<mark>This is the draft for ballot #5773 for RP 10B-2, 3rd Edition</mark>

Testing Well Cements

1   Scope

This standard specifies methods and gives recommendations for the testing of cement slurries and related materials under simulated well conditions.

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 Specification 10A, *Specification for Cements and Materials for Well Cementing*

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

ASTM C109/C109M[1], *Standard Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2 in. or [50 mm] Cube Specimens)*

ASTM C188, *Standard Test Method for Density of Hydraulic Cement*

3   Terms, Definitions, and Symbols

3.1   Terms and Definitions

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

3.1.1
density
absolute density
The mass of a material per unit of volume, not including the intergranular space in the case of a solid.

NOTE 1   Density of a material can be obtained multiplying the relative density of a material by the density of water at 4 °C, 1000 kg/m^3 (8.345 lbm/gal).

3.1.2
absolute volume
The volume occupied by a material, not including the intergranular space in the case of a solid.

NOTE   Absolute volume corresponds to the inverse of the density.

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[1]   ASTM International, 100 Barr Harbor Drive, West Conshohocken, Pennsylvania 19428; www.astm.org.

3.1.3
additive
Material incorporated in a cement slurry to modify or enhance some desired property.

3.1.4
annulus
Space between the pipe and the wellbore wall or an outer pipe.

3.1.5
atmospheric consistometer
Device used to condition fluids prior to some tests and for determining the thickening-time of artic slurries at atmospheric pressure.

3.1.6
batch mix
Process of mixing the entire volume of cement slurry prior to placement in the wellbore.

3.1.7
Bearden units of consistency
B_c
Units used to express consistency of a cement slurry when determined using a pressurized consistometer.

3.1.8
bottomhole circulating temperature
T_{BHC}
Pseudo-stable temperature found at the bottom of the wellbore while the well is being circulated.

3.1.9
bulk density
Mass per unit of volume of a dry material including the air between particles.

3.1.10
cement
Portland cement
Material formed by the grinding of clinker, generally consisting of hydraulic calcium silicates and aluminates and usually containing one or more of the forms of calcium sulfate added during grinding.

NOTE   Clinker: fused materials from the kiln.

3.1.11
cement blend
Mixture of dry cement and other dry materials.

3.1.12
cement class
Designation of classifications of well cement according to defined specifications of API Specification 10A.

3.1.13
compatibility
Capacity to form a fluid mixture that does not undergo undesirable chemical and/or physical reactions.

3.1.14
compressive strength
Strength of a set cement sample measured by the force required to cause it to fail in compression, expressed as force per unit area.

3.1.15
pressurized consistometer
Device used to measure the thickening-time of a cement slurry and also, to condition fluids prior to some tests under temperature and pressure.

3.1.16
continuous-pumping squeeze-cementing
Squeeze-cementing operation that does not involve interruptions in pumping.

3.1.17
drilling fluid
mud
Fluid, generally viscosified and possibly weighted, used to remove cuttings, cool the drill bit, and prevent formation fluids from entering the wellbore during drilling or workover operations.

3.1.18
equivalent sack
Mass of the blend of fly ash or pozzolan and Portland cement that has the same absolute volume as a sack (1 ft^3 ⁰ 3.59 gal) of Portland cement.

NOTE   Only used in U.S. customary (USC) measurements.

3.1.19
filtrate
Liquid that is forced out of a cement slurry during a fluid-loss test.

3.1.20
fly ash
Finely divided residue from the combustion of ground or powdered coal with pozzolanic properties.

NOTE   See Section 14 for further description.

3.1.21
free fluid
Colored or colorless liquid that has separated from a cement slurry when standing in a static condition.

3.1.22
freeze-thaw cycle
A prescribed cycle of exposure of a cement sample to temperatures above and below the freezing point of water or that of the cement filtrate.

3.1.23
heat-up rate
R_h
Rate of slurry temperature change from the surface slurry temperature, T_{SS}, to the predicted bottomhole circulating temperature, T_{PBHC}.

3.1.24
hesitation-pumping squeeze-cementing
Squeeze-cementing operation that involves alternate pumping and non-pumping periods.

3.1.25
load frame
Frame to place a load on set cement samples for determining material properties (i.e. Young's modulus and Poisson's ratio) or failure criteria (i.e. compressive strength and tensile strength) or both.

3.1.26
mix fluid
Mix water containing solid or liquid (or both) additives.

3.1.27
mix water
Fresh water or sea water without solid or liquid (or both) additives.

3.1.28
Non-aqueous fluid
NAF
NAF is a non-aqueous drilling fluid or well circulating fluid. Common NAF systems are diesel, mineral oil, or synthetic fluid-based invert emulsions, or other non-water based fluids.

NOTE   Non-aqueous drilling fluids are also named using symbol NADF

3.1.29
plug cementing
Process of placing a volume of cement in a well to form a plug across the wellbore.

3.1.30
pozzolan
Siliceous or siliceous and aluminous material that, in itself, possess little or no cementitious value but, in finely divided form and in the presence of moisture, reacts with calcium hydroxide to form a cementitious material.

NOTE   See Section 14 for further description.

3.1.31
preflush
Fluid that is not viscosified nor densified with insoluble, solid weighting agents.

3.1.32
pressure-down rate
R_{pd}
Rate at which pressure is reduced from the bottomhole pressure, p_{BH}, to the pressure at the top-of-cement column, p_{TOC}, during a thickening-time test.

3.1.33
pressure-up rate
R_{pu}
Rate at which pressure is increased from the starting pressure, p_{S}, to the bottomhole pressure, p_{BH}, during a thickening-time test.

3.1.34
pressurized curing chamber
Apparatus used for curing a sample of cement under temperature and pressure for subsequent tests such as compressive strength, sedimentation, etc.

3.1.35
pressurized fluid density balance
Beam-type balance used to measure fluid density under pressure to minimize the effects of air entrained in the fluid.

3.1.36
relative density
specific gravity (SG)
Ratio of the mass of a substance to the mass of an equal volume of a standard substance at a reference temperature.

NOTE   The standard substance is usually water; the reference temperature is usually 4 °C.

3.1.37
sack
A unit of measure of cement that occupies a bulk volume of one cubic foot (3.59 gallons).

NOTE 1   Normally 94 lbm for Portland cement.

NOTE 2   Only used in USC measurements.

3.1.38
sedimentation
Separation and settling of solids in a cement slurry.

3.1.39
slurry container
Container used to hold the slurry in a consistometer for conditioning or testing.

3.1.40
slurry stability test
Test to determine the degree of sedimentation and, or free fluid development in a cement slurry.

3.1.41
sonic strength
Compressive strength of a cement sample obtained by measuring the velocity of sound through the cement and computing the strength using a correlation to compressive strength measurements.

3.1.42
spacer
Viscosified fluid that may be densified with insoluble, solid weighting agents and used to separate drilling fluids and cement slurries during cementing operations.

3.1.43
squeeze-cementing
Remedial process in which cement slurry is forced under pressure into a specific portion of the well such as a fracture or opening with the intent to form a bridge of dehydrated cement slurry.

3.1.44
starting pressure
p_S
Initial pressure applied to the cement slurry at the beginning of the thickening-time test.

3.1.45
static fluid-loss test
Test to determine filtrate lost from a cement slurry when placed against a 45 µm (325 US mesh) screen at 6900 kPa (1000 lbf/in.²) differential pressure.

3.1.46
stirred fluid-loss apparatus
Apparatus specially designed to allow for conditioning of the cement slurry within the same cell used to perform a static fluid-loss test.

3.1.47
Thickening-time
Time elapsed from the initial application of pressure and temperature while stirring to the time at which the slurry reaches a consistency deemed sufficient to make it unpumpable (e.g. 70 B_c or 100 B_c) under the test conditions.

3.1.48
transit time
TT
Transit time (sometimes referred to as compressional sonic slowness) is a measurement of sonic velocity that starts when the transmitter is fired and ends when a determined amplitude peak from the waveform arrives at the receiver.

NOTE 1   Transit time is typically expressed in microseconds per inch.

NOTE 2   Transit time is a measurement of sonic velocity and shall not be confused with travel time. Some apparatus may report a travel time through the cement sample and this time shall be converted into transit time by dividing it by the distance between the two sonic sensors.

3.1.49
well-simulation test
Test whose parameters are designed and modified as required to simulate the conditions found in a wellbore.

3.2   Symbols and Abbreviations

3.2.1   Symbols

For the purposes of this document, the symbols given in Table 1 apply. This list is not exhaustive.

Table 1—Symbols

Symbol	Meaning	Units
B_c	consistency	Bearden units
$c_{ad \cdot sol}$	solid additive concentration	percent by BWOC
$d_{rel,i}$	relative density (specific gravity) of cement segment (i)	dimensionless
h_{TOCTVD}	top-of-cement true vertical depth	meters (feet)
h_{TVD}	true vertical depth	meters (feet)
k	consistency index	$Pa \cdot s^n$ ($lbf \cdot s^n/100$ ft²)
m	mass	kilograms (pounds-mass)
$m_{ad.liq}$	liquid additive mass (volume $V_{ad.liq}$)	kilograms per metric ton of cement (pounds-mass per sack)
$m_{ad.liq,600}$	liquid additive mass for 600 mL of slurry	grams
$m_{ad.sol}$	solid additive mass concentration	kilograms per metric ton of cement (pounds-mass per sack)
$m_{ad.sol,600}$	solid additive mass for 600 mL of slurry	grams
$m_{air,i}$	mass of cement segment (i) in air	grams
$m_{bld,sk-eq}$	mass weight of one equivalent sack (3.59 gal) of blend	pounds-mass
m_c	mass of 1 metric ton or 1 sack of cement	1000 kilograms or normally 94 pounds-mass
$m_{cmt,sk}$	mass weight of one sack (3.59 gal) of cement	pounds-mass
$m_{c,600}$	cement mass for 600 mL of slurry	grams
m_{dw}	mass of deionized or distilled water	grams
m_{lfm}	mass of 100 mL of loosely filled material	grams
$m_{poz,sk}$	mass weight of one sack (3.59 gal) of pozzolan	pounds-mass
m_s	slurry mass	kilograms per metric ton of cement (pounds-mass per sack)
$m_{s,600}$	mass of 600 mL of slurry	grams
m_w	mix water mass	kilograms per metric ton of cement (pounds-mass per sack)
$m_{w,600}$	mix water mass for 600 mL of slurry	grams
$m_{water,i}$	mass of cement segment (i) in water	grams
n	flow behavior index	dimensionless
n_s	total number of sedimentation test sample segment	dimensionless
p_{BH}	bottomhole pressure [a]	kilopascals (pounds-force per square inch)
p_{FSQ}	final bottomhole squeeze-cementing pressure	kilopascals (pounds-force per square inch)
p_S	starting pressure	kilopascals (pounds-force per square inch)
p_{SQ}	maximum surface squeeze-cementing pressure	kilopascals (pounds-force per square inch)
p_{TOC}	top-of-cement pressure	kilopascals (pounds-force per square inch)
q	pump rate	cubic meters per minute (barrels per minute or cubic feet per minute)
R_{pd}	rate of pressure decrease	kilopascals per minute (pounds-force per square inch and minute)

Symbol	Meaning	Units
R_{pu}	rate of pressure increase	kilopascals per minute (pounds-force per square inch and minute)
R_{PSQ}	rate of pressure increase to apply the squeeze-cementing pressure	kilopascals per minute (pounds-force per square inch and minute)
$R_{\Delta T}$	rate of temperature change (heat-up or cool-down)	degrees Celsius per minute (degrees Fahrenheit per minute)
T	torque	gram·centimeters
TT	transit time (acoustic slowness)	microseconds per inch
T_{BHC}	bottomhole circulating temperature	degrees Celsius (degrees Fahrenheit)
T_{BHS}	bottomhole static temperature	degrees Celsius (degrees Fahrenheit)
T_{PBHC}	predicted bottomhole circulating temperature	degrees Celsius (degrees Fahrenheit)
T_{PSP}	predicted bottomhole squeeze-cementing or plug-cementing temperature	degrees Celsius (degrees Fahrenheit)
T_{SS}	slurry surface temperature	degrees Celsius (degrees Fahrenheit)
T_{TOCC}	top-of-cement circulating temperature	degrees Celsius (degrees Fahrenheit)
T_{TOCS}	top-of-cement static temperature	degrees Celsius (degrees Fahrenheit)
t	elapsed time	minutes
t_a	time to displace the leading edge of the cement slurry from bottom of the casing to the top of the annular column	minutes
t_{cd}	cool-down time	minutes
t_d	time to displace the leading edge of cement slurry from the surface to the bottom of the wellbore	minutes
t_{sq}	time to apply squeeze-cementing pressure to cement slurry after bottomhole placement	minutes
V_F	volume of free fluid	milliliters
V_M	volume at a receiver specific mark	milliliters
V_a	annular volume of cement	cubic meters (barrels or cubic feet)
$V_{ad.liq}$	liquid additive volume concentration	liters per metric ton of cement (gallons per sack)
$V_{ad.sol}$	solid additive volume (mass $m_{ad.sol}$)	liters per metric ton of cement (gallons per sack)
V_c	volume of 1 metric ton or 1 sack of cement	liters (gallons)
V_{fp}	final packed volume	milliliters
V_p	volume of the pipe	cubic meters (barrels)
V_s	slurry yield (surface conditions)	liters per metric ton of cement (cubic feet per sack)
$V_{s,dh}$	slurry yield at downhole conditions	liters per metric ton of cement (cubic feet per sack)
V_t	volume of filtrate collected at the time nitrogen blows through	milliliters
V_w	mix water volume	liters per metric ton of cement (gallons per sack)
V_{30}	volume of filtrate collected at 30 min	milliliters
Z	acoustic impedance	megarayls
γ	shear rate	reciprocal seconds (s^{-1})
ΔT_P	pseudo-temperature gradient [b]	degrees Celsius per 100 meters (degrees Fahrenheit per 100 feet)
$\Delta\rho_{sc,i}$	density variance of the set cement segment (i)	percent

Symbol	Meaning	Units
μ_p	plastic viscosity	pascal•seconds (centipoises)
$\rho_{ad.liq}$	density of liquid additive	kilograms per liter (pounds-mass per gallon)
$\rho_{ad.sol}$	density of solid additive	kilograms per liter (pounds-mass per gallon)
ρ_{af}	density of annular fluid	kilograms per liter (pounds-mass per gallon)
ρ_{bulk}	average bulk density	kilograms per cubic meter (pounds-mass per cubic feet)
ρ_c	density of cement	kilograms per liter (pounds per gallon)
ρ_{dw}	density of deionized/distilled water	grams per milliliter
ρ_{df}	density of drilling fluid	kilograms per liter (pounds per gallon)
ρ_{LAB}	loose apparent bulk density	kilograms per cubic meter (pounds-mass per cubic feet)
ρ_{PAB}	packed apparent bulk density	kilograms per cubic meter (pounds-mass per cubic feet)
$\rho_{s,m}$	density of slurry measured after mixing	kilograms per liter (pounds-mass per gallon)
ρ_s	density of slurry	kilograms per liter (pounds-mass per gallon)
$\rho_{s,dh}$	density of slurry at downhole conditions	kilograms per liter (pounds-mass per gallon)
ρ_{set}	density of the set cement	kilograms per liter (pounds-mass per gallon)
$\rho_{set,i}$	density of set cement segment (i)	kilograms per liter (pounds-mass per gallon)
ρ_w	density of mix water	kilograms per liter (pounds-mass per gallon)
τ	shear stress	pascals (pounds-force per 100 square feet)
τ_y	yield stress	pascals (pounds-force per 100 square feet)
Φ_{cmt}	ratio of cement in the pozzolan:cement blend	percent
Φ_{poz}	ratio of pozzolan in the pozzolan:cement blend	percent
φ	fluid volume fraction	percent
$v_{ad.abs}$	solid additive absolute volume	gallons per pound
$v_{c.abs}$	cement absolute volume	gallons per pound

a   Hydrostatic pressure at the bottom of the well, calculated using the true vertical depth and the fluid densities in the wellbore.
b   Gradient in °C/100 m (°F/100 ft), calculated from the difference between the maximum recorded bottomhole static temperature and 27 °C (80 °F).

3.2.2   Abbreviations

BWOB   by weight of blend

BWOC   by weight of cement

BWOW   by weight of water

CAS   Chemical Abstracts Service [division of ACS (American Chemical Society)]

HPHT   high pressure, high temperature

LWS   lightweight cement slurry

NAF   non-aqueous fluid

NADF   non-aqueous drilling fluidSSST   spacer surfactant screening test

PPE   personal protective equipment

SG   relative density (specific gravity)

4 Sampling

4.1 General

Samples of the cement, cement blend, solid and liquid additives, and mix water may be required to test a cement slurry in accordance with this standard. Some commonly used sampling techniques are described in this section.

4.2 Sampling Cement at a Field Location

Multiple samples shall be extracted using a suitable device (Figure 1). A composite of the samples shall be prepared, packaged, and labeled (see 4.7). Sample volume shall be sufficient to perform the desired testing.

4.3 Sampling Cement Blends at a Blending Facility

Cement blends may be sampled from the weigh batch mixer (scale tank), bulk transport, or extracted from the flow lines during transfer. The cement and dry additives shall be thoroughly blended prior to sampling. Samples extracted from a flow line during a transfer are preferred and may be taken from a properly installed sample valve, diverted flow sampler, or automatic in-line sampling device [Figure 1 c), d), and e)] using a procedure designed to give a representative sample of the blend being transferred. The samples shall be prepared, packaged, and labeled (see 4.7). Sample volume shall be sufficient to perform the desired testing.

NOTE   Weight batch mixer (scale tank) are devices or systems for the weighing and blending of cement with dry additives for a cementing operation.

4.4 Sampling Dry Cement Additives

Dry cement additive samples may be extracted from a bulk container or sack. Multiple samples should be extracted from the center of the source using a suitable sampling device [Figure 1 a) or b)]. A composite of the samples from the same lot shall be prepared, packaged, and labeled (see 4.7). The volume of each dry cement additive sample shall be sufficient to perform the desired testing.

4.5 Sampling Liquid Cement Additives

Most liquid additives are solutions or suspensions of dry materials. Storage can allow separation of the active ingredients; thus, the active ingredients may float to the top of the container, be suspended as a phase layer, or settle to the bottom. For these reasons, liquid additives shall be thoroughly mixed prior to sampling. The sample shall be extracted from the center of the container using a clean, dry sampling device. A composite of the samples from the same lot shall be prepared, packaged, and labeled (see 4.7). The volume of each liquid additive sample shall be sufficient to perform the desired testing.

4.6 Sampling Mix Water

The mix water shall be sampled from the source or from the tank in which it is stored just prior to mixing. The sample shall be extracted in such a way as to avoid contamination. The sample shall be packaged and labeled (see 4.7). The sample volume shall be sufficient to perform the desired testing.

Dimensions in millimeters (inches) unless otherwise indicated.

b)   Tube Sampler for Bulk Cement

a)   Tube Sampler for Sacked Cement

c)   Automatic Probe Sampler

d)   Modified Diverted-flow Sampler

e)   Top View—Lateral Sampler

Key

1	handle	5	valve
2	dragg tubing	6	flow direction
3	sample tube, extended	7	valve
4	product discharge		
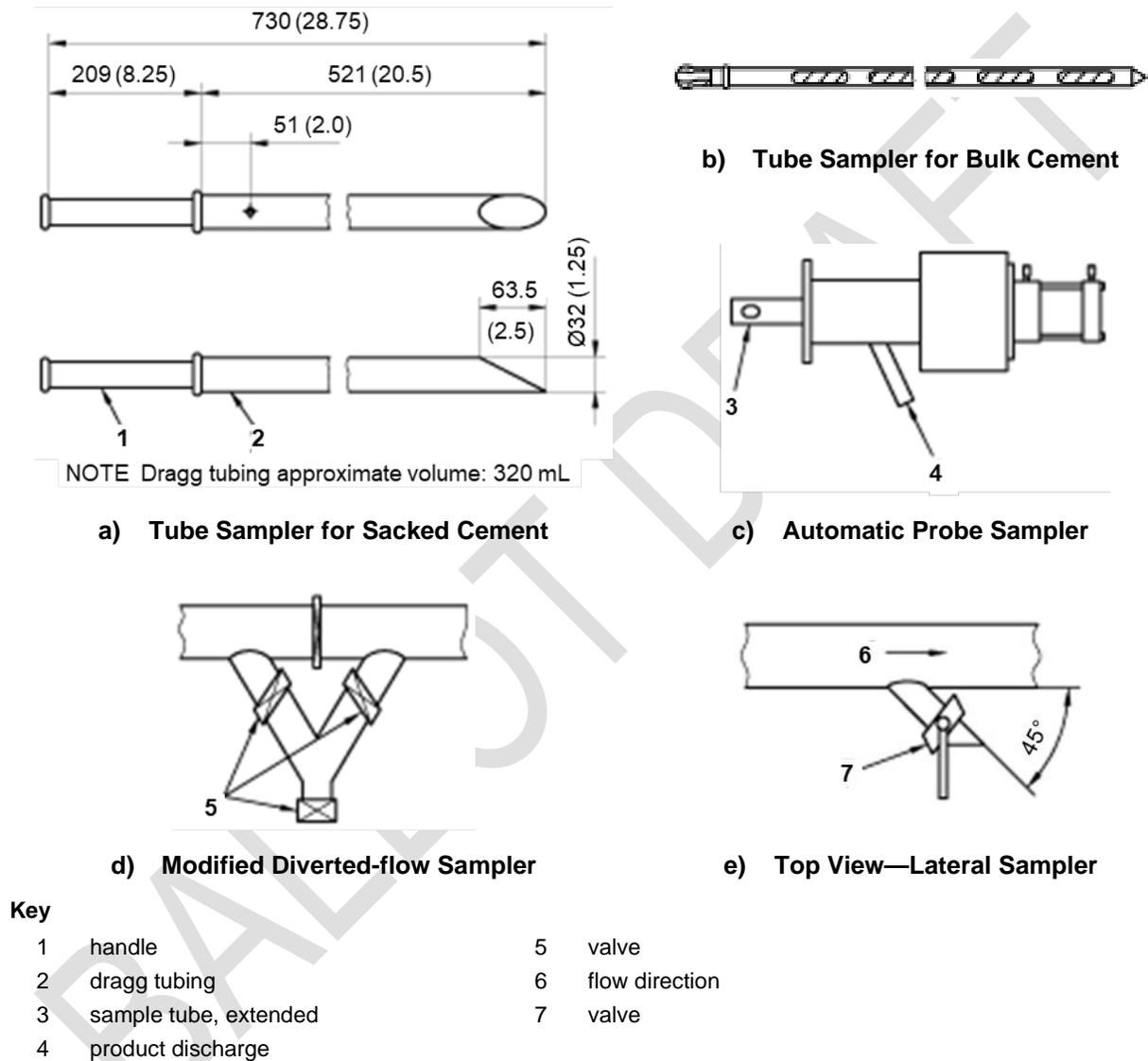
Figure 1—Commonly Used Sampling Devices

4.7 Shipping and Storage

Test samples shall be packaged promptly in unused, clean, airtight, moisture-proof containers suitable for shipping and long-term storage. The containers shall be lined metal, plastic, or other heavy-walled material to assure maximum protection. Resealable plastic bags may be used provided the bag is placed in a protective container prior to shipping to prevent puncturing and to contain all material that may leak out

during shipping. Ordinary cloth sacks, cans, or jars shall not be used. Shipping in glass containers is not recommended.

The body of each blend container shall be clearly labeled and identified with the type of material, lot number, source, and date of sampling. Shipping containers shall also be labeled. The lids of containers shall not be marked since the lids can be readily interchanged and leading to confusion. Any required regulatory identification or documentation shall be enclosed or securely attached to the container. All hazardous material samples shall be packaged and labeled in accordance with all regulatory requirements.

4.8   Sample Preparation Prior to Testing

Upon arrival at the testing location, the samples shall be closely examined to ensure they have remained sealed during shipment and are not contaminated.

If the sample was not shipped in a leak-proof container, the sample shall be transferred into a suitable, leak-proof container. The container shall be properly labeled, dated, and stored in a dry place where room temperatures remain fairly constant. Repackaging shall be indicated. At the time of testing, each sample shall be examined for quality and thoroughly blended just prior to slurry preparation.

Optimum shelf life(s) for all samples shall be determined by the supplier or manufacturer. If unknown, use of any cement additive that has been stored for longer than one year is not recommended.

4.9   Sample Disposal

Sample disposal shall comply with all regulatory requirements.

5   Preparation of Slurry

5.1   General

The preparation of cement slurries varies from that of classical solid/liquid mixtures due to the reactive nature of cement. Shear rate and time at shear are important factors in the mixing of cement slurries. Varying these parameters has been shown to affect slurry performance properties.

The procedure described in this section is recommended for the laboratory preparation of slurries that require no special mixing conditions. If large slurry volumes are needed for a test series, the alternative method for slurry preparation given in Annex A may be used. Annex G (informative) describes alternative procedures which should be used to prepare cement slurries with "fragile" additives such as lightweight microspheres or cement slurries with excess foaming tendencies requiring de-airing.

5.2   Apparatus

5.2.1   General

All apparatuses shall be calibrated as per Annex B.

5.2.2   Balances

Electronic balances shall have, at a minimum, a two (2) decimal place precision reading and conform to requirements specified in Annex B.

Mechanical balances may be used. Weight sets use daily for routine weighing shall conform to requirements specified in annex B.

5.2.3   Mixing device

The mixing device for the preparation of well cement slurries shall be a 1 L (1 qt) size, bottom-drive, blade-type mixer, with a timing device able to measure mixing times and preset times of 15 s (optional) and 35 s. The mixing device shall be able to mix cement slurry at 4000 r/min ±250 r/min rotational speed, and at 12,000 r/min ±500 r/min rotational speed.

An example of a mixing device in common use for preparation of slurries is shown in Figure 2. The blender container and the blender blade, (Figure 3, key 2), shall be constructed of durable, corrosion-resistant material. The mixing-blade assembly (Figure 3) shall be constructed in such manner that the blade can be removed for weighing and changing. The blade shall be mounted with its sharp edge on the leading-edge rotation. According to Annex B, the mixing blade shall be weighed initially and replaced with an unused blade if observed blade deformation has occurred or after blade has lost 10% of its mass. If the mixing device leaks at any time during the mixing procedure, the contents shall be discarded, the leak repaired, and the procedure restarted.

Figure 2—Example of a Typical Cement mixing Device

Key
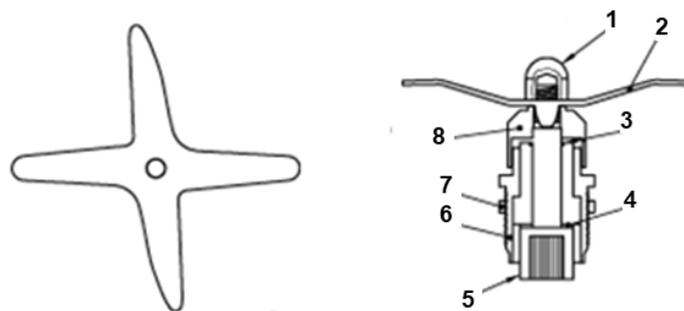
1 cap nut
2 blade (installed with tapered edge down)
3 O-ring
4 thrust washer

5 socket head shaft
6 bearing holder
7 hexagonal nut
8 bearing cap

Figure 3—Common Blade Assembly

5.2.4 Pressurized consistometer or Atmospheric consitometer

Any pressurized consistometer as referenced in Section 9 or Annex C and alternatively an atmospheric consistometer as referenced in API Specification 10A may be used for cement slurry conditioning.

5.2.5 Temperature measuring device

A thermometer or a thermocouple with a minimum range from -20 °C to 105 °C (0 °F to 220 °F), with minimum scale divisions not exceeding 1 °C (2 °F) or a thermocouple with a minimum range of -20 °C to 120 °C (70 °F to 400 °F) may be used as in an atmospheric consistometer.

A thermocouple of range -20 °C up to at least 205 °C (0 °F to at least 400 °F) or above the range limitation of the equipment, shall be used in a pressurized consistometer

5.2.6 Timer

A stopwatch or electric timer with time indicated in minutes and seconds shall be used.

NOTE: Timer incorporated into mixing device shall meet Annex B requirements.

5.3 Procedure

5.3.1 Determination of density of components

5.3.1.1 General

The density of different batches of cement can vary due to natural variations in the composition of the raw materials used in the manufacturing process. Cement density may vary from 3100 kg/m^3 to 3250 kg/m^3 (25.87 lbm/ft^3 to 27.12 lbm/ft^3). This can result in variation in slurry densities by as much as 34 kg/m^3 (0.28 lbm/gal) for slurries with constant water-to-solids ratios. The density of mix water can also vary, depending on the source, resulting in slurry density inconsistencies. Determination of the density of all components of the slurry shall be necessary to properly calculate the required amounts for slurry preparation.

5.3.1.2   Density of cement and dry additives

The density of the cement and any dry additives can be determined by the use of a gas pycnometer (also known as stereopycnometer). Alternatively, a Le Chatelier flask as outlined in ASTM C188 may be used for determining the density of these materials.

5.3.1.3   Density of mix water and liquid additives

The density or relative density of the mix water and any liquid additives may be determined by the use of a hydrometer as outlined in API 13J. Alternatively, a pycnometer may be used for determining the density of these materials.

NOTE   Two types of hydrometers can be used, one type is directly measuring a fluid density (see 3.1.1), and the second one is measuring a relative density (see 3.1.36).

5.3.2   Laboratory slurry design calculations

5.3.2.1   General

For the purpose of laboratory slury design calculations one cubic centimeter is equivalent to one milliliter. Fresh water density at 23 °C (73 °F) is considered at 0.9976 kg/L (8.325 lbm/gal).

NOTE   Fresh water, i.e. tap water, density is denser than deionized water [0.9975 kg/L at 23 °C].

Laboratory cement, additives and mix water requirements (see 5.3.3) may be calculated by use of the formulas based on slurry design recipes Equation (21) and, in SI units Equations (22) to (26) or in USC units Equations (27) to (31). Alternative, suitable equations may also be used to calculate laboratory requirements.

Slurry composition is based on additive concentrations expressed for solids in kilograms per metric ton of cement (1 metric ton = 1000 kg) or in percent by weight of cement (BWOC) and for liquids in liters per metric ton of cement (gallon par sack of cement, see 3.1.39).

In SI units, based on a slurry design that includes solid additives or liquid additives or both, calculations are provided under 5.3.2.2 [Equations (1) to (8)], mix water requirement, if not given, is calculated Equation (9) or (10). The slurry yield (V_s) is calculated based on Equation (3)

In USC units, based on a slurry design that includes solid additives or liquid additives or both, calculations are provided under 5.3.2.3 [Equations (11) to (18)], mix water requirement, if not given is calculated Equation (19) or (20). The slurry yield (V_s) is calculated based on Equation (13).

NOTE   Design slurry calculations and equations based on design concentrations by weight by metric ton of cement or in percent   BWOC and volume by metric ton of cement (sack of cement) are also applicable to slurry design with concentrations expressed by weight or in percent by weight of blend (BWOB) and volume per metric ton of blend (sack of blend, sack weight shall be defined).

Remark—Sodium chloride additive in cement slurry

The measurement of sodium chloride (NaCl) as an additive in cement slurry is normally defined in percent by weight of water (BWOW). Other salts (e.g. calcium chloride, potassium chloride) are defined in percent BWOC unless noted otherwise by the practitioner.

For testing purposes if sodium chloride is included in a slurry formulation, it is recommended to consider mix water as a sodium chloride brine and to calculate mix water mass from brine density and brine volume

requirement. An alternate method to calculate the sodium chloride in the slurry is to use a graph or table containing an equivalent relative density [specific gravity (SG)] for the actual sodium chloride when it is in solution. Using this method, sodium chloride can be treated as any other additive as long as the concentration is BWOW.

5.3.2.2 SI units design calculations

Density of slurry, ρ_s, expressed in kilograms per liter shall be given Equation (1);

$$\rho_s = \frac{m_s}{V_s}$$ (1)

where

m_s is the slurry mass prepared with one metric ton (1000 kg) of cement (including mix water and additives), expressed in kilograms per metric ton of cement, as given Equation (2)

$$m_s = m_c + m_w + \sum m_{ad.liq} + \sum m_{ad.sol}$$ (2)

and

V_s is the slurry yield prepared with one metric ton (1000 kg) of cement, in liters per metric ton of cement, as given by Equation (3)

$$V_s = V_c + V_w + \Sigma V_{ad.liq} + \Sigma V_{ad.sol}$$ (3)

where respectively for Equation (2) and Equation (3)

m_c is the cement mass of one metric ton (1000 kg) of cement, expressed in kilograms;

V_c is the cement volume of one metric ton of cement, expressed in liters;

m_w is the mix water mass, expressed in kilograms per metric ton of cement;

V_w is the mix water volume, expressed in liters per metric tonn of cement;

$\Sigma m_{ad.liq}$ is the sum of masses for all liquid additives ($V_{ad.liq}$), expressed in kilograms per metric ton of cement;

$\Sigma V_{ad.liq}$ is the sum of volume concentrations for all liquid additives, expressed in liters per metric ton of cement;

$\Sigma m_{ad.sol}$ is the sum of mass concentrations for all solid additives, expressed in kilograms per metric ton of cement;

$\Sigma V_{ad.sol}$ is the sum of volumes for all solid additives ($m_{ad.sol}$), expressed in liters per metric ton of cement.

Solid additives can be also expressed by the solid additive concentration, $c_{\text{ad.sol}}$ expressed in percent BWOC. In that case for each solid additive mass of additive per metric ton of cement (m_c = 1000 kg) is given Equation (4):

$$m_{\text{ad.sol}} = 0.01 \times m_c \times c_{\text{ad.sol}} = 10 \times c_{\text{ad.sol}} \tag{4}$$

Masses are related to volumes and volumes to masses:

Mass of mix water, m_w, expressed in kilograms per metric ton of cement is given Equation (5):

$$m_w = V_w \times \rho_w \tag{5}$$

where

ρ_w is the density of mix water, expressed in kilograms per liter.

Volume of one metric ton of cement, V_c (mass m_c), is given Equation (6):

$$V_c = \frac{m_c}{\rho_c} = \frac{1000}{\rho_c} \tag{6}$$

where

ρ_c is the density of cement, expressed in kilograms per liter.

For each additive, liquid or solid:

Mass of a liquid additive, $m_{\text{ad.liq}}$, expressed in kilograms per metric ton of cement is given Equation (7)

$$m_{\text{ad.liq}} = V_{\text{ad.liq}} \times \rho_{\text{ad.liq}} \tag{7}$$

where

$V_{\text{ad.liq}}$ is the liquid additive volume concentration, expressed in liters per metric ton of cement;

$\rho_{\text{ad.liq}}$ is the density of the liquid additive, expressed in kilograms per liter;

and volume of a solid additive, $V_{\text{ad.sol}}$, expressed in liters per metric ton of cement is given Equation (8) :

$$V_{\text{ad.sol}} = \frac{m_{\text{ad.sol}}}{\rho_{\text{ad.sol}}} = \frac{10 \times c_{\text{ad.sol}}}{\rho_{\text{ad.sol}}} \tag{8}$$

where

$m_{\text{ad.sol}}$ is the solid additive mass concentration, expressed in kilograms per metric ton of cement;

$c_{\text{ad.sol}}$ is the solid additive concentration, expressed as a percentage BWOC

$\rho_{\mathrm{ad.sol}}$ is the density of the solid additive, expressed in kilograms per liter

When the mix water requirement is not provided, mix water volume requirement, V_{w}, expressed in liters per metric ton of cement (mass $m_{\mathrm{c}} = 1000$ kg) is calculated solving Equation (1) using Equations (2) to (8). Mix water requirement shall be given Equation (9).

$$V_{\mathrm{w}} = \frac{m_{\mathrm{c}}\left(1 - \rho_{\mathrm{S}}/\rho_{\mathrm{c}}\right) - \Sigma\left[V_{\mathrm{ad.liq}}\left(\rho_{\mathrm{S}} - \rho_{\mathrm{ad.liq}}\right)\right] + \Sigma\left[m_{\mathrm{ad.sol}}\left(1 - \rho_{\mathrm{S}}/\rho_{\mathrm{ad.sol}}\right)\right]}{\left(\rho_{\mathrm{s}} - \rho_{\mathrm{w}}\right)} \tag{9}$$

which is resumed with fresh water at 23 °C (73 °F) (density 0.9976 kg/L) in Equation (10):

$$V_{\mathrm{w}} = \frac{1000 \times \left(1 - \rho_{\mathrm{S}}/\rho_{\mathrm{c}}\right) - \Sigma\left[V_{\mathrm{ad.liq}}\left(\rho_{\mathrm{S}} - \rho_{\mathrm{ad.liq}}\right)\right] + \Sigma\left[m_{\mathrm{ad.sol}}\left(1 - \rho_{\mathrm{S}}/\rho_{\mathrm{ad.sol}}\right)\right]}{\left(\rho_{\mathrm{S}} - 0.9976\right)} \tag{10}$$

where

$V_{\mathrm{ad.liq}}$ is the volume concentration of a liquid additive, expressed in liters per metric ton of cement (1000 kg); all liquid additives shall be considered by the calculation;

$m_{\mathrm{ad.sol}}$ is the mass concentration of a solid additive, expressed in kilograms per metric ton of cement (1000 kg) or calculated by Equation (4) and solid additive concentration, $c_{\mathrm{ad.sol}}$; expressed in percent; all solid additives shall be considered by the calculation;

ρ_{s} is the density of slurry, expressed in kilograms per liter;

ρ_{c} is the density of cement, expressed in kilograms per liter;

ρ_{w} is the density of mix water, expressed in kilograms per liter;

$\rho_{\mathrm{ad.liq}}$ is the density of the liquid additive, expressed in kilograms per liter;

$\rho_{\mathrm{ad.sol}}$ is the density of the solid additive, expressed in kilograms per liter.

Then mix water mass expressed kilograms per metric ton of cement is calculated by Equation (5).

Slurry yield, V_{s}, expressed in liters per metric ton of cement shall be given Equation (3)

5.3.2.3 USC units design calculations

USC equations are based on one sack (one cubic feet $^{\mathrm{o}}$ 3.59 gal) of cement normally considered to be 94 lbm (3.1.39). For a cement blend, sack weight shall be defined by the user and Equation (13) modified accordingly. Density of slurry, ρ_{s}, expressed in pounds-mass per gallon shall be given Equation (11);

$$\rho_s = 0.1337 \times \frac{m_s}{V_s} \tag{11}$$

where

m_s is the slurry mass prepared with one sack of cement (including mix water and additives), as given Equation (12)

$$m_s = m_c + m_w + \sum m_{ad.liq} + \sum m_{ad.sol} \tag{12}$$

and

V_s is the slurry yield prepared with one sack of cement (94 lbm), in cubic feet per sack, as given Equation (13)

$$V_s = 0.1337 \times \left(V_c + V_w + \Sigma V_{ad.liq} + \Sigma V_{ad.sol} \right) \tag{13}$$

where respectively, for Equation (12) and Equation (13)

m_c is the cement mass of one sack of cement, taken to be 94 lbm;

V_c is the cement volume of one sack of cement, expressed in gallons;

m_w is the mix water mass, expressed in pounds-mass per sack of cement;

V_w is the mix water volume, expressed in gallons per sack of cement;

$V_{ad.liq}$ is a liquid additive volume concentration, expressed in gallons per sack of cement, all liquid additives shall be considered by the calculation;

$m_{ad.sol}$ is the mass of a solid additive for one sack of cement based on its concentration, $c_{ad.sol}$, expressed in percent BWOC and given Equation (14), all solid additives shall be considered by the calculation.

where

$$m_{ad.sol} = 0.01 \times m_c \times c_{ad.sol} = 0.94 \times c_{ad.sol} \tag{14}$$

$m_{ad.liq}$ is the mass of the liquid additive, expressed in pounds-mass per sack of cement, all liquid additives shall be considered by the calculation;

$V_{ad.sol}$ is the volume of the solid additive, expressed in gallons per sack of cement, all solid additives shall be considered by the calculation.

NOTE   coefficient 0.1337 is not required in Equations (12) and (13) when cement slurry density is expressed in pounds-mass per gallon and cement slurry yield is expressed in gallons.

Volumes are related to masses, and masses to volumes.

Volume of one sack of cement, V_c, (mass m_c normally taken to 94 lbm) expressed in pounds-mass per sack of cement is given Equation (15):

$$V_c = m_c \times v_{c.abs} = 94 \times v_{c.abs} \tag{15}$$

where

$\quad v_{c.abs}$ is the absolute volume of cement, expressed in gallons per pound-mass;

Volume of mix water, V_w, expressed in gallons per sack of cement is given Equation (16):

$$m_w = V_w \times \rho_w \tag{16}$$

where

$\quad \rho_w$ is the density of mix water, expressed in pounds-mass per gallon;

Mass of a liquid additive, $m_{ad.liq}$, expressed in gallons per sack of cement is given Equation (17)

$$m_{ad.liq} = V_{ad.liq} \times \rho_{ad.liq} \tag{17}$$

$\quad V_{ad.liq}$ is the liquid additive volume concentration, expressed in gallons per sack of cement; all liquid additives shall be considered by the calculation;

$\quad \rho_{ad.liq}$ is the density of the liquid additive, expressed in pounds per gallon

The volume of a solid additive, $V_{ad.sol}$, expressed in gallons per sack of cement (mass m_c normally taken to 94 lbm) is given Equation (18):

$$V_{ad.sol} = 0.01 \times m_c \times c_{ad.sol} \times v_{ad.sol} = 0.94 \times c_{ad.sol} \times v_{ad.sol} \tag{18}$$

where

$\quad c_{ad.sol}$ is the solid additive concentration expressed in percent BWOC.

$\quad v_{ad.abs}$ is the absolute volume of the solid additive, expressed in gallons per pound-mass;

When the mix water requirement is not provided, mix water volume requirement, V_w, expressed in gallons per sack of cement (m_c normally taken to 94 lbm) is calculated solving Equation (11) using Equations (12) to (18). Mix water requirement shall be given Equation (19).

$$V_w = \frac{m_c \times \left[1 - \left(\rho_s \times v_{c.abs}\right)\right] - \Sigma\left[V_{ad.liq}\left(\rho_s - \rho_{ad.liq}\right)\right] + \left(0.01 \times m_c\right) \times \Sigma\left[c_{ad.sol}\left(1 - \left(\rho_s \times v_{ad.abs}\right)\right)\right]}{\left(\rho_s - \rho_w\right)} \tag{19}$$

which is resumed with fresh water at 23 °C (73 °F) (density 8.325 lbm/gal) in Equation (20):

$$V_{\text{w}} = \frac{94 \times \left[1 - \left(\rho_{\text{s}} \times v_{\text{c.abs}}\right)\right] - \Sigma\left[V_{\text{ad.liq}}\left(\rho_{\text{s}} - \rho_{\text{ad.liq}}\right)\right] + 0.94 \times \Sigma\left[c_{\text{ad.sol}}\left(1 - \left(\rho_{\text{s}} \times v_{\text{ad.abs}}\right)\right)\right]}{\left(\rho_{\text{s}} - 8.325\right)}$$

(20)

where

ρ_{s} is the density of slurry, expressed in pounds-mass per gallon;

$v_{\text{c.abs}}$ is the absolute volume of cement, expressed in gallons per pound-mass;

$V_{\text{ad.liq}}$ is a liquid additive volume concentration, expressed in gallons per sack of cement, all liquid additives shall be considered by the calculation;

$\rho_{\text{ad.liq}}$ is the density of the liquid additive, expressed in pounds-mass per gallon;

$c_{\text{ad.sol}}$ is the concentration of a solid additive, expressed in percent BWOC, all solid additives shall be considered by the calculation;

$v_{\text{ad. abs}}$ is the absolute volume of the solid additive, expressed in gallon per pounds-mass;

ρ_{w} is the density of mix water, expressed in pounds-mass per gallon.

Mix water mass expressed pounds-mass per sack of cement is then calculated using Equation (16).

Slurry yield, V_{s}, expressed in cubic feet per sack of cement is given Equation (13).

5.3.3 Laboratory masses—Calculations

5.3.3.1 General

A slurry volume of 600 mL shall be mixed based on mix water, cement, and additives mass requirements expressed in grams.

NOTE   A slurry volume of approximately 600 mL is sufficient to perform most laboratory test procedures while not overfilling the mixing container.

Mass requirements for 600 mL shall be calculated as given Equation (21), based on Equations (1) and (2) expressed in SI units,or Equations (11) and (12) expressed in USC units.

$$m_{\text{s,600}} = m_{\text{c,600}} + m_{\text{w,600}} + \Sigma m_{\text{ad.liq,600}} + \Sigma m_{\text{ad.sol,600}}$$

(21)

where

$m_{\text{s,600}}$ is the mass of 600 mL of slurry to be mixed, expressed in grams

$m_{\text{c,600}}$ is the cement mass to be mixed for 600 mL of slurry, expressed in grams;

$m_{w,600}$ is the mix water mass to be mixed for 600 mL of slurry, expressed in grams;

$\Sigma m_{ad.liq,600}$ is the sum of masses for all liquid additives to be mixed for 600 mL of slurry, expressed in grams;

$\Sigma m_{ad.sol,600}$ is the sum of masses for all solid additives masses to be mixed for 600 mL of slurry, expressed in grams.

5.3.3.2   SI units laboratoty masses calculations

As per slurry composition and yield calculation to mix a 600 mL sample, the mass requirements expressed in grams, shall be calculated as per the Equations (22) to (26).

Mass of cement ($m_{c,600}$) to be mixed as given Equation (22):

$$m_{c,600} = \frac{600}{V_s} \times 1000 \tag{22}$$

into the mass of mix water ($m_{w,600}$) as given Equation (23)

$$m_{w,600} = \frac{600}{V_s} \times V_w \times \rho_w \tag{23}$$

Equation (23) is reduced when mixed with fresh water at 23 °C (density 0.9976 kg/L) to Equation(24):

$$m_{w,600} = \frac{598.56}{V_s} \times V_w \tag{24}$$

and with the masses of each liquid additives ($m_{ad.liq,600}$) as given Equation (25) and each solids additives ($m_{ad.sol,600}$) as given Equation (26):

$$m_{ad.liq,600} = \frac{600}{V_s} \times V_{ad.liq} \times \rho_{ad.liq} \tag{25}$$

$$m_{ad.sol,600} = \frac{600}{V_s} \times m_{ad.sol} \times \rho_{ad.sol} \tag{26}$$

where

V_s is the slurry yield prepared with one metric ton of cement (1000 kg), expressed in liters per metric ton of cement;

V_w is the mix water volume per one metric ton of cement (1000 kg), expressed in liters per metric ton of cement;

ρ_w is the density of mix water, expressed in kilograms per liter;

$V_{\mathrm{ad.liq}}$ is a liquid additive volume concentration per one metric ton of cement (1000 kg), expressed in liters per metric ton of cement;

$\rho_{\mathrm{ad.liq}}$ is the density of a liquid additive, expressed in kilograms per liter;

$m_{\mathrm{ad.sol}}$ is a solid additive mass concentration per one metric ton of cement (1000 kg), expressed in kilograms per metric ton of cement, or as given Equation (4) when solid additive is based on its concentration, $c_{\mathrm{ad.sol}}$, in percent BWOC.

5.3.3.3   USC units laboratoty masses calculations

As per slurry composition and yield calculation, to mix a 600 mL sample, the mass requirements expressed in grams shall be calculated by the following Equations (27) to (31) (based on a 94 lbm sack (3.59 gal) of cement).

Mass of cement ($m_{\mathrm{c,600}}$) to be mixed is given Equation(27)

$$m_{\mathrm{c,600}} = \frac{9.611}{V_{\mathrm{s}}} \times 94 \ = \ \frac{903.4}{V_{\mathrm{s}}} \tag{27}$$

into the mass of mix water ($m_{\mathrm{w,600}}$) given Equation (28)

$$m_{\mathrm{w,600}} = \frac{9.611}{V_{\mathrm{s}}} \times V_{\mathrm{w}} \times \rho_{\mathrm{w}} \tag{28}$$

Equation (27) is reduced when mixed with fresh water at 73 °F (density 8.325 lbm/gal) to Equation (29):

$$m_{\mathrm{w,600}} = \frac{80.01}{V_{\mathrm{s}}} \times V_{\mathrm{w}} \tag{29}$$

and with the masses for each liquid additives ($m_{\mathrm{ad.liq,600}}$) as given Equation (30) and each solids additives ($m_{\mathrm{ad.sol,600}}$) as given Equation (31):

$$m_{\mathrm{ad.liq,600}} = \frac{9.611}{V_{\mathrm{s}}} \times V_{\mathrm{ad.liq}} \times \rho_{\mathrm{ad.liq}} \tag{30}$$

$$m_{\mathrm{ad.sol,600}} = \frac{0.09611}{V_{\mathrm{s}}} \times \ 94 \times \ c_{\mathrm{ad.sol}} = \frac{9.034}{V_{\mathrm{s}}} \times \ c_{\mathrm{ad.sol}} \tag{31}$$

where

V_{s} is the slurry yield prepared with one sack of cement (normally, 94 lbm), expressed in cubic feet per sack of cement;

V_{w} is the mix water volume, expressed in gallons per sack of cement;

ρ_{w} is the density of mix water, expressed in pounds-mass per gallon;

$V_{\text{ad.liq}}$ is aliquid additive volume concentration per one sack of cement, expressed in gallons per sack of cement;

$\rho_{\text{ad.liq}}$ is the density of the liquid additive, expressed in pounds-mass per gallon;

$c_{\text{ad.sol}}$ is the concentration of a solid additive, expressed in percent BWOC.

5.3.4   Temperature of water and cement

The temperature of the mix water, and dry cement or cement blend shall be a representation of field mixing conditions. If field conditions are unknown, the temperature of the mix water (including any premixed additives) and dry cement shall be 23 °C ±1 °C (73 °F ±2 °F) immediately prior to mixing. In all cases, the temperatures of the mix water and dry cement shall be measured and documented.

NOTE   Temperature of mixing and blending devices should be also a representation of field mixing conditions.

5.3.5   Mix water and mix fluid

Water composition can affect slurry performance. Water from the field source shall be used. If field mix water is unavailable, water of similar composition shall be used. If field mix water composition is unknown, deionized, distilled, or tap water may be used. The type of mix water shall be documented in the lab report. The mix water shall be weighed into a clean, dry, blender container. If used, additives may be weighted into the water in the blender container or may be weighed separately and added to the water while agitating at low speed at which a vortex start to be observed without air entrainment. No excess water shall be added to compensate for evaporation or wetting.

5.3.6   Mixing of Cement and Water

A slurry volume of approximately 600 mL shall be sufficient to perform most laboratory test procedures while not overfilling the blender container. Following procedure and instructions shall be applied.

a)   Weigh dry materials and then blend thoroughly and uniformly prior to adding them to the mix fluid.

b)   Place the blender container with the required mass of mix water and liquid additives (if previously added) on the blender base.

c)   Turn on the motor and maintain at 4000 r/min ±250 r/min. If additives are present in the mix water, stir at the above rotational speed to thoroughly disperse them prior to the addition of cement.

    In some cases, additives (dry or liquid) may be added to the mix water in the field. In such cases, the additives shall be added to the mix water while mixing at low speed at which a slight vortex is observed.

    In certain cases, the order of addition of the additives to the mix water can be critical, in which case, the additives shall be mixed in the order that they will be mixed in the field. Document any special mixing procedures and mixing time.

d)   While mixing at 4000 r/min ±250 r/min, add the cement or cement/dry additive blend at a uniform rate in not more than 15 s, if possible.

Some slurry designs may take longer to completely wet the cement blend; however, the time used to add the blend shall be kept to a minimum. If more than 15 s were required to add the cement blend to the water, document that time.

e)   After 15 s or when all the dry materials have been added to the mix water if longer than 15 s, place the cover on the mixing container and continue mixing at 12,000 r/min ±500 r/min for 35 s ±1 s. Measure and document the rotational speed under load.

5.4   Test Fluid Conditioning

5.4.1   General

Conditioning simulates the conditions the test fluid will encounter during placement into the wellbore. Conditioning shall be done according to a schedule that reflects the expected conditions under which the test fluid will be exposed during placement. If possible, the schedule shall use the temperatures and pressures of the well. Alternatively, the conditioning can be done at atmospheric pressure.

5.4.2   Procedure: Pressurized conditioning

The following procedure shall be applied using a pressurized conistometer (see 5.2.4):

a)   After cessation of mixing the slurry according to 5.3.6, pour the test fluid into the slurry container of a pressurized consistometer. Start the test by applying the initial pressure and starting the temperature ramp. No more than 5 min shall have elapsed after cessation of mixing the slurry and the beginning of the test.

b)   Heat to T_{PBHC} in accordance with the pressure/temperature schedule designed to simulate conditions in the well (see 9.4). During the pressure and temperature ramp, the temperature and pressure shall be maintained within ±3 °C (±5 °F) and ±2 MPa (±300lbf/in.²) of the appropriate temperature and pressure target vs elapsed time. Within 10 min after the end of the ramp, temperature and pressure shall be within ±1 °C (±2 °F) and ±0.7 MPa (±100lbf/in.²) of the specified values.

c)   Hold test temperature and pressure for 30 min ±30 s to allow the test fluid temperature to reach equilibrium. This hold time may be modified to simulate cementing operations. Proper note shall be made of this in the test report.

d)   If the temperature is greater than 88 °C (190 °F), cool the test fluid as quickly as possible to 88 °C (190 °F). If the boiling point of water at the testing facility is less than 100 °C (212 °F), adjust temperatures accordingly. Maintain test pressure while decreasing the temperature. When 88 °C (190 °F) is reached, release the pressure slowly [about 1400 kPa/s (200 lbf/in.²•s)].

e)   Remove the slurry container from the consistometer, keeping the container upright so that the oil does not mix with the test fluid. Do not cool the slurry container further after removal from the pressurized consistometer.

f)   Remove the flange ring and the backup plate and syringe or blot oil from the top of the slurry container.

g)   Remove the diaphragm and the support ring. Syringe or blot the top of the test fluid with an absorbent cloth or paper towel to remove any visible oil. If the contamination is severe, discard the test fluid and condition a fresh test fluid.

h)   Remove the paddle and stir the test fluid briskly with a spatula to ensure it is uniform.

i)   Continue with the desired test.

5.4.3   Procedure: Atmospheric-pressure conditioning

Atmospheric consistometer or a pressurized consistometer (see 5.2.4) run at atmospheric pressure may be used. This procedure is limited to a maximum temperature of 88 °C (190 °F). It is preferred that at temperatures above 140 F pressurized conditioning should be used. If the boiling point of water at the testing facility is less than 100 °C (212 °F), adjust conditioning temperatures accordingly. The procedure shall be as per the following.

NOTE   Cement slurry conditioned in an atmospheric consistometer may perform differently than slurry conditioned in a pressurized consistometer regardless of the temperature below the limits of the atmospheric consistometer.

a)   Within 1 min after mixing according to 5.3.6 using test fluid, fill the slurry container of the atmospheric-pressure consistometer to the fill line.

b)   Heat the test fluid from ambient temperature or a temperature that simulates field surface mixing temperature to T_{PBHC} in accordance with the thickening-time schedule that most closely simulates actual field conditions. If the atmospheric consistometer is not capable of heating on a controlled temperature ramp, heat as rapidly as the instrument is capable and record the time to T_{PBHC}. If the atmospheric pressure consistometer is not equipped to measure test fluid temperature, the bath shall be heated in accordance with the appropriate schedule.

c)   With test fluids containing additives that are not affected by sudden temperature increases, the slurry container may be placed in the heating bath preheated to the test temperature [± 3 °C (± 5 °F)] or other initial temperature that is appropriate. Care shall be taken to prevent unusual behavior such as gelation, increase in free fluid, or poor response to additives such as retarders and fluid-loss control agents, when conditioning the fluid.

d)   After the slurry reaches test temperature (the temperature must be verified by measurement), hold the test temperature for 30 min ± 30 s to allow the test fluid temperature to reach equilibrium. This hold time may be modified to simulate cementing operations. However, proper note shall be made of this in the test report.

e)   Remove the paddle and stir the test fluid briskly with a spatula to ensure it is uniform.

f)   Continue with the desired test.

6   Determination of Slurry Density

6.1   Apparatus

The preferred apparatus for measuring the density of a cement slurry should be the pressurized fluid density balance (see Figures 4 and 5). By pressurizing the sample cup, any entrained air is decreased to a negligible volume, thus providing a slurry density measurement more representative of the true slurry density. The apparatus shall be calibrated according to the requirements found in Annex B.

6.2   Procedure

Following procedure shall be used to determine a slurry density using a pressurized fluid density balance (see Figures 4 and 5). The fluid density balance shall be clean and dry.

a)   The sample cup shall be filled to a level slightly below the upper edge of the cup.

b) Place the lid on the cup with the check valve in the down (open) position. Push the lid downward into the mouth of the cup until surface contact is made between the outer skirt of the lid and the upper edge of the cup. Expel excess slurry through the check valve. If slurry does not expel from the check valve, remove lid, add additional slurry. Replace the lid and start again the procedure 6.2 b).

Caution—Slurry can be expelled forcibly, use appropriate personal protective equipment (PPE).

c) After the lid has been placed on the cup, pull the check valve up into the closed position, rinse off the cup and threads with water, and screw the threaded ring on the cup.

d) The pressurizing pump is similar in operation to a syringe. Fill the pump by submerging the nose of the pump assembly in the slurry with the piston rod in the completely downward position. Then draw the piston rod upward, thereby filling the pump cylinder with slurry. Return the piston to the downward position to expel air from the piston and then draw the rod upward to refill the pump cylinder with slurry.

e) Push the nose of the pump onto the mating O-ring surface of the check valve. Pressurize the sample cup by applying a downward force on the pump cylinder housing in order to hold the check valve down (open) and at the same time push the piston rod downward, forcing the slurry into the cup. Maintain approximately 230 N (50 lbf) force or greater on the piston rod (Figure 5 key 6).

f) The check valve in the lid is pressure-actuated, which means the pressure in the cup closes the valve and keeps it closed. Close the valve by gradually lifting the cylinder housing of the pressurizing pump while maintaining pressure on the piston rod. When the check valve closes, release pressure on the piston rod before disconnecting the pump. Check to ensure the valve has closed. Fluid leaking out of the check valve indicates it is not fully closed or the interior O-ring is defective. Correct the problem and restart the test.

g) Rinse off the exterior of the cup and wipe dry. Then place the instrument on the knife edge as illustrated in Figure 5. Move the sliding weight right or left until the beam is balanced. The beam is balanced when the bubble is centered in the sight glass. Obtain the density by reading directly one of the four calibrated scales on the arrow side of the sliding weight**;** density can be read in units of grams per milliliter using relative density (specific gravity) scale, pounds per gallon, pounds per cubic foot, or as a fluid gradient in pounds per square inch per 1000 ft of vertical depth.

h) After the measurement, reconnect the pump assembly and push downward on the pump cylinder housing to release the pressure. Empty the cup and pump assembly and thoroughly clean all components.

Figure 4— Typical Pressurized Fluid Density Balance (balanced at 1.90 SG).
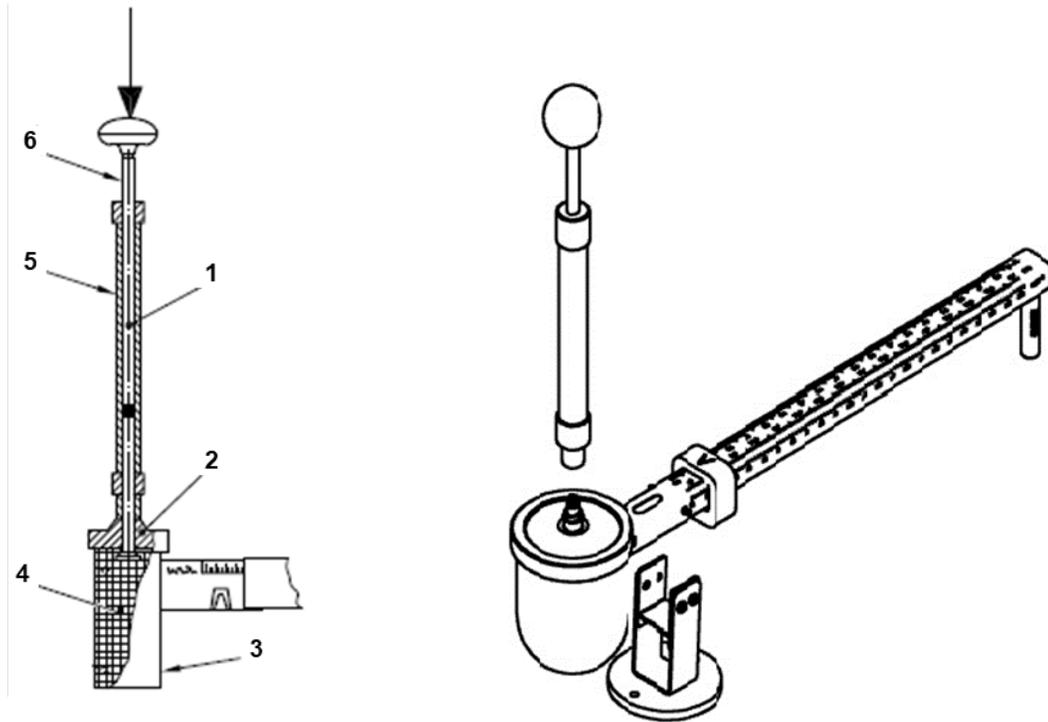
Key

1   pressurizing pump
2   pressurizing valve
3   sample cup
4   slurry sample with entrained air
5   cylinder housing
6   piston rod

Figure 5—Typical Pressurized Fluid Density Balance

7   Well-simulation Compressive-strength Tests

7.1   General

This section presents procedures for well-simulation compressive-strength testing.

7.2   Sampling

Obtain samples of the cement, additives, and mix water in accordance with Section 4.

7.3   Apparatus

7.3.1   General

Unless otherwise noted, all apparatuses shall be calibrated as per the requirements given in Annex B.

Molds and testing machines for compressive strength tests shall conform to the requirements in ASTM C109/C109M, with the exception described in 7.3.2 and 7.3.3.

7.3.2 Cube molds

Cement specimens shall be prepared using 50 mm-cube or 2 in.-cube molds. Exceptions to ASTM C109/C109M shall be as per the following:

a)   Molds may be separable into more than two parts.

b)   Dimensions for 50 mm-cube and 2 in.-cube molds shall be manufactured in accordance with ASTM C109. Molds in use verification shall be as per requirements of Annex B.

The molds shall be made of hard corrosion-resistant materials.

Cube-mold base plate and cover plate shall be of corrosion-resistant materials (brass, stainless steel); they shall have a minimum thickness of 5 mm (0.2 in.) Grooves may be incorporated into the surface of the cover plate contacting the surface of the cement.

7.3.3   Compressive strength-testing machine (load frame)

Exceptions to ASTM C109/C109M shall be as per the followings:

a)   The two surfaces of the bearing blocks shall have a diameter or diagonal of no less than 70.7 mm (2.83 in.)

b)   The bearing blocks shall have a Rockwell hardness of no less than 30 HRc.This is not auditable

The testing load frame bearing block above or beneath the tested specimen shall be free to tilt in any direction. The center of the bearing block shall align with the center of the surface of the specimen, to assure even loading across the sample surfaces. A hardened metal bearing block shall be used beneath or above the specimen, depending on how the swivel bearing is tilting to minimize wear on the platens. The lower platen shall be inscribed to assist in the placement of the specimen in the center of the platen surface.

7.3.4   Water curing bath or tank

Water bath or tank having dimensions permitting the complete immersion of molds for compressive-strength test samples in water, and capable of maintaining the specified test temperatures within ±2 °C (±3 °F) shall

be employed. Two types of water curing baths are used for compressive strength testing, atmospheric unpressurized baths and pressurized baths for high temperature curing under pressure.

7.3.4.1   Atmospheric-pressure curing bath

Unpressurized vessel, having an agitator or circulating system. Atmospheric-pressure curing baths at or below 66 °C (150 °F) may be used for curing samples for compressive-strength testing when higher pressure is not required.

7.3.4.2   Pressurized curing chamber

Suitable for curing samples at the appropriate final test temperature and a pressure of at least 21 MPa (3000 lbf/in.²), the vessel shall be capable of being heated at the desired rate.

7.3.5   Cooling bath

Atmospheric-pressure water bath designed so that the specimen to be cooled from the curing temperature can be completely submerged in water maintained at 27 °C ±3 °C (80 °F ±5 °F).

7.3.6   Temperature-measuring system

7.3.6.1   General

The temperature-measuring system shall meet requirements given Annex B. This includes thermometers, thermocouples, and temperature controllers on water curing bath, curing chambers and cooling bath, and those that are used separate from or are not an integral part of the instrument.

7.3.6.2   Temperature measuring device

A thermometer or a thermocouple with a minimum range from −20 °C up to at least 105 °C (0 °F up to at least 220 °F) or above limitation of the equipment, with minimum scale divisions not exceeding 1 °C (2 °F) or a thermocouple with a minimum range of −20 °C to at least 120 °C (0 °F to at least 250 °F) may be used in a non-pressurized bath.

A thermocouple of range −20 °C to at least 205 °C (0 °F to at least 400 °F) shall be used in a pressurized curing chamber.

7.3.7   Puddling rod, corrosion-resistant rod, typically with a diameter of 6 mm ±2 mm (1/4 in. ±0.08 in.).

7.3.8   Mold-sealing grease, possessing the following properties when subjected to anticipated test temperatures and pressures is suitable for use to seal the exterior contact points of the sample molds:

a)   a consistency to permit ease of application,

b)   good sealing properties to prevent leakage,

c)   water resistance,

d)   inert to the cement, and

e)   non-corrosive in the temperature range of the test.

7.3.9 Mold-release agent (optional)

A thin layer of mold-release agent may be applied to the interior surfaces of the mold to prevent the sample from being damaged when removed from the mold. The mold-release agent shall comply with 7.3.8.

7.3.10 Timers

A stopwatch or electric timer with time indicated in hours, minutes and seconds shall be used.

7.3.11 Caliper

A caliper accurate to 0.1 mm (1/16in.) at minimum shall be used to measure dimensions of cement cube specimens.

7.4 Procedure

7.4.1 Preparation of molds

The base of assembled molds shall be watertight. The interior faces of the molds and the contact surfaces of the plates shall be clean and dry and may be lightly coated with mold release agent. Care shall be taken to ensure there is no bead of sealant on the interior of the mold. See Figure 6.
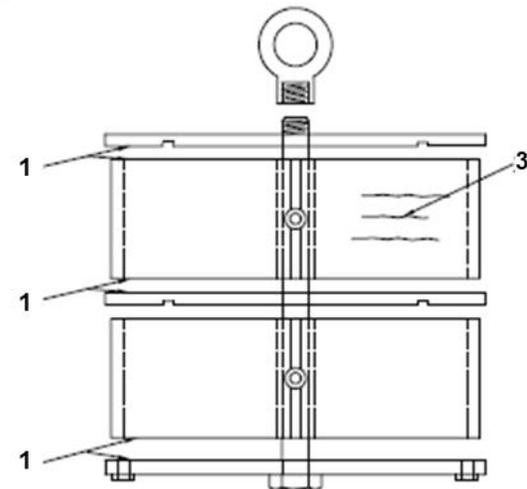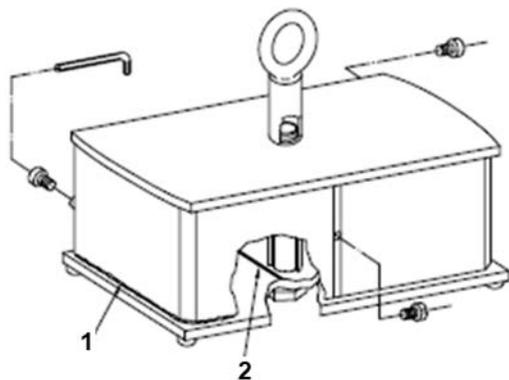
Key

1   grease lightly here
2   remove extruded grease
3   apply mold release agent inside sample cavity

Figure 3—Diagram of Mold Preparation

7.4.2 Preparation of slurry and filling of molds

7.4.2.1 Preparation

Cement slurry shall be prepared in accordance with Section 5. If preconditioning is required, then either 5.4 or 7.5 shall be followed. Section 7.5 is particularly useful to determine the compressive strength at the top of long cement columns.

7.4.2.2 Mold filling

To prepare samples, the following procedure shall be followed:

a) Immediately after mixing or after conditioning as appropriate, pour the cement slurry into each compartment of the prepared molds in a layer approximately one-half of the mold depth.

b) Place the slurry in all the specimen compartments before commencing the puddling operation.

c) Use the puddling rod to puddle efficiently the slurry in an evenly distributed pattern (minimum 27 times per specimen) to remove air bubbles.

d) After puddling the layer, stir the remaining slurry by hand using a puddling rod or spatula to minimize segregation.

e) Fill each mold compartment with cement slurry to overflowing and puddle the same as for the first layer.

f) After puddling, use a straight-edge to strike off the excess slurry level with the top of the mold.

g) Discard specimens in molds that leak.

h) Place a clean, dry cover plate on top of the mold.

No less than three cube-samples shall be used for each test determination.

7.4.3 Curing

7.4.3.1 Curing periods

Curing periods are defined according testing conditions and the temperature of the curing vessel.

For curing temperature at or below 88 °C (190 °F), the curing period is the time elapsed from subjecting the specimens to the specified temperature in the curing vessel (or initiating the temperature/pressure schedule for testing above atmospheric pressure) to testing the cured sample for strength. The strength of the cured samples shall be tested within 45 min at the appropriate time, as specified in Tables2 and 3 or a specific designed schedule.

For curing temperature above 88 °C (190°F), the curing period is the time elapsed from subjecting the specimens to the specified temperature in the curing vessel (or initiating the temperature/pressure schedule for testing above atmospheric pressure) to the point the heating is discontinue and cured samples are allowed to cool.

For specimens cured at atmospheric pressure, the curing period starts when specimens are initially placed in the curing bath preheated to the test temperature.

For specimens cured at above atmospheric pressures, the curing period starts with the initial application of pressure and temperature.

7.4.3.2   Curing at atmospheric pressure

To cure samples at atmospheric pressure the following procedure shall be applied:

a)   After the molds have been filled and covered with the cover plate and within 5 min after mixing of the slurry or slurry conditioning (removal of conditioned slurry from the slurry cup), place them in a curing vessel maintained at the desired curing temperature.

When placed in an atmospheric-pressure water bath, raise the molds off the bottom of the bath using a perforated baffle plate, wire rack, or suitable spacers to allow water to completely circulate around the samples during the curing period.

b)   At 45 min ±5 min prior to the age at which the samples are to be tested, remove the molds from thecuring vessel.

c)   Cured samples shall be removed from their molds and immediately cooled by being immersed in a water-cooling bath maintained at 27 °C ±3 °C (80 °F ±5 °F) for 40 min ±5 min, until the samples are tested.

Do not leave the cured samples out of water for more than 5 min to avoid dehydration.

7.4.3.3   Curing above atmospheric pressure

To cure samples above atmospheric pressure the following procedure shall be applied:

a)   After the molds have been filled and covered with the cover plate and within 5 min after mixing the slurry or after slurry conditioning (removal of conditioned slurry from the slurry cup), place them in a curing vessel at the desired test initiation temperature [normally 27 °C ±3 °C (80 °F ±5 °F or at the desired pre-heated curing vessel temperature)].

b)   Apply heat and pressure in accordance with the test schedule. Cement samples may be cured in accordance with pressure/temperature standard schedules provided either in Table 2 or Table 3, or by a schedule designed to simulate conditions in a specific well. For depths greater than 6600 m or 22,000 ft, user defined schedules shall be used. Schedules shall be interpolated to arrive at the correct depth/static temperature of the well for which the test is performed.

NOTE : Standard API compressive strength schedules are designated by a "Sg number". This appelation shall not misinterpreted as a relative density "SG".

c)   For samples cured at or below 88 °C (190 °F), maintain test temperature and pressure until 45 min ±5 min prior to testing.

d)   For test temperatures above 88 °C (190 °F), maintain the maximum scheduled temperature and pressure until   the desired curing time.

e)   Discontinue heating and allow samples to cool at such a rate that the sample temperature is below 90 °C (194 °F) or less without a reduction in the pressure other than caused by the reduction in temperature.

f) At 45 min ±5 min prior the time at which the cured samples will be tested, release the remaining pressure gradually and remove the cured samples from the curing vessel.

g) Immediately remove the samples from their molds and cooled by being immersed in a water cooling bath maintained at 27 °C ±3 °C (80 °F ±5 °F) for 40 min ±5 min, until the samples are tested.

   Do not leave the cured samples out of water for more than 5 min to avoid dehydration.

h) Record the total elapsed time between the samples are cool down to the time the cube samples are tested [steps item e) to g)].

   NOTE   With curing periods less than 60 hours this time should be critical when compressive strength results are evaluated and compared.

7.4.3.4   Specimen acceptance

Cube-test samples that are damaged shall be discarded prior to testing. If fewer than two test samples are left for determining the compressive strength at any given conditions, a retest shall be made.

7.4.4   Strength testing procedure

To conduct the compressive strength test, the following procedure shall be followed:

a) Remove cured sample from the water-cooling bath that has been maintained at 27 °C ±3°C (80 °F ±5 °F); wipe each sample to remove any loose material from the faces that will be in contact with the bearing blocks of the testing machine.

b) Cube height dimensions shall be measured to ±1.6 mm (50-mm molds) or to ±1/16 in. (2-in. molds) for calculation of the cross-sectional area.

   If the cube height is less than 47.5 mm (cube prepared using a 50-mm mold) or less than 1.9 in. (cube prepared using a 2-in. mold) the cube shall be discarded.

c) Place the cube-sample in the load frame centered below the upper bearing block (i.e. the top surface during curing shall not be in contact with the platen).

   1) The load will be applied to the sample faces that were in contact with the plane (vertical) surfaces of the mold (not in contact with the base or the cover plates).

   2) Prior to the testing of each cube, ascertain that the spherically seated block is free to tilt.

   3) Use no cushioning or bedding materials.

d) Apply the load to the cube-sample:

   Caution—Employ appropriate safety and handling procedures in testing the specimen.

   1. The rate of loading for samples with expected strength greater than 3.5 MPa (500 lbf/in.²) shall be 72 kN/min ±7 kN/min (16,000 lbf/min ±1600 lbf/min). For a nominal 25.8 cm² (4 in.²) sample surface, this rate can be achieved by adjusting the load rate to obtain a gauge indicator change of 18 kN (4000 lbf) in gauge reading in 15 s.

2. The rate of loading for samples with expected strength lower or equal than 3.5 MPa (500 lbf/in.²) shall be 18 kN/min ±2 kN/min (4,000 lbf/min ±400 lbf/min). For a nominal 25.8 cm² (4 in.²) sample surface, this rate can be achieved by adjusting the load rate to obtain a change of 18 kN (4000 lbf) in gauge reading in 1 min.

NOTE Depending on the type of compressive strength test machine employed, it can require some time for the load frame to build up the required load rate after initial contact with the cement sample.

The rate of loading applied to determine compressive strength on cement cube shall be reported.

e) Calculate the compressive strength expressed in megapascals (pounds-force per square inch) as the force required to break the sample divided by the smallest calculated cross-sectional area in contact with the load-bearing plates of the load frame.

f) The compressive strength of all acceptance-test specimens made from the same sample and tested at the same period shall be recorded and averaged to the nearest 0.1 MPa (10 lbf/in.²).

NOTE In case of a reduced number of cube-sample to be crushed, if the calculated compessive strength of any sample appears as an outlier, do not delete it without reasonable cause (e.g., identifyable cause such as suspected faulty cube based on pretest physical examination, unusual failure behavior, clearly unreasonable result), as the outlier may be a result of the other sample data clumping, thus removing the suspected outlier without cause may introduce bias.

h) Average the compressive strength of all acceptable test samples made from the same slurry and tested at the same time. Report compressive strength results to the nearest 0.3 MPa (50 lbf/in.²) and include the test conditions used.

7.5 Determination of Cement Compressive Strength at the Top of a Long Cement Column

7.5.1 Guidelines for Use

This procedure should be especially useful if the predicted bottomhole-circulating temperature (T_{PBHC}) is higher than the static temperature at the top of the cement column (T_{TOCS}).

7.5.2 Procedure

To prepare a cement sample at top of a long cement column the following procedure shall be applied.

a) Prepare a cement slurry in accordance with Section 5. Pour the slurry into the slurry container of a pressurized consistometer, and heat to T_{PBHC}. Apply pressure in accordance with the Table 2, Table 3, or 9.4. Hold at T_{PBHC} and pressure for 60 min to allow the cement temperature to reach equilibrium.

b) Upon completion of the appropriate test schedule, plus 60 min at temperature, change the temperature of the slurry to the top-of-cement circulating temperature (T_{TOCC}) or 88 °C (190 °F), whichever is lower, at a rate of 1.0 °C/min (2.0 °F/min). Use one of the following equations to determine the cool-down time (t_{cd}), expressed in minutes:

— Equation (32) when temperatures are expressed in degrees Celsius:

$$t_{cd} = T_{PBHC} - T_{TOCC} \tag{32}$$

— Equation (33) when temperatures are expressed in degrees Fahrenheit:

$$t_{cd} = \frac{T_{PBHC} - T_{TOCC}}{2.0} \qquad (33)$$

where

T_{PBHC} is the predicted bottomhole circulating temperature, expressed in degrees Celsius or degrees Fahrenheit;

T_{TOCC} is the top-of-cement circulating temperature or 88 °C (190 °F), whichever is lower, expressed in degrees Celsius or degrees Fahrenheit.

Maintain test pressure while decreasing the temperature.

c) When the T_{TOCC} or 88 °C (190 °F) whichever is lower, is reached, release the pressure slowly and remove the slurry container. Minimize oil contamination of the slurry by maintaining the slurry container upright (with the paddle shaft up). Syringe or blot oil from the top of the slurry container.

Do not cool the slurry container further after removal from the pressurized consistometer.

d) Open the slurry container from the top (paddle shaft end) leaving the paddle in place. This eliminates the need for inverting the slurry container and reduces contamination that could be caused by oil migrating through the slurry. Syringe and blot the oil from the top of the slurry with an absorbent cloth or paper towel.

e) Remove the paddle and stir the slurry briskly with a spatula to ensure it is uniform and to resuspend any solids that may have settled.

f) Pour the slurry into prepared molds as specified in 7.4.2.2 and place the molds in a preheated curing vessel [preheated to T_{TOCC} or 88 °C (190 °F), whichever is lower].

Alternatively, a nondestructive sonic test device as described in Section 8 may be used.

g) In no more than 15 min after removing the slurry from the pressurized consistometer, apply pressure simulating well pressure at top-of-cement, within the limitations of the apparatus being used.

h) Adjust temperature of the sample to the final curing temperature (T_{TOCS}) over a time period appropriate to well conditions, while maintaining curing pressure. This may require cooling the slurry to T_{TOCS}. If a time to reach final conditions is not known or specified, use 6 hr.

i) Remove samples as specified in 7.4.3.3.

j) Test the samples for strength in accordance with procedures in 7.4.4 or Section 8.

Table 2—Well-simulation API Test Schedules for Curing Compressive Strength Specimens (SI units)

Temperature Gradient [d] (°C/100 m)	Schedule[a] (SI)	1 Sg	2 Sg	3 Sg	4 Sg	5 Sg	6 Sg	7 Sg	8 Sg	9 Sg	10 Sg	11 Sg	12 Sg
	Depth (m)	300	600	1200	1800	2400	3000	3600	4200	4800	5400	6000	6600
	Pressure[c] (MPa)	5.5	11.0	20.7	20.7	20.7	20.7	20.7	20.7	20.7	20.7	20.7	20.7
	Time to T_{BHC}[b] (min)	13	17	25	32	40	49	57	65	73	81	89	96
	Total Time to T_{BHS}[b] (min)	240	240	240	240	240	240	240	240	240	240	240	240
1.6	T_{BHC}[f] (°C)	27	32	37	44	52	60	62	71	80	90	101	113
	Temp. ramp to T_{BHC} (°C/min)		0.30	0.41	0.52	0.62	0.67	0.61	0.68	0.73	0.78	0.84	0.89
	T_{BHS}[e] (°C)	32	37	46	56	65	75	85	94	104	113	123	133
	Temp. ramp to T_{BHS} (°C/min)	0.02	0.02	0.04	0.06	0.07	0.08	0.12	0.13	0.14	0.15	0.15	0.14
2.0	T_{BHC} (°C)	27	32	38	45	53	63	73	84	96	108	122	137
	Temp. ramp to T_{BHC} (°C/min)		0.30	0.45	0.55	0.64	0.73	0.81	0.88	0.95	1.00	1.07	1.14
	T_{BHS} (°C)	33	39	51	63	75	87	99	111	123	135	147	159
	Temp. ramp to T_{BHS} (°C/min)	0.03	0.03	0.06	0.09	0.11	0.13	0.14	0.15	0.16	0.17	0.17	0.15
2.4	T_{BHC} (°C)	27	32	38	46	57	69	84	97	111	126	143	161
	Temp. ramp to T_{BHC} (°C/min)		0.30	0.45	0.58	0.74	0.85	1.00	1.08	1.15	1.23	1.31	1.39
	T_{BHS} (°C)	34	41	56	70	85	99	113	128	142	157	171	185
	Temp. ramp to T_{BHS} (°C/min)	0.03	0.04	0.08	0.12	0.14	0.16	0.16	0.18	0.19	0.19	0.18	0.17
2.8	T_{BHC} (°C)	27	32	39	48	60	75	94	110	126	144	163	185
	Temp. ramp to T_{BHC} (°C/min)		0.30	0.49	0.65	0.82	0.98	1.17	1.28	1.36	1.45	1.54	1.64
	T_{BHS} (°C)	35	44	61	77	94	111	128	145	161	178	195	212
	Temp. ramp to T_{BHS} (°C/min)	0.04	0.05	0.10	0.14	0.17	0.19	0.18	0.20	0.21	0.21	0.21	0.19

Temperature Gradient [d] (°C/100 m)	Schedule[a] (SI)	1 Sg	2 Sg	3 Sg	4 Sg	5 Sg	6 Sg	7 Sg	8 Sg	9 Sg	10 Sg	11 Sg	12 Sg
	Depth (m)	300	600	1200	1800	2400	3000	3600	4200	4800	5400	6000	6600
	Pressure[c] (MPa)	5.5	11.0	20.7	20.7	20.7	20.7	20.7	20.7	20.7	20.7	20.7	20.7
	Time to T_{BHC}[b] (min)	13	17	25	32	40	49	57	65	73	81	89	96
	Total Time to T_{BHS}[b] (min)	240	240	240	240	240	240	240	240	240	240	240	240
3.2	T_{BHC} (°C)	27	33	39	49	65	84	105	122	141	162	184	208
	Temp. ramp to T_{BHC} (°C/min)		0.36	0.49	0.68	0.94	1.16	1.37	1.46	1.57	1.67	1.77	1.88
	T_{BHS} (°C)	37	46	65	85	104	123	142	161	181	200	219	238
	Temp. ramp to T_{BHS} (°C/min)	0.04	0.06	0.12	0.17	0.19	0.20	0.20	0.23	0.24	0.24	0.23	0.21
3.6	T_{BHC} (°C)	27	33	40	53	74	96	115	135	157	180	205	232
	Temp. ramp to T_{BHC} (°C/min)		0.36	0.53	0.80	1.16	1.40	1.54	1.66	1.78	1.90	2.01	2.13
	T_{BHS} (°C)	38	49	70	92	113	135	157	178	200	221	243	265
	Temp. ramp to T_{BHS} (°C/min)	0.05	0.07	0.14	0.19	0.20	0.20	0.23	0.25	0.26	0.26	0.25	0.23

[a] Sg is API schedule number designation.

[b] Temperature shall be increased on a double temperature ramp to meet the targets designated by the schedule until the 240 min temperature (T_{BHS}) is reached. First ramp to T_{BHC}, duration time to T_{BHC}; second ramp from T_{BHC} to T_{BHS}, duration 240 min minus time to T_{BHC}.

The curing temperature (temperature at 240 min) shall be maintained within ± 2 °C throughout the remainder of the curing period until cooling, if necessary, is required (see 7.4.5).

[c] Test temperature shall be applied as soon as specimens are placed in the pressure vessel and the pressure seal is complete and maintained at the given pressure within the following limits for the duration of the curing period:
    Schedule 1 Sg   5500 kPa ±700 kPa;
    Schedule 2 Sg   11,000 kPa ±1400 kPa;
    Schedule 3 Sg–12 Sg   20,700 kPa ±3400 kPa.

[d] Temperature gradient = (T_{BHS} − 27 °C)/100 m depth.

[e] T_{BHS} = bottomhole static temperature, temperature at 240 min.

[f] T_{BHC} = bottomhole circulating temperature.

Table 3—Well-simulation API Test Schedules for Curing Compressive Strength Specimens (USC units)

Temperature Gradient [d] (°F/100 ft)	Schedule[a] (USC)	1 Sg	2 Sg	3 Sg	4 Sg	5 Sg	6 Sg	7 Sg	8 Sg	9 Sg	10 Sg	11 Sg	12 Sg
	Depth (ft)	1000	2000	4000	6000	8000	10,000	12,000	14,000	16,000	18,000	20,000	22,000
	Pressure[c] (lbf/In.²)	800	1600	3000	3000	3000	3000	3000	3000	3000	3000	3000	3000
	Time to T_{BHC}[b] (min)	13	17	25	33	41	50	58	66	74	82	90	98
	Total Time to T_{BHS}[b] (min)	240	240	240	240	240	240	240	240	240	240	240	240
0.9	T_{BHC}[f] (°F)	80	89	99	112	126	141	148	164	182	201	222	244
	Temp. ramp to T_{BHC} (°F/min)		0.53	0.76	0.97	1.12	1.22	1.17	1.27	1.38	1.48	1.58	1.67
	T_{BHS}[e] (°F)	89	98	116	134	152	170	188	206	224	242	260	278
	Temp. ramp to T_{BHS} (°F/min)	0.04	0.04	0.08	0.11	0.13	0.15	0.22	0.24	0.25	0.26	0.25	0.24
1.1	T_{BHC} (°F)	80	89	100	114	129	146	165	185	207	231	256	284
	Temp. ramp to T_{BHC} (°F/min)		0.53	0.80	1.03	1.20	1.32	1.47	1.59	1.72	1.84	1.96	2.08
	T_{BHS} (°F)	91	102	124	146	168	190	212	234	256	278	300	322
	Temp. ramp to T_{BHS} (°F/min)	0.05	0.06	0.11	0.15	0.20	0.23	0.26	0.28	0.30	0.30	0.29	0.27
1.3	T_{BHC} (°F)	80	90	101	116	135	158	183	207	233	261	291	324
	Temp. ramp to T_{BHC} (°F/min)		0.59	0.84	1.09	1.34	1.56	1.78	1.92	2.07	2.21	2.34	2.49
	T_{BHS} (°F)	93	106	132	158	184	210	236	262	288	314	340	366
	Temp. ramp to T_{BHS} (°F/min)	0.06	0.07	0.14	0.20	0.25	0.27	0.29	0.32	0.33	0.34	0.33	0.30
1.5	T_{BHC} (°F)	80	90	102	118	140	167	201	228	258	291	326	364
	Temp. ramp to T_{BHC} (°F/min)		0.59	0.88	1.15	1.46	1.74	2.09	2.24	2.41	2.57	2.73	2.90
	T_{BHS} (°F)	95	110	140	170	200	230	260	290	320	350	380	410
	Temp. ramp to T_{BHS} (°F/min)	0.07	0.09	0.18	0.25	0.30	0.33	0.32	0.36	0.37	0.37	0.36	0.32

Temperature Gradient [d] (°F/100 ft)	Schedule[a] (USC)	1 Sg	2 Sg	3 Sg	4 Sg	5 Sg	6 Sg	7 Sg	8 Sg	9 Sg	10 Sg	11 Sg	12 Sg
	Depth (ft)	1000	2000	4000	6000	8000	10,000	12,000	14,000	16,000	18,000	20,000	22,000
	Pressure[c] (lbf/In.²)	800	1600	3000	3000	3000	3000	3000	3000	3000	3000	3000	3000
	Time to T_{BHC}[b] (min)	13	17	25	33	41	50	58	66	74	82	90	98
	Total Time to T_{BHS}[b] (min)	240	240	240	240	240	240	240	240	240	240	240	240
1.7	T_{BHC} (°F)	80	91	103	120	146	180	219	250	284	321	360	404
	Temp. ramp to T_{BHC} (°F/min)		0.65	0.92	1.21	1.61	2.00	2.40	2.58	2.76	2.94	3.11	3.31
	T_{BHS} (°F)	97	114	148	182	216	250	284	318	352	386	420	454
	Temp. ramp to T_{BHS} (°F/min)	0.07	0.10	0.21	0.30	0.35	0.37	0.36	0.39	0.41	0.41	0.40	0.35
1.9	T_{BHC} (°F)	80	91	104	126	160	200	236	271	309	350	395	444
	Temp. ramp to T_{BHC} (°F/min)		0.65	0.96	1.39	1.95	2.40	2.69	2.89	3.09	3.29	3.50	3.71
	T_{BHS} (°F)	99	118	156	194	232	270	308	346	384	422	460	498
	Temp. ramp to T_{BHS} (°F/min)	0.08	0.12	0.24	0.33	0.36	0.37	0.40	0.43	0.45	0.46	0.43	0.38

[a] Sg is API schedule number designation.

[b] Temperature shall be increased on a double temperature ramp to meet the targets designated by the schedule until the 240 min temperature (T_{BHS}) is reached. First ramp to T_{BHC}, duration time to T_{BHC}; second ramp from T_{BHC} to T_{BHS}, duration 240 min minus time to T_{BHC}.

The curing temperature (temperature at 240 min) shall be maintained within ±3 °F throughout the remainder of the curing period until cooling, if necessary, is required (see 7.4.5).

[c] Test temperature shall be applied as soon as specimens are placed in the pressure vessel and the pressure seal is complete and maintained at the given pressure within the following limits for the duration of the curing period:
    Schedule 1 Sg   800 lbf/In.² ±100 lbf/In.²;
    Schedule 2 Sg   1600 lbf/In.² ±200 lbf/In.²;
    Schedule 3 Sg–12 Sg   3000 lbf/In.²i ±500 lbf/In.².

[d] Temperature gradient = (T_{BHS} − 80 °F)/100 ft depth.

[e] T_{BHS} = Bottomhole static temperature, temperature at 240 min.

[f] T_{BHC} = Bottomhole circulating temperature.

8 Nondestructive Sonic Determination of Compressive Strength of Cement

8.1 General

This section presents a sonic nondestructive testing procedure to correlate compressive strength to sonic compressional transit time (compressional sonic slowness). Acoustic impedance of the cement may also be calculated. Strength correlations are specific to certain cement compositions. Some cement compositions may not fit the correlations supplied by the manufacturer and may require custom correlations.

8.2 Apparatus

The apparatus shall include the following systems:

a) **Temperature-measuring system**, meeting requirements in Annex B

b) **Sonic signal measuring system**,

c) **A pressure cell**, which can be subjected to controlled temperature and pressure for curing the cement slurry.

d) **A control unit,** to regulate pressure and temperature according to a simulated well schedule.

The apparatus is calibrated according to the requirements in Annex B.

8.3 Sampling

Samples of the cement, additives, and mix water shall be obtained in accordance with Section 4.

8.4 Preparation of Slurry

The slurry shall be prepared in accordance with 5.3 unless preconditioning is appropriate, then use 5.4 or 7.5 shall be followed.

8.5 Procedure

Follow the detailed operating instructions and safety precautions furnished by the manufacturer. The presence of free fluid may change the transit time measurements.

8.6 Curing Time

Curing period begins when transit time is recorded along with pressure and temperature and continues until the test is terminated.

Start the test within 15 minutes after mixing of the slurry or slurry conditioning (removal of conditioned slurry from the slurry cup) with the application of pressure and initiation of the temperature schedule.

8.7 Curing Schedules

Cement samples may be cured in accordance with pressure/temperature schedules provided in Section 7 or using a schedule designed to simulate specific well conditions, such as test at the top-of-cement or following a heating schedule determined by temperature simulation.

NOTE   Planned or unplanned changes in temperature or pressure alter the transit time and consequently the strength result.

8.8 Data Reporting

8.8.1   The transit time shall be monitored and recorded a minimum of once every 4 min. The sonic strength of the cement sample can be determined from transit time correlations. The sonic strength shall be obtained using transit time correlations specific to the cement composition.

The acoustic impedance, Z, of the set cement expressed in megarayls (MRayl; 10^6 kg/m^2•s) may be calculated from Equation (34) in SI units or Equation (35) in USC units.

—   In SI units:

$$Z = 25.4 \times \frac{\rho_{\text{set}}}{\text{TT}}$$

(34)

—   In USC units:

$$Z = 3.044 \times \frac{\rho_{\text{set}}}{\text{TT}}$$

(35)

where

   ρ_{set} is set cement density, expressed in kilograms per liter (pounds-mass per gallon);

   TT   is the transit time (acoustic slowness), expressed in microseconds per inch (µs/in.)

NOTE 1   Set cement density should be determined by a similar procedure as described in 12.5.2 on a cement sample from the curing cell or calculated from a sedimentation test (see 12.5.3).

NOTE 2   If the apparatus output is the time to travel between the sonic sensors inside the pressure cell, transit time is calculated by dividing this travel time expressed in microseconds by the distance between the sensors expressed in inches (see 3.1.51).

8.8.2   After removal of the sample from the curing cell, the sample is occasionally cored and surfaced (top bevel cut off) before being crushed. The result obtained is not comparable to that found using the method described in Section 7 as strengths determined by testing to compressive failure are greatly affected by specimen geometry. The result of such a test shall not be reported as API compressive strength.

9   Well-simulation Thickening-time Tests

9.1   General

Recommended procedures for determining the well-simulation thickening-time of a cement slurry are provided in this section. The results of the laboratory thickening-time test provide an indication of the length of time that a cement slurry will remain pumpable in a well. The laboratory test conditions shall represent the time, temperature, and pressure to which a cement slurry will be exposed during pumping operations.

9.2   Apparatus and Material

9.2.1   Calibration

Measurement of the thickening-time of a cement slurry shall require calibration and maintenance of operating systems of the pressurized consistometer including consistency measurement, temperature

measuring systems, temperature controllers, motor speeds, timers, and gauges as slurry container assembly for wear. All apparatuses shall be calibrated as per the requirements given in Annex B.

9.2.2 Pressurized consistometer

The most commonly used apparatus incorporates a rotating cylindrical slurry container equipped with a stationary paddle assembly, all enclosed in a pressure vessel capable of withstanding well-simulation pressures and temperatures (Figure 7, and API Specification 10A). The slurry container is rotated at a speed of 150 r/min ±15 r/min. An alternative apparatus is described in Annex C.

NOTE   The alternative apparatus used to conduct well-simulation thickening-time tests described in Annex C utilizes a rotating paddle and stationary slurry container. Generalized operating procedures for the alternative apparatus are also found in Annex C.

Figure 4—Typical Pressurized Consistometer

A heating system, capable of raising the temperature of the oil in the pressure vessel at a rate of at least 3 °C/min (5 °F/min) shall be required.

The paddle and all parts of the slurry container exposed to the slurry shall be constructed of corrosion-resistant materials. The paddle and all parts of the slurry container exposed to the slurry shall be constructed according to the dimensions given in API Specification 10A.

The space between the slurry container and the walls of the pressure vessel shall be completely filled with white mineral or synthetic oil. White mineral oil is a colorless, odorless liquid consisting of a mixture of hydrocarbons obtained by distillation. Synthetic oil is a liquid of chemical compounds artificially made (chemically modified petroleum components or synthesized from other raw materials). The selected oil shall safely allow the required 3 °C/min (5°F/min) heat-up rate.

NOTE   Although oils have been used above their flash point in consistometers with appropriate safety design features, it is recommended to select oils (hydrocarbon or synthetic) with suitable properties and a flash point above the maximum operating temperature of the equipment.

To determine the thickening-time, the consistency of a cement slurry shall be measured. The consistency, expressed in Bearden units of consistency (B_C), is determined by the force imposed by the slurry against the paddle and measured as a torque. A potentiometer mechanism and voltage measurement circuit, or its equivalent, is used to determine the torque.

Temperature-measuring systems shall be provided for determining and controlling the temperature of the cement slurry (centerline) (see 9.3.4).

The temperature, pressure and consistency of the cement slurry shall be measured and recorded.

9.3   Thickening-time Test Procedure

9.3.1   Operating instructions

Detailed operating instructions developed by the operator or furnished by the equipment manufacturer, are applicable for this method and shall be followed, provided they conform to the following procedure. Grease may be placed only on the threaded surfaces of the slurry container.

9.3.2   Assembly and filling the slurry container

Inspect and assemble the slurry cup, prepare the slurry as per 5.3, and fill the slurry container by the following steps.

a)   Ensure the cup and threads are clean.

b)   Inspect the diaphragm and replace it if it is damaged.

c)   Assemble the paddle shaft assembly and diaphragm with diaphragm support ring and back-up plate and secure them in the cup sleeve with the flange ring (the paddle and inside of cup sleeve shall not be greased as some greases have been found to affect thickening-time results). Ensure the tapered side of the diaphragm support ring is against the diaphragm for the best seal.

d)   Assemble the base and center plug (pivot bearing) and make sure the paddle turns freely.

e)   Remove the base and pivot bearing. Grease the threads of the cup on the base end to ease removal following the test. Grease the threads of the center plug to ease removal.

f)   With the base end of the slurry container up, prepare the slurry as in 5.3 and fill to the top or leaving two to three threads showing.

g)   strike the cup and remove any air that rises to the top of the slurry.

h)   Install the base until slurry is extruded through the center hole.

i)   Install the center plug (pivot bearing) into the container and tighten it until both the plug and base are tight in the container.

j)   Rinse all cement from the outer surfaces and dry them; and

k)   Recheck the paddle to ensure it turns smoothly.

9.3.3   Initiation of test

To initiate the thickening-time the following procedure shall be applied.

a)   Place the filled slurry container on the drive table in the pressure vessel, start rotation of the slurry container and secure the potentiometer mechanism or other suitable device for measuring consistency so as to engage the paddle shaft drive bar. Begin filling the vessel with oil. The paddle shaft shall not be rotating.

   NOTE   Some consistometers fill with oil faster than others. Begin filling those that fill quickly after the head assembly has been secured in the pressure vessel.

b)   Secure the head assembly in the pressure vessel, inserts the thermocouple through its fitting, and partially engage the threads. After the pressure vessel is completely filled with oil, tighten the threads of the thermocouple.

c)   Begin the thickening-time test by applying the initial pressure and starting the temperature ramp. No more than 5 min shall have elapsed after cessation of mixing of the slurry as defined in 5.3.6 and the beginning of the test. Alternatively, if surface mixing is to be simulated in the consistometer, no more than 5 min shall have elapsed before the beginning of the surface mixing simulation.

9.3.4   Temperature and pressure controls

During the thickening-time test, the temperature and pressure of the cement slurry in the slurry container shall be increase in accordance with the appropriate well-simulation test schedule (9.4). Schedules may be calculated or taken from tables. During the pressure and temperature ramp, the temperature and pressure shall be maintained within ± 3 °C (± 5 °F) and ± 2 MPa (± 300 lbf/in.²) of the appropriate temperature and pressure target vs elapsed time. Within 10 min after the end of the ramp, temperature and pressure shall be within ± 1 °C (± 2 °F) and ± 0.7 MPa (± 100 lbf/in.²) of the specified values.

The temperature of the cement slurry shall be determined by use of a temperature measuring device located in the center of the sample container. The tip of the thermocouple shall be vertically positioned within the paddle shaft in the slurry cup in such a way that it is between 44 mm (1.75 in.) and 89 mm (3.5 in.) above the inside of the base of the sample container. As there are many models of consistometers, each having different dimensions, care must be taken to ensure that the thermocouple used is compatible with the consistometer and the position of the tip of the thermocouple is in the correct location as specified above.

9.3.5   Thickening-time

The thickening-time is the time elapsed from the initial application of pressure and temperature to the time at which the slurry reaches a consistency deemed sufficient to make it unpumpable (e.g. 70 B_c or 100 B_c). The slurry consistency at which the thickening-time test was terminated shall be documented and reported.

9.3.6   Test conclusion and cleanup

Following the determination of the thickening-time, terminate the test and cool the consistometer before releasing the pressure. After releasing the pressure, remove the potentiometer and slurry container and prepare them both for the next test.

For tests at high temperature, the cement may be set. The cement with paddle may be pressed out of the slurry container using a hydraulic press and a suitable frame to mount the container. Take care not to damage the tip of the paddle shaft or the shaft itself. After removal of the set cement with the paddle, carefully chip the cement away from the paddle without damaging the paddle. The orientation of the paddle blades can normally be determined by observing the ends. If the orientation cannot be determined, chip gently on the sides of the cement until the blades of the paddle can be observed. Then, using a small hammer in the area between the paddle sides and the free blades, chip on the cement. After carefully removing all large masses of cement, clean any remaining cement from the blades using a spatula or other blunt instrument.

Clean the slurry cup sleeve and its threads of all cement residue. Inspect the diaphragm, paddle, shaft, shaft point, and pivot area of the plug for damage or wear and replace if necessary.

9.4   Determination of Thickening-time Test Schedules

9.4.1   General

Well-simulation thickening-time test schedules are used to simulate changes in temperature and pressure from surface conditions to bottomhole pressure (p_{BH}) and predicted bottomhole circulating temperature (T_{PBHC}). The temperature in the well can vary with time and volume circulated, volume of the fluid(s) being circulated, the pump rate, pipe size, etc. The temperatures and pressures may be taken from the tables or calculated from equations as described in 9.4.3 to 9.4.5 or may be determined using thermal simulators.

Annex D lists bottomhole circulating temperatures for nominally vertical wells based on correlations. The choice of table to use shall be based upon type of cement job to be performed: casing, liner, plug, continuous—pumping squeeze or hesitation-pumping squeeze. The choice of column and row within a table are based upon average temperature gradient and well measured depth. Schedules may be interpolated to arrive at the correct depth and temperature gradient of the well for which the test is being performed.

The bottomhole circulating temperature correlation used for plugging operations may be based on either the casing and liner well-simulation schedules (Table D.1) or the squeeze-cementing well-simulation schedule (Table D.2), depending on the extent of circulation prior to cementing.

Remark—Plug-cementing well-simulation schedules: Shall be recognized the difference in predicted bottomhole circulating temperatures (T_{PBHC}) that can be used in plugging operations, (between the casing and liner well-simulation schedules and the squeeze-cementing schedules) may be considerable.

9.4.2   Casing and liner well-simulation thickening-time schedules

Casing and liner well-simulation thickening-time schedules shall be determined as follows:

a)   account for surface mixing of the slurry (9.4.3.1), if applicable;

b)   calculate time to displace leading edge of cement slurry to bottom (t_d) (9.4.3.2);

c)   calculate bottomhole pressure (p_{BH}) (9.4.3.5);

d)   determine the starting pressure (p_S) (9.4.3.6);

e)   calculate rate of pressure increase to bottomhole pressure (R_{pu}) (9.4.3.7);

f)   determine the T_{PBHC} for the specific thermal gradient (Table D.1) or, for wells with depth 3050 m (10,000 ft) or greater, use the correlation found in 9.4.3.3;

g)   calculate well-simulation heat-up rate ($R_{\Delta T}$) (9.4.3.4).

Alternatively, a thermal simulator may be used to determine the T_{PBHC} and the heat-up schedule.

Once reached, the final temperature and pressure conditions shall be maintained until the thickening-time test is completed. Alternatively, if a thermal simulation of well temperatures is available, the final

temperature and pressure may be changed to simulate the conditions as the cement flows up the annulus and held at the top-of-cement temperature and pressure (using steps in 9.4.3.9 to 9.4.3.13).

9.4.3   Calculations for casing and liner thickening-time schedules

9.4.3.1   Surface mixing of the cement slurry

If batch mixing is used for the cementing operation, the slurry may be stirred in the consistometer to simulate the time and temperature of the batch mix operation. The time and slurry surface temperature (T_{SS}) may be estimated depending upon the expected conditions at the well site.

The batch mix simulation is done prior to the start of the thickening-time test. The batch mix time shall be reported separately from the thickening-time of the slurry.

EXAMPLE   Total time of test: 4 hr 30 min

Simulated batch mix time: 1 hr

Thickening-time (does not include batch mix simulation): 3 hr 30 min

9.4.3.2   Time to displace leading edge of cement slurry to wellbore bottom

The time required for displacement of slurry to the end of the pipe is calculated as given Equation (36):

$$t_d = \frac{V_p}{q} \tag{36}$$

where

t_d is the time to displace the leading edge of cement slurry to bottom, expressed in minutes;

V_p is the volume of the pipe, expressed in cubic meters (barrels);

q is the pump rate, expressed in cubic meters per minute (barrels per minute).

NOTE   Pipe can contain sections of different sizes and the time must be computed for each section to determine the total time to bottom.

9.4.3.3   Correlation for predicted bottomhole circulating temperatures (T_{PBHC}) for casing or liner jobs at depths greater than 3050 m (10,000 ft)

Although the T_{PBHC} correlation is based upon field measurements, those measurements were made in a small geographical area with a typical surface temperature of 27 °C (80 °F). There can be error associated with its use for predicting the circulating temperature in a well. Whenever possible, measurements of downhole temperatures or computer simulations are preferred over calculated estimates.

The correlation developed for predicting bottomhole circulating temperatures in degrees Celsius shall be given Equation (37):

$$T_{PBHC} = 26.7\,°C + \frac{\left(6.061 \times 10^{-3} \times h_{TVD} \times \Delta T_P\right) - 5.606\ 4}{1.0 - \left(0.049\ 383 \times 10^{-3} \times h_{TVD}\right)} \tag{37}$$

or, in degrees Fahrenheit as Equation (38):

$$T_{\text{PBHC}} = 80\text{ °F} + \frac{\left(6.061 \times 10^{-3} \times h_{\text{TVD}} \times \Delta T_{\text{P}}\right) - 10.091\,5}{1.0 - \left(0.015\,052 \times 10^{-3} \times h_{\text{TVD}}\right)} \tag{38}$$

where

T_{PBHC} is the predicted bottomhole circulating temperature, expressed in degrees Celsius [Equation (36)] or in degrees Fahrenheit [Equation (37)];

h_{TVD} is true vertical depth, expressed in meters [Equation (37)] or in feet [Equation (38)];

ΔT_{P} is the pseudo-temperature gradient, expressed in °C/100 m [Equation (37)] or in °F/100 ft [Equation (38)].

NOTE   This correlation was developed using constants derived from linear regression analysis of data collected in USC units. The correlation using SI units has not been established.

This correlation shall not be used to predict T_{PBHC} for depths less than 3050 m (10,000 ft) because it was developed from data measured on wells greater than 3050 m (10,000 ft) in depth and can give significantly higher T_{PBHC} than the T_{PBHC} found in the tables in Annex D.

9.4.3.4   Rate of heat-up to predicted bottomhole circulating temperature ($R_{\Delta\text{T}}$)

The temperature of the cement slurry shall be increased from the slurry surface temperature (T_{SS}) to the predicted bottomhole circulating temperature (T_{PBHC}) in the time required to displace the leading edge of cement slurry to bottom. The heat-up rate, $R_{\Delta\text{T}}$, expressed in degrees Celsius per minute (degrees Fahrenheit per minute) shall be given Equation (39):

$$R_{\Delta\text{T}} = \frac{T_{\text{PBHC}} - T_{\text{SS}}}{t_{\text{d}}} \tag{39}$$

where

T_{PBHC} is the predicted bottomhole circulating temperature, expressed in degrees Celsius (degrees Fahrenheit);

T_{SS} is the slurry surface temperature, expressed in degrees Celsius (degrees Fahrenheit);

t_{d} is the time to displace the leading edge of cement slurry to bottom, expressed in minutes (9.4.3.2).

9.4.3.5   Bottomhole pressure (p_{BH})

The bottomhole pressure, p_{BH} , can be calculated expressed in kilopascals as given Equation (40):

$$p_{\text{BH}} = 0.009\,81 \times \rho_{\text{af}} \times h_{\text{TVD}} \tag{40}$$

or expressed in pounds-force per square inch as given Equation (41) :

$$p_{\text{BH}} = 0.052 \times \rho_{\text{af}} \times h_{\text{TVD}} \tag{41}$$

where

ρ_{af} is the density of annular fluid, expressed in kilograms per cubic meter (pounds-mass per gallon);

h_{TVD} is the true vertical depth, expressed in meters (feet).

NOTE   The bottomhole pressure shall be calculated based on contributions of all fluids (spacers, weighted pills, etc.) in the annulus.

9.4.3.6   Starting pressure (p_{S})

A minimum starting pressure (p_{S}) of 700 kPa (100 lbf/in.²) is recommended. Other starting pressures, to simulate the pressure expected on the operation, may be applied, as appropriate.

9.4.3.7   Increase in pressure (pressure-up rate) to bottomhole pressure

Pressure on the cement slurry shall be increased to the bottomhole pressure during the test at a pressure-up rate calculated as given Equation (42):

$$R_{\text{pu}} = \frac{p_{\text{BH}} - p_{\text{S}}}{t_{\text{d}}} \tag{42}$$

where

R_{pu} is the rate of pressure increase, expressed in kilopascals per minutes (pounds-force per square inch and minute);

p_{BH} is the bottomhole pressure, expressed in kilopascals (pounds-force per square inch);

p_{S} is the starting pressure, expressed inkilopascals (pounds-force per square inch);

t_{d} is the time to displace the leading edge of cement to bottom, expressed in minutes (9.4.3.2).

9.4.3.8   Time at T_{PBHC} and p_{BH}

If no predicted data are available for the top-of-cement-circulating temperature (T_{TOCC}), the T_{PBHC} and p_{BH} shall be maintained until the completion of the thickening-time test. Skip steps in 9.4.3.9 to 9.4.3.13.

If reliable T_{TOCC} data are available, the cement slurry may be held at the T_{PBHC} and p_{BH} for a given period, such as 30 min, as a built-in safety factor. After a holding period at T_{PBHC} and p_{BH}, the temperature and pressure on the cement slurry may be changed to the top-of-cement-circulating temperature (T_{TOCC}) and pressure (p_{TOC}) using steps in 9.4.3.9 to 9.4.3.13.

9.4.3.9   Time to displace the annular volume to be cemented

The time required to displace the annular volume of cement from the bottom of the casing to the top of the annular cement column shall be calculated as given Equation (43):

$$t_a = \frac{V_a}{q} \tag{43}$$

where

> t_a is the time to displace the leading edge of cement slurry from the bottom of the casing to the top of the annular cement column, expressed in minutes;
>
> V_a is the annular volume of cement, expressed in cubic meters (barrels or cubic feet);
>
> q is the pump rate, expressed in cubic meters per minute (barrels per minute or cubic feet per minute).

9.4.3.10   Rate of temperature change to T_{TOCC}

The temperature of the cement slurry may be changed to the T_{TOCC} at a rate calculated by Equation (44)

$$R_{\Delta T} = \frac{T_{TOCC} - T_{PBHC}}{t_a} \tag{44}$$

where

> $R_{\Delta T}$ is the rate of temperature change to T_{TOCC}, expressed in °C/min (°F/min);
>
> > NOTE   This rate is different than the rate calculated in Equation (39).
>
> T_{PBHC} is the bottomhole circulating temperature, expressed in degrees Celsius (degrees Fahrenheit);
>
> T_{TOCC} is top-of-cement circulating temperature, expressed in degrees Celsius (degrees Fahrenheit);
>
> t_a is the time to displace the leading edge of the cement slurry from the bottom of the casing to the top of the annular cement column, expressed in minutes (9.4.3.9).

NOTE   A positive $R_{\Delta T}$ indicates heat-up; a negative $R_{\Delta T}$ indicates cool-down.

9.4.3.11   Pressure at the top-of-cement (p_{TOC})

Pressure at the top of the cement slurry, p_{TOC}, in the annulus shall be calculated using the following equations:

a)   expressed in kilopascals as given Equation (45):

$$p_{TOC} = 0.009\ 81 \times \rho_{af} \times h_{TOCTVD} \tag{45}$$

b)   expressed in pounds-force per square inch as given Equation (46):

$$p_{TOC} = 0.052 \times \rho_{af} \times h_{TOCTVD} \tag{46}$$

where

p_{TOC} is the pressure at the top of the cement in the annulus, expressed in kilopascals (pounds-force per square inch);

ρ_{af} is the density of annular fluid, expressed in kilograms per cubic meter (pounds-mass per gallon);

h_{TOCTVD} is the true vertical depth at top-of-cement, expressed in meters (feet).

NOTE   The top-of-cement pressure is calculated based on contributions of all fluids (spacers, weighted pills, etc.) in the annulus above the top of cement.

9.4.3.12   Rate of decrease in pressure to pressure at the top-of-cement (R_{pd})

Equation (47) is used to calculate the rate of decrease in pressure (pressure-down rate), R_{pd}, from the bottomhole pressure to the pressure at the top of the cement slurry column, expressed in kilopascals per minute (pounds-force per square inch and minute):

$$R_{pd} = \frac{p_{BH} - p_{TOC}}{t_a} \tag{47}$$

where

p_{BH} is the bottomhole pressure, expressed in kilopascals (pounds-force per square inch);

p_{TOC} is the pressure at the top of the cement column in the annulus, expressed in kilopascals (pounds-force per square inch);

t_a is the time needed to displace the leading edge of cement slurry from the bottom of the casing to the top of the annular cement column, expressed in minutes.

9.4.3.13   Completion of test with simulated temperature change

The cement slurry shall be held at the T_{TOCC} and p_{TOC} until the thickening-time test is completed.

9.4.4   Calculations for determining squeeze-cementing well-simulation schedules

9.4.4.1   General

Instructions contained in 9.4.3.1 to 9.4.3.8, with the exception of 9.4.3.3, shall apply for the calculation of a squeeze-cementing schedule. Squeeze-cementing temperature schedules may be determined according to 9.4.4.2 and 9.4.4.3. A minimum starting pressure (p_S, see 9.4.3.6) of 3400 kPa (500 lbf/in.²) shall be recommended for squeeze-cementing operations.

9.4.4.2   Continuous-pumping squeeze-cementing schedules

The predicted squeeze-cementing temperatures (T_{PSP}) for continuous-pumping squeeze-cementing shall be provided in Table D.2 or be calculated using Equation (50) or Equation (51). Pressure shall be increased directly from surface pressure (p_S) to final squeeze-cementing pressure (p_{FSQ}, see 9.4.4.5), Once reached,

the final temperature (T_{PSP}) and final squeeze-cementing pressure (p_{FSQ}) shall be maintained until the thickening-time test is completed.

9.4.4.3 Hesitation-pumping squeeze-cementing schedules

The predicted squeeze-cementing temperatures (T_{PSP}) and pressures for hesitation-pumping squeeze-cementing shall be determined as for continuous-pumping squeeze-cementing schedules, see 9.4.4.2 and 9.4.4.4.

The differences between the hesitation-pumping and continuous-pumping squeeze-cementing schedules are that for hesitation-pumping squeeze-cementing:

a)   there is a second temperature ramp to static temperature, and

b)   stirring of the slurry is cycled off and on during and after the second temperature ramp.

Stirring of the slurry is cycled off and on during and after the second temperature ramp, following an appropriate sequence. When the final applied squeeze pressure is reached, the slurry container rotation shall be typically stopped for 10 min and then restarted for 5 min. This cycle shall be repeated until the end of the test. The stopped time and rotating time during these "hesitations" should be modified to simulate the length of hesitations and pumping periods anticipated to be used for the squeeze cementing job, if known.

c)   pressure schedules shall follow a two-ramp schedule: first to bottomhole pressure (p_{BH}) and then to final squeeze pressure (p_{FSQ}), see 9.4.4.5.

The temperature and pressure schedule for a hesitation-pumping squeeze-cementing operation can be represented vs time by the Figure 8.

9.4.4.4 Determination of squeeze-cementing pressures

For a squeeze-cementing thickening-time schedule, the test pressure may be increased from p_{BH} to the anticipated final squeeze-cementing pressure (p_{FSQ}), which should include any applied surface squeeze-cementing pressure (p_{SQ}). Final bottomhole squeeze-cementing pressure, p_{FSQ}, expressed in kilopascals (pounds-force per square inch) shall be given Equation (48);

$$p_{FSQ} = p_{SQ} + p_{BH}$$
(48)

where

p_{SQ} is the final (maximum) surface squeeze-cementing pressure, expressed in kilopascals (pounds-force per square inch);

p_{BH} is the placement bottomhole pressure at the beginning of the squeeze-cementing, expressed in kilopascals (pounds-force per square inch).

Squeeze-cementing is conducted as a low-pressure squeeze-cementing operation below fracturing pressure, or as a high-pressure squeeze-cementing operation above fracturing pressure. Whatever the type of squeeze-cementing operation, the final squeeze-cementing pressure (p_{FSQ}) is a function of the well and the zone to be squeezed. Then, the maximum surface final squeeze-cementing pressure (p_{SQ}) used to determine the squeeze-cementing schedule shall be left to the user; typical values can vary from 3400 kPa (500 lbf/in.²) to more than 17,000 kPa (2500 lbf/in.²).
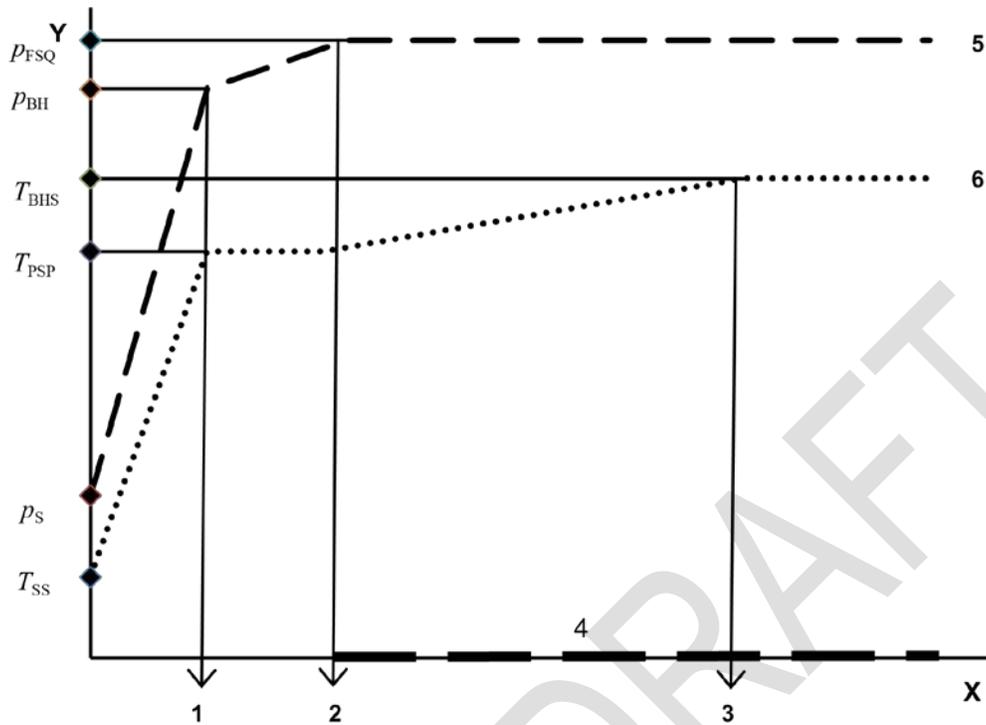
Key

X-axis	hesitation squeeze time	T_{SS}	slurry surface temperature
Y-axis	temperature and pressure	T_{PSP}	squeeze-cementing temperature
1	time to T_{PSP}, squeeze-cementing temperature	T_{BHS}	static temperature
2	time to final squeeze-cementing pressure p_{FSQ} first off/on and start to ramp to T_{BHS} static temperature	p_S	starting pressure
3	total time to T_{BHS} static temperature	p_{BH}	placement bottomhole pressure
4	slurry stirring cycle off/on from time 2 until the end of the test	p_{FSQ}	final squeeze-cementingpressure
5	pressure		
6	temperature		

Figure 8—Typical Hesitation-pumping Squeeze-cementing Pressure and Temperature Schedule

For hesitation-pumping squeeze-cementing application of the surface final squeeze-cementing-pressure (p_{SQ}) results in a second pressure step-up rate (R_{PSQ}), pressure increase, from p_{BH} to $p_{FSQ},$ shall be calculated using Equation (49).

$$R_{PSQ} = \frac{p_{SQ}}{t_{sq}}$$ (49)

where

    R_{PSQ} is the rate of pressure increase during application of the squeeze-cementing pressure, expressed in kilopascals per minute (pounds-force per square inch and minute);

    p_{SQ} is the final surface squeeze-cementing pressure, expressed in kilopascals (pounds-force per square inch);

t_{sq} is the time required to apply the final surface squeeze-cementing pressure, expressed in minutes;

For nontailored squeeze-cementing schedules R_{PSQ} should be calculated based on an anticipated time, t_{sq}, of 15 min.

9.4.4.5 Squeeze-cementing tailored schedules

Equations (36), (39) to (42), under 9.4.3.2, 9.4.3.4 to 9.4.3.7, and Equations (48) to (51), under 9.4.4.5 and 9.4.4.6 shall be used for tailored schedules. These equations can be used to calculate the heat-up rate ($R_{\Delta T}$) and pressure-up rates (R_{pu} and R_{PSQ}) for a squeeze-cementing simulation thickening-time test. In Equation (39), the predicted squeeze-cementing temperature (T_{PSP}) from Equation (50) or Equation (51) shall be substituted for the T_{PBHC}. After reaching the T_{PSP} and p_{FSQ}, the temperature and pressure profiles shall follow the anticipated temperature and pressure profiles for the remainder of the squeeze-cementing operation.

Additionally if the squeeze-cementing operation is expected to use the hesitation-pumping technique, it is recommended that the stirring of the slurry be cycled (see 9.4.4.3, Table D.2 Note 2) using an appropriate sequence. When the final pressure is reached (p_{FSQ}), slurry container rotation shall be stopped for 10 min and then restarted for 5 min. This cycle shall be repeated until the end of the test. The time stopped and time rotating during these "hesitations" should be modified to simulate the length of hesitations and pumping periods anticipated to be used for the squeeze-cementing operation, if they are known. In addition to cycling the stirring after final pressure is reached, the temperature should be increased at 0.1 °C/min (0.2 °F/min) until bottomhole static temperature (T_{BHS}) is reached and then held at T_{BHS}.

9.4.4.6 Correlation to predict squeeze-cementing temperatures

The correlation developed for predicting squeeze-cementing temperatures, T_{PSP}, expressed in degrees Celsius, shall be given in Equation (50):

$$T_{PSP} = 26.7\ °C + \frac{\left(7.649\ 5 \times 10^{-3} \times h_{TVD} \times \Delta T_P\right) - 4.556\ 7}{1.0 - \left(0.026\ 47 \times 10^{-3} \times h_{TVD}\right)} \tag{50}$$

or expressed in degrees Fahrenheit in Equation (51):

$$T_{PSP} = 80\ °F + \frac{\left(7.649\ 5 \times 10^{-3} \times h_{TVD} \times \Delta T_P\right) - 8.202\ 1}{1.0 - \left(8.068 \times 10^{-3} \times h_{TVD}\right)} \tag{51}$$

where

ΔT_P is the pseudo-temperature gradient, expressed in °C/100 m (°F/100 ft);

h_{TVD} is the true vertical depth, expressed in meters (feet).

NOTE   These correlations are valid only for the units shown.

Although the T_{PSP} correlation is based upon field measurements, those measurements were made in a small geographical area and there can be error associated with its use for predicting the squeeze-cementing temperature in a well. Whenever possible, measurements of downhole temperatures or computer simulations are preferred over calculated estimates.

9.4.5   Calculations for determining plug-cementing well-simulation schedules

9.4.5.1   General

Instructions contained in 9.4.3.1 to 9.4.3.8 shall apply for the calculation of a plug-cementing schedule. As plug-cementing operations are conducted through a smaller diameter pipe than casings a minimum starting pressure (p_S, see 9.4.3.6) of 3400 kPa (500 lbf/in.²) shall be recommended.

9.4.5.2   Plug-cementing schedules

If the well will not be circulated prior to cementing or, if area of the wellbore is not subjected to circulation, for example plugging below a retainer, the predicted plug-cementing temperatures (T_{PSP}) shall be provided in Table D.2 or calculated using Equation (50) or Equation (51). Once reached, the final temperature (T_{PSP}) and the bottomhole pressure (p_{BH}) shall be maintained until the thickening-time test is completed.

If the well is circulated, predicted plug-cementing temperature shall be the T_{PBHC} provided in Table D.1 or Equation (37) or Equation (38) under 9.4.3.3.

9.4.5.3   Plug-cementing tailored schedules

Equations (36) to (38), or Equations (39) to (42), under 9.4.3.2 to 9.4.3.7 can be used for plug-cementing tailored schedules. These equations shall be used to calculate respectively, the heat-up rate ($R_{\Delta T}$) and pressure-up rate (R_{pu}) for a plug-cementing simulation thickening-time test.

If the well will not be circulated prior to cementing, or if area of the wellbore is not subjected to circulation, for example plugging below a retainer, in Equation (39), the predicted plug temperature, T_{PSP}, calculated using Equation (50) or Equation (51) may be substituted for the T_{PBHC}.

Because of the short cement columns typically used in plug-cementing, no temperature change, or pressure-down rates to the top of the cement column shall be used. Therefore, the steps in 9.4.3.9 to 9.4.3.13 shall not be used.

10   Static Fluid-loss Tests

10.1 General

This section provides procedures for running static fluid-loss tests using high-pressure, high-temperature (HPHT) non-stirred or stirred fluid-loss cells. For tests at temperatures less than or equal to 88 °C (190 °F), tests may be performed using a non-stirred fluid-loss cell (sometimes called a filter press) after slurry conditioning in an atmospheric or pressurized consistometer, or by using a stirred fluid-loss apparatus. For tests at temperatures greater than 88 °C (190 °F), tests may be performed using a nonstirred fluid-loss cell following conditioning in a pressurized consistometer or by using a stirred fluid-loss apparatus. Regardless of whether the slurry is conditioned in a consistometer or in a stirred fluid-loss apparatus, the fluid loss value is determined under static conditions.

10.2 Apparatus

10.2.1 General

All apparatus used to perform static fluid-loss tests shall be calibrated and/or verified as per requirements given Annex B.

10.2.2 High-pressure, High-temperature (HPHT) Fluid-loss Cells

10.2.2.1   General

HPHT non-stirred fluid-loss cell or stirred fluid-loss apparatus, shall be fitted with a 45 μm (0.00177 in.-325 US mesh) screen with a 22.6 cm^2 (3.5 in.^{2}) filtration area backed by a 250 μm (0.01 in. – 60 US mesh) screen. If a screen with a perforated metal back is used, the end caps shall have radial grooves to provide a flow path for the cement filtrate. The screens shall be replaced when they show visible plugging, wear, damage, or distortion. The screens should be verified by the informative procedure outlined in Annex B and then, should be replaced when they show a flow time outside the tolerances set by the initial verification.

For testing at temperatures greater than 88 °C (190 °F), to prevent cement filtrate from boiling at the test temperature, a filtrate collector (a back-pressure receiver or a condenser) shall be connected to the outlet stem. For slurries containing salts (NaCl, KCl, etc.), a back-pressure receiver shall be used. The use of a condenser can allow the salts to crystallize as it cools and can plug the condenser tubing, giving a false indication of low fluid loss.

The equipment manufacturer's recommendations for maximum temperature, pressure, and volume shall not be exceeded.

10.2.2.2   HPHT non-stirred fluid-loss cells

Two main types of non-stirred HPHT fluid-loss cells can be used for testing cement slurries: HPHT"long" 25.4 cm (10 in.) cells and HPHT "short" 12.7 cm (5 in.) cells. Estimated slurry volumes to be tested are respectively 500 mL and 175 mL. Given the range of cement slurry fluid loss volumes which are measured during the 30-minute test duration, to prevent the slurry from "blowing dry," the long HPHT cell shall be the preferred non-stirred apparatus.

Single- or double-ended long and short cells are available. For safety, double-ended cells shall be preferred as they do not require inversion after preheating and filling.

Slurries that show settlement give erroneous fluid loss results. Care shall be taken to ensure that slurries are properly designed and have minimum tendency to settle.

Single-ended cells should pose both maintenance and safety concerns. It is more difficult to maintain single-ended cells, as they are more difficult to clean after a test. The single-ended cell also poses a safety hazard, as there is no screen protecting the pressurizing hole in the closed end of the cell. This increases the likelihood that the hole can become plugged with cement and trap pressure inside.

10.2.2.3   HPHT stirred fluid-loss apparatus

A stirred fluid-loss apparatus provides a means of conditioning the slurry in the fluid-loss cell itself. For safety concerns, stirred fluid-loss apparatus should be preferred for performing fluid-loss tests at temperatures higher than 88 °C (190 °F) as the stirred fluid-loss apparatus avoids having to handle and transfer hot fluids. Approximate slurry volume to be used in a stirred fluid-loss cell is 250 mL, but the requirements vary depending on the model used.

10.2.3 Temperature measuring system

A thermocouple or temperature measuring device accurate to ±2 °C (±3 °F) or better shall be used. The location of the thermocouple or temperature measuring device shall be noted on the report form. A thermocouple or temperature measuring device inserted in the wall of the fluid-loss cell or immersed in the slurry is preferred.

Metal dial thermometers shall not be used because of their relatively poor accuracy. Glass thermometers are not used because their dimensions do not allow them to fit into the heating jacket or the test cell.

10.2.4 Pressure gauges

Pressure gauges with a scale such that pressure can be read to $\pm$300 kPa ($\pm$50 lbf/in.²) shall be used.

10.2.5 Timer or stopwatch

A timer or stopwatch that is capable of measuring time in seconds and minutes shall be used.

10.3 Safety

Warning—Using appropriate PPE is recommended when operating HTHP equipment. Procedures require the handling of hot, pressurized equipment and materials that are hazardous and can cause serious injury. Only trained personnel can run these tests. Fluid-loss tests shall be performed behind a protective shield. Pressure can be trapped inside the cell, even if the valve stems are open or removed. To prevent plugging of the valve by cement, cooling the fluid-loss cell in the test position is recommended (i.e. with filter cake at the bottom). A screen in the "non-test" end of the cell will help to prevent the hole in the end cap from becoming plugged and trapping pressure. If end caps are difficult to remove, it can indicate pressure remaining in the cell and appropriate precautions shall be taken. Refer to manufacturers safety procedures.

10.4 Performing Static Fluid-loss Test Using Non-stirred Fluid-loss Cell

10.4.1 General

This subsection covers test procedures using HPHT, non-stirred fluid-loss cells ("long" or "short" cells), see Figures 9 and 10 for common non-strirred fluid-loss cells.

Before the test, ensure there is a nitrogen supply with sufficient pressure to pressurize the cell and the back-pressure receiver (if used) and to complete the test [i.e. around a minimum pressure of 8200 kPa (1200 lbf/in.²)].

Conditions of screens, O-rings, stems, and other components used in the test shall be checked prior to assembly. Clean and grease parts, if necessary. At the end of the test, ensure pressure within the test cell has been fully bled off before disassembly. Check the screen condition at the end of the test and repeat the test if necessary.

10.4.2 Preparing the non-stirred fluid-loss cell

The following procedure shall be applied to prepare the non-stirred fluid-loss cell:

a)   Prepare the fluid-loss cell. It shall be clean and dry. For double-ended cells, screens are required in both ends. The cell shall be ready to be filled when slurry conditioning has been completed.

b)   Preheat the cell in the heating jacket to the test temperature or to a maximum of 88 °C $\pm$3 °C (190 °F $\pm$5 °F) for tests at temperatures greater than 88 °C (190 °F).

10.4.3 Mixing and conditioning the slurry

The slurry shall be mixed and conditioned in accordance with Section 5. Note that for tests greater than 88 °C (190 °F), conditioning must be done in a pressurized consistometer at the test temperature.

Record the time to reach the test temperature and conditioning time. For tests at temperature greater than 88 °C (190 °F), record the cooling time.

Figure 9—Common HPHT, Non-stirred Fluid-loss Cell Bodies

**Figure 10—Common Screwed-cap Type, HPHT,
Double-ended Fluid-loss Cell**

10.4.4 Filling the non-stirred fluid-loss cell

The non-stirred fluid-loss cell shall be filled as per the following procedure:

a)   After conditioning, with the bottom valve closed, pour the slurry into the fluid-loss cell to 5.1 cm ±0.6 cm (2 in. ±^{1}/$_4$ in.) below the shoulder on which the screen rests in the long cell or to 2.5 cm ±0.6 cm (1 in. ±^{1}/$_4$ in.) below the shoulder in the short cell. The 45 μm - 325 mesh side of the screen shall be placed so it will be in contact with the cement slurry during testing.

Warning—Overfilling creates a hazard due to thermal expansion (see Table 4). Do not exceed equipment manufacturer's recommendations for maximum temperature, pressure, and volume.

Table 4—Vapor Pressure and Volume Expansion of Water at Temperatures Between 100 °C (212 °F) and 316 °C (600 °F)

Temperature °C (°F)	Water Vapor Pressure kPa (lbf/in.²)	Coefficient of Volume Expansion for Water at Saturation Pressure
100 (212)	100 (14.7)	1.04
121 (250)	210 (30)	1.06
149 (300)	460 (67)	1.09
177 (350)	930 (135)	1.12
204 (400)	1700 (247)	1.16
232 (450)	2910 (422)	1.21
260 (500)	4688 (680)	1.27
288 (550)	7200 (1044)	1.36
316 (600)	10,620 (1541)	1.47

b) Place the screen and O-rings in the cell and secure the end cap to the cell.

c) If the cell is a single-ended cell: invert the cell so that the screen is at the bottom.

10.4.5 Static fluid-loss test at temperature less than or equal to 88 °C (190 °F)

In order to perform a fluid loss test at temperature less than or equal to 88 °C (190°F) the following procedure shall be applied:

a) Place the thermocouple in the thermocouple well in the top of the cell body.

b) Connect the pressure supply line and apply 7000 kPa ±300 kPa (1000 lbf/in.² ±50 lbf/in.²). Open the top valve stem to apply the pressure to the cell.

c) Start the test: Open the bottom valve and start the timer simultaneously. Maintain at the test temperature and 7000 kPa ±300 kPa (1000 lbf/in.² ±50 lbf/in.²) for the duration of the test.

For tests at temperatures less than or equal to 88 °C (190 °F), no more than 6 min shall elapse from the completion of conditioning according to Section 5 until the start of the test (opening the bottom valve stem).

d) Collect the filtrate and record the filtrate volume to a precision of 1 mL at 30 s, 1 min, 5 min, 10 min, 15 min, 20 min, and 30 min, precisely recording the collection time. Alternatively, the filtrate may be continuously weighed to a precision of 1 g or less and recorded. If weighed, measure and report the filtrate relative density at 27 °C (80 °F) and calculate the volume of filtrate from the recorded weights using the relative density.

e) If nitrogen blows through at less than 30 min, record the volume collected and time at which it blows through. Any time nitrogen blows through, even if some liquid is flowing out, it shall be considered the end of the test.

f)   Stop the test after 30 min or when nitrogen blows through.

g)   Report fluid loss results according to 10.6.

The "Fluid loss Results Reporting Form" (Table 5) at the end of Section 10 may be used for recording data and other pertinent information about the test.

10.4.6 Static fluid-loss test at temperature greater than 88 °C (190 °F)

In order to perform a fluid loss test at temperature higher than 88 °C (190°F) the following procedure shall be applied:

a)   For tests at temperature greater than 88 °C (190 °F), after filling the cell, installing the end cap, and inverting, if necessary (single-ended cells only, 10.4.4.3), apply pressure of 3500 kPa ±300 kPa (500 lbf/in.² ±50 lbf/in.²), or sufficient pressure to prevent the fluid from boiling at the test temperature (see Table 4), to the cell. Leave the pressurizing valve open to allow pressure regulation while heating.

b)   Place the thermocouple in the thermocouple well in the top of the cell body.

c)   Attach the filtrate collector (back-pressure receiver or condenser) to the outlet stem. If a back-pressure receiver is used, apply sufficient pressure to the back-pressure receiver to prevent the cement filtrate from boiling at the test temperature [see Table 4, e.g. 700 kPa (100 lbf/in.²) for a test at 150 °C (300 °F)].

d)   Heat the fluid-loss cell to test temperature as fast as the heating jacket will heat. No more than 6 min shall elapse from the time of completion of the conditioning, in accordance with Section 5, to the start of heating. Record the time to reach the test temperature.

   NOTE   In order for the slurry to reach the test temperature, it is sometimes necessary to set the controller temperature higher than the desired test temperature.

e)   Apply 7000 kPa ±300 kPa (1000 lbf/in.² ±50 lbf/in.²) differential pressure to the cell. If a back-pressure receiver is used, the pressure applied is the sum of the pressure applied to the back-pressure receiver plus 7000 kPa ±300 kPa (1000 lbf/in.² ±50 lbf/in.²).

f)   Start the test within 60 s of reaching the test temperature. Open the bottom valve stem and start the timer simultaneously. Maintain at the specified test temperature and 7000 kPa ±300 kPa (1000 lbf/in.² ±50 lbf/in.²) differential pressure for the duration of the test.

g)   Collect the filtrate and record the volume after 30 min or precisely the time when blow through occurs, whichever occurs first. The filtrate is collected after flowing through the condenser (if used) or by draining the fluid from the back-pressure receiver and recording the volume to a precision of 1 mL at 30 s, 1 min, 5 min, 10 min, 15 min, 20 min, and 30 min precisely recording the collection time. If a condenser is used, the volume of fluid in the condenser itself must be accounted for.

h)   If nitrogen blows through or when back pressure (if used) increases due to nitrogen blowing through at less than 30 min, record the volume collected and precisely the time at which nitrogen blows through.

i)   Stop the test after 30 min or when nitrogen blows through. Starting with bottom valve, close all valves to the cell and turn off the heater. Bleed pressure off the lines (but leave pressure in the cell) and disconnect the lines from the cell. Bleed the pressure from, disconnect, and clean the back-pressure receiver.

j)   Report fluid loss results according to 10.6.

The "Fluid-loss Results Reporting Form" (Table 5) at the end of Section 10 may be used for recording data and other pertinent information about the test.

10.4.7 Test completion and cleanup

Test completion and cell cleanup procedure shall be applied as per the following:

a) Cool the cell to a safe handling temperature and carefully release the pressure.

b) After ensuring that all the pressure is released, disassemble the cell and inspect the screen to check for holes, plugging, or damage. If there is damage to the O-ring seals or screen, discard the test results and rerun the test.

c) Measure and report the height of the cake and its consistency (hard, firm, mushy, gelled, etc.). Do not include remaining slurry if gelled**.**

d) Clean and dry the fluid-loss cell in preparation for the next test. Pay particular attention to the O-ring grooves and the O-rings in the cell and on the valve stems. Ensure the hole in the valve stem is not plugged with cement and there is no cement in the fitting on the pressure supply lines. Carefully clean the screen to remove cement and/or additive residue.

Screens shall be replaced when they show visible plugging, wear, damage, or distortion. Screens should be verified by the informative procedure outlined in Annex B. If the screen condition is questionable, it should be possible to re-verify as per B.15.2 as to chemically clean the screen by soaking it in citric acid cleaning solution. Any screen that has been exposed to citric acid wash should be re-verified before use and be replaced when its verified flow time is outside the tolerances set by the initial verification.

NOTE   Cleaning solution should be prepared by mixing monohydrate citric acid (CAS No.5949-29-1) at a concentration of 50 g/L in water.

10.5 Performing a Static Fluid-loss Test Using Stirred Fluid-loss Apparatus

10.5.1 General

This section covers the test procedure using a stirred fluid-loss apparatus (Figures 11 and 12). These apparatuses allow fluid conditioning under high temperature and pressure. For tests at temperature greater than 88 °C (190 °F), a back-pressure receiver or a condenser is required to collect the filtrate. The fluid loss is measured under static conditions.

Before the test, ensure there is a nitrogen supply with sufficient pressure to pressurize the cell and the back-pressure receiver (if used) and to complete the test [i.e. around a minimum pressure of 8200 kPa (1200 lbf/in.²)].

Condition of the screen, O-rings, stems, and other parts used in the test shall be checked prior to assembly and be clean and dry. Grease parts, if necessary. At the end of the test, ensure the pressure within the test cell has been fully bled off before disassembly. Check the screen condition at the end of the test and repeat the test if the screen is damaged. The screens shall be replaced when they show visible plugging, wear, damage, or distortion. The screens should be verified by the informative procedure outlined in Annex B and then, should be replaced when they show a flow time outside the tolerances set by the initial verification.
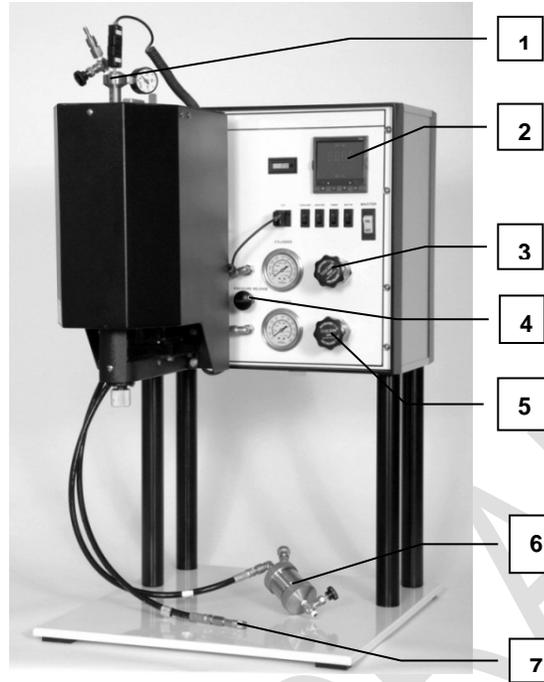
1	top plug, outlet valve	4	pressure release valve	7	nitrogen pressure supply line
2	temperature controller	5	back-pressure regulator		
3	cell pressure regulator	6	back-pressure receiver		

NOTE   The fluid-loss cell is in the stirring position (outlet valve at the top) and the back-pressure receiver is not connected.

Figure 11—Typical Stirred Fluid-loss Apparatus
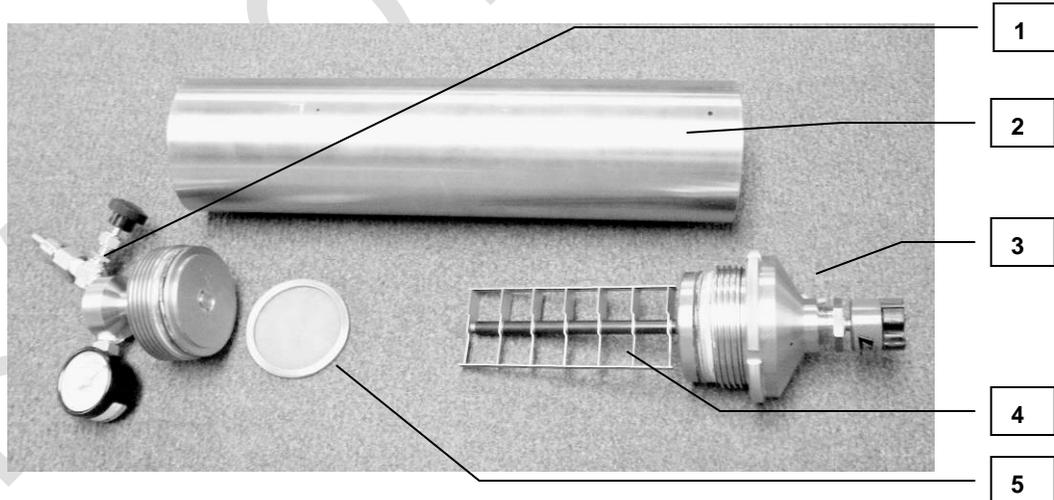
Key

1	top plug, outlet valve	4	paddle
2	fluid-loss cell body	5	screen
3	bottom plug		

NOTE   For slurry conditioning, "top plug" is at the top of the apparatus; after inversion, it is at the bottom.

Figure 12—Cell and Components of a Typical Stirred Fluid-loss Apparatus

10.5.2 Mixing and conditioning the slurry

The following procedure for mixing and conditioning the slurry with a stirred fluid-loss apparatus shall be applied. Prepare the stirred fluid-loss apparatus in accordance with the manufacturer's instructions.

a) Turn on the master switch and program the temperature controller for the thickening-time test schedule that most closely simulates field conditions.

b) Mix the slurry in accordance with 5.3.

c) Pour the slurry into the clean, dry, cell assembly according to the manufacturer's instructions.

   Warning—Overfilling of this device creates a hazard due to thermal expansion (see Table 4). Do not exceed equipment manufacturer's recommendations for maximum temperature, pressure, and volume.

d) Complete assembly of the cell (screen, O-rings, end cap, etc.) in accordance with the manufacturer's instructions. The 45 µm (325 US mesh) side of the screen shall be placed so it will be in contact with the cement slurry during testing.

e) Apply and maintain 3500 kPa ±300 kPa (500 lbf/in.² ±50 lbf/in.²) (or sufficient pressure to prevent the fluid from boiling at the maximum test temperature as listed in Table 4) to the cell. Leave the pressurizing valve open to allow pressure regulation while heating the cell.

f) Start stirring the slurry with the paddle.

g) While stirring with the paddle, begin heating the cell by turning on the heaters and starting the temperature controller. No more than 5 min shall elapse from the end of mixing of the slurry to the beginning of heating.

   Heat the slurry in accordance with the schedule that most closely simulates actual field conditions [programmed into controller, item a)].

   Hold the test temperature for 30 min ±30 s to allow the test fluid temperature to reach equilibrium. Optionally, the slurry may then be conditioned for an additional period appropriate to the operations planned for the well. Monitor pressures closely to prevent over-pressuring the cell.

h) Record the time to reach the test temperature and conditioning time.

i) Once the slurry has reached the specified test temperature and conditioning is complete, stop stirring, close the pressurizing valve (if necessary), disconnect the pressurizing line (if necessary), and disengage the motor drive. Invert the pressure vessel.

j) If required, connect the filtrate collector (back-pressure receiver or condenser) to the test valve below the screen. When a back-pressure receiver is used, apply sufficient pressure to the back-pressure receiver to prevent the cement filtrate from boiling at the test temperature (see Table 4). [e.g. back pressure 700 kPa (100 lbf/in.²) for a test at 150 °C (300 °F)].

k) If it was disconnected, connect the pressurizing line. Apply a differential pressure of 7000 kPa ±300 kPa (1000 lbf/in.² ±50 lbf/in.²). The pressure applied is the sum of the pressure applied to the back-pressure receiver (if used) plus 7000 kPa ±300 kPa (1000 lbf/in.² ±50 lbf/in.²). Open the top valve stem or pressurizing valve to apply the pressure to the cell.

10.5.3 Static fluid-loss filtration in stirred fluid-loss apparatus

The procedure to perform static fluid-loss test in stirred fluid-loss apparatus the following procedure shall be applied:

a)   Start the test within 60 s of inverting the cell. Open the bottom valve and start the timer simultaneously. Maintain at the specified temperature and at a differential pressure of 7000 kPa ±300 kPa (1000 lbf/in.² ±50 lbf/in.²) for the duration of the test.

b)   Collect the filtrate and record the volume to a precision of 1 mL at 30 s, 1 min, 5 min, 10 min, 15 min, 20 min, and 30 min, precisely recording the collection time. Alternatively, the filtrate may be continuously weighed to a precision of 1 g or less and recorded. If weighed, measure and report the filtrate relative density at 27 °C (80 °F) and calculate the volume of filtrate from the recorded weights using the relative density. When a condenser is used, the filtrate volume in the condenser shall be accounted for.

c)   If nitrogen blows through, or when back-pressure (if used) increases due to nitrogen blowing through, at less than 30 min, record the volume collected and precisely time at which nitrogen blows through.

d)   Stop the test after 30 min or when nitrogen blows through. Close all valves to the cell and turn off the heater. Bleed pressure from the lines (but leave pressure in the cell) and disconnect them from the cell. Bleed the pressure from, disconnect, and clean the back-pressure receiver.

e)   Report fluid loss results according to 10.6. The "Fluid loss Results Reporting Form" (Table 5) at the end of Section 10 may be used for recording data and other pertinent information about the test.

10.5.4 Test completion and cleanup

Test completion and cell cleanup procedure shall be applied as per the following:

a)   Cool the cell to a safe handling temperature and carefully release the pressure according to manufacturer's instructions.

b)   Following manufacturer's instructions, remove the cell.

c)   After ensuring that all the pressure has been released, disassemble the cell and inspect the screen to check for holes or damage. If there is damage to the O-ring seals or screen, discard the test results and rerun the test.

   Warning—Pressure can be trapped inside the cell, even if the valve stems are open or removed. To prevent plugging of the valve by cement, cooling the fluid-loss cell in the test position is recommended (i.e. with filter cake at the bottom). If end caps are difficult to remove it can indicate pressure remaining in the cell and appropriate precautions shall be taken. Refer to manufacturer's safety procedures.

d)   Measure and report the height of the cake and its consistency (hard, firm, mushy, gelled, etc.). Do not include remaining slurry if gelled.

e)   Clean and dry the fluid-loss cell in preparation for the next test. Carefully clean the screen to remove cement and/or additive residue.

   Screens shall be replaced when they show visible plugging, wear, damage, or distortion. Screens should be verified by the informative procedure outlined in Annex B. If the screen condition is questionable, it should be possible to re-verify as per B.15.2 as to chemically clean the screen by soaking it in citric acid cleaning solution. Any screen that has been exposed to citric acid wash should

be re-verified before use and be replaced when its verified flow time is outside the tolerances set by the initial verification.

NOTE   Cleaning solution should be prepared by mixing monohydrate citric acid (CAS No.5949-29-1) at a concentration of 50 g/L in water.

10.6 Fluid loss Results and Reporting

10.6.1   Calculate the API Fluid Loss, expressed as milliliters per 30 min as given in Equation (52). For tests that run the entire 30 min without "blowing dry," measure the collected filtrate volume, double the value, and report the doubled value as the API Fluid Loss.

$$\text{API Fluid Loss} = 2V_{30} \tag{52}$$

where

V_{30} is the volume of filtrate collected at 30 min, expressed in milliliters.

For tests that "blow dry" in less than 30 min, use Equation (53) or Equation (54) to determine the Calculated API Fluid Loss.

$$\text{Calculated API Fluid Loss} = 2 \times V_{t} \sqrt{\frac{30}{t}} \tag{53}$$

or, combining the constants, the equation reduces to:

$$\text{Calculated API Fluid Loss} = 10.954 \times \frac{V_{t}}{\sqrt{t}} \tag{54}$$

where

V_{t} is the volume of filtrate collected at the time nitrogen blows through, expressed in milliliters;

t is the elapsed time, expressed in minutes.

NOTE   If t, time nitrogen blows through, is not properly recorded, "Calculated API Fluid Loss" is calculated using the last recorded value before the nitrogen blows through.

10.6.2   When reporting the fluid loss of cement slurries, those for which the fluid loss was measured for a full 30 min shall be reported as "API Fluid Loss," while those for which the fluid "blew dry" in less than 30 min shall be reported as "Calculated API Fluid Loss."

NOTE 1   Slurries with significant sedimentation give erroneous values for fluid loss.

NOTE 2   Fluid-loss tests that do not run a full 30 min have a potential error that becomes greater as the length of the test becomes shorter. Fluid-loss tests that run the full 30 min typically show 5 % variability. Tests that run less than 5 min can have a variability of more than 30 %.

Table 5—Fluid loss Results Reporting Form

Heat-up schedule:	_____ minutes to _____ °C (°F) Test temperature _____ °C (°F)/min]

Conditioning method [ ] Atmospheric   [ ] Pressurized _____ kPa, (lbf/in.²)]

     [ ] Stirred fluid-loss cell

     [ ] Optional extra conditioning _____ minutes

Static cell length [ ] 12.7 cm (5 in.)   [ ] 25.4 cm (10 in.)

Cell type (ends) [ ] Double   [ ] Single

Screen type [ ] 45 µm x 250 µm (325 mesh × 60 mesh)

    [ ] 45 µm x 250 µm (325 mesh × 60 mesh) with perforated metal back

Time (min)	Filtrate [( ) mL or ( ) g]	Time (min)	Filtrate [( ) mL or ( ) g]
$1/2$	_____	15	_____
1	_____	20	_____
5	_____	30	_____
10	_____		

If filtrate weighed, relative density : _____ at 27 °C (80 °F)

API fluid loss	=	_____ mL/30 min
Blowout	=	_____ mL (or g) at _____ min/s
Calculated API fluid loss	=	_____ mL/30 min
Filter cake conditions	=	Thickness [a] _____mm (in.) Consistency [b] _____
Time from end of conditioning to test start	=	_____ min
Temperature	=	Start of test _____ °C (°F)
		End of test _____ °C (°F)
Location of thermocouple	=	[ ] Cell wall [ ] In slurry

Date of calibration of sensors	=		Consistometer	Fluid-loss cell
		Pressure gauge	_____	_____
		Thermocouple	_____	_____

[a] Thickness: of cake only; do not include remaining slurry if gelled.
[b] Consistency: hard, firm, mushy, gelled, etc.

11 Determination of Rheological Properties and Gel Strength at Atmospheric Pressure using a Rotational Viscometer

11.1 General

This procedure shall be used to characterize the rheological behavior of fluids used in well cementing under conditions of atmospheric pressure and at temperatures not exceeding 88 °C (190 °F). Determination of rheological properties of test fluids can be sensitive to the procedure being used. A comparison with rheological properties obtained using other methods is not recommended.

This standardized procedure has been developed to generate reproducible results for a given fluid. It was developed after a careful analysis of many parameters that affect the rheological behavior of fluids. Results may not be accurate when measuring the rheological properties of highly dispersed fluids.

The following conditions are necessary to properly characterize the rheological parameters of a fluid:

a)   the slurry is homogenous, and the shear stress is uniform across the gap,

a)   the flow regime in the annular gap is laminar,

b)   slip at the wall is negligible, and

c)   the fluid exhibits time-independent behavior.

NOTE   Procedure to determine rheological properties and gel strength under pressure is given Annex F.

11.2 Apparatus

11.2.1 Rotational viscometer

11.2.1.1   General

A typical rotational non-pressurized viscometer, also known as a Couette viscometer, shall be used to measure the rheological properties of fluids used in well cementing. This device consists of a cylindrical bob located concentrically within a cylindrical rotor, also referred to as a sleeve. The width of the gap between the bob and the rotor is fixed for a given rotor/bob configuration. The rotor is capable of turning at various specified rotational velocities. The rotation of the rotor acts upon the fluid being tested to impart a torque on the bob. The bob is mounted on a shaft that is connected to a torsion device, which restrains its movement. The deflection of the bob due to torque is measured in degrees. On most viscometers, this angular deflection is read directly from a dial attached to the shaft holding the bob. On some viscometer models however, the deflection is indicated indirectly on a digital display. A schematic of a typical rotational viscometer is shown in Figure 13.

Various sizes of rotors and bobs are available. Table 6 shows the dimensions of the rotors and bobs used for measuring the rheological properties of fluids used in well cementing. The ratio of the radius of the bob to the rotor should be > 0.9 in order to maintain a nominally uniform shear stress across the gap and to keep the slurry homogenous. Thus, for measuring the rheology of well cementing fluids the combination of rotor size R1 and bob size B1 shall be used in most cases. The gap should also be a minimum of ten times wider than the largest particle diameter. This criterion may require the use of a smaller diameter bob, which results in a radius ratio < 0.9. For special slurries containing large diameter particles, a B5 bob is usually sufficient to provide the required gap width. A B2 bob is also available; however, its use shall be only acceptable for measuring fluids containing very large particles
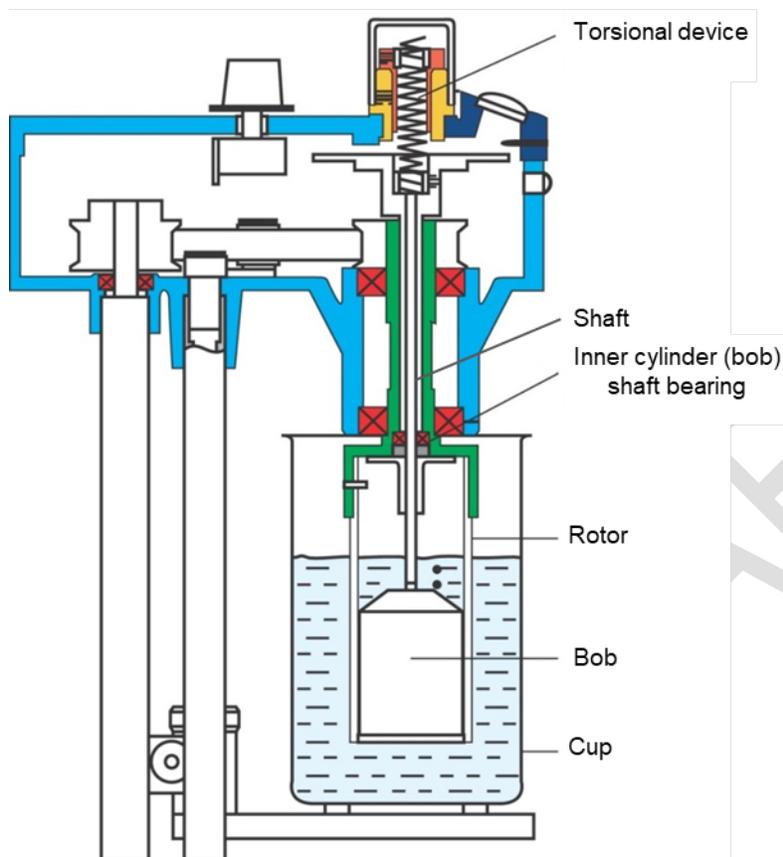
Figure 13—Typical Rotational Viscometer Schematic

Rotor and bob dimensions for an R1-B1 configuration are illustrated in Figure 14. The rotor shall have a scribed line 58.42 mm ±0.10 mm (2.30 in. ±0.004 in.) from the bottom to indicate the proper fill level for the fluid being tested. The rotor should have two rows of 3.16 mm (0.125 in.) diameter holes, spaced 2.09 radians (120°) apart that are centered 3.18 mm (0.125 in.) and 9.53 mm (0.375 in.) below the scribed line. The bob shall have a cylindrical body with a flat, closed base and a tapered top with a truncated cone. The finish on the inner surface of the rotor and the outer surface of the bob shall have a roughness average of 0.2 μm to 0.8 μm (8 μin. to 32 μin.) cross-hatch honed (surface grade N4 to N6).

The rotational viscometer shall be capable of measuring shear stress at shear rates ranging from near zero to at least 511 s^{-1} (for an R1-B1 rotor/bob combination). A minimum of five shear rates spread across the range is recommended.

Table 6—Dimensions of Rotors and Bobs

	Rotor-Bob		
	R1-B1	**R1-B2**	**R1-B5**
Rotor internal diameter	36.83 ± 0.02 (1.450 ± 0.001)	36.83 ± 0.02 (1.450 ± 0.001)	36.83 ± 0.02 (1.450 ± 0.001)
Bob external diameter	34.49 ± 0.02 (1.358 ± 0.001)	24.54 ± 0.02 (0.966 ± 0.001)	31.97 ± 0.02 (1.259 ± 0.001)
Bob length	38.00 ± 0.02 (1.496 ± 0.001)	38.00 ± 0.02 (1.496 ± 0.001)	38.00 ± 0.02 (1.496 ± 0.001)

	Rotor-Bob		
	R1-B1	**R1-B2**	**R1-B5**
Gap width	1.17 ± 0.02 (0.046 ± 0.001)	6.14 ± 0.02 (0.242 ± 0.001)	2.43 ± 0.02 (0.096 ± 0.001)
Bob/rotor radius ratio	0.937	0.666	0.868
NOTE Dimensions are in millimeters (inches)			

Dimensions in millimeters (inches) unless otherwise indicated.
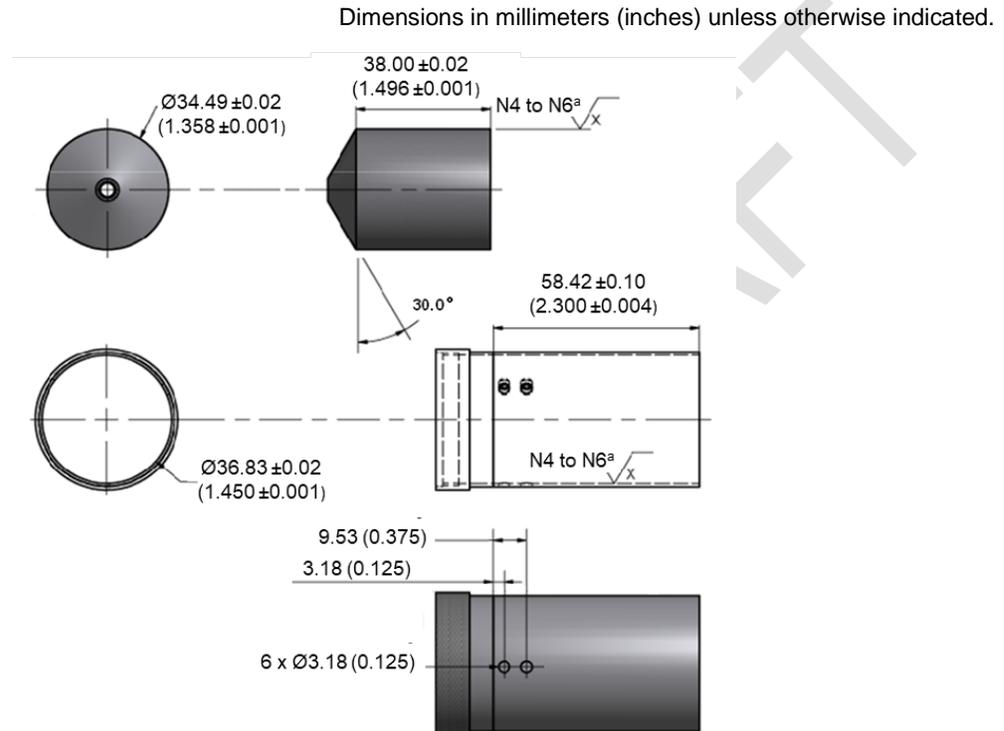
NOTE

[a]   surface roughness N4 to N6: Ra 0.2 μm to 0.8 μm (8 μin. to 32 μ in.).

Figure 14—R1 Rotor and B1 Bob Dimensions

11.2.1.2   Shear rate

Shear rate shall be a function of the rotational velocity of the rotor and the width of the gap between the rotor and the bob. Table 7 shows the calculated shear rate for each rotor-bob combination across a range of rotational velocities.

Table 7—Shear Rate Conversion for Rotor-Bob Combination

Rotational Velocity (r/min)	Rotor-Bob		
	R1-B1	R1-B2	R1-B5
	Shear Rate (s^{-1})		
1	1.70	0.38	0.85
2	3.40	0.75	1.70
3	5.11	1.13	2.55
6	10.21	2.26	5.10
10	17	3.77	8.50
20	34	7.54	17
30	51	11.31	25
60	102	22	51
100	170	38	85
200	340	75	170
300	511	113	255
600	1021	226	511

11.2.1.3   Shear stress

Shear stress is expressed in units of pascals (pounds force per one hundred square feet). It is a measurement of the deflection of the bob that is a function of torque and is dependent upon the dimensions of the bob and the torsional stiffness of the device. Torsion devices of varying torsional stiffness are available from the various manufacturers and their usable ranges are available from them. Table 8 shows the shear stress per degree of dial deflection for the various combinations of rotor-bobs and springs.

Table 8—Shear Stress per Degree of Dial Deflection

Spring Factor	Rotor-Bob		
	R1-B1	R1-B2	R1-B5
	Shear Stress/Degree Pa (lbf/100 ft²)		
F 0.2	0.102 (0.213)	0.202 (0.420)	0.119 (0.248)
F 0.5	0.256 (0.533)	0.504 (1.051)	0.297 (0.620)
F 1.0	0.511 (1.065)	1.008 (2.101)	0.595 (1.239)
F 2.0	1.022 (2.130)	2.016 (4.202)	1.189 (2.479)
F 3.0	1.533 (3.195)	3.024 (6.303)	1.784 (3.718)
F 5.0	2.555 (5.325)	5.040 (10.505)	2.975 (6.195)

11.2.2 Timer or stopwatch

A stopwatch or electric timer with time indicated in seconds shall be used.

11.2.3 Thermometer or Thermocouple

A thermometer or thermocouple capable of measuring temperature within ± 2 °C (± 3 °F) shall be used.

11.3 Calibration

Apparatus required for rheological properties and gel strength measurements shall meet requirements of Annex B. Viscometers are calibrated according to B.3.6, and B.3.7 and according to the manufacturer's instructions.

11.4 Determination of Rheological Properties

This following procedure shall be recommended when using atmospheric pressure viscometers. For safety reasons, do not use atmospheric viscometers at temperatures above 88 °C (190 °F). If the boiling point of water at the test location is less than 100 °C (212 °F), adjust test temperatures accordingly.

a) Using a heated cup assembly large enough to allow good temperature control, place sufficient water into the viscometer cup to cover the rotor to the scribed mark on the rotor without the rotor or bob touching the bottom of the cup. Raise the cup so the water covers the rotor to the scribed mark. The rotor may be dismounted from the viscometer and placed in the cup during heating.

b) Preheat the rotor, bob, and cup to the test temperature.

c) Prepare and condition the test fluid in accordance with Section 5.

d) Dry the rotor, bob, and cup immediately prior to testing and reassemble the rotor and bob on the viscometer. If the cup is not removable, the power to the heated cup shall be disconnected from the wall and special care taken when pouring water from the cup and refilling it.

e) Visually check the rotor and bob for centralization and make any needed corrections before starting the test.

f) Within 5 min after conditioning the slurry (removal of the slurry from the slurry container) pour the conditioned test fluid (fluid verified to be at proper temperature) into the preheated viscometer cup to a level adequate to raise the fluid to the scribed mark on the rotor without the rotor or bob touching the bottom of the cup. Maintain the test fluid at the test temperature ± 3 °C (± 5 °F) for the duration of the test by using a heated cup assembly large enough to allow good temperature control. During conditioning and rheology measurement, make every effort to prevent the test fluid from remaining static.

g) With the rotor turning at the lowest selected shear rate (typically 3 r/min for a R1-B1 combination), raise the cup until the liquid is level with the scribed line on the rotor.

h) Take the initial instrument dial reading after 10 s continuous rotation at that lowest selected shear rate.

i) Take all the remaining dial readings, first in ascending order up to 511 s^{-1} shear rate, i.e. 300 r/min using R1-B1 combination, and immediately change the speed to 200 r/min and then take the remainder of the dial readings in descending order after continuous rotation of 10 s at each speed. Shift to the next speed immediately after taking each dial reading. The highest recommended speed is 300 r/min for a R1-B1 combination. Exposing cement slurries to shear rates above 511 s^{-1} can lead to inconsistent (erroneous) results.

  NOTE   Highest recommended speed using a R1-B5 combination shall be 600 r/min and exposed cement slurries at 511 s^{-1} as using a R1-B1 combination at 300 r/min.

j)   If desired, dial readings at speeds higher than 300 r/min with R1-B1 combination may be taken after ramping up and down and after measuring the gel strength (see 11.5). After taking all the dial readings, record the temperature of the slurry in the viscometer cup.

NOTE   Repeatability of data taken at shear rates at and below 10.2 s^{-1} is often poor, i.e. 6 r/min using R1-B1 combination. At the discretion of the operator, dial readings at and below 10.2 s^{-1} may be omitted from the test, except when measuring gel strength (see 11.5).

k)   Record the following data:

—   rotor, bob, and spring configuration;

—   time, temperature, and method of conditioning;

—   r/min and dial reading at each r/min (raw data without correction for spring factor or rotor/bob gap); and

—   temperature of test fluid at end of rheology measurements.

l)   Calculate the ratio of the dial readings during ramp-up to ramp-down at each speed. This ratio can be used to help qualify certain fluid properties.

When the ratio at all the speeds is close to 1:1, this can suggest that the fluid is a non-settling, non-gelling, time-independent fluid at the test temperature.

Ratios other than 1:1 can suggest settling or gelation of the fluid during the test. If some ramp-down dial readings are lower by more than 5 instrument degrees at the same rotational speed (obtained with the viscometer described in 11.2.1.1 with a spring factor F 1.0), this may be a further indication of settling.

When significant differences in the dial readings indicate that the test fluid is not stable, that is, prone to settling or excessive gelation, adjustments in the composition shall be considered.

m)   Report the fluid rheological measurements as the average of the dial readings [(ramp-up + ramp-down)/2], and the temperatures recorded in item j). Also report the rotor, bob, and spring configuration, as well as the conditioning methods used. An example is shown in Table 9.

n)   For improved reliability of the data, the entire procedure may be repeated two or three times, using freshly prepared fluid each time, and the results averaged.

An example of rheological data report is given in Table 9.

Table 9—Example Rheological Data Report

Rotational Speed r/min	Ramp-up Reading	Ramp-down Reading	Reading Ratio	Average Reading
3	21	24	0.87	22.5
6	40	36	1.11	38
30	65	83	0.78	74
60	84	100	0.84	92
100	100	115	0.87	107.5
200	137	147	0.93	142
300	170	—	—	170
NOTE 1 Configuration: R1-B1-F1.				
NOTE 2 Conditioning method: atmospheric pressure, 30 min after reaching 66 °C.				
NOTE 3 Initial slurry temperature: 66 °C.				
NOTE 4 Final slurry temperature: 63 °C.				
Example of notes about the test above. The information underlined is to be filled in for the test performed				

11.5 Determination of Gel Strength

11.5.1 Gel strength at 10 s and 10 min

The gel strength of a fluid may be measured immediately after determining the rheological properties of the sample or on a separate, freshly prepared fluid.

NOTE   Gel strengths of a fluid are the measurements resulting of the application of the rheological property procedure described under 11.5. These measurements are not comparable to static gel strength of a fluid measured under API 10B-6 or ISO 10426-6 and shall not be used for critical static gel strength determination.

The following procedure shall be used to determine gel strength:

a) Recondition the fluid in the viscometer for 1 min at 300 r/min to disperse the gels and to allow better measurement of the gel strength. For tests on separate, freshly prepared fluids, prepare, condition, and load the fluid into the viscometer as outlined in 11.4 item a) to 11.4 item g) and then condition the fluid for 1 min at 300 r/min.

b) Stop rotation of the rotor.

c) Set the viscometer at the speed equivalent to 5.1 s^{-1} (3 r/min with R1-B1 combination or 6 r/min with R1-B5 combination).

d) 10 s after stopping the rotor, start rotation at 5.1 s^{-1} and record the maximum deflection of the dial observed immediately after starting rotation. Calculate the 10 s gel strength by multiplying the measured value by the factor for the applicable rotor, bob, and spring configuration found in Table 8. Record the fluid temperature, then stop the viscometer for 10 min (or other specified time, see 11.5.2).

NOTE   When the test is performed at high temperature and above approximatively 65 °C (150 °F), during static time to avoid dehydration of the cement slurry and development of a viscous layer at the slurry surface it should be recommended to either put oil on top of the slurry or to cover top of the viscometer cup with a plastic film wrap. In that case the film wrap shall be removed before to go with next item e).

e)   Start rotation at 5.1 s^{-1} 10 min after stopping the rotor and record the maximum deflection of the dial observed immediately after starting rotation. Calculate the 10 min gel strength by multiplying the measured value by the factor for the applicable rotor, bob, and spring configuration found in Table 8.

f)   After taking the maximum deflection dial reading, again record the temperature of the fluid in the viscometer cup

g)   Report the fluid gel strengths at the recorded temperatures (note the rotational velocity, the rotor-bob configuration, and spring factor used in the test).

h)   For improved reliability of the measurements, the entire procedure may be repeated two times or three times using freshly prepared fluid each time. Report the gel strength values as the average of the measurements.

11.5.2 Gel strengths at other static times

Gel strength measurements may be taken at other static times, as desired (example 30 min or other). The test shall have a 10 s gel strength and no more than one other gel strength time. If other gel strengths are required, a new slurry shall be prepared and tested for each set of two gel strength times.

Procedure is similar to gel strength determination at 10 min: Prepare, condition, and load the freshly-prepared fluid into the viscometer as outlined in 11.4 item a) to 11.4 item g) and then condition the fluid for 1 min at 300 r/min. Then follow the whole procedure 11.5.1 item b) to item h), substituting the desired static time for 10 min.

11.6 Characterization of Rheological Behavior

11.6.1 General

To characterize flow behavior (friction pressure, flow regime, etc.) of a fluid in any geometry (pipe, annulus, etc.), a rheological model that best represents the measured data must be selected. Table 7 and Table 8 can be used to convert the raw data measured with the viscometer (rotational velocity and torque) into shear stress and shear rate. The rheological model that best describes the relationship between shear stress and shear rate can then be selected by preparing a plot of shear stress vs shear rate (rheogram).

11.6.2 Newtonian fluids

Fluids for which shear stress is directly proportional to shear rate are called Newtonian. A single viscosity measurement characterizes a Newtonian fluid at a specified temperature and pressure. Water, glycerine, and light oils are common examples of Newtonian fluids.

11.6.3 Non-newtonian fluids

11.6.3.1   General

Fluids for which shear stress is not directly proportional to shear rate are called non-Newtonian. Most drilling fluids, cement slurries, and spacers exhibit non-Newtonian behavior. Several models have been developed to describe non-Newtonian fluids including the Bingham plastic, power law, and Herschel-Bulkley models. It is important to note that no single model can completely describe the behavior of a non-Newtonian fluid across the entire range of shear rates. Selection of the most appropriate model can be made by performing a regression analysis of the shear stress and shear rate data for each model. The model with the best regression coefficient should be selected in most cases.

11.6.3.2 Bingham plastic model

The Bingham plastic model describes a fluid with a shear stress to shear rate ratio that is linear after having exceeded some minimum shear stress. Two parameters, plastic viscosity (μ_p) and yield stress (τ_y), are used to describe a Bingham plastic fluid. A rheogram of a Bingham plastic fluid on Cartesian (rectangular) coordinates is a straight line with a positive slope and a positive shear stress at zero shear rate. The slope of the line is the plastic viscosity and the intercept of the y-axis is the yield stress (also referred to as the yield point).

NOTE   Plastic viscosity is commonly known in the industry by the abbreviation PV and the yiel point by the abbreviation TY.

Plastic viscosity is expressed in pascal•seconds (SI unit) or centipoises (USC unit). Yield point is expressed in pascals (SI unit) or pounds-force per one hundred square feet (USC unit).

The preferred method for calculating the plastic viscosity and yield point of a Bingham plastic fluid is through a mathematical linear regression analysis using all of the data points according to Equation (55) (SI units) or Equation (56) (USC units). The use of a computer program or spreadsheet is recommended to perform the regression analysis. Two-point calculations are not accurate and shall not be used.

In SI units:

$$\tau = \tau_\text{y} + (\mu_\text{p} \times \gamma) \tag{55}$$

In USC units:

$$\tau = \tau_\text{y} + \left[0.002\ 08 \times (\mu_\text{p} \times \gamma) \right] \tag{56}$$

where

   τ is the shear stress, expressed in pascals (pounds-force per 100 square feet);

   τ_y is the Bingham yield stress, expressed in pascals (pounds-force per 100 square feet);

   μ_p is the plastic viscosity, expressed in pascal•seconds (centipoises);

   γ is the shear rate, expressed in reciprocal seconds (s^{-1}).

11.6.3.3 Power law model

The power law model describes a fluid with a shear stress to shear rate ratio that is log-linear and has zero shear stress at zero shear rate. Two parameters, the power law flow behavior index (n) and the consistency index (k) are used to describe a power law fluid. For shear-thinning (pseudo-plastic) fluids, n is greater than zero and less than one. For shear thickening (dilatant) fluids, n is a number greater than one. If n is equal to one the fluid is Newtonian. A rheogram of a power law fluid on Cartesian (rectangular) coordinates is a curved line passing through the origin. When plotted with log-log coordinates, a power law fluid forms a straight line with a positive slope. The preferred method for calculating the behavior index and consistency index of a power law fluid is through a mathematical regression analysis using all of the data points according to Equation (57). The use of a computer program or spreadsheet is recommended to perform the regression analysis. Two-point calculations are not accurate and shall not be used.

In SI or USC units:

$$\tau = k \times \gamma^{n} \tag{57}$$

where

τ is the shear stress, expressed in pascals (pounds-force per 100 square feet);

k is the consistency index, expressed in Pa$\bullet$s^{n} (lbfl.s^{n}/100 ft^2);

γ is the shear rate, expressed in reciprocal seconds (s^{-1});

n is the power law flow behavior index, dimensionless.

11.6.3.4 Herschel-Bulkley Model

The Herschel-Bulkley model combines a portion of the Bingham plastic model with a portion of the power law model. It describes a fluid with a shear stress to shear rate ratio that is log-linear (like a power law fluid) after having exceeded some minimum shear stress (like a Bingham plastic fluid). Three parameters, the Herschel-Bulkley flow behavior index (n), the consistency index (k), and the yield stress (τ_{y}) are used to describe a Herschel-Bulkley fluid. The method for calculating the parameters of a Herschel-Bulkley fluid is through a mathematical regression analysis using all of the data points according to Equation (58). The use of a computer program is recommended to perform this complex data analysis.

In SI or USC units:

$$\tau = \tau_{y} + \left(k \times \gamma^{n} \right) \tag{58}$$

where

τ is the shear stress, expressed in pascals (pounds-force per 100 square feet);

τ_{y} is the Herschel-Bulkley yield stress, expressed in pascals (pounds-force per 100 square feet);;

n is the Herschel-Bulkley flow behavior index, dimensionless;

γ is the shear rate, expressed in reciprocal seconds (s^{-1});

k is the consistency index, expressed in Pa$\bullet$s^{n} (lbf$\bullet$s^{n}/100 ft^2).

It should be noted that the Herschel-Bulkley model's governing equation reduces to other rheological models under certain conditions. When the, the flow behavior index is equal to one the model reduces to the Bingham plastic model. When the yield stress is equal to zero, the Herschel-Bulkley model reduces to the power law model. Consequently, the Herschel-Bulkley model can be considered the unifying model that fits Bingham plastic fluids, power law fluids, and everything in between.

In the Herschel-Bulkley model, the consistency index can be considered analogous to the plastic viscosity term in the Bingham plastic model, although the numerical value will usually differ significantly. Similarly, the fluid yield stress in the Herschel-Bulkley model is analogous to the yield stress (yield point) in the Bingham plastic model but the numerical value will usually be lower.

12 Well-simulation Slurry Stability Tests

12.1 Introduction

The purpose of this test is to determine the static stability of a cement slurry. The cement slurry shall be conditioned to simulate dynamic placement in a wellbore. The slurry is then left static to determine if free fluid separates from the slurry or to determine if the cement slurry experiences particle sedimentation. Free fluid can be formed with minimal sedimentation, and sedimentation can take place without free fluid being formed. Therefore, both the free fluid result and the sedimentation result are required in order to understand the static stability of the slurry under downhole conditions. Excessive free fluid and sedimentation are considered detrimental to cement sheath quality. The amount of free fluid and sedimentation or both that is acceptable varies with the application.

Apparatus required for slurry stability test shall be verified and/or calibrated if not in calibration as per requirements of Annex B.

12.2 Slurry Mixing and Conditioning

Cement slurry shall be prepared and conditionned according to Section 5. If performing the sedimentation test described in 12.5, immediately after mixing the slurry measure the density of the slurry using a pressurized fluid density balance.

After conditioning, proceed with either 12.3 or 12.4 for a free-fluid test or 12.5 for a sedimentation test.

12.3 Free-fluid Test at Atmospheric Pressure with Static Period at Ambient Temperature or Temperature Equal or Less than 88 °C (190 °F)

12.3.1 Test preparation

Free-fluid test at ambient temperature shall require using a graduated cylinder with zero-to-250 mL graduated portion of the cylinder no less than 232 mm (9.1 in.) nor more than 250 mm (9.8 in.) in length, graduated in 2 mL increments or less.

Free-fluid test with heated static period at temperature (T_{PBHC}) equal or less than 88°C (190 °F) shall required following apparatus:

A clear, glass tube shall be required. The ratio of the slurry-filled length to the inside tube diameter shall be greater than 6:1 and less than 8:1, with a mark at the required fill level. The clear tube shall be inert to well cements and shall not deform during the test. The free-fluid test slurry volume shall be between 100 mL and 250 mL, inclusive. To determine free-fluid volume it should be recommended to graduate the tube in 2 mL increments or less below the required fill level mark.

A test chamber shall be preheated (or precooled) for aging the slurry during the static period to T_{PBHC} or 88 °C (190 °F), whichever is cooler. A boiling point for water of 100 °C (212 °F) is assumed. If the boiling point of water due to elevation of the testing location is less than 100 °C (212 °F), adjust the temperature accordingly. This chamber may be an atmospheric heating or cooling bath/oven/jacket/chamber The test chamber shall be able to heat or to cool the entire slurry

NOTE   Optionally graduated cylinder required by free fluid test at ambient temperature should be use for test at temperature equal or less than 88 °C (190 °F).

12.3.2 Free-fluid tests at atmospheric pressure—Procedure

To perform free-fluid tests at atmospheric pressure either with static temperature less than or equal to 88 °C (190 °F) or at ambient temperature, the following procedure shall be applied.

a)   Pour conditioned slurry into the clear tube to the fill mark. Document the slurry volume placed in the tube and the tube dimensions. Cover the opening of the tube to prevent evaporation.

For test at ambient temperature, the cement slurry is poured into the graduated glass cylinder to the 250 mL-mark. Seal the cylinder with plastic film wrap or equivalent material to prevent evaporation.

The test duration is 2 hr, starting from the time the slurry is poured into the tube/cylinder.

b)   Immediately place the clear tube in a heating or cooling chamber that is preheated or precooled to $T_{\text{PBHC}} \pm 3$ °C (± 5 °F).

The tube (graduated cylinder) may be tilted to simulate wellbore angle, if desired. Angles are referenced from vertical in the same way as the deviation angles of wells. Thus, vertical is 0° and horizontal is 90°.

Appropriate precautions shall be taken to ensure the static curing is performed at essentially vibration free conditions.

c)   For heated or cooled test, maintain for the duration of the test the temperature at $T_{\text{PBHC}} \pm 3$ °C (± 5 °F) or at 88 °C ± 3 °C (190 °F ± 5 °F), whichever is the lower.

d)   At 2 hr ± 1 min, measure the volume of free-fluid (clear or colored fluid on top of the cement slurry inside the tube/cylinder) with a precision of 1 mL.

Calculate the volume fraction, φ, of free-fluid, expressed as a percent as given Equation (59).

$$\varphi = \frac{V_{\text{F}}}{V_{\text{S}}} \times 100 \tag{59}$$

where

   V_{F} is the volume of free fluid, expressed in milliliters;

   V_{s} is the initial volume of slurry, expressed in milliliters.

Report the volume fraction as a percent of free-fluid, mixing and conditioning method, time and temperature, tube dimension, test temperature and angle. The form shown as Table 10 may be used.

12.4 Free-fluid Tests under Pressure with Static Temperature Greater than 88 °C (190 °F)

12.4.1 Test preparation

Free-fluid test under pressure with heated static period greater than 88°C (190 °F) shall required following apparatus:

A clear, glass tube as described in 12.3.1 shall be required. The free-fluid test slurry volume shall be between 100 mL and 250 mL, inclusive.

A test chamber shall be preheated for aging the slurry during the static period to T_{PBHC}. This chamber shall be a suitable pressurized heating chamber that uses hydrocarbon oil to transmit heating/cooling to the slurry. Hydrocarbon oil shall have a flash point that satisfactorily meets all safety requirements to perform the test.

12.4.2 Free-fluid tests under pressure—Procedure

To perform free-fluid tests under pressure with static temperature greater than 88 °C (190 °F), the following procedure shall be applied.

a) Pour conditioned slurry into the clear tube to the fill mark. Document the slurry volume placed in the tube and the tube dimensions. To avoid disturbing the slurry when it is placed in the chamber, oil shall be carefully placed in the tube above the slurry.

b) Without covering the tube, place it in an oil-filled heating chamber preheated to 88 °C (190 °F). Optionally, tilt the tube to simulate hole angle (see 12.3.2).

   NOTE   A boiling point for water of 100 °C (212 °F) is assumed. If the boiling point of water due to elevation of the testing location is less than 100 °C (212 °F), adjust the temperature accordingly.

c) Apply sufficient pressure to prevent boiling of the slurry. Further heat the slurry to $T_{\mathrm{PBHC}} \pm 3$ °C (± 5 °F) in the time required to take the slurry from a depth with 88 °C (190 °F) circulating temperature to T_{PBHC}. Some heating chambers may not be able to heat fast enough; in that case heat as fast as possible but minimize overshooting the T_{PBHC}.

d) Maintain the slurry at T_{PBHC} until it is time to cool the chamber to 88 °C (190 °F). The time required to cool various pieces of equipment from elevated temperatures to 88 °C (190 °F) will vary. Maintain sufficient pressure on the curing chamber so the slurry cannot boil (see Table 4).

   The pressure applied can simulate bottomhole conditions, if desired. The formulas found in 9.4 can be used to aid in selecting pressure- and temperature-change rates.

   Take appropriate precautions to ensure that the static curing is performed in essentially vibration-free conditions. Avoid constant pump cycling, in order to prevent vibration.

e) The 2 hr-test period is initiated when the conditioned slurry is poured into the tube. Slurries need to be cooled to 88 °C (190 °F) before the free fluid can be measured. This cooling time is part of the 2 hr-test period.

f) At the end of the 2 hr ±1 min test period, measure the volume of free-fluid (clear or colored fluid on top of the cement slurry inside of the clear tube). Free-fluid for slurries immersed in hydrocarbon oil collects above the cement but below the oil. Measure the volume of the free fluid with a precision of 1 mL.

The volume fraction, as a percent of free-fluid is then calculated in accordance with Equation (59). The results may be reported on a form such as the one in Table 10.

12.5 Sedimentation Test

12.5.1 Preparation

The sedimentation tube (Figure 15) shall have an inner diameter of 25.4 mm ±0.5 mm (1.00 in. ±0.02 in.). The tube length shall be a minimum of 100 mm (3.95 in.). The most common tube length is approximately 200 mm (7.9 in.). The tube shall be inert to well cements and shall not deform during the course of the test. Liberally grease the inside of the tube and all joints to ensure that it is leak-tight and so that the set cement can be removed without damage. Ensure the grease on the inside is smooth so that it does not produce any grease pockets in the cement after the cement has set. Ensure that the grease extrudes to the outside of the tube when the two tube halves are fitted together.

The sedimentation tube and a water-filled chamber (or oven) shall be preheated or precooled to T_{PBHC} ±3 °C (±5 °F) or to 88 °C (190 °F), whichever is cooler.

NOTE   The 88 °C (190 °F) safety temperature assumes a boiling point for water of 100 °C (212 °F).

Dimensions in millimeters (inches).
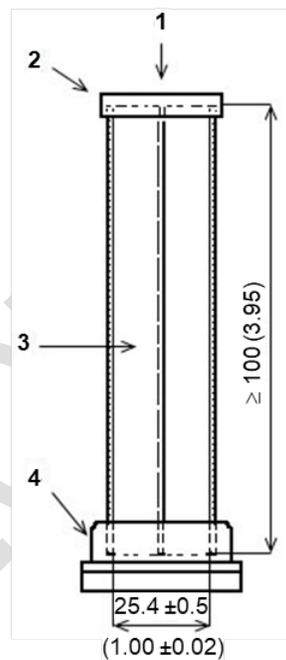
Key

1   vent hole
2   lid
3   split in tube
4   base

NOTE   Common tube length 200 mm (7.9 in.)

Figure 15—Typical Sedimentation Tube

12.5.2 Sedimentation Test Procedure

In order to evaluate cement slurry sedimentation, the following procedure shall be applied.

a)   Pour conditioned slurry into the dry preheated/precooled sedimentation tube until it is approximately 20 mm (0.75 in.) from the top.

     Puddle the slurry in the tube to dislodge any air bubbles, then fill the tube completely. A top closure that allows pressure communication may be used to prevent spillage of the slurry.

b)   Place the filled tube in the preheated/precooled chamber in a vertical position. In this test, the tube is not inclined at the well angle.

c)   Adjust the slurry temperature further to simulate temperature changes in the wellbore. Maintain pressure of 3400 kPa (500 lbf/in.²) minimum, but no lower than that listed in Table 4, to avoid boiling and to compress air bubbles. The pressure applied may simulate bottomhole conditions, if desired. Avoid constant pump cycling in order to minimize vibration. Temperature information in Annex D and schedules in Section 7 can be used to aid in selecting the temperature and pressure.

d)   Cure the slurry for 24 hr or until set before removing it from the chamber.

e)   Cool the chamber to 88 °C (190 °F) or less, if required. Release pressure from the chamber, if required. Remove the tube from the chamber and bring the tube to 27 °C ±3 °C (80 °F ±5 °F) by placing it in a water bath.

f)   After the tube has cooled, remove the cement from the tube. Keep the cement sample immersed in water, as much as possible, to prevent it from drying out. Carefully remove any grease adhering to the specimen.

g)   Measure and record the length of the cement specimen. Mark the specimen approximately 20 mm (0.75 in.) from the bottom and from the top. Divide the section between the marks by further marks into segments of roughly equal length with a minimum of four segments (n segments are prepared). Mark the segments to keep track of their order.

h)   Break or cut the sample at these marks. Return all the segments, including the end pieces, to the water until each is weighed.

i)   To determine the density of the segments, place a beaker containing water on the balance and tare the balance to zero. A balance with a precision of 0.01 g is necessary.

j)   Remove a segment (number i) to be measured from the water and gently dry it.

k)   Place this segment (i) on the balance beside the beaker.

l)   Record the dry mass ($m_{air,i}$) and remove the segment from the balance.

m)   Tare the balance if necessary.

n)   Place a noose of thin string around the segment (i) (dental floss works well).

o)   Suspend the segment in the beaker such that the segment is totally surrounded by water. The segment shall not touch the bottom or sides of the beaker. Air bubbles shall not be clinging to the segment.

p)   Record the wet mass ($m_{water,i}$) with the segment suspended in water.

q)   Remove the segment from the water and tare the balance.

r)   Repeat the procedure item j) to item q) for each segment.

12.5.3 Sedimentation test calculation

By applying Archimede principle, a relative density variance and the difference from top to bottom of sedimentation test sample shall be calculated as per following calculations.

a) Calculate the relative density ($d_{\text{rel,i}}$) of each cement segment (i) (n_s total number of segment) as given Equation (60).

$$d_{\text{rel,i}} = \frac{m_{\text{air,i}}}{m_{\text{water,i}}} \tag{60}$$

where

$m_{\text{air,i}}$ is the mass of cement segment (i) in air (dry mass), expressed in grams;

$m_{\text{water,i}}$ is the mass of cement segment (i) in water (wet mass), expressed in grams.

b) Determine the average relative density d_{rel}, of the set segmented cement sample as given Equation (61)

$$d_{\text{rel}} = \frac{1}{n} \times \sum_{\text{i}=1}^{\text{i}=n} d_{\text{rel,i}} \tag{61}$$

where

n_s is the total number of segment of the sedimentation test sample .

These results should be used to construct a relative density profile in comparison to the average relative density of the sedimentation test sample.compared to the for the entire specimen based on following calculations.

A density profile can be constructed, with densities calculated from relative densities as given item c) Equation (62) expressed in kilograms, or Equation (63) expressed in pounds-mass per gallon.

c) Determine density ($\rho_{\text{set,i}}$) of each cement segment (i) based on its relative density ($d_{\text{rel,i}}$), when expressed in kilograms per liter as per Equation (62) :

$$\rho_{\text{set,i}} = d_{\text{rel,i}} \tag{62}$$

or, when expressed in pounds-mass per gallon as given Equation (63):

$$\rho_{\text{set,i}} = 8.34 \times d_{\text{rel,i}} \tag{63}$$

d) Determine average set cement density of the segmented sample, ρ_{set}, expressed in kilograms per liter as given Equation (62) or expressed in pounds-mass per gallon as given Equation (63) from its relative density d_{rel}, given Equation (61).

NOTE   It is normal for cement slurries to experience a small density increase upon setting.

For each cement segment (i) , the percent density variance, $\Delta\rho_{sc,i}$, between the set cement segment sample and the average set cement shall be calculated as given Equation (64).

$$\Delta\rho_{sc,i} = 100 \times \frac{\rho_{set,i}}{\rho_{set}} \tag{64}$$

where

$\Delta\rho_{sc,i}$ is the density variance of the set cement segment (i), expressed in percent;

$\rho_{set,i}$ is the density of the set cement segment (i) expressed in kilograms per liter (pounds-mass per gallon);

ρ_{set} is the average set cement density expressed in kilograms per liter (pounds-mass per gallon).

The results may be reported on a form such as the one in Table 10. Report should also include following measured variances:

— Difference between average set cement density and slurry density slurry density;

— Difference between set cement segment heavier density and average set cement density;

— Difference between set segment segment lighter density and average set cement density.

The density variances between slurry and set well cements, and the density difference from top to bottom of a sedimentation test sample, can vary greatly and depend on many factors. The amount of density difference that is acceptable varies with the application.

The heating/cooling, pressurizing, and cooling information that is listed in the report form (Table 10) will allow other laboratories to reproduce the test. The information requested is sufficient only if the heating/cooling rate, pressurizing rate, and cool-down rate are linear. If the rates are not linear, specify the exact heating/cooling, pressurizing, and cool-down schedules on the form.

Table 10—Optional Free-fluid and Sedimentation Results-report Form

Slurry Mixing and Conditioning

Cement temperature: _____

Mix water temperature: _____

Slurry initial temperature: _____

Slurry final conditioning temperature: _____

Time to final temperature: _____

Optional additional conditioning period: _____

Initial pressure: _____

Final pressure: _____

Time to final pressure: _____

Free-fluid Test

    Ambient Temperature: _____   250 mL Graduated cylinder:_____

    Free fluid clear glass tube:

       Length of graduated tube section: _____

       Graduated tube inner diameter: _____

    Test angle: _____ (Angle is measured from vertical.)

Test at T_{PBHC}

    Slurry volume (V_s): _____

    Final temperature: _____

    Time to final temperature: _____ min

    Initial test pressure: _____

    Final test pressure: _____

    Time to final test pressure: _____

    Time to cool the slurry to 88 °C (190 °F): _____

Measured free-fluid volume (V_F): _____ mL

Volume fraction free-fluid (φ): _____%

Sedimentation Test

Preheated or precooled chamber temperature: _____

T_{PBHC}: _____

Time from 88 °C (190 °F) to T_{PBHC} (if applicable): _____

Initial test pressure: _____   Final test pressure: _____

Time to final test pressure: _____   Time at T_{BHS}: _____hours

Time to cool the chamber to 88 °C (190 °F): _____

Section 7 schedules employed: _____Yes or _____ No

If Yes, schedule number: _____

Length of sedimentation tube: _____

Length of set specimen: _____

Design slurry density: _____

Measured slurry density: _____(pressurized fluid balance)

Density profile:

Segment No.	Dry Mass $m_{\mathrm{air},i}$	Wet Mass $m_{\mathrm{water},i}$	SG $d_{\mathrm{rel},i}$	Density $\rho_{\mathrm{set},i}$	% Density Variance $\Delta\rho_{\mathrm{sc},i}$
1 (top)					
2					
3					
4					
5					
6					
7					
8					
9					
10					
bottom					
Average	$\Sigma=$	$\Sigma=$	$d_{\mathbf{rel}}=$	$\rho_{\mathbf{set}}=$	
Maximum $\rho_{\mathbf{set,i}} - \rho_{\mathbf{set}} =$					
Minimum $\rho_{\mathbf{set,i}} - \rho_{\mathbf{set}} =$					
NOTE Use as many rows as needed for the number of segments.					

13 Compatibility of Wellbore Fluids

13.1 General

13.1.1 Introduction

This procedure is intended to be used for determining the degree of compatibility of wellbore fluids in cementing operations and includes the examination of rheological properties, gel strength, thickening-time, compressive strength, solids suspension, and spacer surfactant screening. By the use of this procedure, incompatibility tendencies can be determined and the selection of proper preflushes and/or spacers can be made. User discretion shall be exercised in the selection of the portion(s) of the procedure needed and interpretation of the results. The following test procedures shall be the same for preflushes and spacers, therefore the term spacer is used to refer to both fluids.

NOTE   Hereafter, the term "mud" will be used to refer to any wellbore fluid, including drilling fluids, completion fluids, etc.

The user shall consider potential impacts when deciding whether to test mixtures of the mud with the cement. Most of the time, it is presumed that the mud and cement are incompatible and that a spacer will provide adequate separation between the two. The effects of standoff (centralization) of the casing and well geometry and rheologies and flow regimes of the fluids should be carefully considered in the design of the physical displacement process to minimize the chance of cement and mud contact.

13.1.2 Test sequence—Flow chart

The user shall use discretion in the determination of which tests to perform and the sequence in which to perform them. A compatibility testing flow chart, Figure 16, is provided as an aid in optimizing the testing protocol.

13.2 Preparation of Test Fluids

13.2.1 General

13.2.1.1   Observe all proper laboratory safety requirements and procedures for working with fluids under temperature and pressure. Note the flash points of all fluids before testing and ensure proper ventilation in the work area before starting any tests. Special care shall be taken when working with flammable fluids.

13.2.1.2   The maximum temperature for testing at atmospheric pressure shall be 88 °C (190 °F) or safely below the lowest flash point, whichever is lowest. When conditioning is done at a higher temperature under pressure, the fluid shall be be cooled to the test temperature or below flash point, whichever is lower, before releasing the pressure.

NOTE   If the boiling point of water at the testing facility is less than 100 °C (212 °F), adjust maximum testing temperatureat atmospheric pressure accordingly.

13.2.2 Preparation of spacer

13.2.2.1   The spacer shall be freshly prepared in accordance with the supplier's instructions. A 500 mL volume is normally sufficient to perform a single test.

13.2.2.2   Spacer fluids shall be conditioned as detailed in 5.4. Devices which will not expose the fluids to oil are recommended (e.g. stirred fluid-loss cell, roller ovens). Observe all safe-handling procedures for fluids being tested.
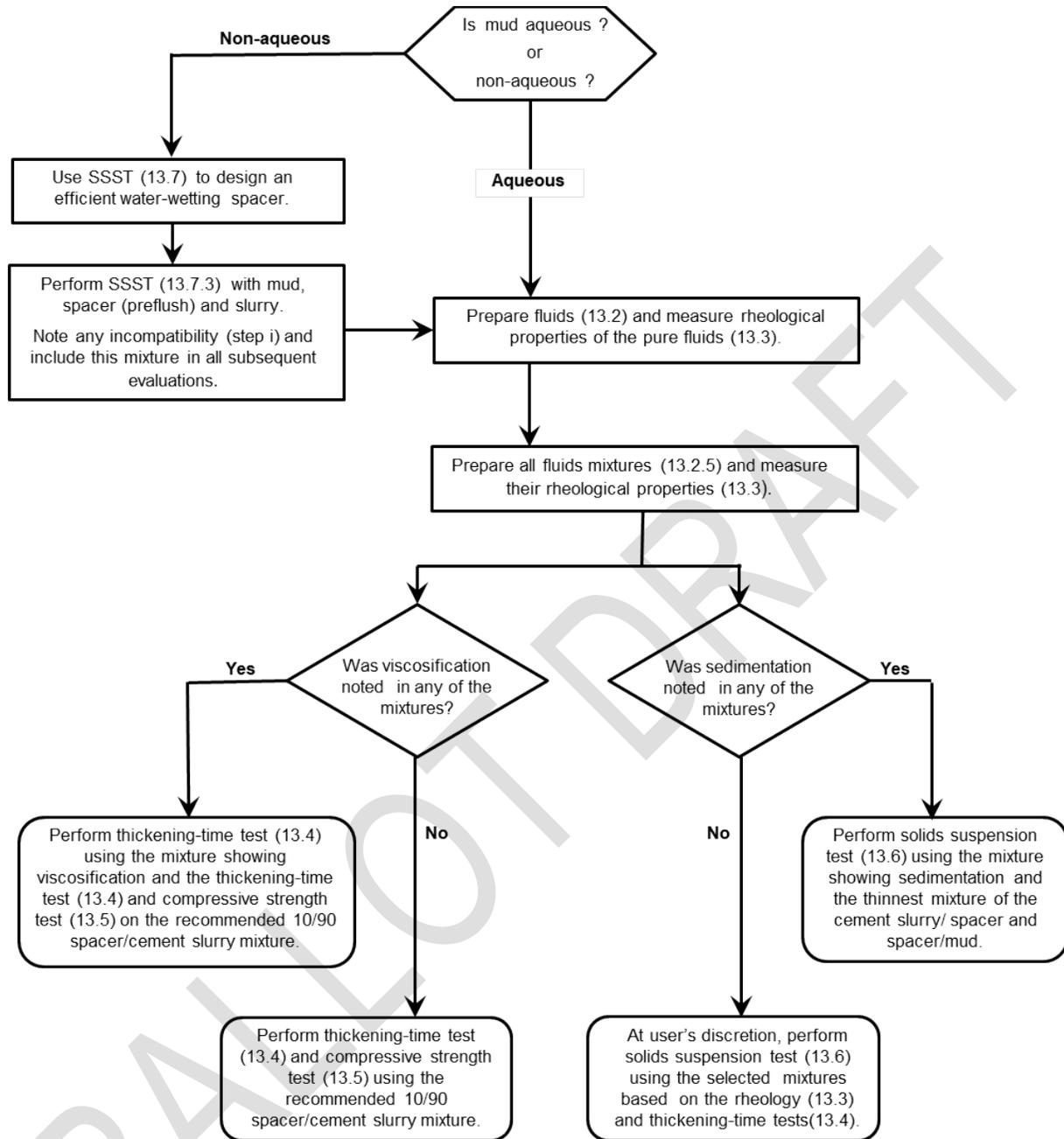
Figure 16—Compatibility Testing Flowchart

13.2.3   Preparation of drilling fluid or other field fluid

13.2.3.1   Representative samples of field mud or other field fluid shall be used. Hereafter, the term "mud" will be used to refer to any wellbore fluid, including drilling fluid, completion fluid, etc... Thoroughly mix mud samples prior to testing. Hot-roll or shear samples shall be recommended by the supplier or in accordance with API 13I .

13.2.3.2   If field mud is not available, laboratory samples may be prepared. Such laboratory-prepared mud samples are not representative of the field mud, since they do not contain drill solids and formation fluids as field muds do. Laboratory-prepared fluid samples shall require additional preparation, such as static aging or hot-rolling, to simulate field fluid properties more closely. Care shall be taken in interpreting results when laboratory-prepared samples are used. Confirmation with a field sample of the mud shall be recommended once it is available.

13.2.3.3   Condition the fluid at T_{PBHC} for 16 hr using a roller oven or for 30 min on a pressurized consistometer, or on a stirred fluid-loss cell, at T_{PBHC} for 30 min on an atmospheric consistometer [maximum of 88 °C (190 °F)].

NOTE   There is danger of contamination with oil when the fluid is conditioned in a pressurized consistometer.

13.2.4 Preparation of cement slurry

A fresh quantity of cement slurry shall be prepared according to Section 5 or Annex A for each test and conditioned according to 5.4.

13.2.5 Preparation of fluid mixtures

13.2.5.1   Mixtures and pure fluids to be used for rheological properties, gel strength, solids suspension, thickening-time, and compressive-strength testing shall be prepared as described in this subsection (see Table 11 or Table 12). Data for uncontaminated test fluids shall be obtained before mixtures are prepared. All fluid mixtures in this section are expressed as volume fraction (percent) of the total mixture.

13.2.5.2   The volume of the mixture shall be sufficient to perform the desired compatibility test procedure.

If there is sufficient quantity of cement, mud, and spacer available, mixing scheme procedure using fresh fluids to prepare each fluid contamination ratio mixture is given Table 11.

Table 11—Drilling Fluid or Cement Slurry to Spacer Mixtures for Compatibility Testing.

No.	Ratio Mud or Cement Slurry to Spacer vol. %	Mixing Scheme
1	100/0	400 L of Mud (or Cement Slurry)
2	95/5	380 mL of Mud (or Cement Slurry) + 20 mL Spacer
3	75/25	300 mL of Mud or Cement Slurry + 100 mL of Spacer
3	50/50	200 L of Mud or (Cement Slurry + 200 mL of Spacer
4	25/75	100 mL of Mud or (Cement Slurry) + 300 mL of Spacer
5	5/95	20 mL of Mud or (Cement Slurry + 380 mL of Spacer
6	0/100	400 mL of Spacer
7	25/50/25 mud/spacer/cement slurry	100 mL of Mud + 200 mL Spacer + 100mL Cement Slurry
NOTE To reduce volume requirements of mud, spacer, and cement slurry to prepare mixtures for compatibility testing, follow procedure and volume requirements given in Table 12		

In order to reduce volume of fresh fluids to be used to prepare each fluid contamination ratio mixture for compatibility testing, a volumetric procedure based on combination of previously tested mixtures is given Table 12.

Procedure using fresh fluids to prepare each fluid contamination ratio mixture (see Table 11) should be preferred over combining previously tested mixtures (see Table 12; e.g. 75/25 and 25/75 to form a 50/50 mixture, and should improve test accuracy and repeatability.

NOTE   According availability of base fluids, Table 12 mixing scheme can be modified based on smaller volumes of fluids.

Table 12—Combination Fresh Fluids and Previously Tested Mixtures for Compatibility Testing.

Mixture No.	Ratio Mud or Cement Slurry to Spacer vol. %	Mixing Scheme
	100% Spacer	Fresh fluid
	100 % Mud (or Cement slurry)	Fresh Fluid
1	95/5	380 mL Mud (or Cement Slurry) + 20 mL of Spacer
2	75/25	300 mL mix No. 1 + 80 mL of Spacer
3	5/95	20 mL Mud (or Cement Slurry) + 380 mL of Spacer
4	25/75	80 mL Mud (or Cement Slurry) + 300 mL of mix No. 3
5	50/50	Equal parts 200 mL of mix No. 2 + 200 mL of mix No. 4
6	25/50/25 Mud/Spacer/Cement Slurry	Equal parts of mix No. 5 Mud/Spacer and mix No. 5 Cement/ Spacer
NOTE Using this procedure, volume requirements are in the range of 500 mL to 550 mL of mud , spacer and cement slurry.		

13.2.5.3   Since compatibility can vary with test temperature, tests may be performed at well-site ambient temperature and/or at T_{PBHC}. Consideration shall be given to the temperature at which mixing will occur in the well when selecting the temperature for compatibility testing. Due to safety considerations, maximum test temperature shall be limited to 88 °C (190 °F) or safely below the lowest flash point of any of the fluids to be tested when using atmospheric pressure procedures.

13.2.5.4   After conditioning the fluids at test temperature and cooling to 88 °C (190 °F), if conditioned at a higher temperature, an appropriately sized spatula or glass rod shall be used to gently stir one test fluid in a beaker or plastic cup as the other test fluid is being added. The minor component shall be added to the major component. Observe the mixture for any signs of incompatibility (e.g. viscosification, clabbering, flocculation, solids settling, solids flotation, or fluid separation) during or after mixing. Signs of incompatibility may be seen during addition that are not observed after the mixture has been completely mixed. Continue stirring until the mix is homogenous, but no less than one minute. If signs of incompatibility are seen, note the approximate volume fraction and include this volume fraction in the testing routine

13.3 Rheological Properties

Rheological properties shall be determined on mixtures of cement slurry/mud (at user's discretion), cement slurry/spacer, and mud/spacer. The recommended ratios are 95/5, 75/25, 50/50, 25/75, and 5/95 for each fluid combination as well as a 25/50/25 mixture of mud/spacer/cement slurry. Any ratio of fluids for which possible incompatibility or viscosity increases are observed during the mixing of the fluids (13.2.5) are also recommended for rheological properties testing. At the user's discretion, the various-ratio mixtures may be prepared in accordance with Table 11 or Table 12 if sample volumes are too limited to permit use of fresh mixtures for each mix ratio. The rheological properties shall be measured in accordance with Section 11. Additional conditioning of the mixtures is not required. Ramp up and ramp down rheology readings shall be taken as per 11.4 g) to 11.4 i). The data may be recorded on a form similar to the one found in Table 13.

After testing, gently pour off the fluid and observe the bottom of the viscometer cup for signs of solids sedimentation.

NOTE   Different sizes of cups are available for use withviscometers. As the cup dimensions may have an impact on the readings obtained, the same size cup shall be used throughout the testing.

Table 13—Rheological Compatibility of Mud, Cement Slurry, and Spacer

Cement slurry:_____

Spacer: _____

Mud type:_____ _____Field Sample (date):_____   Lab Sample:_____

Rotor-Bob/Torsion device configuration: _____

Static time used for gel strength determination: _____

Fluid Mixture	Test Temp.	Average Viscometer Dial Readings r/min							API Gel Strength	Plastic Viscosity μ_p	Yield Stress τ_y
	° C (° F)	300	200	100	60	30	6	3	Pa (lbf/100 ft^2)	Pa•s (cP)	Pa (lbf/100 ft^2)
100 % mud											
95 % mud 5 % spacer											
75 % mud 25 % spacer											
50 % mud 50 % spacer											
25 % mud 75 % spacer											
5 % mud 95 % spacer											
100 % spacer											
95 % spacer 5 % cement											
75 % spacer 25 % cement											
50 % spacer 50 % cement											
25 % spacer 75 % cement											
5 % spacer 95 % cement											
100 % cement											
25 % mud 50 % spacer 25 % cement											

13.4 Thickening-time

13.4.1 Thickening-time tests shall be performed on mixtures of spacer/cement slurry to determine if the spacer causes gelation or other adverse effect on the thickening-time. The recommended ratio is 10/90 spacer/cement slurry. The thickening-time test shall be performed in accordance with Section 9. At the user's discretion, tests may be performed on mixtures of spacer/cement slurry, mud/spacer/cement slurry, and cement slurry/mud at any ratio.

13.4.2 If there are signs of incompatibility with any mixture containing more than 25 % cement slurry during initial mixing with the spatula or rod or when measuring the rheological properties (see 13.3), then the thickest mixture shall be tested on a pressurized consistometer for a minimum of the thickening-time of the cement slurry. This test is in addition to the 10/90 mixture of spacer/cement slurry.

13.5 Compressive Strength

Compressive-strength tests shall be performed on mixtures of spacer/cement slurry. The recommended ratio is 10/90 spacer/cement slurry. The compressive-strength test shall be conducted in accordance with either Section 7 or Section 8. At the user's discretion, tests may be conducted with any mixture of mud/cement slurry, spacer/cement slurry, or mud/spacer/cement slurry.

13.6 Solids Suspension and Gel Strength

13.6.1 General

This procedure is designed to investigate the behavior of fluid mixtures during and following cement slurry placement. Selection of the fluid mixtures and ratios shall be made based on results obtained from the rheology test (13.3) or the thickening-time test (13.4), at the user's discretion.

13.6.2 Procedure

To investigate behavior of fluid mixtures during and following cement slurry placement the following procedure shall be applied.

a) If there were signs of sedimentation or gelation in the rheological properties test, test the ratio(s) for which the sedimentation or gelation was most evident. If there were no signs of sedimentation or gelation, test the mixture with the lowest viscosity.

b) Using the selected mixtures, initiate thickening-time tests in accordance with Section 9.

c) After the temperature has reached T_{PBHC}, observe the consistency, then stop the motor and leave the mixture static for a minimum of 10 minutes or a time period appropriate to a scheduled application following cement slurry placement (e.g. as example, time to set a liner top packer).

d) Start the motor and observe for signs of settling or gelation indicated by a momentary or sustained increase in the consistency.If the pin shears, discontinue the test and proceed to itemf).

e) Continue agitation until 1 hr after reaching T_{PBHC} or until sustained 70 B_{c}, whichever occurs first Observe the consistency and then stop the motor.

   Note on report if the test passed 70 B_{c} or report the consistency when the test is stopped after 1 hr at T_{PBHC}.

f) Immediately cool to 88 °C (190 °F), if necessary and, after a minimum 10 min static period, remove the slurry cup without any agitation.

g)   Remove the top of the slurry cup (end with paddle shaft) without removing the paddle and gently pour the slurry from the cup, observing for signs of sedimentation or gelation.

13.7 Spacer Surfactant Screening Test

13.7.1 Introduction

The spacer surfactant screening test (SSST) procedure is specific to evaluation of a spacer's ability to promote a water-external phase emulsion when mixed with a non-aqueous fluid (NAF). This procedure facilitates the evaluation and the selection of proper spacers and/or surfactants in the spacer. This procedure shall help to:

a)   evaluate the effectiveness of surfactant/spacer systems (screening),

b)   evaluate compatibility of NAF and aqueous spacer mixtures, and

c)   evaluate compatibility of NAF/spacer/cement slurry mixes.

This procedure does not address bulk displacement issues or quantify the degree of "water-wetting" by the spacer. It measures the ability of the spacer and surfactants to convert the mixture to a water-external phase that is critical for water wetting. Additional tests may be required to evaluate the spacer/surfactant's ability to remove mud film from pipe and formation surfaces. The procedure is applicable to aqueous spacer systems only. This procedure is not suitable for evaluating non-aqueous or non-conductive spacer systems or mixtures of surfactants in base oils.

13.7.2 Method and Apparatus

The SSST apparatus provides a continuous measurement of the electrical conductivity between electrode surfaces. Any apparatus that has the ability to measure the conductivity of a fluid while being mixed can be used for this procedure. From the conductivity measurements, the emulsion state of the fluid can be inferred if the titrating spacer fluid is conductive, and the titrated NAF is not. Normally, oil-external fluids are not electrically conductive. Water-based or water-external emulsion spacers are electrically conductive with the conductivity dependent on the solution chemistry.

Apparatus shall be calibrated according to the requirements for blenders in Annex B and according to the manufacturer's instructions.

13.7.3 Procedure

This is an atmospheric pressure test the following procedure shall be applied. Fluids shall be prepared according to 13.2 and conditioned according to 5.4.

a)   Prepare the equipment according to instructions from the supplier.

b)   Clean and dry the test equipment.

c)   Fill the container with water, then heat the SSST fluid container to test conditions to maintain the temperature of the test fluids. Remove and save the heated water before continuing to the next step.

d)   Add conditioned spacer to cover the electrodes.

e)   Measure the fluid temperature with a temperature-measuring device in direct contact with the fluid. Note that the thermocouple supplied with the apparatus may not measure the temperature of the fluid. Record the temperature.

f) Establish the baseline conductivity of the spacer at test temperature. Adjust the conductivity meter setting to a value between 50 % and 75 % of full scale. Note that some instruments use an ohmmeter and adjustment is not necessary. Record the value.

g) Remove the spacer and clean with water from item c).

h) Pour the minimum volume of conditioned, non-aqueous test fluid required to fully immerse the electrodes into the SSST apparatus. Record the starting volume of NAF. Measure the fluid temperature. Begin stirring at a rate sufficient to maintain a vortex. Continue stirring until the NAF is at test temperature and record the temperature. Throughout the test, adjust the stirring rate as required to maintain a vortex. Avoid stirring at rates that would cause air-entrainment that may affect readings and surfactant performance.

i) Slowly titrate the spacer into the NAF as it is being stirred in the test apparatus and periodically record conductivity vs the volume added. Ensure that the conductivity reading is stable before recording the result and adding additional spacer. Observe for signs of incompatibility and note the volume of spacer added if incompatibility is seen.

   Continue to titrate the spacer into the NAF until the conductivity reaches a plateau.

   If a volume of spacer equal to the volume of NAF is added (50 % spacer) and the conductivity plateau has not been reached, one half of the fluid may be removed to prevent overfilling the apparatus as the titration continues. Care must be taken to ensure calculation of spacer percent is based on the volume of spacer added to the volume of NAF remaining in the SSST apparatus. If spacer equal to 75 % of the NAF/spacer mixture is added before full conductivity is reached, consideration shall be given to redesign of the spacer/surfactant formulation. Note that calculations of the spacer added must be based on the amount of NAF that has been left in the apparatus.

j) Graph the test results as conductivity vs the volume percentage of spacer in the NAF/spacer mixture and assess the spacer effectiveness. For example, if 150 mL of spacer is added to a starting fluid volume of 300 mL, the volume is reported as 33 % (150 mL /450 mL).

   Curves showing typical responses are shown in 13.8.7, Figure 17.

k) Evaluate the NAF/spacer fluid titration results according to 13.8.7. If results are acceptable, continue to item l). If not, redesign the spacer and repeat item a) through item j).

l) When the spacer/surfactant mix has been optimized, remove, and set aside all but 300 mL of the final spacer/NAF mixture from item i). While stirring the mix remaining in the blender at low speed (maintain a slight vortex), begin adding freshly prepared and conditioned cement slurry and continue until the volume of cement slurry added equals 300 mL or until an obvious incompatibility is observed. Continue stirring this mixture at low speed (maintain a slight vortex) for a minimum of 2 min and observe the behavior of the mixture. If significant viscosification occurs, redesign the fluids, or take precautions to prevent mixing of all three components during field application.

m) After 30 min to 1 hr, observe the portion set aside in item l) for phase separation, sedimentation, or other signs of incompatibility.

n) Observe the wetting properties of the mixing container when first emptied and rinsed with water only; do not use any additional detergents or soaps for clean-up at this point. Pour a portion of the final mixture that was previously set aside into a clean, dry glass vessel or immerse a clean, dry stirring rod in the mixture. Then rinse the glass vessel or stirring rod under flowing room temperature tap water without using surfactants (detergents or soaps). After rinsing, if the water beads or a greasy film is evident on the interior of the mixing container, glass vessel, or stirring rod, it is an indication that full water wetting was not obtained.

o)   Observe the behavior when the mixing container is cleaned following the test. At times, beading of water when rinsing out the container may indicate that a full water-wetting state was not reached.

p)   Observations made during the SSST may be used to determine mixture ratios for testing for rheological properties, thickening-time, compressive strength, solids suspension, and gel strength according to the procedures described in 13.3 to 13.6.

13.8   Compatibility of Wellbore Fluids—Interpretation

13.8.1   General

The following subsections should suggest methods for evaluating the results of compatibility testing and surfactant/spacer screening.

13.8.2   Mixing

The nature of the fluid as the mixtures being prepared (13.2.5.4) shall be observed. Viscosification, clabbering, flocculation, solids settling, or fluid separation should be an indication of incompatibility of fluids. Any fluids showing such behavior shall be thoroughly tested and modified, where necessary, to eliminate such behavior.

13.8.3   Rheology

13.8.3.1   The rheological data for the mixtures of spacer/mud, spacer/cement slurry, and the mud/spacer/cement slurry shall be evaluated. When the dial reading of a mixture is between the readings of the two uncontaminated fluids at each rotational speed, the fluids are considered rheologically compatible. Otherwise, evaluate the effects of the rheological behavior on job performance at the range of shear rates expected in the well and determine whether further optimization of the spacer is required.

13.8.3.2   Compare the yield point and gel strength of mixtures to those of the base fluids. If there are significant increases in either, mud removal may be made more difficult and redesign of the spacer shall be considered.

13.8.3.3   If any mixture has significant sediment in the bottom of the rheology cup or exhibits fluid or phase separation, the effects of this behavior shall be evaluated on job performance and to consider redesigning the fluids. The solids suspension and static gel strength testing procedure (13.6) shall be performed to further evaluate the mixture.

13.8.3.4   If the fluids are redesigned, the entire compatibility testing protocol shall be repeated with the redesigned fluids.

13.8.3.5   When evaluating the rheological behavior on mud/cement slurry mixtures, the degree of incompatibility emphasizes the importance of an adequate volume of spacer in job design and of a properly optimized spacer/surfactant.

13.8.4   Thickening-time

The consistency and temperature plots for any behavior that might indicate incompatibility shall be examined. Incompatibility may be indicated by temperature oscillation, increase or decrease in the consistency, or change in the thickening-time. The effects of these behaviors shall be evaluated on job performance and to consider redesigning the fluids. If the fluids are redesigned, the entire compatibility testing protocol shall be repeated with the redesigned fluids.

13.8.5 Compressive strength

Any change in the development of compressive strength of mixtures that is inconsistent with the dilution shall be carefully considered. These effects shall be evaluated on well performance and to consider redesigning the fluids. If the fluids are redesigned, the entire compatibility testing protocol shall be repeated for the redesigned fluids.

13.8.6 Solids suspension and gel strength

Sedimentation, phase separation, or significant gel strength development of mixtures shall be evaluated carefully. Consideration shall be given to redesigning the fluids to reduce or eliminate such behavior. If the fluids are redesigned, the entire compatibility testing protocol shall be repeated for the redesigned fluids.

13.8.7 Spacer surfactant screening test (SSST)

13.8.7.1 Spacer/NAF mixes shall be evaluated from the SSST.

A comparison of typical titration curves from the SSST apparatus is shown in Figure 17. Four spacer formulations ("1," "2," "3," and "4") are depicted. Zero conductivity units represent a completely oil-wet state. In this example, the baseline for spacer conductivity is 135 conductivity units (note that some apparatuses may express conductivity as resistivity in ohms). Notice that the endpoint conductivity may exceed the spacer baseline. This is normal and is due to the fact that the water phase of NAF typically has a high salinity and thus results in a higher conductivity than the base spacer once the emulsion has been inverted to a water external phase. It does not indicate that the surfaces are actually water wet. The transitory portion of any of the curves that is greater than zero conductivity units, yet less than the endpoint, is representative of an unstable emulsion that is neither fully water external nor fully oil external. In general, the smaller the volume of spacer required to reach the endpoint, the better the performance will be in the wellbore.

Spacer formulation "1" shows a very slow increase in conductivity as the spacer is titrated into the NAF. The final conductivity is just approaching the original spacer baseline and still has not reached a stable value, indicating that satisfactory spacer performance has not been achieved. The long transition between a completely oil-wet state (i.e. zero conductivity units) and a state approaching the original spacer conductivity is indicative of a poor spacer/surfactant performance. This spacer/surfactant composition shall be considered inadequate.

Spacer formulation "2" exhibits a much steeper slope than Spacer "1" but the endpoint (the plateau on the curve) is reached prior to reaching the spacer baseline. The performance of a freshwater spacer with this type of behavior shall be considered poor.

NOTE   Care shall be taken when interpreting results obtained with spacers containing salts such as KCl. In some cases the electrical conductivity of the spacer can be higher than that of the water phase of the NAF. In such a case the electrical conductivity measured may never reach the baseline conductivity, even when a very efficient spacer is used.

Both Spacers "3" and "4" provide a stable endpoint above the baseline and would be considered effective. While Spacer "3" performs well, the performance of Spacer "4" is superior since the final volume of spacer required to reach the endpoint is less.

13.8.7.2 Mixtures during the SSSTshall be observed and noted any sedimentation, fluid separation, breaking of the emulsion, or a nonhomogeneous appearance, which are indicators of incompatibility or instability of the mix. Also note any unusual thickening or thinning of the mixture as the spacer is added to the mud.

13.8.7.3   Shall be observe the behavior of the mix as cement slurry is being added to the mud/spacer mix. Consideration shall be given to modification of the spacer to reduce or eliminate incompatibility of the three-component mix indicated by sedimentation, fluid separation, breaking of the emulsion, or a nonhomogeneous appearance. When modification of the spacer is impractical or ineffective in eliminating

the incompatibility of the three-component mix, care shall be exercised to ensure that sufficient spacer is used to minimize any chance of mixing of all three fluids in the well. Use of bottom wiper plug(s) and improving casing standoff will help to reduce the potential of mixing of the three fluids in the casing and in the annulus.
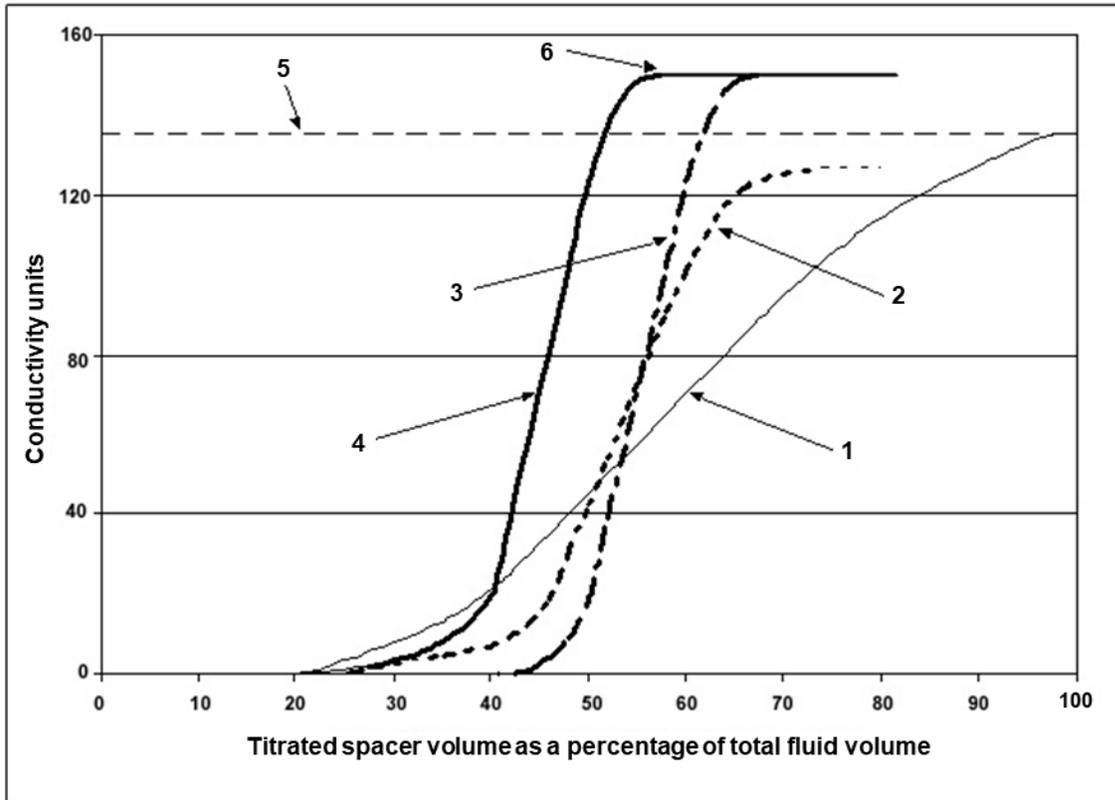
Key

x-axis   titrated spacer volume as a percentage of total fluid volume

y-axis   conductivity units

1   inefficient spacer performance

2   poor spacer performance

3   efficient spacer performance

4   most efficient spacer performance

5   baseline of spacer conductivity

6   endpoint

Figure 17—Typical Conductivity Titration vs Fresh Water Spacer Volume in SSST Apparatus

13.8.7.4   Shall be observed the fluid set aside following the SSST test [13.7.3 step l)] after 30 min to 1 hr. If there are signs of phase separation or sedimentation, redesign the spacer.

14 Pozzolans and Fly Ashes

14.1   General

This section covers the recommended terminology, procedures, and properties for pozzolans and fly ashes used in well cements.

NOTE 1   Pozzolanic materials and fly ashes are constituents of well composite cement classes L, see API Specification 10 A

NOTE 2   Exemplary, pozzolans and fly ashes for use in concrete are also described in ASTM C618, Classes N (pozzolans), and Classes F and C (fly ashes).

14.2 Types of Pozzolan

14.2.1 Pozzolans or pozzolanic materials are described as siliceous (mainly in form of reactive silicon dioxide, SiO_2) or siliceous and aluminous (mainly Al_2O_3) materials, and iron oxide (Fe_2O_3), with small amounts of oxides of other elements which themselves possess little or no cementitious value but which will, in finely divided form and in the presence of moisture, chemically react with calcium hydroxide [$Ca(OH)_2$] at ordinary temperatures to form compounds possessing cementitious properties. These compounds are similar to those that are formed in the hardening of Portland cement.

Although some fly ashes have pozzolanic properties, they are specified separately (see 14.3).

14.2.2   Natural pozzolans are naturally occurring materials of volcanic origin such as volcanic ashes, tuffs, pumices, etc., or sedimentary rocks with suitable chemical and mineralogical composition, and shall conform to 14.2.1.

14.2.3   Natural Calcined Pozzolans are materials of volcanic origin, clays, shales, or sedimentary rocks, which are thermally treated to improve or activate their pozzolanic reactivity and shall conform to 14.2.1.

14.3 Types of Fly Ash

Fly ash is a finely divided residue from the combustion of ground or powdered coal. It is obtained by electrostatic or mechanical separation of fine particles from furnace flue gases.

Fly ash is called artificial pozzolan and is the pozzolan most commonly used in well cement formulations.

According to ASTM C618 class C fly ash is obtained by burning lignite or subbituminous coals. Class C fly ash has pozzolanic properties and also has some cementitious properties. Class C fly ash may have lime contents higher than 10 %.

According to ASTM C618 class F fly ash is produced as the combustion residue from anthracite or bituminous coals. Class F fly ash has pozzolanic properties.

14.4 Physical and Chemical Properties

14.4.1   The physical and chemical characteristics of pozzolans are listed in ASTM C618.

NOTE: For the purposes of the remainder of this section, the term Pozzolan shall refer to both pozzolan and fly ash.

14.4.2   Bulk Density

The average bulk density of pozzolan, is used to select the storage container capacity. The average bulk density is the average of the "loose apparent bulk density" and the "packed apparent bulk density" that may be determined by using the following procedure. The average bulk density can vary between 865 kg/m^3 and 1442 kg/m^3 (54 lbm/ft^3 to 90 lbm/ft^3).

14.4.3 Density

The density of a pozzolan shall be measured using either a Le Chatelier flask, in accordance with ASTM C188, or a gas pycnometer.Certain pozzolans can contain particles with a relative density less than the kerosene or naphtha specified in ASTM C188. Suitable fluids with a density that prevents floating of these particles may be used. Density of a pozzolan shall be calculated knowing density of the fluids used to determine the density in a Le Chatelier flask. Use of a gas pycnometer is preferred for measuring the density of pozzolans containing unusually light particles

14.5 Pozzolan-cement Blend Calculations

14.5.1   Designation of pozzolan-Portland cement blend

When used with Portland cement in well cementing, the amount of pozzolan is based on the absolute volume replacement of a portion of the Portland cement by an equivalent absolute volume of pozzolan. On a sack basis, the resulting mix is referred to as an equivalent sack (see 14.5.3).

The volumes of pozzolan and Portland cement are designated by a ratio of percentages such as 35:65. The first number refers to the volume of pozzolan and the second number refers to the volume of Portland cement in an equivalent sack. A 35:65 blend represents 35 % absolute volume pozzolan mixed with 65 % absolute volume cement.

The designation shall be further clarified as 35:65 Class F fly ash:Class G Cement. Since both fly ash and cement can come from any of a number of sources each having a unique set of properties, the sources of the fly ash and cement shall also be designated.

14.5.2   Blend calculations requirements

14.5.2.1   The densities of the pozzolan and of the Portland cement are required to perform the following calculations. The manufacturer of the pozzolan or cement shall supply the density of the material (or the relative density to be used in calculating the density). The user shall verify the densities reported by the manufacturer (see 5.3.1.2).

14.5.2.2   The weight of a sack of Portland cement (with a volume of 3.59 gal) can vary depending upon the density of the cement. The density of Portland cement can vary between 3100 kg/m^3 (25.87 lbm/gal) and 3250 kg/m^3 (27.12 lbm/gal**).** The correct density value for the selected cement shall be used.

For the purposes of the slurry calculations for pozzolan/Portland cement blends in the following, a sack of cement is defined as 94 lbm of Portland cement with an absolute volume of 3.59 gal. For accurate calculations the mass of a sack of cement (3.59 gal) shall be known.

The density of pozzolan can vary between 15.02 lbm/gal (1800 kg/m^3) and 24.20 lbm/gal (2900 kg/m^3), and the correct value shall be known for the pozzolan material that will be used. (This equates to a pozzolan sack weight of 54 lbm to 87 lbm per 3.59 gal.)

14.5.3 Pozzolan-Portland cement blend —Equivalent sack weight

Pozzolan/Portland cement blend calculations are used to determine the weights of pozzolan and Portland cement to be blended into an equivalent sack (see 3.1.19). The following examples illustrates calculations for blend systems containing pozzolan.

REMARK: Equivalent sack is only used by calculations using U.S. Customary (USC) units.

Given an absolute volume of a sack of cement as 3.59 gal, an equivalent sack of a pozzolan:cement blend will have the same volume but will not weigh 94 lbm. To calculate the weight of the equivalent sack of blend, $m_{\text{bld,sk-eq}}$, the weight is based the weights of pozzolan, and cement blended to prepare one sack of blend and the relation:

$$m_{\text{bld,sk-eq}} = \text{Pozzolan mass} + \text{Cement mass}$$

The absolute volume of an equivalent sack remains 3.59 gal. The weight of the equivalent sack of a pozzolan: cement blend ($m_{\text{bld,sk-eq}}$) shall be calculated as given in Equation (65)

$$m_{\text{bld,sk-equi}} = \frac{\left(m_{\text{poz,sk}} \times \varPhi_{\text{poz}}\right) + \left(m_{\text{cmt,sk}} \times \varPhi_{\text{cmt}}\right)}{100} \tag{65}$$

where

$m_{\text{poz,sk}}$ is the mass weight of one sack (3.59 gal) of pozzolan, expressed in pounds-mass;

$\varPhi_{\text{poz}}$ is the ratio of pozzolan in the pozzolan:cement blend , expressed in percent;

$m_{\text{cmt,sk}}$ is the mass weight of one sack (3.59 gal) of cement, expressed in pounds-mass;

$\varPhi_{\text{cmt}}$ is the ratio of cement in the pozzolan:cement blend , expressed in percent.

Once the mass of the equivalent sack is known, most other additives are based on the mass of this equivalent sack. For blends such as pozzolan **:** Portland cement blends, the convention for determining the amount of additives is percent BWOB, or for liquid additives on gallons per equivalent sacks.

Slurry calculations shall follow 5.3.2 and 5.3.2.3 with USC units where:

— mass of cement, $m_{\text{c,}}$, is replaced by the mass of one equivalent sack of blend, $m_{\text{bld,sk-eq}}$;

— the convention for determining the amount of solid additives is percent BWOB;

— the convention for determining the amont of liquid additives is in gallons per equivalent sack of blend.

EXAMPLE 1

For a 35**:**65 Class F Fly ash:Class G cement blend, the volume of pozzolan is 1.26 gal (35 % of 3.59 gal) and the Portland cement is 2.33 gal (65 % of 3.59 gal).

Calculate the amount of cement in an equivalent sack of blend

Given the weight ($m_{\text{cmt,sk}}$) of a sack (3.59 gal) of cement is 94 lbm, the required weight of cement can be calculated by:

$$\text{Weight of cement} = 94 \times 0.65 = 61.1 \text{ lbm}$$

The density of pozzolan can vary between 15.02 lbm/gal (1800 kg/m^3) and 24.20 lbm/gal (2900 kg/m^3), and the correct value must be known for the pozzolan material that will be used. (This equates to a pozzolan sack weight of 54 to 87 lbm per 3.59 gal.). Using a weight of 74 lbm per sack for the pozzolan ($m_{poz,sk}$), the weight of pozzolan used in an equivalent sack of blend is:

$$\text{Weight of pozzolan} = 74 \times 0.35 = 25.9 \text{ lbm}$$

The weight of an equivalent sack of this blend ($m_{bld,sk-eq}$) is:

$$\text{Weight of Equivalent Sack} = 61.1 + 35.9 = 87 \text{ lbm}$$

EXAMPLE 2

Expanding Example 1, using USC units, if the absolute volume of a sack of cement is 3.62 gal and a 35:65 blend is desired, then the pozzolan is 1.27 gal (35 % of 3.62 gal) and the Portland cement is 2.35 gal (65 % of 3.62 gal). The absolute volume of pozzolan (1.27 gal) and the absolute volume of Portland cement (2.35 gal) are then used to calculate the pounds-mass of each material from the density values of pozzolan and Portland cement.

Expanding further on the example, 2.35 gal $\times$ 26.00 lbm/gal = 61.1 lbm of Portland cement. Pozzolan density can vary between 15.02 lbm/gal (1800 kg/m^3) and 24.20 lbm/gal (2900 kg/m^3), and the correct value must be known for the pozzolan material that will be used. Assume that the density of the pozzolan to be used is 20.50 lbm/ gal. Then the mass of pozzolan used in the blend is 26.0 lbm (1.27 gal $\times$ 20.50 lbm/gal).

Combining the pounds-mass of pozzolan and the pounds-mass of cement gives 87.1 lbm (61.1 lbm + 26.0 lbm) of blend. For this example, 87.1 lbm of blend is referred to as an equivalent sack (see 3.1.20) of 35:65 pozzolan:Portland cement.

EXAMPLE 3

For this example, assume the Example 2 blend has 6 % bentonite and 0.2 % retarder.

The mass of bentonite per equivalent sack of blend is (6 %) $\times$ (87.1 lbm) = 5.23 lbm of bentonite per equivalent sack. The mass of retarder per equivalent sack of blend is (0.2 %) $\times$ (87.1 lbm) = 0.17 lbm per equivalent sack.

The mass of the equivalent sack and the masses of additives in the blend are now known. Slurry density and slurry yield can be calculated if the volume of mix water is known. Conversely, if the desired slurry density is known, then the slurry yield and mix water requirements can be calculated. Once the value for slurry yield is determined, the number of equivalent sacks of blend for a given job can be calculated from the annular volume from pipe and hole configuration, caliper logs, etc.

EXAMPLE 4

To calculate the material requirements, assume the job requires 125 equivalent sacks.
Thus, (125 sk) $\times$ (61.1 lbm/sk) = 7638 lbm of Portland cement would be required.
For the same 125-sack job, (125 sk) $\times$ (2.0 lbm/sk) = 3250 lbm of pozzolan would be required.
The masses of bentonite and retarder required are, respectively, (125 sk) $\times$ (5.23 lbm/sk) = 654.75 lbm and (125 sk) $\times$ (0.17 lbm/sk) = 21.25 lbm.

15 Test Procedure for Arctic Cementing Slurries

15.1 General

This procedure is intended for the testing of cement slurries that are to be placed through formations that are permanently frozen (permafrost). The conditioning temperature for the test equipment, materials to be tested, and the test temperatures shall be controlled to ± 2 °C (± 3 °F). Procedures may be modified, where appropriate, to meet local requirements. Reports of test data must clearly indicate what modifications have been made to the procedure, when modified.

Apparatus shall be verified and or calibrated if not in calibration as per Annex B requirements.

15.2 Preparation of Cement Slurry

Test samples shall be prepared according to Section 5, except that the temperature of the mix water, dry cement or cement blend, and mixing container shall be representative of field mixing conditions. If field conditions are unknown, the cement blend and mixing container shall be cooled to – 7 °C (20 °F) before mixing. Mix water shall be cooled to 1 °C ±1 °C (34 °F ±2 °F), and the slurry temperature immediately after mixing shall be recorded, 4 °C (40 °F) is common in some areas. Each of the above temperatures shall be measured and reported on all tests.

15.3 Fluid Fraction

The fluid fraction shall be expressed as percent by mass of basic dry blend (not including any additives needed for placement such as retarders, fluid loss additives, free fluid control additives, etc.).

15.4 Thickening-time

A thickening-time test shall be performed in a pressurized consistometer at atmospheric pressure. The consistometer must be built or modified to be capable of the test conditions. Particular attention must be paid to an adequate refrigeration system to meet the test requirements and insulation to prevent heat loss and the potential for electrical shorts on the consistency measuring system and of the heater connections due to condensation of moisture. The procedure described in Section 9, Well-simulation thickening-time tests, regarding test preparation and operation of the consistometer shall be followed with appropriate modifications for cold testing. Test conditions shall be consistent with the anticipated conditions in the well or if wellbore conditions are unknown, performed at 4 °C ±1 °C (40 °F ±2 °F). It is acceptable to chill the slurry container to the starting temperature prior to filling the slurry container with slurry

If an atmospheric consistometer is used for the thickening-time test, note that the consistency indication is not equivalent to the consistency measured on a pressurized consistometer.

15.5 Compressive Strength

Specimens shall be cured at – 7 °C ±1 °C (20 °F ±2 °F) and 4 °C ±1 °C (40 °F ±2 °F) or another temperature consistent with the anticipated well conditions for the desired testing period, such as 1 d, 3 d, or 7 d. As is done for thickening-time test schedules, thermal simulators are suggested for development of schedules for curing compressive strength specimens.

The molds shall be cooled to the test temperature, that is – 7 °C (20 °F) or 4 °C (40 °F) or another temperature, as appropriate. Otherwise, the molds shall be prepared as described in Section 7.

Condition the slurry for 90 min in a consistometer at 4 °C ±1 °C (40 °F ±2 °°F) and atmospheric pressure before pouring into the cooled molds for curing. Fill and prepare the molds for curing as described in Section 7.

For curing at temperatures below 0 °C (32 °F), seal the test specimens in a container of fresh water cooled to the test temperature or 2 °C ±1°C((35 °F ±2 °F), whichever is the higher Submerge the sealed container in a mineral oil or glycol bath or in a temperature-controlled freezer at test temperature in a manner consistent with avoiding contamination of the fresh water and specimens. Cure the specimens in the sealed containers at atmospheric pressure as described in Section 7.

At 30 min±5 min prior to the time at which the strength is to be tested, remove the specimens from the molds and submerge them in a water bath maintained at 4 °C ±1 °C (40 °F ±2 °F) until each specimen is tested. At the desired test time (measured from the end of conditioning), remove each sample from the water bath and test for strength according to the procedure described in Section 7. The test for strength shall be performed within 30 min of the time at which the desired age is reached. After testing, the residue,

or an additional test specimen, shall be allowed to warm to ambient temperature and checked to be sure the specimen was set and not just frozen.

Alternatively, specimens may be tested according to the procedure for nondestructive sonic strength testing of cement (Section 8) with appropriate modifications of the apparatus for the temperature of the test. It is acceptable to chill the slurry container to the starting temperature prior to filling the slurry container with slurry

15.6 Strength After Freeze-Thaw Cycling at Atmospheric Pressure

15.6.1 Freeze-thaw cycle testing is normally done to qualify slurry formulations under development and not for routine testing of "standard" cement formulations. If desired, after a basic slurry composition has been qualified according to this procedure, additional testing may be performed using different temperatures or adjusted curing schedules, as appropriate. If such additional testing is performed, reports of test data must clearly indicate what modifications have been made to the procedure.

15.6.2 Following procedure shall be applied to determine strength after freeze thaw cycling at atmospheric pressure:

Prepare the slurry as in 15.2 and prepare the cube molds as specified in 15.5 (condition the slurry as in 15.5).

During the "freeze-thaw" cycling, cure the specimens at atmospheric pressure according to the following 14 days sequence (beginning the cycle on a Monday is convenient for most):

a)   48 hr ±1 hr at 4 °C ±1°C (40 °F ±2°F), days 1–2;

b)   24 hr ±1 hr at – 7 °C ±1°C (20 °F ±2°F), day 3;

c)   24 hr ±1 hr at 4 °C ±1°C (40 °F ±2°F), day 4;

d)   72 hr ±1 hr at 38 °C ±1°C (100 °F ±2°F), days 5–7;

e)   72 hr ±1 hr at 77 °C ±1°C (170 °F ±2°F), days 8–10;

f)   24 hr ±1 hr at 38 °C ±1°C (100 °F ±2°F), day 11;

g)   72 hr ±1 hr at – 7 °C ±1°C (20 °F ±2°F), days 12–14; and

h)   raise to 4 °C ±1°C (40 °F ±2°F) and repeat cycle.

Cure all specimens in molds under water and with the top of the cement exposed to the water.

Test control specimens for strength after 48 hr ±1 hr at 4 °C ±1°C (40 °F ±2°F). Examine the cement cubes and test them for strength after one cycle (14 d) and 3 cycles (42 d).

Annex A
(normative)

Procedure for Preparation of Large Slurry Volumes

A.1 General

This procedure shall be used only when an individual test or a series of tests requires a slurry volume greater than 600 mL. It is not intended to be used in place of Section 5. Note that the mixing energy of this procedure is not the same as that of the procedure in Section 5 and mixing energy can influence results.

A.2 Apparatus

A.2.1 General

All apparatuses shall be the same as outlined in Section 5, except that the mixing device shall be as described in this subsection. All apparatuses shall meet Annex B requirements.

A.2.2 Mixing Device

The mixing device shall have a capacity 4 L to 5 L. The mixing device for preparation of large slurry volumes shall be a bottom-drive, blade-type blender. The mixing container and the mixing blade shall be constructed of corrosion-resistant material. The mixing assembly shall be constructed so that the blade can be separated from the drive mechanism. The mixing blade shall be separated from the mixing assembly and replaced with a new blade before mass loss greater than 10 % has occurred (see B.3.3). Should the mixing device leak at any time during the mixing procedure, the contents shall be discarded, the leak repaired, and the procedure restarted.

An example of a mixing device in common use is shown in Figure A.1.

Figure A.1—Example of a Common Cement-mixing Device for Large Volumes

A.3 Procedure

A.3.1 General

The procedure shall be the same as outlined in Section 5, except as described below.

A.3.2 Laboratory Density and Volume Calculations

The recommended volume for the procedure in Annex A is 3000 mL. Laboratory blend requirements can be calculated by use of the equations found in 5.3.2.5

A.3.3 Mixing Cement and Water

Mixing large volume of cement and water shall be as per the following procedure.

a)   Place the mixing container with the required mass of mix water and any liquid additives (if previously added) on the blender base.

b)   Place the cover of the mixing container. Turn on the motor and maintain at "slow" speed, 4000 r/min, $\pm$250 r/min.

   If additives are present in the mix water, stir at the above rotational speed to thoroughly disperse them in the mix water prior to the addition of the cement. In some cases, additives (dry or liquid) may be added to the mix water in the field. In such cases, the additives shall be added to the mix water while mixing at low speed at which a slight vortex is observed.

   In certain cases, the order of addition of the additives to the mixing water can be critical, in which case the additives shall be mixed in the order that they will be mixed in the field. Document any special mixing procedures and mixing times.While mixing at 4000 r/min, $\pm$250 r/min, add the cement or cement/dry additive blend to the mixing container at a uniform rate in not more than 35 s, if possible.

c)   Some slurry designs can require a longer time to completely wet the cement blend. However, the time used to add the blend shall be kept to a minimum. If a time longer than 35 s was required to add the cement blend to the water, document that time.After 35 s or when all the dry materials have been added to the mix water (if longer than 35 s), continue mixing at 10,000 r/min, $\pm$500 r/min, for 50 s, $\pm$1 s.

   If possible, measure and document the rotational speed under load.

   NOTE   Data generated from the use of a large volume (3000 mL) mix may vary from the 600 mL mixing data and is only recommended when large volumes are required.

Annex B
(normative)

Calibration and Verification of Well Cement Testing Equipment

B.1 Overview

This annex defines the requirements for calibration and verification of measurements by instruments used in testing cement for oil and gas well applications according to the procedures prescribed in this document. Henceforth, the term calibration is used to mean either verification and/or calibration according to the requirements of this document).

Procedures will depend on the specific instrument used for the calibration. Manufacturer's instructions should be followed for the calibration. Calibrations shall be conducted using instruments and devices traceable to national standards.

NOTE   Calibration refers to the verification of an instrument or to the adjustments made to the instrument if it is out of calibration. If a verification shows the instrument to be in calibration, the date and verification data will be the calibration. In this calibration section, the word "verification" pertains to instruments that cannot be changed if they are inaccurate, and the word "calibrate" pertains to instruments that if they are out of calibration as seen by the "verification" of the instrument may be adjusted to get them into calibration.

B.2 General

The following sections set the requirements for interval and accuracy of calibration of devices used in a laboratory conducting tests according to this Standard. The requirements by equipment type are given in Table B.1 and the frequency and allowable tolerances are given in Table B.2.

Volumetric glassware (graduated cylinders, conical flask, and similar equipment) calibration is generally performed by the glassware supplier and may be part of the purchase specification. Glassware users shall obtain documented evidence, where deemed important, of glassware calibration from the supplier. Calibration may be checked gravimetrically before the first use. Periodic recalibration is not required. As specified in B.3.17, a procedure for graduated cylinders applicable to any volumetric glassware is detailed.

Dimensions of apparatuses such as cube molds and consistometers parts may be verified using a caliper accurate to at least 0.1 mm (0.005 in.).

Table B.1—Equipment Calibration Requirements

Measurement Instrument	Equipment															
	Balance	Weight Sets	Handheld Timers	Mixing Device[a] (Blender)	Handheld Thermometers	Viscometers	Pressurized consistometer	Atmospheric Consistometer	Water & Curing Baths	Load Frame	Cube-molds	Ultrasonic Device	Sedimentation Tube	Fluid-loss Cell	Pressure Density Balance	Data Acquisition Chart recorder
Mass	V/C	V		V												
Timer			V	V			V	V						V		V/C
Rotation				V		V	V/C	V/C								
Temperature Measurement					C	V/C	V/C	V/C	V/C			V/C		V/C		C
Dimensions						V					V		V			
Torsion Device						C										
Consistency Measurement							V/C									C
Pressure Gage							V/C		V/C			V/C		V/C		C
Ultrasonic Transducer												V/C				
Screen,Fluid-loss														V		
Auxiliary Display							V	V								
Indication										V/C					V/C	V

NOTE 1   See Table B.2 for frequency and tolerances

NOTE 2   V   Verify accuracy or tolerance; if outside requirements adjust or replace.

        C   Calibrate by making adjustments or creating calibration chart.

        V/C   Verification of calibration may be performed first. If the verification values are acceptable at the time it is verified, the verification will serve as a calibration. If it is not in calibration tolerance, the calibration needs to be performed

[a]   Blender blade mass

Table B.9—Calibration and Verification of Well Cement Testing Equipment

Equipment and Component/Function	Calibration/ Verification	Check Points	Tolerance	Frequency
Balance (each range on multi-range balance)	Calibration	Three points spread evenly across range	±0.1 % of reading for measurements made at 10 g or greater up to the full scale of the balance ±0.01 g for measurement made at or less than 10 g	Annually
Weight sets	Verification	Each piece	±0.1 % of the nominal mass value for weight mass equal or greater than 10 g ±0.01 g of the nominal mass for 10 g or less	Annually (routine usage)
Timers	Verification	12 min	within 5 s in one hour (in 12 min minimum verification period within 1 s)	Annually
Mixing device (1 L blender)	Verification	4000 r/min and 12,000 r/min	±250 r/min @ 4000 r/min and ±250 r/min @ 12,000 r/min	Annually
Large volume mixing device (4 L blender)	Verification	4000 r/min and 10,000 r/min	±250 r/min @ 4000 r/min and ±250 r/min @ 10,000 r/min	Annually
Mixing device—blade mass	Verification	Initially when replaced	Maximum 10 % loss	Minimum 30 days after initial weighing or when abnormal critical wear/deformation is observed
Mixing device—timing controller	Verification	One liter device :35 s Large volume device: 50 s	One liter device: within 1 s in 35 s Large volume device: within 1 s in 50 s	Annually
Temperature controlling systems and displays	Calibration	Three points spanning the range	Maximum of ±1 °C (2 °F)	Quarterly
Thermocouple	Verification	Three points spanning the range	Maximum of ±1 °C (2 °F)	Quarterly
Special J thermocouple	Verification	Three points spanning the range	Maximum of ±1.1 °C (±2 °F) or 0.4 % of the reading whichever is the greater	Quaterly

Equipment and Component/Function	Calibration/ Verification	Check Points	Tolerance	Frequency
Handheld thermometer or portable thermocouple	Calibration	Three points spanning the range	Maximum of ±1 °C (±2 °F)	Quarterly
Viscometer torsion measuring device	Calibration	See Table B.4	Table B.4; Follow manufacturer's instructions (weights or calibration fluid); Reads zero when empty at any speed	Quarterly, and whenever torque measuring component is changed or adjusted
Viscometer rotor speed	Verification	At each speed	Maximum of ±1 % of nominal speed at speeds of 100 r/min and greater ±1 r/min for speeds <100 r/min	Quarterly
Consistometer container speed (or paddle speed)	Verification	At 150 r/min (and 25 r/min with variable speed controls)	±15 r/min at 150 r/min (±5 r/min at 25 r/min)	Quarterly
Slurry container, paddle and shaft [a]	Verification	See API Specification 10A for dimensions	Maximum wear ±1.0 mm (±0.04 in.)	Annually and whenever parts are replaced
Consistency measuring devices	Calibration	Table B.5	±5 B_c or ±0.5 VDC, as appropriate	Monthly and whenever it is adjusted or parts replaced
Potentiometer calibration weight set	Verification	Each piece including holding support washer	±0.1% of mass value	Annually
Recorder - Chart speed	Verification /Calibration	One hour	±2 min in 1 hr	Annually
Pressure measuring device— High range (>17,000 kPa or 2500 lbf/in.²)	Calibration	25 %, 50 %, 75 % of full scale or max user defined working pressure	Using deadweight tester or master gauge; ±1 % of full range or minimum gauge increment, whichever is greater	Annually or when pressure transducer is replaced
Pressure measuring device— Low range (up to 17,000 kPa or 2500 lbf/in.²)	Calibration	3500 kPa and 10,500 kPa (500 lbf/in.² and 1500 lbf/in.²) or equipment maximum allowable working pressure	±300 kPa (±50 lbf/in.²)	Annually or when pressure transducer is replaced
Air pressure gauges	NR[a]			

Equipment and Component/Function	Calibration/ Verification	Check Points	Tolerance	Frequency
Sedimentation Tube	Verification	Tube internal diameter 25.4 mm (1.00 in.)	±0.5 mm (±0.02 in.) using a go / no-go gage	Once every two years
Load frame	Calibration	At 25 %, 50 %, 75 % and 100% of the maximum of the cell range	±2 % of the applied load or one minimum instrument scale division, whichever is greater	Annually
Cube-molds (in use)	Verification	Cube dimensions distance between opposite sides and height of compartment	50-mm molds:distance between opposite sides: ±0.5 mm, height of compartment: +0.25 mm/-0.38 mm. 2-in. molds: distance between opposite side ±0.02 in., height of compartment +0.01 in./-0.015 in..	Once every two years
Sonic measuring system	Calibration	According to manufacturer's instructions	According to manufacturer's specifications	Monthly, and when cells, transducers, or cables are changed
Pressurized density balance— Indicated density	Calibration	Deionized water (1 SG) and in the range 1.80 kg/L to 2.30 kg/L (15 lbm/gal to 19 lbm/gal)	±0.01 kg/L (±0.1 lbm/gal) from actual	Monthly: with water Annually: in the range 1.80 kg/L to 2.30 kg/L (15 lbm/gal to 19 lbm/gal)
Fluid-loss screen [a]	Verification	Each piece	±10% of the intial flow time	Initially and after every 5 tests or after a citric acid wash.
Data acquisition devices	Verification	Same as temperature, pressure sensors, or other measurement devices	Adjust or prepare calibration table	With sensor recorded
Graduated cylinder [b]	Verification /Calibration	5 points	If required prepare calibration table	Before first use
Volumetric glassware [b]	NR[c]			Before first use

[a] Informative procedure

[b] Volumetric glassware including graduated cylinders calibration is done by supplier. and may be part of the purchase specification. Calibration may be checked gravimetrically before the first use. Periodic recalibration is not required.

[c] Not required

The individual performing the verification/calibration shall possess adequate skill and training to properly perform the calibration according to the procedures specified for the device(s) being used.

Records of such calibration shall be maintained in a manner that the data or record cannot be altered after certification. The records shall be maintained at least as long as the data from tests performed during the calibration period on the specific instrument are maintained.

Verifcation and calibration records shall contain, as appropriate:

a)   model name/number, serial number, and local identification number of the instrument;

b)   name, model number, and serial number of instrument used for the calibration;

c)   certification number, date of certification, and date next calibration against national standard is due for the calibration instrument;

d)   calibration inputs from the calibration instrument and the initial (before calibration) and final (calibrated) indications from instrument being calibrated;

e)   indication that instrument passed or failed each calibration point;

f)   condition found/condition left (e.g. out of or in calibration);

g)   adjustments made and results after adjustment;

h)   date of calibration;

i)   name and signature of calibration technician;

j)   if calibration is by technician from an outside service, name of service, location, and phone number.

Some instruments have multiple cells or testing systems within one unit (e.g. dual-cell consistometers). In such cases, each cell or testing system shall be verified and/or calibrated (if not in calibration) individually with a separate certificate showing a unique identifier for each testing system. Components that are common to all of the cells or testing systems need be calibrated only once, but the calibration data shall be indicated on each certificate.

When the device is being used for a test and a verification or a calibration becomes due, the verification or calibration remains valid for the duration of that test and the instrument shall be calibrated at the end of that test.

B.3 Requirements

B.3.1 Balances

Balances shall be verified and/or calibrated (if not in calibration) no less frequently than annually. Balances shall be accurate to ±0.1 % of reading for measurements made at 10 g or greater up to the full scale of the balance. Balances shall be accurate to ±0.01 g of reading for measurement made less than 10 g. Balances shall have two (2) decimal-place precision at a minimum. Each range of a dual range balance shall be calibrated in this fashion. Reference weights (see B.3.2) shall be used for balance verification/calibration

B.3.2   Weight Sets

Weight sets shall be verified against reference weights traceable to a national standard. Weight sets to be verified include, but are not limited to, those used daily (routine weighing) and to calibrate viscometersprings, balances, and consistometer potentiometers (dedicated potentiometer calibration weight set) or their equivalent. Weight sets shall be verified and/or calibrated no less frequently than once annually.

Weights shall conform within ±0.1 % of the nominal mass excepted for weights with a mass equal or less than 10 g (Table B.3). For weights of 10 g and less shall conform within ±0.01 g of the nominal mass. If weights do not conform to the specification, they shall be discarded or sent to the manufacturer for recertification.

NOTE   Annual verification shall be required for weight sets use daily for routine weighing. For reference weight sets use for annual verification, weights should be recertified against certified master weights every five years providing weights sets are kept in a padded dust- and moisture-proof holder and used with tongs with plastic tips, If not the case, reference weight sets shall be recertified every two years.

Table B.3—Permissible Variation in Mass of Weights (routine usage)

Mass g	Permissible variation g
1000	±1.00
500	±0.50
300	±0.30
200	±0.20
100	±0.10
50	±0.05
≤10	±0.01

B.3.3   Timers

Timers on all instruments shall be verified/and or calibrated (if not in calibration) no less frequently than annually. This includes timers on consistometers, handheld timers and any other timer, whether as part of an instrument or separate. Timers shall be accurate within ±5 seconds per hour and shall be verified over a period of no less than 12 minutes. Calibration may use the time signal from the NIST or similar web sites and radio stations. If not within required accuracy, the units shall be adjusted or replaced.

NOTE: Mixing device timing controllers (see B.3.4.3) are not covered by this section.

B.3.4   Mixing Device (Blender)

B.3.4.1   Blender Speed

Mixing device display of rotational speed shall be verified and/or calibrated (if not in calibration) no less frequently than annually. The tolerance measured by a calibrated tachometer shall not deviate more than

the limits given in Table B.2: ±250 r/min at either 4000 r/min low speed and 12,000 r/min high speed for the 1 L (1qt) blender (10,000 r/min high speed for the large volume blender).

However mixing cement slurry, one liter blender [1 L (1 qt)] shall operate at a low speed range of 3750 r/min to 4250 r/min (4000 r/min ±250 r/min) and high speed range of 11,500 r/min to 12,500r/min (12,000 r/min ±500 r/min) and large volume (4 L) blender shall operate at a low speed range of 3750 r/min to 4250 r/min (4000 r/min ±250 r/min) and at high speed range of 9500 r/min to 10,500 r/min (10,000 r/min ±500 r/min).

The indicated speed shall not deviate by more than these limits at either the low speed or the high speed from the true speed observed using a calibrated tachometer.

NOTE   The ability of the controller to maintain speeds within the required range during mixing a cement slurry is not a calibration issue. If the mixing device is unable to control within the required range, the blender motor or controller shall be repaired or replaced, as appropriate. Typical motors available for the 1 L (1 quart) mixer are 3.6 A/0.04HP and the 7 A/0.7 HP; typically, the 3.6 A motor will not maintain the tolerance especially with viscous slurry.

B.3.4.2   Blender Blade

Initial blade weight and dimensions may vary due to the manufacturing process. The weight loss of the blender blade (blade only, not the blade assembly) shall be verified to be no more than 10 % when it is changed. The mixing blade shall be weighed initially (out of the package) and when critical blade deformation or wearing is observed and at least every 30 days after the initial weighing. Replace with an unused blade if observed blade deformation has occurred or after a bled has lost 10 % of its mass.

Users will develop procedures to ensure that the blade is not used with >10 % weight loss compared to its unused (out of the package) weight and will develop a system to confirm weight loss of the blade when it is changed. This method will include a method of identifying the blender container in which the blade is installed. A log of blade weights and dates changed will be maintained with the calibration records. A practical method of judging when a blade is approaching the limit in weight loss is by comparing the blade in the blender to a blade with known weight loss (such as is shown in the picture in Figure B.1). The appearance of wear for a hardened blade may be different than shown in Figure B.1 for a non-hardened blade.
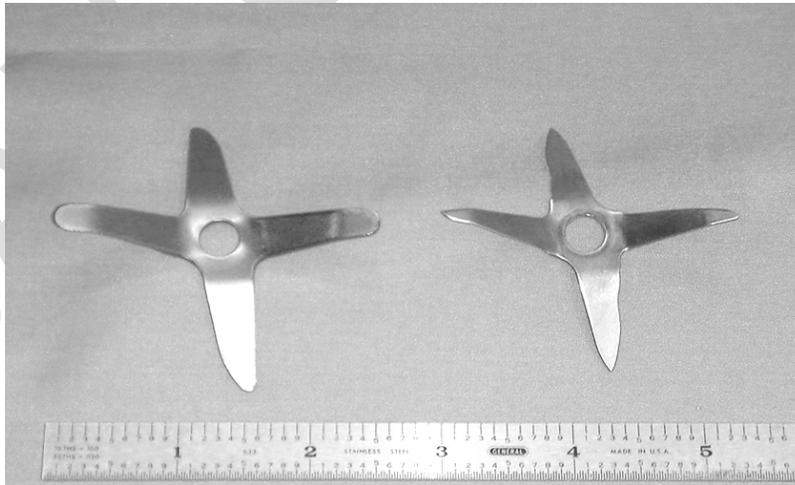
Figure B.1—Worn Blade (right) Compared to a New One (left)

B.3.4.3   Mixing device timing controller

One liter mixing device timing controller device shall be verified against a calibrated timer (stopwatch) (see B.3.3) for high-speed mixing (12,000 rpm) time at 35 s ±1 s no less frequently than annually. Large volume mixing device timing controller device shall be verified for high-speed mixing (10,000 rpm) time at 50 s ±1 s no less frequently than annually.

B.3.5   Temperature Devices

B.3.5.1   Temperature measuring and controlling devices shall be verified and/or calibrated (if not in calibration) no less frequently than quarterly. This includes thermometers and portable thermocouples, thermocouples, and temperature controllers on consistometers, curing chambers, ultrasonic devices, and those that are used separate from or are not an integral part of the instrument. Measurements shall be made at no less than three temperatures spanning the manufacturer or user defined operating range of the equipment on or with which the device is used. The lowest temperature verified/calibrated shall be no more than 5 °C (10 °F) above the minimum and the highest temperature calibrated shall be no more than 5 °C (10 °F) below the maximum of the user-defined operating range of the instrument.

B.3.5.2   Thermometers or thermocouples shall be verified for accuracy by use of a known temperature source (such as a heat block) with a thermometer or thermocouple that is certified and traceable to a national standardization body. Accuracy shall be within ±1 °C (±2 °F) or for special J type thermocouple accuracy shall be ±0.04 % of the temperature indication whichever is the greater. If the thermometer or thermocouple error is greater than that, it shall be replaced by one meeting the accuracy requirements.

Thermocouples mounted in the cylinder wall of a consistometer are exempt from calibration, but during the test, adjustments shall be made to the controlling temperature when control is from the oil thermocouple, so the final temperature of the slurry is within the required test temperature.

B.3.5.3   Temperature controlling systems consist of:

— the sensing device (normally a thermocouple);

— display (may be integral to controller or may be separate);

— the controller that receives input from the sensing device and in turn provides output to the heating or cooling medium. The output circuit is not calibrated in this procedure; and

— all wiring, connectors, etc. between the sensor and the controller.

The temperature controller may be verified and/or calibrated while connected to its thermocouple placed in a calibrated heat source or using a cold junction compensated input device in place of the thermocouple. If the controller is calibrated using a cold junction compensated input device, the thermocouple shall be calibrated independently. The thermometer or thermocouple that is used for the verification of calibration tolerance shall be certified and traceable to a national standardization body. The controller accuracy shall be within ±1 °C (±2 °F). If the system is not within the required accuracy, the source of the inaccuracy shall be determined and the component(s) that is (are) not within the required accuracy shall be adjusted or replaced.

The temperature controller shall be verified for accuracy at an interval no less than quarterly and be checked at no less than three temperatures spanning the operating range of the equipment on or with which the device is used. The lowest temperature calibrated shall be no more than 5 °C (10 °F) above the minimum and the highest temperature calibrated shall be no more than 5 °C (10 °F) below the maximum of the user-defined operating range of the instrument.

Temperature displays that are separate from the controller shall be calibrated by the same methods used to calibrate the controller. Separate displays shall have their own calibration record.

B.3.5.4   Temperature data acquisition: see B.3.17.

B.3.5.5   Guidance for temperature device calibration

This subsection provides additional guidance for temperature device calibration.

B.3.5.5.1   Heating medium

The heating medium shall permit proper immersion of both the test thermocouple (the one being verified/calibrated) and the reference thermocouple or reference thermometer. The medium may be a fluidized solids bath, a heated block, or a furnace. The equipment shall be capable of maintaining a stable temperature that is uniform throughout the test section. If the heat source (heater and instrument) is portable, it should be allowed to stabilize at the ambient temperature prior to being used. This is especially true when the instrument is removed from a car or storage room, etc.

B.3.5.5.2   Procedure

Items listed here are those needing special attention or related to the use of the indicated type of equipment.

⎯ The test and reference thermocouple or thermometer shall be placed as close together in the heating medium as possible.

⎯ After each change of the temperature setting, the temperature of the heating medium shall be allowed to stabilize before reading the reference temperature (or voltage) and the test thermocouple temperature (or voltage). If several thermocouples are checked in sequence, each one shall be allowed to reach a stable reading before making the calibration measurement.

⎯ If the test thermocouple does not accurately sense the temperature, a calibration curve shall be drawn and used to correct the indicated temperatures from the test thermocouple. Occasionally, small inaccuracies in thermocouple response can be compensated for during the calibration of the temperature-measuring system being used in conjunction with the thermocouple (B.3.5.3).

B.3.5.5.3   Calibration of temperature-measuring systems and controllers

B.3.5.5.3.1   Equipment

The verification/calibration of temperature-measuring systems and controllers requires a millivolt source, the correct connecting thermocouple extension cable for the type of thermocouple being used, and possibly a thermometer and a table of reference voltages. Signal sources, or calibrators, are of two types, namely, uncompensated, and cold junction compensated. Several commercial calibrators are available that are cold junction compensated and have a digital display of the temperature equivalent of the millivolt signal being supplied. The accuracy of all calibration equipment shall be traceable to national standards certification. Some older galvanometer type temperature-indicating instruments and controllers require a stronger signal for operation than the newer potentiometric and digital type temperature-measuring systems and controllers, and thus require a calibrator with sufficient signal strength to give an accurate calibration.

An alternate method consists of placing the thermocouple that is connected to the controlling or indicating instrument into a heat source and comparing it with a calibrated thermocouple or thermometer. The temperature of the heat source is then changed as specified in B.3.5.5.2 to certify the controlling or indicating instrument is accurately calibrated.

B.3.5.5.3.2   Procedure

Manufacturer's procedure for calibrating temperature-measuring systems and controllersshall be followed. For greatest accuracy, allow proper warm-up time for calibrators, temperature-measuring systems, and controllers as specified by the manufacturer. The following items need special attention:

— Fit the thermocouple extension cable with a proper thermocouple-grade adapter to permit plugging it into the same receptacle used for connecting the test equipment thermocouple. Take care to ensure the correct polarity of the connections.

— Thermocouple calibrators with cold-junction compensation need only be properly connected with the proper thermocouple extension cable and thermocouple connectors. The temperature-measuring systems and/or controllers using this signal shall display the same temperature within the accuracy of the thermometer or controllers as supplied by the manufacturer.

— Uncompensated thermocouple calibrators require a thermometer to determine the cold-junction temperature of the thermocouple extension cable connection of the calibrator. This cold-junction temperature shall be set on the calibrator by the operator.

— The use of an uncompensated millivolt potentiometer requires that the temperature at the calibrator/thermocouple extension cable terminals be read with a thermometer of known accuracy. The millivolt equivalent of this temperature is then subtracted from the equivalent test millivolt signal to obtain the calibrator millivolt signal used. These voltages may be found in reference millivolt/temperature tables for the type of thermocouple in use.

— The temperature of the calibration instrument should be allowed to stabilize at the ambient temperature at the location where it is being used. This is especially true when the instrument is removed from a car or a storage room, etc.

B.3.5.5.4   Specific procedure for atmospheric consistometer

When verifying the temperature calibration of an atmospheric consitometer, place one slurry container in the unit and leave the second one out. Place the reference thermocouple (attached to a master temperature calibration instrument) in the open hole as close as possible to the atmospheric consistometer thermocouple and heat the consistometer to no less than three temperatures from ambient temperature to 88 °C (190°C) to verify the accuracy of the temperature indication. Calibrate the controller if it is not in calibration. If it is in calibration, use the verification as the calibration and date it for reference.

B.3.5.5.5   Specific procedure for atmospheric pressure water bath

When verifying the temperature calibration of an atmospheric pressure water bath place a stirring device in the bath (if it does not have one) along with a reference thermocouple attached to a master temperature calibration instrument in the water bath. Heat the water bath to no less than Three temperatures from ambient temperature to 88 °C (190 °C) to verify the accuracy of the temperature.

B.3.6   Viscometer Torsion Measuring Systems

Viscometer torsion measuring systems shall be verified and/or calibrated (if not in calibration) using either a dead weight method or a Newtonian fluid having a certified viscosity vs temperature profile. The dead weight method is preferred. Calibration fluids are subject to contamination and are sensitive to temperature; however, an advantage of the use of calibration fluids is that they check the entire measuring system. When calibration fluid is used, viscosity and temperature shall be measured simultaneously and recorded. Verification and/or calibration shall be no less frequently than quarterly or whenever a spring or bearing is installed, changed, or adjusted in the instrument. Using dedicated calibration spool and dead weight method, dial deflection tolerance shall be as indicated in Table B.4.

Table B.4—Viscometer Calibration

Calibration Weight g	Allowable Tolerance in Dial Deflection for Indicated Spring Constant					
	F 0.2	F 0.5	F 1.0	F 2.0	F 3.0	F 5.0
0	0 ±0.5	0 ±0.5	0 ±0.5	0 ±0.5	0 ±0.5	0 ±0.5
10	127 ±2	51 ±1	25 ±1	12.5 ±1	8.5 ±1	5.0 ±1
20	254 ±2	102 ±2	51 ±1	25 ±1	17 ±1	10 ±1
50		254 ±2	127 ±2	64 ±1	42 ±1	25 ±1
100			254 ±2	127 ±2	85 ±1	51 ±1
200				254 ±2	169 ±2	102 ±2
300					254 ±2	152 ±2
500						254 ±2

B.3.7   Instrument Rotation

B.3.7.1   Rotation of viscometer rotors and of consistometer cups (or paddles) measured by a calibrated tachometer shall be verified and/or calibrated (if not in calibration) no less frequently than quarterly.

B.3.7.2   Each viscometer speed shall be within ±1 % of the speed setting for speeds 100 r/min and greater or ±1 r/min for speeds less than 100 r/min

B.3.7.3   Consistometer speeds shall be 150 r/min, ±15 r/min. Additionally, for consistometers with variable speed control, tolerance shall be ±5 r/min at 25 r/min.

B.3.8   Pressurized Consistometer

B.3.8.1   Slurry container (informative procedure)

Pre-test checks visual verification can identify pressurized slurry container, paddle and paddle shaft potential wearing or damages. To judge wear, their key dimensions (see API 10A, dimensions tagged as "INSP"), should be verified, maximum acceptable wear is ±1 mm (±0.04 in.). Dimensions should be verified no less frequently than annually or whenever a paddle or a paddle shaft is replaced. If dimensions are found outside tolerances the slurry container part needs to be discarded.

To verify dimensions a caliper accurate to 0.05 mm (0.002 in.) at minimum is required.

.B.3.8.2   Consistency measurement device or potentiometer

B.3.8.2.1   General

The potentiometer or other consistency-measuring device shall be verified and/or calibrated (if not in calibration) no less frequently than monthly. A new calibration is required whenever repairs or adjustments are made to the device (such as calibration spring, resistor, contact arm). Accuracy shall be maintained within $\pm 5\,B_c$ (if output is in B_c) or ±0.5 V (if output is in volts) across the calibration range. When the calibration is of a system (potentiometer, wiring, voltmeter, etc.), for the calibration to be valid the potentiometer must be kept together with the consistometer with which it has been calibrated. For these

systems, laboratories shall devise a method of identifying the potentiometer with the consistometer on which it is calibrated and ensure it is only used on that consistometer.

Some potentiometer calibration devices allow the potentiometer to be calibrated so that the voltage output is fixed based on applied load (equivalent to B_c). In such a case, provided all potentiometers are adjusted to the same voltage output vs load [or consistency (B_c)], the potentiometers may be interchanged between machines.

B.3.8.2.2   Procedure

A calibration chart or table showing indicated consistency vs input consistency (mass) shall be maintained. The entire range of consistencies shall be checked as indicated in Table B.5. Consistency values shall be reported based on the calibration curve or table.

A weight-loaded device (see Figure B.2 for a typical potentiometer calibration device) is used to produce a series of torque-equivalent values for consistency, defined by Equation (B.1):

$$T = 78.2 + \left(20.02 \times B_c\right) \tag{B.1}$$

where

T is torque, expressed in gram•centimeters;

B_c is consistency expressed in Bearden units.

Dedicated potentiometer calibration weights are used to apply torque to the potentiometer spring, using the radius of the potentiometer frame as a lever arm. Weights cause a deflection, and the resulting DC voltage is recorded and used to determine B_c (alternatively, some instruments display the B_c equivalent directly). Slurry consistencies at ±5 B_c versus equivalent torques (mass of calibration weights at ±0.1 % of indicated value, for potentiometer with a radius of 52 mm ±1mm are provided Table B.5. Operating instructions from the manufacturer shall be followed for proper calibration.

NOTE   Dedicated potentiometer calibration weights shall be verified as per requirements of B.3.2. Each weight isincluding the holding support washer.

Figure B.2—Common Calibrating Device for Pressurized Consistometer Potentiometer

B.3.8.3   Slurry container (or paddle) rotation: see 3.7.3.

B.3.8.4   Temperature-measuring systems and controllers: see 3.5

B.3.8.5   Pressure gages: see 3.9

B.3.8.6   Chart recorder or **Data acquisition:** see 3.16

Table B.5—Slurry Consistency vs Equivalent Torque
(for potentiometer with radius of 52 mm ±1 mm)

Torque Equivalent g●cm	Mass of Added Weights[a] ±0.1% of the indicated mass	Slurry Consistency B_c ±5
260	50	9
520	100	22
780	150	35
1040	200	48
1300	250	61
1560	300	74
1820	350	87
2080	400	100
NOTE 1 For a potentiometer with a radius other than 52 mm ±1 mm, an adaptor ring with a radius of 52 mm ±1 mm or an appropriate table of equivalent tolerances is required. NOTE 2 A consistency reading of a potentiometer may vary no more than ±5 B_c from the slurry consistency shown in this table.		
[a] Mass of added calibration weight includes mass of the holding support washer.		

B.3.9   Pressure Gauges

B.3.9.1   Pressure gauges shall be verified and/or calibrated (if not in calibration) no less frequently than annually using a deadweight tester or a master gauge. The term "gauge" includes pressure-sensing transducers.

B.3.9 2   Gauges designed to measure pressures greater than 17,000 kPa (2500 lbf/in.²) shall be calibrated at a minimum of 25 %, 50 %, and 75 % of full scale or the maximum user defined working pressure of the equipment on or with which it is used. Maximum allowable error is ±1 % of full range or one minimum gauge increment, whichever is greater.

B.3.9.3   Gauges designed to measure pressures up to 17,000 kPa (2500 lbf/in.²) shall be calibrated with allowable error of ±300 kPa (±50 lbf/in.²) at a minimum gauge reading of ± 3500 kPa (± 500 lbf/in.²) and at a gauge reading of ±10,500 kPa (±1500 lbf/in.²) or the maximum allowable working pressure of the equipment on or with which it is used.

B.3.9.4   Calibration of gauges showing pressure of air used to operate pumps and purge pressure vessels of liquid is not required.

B.3.10   Sedimentation Tube

Sedimentation tube used to prepare a column of set cement shall be verified for its internal tolerance no less frequently than every two years. Internal diameter dimensions and tolerance are provided Table B.2. Verification should be done using a go/no go gage with diameters of 24.50 mm x 25.50 mm (0.980 in. x 1.020 in.). If sedimentation tube is out of tolerance, it shall be discarded

NOTE Tolerances for go/no-go gage diameters shall be ±0.02 mm (±0.001 in.).

B.3.11   Load Frame

The load frame used to measure break force of cement specimens shall be verified and/or calibrated no less frequently than annually. Indicated force shall deviate by no more than ±2 % of the applied load or one minimum instrument scale division, whichever is greater, at 9.0 kN (2000 lbf) load and at a minimum of 25 %, 50 %, and 75 % of the range of the load cell or load indicator. With units having multiple indicators for different ranges, each indicator shall be calibrated according to these criteria.

B.3.12   Cube-molds

Cube-molds in use to prepared cement specimens shall be verified no less frequently than every two years.

Cube dimensions to be verified and their tolerances are provided Table B.6. If molds are out of tolerances, molds shall be discarded.

Table B.6 — Dimensions for 50-mm (2-in.) Cube Molds in Use

Specification	Cube Molds
Distance between opposite sides	50.0 mm ±0.5 mm (2.00 in. ±0.02 in.)
Height of compartment	15.00 mm +0.25mm/-0.38 mm (2.00 in. +0.01 in./-0.015 in.)

B.3.13   Ultrasonic Devices

Transducers, cables, and slurry cells shall be calibrated as a system to a specific ultrasonic unit and used together as a set. Changing any one of the three makes a new calibration necessary. Users will devise a system to ensure that cells, cables, and transducers are used as matched sets. The ultrasonic transducers shall be calibrated no less frequently than monthly, according to manufacturer's procedures.

B.3.14   Pressurized Fluid Density Balances

B.3.14.1 Standard procedure

Pressurized fluid density balances shall be verified and/or calibrated (if not in calibration) annually in the range 1.80 kg/L to 2.30 kg/L (15 lbm/gal to 19 lbm/gal) and with water no less frequently than monthly. Tolerance shall be within ±0.01 kg/L (±0.1 lbm/gal). Calibration certificates shall indicate the serial numbers of all components of the balance (cup, cap, balance arm and slide weight, etc.) and indicate the deviation from the calibration point. Manufacturers provide methods for making the calibration with water at the high end. The following procedure should be used for the water calibrations.

a) Thoroughly clean the inside of the sample cup and lid assembly, the indication arm, and the sliding weight. There should be no set cement on the system and there should be no visible signs of wear.

b)   Fill sample cup with deionized water, place lid on cup, pressurize it, and check it for accuracy at 1.0 SG (8.33 lbm/gal).

c)   After it is verified to be correct with water, record the indication with water, remove the water from the sample cup, and dry the cup thoroughly.

d)   Place the lid and ring back on the sample cup.

e)   Attach the calibration fixture (see Figure B.3) on the base of the sample cup. The fixture consists of an all-thread, nuts, and hose clamp. The all-thread should point away from the balance beam.

f)   Adjust the nuts until it is balanced at 1.0 SG (8.33 lbm/gal) and lock the nuts against each other so they will not move.

g)   Remove the lid and fill the sample cup with deionized water.

h)   Replace the lid, pressurize the cup as before, and check the density. The density shall indicate 2.0 SG (16.7 lbm/gal) if it is in calibration at the higher density. Record the indication of the balance.
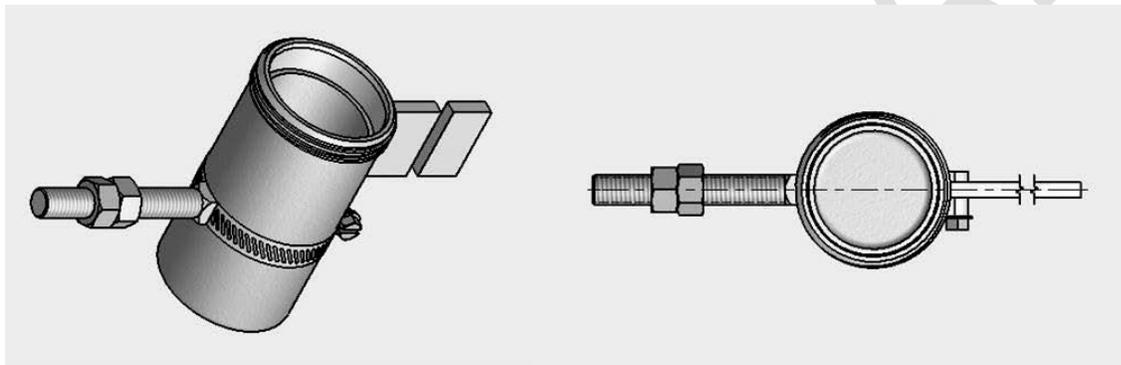
Figure B.3—Fixture for Calibration of Upper Density Range

B.3.14.2 Alternate procedure

An alternate procedure for performing the high-density calibration is by the use of steel or lead pellets (steel is preferred due to lead toxicity). The following procedure may be used for calibration using pellets.

a)   Fill the sample cup with deionized water, place the lid on the cup, pressurize it, and check for accuracy at 1.0 SG (8.33 lbm/gal). Record the indication with water.

b)   After the accuracy is verified to be correct with water, remove the water from the sample cup and dry the cup thoroughly.

c)   Carefully add small pellets to the cup until the unit is balanced at 1.0 SG (8.33 lbm/gal) (start with about 220 g). The pellets must be level in the cup and the lid replaced after each addition or removal of shot when verifying the balance.

d)   After the unit is balanced at 1.0 SG (8.33 lbm/gal) with pellets, remove and carefully weigh the pellets used. Record the weight of the pellets for future calibrations.

e)   Weigh out twice the amount of pellets recorded in Step d), place the pellets in the cup, and level them. Replace the lid and check the reading of the density. The indicated density shall be 2.0 SG (16.7 lbm/gal). Record the indication and the weight of these pellets.

Step c) can be eliminated from future calibrations, if the pellets used in Steps d) and e) are saved in clean sealed containers or if the pellets are weighed out each time according to the recorded values in Steps d) and e). In all cases, it is important that the pellets be level in the cup before each verification.

B.3.15   Fluid-loss Screens (informative subsection)

B.3.15.1   General

Fluid-loss screens shall be verified after being used with water with no less frequently than 5 fluid-loss tests. Tolerance shall be within ±10% of initial flow time. A fluid–loss screen tester (Figure B.4) shall be used to evaluate potential screen damage.

Verification shall be recorded and indicate the deviation from the initial verification point. Verification records shall indicate the test date and the serial numbers of each screen. Manufacturers provide equipment for making the verification check with water.

Detail A

Key

|			
1	cap, screen holder	**5**	o-ring
2	o-ring	**6**	nipple
3	tube, threaded	**7**	valve, ball
4	screen, 45 µm (325 mesh)	**8**	connection, hose
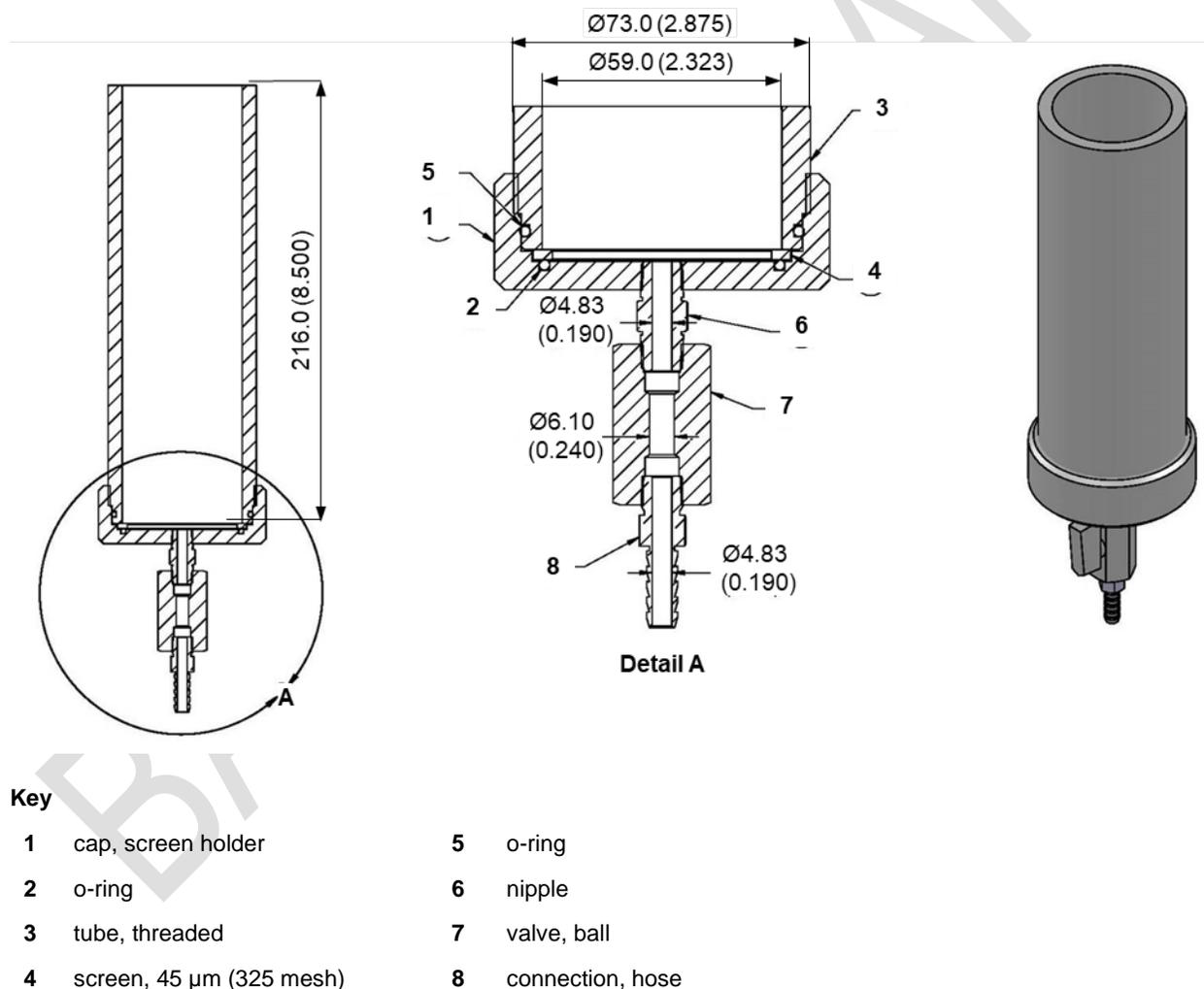
Figure B.4—Typical Fluid-loss Screen Tester Cell

B.3.15.2   Procedure

Fluid-loss screens shall be verified once initially for new screens and after every five fluid-loss tests maximum or after citric acid wash, with 500 g of deionized or distilled water.

The following procedure shall be used for the water verifications of new and used fluid-loss screens. Prior water flow verification visual inspection should be carried out. Fluid-loss screens with visible plugging, wear, damage, or distortion shall be discarded.

a) Assemble the fluid-loss screen tester (see figure with the 45 µm (0.00177 in.- 325 US mesh) screen facing inward and the 250 µm (0.01 in. - 60 US mesh) screen facing downward. Be sure to screw the bottom cap onto the test cylinder firmly to seal the edges of the screen.

b) Clamp the fluid-loss screen tester to a vertical stand such that the bottom valve can be easily operated.

c) With the bottom valve closed, fill the fluid loss screen tester with 500.0 g ±0.1g of tap or deionized or distilled water. Record type of water

d) Open the bottom valve stem and start the timer (stopwatch) simultaneously. Record the time at ±0.1 s) that the water level empties from the test cylinder. The timer shall be stopped when visible observation shows that the water has left the fluid-loss screen surface.

e) Repeat the procedure 3 (three) times to establish repeatability. Record the date, screen serial number and the average flow time.

    NOTE   Users will develop procedures to ensure that screens are identified by a serial number or equivalent.

f) If the average flow time is within tolerance of 10% of the initial flow time (out of the package) the fluid-loss screen can be used for another 5 fluid-loss tests.

    If the flow time is increased by more than 10%, fluid-loss screen should be cleaned in citric acid cleaning solution and then shall be retested [step a) through e)]. If retested average flow time is within 10% of the initial flow time, fluid-loss screen shall be used for fluid-loss testing (5 tests before retesting). If the retested average flow time is not in tolerance, fluid-loss screen shall be discarded.

B.3.16   Data Acquisition and Charts Recorders

B.3.16.1   Data acquisition can be by chart recorder or by electronic recording, such as computer data acquisition, or both.

B.3.16.2   For devices with chart recorders used for recording thickening-times or other data the indication of temperature and pressure, or other measurement shall be recorded during the verification/calibration.

Charts recorder shall be checked at least annually to ensure they vary by no more than 2 min/hr This can be done by running the chart, causing a deflection of the temperature or pressure trace at the beginning and end of one hour according to a calibrated timer, and measuring the deviation on the chart. If the chart can be adjusted to correct indication, the record on the chart should show the indication of a check before calibration and after adjustment of the chart. For those without an adjustment, a calibration table shall be maintained with the instrument and appropriate corrections made to charts of all tests conducted with the instrument. The chart shall be attached to the calibration record for the instrument.

The use of chart recorders is discouraged due to their inaccuracy.

B.3.16.3   Electronic data recording shall be verified by acquiring data during the calibration of the system and checking the accuracy of the data recorded. If the acquisition deviates by more than the limits for

temperature (B.3.5) and pressure (B.3.9), corrections shall be made. For dedicated data acquisition systems, the data acquisition system shall conform to the limits prescribed above for the data they are designed to record.

B.3.17   Volumetric Glassware

B.3.17.1   General

Calibration of pipettes, graduated cylinders, conical flask and similar equipment is generally performed by the glassware supplier and may be part of the purchase specification. Glassware users should obtain documented evidence, where deemed important, of glassware calibration from the supplier. Calibration may be checked gravimetrically before the first use. Periodic recalibration is not required.

B.3.17.2   Procedure—Verification/Calibration of graduated cylinders

The following procedure shall be followed to verified calibration of graduated cylinders. This procedure shall be adapted to any volumetric glassware.

a)   Allow the receiver and deionized (or distilled) water to reach ambient temperature. Record the temperature to the nearest 0.5 °C (1 °F).

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

c)   Fill the receiver with deionized (or distilled) water, while it is on the balance, to the various graduation marks in increments of 2 mL for the 10 mL receiver, in increments of 4 mL for the 20 mL receiver, and in increments of 10 mL for the 50 mL receiver. Use a pipette or syringe to carefully fill the receiver to the desired graduation mark without leaving water droplets on the walls of the receiver.

d)   Record the masses for each incremental volume of water at the specific graduation mark, m_W, to the nearest 0.01 g.

e)   Calculate the volume of the receiver at each mark, V_M as given in Equation (B.2)

$$V_M = \frac{m_{dw}}{\rho_{dw}}$$ (B.2)

where

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

   m_{dw} is the mass of water, expressed in grams;

   ρ_{dw} is the deionized water density, expressed in grams per milliliter, at the test temperature (Table B.7).

Table B.7—Density of Deionized/Distilled Water as a Function of Temperature

Temperature °C	Density ρ_{dw} g/mL	Temperature °F	Density ρ_{dw} g/mL
15.0	0.9991	59	0.9991
15.5	0.9990	60	0.9990
16.0	0.9989	61	0.9989
16.5	0.9989	62	0.9988
17.0	0.9988	63	0.9987
17.5	0.9987	64	0.9986
18.0	0.9986	65	0.9985
18.5	0.9985	66	0.9984
19.0	0.9984	67	0.9983
19.5	0.9983	68	0.9982
20.0	0.9982	69	0.9981
20.5	0.9981	70	0.9980
21.0	0.9980	71	0.9978
21.5	0.9979	72	0.9977
22.0	0.9978	73	0.9976
22.5	0.9977	74	0.9975
23.0	0.9975	75	0.9973
23.5	0.9974	76	0.9972
24.0	0.9973	77	0.9970
24.5	0.9972	78	0.9969
25.0	0.9970	79	0.9968
25.5	0.9969	80	0.9966
26.0	0.9968	81	0.9965
26.5	0.9966	82	0.9963
27.0	0.9965	83	0.9961
27.5	0.9964	84	0.9960
28.0	0.9962	85	0.9958
28.5	0.9961	86	0.9956
29.0	0.9959	87	0.9955
29.5	0.9958	88	0.9953
30.0	0.9956		
30.5	0.9955		
31	0.9953		

BALLOT DRAFT

Annex C
(normative)

Alternative Apparatus for Well-simulation Thickening-time Tests

C.1 General

This annex describes alternative pressurized consistometers for the well-simulation thickening-time testing of cement slurries.

C.2 Apparatus

This consistometer has a rotating paddle and a stationary cup and is constructed such that the cement slurry can be subjected to the temperatures and pressures required by the well-simulation test schedules described in 9.4. The inside dimensions of the cup shall conform to the requirements for the slurry cup defined in API Specification 10A. The rotating paddle shall conform to the dimensions defined in API 10A with the exception that the shaft may be modified to meet the requirements of the drive mechanism of the alternative thickening-time test apparatus. The system isolating the test fluid inside the cup may be a diaphragm as depicted in API Specification 10A or any system suitable of isolating the test fluid from the pressurizing medium.

Paddle torque is sensed by motor load or alternate torque sensors to provide slurry consistency measurements equivalent to those of the typical consistometer described in 9.2. Slurry temperature and pressure controls shall be provided. General schematics of alternative configurations are shown in Figure C.1 and Figure C.2. The apparatus shall be capable of duplicating the test conditions and measurements required of the consistometer described in 9.2.
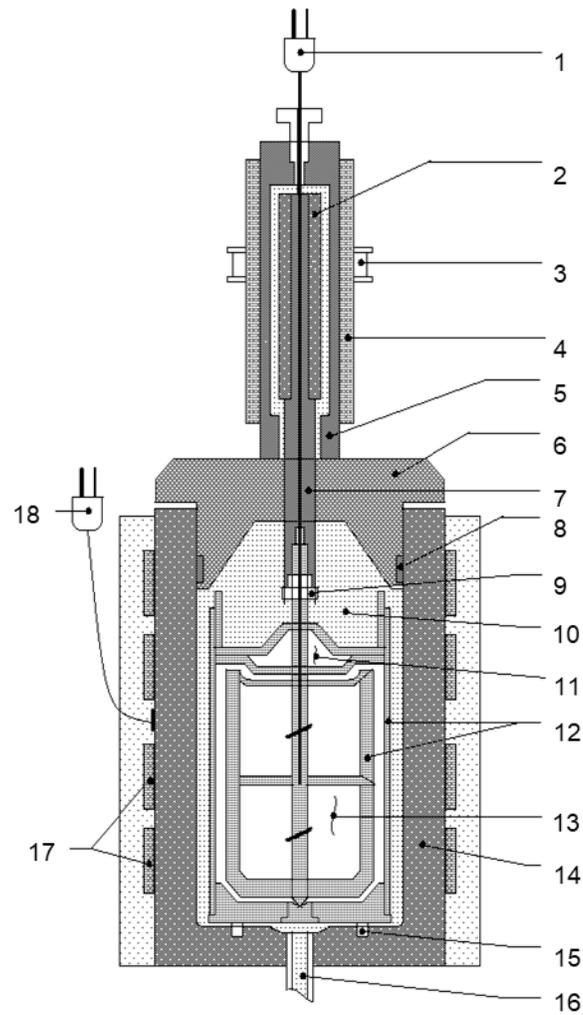
Key

1	slurry temperature sensor	10	pressure medium
2	inner magnet	11	isolation area (slurry/pressure medium)
3	drive pulley	12	slurry container assembly with paddle
4	outer magnet	13	slurry
5	pressure housing	14	pressure chamber
6	lid	15	slurry container retainers
7	drive shaft	16	pressurization port (also fill/empty)
8	lid seal	17	heater elements
9	paddle drive coupling	18	chamber temperature sensor

Figure C.1—Alternative Consistometer Design for Well-Simulation Thickening-time; Example 1
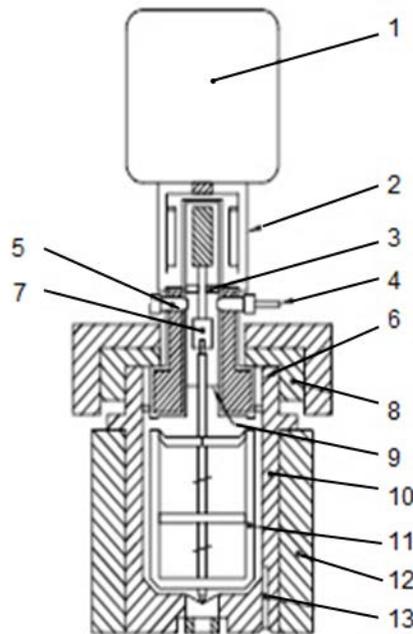
Key

1	motor/generator	8	auxiliary heater jacket
2	magnetic drive	9	oil/cement interface
3	pressure-transmitting seal	10	slurry pressure vessel
4	pressurizing port	11	rotating paddle
5	air vent	12	main heating/cooling jacket
6	auxiliary thermocouple	13	main thermocouple well
7	mechanical drive coupling		

Figure C.2—Alternative Consistometer Design for Well-simulation Thickening-time; Example 2

C.3 Calibration

Apparatuses shall be calibrated according to the requirements in Annex B.

The same requirements for calibration apply to the use of these alternative devices as to the consistometer of 9.2. The equipment manufacturer's procedures for the calibration of the pressurized consistometer, including consistency measurement, temperature measurement, temperature controllers, motor speed, timer, and pressure gauges, shall be followed so long as they conform to the provisions of Annex B.

C.4 Test Procedure

The equipment manufacturer's detailed procedures for the operation and maintenance of the equipment shall be followed and shall satisfy the intent of the general procedures in Section 9. Some modifications may be necessary to accommodate the design variations of the alternative device. Do not exceed manufacturer's safety limits.

Annex D
(informative)

Cementing Temperatures and Schedules

Table D.1—T_{PBHC} for Casing and Liner Well-simulation Tests

SI Units						
Depth h_{TVD} m	Temperature Gradient °C/100 m depth					
	1.60	2.00	2.40	2.80	3.20	3.60
	°C	°C)	°C	°C	°C	°C
300	27	27	27	27	27	27
600	32	32	32	32	33	33
1200	37	38	38	39	39	40
1800	44	45	46	48	49	53
2400	52	53	57	60	64	73
3000	60	63	69	75	84	96
3600	63	73	84	94	105	116
4200	71	84	97	110	123	136
4800	80	96	111	126	142	157
5400	90	109	126	144	162	180
6000	101	122	143	164	184	205
6600	114	137	161	185	209	232

USC Units						
Depth h_{TVD} ft	Temperature Gradient °F/100 ft depth					
	0.9	1.1	1.3	1.5	1.7	1.9
	°F	°F	°F	°F	°F	°F
1000	80	80	80	80	80	80
2000	89	89	90	90	91	91
4000	99	100	101	102	103	104
6000	112	114	116	118	120	126
8000	126	129	135	140	146	160
10,000	141	146	158	167	180	200
12,000	148	165	183	201	219	236
14,000	164	185	207	228	250	271
16,000	182	207	233	258	284	309
18,000	201	231	261	291	321	350
20,000	222	256	291	326	360	395
22,000	244	284	324	364	404	444

NOTE   Predicted bottomhole circulating temperatures are calculated using Equation (37) at depths greater than 3050 m with SI units and using Equation (38) at depths greater than 10,000 ft with USC units.

Table D.2—T_{PSP} for Squeeze-cementing Well-simulation Tests

Depth h_{TVD} m	Temperature Gradient °C/100 m depth					
	1.60	2.00	2.40	2.80	3.20	3.60
	°C	°C	°C	°C	°C	°C
300	27	27	28	29	30	30
600	30	31	33	35	37	39
1200	37	41	45	49	52	56
1800	45	51	57	62	68	74
2400	53	61	69	77	85	92
3000	62	72	82	92	102	111
3600	70	83	95	107	119	131
4200	79	94	108	123	137	152
4800	89	106	122	139	156	173
5400	98	118	137	156	176	195
6000	109	130	152	174	196	218
6600	119	144	168	192	217	241

USC Units						
Depth h_{TVD} ft	Temperature Gradient °F/100 ft depth					
	0.9	1.1	1.3	1.5	1.7	1.9
	°F	°F	°F	°F	°F	°F
1000	80	80	82	83	85	86
2000	86	89	92	95	98	101
4000	100	106	113	119	125	132
6000	115	124	134	144	153	163
8000	130	143	156	169	182	196
10,000	146	163	179	196	213	229
12,000	162	183	203	223	244	264
14,000	179	204	228	252	276	300
16,000	197	225	253	281	309	338
18,000	215	248	280	312	344	376
20,000	234	271	307	344	380	417
22,000	254	295	336	377	418	459

NOTE 1   Predicted squeeze temperatures are calculated using Equation (50) with SI units and calculated using Equation (51) with USC units.

NOTE 2   Hesitation squeeze schedules: After the final squeeze pressure (p_{FSQ}) is reached, the temperature shall be increased to static temperature T_{BHS} at 0.11 °C (0.2 °F) per minute. At this same time the stirring shall be cycled off-on, typically for 10 min off and 5 min on until the test is terminated.

NOTE 3 T_{PSP} shall be the plug cementing well-simulation temperature if the well will not be circulated prior to cementing, or if area of the wellbore is not subjected to circulation.

Annex E
(informative)

Determination of Bulk Density of Solids

E.1   Bulk Density, Absolute Density and Absolute Volume

E.1.1 General

The terms bulk density, absolute density (or density, see 3.1.1), and absolute volume must be understood to prevent confusion.

NOTE   "Density" is generally the term used for "absolute density".

Bulk density and absolute density both have density units, that is, kilograms per cubic meter (pounds-mass per cubic foot, pounds-mass per gallon), etc .

Bulk and absolute density values for Portland cement and other powdered materials are very different.

E.1.2   Bulk Density

Bulk density includes the air space around particles, thus is a smaller number than the absolute density (density of the material itself). Bulk density is used to calculate storage requirements for dry powdered cement or other dry powdered materials such as blends of Portland cement with materials like pozzolan or composite cements.

The bulk density for Portland cement can vary, but it is usually about 1506 kg/m^3 (12.6 lbm/gal or 94 lbm/ft^3).

E.1.3   Absolute Density

Absolute density is the density of the material alone (does not include the air around the particles) and is thus a much larger number than bulk density. It is similar to the relative density and can be obtained by multiplying the relative density of a material by the density of water at 4 °C, 1000 kg/m^3 (8.345 lbm/gal).

E.1.4 Absolute Volume

Absolute volume is the reciprocal of the absolute density and is the volume occupied by a material in a liquid suspension (slurry). In cementing handbooks, an absolute volume "factor" is given for making slurry calculations. Units of absolute volume are volume per mass (cubic meters per kilogram, liters per kilogram or gallons per pound-mass.

Absolute density (or absolute volume) is used to calculate liquid slurry properties such as slurry density, slurry water requirement, and slurry yield.

E.2   Bulk Density Determination Procedure

The average bulk density is the average of the "loose apparent bulk density" and the "packed apparent bulk density" of a powdered material and shall be determined by using the following procedure.

a) Use a clean, dry 100 mL (to contain type) graduated cylinder for the determination of both loose and packed apparent densities. Graduated cylinder shall be verified and/or calibrated as per requirements of Annex B.

   NOTE   Accuracy of the graduated cylinder should be checked by filling with 99.75 g of distilled water, which equals 100 mL volume at 23 °C (99.76 g of distilled water at 73 °F).

b) Place about 200 mL of the sample to be tested in a jar of approximately 1 L (1 qt) volume, seal with a lid, and handshake to "fluff" the material for approximately 30 s.

c) Over an approximate 1 min period, loosely fill the tared graduated cylinder with fluffed material to the 100 mL mark. Weigh the sample and record for calculation of loose apparent bulk density.

d) "Pack" the material from item c) by gently tapping the cylinder on a hard surface, cushioned with a pad to prevent breakage of the cylinder. Record the volume of material after each 100 taps and continue tapping until the compacted volume is unchanged.

e) Record the packed volume (V_{fp}) of material directly from the cylinder graduations. Use this volume to calculate the packed apparent bulk density.

f) Calculate the bulk density in common field units as follows:

   The loose apparent bulk density, ρ_{LAB}, expressed in kilograms per cubic meter is given in Equation (E.1)

   $$\rho_{LAB} = \frac{1000}{100} \times m_{lfm} \tag{E1}$$

   or expressed in pounds-mass per cubic feet in Equation (E.2)

   $$\rho_{LAB} = \frac{62.43}{100} \times m_{lfm} \tag{E2}$$

   The packed apparent bulk density, ρ_{PAB}, expressed in kilograms per cubic meter is given in Equation (E.3)

   $$\rho_{PAB} = \frac{1000}{V_{fp}} \times m_{lfm} \tag{E3}$$

   or expressed in pounds-mass per cubic feet in Equation (E.4)

   $$\rho_{PAB} = \frac{62.43}{V_{fp}} \times m_{lfm} \tag{E4}$$

   where

   m_{lfm} is the mass of 100 mL of loosely filled material, expressed in grams;

V_{fp} is the final packed volume attained from the original 100 mL of loosely filled material, expressed in milliliters

g) Report average bulk density, ρ_{bulk}, expressed in kilograms per cubic meter (pounds-mass per cubic foot) as the average of the loose and packed bulk densities and Equation (E.5).

$$\rho_{\text{bulk}} = \frac{\rho_{\text{PAB}} + \rho_{\text{LAB}}}{2} \tag{E5}$$

where

ρ_{LAB} is the loose apparent bulk density, expressed in kilograms per cubic meter (pounds-mass per cubic foot);

ρ_{PAB} is the packed apparent bulk density, expressed in kilograms per cubic meter (pounds-mass per cubic foot).

The absolute density for Portland cement can vary, but it is usually about 3180 kg/m^3 (26.5 lbm/gal or 198.6 lbm/ft^3).

Annex F
(informative)

Determination of Rheological Properties and Gel Strength Using a Pressurized Rotational Viscometer

F.1 General

This procedure should be used to characterize the rheological behavior of fluids used in well cementing under conditions of greater than atmospheric pressure. Determination of rheological properties of test fluids can be sensitive to the procedure being used. A comparison with rheological properties obtained using other methods is not recommended.

Note   Procedure is applicable for determination of rheological properties at temperature above 88 °C (190°F) which require to pressurize cement slurry to avoid boiling of water.

This standardized procedure has been developed to generate reproducible results for a given fluid. It was developed after a careful analysis of many parameters that affect the rheological behavior of fluids. Results may not be accurate when measuring the rheological properties of highly dispersed fluids.

The following conditions shall be necessary to properly characterize the rheological parameters of a fluid using a rotational viscometer:

a)   the slurry is homogenous, and the shear stress is uniform across the gap,

b)   the flow regime in the annular gap is laminar,

c)   slip at the wall is negligible, and

d)   the fluid exhibits time-independent behavior.

F.2 Apparatus

F.2.1   High-temperature, High-pressure (HTHP) Rotational Viscometer

F.2.2.1. General

A typical rotational HTHP viscometer, also known as a Couette HTHP viscometer, should be used to measure the rheological properties of fluids used in well cementing at simulated downhole conditions. This device consists of a cylindrical bob located concentrically within a cylindrical rotor, also referred to as a sleeve. The width of the gap between the bob and the rotor is fixed for a given rotor/bob configuration. The rotor can turn at various specified rotational velocities. The rotation of the rotor acts upon the fluid being tested to impart a torque on the bob. The bob is mounted on a shaft that is connected to a torsion device, which restrains its movement. The torque measured is then converted into shear stress. In addition, a HTHP viscometer simulates downhole temperatures and pressures.

F.2.2.2 High-temperature, medium-pressure (HTMP) rotational viscometer

A typical rotational HTMP viscometer, is basically similar to a HTHP viscometer. However rather than simulating downhole pressure, the HTMP is typically pressurized using a gas. This pressure is not intended to simulate downhole pressure but is applied to prevent the liquids in the slurry from boiling off during the test. The pressure is typically held constant though out the test.

F.2.2.3   Rotor and bob for HTHP viscometer

Various sizes of rotors and bobs shall be available. Table F1 shows the dimensions of the rotors and bobs used for measuring the rheological properties of fluids used in well cementing. The ratio of the radius of the bob to the rotor should be > 0.9 to maintain a nominally uniform shear stress across the gap and to keep the slurry homogenous. Thus, for measuring the rheology of well cementing fluids the combination of rotor size R1 and bob size B1 should be used in most cases. The gap should also be a minimum of ten times wider than the largest particle diameter. This criterion may require the use of a smaller diameter bob, which results in a radius ratio <0.9. For special slurries containing large diameter particles, a B5 bob should be usually sufficient to provide the required gap width.

Table F.1—Dimensions of Rotors and Bobs

Dimensions in millimeters (inches)

	Rotor-Bob		
	R1-B1	**R1-B2**	**R1-B5**
Rotor internal diameter	36.83 ± 0.02 (1.450 ± 0.001)	$36.83 \pm 0.02.$ (1.450 ± 0.001)	$36.83 \pm 0.02.$ (1.450 ± 0.001)
Bob external diameter	34.49 ± 0.02 (1.358 ± 0.001)	$24.54 \pm 0.02.$ (0.966 ± 0.001)	$31.97 \pm 0.02.$ (1.259 ± 0.001)
Bob length	38.00 ± 0.02 (1.496 ± 0.001)	$38.00 \pm 0.02.$ (1.496 ± 0.001)	$38.00 \pm 0.02.$ (1.496 ± 0.001)
Gap width	1.17 ± 0.02 (0.046 ± 0.001)	$6.14 \pm 0.02.$ (0.242 ± 0.001)	$2.43 \pm 0.02.$ (0.096 ± 0.001)
Bob/rotor radius ratio	0.937	0.666	0.868

F2.2.4   Shear rate

Shear rate is a function of the rotational velocity of the rotor and the width of the gap between the rotor and the bob. Table F.2 shows the calculated shear rate for each rotor-bob combination across a range of rotational velocities.

F2.2.5   Shear stress

Shear stress is expressed in units of pascals (pounds force per one hundred square feet). It is a measurement of the deflection of the bob that is a function of torque and is dependent upon the dimensions of the bob and the torsional stiffness of the device. Torsion devices of varying torsional stiffness are available from the various manufacturers and their usable ranges are available from them.

Table F.2—Shear Rate for Rotor-Bob Combinations

Rotational Velocity (r/min)	Rotor-Bob		
	R1-B1	**R1-B2**	**R1-B5**
	Shear Rate (s^{-1})		
1	1.70	0.38	0.85
2	3.40	0.75	1.70
3	5.11	1.13	2.55
6	10.21	2.26	5.10
10	17	3.77	8.50
20	34	7.54	17
30	51	11.31	25
60	102	22	51
100	170	38	85
200	340	75	170
300	511	113	255
600	1021	226	511

F2.2.6   Calibration

Viscometers shall be calibrated according to the requirements in Annex B and according to the manufacturer's instructions. Calibrations shall include viscometer torsion device and rotor speed as thermocouple, pressure gauges, time measuring devices and data acquisition.

F2.3   Timer or Stopwatch

A stopwatch or electric timer with time indicated in seconds should be used used.

F.3   Determination of Rheological Properties

This procedure shall be recommended when running tests at greater pressure than atmospheric pressure and/or temperature above 88 °C (190 °F).

a)   Prior to preparing the slurry sample, program the viscometer for the test parameters needed for the test, as per manufacturers' instructions.

b)   After preparing the sample according to Section 5, fill the viscometer sample cup as per manufacturers' instructions. If possible, test should begin within 20 min of mixing the slurry. Some equipment or slurries may require more time. If additional time is required, record the total time.

c) With the viscometer rotating at 340 s⁻¹ (see Table F.2), i.e. 300 r/min using R1-B1 combination, heat or cool to T_{PBHC} in accordance with the pressure/temperature schedule designed to simulate conditions in the well. Hold test temperature and pressure for 30 min ±30 s to allow the test fluid temperature to reach equilibrium. This hold time may be modified to simulate cementing operations. Proper note should be made of this in the test report.

d) Change shear rate to 5.1 s⁻¹ (i.e. 3 r/min using R1-B1 combination) and begin recording data after a minimum 20s of continuous rotation at speed. Record and average data for a minimum of 10 s.

e) Take the average of remaining readings, first in ascending order and then in descending order, after a minimum continuous rotation of 20 s at each shear rate. The highest recommended shear rate is 511 s⁻¹ (i.e. 300 r/min using R1-B1 combination). Exposing cement slurries to speeds above 511 s⁻¹. can lead to inconsistent (erroneous) results.

f) If desired, readings at speeds higher than 511 s⁻¹ may be taken after ramping up and down and measuring the gel strength (see F.5). After taking all the readings, record the final temperature of the slurry in theviscometer.

F.4   Multi-point Pressurized Rheology

At user's discretion, multiple temperatures and pressures can be run on a single test, by following F.3 item c) through item f) for each temperature and pressure.

F.5   Determination of Gel Strengths

F.5.1   Gel Strengths at 10 s and at 10 min

The gel strength of a fluid may be measured immediately after determining the rheological properties of the sample or on a separate, freshly prepared fluid.

NOTE   Gel strengths of a fluid are the measurements resulting of the application of the rheological property procedure described under F.3. These measurements are not comparable to static gel strength of a fluid measured under API 10B-6 or ISO 10426-6 and shall not be used for critical static gel strength determination.

The following procedure shall be recommended to determine gel strength:

a) Recondition the fluid in the viscometer for 1 min at 511 s⁻¹ (i.e. 300 r/min using R1-B1 combination). to disperse the gels and to allow better measurement of the gel strength. For tests on separate, freshly prepared fluids, prepare, condition, and load the fluid into the viscometer as outlined in F.3 a) to F.3 c).

b) Stop rotation of the rotor.

c) Set the viscometer at the speed equivalent to 5.1 s⁻¹ (i.e. 3 r/min with R1-B1 combination).

d) 10 s after stopping the rotor, start rotation at 5.1 s⁻¹ and record the maximum shear stress observed. Record the fluid temperature, then stop the viscometer for 10 min (or other specified time, see).

e) 10 min After stopping the rotor, start rotation at 5.1 s⁻¹ and record the maximum shear stress observed immediately after starting rotation.

f) After taking the maximum deflection dial reading, again record the temperature of the fluid in the viscometer cup.

g) Report the slurry gel strengths at the recorded temperatures (note the rotational velocity, the rotor-bob configuration, used in the test).

h) For improved reliability of the measurements, the entire procedure may be repeated two times or three times using freshly prepared fluid each time. Report the gel strength values as the average of the measurements.

F.5.2   Gel Strengths at Other Static Times

Gel strength measurements may be taken at other static times, as desired (example 30 min or other). The test shall have a 10 s gel strength and no more than one other gel strength time. If other gel strengths are required, a new slurry should be prepared and tested for each set of two gel strength times.

Procedure shall be similar to gel strength determination at 10 min: Prepare, condition, and load the freshly prepared fluid into the viscometer as outlined in F.3 a) to F.3 c) and then condition the fluid for 1 min at 300 r/min. Then follow the whole F.5.1 procedure step b) to h), substituting the desired static time for 10 min.

F.6   Rheological properties Recording and Characterization of Rheological Behavior

F.6.1   Data Recording

Following data shall be recorded:

a) Rotor, bob, configuration.

b) Time, temperature, and method of conditioning.

c) Heating or cooling time at T_{PBHC} in viscometer.

d) Pressure at T_{PBHC}

e) Shear rate (r/min) and shear stress at each r/min.

f) Temperature at end of rheology measurements.

g) Gel strengths at 10 s and at 10 min (or at any times) , measurements and average values, and gel strength temperatures.

h) Calculate the ratio of the dial readings during ramp-up to ramp-down at each speed. This ratio can be used to help qualify certain fluid properties.

i) When the ratio at all the speeds is close to 1:1, this can suggest that the fluid is a non-settling, non-gelling, time-independent fluid at the test temperature.

j) Ratios other than 1:1 can suggest settling or gelation of the fluid during the test. If some ramp-down dial readings are lower by more than 5 instrument degrees at the same rotational speed (obtained with the viscometer described in F.2), this may be a further indication of settling.

k) When significant differences in the dial readings indicate that the test fluid is not stable, that is, prone to settling or excessive gelation, adjustments in the composition shall be considered.

l) Report the fluid rheological measurements as the average of the dial readings [(ramp-up + ramp-down)/2], and the temperatures recorded in item j). Also report the rotor and bob configuration, as well as the conditioning methods used. An example is shown in Table F.3.

m) For improved reliability of the data, the entire procedure may be repeated two or three times, using freshly prepared fluid each time, and the results averaged.

n) An example of rheological data report is given in Table F.3.

Table F.3—Example Rheological Data Report

Rotational Speed r/min	Ramp-up Reading	Ramp-down Reading	Reading Ratio	Average Reading
3	21	24	0.87	22.5
6	40	36	1.11	38
30	65	83	0.78	74
60	84	100	0.84	92
100	100	115	0.87	107.5
200	137	147	0.93	142
300	170	—	—	170
NOTE 1 Configuration: R1-B1.				
NOTE 2 Conditioning method: conditioning pressure, 30 min after reaching 66 °C.				
NOTE 3 Initial slurry temperature: 66 °C.				
NOTE 4 Final slurry temperature: 63 °C.				
Example of notes about the test above. The information underlined is to be filled in for the test performed				

F6.2 Characterization of Rheological Behavior

Rheological behavior of the tested cement slurry shall be done following models and equations given in 11.6.

Annex G
(informative)

Alternates Slurry Mixing Procedures

G.1 Overview

This annex is providing alternates slurry mixing procedures which should be recommended to prepare cement slurries requiring specific attentions. Annex G is covering:

a)   Mixing slurries prepared with "fragile" shear sensitive additives. They mainly cover lightweight microspheres used in lightweight cement formulations.

b)   Mixing slurries whith excess foaming tendencies, these slurries should require de-airing.

G.2 Lightweight Cement Slurry (LWS) with "Fragile" Additives

G.2.1 General

Lightweight cement slurries (LWS) with density below 1.50 kg/L to 1.40 kg/L can be mixed with cement blend incorporating ceramic (alumina-silicate) or glass hollow microspheres as lightweight materials. These fragile additives are subject to crushing when exposed to pressure. It results in cement slurries with a lower yield and higher density than the surface and design conditions. Downhole conditions shall be taken at the design stage.

Cement blend quality control shall be provided by the supplier, cement design shall be based on blend sample. In this section only slurry performance tests are covered.

G2.2 LWS Testing Procedures

To obtain reliable and reproducible results, mixing conditions, conditioning (pressure and and temperature schedule) and temperatures of the measurements must be reported on the laboratory report. A commentary section must be filled with any observation or remarks linked with performed tests.

G.2.2.1 Mixing procedures

Section 5 mixing procedure should not be applicable to LWS containing hollow ceramic or glass microspheres. As high shear mixing is breaking a large amount of microspheres, low shear mixing is required. This recommendation to mix LWS is based on keeping the same API mixing energy as for standard slurries (5.89 kJ/kg). The recommended slurry preparation procedure should follow Section 5 with the exceptions for mixing procedure (5.3.4) as the following ones:

a) Prepare blend of cement (cement plus light weight additives and any other solids as required for the formulation to be tested); blend to be prepared is as per the requirement for 600 mL of slurry.

b) Add the cement additives to the mix water under 4000 r/min ±250 r/min;

c) Add the cement blend within 30 seconds while mixing at 4000 r/min ±250 r/min;

d) Continue to mix at the speed of 4000 r/min ±250 r/min for 300 seconds.

If cement blend addition within 30 s time does not bring homogeneous mixture between blend powder and liquid phase, it is recommend adding slowly cement blend to liquid to ensure properly homogenization and record addition time. In that case, total mixing time (adding the cement blend and 300 s mixing time) should be equal to 330 s. Report total mixing time.

The slurry is then ready to be conditioned and tested. Testing will be performed at surface conditions <u>and</u> after conditioning at downhole conditions (T_{PBHC} and p_{BH}, see 5.4.2). Slurry pressure conditioning at downhole conditions shall be required when anticipated a bottom hole pressure higher or equal to 13,800 kPa (2000 lbf/in.²).

G.2.2.2 Slurry conditioning

Slurry characteristics shall be measured at surface conditions and also after conditioning at T_{PBHC} and p_{BH} to take in account the collapse of hollow spheres under pressure and its consequences on final slurry properties.

For downhole pressure not greater than 10,300 kPa (1500 lbf/in.²), atmospheric conditioning (see 5.4.3) should be suitable for that purpose as it is admitted that pressure lower than 10,300 kPa (1500 lbf/in.²) should have no strong incidence on hollow lightweight additives.

G.2.3 Density

G.2.3.1 Density at surface conditions ($\rho_{\mathrm{s,m}}$)

The density of LWS shall be measured using a pressurised mud balance (see Section 6). The density must be taken just after mixing, not after conditioning or heating up. The reading can be slightly different than the calculated design value (±0.01 sg). On job site, this measured value should be the mixing value.

Density after mixing can be considered generally right for LWS slurry at downhole conditions with a maximum downhole pressure up to 7000 kPa to 10,300 kPa (1000 lbf/in.² to 1500 lbf/in.²).

G.2.3.2 Density at downhole conditions ($\rho_{\mathrm{s,dh}}$)

The slurry density shall be measured using the pressurized mud balance after pressurized conditioning at room temperature on a cooled sample. This is the density at which the slurry would be pumped in the well and used to calculate p_{BH} and the maximum ECD.

Then, from the density increase or volume reduction due to microsphere collapse, the yield at bottom hole conditions, $V_{\mathrm{s,dh}}$, expressed in liters per metric ton of cement (or blend) [cubic feet per sack of cement (or blend)] as given in Equation (1) shouldbe calculated and therefore the slurry mixing volume require to obtain the TOC as per design requirement.

$$V_{\mathrm{s,dh}} = \frac{V_{\mathrm{s}} \times \rho_{\mathrm{s,m}}}{\rho_{\mathrm{s,dh}}} \tag{1}$$

where

V_{s} is the yield at surface conditions expressed in liters per metric ton of cement (or blend) [cubic feet per sack of cement (or blend)];

$\rho_{s,m}$ is the cement slurry density measured at surface conditions expressed in kilograms per liter (pounds per gallon);

$\rho_{s,dh}$ is the cement slurry density at downhole conditions, expressed in kilograms per liter (pounds per gallon).

G.2.4   Rheology and Gel Strength

Rheologies and gel strength measurements shall follow requirements and procedures provided Section 11 Due to the size of hollow microspheres and viscosimeter gap measurements should be done generally using the larger gap provided by R1B5 combination. Two sets of measurements should be required.

a)   Rheology at surface conditions: measurements after mixing.

b)   Rheology at downhole conditions after pressurized conditioning

Measurement should be performed in heated cup assembly. The maximum temperature for an atmospheric viscometer shall not exceed 88°C (190°F) for safety reasons and to prevent dehydration of the slurry.

G.2.5   Fluid Loss

Fluid loss measurement shall be performed on conditioned slurry following requirements Section 12.

NOTE   Slurry conditioning in a stirred fluid-loss apparatus is acceptable only if the downhole pressure is below the maximum pressure rating of the stirred fluid-loss apparatus.

G.2.6   Thickening-time

The thickening-time shall be performed using a pressurized consistometer as per standard procedures (Section 9). It is very important to follow the heating up as pressure increases at the same time. Above 27,500 kPa (4000 lbf/in.²) large quantities of lightweight particles start to break, and the slurry properties can be affected.

In case of batch mixing, the simulation should be done in the pressurized consistometer at 80°F (or specified rig temperature) without pressure.

G.2.7   Compressive Strength

Compressive strength of LWS cement shall be determined by crushing API cubes (Section 7) or by a non-destructive sonic testing procedure (Section 8).

Considering slurry volume reduction due to crushing of hollow microsphere at high pressure, according to the crushing resistance of the material, it should be required to fill cube molds or UCA cell with pressurized conditioning slurry at the expected downhole pressure. Pressurized conditioning shall be mandatory if down hole pressure is higher than the curing pressure.

An important sonic test result is the ability to determine acoustic impedance of the tested slurry. Acoustic impedance should be used to calibrate response of acoustic logging tools and to evaluate LWS cement bond.

G.2.8   LWS Stability Tests

G.2.8.1   Free-fluid test

Free-fluid measurements should be performed <u>on conditioned slurry</u> as per described in G.1.2.2.

G.2.8.2 Sedimentation test

Sedimentation test is allowing the evaluate LWS stability Test operating conditions are described in 12.5. Test tube shall be cured it in a pressurised vessel in static conditions under downhole conditions (T_{PBHC} and p_{BH}). It may have interest in running in parallel a tube stability test on fresh mixed cement slurry and on pressurized conditioned slurry (see G.1.2).

This test should provide also a good indication related to free fluid volume and microsphere collapse under pressure: difference of cement column height may be measured between slurry column before setting and set cement column after setting.

G.3 Preparation of Slurries with Foaming Tendencies—De-airing Cement Mixing Procedure

G.3.1 General

Cement slurries mixed according to Section 5 or Annex A. may produce excessive foaming. Excessive foaming may reduce the volume of cement available for testing leading to invalid test results. This procedure applies a vacuum while mixing to reduce the foaming tendency and removed trapped air from cement slurries

Cement slurries should use this procedure when mixed according to Section 5 or Annex A. and the pressurized density measurement, as described in Section 6, results in 0.03 kg/L (0.27 lbm/gal) or greater variation.

NOTE   Mixing energy of this procedure would not be equal to the procedure according to Section 5.

G.3.2 Apparatus

G.3.2.1 General

All apparatuses shall be the same according to Section 5 and Annex A.1 with only the modifications and addition described hereafter.

G.3.2.2 Mixing device

The mixing device shall have a 1-liter (1-quart) capacity for standard volumes or 4 L to 5 L for large volumes. A modified mixing device cover should be fabricated in order to seal the cement slurry container when a vacuum is applied and with a pipe connection at the top to apply the vacuum.

An example of a mixing device for cements that exhibit excessive foaming and requiring de-airing is shown Figure G.1.

G.3.2.3 Vacuum pump

A standard laboratory vacuum pump able to provide a vacuum of 10 kPa $\pm$0.5 kPa (1.5 lbf/in² $\pm$0.7 lbf/in²) should be required. In the case of vacuum provided by a jet pump, its lateral outlet should be mounted with a check valve in order to avoid any potential water reflux toward the vacuum line.

NOTE   Vacuum pressure is not measured and not required regular verification.

Figure G.1—Example of De-airing Mixing Container

G.3.3   Mixing Procedure

G.3.3.1   General

Mixing procedures shall be the same as outlined in Section 5 and Annex A, except as described below.

G.3.3.2   Standard volume mixing

Standard mixing procedure, see 5.3.6, after addition of cement or dry cement/dry additive(s) blend at a uniform rate in no more than 15 s while mixing at 4000 r/min ±250 r/min, shall be replaced by the following.

a)   After 15 s or when all the dry materials have been added to the mix water if longer than 15 s, place the cover on the mixing container and apply vacuum.

   The time used to add the cement blend shall be kept to a minimum. If more than 15 s were required to add the cement blend to the water, document that time.

b)   Continue mixing at 12,000 r/min ± 500 r/min for 35 s ± 1 s. Measure and document the rotational speed under load.

c)   Reduce the mixing velocity to 1000 r/min ± 100 r/min for an additional 2 min ±5 s.

d)   Release vacuum and proceed to cement slurry conditioning and testing.

G.3.3.2   Large volume mixing

Large volume mixing procedure, see A.3.3, after addition of cement or dry cement/dry additive(s) blend at a uniform rate in no more than 35 s while mixing at 4000 r/min ±250 r/min, shall be replaced by the following:

a) After 35 s or when all the dry materials have been added to the mix water if longer than 35 s, place the cover on the mixing container and apply vacuum.

The time used to add the cement blend shall be kept to a minimum. If more than 35 s were required to add the cement blend to the water, document that time.

b) Continue mixing at 10,000 r/min, $\pm$ 500 r/min, for 50 s, $\pm$ 1 s.

c) Reduce the mixing velocity to 1000 r/min $\pm$ 100 r/min for an additional 2 min $\pm$5 s.

d) Release vacuum and proceed to cement slurry conditioning and testing.

NOTE   Data generated from the use of a large volume (3000 mL) mix may vary from the 600 mL mixing data and is only recommended when large volumes of slurry are required.

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