Recommended Practice for Testing Well Cements

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Introduction

Users of this standard should be aware that further or differing requirements may be needed for individual applications. This standard is not intended to inhibit a vendor from offering, or the purchaser from accepting, alternative equipment or engineering solutions for the individual application. This may be particularly applicable where there is innovative or developing technology. Where an alternative is offered, the vendor should identify any variations from this standard and provide details.

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

In this standard, calibrating an instrument refers to assuring the accuracy of the measurement. Accuracy is the degree of conformity of a measurement of a quantity to its actual or true value. Accuracy is related to precision, or reproducibility of a measurement. Precision is the degree to which further measurements or calculations will show the same or similar results. Precision is characterized in terms of the standard deviation of the measurement. The results of calculations or a measurement can be accurate, but not precise, precise but not accurate, neither and both. A result is valid if it is both accurate and precise.

Well cement classes and grades are defined in API Specification 10A.

Warning—The tests specified in this standard require the handling of hot, pressurized equipment and materials that may be hazardous and can cause injury. Do not exceed manufacturer's safety limits. Only trained personnel should perform these tests.

Recommended Practice for 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 13B-1, *Recommended Practice for Field Testing Water-based Drilling Fluids*

API Recommended Practice 13B-2, *Recommended Practice for Field Testing Oil-based Drilling Fluids*

API Recommended Practice 13J, *Testing of Heavy Brines*

ASTM C109/C109M-07 [1], *Standard Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2 in. or [50 mm] Cube Specimens)*

ASTM C188-95, *Standard Test Method for Density of Hydraulic Cement*

ASTM C618-08, *Standard Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use in Concrete*

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
absolute density
Density of a material without the fluid around the particles, 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).

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

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

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

3.1.4
annulus

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

3.1.5
batch mix

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

3.1.6
Bearden units of consistency

B_c

Units used to express consistency of a cement slurry when determined using a pressurized consistometer.

3.1.7
bottomhole circulating temperature

T_{BHC}

Pseudo-stable temperature found at the bottom of the wellbore while the well is being circulated.

3.1.8
bulk density

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

3.1.9
casing cementing

Process of placing cement to fill or partially fill the space between a full string of casing and the wellbore.

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.

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 10A.

3.1.13
cement grade

Designation for denoting the sulfate resistance of a particular cement according to specifications of API 10A.

3.1.14
clinker

Fused materials from the kiln that are interground with calcium sulfate to manufacture cement.

3.1.15
compatibility

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

3.1.16
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.

3.1.17
consistometer
Device used to measure the thickening time of a cement slurry under temperature and pressure.

NOTE An atmospheric consistometer can be used to condition fluids prior to testing and for determining the thickening time of arctic slurries.

3.1.18
continuous-pumping squeeze-cementing operation
Squeeze-cementing operation that does not involve interruptions in pumping.

3.1.19
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.

3.1.20
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) of Portland cement.

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

3.1.21
filtrate
Liquid that is forced out of a cement slurry during a fluid-loss test.

3.1.22
fly ash
Finely divided residue from the combustion of ground or powdered coal with pozzolanic properties.

NOTE See Section 14 for further description.

3.1.23
free fluid
Colored or colorless liquid that has separated from a cement slurry when standing in a static condition.

3.1.24
freeze-thaw cycle
A prescribed cycle of exposure of a cement sample to temperatures above and below the freezing point of water.

3.1.25
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}.

3.1.26
hesitation-squeeze-cementing operation

Method of placement of cement during squeeze-cementing operation that involves alternate pumping and nonpumping periods.

3.1.27
liner cementing

Annular cementing operations for which the top of the casing being cemented does not reach the surface or subsea wellhead.

3.1.28
load frame

Frame to place a load on 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.

3.1.29
mix fluid

Mix water containing solid or liquid (or both) additives.

3.1.30
mix water

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

3.1.31
nonaqueous fluid
NAF

NAF is a nonaqueous 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.

3.1.32
plug cementing

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

3.1.33
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.

3.1.34
preflush

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

3.1.35
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.

3.1.36
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.

3.1.37
pressurized curing chamber
Apparatus used for curing a sample of cement under temperature and pressure for subsequent tests such as compressive strength, sedimentation, etc.

3.1.38
pressurized fluid density balance
Beam-type balance used to measure fluid density under pressure to minimize the effects of air entrained in the fluid.

3.1.39
relative density
specific gravity
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.

3.1.40
sack
A unit of measure of cement that occupies a bulk volume of one cubic foot (normally 94 lbm for Portland cement).

NOTE Only used in USC measurements.

3.1.41
sedimentation
Separation and settling of solids in a cement slurry.

3.1.42
slurry container
Container used to hold the slurry in a consistometer for conditioning or testing.

3.1.43
slurry stability test
Test to determine the degree of sedimentation and/or free fluid development in a cement slurry.

3.1.44
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.

3.1.45
spacer
Viscosified fluid that may be densified with insoluble, solid weighting agents and are used to separate drilling fluids and cement slurries.

3.1.46
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.

3.1.47
starting pressure
p_S
Initial pressure applied to the cement slurry at the beginning of the thickening-time test.

3.1.48
static fluid-loss test
Test to determine filtrate lost from a cement slurry when placed against a 325 mesh screen at 6900 kPa (1000 psi) differential pressure.

3.1.49
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.

3.1.50
thickening time
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).

NOTE The results of a thickening-time test provide an indication of the length of time a cement slurry can remain pumpable under the test conditions.

3.1.51
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. Transit time is typically expressed in microseconds per inch.

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

3.1.52
ton
Term used to express a two thousand (2000) pound mass.

3.1.53
tonne
metric ton
Term used to express a one thousand (1000) kilogram mass.

3.1.54
weigh batch mixer
scale tank
Device or system for the weighing and blending of cement with dry additives for a cementing operation.

3.1.55
well-simulation test
Test whose parameters are designed and modified as required to simulate the conditions found in a wellbore.

3.2 Symbols

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

Table 1—Symbols

Symbol	Meaning
B_{c}	consistency expressed in Bearden units
$C_{\mathrm{ad \cdot sol}}$	solid additive concentration
d_{rel}	relative density
h_{TOCTVD}	top-of-cement true vertical depth
h_{TVD}	true vertical depth
k	consistency index
m	mass
$m_{\mathrm{ad.liq}}$	liquid additive mass
$m_{\mathrm{ad.sol}}$	solid additive mass
m_{air}	cement segment weight in air
m_{c}	cement mass
m_{lfm}	mass of loosely filled material
m_{s}	slurry mass
m_{w}	mix water mass
m_{water}	cement segment weight in water
n	flow behavior index
p_{BH}	bottomhole pressure [a]
p_{FSQ}	final bottomhole squeeze pressure
p_{S}	starting pressure
p_{SQ}	maximum surface squeeze pressure
p_{TOC}	top-of-cement pressure
q	pump rate
$R_{\Delta \mathrm{T}}$	rate of temperature change (heat-up or cool-down)
R_{pd}	rate of pressure decrease
R_{pu}	rate of pressure increase
R_{PSQ}	rate of pressure increase to apply the squeeze pressure
T	torque
T_{BHC}	bottomhole circulating temperature
T_{BHS}	bottomhole static temperature
T_{PBHC}	predicted bottomhole circulating temperature
T_{PSP}	predicted bottomhole squeeze or plug temperature
T_{SS}	slurry surface temperature
T_{TOCC}	top-of-cement circulating temperature
T_{TOCS}	top-of-cement static temperature
TT	transit time (sonic slowness)

Symbol	Meaning
t	elapsed time
t_{a}	time to displace the leading edge of the cement slurry from bottom of the casing to the top of the annular column
t_{d}	time to displace the leading edge of cement slurry from the surface to the bottom of the wellbore
t_{sq}	time to apply squeeze pressure to cement slurry after bottomhole placement
V_{30}	volume of filtrate collected at 30 min
V_{a}	annular volume of cement
$V_{\mathrm{ad.liq}}$	liquid additive volume
$V_{\mathrm{ad.sol}}$	solid additive volume
V_{F}	volume of free fluid
V_{fp}	final packed volume
V_{p}	volume of the pipe
V_{s}	slurry yield
V_{t}	volume of filtrate collected at the time nitrogen blows through
V_{w}	mix water volume
Z	acoustic impedance
γ	shear rate
Δ_{PT}	pseudo-temperature gradient [b]
$\Delta\rho_{\mathrm{sc}}$	density of the set cement relative to the cement slurry
μ_{p}	plastic viscosity
$\rho_{\mathrm{ad.liq}}$	density of liquid additive
$\rho_{\mathrm{ad.sol}}$	density of solid additive
ρ_{af}	density of annular fluid
ρ_{bulk}	average bulk density
ρ_{c}	density of cement
ρ_{df}	density of drilling fluid
ρ_{LAB}	loose apparent bulk density
ρ_{PAB}	packed apparent bulk density
ρ_{s}	density of slurry
ρ_{set}	density of the set cement
ρ_{w}	density of mix water
τ	shear stress
τ_{y}	yield stress
φ	fluid volume fraction
$\upsilon_{\mathrm{ad.abs}}$	solid additive absolute volume
$\upsilon_{\mathrm{c.abs}}$	cement absolute volume

[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).

4 Sampling

4.1 General

Samples of the cement, cement blend, solid and liquid additives, and mixing water may be required to test a 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 should be extracted using a suitable device (Figure 1). A composite of the samples should be prepared, packaged, and labeled (see 4.7). Sample volume should 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 should be thoroughly blended prior to sampling. Samples from the bulk container may be extracted in accordance with 4.2. Samples extracted from a flow line during a transfer may be taken from a properly installed sample valve, diverted flow sampler, or automatic in-line sampling device [Figure 1 c), Figure 1 d), and Figure 1 e)] using a procedure designed to give a representative sample of the blend being transferred. The samples should be prepared, packaged, and labeled (4.7). Sample volume should be sufficient to perform the desired testing.

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 Figure 1 b)]. A composite of the samples from the same lot should be prepared, packaged, and labeled (4.7). The volume of each dry cement additive sample should 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 should be thoroughly mixed prior to sampling. The sample should then be extracted from the center of the container using a clean, dry sampling device. A composite of the samples from the same lot should be prepared, packaged, and labeled (4.7). The volume of each liquid additive sample should be sufficient to perform the desired testing.

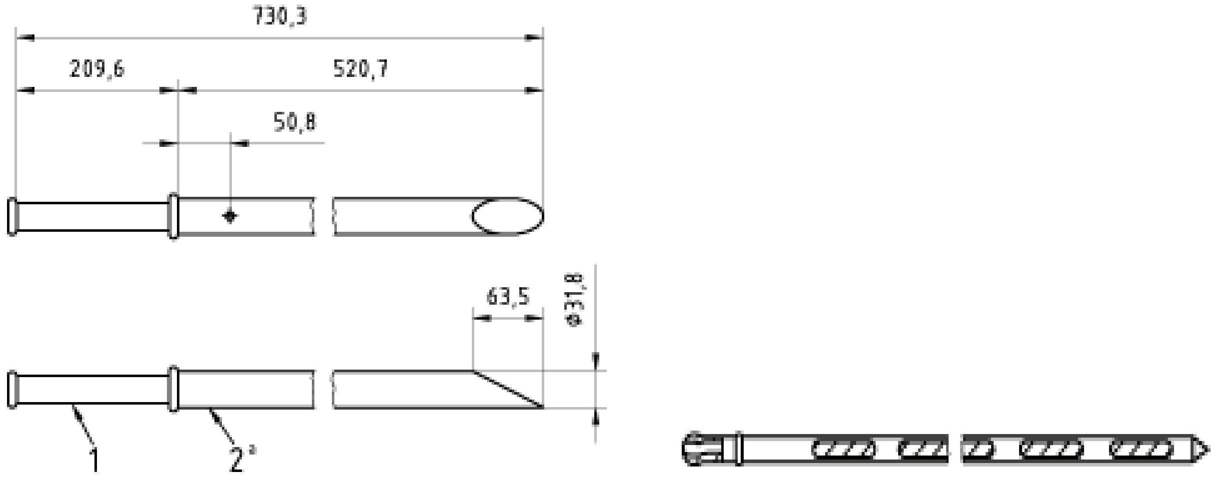
4.6 Sampling Mixing Water

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

4.7 Shipping and Storage

Test samples should be packaged promptly in unused, clean, airtight, moisture-proof containers suitable for shipping and long-term storage. The containers should be lined metal, plastic, or other heavy-walled flexible or rigid 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 should not be used. Shipping in glass containers is not recommended.

Dimensions in millimeters

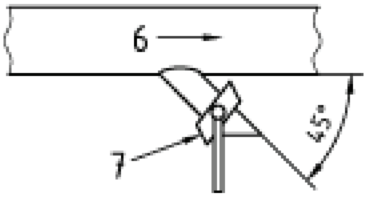


a) Tube Sampler for Sacked Cement

b) Tube Sampler for Bulk Cement

c) Automatic Probe Sampler

d) Modified Diverted-flow Sampler



e) Top View—Lateral Sampler

Key

1 handle
2 dragg tubing
3 sample tube, extended
4 product discharge
5 valve
6 flow direction
7 valve

[a] Approximate volume = 320 ml.

Figure 1—Commonly Used Sampling Devices

The body of each blend container should be clearly labeled and identified with the type of material, lot number, source, and date of sampling. Shipping containers should also be labeled. The lids of containers should not be marked, since the lids can be readily interchanged and thus lead to confusion. Any required regulatory identification or documentation should be enclosed or securely attached to the container. All hazardous material samples should 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 should 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 should be transferred into a suitable, leak-proof container. The container should be properly labeled, dated, and stored in a dry place where room temperatures remain fairly constant. Repackaging should be indicated. At the time of testing, each sample should be examined for quality and thoroughly blended just prior to slurry preparation (see Section 5).

Optimum shelf life(s) for all samples should 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 should 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.

5.2 Apparatus

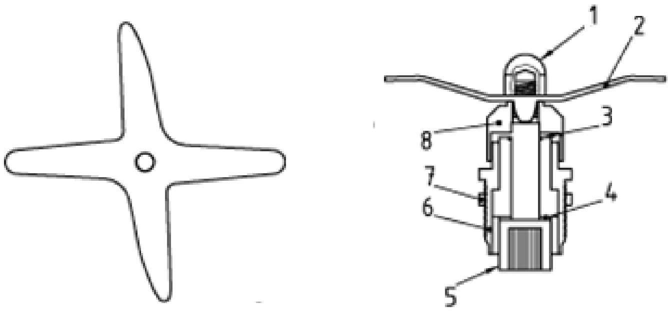
5.2.1 All apparatuses are calibrated as per Annex B.

5.2.2 Electronic or mechanical balances—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 ± 1 % of reading for measurement made less than 10 g. Balances shall have two (2) decimal place precision at a minimum.

5.2.3 Mixing device, of capacity 1 liter (1 quart), having bottom drive and a blade-type blender—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) should be constructed of corrosion-resistant material. The mixing blender assembly should be constructed so that the blade can be separated from the drive mechanism.

The mixing blender blade should be replaced with a new blade before mass loss greater than 10 % has occurred (see B.3.2.2). If the mixing device leaks at any time during the mixing procedure, the contents should be discarded, the leak repaired, and the procedure restarted.

Figure 2—Example of Common Mixing Device



Key

1	cap nut	5	socket head shaft
2	blade (installed with tapered edge down)	6	bearing holder
3	O-ring	7	hexagonal nut
4	thrust washer	8	bearing cap

Figure 3—Common Blade Assembly

5.3 Procedure

5.3.1 Determination of Relative Density (Specific Gravity) of Components

5.3.1.1 General

The relative 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 relative density may vary from 3.10 to 3.25. 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 relative density of mix water can also vary, depending on the source, resulting in slurry density inconsistencies. Determination of the relative density of all components of the slurry is necessary to properly calculate the required amounts for slurry preparation.

5.3.1.2 Relative Density of Cement and Dry Additives

The relative 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 C188-95 may be used for determining the relative density of these materials.

5.3.1.3 Relative Density of Mix Water and Liquid Additives

The 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 relative density of these materials.

5.3.1.4 Laboratory Density and Volume Calculations

5.3.1.4.1 General

For the purpose of these calculations, as a general rule one cubic centimeter is equivalent to one milliliter. Fresh water density at 23 °C (73 °F) is 0.9976 kg/l (8.325 lbm/gal).

A slurry volume of approximately 600 ml shall be sufficient to perform most laboratory test procedures while not overfilling the mixing container. Laboratory blend requirements may be calculated by use of the formulas based on slurry design recipes [Equations (21) to (25) in SI units or Equations (27) to (31) in USC units] under 5.3.1.4.4. Alternative, suitable equations may also be used to calculate laboratory blend requirements.

Calculations [Equations (1) to (8) in SI units or Equations (9) to (15) in USC units] are based on a slurry design that includes solid additives or liquid additives or both. Slurry composition is based on additive concentrations expressed for the solids in kilograms per tonne of cement (metric ton, 1 tonne = 1000 kg) or in percent by weight of cement (BWOC) and for the liquids in liters per tonne of cement (gallon par sack of cement). When water requirement is provided, the slurry yield (V_s) can be easily calculated based on Equation (2) (SI units) or Equation (10) (USC units). If it is not the case the first step should be to calculate water requirement and then slurry yield.

5.3.1.4.2 Slurry Design Calculation in SI Units

$$\rho_\mathrm{s} = \frac{m_\mathrm{s}}{V_\mathrm{s}} \tag{1}$$

$$V_\mathrm{s} = V_\mathrm{c} + V_\mathrm{w} + \Sigma\ V_\mathrm{ad.liq} + \Sigma\ V_\mathrm{ad.sol} \tag{2}$$

$$m_{\mathrm{s}} = m_{\mathrm{c}} + m_{\mathrm{w}} + \sum m_{\mathrm{ad.liq}} + \sum m_{\mathrm{ad.sol}} \tag{3}$$

where

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

V_{s} the slurry yield prepared with one tonne (1000 kg) of cement, in liters per tonne of cement;

V_{c} is the cement volume of one tonne (1000 kg) of cement, expressed in liters;

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

$\Sigma\, V_{\mathrm{ad \cdot liq}}$ is the sum of volumes for all liquid additives, expressed in liters per tonne of cement;

$\Sigma\, V_{\mathrm{ad \cdot sol}}$ is the sum of volumes for all solid additives, expressed in liters per tonne of cement;

m_{s} is the slurry mass prepared with one tonne (1000 kg) of cement, expressed in kilograms per tonne of cement;

m_{c} is the cement mass of one tonne (1000 kg) of cement, expressed in kilograms;

m_{w} is the mix water mass, expressed in kilograms per tonne of cement;

$\Sigma\, m_{\mathrm{ad \cdot liq}}$ is the sum of masses for all liquid additives, expressed in liters per tonne of cement;

$\Sigma\, m_{\mathrm{ad \cdot sol}}$ is the sum of masses for all solid additives, expressed in liters per tonne of cement.

Solid additives can be also expressed by the solid additive concentration, $C_{\mathrm{ad \cdot sol}}$ in percent BWOC. In that case:

$$m_{\mathrm{ad.sol}} = 10 \times C_{\mathrm{ad.sol}} \tag{4}$$

Volumes are related to masses, and masses to volumes, for cement:

$$V_{\mathrm{c}} = 1000 / \rho_{\mathrm{c}} \tag{5}$$

where

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

for mix water:

$$m_{\mathrm{w}} = V_{\mathrm{w}} \times \rho_{\mathrm{w}} \tag{6}$$

where

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

and for cement additives:

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

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

where

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

5.3.1.4.3 Slurry Design Calculation in USC Units

$$\rho_{\text{s}} = 0.133\,7 \times \frac{m_{\text{s}}}{V_{\text{s}}} \tag{9}$$

$$V_{\text{s}} = 0.133\,7 \times \left(V_{\text{c}} + V_{\text{w}} + \Sigma V_{\text{ad.liq}} + \Sigma V_{\text{ad.sol}} \right) \tag{10}$$

$$m_{\text{s}} = m_{\text{c}} + m_{\text{w}} + \sum m_{\text{ad.liq}} + \sum m_{\text{ad.sol}} \tag{11}$$

where

ρ_{s}	is the density of slurry, in pounds per gallon;
V_{s}	the slurry yield prepared with one sack of cement (94 lbm), in cubic feet per sack;
V_{c}	is the cement volume of one sack of cement, expressed in gallons;
V_{w}	is the mix water volume, expressed in gallons per sack of cement;
$V_{\text{ad·liq}}$	is the volume of a liquid additive, expressed in gallons per sack of cement, all liquid additives should be considered by the calculation;
$V_{\text{ad.sol}}$	is the volume of a solid additive, expressed in gallons per sack of cement, all solid additives should be considered by the calculation;
m_{s}	is the slurry mass prepared with one sack of cement (including water and additives), expressed in pounds per sack of cement;
m_{c}	is the cement mass of one sack of cement, taken to be 94 lbm;
m_{w}	is the mix water mass, expressed in pounds per sack of cement;
$m_{\text{ad.liq}}$	is the mass of a liquid additive, expressed in pounds per sack of cement, all liquid additives should be considered by the calculation;
$m_{\text{ad.sol}}$	is the mass of a solid additive for one sack of cement based on its concentration, $C_{\text{ad·sol}}$, expressed in percent BWOC, all solid additives should be considered by the calculation.

where

$$m_{\text{ad.sol}} = 0.01 \times m_{\text{c}} \times C_{\text{ad.sol}} = 0.94 \times C_{\text{ad.sol}}$$

Volumes are related to masses, and masses to volumes, for cement:

$$V_{\text{c}} = 94 \times v_{\text{c.abs}} \tag{12}$$

where

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

for mix water:

$$m_{\text{w}} = V_{\text{w}} \times \rho_{\text{w}} \tag{13}$$

where

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

and for cement additives:

$$m_{\text{ad.liq}} = V_{\text{ad.liq}} \times \rho_{\text{ad.liq}} \tag{14}$$

$$V_{\text{sol.ad}} = 0.01 \times m_{\text{c}} \times C_{\text{ad.sol}} \times v_{\text{c.abs}} = 0.94 \times C_{\text{ad.sol}} \times v_{\text{c.abs}} \tag{15}$$

where

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

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

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

Remarks:

a) USC equations are based on a sack of cement. If a sack is defined as one bulk cubic foot (3.1.40), a cement sack weight would be 94 lb. For a cement blend, sack weight should be defined by the user and in that case, Equation (12) shall be modified accordingly.

b) Salt as an additive is defined in percent by weight of water (BWOW). For testing purposes, it is recommended to consider mixing water as brine and to calculate m_{w} from brine density and brine volume requirements. An alternate method to calculate the salt in the slurry is to use a graph or table containing an equivalent specific gravity for the actual salt when it is in solution. Using this method, salt can be treated as any other additive as long as the concentration is BWOW.

5.3.1.4.4 Water Requirement Calculations

In SI units, mix water requirement is calculated by solving Equation (1) using Equations (2) to (8), mix water requirement is calculated by Equation (16).

$$V_{\text{w}} = \frac{1000\left(1-\rho_{\text{s}}/\rho_{\text{c}}\right)-\Sigma\left[V_{\text{ad.liq}}\left(\rho_{\text{s}}-\rho_{\text{ad.liq}}\right)\right]+\Sigma\left[m_{\text{ad.sol}}\left(1-\rho_{\text{s}}/\rho_{\text{ad.sol}}\right)\right]}{\left(\rho_{\text{s}}-\rho_{\text{w}}\right)} \tag{16}$$

which is resumed with fresh water at 23 °C (73 °F) to:

$$V_{\text{w}} = \frac{1000\left(1-\rho_{\text{s}}/\rho_{\text{c}}\right)-\Sigma\left[V_{\text{ad.liq}}\left(\rho_{\text{s}}-\rho_{\text{ad.liq}}\right)\right]+\Sigma\left[m_{\text{ad.sol}}\left(1-\rho_{\text{s}}/\rho_{\text{ad.sol}}\right)\right]}{\left(\rho_{\text{s}}-0.9976\right)} \tag{17}$$

where

V_{w} is the mix water volume, expressed in liters per tonne of cement (1000 kg);

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

$m_{\text{ad.sol}}$ is the mass of a solid additive, expressed in liters per tonne of cement (1000 kg) or calculated by Equation (4) and solid additive concentration, $C_{\text{ad}\cdot\text{sol}}$; all solid additives should 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;

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

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

In USC units, mix water requirement is calculated solving Equation (9) using Equations (10) to (15), by Equation (18).

$$V_{\text{w}} = \frac{94\left(1-\rho_{\text{s}}\times v_{\text{c.abs}}\right)-\Sigma\left[V_{\text{ad.liq}}\left(\rho_{\text{s}}-\rho_{\text{ad.liq}}\right)\right]+0.94\times\Sigma\left[C_{\text{ad.sol}}\left(1-\rho_{\text{s}}\times v_{\text{ad.abs}}\right)\right]}{\left(\rho_{\text{s}}-\rho_{\text{w}}\right)} \tag{18}$$

which is resumed with fresh water to:

$$V_{\text{w}} = \frac{94\left(1-\rho_{\text{s}}\times v_{\text{c.abs}}\right)-\Sigma\left[V_{\text{ad.liq}}\left(\rho_{\text{s}}-\rho_{\text{ad.liq}}\right)\right]+0.94\times\Sigma\left[C_{\text{ad.sol}}\left(1-\rho_{\text{s}}\times v_{\text{ad.abs}}\right)\right]}{\left(\rho_{\text{s}}-8.325\right)} \tag{19}$$

where

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

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

$v_{\text{c.abs}}$	is the absolute volume of cement, expressed in gallons per pound;
$V_{\text{ad.liq}}$	is the volume of liquid additive, expressed in gallons per sack of cement, all liquid additives should be considered by the calculation;
$\rho_{\text{ad.liq}}$	is the density of liquid additive, expressed in pounds per gallon;
$C_{\text{ad.sol}}$	is the concentration of solid additive, expressed in percent BWOC, all solid additives should be considered by the calculation;
$v_{\text{ad. abs}}$	is the absolute volume of solid additive, expressed in gallon per pounds;
ρ_{w}	is the density of mix water, expressed in pounds per gallon.

5.3.1.4.5 Slurry Yield Calculations

Slurry yield (V_{s}) expressed in liters per tonne of cement (cubic feet per sack) are calculated with Equation (2) and Equation (10) in USC units.

5.3.1.4.6 Laboratory Mass Calculations

A slurry volume of 600 ml should be mixed based on mix water, cement, and additives mass requirements expressed in grams. Mass requirements for 600 ml are calculated based on Equations (20), (1), and (3):

$$m_{\text{s,600}} = m_{\text{c,600}} + m_{\text{w,600}} + \sum m_{\text{ad.liq,600}} + \sum m_{\text{ad.sol,600}} \tag{20}$$

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;
$m_{\text{w,600}}$	is the mix water mass to be mixed for 600 ml of slurry, expressed in grams;
$\Sigma\, m_{\text{ad.liq,600}}$	is the sum of masses for all liquid additives to be mixed for 600 ml of slurry, expressed in grams;
$\Sigma\, m_{\text{ad.sol,600}}$	is the sum of masses for all solid additives masses to be mixed for 600 ml of slurry, expressed in grams.

For SI units as per slurry composition and yield calculation mass requirements are:

Mass of cement ($m_{\text{c,600}}$) to be mixed:

$$m_{\text{c,600}} = \frac{600}{V_{\text{s}}} \times 1000 \tag{21}$$

into the mass of mix water ($m_{\text{w,600}}$)

$$m_{\mathrm{w},600} = \frac{600}{V_\mathrm{s}} \times V_\mathrm{w} \times \rho_\mathrm{w} \tag{22}$$

with fresh water at 23 °C (density 0.9976 kg/l):

$$m_{\mathrm{w},600} = \frac{598.56}{V_\mathrm{s}} \times V_\mathrm{w} \tag{23}$$

and with the masses of liquid additives ($m_{\mathrm{ad.liq},600}$) and solids additives ($m_{\mathrm{ad.sol},600}$):

$$m_{\mathrm{ad.liq},600} = \frac{600}{V_\mathrm{s}} \times V_{\mathrm{ad.liq}} \times \rho_{\mathrm{ad.liq}} \tag{24}$$

$$m_{\mathrm{ad.sol},600} = \frac{600}{V_\mathrm{s}} \times m_{\mathrm{ad.sol}} \tag{25}$$

where

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

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

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

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

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

$m_{\mathrm{ad.sol}}$ is the mass for a solid additive per one tonne of cement (1000 kg), expressed in kilograms per tonne of cement.

or with

$$m_{\mathrm{ad.sol}} = 10 \times C_{\mathrm{ad.sol}} \tag{4}$$

where

$C_{\mathrm{ad.sol}}$ is the concentration of the solid additive, expressed in percent BWOC.

For USC units as per slurry composition and yield calculation, to mix a 600 ml sample, the mass requirements expressed in grams are calculated by the following equations (based on a 94 lbm sack of cement).

Mass of cement ($m_{\mathrm{c},600}$) to be mixed:

$$m_{\mathrm{c},600} = \frac{9.611}{V_\mathrm{s}} \times 94 = \frac{903.4}{V_\mathrm{s}} \tag{26}$$

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

$$m_{\mathrm{w,600}} = \frac{9.611}{V_{\mathrm{s}}} \times V_{\mathrm{w}} \times \rho_{\mathrm{w}} \tag{27}$$

with fresh water at 73 °F (density 8.325 lb/gal):

$$m_{\mathrm{w,600}} = \frac{80.01}{V_{\mathrm{s}}} \times V_{\mathrm{w}} \tag{28}$$

and with the masses of liquid additives ($m_{\mathrm{ad.liq,600}}$) and solids additives ($m_{\mathrm{ad.sol,600}}$):

$$m_{\mathrm{ad.liq,\,600}} = \frac{9.611}{V_{\mathrm{s}}} \times V_{\mathrm{ad.liq}} \times \rho_{\mathrm{ad.liq}} \tag{29}$$

$$m_{\mathrm{ad.sol,600}} = \frac{0.09611}{V_{\mathrm{s}}} \times 94 \times C_{\mathrm{ad.sol}} = \frac{9.034}{V_{\mathrm{s}}} \times c_{\mathrm{ad.sol}} \tag{30}$$

where

V_{s}	is the slurry yield prepared with one sack of cement (94 lbm), expressed in cubic feet per one sack of cement;
V_{w}	is the mix water volume, expressed in gallons per one sack of cement;
ρ_{w}	is the density of mix water, expressed in pounds per gallon;
$V_{\mathrm{ad.liq}}$	is the volume for a liquid additive per one sack of cement, expressed in gallons per one sack of cement;
$\rho_{\mathrm{ad.liq}}$	is the density of a liquid additive, expressed in pounds per gallon;
$C_{\mathrm{ad.sol}}$	is the concentration of the solid additive, expressed in percent BWOC.

5.3.2 Temperature of Water and Cement

The temperature of the mix water, dry cement or cement blend, and mixing and blending devices should be representative of field mixing conditions. If field conditions are unknown, the temperature of the mix water (including any premixed additives) and dry cement should 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 should be measured and documented.

5.3.3 Mix Water and Mix Fluid

Water composition can affect slurry performance. Water from the field source should be used. If field mix water is unavailable, water of similar composition should be used. If field mix water composition is unknown, deionized, distilled, or tap water may be used. The type of mix water should be documented in the lab report. The mix water should 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. No excess water should be added to compensate for evaporation or wetting.

5.3.4 Mixing of Cement and Water

A slurry volume of approximately 600 ml is sufficient to perform most laboratory test procedures while not overfilling the blender container. Weigh dry materials and then blend thoroughly and uniformly prior to adding them to the mix fluid. Place the blender container with the required mass of mix water and liquid additives (if previously added) on the blender base. 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 should be added to the mix water while mixing at low speed. In certain cases, the order of addition of the additives to the mix water can be critical, in which case, the additives should be mixed in the order that they will be mixed in the field. Document any special mixing procedures and mixing time.

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 should be kept to a minimum. If more than 15 s were required to add the cement blend to the water, document that time. 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 ± 250 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 should 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 should use the temperatures and pressures of the well. Alternatively, the conditioning can be done at atmospheric pressure.

5.4.2 Procedure: Pressurized Conditioning

Any consistometer referenced in Section 9 or Annex C may be used.

a) Within 1 min after mixing according to 5.3.4, pour the test fluid into the slurry container of a pressurized consistometer. Start test within 5 min after placing test fluid in the pressurized consistometer.

b) Heat 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.

c) 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 test location 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 psi/s)].

d) 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.

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

f) 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.

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

h) Continue with the desired test.

5.4.3 Procedure: Atmospheric-pressure Conditioning

This procedure is limited to a maximum temperature of 88 °C (190 °F). If the boiling point of water at the test location is less than 100 °C (212 °F), adjust conditioning temperatures accordingly.

a) Within 1 min after mixing according to 5.3.4 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 should 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 should 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 should 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 is the pressurized fluid density balance. 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 is calibrated according to the requirements found in Annex B. The apparatus should be clean and dry.

6.2 Procedure

6.2.1 The sample cup should be filled to a level slightly below the upper edge of the cup.

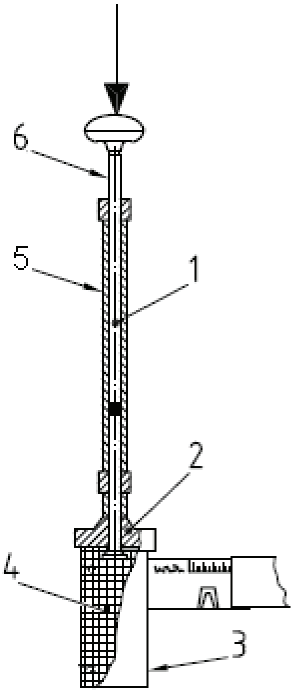
6.2.2 Place the lid on the cup with the check valve in the down (open) position. Push the lid downward into the mouth of the cup until surface contact is made between the outer skirt of the lid and the upper edge of the cup. Expel excess slurry through the check valve.

Caution—Slurry can be expelled forcibly.

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.

6.2.3 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.

6.2.4 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 4).



Key

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

Figure 4—Common Pressurized Fluid Density Balance

6.2.5 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.

6.2.6 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 one of the four calibrated scales on the arrow side of the sliding weight.

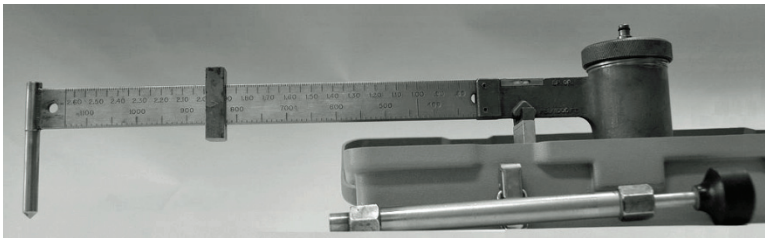


Figure 5—Common Pressurized Fluid Density Balance

6.2.7 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.

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 Cube molds and compressive-strength testing load frame should conform with ASTM C109/C109M-07, except for: (1) the bearing block surface dimension requirement, (2) the bearing block Rockwell hardness requirement, and (3) that molds can be separable into more than two parts.

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

The molds should be made of hard metal and mold tolerance should be verified at least once every two years. The cube mold base plate should be of corrosion-resistant metal; the cover plate should have a minimum thickness of 6 mm (0.25 in.) and be made of corrosion-resistant material. Grooves may be incorporated into the surface of the cover plate contacting the surface of the cement. Glass plates may be used but are not recommended for tests above 110 °C (230 °F) because of the risk of silica replacement

7.3.2 Water curing 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).

The two types of water curing baths are:

a) **atmospheric-pressure curing bath** (unpressurized), 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.

b) **pressurized curing chamber**, suitable for curing samples at the appropriate final test temperature and a pressure of at least 21 MPa (3000 psi). The vessel should be capable of being heated at the desired rate.

7.3.3 Cooling 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).

7.3.4 Temperature-measuring system, three commonly used temperature-measuring systems are as follows:

a) A thermometer, of range – 18 °C to 104 °C (0 °F to 220 °F), with minimum scale divisions not to exceed 1 °C (2 °F) may be used in a nonpressurized bath.

b) A thermocouple, of range – 18 °C to 104 °C (0 °F to 220 °F), accurate to ± 2 °C (± 3 °F) is preferred in a nonpressurized bath.

c) A thermocouple, of range – 18 °C to at least 204 °C (0 °F to at least 400 °F), accurate to ± 2 °C (± 3 °F), should be used in a pressurized curing chamber.

7.3.5 Puddling rod, corrosion-resistant, typically with a diameter of 6 mm (0.25 in.).

7.3.6 Mold-sealing grease, possessing the following properties when subjected to anticipated test temperatures and pressures is suitable for use:

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) noncorrosive in the temperature range of the test.

7.3.7 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 should comply with 7.3.6.

7.4 Procedure

7.4.1 General

Apparatuses are calibrated according to the requirements in Annex B.

7.4.2 Preparation of Molds

The interior faces of the molds and the contact surfaces of the plates are commonly coated with mold release agent, but may be clean and dry. Care should be taken to ensure there is no bead of sealant on the interior of the mold. See Figure 6.

7.4.3 Preparation of Slurry and Filling of Molds

7.4.3.1 Preparation

Prepare the cement slurry in accordance with 5.3. If preconditioning is required, then either 5.4 or 7.5 should be followed. Section 7.5 is particularly useful to determine the compressive strength at the top of long cement columns.

7.4.3.2 Mold Filling

Pour the cement slurry into each chamber of the prepared molds to approximately one-half of the mold depth. Puddle each sample sufficiently with a puddling rod to remove air bubbles. Stir the remaining slurry with a spatula to ensure the slurry is uniform. Fill each mold chamber to overflowing with slurry and puddle the same as the first layer. After puddling, strike off the excess slurry even with the top of the mold using a straight edge. Discard specimens in molds that leak. Place the cover plate on top of the molds. For each test, prepare at least three specimens.

7.4.4 Curing at Atmospheric Pressure

After the molds have been filled and covered and within 5 min after mixing, place them in a curing bath maintained at the desired curing temperature. 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. At 45 min (± 5 min) prior to the age at which the samples are to be tested, remove the molds from the water bath and remove the cured samples from the molds. Immediately immerse the samples in a water cooling bath at 27 °C ± 3 °C (80 °F ± 5 °F) until the samples are tested.

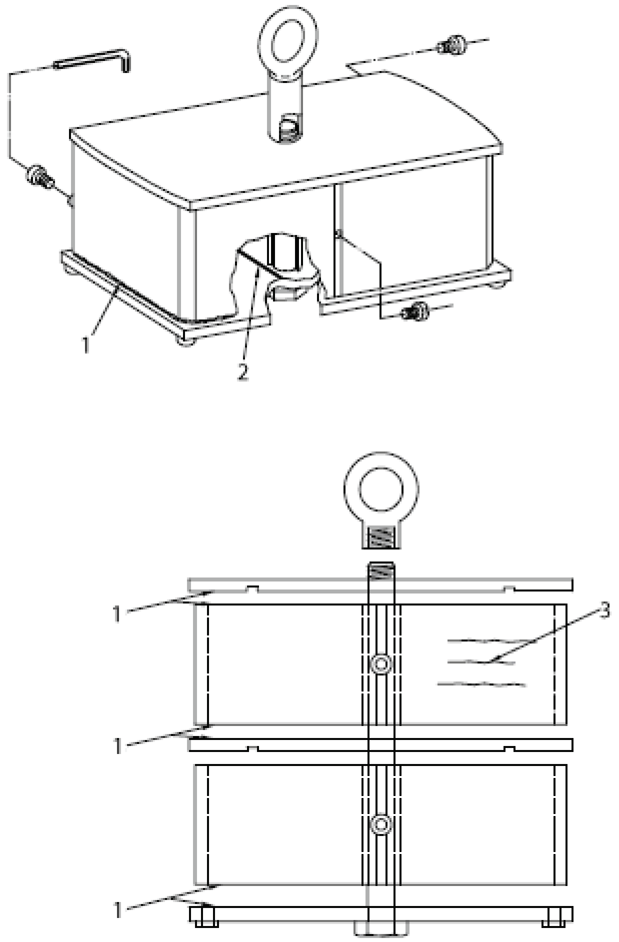
7.4.5 Curing at Pressures Above Atmospheric

Within 5 min after the last slurry mixing, the molds have been filled and covered with the top plate, and immediately placed in a curing vessel at the desired test initiation temperature [normally 27 °C ± 3 °C (80 °F ± 5 °F)], heat and pressure in accordance with the test schedule is applied. Cement samples may be cured in accordance with pressure/temperature schedules provided in Table 2 and Table 3 (see below) 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 must be used. Schedules should be interpolated to arrive at the correct depth/static temperature of the well for which the test is performed.

For samples cured at or below 88 °C (190 °F), maintain test temperature and pressure until 45 min (± 5 min) prior to testing. For test temperatures above 90 °C (194 °F), discontinue heating and allow samples to cool at such a rate that the sample temperature is 90 °C (194 °F) or less 45 min prior to testing. Maintain test pressure on the curing vessel during the cooling process. Release the pressure gradually and remove the molds from the curing vessel. Immediately remove the samples from the molds and immerse them in a water cooling bath at 27 °C ± 3 °C (80 °F ± 5 °F) for 45 min (± 5 min) until the samples are tested.

7.4.6 Test Period

The test period is the time elapsed from subjecting the specimen to heat in the curing vessel to testing the sample for strength. The test should be performed within 45 min of the desired test duration.



Key

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

Figure 6—Diagram of Mold Preparation

7.4.7 Strength Testing

Test specimens immediately after removal from the cooling bath. Measure the specimen height and calculate the minimum surface area in contact with the platen. Place the specimen on the platen with a side of the specimen in contact with the platen (i.e. the top surface during curing should not be in contact with the platen). The test procedure should be in accordance with ASTM C109/C109M-07, except for the following.

a) A compressive-strength testing load frame should be used, and the rate of loading for samples with expected strength greater than 3.5 MPa (500 psi) should be 72 kN ± 7 kN (16,000 lbf ± 1600 lbf) per minute. For a nominal 25.8 cm^2 (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. For samples with expected strength of 3.5 MPa (500 psi) or less, a loading rate of 18 kN ± 2 kN (4000 lbf ± 400 lbf) per minute should be used. For a nominal 25.8 cm^2 (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. Depending on the type of compressive strength load frame employed, it may require some time for the load frame to build up the required load rate after initial contact with the cement sample.

NOTE If the cube height is less than 48 mm (1.9 in.), the cube should be discarded.

b) Report compressive strength as the force required to break the sample divided by the smallest measured cross-sectional area in contact with the load-bearing plates of the load frame. Average the compressive strength of all acceptable test samples (see ASTM C109/C109M-07) made from the same slurry and tested at the same time. Report compressive strength results to the nearest 0.3 MPa (50 psi) 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 is 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

7.5.2.1 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.

7.5.2.2 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), in minutes.

In SI units:

$$t = \frac{T_{\text{PBHC}} - T_{\text{TOCC}}}{1.0\ ^\circ\text{C}} \tag{31}$$

In USC units:

$$t = \frac{T_{\text{PBHC}} - T_{\text{TOCC}}}{2.0\ ^\circ\text{F}} \tag{32}$$

where

t is the elapsed cool-down time, expressed in minutes;

T_{PBHC} is the predicted bottomhole circulating temperature, expressed in °C or °F;

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

Maintain test pressure while decreasing the temperature. When the T_{TOCC} or 88 °C (190 °F) (whichever is lower) is reached, release the pressure slowly and remove the slurry container.

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

7.5.2.4 Pour the slurry into prepared molds as specified in 7.4.3.2 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. In not more than 15 min after removing the slurry from the consistometer, apply pressure simulating well pressure at top-of-cement, within the limitations of the apparatus being used.

7.5.2.5 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.

7.5.2.6 Remove samples as specified in 7.4.5.

7.5.2.7 Test the samples for strength in accordance with procedures in 7.4.7 or Section 8.

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

Temperature Gradient [d] (°C/100 m)	**Schedule** [a] (SI)	**1Sg**	**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)	**1Sg**	**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 a schedule number designation.

[b] The temperature should 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) should be maintained within ± 2 °C throughout the remainder of the curing period until cooling, if necessary, is required (see 7.4.5).

[c] The test temperature should 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 Test Schedules for Curing Compressive Strength Specimens (USC)

Temperature Gradient [d] (°F/100 ft)	**Schedule** [a] (USC)	**1Sg**	**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] (psi)	**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)	1Sg	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] (psi)	**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} (°C)	80	91	103	120	146	180	219	250	284	321	360	404
	Temp. ramp to T_{BHC} (°C/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} (°C)	97	114	148	182	216	250	284	318	352	386	420	454
	Temp. ramp to T_{BHS} (°C/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} (°C)	80	91	104	126	160	200	236	271	309	350	395	444
	Temp. ramp to T_{BHC} (°C/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} (°C)	99	118	156	194	232	270	308	346	384	422	460	498
	Temp. ramp to T_{BHS} (°C/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 a schedule number designation.

[b] The temperature should 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) should be maintained within ± 3 °F throughout the remainder of the curing period until cooling, if necessary, is required (see 7.4.5).

[c] The test temperature should 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 psi ± 100 psi;
Schedule 2 Sg	1600 psi ± 200 psi;
Schedule 3 Sg–12 Sg	3000 psi ± 500 psi.

[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 should include the following systems:

a) **Temperature-measuring system**, calibrated to an accuracy of ± 2 °C (± 3 °F).

b) **Sonic signal measuring system**, calibrated in accordance with the manufacturer's instructions.

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 should be obtained in accordance with Section 4.

8.4 Preparation of Slurry

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

8.5 Procedure

Follow the detailed operating instructions and safety precautions furnished by the manufacturer. The presence of free fluid can cause the slurry to lose contact with the top cell cover plate. This can affect the sonic signal being sent through the slurry.

8.6 Curing Time

The curing period begins with the recording of the transit time and the application of temperature and pressure and continues until the test is terminated. Recording of transit-time data should begin within 5 min after application of temperature and pressure.

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 should 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 should be obtained using transit time correlations specific to the cement composition.

The set cement acoustic impedance may be calculated from Equation (33) (SI) or Equation (34) (USC).

In SI units:

$$Z = 25.4 \times \frac{\rho_{\text{set}}}{\text{TT}} \tag{33}$$

In USC units:

$$Z = 3.046 \times \frac{\rho_{\text{set}}}{\text{TT}} \tag{34}$$

where

Z is the acoustic impedance, expressed in 10^6 kg/m^2·s (MRayl);

ρ_{set} is set cement density, expressed in kg/l (lbm/gal);

TT is the transit time, expressed in µs/in.

NOTE 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 should not be reported as 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 should 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 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, see also API 10A). The slurry container is rotated at a speed of 150 r/min ± 15 r/min. An alternative apparatus is described in Annex C. Apparatuses are calibrated according to the requirements in Annex B.

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.

The paddle and all parts of the slurry container exposed to the slurry should be constructed of corrosion-resistant materials. The space between the slurry container and the walls of the pressure vessel should be completely filled with hydrocarbon or synthetic oil (9.2.2 and API 10A, Section 10.1).

The temperature and consistency of the cement slurry (9.3.2) should be measured.

To determine the thickening time, the consistency of a cement slurry is 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, or its equivalent, is used to determine the torque.

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

9.2.2 Oil, selected to have the following physical properties:

Viscosity range = 6 mm^2/s to 79 mm^2/s at 38 °C (100 °F) or 6 cSt to 79 cSt at 38 °C (100 °F) or 45 SSU to 360 SSU at 38 °C (100 °F)

Specific heat = 1.9 kJ/(kg·K) to 2.5 kJ/(kg·K) (0.45 Btu/(lb·°F) to 0.60 Btu/(lb·°F))

Thermal conductivity = 0.112 W/(m·K) to 0.138 W/(m·K) [0.065 Btu/(h·ft^2·°F/ft) to 0.08 Btu/(h·ft^2·°F/ft)]

Specific gravity = 0.83 to 0.93

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.

9.2.3 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).

Figure 7—Typical Pressurized Consistometer

9.3 Test Procedure

9.3.1 Operating Instructions

Detailed operating instructions, as furnished by the equipment manufacturer, are applicable under this method and should be followed, provided they conform to the following procedure.

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 should 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

9.3.3.1 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 should 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.

9.3.3.2 Secure the head assembly in the pressure vessel, insert 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.

9.3.3.3 Begin the thickening time test by applying the initial pressure and starting the temperature ramp. No more than 5 min should have elapsed after cessation of mixing of the slurry as defined in 5.3.4 and the beginning of the test. Alternatively, if surface mixing is to be simulated in the consistometer, no more than 5 min should have elapsed before the beginning of the surface mixing simulation.

9.3.4 Temperature and Pressure Controls

During the thickening time test, increase the temperature and pressure of the cement slurry in the slurry container 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 should be maintained within ± 3 °C (± 6 °F) and ± 2 MPa (± 300 psi) of the appropriate temperature and pressure target vs elapsed time. Within 10 min after the end of the ramp, temperature and pressure should be within ± 1 °C (± 2 °F) and ± 0.7 MPa (± 100 psi) of the specified values. Determine the temperature of the cement slurry by use of a temperature measuring device located in the center of the sample container. The tip of the thermocouple should 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 should 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 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 9.4.2 to 9.4.5.1 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 is based upon type of cement job to be performed: casing, liner, plug, continuous—pumping squeeze or hesitation 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.

Caution—Plug cementing well-simulation schedules: Recognize 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 Schedules

Casing and liner well-simulation thickening-time schedules should 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 ($R_{\Delta \mathrm{T}}$) (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 should 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 Schedules

9.4.3.1 Surface Mixing of the 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 should be reported separately from the thickening time of the slurry. For example:

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 follows:

$$t_\mathrm{d} = \frac{V_\mathrm{p}}{q} \tag{35}$$

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 (bbl);

q is the pump rate, expressed in cubic meters per minute (bbl/min).

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 is given by:

$$T_{\mathrm{PBHC}} = 26.7\ ^{\circ}\mathrm{C} + \frac{(0.006061 \times h_{\mathrm{TVD}} \times \Delta_{\mathrm{PT}}) - 5.6064}{1.0 - (0.000049383 \times h_{\mathrm{TVD}})} \tag{36}$$

or, in degrees Fahrenheit,

$$T_{\mathrm{PBHC}} = 80\ ^{\circ}\mathrm{F} + \frac{(0.006061 \times h_{\mathrm{TVD}} \times \Delta_{\mathrm{PT}}) - 10.0915}{1.0 - (0.000015052 \times h_{\mathrm{TVD}})} \tag{37}$$

where

T_{PBHC} is the predicted bottomhole circulating temperature, expressed in °C [Equation (36)] or °F [Equation (37)];

h_{TVD} is true vertical depth, expressed in meters [Equation (36)] or feet [Equation (37)];

Δ_{PT} is the pseudo-temperature gradient, expressed in °C/100 m [Equation (36)] or °F/100 ft [Equation (37)].

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 should 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

The temperature of the cement slurry should 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 can be calculated using the following equation:

$$R_{\Delta \mathrm{T}} = \frac{T_{\mathrm{PBHC}} - T_{\mathrm{SS}}}{t_{\mathrm{d}}} \tag{38}$$

where

$R_{\Delta \mathrm{T}}$ is the rate of temperature change, expressed in °C/min (°F/min);

T_{PBHC} is the predicted bottomhole circulating temperature, expressed in °C (°F);

T_{SS} is the slurry surface temperature, expressed in °C (°F);

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

The bottomhole pressure can be calculated as follows:

In SI units:

$$p_{BH} = 0.00981 \times \rho_{af} \times h_{TVD} \tag{39}$$

In USC units:

$$p_{BH} = 0.052 \times \rho_{af} \times h_{TVD} \tag{40}$$

where

p_{BH} is the bottomhole pressure, expressed in kPa (psi);

ρ_{af} is the density of annular fluid, expressed in kg/m^3 (lbm/gal);

h_{TVD} is the true vertical depth, expressed in m (ft).

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

9.4.3.6 Starting Pressure

A minimum starting pressure (p_S) of 700 kPa (100 psi) 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 should be increased to the bottomhole pressure during the test at a pressure-up rate calculated as follows:

$$R_{pu} = \frac{p_{BH} - p_S}{t_d} \tag{41}$$

where

R_{pu} is the rate of pressure increase, expressed in kPa/min (psi/min);

p_{BH} is the bottomhole pressure, expressed in kPa (psi);

p_S is the starting pressure, expressed in kPa (psi);

t_d is the time to displace the leading edge of cement to bottom, expressed in min (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} should 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 is calculated as follows:

$$t_{\text{a}} = \frac{V_{\text{a}}}{q} \tag{42}$$

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 min;

V_{a} is the annular volume of cement, expressed in m^3 (bbl or ft^3);

q is the pump rate, expressed in m^3/min (bbl/min or ft^3/min).

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 the equation

$$R_{\Delta\text{T}} = \frac{T_{\text{TOCC}} - T_{\text{PBHC}}}{t_{\text{a}}} \tag{43}$$

where

$R_{\Delta\text{T}}$ 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 (38).

T_{PBHC} is the bottomhole circulating temperature, expressed in °C (°F);

T_{TOCC} is top-of-cement circulating temperature, expressed in °C (°F);

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 min.

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

9.4.3.11 Pressure at the Top-of-Cement

Pressure at the top of the cement slurry is calculated using the following equation:

In SI units:

$$p_{\mathrm{TOC}} = 0.00981 \times \rho_{\mathrm{af}} \times h_{\mathrm{TOCTVD}} \tag{44}$$

In USC units:

$$p_{\mathrm{TOC}} = 0.052 \times \rho_{\mathrm{af}} \times h_{\mathrm{TOCTVD}} \text{ (USC)} \tag{45}$$

where

p_{TOC} is the pressure at the top of the cement in the annulus, expressed in kPa (psi);

ρ_{af} is the density of annular fluid, expressed in kg/m^3 (lbm/gal);

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

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

The following equation is used to calculate the rate of decrease in pressure (pressure-down rate) from the bottomhole pressure to the pressure at the top of the cement slurry column:

$$R_{\mathrm{pd}} = \frac{p_{\mathrm{BH}} - p_{\mathrm{TOC}}}{t_{\mathrm{a}}} \tag{46}$$

where

R_{pd} is the rate of pressure decrease, expressed in kPa/min (psi/min);

p_{BH} is the bottomhole pressure, expressed in kPa (psi);

p_{TOC} is the pressure at the top of the cement column in the annulus, expressed in kPa (psi);

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 min.

9.4.3.13 Completion of Test with Simulated Temperature Change

The cement slurry should 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, 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 psi) is recommended for squeeze-cementing operations.

9.4.4.2 Continuous-pumping Squeeze-cementing

The predicted squeeze cementing temperatures (T_{PSP}) for continuous-pumping squeeze-cementing is provided in Table D.2 or can be calculated using Equation (49) or Equation (50). Pressure is increased directly from surface pressure (p_{S}) to final squeeze pressure (p_{FSQ}, see 9.4.4.5), Once reached, the final temperature (T_{PSP}) and final squeeze pressure (p_{FSQ}) should be maintained until the thickening-time test is completed.

9.4.4.3 Hesitation-pumping Squeeze Schedules

The predicted squeeze-cementing temperatures (T_{PSP}) and pressures for hesitation-pumping squeeze-cementing are determined as per 9.4.4.2 continuous-pumping squeeze schedules.

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

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 is typically stopped for 10 min and then restarted for 5 min. This cycle is 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 should 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 pressure (p_{FSQ}), which would include any applied surface squeeze pressure (p_{SQ}).

$$p_{\mathrm{FSQ}} = p_{\mathrm{SQ}} + p_{\mathrm{BH}} \tag{47}$$

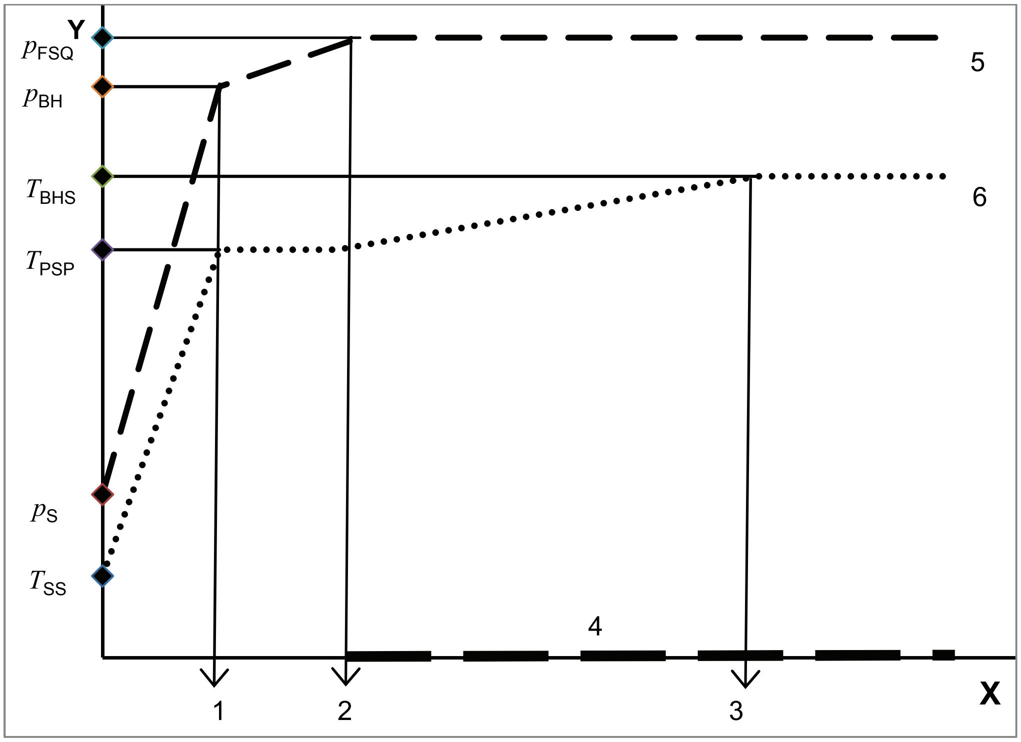
where

p_{FSQ} is the final bottomhole squeeze pressure, expressed in kPa (psi);

p_{SQ} is the final surface squeeze pressure, expressed in kPa (psi);

p_{BH} is the placement bottomhole pressure at the beginning of the squeeze, expressed in kPa (psi).

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 pressure (p_{FSQ}) is a function of the well and the zone to be squeezed. Then, the maximum surface final squeeze pressure (p_{SQ}) used to determine the squeeze cementing schedule is left to the user; typical values can vary from 3400 kPA (500 psi) to more than 17,000 kPa (2500 psi).



Key

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

Figure 8—Typical Hesitation Squeeze Pressure and Temperature Schedule

For squeeze-cementing hesitation squeeze application of the surface final squeeze pressure (p_{SQ}) results in a second pressure step-up rate (R_{PSQ}), pressure increase, from p_{BH} to p_{FSQ}, is calculated using Equation (48).

$$R_{\mathrm{PSQ}} = \frac{p_{\mathrm{SQ}}}{t_{\mathrm{sq}}} \tag{48}$$

where

R_{PSQ} is the rate of pressure increase during application of the squeeze pressure, expressed in kPa/min (psi/min);

p_{SQ} is the final surface squeeze pressure, expressed in kPa (psi);

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

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

9.4.4.5 Squeeze-cementing Tailored Schedules

Equations (35), (38) to (41), under 9.4.3.2, 9.4.3.4 to 9.4.3.7, and Equations (47) to (50), under 9.4.4.5 should be used for tailored schedules. These equations can be used to calculate the heat-up rate ($R_{\Delta\mathrm{T}}$) and pressure-up rates (R_{pu} and R_{PSQ}) for a squeeze simulation thickening-time test. In Equation (38), the predicted squeeze temperature (T_{PSP}) from Equation (49) or Equation (50) should be substituted for the T_{PBHC}. After reaching the T_{PSP} and p_{FSQ}, the temperature and pressure profiles should follow the anticipated temperature and pressure profiles for the remainder of the squeeze operation.

Additionally, 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, if the squeeze-cementing operation is expected to use the hesitation technique. When the final pressure is reached (p_{FSQ}), slurry container rotation is stopped for 10 min and then restarted for 5 min. This cycle should 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 job, if they are known. In addition to cycling the stirring after final pressure is reached, the temperature is 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 Temperatures

The correlation developed for predicting squeeze temperatures, in Celsius, is given by:

$$T_{\mathrm{PSP}} = 26.7\ ^{\circ}\mathrm{C} + \frac{(0.0076495 \times h_{\mathrm{TVD}} \times \Delta_{\mathrm{PT}}) - 4.5567}{1.0 - (0.00002647 \times h_{\mathrm{TVD}})} \tag{49}$$

or in Fahrenheit:

$$T_{\mathrm{PSP}} = 80\ ^{\circ}\mathrm{F} + \frac{(0.0076495 \times h_{\mathrm{TVD}} \times \Delta_{\mathrm{PT}}) - 8.2021}{1.0 - (0.000008068 \times h_{\mathrm{TVD}})} \tag{50}$$

where

T_{PSP} is the predicted bottomhole squeeze temperature, expressed in °C (°F);

Δ_{PT} is the pseudo-temperature gradient, expressed in °C/100 m (°F/100 ft);

h_{TVD} is the true vertical depth, expressed in m (ft).

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 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 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 psi) is 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}) is provided in Table D.2 or can be calculated using Equation (49) or Equation (50). Once reached, the final temperature (T_{PSP}) and the bottomhole pressure (p_{BH}) should be maintained until the thickening-time test is completed. If the well is circulated, predicted plug-cementing temperature should be the T_{PBHC} provided in Table D.1 or Equation (36) or Equation (37) under 9.4.3.3.

9.4.5.3 Plug-cementing Tailored Schedules

Equation (35), Equation (36), or Equations (37) to (41), under 9.4.3.2 to 9.4.3.7 can be used for plug-cementing tailored schedules. These equations can be used to calculate the heat-up rate ($R_{\Delta\mathrm{T}}$) and pressure-up rate (R_{pu}) (respectively) 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 (38), the predicted plug temperature, T_{PSP}, calculated using Equation (49) or Equation (50) 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 should be used. Therefore, the steps in 9.4.3.9 to 9.4.3.13 should 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 nonstirred or stirred fluid-loss cells. For tests at temperatures less than or equal to 88 °C (190 °F), tests may be performed using a nonstirred 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 High-pressure, High-temperature Fluid-loss Cells

10.2.1.1 General

High-pressure, high-temperature nonstirred fluid-loss cell or stirred fluid-loss apparatus, are fitted with a 45 µm (325 mesh) screen with a 22.6 cm^2 (3.5 $in.^2$) filtration area backed by a 250 µm (60 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.

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) is 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 should not be exceeded.

10.2.1.2 High-pressure, High-temperature Nonstirred Fluid-loss Cells

Two main types of nonstirred high-pressure, high-temperature fluid-loss cells can be used for testing cement slurries: high-pressure, high-temperature “long” 25.4 cm (10 in.) cells and high-pressure, high-temperature “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 high-pressure, high-temperature cell is the preferred nonstirred apparatus.

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

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

Single-ended cells 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.

10.2.1.3 High-pressure, High-temperature 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 is 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.

10.2.2 Temperature Measuring System

A thermocouple or temperature measuring device accurate to ± 2 °C (± 3 °F) or better should 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.

10.2.3 Pressure gauges

Pressure gauges with a scale such that pressure can be read to ± 300 kPa (± 50 psi) should be used.

10.2.4 Timer or stopwatch

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

10.3 Safety

Warning—These procedures require the handling of hot, pressurized equipment and materials that are hazardous and can cause injury. Only trained personnel shall run these tests. Fluid-loss tests should 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 "nontest" 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 should be taken. Refer to manufacturers safety procedures.

10.4 Performing Static Fluid-loss Test Using Nonstirred Fluid-loss Cell

10.4.1 General

Apparatus and instruments are calibrated according to the requirements in Annex B.

This subsection covers test procedures using high-pressure, high-temperature, nonstirred fluid-loss cells ("long" or "short" 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 psi)].

Conditions of screens, O-rings, stems, and other components used in the test should 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 Nonstirred Fluid-loss Cell

10.4.2.1 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.

10.4.2.2 Preheat the cell in the heating jacket to the test temperature or to a maximum of 88 °C ± 3 °C (190 °F ± 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.

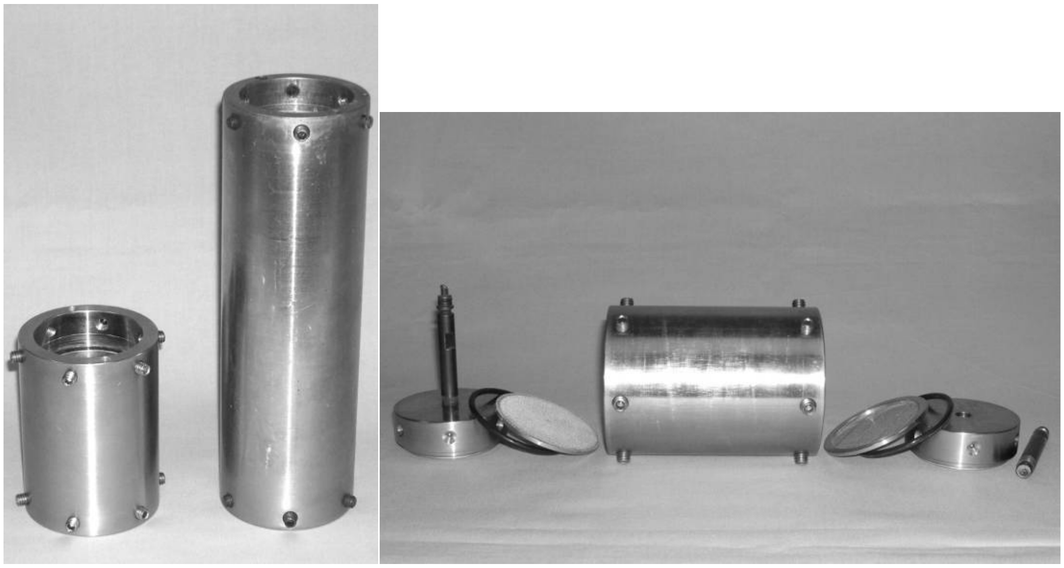


Figure 9—Common High-temperature, High-pressure, Nonstirred Fluid-loss Cell Bodies

Figure 10—Common Screwed-cap Type, High-temperature, High-pressure, Double-ended Fluid-loss Cell

10.4.4 Filling the Nonstirred Fluid-loss Cell

10.4.4.1 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 325 mesh side of the screen should 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 (psi)	**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

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

10.4.4.3 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)

10.4.5.1 Place the thermocouple in the thermocouple well in the top of the cell body.

10.4.5.2 Connect the pressure supply line and apply 7000 kPa ± 300 kPa (1000 psi ± 50 psi). Open the top valve stem to apply the pressure to the cell.

10.4.5.3 Start the test**:** Open the bottom valve and start the timer simultaneously. Maintain at the test temperature and 7000 kPa ± 300 kPa (1000 psi ± 50 psi) 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).

10.4.5.4 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. 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.

10.4.5.5 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 should be considered the end of the test.

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

10.4.5.7 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)

10.4.6.1 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 psi ± 50 psi), 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.

10.4.6.2 Place the thermocouple in the thermocouple well in the top of the cell body.

10.4.6.3 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 psi) for a test at 150 °C (300 °F)].

10.4.6.4 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.

10.4.6.5 Apply 7000 kPa ± 300 kPa (1000 psi ± 50 psi) 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 psi ± 50 psi).

10.4.6.6 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 psi ± 50 psi) differential pressure for the duration of the test.

10.4.6.7 Collect the filtrate and record the volume after 30 min or 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. If a condenser is used, the volume of fluid in the condenser itself must be accounted for.

10.4.6.8 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 time at which nitrogen blows through.

10.4.6.9 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.

10.4.6.10 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 Clean-up

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

10.4.7.2 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.

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

10.4.7.4 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.

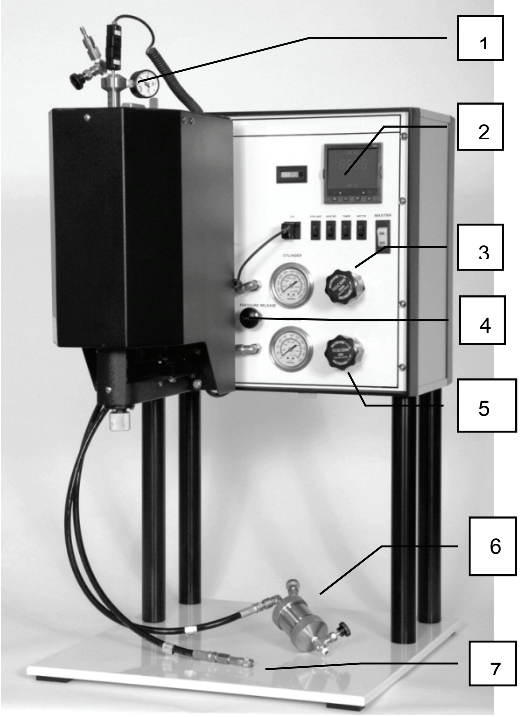
10.5 Performing a Static Fluid-loss Test Using Stirred Fluid-loss Apparatus

10.5.1 General

Apparatuses and instruments are calibrated according to the requirements in Annex B.

This section covers the test procedure using a stirred fluid-loss apparatus. 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 psi)].



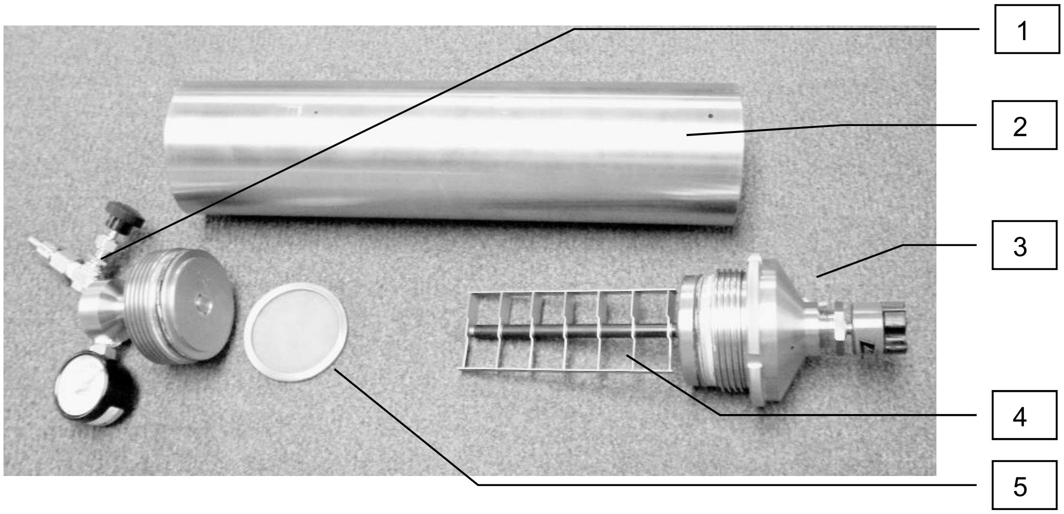
Key

1 top plug, outlet valve
2 temperature controller
3 cell pressure regulator
4 pressure release valve
5 back-pressure regulator
6 back-pressure receiver
7 nitrogen pressure supply line

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—Common Stirred Fluid-loss Apparatus

Condition of the screen, O-rings, stems, and other parts used in the test should 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.



Key

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

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

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

10.5.2 Mixing and Conditioning the Slurry

10.5.2.1 Prepare the stirred fluid-loss apparatus in accordance with the manufacturer’s instructions.

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

10.5.2.3 Mix the slurry in accordance with 5.3.

10.5.2.4 Pour the slurry into the clean, dry, cell 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.

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

10.5.2.6 Apply and maintain 3500 kPa ± 300 kPa (500 psi ± 50 psi) (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.

10.5.2.7 Start stirring the slurry with the paddle.

10.5.2.8 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 in the step in 10.5.2.2). 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.

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

10.5.2.9 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.

10.5.2.10 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 psi) for a test at 150 °C (300 °F)].

10.5.2.11 If it was disconnected, connect the pressurizing line. Apply 7000 kPa ± 300 kPa (1000 psi ± 50 psi) differential pressure. The pressure applied is the sum of the pressure applied to the back-pressure receiver (if used) plus 7000 kPa ± 300 kPa (1000 psi ± 50 psi). 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

10.5.3.1 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 7000 kPa ± 300 kPa (1000 psi ± 50 psi) differential pressure for the duration of the test.

10.5.3.2 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. 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.

10.5.3.3 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 time at which nitrogen blows through.

10.5.3.4 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.

10.5.3.5 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 Clean-up

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

10.5.4.2 Following manufacturer’s instructions, remove the cell.

10.5.4.3 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 should be taken. Refer to manufacturers safety procedures.

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

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

10.6 Fluid-loss Results and Reporting

10.6.1 Calculate the API Fluid Loss, expressed as milliliters per 30 min. 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.

$$\text{API Fluid Loss} = 2V_{30} \tag{51}$$

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 (52) or Equation (53) to determine the Calculated API Fluid Loss.

$$\text{Calculated API Fluid Loss} = 2V_t\sqrt{\frac{30}{t}} \tag{52}$$

or, combining the constants, the equation reduces to:

$$\text{Calculated API Fluid Loss} = 10.954 \times \frac{V_t}{\sqrt{t}} \tag{53}$$

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, (psi)]
[ ] 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
[ ] 325 mesh × 60 mesh
[ ] 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] _______ 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 Using a Rotational Viscometer

11.1 General

This procedure can 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:

- the slurry is homogenous and the shear stress is uniform across the gap,
- the flow regime in the annular gap is laminar,
- slip at the wall is negligible, and
- the fluid exhibits time-independent behavior.

11.2 Apparatus

11.2.1 Rotational Viscometer

11.2.1.1 General

A typical rotational nonpressurized viscometer, also known as a Couette viscometer, should 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 spring, which restrains its movement. The angle of 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 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 is usually sufficient to provide the required gap width. A B2 bob is also available; however, its use is only acceptable for measuring fluids containing very large particles [i.e. diameters greater than 0.243 mm (0.0096 in.) but smaller than 0.614 mm (0.0242 in.)].

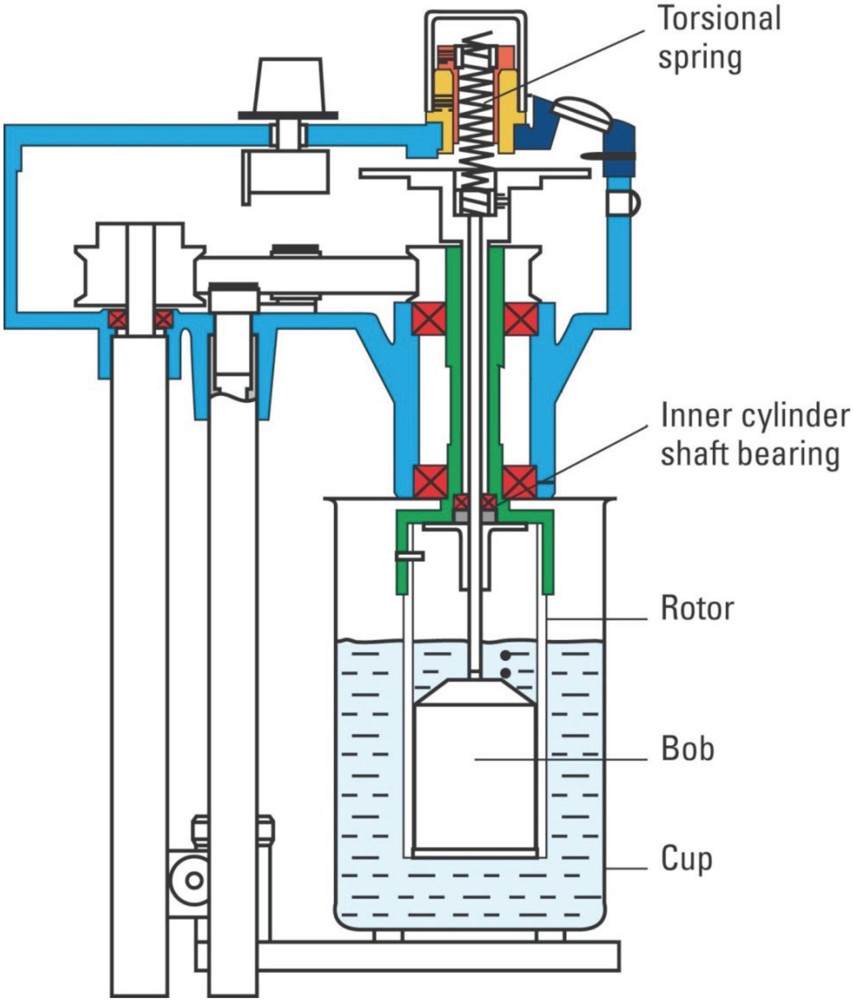


Figure 13—Typical Rotational Viscometer Schematic

Table 6—Dimensions of Rotors and Bobs

Dimension in millimeters (inches)

	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)
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

Rotor and bob dimensions for an R1-B1 configuration are illustrated in Figure 14. The rotor should have a scribed line 58.42 mm ± 0.1 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 3.16 mm (0.125 in.) and 9.51 mm (0.375 in.) below the scribed line. The bob should 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 should have a roughness average of 8 to 32 cross-hatch honed.

The rotational viscometer should 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.

Dimensions in millimeters

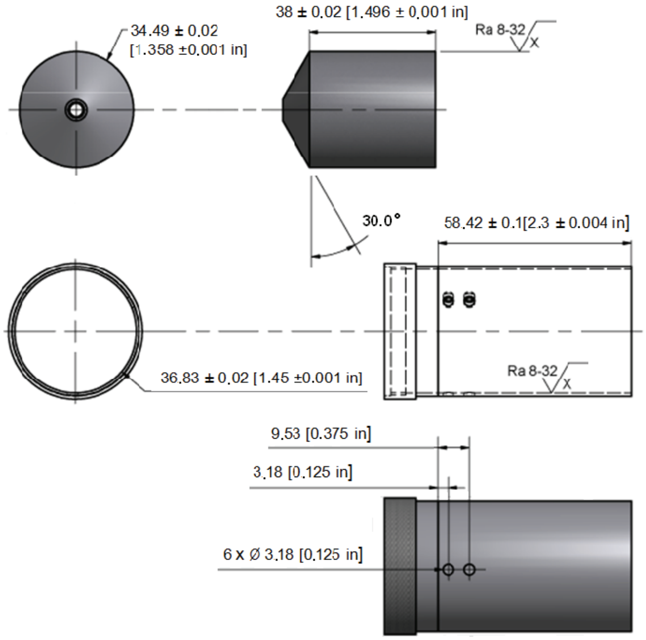


Figure 14—Rotor and Bob Dimensions (R1-B1)

11.2.1.2 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 7 shows the calculated shear rate for each rotor-bob combination across a range of rotational velocities.

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 spring. Springs of varying torsional stiffness are available. Table 8 shows the shear stress per degree of dial deflection for the various combinations of rotors, bobs, and springs. Table 9 shows the maximum shear stress that can be measured for the various combinations of rotors, bobs, and springs. An F 1.0 spring is suitable for measuring the rheology of most well cementing fluids. Fluids that have very high viscosity, such as high density cement slurries or slurries with a high polymer loading, may require the use of an F 2.0 or F 3.0 spring in order to obtain a shear stress dial reading at the maximum recommended rotational speed of 300 r/min. Fluids having very low viscosities may be measured with better accuracy using an F 0.5 or F 0.2 spring.

Table 7—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

Table 8—Shear Stress per Degree of Dial Deflection

Spring Factor	**Rotor-Bob**		
	R1-B1	**R1-B2**	**R1-B5**
	Shear Stress/Degree in pascals (lbf/100 ft^2)		
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)

Table 9—Maximum Shear Stress for Various Configurations (300° Maximum Deflection)

Spring Factor	**Rotor-Bob**		
	R1-B1	**R1-B2**	**R1-B5**
	Maximum Shear Stress in pascals (lbf/100 ft^2)		
F 0.2	31 (64)	61 (126)	36 (74)
F 0.5	76 (160)	151 (315)	89 (186)
F 1.0	153 (320)	302 (630)	179 (372)
F 2.0	306 (640)	605 (1261)	357 (474)
F 3.0	459 (960)	907 (1891)	535 (1115)

11.2.2 Stopwatch or Electric Timer

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

11.2.3 Thermometer or Thermocouple

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

11.3 Calibration

Rheometers are calibrated according to the requirements in Annex B and according to the manufacturer's instructions.

11.4 Determination of Rheological Properties

11.4.1 This procedure is 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.

11.4.2 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 rheometer and placed in the cup during heating.

11.4.3 Preheat the rotor, bob, and cup to the test temperature.

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

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

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

11.4.7 Immediately 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.

11.4.8 With the rotor turning at 3 r/min, raise the cup until the liquid is level with the scribed line on the rotor.

11.4.9 Take the initial instrument dial reading after 10 s continuous rotation at 3 r/min.

11.4.10 Take all the remaining dial readings, first in ascending order and then 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. Exposing cement slurries to speeds above 300 r/min can lead to inconsistent (erroneous) results.

11.4.11 If desired, dial readings at speeds higher than 300 r/min 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. 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).

11.4.12 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.

11.4.13 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 nonsettling, nongelling, 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 should be considered.

11.4.14 Report the fluid rheological measurements as the average of the dial readings [(ramp-up + ramp-down)/2], and the temperatures recorded in 11.4.11. Also report the rotor, bob, and spring configuration, as well as the conditioning methods used. An example is shown in Table 10.

11.4.15 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.

Table 10—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.

11.5 Determination of Gel Strength

11.5.1 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.

11.5.2 Recondition the fluid in the viscometer for 1 min at 300 r/min to disperse the gels and 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 11.4.2 to 11.4.8 and then condition the fluid for 1 min at 300 r/min.

11.5.3 Stop rotation of the rotor.

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

11.5.5 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.

11.5.6 Record the fluid temperature, then stop the viscometer for 10 min.

11.5.7 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.

11.5.8 Gel strength measurements may be taken at other static times, as desired. For other static times, follow the procedure of 11.5.6 and 11.5.7, substituting the desired static time for 10 min.

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

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

11.5.11 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.

11.6 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).

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

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 (54) (SI units) or Equation (55) (USC units). The use of a computer program or spreadsheet is recommended to perform the regression analysis. Two point calculations are not accurate and should not be used.

In SI units:

$$\tau = \tau_{\mathrm{y}} + (\mu_{\mathrm{p}} \times \gamma) \tag{54}$$

In USC units:

$$\tau = \tau_{\mathrm{y}} + 0.00208(\mu_{\mathrm{p}} \times \gamma) \tag{55}$$

where

τ is the shear stress, expressed in Pa (lbf/100 ft^2);

τ_{y} is the Bingham yield stress, expressed in Pa (lbf/100 ft^2);

μ_{p} is the plastic viscosity, expressed in Pa·s (cP);

γ is the shear rate, expressed in 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 (56). The use of a computer program or spreadsheet is recommended to perform the regression analysis. Two point calculations are not accurate and should not be used.

$$\tau = k \times \gamma^{n} \quad \text{(SI or USC)} \tag{56}$$

where

τ is the shear stress, expressed in Pa (lbf/100 ft^2);

k is the consistency index, expressed in Pa·s^n (lbf·s^n/100 ft^2);

γ is the shear rate, expressed in 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 (57). The use of a computer program is recommended to perform this complex data analysis.

$$\tau = \tau_y + k \times \gamma^{n} \quad \text{(SI or USC)} \tag{57}$$

where

τ is the shear stress, expressed in Pa (lbf/100 ft^2);

τ_y is the Herschel-Bulkley yield stress, expressed in Pa (lbf/100 ft^2);

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

γ is the shear rate, expressed in s^{-1};

k is the consistency index, expressed in Pa·s^n (lbf·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 yield stress equals the yield point, the flow behavior index becomes 1 and 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 is 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.

12.2 Slurry Mixing and Conditioning

Prepare the cement slurry and condition it 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 with Heated Static Period

12.3.1 Prepare for Test

A clear, glass tube is required for this test. The ratio of the slurry-filled length to the inside tube diameter should be greater than 6:1 and less than 8:1, with a mark at the required fill level. The clear tube should be inert to well cements and should not deform during the test. The free-fluid test slurry volume should be between 100 ml and 250 ml, inclusive.

Preheat (or precool) a test chamber for aging the slurry during the static period to T_{PBHC} ± 3 °C (± 5 °F) 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 is less than 100 °C (212 °F), adjust the temperature accordingly. This chamber may be an atmospheric heating or cooling bath/oven/jacket/chamber or a suitable pressurized heating/cooling chamber that uses hydrocarbon oil to transmit heating/cooling to the slurry.

A bath/oven/jacket/chamber or pressurized chamber is designated hereafter in this section as a chamber. When hydrocarbon oil is used, the oil should have a flash point that satisfactorily meets all safety requirements to perform the test.

12.3.2 Free-fluid Tests at Atmospheric Pressure with Static Temperature Less Than or Equal to 88 °C (190 °F)

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. Immediately place the tube in a heating or cooling chamber that is preheated or precooled to T_{PBHC}. The chamber must be able to heat or cool the entire slurry. The tube 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 should be taken to ensure the static curing is performed at essentially vibration free conditions.

Maintain the temperature at T_{PBHC} or 88 °C ± 3 °C (190 °F ± 5 °F), whichever is the lower, for the duration of the test. The test duration is 2 hr, starting from the time the slurry is poured into the tube. At 2 hr ± 1 min, measure the volume of free fluid (clear or colored fluid on top of the cement slurry inside the tube) with a precision of 0.2 ml.

Calculate the volume fraction, φ, of free fluid, expressed as a percent.

$$\varphi = \frac{V_{\text{F}}\,(100)}{V_{\text{s}}} \tag{58}$$

where

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

V_{s} is the initial volume of slurry, expressed in ml.

Report the % free fluid, conditioning method, time and temperature, tube dimension, test temperature and angle. The form shown as Table 11 may be used.

12.3.3 Free-fluid Tests Under Pressure with Static Temperature Greater Than or Equal to 88 °C (190 °F)

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 should be carefully placed in the tube above the slurry. 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. Apply sufficient pressure to prevent boiling of the slurry. Further heat the slurry to T_{PBHC} ± 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}. 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 (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.

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. 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 0.2 ml.

The volume fraction, as a percent of free fluid is then calculated in accordance with Equation (58). The results may be reported on a form such as the one in Table 12.

12.4 Free-fluid Test with Static Period at Ambient Temperature

Use 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.

Pour the slurry into the graduated glass cylinder to the 250 ml mark. The 2 hr test period is initiated when the conditioned slurry is poured into the cylinder. Seal the cylinder with plastic film wrap or equivalent material to prevent evaporation. The cylinder may be inclined at an angle to simulate wellbore deviation. Angles are referenced from vertical in the same way as the deviation angles of wells. Thus, vertical is 0° and horizontal is 90°. Take appropriate precautions to ensure that static curing is performed in essentially vibration-free conditions.

At 2 hr ± 1 min, measure the volume of free fluid (clear or colored fluid on top of the cement slurry) with a precision of 0.2 ml.

Calculate the volume fraction of free fluid (φ), as a percent, in accordance with Equation (58). The results may be reported on a form such as the one in Table 11.

12.5 Sedimentation Test

12.5.1 Preparation

The sedimentation tube should have an inner diameter of 25 mm ± 0.5 mm (1 in. ± 0.02 in.). The tube length should be a minimum of 100 mm (3.9 in.). The most common tube length is approximately 200 mm (7.9 in.) (see Figure 15). The tube should be inert to well cements and should 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.

Preheat or precool the sedimentation tube and a water-filled chamber (or oven) to T_{PBHC} ± 3 °C (± 5 °F) or 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).

12.5.2 Sedimentation Test Procedure

12.5.2.1 Pour conditioned slurry into the dry preheated/precooled sedimentation tube until it is approximately 20 mm (0.75 in.) from the top.

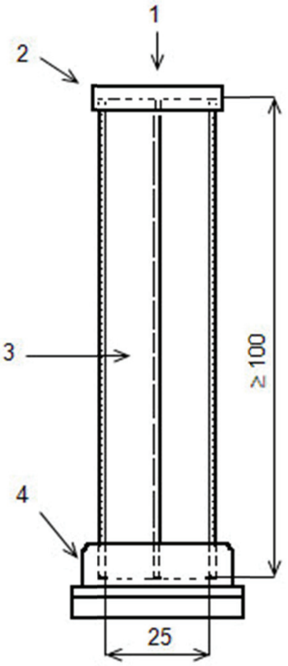
Puddle the slurry in the tube to dislodge any air bubbles, then fill the tube completely. A top closure that allows pressure communication may be used to prevent spillage of the slurry.

12.5.2.2 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.

12.5.2.3 Adjust the slurry temperature further to simulate temperature changes in the wellbore. Maintain pressure of 500 psi 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.

12.5.2.4 Cure the slurry for 24 hr or until set before removing it from the chamber.

Dimensions in millimeters



Key

1 vent hole
2 lid
3 split in tube
4 base

Figure 15—Typical Sedimentation Tube

12.5.2.5 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.

12.5.2.6 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.

12.5.2.7 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. Mark the segments to keep track of their order.

12.5.2.8 Break or cut the sample at these marks. Return all the segments, including the end pieces, to the water until each is weighed.

12.5.2.9 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.

12.5.2.10 Remove a segment to be measured from the water and gently dry it.

12.5.2.11 Place this segment on the balance beside the beaker.

12.5.2.12 Record the dry mass (m_{air}) and remove the segment from the balance.

12.5.2.13 Tare the balance if necessary.

12.5.2.14 Place a noose of thin string around the segment (dental floss works well).

12.5.2.15 Suspend the segment in the beaker such that the segment is totally surrounded by water. The segment should not touch the bottom or sides of the beaker. Air bubbles should not be clinging to the segment.

12.5.2.16 Record the wet mass (m_{water}) with the segment suspended in water.

12.5.2.17 Remove the segment from the water and tare the balance.

12.5.2.18 Repeat the procedure for each segment.

12.5.2.19 By applying Archimedes Principle, calculate the relative density of each cement segment.

$$d_{\text{rel}} = \frac{m_{\text{air}}}{m_{\text{water}}} \tag{59}$$

where

m_{air} is the cement segment mass in air (dry mass), expressed in g;

m_{water} is the cement segment mass in water (wet mass), expressed in g.

The results are used to construct a density profile for the entire specimen.

NOTE It is normal for cement slurries to experience a small density increase upon setting.

Calculate the percent density variance between the liquid slurry sample and the set cement sample.

$$\Delta\rho_{\text{sc}} = \frac{\rho_{\text{set}}}{\rho_{\text{s}}}(100) \tag{60}$$

where

$\Delta\rho_{\text{sc}}$ is the density of the set cement relative to the cement slurry density (%);

ρ_{set} is the density of the set cement segment (in any units);

ρ_{s} is the density of the cement slurry (in same units as for set cement).

The density variances between slurry and set well cements, and the density difference from top to bottom, 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 11) 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 11—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

Length of graduated tube section: _______

Graduated tube inner diameter: ___________

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: __________

Test angle: __________ (Angle is measured from vertical.)

Time to cool the slurry to 88 °C (190 °F): ___________

Ambient static period

Test angle: ________________ (Angle is measured from vertical.)

Measured free fluid volume (V_{F}): ____________ ml

Vol. 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: __________

Measured slurry density: __________________ (pressurized fluid density balance)

Calculated slurry density: __________

Density profile:

Segment No.	**Dry Mass** (m_{air})	**Wet Mass** (m_{water})	**SG** (ρ_{set})	**Density**	**% Density Difference** ($\Delta\rho_{\mathrm{sc}}$)
1 (top)					
2					
3					
4					
5					
6					
7					
8					
9					
10					
bottom					

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 should be exercised in the selection of the portion(s) of the procedure needed and interpretation of the results. The following test procedures are the same for preflushes and spacers, therefore the term spacer is used to refer to both fluids.

The user should 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 should 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 should be taken when working with flammable fluids.

13.2.1.2 The maximum temperature for testing at atmospheric pressure is 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 must be cooled to the test temperature or below flash point, whichever is lower, before releasing the pressure.

13.2.2 Preparation of Spacer

13.2.2.1 The spacer should 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 Condition spacer fluids 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.

13.2.3 Preparation of Drilling Fluid or Other Field Fluid

13.2.3.1 Representative samples of field mud or other field fluid should 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 as recommended by the supplier or in accordance with API 13B-1 or API 13B-2, as applicable.

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 require additional preparation, such as static aging or hot-rolling, to more closely simulate field fluid properties. Care should be taken in interpreting results when laboratory-prepared samples are used. Confirmation with a field sample of the mud is recommended once it is available.

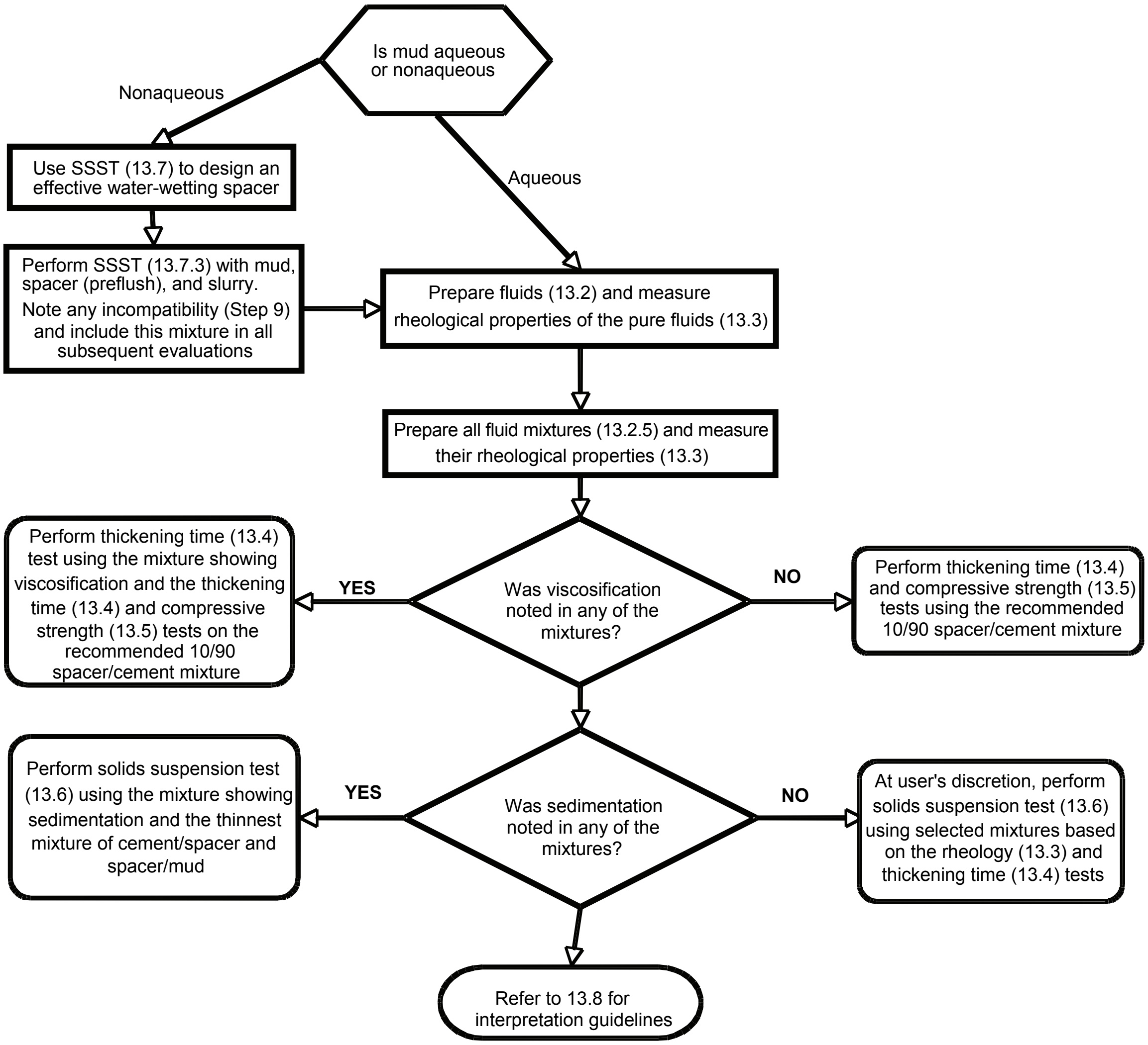


Figure 16—Compatibility Testing Flowchart

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, or 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.

Table 12—Mixtures for Testing

No.	Ratio Mud or Cement Slurry to Spacer (vol. %)	Mixing Scheme
1	95/5	380 ml mud or cement slurry/20 ml spacer
2	75/25	300 ml mix No. 1 plus 80 ml of spacer
3	5/95	20 ml mud or cement slurry/380 ml spacer
4	25/75	80 ml mud or cement slurry plus 300 ml of mix No. 3
5	50/50	Equal parts (200 ml) of mix No. 2 and mix No. 4
6	25/50/25 mud/spacer/cement slurry	Equal parts of mix No. 5 mud/spacer and mix No. 5 cement slurry/spacer

13.2.4 Preparation of Cement Slurries

Prepare a fresh quantity of cement slurry according to Section 5 or Annex A for each test and condition according to 5.4.

13.2.5 Preparation of Fluid Mixtures

13.2.5.1 Mixtures to be used for rheological properties, gel strength, solids suspension, thickening time, and compressive-strength testing should be prepared as described in this subsection (see Table 12). Data for uncontaminated test fluids should 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 should be sufficient to perform the desired test procedure.

13.2.5.3 Since compatibility can vary with test temperature, tests may be performed at well-site ambient temperature and/or T_{PBHC}. Consideration should 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 should 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, use an appropriately sized spatula or glass rod to gently stir one test fluid in a beaker or plastic cup as the other test fluid is being added. The minor component should 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. If signs of incompatibility are seen, note the approximate volume fraction and include this volume fraction in the testing routine. If there is sufficient quantity of cement, mud, and spacer available, using fresh fluids to prepare each fluid contamination ratio is preferred over combining previously tested mixtures (e.g. 75/25 and 25/75 to form a 50/50 mixture) and will improve test accuracy and repeatability.

13.3 Rheological Properties

Rheological properties should 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) is also recommended for rheological properties testing. At the user's discretion, the various-ratio mixtures may be prepared in accordance with Table 12 if sample volumes are too limited to permit use of fresh mixtures for each mix ratio. The rheological properties should be measured in accordance with Section 11. Additional conditioning of the mixtures is not required. Ramp up and ramp down rheology readings should be taken as per 11.4.8 to 11.4.10. 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 rheometer cup for signs of solids sedimentation.

NOTE Different sizes of cups are available for use with rheometers. As the cup dimensions may have an impact on the readings obtained, the same size cup should be used throughout the testing.

13.4 Thickening Time

13.4.1 Thickening-time tests should 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 should 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 test the thickest mixture 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 should be performed on mixtures of spacer/cement slurry. The recommended ratio is 10/90 spacer/cement slurry. The compressive-strength test should 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 should 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

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 10 min.

d) Start the motor and observe for signs of settling or gelation indicated by a momentary or sustained increase in the consistency.

e) Continue agitation until 1 hr after reaching T_{PBHC}. Observe the consistency and then stop the motor.

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.

Table 13—Rheological Compatibility of Mud, Cement Slurry, and Spacer

Cement slurry: ____________________

Spacer: ____________________

Mud: ____________________

Rotor bob/spring configuration: _________

Static time used for gel strength determination: _________

Fluid Mixture	**Test Temp**	**Average Viscometer Dial Readings** (r/min)							**API Gel Strength**	μ_p (PV)	τ_y (YP)
	°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											

13.7 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 NAF. This procedure facilitates the evaluation and the selection of proper spacers and/or surfactants in the spacer. This procedure will 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 nonaqueous or nonconductive 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.

Calibrate the apparatus 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. Fluids should 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 Step c).

h) Pour the minimum volume of conditioned, nonaqueous 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 titrating 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 should 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.

Curves showing typical responses are shown in 13.8.7, Figure 17.

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).

k) Evaluate the nonaqueous/spacer fluid titration results according to 13.8.7. If results are acceptable, continue to Step l). If not, redesign the spacer and repeat Steps a) through 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 Step 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 Step 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 Interpretation

13.8.1 General

The following subsections suggest methods for evaluating the results of compatibility testing and surfactant/spacer screening.

13.8.2 Mixing

Observe the nature of the fluid as the mixtures are being prepared (13.2.5.4). Viscosification, clabbering, flocculation, solids settling, or fluid separation is an indication of incompatibility of fluids. Any fluids showing such behavior should be thoroughly tested and modified, where necessary, to eliminate such behavior.

13.8.3 Rheology

13.8.3.1 Evaluate the rheological data for the mixtures of spacer/mud, spacer/cement slurry, and the mud/spacer/cement slurry. 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 should 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, evaluate the effects of this behavior on job performance and consider redesigning the fluids. Perform the solids suspension and static gel strength testing procedure (13.6) to further evaluate the mixture.

13.8.3.4 If the fluids are redesigned, the entire compatibility testing protocol should 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

Examine the consistency and temperature plots for any behavior that might indicate incompatibility. Incompatibility may be indicated by temperature oscillation, increase or decrease in the consistency, or change in the thickening time. Evaluate the effects of these behaviors on job performance and consider redesigning the fluids. If the fluids are redesigned, the entire compatibility testing protocol should 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 should be carefully considered. Evaluate these effects on well performance and consider redesigning the fluids. If the fluids are redesigned, the entire compatibility testing protocol should be repeated for the redesigned fluids.

13.8.6 Solids Suspension and Gel Strength

Sedimentation, phase separation, or significant gel strength development of mixtures should be evaluated carefully. Consideration should be given to redesigning the fluids to reduce or eliminate such behavior. If the fluids are redesigned, the entire compatibility testing protocol should be repeated for the redesigned fluids.

13.8.7 Spacer Surfactant Screening Test

13.8.7.1 Evaluate spacer/NAF mixes 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 should be considered inadequate.

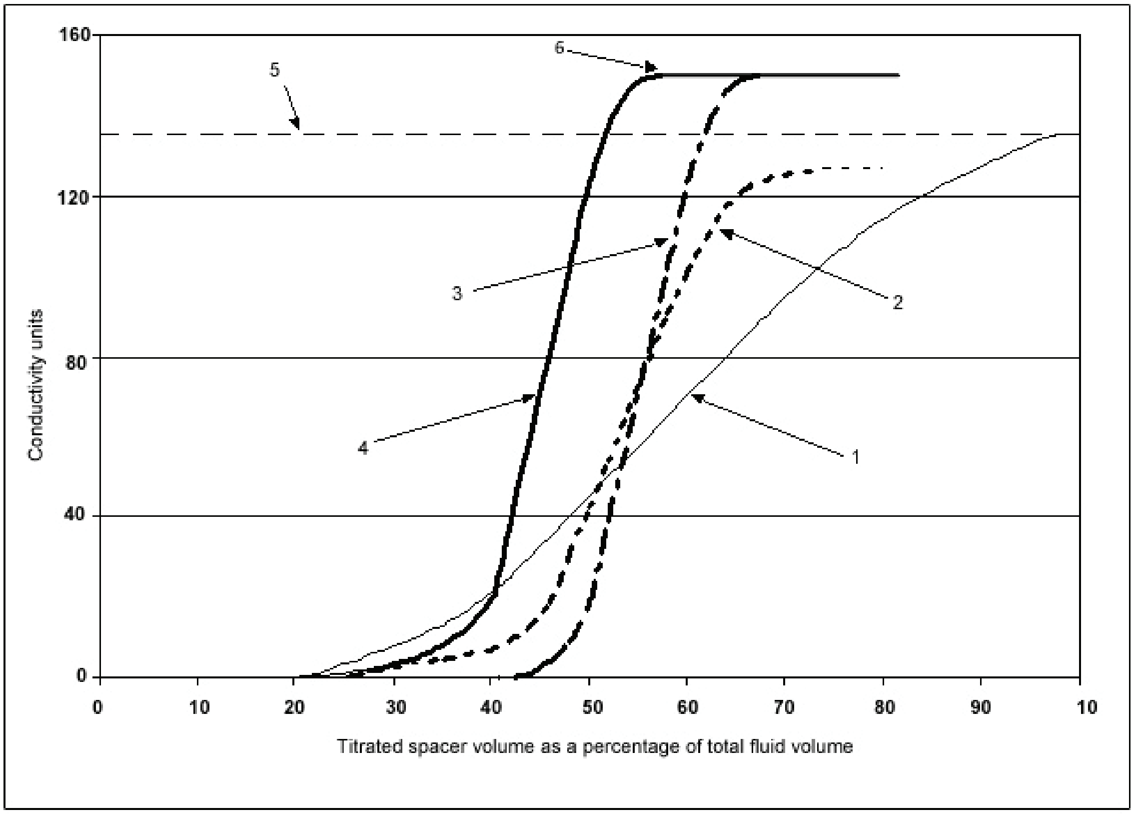
Spacer formulation "2" exhibits a much steeper slope than Spacer "1" but the endpoint (the plateau on the curve) is reached prior to reaching the spacer baseline. The performance of a fresh water spacer with this type of behavior should be considered poor.

NOTE Care should 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 Observe mixtures during the SSST. Note 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 Observe the behavior of the mix as cement slurry is being added to the mud/spacer mix. Consideration should 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 should 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 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 Percent in SSST Apparatus

13.8.7.4 Observe the fluid set aside following the SSST test [13.7.3 l)] after 30 min to 1 hr. If there are signs of phase separation or sedimentation, redesign the spacer.

14 Pozzolans

14.1 General

This section covers the recommended terminology, procedures, and properties for pozzolans used in well cements.

14.2 Types of Pozzolan

14.2.1 Pozzolan is defined in 3.1.32 and is further described by ASTM as siliceous and aluminous materials that, in themselves, possess little or no cementitious value but, in finely divided form at ordinary temperatures and in the presence of moisture, will chemically react with calcium hydroxide to form compounds possessing cementitious properties.

14.2.2 Class N pozzolans are naturally occurring materials such as volcanic ashes, tuffs, pumices, etc.

14.2.3 Fly ash is defined in 3.1.22 and is further described by ASTM as finely divided residue that results from the combustion of ground or powdered coal. Fly ash is called artificial pozzolan and is the pozzolan most commonly used in well cement formulations.

Class F fly ash is produced as the combustion residue from anthracite or bituminous coals. Class F fly ash has pozzolanic properties.

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 %.

14.3 Physical and Chemical Properties

14.3.1 The physical and chemical characteristics of pozzolans are listed in ASTM C618-08.

14.3.2 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).

a) Use a clean, dry 100 ml (TC type) graduated cylinder for the determination of both loose and packed apparent densities. Check the accuracy of the graduated cylinder by filling with 99.75 g of distilled water, which equals 100 ml volume at 23 °C (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 hand shake 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 Step 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. Record the packed volume (V_{fp}) of material directly from the cylinder graduations. Use this volume to calculate the packed apparent bulk density.

e) Calculate the bulk density in common field units as follows:

$$\rho_{\mathrm{LAB}} = \frac{1000}{100} \times m_{\mathrm{lfm}} \text{ (SI)} \tag{61}$$

or

$$\rho_{\mathrm{LAB}} = \frac{62.43}{100} \times m_{\mathrm{lfm}} \text{ (USC)} \tag{62}$$

and

$$\rho_{\text{PAB}} = \frac{1000}{V_{\text{fp}}} \times m_{\text{lfm}} \text{ (SI)} \tag{63}$$

or

$$\rho_{\text{PAB}} = \frac{62.43}{V_{\text{fp}}} \times m_{\text{lfm}} \text{ (USC)} \tag{64}$$

where

ρ_{LAB} is the loose apparent bulk density, expressed in kg/m^3 (lbm/ft^3);

ρ_{PAB} is the packed apparent bulk density, expressed in kg/m^3 (lbm/ft^3);

m_{lfm} is the mass of 100 ml of loosely filled material, expressed in g;

V_{fp} is the final packed volume attained from the original 100 ml of loosely filled material, expressed in ml.

f) Report average bulk density as the average of the loose and packed bulk densities.

$$\rho_{\text{bulk}} = (\rho_{\text{PAB}} + \rho_{\text{LAB}})/2 \tag{65}$$

where

ρ_{bulk} is the average bulk density, expressed in kg/m^3 (lbm/ft^3);

ρ_{LAB} is the loose apparent bulk density, expressed in kg/m^3 (lbm/ft^3);

ρ_{PAB} is the packed apparent bulk density, expressed in kg/m^3 (lbm/ft^3).

14.3.3 The relative density of a pozzolan should be measured using either a Le Chatelier flask, in accordance with ASTM C188-95, or a gas pycnometer. Certain pozzolans can contain particles with a relative density less than the kerosene or naphtha specified in ASTM C188-95. Suitable fluids with a relative density that prevents floating of these particles may be used. Use of a gas pycnometer is preferred for measuring the relative density of pozzolans containing unusually light particles.

14.4 Slurry Calculations

14.4.1 The terms bulk density, absolute density, and absolute volume must be understood to prevent confusion.

Bulk density and absolute density both have density units, that is, kg/m^3 (lbm/ft^3, lbm/gal), etc.

Bulk and absolute density values for Portland cement and other powdered materials are very different. Bulk density includes the air space around particles, thus it 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. 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).

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).

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 (m^3/kg, l/kg or gal/lbm).

Absolute density (or absolute volume) is used to calculate liquid slurry properties such as slurry density, slurry water requirement, and slurry yield. 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).

14.4.2 The absolute densities of the pozzolan and of the Portland cement are required to perform the following calculations. The manufacturer of the pozzolan or cement should supply the absolute density of the material (or the relative density to be used in calculating the absolute density as previously stated). The user should verify the absolute densities reported by the manufacturer.

14.4.3 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 14.4.6). These volumes are designated by a ratio of percentages such as 35:65. The first number refers to pozzolan and the second number refers to Portland cement. A 35:65 blend represents 35 % absolute volume pozzolan mixed with 65 % absolute volume cement. This designation is often not specific enough and should be further specified. For example, 35:65 Class F fly ash: Class G Cement would be a better designation. Since both fly ash and cement can come from any of a number of suppliers, and each will have a unique set of properties, the sources of the fly ash and cement should also be designated.

14.4.4 As a starting point for performing slurry calculations for pozzolan/Portland cement blends, a sack of cement is defined as 94 lbm of Portland cement.

14.4.5 The absolute volume of a sack of Portland cement (94 lbm) can vary depending upon the absolute density of the cement. The absolute 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 absolute density value for the selected cement should be used.

14.4.6 The following examples illustrate the calculations.

EXAMPLE 1 An example calculation, in USC units, for obtaining the absolute volume, $V_{\text{c,sk}}$, of a sack of Class G cement with an absolute density of 26.00 lbm/gal is as follows:

$$V_{\text{c,sk}} = \frac{94\ \text{lbm}}{26.00\ \text{lbm/gal}} = 3.62\ \text{gal}$$

Once the absolute volume of the sack of cement is known, then the pozzolan and Portland cement percentages of the absolute volume can be calculated.

EXAMPLE 2 Expanding on 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 of each material from the absolute density values of pozzolan and Portland cement. Expanding further on the example, 2.35 gal × 26.00 lbm/gal = 61.1 lbm of Portland cement. Pozzolan absolute 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 absolute 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 × 20.50 lbm/gal).

Combining the pounds of pozzolan and the pounds 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.

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 by weight of blend (% BWOB).

EXAMPLE 3 For this example, assume the blend has 6 % bentonite and 0.2 % retarder. The mass of bentonite per equivalent sack of blend is (6 %) × (87.1 lbm) = 5.23 lbm of bentonite per equivalent sack. The mass of retarder per equivalent sack of blend is (0.2 %) × (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) × (61.1 lbm/sk) = 7638 lbm of Portland cement would be required. For the same 125-sack job, (125 sk) × (2.0 lbm/sk) = 3250 lbm of pozzolan would be required. The masses of bentonite and retarder required are, respectively, (125 sk) × (5.23 lbm/sk) = 654.75 lbm and (125 sk) × (0.17 lbm/sk) = 21.25 lbm.

14.5 Bulk Volume of a Blend

14.5.1 The bulk volume of a blend of pozzolan and Portland cement will vary depending on the amount of small-particle packing between larger particles during the blending operation and on other factors such as humidity, vibration, time, air content, and the compacting force on the materials.

14.5.2 The bulk volume of the pozzolan:cement blend can be determined in the same manner as for the pozzolan as described in 14.3.2 a) to 14.3.2 f).

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 should 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.

15.2 Preparation of Cement Slurry

Test samples should be prepared according to Section 5, except that the temperature of the mix water, dry cement or cement blend, and mixing container should be representative of field mixing conditions. If field conditions are unknown, the cement blend and mixing container should be cooled to – 7 °C (20 °F) before mixing. Mix water should be cooled to 1 °C (34 °F), and the slurry temperature immediately after mixing should be recorded; 4 °C (40 °F) is common in some areas. Each of the above temperatures should be measured and reported on all tests.

15.3 Fluid Fraction

The fluid fraction should 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 should 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 should be followed with appropriate modifications for cold testing. Test conditions should be consistent with the anticipated conditions in the well or if wellbore conditions are unknown, performed at 4 °C (40 °F).

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.

NOTE There is no procedure for calibrating an atmospheric pressure consistometer potentiometer.

15.5 Compressive Strength

Specimens should be cured at – 7 °C (20 °F) and 4 °C (40 °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 should 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 should be prepared as described in Section 7.

Condition the slurry for 90 min in a consistometer at 4 °C (40 °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 (35 °F), whichever is 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 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 (40 °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 should be performed within 30 min of the time at which the desired age is reached. After testing, the residue, or an additional test specimen, should 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.

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 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 sequence (beginning the cycle on a Monday is convenient for most):

a) 48 hr at 4 °C (40 °F), days 1–2;

b) 24 hr at – 7 °C (20 °F), day 3;

c) 24 hr at 4 °C (40 °F), day 4;

d) 72 hr at 38 °C (100 °F), days 5–7;

e) 72 hr at 77 °C (170 °F), days 8–10;

f) 24 hr at 38 °C (100 °F), day 11;

g) 72 hr at – 7 °C (20 °F), days 12–14; and

h) raise to 4 °C (40 °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 at 4 °C (40 °F). Examine the cement cubes and test them for strength after 1 and 3 cycles (14 d and 42 d).

Annex A
(normative)

Procedure for Preparation of Large Slurry Volumes

A.1 General

This procedure should 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 should be the same as outlined in Section 5, except that the mixing device should be as described in this subsection.

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 should be a bottom-drive, blade-type blender. The mixing container and the mixing blade should be constructed of corrosion-resistant material. The mixing assembly should be constructed so that the blade can be separated from the drive mechanism. The mixing blade should 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 should 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 should 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 5.3.1.4.

A.3.3 Mixing Cement and Water

Place the mixing container with the required mass of mix water and any liquid additives (if previously added) on the blender base. Place the cover of the mixing container. Turn on the motor and maintain at "slow" speed, 4000 r/min, ± 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 should be added to the mix water while mixing at low speed. In certain cases, the order of addition of the additives to the mixing water can be critical, in which case the additives should 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, ± 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. Some slurry designs can require a longer time to completely wet the cement blend. However, the time used to add the blend should 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, ± 250 r/min, for 50 s, ± 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 calibration or verification according to the requirements of this API recommended practice. 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.

B.2 General

The following sections prescribe the requirements for interval and accuracy of calibration of devices used in a laboratory conducting tests according to this API recommended practice. Table B.1 lists the requirements by equipment type and Table B.2 lists the frequency and allowable tolerances.

Table B.1—Equipment Calibration Requirements

	Equipment												
Measurement Instrument	**Balance**	**Blender**	**HPHT Consistometer**	**Atmospheric Consistometer**	**Rheometer**	**Fluid Loss Cell**	**Ultrasonic Devices**	**Handheld Timers**	**Handheld Thermometers**	**Load Frame**	**Pressure Density Balance**	**Weight Sets**	**Chart (where Used)**
Mass	C											V	
Timer		V	V	V		V		V					C (speed)
Rotation		V	V/C	V/C	V								
Temperature measurement			C	C	C	C	C		C				C
Torsion device					C								
Consistency measurement			C										C
Pressure gauge			C			C	C						C
Ultrasonic transducer							C						
Auxiliary display			V	V									
Indication										C	C		

V—verify accuracy or tolerance; if outside requirement, adjust or replace.
C—calibrate by making adjustments or by creating calibration chart.
See Table B.2 for frequency and tolerances.

Table B.2—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 ± 1 % of reading for measurement made less than 10 g	Annually
Blender (1 l)	Verification	4000 r/min and 12,000 r/min	± 100 r/min @ 4000 r/min and ± 250 r/min @ 12,000 r/min	Annually
Large volume blender (4 l)	Verification	4000 r/min and 10,000 r/min	± 100 r/min @ 4000 r/min and ± 250 r/min @ 10,000 r/min	Annually
Blender blade mass	Verification	NA	Maximum 10 % loss	When replaced
Timers	Verification	2 min	within 1 s in 2 min	Annually
Chart speed	Calibration	1 hr	± 2 min in 1 hr	Annually
Temperature controlling systems and displays	Calibration	Three points spanning the range	± 1 °C (2 °F)	Quarterly
Thermocouple	Verification	Three points spanning the range	± 1 °C (2 °F)	Quarterly
Handheld thermometer or portable thermocouple	Calibration	Three points spanning the range	± 1 °C (2 °F)	Quarterly
Viscometer torsion measuring device	Calibration	Table B.3	Table B.3; 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
Consistometer cup 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

Equipment and Component/Function	Calibration/ Verification	Check Points	Tolerance	Frequency
Rheometer rotor speed	Verification	At each speed	± 1 % of nominal speed at speeds of 100 r/min and greater ± 1 r/min for speeds <100 r/min	Quarterly
Consistency measuring devices	Calibration	Table B.4	± 5 B_c or ± 0.5 VDC, as appropriate	Monthly and whenever it is adjusted or parts replaced
Sonic measuring system	Calibration	According to manufacturer's instructions	According to manufacturer's specifications	Monthly, and when cells, transducers, or cables are changed
Pressure measuring device—High range (>17,000 kPa or 2500 psi)	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
Pressure measuring device—Low range (up to 17,000 kPa or 2500 psi) NOTE Calibration of air pressure gauges is not required	Calibration	3500 kPa and 10,500 kPa (500 psi and 1500 psi)	± 300 kPa (± 50 psi)	Annually
Load frame	Calibration	At 9.0 kN (2000 lbf) and 25 %, 50 %, and 75 % of maximum	± 2 % of the indicated force or one minimum instrument scale division, whichever is greater	Annually
Cube molds	Verification	As per ASTM/C109M or EN 196-1	As per ASTM/C109M or EN 196-1	Once every two years
Pressurized density balance—Indicated density	Calibration	Water and at 1800 kg/m^3 to 2300 kg/m^3 (15 lbm/gal to 19 lbm/gal)	± 10 kg/m^3 (0.1 lbm/gal) from actual	With water monthly and at 1800 kg/m^3 to 2300 kg/m^3 (15 lbm/gal to 19 lbm/gal) annually
Weight sets	Calibration	Each piece	0.1 % of nominal value	Annually
Data acquisition devices	Verification	Same as T and P	Adjust or prepare calibration table	With sensor recorded

The individual performing the 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.

Calibration records shall contain, as appropriate:

- model name/number, serial number, and local identification number of the instrument;
- name, model number, and serial number of instrument used for the calibration;
- certification number, date of certification, and date next calibration against national standard is due for the calibration instrument;
- calibration inputs from the calibration instrument and the initial (before calibration) and final (calibrated) indications from instrument being calibrated;
- indication that instrument passed or failed each calibration point;
- condition found/condition left (e.g. out of or in calibration);
- adjustments made and results after adjustment;
- date of calibration;
- name and signature of calibration technician;
- 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 calibrated 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 calibration becomes due, the calibration remains valid for the duration of that test and the instrument should be calibrated at the end of that test.

B.3 Requirements

B.3.1 Balance

Balances shall be calibrated 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 ± 1 % 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.

B.3.2 Blender

B.3.2.1 Blender Speed

Blender display of rotational speed shall be calibrated no less frequently than annually. A one liter blender 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 11,750 r/min to 12,250 r/min (12,000 r/min ± 250 r/min). A 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 high speed range of 9750 r/min to 10,250 r/min (10,000 r/min ± 250 r/min).

The indicated speed shall not deviate by more than 250 r/min at the low speed and 250 r/min at the high speed from the true speed observed using a calibrated tachometer. Note that the ability of the controller to maintain speeds within the required range is not a calibration issue. If the blender is unable to control within the required range, the blender motor or controller shall be repaired or replaced, as appropriate.

B.3.2.2 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. 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 system 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 nonhardened blade.

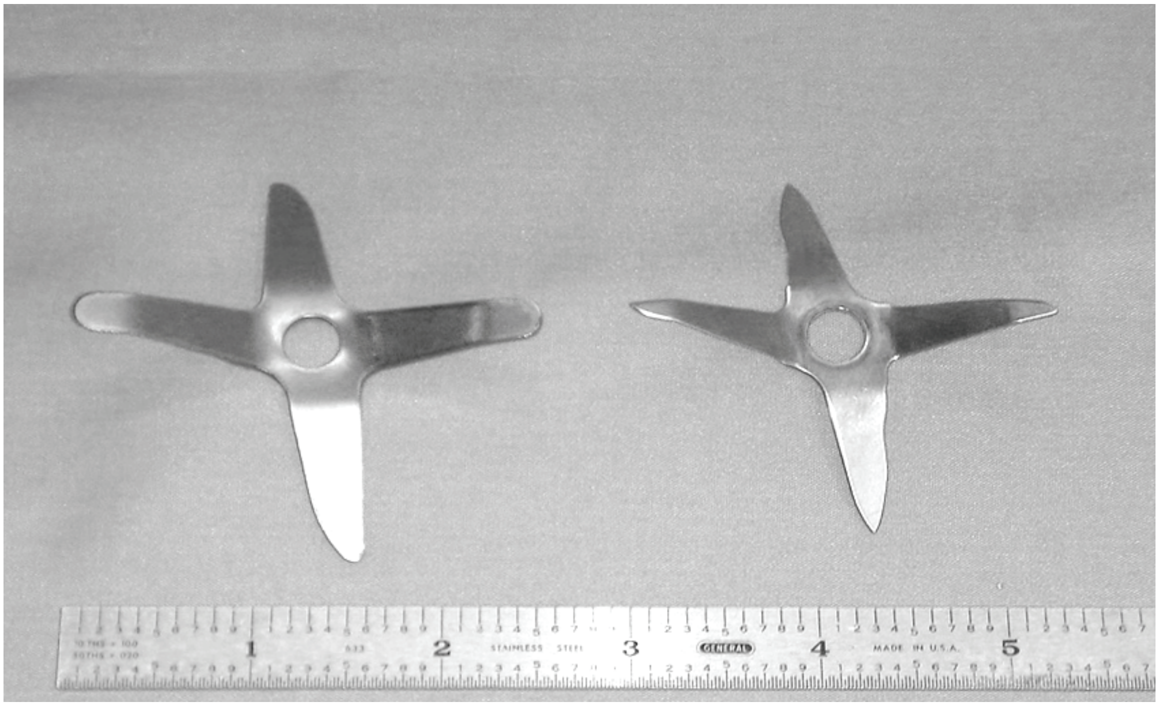


Figure B.1—Worn Blade (right) Compared to a New One (left)

B.3.3 Timers

Timers on all instruments shall be calibrated no less frequently than annually. This includes timers on consistometers, blender controllers, handheld timers and any other timer, whether as part of an instrument or separate. Accuracy shall be within 1 s in 2 min.

B.3.4 Recorders

Charts used for recording thickening times or other data 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. The chart on which this recording is made shall be maintained with the calibration record for the instrument.

B.3.5 Temperature Devices

B.3.5.1 Temperature measuring and controlling devices shall be calibrated no less frequently than quarterly. This includes thermometers, 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 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). 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 should 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 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 calibration 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 should 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 See B.3.14 for temperature data acquisition.

B.3.5.5 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 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 calibration of temperature-measuring systems and controllers requires a millivolt source, the correct connecting thermocouple extension cable for the type 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 in B.3.5.5.2 to certify the controlling or indicating instrument is accurately calibrated.

B.3.5.5.3.2 Procedure

Follow the manufacturer’s procedure for calibrating temperature-measuring systems and controllers. 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.3.6 Viscometer Torsion Measuring Systems

Viscometer torsion measuring systems shall be calibrated 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. Calibration shall be no less frequently than quarterly or whenever a spring or bearing is installed, changed, or adjusted in the instrument. Accuracy shall be as indicated in Table B.3.

Table B.3—Rheometer Calibration

Mass (g)	**Allowable Tolerance in Dial Deflection for Indicated Spring Constant**			
	0.2	**0.5**	**1.0**	**2.0**
0	0 ± 0.5	0 ± 0.5	0 ± 0.5	0 ± 0.5
10	127 ± 2	51 ± 1	25 ± 1	12.5 ± 1
20	254 ± 2	102 ± 2	51 ± 1	25 ± 1
50		254 ± 2	127 ± 2	64 ± 1
100			254 ± 2	127 ± 2
200				254 ± 2

B.3.7 Instrument Rotation

B.3.7.1 Rotation of consistometer cups (or paddles) and of rheometer rotors shall be calibrated no less frequently than quarterly.

B.3.7.2 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.3.7.3 Each rheometer 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.3.8 Consistency Measurement Device or Potentiometer

The potentiometer or other consistency-measuring device shall be calibrated no less frequently than monthly. A new calibration is required whenever repairs or adjustments are made to the device. Accuracy shall be maintained within ± 5 B_c (if output is in B_c) or ± 0.5 volt (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 should 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.

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.4. Consistency values shall be reported based on the calibration curve or table.

A weight-loaded device (Figure B.2) is used to produce a series of torque-equivalent values for consistency, defined by:

$$T = 78.2 + 20.02 \times B_\mathrm{c} \tag{B.1}$$

where

T is torque, expressed in gram-centimeters (g·cm);

B_c is consistency expressed in Bearden units.

Weights are used to apply the 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). Operating instructions from the manufacturer should be followed for proper calibration.

Figure B.2—Common Calibrating Device for Pressurized Consistometer Potentiometer

Table B.4—Slurry Consistency vs Equivalent Torque (for Potentiometer with Radius of 52 mm ± 1 mm)

Torque Equivalent (g·cm)	**Mass of Added Weights** (g ± 0.1)	**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

For a potentiometer with a radius other than (52 ± 1) mm, an adaptor ring with a radius of (52 ± 1) mm or an appropriate table of equivalent tolerances is required.

NOTE A consistency reading of a potentiometer may vary no more than ± 5 B_c from the slurry consistency shown in this table.

B.3.9 Ultrasonic Devices

Transducers, cables, and slurry cells must 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.3.10 Pressure Gauges

B.3.10.1 Pressure gauges shall be calibrated no less frequently than annually using a deadweight tester or a master gauge. The term gauge includes pressure-sensing transducers.

B.3.10.2 Gauges designed to measure pressures greater than 17,000 kPa (2500 psi) 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.3.10.3 Gauges designed to measure pressures up to 17,000 kPa (2500 psi) shall be calibrated with allowable error of ± 300 kPa (± 50 psi) at a minimum gauge reading of ± 3500 kPa (± 500 psi) and at a gauge reading of ± 10,500 kPa (± 1500 psi) or the maximum allowable working pressure of the equipment on or with which it is used.

B.3.10.4 Calibration of gauges showing pressure of air used to operate pumps and purge pressure vessels of liquid is not required.

B.3.11 Load Frame

The load frame used to measure break force of cement specimens shall be 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 Pressurized Fluid Density Balances

B.3.12.1 Standard Procedure

Pressurized fluid density balances shall be calibrated annually in the range 1800 kg/m^3 to 2300 kg/m^3 (15 lbm/gal to 19 lbm/gal) and with water no less frequently than monthly. Tolerance shall be within ± 10 kg/m^3 (± 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 can 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 water, place lid on cup, pressurize it, and check it for accuracy at 1.0 specific gravity (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 specific gravity (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 water.

h) Replace the lid, pressurize the cup as before, and check the density. The density should indicate 2.0 specific gravity (16.7 lbm/gal) if it is in calibration at the higher density. Record the indication of the balance.

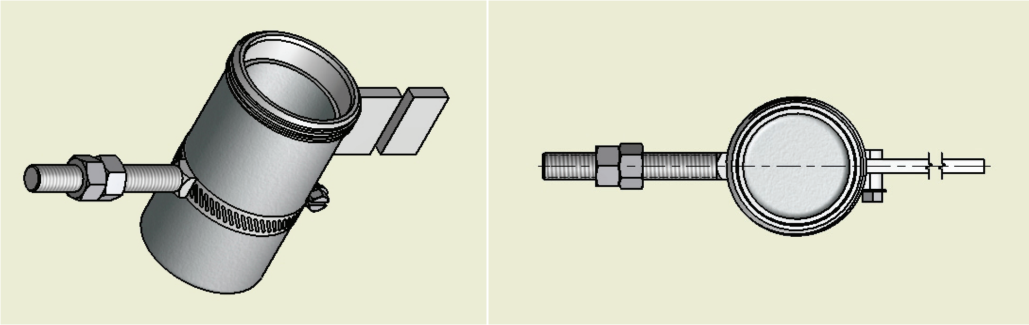


Figure B.3—Fixture for Calibration of Upper Density Range

B.3.12.2 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 water, place the lid on the cup, pressurize it, and check for accuracy at 1.0 specific gravity (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 specific gravity (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 specific gravity (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 should be 2.0 specific gravity (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.3.13 Weight Sets

Weight sets should be calibrated against certified weights traceable to a national standard. Weight sets to be calibrated include, but are not limited to, those used to calibrate rheometer springs, balances, and consistometer potentiometers or their equivalent. Weight sets should be calibrated no less frequently than once annually. Weights should conform within ± 0.1 % of the nominal weight.

B.3.14 Data Acquisition

B.3.14.1 Data acquisition can be by chart recorder or by electronic recording, such as computer data acquisition, or both.

B.3.14.2 For devices with chart recorders, the indication of temperature and pressure should be recorded during the calibration. 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 should be maintained with the instrument and appropriate corrections made to charts of all tests conducted with the instrument. The chart should be attached to the calibration record. The use of chart recorders is discouraged due to their inaccuracy.

B.3.14.3 Electronic data recording should 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.3.10), corrections should be made. For dedicated data acquisition systems, the data acquisition system should conform to the limits prescribed above for the data they are designed to record.

Annex C

(normative)

Alternative Apparatus for Well-simulation Thickening-time Tests

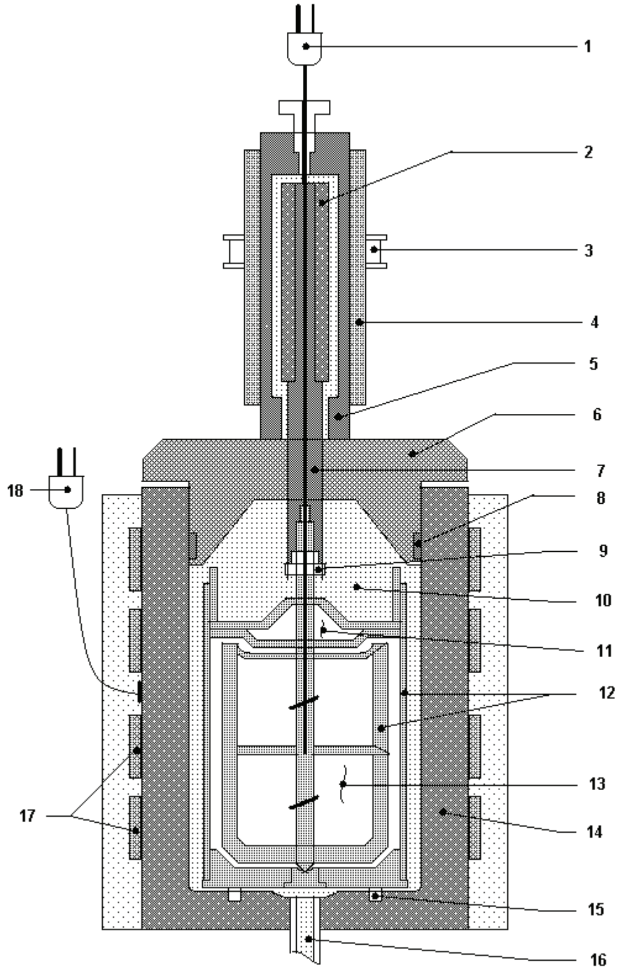
C.1 General

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 10A, Section 10. The rotating paddle shall conform to the dimensions defined in API 10A, Section 10, 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 Figure 10 and Figure 11 of API 10A or any system suitable of isolating the test fluid from the pressurizing medium.

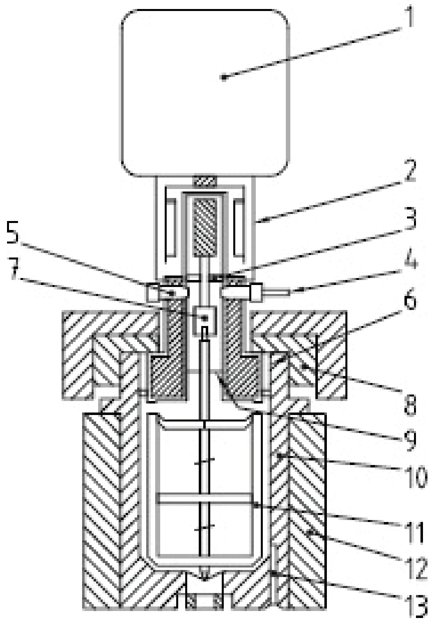
Paddle torque is sensed by motor load or alternate torque sensors to provide slurry consistency measurements equivalent to those of the typical consistometer described in 9.2. Slurry temperature and pressure controls shall be provided. General schematics of alternative configurations are shown in Figure 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
2	magnetic drive
3	pressure-transmitting seal
4	pressurizing port
5	air vent
6	auxiliary thermocouple
7	mechanical drive coupling
8	auxiliary heater jacket
9	oil/cement interface
10	slurry pressure vessel
11	rotating paddle
12	main heating/cooling jacket
13	main thermocouple well

Figure C.2—Alternative Consistometer Design for Well-simulation Thickening Time, Example 2

C.3 Calibration

Apparatuses are 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, should 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 should be followed and should 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 (36) at depths greater than 3050 m with SI units and using Equation (37) at depths greater than 10,000 ft with USC units.

Table D.2—T_{PSP} for Squeeze-cementing 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	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 (49) with SI units and calculated using Equation (50) with USC units.

NOTE 2 Hesitation squeeze schedules: After the final squeeze pressure (p_{FSQ}) is reached, the temperature should be increased to static temperature T_{BHS} at 0.11 °C (0.2 °F) per minute. At this same time the stirring should be cycled off-on, typically for 10 min off and 5 min on until the test is terminated.

NOTE 3 T_{PSP} should 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.

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