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Erdöl- und Erdgasindustrie – Zemente und Materialien für die Zementation von Tiefbohrungen – Teil 2: Prüfung von Bohrloch-Zemente (ISO 10426-2:2003 + Amd. 1:2005); Englische Fassung EN ISO 10426-2:2003 + A1:2005

Petroleum and natural gas industries – Cements and materials for well cementing – Part 2: Testing of well cements (ISO 10426-2:2003 + Amd. 1:2005); English version EN ISO 10426-2:2003 + A1:2005

Industries du pétrole et du gaz naturel – Ciments et matériaux pour la cimentation des puits – Partie 2: Essais de ciment pour puits (ISO 10426-2:2003 + Amd. 1:2005); Version anglaise EN ISO 10426-2:2003 + A1:2005

Gesamtumfang 180 Seiten

Normenausschuss Erdöl- und Erdgasgewinnung (NÖG) im DIN

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Nationales Vorwort

Diese Europäische Norm wurde vom Technischen Komitee CEN/TC 12 „Materialien, Ausrüstungen und Offshore-Bauwerke für die Erdöl- und Erdgasindustrie“ (Sekretariat: Frankreich) erstellt. Es handelt sich dabei um die unveränderte Übernahme von EN ISO 10426-2:2003 einschließlich der Änderung 1:2005, erarbeitet von ISO/TC 67 „Materialien, Ausrüstungen und Offshore-Bauwerke für die Erdöl- und Erdgasindustrie; Unterkomitee SC 3, Bohrspülungen, Komplettierungsflüssigkeiten und Zemente“ (Sekretariat: Norwegen).

Für Deutschland hat hieran der Arbeitskreis NÖG 12/67 AK 3 „Bohrspülung und Zemente“ im Normenausschuss Erdöl- und Erdgasgewinnung (NÖG) mitgearbeitet.

Diese Europäische Norm enthält unter Berücksichtigung des DIN-Präsidialbeschlusses 13/1983 nur die englische Originalfassung der ISO-Norm.

Die Änderung (ISO 10426-2:2003/Amd.1:2005 übernommen als EN ISO 10426-2:2003/A1:2005) wurde in diese Ausgabe eingearbeitet und durch einen senkrechten Strich am Rand im Text gekennzeichnet. Hierbei handelt es sich um die zusätzlich aufgenommenen Abschnitte 3.1.52 und 16.8.

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Industries du pétrole et du gaz naturel — Ciments et matériaux pour la cimentation des puits — Partie 2: Essais de ciment pour puits (ISO 10426-2:2003 + A1:2005)

Erdöl- und Erdgasindustrie — Zemente und Materialien für die Zementation von Tiefbohrungen — Teil 2: Prüfung von Bohrloch-Zemente (ISO 10426-2:2003 + A1:2005)

This European Standard was approved by CEN on 1 October 2003.

This admentment A1 modifies the European Standard EN ISO 10426-2:2003; it was approved by CEN on 13 August 2005.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

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Foreword

This document (EN ISO 10426-2:2003) has been prepared by Technical Committee ISO/TC 67 "Materials, equipment and offshore structures for petroleum and natural gas industries" in collaboration with Technical Committee CEN/TC 12 "Materials, equipment and offshore structures for petroleum and natural gas industries", the secretariat of which is held by AFNOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by April 2004, and conflicting national standards shall be withdrawn at the latest by April 2004.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and the United Kingdom.

Endorsement notice

The text of ISO 10426-2:2003 has been approved by CEN as EN ISO 10426-2:2003 without any modifications.

Foreword Amendment 1

This document (EN ISO 10426-2:2003/A1:2005) has been prepared by Technical Committee ISO/TC 67 "Materials, equipment and offshore structures for petroleum and natural gas industries" in collaboration with Technical Committee CEN/TC 12 "Materials, equipment and offshore structures for petroleum and natural gas industries", the secretariat of which is held by AFNOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by March 2006, and conflicting national standards shall be withdrawn at the latest by March 2006.

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Endorsement notice

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Introduction

This part of ISO 10426 is based on API RP 10B, 22nd edition, December 1997, addendum 1, October 1999.

Users of this part of ISO 10426 should be aware that further or differing requirements may be needed for individual applications. This part of ISO 10426 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 part of ISO 10426 and provide details.

In this part of ISO 10426, where practical, US Customary units are included in brackets for information.

Well cement classes and grades are defined in ISO 10426-1.

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1 Scope

This part of ISO 10426 specifies requirements 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.

ISO 10414-1, *Petroleum and natural gas industries — Field testing of drilling fluids — Part 1: Water-based fluids*

API RP 13J, *Testing of heavy brines (second edition), March 1996*

ASTM C 109, *Standard test method for compressive strength of hydraulic cement mortars (using 2 in. or [50 mm] cube specimens)*

ASTM C 188, *Standard test method for density of hydraulic cement*

3 Terms, definitions and symbols

3.1 Terms and definitions

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

3.1.1
absolute volume
reciprocal of absolute density

NOTE It is expressed as volume per unit mass.

3.1.2
additive
material added to a cement slurry to modify or enhance some desired property

NOTE Common properties that are modified include: setting time (by use of retarders or accelerators), fluid loss control, viscosity, etc.

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3.1.3
annulus
space surrounding the pipe in the wellbore

NOTE The outer wall of the annular space may be either surface or casing

3.1.4
assumed surface temperature
T_{AS}
assumed temperature at surface used for calculating a pseudo-temperature gradient

3.1.5
batch mixing
process of mixing and holding a volume of cement slurry prior to placement in the wellbore

3.1.6
Bearden units of consistency
units used to express consistency of a cement slurry when determined on a pressurized consistometer

NOTE The symbol for consistency when expressed in Bearden units is B_C.

3.1.7
blowout
point in time at which nitrogen flows through the sample in a fluid loss test

3.1.8
bulk density
mass per unit volume of a dry material containing entrained air

3.1.9
casing cementing
complete or partial annular cementing of a full casing string

3.1.10
cement
Portland cement
ground clinker generally consisting of hydraulic calcium silicates and aluminates and usually containing one or more of the forms of calcium sulfate as an interground addition

NOTE 1 Hydraulic calcium silicates and aluminates are those which harden under water.

NOTE 2 Interground additions are added before grinding, rather than after grinding.

3.1.11
cement class
cement type
designation achieved using the ISO system of classifications of well cement in accordance with its intended use

NOTE See ISO 10426-1 for further information.

3.1.12
cement grade
designation achieved using the ISO system for denoting the sulfate resistance of a particular cement

NOTE See ISO 10426-1 for further information.

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3.1.13
cement blend
mixture of dry cement and other dry materials

3.1.14
clinker
fused materials from the kiln in cement manufacturing that are interground with calcium sulfate to make 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 crush it

NOTE It is expressed as force per unit area.

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

3.1.18
continuous-pumping squeeze-cementing operation
squeeze-cementing operation that does not involve cessation of pumping

3.1.19
equivalent sack
mass of the blend of Portland cement and fly ash or pozzolan that has the same absolute volume as 42,63 kg (94 lbs) of Portland cement

3.1.20
filtrate
liquid that is forced out of a cement slurry during a fluid loss test

3.1.21
fly ash
powdered residue from the combustion of coal having pozzolanic properties

NOTE See Clause 17 for further description.

3.1.22
free fluid
coloured or colourless liquid which has separated from a cement slurry

3.1.23
freeze-thaw cycle
test involving a cement sample that is alternately exposed to temperatures above and below the freezing point of water

3.1.24
hesitation-pumping squeeze-cementing operation
squeeze-cementing operation that incorporates discontinuous pumping of the cement slurry

NOTE The slurry is placed into the well, the pumps are stopped for some period of time, then a volume of slurry is again pumped. The process is repeated until a predetermined pressure is reached or the volume of cement slurry has been completely pumped.

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3.1.25
heat-up rate
R_h
rate of slurry temperature change on going from the surface temperature, T_{SS}, to the predicted bottom-hole circulating temperature, T_{PBHC}

3.1.26
liner cementing
annular cementing operations for which the top of the casing being cemented is not at the top the wellbore

3.1.27
mud
fluid that is circulated through the wellbore during drilling or workover operations

3.1.28
mud balance
beam-type balance used to measure fluid density at atmospheric pressure

3.1.29
neat cement slurry
cement slurry consisting of only cement and water

3.1.30
pressure-down rate
R_{pd}
rate at which pressure is reduced from the bottom-hole pressure, p_{BH}, to the pressure at the top of cement column, p_{TOC}, during a thickening-time test

3.1.31
permeability
measure of the capacity of a porous medium to allow flow of fluids or gases

NOTE Permeability is usually expressed in millidarcy, mD.

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 which in finely divided form reacts with calcium hydroxide to form a cementitious material

NOTE See Clause 17 for further description.

3.1.34
preflush, noun
fluid containing no insoluble weighting agents used to separate drilling fluids and cementing slurries

3.1.35
pressure vessel
vessel in a consistometer into which the slurry container is placed for the thickening-time test

3.1.36
pressurized curing vessel
vessel used for curing a sample of cement under temperature and pressure for compressive strength testing

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3.1.37
pressure-up rate
R_{pu}
rate at which pressure is increased from the starting pressure to the bottom-hole pressure during a thickening-time test

3.1.38
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.

3.1.39
sedimentation
separation and settling of solids in a cement slurry

3.1.40
slurry container
container in a pressurized consistometer used to hold the slurry for conditioning purposes or for thickening-time test

3.1.41
sonic strength
extent of strength development of a cement sample calculated by measuring the velocity of sound through it

NOTE The calculation is based on specific mathematical correlations and not on direct measurements of strength.

3.1.42
starting pressure
p_S
initial pressure applied to the test sample at the beginning of the thickening-time test

NOTE p_S is also used to determine the pressure-up rate.

3.1.43
spacer
fluid containing insoluble weighting materials that is used to separate drilling fluids and cementing slurries

3.1.44
squeeze-cementing
remedial process in which cementing material is forced under pressure into a specific portion of the well such as a fracture or opening

3.1.45
static fluid loss test
test to determine fluid lost from a cement slurry when placed against a 325 mesh screen at 6 900 kPa (1 000 psi) differential pressure

3.1.46
static stability test
test to determine the degree of sedimentation and free fluid development in a cement slurry

3.1.47
stirred fluid-loss cell
cell specially designed to allow for conditioning of the cement slurry within the same cell used to perform a static fluid loss test

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3.1.48
strength retrogression
reduction in compressive strength and increase in permeability of a cement caused by exposure to temperatures exceeding 110 °C (230 °F)

3.1.49
thickening time
time required for a cement slurry to develop a selected Bearden consistency value

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.

3.1.50
weigh batch mixer
scale tank
device or system for the weighing and blending of cement with dry additives

3.1.51
well simulation test
test whose parameters are designed and modified as required to simulate the conditions found in a wellbore

3.1.52
water-wetting capability
capability of a fluid to alter the quality or state of being water wetted

Note A fully water-wet state is considered most desirable to provide cement bonding.

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3.2 Symbols

For the purposes of this part of ISO 10426, the symbols given in Table 1 apply. This list is non-exhaustive.

Table 1 — Symbols

Symbol	Meaning
h_{TOCTVD}	top-of-cement true vertical depth
p_{BH}	bottom-hole pressure[b]
p_S	starting pressure
p_{TOC}	top-of-cement pressure
T_{AS}	assumed surface temperature
T_{BHC}	bottom-hole circulating temperature[a]
T_{BHS}	bottom-hole static temperature
T_{PBHC}	predicted bottom-hole circulating temperature
T_{MRBHS}	maximum recorded bottom-hole temperature after a static period
T_{MNRBHC}	minimum recorded bottom-hole temperature after sufficient circulation in the well to obtain a stabilized or steady-state temperature
T_{PS}	predicted squeeze temperature
∇_{PT}	pseudo-temperature gradient[c]
T_{PU}	pseudo-undisturbed temperature
T_{RS}	recorded squeeze temperature
T_{SS}	slurry surface temperature
T_{TOCC}	top-of-cement circulating temperature
T_{TOCS}	top-of-cement static temperature
T_{TOC}	top-of-cement column temperature
T_{UF}	undisturbed formation temperature
t_a	time to displace the leading edge of the cement slurry from bottom of the casing to the top of the annular cement column
t_d	time to displace the leading edge of cement slurry to the bottom of the wellbore or other predetermined location in the well

a The T_{BHC} can vary with time, fluid being circulated, pump rate, pipe size, etc.

b Hydrostatic pressure at the bottom of the well, calculated from the true vertical depth and the fluid densities in the wellbore.

c Gradient in °C/100 m (°F/100 ft), calculated from the difference between the maximum recorded bottom-hole static temperature (T_{MRBHS}) and the T_{AS}.

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4 Sampling

4.1 General

For cement blends, the purpose for which samples are taken shall be considered. In many cases, samples of the cement, cement blend, solid and liquid additives, and mixing water may be required to test a slurry in accordance with this part of ISO 10426. The best available sampling technology shall be employed to ensure accurate samples are taken. Some commonly used sampling techniques are described in this clause.

NOTE API documents prior to API RP10B, 22nd Edition, December 1997 have dealt only with sampling unblended cement in accordance with the procedure outlined in ASTM C 183.

4.2 Sampling cement at field location

When sampling from bulk tanks, transport containers or sacks, the cement shall be dry and uniform. Multiple samples shall be extracted using a suitable device (Figure 1). A composite of the samples shall be prepared, packaged and labelled (see 4.7). Average sample volume shall be 8 l to 20 l. Suggested sampling procedures are also outlined in ASTM C 183.

4.3 Sampling cement blends at field location

Cement blends may be sampled from the weigh batch mixer (scale tank), bulk transport or extracted from the flow lines during transfer. The cement and dry additives shall be thoroughly blended prior to sampling. This can be done by transferring the cement (air blowing) from the weigh batch mixer to some other container three to six times. 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). The samples shall be prepared, packaged, and labelled (4.7). Sample volume shall be sufficient to perform the desired testing.

4.4 Sampling dry cement additives at field location

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

4.5 Sampling liquid cement additives at field location

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

4.6 Sampling mixing water

The mixing water shall be sampled from the source. The sample shall be extracted in such a way as to avoid contamination. The sample shall be packaged and labelled (4.7). The sample volume shall be sufficient to perform the desired testing.

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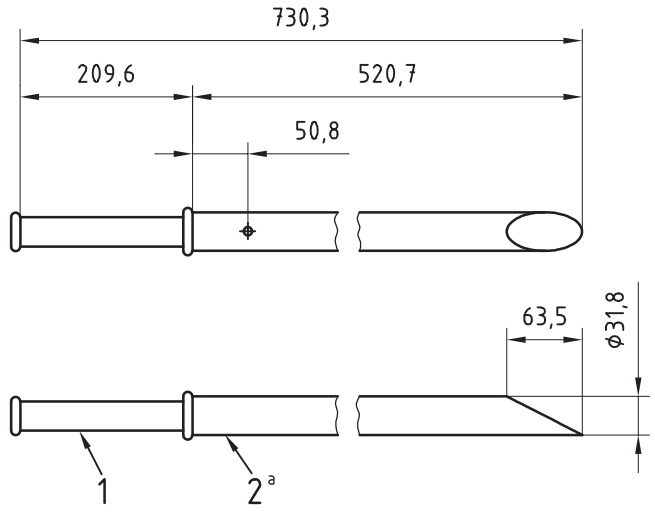
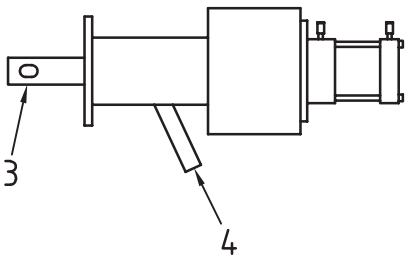
Dimensions in millimetres

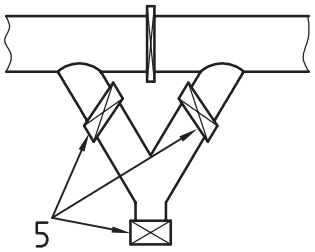


a) Tube sampler for sacked cement

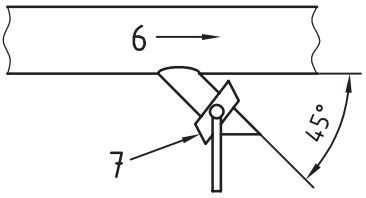
b) Tube sampler for bulk cement



c) Automatic probe sampler



d) Modified diverted-flow sampler



e) Top view – Lateral sampler

Key

1 hardwood handle
2 Dragg tubing
3 sample tube extended
4 product discharge
5 2,54 cm (1 in) ball valve
6 flow direction
7 2,54 cm (1 in) ball valve

[a] Approximate volume = 320 ml

Figure 1 — Commonly used sampling devices

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4.7 Shipping and storage

Test samples shall be packaged promptly in clean, airtight, moisture-proof containers suitable for shipping and long-term storage. The containers shall be lined metal, plastic, or some other heavy-walled flexible or rigid material to assure maximum protection. Re-sealable plastic bags may be used provided the bag is placed in a protective container prior to shipping to prevent puncturing, and to contain all material that may leak out during shipping. Ordinary cloth sacks, cans or jars shall not be used. Shipping in glass containers is not recommended.

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

4.8 Sample preparation prior to testing

Upon arrival at the testing location the samples shall be closely examined to ensure they have remained sealed during shipment and are not contaminated. Each sample shall be thoroughly blended just prior to slurry preparation. (Clause 5)

For storage, each sample shall be transferred into a suitable, leak-proof container (if one has not been used in shipping), properly labelled and dated, and stored in a dry place where room temperatures remain fairly constant. At the time of testing, each sample shall be examined for quality and thoroughly blended just prior to slurry preparation.

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

4.9 Sample disposal

Sample disposal shall comply with all regulatory requirements.

5 Preparation of slurry

5.1 General

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

The procedure described in this Clause is recommended for the laboratory preparation of slurries that require no special mixing conditions. If large slurry volumes are needed, the alternative method for slurry preparation given in Annex A may be used.

5.2 Apparatus

5.2.1 **Electronic balances**, with an accuracy of within ± 0,1 % of the indicated load.

Balances shall be calibrated frequently enough to ensure accuracy, and at least annually.

5.2.2 **Mechanical balances**, with weights having an accuracy within ± 0,1 % of the weight indicated.

5.2.3 **Mixing device**, of capacity 1 litre (1 quart), having bottom drive and a blade-type mixer.

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Examples of mixing devices in common use for preparation of slurries are shown in Figure 2. The mixing container and the mixing blade shall be constructed of corrosion-resistant material (Figure 3). The mixing assembly shall be constructed so that the blade can be separated from the drive mechanism.

The mixing blade shall be separated from the mixing assembly and weighed prior to use and replaced with a new blade when 10 % mass loss has occurred. The blade shall also be visually inspected for damage prior to each use and replaced as necessary.

If the mixing device leaks at any time during the mixing procedure, the contents shall be discarded, the leak repaired and the procedure restarted.

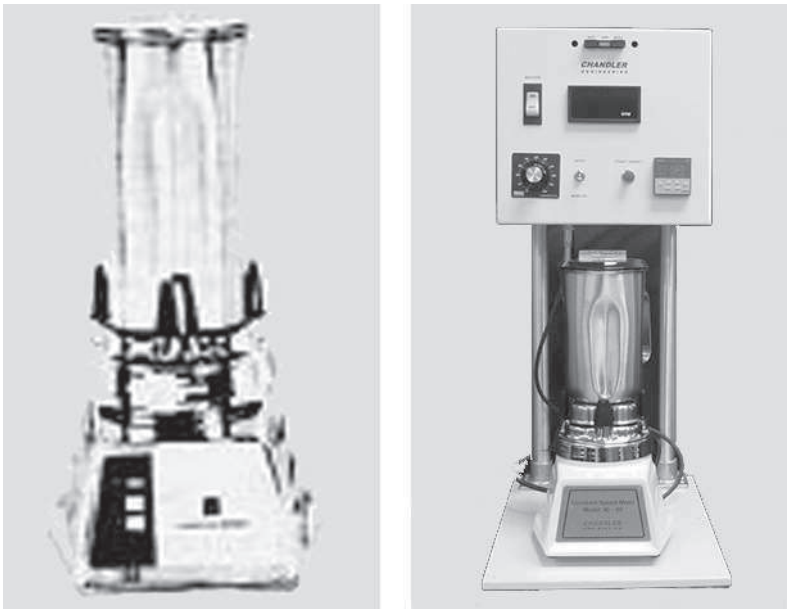

Figure 2 — Common mixing devices

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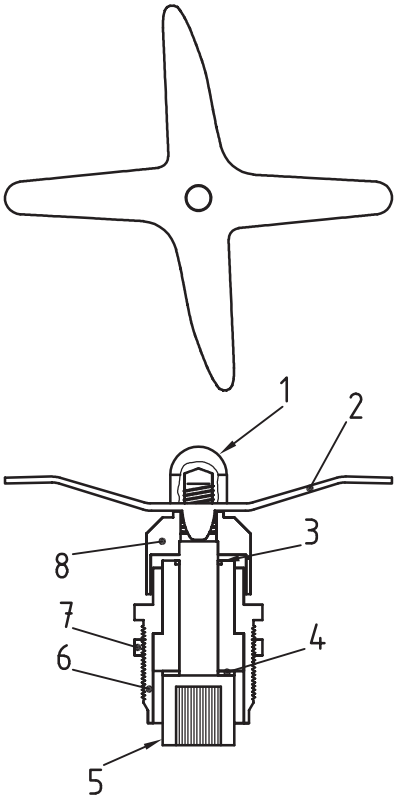

Key

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

Figure 3 — 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 changes in the composition of the raw materials used in the manufacturing process. Studies have shown that cement relative density may vary from 3,10 to 3,25. This variability could result in deviation of slurry densities by as much as 0,033 kg/l for slurries with constant water-to-solids ratio. 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.

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5.3.1.2 Relative density of cement and dry additives

The relative density of the cement and any dry additives may be determined by the use of a Le Chatelier flask as outlined in ASTM C 188. Alternatively, a pycnometer 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 shall be determined by the use of a hydrometer as outlined in API RP 13J. Alternatively, a pycnometer may be used for determining the relative density of these materials.

5.3.1.4 Laboratory density and volume calculations

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 following formulas. Alternative, suitable equations may also be used to calculate laboratory blend requirements.

For the purpose of these calculations, assume that relative density is equal to density expressed in grams per millilitre.

$$V_s = V_c + V_w + V_a$$

$$m_s = m_c + m_w + m_a$$

$$\rho_s = m_s / V_s$$

where

V_s is the slurry volume, in millilitres;

V_c is the cement volume, in millilitres;

V_w is the water volume, in millilitres;

V_a is the additive volume, in millilitres;

ρ_s is the density of slurry, in grams per millilitre;

m_s is the slurry mass, in grams;

m_c is the cement mass, in grams;

m_w is the water mass, in grams;

m_a is the additive mass, in grams;.

$$V_c = m_c / \rho_c$$

where ρ_c is the density of cement, in grams per millilitre;

$$V_w = m_w / \rho_w$$

where ρ_w is the density of water, in grams per millilitre;

$$V_a = m_a / \rho_a$$

where ρ_a is the density of additive, in grams per millilitre.

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5.3.2 Temperature of water and cement

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

5.3.3 Mix water

Water composition can affect slurry performance. Water from the field source shall be used. If field mix water is unavailable, a water of similar composition shall be used. If field mix water composition is unknown, deionized, distilled, or tap water may be used. The mix water and any liquid additives shall be weighed into a clean, dry, mixing container. No excess water shall be added to compensate for evaporation or wetting.

5.3.4 Mixing of cement and water

Weigh dry materials and then blend thoroughly and uniformly prior to adding them to the mixing fluid. Place the mixing container with the required mass of mix water and any liquid additives on the mixer base. Turn on the motor and maintain at 4 000 r/min ± 200 r/min (66,7 r/s ± 3,3 r/s). 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 certain cases, the order of addition of the additives to the mixing water can be critical. Document any special mixing procedures and mixing time. Add the cement or cement/dry additive blend at a uniform rate, in not more than 15 s if possible. Some slurry designs may take longer to completely wet the cement blend, however, the time used to add the blend shall be kept at a minimum. When all the dry materials have been added to the mix water, place the cover on the mixing container and continue mixing at 12 000 r/min ± 500 r/min (200 r/s ± 8,3 r/s) for 35 ± 1 s. Measure and document the rotational speed under load.

6 Determination of slurry density

6.1 Preferred apparatus

The preferred method for measuring the density of a cement slurry is by using the pressurized fluid density balance. The pressurized fluid density balance is similar in operation to the conventional mud balance, the difference being that the slurry can be placed in a fixed volume sample cup under pressure.

The purpose of placing the sample under pressure is to minimize the effect of entrained air upon slurry density measurements. A major problem found in the measurement of cement slurry densities is that often these fluids have a considerable amount of air entrained when initially mixed. By pressurizing the sample cup, any entrained air is decreased to a negligible volume, thus providing a slurry density measurement more closely in agreement with that which will be found under downhole conditions.

6.2 Calibration

The calibration of the apparatus shall be verified by placing water or fluids of known density in the sample cup or by using manufacturer-specified weights for equivalent densities placed in the sample cup.

6.3 Procedure

6.3.1 The sample cup shall be filled to a level slightly below the upper edge of the cup [6 mm ± 0,5 mm (1/4 in)].

6.3.2 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.

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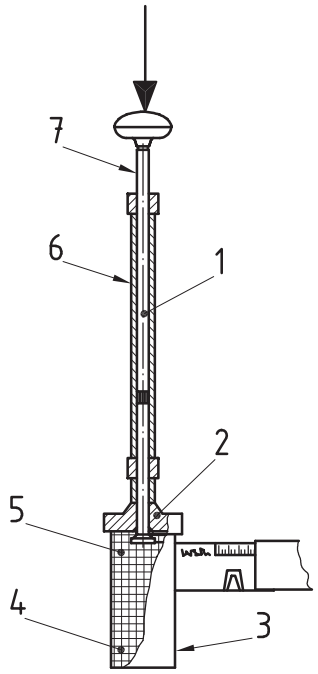
When the lid has been placed on the cup, pull the check valve up in the closed position, rinse off the cup and threads with water, and screw the threaded cap on the cup.

6.3.3 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 inward position. Then draw the piston rod upward, thereby filling the pump cylinder with slurry.

6.3.4 Push the nose of the pump onto the mating O-ring surface of the check valve. Pressurize the sample cup by maintaining a downward force on the pump cylinder housing in order to hold the check valve down (open) and at the same time force the piston rod inward. Maintain approximately 230 N (50 lbf) force or greater on the piston rod, see Figure 4.



Key

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

Figure 4 — Fluid density balance

6.3.5 The check valve in the lid is pressure-actuated, which means the pressure in the cup keeps the valve closed. Close the valve initially by gradually lifting the cylinder housing of the pressurizing pump while applying pressure to the piston rod. When the check valve closes, release pressure on the piston rod before disconnecting the pump.

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6.3.6 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 attached bubble is centred between the two scribed marks. Obtain the density by reading one of the four calibrated scales on the arrow side of the sliding weight.

Figure 5 — Pressurized fluid density balance

6.3.7 To push the valve downward and release the pressure, reconnect the pump assembly and push downward on the pump cylinder housing.

Then empty the cup and pump assembly of its contents, and thoroughly clean all components.

For best operation, lightly grease the valve, lid and cylinder.

6.4 Alternative apparatus and procedure

6.4.1 Alternative apparatus

Cement slurry density may alternatively be determined by use of the mud balance.

6.4.2 Alternative procedure

The procedure for using a mud balance shall be in accordance with the latest edition of ISO 10414-1 except that the slurry, after being poured into the mud balance cup, shall be puddled 25 times to eliminate entrapped air.

7 Well-simulation compressive strength tests

7.1 General

This clause presents procedures for well-simulation compressive strength testing. Well-simulation compressive strength tests are not required for compliance with ISO 10426-1.

The well-simulation compressive strength tests described in this clause may be used to test cements or cement blends for resistance to thermally induced strength retrogression. To do this, the cement or cement blend sample is exposed to temperature and pressure for varying periods of time and observed for changes in compressive strength. The procedure involves comparing the compressive strength observed after some initial period (such as 24 h, 48 h or 72 h) with the compressive strength observed after some extended period or periods (such as 28 d). Cements or cement blends that exhibit lower compressive strength after extended ageing may be considered to exhibit strength retrogression. The commonly cited temperature threshold for thermally induced strength retrogression is 110 °C (230 °F), although deviation from this value has been reported.

7.2 Sampling

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

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7.3 Preparation of slurry

Prepare the slurry in accordance with Clause 5.

7.4 Apparatus

7.4.1 Cube moulds and compressive-strength testing machine, conforming to the requirements of ASTM C 109, except that the moulds may be separable into more than two parts.

Moulds and testing machine for compressive strength tests shall conform to the requirements of ASTM C 109, except that the moulds may be separable into more than two parts. The mould tolerances shall be verified. Calibrate the testing device to be accurate to ± 1 % of the load range to be measured. The moulds and testing device shall be calibrated at least every two years.

7.4.2 Cube mould base and cover plates, of corrosion-resistant material.

The base plate shall be of metal; the cover plate shall have a minimum thickness of 6 mm (¼ in). Grooves may be incorporated into the surface of the cover plate contacting the surface of the cement. Glass plates are not recommended for tests above 110 °C (230 °F) because of the risk of silica replacement.

7.4.3 Water curing bath or tank, having dimensions permitting the complete immersion of moulds 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 bath**, suitable for curing samples at the appropriate final test temperature and recommended as capable of maintaining a pressure of at least 20 700 kPa (3 000 psi).

   The vessel shall be capable of being heated at the desired rate.

7.4.4 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).

7.4.5 Temperature-measuring system, calibrated to an accuracy of ± 2 °C (± 3 °F) no less frequently than monthly.

The calibration procedure described in Annex B is commonly used. 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 non-pressurized vessel.

b) A thermocouple, of range –18 °C to 104 °C (0 °F to 220 °F), calibrated to an accuracy of ± 2 °C (± 3 °F) is preferred in a non-pressurized vessel.

c) A thermocouple, of range –18 °C to at least 204 °C (0 °F to at least 400 °F), calibrated to an accuracy of ± 2 °C (3 °F), shall be used in pressurized vessels.

7.4.6 Puddling rod, corrosion-resistant, typically with a width of 6 mm ± 0,5 mm (1/4 in).

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7.4.7 Mould-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.

e) non-corrosive in the temperature range –18 °C to at least 204 °C (0 °F to at least 400 °F).

7.4.8 Mould-release agent (optional).

A thin layer of mould-release agent may be applied to the interior surfaces of the mould to prevent the sample from being damaged when removed from the mould.

7.5 Procedure

7.5.1 Preparation of moulds

The interior faces of the moulds and the contact surfaces of the plates are commonly coated with mould release agent, but may be clean and dry. The assembled moulds shall be water tight. Care shall be taken to ensure there is no bead of sealant on the interior of the mould (Figure 6).

7.5.2 Preparation and placement of cement slurry

7.5.2.1 Preparation

Prepare the cement slurry in accordance with Clause 5.

7.5.2.2 Placement

Pour the cement slurry into the prepared moulds to approximately one-half of the mould depth. Puddle each sample approximately 30 times with a puddling rod after all mould chambers have received slurry. Stir the remaining slurry by hand to re-suspend the components. Fill each sample mould 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 mould using a straight edge. Discard specimens in moulds that leak. Place the cover plate on top of the moulds. For each test, use at least three specimens.

7.5.3 Curing at atmospheric pressure

After the moulds have been filled and covered with the cover plate, immediately place them in a water curing bath maintained at the desired curing temperature. Raise the moulds off the bottom of the bath using a perforated baffle plate or suitable spacers to allow water to completely circulate around the samples during the curing period. At approximately 45 min prior to the age at which the samples are to be tested, remove the moulds from the water bath and remove the cured samples from the moulds. Immediately immerse the samples in a water cooling bath at 27 °C ± 3 °C (80 °F ± 5 °F) until the samples are tested.

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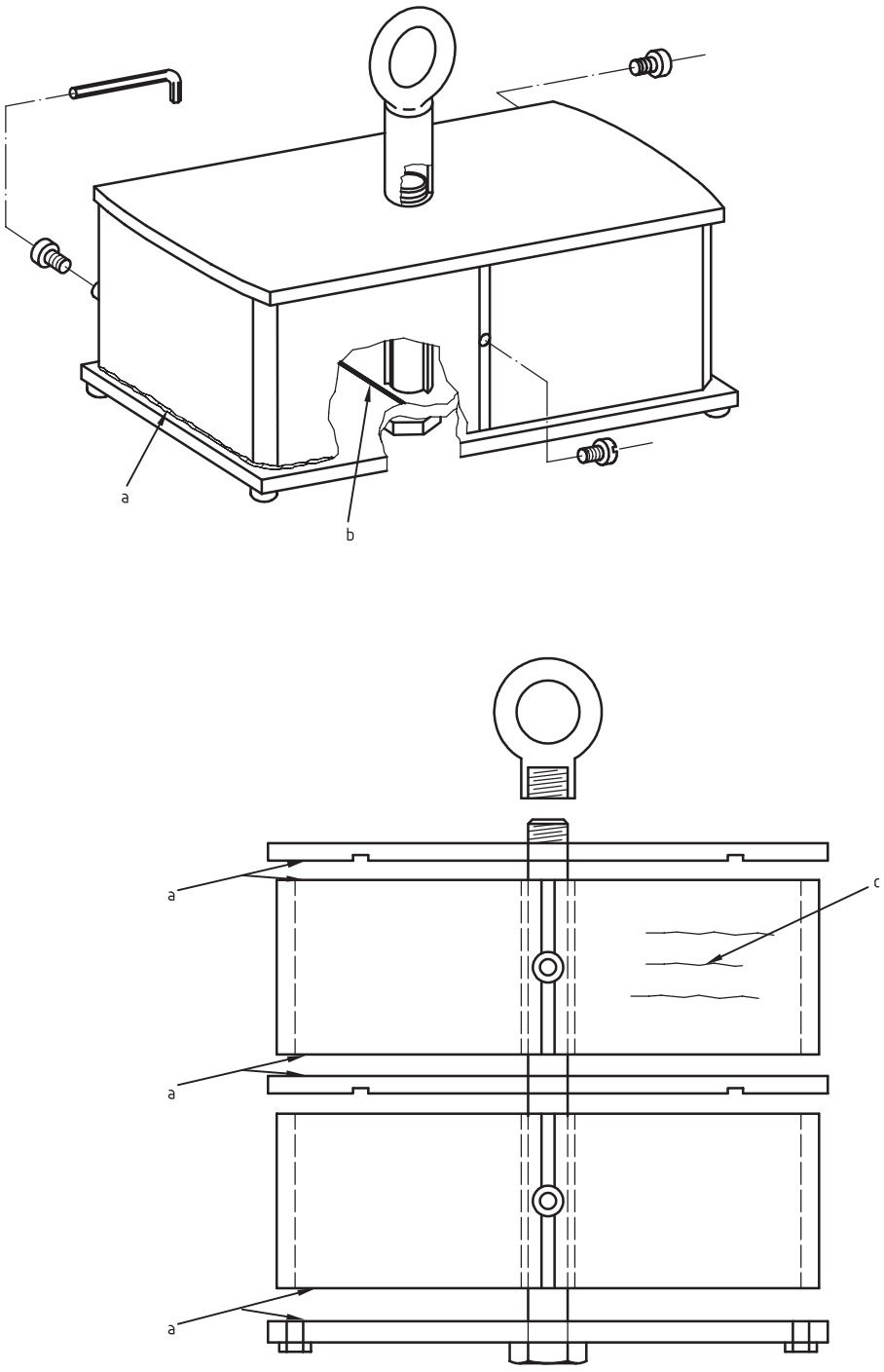

a Grease lightly here.

b Remove extruded grease.

c Apply mould release agent inside sample cavity.

Figure 6 — Diagram of mould preparation

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7.5.4 Curing at pressures above atmospheric

After the moulds have been filled and covered with the top plate, immediately place them in a pressurized curing vessel at the desired test initiation temperature [normally 27 °C ± 3 °C (80 °F ± 5 °F)]. Apply heat and pressure in accordance with the test schedule. Cement samples may be cured in accordance with pressure/temperature schedules provided in Table 2 (see below) or to a schedule designed to simulate conditions in a specific well.

For samples cured at or below 90 °C (194 °F), maintain test temperature and pressure until 45 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. At 45 min prior to testing the samples, release the pressure gradually and remove the moulds from the curing vessel. Immediately remove the samples from the moulds and place them in a water bath maintained at 27 °C ± 3 °C (80 °F ± 5 °F) until the samples are tested.

7.5.5 Test period

The test period is the time elapsed from subjecting the sample to heat in the curing vessel to testing the sample for strength.

7.5.6 Sample testing

Test samples immediately after removal from the cooling bath. The test procedure shall be in accordance with ASTM C 109, except for the following aspects.

a) A compressive strength testing device shall be used, and the rate of loading for samples with strength greater than 3,5 MPa (500 psi) shall be 71,7 kN ± 7,2 kN (16 000 lbf ± 1 600 lbf, 4 000 psi ± 400 psi) per minute. For a nominal 2 580,64 mm^2 (4 in^2) sample surface, this rate can be achieved by adjusting the load rate to obtain a gauge indicator travel from 8,9 kN to 26,8 kN (2 000 lbf and 6 000 lbf) gauge reading in 15 s. For samples with strength of 3,5 MPa (500 psi) or less, a loading rate of 17,9 kN ± 1,8 kN (1 000 psi ± 100 psi, 4 000 lbf ± 400 lbf) per minute shall be used. For a nominal 4 in^2 sample surface, this rate can be achieved by adjusting the load rate to obtain a gauge indicator travel from 8,9 kN to 26,8 kN (2 000 lbf and 6 000 lbf) gauge reading in 1 min. Make no adjustment in the controls of the testing machine while a sample is yielding before failure.

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 compression tester. Average the compressive strength of all acceptable test samples (see ASTM C 109) made from the same slurry and tested at the same time. Report compressive strength results to the nearest 0,1 MPa (10 psi) and include the test schedule used.

7.6 Determination of cement compressive strength at the top of long cement columns

7.6.1 Guidelines for use

Use this procedure if the bottom-hole circulating temperature (T_{BHC}) is higher than the static temperature at the top of the cement column (T_{TOCS}).

7.6.2 Procedure

7.6.2.1 Prepare a cement slurry in accordance with Clause 5. Pour the slurry into the slurry container of a pressurized consistometer, and heat to T_{BHC} in accordance with the pressure/temperature schedules provided in Annex E or a schedule designed to simulate conditions in a specific well. Hold test temperature and pressure for 60 min to allow the cement temperature to reach equilibrium.

7.6.2.2 Upon completion of the appropriate test schedule, plus the 60 min to reach equilibrium, cool to the top-of-cement circulating temperature (T_{TOCC}) or 90 °C (194 °F), whichever is lower, at a rate of 1,0 °C/min (2,0 °F/min). Use the following equation to determine the cool-down time (t), in minutes.

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For SI units:

$$t = \frac{T_{BHC} - T_{TOCC}}{1{,}0\ °C} \quad (1)$$

For US customary units:

$$t = \frac{T_{BHC} - T_{TOCC}}{2{,}0\ °F} \quad (2)$$

where:

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

T_{BHC} is the bottom-hole circulating temperature, expressed in °C or °F

T_{TOCC} is the top-of-cement circulating temperature, expressed in °C or °F.

Decrease the temperature while maintaining test pressure. When the T_{TOCC} or 90 °C (190 °F) (whichever is lower) is reached, release the pressure slowly and remove the slurry container.

7.6.2.3 Take care to minimize oil contamination of the slurry. Open the slurry container from the top (while 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. Blot the top of the slurry with an absorbent cloth or paper towel to remove any visible oil. Transfer the slurry three (3) times between the slurry container and a beaker, to re-suspend any solids that may have settled.

7.6.2.4 Pour the slurry into prepared moulds as specified in 7.5.2.2, and place the moulds in a preheated curing vessel [preheated to T_{TOCC} or 90 °C (194 °F), whichever is lower]. A non-destructive sonic test device as described in Clause 8 may also be used. No longer than 15 min after removing the slurry from the consistometer, apply 20 700 kPa ± 3 400 kPa (3 000 psi ± 500 psi) curing pressure.

7.6.2.5 Adjust sample temperature T_{TOCS} to the final curing temperature over a time period appropriate to well conditions, while maintaining curing pressure. If a time to reach final conditions is not known or specified, use 6 h.

7.6.2.5 Remove samples as specified in 7.5.4.

7.6.2.6 Test the samples for strength in accordance with procedures in 7.5.6 or Clause 8.

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Table 2 — Well-simulation test schedules for curing compressive strength specimens

1	2	3	4		5		6		7		8		9	
Schedule[a]	**Elapsed time**[b]	**Pressure**[c]	**Temperature gradient, °C/100 m depth** (°F/100 ft depth)[d]											
			°C	(°F)	°C	(°F)	°C	(°F)	°C	(°F)	°C	(°F)	°C	(°F)
	min	kPa (psi)	1,6	(0,9)	2	(1,1)	2,4	(1,3)	2,7	(1,5)	3,1	(1,7)	3,5	(1,9)
			Temperature, °C (°F)											
1 Sg 305 m (1 000 ft)	15	5 500 (800)	27	(80)	27	(80)	27	(80)	27	(80)	27	(80)	27	(80)
	30		27	(81)	27	(81)	27	(81)	27	(81)	27	(81)	27	(81)
	60		28	(82)	28	(82)	28	(83)	28	(83)	29	(84)	29	(84)
	90		28	(83)	29	(84)	29	(84)	29	(85)	30	(86)	30	(86)
	120		29	(84)	29	(85)	30	(86)	31	(87)	31	(88)	32	(89)
	150		29	(85)	31	(87)	31	(88)	32	(89)	32	(90)	33	(91)
	180		31	(87)	31	(88)	32	(90)	33	(91)	34	(93)	34	(94)
	210		31	(88)	32	(90)	33	(91)	34	(93)	35	(95)	36	(96)
	240		32	(89)	33	(91)	34	(93)	35	(95)	36	(97)	37	(99)
2 Sg 610 m (2 000 ft)	15	11 000 (1 600)	31	(88)	31	(88)	32	(89)	32	(89)	32	(90)	32	(90)
	30		32	(90)	32	(90)	33	(91)	33	(91)	33	(92)	34	(93)
	60		33	(91)	33	(92)	34	(93)	34	(94)	35	(95)	36	(96)
	90		33	(92)	34	(93)	35	(95)	36	(97)	37	(99)	38	(100)
	120		34	(93)	35	(95)	36	(97)	37	(99)	39	(102)	39	(103)
	150		34	(94)	36	(97)	38	(100)	39	(102)	41	(105)	42	(107)
	180		36	(96)	37	(99)	39	(102)	41	(105)	42	(108)	44	(111)
	210		36	(97)	38	(100)	40	(104)	42	(107)	44	(111)	46	(114)
	240		37	(98)	39	(102)	41	(106)	43	(110)	46	(114)	48	(118)
3 Sg 1 220 m (4 000 ft)	15	20 700 (3 000)	33	(91)	33	(92)	34	(93)	34	(93)	34	(94)	34	(94)
	30		37	(99)	38	(101)	39	(102)	39	(103)	40	(104)	41	(105)
	60		39	(102)	40	(104)	41	(106)	42	(108)	43	(110)	44	(112)
	90		40	(104)	42	(107)	43	(110)	45	(113)	47	(117)	49	(120)
	120		42	(107)	44	(111)	46	(115)	48	(119)	51	(123)	53	(127)
	150		43	(109)	46	(114)	48	(119)	51	(124)	54	(129)	57	(134)
	180		44	(111)	47	(117)	51	(123)	54	(129)	57	(135)	61	(141)
	210		46	(114)	49	(121)	53	(128)	57	(135)	61	(142)	65	(149)
	240		47	(116)	51	(124)	56	(132)	60	(140)	64	(148)	69	(156)
4 Sg 1 830 m (6 000 ft)	15	20 700 (3 000)	35	(95)	35	(95)	36	(96)	36	(97)	37	(98)	38	(101)
	30		43	(109)	44	(111)	45	(113)	46	(115)	47	(116)	50	(122)
	45		45	(113)	47	(116)	48	(118)	49	(121)	51	(124)	54	(130)
	60		46	(115)	48	(118)	49	(121)	52	(125)	53	(128)	57	(135)
	90		48	(118)	51	(123)	53	(128)	56	(132)	58	(137)	63	(145)
	120		49	(121)	53	(127)	57	(134)	60	(140)	63	(146)	68	(155)
	150		51	(124)	56	(132)	60	(140)	64	(147)	68	(155)	73	(164)
	180		53	(128)	58	(137)	63	(146)	68	(155)	73	(164)	79	(174)
	210		55	(131)	61	(141)	67	(152)	72	(162)	78	(173)	84	(184)
	240		57	(134)	63	(146)	70	(158)	77	(170)	83	(182)	90	(194)

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Table 2 — *(continued)*

1	2	3	4		5		6		7		8		9	
Schedule[a]	**Elapsed time**[b]	**Pressure**[c]	**Temperature gradient, °C/100 m depth** (°F/100 ft depth)[d]											
			°C	(°F)	°C	(°F)	°C	(°F)	°C	(°F)	°C	(°F)	°C	(°F)
	min	kPa (psi)	1,6	(0,9)	2	(1,1)	2,4	(1,3)	2,7	(1,5)	3,1	(1,7)	3,5	(1,9)
			Temperature, °C (°F)											
5 Sg 2 440 m (8 000 ft)	15	20 700 (3 000)	36	(97)	37	(98)	38	(100)	39	(102)	40	(104)	43	(109)
	30		46	(114)	47	(116)	49	(120)	51	(124)	53	(128)	59	(139)
	45		53	(127)	54	(130)	58	(136)	61	(141)	64	(147)	72	(161)
	60		53	(128)	56	(133)	60	(140)	63	(146)	67	(153)	75	(167)
	90		56	(132)	59	(139)	64	(147)	68	(155)	73	(163)	81	(178)
	120		58	(136)	62	(144)	68	(154)	73	(164)	79	(174)	87	(189)
	150		60	(140)	66	(150)	72	(162)	78	(173)	84	(184)	93	(199)
	180		62	(144)	69	(156)	76	(169)	83	(182)	91	(195)	99	(210)
	210		64	(148)	72	(162)	81	(177)	88	(191)	96	(205)	105	(221)
	240		67	(152)	76	(168)	84	(184)	93	(200)	102	(216)	111	(232)
6 Sg 3 050 m (10 000 ft)	15	20 700 (3 000)	37	(98)	38	(100)	39	(103)	41	(106)	43	(110)	47	(116)
	30		47	(116)	49	(120)	53	(127)	56	(132)	60	(140)	67	(152)
	45		57	(134)	59	(139)	66	(150)	70	(158)	77	(170)	87	(188)
	60		61	(142)	64	(148)	72	(161)	77	(170)	84	(184)	96	(204)
	90		63	(146)	68	(155)	76	(169)	82	(180)	91	(195)	102	(215)
	120		66	(151)	72	(162)	81	(177)	88	(190)	97	(206)	108	(226)
	150		69	(156)	76	(169)	85	(185)	93	(200)	103	(217)	114	(237)
	180		72	(161)	80	(176)	90	(194)	99	(210)	109	(228)	120	(248)
	210		74	(165)	84	(183)	94	(202)	104	(220)	115	(239)	126	(259)
	240		77	(170)	88	(190)	99	(210)	110	(230)	121	(250)	132	(270)
7 Sg 3 660 m (12 000 ft)	15	20 700 (3 000)	37	(98)	39	(102)	42	(107)	44	(111)	47	(116)	49	(120)
	30		46	(115)	51	(124)	56	(133)	62	(143)	67	(152)	72	(161)
	45		56	(133)	63	(146)	71	(160)	79	(174)	87	(188)	94	(201)
	60		64	(148)	74	(166)	84	(184)	94	(202)	104	(220)	114	(237)
	90		68	(155)	78	(173)	89	(192)	99	(211)	110	(230)	121	(249)
	120		72	(162)	83	(181)	94	(201)	105	(221)	116	(241)	127	(261)
	150		76	(168)	87	(189)	99	(210)	111	(231)	122	(252)	133	(272)
	180		79	(175)	92	(197)	104	(219)	116	(241)	128	(263)	140	(284)
	210		83	(181)	96	(204)	108	(227)	121	(250)	134	(273)	147	(296)
	240		87	(188)	100	(212)	113	(236)	127	(260)	140	(284)	153	(308)

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Table 2 — *(continued)*

1	2	3	4		5		6		7		8		9	
Schedule[a]	**Elapsed time**[b]	**Pressure**[c]	**Temperature gradient, °C/100 m depth** (°F/100 ft depth)[d]											
			°C	(°F)	°C	(°F)	°C	(°F)	°C	(°F)	°C	(°F)	°C	(°F)
	min	kPa (psi)	1,6	(0,9)	2	(1,1)	2,4	(1,3)	2,7	(1,5)	3,1	(1,7)	3,5	(1,9)
			Temperature, °C (°F)											
8 Sg 4 270 m (14 000 ft)	15	20 700 (3 000)	37	(99)	40	(104)	43	(109)	46	(114)	48	(119)	51	(123)
	30		48	(118)	53	(128)	59	(138)	64	(147)	69	(157)	75	(167)
	45		58	(137)	67	(152)	75	(167)	83	(181)	91	(196)	99	(210)
	60		69	(156)	79	(175)	91	(195)	102	(215)	113	(235)	123	(254)
	75		74	(166)	87	(188)	99	(210)	111	(231)	123	(254)	135	(275)
	90		77	(170)	89	(192)	102	(215)	114	(237)	126	(259)	138	(281)
	120		81	(177)	93	(200)	107	(224)	119	(247)	133	(271)	146	(294)
	150		84	(184)	98	(209)	112	(234)	126	(258)	139	(283)	153	(307)
	180		89	(192)	103	(217)	117	(243)	132	(269)	146	(295)	160	(320)
	210		93	(199)	108	(226)	123	(253)	137	(279)	152	(306)	167	(333)
	240		97	(206)	112	(234)	128	(262)	143	(290)	159	(318)	174	(346)
9 Sg 4 880 m (16 000 ft)	15	20 700 (3 000)	38	(101)	41	(106)	44	(111)	47	(116)	49	(121)	52	(126)
	30		49	(121)	55	(131)	61	(142)	67	(152)	73	(163)	78	(173)
	45		61	(142)	69	(157)	78	(173)	87	(188)	96	(204)	104	(219)
	60		73	(163)	84	(183)	96	(204)	107	(224)	118	(245)	130	(266)
	75		83	(182)	97	(207)	112	(233)	126	(258)	140	(284)	154	(309)
	90		86	(186)	100	(212)	114	(238)	129	(264)	144	(291)	158	(316)
	120		90	(194)	105	(221)	120	(248)	135	(275)	151	(303)	166	(330)
	150		94	(201)	109	(229)	126	(258)	141	(286)	157	(315)	173	(343)
	180		98	(209)	114	(238)	131	(268)	148	(298)	164	(327)	181	(357)
	210		102	(216)	119	(247)	137	(278)	154	(309)	171	(340)	188	(370)
	240		107	(224)	124	(256)	142	(288)	160	(320)	178	(352)	196	(384)
10 Sg 5 490 m (18 000 ft)	15	20 700 (3 000)	39	(102)	42	(108)	45	(113)	48	(119)	51	(124)	54	(129)
	30		51	(124)	57	(135)	63	(146)	69	(157)	76	(168)	82	(179)
	45		63	(146)	73	(163)	82	(179)	61	(196)	100	(212)	109	(228)
	60		76	(169)	88	(190)	100	(212)	112	(234)	124	(256)	137	(278)
	75		88	(191)	103	(218)	119	(246)	134	(273)	149	(300)	164	(327)
	90		95	(203)	112	(233)	129	(264)	146	(294)	162	(324)	179	(354)
	120		99	(211)	117	(242)	134	(274)	152	(305)	169	(337)	186	(367)
	150		104	(219)	122	(251)	140	(284)	158	(316)	176	(349)	194	(381)
	180		108	(226)	127	(260)	146	(294)	164	(328)	183	(361)	202	(395)
	210		112	(234)	132	(269)	151	(304)	171	(339)	190	(374)	209	(408)
	240		117	(242)	137	(278)	157	(314)	177	(350)	197	(386)	217	(422)

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Table 2 — *(continued)*

1	2	3		4		5		6		7		8		9	
Schedule[a]	**Elapsed time**[b]	**Pressure**[c]		**Temperature gradient, °C/100 m depth** (°F/100 ft depth)[d]											
				°C	(°F)	°C	(°F)	°C	(°F)	°C	(°F)	°C	(°F)	°C	(°F)
	min	kPa	(psi)	1,6	(0,9)	2	(1,1)	2,4	(1,3)	2,7	(1,5)	3,1	(1,7)	3,5	(1,9)
				Temperature, °C (°F)											
11 Sg 6 100 m (20 000 ft)	15	20 700	(3 000)	40	104	43	(109)	46	(115)	49	(121)	53	(127)	56	(133)
	30			53	127	59	(139)	66	(150)	72	(162)	78	(173)	85	(185)
	45			66	151	76	(168)	86	(186)	95	(203)	104	(220)	114	(238)
	60			79	175	92	(197)	105	(221)	118	(244)	131	(267)	143	(290)
	75			92	198	108	(227)	124	(256)	141	(285)	156	(313)	173	(343)
	90			106	222	124	(256)	144	(291)	163	(326)	182	(360)	202	(395)
	120			110	230	129	(265)	149	(301)	169	(337)	189	(372)	209	(408)
	150			114	237	134	(274)	155	(311)	176	(348)	196	(384)	216	(421)
	180			118	245	139	(282)	160	(320)	181	(358)	202	(396)	223	(434)
	210			122	252	144	(291)	166	(330)	187	(369)	209	(408)	231	(447)
	240			127	260	149	(300)	171	(340)	193	(380)	216	(420)	238	(460)
12 Sg 6 100 m (20 000 ft)	15	20 700	(3 000)	41	(105)	44	(111)	47	(117)	51	(123)	54	(130)	58	(136)
	30			54	(130)	61	(142)	68	(155)	75	(167)	82	(179)	88	(191)
	45			68	(155)	79	(174)	89	(192)	99	(210)	109	(229)	119	(247)
	60			82	(180)	96	(205)	109	(229)	123	(254)	137	(278)	151	(303)
	75			97	(206)	113	(236)	131	(267)	147	(297)	164	(328)	182	(359)
	90			111	(231)	131	(267)	151	(304)	172	(341)	192	(378)	212	(414)
	105			119	(246)	141	(286)	163	(326)	186	(366)	208	(406)	231	(447)
	120			121	(249)	143	(290)	166	(331)	188	(371)	211	(412)	233	(452)
	150			124	(256)	148	(298)	171	(339)	194	(381)	217	(422)	240	(464)
	180			129	(264)	152	(306)	176	(348)	199	(391)	223	(433)	246	(475)
	210			133	(271)	157	(314)	181	(357)	204	(400)	228	(443)	253	(487)
	240			137	(278)	161	(322)	186	(366)	210	(410)	234	(454)	259	(498)

[a] Sg is a schedule number designation.

[b] The temperature shall be increased in equal amounts at 15 min intervals until the 4 h (240 min) temperature is reached.

The 4 h temperature shall be maintained until completion of test.

Final temperature shall be maintained within ± 2 °C (± 3 °F) throughout the remainder of the curing period.

[c] The test pressure shall be applied as soon as specimens are placed in the pressure vessel and maintained at the given pressure within the following limits for the duration of the curing period:

Schedule 1 Sg 5 500 kPa ± 700 kPa (800 psi ± 100 psi).

Schedule 2 Sg 11 000 kPa ± 1 400 kPa (1 600 psi ± 200 psi).

Schedule 3 Sg-11 Sg 20 700 kPa ± 3 400 kPa (3 000 psi ± 500 psi).

[d] Temperature gradient = (T_{BHS} – 27 °C) / 100 m depth or (T_{BHS} – 80 °F) / 100 ft depth

T_{BHS} = Bottom-Hole Static Temperature

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8 Non-destructive sonic testing of cement

8.1 General

This clause presents testing procedures for the non-destructive sonic testing of cement. The apparatus transmits a sonic signal through the cement being tested. The signal transit time can be correlated to cement properties such as the time and extent of strength development.

Non-destructive sonic testing of cement is not required for compliance with ISO 10426-1.

8.2 Apparatus

8.2.1 Curing cell, which can be subjected to controlled temperature and pressure for curing the cement slurry.

The cell shall include the following systems:

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

Calibration shall be no less frequent than monthly and may be performed in accordance with the procedure described in Annex B.

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

8.3 Sampling

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

8.4 Preparation of slurry

The slurry shall be prepared in accordance with Clause 5.

Excessive free fluid can impair the accuracy of this test. Free fluid in a slurry can cause cement to lose contact with the top cell cover plate and affect the signal being sent through the cement. The percentage of free fluid can be determined in accordance with 15.4 and 15.5.

8.5 Procedure

Follow the detailed operating instructions and safety precautions furnished by the manufacturer.

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 shall 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 Clause 7 or with a schedule designed to simulate specific well conditions.

NOTE Planned or unplanned changes in temperature or pressure alter the transit time.

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8.8 Data reporting

8.8.1 The transit time shall be continuously monitored. The strength of the cement sample shall be obtained from transit time correlations.

8.8.2 After removal of the sample from the curing cell, it is occasionally surfaced (cut into cube) and crushed. The result obtained is not comparable to that found using the method described in Clause 7. The result shall not be reported as ISO 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 clause. The results of the laboratory thickening-time test provide an indication of the length of time that a cement slurry will remain pumpable in a well. The laboratory test conditions shall represent the time, temperature and pressure to which a cement slurry will be exposed during pumping operations.

Well-simulation thickening-time tests are not required for compliance with ISO 10426-1.

9.2 Apparatus and material

9.2.1 Pressurized consistometer, see Figure 7.

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. The slurry container is rotated at a speed of 150 r/min ± 15 r/min. An alternative apparatus is described in Annex D.

The paddle and all parts of the slurry container exposed to the slurry shall be constructed of corrosion-resistant materials. The space between the slurry container and the walls of the pressure vessel shall be completely filled with hydrocarbon oil (9.2.2).

The temperature and consistency of the cement slurry (9.3.2) shall be measured. See Annex B for temperature-measuring systems.

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

9.2.2 Hydrocarbon oil, selected to have the following physical properties:

Viscosity = 7 mm/s^2 to 75 mm/s^2 at 38 °C (7 cSt to 75 cSt at 100 °F)

Specific heat = 2,1 kJ/(kg·K) to 2,4 kJ/(kg·K) (0,5 Btu/lb·°F to 0,58 Btu/lb·°F)

Thermal conductivity = 0,119 W/(m·K) to 0,133 W/(m·K) (0,068 5 Btu/h·ft^2·°F/ft to 0,077 0 Btu/h·ft^2·°F/ft)

Relative density = 0,85 to 0,91

A synthetic oil with suitable properties shall be used only if the test temperature exceeds the flash point of the hydrocarbon oil.

9.2.3 Heating system, capable of raising the temperature of the oil bath at a rate of at least 3 °C (5 °F) per minute.

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Temperature-measuring systems shall be provided for determining the temperature of the oil bath and also that of the cement slurry (see Annex B).

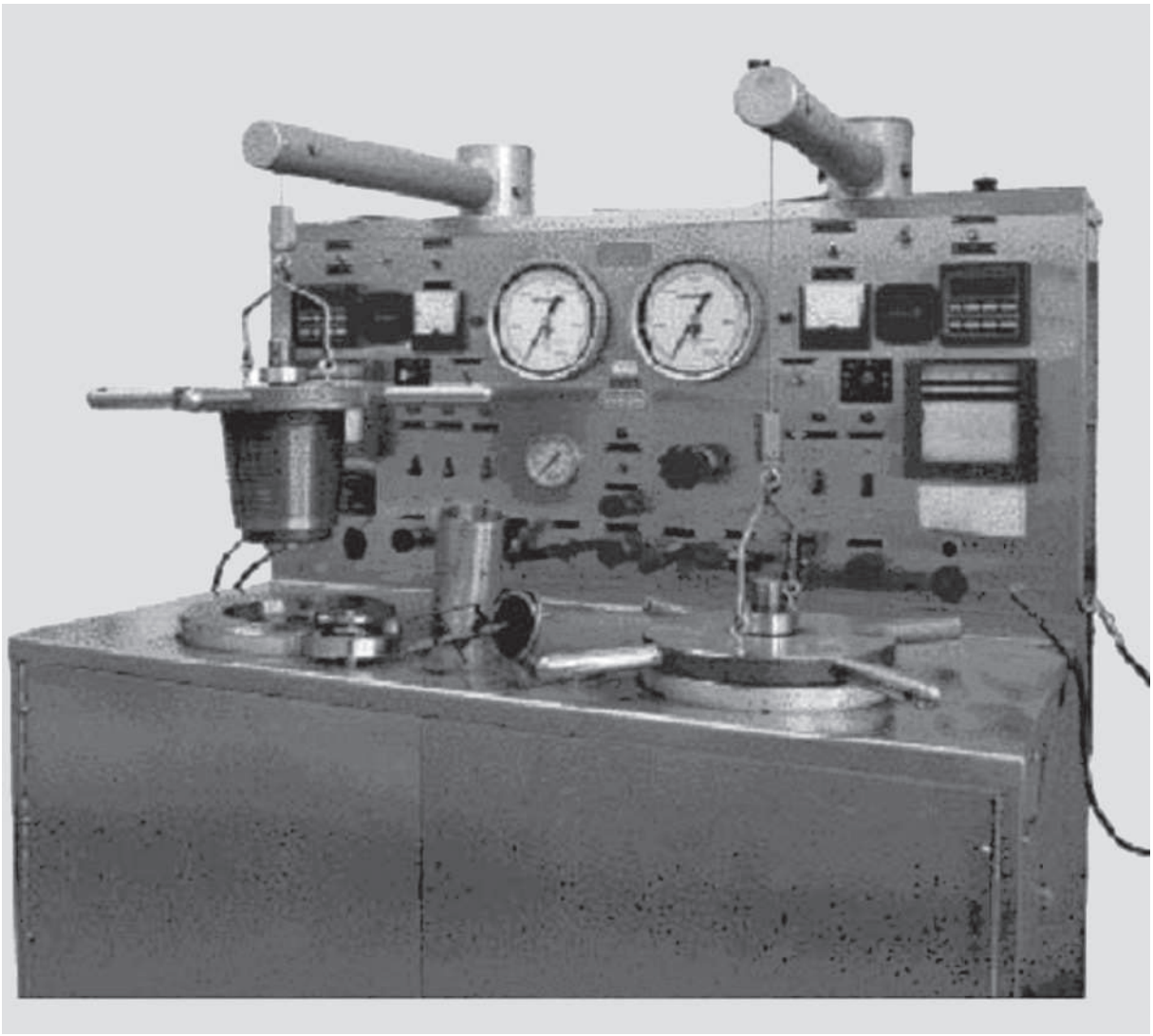

Figure 7 — Typical pressurized consistometer

9.3 Calibration

9.3.1 General

Measurement of the thickening time of a cement slurry requires calibration and maintenance of operating systems of the pressurized consistometer, including consistency measurement, temperature-measuring systems, temperature controllers, motor speed, timer and pressure gauges.

9.3.2 Consistency

Consistency of a cement slurry is expressed in Bearden units (B_c). This value is determined by a potentiometer mechanism and voltage-measurement circuit. These shall be calibrated monthly and whenever the calibration spring, resistor or contact arm is adjusted or replaced. The following calibration method shall be used.

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A weight-loaded device (Figure 8), for typical potentiometer calibration, is used to produce a series of torque-equivalent values for consistency, defined by the following equation:

$$T = 78{,}2 + 20{,}02\ B_c \tag{3}$$

where:

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

B_c consistency expressed in Bearden units.

Weights are used to apply torque to the potentiometer spring, using the radius of the potentiometer frame as a lever arm. As weights are added, the contact arm is deflected and the resulting DC voltage is recorded and used to determine B_c (see manufacturer's instruction manual for procedures). An example is given in Table 3. Some devices display B_c directly.

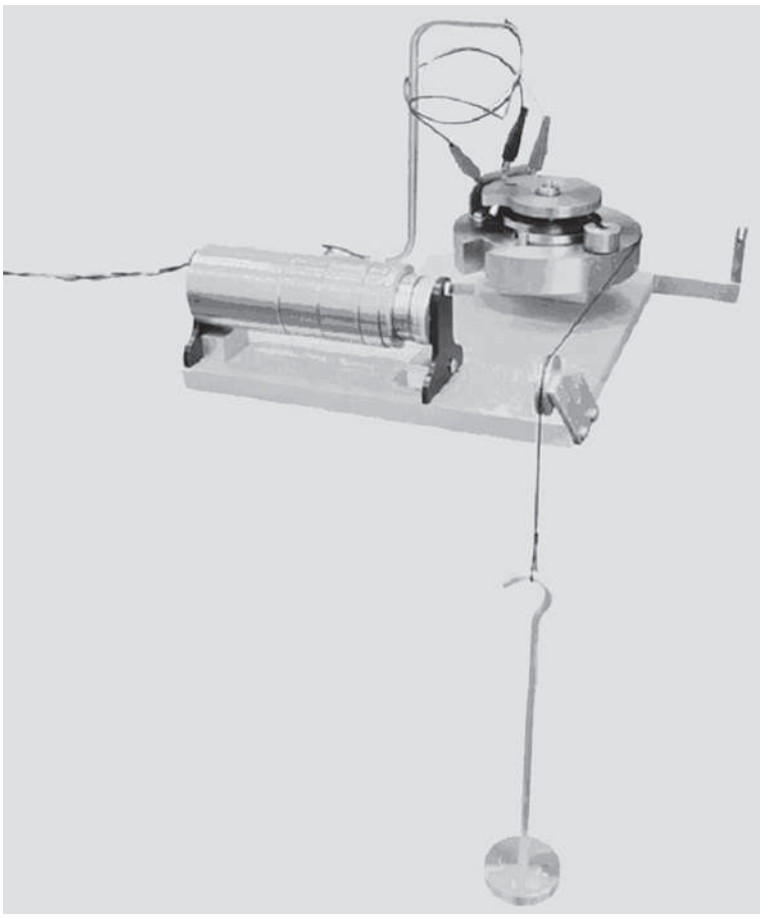

Figure 8 — Typical calibrating device for pressurized consistometer

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Table 3 — Slurry consistency vs. equivalent torque (for a potentiometer mechanism with a radius of 52 mm ± 1 mm)[a]

Calculated torque equivalent g·cm	Mass of added weights g ± 0,1	Calculated slurry consistency B_c
260	50	9
520	100	22
780	150	35
1 040	200	48
1 300	250	61
1 560	300	74
1 820	350	87
2 080	400	100

[a] For a potentiometer mechanism with a radius other than 52 mm ± 1 mm, an appropriate table with equivalent tolerances shall be developed.

9.3.3 Temperature-measuring system

The temperature-measuring device system shall be calibrated to an accuracy of ± 2 °C (± 3 °F). Calibration shall be no less frequent than monthly and may be performed in accordance with the procedure described in Annex B.

9.3.4 Motor speed

Rotational speed of the slurry container shall be 150 r/min ± 15 r/min (2,5 rev/s ± 0,25 rev/s) and shall be checked no less frequently than every three months.

9.3.5 Timer

Timers shall be accurate within ± 30 s per h and shall be checked for accuracy every six months.

9.3.6 Pressure-measuring devices

Calibration shall be conducted annually against a deadweight tester or master gauge to 0,25 % of full range at a minimum of 25 %, 50 % and 75 % of full scale.

9.4 Test procedure

9.4.1 Operating instructions

WARNING This procedure requires the handling of hot, pressurized equipment and materials that are hazardous and can cause injury. Do not exceed manufacturer's safety limits. Only trained personnel shall run these tests.

Detailed operating instructions, as furnished by the equipment manufacturer, are applicable under this method and shall be followed, provided they conform to the procedure contained in this part of ISO 10426.

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9.4.2 Assembly and filling the slurry container

Prepare and fill the slurry container via the following steps:

a) clean and lubricate cup threads;

b) inspect diaphragm;

c) assemble paddle shaft assembly and secure it in the cup sleeve with flange ring;

d) make sure the paddle turns freely;

e) invert the slurry container assembly and fill to within 6 mm (1/4 in) of the top;

f) strike to remove air;

g) screw in base plate and make sure slurry is extruded through the centre hole;

h) screw centre plug (pivot bearing) into the container;

i) wipe all cement from the outer surfaces;

j) recheck the paddle to make sure it turns smoothly;

k) load the slurry container assembly into the consistometer.

NOTE Slurry segregation can occur during the filling operation. This can be reduced by stirring the slurry in the mixing container with a spatula while pouring. Segregation will be less of a problem if the time from cessation of mixing to completing the filling operation is kept to a minimum.

9.4.3 Initiation of test

Place the filled slurry container (9.4.2) on the drive table in the pressure vessel, start rotation of the slurry container and secure the potentiometer mechanism so as to engage the paddle shaft drive bar. Begin filling the vessel with oil. At this point, the paddle shaft shall not be rotating.

Next, securely close the head assembly of the pressure vessel, insert the temperature-sensing device through the hole in head assembly and partially engage the threads. After the pressure vessel is completely filled with oil, tighten the threads of the temperature-sensing device.

Begin operating the apparatus 5 min $\pm$ 15 s after cessation of mixing of the slurry as defined in 5.3.4.

9.4.4 Temperature and pressure controls

During the test period, increase the temperature and pressure of the cement slurry in the slurry container in accordance with the appropriate well-simulation test schedule (9.5). Schedules may be calculated or taken from tables. Determine the temperature of the cement slurry for specification testing by use of an ASTM E 220 classification “special” Type J thermocouple located in the centre of the slurry container.

9.4.5 Thickening time

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

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9.5 Determination of test schedule

9.5.1 General

Well-simulation thickening-time test schedules may be taken from the tables or calculated from equations as described in 9.5.2 to 9.5.4.13. Background and supporting information for these schedules is contained in Annex C.

The schedules in Annex E are based upon nominally vertical wells. The choice of table to use is based upon well depth. The choice of column within a table is based upon thermal gradient.

9.5.2 Casing well-simulation schedules

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

— account for surface mixing of the slurry as provided in 9.5.4.1;

— calculate time to displace leading edge of cement slurry to bottom (9.5.4.2);

— calculate bottom-hole pressure (9.5.4.5);

— determine the starting pressure (9.5.4.6);

— calculate pressure-up rate to bottom-hole pressure (9.5.4.7);

— determine the T_{BHC} for the specific thermal gradient (Table E.1);

— calculate well simulation heat-up rate by subtracting T_{BHC} from ambient temperature and dividing by time to bottom.

Once reached, the final temperature and pressure conditions shall be maintained until the thickening-time test is completed.

9.5.3 Liner well-simulation schedules

Liner well-simulation thickening-time schedules shall be determined as follows:

— account for surface mixing of the slurry as provided in 9.5.4.1;

— calculate time to displace leading edge of cement slurry to bottom (9.5.4.2);

— calculate bottom-hole pressure (9.5.4.5);

— determine the starting pressure (9.5.4.6);

— calculate pressure-up rate to bottom-hole pressure (9.5.4.7);

— determine the T_{BHC} for the specific thermal gradient (Table E.2);

— calculate well simulation heat-up rate by subtracting T_{BHC} from ambient temperature and dividing by time to bottom.

Once reached, the final temperature and pressure conditions shall be maintained until the thickening-time test is completed.

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9.5.4 Calculations for casing and liner schedules

9.5.4.1 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. The time and slurry surface temperature (T_{SS}) may be estimated depending upon the expected conditions at the well site.

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

EXAMPLE Simulated batch mixing time: 1 h

Thickening time (does not include batch mixing simulation): 3 h 30 min

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

The rate of slurry displacement is calculated as follows:

$$t_d = \frac{V_p}{q} \tag{4}$$

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 metres;

q is the rate at which the fluid slurry is pumped, expressed in cubic metres per minute.

9.5.4.3 Correlation for predicted bottom-hole circulating temperatures (T_{PBHC}) for casing or liner jobs at depths deeper than 3 048 m (10 000 ft)

The correlation developed for predicting bottom-hole circulating temperatures is given, in degrees Celsius by:

$$T_{PBHC} = \frac{\left[\left(T_{AS} + \frac{\left(0{,}006\,061 \times h_{TOCTVD} \times \nabla_{PT}\right) - 10{,}091\,5}{1{,}0 - \left(0{,}000\,015\,052 \times h_{TOCTVD}\right)}\right) - 32\right]}{1{,}8} \tag{5}$$

or, in degrees Fahrenheit,

$$T_{PBHC} = T_{AS} + \frac{\left(0{,}006\,061 \times h_{TOCTVD} \times \nabla_{PT}\right) - 10{,}091\,5}{1{,}0 - \left(0{,}000\,015\,052 \times h_{TOCTVD}\right)} \tag{6}$$

where

T_{PBHC} is the predicted bottom-hole circulating temperature, expressed in °C [Equation (5)] or °F [Equation (6)];

T_{AS} is the assumed surface temperature, expressed in °F;

h_{TOCTVD} is the top-of-column true vertical depth, expressed in feet;

∇_{PT} is the pseudo-temperature gradient, expressed in °F/100 ft.

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

This correlation shall not be used to predict T_{PBHC} for depths of 3 048 m (10 000 ft) or shallower, because it can give significantly higher T_{PBHC} than the T_{PBHC} found in the tables in Annex E. A different correlation is used for the predicted squeeze temperature (T_{PS}). This correlation can be found in 9.5.6.1.

Although the T_{PBHC} correlation is based upon field measurements, there can be error associated with its use for predicting the circulating temperature in a well. The error range between this correlation and the field-measured data from which the correlation was derived is shown in Annex C. The standard deviation is 9,2 °C (16,6 °F). Whenever possible, measurements of downhole temperatures are preferred over calculated estimates.

9.5.4.4 Rate of heat-up to predicted bottom-hole circulating temperature

The temperature of the cement slurry shall be increased from the slurry surface temperature (T_{SS}) to the predicted bottom-hole 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:

$$R_{\Delta T} = \frac{T_{PBHC} - T_{SS}}{t_d} \tag{7}$$

where

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

T_{PBHC} is the predicted bottom-hole 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.

9.5.4.5 Bottom-hole pressure

The bottom-hole pressure can be calculated in SI units (or US Customary units) as follows:

$$p_{BH} = g \times \rho_{df} \times h_{TOCTVD} \tag{8}$$

where

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

g is the acceleration of freefall, expressed in metres per second squared (feet per second squared);

ρ_{df} is the density of drilling fluid, expressed in kilograms per cubic metre (pounds per gallon);

h_{TOCTVD} is the top-of-column true vertical depth, expressed in metres (feet).

NOTE The bottom-hole pressure can be calculated to account for contributions of other fluids (spacers, weighted pills, etc.) in the annulus.

9.5.4.6 Starting pressure

The starting pressure (p_S) is the estimated pressure to which the leading edge of cement slurry is subjected as it leaves the cementing head.

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9.5.4.7 Increase in pressure (pressure-up rate) to bottom-hole pressure

Pressure on the cement slurry shall be increased to the bottom-hole pressure during the test at a pressure-up rate calculated as follows:

$$R_{pu} = \frac{p_{BH} - p_S}{t_d} \tag{9}$$

where:

R_{pu} is the pressure-up rate, expressed in kilopascals per minute;

p_{BH} is the bottom-hole pressure, expressed in kilopascals;

p_S is the starting pressure, expressed in kilopascals;

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

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

If no measured data are available for the top-of-cement-column temperature (T_{TOC}), the T_{PBHC} and p_{BH} shall be maintained until the completion of the thickening-time test. Skip steps in 9.5.4.9 to 9.5.4.13 below.

If reliable T_{TOC} data is 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-column temperature (T_{TOC}) and pressure (p_{TOC}) using steps in 9.5.4.9 to 9.5.4.13.

9.5.4.9 Time to displace the annular volume to be cemented

The time required to displace the annular volume from the bottom of the casing to the top of the annular cement column is calculated as follows:

$$t_a = \frac{V_a}{q} \tag{10}$$

where

V_a is the annular volume, expressed in cubic metres;

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

q is the fluid pump rate, expressed in cubic metres per minute.

9.5.4.10 Rate of temperature change to T_{TOC}

The temperature of the cement slurry may be changed to the T_{TOC} at a rate calculated by the following equation:

$$R_{\Delta T} = \frac{T_{TOC} - T_{PBHC}}{t_a} \tag{11}$$

where:

$R_{\Delta T}$ is the rate of temperature change to T_{TOC}, expressed in °C per minute;

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T_{PBHC} is the bottom-hole circulating temperature, expressed in °C;

T_{TOC} is top of cement column temperature, expressed in °C;

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

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

9.5.4.11 Pressure at the top of cement column

Pressure at the top of the cement slurry column is calculated in SI units (or US Customary units) using the following equation:

$$p_{TOC} = g \times \rho_{df} \times h_{TOCTVD} \tag{12}$$

where:

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

g is the acceleration of freefall, expressed in metres per second squared (feet per second squared);

ρ_{df} is the density of drilling fluid, expressed in kilograms per cubic metre (pounds per gallon);

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

The top of cement pressure may be calculated to account for contributions of other fluids (spacers, weighted pills, etc.) in the annulus.

9.5.4.12 Rate of change in pressure down to p_{TOC}

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

$$R_{pd} = \frac{p_{BH} - p_{TOC}}{t_a} \tag{13}$$

where

R_{pd} is the rate of pressure change (down), expressed in kilopascals per minute;

p_{TOC} is the pressure at the top of the cement column in the annulus, expressed in kilopascals;

p_{BH} is the bottom-hole pressure, expressed in kilopascals;

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

9.5.4.13 Completion of test with simulated temperature change

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

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9.5.5 Tables of squeeze-cementing schedules

9.5.5.1 Continuous-pumping squeeze schedules

Tabular well-simulation thickening-time schedules for continuous-pumping squeeze-cementing operations are provided in Table E.3. Once reached, the final temperature and pressure conditions shown in the schedules shall be maintained until the thickening-time test is completed.

9.5.5.2 Hesitation-pumping squeeze schedules

Tabular well-simulation thickening-time schedules for hesitation-pumping squeeze-cementing operations are provided in Table E.4. The differences between the hesitation- and continuous-pumping schedules are that, for hesitation-pumping

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

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

9.5.6 Tailored squeeze schedules

9.5.6.1 Correlation to predict squeeze temperatures

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

$$T_{PS} = \frac{\left[\left(T_{AS} + \frac{(0{,}007\,6495 \times h_{TOCTVD} \times \nabla_{PT}) - 8{,}202\,1}{1{,}0 - (0{,}000\,008\,068 \times h_{TOCTVD})}\right) - 32\right]}{1{,}8} \tag{14}$$

or, for US Customary units:

$$T_{PS} = T_{AS} + \frac{(0{,}007\,6495 \times h_{TOCTVD} \times \nabla_{PT}) - 8{,}202\,1}{1{,}0 - (0{,}000\,008\,068 \times h_{TOCTVD})} \tag{15}$$

where

T_{PS} is the predicted bottom-hole squeeze temperature, expressed in °C [Equation (14)] or °F [Equation (15)];

∇_{PT} is the pseudo-temperature gradient, expressed in °F per 100 ft;

h_{TOCTVD} is the true vertical depth, expressed in feet;

T_{AS} is the assumed surface temperature, expressed in °F.

NOTE These correlations are valid only for the units shown.

Although the T_{PS} correlation is based upon field measurements, error can be associated with its use for predicting the squeeze temperature in a well. The error range between this correlation and the field-measured data from which the correlation was derived is shown in Annex C. The standard deviation is 7,2 °C (13 °F). Whenever possible, measurements of downhole temperatures are preferred over calculated estimates.

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9.5.6.2 Equations for tailored schedules

Equations (5) through (9) under 9.5.4.3 through 9.5.4.7 shall be used for tailored schedules. These equations can be used to calculate the heat-up rate ($R_{\Delta T}$) and pressure-up rate (R_{pu}) for a squeeze simulation thickening-time test. In Equation (7), the predicted squeeze temperature, T_{PS}, shall be substituted for the T_{PBHC}. After reaching the T_{PS} and p_{BH}, the temperature and pressure profiles shall 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, using an appropriate sequence, to simulate a hesitation technique if this is anticipated for the squeeze-cementing operation.

9.5.7 Plug-cementing tailored schedules

Equations (14) and (15) under 9.5.6.1 can be used for plug-cementing tailored schedules. These equations can be used to calculate the heat-up rate ($R_{\Delta T}$) and pressure-up rate (R_{pu}) for a plug-cementing simulation thickening-time test. Because of the short cement columns typically used in plug-cementing, no temperature change or pressure-down rates to the top of the cement column shall be used. Therefore, steps 9.5.4.9 through 9.5.4.13 shall not be used.

10 Static fluid-loss tests

10.1 General

This clause provides several procedures for running static fluid-loss tests. For tests at temperatures less than or equal to 88 °C (190 °F), testing may be performed using a static fluid-loss cell after slurry conditioning in an atmospheric or pressurized consistometer, or by using a stirred fluid-loss cell. For tests at temperatures greater than 88 °C (190 °F), testing may be performed using a static fluid-loss cell following conditioning in a pressurized consistometer or by using a stirred fluid-loss cell. Regardless of whether the slurry is conditioned in a consistometer or in a stirred fluid-loss cell, the fluid-loss value is determined under static conditions.

10.2 Apparatus

10.2.1 High temperature, high pressure fluid-loss cell or stirred fluid-loss cell, 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 wear, damage or distortion.

The equipment manufacturer's recommendations for maximum temperatures, pressures and volumes should not be exceeded.

10.2.2 ASTM E220 classification "special" Type J thermocouple, mounted in the wall of the cell or immersed in the slurry, for measuring the temperature of the cement slurry.

Thermocouples and displays on consistometers and fluid-loss cells shall be calibrated in accordance with Annex B.

The location of the thermocouple shall be noted on the report form. A thermocouple mounted in the heating jacket measures the temperature of the jacket. This temperature is usually higher than the temperature inside the fluid-loss cell.

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

10.2.3 Pressure gauges, having a scale such that pressure can be read to ± 300 kPa (± 50 psi).

Gauges shall be calibrated annually.

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10.3 Safety

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.

10.4 Mixing procedure

The slurry shall be mixed in accordance with Clause 5.

10.5 Conditioning procedures

All slurry conditioning shall start at 27 °C ± 1 °C (80 °F ± 2 °F), or at a temperature appropriate for the well conditions, and be heated in accordance with the appropriate schedule.

10.6 Procedures for testing at temperatures ⩽ 88 °C (190 °F)

10.6.1 Atmospheric-pressure conditioning

10.6.1.1 Within 1 min after mixing, place the slurry into the container of the atmospheric-pressure consistometer.

10.6.1.2 Heat the slurry to T_{PBHC} or T_{PS} in accordance with the thickening-time schedule that most closely simulates actual field conditions. (See optional step 10.6.4.) If the atmospheric-pressure consistometer is not equipped to measure slurry temperature, the bath shall be heated in accordance with the appropriate schedule.

It is preferred that the slurry, and not the bath, be heated in accordance with the appropriate schedule.

10.6.1.3 After conditioning, remove the paddle and stir the slurry briskly with a spatula to ensure a uniform slurry.

10.6.1.4 Fill the fluid-loss cell as specified in 10.8.

10.6.2 Pressurized conditioning

10.6.2.1 Any consistometer referenced in Clause 9 or Annex D may be used. The following procedure applies to the most commonly used equipment.

10.6.2.2 Place the slurry in the container of the pressurized consistometer in accordance with the procedure in 9.4.

10.6.2.3 Apply pressure and heat in accordance with the thickening-time schedule which most closely simulates actual field conditions. (See optional step 10.6.4.)

10.6.2.4 At the end of the schedule, turn off the heaters and release the pressure slowly [about 1 400 kPa/s (200 psi/s)].

10.6.2.5 Remove the slurry container from the consistometer, keeping the container upright so that the oil does not mix with the slurry.

10.6.2.6 Remove the top locking ring, drive bar and collar from the shaft and the diaphragm cover.

10.6.2.7 Syringe and blot the oil from the top of the diaphragm.

10.6.2.8 Remove the diaphragm and the support ring.

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10.6.2.9 Syringe and blot any remaining oil from the top of the slurry. If the contamination is severe, discard the slurry and begin the test again.

10.6.2.10 Remove the paddle and stir the slurry briskly with a spatula to ensure a uniform slurry.

10.6.2.11 Fill the fluid-loss cell as specified in 10.8.

10.6.3 Stirred fluid-loss cell conditioning

10.6.3.1 Prepare the stirred fluid-loss cell in accordance with manufacturer's instructions.

10.6.3.2 After mixing in accordance with Clause 5, pour the slurry into a clean, dry, stirred fluid-loss cell, in accordance with manufacturer instructions.

10.6.3.3 Complete the assembly of the fluid-loss cell (screen, O-rings, end cap, etc.).

10.6.3.4 Apply 3 450 kPa ± 300 kPa (500 psi ± 50 psi) to the cell. Do not close the pressurizing valve.

10.6.3.5 While agitating with the paddle, heat the slurry in accordance with the thickening-time schedule which most closely simulates actual field conditions.

10.6.3.6 Once the slurry has reached the specified temperature (and been conditioned for an optional additional period as described in 10.6.4), if required, close the test valve and invert the pressure vessel, reconnect the nitrogen to the top valve and repressurize the nitrogen supply line (if disconnected), and open the top valve slowly.

10.6.3.7 Apply 7 000 kPa ± 300 kPa (1 000 psi ± 50 psi) differential pressure to the test cell.

10.6.3.8 Open the test valve below the screen to begin the test as specified in 10.9.

10.6.4 Extra conditioning at test temperature (optional)

Follow the procedure described in 10.6.1.2, 10.6.2.3 or 10.6.3.6 above, then hold the slurry at the specified temperature and pressure for 30 min ± 30 s or other desired conditioning period before proceeding to the next step. Document the conditioning period used.

10.7 Procedures for testing at temperatures > 88 °C (190 °F)

10.7.1 Conditioning using pressurized consistometer

10.7.1.1 Any consistometer referenced in Clause 9 or Annex D may be used. The following procedure applies to the most commonly used equipment.

10.7.1.2 Place the slurry in the container of the pressurized consistometer and begin a thickening-time test in accordance with the procedure in 9.4.

10.7.1.3 Apply pressure and heat in accordance with the thickening-time schedule which most closely simulates actual field conditions.

10.7.1.4 At the end of the schedule, turn off the heaters and cool as quickly as practical.

10.7.1.5 After the slurry has cooled to approximately 88 °C (190 °F), release the pressure slowly (about 1 400 kPa/s (200 psi/s).

10.7.1.6 Remove the slurry container from the consistometer, keeping the container upright so that oil does not mix with the slurry.

10.7.1.7 Remove the top locking ring, drive bar and collar from the shaft and the diaphragm cover.

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10.7.1.8 Syringe and blot the oil from the top of the diaphragm.

10.7.1.9 Remove the diaphragm and the support ring.

10.7.1.10 Syringe and blot any remaining oil from the top of the slurry. If the contamination is severe, discard the slurry and begin the test again.

10.7.1.11 Remove the paddle and stir the slurry briskly with a spatula to ensure a uniform slurry.

10.7.1.12 Fill the fluid-loss cell as specified in 10.8.

WARNING Overfilling of this device creates a hazard due to thermal expansion (see Table 4). Do not exceed equipment manufacturer's recommendations for maximum temperatures, pressures and volumes.

Table 4 — Vapour pressure and volume expansion of water at temperatures between 100 °C (212 °F) and 316 °C (600 °F)

Temperature °C (°F)	Water vapour 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)	1 700 (247)	1,16
232 (450)	2 910 (422)	1,21
260 (500)	4 690 (680)	1,27
288 (550)	7 200 (1 044)	1,36
316 (600)	10 620 (1 541)	1,47

10.7.2 Conditioning using stirred fluid-loss cell

10.7.2.1 Prepare the stirred fluid-loss cell in accordance with manufacturer's instructions.

10.7.2.2 Pour the slurry into a clean, dry, fluid-loss cell following 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 temperatures, pressures and volumes.

10.7.2.3 Complete the assembly of the stirred fluid-loss cell (screen, O-rings, end cap, etc.) in accordance with manufacturer's instructions.

10.7.2.4 Apply and maintain 3 500 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. Do not close the pressurizing valve.

10.7.2.5 While agitating with the paddle, heat the slurry in accordance with the thickening-time schedule which most closely simulates actual field conditions. Monitor pressures closely to prevent over-pressurizing the cell.

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10.7.2.6 Once the slurry has reached the specified temperature (and been conditioned for an optional period as described in 10.6.4), stop stirring, close the pressurizing valve, invert the pressure vessel, reconnect the nitrogen and repressurize the nitrogen supply line (if disconnected), and open the top valve slowly.

10.7.2.7 Connect the back-pressure receiver or condenser to the test valve below the screen. 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 (Table 4).

10.7.2.8 Apply 7 000 kPa ± 300 kPa (1 000 psi ± 50 psi) differential pressure to the pressure vessel.

10.7.2.9 Open the test valve below the screen and begin the test as specified in 10.9.

10.8 Filling the static fluid-loss cell

10.8.1 Prepare the fluid-loss cell. It shall be ready to be filled when the slurry conditioning period has been completed. It shall be clean and dry.

10.8.2 Preheat the fluid-loss cell to the test temperature of 88 °C ± 3 °C (190 °F ± 5 °F) for tests at 88 °C (190 °F) or greater.

10.8.3 With the test pressure supply valve closed, pour the slurry into the fluid-loss cell to 2,5 cm ± 0,6 cm (1 in ± $^1/_4$ in) below the shoulder on which the screen rests in the 12,7 cm (5 in) cell, or 5,1 cm ± 0,6 cm (2 in ± $^1/_4$ in) below the shoulder in the 25,4 cm (10 in) cell.

WARNING Overfilling of this device creates a hazard due to thermal expansion (see Table 4).

10.8.4 Place the screen and O-rings in the cell, secure the end cap in the cell. Apply pressure of 3 500 ± 300 kPa or 500 psi ± 50 psi. Do not close the test valve.

10.8.5 Heating (non-stirred cell)

10.8.5.1 For tests at temperatures ⩽ 88 °C (190 °F), start the test as quickly as possible, but no more than 6 min shall elapse from the time of completion of conditioning to the start of the test (opening the bottom valve, 10.9.1). Completion of conditioning is the end of the heat-up schedule (plus optional extra conditioning).

10.8.5.2 For tests at temperatures > 88 °C (190 °F), heat the fluid-loss cell to the test temperature as fast as the heating jacket will heat. No more than 6 min shall elapse from the time of completion of conditioning to the start of heating. Completion of conditioning is the end of the heat-up schedule (plus optional extra conditioning) and cooling. 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.

10.8.6 Close the fluid-loss cell top valve, bleed the pressure from the supply line and disconnect the nitrogen line.

10.8.7 Invert the cell so that the screen is on the bottom.

10.8.8 Attach the 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 (Table 4).

10.8.9 Connect the nitrogen line and apply a differential pressure of 7 000 kPa ± 300 kPa (1 000 psi ± 50 psi). Open the top fluid-loss cell valve to apply and maintain 7 000 kPa ± 300 kPa (1 000 psi ± 50 psi) differential pressure to the cell.

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