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Entwurf

**Erdöl- und Erdgasindustrie –
Zemente und Materialien für die Zementation von Tiefbohrungen –
Teil 2: Prüfung von Bohrloch-Zemente (ISO/DIS 10426-2:2010);
Englische Fassung prEN ISO 10426-2:2010, nur auf CD-ROM**

Petroleum and natural gas industries –
Cements and materials for well cementing –
Part 2: Testing of well cements (ISO/DIS 10426-2:2010);
English version prEN ISO 10426-2:2010; only on CD-ROM

Industries du pétrole et du gaz naturel –
Ciments et matériaux pour la cimentation des puits –
Partie 2: Essais de ciment pour puits (ISO/DIS 10426-2:2010);
Version anglaise prEN ISO 10426-2:2010, seulement en CD-ROM

Anwendungswarnvermerk

Dieser Norm-Entwurf mit Erscheinungsdatum 2010-06-21 wird der Öffentlichkeit zur Prüfung und Stellungnahme vorgelegt.

Weil die beabsichtigte Norm von der vorliegenden Fassung abweichen kann, ist die Anwendung dieses Entwurfes besonders zu vereinbaren.

Stellungnahmen werden erbeten

- vorzugsweise als Datei per E-Mail an noeg@din.de in Form einer Tabelle. Die Vorlage dieser Tabelle kann im Internet unter www.din.de/stellungnahme abgerufen werden;
- oder in Papierform an den Normenausschuss Erdöl- und Erdgasgewinnung (NÖG) im DIN, 10772 Berlin (Hausanschrift: Burggrafenstr. 6, 10787 Berlin).

Die Empfänger dieses Norm-Entwurfs werden gebeten, mit ihren Kommentaren jegliche relevante Patentrechte, die sie kennen, mitzuteilen und unterstützende Dokumentationen zur Verfügung zu stellen.

Gesamtumfang 121 Seiten

Normenausschuss Erdöl- und Erdgasgewinnung (NÖG) im DIN

Nationales Vorwort

Dieses Dokument (EN ISO 10426-2:2010) wurde vom ISO/TC 67 „Materials, equipment and offshore structures for petroleum, petrochemical and natural gas industries“, Subkomitee SC 3 „Drilling and completion fluids, and well cements“ in Zusammenarbeit mit dem Technischen Komitee CEN/TC 12 „Materialien, Ausrüstungen und Offshore-Bauwerke für die Erdöl-, petrochemische und Erdgasindustrie“ (Sekretariat AFNOR, Frankreich) erarbeitet.

Für Deutschland hat hieran der Arbeitskreis NA 109-00-01-03 AK „Bohrspülung und Zemente - Spiegelausschuss zu ISO/TC 67/SC 3“ im Normenausschuss Erdöl- und Erdgasgewinnung (NÖG) des DIN Deutsches Institut für Normung e. V. mitgearbeitet.

Diese Europäische Norm enthält unter Berücksichtigung des DIN-Präsidialbeschlusses 1/2004 nur die englische Originalfassung der ISO Norm.

Diese Norm enthält neben den gesetzlichen Einheiten auch die Einheiten „°F“, „bbl“, „BTU/(lb·°F)“, „ft“, „gal“, „lb/ft³“, „lbf“, „psi“, „qt“ und „r/min“, die im Deutschen Normenwerk nicht zugelassen sind. Es wird ausdrücklich darauf hingewiesen, dass die Anwendung dieser Einheiten im nationalen amtlichen und geschäftlichen Verkehr aufgrund des Gesetzes über Einheiten im Messwesen nicht zulässig ist.

Umrechnung:

Nicht-SI-Einheit	SI-Einheit	Umrechnung	
°F	°C	°C	= (5/9) (°F-32)
bbl	dm³	1 bbl	= 158,987 dm³
BTU/(lb·°F)	kJ/(kg·K)	1 Btu/(lbm °F)	= 4,186 8 kJ/ (kg K)
ft	m	1 ft	= 0,3048 m
gal (galone)	l	1 gal	= 3,785 l
lb/ft³	kg/m³	1 lb/ft³	= 16,018 kg/m³
lbf	N	1 lbf	= 4,448 222 N
psi (psig)	kPa	1 psi	= 6,894 757 kPa
qt (quart)	l	1 quart	= 0,946 l
r/min	min⁻¹	1 r/min	= 1 min⁻¹

Für die in diesem Dokument zitierten Internationalen Normen wird im Folgenden auf die entsprechenden Deutschen Normen hingewiesen:

ISO 10414-1	siehe	DIN EN ISO 10414-1
ISO 10414-2	siehe	DIN EN ISO 10414-2
ISO 10426-1	siehe	DIN EN ISO 10426-1
ISO 13503-3	siehe	DIN EN ISO 13503-3

ISO 10426 *Petroleum and natural gas industries — Cements and materials for well cementing* besteht aus den folgenden Teilen:

- *Part 1: Specification*
- *Part 2: Testing of well cements*
- *Part 3: Testing of deepwater well cement formulations*
- *Part 4: Preparation and testing of foamed cement slurries at atmospheric pressure*
- *Part 5: Determination of shrinkage and expansion of well cement formulations at atmospheric pressure*
- *Part 6: Methods for determining the static gel strength of cement formulations*

Änderungen

Gegenüber DIN EN ISO 10426-2:2006-02 wurden folgende Änderungen vorgenommen:

- a) Aktualisierung der Normativen Verweisungen;
- b) Erweiterung der Symboldefinitionen;
- c) Aktualisierung von Bild 1 „Common mixing device“;
- d) Neuer Abschnitt 5.4 „Test fluid conditioning“;
- e) Streichung des alten Abschnitts 9.3 „Calibration“;
- f) Neues Bild 8 „Typical hesitation squeeze pressure and temperature schedule“;
- g) Neues Bild 9 „Common high temperature, high pressure non-stirred fluid-loss cell bodies“;
- h) Neues Bild 10 „Common screwed-cap type, high temperature, high pressure double-ended fluid-loss cell“;
- i) Neues Bild 11 „Common stirred fluid-loss apparatus“;
- j) Neues Bild 12 „Cell and components of common stirred fluid-loss apparatus“;
- k) Streichung des alten Abschnitts 11 „Permeability tests“;
- l) Neues Bild 13 „Typical rotational viscometer schematic“;
- m) Neues Bild 14 „Rotor and bob dimensions (R1-B1)“;
- n) Streichung der alten Bilder 11 „Illustration of shear stress-shear rate behaviour of various fluids on a linear plot“ und 12 „Illustration of shear stress-shear rate behaviour of various fluids on a log-log plot“;
- o) Streichung des alten Abschnitts 13 „Calculation of pressure drop and flow regime for cement slurries in pipes and annuli“;
- p) Neues Bild 16 „Compatibility testing flowchart“;
- q) Neues Bild 17 „Typical conductivity titration vs. fresh water spacer volume percent in SSST apparatus“;
- r) Neuer normativer Anhang B „Calibration and verification of well cement testing equipment“.

Nationaler Anhang NA (informativ)

Begriffe und Symbole

Die Benennung der folgenden Begriffe und Symbole sind identisch mit der Benennung in der englischen Fassung.

3.1 Begriffe

Für die Anwendung dieses Dokuments gelten die folgenden Begriffe.

3.1.1

Reindichte

Dichte eines Materials ohne die dessen Partikel umgebende Flüssigkeit. Sie ist der relativen Dichte ähnlich und lässt sich durch Multiplizieren der relativen Dichte eines Materials mit der Dichte von Wasser bei 4 °C, d. h. 1 000 kg/m³ (8,345 4 lbm/gal) berechnen

3.1.2

absolutes Volumen

Kehrwert der Reindichte

3.1.3

Zement-Zusatz

Zusatz

Material, das einer Zementbrühe hinzugefügt wird, um eine gewünschte Eigenschaft zu verändern oder zu verbessern

3.1.4

Ringkammer

Raum zwischen Rohr und Bohrlochwand oder einem äußeren Rohr

3.1.5

Chargenmischen

Prozess, bei dem das gesamte Volumen der Zementbrühe gemischt wird, bevor es in das Bohrloch gegeben wird

3.1.6

Bearden-Einheiten der Konsistenz

B_c

Einheiten für die Angabe der Konsistenz einer Zementbrühe, wenn diese in einem druckbeaufschlagten Konsistometer bestimmt wird

3.1.7

Umlauftemperatur am Bodenloch

T_{BHC}

Temperatur, die am Boden des Bohrlochs gemessen wird, wenn Flüssigkeit in diesem Bohrloch umläuft

3.1.8

Schüttdichte

volumenbezogene Masse eines Materials im Trockenzustand einschließlich der zwischen dessen Partikeln anstehenden Luft

3.1.9**Futterrohrzementierung**

Prozess des Einbringens von Zement zur vollständigen oder teilweisen Verfüllung des Raums zwischen einer vollständigen Abfolge von Futterrohren und dem Bohrloch

3.1.10**Zement****Portlandzement**

durch das Mahlen von Klinker gewonnenes Material, das im Wesentlichen aus hydraulischen Calciumsilikaten und -aluminaten besteht und üblicherweise eine oder mehrere Form(en) von Calciumsulfat enthält, die während des Mahlens zugegeben wird (werden)

3.1.11**Zementmischung**

Gemisch aus Trockenzement und anderen trockenen Materialien

3.1.12**Zementklasse**

dem ISO-Klassifikationssystem entsprechende Bezeichnung von Bohrlochzement nach festgelegten Güteanforderungen von ISO 10426-1

3.1.13**Zementgüte**

dem ISO-System entsprechende Bezeichnung zur Angabe der Beständigkeit eines bestimmten Zements gegen Sulfate nach Güteanforderungen von ISO 10426-1

3.1.14**Klinker**

im Ofen erzeugte geschmolzene Materialien, die für die Herstellung von Zement gemeinsam mit Calciumsulfat vermahlen werden

3.1.15**Kompatibilität**

Fähigkeit, ein flüssiges Gemisch zu bilden, das keine unerwünschten chemischen und/oder physikalischen Reaktionen zeigt

3.1.16**Druckfestigkeit**

Festigkeit einer erstarrten Zementprobe, die anhand der Druckkraft gemessen wird, die erforderlich ist, um diese Probe bersten zu lassen. Die Druckfestigkeit wird als Kraft je Flächeneinheit angegeben

3.1.17**Konsistometer**

Gerät, mit dem sich die Verdickungszeit einer Zementbrühe unter bestimmten Temperatur- und Druckbedingungen messen lässt

ANMERKUNG Druckbeaufschlagte Konsistometer können zur Konditionierung von Flüssigkeiten vor Beginn der Prüfung und zur Bestimmung der Verdickungszeit von arktischen Zementbrühen verwendet werden.

3.1.18**Einpresszementieren mit kontinuierlichem Pumpen**

Prozess des Einpresszementierens, bei dem ununterbrochen gepumpt wird

3.1.19**Bohrflüssigkeit**

Flüssigkeit, die beim Bohren des Bohrlochs dazu verwendet wird, das Bohrklein zu entfernen, den Bohrkopf zu kühlen und aus der Formation austretende Flüssigkeiten am Eindringen in das Bohrloch zu hindern

3.1.20**Sackäquivalent**

Masse des Gemischs aus Flugasche oder Puzzolanerde und Portlandzement, die dasselbe absolute Volumen wie ein Sack Portlandzement (94 lbm) hat

3.1.21**Filtrat**

Flüssigkeit, die während einer Filtrationsverlustprüfung aus einer Zementbrühe herausgepresst wird

3.1.22**Flugasche**

fein verteilter Rückstand, der bei der Verbrennung von gemahlener oder pulverisierter Kohle anfällt. Flugasche hat puzzolanische Eigenschaften

ANMERKUNG Für eine nähere Beschreibung siehe Abschnitt 14.

3.1.23**ungebundene Flüssigkeit**

farbige oder farblose Flüssigkeit, die von einer Zementbrühe abgesondert wird, wenn sie unter statischen Bedingungen stehen gelassen wird

3.1.24**Frost-Tau-Wechsel**

festgelegter Zyklus von Temperaturbedingungen über und unter dem Gefrierpunkt von Wasser, dem eine Zementprobe ausgesetzt wird

3.1.25**Aufheizrate**

R_h

Geschwindigkeit der Änderung der Temperatur einer Zementbrühe von der Oberflächentemperatur T_{SS} bis auf die vorhergesagte Umlauftemperatur am Bodenloch, T_{TPBHC}

3.1.26**verzögertes Einpresszementieren**

(en: hesitation-squeeze-cementing operation)

Verfahren des Einbringens von Zement während eines Einpresszementiervorgangs, bei dem abwechselnd gepumpt bzw. nicht gepumpt wird

3.1.27**Überzugzementieren**

(en: liner cementing)

Vorgang des Zementierens in Ringform, bei dem der obere Teil der zu zementierenden Schale die Oberfläche nicht erreicht

3.1.28**Lastrahmen**

Rahmen, mit dessen Hilfe Zementblöcke zur Bestimmung ihrer Druckfestigkeit so lange mit einer Last beaufschlagt werden, bis sie bersten

3.1.29**Schlamm**

Flüssigkeit, die im Wesentlichen viskosiert und gegebenenfalls beschwert ist und bei Bohr- oder Instandsetzungs-(Workover-)vorgängen im Bohrloch umläuft

3.1.30**Abdichtzementieren**

(en: plug cementing)

Prozess des Einbringens eines Zementvolumens in ein Bohrloch, um das Bohrloch zu verschließen

3.1.31**Puzzolan**

kieselhaltige oder kiesel- und tonerdehaltiges Material, das als solches nur geringen oder gar keinen Zementwert hat, das jedoch in fein gemahlener Form und in Anwesenheit von Feuchtigkeit mit Calciumhydroxid reagiert und so ein zementartiges Material bildet

ANMERKUNG Für eine nähere Beschreibung siehe Abschnitt 14.

3.1.32**Vorspülmittel**

Flüssigkeit, die weder viskosiert noch durch unlösliche, feste Beschwerungsmittel verdichtet ist

3.1.33**Druckabfallrate** R_{pd}

Geschwindigkeit, mit der der Druck bei einer Prüfung der Verdickungszeit vom Druck am Bodenloch p_{BH} zum Druck am oberen Ende der Zementsäule p_{TOC} hin abfällt

3.1.34**Druckanstiegsrate** R_{pu}

Geschwindigkeit, mit der der Druck bei einer Prüfung der Verdickungszeit vom Anfangsdruck zum Druck am Bodenloch hin ansteigt

3.1.35**druckbeaufschlagte Nachbehandlungskammer**

Gerät, das zur Nachbehandlung einer Zementprobe unter bestimmten Temperatur- und Druckbedingungen für spätere Prüfungen, wie z. B. die der Druckfestigkeit, der Sedimentation usw., verwendet wird

3.1.36**druckbeaufschlagte Dichtewaage für Flüssigkeiten**

Waage vom Typ Balkenwaage, die zur Messung der Flüssigkeitsdichte unter Druck verwendet wird, um die Auswirkungen von in der Flüssigkeit eingeschlossener Luft auf ein Mindestmaß zu reduzieren

3.1.37**relative Dichte****spezifische Dichte**

Verhältnis der Masse einer Substanz zur Masse eines gleichen Volumens einer Normsubstanz bei einer bestimmten Bezugstemperatur

ANMERKUNG Als Standardsubstanz wird in der Regel Wasser verwendet; die Bezugstemperatur beträgt in der Regel 4 °C.

3.1.38**Sack**

Maßeinheit für Zement, der ein Schüttvolumen von einem Kubikfuß einnimmt (üblicherweise 94 lbm für Portlandzement)

3.1.39**Sedimentation**

Entmischung und Absetzen von Feststoffen in einer Zementbrühe

3.1.40**Behälter für Zementbrühe**

Behälter, der dazu verwendet wird, die Zementbrühe für Konditionierungs- oder Prüfzwecke in einem Konsistometer zu halten

3.1.41**Stabilitätsprüfung für Zementbrühe**

Prüfung zur Bestimmung des Sedimentationsgrades und/oder der Entstehung ungebundener Flüssigkeiten in einer Zementbrühe

3.1.42**Schallfestigkeit**

Druckfestigkeit einer Zementprobe, die durch Messung der Geschwindigkeit des Schalls beim Durchlaufen des Zements und rechnerische Korrelation mit den Druckfestigkeitsmesswerten erhalten wird

3.1.43**Trennsubstanz**

viskosierte Flüssigkeit, die sich durch unlösliche, feste Beschwerungsmittel verdichten lässt. Trennsubstanzen werden verwendet, um Bohrflüssigkeiten und Zementbrühen zu trennen

3.1.44**Einpresszementieren**

(en: squeeze-cementing)

Hilfsprozess, bei dem Zementbrühe unter Druck in einen bestimmten Teil des Bohrlochs, wie z. B. in einen Riss oder eine Öffnung gepresst wird

3.1.45**Anfangsdruck**

P_S

Druck, der zu Beginn der Prüfung der Verdickungszeit auf die Prüfprobe aufgebracht wird

3.1.46**statische Flüssigkeitsverlustprüfung**

Prüfung zur Bestimmung des Verlustes einer Zementbrühe an Filtrat, wenn diese bei einem Differenzdruck von 6 900 kPa (1 000 psi) auf ein 325er Maschensieb gegeben wird

3.1.47**statische Stabilitätsprüfung**

Prüfung zur Bestimmung des Sedimentationsgrades und der Entstehung ungebundener Flüssigkeiten in einer Zementbrühe

3.1.48**Prüfgerät mit Rührfunktion für die Flüssigkeitsverlustprüfung**

Prüfgerät, das speziell so ausgelegt ist, dass die Zementbrühe im selben Behälter konditioniert werden kann, in dem sie der statischen Flüssigkeitsverlustprüfung unterzogen wird

3.1.49**Verdickungszeit**

Zeit, die von der ersten Anwendung von Druck und Temperatur bis zu dem Moment verstreicht, in dem die Brühe eine Konsistenz erreicht, von der an sie als nicht mehr pumpfähig erachtet wird (z. B. 70 B_c oder 100 B_c)

ANMERKUNG Die Ergebnisse einer Prüfung der Verdickungszeit liefern einen Hinweis auf die Dauer des Zeitraums, über den die betreffende Zementbrühe unter Prüfbedingungen pumpfähig ist.

3.1.50**Chargenwägemischer****Waagentank**

Gerät oder System zum Wägen und Mischen von Zement mit trockenen Zusätzen für einen Zementiervorgang

3.1.51**Bohrlochsimulationsprüfung**

Prüfung, deren Parameter nach Bedarf beim Entwurf angesetzt und geändert werden, um die in einem Bohrloch herrschenden Bedingungen nachzubilden

3.2 Symbole

Für die Anwendung des vorliegenden Teils von ISO 10426 gelten die in Tabelle 1 angegebenen Symbole. Diese Liste erhebt keinen Anspruch auf Vollständigkeit.

Tabelle 1 — Symbole

Symbol	Bedeutung
B_C	Konsistenz, angegeben in Bearden-Einheiten
g	Beschleunigung im freien Fall
h_{TOCTVD}	wahrer Wert der vertikalen Tiefe ab der Zementoberfläche
h_{TVD}	wahrer Wert der vertikalen Tiefe
k	Konsistenzindex
m	Masse
m_a	Masse der Zusätze
m_C	Masse des Zements
m_S	Masse der Zementbrühe
m_W	Masse des Wassers
n	dimensionsloser Index des Fließverhaltens
p_{BH}	Druck am Bodenloch ^a
p_S	Anfangsdruck
p_{TOC}	Druck an der Zementoberfläche
q	Pumpgeschwindigkeit
$R_{\Delta T}$	Geschwindigkeit der Temperaturänderung
R_{pd}	Druckabfallrate
R_{pu}	Druckanstiegsrate
T	Drehmoment
TT	Übergangszeit
T_{BHC}	Umlauftemperatur am Bodenloch
T_{BHS}	statische Temperatur am Bodenloch

Symbol	Bedeutung
T_{PBHC}	vorhergesagte Umlauftemperatur am Bodenloch
T_{PS}	vorhergesagte Einpresstemperatur
T_{PP}	vorhergesagte Abdichttemperatur
T_{PSP}	vorhergesagte Einpress- oder Abdichttemperatur am Bodenloch
Δ_{PT}	unechtes Temperaturgefälle ^b
T_{SS}	Oberflächentemperatur der Zementbrühe
T_{TOCC}	Umlauftemperatur an der Zementoberfläche
T_{TOCS}	statische Temperatur an der Zementoberfläche
t	verstrichene Zeit
t_a	Zeit, die für die Verlagerung der Stirnfläche der Zementbrühe vom Boden der Schalung bis zur Oberkante der Ringsäule benötigt wird
t_d	Zeit, die für die Verlagerung der Stirnfläche der Zementbrühe von der Oberfläche bis zum Boden des Bohrlochs benötigt wird
V_{fp}	Endvolumen im gepackten Zustand
V_{30}	Volumen des aufgefangenen Filtrats nach 30 min
V_{abs}	absolutes Volumen der Zusätze
V_a	Volumen des Zements in der Ringkammer
V_c	absolutes Volumen des Zements
V_F	Volumen der ungebundenen Flüssigkeit
V_p	Volumen des Rohrs
V_s	Volumen der Zementbrühe
V_t	Volumen des zum Zeitpunkt des Stickstoffdurchblasens aufgefangenen Filtrats
V_w	Wasservolumen
Z	Schallimpedanz
γ	Schergeschwindigkeit
μ_p	plastische Viskosität
$\Delta\rho_{sc}$	Dichte des abgebundenen Zements im Verhältnis zur Dichte der Zementbrühe
ρ_a	Dichte des Zusatzes
ρ_{bulk}	durchschnittliche Schüttdichte
ρ_c	Dichte des Zements
ρ_{af}	Dichte der in der Ringkammer anstehenden Flüssigkeit
ρ_{df}	Dichte der Bohrflüssigkeit

Symbol	Bedeutung
ρ_{LAB}	Schüttgewicht im losen Zustand
ρ_{PAB}	Schüttgewicht im gepackten Zustand
ρ_s	Dichte der Zementbrühe
ρ_{set}	Dichte des erstarrten Zementsegments
ρ_w	Dichte von Wasser
τ	Scherspannung
τ_y	Fließspannung
φ	Flüssigkeitsanteil
^a Hydrostatischer Druck am Boden des Bohrlochs, der mit Hilfe des wahren Werts der vertikalen Tiefe und der Flüssigkeitsdichten im Bohrloch berechnet wird. ^b Gefälle in °C/100 m (°F/100 Fuß), das anhand des Unterschieds zwischen der höchsten aufgezeichneten statischen Temperatur am Bodenloch und 80 °F berechnet wird.	

Nationaler Anhang NB (informativ)

Literaturhinweise

DIN EN ISO 10414-1, *Erdöl- und Erdgasindustrie — Feldprüfung von Bohrspülungen — Teil 1: Flüssigkeiten auf Wasserbasis*

DIN EN ISO 10414-2, *Erdöl- und Erdgasindustrie — Feldprüfung von Bohrflüssigkeiten — Teil 2: Flüssigkeiten auf Ölbasis*

DIN EN ISO 10426-1, *Erdöl- und Erdgasindustrie — Zemente und Materialien für die Zementation von Tieflochbohrungen — Teil 1: Anforderungen*

DIN EN ISO 13503-3, *Erdöl- und Erdgasindustrie — Komplettierungsflüssigkeiten und -materialien — Teil 3: Prüfung von schweren Solen*

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English Version

**Petroleum and natural gas industries —
Cements and materials for well cementing —
Part 2: Testing of well cements
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Industries du pétrole et du gaz naturel —
Ciments et matériaux pour la cimentation des puits —
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(ISO/DIS 10426-2:2010)

Erdöl- und Erdgasindustrie —
Zemente und Materialien für die Zementation
von Tiefbohrungen —
Teil 2: Prüfung von Bohrloch-Zemente
(ISO/DIS 10426-2:2010)

This draft European Standard is submitted to CEN members for parallel enquiry. It has been drawn up by the Technical Committee CEN/TC 12.

If this draft becomes a European Standard, 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.

This draft European Standard was established by CEN in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN Management Centre has the same status as the official versions.

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Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

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COMITÉ EUROPÉEN DE NORMALISATION
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Foreword

This document (prEN ISO 10426-2:2010) has been prepared by Technical Committee ISO/TC 67 "Materials, equipment and offshore structures for petroleum, petrochemical and natural gas industries" in collaboration with Technical Committee CEN/TC 12 "Materials, equipment and offshore structures for petroleum, petrochemical and natural gas industries" the secretariat of which is held by AFNOR.

This document is currently submitted to the parallel Enquiry.

This document will supersede EN ISO 10426-2:2003.

Endorsement notice

The text of ISO/DIS 10426-2:2010 has been approved by CEN as a prEN ISO 10426-2:2010 without any modification.

Introduction

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. The units do not necessarily represent a direct conversion of SI to US Customary units, or US Customary to SI. Consideration has been given to the precision of the instrument making the measurement. For example, thermometers are typically marked in one degree increments, thus temperature values have been rounded to the nearest degree.

In this part of ISO 10426, calibrating an instrument refers to assuring the accuracy of the measurement. Accuracy is the degree of conformity of a measurement of a quantity to its actual or true value. Accuracy is related to precision, or reproducibility of a measurement. Precision is the degree to which further measurements or calculations will show the same or similar results. Precision is characterized in terms of the standard deviation of the measurement. The results of calculations or a measurement can be accurate, but not precise, precise but not accurate, neither and both. A result is valid if it is both accurate and precise.

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

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

1 Scope

This part of ISO 10426 specifies methods and gives recommendations for the testing of cement slurries and related materials under simulated well conditions.

2 Normative references

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

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

ISO 10414-2, *Petroleum and natural gas industries — Field testing of drilling fluids — Part 2: Oil-based fluids*

ISO 10426-1, *Petroleum and natural gas industries — Cements and materials for well cementing — Part 1: Specification*

ISO 13503-3, *Petroleum and natural gas industries — Completion fluids and materials — Part 3: Testing of heavy brines*

ASTM C 109/C 109M-07, *Standard test method for compressive strength of hydraulic cement mortars (using 2 in. or [50 mm] cube specimens)*

ASTM C 188-95, *Standard test method for density of hydraulic cement*

ASTM C 618-08, *Standard Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use in Concrete*

ASTM E 220-07a, *Standard Test Method for Calibration of Thermocouples by Comparison Techniques*

3 Terms, definitions, and symbols

3.1 Terms and definitions

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

3.1.1

absolute density

density of a material without the fluid around the particles. It is similar to the relative density and can be obtained by multiplying the relative density of a material by the density of water at 4 °C, 1000 kg/m³ (8,345 4 lbm/gal)

3.1.2

absolute volume

reciprocal of absolute density

3.1.3

additive

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

3.1.4

annulus

space between the pipe and the wellbore wall or an outer pipe

3.1.5

batch mix

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

3.1.6

Bearden units of consistency

B_c

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

3.1.7

bottomhole circulating temperature (T_{BHC})

temperature found at the bottom of the wellbore while the well is being circulated

3.1.8

bulk density

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

3.1.9

casing cementing

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

3.1.10

cement

Portland cement

material formed by the grinding of clinker generally consisting of hydraulic calcium silicates and aluminates and usually containing one or more of the forms of calcium sulfate added during grinding

3.1.11

cement blend

mixture of dry cement and other dry materials

3.1.12

cement class

designation under the ISO system of classifications of well cement according to defined specifications of ISO 10426-1

3.1.13

cement grade

designation under the ISO system for denoting the sulfate resistance of a particular cement according to specifications of ISO 10426-1

3.1.14

clinker

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

3.1.15

compatibility

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

3.1.16

compressive strength

strength of a set cement sample measured by the force required to cause it to fail in compression. Compressive strength is expressed as force per unit area.

3.1.17

consistometer

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

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

3.1.18

continuous-pumping squeeze-cementing operation

squeeze-cementing operation that does not involve interruptions in pumping

3.1.19

drilling fluid

fluid used during the drilling of the well to remove cuttings, cool the drill bit and prevent formation fluids from entering the wellbore

3.1.20

equivalent sack

mass of the blend of fly ash or pozzolan and Portland cement that has the same absolute volume as a sack (94 lbm) of Portland cement

3.1.21

filtrate

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

3.1.22

fly ash

finely divided residue from the combustion of ground or powdered coal. Fly ash has pozzolanic properties

NOTE See Clause 14 for further description.

3.1.23

free fluid

coloured or colourless liquid that has separated from a cement slurry when standing in a static condition

3.1.24

freeze-thaw cycle

a prescribed cycle of exposure of a cement sample to temperatures above and below the freezing point of water

3.1.25

heat-up rate

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

3.1.26

hesitation-squeeze-cementing operation

method of placement of cement during squeeze-cementing operation that involves alternate pumping and non-pumping periods

3.1.27

liner cementing

annular cementing operations for which the top of the casing being cemented does not reach the surface

3.1.28

load frame

frame to place a load on cement blocks until they fail, in order to determine their compressive strength

3.1.29**mud**

a fluid, generally viscosified and possibly weighted, that is circulated through the wellbore during drilling or workover operations

3.1.30**plug cementing**

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

3.1.31**pozzolan**

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

NOTE See Clause 14 for further description.

3.1.32**preflush**

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

3.1.33**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

3.1.34**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

3.1.35**pressurized curing chamber**

apparatus used for curing a sample of cement under temperature and pressure for subsequent tests such as compressive strength, sedimentation, and so forth

3.1.36**pressurized fluid density balance**

beam-type balance used to measure fluid density under pressure to minimize the effects of air entrained in the fluid

3.1.37**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.

3.1.38**sack**

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

3.1.39**sedimentation**

separation and settling of solids in a cement slurry

3.1.40

slurry container

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

3.1.41

slurry stability test

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

3.1.42

sonic strength

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

3.1.43

spacer

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

3.1.44

squeeze-cementing

remedial process in which cement slurry is forced under pressure into a specific portion of the well such as a fracture or opening

3.1.45

starting pressure

p_s

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

3.1.46

static fluid loss test

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

3.1.47

static stability test

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

3.1.48

stirred fluid-loss apparatus

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

3.1.49

thickening time

time elapsed from the initial application of pressure and temperature to the time at which the slurry reaches a consistency deemed sufficient to make it unpumpable (for example, 70 B_c or 100 B_c).

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

3.1.50

weigh batch mixer

scale tank

device or system for the weighing and blending of cement with dry additives for a cementing operation

3.1.51

well simulation test

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

3.2 Symbols

For the purposes of this part of ISO 10426, the symbols given in Table 1 apply. This list is not exhaustive.

Table 1 — Symbols

Symbol	Meaning
B_c	consistency expressed in Bearden units
g	acceleration of freefall
h_{TOCTVD}	top-of-cement true vertical depth
h_{TVD}	true vertical depth
k	consistency index
m	mass
m_a	additive mass
m_c	cement mass
m_s	slurry mass
m_w	water mass
n	dimensionless flow behaviour index
p_{BH}	bottom-hole pressure ^a
p_s	starting pressure
p_{TOC}	top-of-cement pressure
q	pump rate
$R_{\Delta T}$	rate of temperature change
R_{pd}	rate of pressure decrease
R_{pu}	rate of pressure increase
T	torque
TT	transit time
T_{BHC}	bottom-hole circulating temperature
T_{BHS}	bottom-hole static temperature
T_{PBHC}	predicted bottom-hole circulating temperature
T_{PS}	predicted squeeze temperature
T_{PP}	predicted plug temperature
T_{PSP}	predicted bottom-hole squeeze or plug temperature
Δ_{PT}	pseudo-temperature gradient ^b
T_{SS}	slurry surface temperature
T_{TOCC}	top-of-cement circulating temperature
T_{TOCS}	top-of-cement static temperature
t	elapsed time
t_a	time to displace the leading edge of the cement slurry from bottom of the casing to the top of the annular column
t_d	time to displace the leading edge of cement slurry from the surface to the bottom of the wellbore

Table 1 (continued)

Symbol	Meaning
V_{fp}	final packed volume
V_{30}	volume of filtrate collected at 30 minutes
V_{abs}	additive absolute volume
V_a	annular volume of cement
V_c	cement absolute volume
V_F	volume of free fluid
V_p	volume of the pipe
V_s	slurry volume
V_t	volume of filtrate collected at the time nitrogen blows through
V_w	water volume
Z	acoustic impedance
γ	shear rate
μ_p	plastic viscosity
$\Delta\rho_{sc}$	density of the set cement relative to the cement slurry
ρ_a	density of additive
ρ_{bulk}	average bulk density
ρ_c	density of cement
ρ_{af}	density of annular fluid
ρ_{df}	density of drilling fluid
ρ_{LAB}	loose apparent bulk density
ρ_{PAB}	packed apparent bulk density
ρ_s	density of slurry
ρ_{set}	density of the set cement segment
ρ_w	density of water
τ	shear stress
τ_y	yield stress
φ	fluid fraction
a Hydrostatic pressure at the bottom of the well, calculated using the true vertical depth and the fluid densities in the wellbore. b Gradient in °C/100 m (°F/100 ft), calculated from the difference between the maximum recorded bottom-hole static temperature and 80 °F.	

4 Sampling

4.1 General

Samples of the cement, cement blend, solid and liquid additives, and mixing water may be required to test a slurry in accordance with this part of ISO 10426. Some commonly used sampling techniques are described in this clause.

4.2 Sampling cement at a field location

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

4.3 Sampling cement blends at a blending facility

Cement blends may be sampled from the weigh batch mixer (scale tank), bulk transport, or extracted from the flow lines during transfer. The cement and dry additives should be thoroughly blended prior to sampling. Samples from the bulk container may be extracted in accordance with 4.2. Samples extracted from a flow line during a transfer may be taken from a properly installed sample valve, diverted flow sampler, or automatic in-line sampling device (Figure 1c, 1d and 1e) using a procedure designed to give a representative sample of the blend being transferred. The samples should be prepared, packaged, and labelled (4.7). Sample volume should be sufficient to perform the desired testing.

4.4 Sampling dry cement additives

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

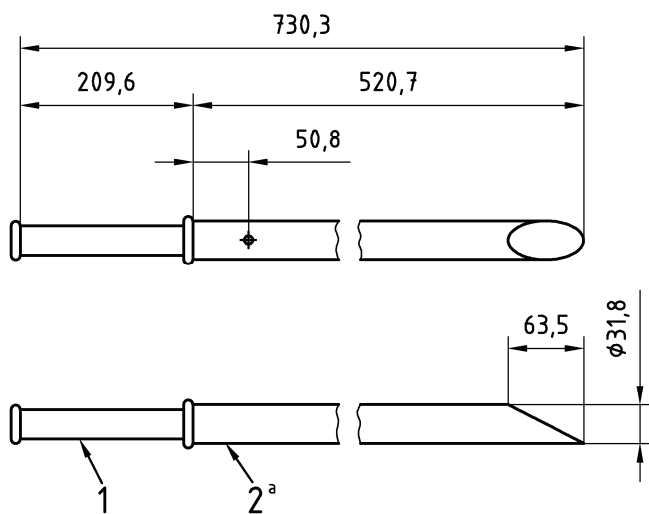
4.5 Sampling liquid cement additives

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

4.6 Sampling mixing water

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

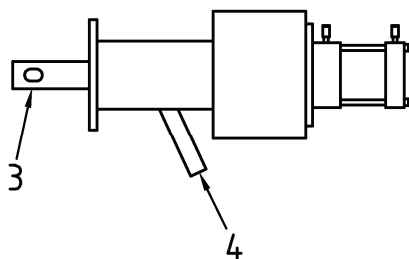
Dimensions in millimetres



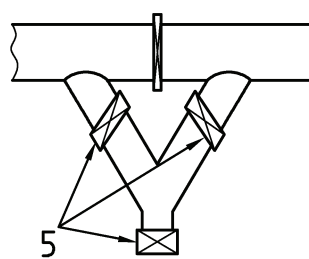
a) Tube sampler for sacked cement



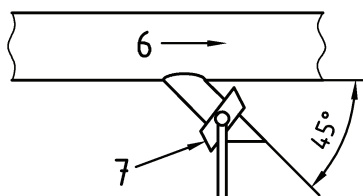
b) Tube sampler for bulk cement



c) Automatic probe sampler



d) Modified diverted-flow sampler



e) Top view – Lateral sampler

Key

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

^a Approximate volume = 320 ml

Figure 1 — Commonly used sampling devices

4.7 Shipping and storage

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

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

4.8 Sample preparation prior to testing

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

If the sample was not shipped in a leak-proof container, the sample should be transferred into a suitable, leak-proof container. The container should be properly labelled, dated, and stored in a dry place where room temperatures remain fairly constant. Repackaging should be indicated. At the time of testing, each sample should be examined for quality and thoroughly blended just prior to slurry preparation (see Clause 5).

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

4.9 Sample disposal

Sample disposal should comply with all regulatory requirements.

5 Preparation of slurry

5.1 General

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

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

5.2 Apparatus

5.2.1 All apparatus are calibrated as per Annex B.

5.2.2 Electronic balances, with an accuracy within $\pm 0,1$ % of the indicated load.

5.2.3 Mechanical balances, with weights having an accuracy within $\pm 0,1$ % of the indicated load.

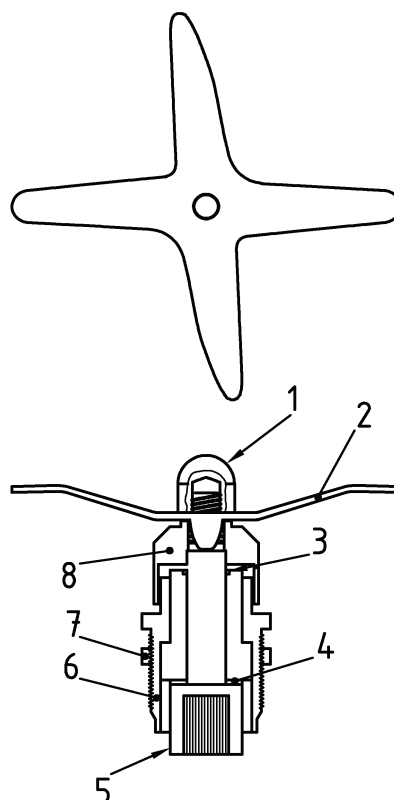
5.2.4 Mixing device, of capacity 1 litre (1 quart), having bottom drive and a blade-type mixer.

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

The mixing blade should be replaced with a new blade before mass loss greater than 10 % has occurred (see B.3.3). If the mixing device leaks at any time during the mixing procedure, the contents should be discarded, the leak repaired, and the procedure restarted.



Figure 2 — Common mixing device



Key

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

Figure 3 — Common blade assembly

5.3 Procedure

5.3.1 Determination of relative density (specific gravity) of components

5.3.1.1 General

The relative density of different batches of cement can vary due to natural variations in the composition of the raw materials used in the manufacturing process. Cement relative density may vary from 3,10 to 3,25. This can result in variation in slurry densities by as much as 34 kg/m^3 (0,28 lbm/gal) for slurries with constant water-to-solids 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.

5.3.1.2 Relative density of cement and dry additives

The relative density of the cement and any dry additives can be determined by the use of a gas pycnometer. Alternatively, a Le Chatelier flask as outlined in ASTM C 188-95 may be used for determining the relative density of these materials.

5.3.1.3 Relative density of mix water and liquid additives

The relative density of the mix water and any liquid additives may be determined by the use of a hydrometer as outlined in ISO 13503-3. Alternatively, a gas 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 is 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.

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

$$V_s = V_c + V_w + V_{abs} \quad (1)$$

$$m_s = m_c + m_w + m_a \quad (2)$$

$$r_s = m_s / V_s \quad (3)$$

where

V_s is the slurry volume, in millilitres;

V_c is the cement absolute volume, in millilitres;

V_w is the water volume, in millilitres;

V_{abs} is the additive absolute 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.

$$V_c = m_c / r_c \quad (4)$$

where

ρ_c is the density of cement, in grams per millilitre.

$$V_w = m_w / \rho_w \quad (5)$$

where

ρ_w is the density of water, in grams per millilitre.

$$V_a = m_a / \rho_a \quad (6)$$

where

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

5.3.2 Temperature of water and cement

The temperature of the mix water, dry cement or cement blend, and mixing and blending devices should be representative of field mixing conditions. If field conditions are unknown, the temperature of the mix water (including any pre-mixed additives) and dry cement should be $23\text{ °C} \pm 1\text{ °C}$ ($73\text{ °F} \pm 2\text{ °F}$) immediately prior to mixing. In all cases, the temperatures of the mix water and dry cement should be measured and documented.

5.3.3 Mix water

Water composition can affect slurry performance. Water from the field source should be used. If field mix water is unavailable, water of similar composition should be used. If field mix water composition is unknown, deionized, distilled, or tap water may be used. The type of mix water should be documented in the lab report. The mix water should be weighed into a clean, dry, mixing container. If used, liquid additives may be weighted into the water in the mixing container or may be weighed separately and added to the water while agitating at low speed. No excess water should be added to compensate for evaporation or wetting.

5.3.4 Mixing of cement and water

Weigh dry materials and then blend thoroughly and uniformly prior to adding them to the mix fluid. Place the mixing container with the required mass of mix water and liquid additives (if previously added) on the mixer base. Turn on the motor and maintain at $4\,000\text{ r/min} \pm 200\text{ r/min}$. If additives are present in the mix water, stir at the above rotational speed to thoroughly disperse them prior to the addition of cement. In some cases, additives (dry or liquid) may be added to the mix water in the field. In such cases, the additives should be added to the mix water while mixing at low speed. In certain cases, the order of addition of the additives to the mix water can be critical, in which case, the additives should be mixed in the order that they will be mixed in the field. Document any special mixing procedures and mixing time.

While mixing at $4\,000\text{ r/min} \pm 200\text{ r/min}$, add the cement or cement/dry additive blend at a uniform rate in not more than 15 s, if possible. Some slurry designs may take longer to completely wet the cement blend, however, the time used to add the blend should be kept to a minimum. If more than 15 s were required to add the cement blend to the water, document that time. After 15 s or when all the dry materials have been added to the mix water, if longer than 15 s, place the cover on the mixing container and continue mixing at $12\,000\text{ r/min} \pm 500\text{ r/min}$ for $35\text{ s} \pm 1\text{ s}$. Measure and document the rotational speed under load.

5.4 Test fluid conditioning

5.4.1 General

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

5.4.2 Procedure: pressurized conditioning

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

- a) Within 1 min after mixing according to 5.3.4, pour the test fluid into the slurry container of a pressurized consistometer.
- b) Heat to T_{PBHC} in accordance with the pressure/temperature schedule designed to simulate conditions in the well. Hold test temperature and pressure for 30 min $\pm$ 30 s to allow the test fluid temperature to reach equilibrium. This hold time may be modified to simulate cementing operations. Proper note should be made of this in the test report.
- c) If the temperature is greater than 88 °C (190 °F), cool the test fluid as quickly as possible to 88 °C (190 °F). If the boiling point of water at the test location is less than 100 °C (212 °F), adjust temperatures accordingly. Maintain test pressure while decreasing the temperature. When 88 °C (190 °F) is reached, release the pressure slowly (about 1 400 kPa/s [200 psi/s]).
- d) Remove the slurry container from the consistometer, keeping the container upright so that the oil does not mix with the test fluid.
- e) Remove the flange ring and the backup plate and syringe or blot oil from the top of the slurry container.
- f) Remove the diaphragm and the support ring. Syringe or blot the top of the test fluid with an absorbent cloth or paper towel to remove any visible oil. If the contamination is severe, discard the test fluid and condition a fresh test fluid.
- g) Remove the paddle and stir the test fluid briskly with a spatula to ensure it is uniform.
- h) Continue with the desired test.

5.4.3 Procedure: atmospheric-pressure conditioning

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

- a) Within 1 min after mixing according to 5.3.4, fill the slurry container with test fluid to the fill line of the atmospheric-pressure consistometer.
- b) Heat the test fluid from ambient temperature or a temperature that simulates field surface mixing temperature to T_{PBHC} in accordance with the thickening-time schedule that most closely simulates actual field conditions. If the atmospheric consistometer is not capable of heating on a controlled temperature ramp, heat as rapidly as the instrument is capable and record the time to T_{PBHC} . If the atmospheric-pressure consistometer is not equipped to measure test fluid temperature, the bath should be heated in accordance with the appropriate schedule.
- c) With test fluids containing additives that are not affected by sudden temperature increases, the slurry container may be placed in the heating bath pre-heated to the test temperature (± 3 °C [± 5 °F]) or other initial temperature that is appropriate. Care should be taken to prevent unusual behaviour such as gelation, increase in free fluid, or poor response to additives such as retarders and fluid loss control agents, when conditioning the fluid.
- d) After the slurry reaches test temperature (the temperature must be verified by measurement), hold the test temperature for 30 min $\pm$ 30 s to allow the test fluid temperature to reach equilibrium. This hold time may be modified to simulate cementing operations. However, proper note should be made of this in the test report.
- e) Remove the paddle and stir the test fluid briskly with a spatula to ensure it is uniform.
- f) Continue with the desired test.

6 Determination of slurry density

6.1 Apparatus

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

6.2 Procedure

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

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

CAUTION — Slurry can be expelled forcibly.

After 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 ring on the cup.

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

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

Key

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

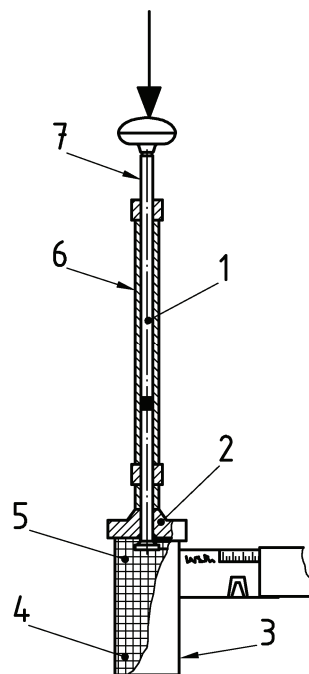


Figure 4 — Common pressurized fluid density balance

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

6.2.6 Rinse off the exterior of the cup and wipe dry. Then place the instrument on the knife edge as illustrated in Figure 5. Move the sliding weight right or left until the beam is balanced. The beam is balanced when the bubble is centered in the sight glass. Obtain the density by reading one of the four calibrated scales on the arrow side of the sliding weight.



Figure 5 — Common pressurized fluid density balance

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

7 Well-simulation compressive strength tests

7.1 General

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

7.2 Sampling

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

7.3 Apparatus

7.3.1 Cube moulds and compressive-strength testing load frame, moulds and testing machine for compressive strength tests should conform with ASTM C 109/C 109M-07, except for: (1) the bearing block surface dimension requirement, (2) the bearing block Rockwell hardness requirement and (3) that moulds can be separable into more than two parts.

The testing load frame should have an upper bearing that is spherically seated and free to tilt in any direction. The center of the sphere should align with the center of the surface of the block in contact with the specimen, to assure even loading across the sample surfaces. A hardened metal bearing block should be used beneath the specimen to minimize wear on the lower platen. The platen should be inscribed to assist in the placement of the specimen in the center of the platen surface. The apparatus is calibrated according to the requirements found in Annex B.

The moulds should be made of hard metal and mould tolerance should be verified. The cube mould base plate should be of corrosion-resistant metal; the cover plate should have a minimum thickness of 6 mm (0,25 in) and made of corrosion-resistant material. Grooves may be incorporated into the surface of the cover plate contacting the surface of the cement. Glass plates may be used but are not recommended for tests above 110 °C (230 °F) because of the risk of silica replacement

7.3.2 Water curing bath or tank, having dimensions permitting the complete immersion of moulds for compressive-strength test samples in water, and capable of maintaining the specified test temperatures within $\pm 2\text{ °C}$ ($\pm 3\text{ °F}$).

The two types of water curing baths are:

- a) **atmospheric-pressure curing bath** (unpressurized), having an agitator or circulating system. Atmospheric-pressure curing baths at or below 66 °C (150 °F) may be used for curing samples for compressive-strength testing when higher pressure is not required.
- b) **pressurized curing chamber**, suitable for curing samples at the appropriate final test temperature and a pressure of at least 21 MPa ($3\,000\text{ psi}$). The vessel should be capable of being heated at the desired rate.

7.3.3 Cooling bath, designed so that the specimen to be cooled from the curing temperature can be completely submerged in water maintained at $27\text{ °C} \pm 3\text{ °C}$ ($80\text{ °F} \pm 5\text{ °F}$).

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

- a) A thermometer, of range -18 °C to 104 °C (0 °F to 220 °F), with minimum scale divisions not to exceed 1 °C (2 °F) may be used in a non-pressurized bath.
- b) A thermocouple, of range -18 °C to 104 °C (0 °F to 220 °F), accurate to $\pm 2\text{ °C}$ ($\pm 3\text{ °F}$) is preferred in a non-pressurized bath.
- c) A thermocouple, of range -18 °C to at least 204 °C (0 °F to at least 400 °F), accurate to $\pm 2\text{ °C}$ ($\pm 3\text{ °F}$), should be used in a pressurized curing chamber.

7.3.5 Puddling rod, corrosion-resistant, typically with a diameter of 6 mm ($0,25\text{ in.}$).

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

7.3.7 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. The mould-release agent should comply with 7.3.6.

7.4 Procedure

Apparati are calibrated according to the requirements in Annex B

7.4.1 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 should be water tight. Care should be taken to ensure there is no bead of sealant on the interior of the mould. See Figure 6.