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Petroleum and natural gas industries — Cements and materials for well cementing —

Part 1:
Specification

Industries du pétrole et du gaz naturel — Ciments et matériaux pour la cimentation des puits —

Partie 1: Spécification

Reference number
ISO 10426-1:2009(E)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 10426-1 was prepared by Technical Committee ISO/TC 67, *Materials, equipment and offshore structures for petroleum, petrochemical and natural gas industries*, Subcommittee SC 3, *Drilling and completion fluids, and well cements*.

This third edition cancels and replaces the second edition (ISO 10426-1:2005), which has been technically revised.

ISO 10426 consists of the following parts, under the general title *Petroleum and natural gas industries — Cements and materials for well cementing*:

- *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*

Introduction

This part of ISO 10426 is based on ISO 10426-1:2005 with the intent that the 24th edition of API Spec 10A will be identical to this part of ISO 10426.

It is necessary that users of this part of ISO 10426 be aware that further or differing requirements can be required 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 can be particularly applicable where there is innovative or developing technology. Where an alternative is offered, it is the responsibility of the vendor to identify any variations from this part of ISO 10426 and provide details.

In this part of ISO 10426, where practical, US Customary (USC) or other units are included in brackets for information. The units do not necessarily represent a direct conversion of SI to USC units, or USC to SI. Consideration has been given to the precision of the instrument making the measurement. For example, thermometers are typically marked in 1° 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 or both. A result is valid if it is both accurate and precise.

Petroleum and natural gas industries — Cements and materials for well cementing —

Part 1: Specification

1 Scope

This part of ISO 10426 specifies requirements and gives recommendations for six classes of well cements, including their chemical and physical requirements and procedures for physical testing.

This part of ISO 10426 is applicable to well cement classes A, B, C and D, which are the products obtained by grinding Portland cement clinker and, if needed, calcium sulfate as an interground additive. Processing additives can be used in the manufacture of cement of these classes. Suitable set-modifying agents can be interground or blended during manufacture of class D cement.

This part of ISO 10426 is also applicable to well cement classes G and H, which are the products obtained by grinding clinker with no additives other than one or more forms of calcium sulfate, water or chemical additives as required for chromium (VI) reduction.

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 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

ISO 13500, *Petroleum and natural gas industries — Drilling fluid materials — Specifications and tests*

ISO 24450, *Laboratory glassware — Wide-necked boiling flasks*

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

ASTM C115, *Standard Test Method for Fineness of Portland Cement by the Turbidimeter*

ASTM C465, *Standard Specification for Processing Additions for Use in the Manufacture of Hydraulic Cements*

ASTM E1404-94(2008), *Standard Specification for Laboratory Glass Conical Flasks*

EN 196-1, *Methods of testing cement — Part 1: Determination of strength*

EN 196-2, *Methods of testing cement — Part 2: Chemical analysis of cement*

EN 196-6, *Methods of testing cement — Part 6: Determination of fineness*

EN 196-7, *Methods of testing cement — Part 7: Methods of taking and preparing samples of cement*

3 Terms and definitions

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

3.1
additive
material added to a cement slurry to modify or enhance some desired property

NOTE Properties that are commonly modified include setting time (by use of retarders or accelerators), fluid loss, viscosity, etc.

3.2
atmospheric pressure consistometer
device used for stirring and conditioning the cement slurry

NOTE The device is not intended to measure thickening time.

3.3
Bearden unit of consistency
B_c
measure of the consistency of a cement slurry when determined on a pressurized consistometer

3.4
cement
Portland cement
ground clinker generally consisting of hydraulic calcium silicates and aluminates and usually containing one or more forms of calcium sulfate as an interground additive

3.5
cement class
designation achieved under the ISO system for classification of well cement according to its intended use

3.6
cement grade
designation achieved under the ISO system for denoting the sulfate resistance of a particular cement

3.7
cement blend
mixture of dry cement and other dry materials

3.8
clinker
fused materials produced in the kiln during cement manufacturing that are interground with calcium sulfate to make cement

3.9
compressive strength
force per unit area required to cause a set cement sample to fail under compression

3.10
consistometer
device used to measure the thickening time of a cement slurry under temperature and under pressure

3.11
filtrate
liquid that is forced out of a cement slurry during a fluid loss test

3.12
free fluid
coloured or colourless liquid that separates from a cement slurry under static conditions

3.13
slurry container
slurry cup
container in an atmospheric or pressurized consistometer used to hold the slurry for conditioning purposes or for the thickening-time test

3.14
thickening time
time after which the consistency of a cement slurry has become so high that the slurry is considered unpumpable

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

4 Requirements

4.1 Specification, chemical and physical requirements

4.1.1 Classes and grades

4.1.1.1 General

Well cement shall be specified using classes A, B, C, D, G and H and the grades: ordinary (O), moderate sulfate-resistant (MSR) and high sulfate-resistant (HSR).

A well cement that has been manufactured and supplied in accordance with this part of ISO 10426 may be mixed and placed in the field using water ratios or additives at the user's discretion. It is not intended that manufacturing compliance with this part of ISO 10426 be based on such field conditions.

Processing additives, set modifying agents or chemical additives used to reduce chromium (VI) shall not prevent a well cement from performing its intended functions.

4.1.1.2 Class A

This product is obtained by grinding clinker, consisting essentially of hydraulic calcium silicates, usually containing one or more forms of calcium sulfate as an interground additive. At the option of the manufacturer, processing additives may be used in the manufacture of class A cement, provided that such materials in the amounts used have been shown to meet the requirements of ASTM C465.

This product is intended for use when special properties are not required and is available only in O grade, similar to ASTM C150, type I.

4.1.1.3 Class B

This product is obtained by grinding clinker, consisting essentially of hydraulic calcium silicates, usually containing one or more forms of calcium sulfate as an interground additive. At the option of the manufacturer, processing additives may be used in the manufacture of class B cement, provided that such materials in the amounts used have been shown to meet the requirements of ASTM C465.

This product is intended for use when conditions require moderate or high sulfate resistance and is available in both MSR and HSR grades, similar to ASTM C150, type II.

4.1.1.4 Class C

This product is obtained by grinding clinker, consisting essentially of hydraulic calcium silicates, usually containing one or more forms of calcium sulfate as an interground additive. At the option of the manufacturer, processing additives may be used in the manufacture of class C cement, provided that such materials in the amounts used have been shown to meet the requirements of ASTM C465.

This product is intended for use when conditions require high, early strength and is available in O, MSR and HSR grades, similar to ASTM C150, type III.

4.1.1.5 Class D

This product is obtained by grinding clinker, consisting essentially of hydraulic calcium silicates, usually containing one or more forms of calcium sulfate as an interground additive. At the option of the manufacturer, processing additives may be used in the manufacture of class D cement, provided that such materials in the amounts used have been shown to meet the requirements of ASTM C465. Further, at the option of the manufacturer, suitable set-modifying agents may be interground or blended during manufacture.

This product is intended for use under conditions of moderately high temperatures and pressures and is available in MSR and HSR grades.

4.1.1.6 Class G

This product is obtained by grinding clinker, consisting essentially of hydraulic calcium silicates, usually containing one or more forms of calcium sulfate as an interground additive. No additives other than calcium sulfate or water, or both, shall be interground or blended with the clinker during manufacture of class G well cement.

This product is intended for use as a basic well cement and is available in MSR and HSR grades.

4.1.1.7 Class H

This product is obtained by grinding clinker, consisting essentially of hydraulic calcium silicates, usually containing one or more forms of calcium sulfate as an interground additive. No additives other than calcium sulfate or water, or both, shall be interground or blended with the clinker during manufacture of class H well cement.

This product is intended for use as a basic well cement and is available in MSR and HSR grades.

4.1.2 Chemical requirements

Well cements shall conform to the respective chemical requirements of classes and grades referenced in Table 1. It is not intended that this manufacturing compliance be applicable to field conditions. This part of ISO 10426 is not applicable to the use of cements that do not conform to the chemical requirements of classes and grades as set forth in Table 1.

Chemical analyses of hydraulic cements shall be carried out as specified in EN 196-2.

NOTE For the purposes of this provision, ASTM C114 is equivalent to EN 196-2.

Table 1 — Chemical requirements

	Cement class					
	A	B	C	D	G	H
Ordinary grade (O)						
Magnesium oxide (MgO), maximum, percent	6,0	NA[a]	6,0	NA	NA	NA
Sulfur trioxide (SO_3), maximum, percent[b]	3,5	NA	4,5	NA	NA	NA
Loss on ignition, maximum, percent	3,0	NA	3,0	NA	NA	NA
Insoluble residue, maximum, percent	0,75	NA	0,75	NA	NA	NA
Tricalcium aluminate (C_3A), maximum, percent[d]	NR[c]	NA	15	NA	NA	NA
Moderate sulfate-resistant grade (MSR)						
Magnesium oxide (MgO), maximum, percent	NA	6,0	6,0	6,0	6,0	6,0
Sulfur trioxide (SO_3), maximum, percent[b]	NA	3,0	3,5	3,0	3,0	3,0
Loss on ignition, maximum, percent	NA	3,0	3,0	3,0	3,0	3,0
Insoluble residue, maximum, percent	NA	0,75	0,75	0,75	0,75	0,75
Tricalcium silicate (C_3S) maximum, percent[d]	NA	NR	NR	NR	58	58
minimum, percent[d]	NA	NR	NR	NR	48	48
Tricalcium aluminate (C_3A), maximum percent[d]	NA	8	8	8	8	8
Total alkali content, expressed as sodium oxide (Na_2O) equivalent, maximum, percent[e]	NA	NR	NR	NR	0,75	0,75
High sulfate-resistant grade (HSR)						
Magnesium oxide (MgO), maximum, percent	NA	6,0	6,0	6,0	6,0	6,0
Sulfur trioxide (SO_3), maximum, percent[b]	NA	3,0	3,5	3,0	3,0	3,0
Loss on ignition, maximum, percent	NA	3,0	3,0	3,0	3,0	3,0
Insoluble residue, maximum, percent	NA	0,75	0,75	0,75	0,75	0,75
Tricalcium silicate (C_3S) maximum, percent[d]	NA	NR	NR	NR	65	65
minimum, percent[d]	NA	NR	NR	NR	48	48
Tricalcium aluminate (C_3A), maximum, percent[d]	NA	3	3	3	3	3
Tetracalcium aluminoferrite (C_4AF) plus twice the tricalcium aluminate (C_3A), maximum, percent[d]	NA	24	24	24	24	24
Total alkali content expressed as sodium oxide (Na_2O) equivalent, maximum, percent[e]	NA	NR	NR	NR	0,75	0,75

[a] NA indicates “not applicable”.

[b] When the tricalcium aluminate content (expressed as C_3A) of the cement is 8 % or less, the maximum SO_3 content shall be 3 %, or 3,5 % for class C cement.

[c] NR indicates “no requirement”.

[d] The expressing of chemical limitations by means of calculated assumed compounds does not necessarily mean that the oxides are actually or entirely present as such compounds. The compounds are calculated according to the ratio of the mass percentages of Al_2O_3 to Fe_2O_3, where w is the percentage mass fraction of the compound indicated in the subscript:

— When $w_{Al_2O_3}/w_{Fe_2O_3}$ is greater than 0,64, the compounds shall be calculated as follows:

$$C_3A = 2{,}65w_{Al_2O_3} - 1{,}69w_{Fe_2O_3}$$

$$C_3S = 4{,}07w_{CaO} - 7{,}60w_{SiO_2} - 6{,}72w_{Al_2O_3} - 1{,}43w_{Fe_2O_3} - 2{,}85w_{SO_3}$$

$$C_4AF = 3{,}04w_{Fe_2O_3}$$

— When $w_{Al_2O_3}/w_{Fe_2O_3}$ is 0,64 or less, the C_3A content is zero.

— The C_3S and C_4AF shall be calculated as follows:

$$C_3S = 4{,}07w_{CaO} - 7{,}60w_{SiO_2} - 4{,}48w_{Al_2O_3} - 2{,}86w_{Fe_2O_3} - 2{,}85w_{SO_3}$$

$$C_4AF = 3{,}04w_{Fe_2O_3}$$

[e] The sodium oxide equivalent, expressed as Na_2O equivalent, shall be calculated by the formula:

Na_2O equivalent is equal to $0{,}658w_{K_2O} + w_{Na_2O}$.

4.1.3 Physical and performance requirements

Well cement shall conform to the respective physical and performance requirements specified in Table 2 and in Clauses 6 through 10.

Table 2 — Summary of physical and performance requirements

Well cement class				**A**	**B**	**C**	**D**	**G**	**H**
Mix water, % mass fraction of cement (Table 5)				46	46	56	38	44	38
Fineness tests (alternative methods) (Clause 6)									
Turbidimeter (specific surface, minimum, m^2/kg)				150	160	220	NR[a]	NR	NR
Air permeability (specific surface, minimum, m^2/kg)				280	280	400	NR	NR	NR
Free-fluid content, maximum, percent (Clause 8)				NR	NR	NR	NR	5,9	5,9
Compressive strength test (8 h curing time)	**Schedule number** Table 6	**Final curing temperature** °C (°F)	**Curing pressure** MPa (psi)	**Minimum compressive strength** MPa (psi)					
(Clause 9)	NA[b]	38 (100)	atm.	1,7 (250)	1,4 (200)	2,1 (300)	NR	2,1 (300)	2,1 (300)
(Clause 9)	NA	60 (140)	atm.	NR	NR	NR	NR	10,3 (1 500)	10,3 (1 500)
(Clause 9)	6S	110 (230)	20,7 (3 000)	NR	NR	NR	3,4 (500)	NR	NR
Compressive strength test (24 h curing time)	**Schedule number** (Table 6)	**Final curing temperature** °C (°F)	**Curing pressure** MPa (psi)	**Minimum compressive strength** MPa (psi)					
(Clause 9)	NA	38 (100)	atm.	12,4 (1 800)	10,3 (1 500)	13,8 (2 000)	NR	NR	NR
(Clause 9)	4S	77 (170)	20,7 (3 000)	NR	NR	NR	6,9 (1 000)	NR	NR
(Clause 9)	6S	110 (230)	20,7 (3 000)	NR	NR	NR	13,8 (2 000)	NR	NR
Thickening-time test	**Specification test schedule number** Tables 9 through 11	**Maximum consistency** (15 min to 30 min stirring period) B_c[c]		**Thickening time** (minimum/maximum) min					
(Clause 10)	4	30		90[d]	90[d]	90[d]	90[d]	NR	NR
(Clause 10)	5	30		NR	NR	NR	NR	90[d]	90[d]
(Clause 10)	5	30		NR	NR	NR	NR	120[e]	120[e]
(Clause 10)	6	30		NR	NR	NR	100[d]	NR	NR

[a] NR indicates “no requirement”.

[b] NA indicates “not applicable”.

[c] Bearden units of consistency, B_c, obtained on a pressurized consistometer as defined in Clause 10 and calibrated in accordance with the same clause.

[d] Minimum thickening time.

[e] Maximum thickening time.

4.2 Sampling frequency, timing of tests, and equipment

4.2.1 Sampling frequency

4.2.1.1 For well cement classes C, D, G, and H, a sample for testing shall be taken by either of the following methods:

a) over an interval of 24 h;

b) on a 1 000 ton (maximum) production run.

4.2.1.2 For well cement classes A and B, a sample for testing shall be taken by either of the following methods:

a) over a 14-day interval;

b) on a 25 000 ton (maximum) production run.

4.2.1.3 These samples shall represent the product as produced. At the choice of the manufacturer, either sampling method may be used.

4.2.2 Time from sampling to testing

Each sample shall be tested for conformance to this part of ISO 10426. All tests shall be completed within seven working days after sampling.

4.2.3 Specified equipment

Equipment used for testing well cements shall comply with Table 3. Dimensions shown in Figures 5 through 7 and Figures 10 through 12 are for the purposes of manufacturing the cement-specification test equipment. Dimensional recertification is not required.

4.2.4 Calibration

Equipment calibrated in accordance with the requirements of this part of ISO 10426 is considered accurate if the calibration is within the specified limits.

Table 3 — Specification test equipment for well-cement manufacturers

Test or preparation	Well cement classes	Clause reference	Required equipment
Sampling	All	5	Apparatus as specified in EN 196-7 NOTE For the purposes of this provision, ASTM C183 is equivalent to EN 196-7.
Fineness	A, B, C	6	Turbidimeter and auxiliary equipment as specified in ASTM C115 or air permeability apparatus and auxiliary equipment as specified in EN 196-6. NOTE For the purposes of this provision, ASTM C204 is equivalent to EN 196-6.
Slurry preparation	All	7	Apparatus as specified in 7.1
Free fluid	G, H	8	Apparatus as specified in 8.1
Atmospheric pressure compressive strength	A, B, C, G, H	9	Apparatus as specified in 9.1, except pressure vessel of 9.1.3.2
Pressure cured compressive strength	D	9	Apparatus as specified in 9.1,except curing bath of 9.1.3.3
Thickening time	All	10	Pressurized consistometer specified in 10.1

5 Sampling procedure

One or more of the procedures in accordance with EN 196-7 shall be used to secure a sample of well cement for specification testing purposes.

NOTE For the purposes of this provision, ASTM C183 is equivalent to EN 196-7.

6 Fineness tests

6.1 Procedure

Tests for fineness of well cement shall be carried out in accordance with either the procedure in ASTM C115 for the turbidimeter test or the procedure in EN 196-6 by air permeability apparatus for the air permeability test.

NOTE For the purposes of this provision, ASTM C204 is equivalent to EN 196-6.

6.2 Requirements

Acceptance requirements for the fineness test are a minimum specific surface area (expressed in square metres per kilogram) as given in Table 2. Classes D, G and H cements have no fineness requirement.

At the discretion of the manufacturer, either of the two fineness test methods (turbidimeter or air permeability test) shall be used to determine the fineness.

7 Preparation of slurry for free-fluid, compressive strength and thickening-time tests

7.1 Apparatus

7.1.1 Scales

The indicated load on scales shall be accurate within 0,1 % of the indicated load. Annual calibration is required.

7.1.2 Mass of reference weights

The mass of the reference weights shall be accurate to within the tolerance shown in Table 4. On beam-type scales where the reference weights are on the beam, the indicated masses shall conform to the requirements given in 7.1.1.

Table 4 — Permissible variation in mass of reference weights

Mass g	Permissible variation g
1 000	± 1,00
500	± 0,50
300	± 0,30
200	± 0,20
100	± 0,10
50	± 0,05

7.1.3 Sieves

A No. 20 wire cloth sieve (openings 850 µm), in accordance with the requirements given in ISO 3310-1, shall be used for sieving cement prior to slurry preparation.

NOTE For the purposes of this provision, ASTM E11 is equivalent to ISO 3310-1.

7.1.4 Mixing devices

The mixing device for the preparation of well cement slurries shall be a 1 l (1 qt) size, bottom-drive, blade-type mixer.

An example of a mixing device in common use is shown in Figure 1. The mixing-blade assembly and mixing container shall be constructed of durable, corrosion-resistant material. The mixing-blade assembly (see Figure 2) shall be constructed in such a manner that the blade can be removed for weighing and changing. The mixing blade shall be weighed initially and thereafter periodically, and replaced with an unused blade before a 10 % mass loss has occurred. Replace the blade if obvious blade deformation has occurred. If the mixing device leaks at any time during the mixing procedure, the contents shall be discarded, the leak shall be repaired and the procedure shall be restarted.

The mixing device shall be calibrated annually to a tolerance of ± 200 r/min (± 3,3 r/s) at 4 000 r/min (66,7 r/s) rotational speed, and ± 500 r/min (± 8,3 r/s) at 12 000 r/min (200 r/s) rotational speed.

Figure 1 — Example of a typical cement-mixing device

Figure 2 — Mixing-blade assembly

7.2 Procedure

7.2.1 Sieving

Prior to mixing, the cement shall be sieved using the method described in EN 196-7 using a sieve defined in 7.1.3.

NOTE For the purposes of this provision, ASTM C183 is equivalent to EN 196-7.

7.2.2 Temperature of water and cement

The temperature of the mix water in the container within 60 s prior to mixing shall be 23 °C ± 1 °C (73 °F ± 2 °F) and the temperature of the cement within 60 s prior to mixing shall be 23 °C ± 1 °C (73 °F ± 2 °F).

7.2.3 Mix water

Distilled or de-ionized water shall be used for testing. The mix water shall be weighed directly into a clean, dry mixing container. No water shall be added to compensate for evaporation, wetting, etc.

7.2.4 Mixing quantities

The quantities of slurry component shown in Table 5 shall be used for testing. The use of the quantities of components shown in Table 5 results in mix-water percentages (based on the mass of dry cement) consistent with water percentages shown in Table 2.

Table 5 — Slurry requirements

Components	Classes A and B g	Class C g	Classes D and H g	Class G g
Mix water	355 ± 0,5	383 ± 0,5	327 ± 0,5	349 ± 0,5
Cement	772 ± 0,5	684 ± 0,5	860 ± 0,5	792 ± 0,5

7.2.5 Mixing cement and water

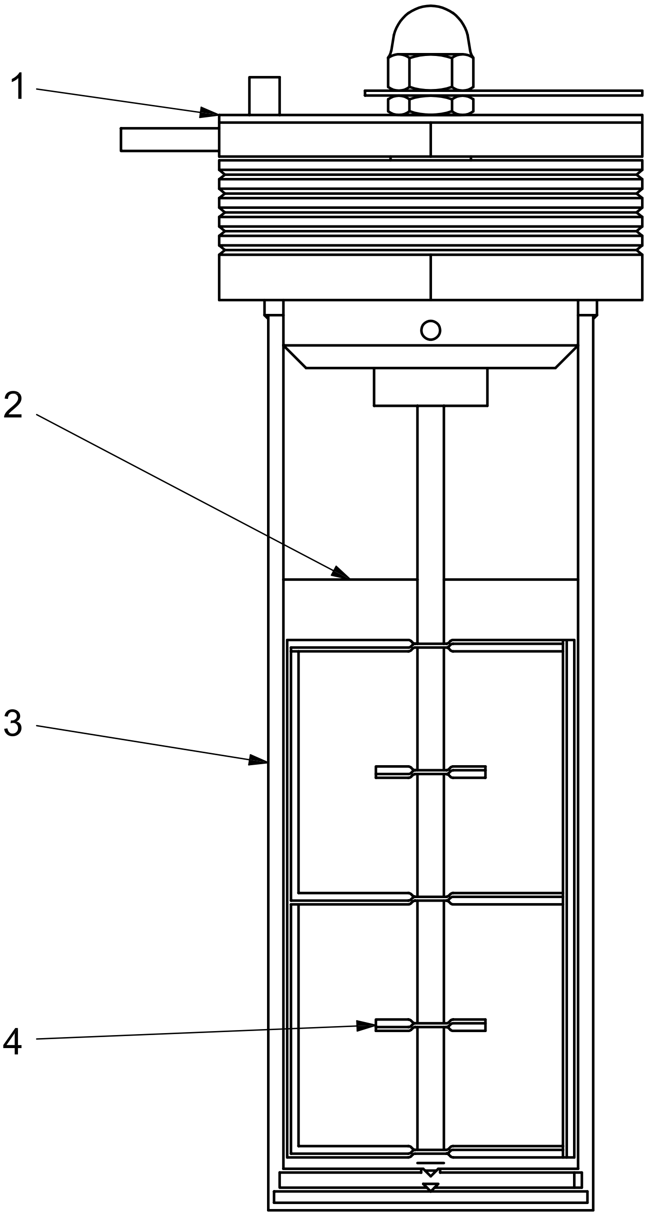
The mixing container with the required mass of mix water, as specified in Table 5, shall be placed on the mixer base, the motor turned on and maintained at 4 000 r/min ± 200 r/min (66,7 r/s ± 3,3 r/s) while the cement sample is added at a uniform rate during no more than 15 s. After 15 s at 4 000 r/min ± 200 r/min (66,7 r/s ± 3,3 r/s), 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 s ±1 s.

8 Free-fluid test (formerly free water)

8.1 Apparatus

8.1.1 Consistometer

The atmospheric pressure consistometer or the pressurized consistometer described in 10.1 (run at atmospheric pressure) shall be used for stirring and conditioning the cement slurry for the determination of free-fluid content. The atmospheric consistometer consists of a rotating cylindrical slurry container, equipped with an essentially stationary paddle assembly, in a temperature-controlled liquid bath. It shall be capable of maintaining the temperature of the bath at 27 °C ± 2 °C (80 °F ± 3 °F) and of rotating the slurry container at a speed of 150 r/min ±15 r/min (2,5 r/s ± 0,25 r/s) during the stirring and conditioning period for the slurry. The paddle and all parts of the slurry container exposed to the slurry shall be constructed of corrosion-resistant materials. See Figures 3 through 6.



Key

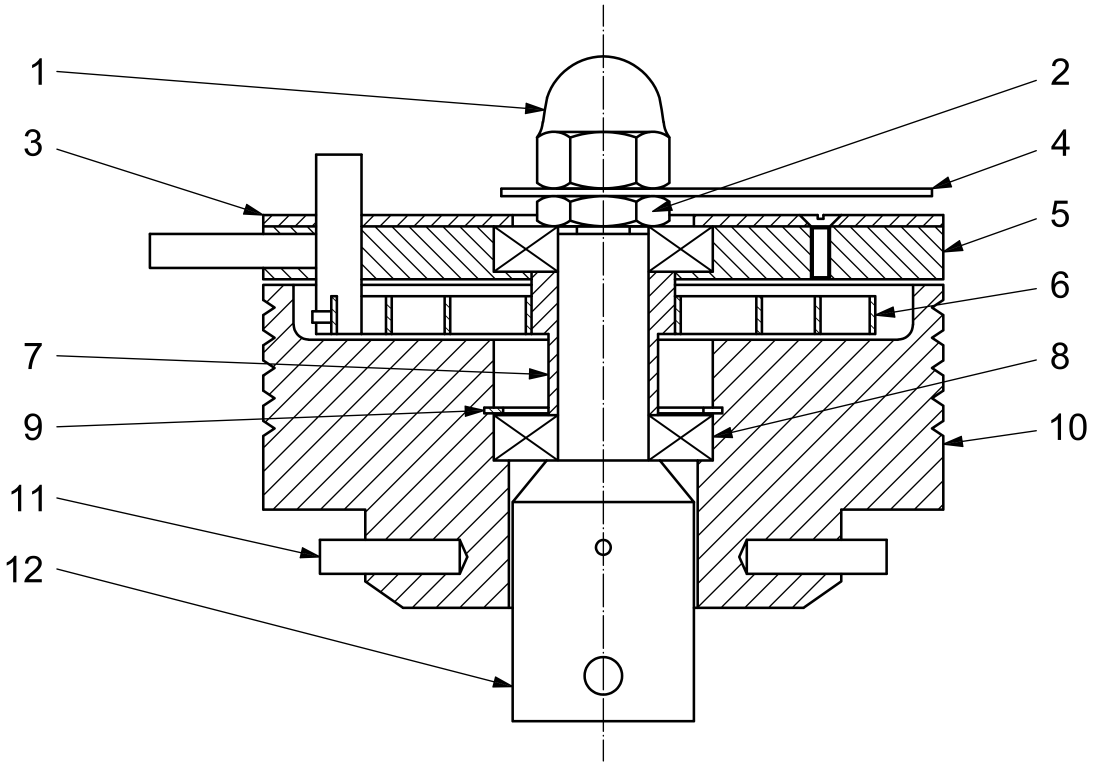
1 lid (see Figure 4)

2 fill indicating groove

3 slurry container (see Figure 5)

4 paddle (see Figure 6)

Figure 3 — Typical container assembly for an atmospheric pressure consistometer

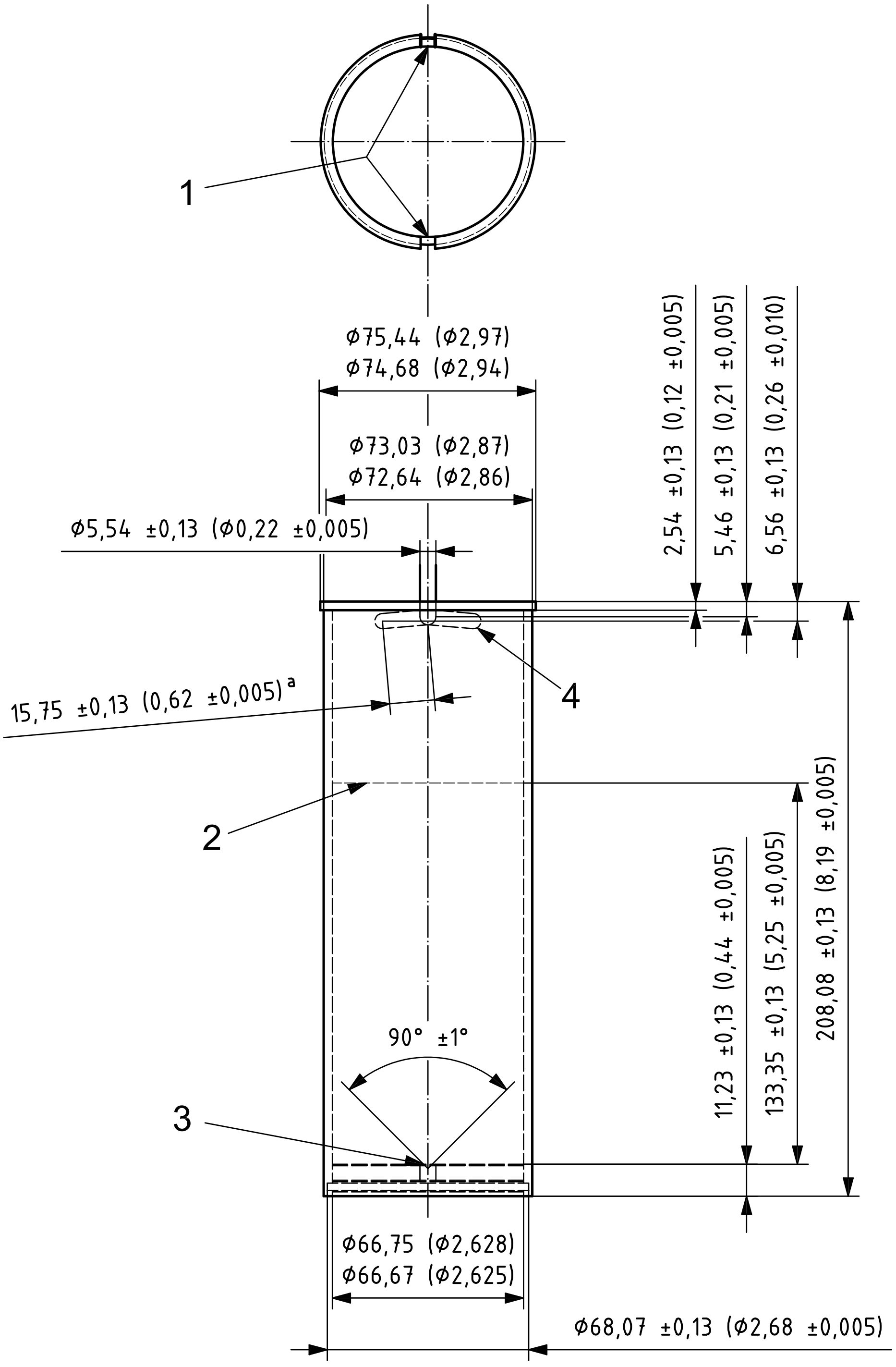


Key

1	cap lock nut
2	centre lock reverse jam nut
3	dial
4	pointer
5	dial and base assembly
6	spring
7	collar
8	bearing
9	retaining ring
10	lid
11	roll pin
12	shaft

Figure 4 — Typical lid and mechanism for an atmospheric pressure consistometer

Dimensions in millimetres (inches) unless otherwise indicated



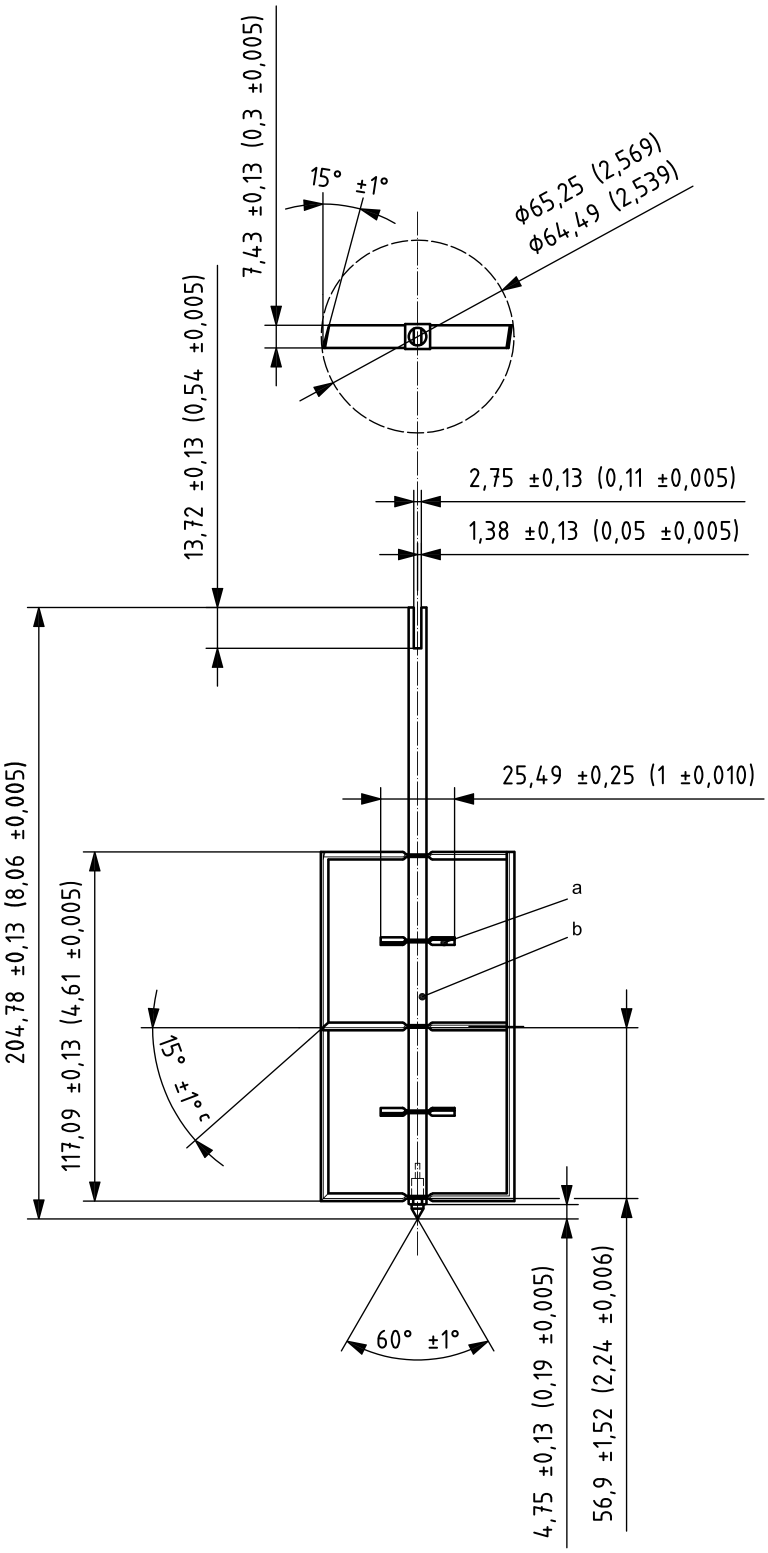
Key

1 two slots 180° apart
2 fill-level indicating groove
3 pivot bearing
4 opposite side

[a] Typical.

Figure 5 — Typical container for an atmospheric pressure consistometer

Dimensions in millimetres (inches) unless otherwise indicated



[a] Paddle material is 300 series stainless steel 1,0 mm × 7,9 mm (0,04 in × 0,313 in).

[b] Shaft material is 400 series steel 6,4 mm × 211,1 mm (0,25 in × 8,313 in) annealed and ground.

[c] Typical.

Figure 6 — Typical paddle for an atmospheric pressure consistometer

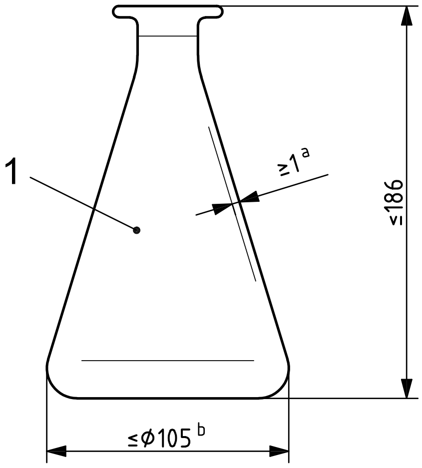
8.1.2 Scales

Scales shall meet the requirements set in 7.1.1.

8.1.3 Test flask

A 500 ml conical flask, in accordance with ASTM E1404-94(2008), type I, class 2, or with ISO 24450 shall be used. See Figure 7.

Dimensions in millimetres



[a] Wall thickness.

[b] Outside diameter (at widest point).

NOTE The conical flask for free-fluid measurement is an ASTM conical flask, type 1, class 2, with a capacity of 500 ml.

Figure 7 — The ASTM conical flask for free-fluid measurement

8.2 Calibration

8.2.1 Temperature measuring system

Temperature-measuring and -controlling devices shall be calibrated no less frequently than quarterly. This includes thermometers, thermocouples and temperature controllers on consistometers, curing chambers and ultrasonic devices, and those that are used separate from or are not an integral part of the instrument. Measurements shall be made at no fewer than three temperatures spanning the manufacturer- or user-defined operating range of the equipment on or with which the device is used. The lowest temperature calibrated shall be no more than 5 °C (10 °F) above the minimum and the highest temperature calibrated shall be no more than 5 °C (10 °F) below the maximum of the user-defined operating range of the instrument. Thermometers or thermocouples shall be calibrated by the use of a known temperature source (such as a heat block) with a thermometer or thermocouple that is certified and traceable to a national standardization body. Accuracy shall be within 2 °C (3 °F). If the error in the thermometer or thermocouple is greater than that, it shall be replaced by one meeting the accuracy requirements. Thermocouples mounted in the cylinder wall of a consistometer, if not used for controlling the temperature in the consistometer, are exempt from calibration. More detailed information can be found in Annex A.

8.2.2 Slurry container rotational speed

The rotational speed shall be 150 r/min ± 15 r/min (2,5 r/s ± 0,25 r/s). The rotational speed of the slurry container shall be checked no less frequently than quarterly, and corrected if found to be outside this range.

8.2.3 Timer

The timer shall be accurate to within ± 30 s per hour. It shall be checked for accuracy no less frequently than annually, and corrected or replaced if found to be less accurate.

8.3 Procedure

8.3.1 Prepare the slurry in accordance with the procedure in Clause 7.

8.3.2 Fill a clean and dry consistometer slurry container to the fill groove.

8.3.3 Assemble the slurry container and associated parts, place them in the consistometer and start the motor according to the operating instructions of the manufacturer. The interval between completion of mixing and starting of the consistometer shall not exceed 1 min.

8.3.4 Stir the slurry in the consistometer for a period of 20 min ± 30 s. Maintain the temperature of the slurry at 27 °C ± 2 °C (80°F ± 3 °F) and atmospheric pressure throughout the stirring period.

8.3.5 Transfer 790 g ± 5 g of class H slurry or 760 g ± 5 g of class G slurry directly into the clean, dry 500 ml conical flask within 1 min of the end of stirring. Record the actual mass transferred. Seal the flask to prevent evaporation.

8.3.6 Set the slurry-filled flask on a surface that is nominally level and vibration-free. The laboratory air temperature to which the slurry-filled flask is exposed shall be 23 °C ± 3 °C (73 °F ± 6 °F). The temperature sensor for measuring air temperature shall meet the requirements of 8.2.1. Let the slurry-filled flask remain undisturbed for a period of 2 h ± 5 min.

8.3.7 At the end of the 2 h ± 5 min period, remove the supernatant fluid that has developed with a pipette or syringe. Measure the volume of supernatant fluid to an accuracy of ± 0,1 ml and record it as “millilitres free fluid.”

8.3.8 Convert the millilitres free fluid to a percentage of starting slurry volume (~400 ml depending on the recorded initial mass) and express that value as percent free fluid.

8.4 Calculation of percent free fluid

The volume fraction, φ, of free fluid in the slurry, expressed as a percentage, is then calculated using Equation (1):

$$\varphi = \frac{V_{\mathrm{FF}} \cdot \rho}{m_{\mathrm{S}}} \times 100 \qquad (1)$$

where

V_{FF} is the volume of free fluid (supernatant fluid) collected, expressed in millilitres;

ρ is the specific gravity of slurry, equal to 1,98 for class H at 38 % water and 1,91 for class G at 44 % water; if the specific gravity of the base cement is other than the typical value of 3,18 ± 0,04, the actual specific gravity of slurry should be calculated and used;

m_{S} is the initially recorded (starting) mass of the slurry, expressed in grams.

EXAMPLE Calculation of percent free fluid:

$m_{\mathrm{S}} = 791{,}7$ g

$V_{\mathrm{FF}} = 15{,}1$ ml

$\rho = 1{,}98$ g/cm^3 (class H)

$\varphi = 15{,}1 \times (1{,}98) \times 100/791{,}7$

$\varphi = 3{,}78$

NOTE Millilitres and cubic centimetres are assumed to be equal for purposes of calculation.

8.5 Acceptance requirements

The free fluid for classes G and H well cements shall not exceed 5,9 %.

9 Compressive strength tests

9.1 Apparatus

9.1.1 Cube moulds and compressive strength-testing machine

Moulds and testing machine for compressive strength tests shall conform to the requirements in ASTM C109/C109M or EN 196-1, except for

a) the bearing block surface dimension requirement;

b) the bearing block Rockwell hardness requirement;

c) the moulds, which may be separable into more than two parts.

The moulds shall be checked for tolerances at least once every two years. The load frame used to measure the break force of cement specimens shall be calibrated no less frequently than annually. Indicated force shall deviate by no more than 2 % of the applied load or one minimum instrument scale division, whichever is greater, at 9,0 kN (2 000 lbf) load and at a minimum of 25 %, 50 % and 75 % of the range of the load cell or load indicator. With units having multiple indicators for different ranges, each indicator shall be calibrated according to these criteria.

9.1.2 Cube mould base and cover plates

Generally, plate glass, brass or stainless steel plates having a minimum thickness of 6 mm (1/4 in) are used. Cover plates may be grooved on the surface that contacts the top of the cement.

9.1.3 Water curing bath

9.1.3.1 General

A curing bath or tank having dimensions that allow the complete immersion of a compressive strength mould(s) in water and capable of maintaining the prescribed test temperatures within ± 2 °C (± 3 °F) shall be employed. The two types of water curing baths are described in 9.1.3.2 and 9.1.3.3.

9.1.3.2 Atmospheric pressure curing bath

An atmospheric pressure curing bath is a vessel for curing specimens at atmospheric pressure and temperatures of 66 °C (150 °F) or less, having an agitator or circulating system.

9.1.3.3 Pressurized curing bath

A pressurized curing bath is a vessel suitable for curing specimens at temperatures up to 110 °C (230 °F) and under pressure that can be controlled at 20,7 MPa ± 3,45 MPa (3 000 psi ± 500 psi). The vessel shall be capable of fulfilling the appropriate specification schedule given in Table 6.

9.1.4 Cooling bath

The cooling bath dimensions shall be such that the specimens being cooled from the curing temperature can be completely submerged in water maintained at 27 °C ± 3 °C (80 °F ± 6 °F).

9.1.5 Temperature-measuring system

9.1.5.1 General

The temperature-measuring system shall be calibrated to an accuracy of ± 2 °C (± 3 °F) no less frequently than tri-monthly. The procedure described in Annex A is commonly used. Two commonly used temperature-measuring systems are described in 9.1.5.2 and 9.1.5.3.

9.1.5.2 Thermometer

A thermometer with a range from 21 °C to 82 °C (70 °F to 180 °F), with minimum scale divisions not exceeding 1 °C (2 °F) may be used.

9.1.5.3 Thermocouple

A thermocouple system with the appropriate range may be used.

9.1.6 Puddling rod

A corrosion-resistant puddling rod of nominal diameter 6 mm (1/4 in) is typically used.

9.1.7 Sealant

A sealant having a consistency that permits ease of use, good sealing properties to prevent leakage, water resistance, inertness to the cement, and is non-corrosive when subjected to the curing temperatures and pressures specified in Table 7, is commonly used to seal the exterior contact points of the specimen mould.

9.2 Procedure

9.2.1 Preparation of moulds

The assembled moulds shall be watertight. The interior faces of the moulds and the contact surfaces of the plates shall be clean and dry, and they may be lightly coated with release agent.

9.2.2 Preparation and placement of slurry

9.2.2.1 Slurry

Prepare the cement slurry in accordance with Clause 7.

9.2.2.2 Placing slurry in moulds

Place the slurry in the prepared moulds in a layer approximately one-half of the mould depth. Use the puddling rod to puddle the slurry in an evenly distributed pattern, 27 times per specimen. Place the slurry in all the specimen compartments before commencing the puddling operation. After puddling the layer, stir the remaining slurry by hand, using a puddling rod or spatula to minimize segregation. Fill the moulds to overflowing and puddle the same as for the first layer. After puddling, use a straight-edge to strike off the excess slurry level with the top of the mould. Discard specimens in moulds that leak. Place a clean, dry cover plate on top of the mould. No less than three specimens shall be used for each test determination.

9.2.2.3 Pressure and temperature schedules

For classes A, B, C, G and H cements, place the specimens in the water bath preheated to the final curing temperature for tests at atmospheric pressure, within 5 min after mixing (see Table 2).

For class D cement, place the specimens in the pressure vessel in water at 27 °C ± 3 °C (80 °F ± 6 °F), and within 5 min after mixing, apply temperature and pressure according to Table 6.

Table 6 — Specification schedules for pressurized curing of specimens

Schedule number	**Curing pressure**[a] MPa (psi)	**Elapsed time from first application of heat and pressure** h: min (± 2 min)										
		0:00	0:30	0:45	1:00	1:15	1:30	2:00	2:30	3:00	3:30	4:00
		Temperature °C (°F)										
4S	20,7 (3 000)	27 (80)	47 (116)	49 (120)	51 (124)	53 (128)	55 (131)	59 (139)	64 (147)	68 (155)	72 (162)	77 (170)
6S	20,7 (3 000)	27 (80)	56 (133)	64 (148)	68 (154)	72 (161)	75 (167)	82 (180)	89 (192)	96 (205)	103 (218)	110 (230)

[a] The test pressure of 20,7 MPa ± 3,4 MPa (3 000 psi ± 500 psi) shall be applied as soon as the specimens are placed in the pressure vessel and maintained for the duration of the curing period. The 4 h temperature shall be maintained within ± 2 °C (± 3 °F) throughout the remainder of the curing period and completion of the test.

9.2.3 Curing

9.2.3.1 Curing periods

The curing period is the time elapsed from subjecting the specimens to the specified temperature in the curing vessel (or initiating the temperature/pressure schedule in Table 6) to testing the specimen for strength. The strength of the specimen shall be tested at the appropriate time, as specified in Table 7.

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

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

9.2.3.2 Specimen cooling

Specimens cured at 60 °C (140 °F) and below shall be removed from the curing bath 45 min ± 5 min before the time at which they will be tested. Specimens shall be removed from their moulds and cooled by being placed in a water bath maintained at 27 °C ± 3 °C (80 °F ± 5 °F) for 40 min. Do not leave the specimen out of water for more than 5 min to avoid dehydration. For specimens cured at temperatures equal to or greater than 77 °C (170 °F), maintain the maximum scheduled temperature and pressure specified in Table 7 until 1 h and 45 min ± 5 min prior to the time at which the specimens will be tested, at which point discontinue heating. During the next 60 min ± 5 min, decrease the temperature to 77 °C (170 °F), or less, without a reduction in the pressure other than that caused by the reduction in temperature. At 45 min ± 5 min prior to the time at which the specimens will be tested, release the pressure remaining and remove the specimens from the moulds. Cool the specimens by transferring them to a water bath maintained at 27 °C ± 3 °C (80 °F ± 6 °F) for 40 min ± 5 min.

9.2.3.3 Specimen acceptance

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

9.3 Test procedure (after ASTM C109/C109M)

9.3.1 Remove specimens from the water bath or the cooling bath that has been maintained at 27 °C ± 3°C (80 °F ± 5 °F). Wipe each specimen to remove any loose material from the faces that will be in contact with the bearing blocks of the testing machine.

9.3.2 The dimensions of the test faces shall be measured to ± 1,0 mm (± 1/16 in) for calculation of the cross-sectional area. Apply the load to the specimen faces that were in contact with the plane (vertical) surfaces of the mould (not in contact with the base or the cover plates). Place the specimen in the testing machine below the upper bearing block. Prior to the testing of each cube, ascertain that the spherically seated block is free to tilt. Use no cushioning or bedding materials.

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

9.3.3 The rate of loading shall be 72 kN/min ± 7 kN/min (16 000 lbf/min ± 1 600 lbf/min) for specimens expected to have a strength greater than 3,4 MPa (500 psi). For specimens expected to have strength less than 3,4 MPa (500 psi), a 18 kN/min ± 2 kN/min (4 000 lbf/min ± 400 lbf/min) rate shall be used. Depending on the type of compressive strength test machine employed, it can require some time for the load frame to build up the required load rate after initial contact with the cement sample.

9.3.4 Calculate the compressive strength expressed in megapascals (pounds force per square inch).

9.4 Compressive strength acceptance criteria

The compressive strength of all acceptance-test specimens made from the same sample and tested at the same period shall be recorded and averaged to the nearest 50 kPa (10 psi). At least two-thirds of the original individual specimens and the average of all the specimens tested shall meet or exceed the minimum compressive strength specified in Table 7. A retest shall be made if fewer than two strength values are left for determining the compressive strength at any given period.

Table 7 — Compressive strength specification requirements

Cement class	Schedule number	Final curing temperature[a] °C (°F)	Curing pressure[b] MPa (psi)	Minimum compressive strength at indicated curing period	
				8 h ± 15 min MPa (psi)	24 h ± 15 min MPa (psi)
A	—	38 (100)	atm.	1,7 (250)	12,4 (1 800)
B	—	38 (100)	atm.	1,4 (200)	10,3 (1 500)
C	—	38 (100)	atm.	2,1 (300)	13,8 (2 000)
D	4S	77 (170)	20,7 (3 000)	NR[c]	6,9 (1 000)
	6S	110 (230)	20,7 (3 000)	3,4 (500)	13,8 (2 000)
G, H	—	38 (100)	atm.	2,1 (300)	NR
	—	60 (140)	atm.	10,3 (1 500)	NR

[a] The curing temperature shall be maintained at the indicated temperature ± 2 °C (± 3 °F).

[b] The test pressure shall be applied as soon as specimens are placed in the pressure vessel, and maintained at the given pressure within ± 3,4 MPa (± 500 psi) for schedules 4S and 6S.

[c] NR indicates “no requirement”.

10 Thickening-time tests

10.1 Apparatus

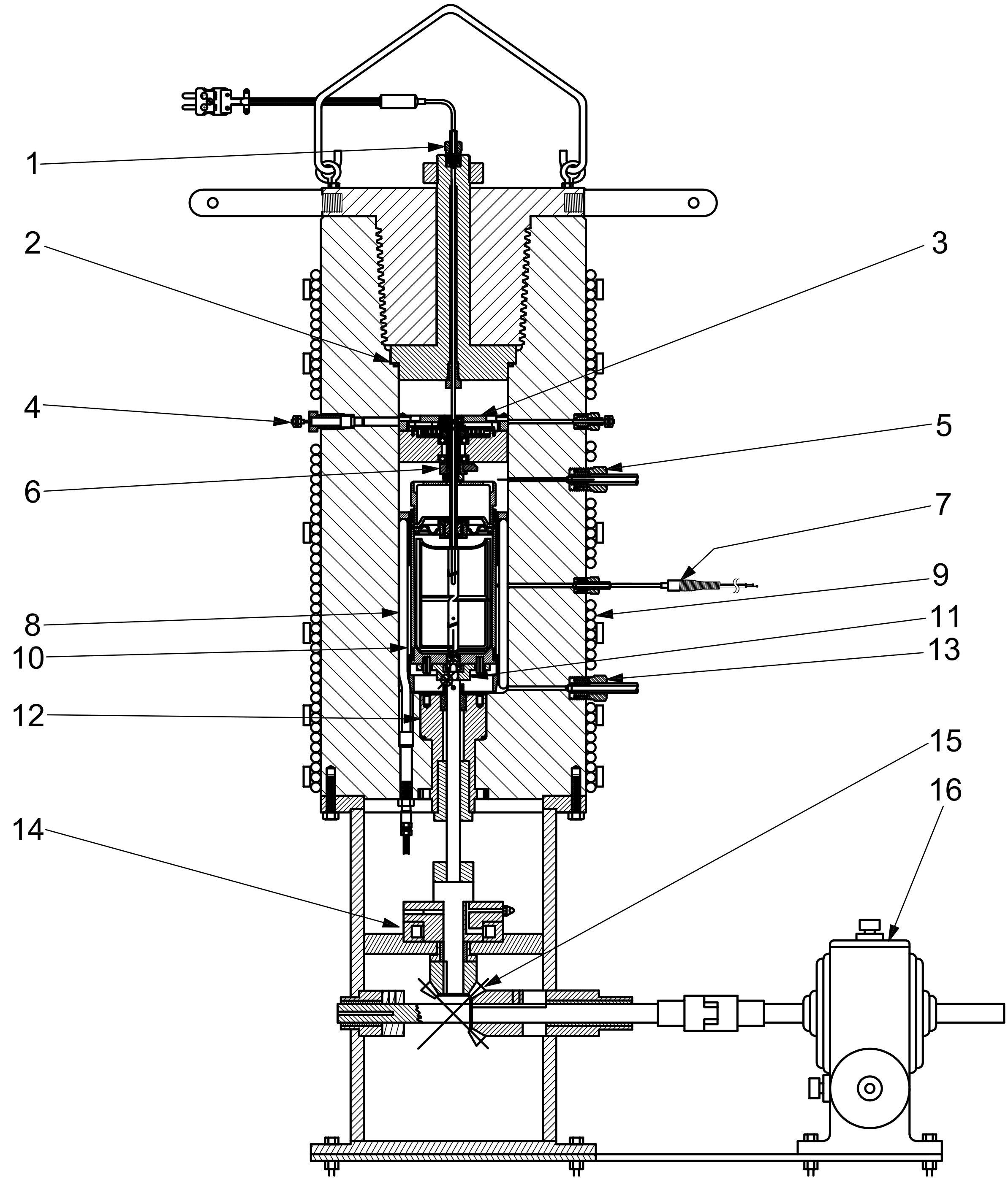
A pressurized consistometer (see Figures 8 and 9) shall consist of a rotating cylindrical slurry container (see Figures 10 and 11) equipped with a stationary paddle assembly (see Figure 12) enclosed in a pressure vessel capable of withstanding the pressures and temperatures described in Tables 9 through 11.

The space between the slurry container and the walls of the pressure vessel shall be completely filled with a hydrocarbon oil. The selected oil shall have the following physical properties:

- viscosity range: 6 mm^2/s to 79 mm^2/s at 38 °C (100 °F) or 6 cSt to 79 cSt at 38 °C (100 °F); or

  45 SSU to 360 SSU at 38 °C(100 °F);
- specific heat: 1,9 kJ/(kg·K) to 2,5 kJ/(kg·K) (0,45 Btu/lb·°F to 0,60 Btu/lb·°F);
- thermal conductivity: 0,112 W/(m·K) to 0,138 W/(m·K) [0,065 Btu/(h·ft^2·°F/ft) to 0,08 Btu/(h·ft^2·°F/ft)];
- specific gravity: 0,83 to 0,93.

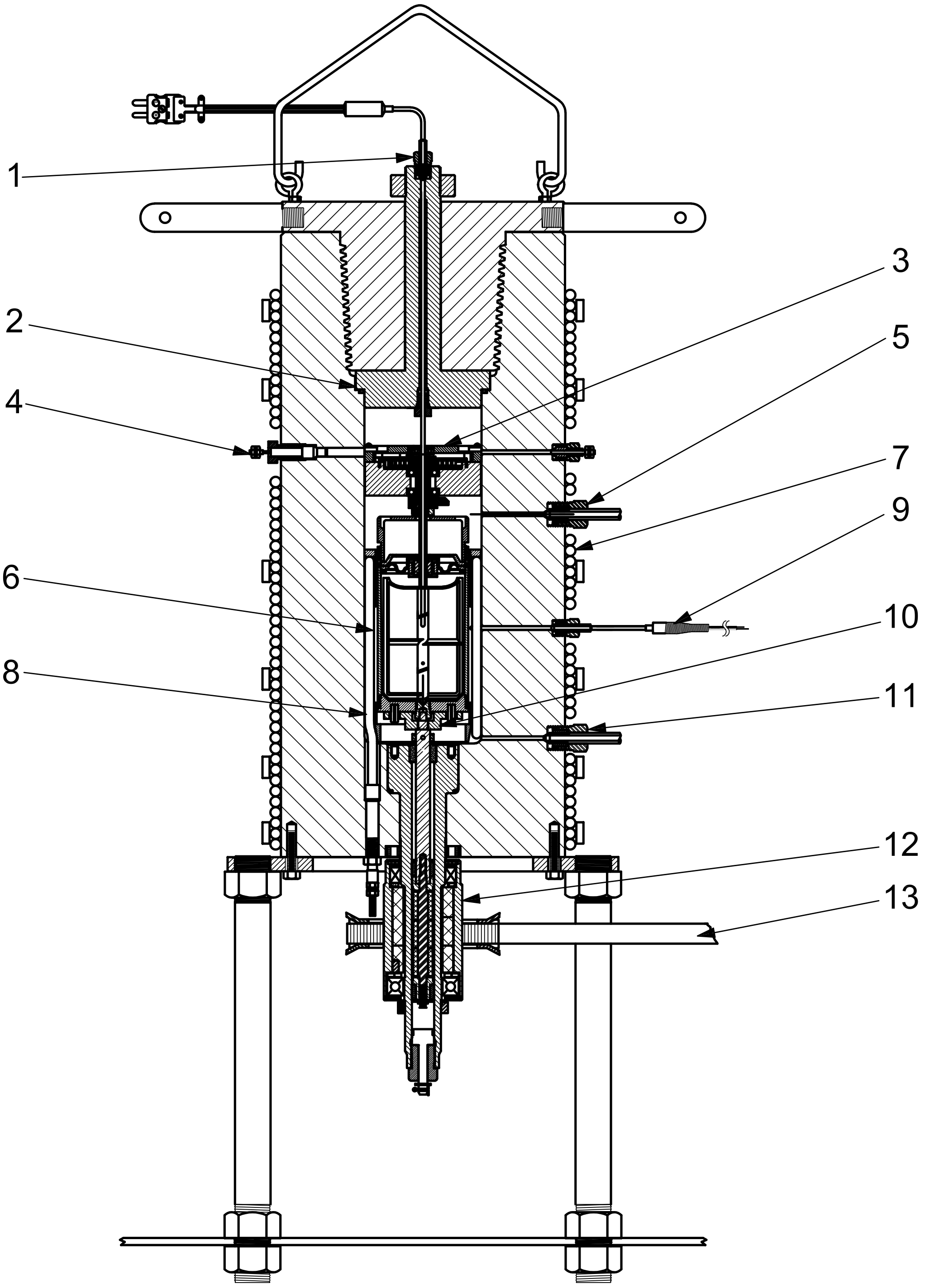
A heating system capable of raising the temperature of this oil bath at the rate of at least 3 °C/min (5 °F/min) is required. A temperature-measuring system shall be provided for determining and controlling the temperature of the cement slurry (centreline). The slurry container is rotated at a speed of 150 r/min ± 15 r/min. The consistency of the slurry (see 10.2.2.1) shall be measured. The paddle and all parts of the slurry container exposed to the slurry shall be constructed according to the dimensions given in Figures 10 through 12.



Key

1 slurry-container thermocouple
2 sealing ring
3 potentiometer mechanism
4 contact pin
5 pressure port
6 drive bar
7 wall thermocouple
8 heater
9 cooling coil
10 slurry container
11 container drive table (rotates anticlockwise)
12 removable packing cartridge
13 pressure port
14 thrust bearing
15 mitre gears
16 gear reducer

Figure 8 — Typical gear drive consistometer for pressurized specification thickening-time test

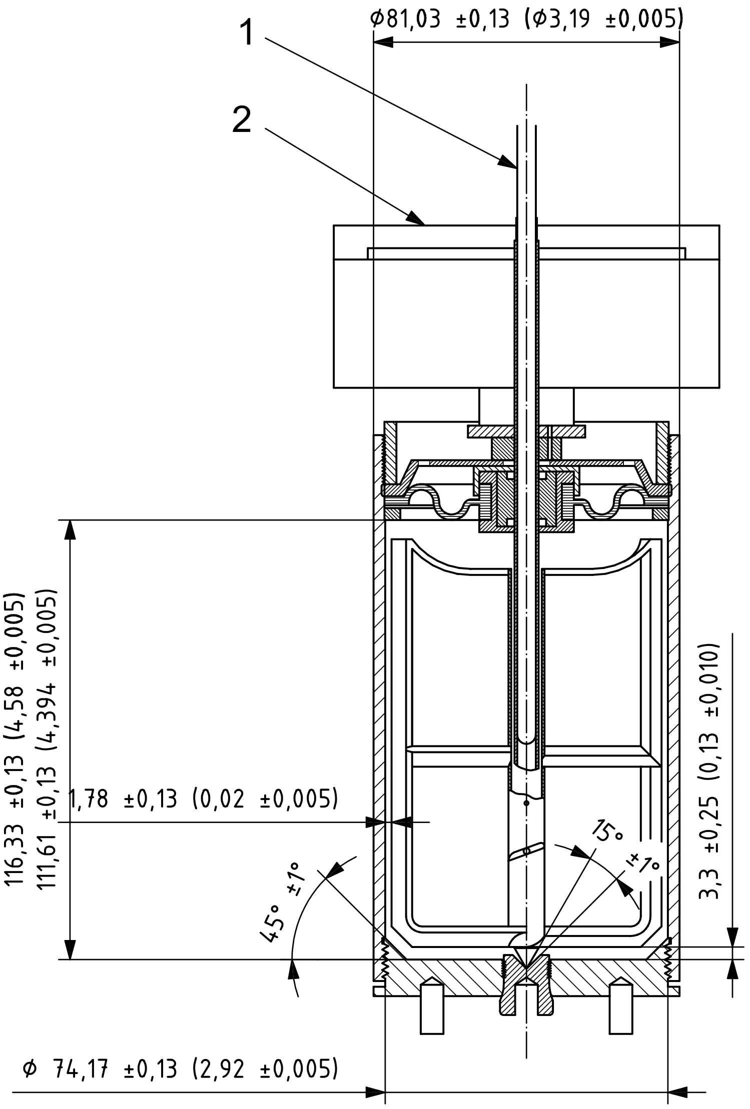


Key

1	slurry-container thermocouple	6	slurry container	11	pressure port
2	sealing ring	7	cooling coil	12	magnetic drive
3	potentiometer mechanism	8	heater	13	drive belt
4	contact pin	9	wall thermocouple		
5	pressure port	10	container drive table (rotates anticlockwise)		

Figure 9 — Typical magnetic drive consistometer for pressurized specification thickening-time tests

Dimensions in millimetres (inches) unless otherwise indicated



Key

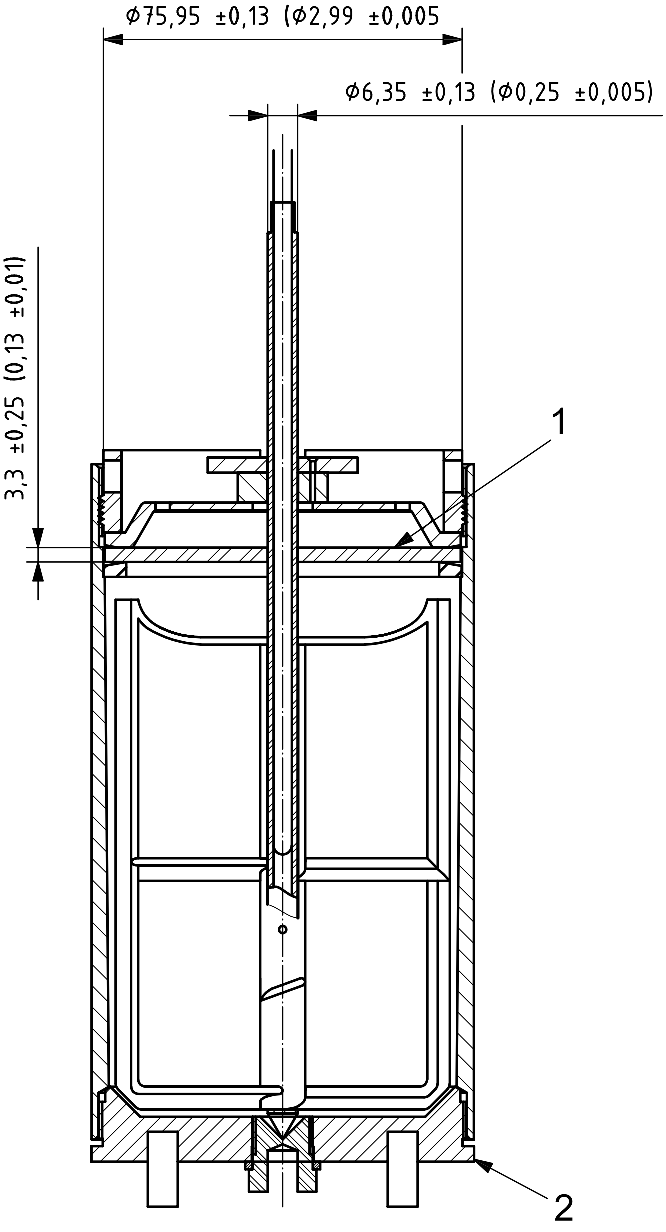
1 temperature measuring device

2 potentiometer mechanism

NOTE The material is stainless steel, except the diaphragm and the hub.

Figure 10 — Typical slurry container assembly for a pressurized consistometer

Dimensions in millimetres (inches)



Key

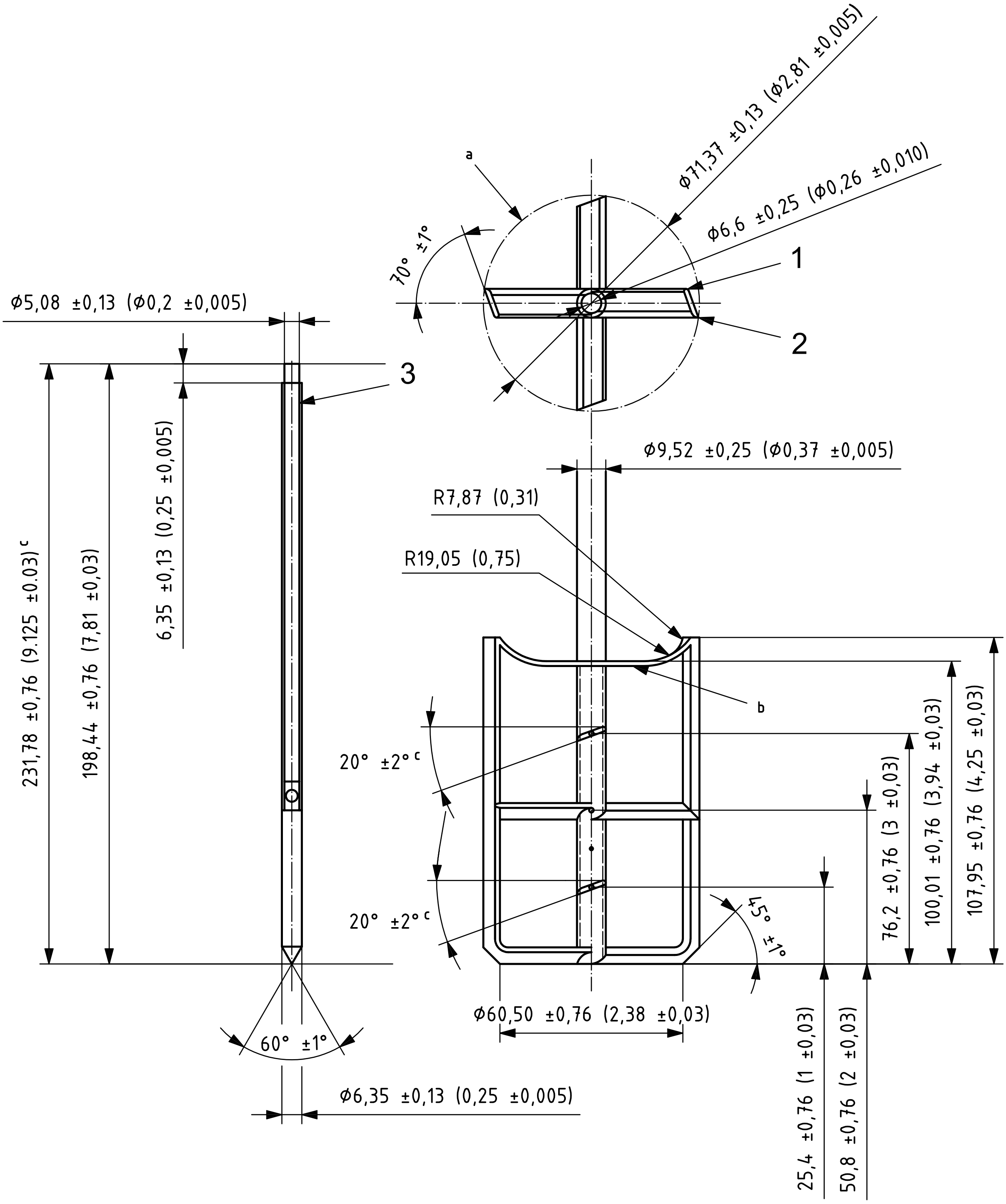
1 diaphragm

2 container base

NOTE The material is stainless steel, except the diaphragm and the hub.

Figure 11 — Typical slurry container assembly and paddle for a pressurized consistometer

Dimensions in millimetres (inches) unless otherwise indicated



Key

1 trailing edge
2 leading edge
3 paddle shaft

NOTE 1 The paddle material is stainless steel 1,6 mm × 9,5 mm (0,062 5 in × 0,375 in).

NOTE 2 Taper all leading edges out and down, and round all trailing edges.

[a] Rotate the slurry container table anti-clockwise when viewed from top of paddle.

[b] The plane of the top of the paddle brace shall be perpendicular to the shaft at all points of contact.

[c] Typical.

Figure 12 — Typical paddle for a pressurized consistometer slurry container

10.2 Calibration

10.2.1 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 gauges.

10.2.2 Consistency

10.2.2.1 Consistency of a cement slurry is expressed in Bearden units of consistency, B_c. This value shall be determined by a potentiometer mechanism and voltage measurement circuit that has been calibrated within one month prior to use, and whenever the calibration spring, resistor or contact arm is adjusted or replaced. The calibration method described in 10.2.2.2 shall be used.

10.2.2.2 A reference weight-loaded device (see Figure 13 for a typical potentiometer calibrating device and Figure 14 for a typical potentiometer mechanism) is used to produce a series of torque equivalent values for consistency for calibration. Reference weights are used to apply torque to the potentiometer spring, using the radius of the potentiometer frame as a lever arm. As reference weights are added, the spring is deflected and resulting DC voltage and/or B_c increases. See Table 8.

NOTE See manufacturer's instruction manual for procedures.

The calibrated torque-equivalent values, T, expressed in gram centimetres, are defined by Equation (2):

$$T = 78{,}2 + 20{,}02 B_\mathrm{c} \tag{2}$$

where B_c is the consistency, expressed in Bearden units.

Table 8 — Slurry consistency vs. equivalent torque
(for a potentiometer[a] mechanism with a radius of 52 mm ±1 mm)

Torque equivalent g·cm	Mass of reference weights g ± 0,1 g	Slurry consistency B_c ± 5
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 different radius, an appropriate table with equivalent tolerances shall be used.

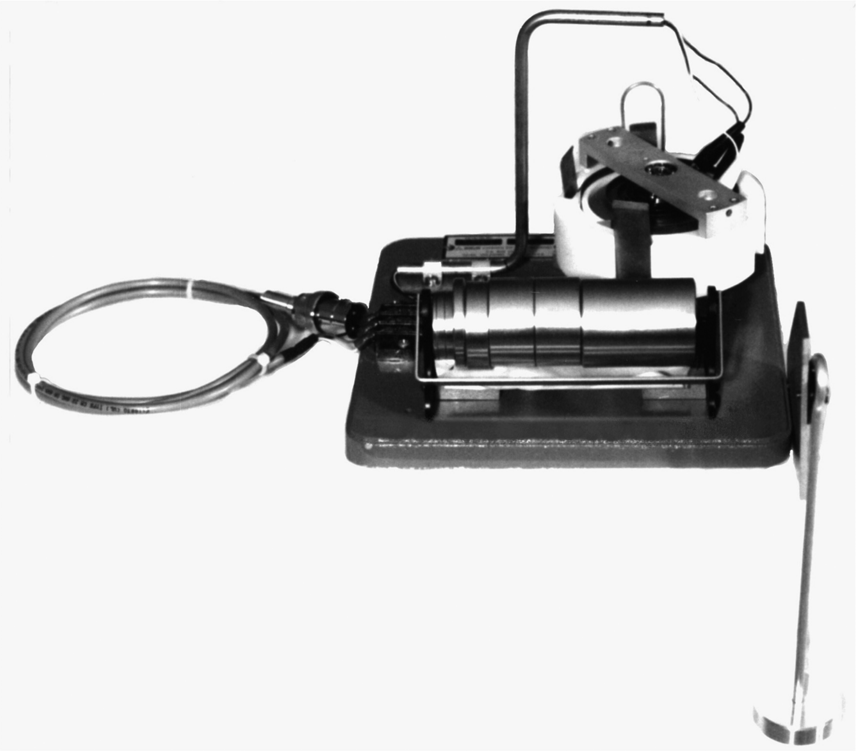
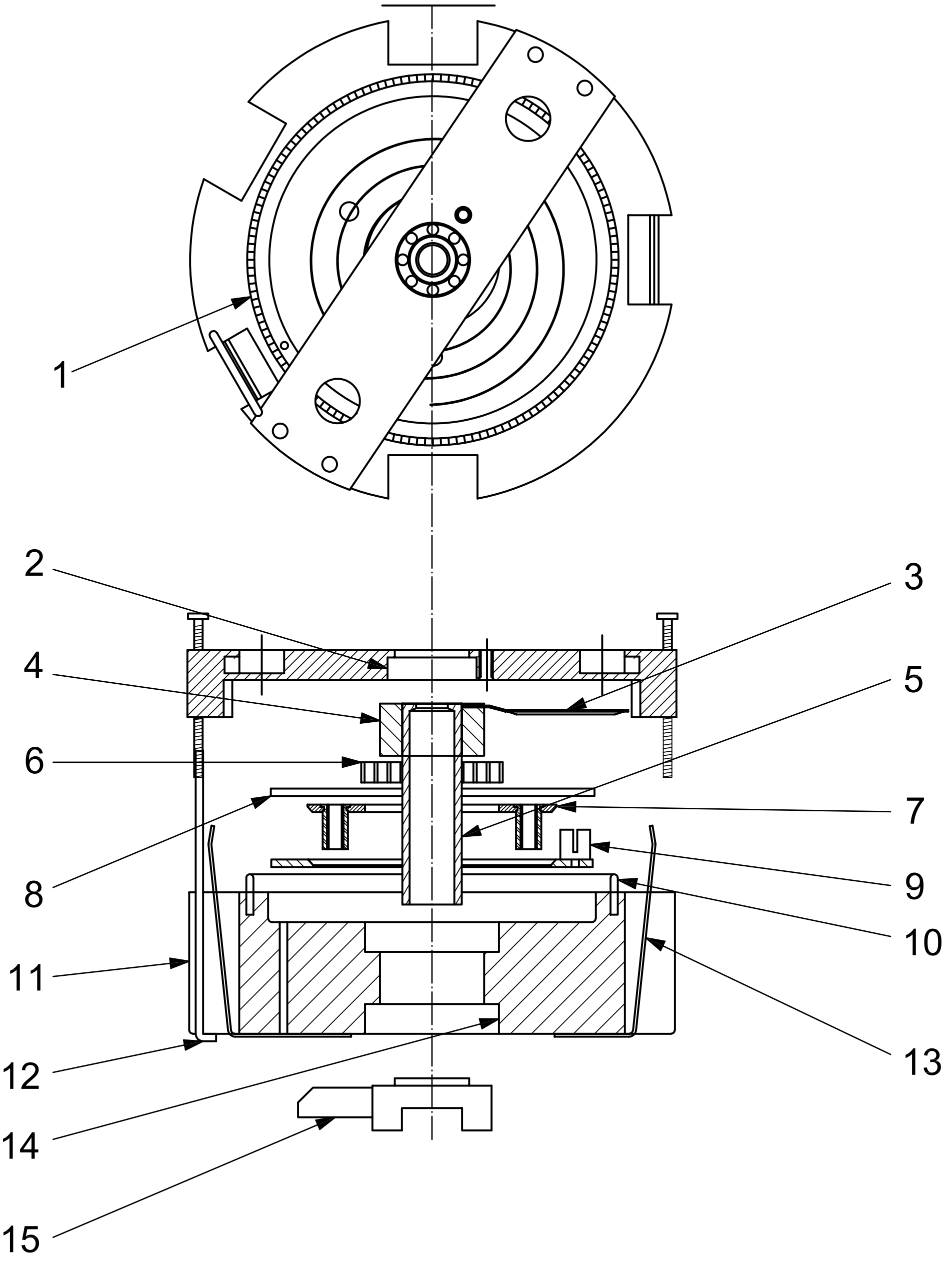


Figure 13 — Typical potentiometer calibrating device



Key

1 resistor
2 bearing shaft
3 arm contact
4 collar, spring
5 sleeve, spring
6 retainer, shaft bearing spring
7 clamp, spring adjustor
8 insulator
9 adjustor, spring
10 resistor
11 frame, mount
12 stop, mount frame
13 contact springs
14 bearing, frame
15 arm, stop

Figure 14 — Typical potentiometer mechanism for a pressurized consistometer

10.2.3 Temperature-measuring system

The temperature-measuring system shall be calibrated to an accuracy of ± 2 °C (± 3 °F). Calibration shall be no less frequently than every three months. The procedure described in Annex A is commonly used.

10.2.4 Motor speed

The motor shall rotate the slurry container at 150 r/min ± 15 r/min (2,5 r/s ± 0,25 r/s) and shall be checked annually.

10.2.5 Timer

Timers shall be accurate to within ± 30 s per hour and shall be checked annually.

10.2.6 Pressure-measuring system

Calibration shall be conducted annually against a dead-weight tester or master gauge. Gauges shall be calibrated at 17 MPa, 34 MPa, and 52 Mpa ± 1,7 MPa (2 500 psi, 5 000 psi, and 7 500 psi ± 250 psi).

10.3 Procedure

10.3.1 Operating instructions

Detailed operating instructions developed by the operator, or furnished by the equipment manufacturer, are applicable for this method and shall be followed, provided they conform to the specifications contained in this part of ISO 10426. Grease may be placed only on the threaded surfaces of the slurry container.

10.3.2 Filling of slurry container

10.3.2.1 Pour the slurry (prepared in accordance with Clause 7) into the inverted slurry container.

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 is less of a problem when the time from cessation of mixing to completing the filling operation is kept to a minimum.

10.3.2.2 When the slurry container is full, strike the outside of the container to remove entrapped air.

10.3.2.3 Then, secure the slurry container base in place.

10.3.2.4 Then, secure the centre plug (pivot bearing) into the container base.

10.3.3 Initiation of test

10.3.3.1 Place the slurry container on the drive table in the pressure vessel, start rotation of the slurry container, place the potentiometer mechanism so as to engage the shaft drive bar, and begin filling the vessel with oil.

10.3.3.2 Close the head assembly of the pressure vessel securely, insert the thermocouple, and partially engage its threads.

10.3.3.3 After the pressure vessel is completely filled with oil, tighten the threads of the thermocouple.

10.3.3.4 Initiate the test by applying pressure and temperature within 5 min after cessation of mixing.

10.3.4 Temperature and pressure control

During the test period, increase the temperature and pressure of the cement slurry in the slurry container in accordance with the appropriate specification schedule given in Table 9, Table 10, or Table 11. During the pressure and temperature ramp of schedules 4, 5 and 6, the temperature and pressure shall be maintained within ± 3 °C (± 6 °F) and ± 2 MPa (± 300 psi), respectively, of the appropriate elapsed time versus temperature and pressure target. Within 10 min after the end of the ramp, the temperature and pressure shall be within ± 1 °C (± 2 °F) and ± 0,7 MPa (± 100 psi), respectively, of the specified values. Determine the temperature of the cement slurry for specification testing by the use of a temperature-measuring device located in the centre of the sample container.

The tip of the thermocouple shall be vertically positioned within the paddle shaft in the slurry cup in such a way that it is between 45 mm (1,75 in) and 89 mm (3,5 in) above the inside of the base of the sample container. As there are many models of consistometers having different dimensions, care shall be taken to ensure that the thermocouple used is compatible with the consistometer and that the position of the tip of the thermocouple is in the correct location specified above.

Table 9 — Schedule 4 specification thickening-time test for classes A, B, C and D cement

Elapsed time min	**Pressure** MPa (psi)	**Temperature** °C (°F)
0	5,2 (750)	27 (80)
2	7,6 (1 100)	28 (83)
4	9,7 (1 400)	31 (87)
6	11,7 (1 700)	32 (90)
8	13,8 (2 000)	34 (93)
10	15,9 (2 300)	36 (97)
12	17,9 (2 600)	38 (100)
14	20,0 (2 900)	39 (103)
16	22,1 (3 200)	41 (106)
18	24,8 (3 600)	43 (110)
20	26,7 (3 870)	45 (113)

Table 10 — Schedule 5 specification thickening-time test for classes G and H cement

Elapsed time min	**Pressure** MPa (psi)	**Temperature** °C (°F)
0	6,9 (1 000)	27 (80)
2	9,0 (1 300)	28 (83)
4	11,1 (1 600)	30 (86)
6	13,1 (1 900)	32 (90)
8	15,2 (2 200)	34 (93)
10	17,3 (2 500)	36 (96)
12	19,3 (2 800)	37 (99)
14	21,4 (3 100)	39 (102)
16	23,4 (3 400)	41 (106)
18	25,5 (3 700)	43 (109)
20	27,6 (4 000)	44 (112)
22	29,6 (4 300)	46 (115)
24	31,7 (4 600)	48 (119)
26	33,8 (4 900)	50 (122)
28	35,6 (5 160)	52 (125)

Table 11 — Schedule 6 specification thickening-time test for Class D cement

Elapsed time min	Pressure MPa (psi)	Temperature °C (°F)
0	8,6 (1 250)	27 (80)
2	11,0 (1 600)	29 (84)
4	13,1 (1 900)	31 (87)
6	15,9 (2 300)	33 (91)
8	17,9 (2 600)	34 (94)
10	20,7 (3 000)	37 (98)
12	22,8 (3 300)	38 (101)
14	25,5 (3 700)	41 (105)
16	27,6 (4 000)	42 (108)
18	30,3 (4 400)	44 (112)
20	32,4 (4 700)	47 (116)
22	35,2 (5 100)	48 (119)
24	37,2 (5 400)	51 (123)
26	39,3 (5 700)	52 (126)
28	42,1 (6 100)	54 (130)
30	44,1 (6 400)	56 (133)
32	46,9 (6 800)	58 (137)
34	49,0 (7 100)	60 (140)
36	51,6 (7 480)	62 (144)

10.4 Thickening time and consistency

Record the elapsed time between the initial application of pressure and temperature (initiation of the test) to the pressurized consistometer and the time at which a consistency of 100 B_c is reached, as the thickening time for the test.

Report the maximum consistency during the 15 min to 30 min period after the initiation of the test.

10.5 Specification acceptance requirements

The acceptance requirements for the maximum consistency during the 15 min to 30 min period after the initiation of the test shall be 30 B_c for all classes of cement manufactured in accordance with this part of ISO 10426. The acceptance requirement for the thickening time shall be in accordance with Table 12.

11 Marking

The following information shall be marked on or made available with each shipment of well cement. For sacked cement, the information required shall be marked on each sack; for bulk cement, the information required shall be marked or attached to the bill of lading on each shipment:

a) manufacturer's name;

b) class and sulfate-resistance grade of cement;

c) net mass.

Table 12 — Thickening time acceptance requirement

Class	Schedule	Minimum thickening time min	Maximum thickening time min
A	4	90	NR[a]
B	4	90	NR
C	4	90	NR
D	4	90	NR
	6	100	NR
G	5	90	120
H	5	90	120

[a] NR indicates “no requirement.”

12 Packing

Well cement shall be furnished in bulk or in sacks.

Each sack shall contain a specified net mass of ± 2 %. The average net mass of 5 % of all sacks in a shipment, taken at random, shall not be less than the specified mass.

A cement sack should be resistant to moisture, resistant to damage during handling and easy to cut during transfer to bulk facilities. Cement sacks should typically consist of up to six paper layers with a minimum areic mass of 70 g/m^2 (0.014 lbm/ft^2) each with up to two polyethylene or polypropylene layers with an areic mass of 15 g/m^2 (0.003 lbm/ft^2) to 24 g/m^2 (0.005 lbm/ft^2) each included between the first and fifth paper layers. Up to two asphalt or bitumen layers may be included to further improve resistance to damage.

Flexible bulk cement containers should provide an acceptable tensile strength (safety factor 5 to 1 minimum). They should also be resistant to ultraviolet radiation when polyethylene or polypropylene layers are used and be suitably moisture-proof.

13 Bentonite

Bentonite is a naturally occurring clay mineral, composed primarily of smectite. Non-treated bentonite, for use in well cementing, is dried and ground, but not chemically treated during processing.

Bentonite meeting the requirements of this part of ISO 10426 for use in well cementing shall meet all the requirements for non-treated bentonite in accordance with ISO 13500.

Table 13 — Bentonite acceptance requirements

Requirement	Specification
Yield point/plastic viscosity ratio	1,5 maximum
Dispersed plastic viscosity	10 cP minimum
Dispersed filtrate volume	12,5 ml maximum

NOTE See ISO 13500 for test procedures.

Annex A
(informative)

Calibration procedures for thermocouples, temperature measuring systems, and controllers

A.1 Calibration methods

There are several satisfactory methods for calibrating thermocouples, including methods supplied by equipment manufacturers. See ASTM E220 for a more complete discussion of these procedures. No ASTM procedures for calibration of temperature measuring systems are available.

A.2 Thermocouple calibration

A.2.1 Apparatus

A.2.1.1 General

The individual pieces of apparatus required to carry out the calibration depend on the particular technique selected. Those features that need special attention, regardless of the technique, are highlighted in A.2.1.2 to A.2.1.4.

A.2.1.2 Heating environment

The heating medium should permit proper immersion of both the test thermocouple (the one being calibrated) and the reference thermocouple or reference thermometer. The apparatus should be capable of maintaining a stable temperature that is uniform throughout the test section.

A.2.1.3 Temperature measurement

The reference temperature of the heating medium may be measured by using either a thermometer or a thermocouple. The accuracy of the reference measuring device should be traceable to the reference of the national body responsible for standards of temperature measurement, for example the NBS certification in the USA.

A.2.1.4 Thermocouple voltage output

If a thermocouple is used to sense the reference temperature, the voltage output from the reference thermocouple and test thermocouple should be determined as described in applicable national standards, such as ASTM E220. In this case, tables of temperature vs. voltage for the type of thermocouple being used should be consulted to determine the temperature. Alternatively, a direct-reading, temperature-compensated readout instrument may be used. The accuracy of the instrument should be traceable to national standards certification.

A.2.2 Procedure

A.2.2.1 With the exception of the indicating instruments, the specific procedures are detailed in applicable national standards, such as ASTM E220. The items in A.2.2.2 through A.2.2.6 require special attention or are related to the use of the indicating type of equipment.

A.2.2.2 The test and reference thermocouples or thermometers should be placed as close together as possible in the heating medium.

A.2.2.3 After each change in heating level, the temperature should be allowed to remain at a stable value for 15 min before reading the reference temperature (or voltage) and the test thermocouple temperature (or voltage).

A.2.2.4 Several (more than three) test temperatures that span the operating range of the equipment should be used in the calibration procedure.

A.2.2.5 If the test thermocouple does not accurately sense the temperature, a calibration curve should be drawn and used to correct the indicated temperatures from the test thermocouple. Occasionally, small inaccuracies in thermocouple response can be compensated for during the calibration of the temperature measuring system being used in conjunction with the thermocouple (see A.3).

A.2.2.6 If the test thermocouple error is greater than that specified by the manufacturer, the thermocouple should be replaced by one that meets the thermocouple accuracy limits. The ASTM E220 classification “special” type J thermocouple has error limits equal to or better than ± 1 °C (± 2 °F) up to 277 °C (530 °F).

A.3 Calibration of temperature measuring systems and controllers

A.3.1 Apparatus

The calibration of temperature-measuring systems and controllers requires a millivolt source, the correct connecting thermocouple extension cable for the type of thermocouple being used and, possibly, a thermometer and a table of reference voltages. Signal sources, or calibrators, are of two types, namely, uncompensated and cold-junction-compensated. Several commercial calibrators are available that are cold-junction-compensated and have a digital display of the temperature equivalent to the millivolt signal being supplied. The accuracy of all calibration apparatus should be traceable to national-standards certification. Some older galvanometer-type temperature-indicating instruments and controllers require a stronger signal for operation than the newer potentiometric and digital-type temperature measuring systems and controllers and, therefore, require a calibrator with sufficient signal strength to give an accurate calibration.

A.3.2 Procedure

A.3.2.1 The manufacturer's procedure for calibrating temperature-measuring systems and controllers should be followed. The items in A.3.2.2 to A.3.2.5 require special attention.

A.3.2.2 The thermocouple extension cable should be fitted with a proper thermocouple grade adapter to permit plugging it into the same receptacle used for connecting the test equipment thermocouple. Care should be taken to ensure the correct polarity of the connections. Calibrators, temperature-measuring systems, and controllers should be allowed proper warm-up time, as specified by the manufacturer, for greatest accuracy.

A.3.2.3 It is only necessary that thermocouple calibrators with cold-junction compensation be properly connected with the proper thermocouple extension cable and thermocouple connectors. The temperature-measuring systems and/or controllers using this signal should have the same temperature readout, within the accuracy of the temperature or controllers as supplied by the manufacturer.

A.3.2.4 Uncompensated thermocouple calibrators require a thermometer to determine the cold-junction temperature of the thermocouple extension cable connection of the calibrator. This cold-junction temperature is set on the calibrator by the operator.

A.3.2.5 The use of an uncompensated millivolt potentiometer requires that the temperature at the calibrator/thermocouple extension cable terminals be read with a thermometer of known accuracy. The millivolt equivalent of this temperature is then subtracted from the equivalent test millivolt signal to obtain the calibrator millivolt signal used. These voltages may be found in reference mV/temperature tables for the type of thermocouple in use.

Bibliography

[1] ASTM C150, *Standard Specification for Portland Cement*

[2] ASTM E220, *Standard Test Method for Calibration of Thermocouples by Comparison Techniques*

[3] ASTM C114, *Standard Test Methods for Chemical Analysis of Hydraulic Cement*

[4] ASTM C183, *Standard Practice for Sampling and the Amount of Testing of Hydraulic Cement*

[5] ASTM C204, *Standard Test Methods for Fineness of Hydraulic Cement by Air-Permeability Apparatus*

[6] ASTM E11, *Standard Specification for Wire Cloth and Sieves for Testing Purposes*

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