

Cements and Materials for Well Cementing

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Introduction

It is necessary that users of this specification be aware that further or differing requirements can be required for individual applications. This specification 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 specification and provide details.

In this specification, where practical, US Customary (USC) 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 units. 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 specification, 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.

Cements and Materials for Well Cementing

1 Scope

1.1 General

This document specifies requirements and gives recommendations for six classes of well cements, and two classes of composite well cements including their chemical and physical requirements, and procedures for physical testing.

This specification 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 (CaSO_4) 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 the manufacture of class D cement. Annex B describes composite well cement classes K and L, which are the products obtained by intergrinding Portland cement clinker and one or more forms of CaSO_4 with composite constituents, or by subsequent blending of separately produced Portland cement with separately processed composite constituents. Composite constituents are also specified in Annex B.

This specification 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 CaSO_4 , water, or chemical additives as required for chromium (VI) reduction.

1.2 Use of Metric SI and US Customary Units

This document contains metric SI and US customary oilfield units. For the purposes of this document, the conversion between the systems is not exact and has been intentionally rounded to allow for ease of use in calibration and measurement.

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 applies (including any addenda/errata). However, not all documents listed may apply to your specific needs. The body of the standard should be referred to for how these documents are specifically applied.

API Specification 13A, *Specification for Drilling Fluid Materials*

ASTM¹ C109/C109M, *Standard Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or [50-mm] Cube Specimens)*

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

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

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

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

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

¹ ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, www.astm.org.

ASTM E1404, *Standard Specification for Laboratory Glass Conical Flasks*

EN² 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*

ISO³ 24450, *Laboratory glassware — Wide-necked boiling flasks*

3 Terms, Definitions, Acronyms, and Abbreviations

3.1 Terms and Definitions

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

3.1.1

additive

Material added to a cement slurry to modify its properties.

3.1.2

atmospheric pressure consistometer

Device used for pre-conditioning a cement slurry under temperature and at atmospheric pressure.

3.1.3

Bearden units of consistency

B_c

Measure of the consistency of a cement slurry when determined on a pressurized consistometer.

3.1.4

blending

A process in which two or more ingredients are combined into an intimate and uniform product of finely divided dry material, as by special blending equipment.

3.1.5

cement

Portland cement

Ground clinker generally consisting of hydraulic calcium silicates and aluminates, and usually containing one or more forms of CaSO_4 as an interground additive.

3.1.6

cement class

Designation for classification of well cement according to its intended use.

3.1.7

clinker

Fused materials produced in the kiln during cement manufacturing that are interground with CaSO_4 to make cement.

² European Committee for Standardization, Rue de la Science 23, B-1040 Brussels, Belgium; www.cen.eu.

³ International Organization for Standardization, BIBC II, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland; www.iso.org.

3.1.8**compressive strength**

Measured strength of a set cement sample calculated by the force required to cause it to fail in compression, expressed as a force per unit of area.

3.1.9**free-fluid****(free-water)**

Colored or colorless liquid that separates from a cement slurry under static conditions.

3.1.10**grade**

Designation achieved under the API system for denoting the sulfate resistance of a particular cement.

3.1.11**intergrinding**

A process involving grinding Portland cement clinker with suitable other components to produce finely divided dry material.

3.1.12**load frame**

Frame to place a load on cement samples for determining compressive strength of cement.

3.1.13**pressurized consistometer**

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

3.1.14**slurry container****(slurry cup)**

Container in an atmospheric or pressurized consistometer used to hold the slurry for pre-conditioning purpose or for the thickening-time test.

3.1.15**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.

3.2 Acronyms and Abbreviations

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

ASTM American Society for Testing and Materials

C₃A tricalcium aluminate

C₄AF tetracalcium aluminoferrite

CaSO₄ calcium sulfate

C₃S tricalcium silicate

EN European Committee for Standardization

HSR	high sulfate-resistant
INSP	inspection (dimension requiring verification)
ISO	International Organization for Standardization
MSR	moderate sulfate-resistant
NIST	National Institute of Standards and Technology
O	ordinary
OD	outside diameter

4 Requirements

4.1 Specification, Chemical, and Physical Requirements

4.1.1 Classes and Grades

4.1.1.1 General

Well cements shall be specified using the classes A, B, C, D, G, or H and the grades ordinary (O), moderate sulfate-resistant (MSR), and high sulfate-resistant (HSR). Composite well cements shall be specified using classes K and L, consisting of Portland cements and additional composite constituents according to Annex B.

A well cement, or composite well cement, that has been manufactured and supplied in accordance with this specification may be mixed and placed in the field using alternate water ratios and/or additives. It is not intended that manufacturing compliance with this specification 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 Well Cement Testing by the Manufacturer

To conform to this standard, the manufacturer shall perform testing of well cements to the following:

- at a laboratory under a Quality Management System that conforms to a nationally or internationally recognized standard, such as API Specification Q1 or ISO 9001;
- at the same place of manufacturing as the Portland cement component in the case of composite well cements;
- on unmodified samples taken in accordance with 4.2, including composite samples, defined as a sample blended in the laboratory from multiple samples that are taken from the product in accordance with 4.2.

NOTE This clause does not prohibit users of well cement from independently performing specification testing according to this standard.

4.1.1.3 Class A

This product is obtained by grinding clinker that consists of hydraulic calcium silicates and contains one or more forms of CaSO_4 as an interground additive. Processing additives used in the manufacture of Class A cement shall meet the requirements given in ASTM C465.

NOTE This product is intended for use when special properties are not required and is available only in O grade, similar to the requirements given in ASTM C150 for Type I cement.

4.1.1.4 Class B

This product is obtained by grinding clinker that consists of hydraulic calcium silicates and contains one or more forms of CaSO_4 as an interground additive. Processing additives used in the manufacture of Class B cement shall meet the requirements given in ASTM C465.

NOTE 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 the requirements given in ASTM C150 for Type II cement.

4.1.1.5 Class C

This product is obtained by grinding clinker that consists of hydraulic calcium silicates and contains one or more forms of CaSO_4 as an interground additive. Processing additives used in the manufacture of Class C cement shall meet the requirements given in ASTM C465.

NOTE This product is intended for use when conditions require high early strength, and is available in O, MSR, and HSR grades, similar to the requirements given in ASTM C150 for Type III cement.

4.1.1.6 Class D

This product is obtained by grinding clinker that consists of hydraulic calcium silicates and contains one or more forms of CaSO_4 as an interground additive. Processing additives used in the manufacture of Class D cement shall meet the requirements given in ASTM C465.

Further, at the option of the manufacturer, suitable set-modifying agents may be interground or blended during manufacture.

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

4.1.1.7 Class G

This product is obtained by grinding clinker that consists of hydraulic calcium silicates and contains one or more forms of CaSO_4 as an interground additive. No additives other than CaSO_4 , water, or chemical additives as required for chromium (VI) reduction shall be interground or blended with the clinker during manufacture of Class G well cement, provided that such additives do not prevent the well cement from performing its intended purpose.

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

4.1.1.8 Class H

This product is obtained by grinding clinker that consists of hydraulic calcium silicates and contains one or more forms of CaSO_4 as an interground additive. No additives other than CaSO_4 , water, or chemical additives as required for chromium (VI) reduction shall be interground or blended with the clinker during manufacture of Class H well cement, provided that such additives do not prevent the well cement from performing its intended purpose.

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

4.1.1.9 Composite Well Cements

See Annex B for the specifications of composite well cements.

^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 Added mineral ingredients shall have a calcium carbonate content greater or equal than 75 %.

^d NR indicates "no requirement."

^e 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 (in percent) are calculated according to the ratio of the mass percentages of aluminum oxide to ferric oxide (Al_2O_3 to Fe_2O_3), where w is the percentage mass fraction of the compound indicated in the subscript:

1) When $w_{Al_2O_3}/w_{Fe_2O_3}$ is greater than 0.64, the compounds shall be calculated as follows:

$$i) \quad C_3A = 2.65 w_{Al_2O_3} - 1.69 w_{Fe_2O_3}$$

$$ii) \quad C_3S = 4.07 w_{CaO} - 7.60 w_{SiO_2} - 6.72 w_{Al_2O_3} - 4.43 w_{Fe_2O_3} - 2.85 w_{SO_3}$$

$$iii) \quad C_4AF = 3.04 w_{Fe_2O_3}$$

2) When $w_{Al_2O_3}/w_{Fe_2O_3}$ is 0.64 or less, the C_3A content is zero, and the C_3S and C_4AF shall be calculated as follows:

$$i) \quad C_3S = 4.07 w_{CaO} - 7.60 w_{SiO_2} - 4.48 w_{Al_2O_3} - 2.86 w_{Fe_2O_3} - 2.85 w_{SO_3}$$

$$ii) \quad C_4AF = 3.04 w_{Fe_2O_3}$$

^f The sodium oxide equivalent, expressed as Na_2O equivalent, shall be calculated by the formula:

$$Na_2O \text{ equivalent} = 0.658 w_{K_2O} + w_{Na_2O}$$

4.1.3 Physical and Performance Requirements

Well cement shall conform to the respective physical and performance requirements specified in Table 2 and in Sections 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 4)				46	46	56	38	44	38
Fineness tests (alternative methods) (Section 6)									
Turbidimeter (specific surface, minimum, m ² /kg)				150	160	200	NR ^a	NR	NR
Air permeability (specific surface, minimum, m ² /kg)				280	280	400	NR	NR	NR
Free-fluid content , maximum, percent (Section 8)				NR	NR	NR	NR	5.9	5.9
Compressive Strength Test Curing time: 8 hr	Schedule Number Table 7	Final Curing Temperature °C (°F)	Curing Pressure MPa (lbf/in. ²)	Minimum Compressive Strength MPa (lbf/in. ²)					
(Section 9)	NA ^b	38 (100)	atmospheric	1.7 (250)	1.4 (200)	2.1 (300)	NR	2.1 (300)	2.1 (300)
(Section 9)	NA	60 (140)	atmospheric	NR	NR	NR	NR	10.3 (1500)	10.3 (1500)
(Section 9)	6S	110 (230)	20.7 (3000)	NR	NR	NR	3.4 (500)	NR	NR
Compressive Strength Test Curing time: 24 hr	Schedule Number Table 7	Final Curing Temperature °C (°F)	Curing Pressure MPa (lbf/in. ²)	Minimum Compressive Strength MPa (lbf/in. ²)					
(Section 9)	NA	38 (100)	atmospheric	12.4 (1800)	10.3 (1500)	13.8 (2000)	NR	NR	NR
(Section 9)	4S	77 (170)	20.7 (3000)	NR	NR	NR	6.9 (1000)	NR	NR
(Section 9)	6S	110 (230)	20.7 (3000)	NR	NR	NR	13.8 (2000)	NR	NR

Well Cement Class			A	B	C	D	G	H
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) minutes					
(Section 10)	4	30	90/NR	90/NR	90/NR	90/NR	NR	NR
(Section 10)	5	30	NR	NR	NR	NR	90/120	90/120
(Section 10)	6		NR	NR	NR	100/NR	NR	NR
<p>^a NR indicates "no requirement."</p> <p>^b NA indicates "not applicable."</p> <p>^c Bearden units of consistency, B_c, obtained on a pressurized consistometer as defined in Section 10 and calibrated in accordance with Annex C.</p>								

4.2 Sampling Frequency, Timing of Tests, and Equipment

4.2.1 Sampling Frequency

4.2.1.1 Well Cement Classes C, D, G, and H

A sample for testing shall represent the product as produced. At the discretion of the manufacturer, a sample shall be taken by either of the following methods:

- over an interval of 24 h; or
- on a 1000-ton (maximum) production run.

4.2.1.2 Well Cement Classes A and B

A sample for testing shall represent the product as produced. At the discretion of the manufacturer, a sample shall be taken by either of the following methods:

- over a 14-day continuous production interval; or
- on a 25,000 ton (maximum) production run.

4.2.2 Time from Sampling to Testing

Each sample shall be tested for conformance to this specification. 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 14 are for the purposes of manufacturing the cement-specification test equipment. Dimensional recertification is not required, with the exception of key dimensions tagged "INSP," which shall be verified to judge wear (see Annex C).

Table 3—Specification Test Equipment for Well-Cement Manufacturers

Test or preparation	Well cement Classes	Section Reference	Required Equipment
Sampling	All	5	Apparatus as specified in ASTM C183 NOTE For the purposes of this provision, EN 196-7 is equivalent to ASTM C183.
Fineness	A, B, C	6	Turbidimeter and auxiliary equipment as specified in ASTM C115 or air permeability apparatus and auxiliary equipment as specified in ASTM C 204. NOTE For the purposes of this provision, EN 196-6 is equivalent to ASTM C204.
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
Pressure cured compressive strength	D	9	Apparatus as specified in 9.1
Thickening time	All	10	Pressurized consistometer specified in 10.1

4.2.4 Calibration

Equipment shall be calibrated in accordance with the requirements in Annex C.

5 Sampling Procedure

One or more of the procedures in accordance with ASTM C183 shall be used to secure a sample of well cement for specification testing purposes.

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

6 Fineness Tests

6.1 Procedure

Tests for fineness of well cement shall be in accordance with either the procedure in ASTM C115 for the turbidimeter test or the procedure in ASTM C204 for the air permeability test that is at the discretion of the manufacturer.

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

6.2 Requirements

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

7 Preparation of Slurry for Free-fluid, Compressive Strength and Thickening-time Tests

7.1 Apparatus

7.1.1 General

Unless otherwise noted, all apparatuses shall be calibrated according to Annex C.

7.1.2 Electronic or Mechanical Balances

Balances shall have, at a minimum, a two-decimal-place precision reading and conform to requirements specified in Annex C.

7.1.3 Weight Sets

Weight sets include sets used daily for routine weighing and reference weight sets used for calibration. Weight sets shall conform to requirements specified in Annex C.

7.1.4 Sieves

An 850 μm wire cloth sieve (US sieve No. 20), shall be used for sieving cement prior to slurry preparation.

NOTE The sieve does not require verification.

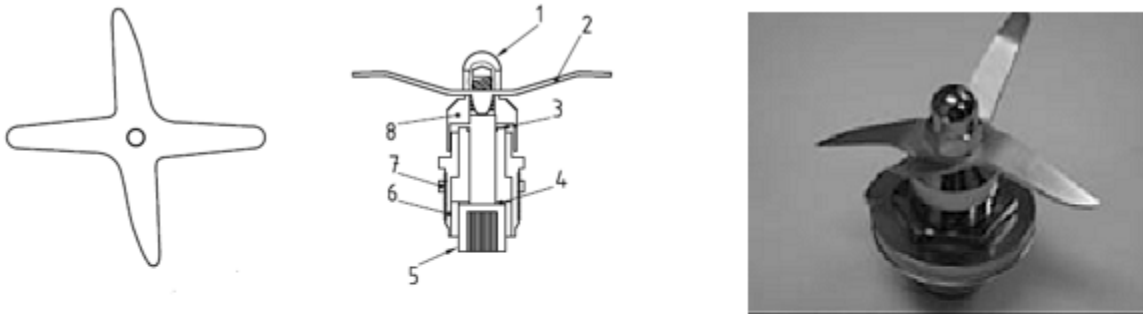
7.1.5 Mixing Devices

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

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 blade shall be mounted with its sharp edge on the leading-edge rotation. 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. According to Annex C, the mixing blade shall be weighed initially and replaced with an unused blade if observed blade deformation has occurred or after a blade has lost 10 % of its mass.



Figure 1—Example of a Typical Cement-Mixing Device



Key

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

Figure 2—Common Blade Assembly

7.2 Procedure

7.2.1 Sieving

Prior to mixing, the cement shall be sieved using the method described according to ASTM C183 using a sieve defined in 7.1.4.

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

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 $23\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$ ($73\text{ }^{\circ}\text{F} \pm 2\text{ }^{\circ}\text{F}$), and the temperature of the cement within 60 s prior to mixing shall be $23\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$ ($73\text{ }^{\circ}\text{F} \pm 2\text{ }^{\circ}\text{F}$).

7.2.3 Mix Water

Distilled or deionized 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, or other losses.

7.2.4 Mixing Quantities

The quantities of slurry component shown in Table 4 shall be used for testing. The use of the quantities of components shown in Table 4 results in mix-water percentages (based on the mass of dry cement) consistent with the water percentages shown in Table 2.

Table 4—Slurry Requirements

Components	Class A and B g	Class C g	Class D and H g	Class G g
Mix water	355 \pm 0.5	383 \pm 0.5	327 \pm 0.5	349 \pm 0.5
Cement	772 \pm 0.5	684 \pm 0.5	860 \pm 0.5	792 \pm 0.5

For composite well cement classes without specified mix-water percentages (see Annex B), the quantities of composite well cement and mix water shall be measured at ± 0.5 g based on a calculated slurry volume of 600 mL.

7.2.5 Mixing Cement and Water

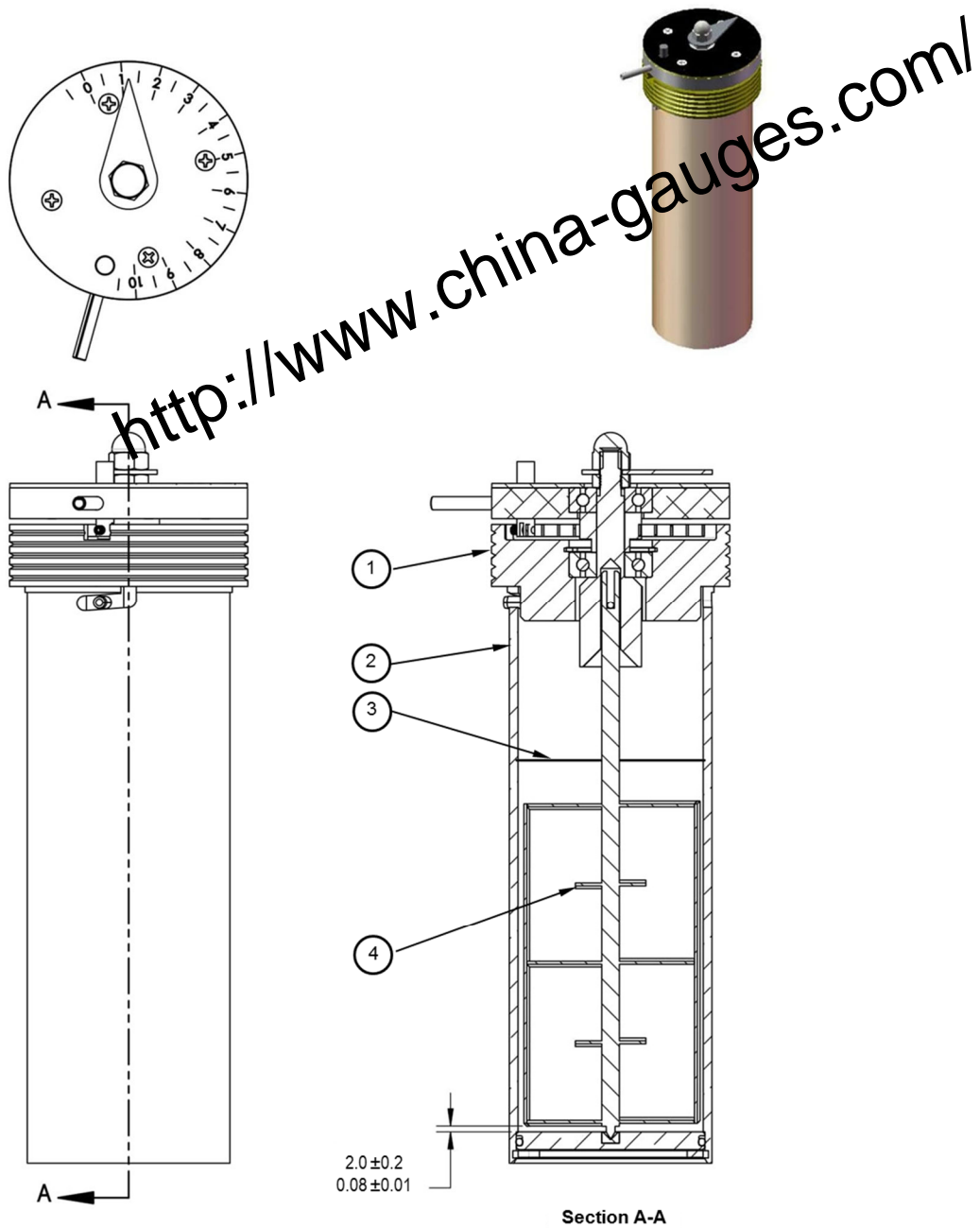
The mixing container with the required mass of mix water, as specified in Table 4, shall be placed on the mixer base, with the motor turned on and maintained at $4000\text{ r/min} \pm 250\text{ r/min}$ while the cement sample is added at a uniform rate for no more than 15 s. After 15 s at $4000\text{ r/min} \pm 250\text{ r/min}$, place the cover on the mixing container and continue mixing at $12,000\text{ r/min} \pm 500\text{ r/min}$ for $35\text{ s} \pm 1\text{ s}$.

8 Free-fluid Test (free-water)

8.1 Apparatus

8.1.1 Atmospheric Pressure Consistometer

The atmospheric pressure consistometer (see Figures 3 through 6) 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 pressure consistometer consists of a rotating cylindrical slurry container, equipped with a fixed paddle assembly, in a temperature-controlled liquid bath. It shall be capable of maintaining the temperature of the bath and the slurry container rotating speed requirements as specified in Annex C. The paddle and all parts of the slurry container exposed to the slurry shall be constructed of corrosion-resistant materials.

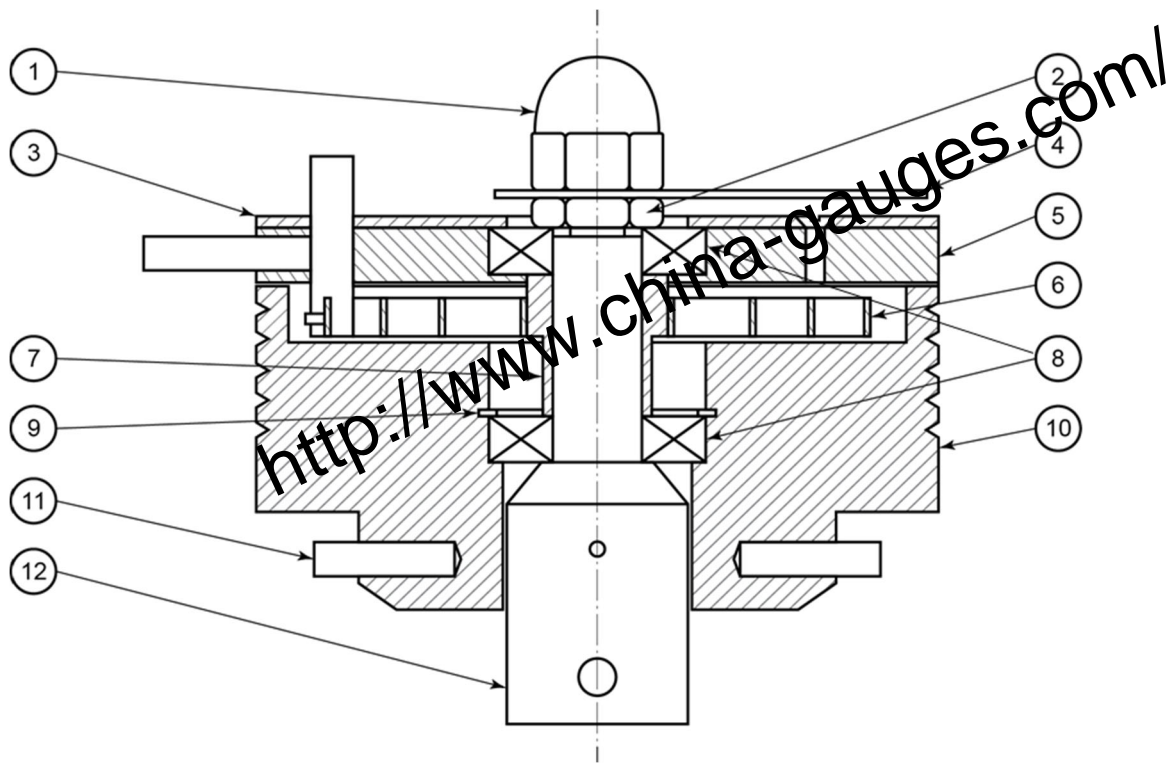


Key

- | | |
|-----------------------------------|-------------------------------|
| 1 lid (see Figure 4) | 3 fill-level indicator groove |
| 2 slurry container (see Figure 5) | 4 paddle (see Figure 6) |

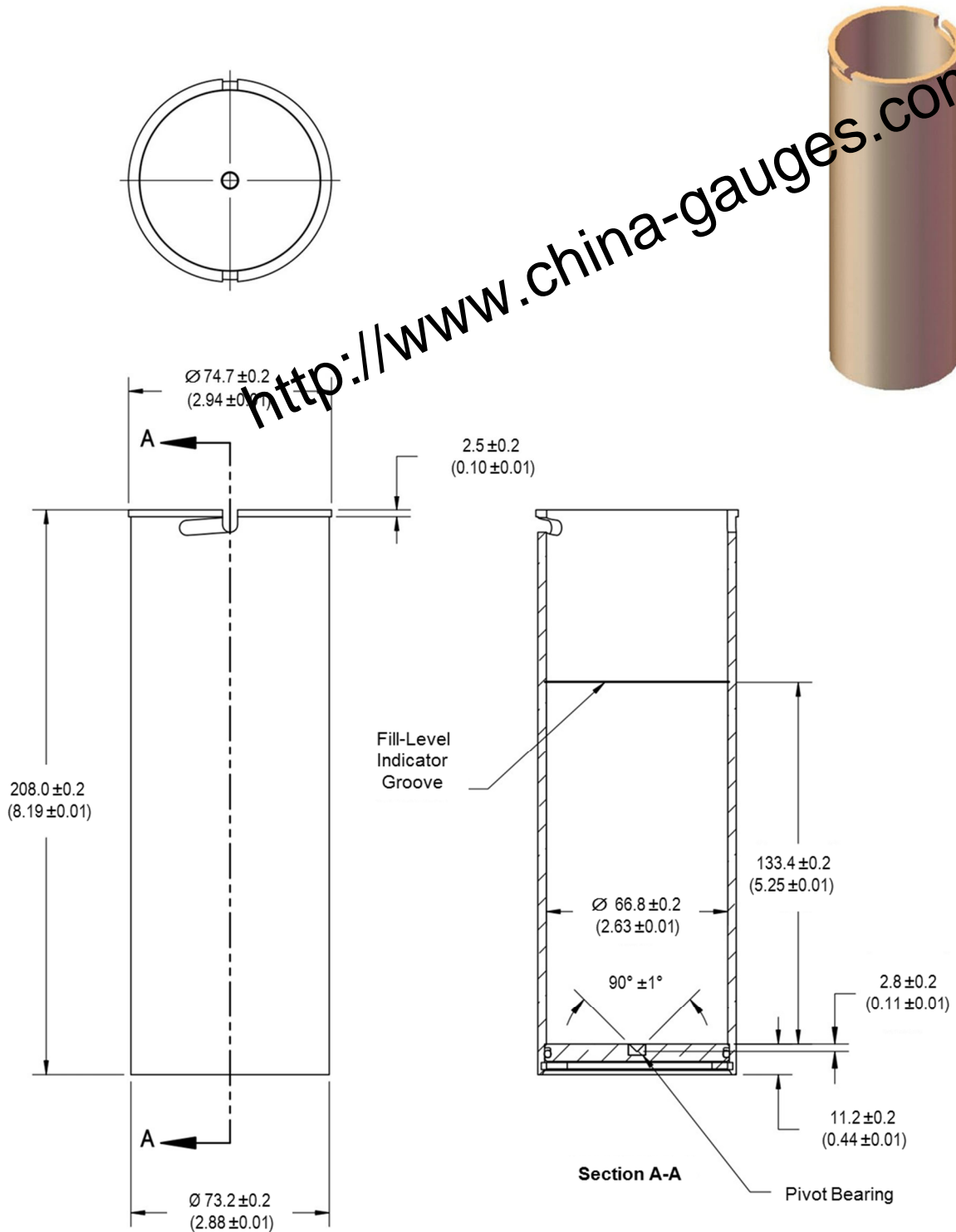
NOTE Dimensions are provided for manufacturing reference only and are in mm (in.) unless otherwise indicated.

Figure 3—Container Assembly for an Atmospheric Pressure Consistometer

**Key**

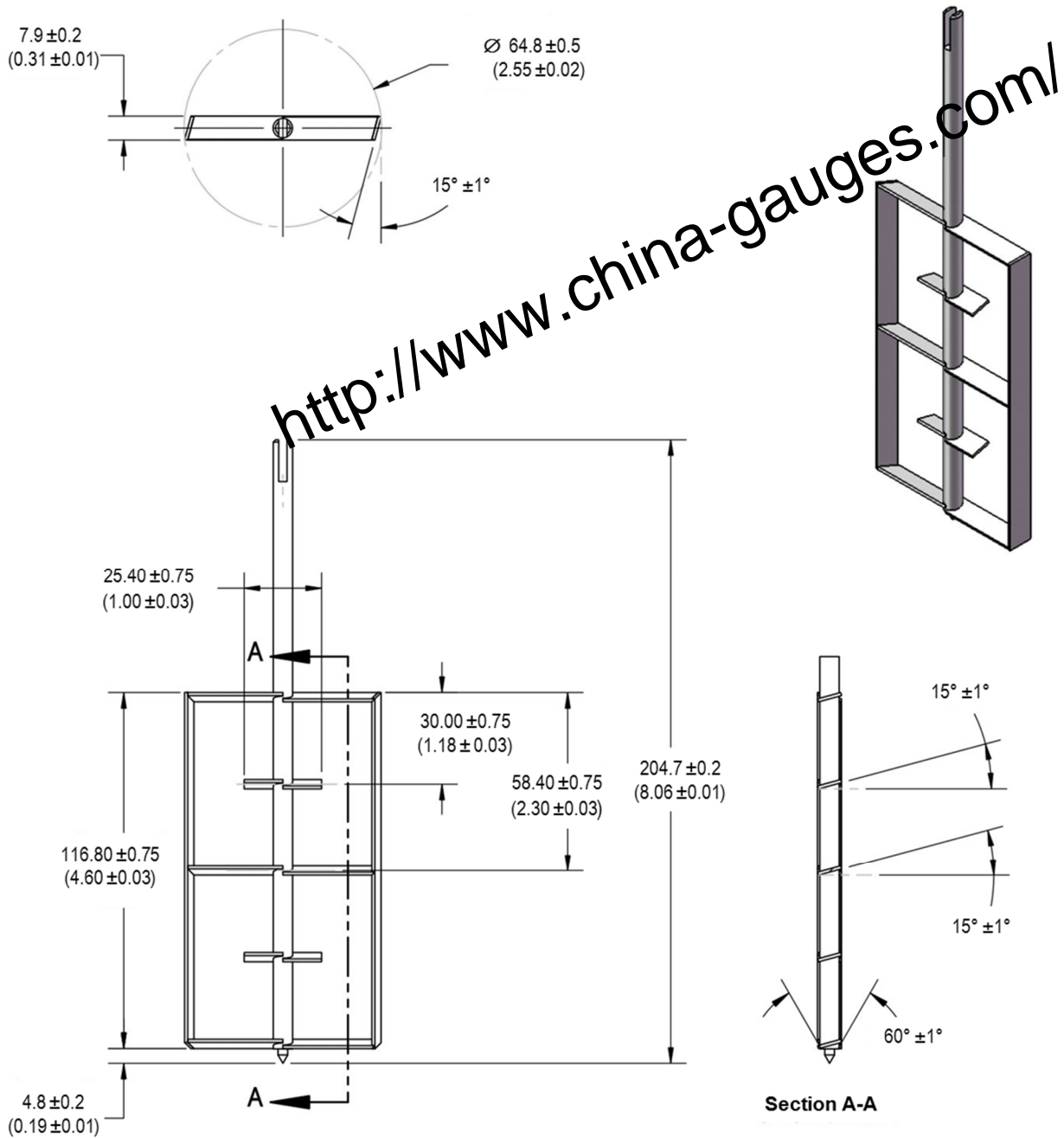
1	cap lock nut	7	collar
2	center lock reverse jam nut	8	bearing
3	dial	9	retaining ring
4	pointer	10	lid
5	dial and base assembly	11	roll pin
6	spring	12	shaft

Figure 4—Typical Lid and Mechanism for an Atmospheric Pressure Consistometer (see Figure 3)



NOTE Dimensions are provided for manufacturing reference only in mm (in.) unless otherwise indicated.

Figure 5—Slurry Container for an Atmospheric Pressure Consistometer (see Figure 3)



NOTE 1 Paddle material: stainless steel, 1.0 mm ± 0.1 mm (0.04 in. ± 0.005 in.).

NOTE 2 Shaft material: stainless steel, 6.4 mm ± 0.1 mm (0.25 in. ± 0.005 in.).

NOTE 3 Dimensions are provided for manufacturing reference only and are in mm (in.) unless otherwise indicated.

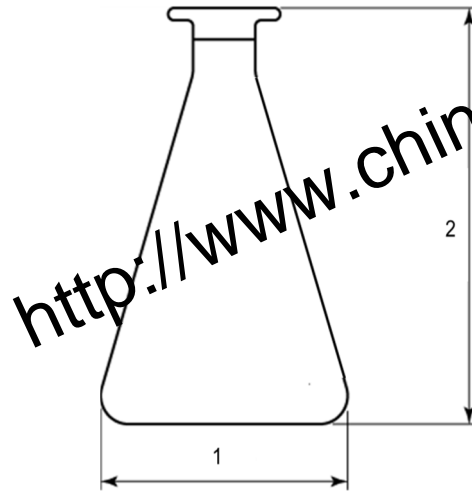
Figure 6—Paddle for an Atmospheric Pressure Consistometer (see Figure 3)

8.1.2 Balances

Balances shall have at a minimum two-decimal-place precision reading and conform to requirements as specified in Annex C.

8.1.3 Test Flask

A wide-mouth general-purpose conical glass flask (Erlenmeyer) with a capacity of 500 mL shall be used; see Figure 7. The conical flask shall be in accordance with ASTM E1404, type I, class 2, or with ISO 24450; see Table 5.



Key

- 1 body outside diameter (OD at widest point).
- 2 overall height

Figure 7—Typical Conical 500-mL Flask for Free-fluid Measurement

Table 5—Dimensions for a 500-mL Conical Flask without Caps or Stoppers

Specification	ASTM type 1, class 2 Conical Flask	ISO Wide-necked Conical Flask
Body OD widest point	105 mm (4.13 in.) (maximum)	105 mm \pm 2 mm (4.13 in. \pm 0.08 in.)
Overall height	186 mm (7.32 in.) (maximum)	175 mm \pm 4 mm (6.89 in. \pm 0.16 in.)

8.2 Calibration

8.2.1 General

Unless otherwise noted, all apparatuses shall be calibrated to the requirements given in Annex C.

8.2.2 Temperature Measuring System

Temperature-measuring and -controlling devices includes thermometers, digital thermometers, thermocouples, temperature controllers on consistometers, and those that are used separate from or are not an integral part of the instrument. Thermocouples mounted in the cylinder wall of a consistometer, not used for controlling the temperature in the consistometer, are permanently exempt from calibration (see Annex C). The calibration procedure for a thermocouple as specified in Annex D is also commonly used.

8.2.3 Slurry Container Rotational Speed

The rotational speed shall be 150 r/min \pm 15 r/min (see Annex C).

8.2.4 Timer

The timer shall be accurate to within ± 5 seconds per hour. If not within required accuracy, the units shall be adjusted or replaced.

8.3 Procedure

The following procedure describes how the free-fluid test shall be performed:

- a) Prepare the slurry in accordance with Section 7.
- b) For an atmospheric pressure consistometer, fill a clean and dry consistometer slurry container to the fill groove, or as specified in 10.3.2 for use of a pressurized consistometer.
- c) Assemble the slurry container and associated parts, place them in the consistometer, and start the motor according to the operating instructions of the manufacturer so that the interval between completion of mixing and starting of the consistometer shall not exceed 5 minutes.
- d) Stir the slurry in the consistometer for a period of 20 min ± 30 s while the temperature of the slurry is 27 °C ± 2 °C (80 °F ± 3 °F) and atmospheric pressure throughout the stirring period.
- e) 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.
 - 1) Record the actual mass transferred, m_S .
 - 2) Seal the flask to prevent evaporation.
- f) Set the slurry-filled flask on a surface that is nominally level and vibration-free.
 - 1) The air temperature to which the slurry-filled flask is exposed shall be 23 °C ± 3 °C (73 °F ± 5 °F).
 - 2) The temperature sensor for measuring air temperature shall meet the requirements given in 8.2.2.
 - 3) Let the slurry-filled flask remain undisturbed for a period of 2 h ± 5 min.
- g) Remove the supernatant fluid that has developed with a pipette or syringe at the end of the 2 h ± 5 min period.
 - 1) Measure the volume of supernatant fluid to an accuracy of ± 0.1 mL.
 - 2) Record the measure volume as “milliliters free-fluid”, V_{FF} .

Convert the milliliters 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):

$$\varphi = \frac{V_{FF} \times \rho}{m_S} \times 100 \quad (1)$$

where

- V_{FF} is the volume of free-fluid (supernatant fluid) collected, expressed in milliliters;
- ρ is the density of slurry, equal to 1.98 g/cm³ for Class H at 38 % water and 1.91 g/cm³ for Class G at 44 % water; if the density of the base cement is other than the typical value of 3.13 g/cm³ ±0.04 g/cm³, 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:

$$m_S = 791.7 \text{ g}$$

$$V_{FF} = 15.1 \text{ mL}$$

$$\rho = 1.98 \text{ g/cm}^3 \text{ (Class H)}$$

$$\phi = [(15.1 \text{ mL} \times 1.98 \text{ g/cm}^3) / 791.7 \text{ g}] \times 100$$

$$\phi = 3.78 \%$$

NOTE Milliliters and cubic centimeters are equal and interchangeable.

8.5 Acceptance Requirements

The free-fluid for Class G and Class H well cements shall not exceed 5.9 % of the slurry volume.

9 Compressive Strength Tests

9.1 Apparatus

9.1.1 General

Unless otherwise noted, all apparatuses shall be calibrated to the requirements given in Annex C.

9.1.2 Cube Molds and Compressive Strength-testing Machine

Cement specimens are prepared using 50-mm or 2-in. cube-molds. Molds and testing machines for compressive strength tests shall conform to the requirements given in ASTM C109/C109M, except for the following:

- a) cube-molds.
 - 1) Molds may be separable into more than two parts.
 - 2) Dimensions for 50 mm and 2 in. cube molds shall be manufactured in accordance with ASTM C109. Except for Table 6 dimensions, no other dimensions are auditable.

Table 6—Dimensions for 50-mm and 2-in. Cube-molds in Use

	50-mm Cube	2-in. Cube
Distance between opposite sides	50.0 mm ±0.5 mm	2.00 in. ±0.02 in.
Height of compartments	50.0 mm +0.25mm/-0.38 mm	2 in. +0.01 in./-0.015 in.

- b) Compressive strength-testing machine (load frame).
- 1) The two surfaces of the bearing blocks shall have a diameter or diagonal of no less than 70.7 mm (2.83 in.).
 - 2) The bearing blocks should have a Rockwell hardness of no less than 30 HRc. This is not additible.

9.1.3 Cube Mold Base and Cover Plates

Glass, brass, or stainless steel plates having a minimum thickness of 5 mm (0.2 in.) are used. Cover plates may be grooved on the surface that contacts the top of the cement.

9.1.4 Water Curing Baths

9.1.4.1 General

A curing bath or tank having dimensions that allow the complete immersion of a compressive strength mold(s) in water and are capable of maintaining the prescribed test temperatures within ± 2 °C (± 3 °F) shall be used. The two types of water curing baths shall have the requirements given in 9.1.4.2 and 9.1.4.3.

9.1.4.2 Atmospheric Pressure Water 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 that has a circulating system.

9.1.4.3 Pressurized Curing Bath (curing chamber)

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 \pm 3.4 MPa (3000 lbf/in.² \pm 500 lbf/in.²). The vessel shall be capable of fulfilling the appropriate specification schedule given in Table 7.

9.1.5 Water 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 \pm 3 °C (80 °F \pm 5 °F).

9.1.6 Temperature-measuring System

9.1.6.1 General

The temperature-measuring system shall meet the requirements shown in Annex C. This includes thermometers or digital thermometers, thermocouples, temperature controllers on water curing baths, curing chambers, cooling baths, and those that are used separate from or are not an integral part of the instrument. The calibration procedure described in Annex D is commonly used.

9.1.6.2 Temperature-measuring Device

A thermometer or digital thermometer with a minimum range from 20 °C to 90 °C (70 °F to 180 °F), with minimum scale divisions not exceeding 1 °C (2 °F), or a thermocouple with a minimum range of 20 °C to 120 °C (70 °F to 250 °F) shall be used.

9.1.7 Puddling Rod

A corrosion-resistant puddling rod of diameter 6 mm \pm 2 mm (0.25 in. \pm 0.08 in.) shall be used.

9.1.8 Sealant

Sealants are commonly used to seal the exterior contact points of the specimen mold and have a consistency that permits ease of use, good sealing properties to prevent leakage, water resistance, and inertness to the cement. Sealants are non-corrosive when subjected to the curing temperatures and pressures given in Table 6.

9.1.9 Mold-release Agent (optional)

A thin layer of mold-release agent may be applied to the interior surfaces of the mold to prevent the sample from being damaged when removed from the mold. The mold-release agent should comply with 9.1.8.

9.1.10 Timer

The timer shall be accurate to within ± 5 seconds per hour. If not within required accuracy, the units shall be adjusted or replaced.

9.1.11 Caliper Device

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

9.2 Procedure

9.2.1 Preparation of Molds

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

9.2.2 Preparation and Placement of Slurry

9.2.2.1 Slurry

Three or more specimens of cement slurry shall be prepared in accordance with Section 7.

9.2.2.2 Placing Slurry in Molds

To prepare each sample, the following procedure shall be followed.

- a) Place the slurry in the prepared molds in a layer approximately one-half of the mold depth.
- b) Place the slurry in all the specimen compartments before commencing the puddling operation.
- c) Use the puddling rod to puddle the slurry in an evenly distributed pattern (27 times per specimen).
- d) After puddling the first layer, stir the remaining slurry by hand, using a puddling rod or spatula to minimize segregation.
- e) Fill the molds to overflowing and puddle the same as for the first layer.
- f) After puddling, use a straight-edge to strike off the excess slurry level with the top of the mold.
- g) Discard specimens in molds that leak.
- h) Place a prepared cover plate on top of the mold.

9.2.2.3 Pressure and Temperature Schedules

For Class A, B, C, G, and H cements, place the specimens in the water bath preheated to the final curing temperature (see Table 8) for tests at atmospheric pressure within 5 min after mixing.

For Class D cement, within 5 min after mixing, place the specimens in the pressure vessel filled with water at $27\text{ }^{\circ}\text{C} \pm 3\text{ }^{\circ}\text{C}$ ($80\text{ }^{\circ}\text{F} \pm 5\text{ }^{\circ}\text{F}$), and apply the temperature and pressure given in Table 7.

Table 7—Specification Schedules for Pressurized Curing of Class D Cement Specimens

Schedule Number	Curing Pressure ^a MPa (lbf/in. ²)	Elapsed Time from First Application of Heat and Pressure 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 $^{\circ}\text{C}$ ($^{\circ}\text{F}$)										
4S	20.7 (3000)	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 (3000)	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 $20.7\text{ MPa} \pm 3.4\text{ MPa}$ ($3000\text{ lbf/in.}^2 \pm 500\text{ lbf/in.}^2$) 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-hour temperature shall be maintained within $\pm 2\text{ }^{\circ}\text{C}$ ($\pm 3\text{ }^{\circ}\text{F}$) 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 given in Table 7) to testing the specimen for strength. The strength of the specimen shall be tested at the appropriate time, shown in Table 8.

For specimens cured at atmospheric pressure (Class A, B, C, G, and H cements), the curing period starts when specimens are initially placed in the curing bath preheated to the test temperature. The test temperature shall be maintained at $\pm 2\text{ }^{\circ}\text{C}$ ($\pm 3\text{ }^{\circ}\text{F}$).

For specimens cured at above atmospheric pressure (Class D cement), the curing period starts with the initial application of pressure and temperature.

9.2.3.2 Specimen Cooling

Specimens cured at $60\text{ }^{\circ}\text{C}$ ($140\text{ }^{\circ}\text{F}$) and below shall be removed from the curing bath $45\text{ min} \pm 5\text{ min}$ before the time at which they will be tested. Specimens shall be removed from their molds and cooled by being placed in a water cooling bath maintained at $27\text{ }^{\circ}\text{C} \pm 3\text{ }^{\circ}\text{C}$ ($80\text{ }^{\circ}\text{F} \pm 5\text{ }^{\circ}\text{F}$) for $40\text{ min} \pm 5\text{ min}$. Do not leave the specimen out of water for more than 5 min to avoid dehydration.

For Class D specimens cured at temperatures equal to or greater than $77\text{ }^{\circ}\text{C}$ ($170\text{ }^{\circ}\text{F}$) under pressure, maintain the maximum scheduled temperature and pressure given in Table 8 until $105\text{ min} \pm 5\text{ min}$ prior to the time at which the specimens will be tested; at that point, discontinue heating. During the next $60\text{ min} \pm 5\text{ min}$, decrease the temperature to $77\text{ }^{\circ}\text{C}$ ($170\text{ }^{\circ}\text{F}$) or less, without a reduction in the pressure other than that caused by the reduction in temperature. At $45\text{ min} \pm 5\text{ min}$ prior to the time at which the specimens will be tested, release the remaining

pressure and remove the specimens from the molds. Cool the specimens by transferring them to a water cooling bath maintained at $27\text{ }^{\circ}\text{C} \pm 3\text{ }^{\circ}\text{C}$ ($80\text{ }^{\circ}\text{F} \pm 5\text{ }^{\circ}\text{F}$) for $40\text{ min} \pm 5\text{ min}$. Do not leave specimens out of water for more than 5 min to avoid dehydration.

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

In order to conduct the compressive strength test, the following procedure shall be followed.

- a) Remove specimens from the water bath or the cooling bath that has been maintained at $27\text{ }^{\circ}\text{C} \pm 3\text{ }^{\circ}\text{C}$ ($80\text{ }^{\circ}\text{F} \pm 5\text{ }^{\circ}\text{F}$); wipe each specimen to remove any loose material from the faces that will be in contact with the bearing blocks of the testing machine.
- b) The dimensions of the cube height shall be measured to $\pm 1.6\text{ mm}$ (50-mm mold) or to $\pm 1/16\text{ in.}$ (2-in. mold) for calculation of the cross-sectional area.
- c) If the cube height is less than 47.5 mm (cube prepared using a 50-mm mold) or less than 1.9 in. (cube prepared using a 2-in. mold), the cube should be discarded.
- d) Place the specimen in the testing machine centered below the upper bearing block.
 - 1) The load will be applied to the specimen faces that were in contact with the plane (vertical) surfaces of the mold (not in contact with the base or the cover plates).
 - 2) Prior to the testing of each cube, ascertain that the spherically seated block is free to tilt.
 - 3) Use no cushioning or bedding materials.
- e) Apply the load to the cube-specimen. The rate of loading shall be $18\text{ kN/min} \pm 2\text{ kN/min}$ ($4000\text{ lbf/min} \pm 400\text{ lbf/min}$) for specimens that were cured for 8 hours at $38\text{ }^{\circ}\text{C}$ ($100\text{ }^{\circ}\text{F}$). For all other specimens cured at any other temperature, time, or pressure, the rate of loading shall be $72\text{ kN/min} \pm 7\text{ kN/min}$ ($16,000\text{ lbf/min} \pm 1600\text{ lbf/min}$).

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

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

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 0.1 MPa (10 lbf/in.^2). 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 given in Table 8. A retest shall be made if fewer than two strength values are left for determining the compressive strength at any given period.

Table 8—Compressive Strength Specification Requirements

Cement Class	Schedule Number	Final Curing Temperature ^a °C (°F)	Curing Pressure ^b MPa (lbf/in. ²)	Minimum Compressive Strength at Indicated Curing Period	
				8 h ±15 min ^c MPa (lbf/in. ²)	24 h ±15 min ^c MPa (lbf/in. ²)
A	—	38 (100)	atmospheric	1.4 (200)	12.4 (1800)
B	—	38 (100)	atmospheric	1.4 (200)	10.3 (1500)
C	—	38 (100)	atmospheric	2.1 (300)	13.8 (2000)
D	4S	77 (170)	20.7 (3000)	NR ^d	6.9 (1000)
	6S	110 (230)	20.7 (3000)	3.4 (500)	13.8 (2000)
G, H	—	38 (100)	atmospheric	2.1 (300)	NR
	—	60 (140)	atmospheric	10.3 (1500)	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 lbf/in.²) for schedules 4S and 6S.

^c Time includes cooling time of 40 min ±5 min.

^d NR indicates “no requirement.”

10 Thickening-time Tests

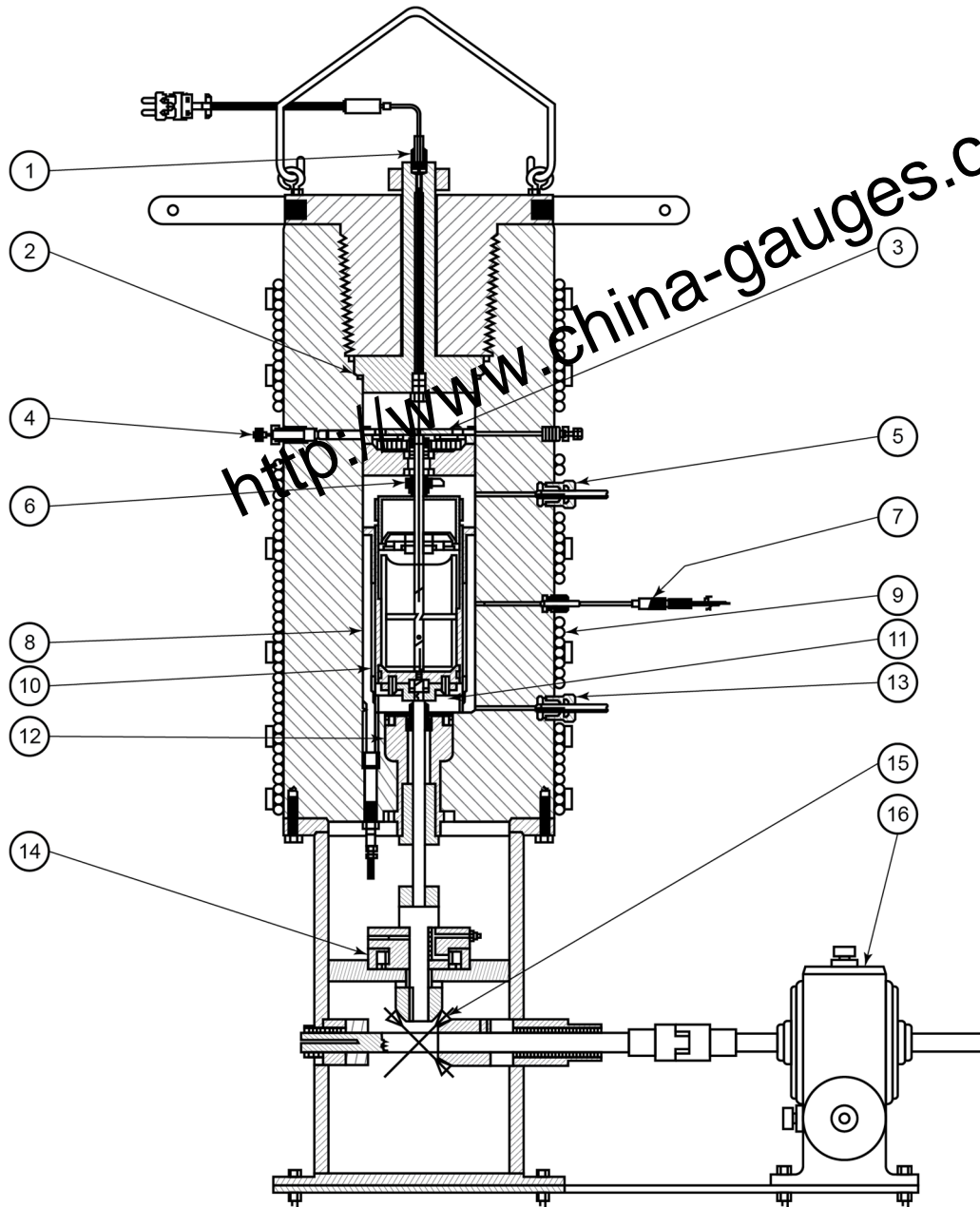
10.1 Apparatus

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 Figures 12 and 13) enclosed in a pressure vessel capable of withstanding the pressures and temperatures shown in Tables 9 through 11.

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 (centerline). The slurry container is rotated at a speed of 150 r/min ±15 r/min. The consistency of the slurry (see 10.2.2) 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 13.

The space between the slurry container and the walls of the pressure vessel shall be completely filled with white mineral or synthetic oil. Synthetic oil is a liquid consisting of chemical compounds artificially made (chemically modified petroleum components or synthesized from other raw materials). White mineral oil is a colorless, odorless liquid consisting of a mixture of hydrocarbons obtained by distillation.

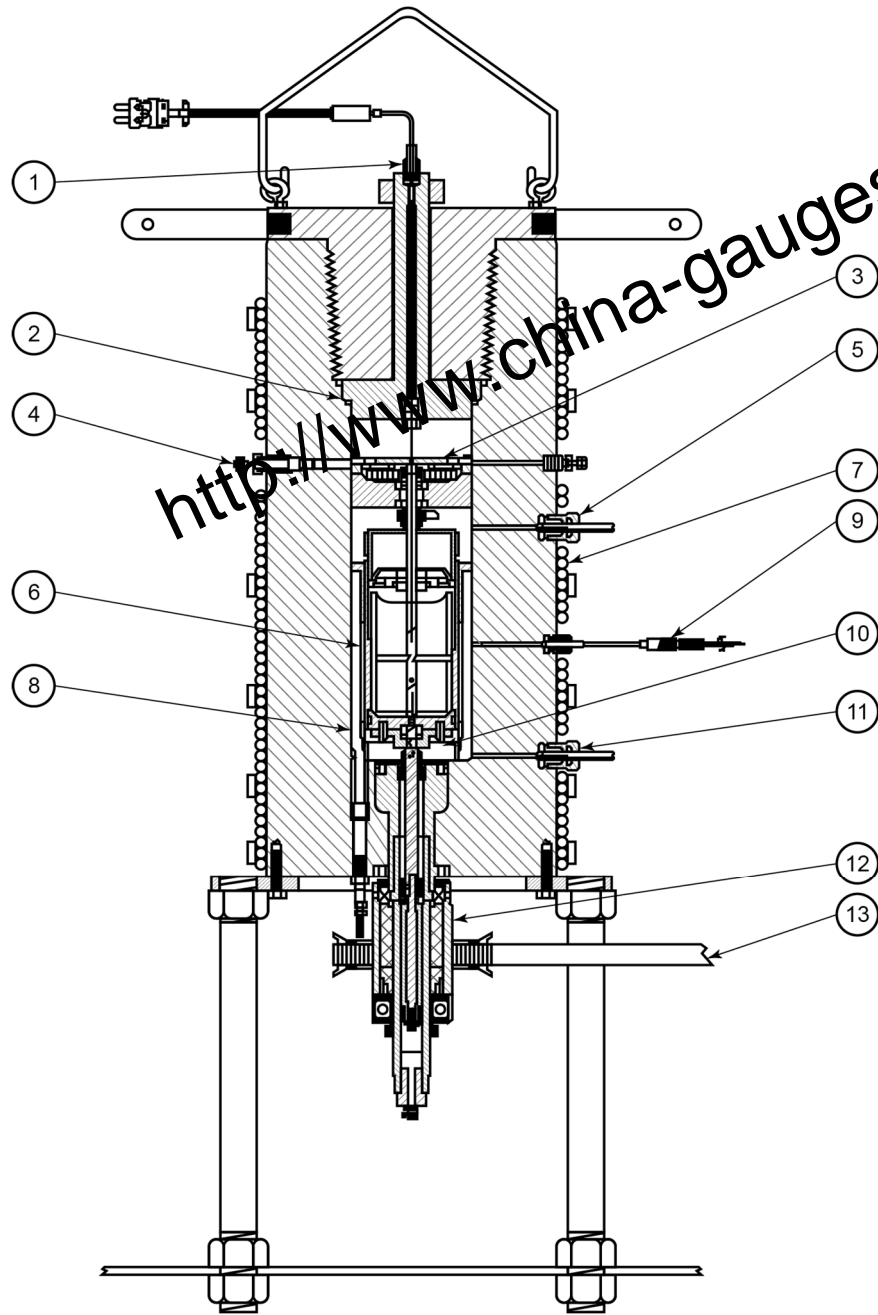
The selected oil shall safely allow the required 3 °C/min heat-up rate.



Key

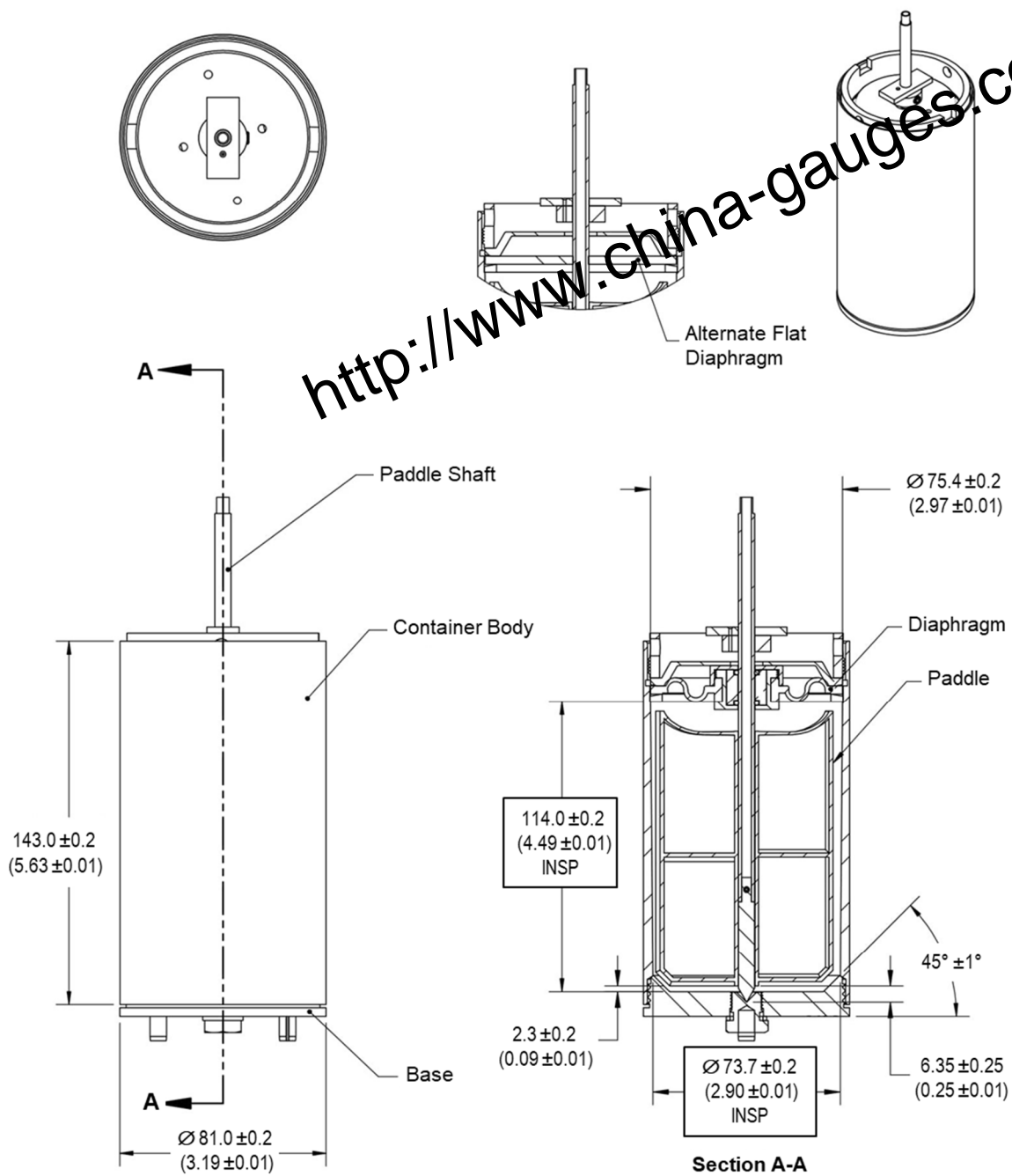
1	slurry-container thermocouple	7	wall thermocouple	13	pressure port
2	sealing ring	8	heater	14	thrust bearing
3	potentiometer mechanism	9	cooling coil	15	miter gears
4	contact pin	10	slurry container	16	gear reducer
5	pressure port	11	container drive table (rotates counterclockwise)		
6	drive bar	12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

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 counterclockwise)		

Figure 9—Typical Magnetic Drive Consistometer for Pressurized Specification Thickening-time Test

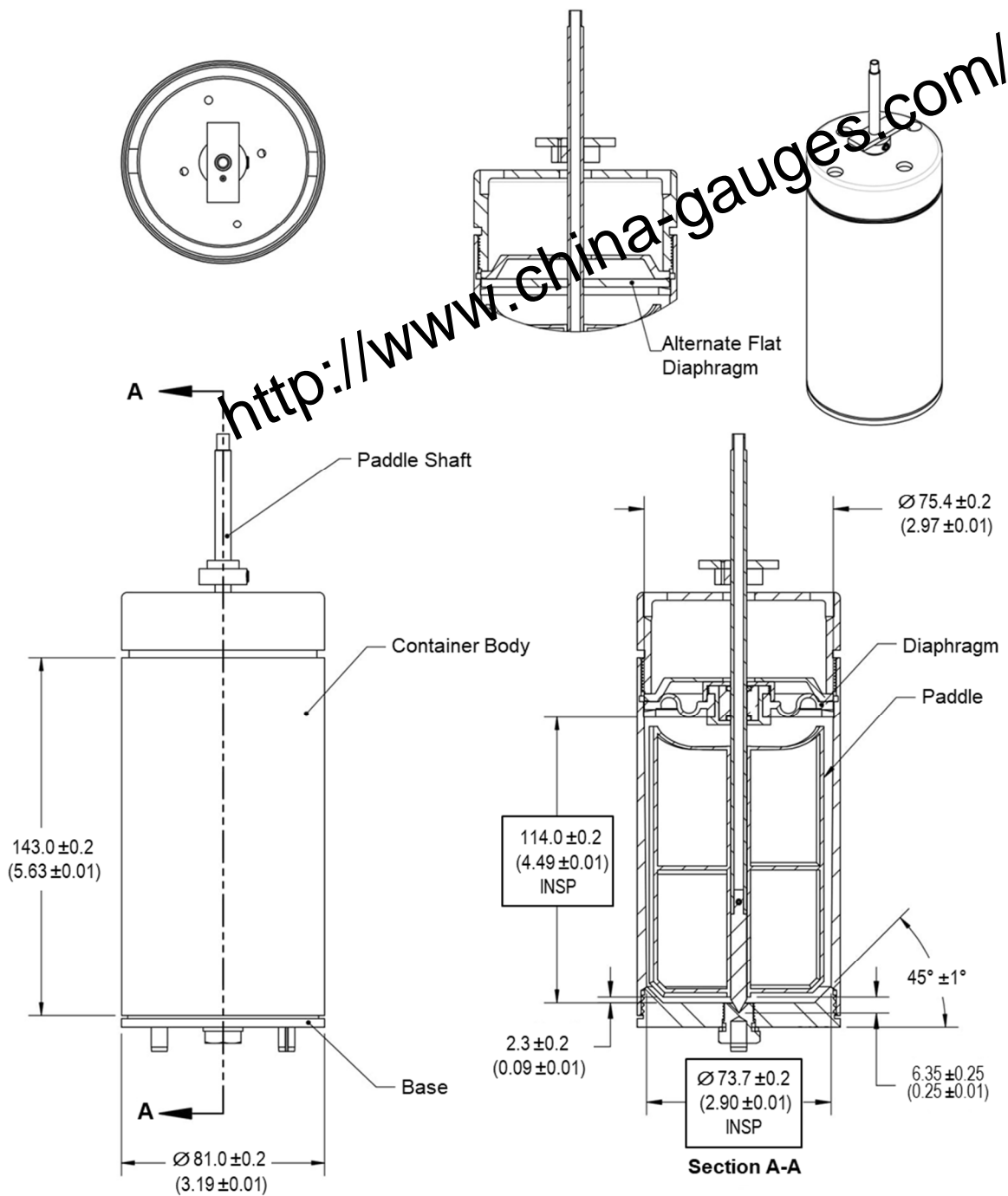
**Key**

INSP inspection

NOTE 1 Dimensions provided are for manufacturing reference only and are in mm (in.) unless otherwise indicated.

NOTE 2 Dimensions tagged as "INSP" for use with evaluating wear. Maximum "INSP" wear: ±1.0 mm (±0.04 in.).

Figure 10—Slurry Container Assembly for a Pressurized Consistometer

**Key**

INSP inspection

NOTE 1 Dimensions provided are for manufacturing reference only and are in mm (in.) unless otherwise indicated.

NOTE 2 Dimensions tagged as "INSP" for use with evaluating wear. Maximum "INSP" wear: ± 1.0 mm (± 0.04 in.).**Figure 11—Slurry Container Assembly for a High-temperature Pressurized Consistometer**

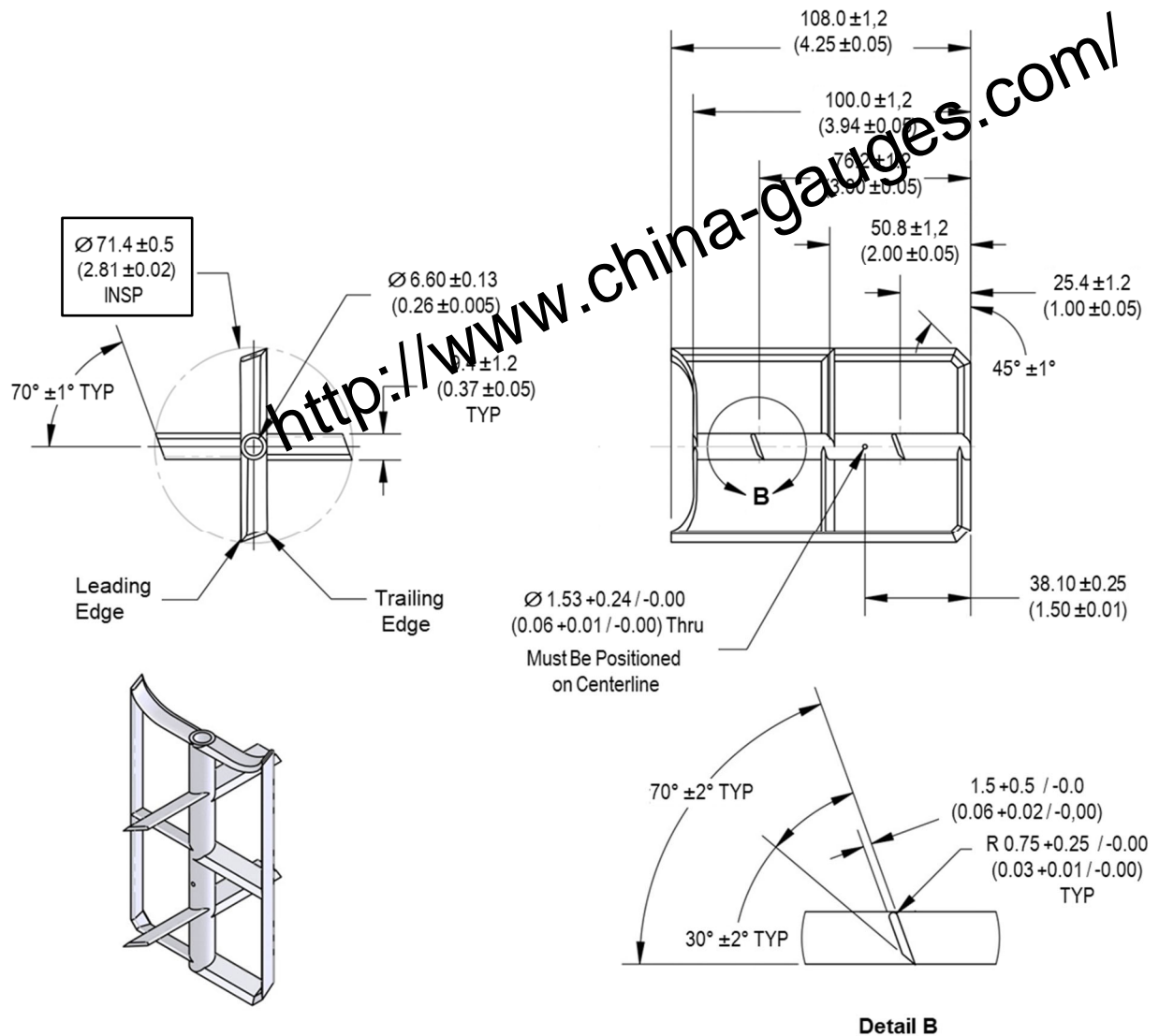
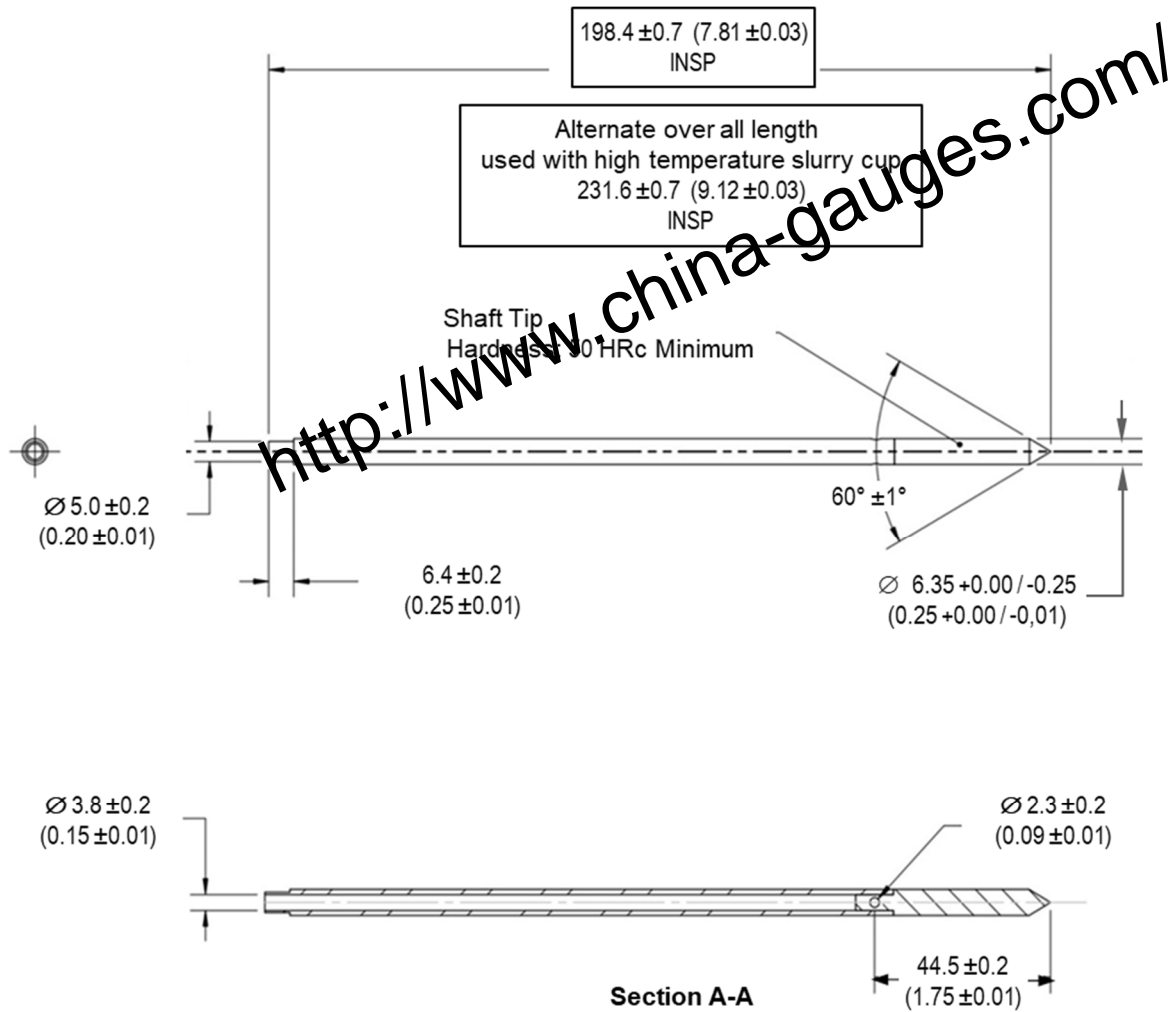


Figure 12—Paddle for a Pressurized Slurry Container



NOTE 1 Dimensions provided are for manufacturing reference only and are in mm (in.) unless otherwise indicated.

NOTE 2 Dimensions tagged as "INSP" for use with evaluating wear. Maximum "INSP" wear: ± 1.0 mm (± 0.04 in.).

Figure 13—Paddle Shaft for a Pressurized Slurry Container

10.2 Calibration

10.2.1 General

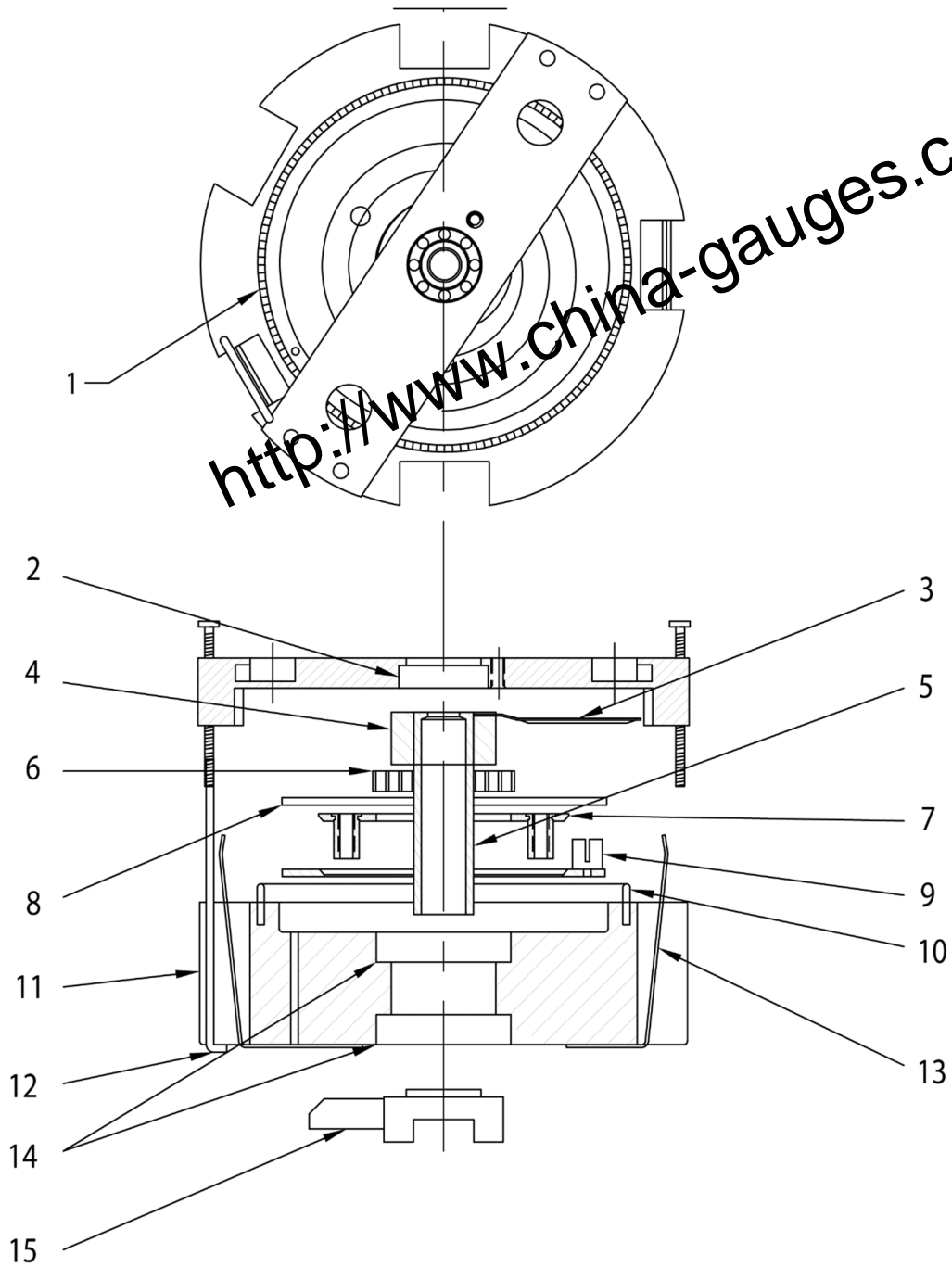
Measurement of the thickening-time of a cement slurry requires calibration and maintenance of operating systems of the pressurized consistometer, including consistency measurement, temperature-measuring systems, temperature controllers, motor speeds, timers, and gauges, as well as the slurry container assembly for wear. All apparatuses are calibrated according to Annex C.

10.2.2 Consistency

Consistency of cement slurry is expressed in Bearden units of consistency, B_c . This value shall be determined by a torque measurement device (see Figure 14 for a typical potentiometer mechanism) and voltage measurement circuit that has been calibrated according to Annex C.

The calibration method as specified in Annex C shall be used, as well as procedures in the potentiometer manufacturer's instructions manual. A weight-loaded device (see Figure C.2 for a typical potentiometer calibrating device) is used to produce a series of torque equivalent values for consistency for calibration. Dedicated potentiometer calibration weights are used to apply torque to the potentiometer spring, using the radius of the potentiometer frame as a lever arm. As dedicated calibration weights are added, the spring is deflected and resulting DC voltage and/or B_c increases.

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Key

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

Figure 14—Typical Potentiometer Mechanism for a Pressurized Consistometer

10.2.3 Temperature-measuring System

The temperature-measuring system shall be accurate to ± 1 °C (± 2 °F); see Annex C. Calibration procedure details as given in Annex D are commonly used.

10.2.4 Motor Speed

The motor shall meet the requirements given in Annex C.

10.2.5 Timers

Timers shall be accurate to within ± 5 seconds per hour. If not within required accuracy, the units shall be adjusted or replaced.

10.2.6 Pressure-measuring System

Calibration shall be conducted against a dead-weight tester or master gauge; see Annex C.

10.2.7 Slurry Container Assembly

A caliper accurate to 0.1 mm (0.005 in.) at minimum is required to verify pressurized consistometer key dimensions (dimensions tagged as "INSP" shown in Figures 10 through 13) for wear of slurry container assembly parts; see Annex C.

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 specification. Grease may be placed only on the threaded surfaces of the slurry container.

10.3.2 Assembling and Filling the Slurry Container

Inspect and assemble the slurry cup, prepare the slurry according to Section 7, and fill the slurry container by the following steps:

- 1) Ensure the cup and threads are clean.
- 2) Inspect the diaphragm and replace it if it is damaged.
- 3) Assemble the paddle shaft assembly and diaphragm with diaphragm support ring and backup plate, and secure them in the cup sleeve with the flange ring (the paddle and inside of the cup sleeve should not be greased, as some greases have been found to affect thickening-time results). Ensure the tapered side of the diaphragm support ring is against the diaphragm for the best seal.
- 4) Assemble the base and center plug (pivot bearing) and make sure the paddle turns freely.
- 5) Remove the base and pivot bearing. Grease the threads of the cup on the base end to ease removal following the test. Grease the threads of the center plug to ease removal.
- 6) With the base end of the slurry container up, prepare the slurry according to Section 7 and fill to the top (or leaving two to three threads showing);
- 7) Strike the cup and remove any air that rises to the top of the slurry.

- 8) Install the base until slurry is extruded through the center hole.
- 9) Install the center plug (pivot bearing) into the container and tighten it until both the plug and base are tight in the container.
- 10) Rinse all cement from the outer surfaces and dry them.
- 11) Recheck the paddle to ensure it turns smoothly.

10.3.3 Initiation of Test

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

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

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

10.3.3.3 Begin the thickening-time test by applying the initial pressure and starting the temperature ramp. No more than 5 min should have elapsed after cessation of mixing of the slurry as specified in 7.2.5 and the beginning of the test.

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 Tables 9, 10, and 11. During the ramp of schedules 4, 5, and 6, the temperature and pressure shall be maintained within ± 3 °C (± 5 °F) and ± 2 MPa (± 300 lbf/in.²/lbf/in.²) of their targets, respectively, versus the appropriate elapsed time. 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 lbf/in.²/lbf/in.²), 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 center of the sample container.

The tip of the thermocouple shall be vertically positioned within the paddle shaft in the slurry cup in such a way that it is between 45 mm (1.77 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 (lbf/in. ² /lbf/in. ²)	Temperature °C (°F)
0	5.2 (750)	27 (80)
2	7.6 (1100)	28 (83)
4	9.7 (1400)	31 (87)
6	11.7 (1700)	32 (90)
8	13.8 (2000)	34 (93)
10	15.9 (2300)	36 (97)
12	17.9 (2600)	38 (100)
14	20.0 (2900)	39 (103)
16	22.1 (3200)	41 (106)
18	24.8 (3600)	43 (110)
20	26.7 (3870)	45 (113)

Table 10—Schedule 5: Specification Thickening-time Test for Classes G and H Cement

Elapsed Time min	Pressure MPa (lbf/in. ² /lbf/in. ²)	Temperature °C (°F)
0	6.9 (1000)	27 (80)
2	9.0 (1300)	28 (83)
4	11.1 (1600)	30 (86)
6	13.1 (1900)	32 (90)
8	15.2 (2200)	34 (93)
10	17.3 (2500)	36 (96)
12	19.3 (2800)	37 (99)
14	21.4 (3100)	39 (102)
16	23.4 (3400)	41 (106)
18	25.5 (3700)	43 (109)
20	27.6 (4000)	44 (112)
22	29.6 (4300)	46 (115)
24	31.7 (4600)	48 (119)
26	33.8 (4900)	50 (122)
28	35.6 (5160)	52 (125)

Table 11—Schedule 6: Specification Thickening-time Test for Class D Cement

Elapsed Time min	Pressure MPa (lbf/in. ² /lbf/in. ²)	Temperature °C (°F)
0	8.6 (1250)	27 (80)
2	11.0 (1600)	29 (84)
4	13.1 (1900)	31 (88)
6	15.9 (2300)	33 (91)
8	17.8 (2500)	34 (94)
10	20.7 (3000)	37 (98)
12	22.8 (3300)	38 (101)
14	25.5 (3700)	41 (105)
16	27.6 (4000)	42 (108)
18	30.3 (4400)	44 (112)
20	32.4 (4700)	47 (116)
22	35.2 (5100)	48 (119)
24	37.2 (5400)	51 (123)
26	39.3 (5700)	52 (126)
28	42.1 (6100)	54 (130)
30	44.1 (6400)	56 (133)
32	46.9 (6800)	58 (137)
34	49.0 (7100)	60 (140)
36	51.6 (7480)	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 specification. The acceptance requirement for the thickening time is given in Table 12.

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."

11 Marking

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. The following information shall be marked on or made available with each shipment of well cement:

- a) manufacturer's name;
- b) class and sulfate-resistance grade of cement;
- c) net mass.

Licensed API cement manufacturers wishing to mark their products with the API Monogram shall follow marking requirements according to Annex A.

12 Packing

Well cement shall be furnished in bulk or in sacks.

Each sack shall contain a specified net mass of $\pm 2\%$. 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 shall be resistant to moisture, resistant to damage during handling, and easy to cut during transfer to bulk facilities.

NOTE Cement sacks frequently consist of up to six paper layers, with a minimum areic mass of 70 g/m² (0.014 lbm/ft²), each with up to two polyethylene or polypropylene layers with an areic mass of 15 g/m² (0.003 lbm/ft²) to 24 g/m² (0.005 lbm/ft²) each included between the first and fifth paper layers. Up to two asphalt or bitumen layers are included to further improve resistance to damage.

Flexible bulk cement containers should provide an acceptable tensile strength (safety factor of 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 specification for use in well cementing shall meet all the requirements for non-treated bentonite in accordance with API Specification 10A.

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Annex A

The information in this annex has been intentionally removed.

See API Specification Q1, Annex A or the API website for information pertaining to the API Monogram Program and use of the API Monogram on applicable products.

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Annex B (normative)

Composite Well Cements

B.1 General

Composite well cements consist of Portland cement and additional constituent(s) such as silica, fly ash (coal ash), expanded shale, clay, or any other natural, processed, or man-made materials that are either blended or interground. Such composite well cements have been manufactured by cement manufacturers and others, and have a long and successful history of use for cementing oil and gas wells. Such composite well cements are appropriate for cementing oil or gas wells, used by themselves or when blended with other materials to enhance their properties to meet the well conditions, and all appropriate design and regulatory requirements for mixing, placement, and performance in the well.

This annex defines specifications for certain composite well cements that are manufactured by facilities having a fully implemented, recognized quality management system in place, but does not preclude the use of other composite well cements as described above for use in wells when such composite well cements meet the requirements of the well.

The physical and chemical specifications for composite well cements described in this annex may also be used for reference testing of field blended composite well cements before tailoring them to the specific well cement conditions.

B.2 Specification, Chemical, and Physical Requirements

B.2.1 Classes and Grades

B.2.1.1 General

Composite well cements according to this annex shall be specified using Classes K and L. Class K is available in different grades: ordinary (O), moderate sulfate-resistant (MSR), and high sulfate-resistant (HSR).

Composite well cement by this annex shall be manufactured at facilities having a fully implemented, recognized quality management system in place such as, but not limited to, API Specification Q1.

Composite well cement is obtained by intergrinding Portland cement clinker and one or more forms of CaSO_4 with composite constituents as specified in B.2.2, or by subsequent blending of separately produced Portland cement with separately processed composite constituents as specified in B.2.2.

Composite well cement that has been manufactured and supplied in accordance with this annex 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 API Specification 10A be based on such field conditions.

Processing additives, set modifying agents, or chemical additives to reduce chromium (VI) are permitted, provided that such additives do not prevent the composite well cement from performing its intended functions.

B.2.1.2 Class K

Class K composite well cement is obtained by intergrinding Portland cement clinker and one or more forms of CaSO_4 with silica as specified in B.2.2.1, or by subsequent blending of separately produced Portland cement with separately ground silica flour as specified in B.2.2.1. At the manufacturer's discretion, another constituent

(additive) as specified in B.2.2 may be interground or interblended. When added, such other constituents shall be reported to the final user by content and type.

This product is intended for use as a basic well cement and is available in O, MSR, and HSR grades, depending on the C_3A content of the Portland cement clinker being interground or Portland cement being blended to produce the product.

B.2.1.3 Class L

Class L composite well cement is obtained by intergrinding Portland cement clinker and one or more forms of $CaSO_4$ with fly ash as specified in B.2.2.3; with other pozzolanic material as specified in B.2.2.2; by blending separately produced Portland cement with separately processed fly ash as specified in B.2.2.3; or with other pozzolanic material as specified in B.2.2.2. At the manufacturer's discretion, free-fluid at the manufacturer's design water ratio may be adjusted by using bentonite or other materials that provide suitable particle-suspending properties that, when added to the final product, shall be reported to the final user by content and type.

This product is intended for use under conditions when a low-density cement slurry is desired.

B.2.2 Composite Constituents (Other than Portland Cement or Clinker)

B.2.2.1 Silica

Silica is synonymous with silicon dioxide (SiO_2). Silica exists in different forms that can be crystalline (most commonly as quartz) as well as non-crystalline (amorphous).

NOTE 1 A special form of amorphous silica is silica fume specified, e.g. in EN 197-1.

NOTE 2 Amorphous silica is not considered in the context of this specification.

The term silica in the context of this specification refers only to crystalline silica with a silicon dioxide content of at least 90 %, the remainder being minerals that naturally occur in conjunction with silicon dioxide. Silica flour or processed silica refers to crystalline silica being produced to a fineness that makes it suitable as a constituent for composite well cement Class K according to this annex.

B.2.2.2 Pozzolan Constituents

B.2.2.2.1 General

Pozzolan materials are described as siliceous (mainly in form of reactive silicon dioxide, SiO_2) or siliceous and aluminous (mainly Al_2O_3) materials, and iron oxide (Fe_2O_3), with small amounts of oxides of other elements that themselves possess little or no cementitious value but that will, in finely divided form and in the presence of moisture, chemically react with calcium hydroxide [$Ca(OH)_2$] at ordinary temperatures to form compounds possessing cementitious properties. These compounds are similar to those that are formed in the hardening of Portland cement.

Before use for manufacturing of API composite well cements according to this annex, pozzolan materials shall be correctly prepared, e.g. selected, homogenized, dried, or heat-treated and comminuted, depending on their state of production or delivery.

Although some fly ashes have pozzolan properties, they are specified separately (see B.2.2.3).

B.2.2.2.2 Natural Pozzolanic Constituents

Natural pozzolanic constituents are naturally occurring materials of volcanic origin, such as volcanic ashes, tuffs, pumices, etc., or sedimentary rocks with suitable chemical and mineralogical composition, and shall conform to the requirements given in B.2.2.2.1.

B.2.2.2.3 Natural Calcined Pozzolanic Constituents

Natural calcined pozzolanic constituents are materials of volcanic origin, clays, shales, or sedimentary rocks that are thermally treated to improve or activate their pozzolanic reactivity and shall conform to the requirements given in B.2.2.2.1.

B.2.2.3 Fly Ashes

Fly ash is the finely divided residue that results from the combustion of ground or powdered coal. It is obtained by electrostatic or mechanical separation of the particles from furnace flue gases.

Fly ash is called artificial pozzolan and is the pozzolan most commonly used in well cement formulations.

According to ASTM C618-08, a Class C fly ash is obtained by burning lignite or subbituminous coals. Class C fly ash has pozzolanic properties and also has some cementitious properties. Class C fly ash may have lime content higher than 10 %.

According to ASTM C618-08, a Class F fly ash is produced as the combustion residue from anthracite or bituminous coals. Class F fly ash has pozzolanic properties.

B.2.3 Chemical Requirements

Composite well cements shall conform to the respective chemical requirements as stipulated in the following clauses.

B.2.3.1 Class K

The manufacturer of Class K is required to calculate and make available to the end user the CaO-to-SiO₂ mole ratio of the finished product. This determines the capability of the product to prevent or lessen strength retrogression at elevated well temperatures. Usually, such capability is given at a ratio lower than 1.25. For blended Class K composite well cements, this is usually accomplished by adding high-purity silica in the magnitude of 35 % to 40 % by weight of cement.

Chemical analysis of hydraulic cements shall be carried out as shown in ASTM C114 or EN 196-2, e.g. by X-ray fluorescence analysis. The CaO-to-SiO₂ mole ratio is calculated using the following formula:

$$\frac{CaO}{SiO_2} \text{ mole ratio} = \frac{m_{CaO} / M_{CaO}}{m_{SiO_2} / M_{SiO_2}} \quad (B.1)$$

where

m_{CaO} is the mass fraction of CaO in a sample of composite well cement, expressed in grams;

m_{SiO_2} is the mass fraction of SiO₂ in a sample of composite well cement, expressed in grams;

M_{CaO} is the molar mass of calcium oxide (56.08 g/mol);

M_{SiO_2} is the molar mass of silicon dioxide (60.09 g/mol).

NOTE In cement chemistry terms, CaO is abbreviated as C and SiO₂ is abbreviated as S. Therefore, CaO/SiO₂ often expressed as C/S mole ratio.

EXAMPLE CaO/SiO₂ mole ratio calculation in a sample of composite well cement of 100 g:

$m_{CaO} = 47.43$ g, here in 100 g of a composite sample (= 47.43 %)

$m_{SiO} = 41.51$ g, here in 100 g of a composite sample (= 41.51 %)

$$\frac{CaO}{SiO_2} \text{ mole ratio} = \frac{47.43 \text{ g} / 77.08 \text{ g/mol}}{41.51 \text{ g} / 60.09 \text{ g/mol}} = \frac{0.615 \text{ mol}}{0.691 \text{ mol}} = 1.22$$

Class K composite well cement is further specified by sulfate-resistance grades ordinary (O), moderate sulfate-resistant (MSR), and high sulfate-resistant (HSR). The sulfate-resistance grade of the finished Class K composite well cement depends on the percentage mass fraction of C₃A of the Portland cement clinker or the Portland cement—the first in case the composite well cement is manufactured by intergrinding the composite constituents, and the latter in case the composite well cement is manufactured by blending Portland cement with the composite constituents. The assignment of sulfate-resistance grades of Class K to the C₃A content of Class K clinker or cement—whatever production method is chosen—is the same as for Classes A to H. For calculation of percentage mass fraction of C₃A of clinker and cement, the formulas as specified in Section 4, Table 1 apply.

NOTE Generally, the formulas of Section 4, Table 1 apply only to the finished cement as an interground blend of Portland cement clinker and gypsum. When applying them to clinker only, the error due to the absence of gypsum can be considered as marginal (negligible) for the purpose of this annex.

B.2.3.2 Class L

The manufacturer of Class L shall calculate and make available to the end user the CaO-to-SiO₂ mole ratio of the finished product.

No further chemical requirements are set for Class L composite well cement.

B.2.4 Physical and Performance Requirements

Composite well cements shall conform to the respective physical and performance requirements given in Table B.1 and in accordance with Section 8 through 10. When reporting results of specification testing, the manufacturer shall always state them in conjunction with the applied mix water percentage at which these tests were performed.

The manufacturer of Class L shall also state the slurry density⁴ at the design mix water percentage and at which mix water percentage the performance tests were performed.

Users of composite well cement may mix at different mix water percentages and with other modifying additives to meet the performance requirements of the well in which it is to be applied.

⁴ For slurry density preferred method of determination, see API 10B-2.

Table B.1—Physical and Performance Requirements for Composite Well Cements

Composite Well Cement Class				K	L
Mix water as per manufacturer's requirements				a	a
Free-fluid content, maximum, percent (Section 8)				5.9	5.9 ^c
Compressive Strength Test Curing time: 8 h	Schedule Number	Final Curing Temperature °C (°F)	Curing Pressure	Minimum Compressive Strength MPa (lbf/in. ² /lbf/in. ²)	
(Section 9)	NA ^d	60 (140)	atmospheric	3.5 (500)	NA
Compressive Strength Test Curing time: 24 h	Schedule Number	Final Curing Temperature °C (°F)	Curing Pressure	Minimum Compressive Strength MPa (lbf/in. ² /lbf/in. ²)	
(Section 9)	NA	38 (100)	atmospheric	NA	1.7 (250)
Thickening-time Test	Specification Test Schedule Number	Maximum Consistency (15 to 30 minutes stirring period) B _c ^e		Thickening Time (minimum/maximum) minutes	
(Section 10)	4 (Table 9)	30		NA	90/NA
(Section 10)	5 (Table 10)	30		70/140	NA
<p>^a The manufacturer shall indicate on certificates of conformance the design mix water percentage for the composite well cement and which performance tests are performed.</p> <p>^b Mix water percentage by slurry density desired. The manufacturer shall indicate on certificates of conformance the slurry density at the design mix water percentage and at which performance tests are performed.</p> <p>^c Free-fluid may be adjusted by use of bentonite (see B.2.1.3) or other water-adsorbing materials.</p> <p>^d NA indicates "not applicable."</p> <p>^e Bearden units of consistency, B_c, obtained on a pressurized consistometer as defined in Section 10 and calibrated in accordance with Annex C.</p>					

Annex C (normative)

Calibration and Verification of Well Cement Testing Equipment

C.1 Overview

This annex defines the requirements for calibration and verification of measurements by instruments used in testing cement for oil and gas well applications according to the procedures prescribed in this document. Henceforth, the term “calibration” is used to mean either verification and/ or calibration according to the requirements of this document. Procedures will depend on the specific instrument used for the calibration. Manufacturer’s instructions should be followed for the calibration. Calibrations shall be conducted using instruments and devices traceable to national standards.

NOTE Calibration refers to the verification of an instrument or to the adjustments made to the instrument if it is out of calibration. If a verification shows the instrument to be in calibration, the date and verification data will be the calibration. In this calibration section, the word “verification” pertains to instruments that cannot be changed if they are inaccurate, and the word “calibrate” pertains to instruments that, if they are out of calibration as seen by the “verification” of the instrument, may be adjusted to get them into calibration.

C.2 General

The following sections set the requirements for interval and accuracy of calibration of devices used in a laboratory conducting tests according to this API specification. The requirements by equipment type are given in Table C.1 and the frequency and allowable tolerances are given in Table C.2.

Volumetric glassware (graduated cylinders, conical flasks, and similar equipment) calibration is generally performed by the glassware supplier, and may be part of the purchase specification. Glassware users should obtain documented evidence, where deemed important, of glassware calibration from the supplier. Calibration may be checked gravimetrically before the first use. Periodic recalibration is not required. As specified in C.3.14, a procedure for graduated cylinders applicable to any volumetric glassware is detailed.

Dimensions of apparatuses such as cube molds and consistometer parts for wear may be verified using a caliper accurate to at least 0.1 mm (0.005 in.).

Table C.1—Equipment Calibration Requirements

Measurement Instrument	Balance	Mixing Device	HPHT Consistometer	Atmospheric Pressure Consistometer	Handheld Timers	Handheld Thermometers	Atmospheric and Pressurized Water Baths	Cube Molds	Level Frame	Weights	Chart Recorder (where used)
Mass	V/C	V ^a								V	
Timer		V	V	V							V/C (speed)
Rotation		V	V/C	V/C							
Temperature			V/C	V/C		V/C	V/C				V/C
Dimensions			V					V			
Consistency			V/C								V/C
Pressure gauge			V/C				V/C				V/C
Auxiliary display			V	V							
Indication									V/C		
NOTE V/C indicates the verification of calibration may be performed first. If the verification values are acceptable at the time it is verified, the verification will serve as a calibration. If it is not in calibration tolerance, the calibration needs to be performed.											
^a Blender blade mass											

Table C.2—Verification and Calibration of Well Cement Testing Equipment

Equipment and Component/Function	Verification/Calibration	Check Points	Tolerance	Frequency
Balance (each range on multi-range balance)	Calibration	Three points spread evenly across range	± 0.1 % of reading for measurements made at 10 g or greater, up to the full scale of the balance 0.01 g of reading for measurement made less than 10 g	Annually
Weight sets	Verification	Each piece	0.1 % of nominal mass value ± 0.01 g of the nominal mass for 10 g or less.	Routine usage: annually Reference weight sets: once every 2 years (or 5 years; see C.3.2)
Potentiometer calibration weight set	Verification	Each piece Note: Include holding support washer	0.1 % of mass value	Annually
Timers	Verification	12 min	Within 5 s in one hour (minimum verification period: within 1 s in 12 min)	Annually
Chart speed	Calibration	One hour	± 2 min in one hour	Annually
Mixing device (1 L)	Verification	4000 r/min and 12,000 r/min	± 250 r/min @ 4000 r/min and ± 250 r/min @ 12,000 r/min	Annually
Mixing device blade mass	Verification	Initially when replaced	Maximum 10 % loss	Prior each production run tested or every 30 days, whichever is greater
Blender timing device	Verification	35 s	Within 1 s in 35 s	Annually
Temperature controlling systems and displays	Calibration	Three points spanning the range	Maximum of ± 1 °C (± 2 °F)	Quarterly
Thermocouple	Verification	Three points spanning the range	Maximum of ± 1 °C (± 2 °F)	Quarterly
Handheld thermometer or digital thermometer	Calibration	Three points spanning the range	Maximum of ± 1 °C (± 2 °F)	Quarterly

Equipment and Component/Function	Verification/Calibration	Check Points	Tolerance	Frequency
Consistometer container speed (or paddle)	Verification	At 150 r/min	± 15 r/min at 150 r/min	Quarterly
Slurry container	Verification	Figure 10 or Figure 11 (INSP [®] dimensions)	Maximum wear ± 1.0 mm (± 0.04 in.)	Annually
Slurry container paddle and shaft	Verification	Figures 12 and 13 (INSP [®] dimensions)	Maximum wear ± 1.0 mm (± 0.04 in.)	Annually and whenever parts are replaced
Consistency measuring devices	Calibration	Table C.4	$\pm 5 B_c$ or ± 0.5 V DC, as appropriate	Monthly and whenever it is adjusted, or parts replaced
High range pressure gauge or pressure measuring device ($> 17,000$ kPa or 2500 lbf/in. ²) ^a	Calibration	17MPa, 34 MPa, and 52 MPa (2500 lbf/in. ² , 5000 lbf/in. ² and 7500 lbf/in. ²) or 25 %, 50 %, 75 % of full scale or maximum user-defined working pressure	Using deadweight tester or master gauge; ± 1 % of full range or minimum gauge increment, whichever is greater	Annually or when pressure transducer is replaced
Low range pressure gauge or pressure measuring device— (up to 17,000 kPa or 2500 lbf/in. ²)	Calibration	3500 kPa (500 lbf/in. ²) at minimum and 10,500 kPa (1500 lbf/in. ²) or at the maximum allowable working pressure or the user defined working pressure	± 300 kPa (± 50 lbf/in. ²)	Annually or when pressure transducer is replaced
Load frame	Calibration	At 9.0 kN (2000 lbf) and at 25 %, 50 % and 75 % of the maximum load	± 2 % of the indicated force or one minimum instrument scale division, whichever is greater	Annually
Cube molds (in use)	Verification	Cubes dimensions	50-mm molds: distance between opposite side ± 0.5 mm, height of compartment $+0.25$ mm/ -0.38 mm 2-in molds: distance between opposite side ± 0.02 in., height of compartment: $+0.01$ in./ -0.015 in.	Once every two years
Data acquisition devices	Verification	Same as T and P	Adjust or prepare calibration table	With sensor recorded

Equipment and Component/Function	Verification/Calibration	Check Points	Tolerance	Frequency
Volumetric glassware ^b ,	NR ^c			Before first use
Graduated cylinder ^b ,	Verification/calibration	5 points	If required, prepare calibration table	Before first use
<p>NOTE Calibration of air pressure gauges is not required.</p> <p>^a Check points at 17MPa, 34 MPa, and 52 MPa (2500 lbf/in.², 5000 lbf/in.² and 7500 lbf/in.²) are the minimum requirements for high range pressure gauges or pressure measuring devices used for test conditions as specified in API 10A.</p> <p>^b Volumetric glassware (including graduated cylinders) calibration is done by supplier and may be part of the purchase specification. Calibration may be checked gravimetrically before the first use. Periodic recalibration is not required.</p> <p>^c Not required</p>				

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The individual performing the verification/calibration shall possess adequate skill and training to properly perform the calibration according to the procedures specified for the device(s) being used.

Records of such calibration shall be maintained in a manner where the data or record cannot be altered after certification. The records shall be maintained at least as long as the data from tests performed during the calibration period on the specific instrument are maintained.

Verification and calibration records shall contain, as appropriate:

- model name/number, serial number, and local identification number of the instrument;
- name, model number, and serial number of instrument used for the calibration;
- certification number, date of certification, and date of next calibration against national standard is due for the calibration instrument;
- calibration inputs from the calibration instrument and the initial (before calibration) and final (calibrated) indications from instrument being calibrated;
- indication that instrument passed or failed each calibration point;
- condition found/condition left (e.g. out of or in calibration);
- adjustments made and results after adjustment;
- date of calibration;
- name and signature of calibration technician;
- if calibration is by technician from an outside service, name of service, location, and phone number.

Some instruments have multiple cells or testing systems within one unit (e.g. dual-cell consistometers). In such cases, each cell or testing system shall be verified and/or calibrated (if not in calibration) individually, with a separate certificate showing a unique identifier for each testing system. Components that are common to all of the cells or testing systems need be calibrated only once, but the calibration data shall be indicated on each certificate.

When the device is being used for a test, and a verification or calibration becomes due, the verification or calibration remains valid for the duration of that test and the instrument should be calibrated at the end of that test.

C.3 Requirements

C.3.1 Balances

Balances shall be verified and/or calibrated (if not in calibration) no less frequently than annually. Balances shall be accurate to ± 0.1 % of reading for measurements made at 10 g or greater, up to the full scale of the balance. Balances shall be accurate to ± 0.01 g of reading for measurement made less than 10 g. Balances shall have two-decimal-place precision at a minimum. Each range of a dual range balance shall be calibrated in this fashion.

C.3.2 Weight Sets

Weight sets should be verified against reference weights traceable to a national standard. Weight sets to be verified include, but are not limited to, those used daily (routine weighing), and those used to calibrate consistometer potentiometers (dedicated potentiometer calibration weight set) or their equivalent.

Weight sets should be verified and/or calibrated no less frequently than once annually. Weights should conform within $\pm 0.1\%$ of the nominal weight except for weights with a mass equal or less than 10 g (see Table C.3). Weights of 10 g and less should conform within ± 0.01 g of the nominal mass. If weights do not conform to the specification, they should be discarded or sent to the manufacturer for recertification.

NOTE 1 Dedicated potentiometer calibration weights include the holding support washer.

NOTE 2 Annual verification should be required for weight sets used daily for routine weighing. For reference weight sets used for annual verification, weights should be recertified against certified master weights every five years providing weights sets are kept in a padded dust- and moisture-proof holder and used with tongs with plastic tips. If not the case, reference weight sets should be recertified every two years.

Table C.3—Permissible Variation in Mass of Reference Weights

Mass g	Permissible Variation g
1000	± 1.00
500	± 0.50
300	± 0.30
200	± 0.20
100	± 0.10
50	± 0.05
≤ 10	± 0.01

C.3.3 Mixing Device (Blender)

C.3.3.1 Mixing Device Speed

Mixing device display of rotational speed shall be verified and/or calibrated (if not in calibration) no less frequently than annually. The tolerance measured by a calibrated tachometer shall not deviate more than the limits given in Table C.2: ± 250 r/min at either 4000 r/min low speed and at 12,000 r/min high speed.

However, when mixing a cement slurry, the one liter [1 L (1 qt)] blender shall operate at a low-speed range of 3750 r/min to 4250 r/min (4000 r/min \pm 250 r/min) and high-speed range of 11,500 r/min to 12,500 r/min (12,000 r/min \pm 500 r/min). The indicated speed shall not deviate by more than these limits at either the low speed or the high speed from the true speed observed using a calibrated tachometer. If the mixing device is unable to control within the required range, the blender motor or controller shall be repaired or replaced, as appropriate.

NOTE The ability of the controller to maintain speeds within the required range during mixing a cement slurry is not a calibration issue. Typical motors available for the 1 L (1 quart) mixer are 3.6 A/0.04HP and the 7 A/0.7 HP; typically, the 3.6 A motor will not maintain the tolerance, especially with viscous slurry.

C.3.3.2 Blender Blade

Initial blade weight and dimensions may vary due to the manufacturing process. The weight loss of the blender blade (blade only, not the blade assembly) shall be verified to be no more than 10 % when it is changed. The mixing blade shall be weighed initially and prior to each production run tested or every 30 days, whichever is greater. Replace with an unused blade if observed blade deformation has occurred or after a blade has lost 10 % of its mass.

Users will develop procedures to ensure that the blade is not used with $\geq 10\%$ weight loss compared to its unused (out of the package) weight, and will develop a system to confirm weight loss of the blade when it is changed. This system will include a method of identifying the blender container in which the blade is installed. A log of blade weights and dates changed will be maintained with the calibration records. A practical method of judging when a blade is approaching the limit in weight loss is by comparing the blade in the blender to a blade with known weight loss (see Figure C.1). The appearance of wear for a hardened blade may be different than shown in Figure C.1 for a non-hardened blade.

C.3.3.3 Blender Timing Controller

The blender timing controller device shall be verified against a calibrated timer (stopwatch) (see C.3.4), for 12,000 r/min high speed mixing time at $35 \text{ s} \pm 1 \text{ s}$.

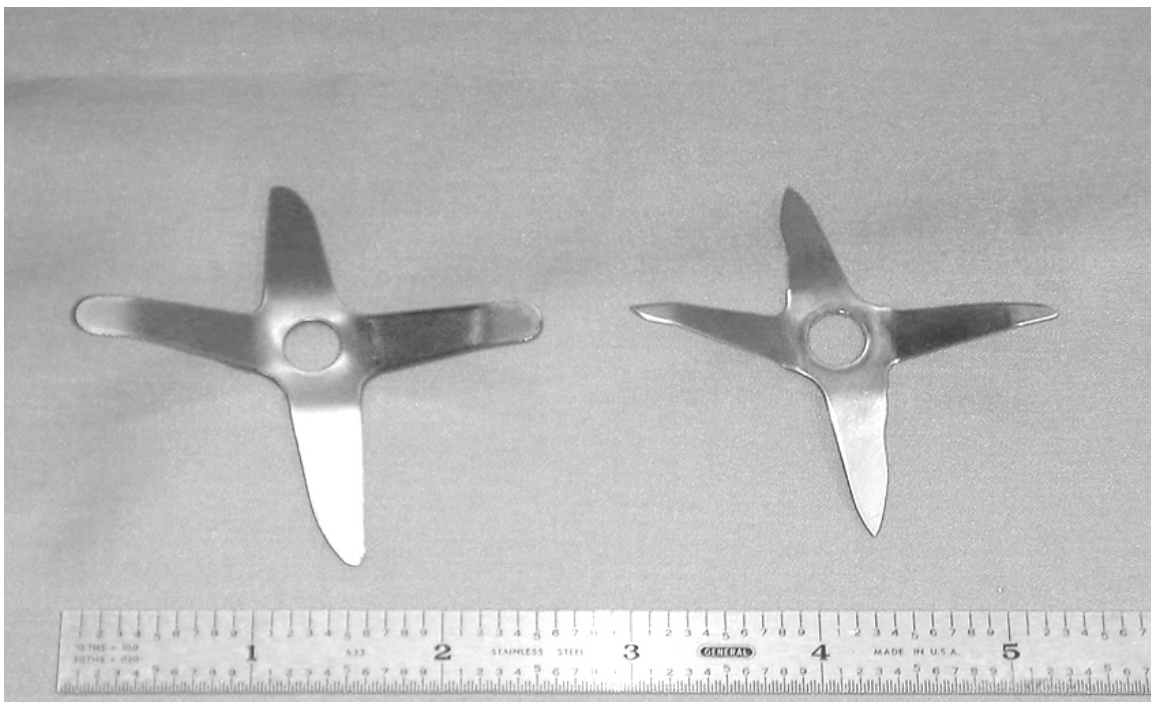


Figure C.1—Worn Blade (right) Compared to a New One (left)

C.3.4 Timers

Timers on all instruments shall be verified/and or calibrated (if not in calibration) no less frequently than annually. This includes timers on consistometers, handheld timers, and any other timer, whether as part of an instrument or separate. Timers shall be accurate to within ± 5 seconds per hour and shall be verified over a period of no less than 12 minutes. Calibration may use the time signal from the NIST or similar websites and radio stations. If not within required accuracy, the units shall be adjusted or replaced.

NOTE Blender timing devices (see C3.3.3) are not covered by this section.

C.3.5 Recorders

Charts used for recording thickening times or other data shall be checked at least annually to ensure they vary by no more than 2 min/h. This can be done by running the chart, causing a deflection of the temperature or pressure trace at the beginning and end of one hour according to a calibrated timer, and measuring the deviation on the chart. The chart on which this recording is made shall be maintained with the calibration record for the instrument (as applicable: temperature, pressure, and/or consistency).

C.3.6 Temperature Devices

C.3.6.1 Temperature-measuring and -controlling devices shall be verified and/or calibrated (if not in calibration) no less frequently than quarterly. This includes thermometers, thermocouples, and temperature controllers on consistometers, curing chambers, and those that are used separate from or are not an integral part of the instrument. Measurements shall be made at no less than three temperatures spanning the manufacturer- or user-defined operating range of the equipment on or with which the device is used. The lowest temperature calibrated shall be no more than 5 °C (10 °F) above the minimum and the highest temperature calibrated shall be no more than 5 °C (10 °F) below the maximum of the user-defined operating range of the instrument.

C.3.6.2 Thermometers or thermocouples shall be verified for accuracy by use of a known temperature source (such as a heat block) with a thermometer or thermocouple that is certified and traceable to a national standardization body. Accuracy shall be within ± 1 °C (± 2 °F) or for special J type thermocouple accuracy shall be ± 1.1 °C (± 2 °F). If the thermometer or thermocouple error is greater than that, it shall be replaced by one meeting the accuracy requirements. Thermocouples mounted in the cylinder wall of a consistometer are exempt from calibration, but during the test, adjustments should be made to the controlling temperature when control is from the oil thermocouple, so the final temperature of the slurry is within the required test temperature.

C.3.6.3 Temperature Control System

Temperature-controlling systems consist of:

- the sensing device (normally a thermocouple);
- display (may be integral to controller or may be separate);
- the controller that receives input from the sensing device and, in turn, provides output to the heating or cooling medium. The output circuit is not calibrated in this procedure; and
- all wiring, connectors, etc. between the sensor and the controller.

The temperature controller may be verified and/or calibrated while connected to its thermocouple placed in a calibrated heat source or using a cold-junction compensated input device in place of the thermocouple. If the controller is calibrated using a cold-junction compensated input device, the thermocouple shall be calibrated independently. The thermometer or thermocouple that is used for the verification of calibration tolerance shall be certified and traceable to a national standardization body. The controller accuracy shall be within ± 1 °C (± 2 °F). If the system is not within the required accuracy, the source of the inaccuracy should be determined and the component(s) that is (are) not within the required accuracy shall be adjusted or replaced.

The temperature controller shall be verified for accuracy at an interval no less than quarterly and be checked at no less than three temperatures spanning the operating range of the equipment on or with which the device is used. The lowest temperature calibrated shall be no more than 5 °C (10 °F) above the minimum and the highest temperature calibrated shall be no more than 5 °C (10 °F) below the maximum of the user-defined operating range of the instrument.

Temperature displays that are separate from the controller shall be calibrated by the same methods used to calibrate the controller. Separate displays shall have their own calibration record.

C.3.6.4 Temperature Data Acquisition

See C.3.13 for temperature data acquisition.

C.3.6.5 Guidance for Temperature Device Calibration

This subsection provides additional guidance for temperature device calibration.

C.3.6.5.1 Heating Medium

The heating medium shall permit proper immersion of both the test thermocouple (the one being verified/calibrated) and the reference thermocouple or thermometer. The medium may be a fluidized solids bath, a heated block, or a furnace. The equipment shall be capable of maintaining a stable temperature that is uniform throughout the test section. If the heat source (heater and instrument) is portable, it should be allowed to stabilize at the ambient temperature prior to being used. This is especially true when the instrument is removed from a car or storage room, etc.

C.3.6.5.2 Procedure

Items listed here are those needing special attention or related to the use of the indicated type of equipment.

- The test and reference thermocouple or thermometer shall be placed as close together in the heating medium as possible.
- After each change of the temperature setting, the temperature of the heating medium shall be allowed to stabilize before reading the reference temperature (or voltage) and the test thermocouple temperature (or voltage). If several thermocouples are checked in sequence, each one shall be allowed to reach a stable reading before making the calibration measurement.
- If the test thermocouple does not accurately sense the temperature, a calibration curve shall be drawn and used to correct the indicated temperatures from the test thermocouple. Occasionally, small inaccuracies in thermocouple response can be compensated for during the calibration of the temperature-measuring system being used in conjunction with the thermocouple (see C.3.6.3).

C.3.6.5.3 Verification and Calibration of Temperature-measuring Systems and Controllers

C.3.6.5.3.1 Equipment

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

An alternate method consists of placing the thermocouple that is connected to the controlling or indicating instrument into a heat source and comparing it with a calibrated thermocouple or thermometer. The

temperature of the heat source is then changed as specified in C.3.6.5.2 to certify that the controlling or indicating instrument is accurately calibrated.

C.3.6.5.3.2 Procedure

- Follow the manufacturer's procedure for calibrating temperature-measuring systems and controllers. For greatest accuracy, allow proper warm-up time for calibrators, temperature-measuring systems, and controllers as specified by the manufacturer. The following items need special attention:
 - Fit the thermocouple extension cable with a proper thermocouple-grade adapter to permit plugging it into the same receptacle used for connecting the test equipment thermocouple. Take care to ensure the correct polarity of the connections.
 - Thermocouple calibrators with cold-junction compensation need only be properly connected with the proper thermocouple extension cable and thermocouple connectors. The temperature-measuring systems and controllers using this signal shall display the same temperature within the accuracy of the thermometer or controllers as supplied by the manufacturer.
 - Uncompensated thermocouple calibrators require a thermometer to determine the cold-junction temperature of the thermocouple extension cable connection of the calibrator. This cold-junction temperature shall be set on the calibrator by the operator.
 - The use of an uncompensated millivolt potentiometer requires that the temperature at the calibrator/thermocouple extension cable terminals be read with a thermometer of known accuracy. The millivolt equivalent of this temperature is then subtracted from the equivalent test millivolt signal to obtain the calibrator millivolt signal used. These voltages may be found in reference millivolt/temperature tables for the type of thermocouple in use.
 - The temperature of the calibration instrument should be allowed to stabilize at the ambient temperature at the location where it is being used. This is especially true when the instrument is removed from a car or a storage room, etc.

C.3.6.5.4 Specific Procedure for Atmospheric Pressure Consistometer

When verifying the temperature calibration of an atmospheric pressure consistometer, place one slurry container in the unit and leave the second one out. Place the reference thermocouple (attached to a master temperature calibration instrument) in the open hole as close as possible to the atmospheric pressure consistometer thermocouple, and heat the consistometer to no less than three temperatures from ambient temperature to 88 °C (190 °F) to verify the accuracy of the temperature indication. Calibrate the controller if it is not in calibration. If it is in calibration, use the verification as the calibration and date it for reference.

C.3.6.5.5 Specific Procedure for Atmospheric Pressure Water Bath

When verifying the temperature calibration of an atmospheric pressure water bath, place a stirring device in the bath (if it does not have one) along with a reference thermocouple attached to a master temperature calibration instrument in the water bath. Heat the water bath to no less than three different temperatures from ambient temperature to 88 °C (190 °F) to verify the accuracy of the temperature indication. Calibrate the controller if it is not in calibration. If it is in calibration, use the verification as the calibration and date it for reference.

C.3.7 Instrument Rotation

Rotation of consistometer containers (or paddles) at 150 r/min shall be verified and/or calibrated (if not in calibration) no less frequently than quarterly. The tolerance measured by a calibrated tachometer shall not deviate more than ± 15 r/min.

C.3.8 Pressurized Consistometer Slurry Container Assembly

Pressurized slurry container, paddle, and paddle shaft key dimensions (dimensions tagged as "INSP"; see Figures 10 to 13) shall be verified to judge wear; the maximum acceptable wear is 1 mm (0.04 in.). Dimensions shall be verified no less frequently than annually, whenever a paddle or a paddle shaft is replaced. If dimensions are found outside tolerances, the slurry container part needs to be discarded. For these pressurized consistometer parts, laboratories should devise a method of identifying them.

To verify dimensions, a caliper accurate to 0.05 mm (0.002 in.) at minimum is required.

C.3.9 Consistency Measurement Device or Potentiometer

C.3.9.1 General

The potentiometer or other consistency-measuring device shall be verified and/or calibrated (if not in calibration) no less frequently than monthly. A new calibration is required whenever repairs or adjustments are made to the device (such as calibration spring, resistor, or contact arm). Accuracy shall be maintained within $\pm 5 B_c$ (if output is in B_c) or ± 0.5 V (if output is in volts) across the calibration range. When the calibration is of a system (potentiometer, wiring, voltmeter, etc.), for the calibration to be valid, the potentiometer shall be kept together with the consistometer with which it has been calibrated. For these systems, laboratories should devise a method of identifying the potentiometer with the consistometer on which it is calibrated and ensure it is only used on that consistometer.

Some potentiometer calibration devices allow the potentiometer to be calibrated so that the voltage output is fixed based on applied load (equivalent to B_c). In such a case, provided all potentiometers are adjusted to the same voltage output vs load [or consistency (B_c)], the potentiometers may be interchanged between machines.

C.3.9.2 Procedure

A calibration chart or table showing indicated consistency vs input consistency (mass) shall be maintained. The entire range of consistencies shall be checked as given in Table C.4. Consistency values shall be reported based on the calibration curve or table.

A weight-loaded device (see Figure C.2 for a typical potentiometer calibrating device and Figure 14 for a typical potentiometer) is used to produce a series of torque-equivalent values for consistency, defined by:

$$T = 78.2 + (20.02 \times B_c) \quad (\text{C.1})$$

where

- T is torque, expressed in gram•centimeters (g•cm);
 B_c is consistency, expressed in Bearden units.

Dedicated potentiometer calibration weights are used to apply torque to the potentiometer spring, using the radius of the potentiometer frame as a lever arm. Weights cause a deflection, and the resulting DC voltage is recorded and used to determine B_c (alternatively, some instruments display the B_c equivalent

directly). Slurry consistencies at $\pm 5 B_c$ versus equivalent torques (mass of dedicated calibration weights at $\pm 0.1\%$ of the indicated mass; see C.3.2) for a potentiometer with a radius of $52 \text{ mm} \pm 1 \text{ mm}$ are given in Table C.4. Operating instructions from the manufacturer should be followed for proper calibration.

NOTE Dedicated calibration weights include the holding support washer.



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

Table C.4—Slurry Consistency vs Equivalent Torque (for Potentiometer with Radius of $52 \text{ mm} \pm 1 \text{ mm}$)

Torque Equivalent (g•cm)	Mass of Added Calibration Weights ^a $\pm 0.1\%$ of the indicated mass	Slurry Consistency ($B_c \pm 5$)
260	50	9
520	100	22
780	150	35
1040	200	48
1300	250	61
1560	300	74
1820	350	87
2080	400	100

NOTE 1 For a potentiometer with a radius other than $52 \text{ mm} \pm 1 \text{ mm}$, an adaptor ring with a radius of $52 \text{ mm} \pm 1 \text{ mm}$ or an appropriate table of equivalent tolerances is required.

NOTE 2 A consistency reading of a potentiometer may vary no more than $\pm 5 B_c$ from the slurry consistency shown in this table.

^a Mass of added calibration weight includes mass of the holding support washer.

C.3.10 Pressure Gauges and Pressure-measuring Devices

C.3.10.1 Pressure gauges shall be verified and/or calibrated (if not in calibration) no less frequently than annually using a deadweight tester or a master gauge. The term “gauge” includes pressure-sensing transducers.

C.3.10.2 Gauges designed to measure pressures greater than 17,000 kPa (2500 lbf/in.²) shall be calibrated at a minimum of 25 %, 50 %, and 75 % of full scale or the maximum user-defined working pressure of the equipment on or with which it is used. Maximum allowable error is ± 1 % of full range or \pm one minimum gauge increment, whichever is greater.

High-range gauges used for API 10A testing shall be calibrated at 17 MPa, 34 MPa, and 52 MPa (2500 lbf/in.², 5000 lbf/in.², and 7500 lbf/in.²), with a maximum defined working pressure of the equipment of 69 MPa (10,000 lbf/in.²).

C.3.10.3 Gauges designed to measure pressures up to 17,000 kPa (2500 lbf/in.²) shall be calibrated with allowable error of ± 300 kPa (± 50 lbf/in.²) at a minimum gauge reading of 3500 kPa (500 lbf/in.²) and at a gauge reading of 10,500 kPa (1500 lbf/in.²), or at the maximum allowable working pressure of the equipment or on the user-defined working pressure, whichever is greater.

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

C.3.11 Load Frame

The load frame used to measure the break force of cement specimens shall be verified and/or calibrated (if not in calibration) no less frequently than annually. Indicated force shall deviate by no more than ± 2 % of the applied load or one minimum instrument scale division, whichever is greater, at 9.0 kN (2000 lbf) load and at a minimum of 25 %, 50 %, and 75 % of the range of the load cell or load indicator. With units having multiple indicators for different ranges, each indicator shall be calibrated according to these criteria.

C.3.12 Cube Molds

Cube molds used to prepare cement specimens shall be verified no less frequently than two years.

Cube dimension tolerances are given in Table C.5. If molds are out of tolerances, molds should be discarded.

Table C.5—Density of Distilled Water as a Function of Temperature

Temperature °C	Density g/mL	Temperature °F	Density g/mL
15.0	0.9991	59	0.9991
15.5	0.9990	60	0.9990
16.0	0.9989	61	0.9989
16.5	0.9989	62	0.9988
17.0	0.9988	63	0.9987
17.5	0.9987	64	0.9986
18.0	0.9986	65	0.9985
18.5	0.9985	66	0.9984
19.0	0.9984	67	0.9983
19.5	0.9983	68	0.9982
20.0	0.9982	69	0.9981
20.5	0.9981	70	0.9980
21.0	0.9980	71	0.9978
21.5	0.9979	72	0.9977
22.0	0.9978	73	0.9976
22.5	0.9977	74	0.9975
23.0	0.9975	75	0.9973
23.5	0.9974	76	0.9972
24.0	0.9973	77	0.9970
24.5	0.9972	78	0.9969
25.0	0.9970	79	0.9968
25.5	0.9969	80	0.9966
26.0	0.9968	81	0.9965
26.5	0.9966	82	0.9963
27.0	0.9965	83	0.9961
27.5	0.9964	84	0.9960
28.0	0.9962	85	0.9958
28.5	0.9961	86	0.9956
29.0	0.9959	87	0.9955
29.5	0.9958	88	0.9953
30.0	0.9956		
30.5	0.9955		
31	0.9953		

C.3.13 Data Acquisition

C.3.13.1 Data acquisition can be by chart recorder or by electronic recording, such as computer data acquisition, or both. Data recorded are not limited to temperature, pressure, consistency, or load.

C.3.13.2 For consistency measurement devices with chart recorders, the indication of temperature and pressure should be recorded during the calibration. If the chart can be adjusted to correct indication, the record on the chart should show the indication of a verification check before calibration and after

adjustment of the chart. For those without an adjustment, a calibration table should be maintained with the instrument, and appropriate corrections made to charts of all tests conducted with the instrument. The chart should be attached to the calibration record. The use of chart recorders is discouraged due to their inaccuracy.

C.3.13.3 Electronic data recording should be verified by acquiring data during the calibration of the system and checking the accuracy of the data recorded. If the acquisition deviates by more than the limits for temperature (see C.3.6) and pressure (see C.3.10), corrections should be made. For dedicated data acquisition systems, the data acquisition system should conform to the limits prescribed above for the data they are designed to record.

C.3.14 Volumetric Glassware

C.3.14.1 General

Calibration of pipettes, graduated cylinders, conical flasks, and similar equipment is generally performed by the glassware supplier and may be part of the purchase specification. Glassware users should obtain documented evidence, where deemed important, of glassware calibration from the supplier. Calibration may be checked gravimetrically before the first use. Periodic recalibration is not required.

C.3.14.2 Procedure—Verification/Calibration of Graduated Cylinders

The following procedure shall be followed to verified calibration of graduated cylinders. This procedure may be adapted to any volumetric glassware.

- a) Allow the receiver and deionized (or distilled) water to reach ambient temperature. Record the temperature to the nearest 0.5 °C (1 °F).
- b) Place the clean, empty receiver, with its base, on the balance and tare to zero.
- c) Fill the receiver with deionized (or distilled) water, while it is on the balance, to the various graduation marks in increments of 2 mL for the 10-mL receiver, in increments of 4 mL for the 20-mL receiver, and in increments of 10 mL for the 50-mL receiver. Use a pipette or syringe to carefully fill the receiver to the desired graduation mark without leaving water droplets on the walls of the receiver.
- d) Record the masses for each incremental volume of water at the specific graduation mark, m_W , to the nearest 0.01 g.
- e) Calculate the volume of the receiver at each mark, V_M :

$$V_M = \frac{m_W}{\rho_W} \quad (\text{C.2})$$

where

V_M is the receiver volume at a specific mark, expressed in milliliters;

m_W is the mass of water, expressed in grams;

ρ_W is the water density, expressed in grams per milliliter, at the test temperature (see Table C.6).

Annex D (informative)

Calibration Procedures for Thermocouples, Temperature-measuring Systems, and Controllers

D.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.

D.2 Thermocouple Calibration

D.2.1 Apparatus

D.2.1.1 General

The individual pieces of the apparatus required to carry out the calibration depend on the particular technique selected. Those features that need special attention, regardless of the technique, have the requirements given in D.2.1.2 through D.2.1.4.

D.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.

D.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 a national/international standardization body.

D.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; see 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.

D.2.2 Procedure

With the exception of the indicating instruments, the specific procedures are detailed in applicable national standards; see ASTM E220. The following items require special attention or are related to the use of the indicating type of equipment.

- a) The test and reference thermocouples or thermometers should be placed as close together as possible in the heating medium.

- b) 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).
- c) Several (minimum three) test temperatures that span the operating range of the equipment should be used in the calibration procedure.
- d) 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 requirements given in ASTM E230 for classification of "special" Type J thermocouple have error limits equal to or better than ± 1.1 °C (± 2 °F) up to 275 °C (530 °F) and 0.4 % of the reading above 400 °F.

D.3 Calibration of Temperature-measuring Systems and Controllers

D.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 with a table of reference voltages. Signal sources, or calibrators, are of two types: 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 apparatuses should be traceable to a national standard 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.

D.3.2 Procedure

The manufacturer's procedure for calibrating temperature-measuring systems and controllers should be followed. The following items require special attention.

- a) 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.
- b) 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.
- c) 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.

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.

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