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Oil and gas industries including lower carbon energy — Cements and materials for well cementing —

Part 5:
Determination of shrinkage and expansion of well cement formulations

Industries du pétrole et du gaz, y compris les énergies à faible teneur en carbone — Ciments et matériaux pour la cimentation des puits —

Partie 5: Détermination du retrait et de l'expansion des formulations de ciments pour puits

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Foreword

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This document was prepared by Technical Committee ISO/TC 67, *Oil and gas industries including lower carbon energy*, Subcommittee SC 3, *Drilling and completion fluids, well cements and treatment fluids,* in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 12, *Oil and gas industries including lower carbon energy,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 10426-5:2004), which has been technically revised.

The main changes are as follows:

— addition of the Introduction, with background information on expansion and shrinkage;

— addition of annular ring test under impermeable conditions at atmospheric pressure;

— inclusion of an informative annex describing a method to determine the stress generated by expansion under confined conditions at elevated temperature and pressure;

— inclusion of an informative annex describing the annular ring test at elevated pressure.

A list of all parts in the ISO 10426 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

When Portland cement reacts with water, there is an overall reduction in the absolute volume of components:

$$V_c + V_w > V_{ch} \quad (1)$$

where

V_c is the volume of cement;

V_w is the volume of water;

V_{ch} is the volume of cement hydrates.

In this document the absolute volume decrease $[(V_c + V_w) - V_{ch}]$ is referred to as hydration shrinkage, although in other documents it can also be referred to as chemical shrinkage, total chemical contraction, or hydration volume reduction.

Depending on the exposure conditions, presence of external stresses during setting and, most importantly, access to external water, the hydration shrinkage may lead to bulk shrinkage of the set cement.

The change in the sample dimensions is referred to as bulk shrinkage or expansion. Bulk shrinkage and expansion of the cement refer to the result of the measurement of a linear dimensional change or volume change. The volume to which all volume changes are related is the volume of the slurry immediately after mixing and emplacement in the experimental equipment. For small values of shrinkage or expansion, typically the case in well cement systems, the fractional volume dimensional change can be approximated as 3 times the fractional linear dimensional change.

Bulk shrinkage may cause:

— formation of a micro-annulus, potentially affecting cement evaluation logs;

— loss of zonal isolation leading to crossflow or sustained casing pressure;

— lack of a hydraulic seal when using cement inflatable packers;

— poor sealing of abandonment plugs.

Additives are available that can overcome the effects of hydration shrinkage and generate bulk expansion of set cement. In plug applications, bulk expansion of cement generates stress at the cement-rock or cement-formation interface. A method of measuring the stress generated by expansion in a plug-type geometry is given in Annex A.

In this document, SI units are used; and where practical, U.S. customary units are included in brackets for information.

This document is based on API Technical Report 10TR 2.

Oil and gas industries including lower carbon energy — Cements and materials for well cementing —

Part 5:
Determination of shrinkage and expansion of well cement formulations

1 Scope

This document provides the methods for the testing of well cement formulations to determine the dimension changes during the curing process (cement hydration) at atmospheric and elevated pressure and the stress generated by expansion in a confined environment under elevated temperature and pressure.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

API Specification 10A, *Cements and Materials for Well Cementing*

API Recommended Practice 10B-2, *Recommended Practice for Testing Well Cements*

3 Terms and definitions

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

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
bulk expansion
increase in the external volume or dimensions of a cement sample

3.2
bulk shrinkage
decrease in the external volume or dimensions of a cement sample

3.3
CEA
cement expansion additive
additive used in a cement slurry formulation to provide *bulk expansion* (3.1), or reduce *bulk shrinkage* (3.2)

3.4
hydration shrinkage
difference in the volume between the hydration products and the volume of the dry cement, additives and water

3.5
radial interface stress
stress generated at the interface between the set cement and casing or borehole wall due to *bulk shrinkage* (3.2) or *bulk expansion* (3.1) of the cement

3.6
UCA
ultrasonic cement analyser
instrument used for the non-destructive sonic determination of compressive strength of cement

4 Determination of shrinkage or expansion under conditions of free access to water at atmospheric pressure — Annular ring test

4.1 General

The annular expansion mould is a device suitable for measuring only the linear bulk shrinkage or expansion properties of a cement formulation. The magnitude of expansion depends on the amount and type of expanding agent, cement powder, slurry design and curing condition (pressure, temperature, time, fluid access). It should be noted that expansion is strongly affected by boundary conditions. The chemical process of synthetic mineral growth is strongly controlled by the state of stress and growth tends to occur relatively more in low stress locations, for example, in pore spaces within the cement matrix. Therefore, the degree of cement shrinkage and expansion is dependent on several conditions, not all of which can be uniquely defined. The test does not fully represent the annulus of a well.

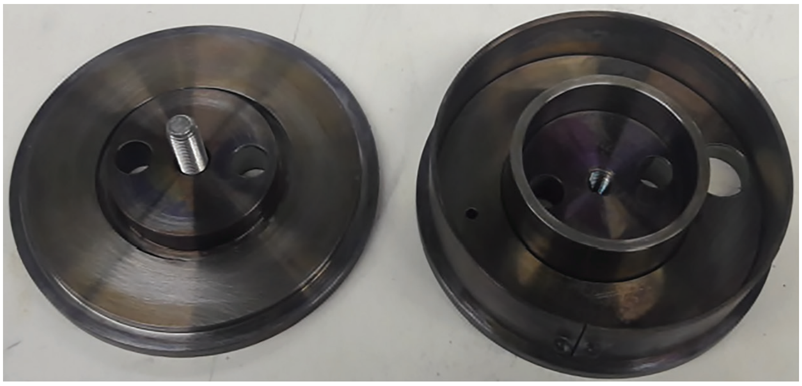
A method for determining the shrinkage or expansion at pressures above atmospheric pressure is given in Annex B.

4.2 Apparatus

4.2.1 Mould

4.2.1.1 General

Use corrosion-resistant material (e.g. stainless steel). The outer diameter (OD) of the internal ring shall be 50,8 mm ± 0,3 mm (2,0 in ± 0,01 in) and the OD of the external ring shall be 88,9 mm ± 0,3 mm (3,5 in ± 0,01 in). See Figures 1, and 2.



1 2

Key

1 bottom plate

2 inner and outer rings placed on the top plate (step d of 4.3.1)

Figure 1 — Typical mould assembly

Dimensions in millimetres (inches)

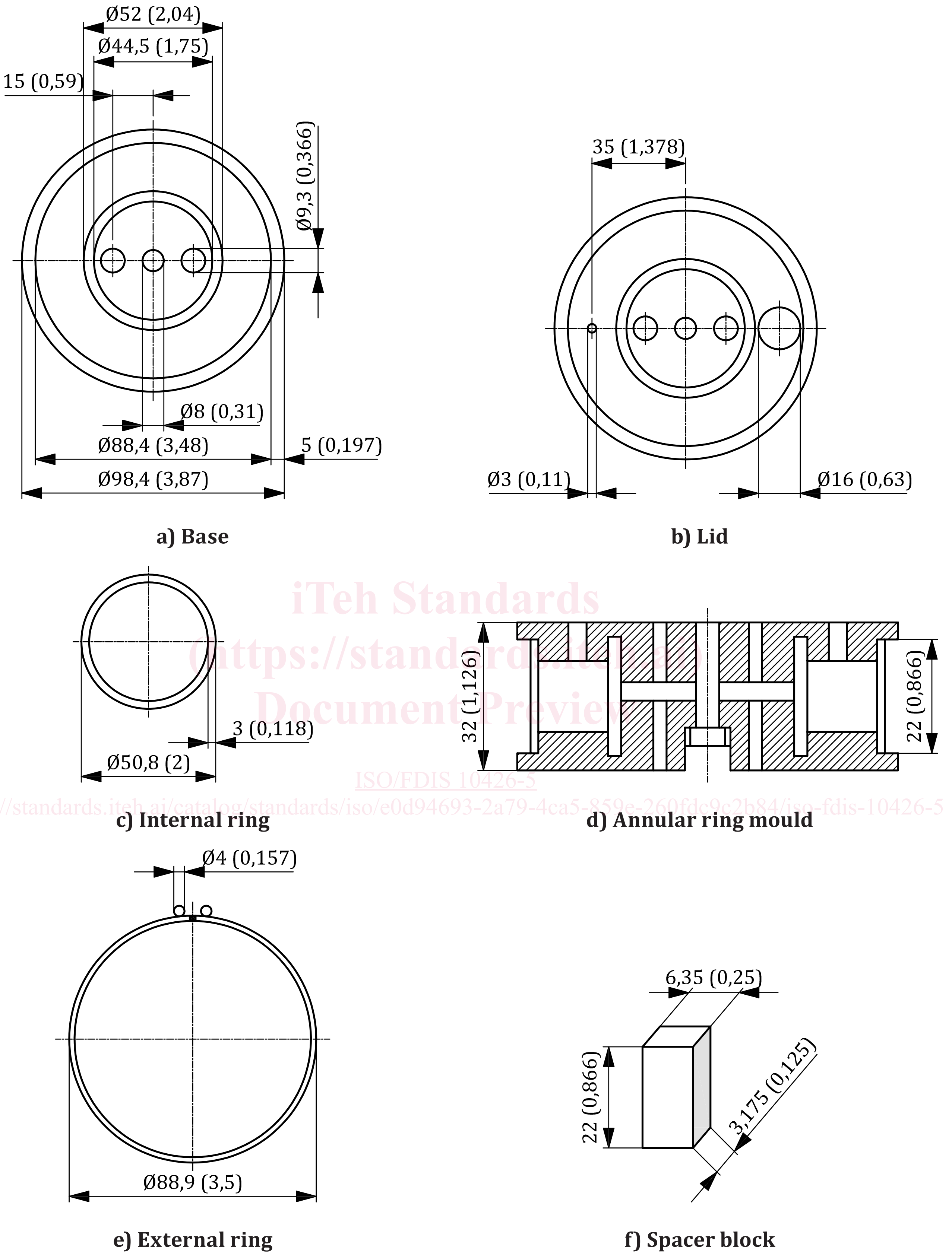
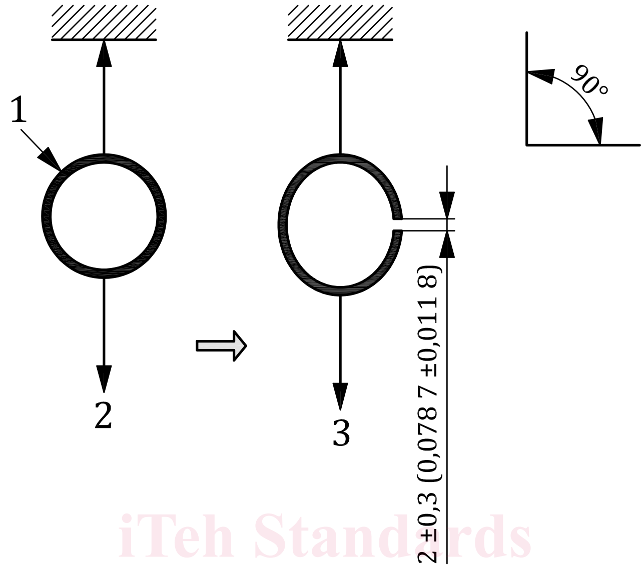


Figure 2 — Schema of typical mould assembly parts

4.2.1.2 Mould verification

The resilience of the ring of the mould shall be verified annually. If the ring is dropped or damaged during use, then the resilience shall be verified. The resilience shall be such that the mass of 1 000 g ± 1 g applied as shown in Figure 3 shall increase the distance between the two steel measurement balls (see Figure 8) by 2 mm ± 0,3 mm (0,078 7 in ± 0,011 8 in) without permanent deformation. The load shall be applied perpendicular to the gap (90°). The readings shall be repeated at least three times to obtain an average value with a standard deviation of 0,05 mm (0,002 in).

Dimensions in millimetres (inches)



Key

1 ring

2 mass, 0 g

3 mass, 1 000 g ± 1 g

Figure 3 — Schema of a calibration measurement of the ring — Resilience test

4.2.1.3 Spacer block

The spacer block shall be used only in the case of shrinkage measurement. It is used to slightly increase the diameter of the outer ring prior to slurry-pouring and to measure shrinkage by removing it once the cement has set. The dimensions of the block shall be (3,2 mm ± 0,1 mm) × (6,4 mm ± 0,1 mm) (0,125 in × 0,25 in) and 22,0 mm (0,866 in) tall; see Figure 2. To ensure that the spacer block's thermal expansion properties are the same as those of the expandable outer ring, the block shall be made of the same material as the mould (e.g. stainless steel).

4.2.2 Water curing bath

A curing bath or tank having dimensions suitable for the complete immersion of a mould(s) in water and which can be maintained within ±2 °C (±3 °F) of the prescribed test temperature shall be employed. The curing bath is an atmospheric-pressure apparatus (bath) for curing specimens at a temperature of up to 88 °C (190 °F). It shall have an agitator or circulating system.

4.2.3 Temperature-measuring system

The temperature-measuring system shall be calibrated to an accuracy of ±1 °C (±2 °F). Calibration shall be no less frequent than quarterly. The procedure described in API Recommended Practice 10B-2 should be used.

4.2.4 Atmospheric-pressure consistometer

The atmospheric-pressure consistometer shall meet the requirements of the apparatus defined in API Specification 10A.

4.2.5 Micrometer

A micrometer with a digital step of 0,001 mm (0,000 05 in) or smaller shall be used to measure the separation of the measurement balls. The micrometer shall be calibrated to an accuracy of ±0,005 mm (0,000 2 in) no less frequently than annually.

4.3 Procedure

4.3.1 Preparation of the mould

The assembled moulds shall be watertight to avoid leakage. The interior faces of the moulds and contact surfaces of the plates may be lightly coated with a release agent. Alternatively, the interior faces of the moulds and contact surfaces of the plates may be left clean and dry. In the case of a shrinkage test, place the spacer block inside the split of the outer ring. Prepare the mould as follows.

a) Clean the mould thoroughly.

b) Place a bead of grease on the upper and lower plates where the inner stationary ring and the outer expandable ring touch. For tests using the spacer block to measure shrinkage, it is essential that the beads of grease be sufficient to completely fill the space between the outer ring and the mating parts on the top and bottom plates. If cement penetrates these gaps, it prevents inwards movement of the outer ring giving erroneous shrinkage measurements.

c) If desired, apply a very thin film of release agent to the inner and outer rings and to the surface of the top and bottom covers that contact the cement.

d) With the top cover inverted, place the inner and outer rings on the top cover.

e) To test for shrinkage, coat a spacer block with grease and place the block with the small side between the split in the outer expandable ring; see Figures 4 and 5.

f) Place the bottom cover over the inner and outer rings.

g) Insert the bolt into the centre hole and tighten the bolt to hold the mould together.

h) Verify that the expandable outer ring rotates freely and place the big hole adjacent to the split (Figure 8).

i) Place a small amount of grease between the split in the outer expandable ring. The grease forms a seal and prevents the slurry from leaking before it sets.