



**International  
Standard**

**ISO 10426-5**

**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*

**Second edition  
2024-09**

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# Contents

	Page
<b>Foreword</b> .....	<b>iv</b>
<b>Introduction</b> .....	<b>v</b>
<b>1 Scope</b> .....	<b>1</b>
<b>2 Normative references</b> .....	<b>1</b>
<b>3 Terms and definitions</b> .....	<b>1</b>
<b>4 Determination of shrinkage or expansion under conditions of free access to water at atmospheric pressure — Annular ring test</b> .....	<b>2</b>
4.1 General.....	2
4.2 Apparatus.....	2
4.2.1 Mould.....	2
4.2.2 Water curing bath.....	4
4.2.3 Temperature-measuring system.....	4
4.2.4 Atmospheric-pressure consistometer.....	5
4.2.5 Micrometer.....	5
4.3 Procedure.....	5
4.3.1 Preparation of the mould.....	5
4.3.2 Preparation of slurry.....	6
4.3.3 Filling of the mould.....	6
4.3.4 Test period.....	6
4.4 Measurement and calculations.....	7
<b>5 Determination of shrinkage or expansion under impermeable condition at atmospheric pressure — Annular ring test in a re-sealable bag</b> .....	<b>9</b>
5.1 General.....	9
5.2 Apparatus.....	9
5.2.1 General.....	9
5.2.2 Re-sealable bag.....	10
5.3 Procedure.....	10
5.4 Measurement and calculations.....	10
<b>6 Determination of bulk shrinkage or expansion under impermeable condition and atmospheric pressure — Membrane test</b> .....	<b>10</b>
6.1 General.....	10
6.2 Apparatus.....	11
6.2.1 Membrane.....	11
6.2.2 Water curing bath.....	11
6.2.3 Temperature-measuring system.....	11
6.2.4 Electronic scales.....	11
6.3 Procedure.....	11
6.3.1 Preparation of the membrane.....	11
6.3.2 Preparation of slurry.....	11
6.3.3 Filling of the membrane.....	11
6.3.4 Curing.....	11
6.4 Measurement and calculations.....	12
<b>Annex A (informative) Determination of stress generated by expansion at elevated pressure and temperature</b> .....	<b>14</b>
<b>Annex B (informative) Determination of shrinkage or expansion under conditions of free access to water at elevated pressure — Annular ring test</b> .....	<b>20</b>
<b>Bibliography</b> .....	<b>22</b>

## Foreword

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

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see [www.iso.org/directives](http://www.iso.org/directives)).

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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](http://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.

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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

### 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.

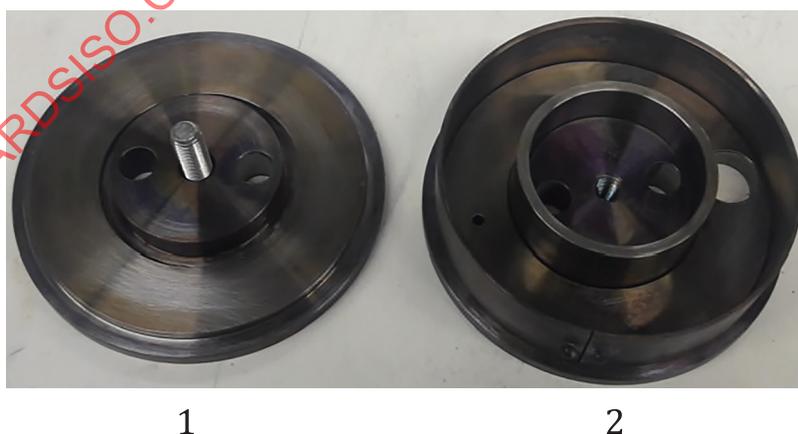
A method for determining the shrinkage or expansion at pressures above atmospheric pressure is given in [Annex B](#).

### 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  $50,8 \text{ mm} \pm 0,3 \text{ mm}$  ( $2,0 \text{ in} \pm 0,01 \text{ in}$ ) and the OD of the external ring shall be  $88,9 \text{ mm} \pm 0,3 \text{ mm}$  ( $3,5 \text{ in} \pm 0,01 \text{ in}$ ). See [Figures 1](#), and [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

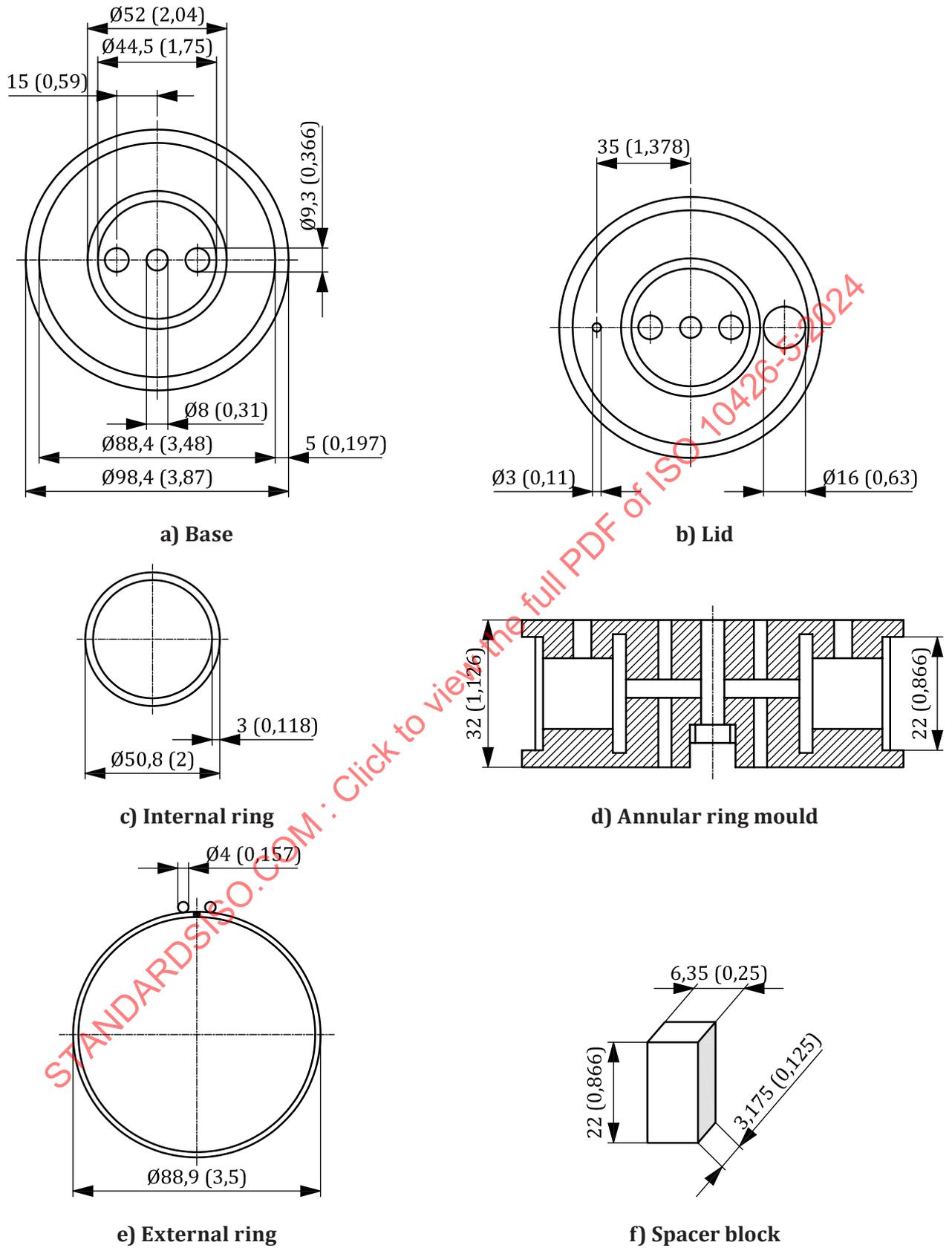
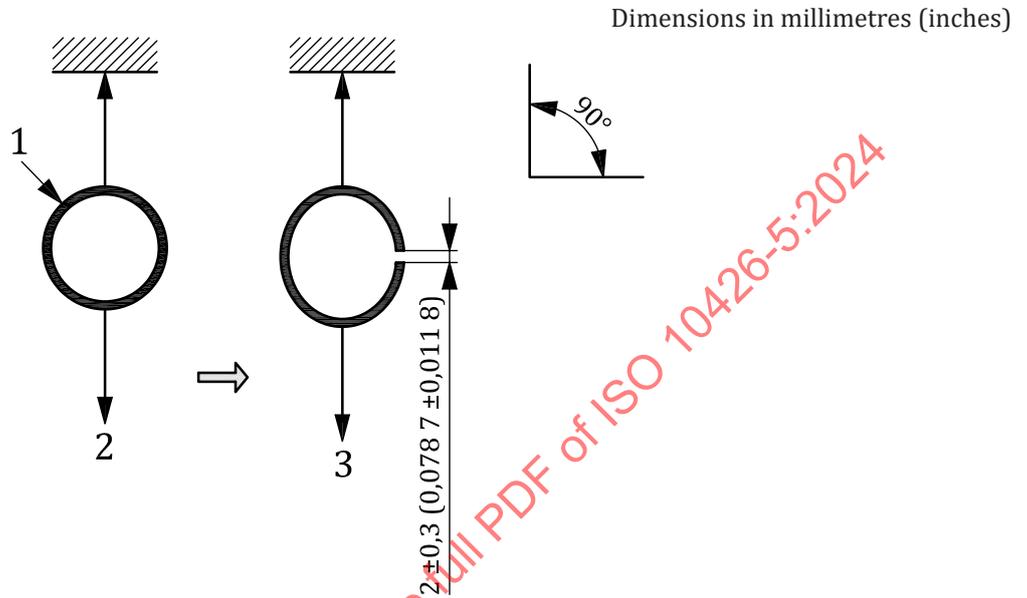


Figure 2 — Schema of typical mould assembly parts

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  $1\ 000\text{ g} \pm 1\text{ g}$  applied as shown in Figure 3 shall increase the distance between the two steel measurement balls (see Figure 8) by  $2\text{ mm} \pm 0,3\text{ mm}$  ( $0,078\ 7\text{ in} \pm 0,011\ 8\text{ in}$ ) without permanent deformation. The load shall be applied perpendicular to the gap ( $90^\circ$ ). The readings shall be repeated at least three times to obtain an average value with a standard deviation of  $0,05\text{ mm}$  ( $0,002\text{ in}$ ).



Key

- 1 ring
- 2 mass, 0 g
- 3 mass,  $1\ 000\text{ g} \pm 1\text{ g}$

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  $(3,2\text{ mm} \pm 0,1\text{ mm}) \times (6,4\text{ mm} \pm 0,1\text{ mm})$  ( $0,125\text{ in} \times 0,25\text{ in}$ ) and  $22,0\text{ mm}$  ( $0,866\text{ in}$ ) 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  $\pm 2\text{ }^\circ\text{C}$  ( $\pm 3\text{ }^\circ\text{F}$ ) 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  $88\text{ }^\circ\text{C}$  ( $190\text{ }^\circ\text{F}$ ). 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  $\pm 1\text{ }^\circ\text{C}$  ( $\pm 2\text{ }^\circ\text{F}$ ). 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  $\pm 0,005$  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.

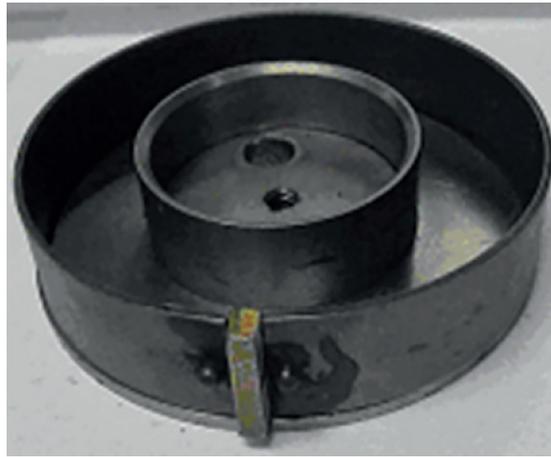


Figure 4 — Mould with a spacer block (top view)

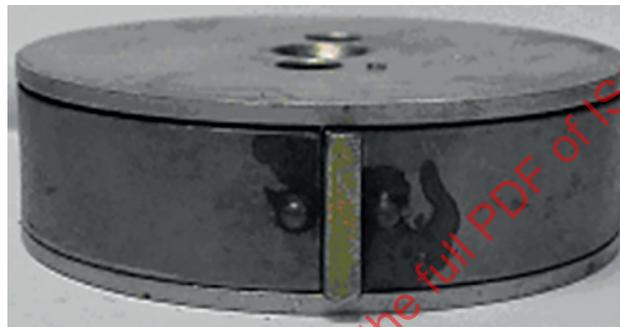


Figure 5 — Mould with a spacer block (side view)

#### 4.3.2 Preparation of slurry

The cement slurry shall be mixed and conditioned in accordance with the procedures described in API Recommended Practice 10B-2.

#### 4.3.3 Filling of the mould

After conditioning, the slurry shall be re-stirred with a spatula to ensure uniformity. The slurry is poured into the large fill hole at the outer portion of the top of the ring mould. The small hole in the top of the mould is for venting air from the mould during filling. The mould is filled until the slurry exits the small hole. On some more viscous slurries, the mould can need to be tapped or vibrated to ensure it is filled. Once the slurry is poured, the mould is placed in a water bath that has been preheated to the test temperature. The slurry is in contact with water during the entire test. Water entry compensates for any inner shrinkage if the cement matrix is permeable. If the set cement expands, the outside diameter of ring expands.

#### 4.3.4 Test period

The test period is the elapsed time from subjecting the sample to test temperature in the atmospheric consistometer to the time of final measurement at the end of curing time. Measurements may be made at intermediate times to determine the expansion profile. In this case the cells are removed from the water bath for the measurement and then returned to the bath until the subsequent measurement is scheduled. The time from the start of the test period to the time of measurement is noted along with the measurement.

For a test using the spacer block, the spacer block shall be removed after the cell is first removed from the water bath and before the first measurement is made. The spacer block is not replaced for subsequent measurements.

#### 4.4 Measurement and calculations

Before curing in the atmospheric bath, an initial measurement is taken with a micrometer immediately after the mould is filled with slurry. The micrometer, which may be mounted on a height-adapter block, is opened and placed on a smooth, flat surface beside the mould to measure the distance between the outside of the steel balls attached to each side of the split in the expandable ring (with the spacer block in place, if used).

After curing, another measurement is taken in the same manner as the initial measurement. If the spacer block is used, it should be carefully removed to take the measurement and not replaced if subsequent measurements are required. Do not expand the outer expandable ring while removing the spacer block. The distance between the two steel balls is measured with a micrometer with a precision of 0,02 mm (0,001 in). This measurement shall be performed immediately (less than 5 min) after removal from the curing bath.

[Figures 6, 7, 8, 9](#) and [10](#) show one example of a height-adapter block used with a micrometer. The use of a height-adapter block improves the reproducibility of the measurements.



Figure 6 — Micrometer and components of a height-adapter block



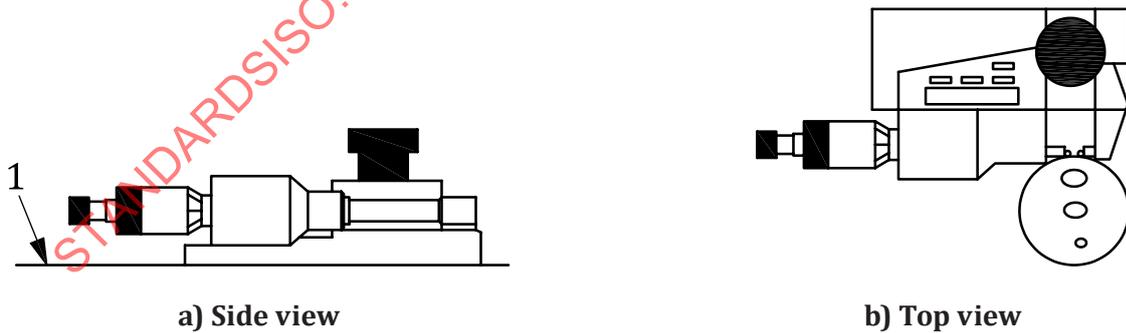
Figure 7 — Micrometer mounted to a height-adapter block



Figure 8 — Micrometer measuring



Figure 9 — Micrometer alignment



a) Side view

b) Top view

Key

1 table top

Figure 10 — Schematic of micrometer and height-adaptor block

The percent circumferential change (shrinkage or expansion) is calculated from measurements in SI units as follows:

$$l_{\Delta} = (l_{f,SI} - l_{i,SI}) \times 0,358 \quad (2)$$

where

$l_{\Delta}$  is the circumferential change, expressed in per cent, of the cement sample;

$l_{f,SI}$  is the final distance, expressed in millimetres, after curing;

$l_{i,SI}$  is the initial distance, expressed in millimetres.

Alternatively, the percent circumferential change (shrinkage or expansion) can be calculated from measurements in U.S. customary units as follows:

$$l_{\Delta} = (l_{f,US} - l_{i,US}) \times 9,095 \quad (3)$$

where

$l_{\Delta}$  is the circumferential change, expressed in per cent, of the cement sample;

$l_{f,US}$  is the final distance, expressed in inches, after curing;

$l_{i,US}$  is the initial distance, expressed in inches.

NOTE 1 It is possible to measure either shrinkage or expansion with this test; a positive  $l_{\Delta}$  indicates expansion; a negative  $l_{\Delta}$  indicates shrinkage.

NOTE 2 Given the reproducibility of the measurement of the separation of the measurement balls, values of  $l_{\Delta} \leq 0,1$  % do not necessarily indicate expansion.

The coefficients used in [Formulae \(2\)](#) and [\(3\)](#) are calculated considering an inner diameter of 88,9 mm (3,5 in) for the outer expansion ring.

It is assumed the expansion is expressed by the change in circumference as measured by the change in distance between the balls. The measurement is a chord and not an arc, but since the difference is small, it can be neglected.

The effect of thermal expansion of the cell on the measurements can be ignored. For a typical stainless steel (thermal expansion coefficient  $17 \times 10^{-6} \text{ }^{\circ}\text{C}^{-1}$ ), a  $10 \text{ }^{\circ}\text{C}$  ( $18 \text{ }^{\circ}\text{F}$ ) change in temperature between the initial and subsequent measurement gives a difference of 0,02 % due to thermal expansion.

## 5 Determination of shrinkage or expansion under impermeable condition at atmospheric pressure — Annular ring test in a re-sealable bag

### 5.1 General

This test uses the annular expansion ring to determine the expansion or shrinkage of cement systems at atmospheric pressure without contact with water. The expansion ring is placed inside a re-sealable plastic bag which is then placed in a water bath. The re-sealable bag prevents evaporation of water.

### 5.2 Apparatus

#### 5.2.1 General

The method uses the same equipment as described in [4.2](#) except for the re-sealable bag.

### 5.2.2 Re-sealable bag

A re-sealable bag is a flexible, rectangular, and transparent storage bag. These bags are sealed via interlocking flanged profiles. The mechanism is activated either manually or via a zipper mechanism. The bags are usually manufactured from polyethylene which is suitable for application up to 85 °C (185 °F).

The dimensions of the re-sealable bag should be sufficient to allow the ring mould to be placed inside but without too much excess space (see [Figure 11](#)). A suitable bag dimension is 25 cm × 25 cm. The thickness of the bag walls should be a minimum of 0,075 mm (0,003 in).



Figure 11 — Ring mould inserted in a re-sealable bag

### 5.3 Procedure

The procedure for determination of shrinkage or expansion under conditions of free access to water at atmospheric pressure ([4.3](#)) is used, except that the ring mould is sealed in the re-sealable storage bag before it is placed into a water bath. As much air as possible should be squeezed from the bag before sealing.

Each time the cell and the re-sealable bag are removed from the water bath, the bag shall be examined to ensure that there is no water ingress. Any time that water, other than condensation, is seen inside the bag the test should be stopped.

### 5.4 Measurement and calculations

The procedure for the measurement and calculations given in [4.4](#) shall be followed.

## 6 Determination of bulk shrinkage or expansion under impermeable condition and atmospheric pressure — Membrane test

### 6.1 General

The purpose of this test method is to measure the bulk expansion or shrinkage when the cement is unable to absorb water or gas. This is commonly the case when the cement is placed within impermeable boundaries in a well.

## 6.2 Apparatus

### 6.2.1 Membrane

An impermeable flexible membrane is used to measure shrinkage or expansion under impermeable conditions. The membrane is sealed by tying a knot at the top. The material of the membrane shall not react in a high-pH environment and shall be capable of containing at least  $150 \text{ ml} \pm 30 \text{ ml}$  of slurry.

### 6.2.2 Water curing bath

A curing bath or tank having dimensions suitable for the complete immersion of the filled membrane in water and which can be maintained within  $\pm 2 \text{ }^\circ\text{C}$  ( $\pm 3 \text{ }^\circ\text{F}$ ) of the prescribed test temperatures shall be employed.

### 6.2.3 Temperature-measuring system

The temperature-measuring system shall be calibrated to an accuracy of  $\pm 1 \text{ }^\circ\text{C}$  ( $\pm 2 \text{ }^\circ\text{F}$ ). Calibration shall be no less frequent than quarterly. The procedure described in API Recommended Practice 10B-2 shall be used.

### 6.2.4 Electronic scales

Electronic scales with a precision of 0,01 g shall be used (preferably with data acquisition interfaced with a computer).

## 6.3 Procedure

### 6.3.1 Preparation of the membrane

To reduce the permeability of the membrane, the internal and external surface shall be covered with a thin layer of silicone grease; do not use spray grease, since the solvent can adversely affect the permeability of membrane.

### 6.3.2 Preparation of slurry

The cement slurry shall be mixed and conditioned in accordance with the procedures described in API Recommended Practice 10B-2.

Settling of and free-fluid formation in the slurry shall be avoided with a properly optimized slurry composition. The free-fluid test shall be performed in accordance with API Recommended Practice 10B-2. The slurry shall exhibit zero free fluid.

### 6.3.3 Filling of the membrane

After conditioning, the slurry shall be re-stirred with a spatula to ensure uniformity. The slurry is then poured into an impermeable flexible membrane, which is sealed by tying a knot at the top.

Extreme care shall be taken so that no air is entrapped in the slurry or at the top of the membrane before sealing.

Make sure the knot is tight so that no water from the bath can access the slurry during the test.

The volume of slurry shall be  $150 \text{ ml} \pm 30 \text{ ml}$ . Determine the mass in air of the sealed membrane containing cement with a precision of  $\pm 0,01 \text{ g}$ .

### 6.3.4 Curing

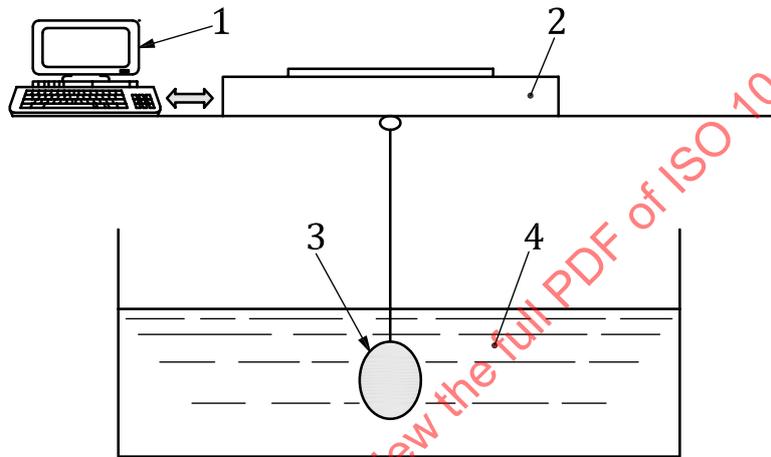
Once the membrane has been prepared and weighed in air, it is placed in a fine net, suspended from the balance hook, and completely immersed in a water bath that has been preheated to the test temperature; see [Figure 12](#).

Attention shall be paid to prevent air from becoming attached to the external surfaces of the membrane or the net.

Expansion that is evident before the cement has reached the temperature of the bath (beginning of test) can correspond to thermal expansion of the air that has been trapped. In this case, the test is invalid and shall be repeated.

The amount of water lost through evaporation in the bath is not critical at 35 °C (95 °F). A thin layer of oil should be placed on the water to minimize evaporation. The oil must not contact the membrane as some membranes are degraded by oil. The water evaporation becomes significant at higher temperatures. A reference level is chosen for the water bath and water at the same test temperature  $\pm 2$  °C ( $\pm 3$  °F) is regularly added.

If water penetrates the sample during the curing due to leaks in the membrane, this gives the appearance of strongly accelerated shrinkage. At this point, the integrity of the membrane should be verified (some of them dissolve after a few days' exposure to the cement); and the test should be repeated with a new membrane (see 6.2.1).



**Key**

- 1 data-acquisition system
- 2 balance or load cell
- 3 slurry in impermeable membrane
- 4 bath with temperature control

**Figure 12 — Schematic of apparatus set-up**

**6.4 Measurement and calculations**

The measured mass  $m_m$ , of the membrane containing the cement in the water bath, is equal to the mass,  $m_a$ , in air, minus the buoyancy due to the water.

The initial measured mass,  $m_{im}$ , is recorded immediately after the membrane is immersed in the water bath; subsequent measured masses are recorded with a precision of  $\pm 0,01$  g. If using data acquisition, masses should be recorded at least every 10 min. An increase in the measured mass corresponds to a decrease of the volume (shrinkage).

Because the membrane test can result in either a decrease or an increase of the external volume, the volumetric change measured with the membrane is called percent bulk change and noted as  $V_{bc}$ .

To calculate the initial and final volumes,  $V_i$  and  $V_f$  respectively, and then the shrinkage or expansion, one assumes that the mass and volume of the membrane and the net are very small and negligible.

Therefore, a positive value corresponds to a bulk expansion, while a negative value corresponds to a bulk shrinkage.

## ISO 10426-5:2024(en)

The percent bulk change (shrinkage or expansion) is calculated in accordance with [Formulae \(4\)](#) to [\(6\)](#):

$$V_{bc} = 100 \times (V_f - V_i) / V_i \quad (4)$$

where

$V_i$  is the initial volume, expressed in millilitres, of the cement slurry;

$V_f$  is the final volume, expressed in millilitres, of the cement slurry.

$$V_i = (m_{ai} - m_{im}) / \rho_w \quad (5)$$

where

$m_{ai}$  is the initial mass, expressed in grams, in air of the membrane containing the cement slurry;

$m_{im}$  is the initial measured mass, in water, expressed in grams, of the membrane containing cement slurry;

$\rho_w$  is the density, expressed in grams/millilitre, of water.

$$V_f = (m_{af} - m_{fm}) / \rho_w \quad (6)$$

where  $m_{fm}$  is the final measured mass, in water, expressed in grams, of the membrane containing cement slurry.

The test period is the elapsed time from subjecting the sample to curing in the atmospheric consistometer to the time of final measurement at the end of curing time.

The mass at the end of the test ( $m_{af}$ ), expressed in grams, in air of the membrane containing the set cement shall be measured.

If  $m_{af}$  differs from  $m_{ai}$  by more than 0,1 g, the test is not valid, and the results shall be discarded. An increase in mass ( $m_{af} > m_{ai}$ ) indicates that water has penetrated the membrane; and a decrease in mass ( $m_{af} < m_{ai}$ ) indicates that slurry has leaked from the membrane.

## Annex A (informative)

# Determination of stress generated by expansion at elevated pressure and temperature

### A.1 General

The methods described in [Clauses 4](#) to [6](#) are used to determine the shrinkage and expansion of cement systems with relatively low stress boundary conditions. However, in many well applications, cement systems are placed in situations with rigid boundaries, for example, as plugs inside casing, cement placed between two casing strings or between a casing and a rock with a relatively high Young's modulus. In these situations, cement expansion induces stress at the interfaces with the casing strings or rock, rather than a significant increase in volume. The stress generated at the interfaces is an important parameter in the sealing efficiency of the cement. For a given cement system, generally the higher the radial interface stress, the better the sealing efficiency.

The method described in this annex allows the radial interface stress to be measured using a modified UCA vessel. A piezoresistive pressure transducer or equivalent is mounted in the side wall of the UCA pressure vessel. The pressure transducer measures the stress generated by cement with bulk volume expansion superimposed on the hydrostatic pressure of the UCA test. The superimposed stress measured is typically up to 10 MPa (1 450 lbf/in<sup>2</sup>). A minimum of 1 MPa (145 lbf/in<sup>2</sup>) is generally required to significantly reduce the leakage rate in laboratory cement plug sealing tests.

The method does not differentiate between systems that shrink and systems that do not show any bulk volume change. In each case the transducer indicates the pressure of the UCA pump.

Due to the small sample size and access to external fluid, the stress generated by the cement is under conditions where the pores remain saturated with water (no pore pressure decrease) and there is little or no temperature change during setting.

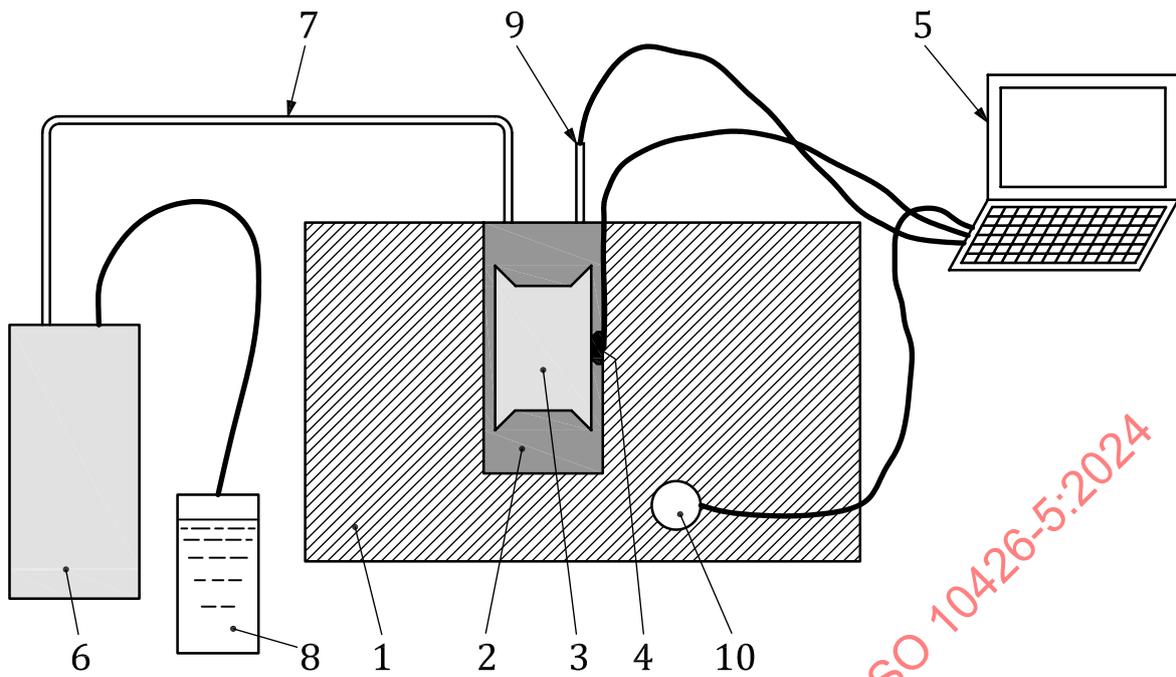
### A.2 Apparatus

#### A.2.1 General

The apparatus consists of a modified UCA apparatus, with a pressure transducer in the side wall to measure radial interface stress. [Figure A.1](#) shows a schematic of a modified UCA apparatus. The main differences with a conventional UCA apparatus are:

- a) pulse-free pressurising pump;
- b) modified UCA cell with a pressure transducer fitted in the UCA side wall.

A conventional UCA heating and control system can be used; and the modified cell also allows the usual UCA compressive strength measurement to be made simultaneously.



**Key**

- |   |                                      |    |                               |
|---|--------------------------------------|----|-------------------------------|
| 1 | UCA unit                             | 6  | pulse-free high-pressure pump |
| 2 | modified UCA vessel                  | 7  | high-pressure water line      |
| 3 | cement sample at downhole conditions | 8  | water reservoir               |
| 4 | wall mounted pressure transducer     | 9  | temperature probe             |
| 5 | data acquisition system              | 10 | acoustic UCA signal           |

**Figure A.1 — UCA setup with modified vessel and external pressure pump**

**A.2.2 Pulse-free pressurising pump**

As the expansion stress is superimposed on the UCA test pressure, the applied water pressure should remain sufficiently constant throughout the test to allow the radial interface stress to be visible above any pressure pulses from the control pump. A pressurising pump that maintains pressure pulses below  $\pm 0,05$  MPa ( $\pm 7$  lbf/in<sup>2</sup>) is suitable for this application.

Typical internal pumps supplied with conventional UCA equipment do not meet this requirement.

**A.2.3 Modified UCA cell**

A modified UCA cell is shown in [Figure A.2](#) and [A.3](#). The pressure transducer is mounted flush with the inside of the UCA cell at mid-height; and the outer side of the cell includes a slot for the transducer cables to exit the main UCA unit.

**WARNING — The modified pressure vessel has a lower rating than the conventional UCA cell, therefore the over pressure rupture disc of the UCA apparatus must be changed. The manufacturer provides information on the maximum working pressure limit of the cell and how to change the over pressure rupture disc.**



Figure A.2 — Modified UCA vessel with on the right a zoom-in on the slot for transducer cables (outside view)



Figure A.3 — Modified UCA vessel with on the right a zoom-in on the pressure transducer (inside view)

The side wall pressure transducer is a piezoresistive pressure transducer which provides a linear voltage output with pressure.

Generate a calibration curve at a minimum of 5 %, 25 %, 50 %, 75 % and 95 % of the full range of the pressure transducer, or the maximum user defined working pressure of the cell, by comparison with a master gauge.

Calibrate the pressure transducer no less frequently than annually. A new calibration shall be carried out whenever repairs or adjustments are made to the device.

The maximum allowable deviation from linearity of the calibration points is  $\pm 1$  % of full range of the pressure transducer.

## A.3 Procedure

### A.3.1 Preparation of the vessel

Prepare the test cell in accordance with the manufacturer's instructions.

### A.3.2 Preparation of slurry

The cement slurry shall be mixed and conditioned in accordance with the procedure described in API Recommended Practice 10B-2.

Condition the cement slurry to simulate placement of the slurry into the wellbore.

### A.3.3 Initiation of the test

After mixing and conditioning, the cement slurry should be poured into the UCA vessel. Follow the detailed operating instructions and safety precautions supplied by the manufacturer. This includes applying a light coating of grease to the pressure transducer ([Figure A.3](#)). This light coating does not influence the measurement and prevents transducer damage when removing the cured cement from the vessel.

Ramp the pressure and temperature to the final conditions using an appropriate schedule to simulate specific well conditions. Once the final temperature and pressure have been reached, maintain them for the duration of the test. The pressure should be ramped to planned test pressure and allowed to stabilize before any hydration of the cement starts.

The duration of the test depends on the application and is decided on a case-by-case basis

### A.3.4 Termination of the test

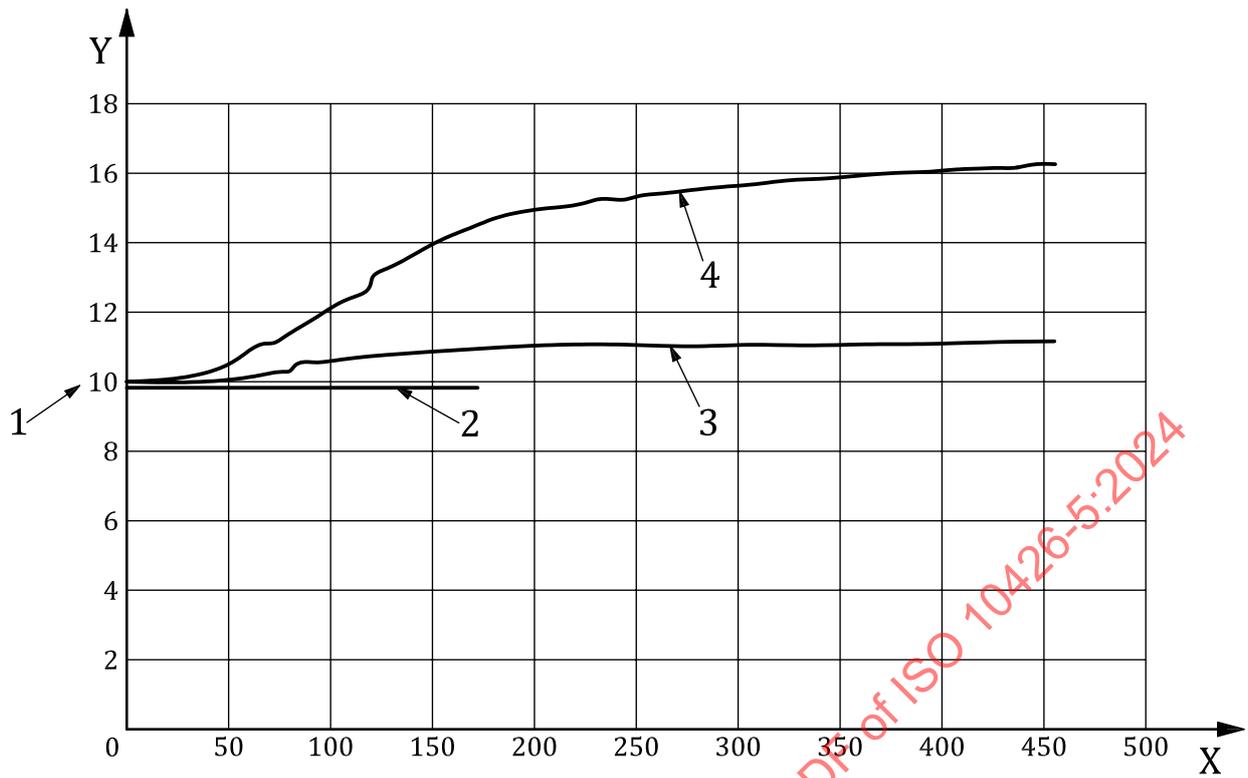
At the end of the test cool the vessel to room temperature while maintaining pressure in the cell. Follow the manufacturer's instructions to remove all pressure from the cell. Then follow the manufacturer's instructions to carefully disconnect the cell and remove the set cement.

## A.4 Measurement

The measurement procedure consists of the following steps.

- a) Confirm that the temperature, pressure, ultrasonic, and radial interface stress signals are correctly being measured after the vessel has been placed in the UCA unit and the sensors connected.
- b) During the pressure and temperature ramp the performance of the wall mounted pressure transducer can be checked as prior to setting of the cement the pressure indicated by the wall mounted transducer should be equal to the UCA pressure.
- c) Record all signals for the duration of the test.

[Figure A.4](#) shows the recorded radial interface stress as a function of time for three cement slurries cured at 10 MPa (1450 lbf/in<sup>2</sup>) and 70 °C (158 °F) in a modified UCA vessel. In case no or limited radial interface stress builds up over time, the chances on the formation of micro-annuli parallel to a plug and abandonment (P&A) plug increase.



**Key**

- |   |                       |   |                                |
|---|-----------------------|---|--------------------------------|
| X | time (hours)          | 2 | well cement recipe without CEA |
| Y | wall stress (MPa)     | 3 | well cement with 3 % BWOC CEA  |
| 1 | curing pressure (MPa) | 4 | well cement with 10 % BWOC CEA |

**Figure A.4 — Typical plot of radial interface stress recorded as a function of time for three slurry types**

[Figure A.5](#) shows an example of the data as generated by the modified UCA vessel.

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