Recommended Practice on Determination of Shrinkage and Expansion of Well Cement Formulations at Atmospheric Pressure

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ISO 10426-5:2003 (Identical), Petroleum and natural gas industries—Cements and materials for well cementing—Part 5: Determination of shrinkage and expansion of well cement formulations at atmospheric pressure

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This American National Standard is under the jurisdiction of the API Subcommittee on Well Cements Sc10. This standard is considered identical to the English version of ISO 10426-5:2004. ISO 10426-5 was prepared by Technical Committee ISO/TC 67, SC 3 drilling completion and well cements.

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Contents

Foreword

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

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

ISO 10426-5 was prepared by Technical Committee ISO/TC 67, *Materials, equipment and offshore structures for petroleum, petrochemical and natural gas industries*, Subcommittee SC 3, *Drilling and completion fluids, and well cements*.

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

— *Part 1: Specification*

— *Part 2: Testing of well cements*

— *Part 3: Testing of deepwater well cement formulations*

— *Part 4: Preparation and testing of foamed cement slurries at atmospheric pressure*

— *Part 5: Determination of shrinkage and expansion of well cement formulations at atmospheric pressure*

Introduction

Dimensional changes in oil- and gas-well cements after placement, phenomena often referred to as shrinkage, (when the dimensional change corresponds to a decrease in cement volume) have often been used to explain various wellbore phenomena including

— a microannulus, leading to a bad bond as demonstrated by the bond log;

— interzonal communication, resulting in costly remedial operations;

— lack of a hydraulic seal when utilizing cement inflatable packers.

Attempts have been made to find additives that decrease cement shrinkage (shrinkage being a fundamental characteristic of Portland cement) The best solution for shrinkage thus far has been the identification of additives that favour the expansion of the cement. However, even if cement expands dimensionally, it will still shrink internally. In this case, the bulk expansion of the cement sample is simply superimposed on an inner shrinkage that will affect the porosity of the sample.

Shrinkage and expansion in cement result from the formation of hydration products having a density different from the compounded density of the reaction components. This can result in the following:

— change in pore volume;

— change in pore pressure;

— change in sample dimensions;

— change in internal stress.

In a closed cell with a non-deformable boundary, the volume of hydrates produced during the chemical reaction is less than the volume of dry compounds plus water. The change in volume of hydrates will be referred to as inner hydration shrinkage. The change in the sample dimensions will be referred to as bulk shrinkage or expansion. Bulk shrinkage and expansion of cement refer to the result of the measurement of 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.

In this part of ISO 10426, units are given as SI, and where practical, U.S. Customary units are included in brackets for information.

Users of this part of ISO 10426 should be aware that further or differing requirements might be needed for individual applications. This part of ISO 10426 is not intended to inhibit a vendor from offering, or the purchaser from accepting, alternative equipment or engineering solutions for the individual application. This can be particularly applicable where there is innovative or developing technology. Where an alternative is offered, the vendor should identify any variations from this International Standard and provide details.

This part of ISO 10426 is based on API Technical Report 10TR 2 [1].

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

Part 5: Determination of shrinkage and expansion of well cement formulations at atmospheric pressure

1 Scope

This part of ISO 10426 provides the methods for the testing of well cement formulations to determine the dimension changes during the curing process (cement hydration) at atmospheric pressure only. This is a base document, because under real well cementing conditions shrinkage and expansion take place under pressure and different boundary conditions.

2 Normative references

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

ISO 10426-2:2003, *Petroleum and natural gas industries — Cements and materials for well cementing — Part 2: Testing of well cements*

3 Terms and definitions

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

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

4 Sampling

4.1 General

Samples of the neat cement or cement blend, solid and liquid additives, and mixing water are required to test a slurry according to this part of ISO 10426. Accordingly, the best available sampling technology should be employed to ensure the laboratory test conditions and materials match as closely as possible those found at the wellsite. Some commonly used sampling devices are shown in ISO 10426-2:2003, Figure 1.

4.2 Method

Applicable sampling techniques for the fluids and materials used are specified in ISO 10426-2:2003, Clause 4.

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

5.1 General information

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 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 mineral growth is strongly controlled by the state of stress and mineral growth will tend to occur where the stress value is the lowest, i.e. in pore space or empty spaces. Therefore, the degree of cement shrinkage and expansion is dependent on a number of conditions, not all of which can be uniquely defined. The test does not represent fully the annulus of a well.

5.2 Apparatus

5.2.1 Mould

5.2.1.1 General

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

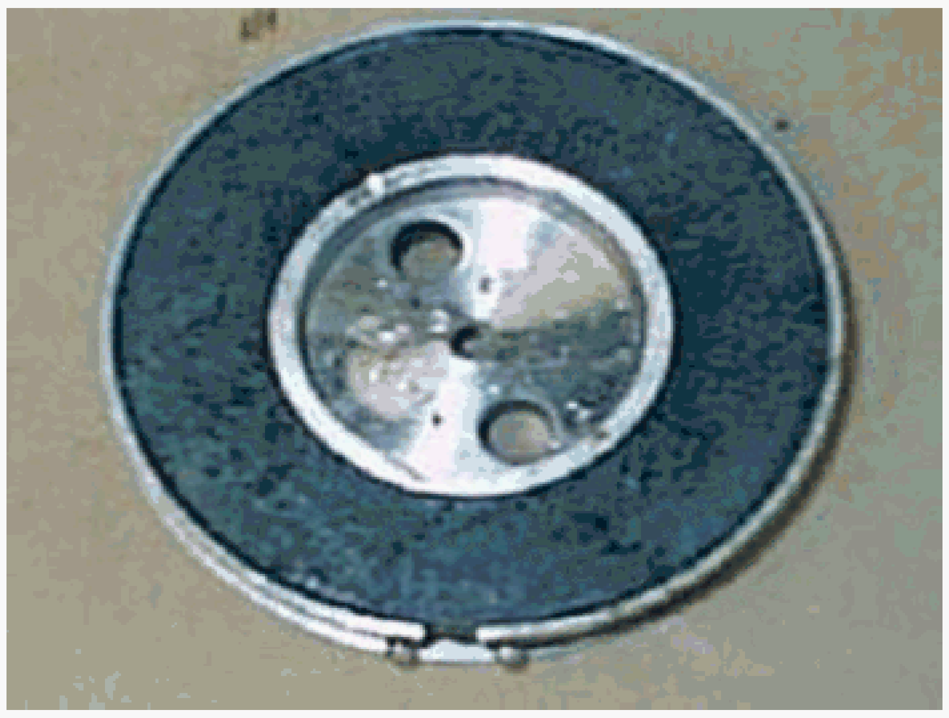


Figure 1 — Typical mould assembly (top view)

Figure 2 — Typical mould assembly (side view)

Dimensions in millimetres (inches)

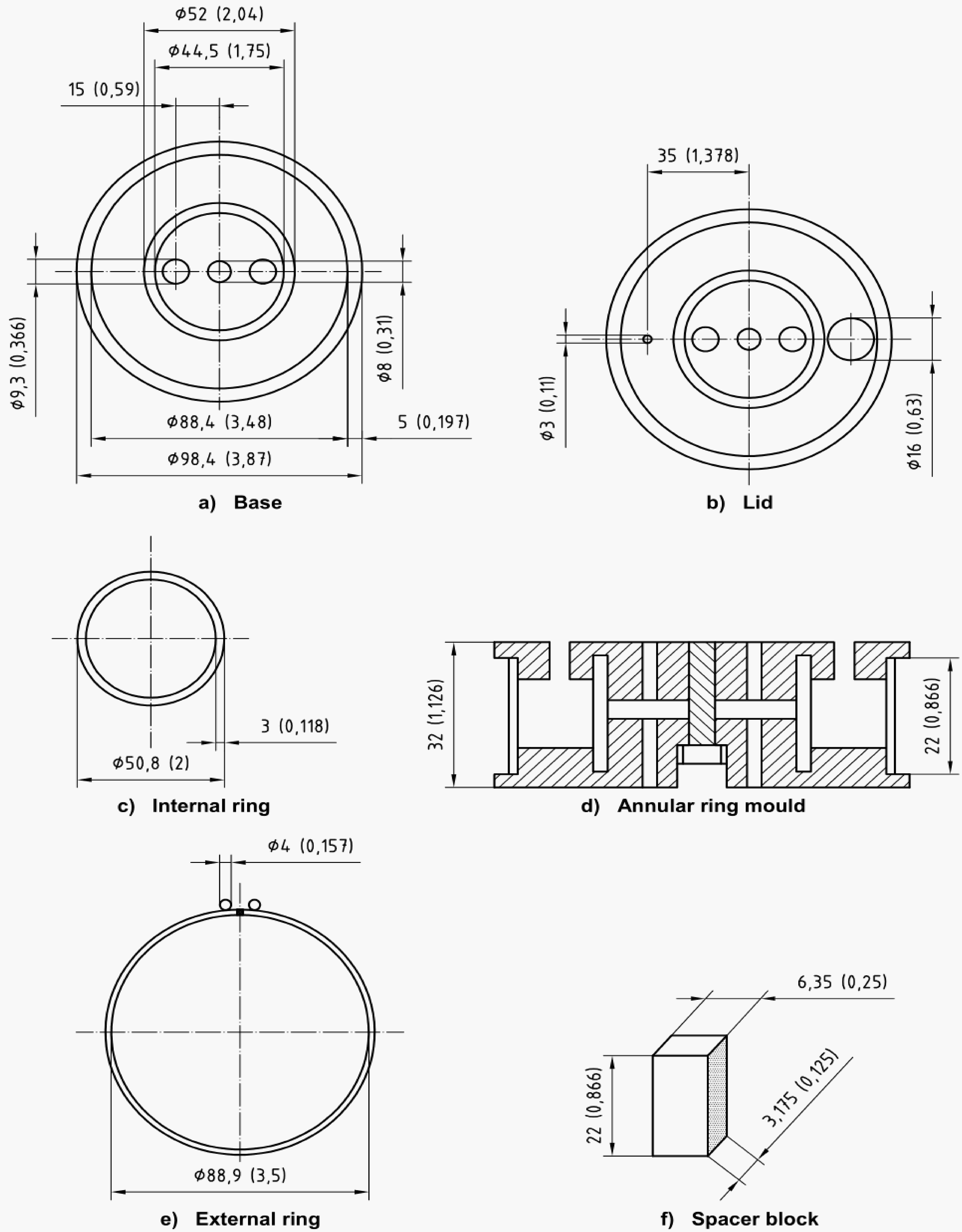


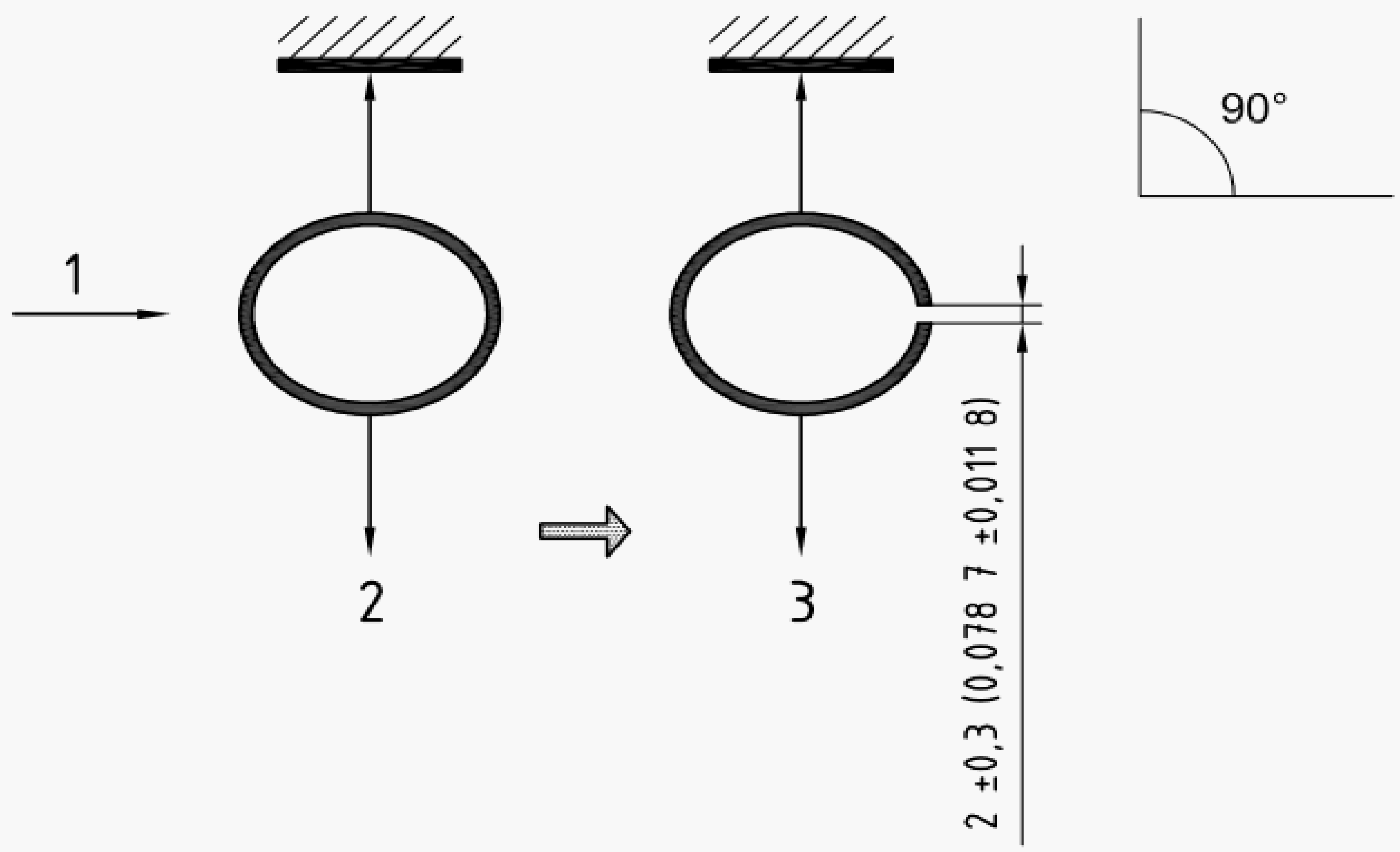
Figure 3 — Schema of typical mould assembly parts

5.2.1.2 Mould calibration

The resilience of the ring of the mould shall be calibrated annually. The resilience shall be such that the mass of 1 000 g ± 1 g (2,204 6 lb ± 0,002 lb) applied as shown in Figure 4 shall increase the distance between the two steel measurement balls (see Figure 9) by 2 mm ± 0,3 mm (0,078 7 in ± 0,011 8 in) without permanent deformation.

Attention must be paid to ensure that the load is applied perpendicular to the gap (90°) in order to avoid errors, which might be easily made. 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



Key

1 ring

2 mass, 0 g

3 mass, 1 000 g ± 1 g (2,204 6 lb ± 0,002 lb)

Figure 4 — Schema of a calibration measurement of the ring — Resilience test

5.2.1.3 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 starts to set. The dimensions of the block shall be 3,175 mm × 3,175 mm to 6,35 mm × 6,35 mm (0,125 in × 0,125 in to 0,25 in × 0,25 in) and 22,0 mm (0,866 in) tall; see Figure 3. 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).

5.2.2 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.

5.2.3 Cooling bath

The cooling-bath dimensions shall be such that the specimen to be cooled from the curing temperature can be completely submerged in water maintained at 27 °C ± 3 °C (80 °F ± 5 °F).

5.2.4 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 monthly. The procedure described in ISO 10426-2:2003, Annex A, is commonly used.

5.2.4.1 Thermometer

A thermometer with a range including 21 °C to 100 °C (70 °F to 212 °F) with minimum scale divisions not exceeding 1 °C (2 °F) should be used.

5.2.4.2 Thermocouple

A thermocouple system with the appropriate range should be used.

5.2.5 Consistometer

The atmospheric-pressure consistometer shall be used for stirring and conditioning the cement slurry. The consistometer consists of a rotating cylindrical slurry container, equipped with an essentially stationary paddle assembly, in a temperature-controlled liquid bath. The consistometer shall be capable of maintaining the temperature of the bath within ± 2 °C (± 3 °F) of the test temperature and of rotating the slurry container at a speed of 2,5 r/s ± 0,25 r/s (150 rpm ± 15 rpm) during the stirring and conditioning period for the slurry. The paddle and all parts of the slurry container exposed to the slurry shall be constructed of corrosion-resistant materials (see ISO 10426-1:2000).

5.3 Procedure

5.3.1 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.

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

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

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

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

g) Verify that the expandable outer ring rotates freely and place the big hole adjacent to the split (Figure 9).

h) Place a small amount of high-temperature grease between the split in the outer ring; the grease will seal the split and prevent the slurry from leaking before it sets.

i) 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 5 and 6.

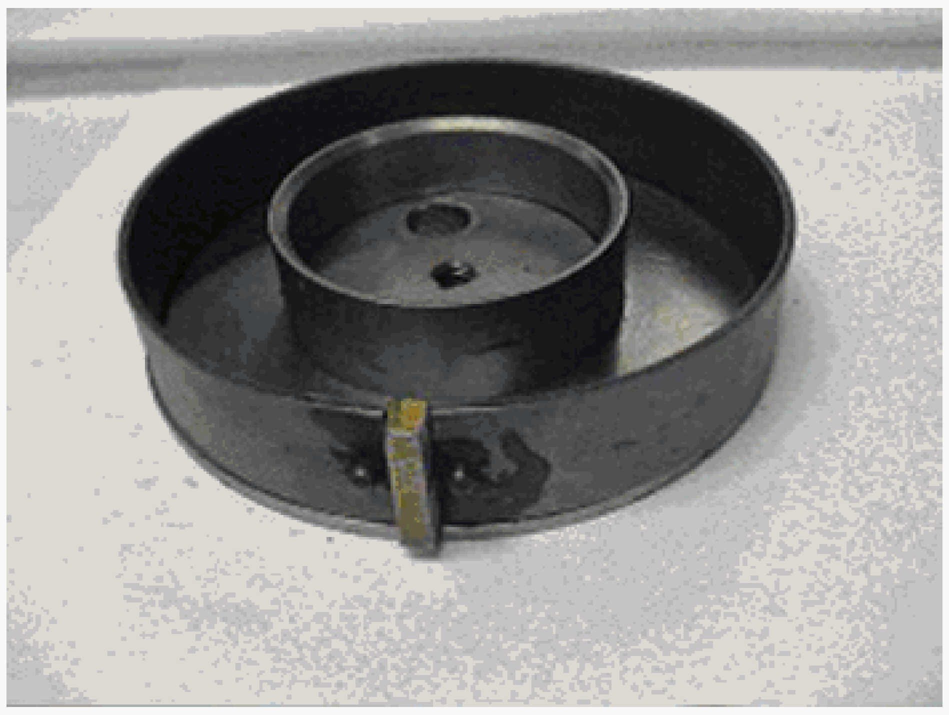


Figure 5 — Mould with a spacer block (top view)

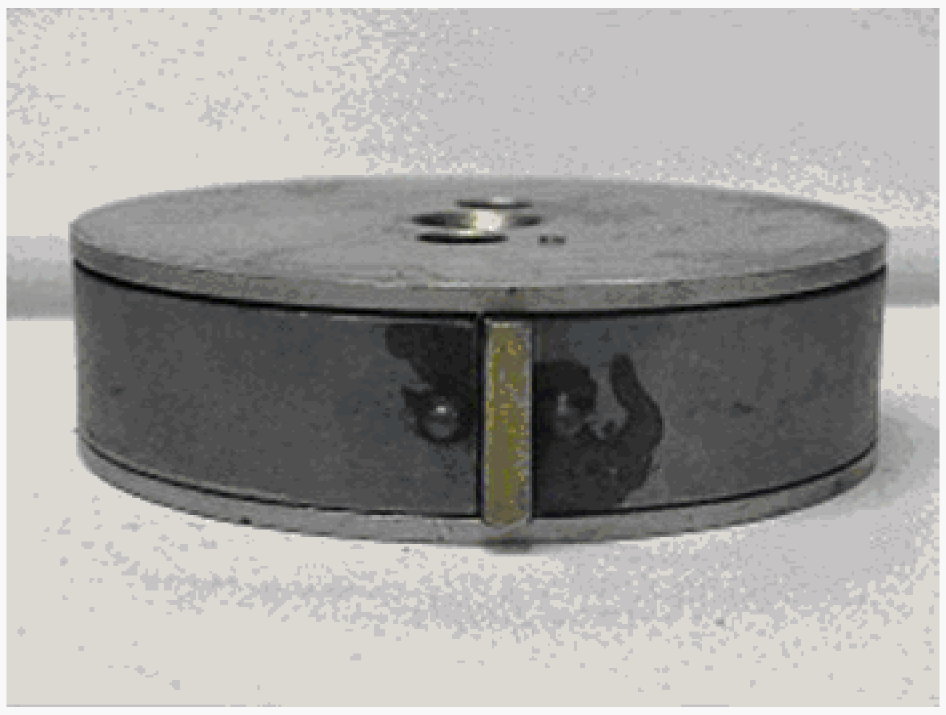


Figure 6 — Mould with a spacer block (side view)

5.3.2 Preparation of slurry

5.3.2.1 Mixing

Test samples shall be prepared in accordance with ISO 10426-2:2003, Clause 5. The mixing device shall be calibrated annually to a tolerance of ± 3,3 r/s (200 r/min) at 66,7 r/s (4 000 r/min) and ± 8,3 r/s (500 r/min) at 200 r/s (12 000 r/min). In addition to accurately controlling the mixing speed, the wear of the mixing blade should be monitored. As such, when the mixer blade has lost more than 10 % of its original mass, it shall be replaced.

If larger slurry volumes are needed, an alternate method for slurry preparation is found in ISO 10426-2:2003, Annex A. As required, the density of the cement slurry can be determined by methods found in ISO 10426-2:2003, Clause 6.

5.3.2.2 Slurry conditioning

After mixing, the cement slurry should be poured immediately into the slurry cup of an atmospheric consistometer for conditioning. The slurry cup should be initially at ambient temperature to avoid the possibility of thermally shocking temperature-sensitive slurries. The slurry shall be then heated to the desired test temperature of up to 88 °C (190 °F). The thickening time schedule that most closely simulates actual field conditions shall be followed; see ISO 10426-2:2003.

5.3.3 Curing at atmospheric pressure

The slurry, after it is heated at the desired test temperature, 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 (preheated to the test temperature in an oven). 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 thicker slurries, the mould might need to be tapped or vibrated to ensure it is completely filled. If the slurry is too thick to pour freely, use a modified syringe to transfer the slurry into the mould through the large hole in the top of the ring mould. To make the modified syringe, cut off the tip of a 60 ml syringe and widen the opening to approximately 6,5 mm to 9,5 mm (1/4 in to 3/8 in). With the plunger inside the syringe, place the syringe into the thick slurry, slowly pull the plunger out to suck the slurry inside the syringe, and then eject it into the large hole of the mould. Repeat this process until the mould is filled completely.

Once the slurry is poured, the mould is placed into a water bath that has been preheated to the test temperature. The slurry is in contact with water during the entire test. After curing, the sample is cooled to below 77 °C (170 °F).

Water entry will compensate for any inner shrinkage as long as the cement matrix is permeable. If the cement expands during the hydration period the outside diameter of ring will expand.

5.3.4 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 periodically to determine only the expansion and not the shrinkage profile.

5.4 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 (mounted on a height-adapter block) is opened and placed on the 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, carefully remove it. 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 atmospheric curing bath or the 77 °C (170 °F) cooling bath to prevent the specimen from cooling significantly and causing an erroneous measurement.

The micrometer shall be mounted on a block that can be placed on a counter top beside the mould so the measuring contact balls on the expandable ring are centred between the micrometer's measuring contacts (Figures 7, 8, 9, 10 and 11). The use of a micrometer without the use of a special mount can result in erratic measurements.

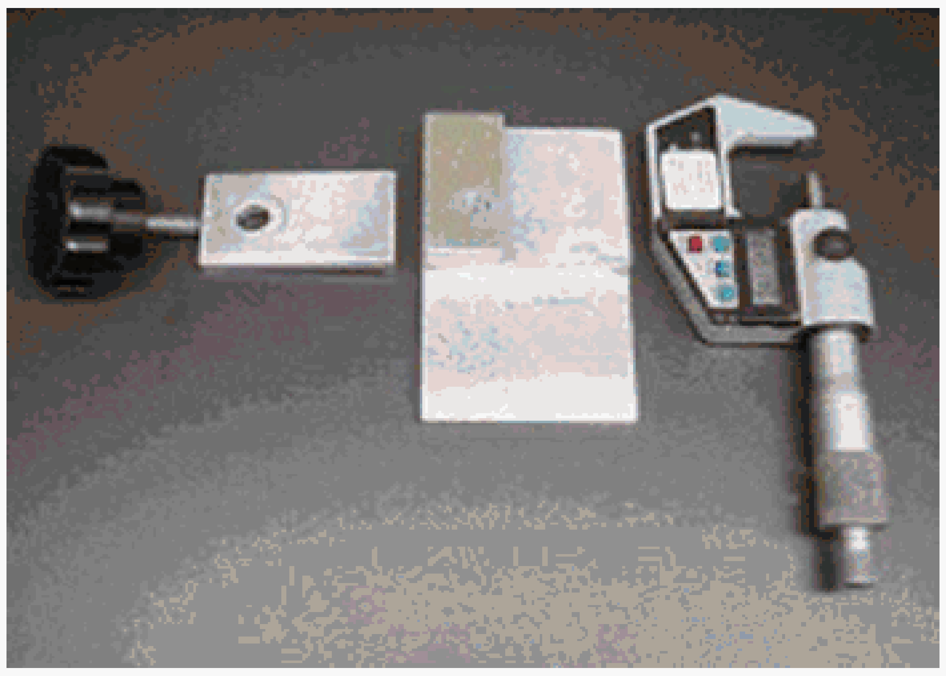


Figure 7 —Micrometer and adapter pieces

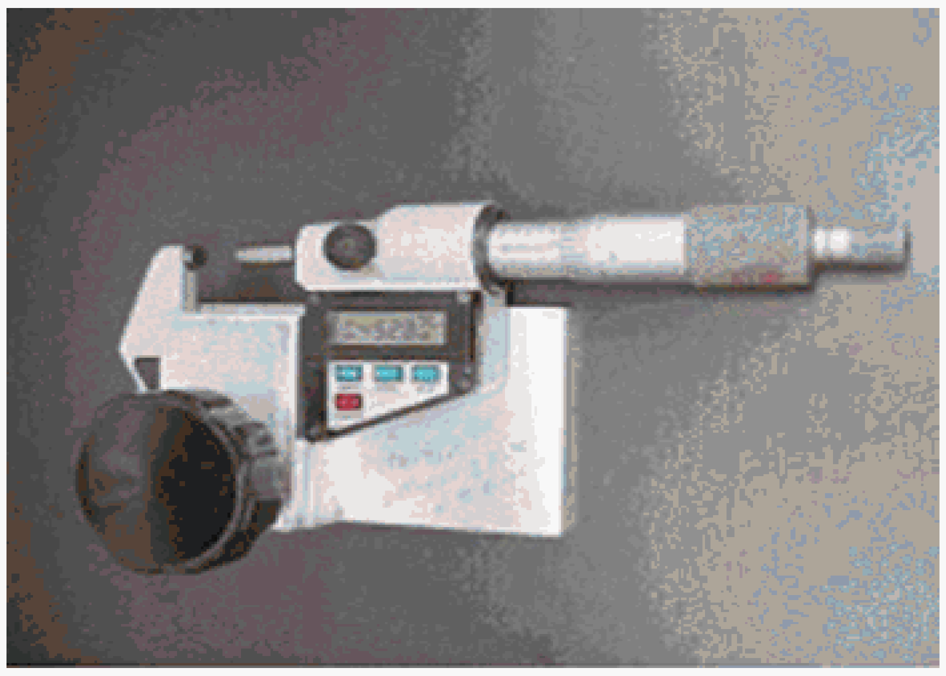


Figure 8 — Micrometer mounted to adapter

Figure 9 — Micrometer measuring

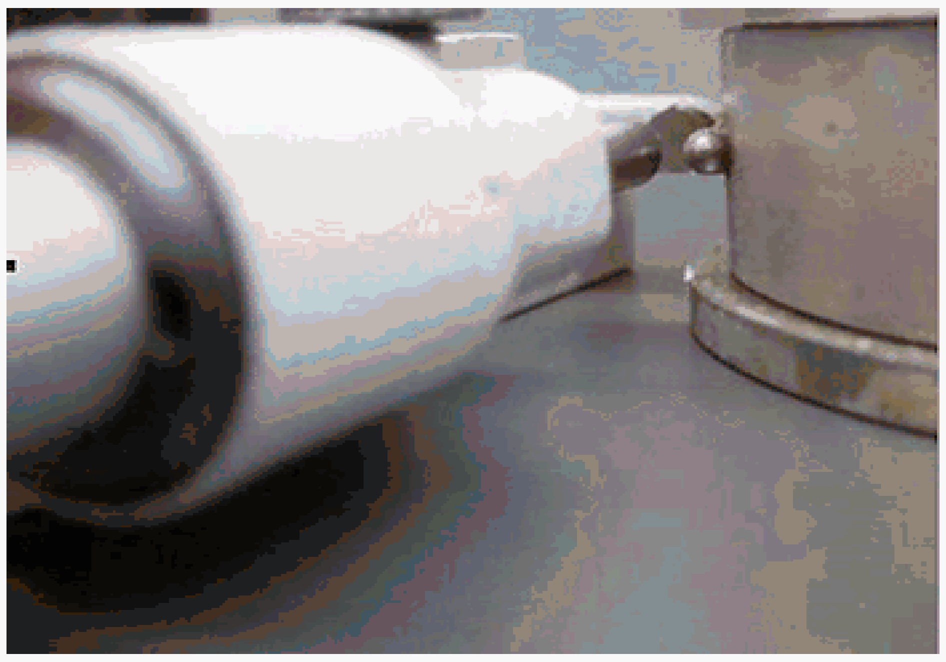
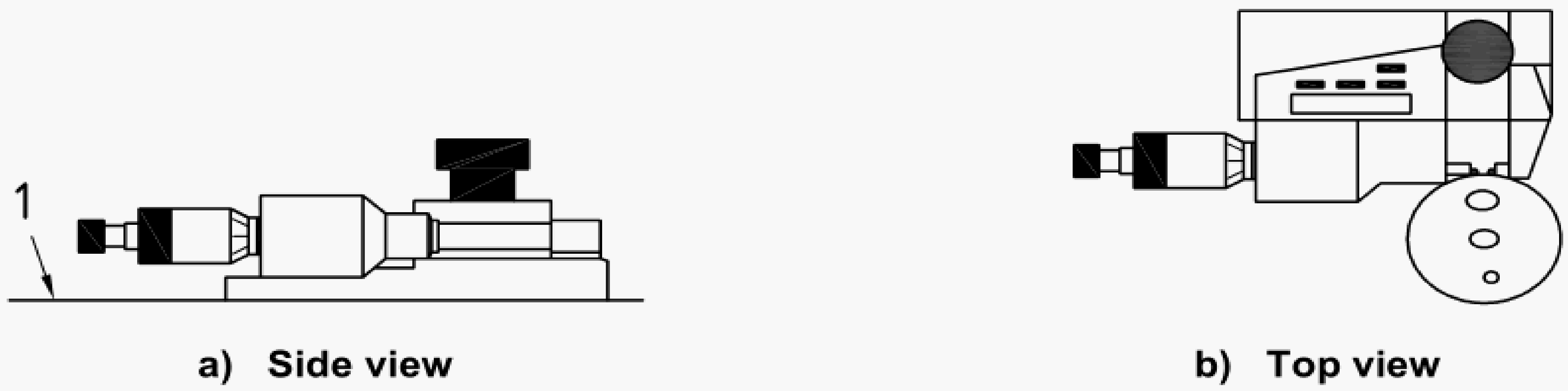


Figure 10 — Micrometer alignment



Key

1 table top

Figure 11 — Scheme of micrometer and adapter pieces

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

$$l_{\Delta,SI} = (l_{f,SI} - l_{i,SI}) \times 0{,}358 \quad (1)$$

where

$l_{\Delta,SI}$ is the circumferential change, expressed in percent, 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:

$$l_{\Delta,US} = (l_{f,US} - l_{i,US}) \times 9{,}095 \quad (2)$$

where

$l_{\Delta,US}$ is the circumferential change, expressed in percent, 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 It is possible to measure either shrinkage or expansion with this test, a positive $l_{\Delta,US}$ or $l_{\Delta,SI}$ indicates expansion, a negative $l_{\Delta,US}$ or $l_{\Delta,SI}$ indicates shrinkage.

The coefficients used in Equations (1) and (2) 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 actually a chord and not an arc, but because the difference is small, it can be neglected. Because of the small distance between the measurement balls, no correction is needed for extrapolating from the initial measurement of the mould at room temperature to the measurement at the actual test temperature. The theoretical correction for the metal used in the moulds is 0,005 % for each 35 °C (100 °F) above the initial room temperature measurement.

The result shall be reported with the corresponding test period.

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

6.1 General information

The purpose of this test method is to measure the bulk expansion or shrinkage when the cement is unable to imbibe 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 ml ± 30 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 ± 2 °C (± 3 °F) of the prescribed test temperatures shall be employed. The water shall be distilled or deionized and shall be de-aerated.

6.2.3 Temperature-measuring system

6.2.3.1 General

The temperature-measuring system shall be calibrated with an accuracy of ± 1 °C (± 2 °F). Calibration shall be no less frequent than monthly. The procedure described in ISO 10426-2:2003, Annex A, is commonly used.

6.2.3.2 Thermometer

A thermometer with a range including 21 °C to 100 °C (70 °F to 212 °F) with minimum scale divisions not exceeding 1 °C (2 °F) should be used.

6.2.3.3 Thermocouple

A thermocouple system with the appropriate range should 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 and placement of slurry

6.3.1.1 Mixing

Test samples shall be prepared according to ISO 10426-2:2003, Clause 5. The mixing device shall be calibrated annually to a tolerance of ± 3,3 r/s (200 r/min) at 66,7 r/s (4 000 r/min) and ± 8,3 r/s (500 r/min) at 200 r/s (12 000 r/min). In addition to accurately controlling the mixing speed, the wear of the mixing blade should be monitored. As such, when the mixer blade has lost more than 10 % of its original mass, it shall be replaced.

If larger slurry volumes are needed, an alternate method for slurry preparation is found in ISO 10426-2:2003, Annex A. As required, the density of the cement slurry can be determined by methods found in ISO 10426-2:2003, Clause 6.

Settling of and free-fluid formation in the slurry shall be avoided with a properly optimized slurry composition. The slurry shall exhibit zero free water.

6.3.1.2 Placing the slurry in the membrane

Condition the slurry in the atmospheric consistometer (see 5.2.5) for a period of 20 min ± 30 s. The bath temperature shall be maintained at 27°C ± 2 °C (80 °F ± 3 °F) throughout the stirring period.

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

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

In order to improve the impermeability of the membrane, the internal and external surface shall be covered with a thin layer of silicon grease; do not use spray grease, since the solvent can adversely affect the impermeability of membrane.

The volume of slurry shall be 150 ml ± 30 ml. Determine the mass in air of the sealed membrane containing cement with a precision of ± 0,01 g.

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

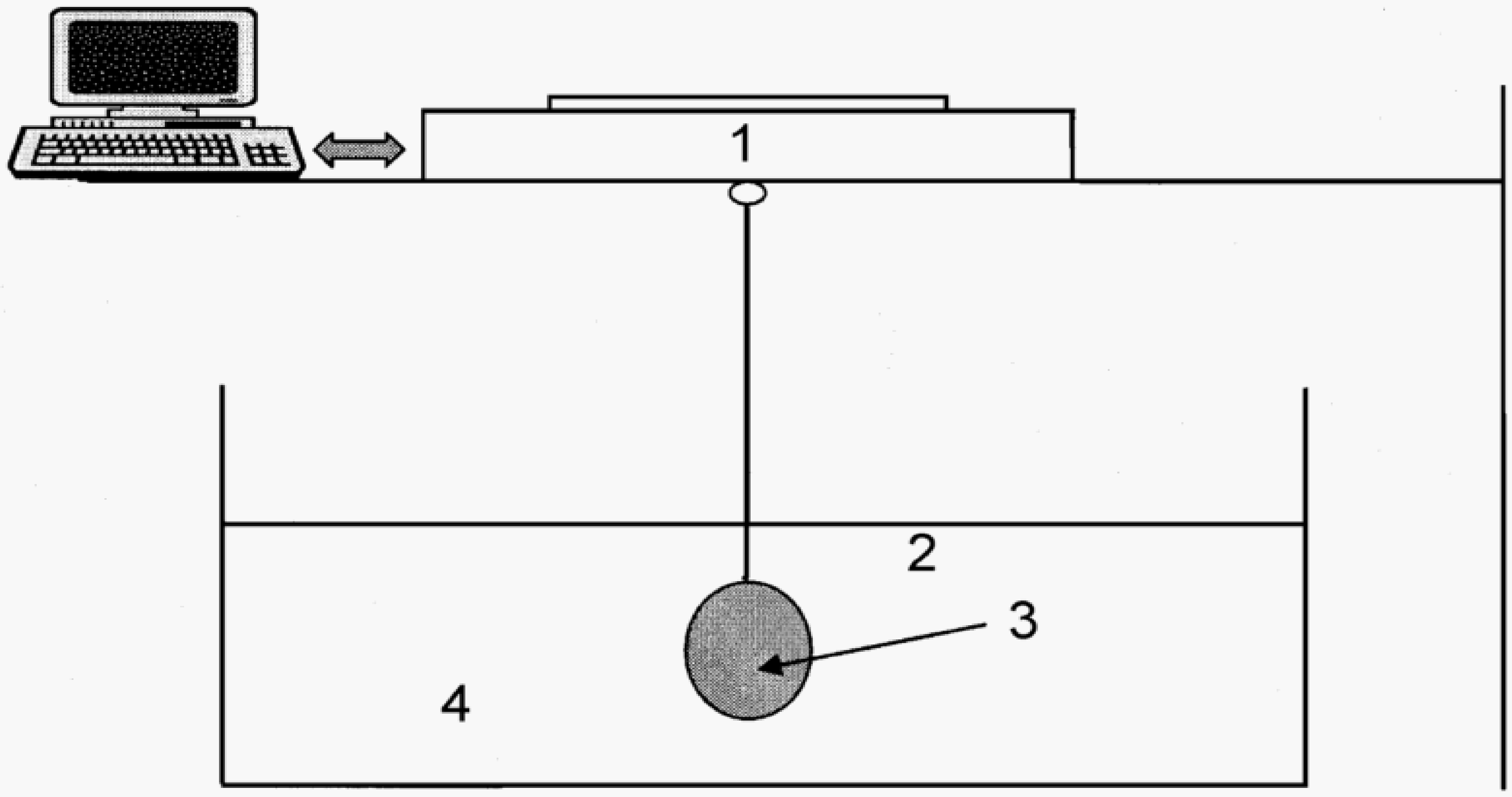
6.3.2 Curing

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 preventing air from becoming attached to the external surfaces of the membrane or the net.

The amount of water lost through evaporation in the bath is not critical at 35 °C (95 °F). It is recommended to place a thin layer of oil on the water to minimize evaporation. The water evaporation becomes significant at higher temperatures. A reference level is chosen for the water bath and water at the same test temperature ± 2 °C (± 3 °F) is regularly added.

If water penetrates into the sample during the curing due to leaks in the membrane, this will give the appearance of strongly accelerated shrinkage. It is important at this point to verify the integrity of the membrane (some of them dissolve after a few days' exposure to the cement) and repeat the test with a new membrane (see 6.2.1).



Key

1 data-acquisition system

2 balance or load cell

3 slurry in impermeable membrane

4 thermostated bath

Figure 12 — Scheme 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 times the acceleration of gravity, minus the buoyancy of the water. The buoyancy force, F_b. is equal to the volume of the membrane containing the cement times the specific density of water, e.g. 1,00.

The initial measured mass, m_{im}, is recorded immediately after the membrane is immersed in the water bath; subsequent measured weights are recorded every 10 min with a precision of $\pm$ 0,01 g. 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.

The percent bulk change (shrinkage or expansion) is calculated in accordance with Equations (3) to (5):

$$V_{bc} = 100 \times (V_f - V_i) / V_i \quad (3)$$

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.

$$V_i = m_a - m_{im} \quad (4)$$

where

m_a is the mass, expressed in grams, in air of the membrane containing the cement slurry;

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

$$V_f = m_a - m_{fm} \quad (5)$$

where

m_{fm} is the final measured mass, 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.

Bibliography

[1] API Technical Report 10TR 2, *Shrinkage and Expansion in Oilwell Cements*, First Edition, July 1997

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