

Recommended Practice on Testing of Deepwater Well Cement Formulations

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



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API Foreword

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

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Foreword

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

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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 10426-3 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*

The following part is under preparation:

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

Introduction

The test methods contained in this part of ISO 10426, though generally based on ISO 10426-2, take into account the specialized sampling/testing requirements and unique downhole temperature profiles found in deepwater wells. ISO 10426-2 contains no applicable well simulation schedules for deepwater cementing operations.

In a deepwater cementing environment, a number of factors impact the thermal history of the cement slurry. These factors include: water depth, mud-line temperature, geothermal gradient, the presence or absence of a drilling riser, drilling fluid temperature, ocean current velocity, presence of thermoclines (layers of ocean water separated by temperature), ambient sea-surface temperature, cement mix-water temperature, bulk cement temperature, cement mixing rate, cement heat of hydration, displacement rate, prior circulating and static event history, drill pipe size and mass, casing size and mass, and hole size.

Given the number of variables impacting the thermal history of a cement formulation during placement and curing, and the interdependence of many of those variables, the user is directed to employ numerical heat-transfer simulation or actual field measurement to determine the test temperature and the temperature/pressure schedule for the test methods contained in this part of ISO 10426. In this way, the testing of the cement formulation can reflect as closely as possible the actual temperature profile found during field cementing operations.

Numerical modelling may be used to determine the relative magnitude of the input variables so that “most likely” and “less likely” scenarios of temperature history can be assessed. The values of some input variables may not be known precisely and a range of possible values should be employed. Physical laboratory testing can then be conducted at “most likely” conditions, with some additional testing at “less likely” conditions to determine the sensitivity to well conditions. Sound engineering judgement can then be applied to assess the risks.

These procedures serve not only for the testing of well cements under deepwater well conditions, but may also be used in those circumstances where low seafloor temperatures are found at shallow water depths.

Well cements that can be used in deepwater well cementing can include those of ISO Classes A, C, G or H (as given in ISO 10426-1^[1]), high-alumina cement, appropriate foamed cements, various types of ductile cement compositions, etc. In each deepwater well cementing operation, the cement chosen needs to be fit for purpose.

In this part of ISO 10426, where practical, United States customary (USC) units are included in parentheses for information.

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

Part 3: Testing of deepwater well cement formulations

1 Scope

This part of ISO 10426 provides procedures for testing well cements and cement blends for use in the petroleum and natural gas industries in a deepwater environment.

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*

ASTM C 109, *Standard test method for compressive strength of hydraulic cement mortars*

3 Terms and definitions

For the purposes of this part of ISO 10426, the terms and definitions given in ISO 10426-2 apply.

4 Sampling

Samples of the neat cement or cement blend, solid and liquid additives, and mixing water are required to test a slurry in accordance with 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 well site. Additionally, the temperature of the mix water, cement or cement blends should be measured with a thermocouple or thermometer capable of measuring temperature with an accuracy of $\pm 2\text{ }^{\circ}\text{C}$ ($\pm 3\text{ }^{\circ}\text{F}$). These temperatures should be recorded. Temperature-measuring devices shall be calibrated (in the case of a thermocouple) or checked (in the case of a thermometer) annually.

NOTE Some commonly used sampling devices and techniques can be found in ISO 10426-2.

5 Preparation of slurry

5.1 Preparation of conventional cement slurry

Prepare the test samples in accordance with Clause 5 of ISO 10426-2:2003. The laboratory temperature of the cement sample, additives, and mix water should be within $\pm 2\text{ }^{\circ}\text{C}$ ($\pm 3\text{ }^{\circ}\text{F}$) of the respective temperature anticipated at the well site. The temperature of the mixing container should approximate that of the mix water being used in the slurry design. The mixing device shall be calibrated annually to a tolerance of $\pm 200\text{ r/min}$ at 4 000 r/min rotational speed and $\pm 500\text{ r/min}$ at 12 000 r/min rotational speed.

If larger slurry volumes are needed, an alternative method for slurry preparation can be found in Annex A of ISO 10426-2:2003.

NOTE The density of the cement slurry can be determined by methods found in Clause 6 of ISO 10426-2:2003.

5.2 Preparation of speciality cement slurries

Cementing operations in deepwater environments may require the use of speciality cements, including foamed cement or microsphere-containing cement formulations. The preparation and testing of these speciality cement formulations should be undertaken using the best available methods or methods mutually agreed upon by the service provider and the end user.

6 Strength tests for deepwater well cements

6.1 General information

The strength development of cement used in a deepwater cementation can be influenced by many factors, including heat of hydration, casing/wellbore size, final slurry location (annulus or shoe track), and initial slurry temperature. Given the number of variables contributing to the rate of strength development in a deepwater well, the temperature and pressure schedule should be determined by means of numerical heat transfer simulation or by field measurement from an offset well(s). In this way, the test schedule can reflect as closely as possible the actual temperature and pressure profiles found after placement.

The preferred method for determining the strength of deepwater cement is by means of a non-destructive sonic test method. Non-destructive sonic testing of a cement slurry may be conducted by methods provided in Clause 8 of ISO 10426-2:2003. Speciality cement systems, as described in 5.2, may employ destructive testing to determine compressive strength.

The energy produced by hydrating cement generates a considerable amount of heat. In a large annulus typically found in the top-hole section of a deepwater well, it is expected that the hydration exotherm (thermal mass effect) may raise the temperature within the annulus significantly. As such, a general guideline is to conduct the strength test at a low curing temperature for only as long as the cement remains unset. Once initial strength of 345 kPa (50 psi) is reached, the curing temperature may be raised to reflect the hydration exotherm.

6.2 Sampling methods

The sampling methods for strength testing are provided in Clause 4.

6.3 Preparation of slurry

The slurry shall be prepared in accordance with Clause 5.

6.4 Non-destructive sonic testing

6.4.1 Apparatus for non-destructive testing

CAUTION — Care should be exercised to ensure that excess condensation caused by chilling the test apparatus does not cause electrical or other damage, which may create safety hazards.

The apparatus transmits a sonic signal through the cement, which can be correlated to cement properties such as the time and extent of strength development. In order to simulate conditions common to deepwater cementing, the apparatus shall possess sufficient cooling capacity to perform strength testing at temperatures anticipated in the wellbore.

Excessive free fluid can impair the accuracy of the non-destructive sonic test. Free fluid in a slurry can inhibit contact with the top cell cover and affect the sonic signal being transmitted through the cement. Free fluid is determined according to the method provided in Clause 9. The test initiation temperature of the slurry should reflect as closely as possible the temperature conditions found during the field mixing operation.

6.4.1.1 Curing cell, in which the slurry temperature and pressure can be controlled according to the appropriate schedule.

A pressure vessel suitable for curing samples at a test temperature anticipated on the well and capable of maintaining pressure shall be used. As pressure is known to have an effect on strength development, the pressure appropriate for the placement conditions should be used for testing. Do not exceed the pressure limitations of the apparatus.

6.4.1.2 Temperature-measuring system, in accordance with 8.2.1 of ISO 10426-2:2003.

6.4.1.3 Sonic signal-measuring system, in accordance with 8.2.2 of ISO 10426-2:2003.

6.4.2 Procedure

Operate the apparatus according to the manufacturer's instructions. To better simulate the temperature profile found in a deepwater well, ramp the curing cell temperature from surface mixing temperature to the desired test temperature according to a specific schedule determined by thermal simulation. Alternatively, chill the curing cell to the desired test temperature or below, before the slurry is placed into the curing cell. The slurry may also be conditioned in accordance with 6.4.3 or 6.4.4.

The test period begins with the recording of sonic data and the application of pressure and continues until the test is terminated. Begin recording the sonic data within 5 min after the application of pressure. The pressure ramp should simulate the pressure conditions to which the cement shall be exposed during placement.

6.4.3 Conditioning of consistometer for atmospheric pressure testing

After the slurry has been prepared, place it into an atmospheric consistometer slurry container that has been chilled to the desired test temperature. At user discretion, the temperature of the slurry container and/or the cooling fluid within the atmospheric consistometer may be lower than the bottomhole test temperature in order to promote a more rapid cool-down. Place the cup into a chilled atmospheric consistometer and condition for 20 min. After 20 min, verify the temperature, remove the paddle from the cup and stir the slurry briskly with a spatula to ensure a uniform slurry. If the cement slurry has not reached the desired test temperature, continue conditioning until the desired test temperature has been reached. It is permissible for the sample to be further conditioned for a period of time to simulate placement of the slurry into the well. Note the actual time to reach the desired test temperature and time of further conditioning.

6.4.4 Conditioning of pressurized consistometer

After the slurry has been prepared, place it into a pressurized consistometer slurry container that has been chilled to the desired test temperature. At user discretion, the temperature of the slurry container and/or the oil within the pressurized consistometer may be lower than the bottomhole test temperature in order to promote a

more rapid cool-down. Place the slurry container in the pressurized consistometer and ramp the sample to the desired test temperature and pressure. Once the desired test temperature and pressure is reached, it is permissible for the sample to be further conditioned for a period of time to simulate placement of the slurry in the well. Before transferring the sample to the non-destructive apparatus, blot any oil that may have invaded the pressurized consistometer slurry container during the conditioning period. Note the actual time to reach the desired test temperature and pressure and time of further conditioning.

6.5 Destructive testing

6.5.1 Sampling methods

The methods provided in Clause 4 shall be used for obtaining samples for strength testing.

6.5.2 Preparation of slurry

The slurry shall be prepared in accordance with Clause 5.

6.5.3 Conditioning of consistometer for atmospheric pressure testing

After the slurry has been prepared, place it into a consistometer slurry container for atmospheric pressure testing that has been chilled to the desired test temperature. At user discretion, the temperature of the slurry container and/or the cooling fluid within the atmospheric pressure consistometer may be lower than the bottomhole test temperature in order to promote a more rapid cool-down. Place the cup into a chilled atmospheric pressure consistometer and condition for 20 min. After 20 min, verify the temperature, remove the paddle from the cup and stir the slurry briskly with a spatula to ensure a uniform slurry. If the cement slurry has not reached the desired test temperature, continue conditioning until the desired test temperature has been reached. It is permissible for the sample to be further conditioned for a period of time to simulate placement of the slurry into the well. Note the actual time to reach the desired test temperature and time of further conditioning.

6.5.4 Conditioning of pressurized consistometer

After the slurry has been prepared, place it into a pressurized consistometer slurry container that has been chilled to the desired test temperature. At user discretion, the temperature of the slurry container and/or the oil within the pressurized consistometer may be lower than the bottomhole test temperature in order to promote a more rapid cool-down. Place the slurry container in the pressurized consistometer and ramp the sample to the desired test temperature and pressure. Once the desired test temperature and pressure is reached, it is permissible for the sample to be further conditioned for a period of time that simulates placement of the slurry into the well. Before placement of the sample into the cube moulds, blot any oil that may have invaded the pressurized consistometer slurry container during the conditioning period. Note the actual time to reach the desired test temperature and pressure and time of further conditioning.

6.5.5 Apparatus and reagents for destructive testing

6.5.5.1 Cube moulds, in agreement with the principles defined in 7.4.1 of ISO 10426-2:2003.

Moulds of other dimensions and geometries are permissible provided the dimensions or geometries are reported.

NOTE Compressive strengths determined with one mould dimension or geometry may not correspond to those using a different mould dimension or geometry.

6.5.5.2 Compressive strength-testing device, in accordance with 7.4.6 of ISO 10426-2:2003.

6.5.5.3 Base and cover plates, in accordance with 7.4.6 of ISO 10426-2:2003.

Base and cover plates for moulds with dimensions or geometries other than those defined in 7.4.6 of ISO 10426-2:2003 shall follow the principles defined in 7.4.6 of ISO 10426-2:2003.

- 6.5.5.4 Puddling rod**, in accordance with 7.4.6 of ISO 10426-2:2003.
- 6.5.5.5 Mould-sealing grease**, in accordance with 7.4.7 of ISO 10426-2:2003.
- 6.5.5.6 Mould-release agent**, in accordance with 7.4.8 of ISO 10426-2:2003.
- 6.5.5.7 Atmospheric pressure curing apparatus (bath)**, for curing cement samples (optional).

Because this test method does not simulate the actual downhole pressure conditions, the results obtained from this method can vary from those of the ultrasonic or pressurized curing method. The apparatus (bath) should be equipped with an agitator or circulating system. At temperatures above 0 °C (32 °F), water may be used as the curing medium. The atmospheric pressure vessel (bath) shall be capable of cooling the samples to the desired temperature and maintaining this temperature for the duration of the test. If glycol or mineral oil serves as the cooling medium for curing at temperatures below 0 °C (32 °F), seal the test specimens in a container of water to avoid contamination by the glycol or mineral oil. If the slurry contains freeze-depressants, then the same type and concentration of freeze-depressant as used in the slurry may be added to the water. Submerge the sealed container in an atmospheric pressure apparatus (bath) in such a way as to avoid contamination of the water and specimens by the cooling medium. A thermocouple, range –18 °C to 104 °C (0 °F to 220 °F) calibrated to an accuracy of ± 1 °C (± 2 °F) is preferred in a non-pressurized apparatus. A thermometer, scale range –18 °C to 104 °C (0 °F to 220 °F) calibrated to an accuracy of ± 1 °C (± 2 °F) may be used in a non-pressurized apparatus.

6.5.5.8 Pressurized curing apparatus, suitable for curing samples typically at a test temperature at or above –7 °C (20 °F) and capable of maintaining a pressure of at least 20 700 kPa (3 000 psi).

The apparatus shall be capable of cooling the samples to the testing temperature and maintaining that temperature for the duration of the test. If glycol or mineral oil serve as the cooling medium for curing at temperatures below 0 °C (32 °F), seal the test specimens in a container of water to avoid contamination by the glycol or mineral oil. If the slurry contains freeze depressants; add to the container of water the same type and concentration of freeze depressant as used in the slurry. The thermocouple in the pressurized curing apparatus shall have a range of –18 °C to 204 °C (0 °F to 400 °F) and be calibrated to an accuracy of ± 2 °C (± 3 °F).

6.5.6 Procedure

6.5.6.1 Curing schedule

The temperature of the slurry should be cooled/heated on a schedule to simulate the conditions of curing.

6.5.6.2 Preparation of moulds

Coat the interior faces of the moulds and contact surfaces of the plates with a mould release agent. The assembled moulds shall be watertight. The moulds may be pre-chilled before being filled with slurry to allow for a more rapid cool-down of the slurry to the desired test temperature.

6.5.6.3 Placement

After preparation and conditioning, pour the cement slurry into the moulds to one-half of the mould depth. Puddle each sample approximately 30 times with a puddling rod after all chambers have received slurry. Stir the remaining slurry by hand. Completely fill each sample mould with slurry and puddle as before. After the chambers are completely filled and puddled, place the cover plate on top of the moulds. Visually verify that the moulds are not leaking.

6.5.6.4 Curing in the atmospheric pressure vessel (bath)

After the moulds have been filled with cement slurry and covered with the top plate, immediately place them in the atmospheric pressure curing vessel (or bath). To better simulate the temperature profile found in a deepwater well, the curing temperature can be ramped from surface mixing temperature to the desired test

temperature according to a specific schedule determined by thermal simulation. Alternatively, the moulds may be placed in the atmospheric curing vessel (or bath) which has been pre-chilled at or slightly below the desired test temperature. At user discretion, the temperature of the fluid in the curing vessel (bath) may be raised from the original test temperature in order to simulate the effects of heating caused by the hydration exotherm. Note the actual time and temperature schedule.

6.5.6.5 Curing in the pressurized vessel

After the moulds have been filled with cement slurry and covered with the top plate, immediately place them in the pressurized curing vessel. To better simulate the temperature profile found in a deepwater well, the curing temperature can be ramped from surface mixing temperature to the desired test temperature according to a specific schedule determined by thermal simulation. Alternatively, the moulds may be placed in the pressurized curing vessel, which has been pre-chilled to or slightly below the desired test temperature. Apply pressure consistent with that anticipated during the actual cementing operation. (If the anticipated downhole pressure is greater than the operating pressure limit of the apparatus, hold the final pressure at the operating pressure limit). Do not exceed the operating pressure limits of the apparatus. Note the actual time, pressure and temperature schedules. Within the pressure limitations of the apparatus, the pressure ramp should simulate the conditions under which the cement can be exposed during placement.

6.6 Strength determination

6.6.1 General

Upon completion of the test period, release the pressure gradually (if applicable) and remove the moulds from the vessel. Remove the cement samples from the moulds and immediately test the sample in accordance with ASTM C 109.

6.6.2 Procedure

Use a compressive strength-testing device. The rate of loading for samples with an anticipated strength of greater than 3,5 MPa (500 psi) should be $71,7 \text{ kN/min} \pm 7,2 \text{ kN/min}$ ($4\,000 \text{ psi/min} \pm 400 \text{ psi/min}$). For anticipated strengths of less than 3,5 MPa (500 psi), the rate of loading for samples should be $17,9 \text{ kN/min} \pm 1,8 \text{ kN/min}$ ($1\,000 \text{ psi/min} \pm 100 \text{ psi/min}$).

Report the compressive strength as the force required to break the sample divided by the smallest cross-sectional area in contact with the load-bearing plates of the compression tester. In accordance with ASTM C 109, average the compressive strength of all the acceptable test samples made from the same slurry and tested at the same time. Report compressive strength to the nearest 0,1 MPa (10 psi).

Additional information to be recorded includes

- a) initial temperature,
- b) final temperature,
- c) initial pressure,
- d) final pressure
- e) time to final temperature and pressure,
- f) any other temperature and pressure variations to reflect cement placement and curing conditions.

The samples of cement should be visually inspected prior to testing. Discard any samples that have been damaged during removal from the moulds or exhibit cracking or spalling.

7 Thickening-time tests for deepwater well cements

7.1 General

The thickening-time test provides an indication of the length of time that a cement slurry will remain pumpable in a well. The laboratory test conditions should represent, as closely as possible, the time, temperature and pressure conditions to which a cement slurry will be exposed during placement in the well.

7.2 Sampling

The methods provided in Clause 4 shall be used for obtaining samples for thickening-time tests.

7.3 Preparation of slurry

The cement slurry shall be prepared in accordance with Clause 5.

7.4 Apparatus

7.4.1 Pressurized consistometer, commonly incorporating a rotating cylindrical slurry container equipped with a stationary paddle assembly, all enclosed in a pressure vessel capable of withstanding the simulated well pressures.

Alternatively, rotating paddle type consistometers as described in Annex D of ISO 10426-2:2003 may be used. In order to simulate conditions common to deepwater cementing, the apparatus shall possess sufficient cooling capacity to perform thickening-time tests at temperatures below ambient.

Calibrate pressurized consistometers in accordance with 9.3 of ISO 10426-2:2003.

7.4.2 Temperature and pressure control devices.

During the test, the conditions of pressure and temperature placed on the cement slurry should simulate the anticipated profiles found during the placement of the slurry. Record the temperatures and pressures found during the test.

Programmable devices shall be used to control the test temperature and record the measured parameters. The temperature of the cement slurry shall be determined by the use of a Type J thermocouple (ASTM classification, special) located in the centre of the slurry container. The tip of the thermocouple shall be vertically positioned between 44,5 mm (1,75 in) and 88,9 mm (3,50 in) above the inside of the base of the slurry container.

As there are many models of consistometers having different dimensions, care should be taken to ensure that the thermocouple used is compatible with the consistometer and the position of the tip of the thermocouple is in the correct location as specified above.

7.5 Test procedures

7.5.1 Consistency

Slurry consistency shall be as defined and determined in accordance with 9.3.1 of ISO 10426-2:2003.

7.5.2 Operating instructions/assembling and filling the slurry container

Operating instructions, assembling and filling the slurry container shall comply with 9.4.1 and 9.4.2 of ISO 10426-2:2003, except that the slurry container may be pre-chilled to or below the desired test temperature to allow for a more rapid ramp-down of the slurry to the desired test temperature.

7.5.3 Initiation of test

Start the test within 5 min after slurry preparation. Place the slurry container on the drive table in the pressure vessel, and start rotation of the slurry container. Secure the potentiometer mechanism so as to engage the paddle shaft drive bar. When properly engaged, the paddle shaft should not be rotating. Finish filling the vessel with oil and secure the head assembly. Insert the thermocouple through the hole in the head assembly and partially tighten the threads. After the pressure vessel is completely filled with oil, tighten the threads of the thermocouple. Record the initial temperature reading and slurry consistency in Bearden units of consistency (B_c).

In order to more closely replicate the temperature profile found during deepwater cementing operations, the pressurized consistometer pressure vessel and oil may be pre-chilled.

7.5.4 Thickening time

The thickening time is the time elapsed from the initial application of pressure to the time at which the slurry reaches a consistency deemed sufficient to make it unpumpable, such as 70 B_c or 100 B_c . Record the slurry consistency at which the thickening time was determined, the thickening time, the surface retention time and pressure/temperature schedules.

7.5.5 Surface mixing of slurry

If batch mixing is used for the cementing operation, stir the slurry in an atmospheric pressure consistometer for a time period and temperature reflective of conditions expected at the well site. Once the batch mix period has been completed, transfer the slurry to a pressurized consistometer and initiate the test in accordance with 7.5.1. A pressurized consistometer may be used to simulate the batch mixing of the slurry, provided the temperature of the cement slurry remains at the temperature anticipated at the time of mixing and that no pressure is applied during the simulated surface-mixing period.

8 Static or stirred fluid-loss test

8.1 Apparatus

8.1.1 Fluid-loss cell, equipped with a means of cooling.

8.2 Sampling

The methods provided in Clause 4 shall be used for obtaining samples for fluid-loss testing.

8.3 Preparation of slurry

The cement slurry shall be prepared in accordance with Clause 5.

8.4 Conditioning procedures

8.4.1 Conditioning of consistometer for atmospheric pressure testing

After the slurry has been prepared, place it into a consistometer slurry container for atmospheric pressure testing that has been chilled to the desired test temperature. Place the cup into the chilled atmospheric pressure consistometer and condition for 20 min. After 20 min, verify the temperature, remove the paddle from the cup and stir the slurry briskly with a spatula to ensure a uniform slurry. If the cement slurry has not reached the desired test temperature, continue conditioning until the desired test temperature has been reached. Note the actual time to reach the desired test temperature. At user discretion, the temperature of the slurry container and/or the fluid within the atmospheric pressure consistometer may be lower than the bottomhole test temperature in order to promote a more rapid cool-down. Once the desired test temperature is

reached, the sample may also be conditioned for a period of time that simulates the placement of the slurry in the well.

8.4.2 Conditioning of pressurized consistometer

After the slurry has been prepared, place it into a pressurized consistometer slurry container that has been chilled to the desired test temperature. Place the slurry container in the pressurized consistometer and ramp the sample to the desired test temperature and pressure. If additional conditioning is desired, refer to 10.6.4 of ISO 10426-2. At user discretion, the temperature of the slurry container and/or the oil within the pressurized consistometer may be lower than the bottomhole test temperature in order to promote a more rapid cool-down. Blot any oil that may have invaded the pressurized consistometer slurry container during the conditioning period.

8.4.3 Conditioning using stirred fluid-loss cell

After the slurry has been prepared, place it in a stirred fluid-loss cell and ramp to the anticipated test temperature. At user discretion, the temperature of the fluid-loss cell may be lower than the test temperature in order to promote a more rapid cool-down. After conditioning in the stirred fluid-loss cell, the fluid-loss test shall be performed in accordance with 10.7.2.1 through 10.7.2.9 of ISO 10426-2:2003.

8.4.4 Filling the fluid-loss cell

After removing the sample from the atmospheric pressure or pressurized consistometer, the sample shall be placed in a fluid-loss cell that has been chilled to the desired test temperature. Fill the fluid-loss cell as specified in 10.8 of ISO 10426-2:2003.

8.4.5 Fluid loss test

The fluid loss test shall be performed in accordance with 10.9 of ISO 10426-2:2003.

9 Deepwater well-simulation free fluid and slurry stability tests

9.1 General

The purpose of this test is to determine the static (quiescent) stability of a cement slurry. Further information concerning the purpose and significance of this test may be found in Clause 15 of ISO 10426-2:2003.

9.2 Sampling

The methods provided in Clause 4 shall be used for obtaining samples for free fluid and slurry stability testing.

9.3 Slurry preparation

The cement slurry shall be prepared in accordance with Clause 5.

9.4 Test procedure

After the slurry has been prepared, place it into an atmospheric pressure consistometer slurry container that has been chilled to the desired test temperature. Place the chilled slurry container into a chilled atmospheric pressure consistometer and condition for 20 min. After 20 min, verify the temperature, remove the paddle from the slurry container and stir the slurry briskly with a spatula to ensure a uniform slurry. If the cement slurry has not reached the desired test temperature, continue conditioning until the desired test temperature has been reached. Note the actual time to reach the desired test temperature. The temperature of the slurry container and/or the fluid within the atmospheric consistometer may be lower than the bottomhole test temperature in order to promote a more rapid cool-down. Alternatively, the sample may be placed in a pressurized

consistometer and ramped to the desired test temperature and pressure. If additional conditioning is desired, refer to 10.6.4 of ISO 10426-2:2003. The temperature of the slurry container and/or the oil within the pressurized consistometer may be lower than the desired test temperature in order to promote a more rapid cool-down. Blot any oil that may have invaded the pressurized consistometer slurry container during the conditioning period.

Once the slurry has been conditioned, conduct the free fluid test in accordance with 15.4.1 of ISO 10426-2:2003. Test for sedimentation in accordance with 15.6 of ISO 10426-2:2003. Keep the graduated glass cylinder or the sedimentation tube in a water bath or vessel, which is cooled to the desired test temperature.

10 Determination of rheological properties and gel strength using a rotational viscometer

10.1 General

The purpose of this procedure is to determine the rheological and gel strength behaviour of cement slurries used in deepwater applications.

10.2 Sampling

The methods provided in Clause 4 shall be used for obtaining samples of cement used in rheological and gel strength testing.

10.3 Preparation of slurry

The cement slurry shall be prepared according to Clause 5.

10.4 Apparatus

The apparatus used shall be in accordance with 12.1.1 of ISO 10426-2:2003.

10.5 Procedure

10.5.1 Place the slurry into an atmospheric pressure consistometer slurry container that has been chilled to the desired test temperature. Place the slurry container into a chilled atmospheric consistometer and condition for 20 min. After 20 min, verify the temperature, remove the paddle from the slurry container and stir the slurry briskly with a spatula to ensure a uniform slurry. If the cement slurry has not reached the desired test temperature, continue conditioning until the desired test temperature has been reached. Note the actual time to reach the desired test temperature. The temperature of the slurry container and/or the fluid within the atmospheric consistometer may be lower than the test temperature in order to promote a more rapid cool-down. Alternatively, the sample may be placed in a pressurized consistometer and ramped to the desired test temperature and pressure. If additional conditioning is desired, refer to 10.6.4 of ISO 10426-2:2003. Blot any oil that may have invaded the pressurized consistometer cup during the conditioning period. After the oil is blotted, remove the slurry paddle from the slurry container and stir briskly with a spatula to ensure a uniform slurry. At user discretion, the temperature of the slurry container and/or the fluid within the pressurized consistometer may be lower than the bottomhole test temperature in order to promote a more rapid cool-down.

10.5.2 Pour the cement slurry immediately into the viscometer cup. The viscometer cup, sleeve and bob should be chilled to the desired test temperature.

10.5.3 With the sleeve turning at the lowest rotational speed, raise the cup until the liquid level is at the fill line on the sleeve.

10.5.4 Record the temperature of the slurry in the viscometer cup before taking the first reading. Record the initial dial reading 10 s after continuous rotation at the lowest speed. All remaining readings shall be taken first

in ascending order of speed, and then in descending order, after continuous rotation of 10 s at each speed. Shift to the next speed immediately after taking each reading. The highest reading should be taken at a shear-rate (equivalent speed) of 511 s^{-1} .

10.5.5 For gel strength determination, test the slurry as defined in 10.5, then test the slurry in accordance with 12.4 of ISO 10426-2:2003.

11 Compatibility of wellbore fluids

11.1 General

This procedure is used to determine the degree of compatibility of wellbore fluids intended for use in deepwater applications. This procedure calls for the measurement of fluid rheology, static gel strength, thickening time, strength, fluid loss and solids suspension. By use of this procedure, the selection of preflushes and/or spacers can be made.

The terms used in this procedure are defined in Clause 16 of ISO 10426-2:2003.

11.2 Preparation of test fluids

11.2.1 Preparation of spacer

The spacer should be freshly prepared and aged in accordance with the supplier's instructions and anticipated well surface-location conditions.

11.2.2 Preparation of drilling fluid

Representative field-drilling fluid should be used. Thoroughly mix drilling fluid samples prior to testing. If representative field-drilling fluid is not available, prepare the drilling fluid in the laboratory according to the supplier's instructions.

11.2.3 Preparation of cement slurries

The cement slurries shall be prepared in accordance with Clause 5.

11.2.4 Preparation of fluid mixtures

The fluid mixtures shall be prepared in accordance with 16.3.4 of ISO 10426-2:2003.

The typical ratios are 95/5, 75/25, 50/50, 25/75, and 5/95 for each fluid combination, as well as a 25/50/25 mixture of drilling fluid/spacer/cement. The specific mixing volume for the various ratios of drilling fluid/spacer/cement is given in Table 6 of ISO 10426-2:2003. Record the data in Table 7 of ISO 10426-2:2003.

11.3 Property determination

11.3.1 Rheology

Rheological properties of the mixtures shall be determined by the method provided in 10.5.

11.3.2 Thickening time

Thickening-time tests of the fluid mixtures shall be performed by the method provided in Clause 7 of this part of ISO 10426. The typical ratios are 95 % (volume fraction) drilling fluid or cement and 5 % (volume fraction) spacer and 75 % (volume fraction) drilling fluid or cement and 25 % (volume fraction) spacer.

11.3.3 Strength testing

Strength testing of the fluid mixtures shall be conducted by either of the methods provided in Clause 6. The specific fluid mixture to be tested can vary with fluid compatibility and well conditions.

11.3.4 Fluid loss

The typical ratios are 95 % (volume fraction) drilling fluid or cement and 5 % (volume fraction) spacer and 75 % (volume fraction) drilling fluid or cement and 25 % (volume fraction) spacer. The fluid loss test shall be conducted in accordance with the method provided in Clause 8. At user discretion, tests may be run on other mixtures of cement/spacer, cement/drilling fluid and cement/drilling fluid/spacer.

11.3.5 Solids suspension and static gel strength

This procedure is used to investigate the behaviour of fluid mixtures during and after cement slurry placement.

Initiate a thickening time test in accordance with the method provided in Clause 7. When the desired test temperature has been reached, record the consistency in Bearden units B_c and then cease agitation. After an elapsed time of 10 min, resume the stirring and observe and record any momentary gel strength development, as indicated by an increase in consistency, or solids settling, as indicated by a deflection in consistency at the moment of start-up. Continue agitation until one-half of the thickening time of the base cement slurry has been reached. Record the consistency in Bearden units B_c and cease agitation. After an elapsed time of 10 min, resume stirring and observe and record any momentary gel strength development or solids settling. This cycle may be repeated as often as desired at user discretion. When removing the slurry container from the consistometer, take care not to invert the cup. Carefully disassemble the slurry container and note the condition of the slurry, especially gelation and/or solids settling.

Bibliography

- [1] ISO 10426-1, *Petroleum and natural gas industries — Cements and materials for well cementing — Part 1: Specification*
- [2] API Recommended Practice 10B, December 1997, *Recommended practice for testing well cements* (22nd edn.): Addendum 1, October 1999: Addendum 2, November 2000



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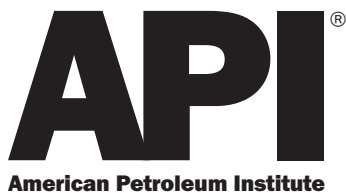
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