Recommended Practice on Determining the Static Gel Strength of Cement Formulations

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ISO 10426-6:2008 (Identical), Petroleum and natural gas industries—Cements and materials for well cementing—Part 6: Methods for determining the static gel strength of cement formulations

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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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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 10426-6 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*

— *Part 6: Methods for determining the static gel strength of cement formulations*

Introduction

Characterizing the static gel-strength (SGS) development of a cement slurry is an important design parameter in specific cementing environments. These include shallow-water flow mitigation, plugging operations and certain annular flow circumstances. Determining the gel-strength characteristics of a cement slurry allows the user to ascertain if the cement design is fit for a particular intended purpose. Historically, the SGS of a cement slurry was determined by a method using a couette-type rotational viscometer. More recently, specialized instruments, including a rotating-type apparatus, an intermittent rotation-type apparatus and an ultrasonic-type apparatus (removed in the API adoption, please refer to Clause 7), have been used to measure the gel-strength development of a static cement slurry. This part of ISO 10426 provides the testing protocol for determining SGS using these two types of instruments.

It is necessary to note that, due to differences in sample size, apparatus configuration and method of SGS determination, there can be considerable variance in results obtained by the three types of instruments described in this part of ISO 10426.

CAUTION — Caution is necessary when using static gel-strength development testing results as the single or predominant engineering parameter of a cement slurry design or technical evaluation.

In this part of ISO 10426, where practical, U.S. Customary (USC) units are included in brackets for information. The units do not necessarily represent a direct conversion of SI to USC, or USC to SI, units. Consideration has been given to the precision of the instrument making the measurement. For example, thermometers are typically marked in one degree increments, thus temperature values have been rounded to the nearest degree.

In this part of ISO 10426, calibrating an instrument refers to ensuring the accuracy of the measurement. Accuracy is the degree of conformity of a quantity to its actual or true value. Accuracy is related to precision, or reproducibility of a measurement. Precision is the degree to which further measurements or calculations show the same or similar results. Precision is characterized in terms of the standard deviation of the measurement. The results of calculations or a measurement can be accurate but not precise, precise but not accurate, neither or both. A result is valid if it is both accurate and precise.

Annex A of this part of ISO 10426 is for information only.

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

Part 6:
Methods for determining the static gel strength of cement formulations

1 Scope

This part of ISO 10426 specifies requirements and provides test methods for the determination of static gel strength (SGS) of cement slurries and related materials under simulated well 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 standard (including any amendments) applies.

API 10B-2/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 part of ISO 10426, the following terms and definitions apply.

3.1
bottom-hole pressure
P_{BH}
Hydrostatic pressure at the bottom of the well calculated from the true vertical depth and the fluid densities in the wellbore.

3.2
bottom-hole circulating temperature
T_{PBHC}
Maximum temperature encountered in a wellbore during cement slurry placement.

3.3
critical static gel strength
CSGS
Specific static gel strength of a cement in which hydrostatic-pressure equilibrium is reached between the decayed hydrostatic pressure transmission of the cement column (and other fluids in the annulus) and the pore pressure of the formation.

See Annex A.

NOTE The critical static gel strength is measured in pascals or newtons per square metre (pounds force per 100 square feet).

3.4
critical static gel-strength period
CSGSP
Time interval required for the cement to progress from the critical static gel strength value to a static gel strength of 250 Pa (500 lbf/100 ft^2).

3.5
static gel strength
SGS
Shear strength (stress) measurement derived from force required to initiate flow of a fluid.

NOTE The static gel strength is measured in pascals or newtons per square metre (pounds force per 100 square feet).

4 Sampling

4.1 General

Samples of the dry 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.

4.2 Method

Applicable sampling techniques for the dry cement or cement blend, solid and liquid additives and mixing water used in typical cementing operations can be found in ISO 10426-2:2003, Clause 4. If required, the temperature of the mix water, cement or cement blends, and liquid additives may be measured with a thermocouple or thermometer capable of measuring temperature with an accuracy of ± 2 °C (± 4 °F). These temperatures should be recorded. Temperature-measuring devices shall be calibrated (in the case of a thermocouple) no less frequently than every three months or checked (in the case of a thermometer) annually.

NOTE Descriptions of commonly used sampling devices can be found in ISO 10426-2:2003, Figure 1.

5 Preparation

Prepare the test samples in accordance with ISO 10426-2:2003, Clause 5.

If larger slurry volumes are needed, an alternative method for slurry preparation is found in ISO 10426-2:2003, Clause A.1.

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

6 Test method using rotating-type static gel strength apparatus

6.1 Apparatus

The apparatus contains a pressure chamber that can be heated and pressurized according to a simulated cement job schedule. The SGS is calculated from the torque required to rotate a paddle of known geometry at very low speed. The rotation speed of the paddle during the SGS measurement portion of the test is normally a continuous 0,000 009 2 r/s (0,2°/min). The initial stirring to simulate placement in the well is typically conducted at 2,5 r/s ± 0,25 r/s (150 r/min ± 15 r/min). The rotating-type static gel strength apparatus shall be calibrated according to the manufacturer's instructions. During the test period, the temperature and pressure of the slurry in the test cell is increased in accordance with the appropriate well-simulation test schedule (see 6.2.2). Determine the temperature of the cement slurry by use of an ASTM E220 classification “special” type J thermocouple located in the centre of the testing cell. The temperature-measuring system shall be calibrated to an accuracy of ± 2 °C (± 4 °F). Calibration shall be performed no less frequently than every three months.

NOTE Changing the rotational speed of the apparatus can be required depending on slurry design. The permissible range of rotational speed for the apparatus is 0,000 006 9 r/s (0,15°/min) to 0,000 023 1 r/s (0,5°/min).

6.2 Test procedure

6.2.1 If there is a batch mixing time being used for the job, the test schedule should include this segment. The slurry should be exposed to the anticipated temperature conditions during the batch mixing time. The pressure at this time shall be atmospheric. The stirring is typically maintained at 2,5 r/s ± 0,25 r/s (150 r/min ± 5 r/min). If there is no batch mixing time, omit this step.

6.2.2 Calculate the expected time to bottom and the expected placement time required to displace the cement to the flow zone. Ramp the cement slurry to bottom-hole circulating temperature (T_{PBHC}) and the bottom-hole pressure (P_{BH}) in the expected time to bottom. Then ramp to the circulating temperature and pressure at the flow zone. The time interval to ramp to the circulating temperature and pressure at the flow zone is the expected placement time to the flow zone, minus the expected time to bottom. After the circulating temperature at the flow zone is reached, hold at the specified temperature and pressure for 5 min ± 30 s to allow for temperature stabilization. Maintain paddle rotation at 2,5 r/s ± 0,25 r/s (150 r/min ± 15 r/min). In cases when an extended period of fluidity is expected, the test temperature may be increased to static temperature at the flow zone in 240 min after reaching the circulating temperature at the flow zone plus 5 min for temperature stabilization. During the placement simulation, the temperature and pressure shall be maintained within 3 °C (5 °F) and 2 MPa (300 psi) of the appropriate elapsed time versus temperature and pressure targets. Within 10 min after the end of the ramp, the temperature and pressure shall be within ± 1 °C (± 2 °F) and 0,7 MPa (100 psi) of the specified values.

NOTE During the time of stirring at API/ISO rotational speeds [2,5 r/s ± 0,25 r/s (150 r/min ± 15 r/min)], the test gives an indication of slurry consistency. It is not an exact slurry thickening time consistency since the paddle does not conform to the required dimensions for determining thickening time.

6.2.3 For the SGS determination, at the end of the slurry placement simulation, the rotational speed is changed from the typical 2,5 r/s ± 0,25 r/s (150 r/min ± 15 r/min) to 0,000 009 2 r/s (0,2°/min) or other permissible rotational speed. Maintain circulating temperature and pressure at the zone of interest. During SGS determination the temperature and pressure shall be maintained within ± 1 °C (± 2 °F) and ± 0,7 MPa (± 100 psi) of the target values.

6.2.4 Record the initial SGS and the elapsed time when the sample is placed in SGS determination mode from the previous placement simulation. Record the time to 50 Pa (100 lbf/100 ft^2), 100 Pa (200 lbf/100 ft^2), 150 Pa (300 lbf/100 ft^2), 200 Pa (400 lbf/100 ft^2) and 250 Pa (500 lbf/100 ft^2) SGS and, CSGS. Where applicable, determine the critical static gel strength period (CSGSP) by measuring the time required for the cement to progress from the critical static gel strength (CSGS) value (see Annex A) to a SGS of 250 Pa (500 lbf/100 ft^2). The manufacturer, model and rotational speed of the apparatus used to make the SGS determination shall be reported.

7 [Removed]

Because, as a general rule, patents the use of which would be required for compliance with that standard should not be included in API standards, this clause of ISO 10426-6 is omitted from this API standard.

8 Test method using intermittent rotation-type static gel strength apparatus

8.1 Apparatus

The cement slurry being tested is maintained in a static condition in a pressure chamber at a controlled temperature and pressure. The SGS is calculated from the torque required to rotate a paddle of known geometry intermittently at very low speed. This apparatus typically operates intermittently at 0,000 16 r/s ± 0,000 005 r/s (0,01 r/min ± 0,003 r/min) for 6 s after an interval of time adjustable between 1 min and 10 min during the SGS-testing phase. Generally, an intermittent rotation every 3 min is used during the SGS-testing phase. The intermittent rotation-type static gel strength apparatus shall be calibrated according to the manufacturer's instructions. During the test period, the temperature of the slurry in the test cell is increased in accordance with the appropriate well simulation test schedule (see 8.2.4). Determine the temperature of the cement slurry by use of an ASTM E220 classification "special" type J thermocouple located in the cement slurry. The temperature-measuring system shall be calibrated to an accuracy of ± 2 °C (± 4 °F). Calibration shall be performed no less frequently than every three months.

8.2 Pressurized conditioning test procedure

8.2.1 Any consistometer referenced in ISO 10426-2:2003, 9.2.1 or Annex D, may be used to condition the slurry.

8.2.2 Place the slurry in the container of the pressurized consistometer and begin a thickening time test according to the procedure given in ISO 10426-2:2003, 9.4.3.

8.2.3 If there is a batch mixing time being used for the job, the test schedule should include this time period. The slurry should be exposed to the anticipated temperature conditions during the batch mixing time for the job. The pressure at this time shall be atmospheric.

8.2.4 Calculate the expected time to bottom and the expected placement time required to displace the cement to the flow zone. Ramp the cement slurry to bottom-hole circulating temperature (T_{PBHC}) and the bottom-hole pressure (P_{BH}) in the expected time to bottom. Then ramp to the circulating temperature and pressure at the flow zone. The time interval to ramp to the circulating temperature and pressure at the flow zone is the expected placement time to the flow zone, minus the expected time to bottom. After the circulating temperature at the flow zone is reached, hold at the specified temperature and pressure for 5 min ± 30 s to allow for temperature stabilization. Maintain paddle rotation at 2,5 r/s ± 025 r/s (150 r/min ± 15 r/min). During the placement simulation, the temperature and pressure shall be maintained within 3 °C (5 °F) and 2 MPa (300 psi) of the appropriate elapsed time versus temperature and pressure targets. Within 10 min after the end of the ramp, the temperature and pressure shall be within 1 °C (2 °F) and 0,7 MPa (100 psi) of the specified values. For safety, if the conditioning temperature is greater than 88 °C (190 °F), turn the heater off and cool the slurry as quickly as practical to approximately 88 °C (190 °F) before removing the slurry from the pressurized consistometer. This 88 °C (190 °F) safety temperature assumes a boiling point of water at 100 °C (212 °F). If the boiling point of water at the test location is less than 100 °C (212 °F), adjust the transfer temperature accordingly.

The 88 °C (190 °F) safety temperature assumes a boiling point for water of 100 °C (212 °F). If the boiling point of water in the test locale is less than 100 °C (212 °F), adjust this safety temperature accordingly.

8.2.5 Release the pressure slowly [about 1 400 Pa/s (200 psi/s)]. Remove the slurry container from the consistometer, keeping the container upright so that the oil does not mix with the slurry. Remove the top locking ring, drive bar and collar from the shaft and the diaphragm cover. Syringe and blot oil from the top of the diaphragm. Remove the diaphragm and the support ring. Syringe and blot any remaining oil from the top of the slurry. If the contamination is severe, discard the slurry and begin the test again. Remove the paddle and stir the slurry briskly with a spatula to ensure a uniform slurry.

8.2.6 Using the conditioned slurry, fill the sample holder of the gel strength test apparatus preheated to the lesser of the circulating temperature at the flow zone or 88 °C (190 °F), according to the manufacturer's instructions. This transfer shall be completed in no more than 5 min after removal of the sample from the pressurized consistometer. Pressurize the sample to the pressure of the flow zone or to the limit of the apparatus. If the temperature of the flow zone is greater than 88 °C (190 °F), ramp to the circulating temperature at the flow zone at a rate of 2 °C/min (4 °F/min). During SGS determination, the temperature and pressure shall be maintained within 1 °C (2 °F) and 0,35 MPa (50 psi) of the target values. In cases when an extended period of fluidity is expected, the test temperature may be increased to the static temperature at the flow zone in 240 min after reaching the circulating temperature at the flow zone.

8.2.7 Record the SGS value immediately after putting the apparatus in operation. Then record the SGS value when the temp at the flow is reached. Record the time to 50 Pa (100 lbf/100 ft^2), 100 Pa (200 lbf/100 ft^2), 150 Pa (300 lbf/100 ft^2), 200 Pa (400 lbf/100 ft^2), 250 Pa (500 lbf/100 ft^2) SGS and CSGS. Where applicable, determine the CSGSP by measuring the time required for the cement to progress from the CSGS value (see Annex A) to a SGS of 250 Pa (500 lbf/100 ft^2). The manufacturer, model of the apparatus and type of conditioning used in making the SGS determination shall be reported.

8.3 Atmospheric pressure conditioning test procedure

8.3.1 Within 1 min after mixing, place the slurry into the container of the atmospheric pressure consistometer.

8.3.2 If there is a batch mixing time being used for the job, the test schedule should include this segment. The slurry should be exposed to the anticipated temperature conditions during the batch mixing time for the job. The stirring is typically maintained at 2,5 r/s ± 0,25 r/s (150 r/min ± 5 r/min). If there is no batch mixing time, omit this step.

8.3.3 Calculate the expected time to bottom and the expected placement time required to displace the cement to the flow zone. Ramp the cement slurry to the lesser of the T_{PBHC} or 88 °C (190 °F) in the expected time to bottom. Then ramp to the circulating temperature at the flow zone. After the circulating temperature at the flow zone is reached, hold at the specified temperature for 5 min ± 30 s to allow for temperature stabilization. The stirring is typically maintained at 2,5 r/s ± 0,25 r/s (150 r/min ± 15 r/min). The time interval to ramp to the circulating temperature at the zone of interest is the expected placement time minus the expected time to bottom. During the placement simulation, the temperature shall be maintained within ± 3 °C (± 5 °F) of the appropriate elapsed time versus temperature and pressure targets. Within 10 min of the end of the ramp, the temperature shall be within ± 1 °C (± 2 °F) of the specified values. Once the circulating temperature at the flow zone or 88 °C (190 °F) is reached, remove the paddle and stir the slurry briskly with a spatula to ensure a uniform slurry.

For safety, this 88 °C (190 °F) safety temperature assumes a boiling point of water at 100 °C (212 °F). If the boiling point of water at the test location is less than 100 °C (212 °F), adjust the transfer temperature accordingly.

8.3.4 Using the conditioned slurry, fill the sample holder of the gel strength test apparatus preheated to the lesser of the temperature at the flow zone or 88 °C (190 °F) according to the manufacturer's instructions. This transfer shall be completed and the apparatus put in operation in no more than 5 min after removal of the sample from the consistometer. Pressurize the sample to the pressure of the zone or to the operating limit of the apparatus. If the temperature at the flow zone is greater than 88 °C (190 °F), ramp to the circulating temperature at the flow zone at a rate of 2 °C/min (4 °F/min). During SGS determination, the temperature and pressure shall be maintained within ± 1 °C (± 2 °F) and ± 0,35 MPa (± 50 psi) of the target values. In cases when an extended period of slurry fluidity is expected, the test temperature may be increased to the static temperature at the flow zone in 240 min after reaching the circulating temperature at the flow zone.

8.3.5 Record the SGS value immediately after putting the apparatus in operation. Then record the SGS value when the temp at the flow is reached. Record the time to 50 Pa (100 lbf/100 ft^2), 100 Pa (200 lbf/100 ft^2), 150 Pa (300 lbf/100 ft^2), 200 Pa (400 lbf/100 ft^2), 250 Pa (500 lbf/100 ft^2) SGS, and CSGS. Where applicable, determine the CSGSP by measuring the time required for the cement to progress from the CSGS value (see Annex A) to a SGS of 250 Pa (500 lbf/100 ft^2). The manufacturer, model of the apparatus and type of conditioning used in making the SGS determination shall be reported.

Annex A
(informative)

Critical static gel strength — Additional information

One method of minimizing the period of susceptibility of the well to pressure losses by gel strength development is to minimize the time during which an underbalanced condition exists in the wellbore before the cement has developed sufficient SGS to resist invasion by the well fluids.

In a wellbore environment, the hydrostatic pressure of the fluid(s) in the annulus counteracts the flow potential of a formation (as a function of formation pore pressure). With settable fluids, such as cement, the effective hydrostatic pressure in the annulus (counteracting the pore pressure of the formation) declines as the slurry transitions from a fully hydraulic fluid into a gelled state, then into a solid material. A point of equilibrium is reached at which the pore pressure of the formation equals the decayed hydrostatic pressure exerted by the cement plus the full hydrostatic pressure contributions of the other fluids in the annulus. This state of equilibrium is known as the critical static gel strength. Once the critical static gel strength is reached, any further gellation of the cement produces an underbalanced condition conducive to flow. The critical static gel strength for a cement employed in flow mitigation is a function of wellbore geometry, length of cement column above the flow zone, hydrostatic pressure of fluids above the cement, cement density and flow zone pore pressure. The critical static gel strength is also known in the industry as “critical wall shear stress”. The CSGS can be calculated as $X_{\text{CSGS,SI}}$ in SI units of pascals, as given by Equation (A.1), and as $X_{\text{CSGS,USC}}$ in USC units of pounds per 100 square feet, as given by Equation (A.2):

$$X_{\text{CSGS,SI}} = (p_{\text{fo}})(0{,}027)/(L/D_{\text{eff}}) \tag{A.1}$$

where

p_{fo} is the fluid overbalance pressure, expressed in pascals;

0,027 is the conversion factor;

L is the length of the cement column, expressed in metres;

D_{eff} is the effective diameter of the wellbore, expressed in centimetres.

$$X_{\text{CSGS,USC}} = (p_{\text{fo}})(300)/(L/D_{\text{eff}}) \tag{A.2}$$

where

p_{fo} is the fluid overbalance pressure, expressed in pounds per square foot;

300 is the conversion factor;

L is the length of the cement column, expressed in feet;

D_{eff} is the effective diameter of the wellbore, expressed in inches.

NOTE The fluid overbalance pressure is determined by subtracting the pore pressure at the flow interval from the annular fluid hydrostatic pressure at the flow zone. In deepwater environments with no marine riser in place, the hydrostatic pressure of the seawater above the mud line is included in the annular fluid pressure hydrostatic pressure calculation. The effective diameter of the wellbore equals the diameter of the open hole minus the diameter of the casing.

Bibliography

[1] ASTM E220, *Standard Test Method for Calibration of Thermocouples By Comparison Techniques*

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