Ultra-Lightweight Cement

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Abstract

The objective of this project is to develop an improved ultra-lightweight cement using ultra-lightweight hollow glass spheres (ULHS). This report includes results from laboratory testing of ULHS systems along with other lightweight cement systems, including foamed and sodium silicate slurries. During this project quarter, a comparison study of the three cement systems examined the effect that cement drillout has on the three cement systems. Testing to determine the effect of pressure cycling on the shear bond properties of the cement systems was also conducted. This report discusses testing that was performed to analyze the alkali-silica reactivity of ULHS in cement slurries.

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Corrigendum

One of the cement types used in the project's tests has been inaccurately referred to as Class A in the past quarterly progress reports. The cement is actually Type I cement. Although Class A and Type I cements are very similar, they are still different cements.

In the past progress reports, the "Class A" designation should be replaced with "Type I" when in the context of the cement that is being tested.

Introduction

Oilwell cementing involves placing a pumpable slurry of Portland cement, additives, and water into a wellbore. The slurry is pumped into the annular space between the borehole and a steel pipe (called a casing) that acts as a conduit from the reservoir to the surface. The cement sets in place (1) to support the casing in the hole, (2) to isolate various formations from one another, and (3) to control fluid movement within the well.

Typically, cement fluid density is anywhere from 12 to 17 lb/gal. Certain conditions that require the application of low-density cements can be encountered during the well construction process. Lower density is required (1) to limit hydrostatic pressure on the formation, and (2) to prevent the formation from fracturing and imbibing the well fluid. This phenomenon, known as lost circulation, increases drilling and completion times and increases construction cost because of expensive remedial treatments. The upper sections of the well are where lost circulation most commonly occurs. These include surface and intermediate casings. Because formations covered by these casings are relatively close to the Earth's surface, application temperatures for these low-density cements are low.

The minimum practical density achievable with conventional cements and additives is approximately 11 lb/gal. At this density, the slurry's stability and set cement's strength and permeability are only marginally acceptable. Water is the primary density-reducing material in these conventional cements. Additional water dilutes the cement, causing low strength and high permeability. Lower temperature, such as those in the upper well sections, delays strength development. To obtain lower cement density or greater cement strength, ultra-lightweight materials must be mixed into the slurry.

Ultra-lightweight hollow spheres (ULHS) are excellent candidate materials for producing ultra-lightweight cements. These small hollow glass beads effectively trap air in the slurry, thereby lowering the slurry density without the addition of water to the slurry.

This project is designed to develop cementing systems using ULHS through a carefully designed program of modeling, design, laboratory testing, and field testing.

Executive Summary

Laboratory testing during the seventh quarter was a continuation and extension of the testing that has been previously conducted. Testing compared slurries containing ULHS to foamed and sodium silicate cement systems. Comparison studies examined shear bond

strength after pressure-cycling conditions. Studies also simulated drillout operations to evaluate the resilience of the three cement systems after drillout procedures.

Because of the large volume of cement required for this project, a quality-control process has been implemented. Information and data gathered for the program are presented.

Cement Quality-Control Program

Because of the large quantities of cement used in the course of this project, a quality – control (QC) program was initiated. Upon receipt, each bucket of cement is labeled with a materials log number and date. When a bucket is first opened for use, the date of opening is recorded in the materials log. This log number is referenced on the laboratory sheets for each test performed. Where applicable, tests are conducted according to API Specification 10A¹. Additionally, several tests tailored for the test conditions and materials (rheology and low-temperature compressive-strength development) are included in this QC program.

For the QC program the Class A test specifications were used for the Type I cement. The Type I and Class H cement performance requirements are presented in the original API Specification 10A (January 1995) before Addendum 1, which changed the free fluid procedure. The free fluid procedure comes from the January 1995 edition that uses graduated cylinders. Because the Lightweight Oilwell cement is not API cement, it is tested according to QC procedures developed by the manufacturer.

The physical requirements used to test each cement are listed in **Table 1**. Calcium chloride (CaCl_2) is being used to accelerate the rate of compressive-strength development at low temperatures. **Table 2** shows the temperatures and concentrations at which calcium chloride is used. Calcium chloride was selected because it is one of the most effective and commonly used cement accelerators.

Table 1—Cement slurry compositions for quality-control testing program

Cement	Mix Water (% BWOC)	Density (lb/gal)	Cement (g)	Water (mL)	Test
Type I	46	15.6	772	355	API Series of Tests
TXI Lightweight	75	13.2	541	406	Thickening Time & Compressive Strength
TXI Lightweight	105	12.1	426	447	Free Fluid
Class H	38	16.4	860	327	API Series of Tests

Table 2—Percent CaCl₂ for low-temperature compressive strengths

Temperature (°F)	CaCl ₂ (% BWOC)
80	0.0
60	1.0
45	2.0

These quality-control tests have been conducted to provide a baseline for each type of cement. This QC data will provide a basis of comparison for other data for this project. After several cycles of the complete QC program had been performed, the data showed no significant variations and remained within the vendors' specifications. Testing then proceeded with a less-stringent program of scheduled thickening-time tests. Each time a new bucket is opened, a thickening-time test is conducted. Additionally, a thickening-time test is performed once a month on all opened buckets. The thickening-time test was chosen as a gauge to detect abnormal cement performance. **Appendix A** shows the QC program data.

Application Scenarios

One way to gauge the performance of cements made with ULHS is to compare them to other ultra-lightweight cement systems. Members of the project advisory board decided to compare ULHS cements to foamed cement and a conventional sodium silicate slurry.

Seventh-quarter testing focused on a cementing scenario referred to as Application 2. Application 2 conditions simulated a typical land-based surface-pipe cementing job with the following conditions:

- bottomhole circulating temperature (BHCT) of 78°F
- bottomhole static temperature (BHST) of 96°F
- bottomhole pressure (BHP) of 1,000 psi

As detailed in the first quarterly report, these conditions are average values obtained from *Worldwide Cementing Practices*². Application 2 cement slurries were designed to achieve a thickening time of 4 to 6 hr with 1.0% maximum free fluid.

Of the different ULHS classes studied in this project, 6K beads were chosen because they are able to withstand the hydrostatic pressures associated with the different simulation applications.

Most tests were performed according to the API RP 10B³ method. To adapt to ULHS and foamed slurries, some preparation and testing methods were modified. To minimize bead breakage caused by high shear from API blending procedures, mixing procedures were modified for the ULHS slurries. The following blending procedure was used for the ULHS slurries:

1. Weigh out the appropriate amounts of the cement sample and additives, water, and ULHS into separate containers.

2. Mix the cement slurry (without ULHS) according to Section 5.3.5 of API RP 10B.³
3. Pour the slurry into a metal mixing bowl and slowly add ULHS while continuously mixing by hand with a spatula. Mix thoroughly.
4. Pour this slurry back into the Waring blender and mix at 4,000 rev/min for 35 seconds to mix and evenly distribute the contents.

Testing methods for foamed slurries were also modified. For example, thickening time is performed on unfoamed slurries only. Because the air in the foam does not affect the hydration rate, the slurry was prepared per API RP 10B³, then the foaming surfactants were mixed into the slurry by hand without foaming the slurry. This and the other non-standard procedures for testing foamed cement are presented in **Appendix B**.

Table 3 presents slurry compositions for the 11.5-lb/gal slurries tested during this project quarter. To adapt each slurry to the application conditions and to meet the thickening time and free fluid requirements, additives, such as slurry extenders, accelerators, and foaming agents, were added to the slurries. The additives are widely available, generic additives, and are not specific to any particular service company. These additives include sodium silicate, calcium chloride, Witcolate[®] 7093 (a foaming agent), and Aromox[®] C/12 (a foam stabilizer).

Table 3—Slurry compositions for Application 2 slurries

Density: 11.5-lb/gal, Application 2				
Cement Type	Slurry System	Measured Density (lb/gal)	Water Content (gal/sk)	Additive Concentrations
Type I	ULHS	11.5	7.09	16.2% BWOC (6K) ULHS
	Foamed	15.6 [†] (11.5 [‡])	5.20	0.03 gal/sk Witcolate [®] 7093; 0.02 gal/sk Aromox [®] C/12
	Sodium Silicate	11.5	16.87	3.0% BWOC Sodium Silicate
TXI Lightweight	ULHS	11.5	6.50	2.0% BWOC CaCl ₂ ; 11.9% BWOC (6K) ULHS
	Foamed	13.0 [†] (11.5 [‡])	7.00	0.03 gal/sk Witcolate [®] 7093; 0.02 gal/sk Aromox [®] C/12
	Sodium Silicate	11.5	12.11	3.0% BWOC Sodium Silicate

[†]Unfoamed slurry density

[‡]Foamed slurry density

Because of Application 2's low temperatures, calcium chloride (CaCl₂) was used with some foamed and ULHS slurries to accelerate hydration. CaCl₂ was not used with the sodium silicate slurries because of viscosity issues specific to this slurry. The only viable

accelerators for sodium silicate slurries are company-specific and not widely available in a generic form.

Shear Bond

Testing continued with the evaluation of the shear bond strength of the three cement systems. These studies investigated the effect of restraining force on shear bond strength. Special shear bond strength testing was done to determine the effect of pressure cycling on shear bond strength.

Samples were cured in a pipe-in-pipe configuration (**Figure 1**). The pipe-in-pipe configuration consisted of a sandblasted internal pipe with an outer diameter (OD) of $1\frac{1}{16}$ in. and a sandblasted external pipe with an internal diameter (ID) of 3 in. For the pressure cycling tests, the internal pipe of the shear bond samples is a 40/41 coiled tubing pipe. Other dimensions & properties of the coiled tubing include: wall thickness of 0.087 in., internal yield pressure of 11,480 psi, and hydro test pressure of 9,200 psi.

Both the internal and external pipes were 6 in. long. To center the internal pipe within the external pipe, a contoured base and top were used. The base extended 1 in. into the annulus and cement filled 4 in. of the annulus. The top 1 in. of annulus contained water.

Figure 1—Cross section of pipe-in-pipe configuration for shear bond tests

Application 2 cement systems were used for the shear bond strength tests. Special shear bond strength testing was done to determine the effect of pressure cycling on shear bond strength. The pressure cycling procedure was designed to simulate conditions encountered during production of a well similar to the Application 2 scenario. The samples were first cured for seven days in a 96°F water bath at atmospheric pressure. They were then subjected to five periods of pressure cycling in which the interior pipe is pressured to 5,000 psi for 10 minutes and then allowed to rest at 0 psi for 10 minutes.

Shear bond measures the stress needed to break the bond between the cement and the internal pipe. Shear bond was measured with the aid of a test jig that creates a platform

for the base of the cement to rest against as force is applied to the internal pipe. (**Figure 2**) Shear bond force is the force required to move the internal pipe. The pipe was pressed until the bond was broken. The shear bond strength is the force required to break the bond (move the pipe) divided by the surface area between the internal pipe and the cement.

Figure 2—Configuration for testing shear bond strength

Table 4 presents the seven-day shear bond strengths for the three cement systems in the pipe-in-pipe configuration. The results of the pressure-cycled pipe-in-pipe shear bonds are also presented in **Table 4**. Current testing is also investigating pressure-cycled shear bonds in a pipe-in-soft configuration. The pipe-in-soft configuration allows the cement to cure in a less-rigid, lower-restraint environment. Results from the pipe-in-soft tests will be presented in the next progress report.

Table 4—Shear bond strength data of lightweight cement systems that have and have not been exposed to pressure cycling

Density: 11.5 lb/gal, Application 2, Baseline (Uncycled)					
Cement Type	Slurry System	Pipe-in-Pipe Shear Bond Strength (psi)			
		Sample 1	Sample 2	Sample 3	Average
Type I	ULHS	466	365	375	402
	Sodium Silicate	173	166	115	151
	Foamed	377	380	595	451
TXI Lightweight	ULHS	120	118	119	119
	Sodium Silicate	37	44	44	42
	Foamed	127	120	136	128
Density: 11.5 lb/gal, Application 2, Pressure Cycled					
Cement Type	Slurry System	Pipe-in-Pipe Shear Bond Strength (psi)			
		Sample 1	Sample 2	Sample 3	Average
Type I	ULHS	332	273	257	287
	Sodium Silicate	130	103	140	124
	Foamed	308	275	333	305
TXI Lightweight	ULHS	127	104	107	113
	Sodium Silicate	164	140	122	142
	Foamed	296	251	304	284

Drillout Testing

Testing was performed to evaluate the competence of ULHS and other lightweight cement systems during drillout of a cemented shoe joint. To simulate the drillout conditions, 3-ft models were used. Each model consisted of an inner pipe (4.500-in. OD, 4.026-in. ID) concentrically centered within an outer pipe (8.625-in. OD, 7.98-in. ID). To join the pipes together at the base and to make a watertight base, a plate was welded to the bottom of each pipe. The plate had a concentrically centered, raised circular surface with a diameter slightly less than the ID of the inner pipe. The raised circular surface in the middle was designed to center the inner pipe within the outer pipe. **Figure 3** illustrates how the inner pipe and outer pipe are joined together at the weld plate.

Figure 3—Diagram of model for drillout testing

To test the competence of the cement in the annulus, testing was done to measure the fluid flow in the annulus before and after drillout. This annular fluid flow accounts for the flow through the cement (permeability) as well as any flow around the cement such as cracks or a microannulus. Using the fluid flow data, calculations were also performed to determine an equivalent bulk permeability; the bulk permeability assumes that the fluid flows through the cement and no fluid flows through cracks or a microannulus.

On the outside of the outer pipe, a 0.25-in. hole (with fitting) served as the pressure inlet port during fluid flow testing. The port was located 1 in. from the bottom of the pipe, just above the weld plate. During fluid flow testing, a cap was used to seal off the inner pipe. During annular fluid flow testing an annular disc was used to isolate the annulus. The annular disc had an outlet port for collecting fluid that permeated through the cement.

Figure 4 depicts the drillout model equipped with the cap for the inner pipe and the annular disc. It is in this configuration that the annular fluid flow of the cement is measured. **Figure 4** shows the drillout model for the case where the cement in the inner pipe has already been drilled; however, the same equipment is also used prior to drillout for fluid flow measurements of the annulus.

**Figure 4—Diagram of model equipped for fluid flow testing
(after cement in inner pipe has been drilled out)**

To prepare the drillout models, the pressure inlet port was capped and then sand was placed to a depth of 3 in. in the bottom of the annular space between the inner and outer pipes. The cement that was to be tested was mixed in the laboratory and poured into the inner pipe and the annulus to within 3 in. of the top of the pipes.

The three different cement slurries that were tested are shown in **Table 5**. They were all designed for a final density of 11.5 lb/gal. For the foamed slurry, the unfoamed slurry density was 15.0 lb/gal and then the slurry was foamed (and verified) to 11.5 lb/gal using methods specified in **Appendix B**.

Table 5—Composition for lightweight cement systems used in drillout testing

Cement Type	Slurry System	Water Content (gal/sk)	Additive Concentrations
Type I	ULHS	7.09	16.2% BWOC (6K) ULHS
	Foamed	6.02	0.03 gal/sk Witcolate® 7093; 0.02 gal/sk Aromox® C/12
	Sodium Silicate	16.87	3.0% BWOC Sodium Silicate

Water was then added on top of the cement up to the top rim of the pipes. The cement was cured for 7 days at atmospheric pressure and room temperature (approximately 75°F). On the eighth day, an initial annular fluid flow test was run on each sample.

The annular fluid flow testing consisted of:

1. Replace the water on top of the cement with new water.
2. Screw the cap on the inner pipe.
3. Screw the annular disc between the inner and outer pipes. As the disc is screwed on, make sure water comes out the outlet port and that there are not any air bubbles. If necessary, use a syringe to add water.
4. Attach the outlet hose and collection beaker.
5. Remove the cap on the inlet port.
6. Attach the inlet water system to the inlet port.
7. Adjust the water pressure to the differential test pressure. The water pressure was supplied by a regulated nitrogen system.
8. Allow the system to stay at the test pressure for the required amount of time. This time changes for each circumstance.
9. After the flow rate has stabilized, measure the flow rate of the fluid flowing out the outlet port.

The initial fluid flow testing performed on the eighth day was performed at a differential test pressure of 100 psi. The sodium silicate cement had immediate water flow and after the flow had stabilized a flow rate of 134 cc/min was measured. The foamed cement and ULHS cements had zero flow after 24 hours. The results from the initial and other fluid flow tests are presented in **Table 6**. **Table 7** presents the equivalent bulk permeability of the tests. The bulk permeability is based on the fluid flowing through the cement (i.e., no microannulus or cracks).

Table 6—Annular fluid flow of drillout tests

	Annular Flow Rate (cc/min)			
	8 th Day	10 th Day	11 th Day	
Differential Test Pressure (psi):	100	100	100	500
Sodium Silicate	134	137	—	—
ULHS	0	0	0	80
Foamed	0	0	0	100

Table 7—Equivalent bulk permeability of drillout tests

	Equivalent Bulk Permeability (md)			
	8 th Day	10 th Day	11 th Day	
Differential Test Pressure (psi):	100	100	100	500
Sodium Silicate	88	90	—	—
ULHS	0	0	0	10
Foamed	0	0	0	12

On the ninth day, the cement inside the inner pipe of each model was drilled out. Each model was clamped down in a horizontal direction. A 3-7/8-in. mill tooth roller cone bit

was used to drill out the cement. The weight on bit was 2500 pounds and the speed was 70 rpm. Water (the drilling fluid) was circulating at a rate of 70 gal/min. The rate of penetration for the sodium silicate cement averaged roughly 1,000 ft/hr; the ULHS cement averaged 100 ft/hr; the foamed cement averaged 150 ft/hr.

On the tenth day another annular flow test was performed on each sample using 100 psi of differential test pressure. The sodium silicate cement had immediate water flow and after the flow had stabilized a flow rate of 137 cc/min was measured. The foamed cement and ULHS cements had zero flow after 24 hours. Because the sodium silicate cement system had annular fluid flow prior to the drillout, no further fluid flow testing was performed on the sodium silicate cement.

On the eleventh day, the inner pipe was subjected to eight periods of pressure cycling in which the inner pipe is pressured to 2,000 psi and held for 10 minutes and then allowed to rest at 0 psi for 10 minutes. Annular fluid flow testing was then performed on the ULHS and foamed cements with 100-psi differential test pressure; both cements had zero flow. The test pressure was then increased to 500 psi. Water flowed at that point for both cement systems. After the fluid flow stabilized, the fluid flow for the ULHS cement was 80 cc/min and the fluid flow for the foamed cement was 100 cc/min. The test pressure was then increased to 1,000 psi. After the fluid flow stabilized, the fluid flow for the ULHS cement was 300 cc/min and the fluid flow for the foamed cement was 140 cc/min.

Expansion Testing

Work done previously in the project led to interest in expansion of neat cements and cement slurries with ULHS. Neat cement slurries are slurries that are mixed according to the manufacturer's recommended amount of water and they contain no other additives.

Table 8 presents the composition of the slurries that were tested.

Table 8—Composition of cement slurries used for expansion testing

Slurry Type	Water Content (gal/sk)	Density (lb/gal)	Additional Contents
Neat TXI Lightweight	6.07	13.5	—
Neat Type I	5.19	15.6	—
TXI Lightweight with ULHS	6.50	11.5	2.0% BWOC CaCl ₂ ; 11.9% BWOC (6K) ULHS

Three different test methods were used: linear bar expansion, annular ring expansion, and cylindrical sleeve expansion. The linear bar and annular ring methods measure linear expansion; the cylindrical sleeve expansion measures bulk expansion. For each method used, the samples are cured and stored in 100°F water. In all cases, there was free access by the cement matrix to water, albeit in the ring and cylinder tests to a limited exterior surface. Expansion measurements are taken at 1, 3, and 7 days.

▪ Linear Bar Expansion

The linear bar expansion testing method is from ASTM C490⁴. The bars were molded to have initial dimensions of 1×1×11¼ in. with a 10-in. gage length (comparator bar). The length of the bars is measured at 1, 3, and 7 days of curing. The linear expansion is calculated as follows.

$$L = \frac{(L_x - L_i)}{G} \times 100$$

where:

L = change in length at x age, %,

L_x = comparator reading of specimen at x age minus comparator reading of reference bar at x age; in.,

L_i = initial comparator reading of specimen minus comparator reading of reference bar at that same time; in.,

G = nominal gage length (10 in.).

▪ Annular Ring Expansion

The annular ring expansion testing method is from API Technical Report 10TR2⁵. The test model consists of an inner ring and outer ring. The solid ring OD is 2 in. and the outer expansion ring ID is 3.5 in. Two steel balls are attached to each side of the split on the expandable ring. These steel balls are used for taking measurements. **Figure 5** shows the configuration of the annular ring expansion test mold.

**Figure 5—Diagram of test mold for annular ring expansion testing
(reprinted from API Technical Report 10TR2⁵)**

When the cement is initially poured into the annular ring mold, an initial measurement of the spacing of the balls is taken with a micrometer for calibration of the annular ring. The distance between the outside of the steel balls is the measurement that's monitored. Measurements are also taken at 1, 3, and 7 days of curing. The percent linear expansion can be calculated as follows:

$$\%L_{ex} = (M_t - M_i) \times \pi \times ID_e$$

where:

$\%L_{ex}$ = percent linear expansion, %,

M_t = micrometer measurement after curing at time t, in.,

M_i = initial micrometer measurement (before curing), in.,

ID_e = inner diameter of expansion ring, in.

▪ Cylindrical Sleeve Expansion

The cylindrical sleeve expansion used for these expansion tests is a modified version of the cylindrical sleeve method in API Technical Report 10TR2. The customized sleeve expansion model is a brass sleeve that is 1.63 in. long and has two splits down the side. As seen in **Figure 6**, eight steel balls (four sets of two balls) are welded along the split.

Figure 6—Diagram of test mold for cylindrical sleeve expansion testing

To measure expansion with the cylindrical sleeve expansion molds, a caliper is used to take an initial measurement of the distance between the two balls for each of the four sets. At 1, 3, and 7 days of curing, measurements are also taken. The measurements between the two steel balls in each set are used to track the expansion of the diameter of the sleeve, which can then be used to determine the bulk expansion. The calculations involved with the cylindrical sleeve expansion testing are as follows.

$$Cx = Ci + (Mx1 - Mi1) + (Mx2 + Mi2)$$

where:

Cx = circumference at x age, in.

Ci = initial circumference

$Mx1$ = caliper measurement at x age—left side average of two, in.

$Mi1$ = initial caliper measurement—left side average of two, in.

$Mx2$ = caliper measurement at x age—right side average of two, in.

$Mi2$ = initial caliper measurement—right side average of two, in.

$$V_x = L \left(\frac{Cx}{\pi} \right)^2 \times \frac{\pi}{4}$$

where:

V_x = volume at x age, in.³,

L = length of sample, (1.625 in.),

$$\%B_{ex} = 100 \times \frac{V_x - V_i}{V_i}$$

where:

$\%B_{ex}$ = percent bulk expansion, %,

V_i = initial volume, (2.87 in.³).

Table 9 presents a summary of the expansion testing that was done. All cement samples were cured and stored in a water bath maintained at 100°F.

Table 9—Summary of expansion testing

Linear Bar Expansion			
Slurry Type	Percent Linear Expansion		
	1 Day	3 Days	7 Days
Neat TXI Lightweight	0.000	0.007	0.007
Neat Type I	0.000	0.016	0.016
Annular Ring Expansion			
Slurry Type	Percent Linear Expansion		
	1 Day	3 Days	7 Days
Neat TXI Lightweight	0.000	0.240	0.240
Neat Type I	0.000	0.048	0.049
TXI Lightweight with ULHS	0.164	0.188	0.201
Cylindrical Sleeve Expansion			
Slurry Type	Percent Bulk Expansion		
	1 Day	3 Days	7 Days
Neat TXI Lightweight	0.109	0.115	0.115
TXI Lightweight with ULHS	0.148	0.235	0.331

Alkali Silica Reactivity

To get a better understanding of the possible alkali-silica reactivity (ASR) between ULHS and Portland cement, Halliburton Energy Services was commissioned to analyze theoretical alkali levels in cement slurries that contain ULHS. The objective of the study was to determine the potential for ASR based on theoretical determination of cement alkali content.

It has been reported⁶ that states the alkali levels of cracked concrete typically range from 5.0 to 9.0 kg/m³ and these values were the basis for the calculations undertaken. In reality, there are many other operative mechanisms that may have contributed to the cracking of the previous concretes, rendering the correlation with alkali level tenuous.

For example, it has been clearly demonstrated that certain aggregates of carbonate composition also participate in reactions with alkalis, undergoing the so-called alkali carbonate reaction (ACR) and producing detrimental expansion and cracking. Other known forces that are generally found to be present in cracked concrete are freezing and thawing, stresses associated with concrete dryness, formation of ettringite, and even traffic loading.

The method used to perform the analysis is based on theoretical slurry compositions and can readily be applied to actual slurry formulations. This theoretical approach is, however, limited to ordinary Portland cements, and as such, cannot be used for blended cements or pozzolanic blended cements.

Although soluble alkali and total alkali content were used for the computation of alkali concentration in slurries ranging in density between 6 to 12 lb/gal, the voluminous ASR technical literature overwhelmingly considers the initial soluble alkali content as the critical threshold level for establishing potential interaction with reactive materials.

The following two tables summarize the data gathered from the analysis. Near the top of each of these tables is listed the total alkali content and the soluble alkali content for each cement. The cement manufacturer provided the total alkali content of the cement. Halliburton Energy Services performed analysis to determine soluble alkali content using ASTM C114.⁷

Calculations to determine the potential alkali contents in slurries of different densities were based on theoretical slurry formulations using 4K and 10K ULHS, cement, and water. The following two tables show varying amount of ULHS and varying densities. The alkali content for each slurry was then calculated using the following equation:

$$A = B \times \frac{C}{W} \times 10$$

where:

- A = alkali content of slurry, kg/m³,
- B = % Na₂O_e (soluble or total) for the cement used,
- C = weight of cement in the slurry, g,
- W = volume of water in the slurry, mL.

As indicated by the expression above, the cement-to-water ratio determines the critical alkali concentration. Since the alkali concentration in a slurry is related to the original alkali content of the cement and to the amount of water included in the formulation, a higher cement-to-water ratio (higher density slurries) corresponds to a higher alkali concentration in the slurry.

Table 10—Alkali concentration of Class H cement slurries with ULHS

Cement: soluble alkali (Na ₂ O _e) - 0.066%							
Cement: total alkali (Na ₂ O _e) - 0.325%							
Slurry Density (lb/gal)	Yield (ft ³ /sk)	Water (gal/sk)	C/W (g/mL)	ULHS Type		Soluble Alkali (kg/m ³)	Total Alkali (kg/m ³)
				4K	10K		
				(%BWOC)			
14	1.33	4.86	2.32	5.10		1.53	7.54
14	1.39	5.24	2.15		8.43	1.42	6.99
13	1.49	5.19	2.17	7.99		1.43	7.05
13	1.60	5.82	1.94		13.56	1.28	6.31
12	1.69	5.60	2.01	11.66		1.33	6.53
12	1.87	6.60	1.71		20.48	1.13	5.56
11	1.95	6.15	1.83	16.48		1.21	5.95
11	2.27	7.71	1.46		30.31	0.96	4.75
10	2.32	6.89	1.64	23.09		1.08	5.33
10	2.88	9.41	1.20		45.40	0.79	3.90
9	2.84	7.97	1.41	32.69		0.93	4.58
8	3.67	9.69	1.16	47.93		0.77	3.77

Table 11—Alkali concentration of Type I cement slurries with ULHS

Cement: soluble alkali (Na ₂ O _e) - 0.179%							
Cement: total alkali (Na ₂ O _e) - 0.479%							
Slurry Density (lb/gal)	Yield (ft ³ /sk)	Water (gal/sk)	C/W (g/mL)	ULHS Type		Soluble Alkali (kg/m ³)	Total Alkali (kg/m ³)
				4K	10K		
				(%BWOC)			
14	1.42	5.89	1.91		6.17	3.42	9.15
13	1.54	5.95	1.89	6.73		3.38	9.05
12	1.92	7.27	1.55		18.49	2.77	7.42
11	2.02	6.94	1.62	15.52		2.90	7.76
10	2.94	10.15	1.11		43.97	1.99	5.32
9	2.94	8.83	1.28	32.3		2.29	6.13
7	5.39	13.86	0.81	46.97		1.45	3.88

It should be emphasized that the data in the tables are calculated values based on simple slurry formulations.

We plan to design actual experiments under downhole conditions to ascertain any potential reactivity between ULHS and various levels of alkali content in lightweight cement slurries.

Software Program

To assist in designing cement slurries made with ULHS, a software program is being created. The program does not simulate cement jobs and fluid dynamics. Instead, the program focuses on the static final placement of the fluids (i.e., the slurry composition and slurry properties such as yield and density).

As suggested by project participants the foamed cement portion of the software program has been removed. Most service companies have a program that deals with foamed cement.

A beta version of the program will be distributed to the project participants near the end of July. This will give the participants a chance to test the program to see if there are any glitches.

Plans for Final Project Quarter

- **Field Job**

A field job using ULHS in a cement slurry is planned for the middle of July. The candidate well is located in Natrona County, Wyoming, on land owned by Rocky Mountain Oilfield Testing Center. The cementing application is for the production casing with a total depth of 5,900 feet. The ULHS slurry will cover from 4,150 to 2,635 ft. The cement that will be used is TXI Lightweight. The planned slurry design will contain 25% BWOC ULHS (6K), 0.2% BWOC proprietary retarder, and 7.57 gal/sk water for a design density of 10.0 lb/gal.

- **Software Program**

A beta version of the ULHS slurry design program will be distributed to the project participants near the end of July. This will give the participants a chance to test the program to see if there are any glitches. Any suggestions or comments about the program should be e-mailed to Fred Sabins.

- **Annular Seal Testing**

This project will also utilize other test methods to evaluate each of the three lightweight cement systems in their ability to maintain a seal under downhole stresses. An annular seal model will be used to measure bulk permeability across a cement system that has

been stressed from temperature or pressure cycling. As with previous testing, a pipe-in-pipe and pipe-in-soft configuration will simulate high and low restraints, respectively. **Figure 7** is a schematic of the pipe-in-soft configuration of the annular seal model.

Figure 7—Cross section of annular seal model for pipe-in-soft configuration

The inner pipe of the model will be the main conduit for the stressing medium. For instance, the inner pipe can contain heated fluids while the remainder of the system is at a different temperature; this simulates the hotter formation fluids that can be experienced during production. The inner pipe of the model can also be pressured up to 5,000 psi, simulating casing pressure testing which is believed by many to lead to loss of annular seal because of stresses placed on the cement due to the expanding and contracting casing.

The annular seal testing will be performed after each time the interior conduit pipe is pressurized. For the temperature cycling, the annular seal testing will be done after each temperature cycle.

In the pipe-in-soft configuration, the rubber sleeve surrounding the cement is able to withstand 25 psi. During the annular seal test, pressure can then be applied to the outside of the rubber sleeve, allowing the sleeve to make a fluid-tight seal on the outside of the cement. Pressurized nitrogen gas (<25 psi) can then be applied axially across the cement and the only paths for fluid flow is through cement or along the interface between the cement and the inner pipe. Any exiting nitrogen flow rate can be monitored and measured.

Conclusions

- In general, pressure cycling did not detrimentally affect the shear bond strength of the lightweight cement systems cured in a pipe-in-pipe configuration.
- Drillout testing revealed no significant faults with the ULHS or foamed cement systems.
- Preliminary expansion testing showed some expansion associated with neat TXI Lightweight cement and a slurry with TXI Lightweight cement and ULHS.
- Analysis testing of alkali content of ULHS slurries showed that there might be a potential for ASR. Investigations into ASR of ULHS cement slurries are continuing.

List of Acronyms and Abbreviations

API—American Petroleum Institute
ASR—alkali-silica reactivity
ASTM—American Society for Testing and Materials
Bc—Bearden units of consistency
BHCT—bottomhole circulating temperature
BHST—bottomhole static temperature
BWOC—by weight of cement
CaCl₂—chemical formula for calcium chloride
cp—centipoise
gal—gallon
H₂O—chemical formula for water
ID—inner diameter
in.—inch
J—Joule
lb—pound
md—millidarcy
min—minute
OD—outer diameter
psi—pound per square inch
rev—revolution
s—second
sk—sack of cement
QC—quality control
TXI—Texas Industries
TXI LW—manufactured lightweight cement available from TXI
ULHS—ultra-lightweight hollow (glass) spheres
3K—3,000-psi designation
6K—6,000-psi designation

References

1. API Specification 10A: "Specifications for Cements and Materials for Well Cementing," 22nd Edition, American Petroleum Institute, Washington, D.C., January 1995.
2. *Worldwide Cementing Practices*, First Edition, American Petroleum Institute, January 1991.
3. API Recommended Practice 10B: "Recommended Practice for Testing Well Cements," 22nd Edition, American Petroleum Institute, Washington, D.C., December 1997.
4. ASTM C 490-00a: "Standard Practice for Use of Apparatus for the Determination of Length Change of Hardened Cement Paste, Mortar, and Concrete," ASTM International, West Conshohocken, PA, 2002.
5. API Technical Report 10TR2: "Shrinkage and Expansion in Oilwell Cements," First Edition, American Petroleum Institute, Washington, D.C., July 1997.
6. "Alkali-silica reaction in concrete," *Structure and Performance of Cement*, Edited by J. Bensted and P. Barnes, Spon Press, p. 267, 2002.
7. ASTM C 114-99: "Standard Test Methods for Chemical Analysis of Hydraulic Cement," ASTM International, West Conshohocken, PA, 2000.

Appendix A—Quality Control Testing

TXI Lightweight Cement

TXI Information			CSI Information					Free Fluid			Rheology									
Cmt Type	Production Date	Date Received	CSI Log#	Bucket Opened	Test Date	Water Conc. (%bwoc)	mL	% by volume	Water Conc. (%bwoc)	Temp (°F)	300 RPM	200 RPM	100 RPM	60 RPM	30 RPM	6 RPM	3 RPM	10 sec G.S.	10 min G.S.	
LW	09/18/00	11/06/00	C-108 B-1	11/07/00	11/15/00	105	2	0.8	75	80	57	50	42	38	33	22	12	13	120	
LW	09/18/00	11/06/00	C-108 B-2	12/05/00	01/04/01	105	2	0.8	75	80	58	52	45	38	32	21	13	13	125	
LW	09/18/00	11/06/00	C-108 B-11	06/24/01	06/26/01	105	2	0.8	75	80	60	55	47	39	21	20	15	15	127	
LW	09/18/00	11/06/00	C-108 B-12	07/03/01																
LW	09/18/00	11/06/00	C-108 B-7	09/05/01																
LW	09/18/00	11/06/00	C-108 B-14	09/24/01																
LW	09/18/00	11/06/00	C-108 B-13	12/04/01																
LW	09/18/00	11/06/00	C-108 B-15	01/22/02																
LW	09/18/00	11/06/00	C-108 B-16	05/15/02																
LW	09/18/00	11/06/00	C-108 B-18	02/25/02																

CSI Information			Water Conc. (%bwoc)	45°F Strengths			60°F Strengths			80°F Strengths			120°F Strengths			Thickening Time			
Cmt Type	CSI Log#	Test Date		Time (Hr)	CaCl ₂ (%bwoc)	CS (psi)	Time (Hr)	CaCl ₂ (%bwoc)	CS (psi)	Time (hr)	CS (psi)	Time (Hr)	CS (psi)	Sch #	Initial Bc	Time to 70 Bc	Time to 100 Bc		
LW	C-108 B-1	11/15/00	24	2.0	166	24	1.0	254	24	523	24	1579	5	6	2:02	2:20			
LW	C-108 B-2	01/04/01	24	2.0	161	24	1.0	221	24	441	24	1225	5	5	2:00	2:17			
LW	C-108 B-11	06/26/01	24	2.0	158				24	501			5	9	1:57	2:18			
LW	C-108 B-12	08/14/01											5	7	2:03	2:22			
LW	C-108 B-7	09/10/01											5	6	1:57	2:17			
LW	C-108 B-14	10/16/01											5	4	2:07	2:26			
LW	C-108 B-14	11/13/01											5	5	2:05	2:25			
LW	C-108 B-14	12/20/01											5	7	2:03	2:22			
LW	C-108 B-13	01/11/02											5	8	1:58	2:12			
LW	C-108 B-15	02/08/02											5	7	2:05	2:15			
LW	C-108 B-18	03/04/02											5	13	2:11	2:27			
LW	C-108 B-18	04/09/02											5	23	1:54	2:16			
LW	C-108 B-16	05/16/02											5	19	2:00	2:12			
LW	C-108 B-18	06/26/02											5	24	1:53	2:10			

TXI Class H Cement

TXI Information			CSI Information					Water	Free Fluid		Rheology									
Cmt Type	Prod. Date	Date Received	CSI Log #	Bucket Opened	Test Date	Conc. (%bwoc)	mL	% by volume	Temp (°F)	300 RPM	200 RPM	100 RPM	60 RPM	30 RPM	6 RPM	3 RPM	10 sec G.S.	10 min G.S.		
Class H	09/27/00	11/06/00	C-108 A-1	11/06/00	11/07/00	38.0	1.9	0.8	80	89	72	53	45	37	16	10	---	---		
Class H	09/27/00	11/06/00	C-108 A-1	11/06/00	11/21/00	38.0	3.0	1.2	80	85	70	53	45	38	14	8	9	19		
Class H	09/27/00	11/06/00	C-108 A-2	11/06/00	01/11/01	38.0	3.0	1.2	80	95	80	60	51	42	20	12	15	26		
Class H	09/27/00	11/06/00	C-108 A-3	02/22/01	---	---	---	---	---	---	---	---	---	---	---	---	---	---		
Class H	09/27/00	11/06/00	C-108 A-4	06/26/01	---	---	---	---	---	---	---	---	---	---	---	---	---	---		
Class H	09/27/00	11/06/00	C-108 A-6	07/20/01	---	---	---	---	---	---	---	---	---	---	---	---	---	---		
Class H	09/27/00	11/06/00	C-108 A-7	08/27/01	---	---	---	---	---	---	---	---	---	---	---	---	---	---		
Class H	09/27/00	11/06/00	C-108 A-8	01/14/02	---	---	---	---	---	---	---	---	---	---	---	---	---	---		
Class H	09/27/00	11/06/00	C-108 A-10	06/11/02	---	---	---	---	---	---	---	---	---	---	---	---	---	---		

CSI Information			Water Conc. (%bwoc)	100°F Strengths		140°F Strengths		Thickening Time		
Cement Type	CSI Log#	Test Date		Time (Hr)	CS (psi)	Time (Hr)	CS (psi)	Sch #	Initial Bc	Time to 100 Bc
Class H	C-108 A-1	11/07/00	38.0	8	524	8	2088	---	---	---
Class H	C-108 A-1	11/29/00	38.0	8	480	8	2249	5	12	2:03
Class H	C-108 A-1	02/22/01	38.0	---	---	8	1617	---	---	---
Class H	C-108 A-1	02/22/01	38.0	---	---	8	1550	---	---	---
Class H	C-108 A-2	01/11/01	38.0	8	1201	8	1964	5	7	1:54
Class H	C-108 A-2	01/12/01	38.0	8	1039	8	1756	---	---	---
Class H	C-108 A-2	02/12/01	38.0	8	424	8	1717	---	---	---
Class H	C-108 A-3	02/22/01	38.0	---	---	8	1567	---	---	---
Class H	C-108 A-3	02/22/01	38.0	---	---	8	1580	---	---	---
Class H	C-108 A-4	06/29/01	38.0	---	---	---	---	5	17	2:00
Class H	C-108 A-6	08/13/01	38.0	---	---	---	---	5	15	1:50
Class H	C-108 A-7	09/06/01	38.0	---	---	---	---	5	19	2:00
Class H	C-108 A-3	10/09/01	38.0	---	---	---	---	5	12	1:53
Class H	C-108 A-3	11/13/01	38.0	---	---	---	---	5	13	1:50
Class H	C-108 A-3	12/21/01	38.0	---	---	---	---	5	14	1:45
Class H	C-108 A-3	01/11/02	38.0	---	---	---	---	5	13	2:00
Class H	C-108 A-8	02/08/02	38.0	---	---	---	---	5	28	1:55
Class H	C-108 A-8	03/05/02	38.0	---	---	---	---	5	14	1:55
Class H	C-108 A-8	04/04/02	38.0	---	---	---	---	5	24	2:00
Class H	C-108 A-8	05/15/02	38.0	---	---	---	---	5	22	1:50
Class H	C-108 A-10	06/25/02	38.0	---	---	---	---	5	26	1:50

TXI Type I Cement

TXI Information			CSI Information			Water Conc. (%bwoc)	Rheology									
Cement Type	Production Date	Date Received	CSI Log #	Bucket Opened	Test Date		Temp (°F)	300 RPM	200 RPM	100 RPM	60 RPM	30 RPM	6 RPM	3 RPM	10 sec G.S.	10 min G.S.
Type I	09/27/00	11/06/00	C-113 A-1	11/09/00	11/17/00	46.0	80	75	62	48	40	33	17	10	10	19
Type I	09/27/00	11/06/00	C-113 A-2	11/20/00	11/20/00	46.0	80	80	65	49	40	34	17	10	10	19
Type I	09/27/00	11/06/00	C-113 A-3	12/04/00	12/05/00	46.0	80	78	64	50	40	32	16	10	10	19
Type I	09/27/00	11/06/00	C-113 A-4	01/09/01	01/09/01	46.0	80	80	65	52	40	33	17	11	10	20
Type I	09/27/00	11/06/00	C-113 A-5	11/09/00	---	---	---	---	---	---	---	---	---	---	---	---
Type I	03/10/01	03/19/01	C-189 A-1	03/20/01	03/20/01	46.0	80	77	63	51	39	32	17	10	10	20
Type I	03/10/01	03/19/01	C-189 A-5	06/01/01	---	---	---	---	---	---	---	---	---	---	---	---
Type I	03/10/01	03/19/01	C-189 A-8	07/11/01	---	---	---	---	---	---	---	---	---	---	---	---
Type I	03/10/01	03/19/01	C-189 A-6	10/16/01	---	---	---	---	---	---	---	---	---	---	---	---
Type I	03/10/01	03/19/01	C-189 A-17	08/30/01	---	---	---	---	---	---	---	---	---	---	---	---
Type I	03/10/01	03/19/01	C-189 A-11	01/17/02	---	---	---	---	---	---	---	---	---	---	---	---
Type I	03/10/01	03/19/01	C-189 A-12	02/17/02	---	---	---	---	---	---	---	---	---	---	---	---
Type I	03/10/01	03/19/01	C-189 A-13	02/28/02	---	---	---	---	---	---	---	---	---	---	---	---
Type I	03/10/01	03/19/01	C-189 A-14	03/27/02	---	---	---	---	---	---	---	---	---	---	---	---
Type I	04/19/02	05/02/02	C-418-A	05/02/02	---	---	---	---	---	---	---	---	---	---	---	---
Type I	04/19/02	05/02/02	C-418-B	06/19/02	---	---	---	---	---	---	---	---	---	---	---	---

CSI Information			Water Conc. (%bwoc)	100°F Strengths			45°F Strengths			60°F Strengths			80°F Strengths			Thickening Time		
Cement Type	CSI Log #	Test Date		Time (Hr)	CS (psi)	Time (Hr)	CaCl ₂ (%bwoc)	CS (psi)	Time (Hr)	CaCl ₂ (%bwoc)	CS (psi)	Time (Hr)	CS (psi)	Time (Hr)	Test Date	Sch #	Int. Bc	Time to 100 Bc
Type I	C-113 A-1	11/17/00	46.0	---	---	---	---	---	---	---	---	24	1646	11/15/00	4	9	2:17	---
Type I	C-113 A-2	11/27/00	46.0	8	754	24	2607	2	737	24	1	1194	1689	01/05/01	4	8	2:21	---
Type I	C-113 A-2	02/12/01	46.0	---	---	---	---	---	---	---	---	24	2022	---	---	---	---	---
Type I	C-113 A-3	12/05/00	46.0	---	---	24	2585	---	---	---	---	---	---	---	---	---	---	---
Type I	C-113 A-4	01/17/01	46.0	8	729	24	2527	2	1042	---	---	---	---	01/17/01	4	4	2:09	---
Type I	C-113 A-5	02/15/01	46.0	8	931	24	2568	---	---	---	---	---	---	---	---	---	---	---
Type I	C-113 A-5	02/22/01	46.0	---	---	24	2334	---	---	---	---	---	---	---	---	---	---	---
Type I	C-113 A-5	02/22/01	46.0	---	---	24	2590	---	---	---	---	---	---	---	---	---	---	---
Type I	C-113 A-5	02/22/01	46.0	---	---	24	2250	---	---	---	---	---	---	---	---	---	---	---
Type I	C-189 A-1	03/20/01	46.0	8	916	24	2364	---	---	24	1	1238	1737	03/20/01	4	12	1:42	---
Type I	C-189 A-5	06/29/01	46.0	---	---	---	---	---	---	---	---	---	---	06/29/01	4	10	1:37	---
Type I	C-189 A-8	08/15/01	46.0	---	---	---	---	---	---	---	---	---	---	08/15/01	4	6	1:50	---
Type I	C-189 A-17	09/12/01	46.0	---	---	---	---	---	---	---	---	---	---	09/12/01	4	6	2:00	---
Type I	C-189 A-6	10/16/01	46.0	---	---	---	---	---	---	---	---	---	---	10/16/01	4	7	1:41	---
Type I	C-189 A-6	11/13/01	46.0	---	---	---	---	---	---	---	---	---	---	11/13/01	4	7	1:53	---
Type I	C-189 A-6	12/21/01	46.0	---	---	---	---	---	---	---	---	---	---	12/21/01	4	6	1:46	---
Type I	C-189 A-11	01/14/02	46.0	---	---	---	---	---	---	---	---	---	---	01/14/02	4	8	1:42	---
Type I	C-189 A-12	02/11/02	46.0	---	---	---	---	---	---	---	---	---	---	02/11/02	4	15	1:40	---
Type I	C-189 A-13	03/04/02	46.0	---	---	---	---	---	---	---	---	---	---	03/04/02	4	14	2:10	---
Type I	C-189 A-14	04/02/02	46.0	---	---	---	---	---	---	---	---	---	---	04/02/02	4	21	1:46	---
Type I	C-418-A	05/03/02	46.0	---	---	---	---	---	---	---	---	---	---	05/03/02	4	27	2:15	---
Type I	C-418-B	06/25/02	46.0	---	---	---	---	---	---	---	---	---	---	06/25/02	4	32	2:00	---

Appendix B—Laboratory Procedures for Foamed Cement

The working draft of ISO 10426-4¹ outlines the recommended practices for the atmospheric generation and testing of foamed cement slurries and their corresponding unfoamed base slurries. The procedures discussed in this appendix and used for this project were borrowed from ISO 10426-4.

B.1 Preparing Unfoamed Base Slurry

B.1.1 Calculation of Base Cement With and Without Surfactants

Because the final slurry for foamed cement contains surfactant(s), these materials cannot be added to the base slurry for initial mixing. This will require that the density of the base slurry be adjusted to compensate for the later addition of the surfactant(s) prior to foaming.

Example: Slurry Design: Class G Cement + 0.2 gal/sk Surfactant
Base slurry density = 14.5 lb/gal
Surfactant weight = 10 lb/gal

Base Slurry Calculations:	<u>Weight</u>	<u>Volume</u>
Cement	94 lb	3.59 gal
Surfactant	2 lb (0.2 gal * 10 lb/gal)	0.2 gal
Water	<u>55.39 lb</u>	<u>6.65 gal</u>
Total	151.39 lb	10.44 gal

Calculation of True Weight % Contributions:

Cement	62.1 %	(94/151.39)
Surfactant	1.3 %	(2/151.39)
Water	36.6 %	(55.39/151.39)

Slurry without Surfactants:	<u>Weight</u>	<u>Volume</u>
Cement	94 lb	3.59 gal
Water	<u>55.39 lb</u>	<u>6.65 gal</u>
Total	149.39 lb	10.24 gal

Slurry Density without Surfactants: 149.39/10.24 = 14.59 lb/gal

B.2 Equipment

B.2.1 Blender Container

A special blending container is required for preparing foamed cement at ambient pressure in the laboratory. (A typical blending container is shown in Figure B.1) The blending container is similar to the one used for standard slurry preparation except that it has a threaded cap with an O-ring seal. The cap has a small hole (approx. 3/4-in. diameter) in the center fitted with a removable plug that has an O-ring seal.

B.2.2 Multi-Blade Assembly

The multi-blade assembly is what is used during this project. The multi-blade or stacked-blade assembly is constructed of a series of assemblies, each blade corresponding to the requirements of ISO 10426-2², clause 5. The assembly consists of five (5) standard blades attached to a central shaft, and spaced equally throughout the mixing container. A typical assembly is shown in Figure B.1.

Fig. B.1—Example of a typical blending container

B.3 Container Volume

Accurate determination of the volume of the blending container is critical to this procedure. The calculations for slurry volume and foamed cement density are based on this volume determination. Weigh the clean, dry, blending container (including mixing assembly, screw-on lid and screw-in plug for the lid). Remove the screw-on lid from the mixing container and then remove the screw-in plug from the lid. Fill the mixing container with water and then screw the lid on tightly. Pour additional water into the hole in the lid for the plug until the container is completely filled, and then screw the plug tightly into the lid. Wipe the excess water that exits from the plug's vent hole, and then weigh the container again. The weight of the water inside the container is then divided by the density of the water to determine an accurate volume for the mixing container.

B.4 Preparing Base Cement Slurry

This method assumes that the base slurry as described in Section B.1.1 is being prepared in a separate mixing container, and this slurry is then to be weighed into the mixing container described in Section B.2.1. To prepare sufficient volume may require multiple mixes with the standard mixing procedure.

Base slurries containing all additives except foaming surfactant(s) should be prepared according to ISO 10426-2², clause 5. When possible, the temperature of the cement sample, additives, and mix water should be within $\pm 2^{\circ}\text{C}$ (3°F) of the respective temperatures recorded from 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 should be calibrated annually to a tolerance of ± 3.3 rev/s (200 rpm) at 66.7 rev/s (4,000 rpm) and ± 8.3 rev/s (500 rpm) at 200 rev/s (12,000 rpm). As required, the density of the unfoamed cement slurry can be determined by methods found in ISO 10426-2², clause 6.

B.5 Determining Slurry Volumes and Weights

B.5.1 Slurry Volume

Determine the volume of unfoamed cement slurry to be mixed. The total volume of unfoamed cement slurry should include the volume of the surfactant(s) to be added to the slurry. The surfactant(s) is to be added after the initial mixing of the base slurry. The volume of unfoamed slurry to be placed in the container may be determined by the following procedure.

When it is desired to foam a slurry with a specific amount of gas per unit volume of slurry (foam quality), the resultant density of the foamed slurry must be determined. This can be calculated by Equation 1.

$$FD = (100 - \%G) \div 100 \times UFDS \quad (1)$$

Where:

FD	=	Foamed density of the slurry
%G	=	Percentage of gas in final foamed slurry
UFDS	=	Unfoamed slurry density with surfactant(s)

When a desired foamed slurry density is known or after calculating it with Equation 1, determine the grams of cement slurry including surfactant(s) that is to be placed into the foam blender to prepare the foamed slurry. This can be calculated by Equation 2.

$$GUFS = CV \times FD \quad (2)$$

Where:

GUFS	=	Grams of unfoamed slurry including surfactant(s) to be placed into the foam mixer
CV	=	Container volume of foam mixer (mL)
FD	=	Foamed density of the slurry (g/mL)

Example:

Container volume	=	1170 mL
Base slurry density	=	14.5 lb/gal (1.74 g/mL)
Foamed cement density	=	10.0 lb/gal (1.2 g/mL)
Unfoamed slurry weight	=	$1170 \text{ mL} \times 1.2 \text{ g/mL} = 1404 \text{ g}$

B.5.2 Surfactant(s) and Slurry Weight

The surfactant(s) weight is determined by taking the unfoamed slurry weight and multiplying by the percent by weight of surfactant(s). The slurry weight is determined by taking the unfoamed slurry weight and subtracting the surfactant(s) weight. This can be calculated by Equation 4.

$$GS = GUFS \times (\% \text{Surfactant} / 100) \quad (3)$$

Where: GS = Grams of surfactants (total) to place into the foam mixer with the unfoamed slurry without surfactant(s)
GUFS = Total grams unfoamed slurry prepared in Section B.1

$$GUSM = GUFS - GS \quad (4)$$

Where: GUSM = Grams of unfoamed slurry without surfactant(s) to be placed into the mixer.

Example: Unfoamed slurry weight = 1404.1 g
Percent by weight of surfactant = 1.3 %
Surfactant weight = $1404.1 \times 0.013 = 18.5$ g
Slurry weight = $1404.1 - 18.5 = 1385.6$ g

B.6 Preparing the Atmospheric Foamed Slurry

Based on the volume calculated in Section B.5.1, weigh the appropriate amount of the prepared slurry into the special mixing container. Add the calculated amount of surfactant(s). The final weight of the cement slurry and added surfactant(s) should be checked against the final desired base slurry density. Before foaming, verify that the total weight of the slurry and added surfactant(s) corresponds to the weight calculated in Section B.5.2.

B.6.1 Generating a Foamed Cement

Make sure the mixing container is sealed. Using the blade assembly described in Section B.2.2, the slurry should be mixed at the 12,000 rpm setting for 15 seconds. Because of the increase in slurry volume and viscosity, the maximum rpm of the blender could be less than 12,000 rpm. The maximum attainable rpm will depend on the power of the blender, slurry density, and foam quality. Record and report the final rpm of the mixer.

During the mixing, there will be a noticeable change in the sound (pitch) from the blender. After mixing, there may be some slight pressure in the mixing container because of temperature increases and energy imparted to the foam during the foaming process. Be careful when removing the top of the mixing container. After mixing, open the sampling port or container lid, and verify that the slurry completely fills the slurry-mixing container. If the slurry does not fill the mixing container at the end of the 15-second

mixing, it is doubtful the slurry will foam properly under field conditions. The slurry should be redesigned.

B.7 Atmospheric Testing of Foamed Cement Slurries

Because of the high air entrainment in a foamed cement slurry, it is necessary to modify some of the standard testing procedures to prevent obtaining erroneous test results.

B.7.1 Determining Foamed Slurry Density

The density of the foamed slurry should be determined by pouring it into a container with a large open top that has a known volume when completely filled. Weigh the container, pour the foamed slurry into the container, and level the top with a straight blade. Wipe the outside of the container clean, and weigh the container with the foamed slurry. The density of the foamed slurry in the container is determined by dividing the slurry mass by the container volume and converting to the appropriate density units.

NOTE: Downhole densities may be different than designed values depending on pressure and temperature conditions. Special software is used to predict downhole densities.

B.7.2 Determining Slurry Stability

B.7.2.1 Unset Slurry Stability

Evaluate the foam stability by pouring a sample of the foamed cement slurry into a container or graduated cylinder for 2 hours of continued evaluation. Cover or seal the top of the container to prevent drying or dehydration of the sample. Since the main purpose of this test is to check for settling and stability in the foamed slurry, the visual appearance of the foamed slurry (such as free fluid, settling, or bubbles concentrated in a specific area) must be noted. If desired, density measurements may be made of the foam at multiple locations in the cylinder after the 2-hour period. To determine the density of the slurry at various locations in the cylinder, a large syringe with a Tygon tube on it can be used to remove small portions from the top, middle, and bottom. The removed slurry can then be transferred to a smaller graduated cylinder to determine the weight of a known volume of the slurry. From there, the specific gravity and density can be determined.

Pour the foamed slurry into a standard 250-mL graduated cylinder that is used for free-fluid testing. Cover the top of the cylinder to prevent dehydration, place it onto the counter-top, and visually examine it during the 2-hour period. The cylinder cannot be cured at temperatures above the ambient temperature at which the foamed slurry was prepared because an increase in temperature will increase the bubble size and may have an effect on the slurry stability.

B.7.2.2 Set Slurry Stability

Check foam stability by curing samples until they are set for density gradient measurement throughout the sample. These may be cured in non-greased, covered 50.8-mm (2-in.) diameter, 101.6-mm (4-in.) tall cylinders or any appropriate covered container. Use of grease or other mold-release agents should be avoided as these

materials may affect the stability of the foamed cement.

Cut or break the samples into sections, mark them from the top to the bottom, and measure the specific gravity of each section. The specimen should not be cut with a saw that uses water. The use of water may cause the specimen to absorb water and change the density of the specimen. Large variations in density from sample top to bottom are an indication of instability. When determining the specific gravity by Archimedes principal, it is recommended that a beaker of fresh water be placed on a scale and tared. The specimen is placed into a loop of fine string (or thread) and suspended in the water for the first measurement for determining the volume of the specimen (V). The volume of the specimen (mL) will be equal to the weight of the water displaced by the specimen when suspended in the water. The weight of the specimen being suspended in the water must be determined quickly to prevent the specimen from absorbing water and giving erroneous results. The specimen is then lowered to rest on the bottom of the beaker of water to obtain the actual weight of the specimen (W). The specific gravity (SG) is then determined by dividing the weight, W (in grams) by volume, V (in mL). The slurry density can also be determined ($SG \times 8.33 = \text{lb/gal}$).

Signs of foam instability include the following:

- More than a trace of free fluid.
- Bubble breakout noted by bubbles appearing on the surface of the sample.
- Excessive gap at the top of the specimen. Minor meniscus effects are normal.
- Visual signs of density segregation as indicated by streaking or light to dark color change from top to bottom.
- Large variations in density from sample top to bottom.

B.7.3 Determining Compressive Strength

The foamed cement slurry is poured into a curing mold that can be sealed. The sealing lid prevents the foamed slurry from expanding out of the curing mold as it is heated. This expansion can result in an undesired density decrease. The mold can be a standard 50.8-mm (2-in.) cube mold with a cover clamped to the top.

The sealed mold containing the foamed cement slurry is then placed into an atmospheric water bath, cured, and the strength is determined as specified by API. The temperature is normally limited to approximately 65°C (149°F), but can sometimes be increased to 90°C (194°F) if there is sufficient seal to prevent the slurry from expanding out of the curing mold.

B.8 Determining Other Tests on Base Unfoamed Slurry

A slurry that is foamed at atmospheric pressure should not be tested under pressure. Applying pressure to a foamed slurry prepared at atmospheric pressure will compress the foam, changing the density and gas ratio. This can also allow contamination when tested in a HPHT consistometer for thickening time.

For the following tests, the base unfoamed slurry without the surfactant(s) is prepared according to ISO 10426-2², clause 5. After the slurry is prepared, the mixer is stopped

and the surfactant(s) added and stirred gently with a spatula to distribute it uniformly in the slurry. It is recommended the slurry be transferred gently from the mixing container to a beaker and back three times to ensure a uniform distribution. The use of a small amount of material for preventing/breaking air entrainment in slurries that are not foamed is permitted for these tests. Materials to prevent/break air entrainment should not be used in any foamed slurries.

B.8.1 Determining Thickening Time

Since the surfactant(s) will affect the thickening time, and the foam itself does not affect the thickening time of a cement slurry, the thickening time test is normally performed using a standard HPHT consistometer on the base unfoamed cement slurry containing the surfactant(s).

The thickening time test of the unfoamed slurry containing the surfactant(s) will be performed using the procedures in ISO 10426-2², clause 9.

B.8.2 Determining Fluid Loss

Fluid-loss tests performed with a foamed cement prepared at atmospheric pressure will not yield reliable results. The fluid loss values obtained from a foamed cement slurry will be slightly less than that of the base unfoamed cement slurry. The fluid loss of the base unfoamed cement is normally used as an indication of the fluid loss of the foamed cement slurry.

The static fluid-loss test of the unfoamed slurry containing the surfactant(s) is performed using the procedures in ISO 10426-2², clause 10.

B.8.3 Determining Rheological Properties

With the concentration of gas in a foamed slurry changing continuously during pumping of the job, it is impractical to perform rheological testing at all the foam quality concentrations that are needed to model the frictional pressures during pumping of a foamed slurry. Use of a rotational viscometer will result in separation of the gas from the slurry, causing erroneous results. Correlations can be used to convert the rheological properties of the base unfoamed slurry to that of a foamed cement with varying foam qualities to simulate the job.

The rheological test of the unfoamed slurry containing the surfactant(s) is performed using the procedures in ISO 10426-2², clause 12.

References

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- ¹ ISO 10426-4: "Petroleum and Natural Gas Industries—Cements and Materials for Well Cementing, Part 4: Recommended Practice for Atmospheric Foam Cement Slurry Preparation," working draft 2001.
 - ² ISO 10426-2: "Petroleum and Natural Gas Industries—Cements and Materials for Well Cementing, Part 2: Recommended Practice for Testing of Well Cements," 1998.