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## Laboratory Cement Test Report-13.5 Tail Slurry PB 3 @ 202 BHCT

Fluid No : GCO 11PB046001	Client : Encana	Location / Rig :	Signatures
Date : Mar-23-2011	Well Name : SGU 8513F-24 F25 496	Field : Story Gulch	Joseph Eslinger

Job Type	Longstring	Depth	12750.0 ft	TVD	12500.0 ft
BHST	260 degF	BHCT	202 degF	BHP	8125 psi
Starting Temp.	80 degF	Time to Temp.	01:00 hr:mn	Heating Rate	2.03 degF/min
Starting Pressure	725 psi	Time to Pressure	01:00 hr:mn	Schedule	9.8-4

## Composition

Slurry Density	13.50 lb/gal	Yield	2.90 ft <sup>3</sup> /sk	Mix Fluid	14.395 gal/sk
Solid Vol. Fraction	33.6 %	Porosity	66.4 %	Slurry type	Conventional

## Blend 15/85 Poz/A

Code	Mass Per Sack
D901	80.0 lb
D035	11.0 lb

Code	Concentration	Sack Reference	Component	Blend Density	Lot Number
15/85 Poz/A		91.0 lb of BLEND	Blend	190.94 lb/ft <sup>3</sup>	
Fresh water	14.395 gal/sk		Base Fluid		
D151	50.000 %BWOB		CaCO <sub>3</sub> weight		
D178	35.000 %BWOB		Silica		
D020	3.000 %BWOB		Extender		
D112	1.100 %BWOB		Fluid loss		
D201	0.600 %BWOB		Retarder		
D046	0.200 %BWOB		Antifoam		

## Rheology (Average readings)

(rpm)	(deg)	(deg)
300	98.0	81.0
200	72.0	70.0
100	43.5	60.5
60	30.5	48.0
30	21.5	40.5
6	12.5	28.5
3	10.5	24.0
10 sec Gel	11	29
10 min Gel	28	35
1 min Stirring	97	77
Temperature	80 degF	180 degF
	Pv: 85.506 cP	Pv: 43.340 cP
	Ty: 13.80 lbf/100ft <sup>2</sup>	Ty: 40.55 lbf/100ft <sup>2</sup>

## Thickening Time

Consistency	Time
POD :	03:27 hr:mn
30 Bc	03:41 hr:mn
50 Bc	03:45 hr:mn
70 Bc	03:48 hr:mn
Remark : Thickening time do not include batch time	

## Free Fluid

0.0 mL/250mL	in 2 hrs
At 180 degF and 0 deg incl.	
Sedimentation	None

## Fluid Loss

API Fluid Loss	298 mL
136 mL in 25 min at 180 degF	and 1000 psi

## UCA Compressive Strength

Time	CS
04:20 hr:mn	500 psi
05:05 hr:mn	1000 psi
24:00 hr:mn	1797 psi
193:00 hr:mn	1530 psi

## Comments

General Comment :
Fann Reading Comment :
Thickening Time Comment :
Other test Comment : ; ; ; ;

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Client :  
Formation :  
Country :

Well :  
District :

Schlumberger

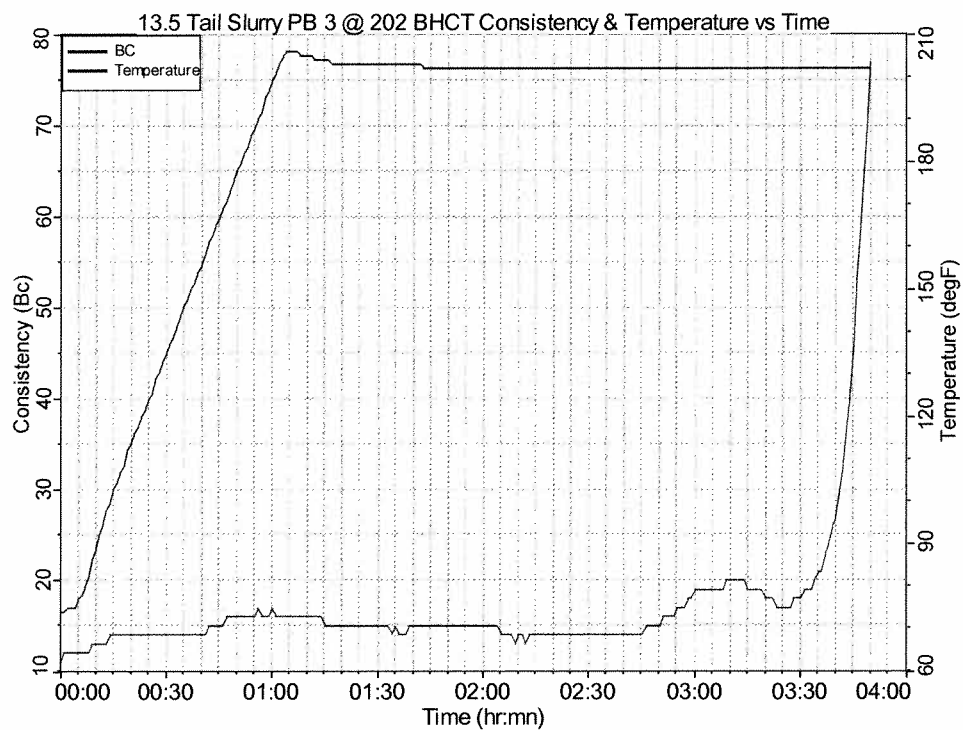
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LabDB \*

Client : Encana  
Well : SGU 8513F-24 F25 496  
String : Longstring  
Rig :  
Country : USA



11PB046.cfw 03-24-2011 LoadCase Untitled Version wca-cem461\_08

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COGCC/Rifle Office

# Laboratory Cement Test Report

## 13.5 Tail Slurry PB 1 @ 260 BHST

Fluid No :	Client :	Encana	Location / Rig :
Date : Apr-21-2011	Well Name :	SGU 8513F-24	Field :
		F25 496	

Signatures

Aida Vazquez

Job Type	Longstring	Depth	12750.0 ft	TVD	12500.0 ft
BHST	260 degF	BHCT	202 degF	BHP	8125 psi
Starting Temp.	80 degF	Time to Temp.	01:00 hr:mn	Heating Rate	2.03 degF/min
Starting Pressure	725 psi	Time to Pressure	01:00 hr:mn	Schedule	9.8-4

**Composition**

Slurry Density	13.50 lb/gal	Yield	2.90 ft <sup>3</sup> /sk	Mix Fluid	14.394 gal/sk
Solid Vol. Fraction	33.6 %	Porosity	66.4 %	Slurry type	Conventional

**Blend 85:15 PozA**

Code	Mass Per Sack
A	80 lb
D035	11 lb

Code	Concentration	Sack Reference	Component	Blend Density	Lot Number
85:15 PozA		91 lb of BLEND	Blend	190.94 lb/ft <sup>3</sup>	
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D151	50.000 %BWOB		CaCO <sub>3</sub> weight		
D178	35.000 %BWOB		Silica		
D020	3.000 %BWOB		Extender		
D112	1.100 %BWOB		Fluid loss		
D201	0.600 %BWOB		Retarder		
D046	0.200 %BWOB		Antifoam		

**Acid Solubility**

Sample	Wt Before HCl	Wt After 3 Hrs	Wt After 6 Hrs	% Solubility
1	213.30 g	n/a	130.76 g	38.82%
2	213.72 g	117.22 g	n/a	45.04%

Note: This is a pilot test. Field may differ after testing. Please read field report carefully and compare to pilot report and load out. Contact the laboratory with any questions or concerns.

Schlumberger Private



**SPE 114759**

## **Test Method To Optimize Acid-Soluble Cement For Unconventional Gas Completions**

K. Luke, SPE, and K. Soucy, SPE, Trican Well Service Ltd.

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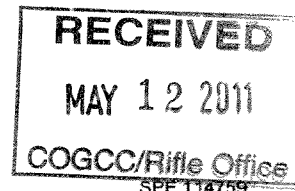
### **Abstract**

In recent developments of unconventional gas reservoirs, there has been a move towards drilling horizontally to increase formation contact with the objective to increase production. A longer horizontal extension gives greater formation contact. However, these extended laterals must be cemented which has caused constraints on optimal reservoir production. Studies have indicated that conventional cements are one of the primary causes resulting in reduced fracture initiation.

Acid-soluble cements have been advocated for use in unconventional gas reservoirs as a means to reduce the rate of inefficient fracture initiations. The logic behind using acid-soluble cement was based on field study examinations completed in 2005. These studies indicated that more than 25% of cemented horizontal laterals experienced fracture initiation problems compared to 4% for uncemented horizontal laterals. In acidizing acid-soluble cement in the area of strategically placed clusters, conditions approaching open hole are obtained similar to that of uncemented horizontal laterals. Cement remaining between clusters provides sufficient zonal isolation for well bore stability.

There are no standard tests to define the characteristics of acid-soluble cements such as acid solubility or rate of dissolution that are factors crucial to obtaining good cement removal and minimal near well-bore issues. In this context a prototype test method has been developed to more closely simulate dissolution of acid-soluble cement for unconventional gas reservoir applications. Data obtained indicate the benefits of such a method to define the most appropriate acid-soluble cements, acid treatments and develop improved systems.

Published test parameters vary considerably and given the number of variables involved in dissolution techniques there is a question as to what defines a good acid-soluble cement. Development of a test method that simulates down hole conditions provides a tool to design acid-soluble cement systems to optimize open hole around perforations and ultimately minimize fracture initiation problems.



## Introduction

Activity in exploitation of unconventional gas reservoirs has surged with the successes observed in such prospects as the Barnett Shale located within the Fort Worth basin Texas. The economics of the play has opened up the arena to other similar producing formations with an estimated 350 wells drilled in 2007 in the Woodford Shale that extends from Kansas to west Texas and in the potential development of the Horn River Basin/Muskwa Shale in British Columbia, Canada.

Implementation of horizontal wells in the early 2000's offered an economic solution in the Barnett Shale with the first wells providing three times the estimated ultimate recovery at twice the well cost compared to vertical wells. The industry trend has been towards longer laterals and multiple stage stimulations. Slick water fracturing treatments that have five to seven stages pumped at 8 – 13 m<sup>3</sup>/min with 1,890 – 3,790 m<sup>3</sup> fracturing fluid volumes and 113 - 320 tonnes proppant are not uncommon. Horizontal lateral lengths range from 450 to over 1500 m, most typically around 610 m, with the goal to maximise formation contact while containing the fracture treatment within the shale interval. Short laterals are not required to be cemented whereas the longer laterals are. Cemented laterals have been identified as a key parameter in strategies to obtain a successful completion along with perforation phasing, perforating lengths, and cluster spacing (Ketter et al., 2006; Leonard et al., 2007). Optimal perforation phasing for the Barnett Shale that aligns the perforations with the preferred fracture plane is 0°/180° phasing though 60° phasing is considered another viable option. The length of the perforation cluster is deemed crucial in determining whether a single dominant fracture is likely to form as opposed to creation of multiple competing fractures (El Rabaa, 1989). A cluster length of less than four times the wellbore diameter has proven to be effective (Ketter et al., 2006). Furthermore the spacing between perforation clusters has to be sufficient to enhance individual fracture growth and to prevent individual fractures from linking up. A spacing at 1.5 times the fracture height or greater has been determined by microseismic studies to be optimal (Fisher, 2004).

Acid-soluble cement (ASC) was implemented in cementing horizontal laterals in unconventional gas reservoirs after a study revealed that 1 in 4 of the longer cemented laterals experienced fracture initiation problems compared to 1 in 25 for the short horizontal laterals that were uncemented (Ketter et al., 2006). The reason for this is that when designing limited entry 60 degree phased perforations fluid will exit the well bore on the side of the hole. Fracturing difficulties arise when perforations are not aligned with the top and bottom of the hole and cement in the annulus can cause a choke effect that restricts fluid flow. The concept of using ASC was to minimize this effect by dissolving the ASC around the perforation cluster on acidizing with hydrochloric acid to produce conditions approaching that of an uncemented lateral or open hole while leaving cement between perforation clusters to provide annular isolation and well bore stability. Fracture complexity issues are considered to be addressed in the cementing program by using ASC (Vulgamore et al., 2007).

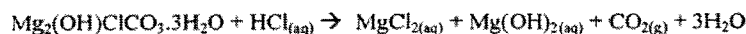
ASCs have proven successful in cementing horizontal laterals in unconventional gas wells as determined by a decrease in surface treating pressures as the acid hits the perforations and the cement dissolves and by both chemical tracer and cement bond logs. There is no defined test method to determine the most appropriate ASC slurry design with optimum performance in providing zonal isolation between perforations and maximum solubility in the perforation zone. Several test methods exist for determining the solubility of conventional and ASCs, both qualitative and quantitative. Comparison of data is limited because of the number of variables involved in each test method and comparison of slurry designs can only be made when a set of variables is fixed for a given test method. None of the test methods represent what occurs on acidizing ASC in horizontal laterals. A test method has been developed to better understand the impact of ASC on horizontal laterals with 60 degree phased perforations and to improve on ASC slurry design. The acid used in the test has been limited to 15% HCl as this is the acid most commonly used in unconventional gas wells.

## Acid-soluble cement

ASCs were initially developed as a solution for severe lost circulation across producing formations. The purpose was to block fractures, channels or pores by producing a solid plug or bridge across the formation that possessed compressive strength and decreased permeability to prevent dislodgment and prevent seepage losses that could then be removed by acid treatment without causing damage to the producing formation. A number of different ASCs were developed and are available for use. These are either some form of modified Sorel cement (Sweatman and Scoggins, 1990; Vincent et al., 1992; Bour et al., 1993; King et al., 1993; King and Totten, 1994) or conventional well cement containing an acid soluble component (Ketter et al., 2006; Carpenter and Wilton, 1996).

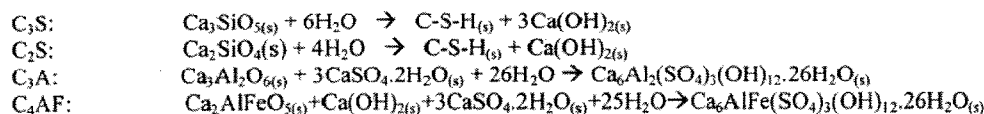
Sorel or magnesium oxychloride cements are formed from the combination of fine particles of calcined MgO and a magnesium chloride solution. The reaction products that produce bonding are Mg(OH)<sub>2</sub>, 3Mg(OH)<sub>2</sub>.MgCl<sub>2</sub>.8H<sub>2</sub>O and 5Mg(OH)<sub>2</sub>.MgCl<sub>2</sub>.8H<sub>2</sub>O up to 100°C and 9Mg(OH)<sub>2</sub>.MgCl<sub>2</sub>.8H<sub>2</sub>O and 2Mg(OH)<sub>2</sub>.MgCl<sub>2</sub>.8H<sub>2</sub>O above 100°C. These phases will disintegrate on exposure to acid due to leaching of MgCl<sub>2</sub>. The acid solubility of magnesium oxychloride cements can be enhanced significantly by addition of magnesium or calcium carbonates. The magnesium and calcium carbonates react with

the magnesium oxychloride to produce carbonated phases such as  $\text{Mg}_2(\text{OH})\text{ClCO}_3 \cdot 3\text{H}_2\text{O}$  and  $\text{Mg}_5(\text{CO}_3)_4(\text{OH})_2 \cdot 4\text{H}_2\text{O}$ . Addition of acid to these carbonated phases causes them to solubilise according to the following reactions:

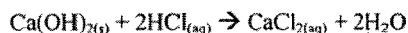


As well as enhancing the acid solubility of magnesium oxychloride systems, modifications to the chemistry can also be made to improve properties required for well cementing, such as compressive strength, temperature stability and cement density. The temperature stability can be improved by forming magnesium oxysulfate bonding phases rather than magnesium oxychloride phases that are also soluble in acid. The magnesium oxysulfate phases formed at 30 – 120°C on reacting calcined MgO with a solution of  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  or alternatively adding  $\text{CaSO}_4$  to a solution of  $\text{MgCl}_2$  are  $5\text{Mg}(\text{OH})_2 \cdot \text{MgSO}_4 \cdot 2 \cdot 3\text{H}_2\text{O}$ ,  $3\text{Mg}(\text{OH})_2 \cdot \text{MgSO}_4 \cdot 8\text{H}_2\text{O}$ ,  $\text{Mg}(\text{OH})_2 \cdot \text{MgSO}_4 \cdot 5\text{H}_2\text{O}$ ,  $\text{Mg}(\text{OH})_2 \cdot 2\text{MgSO}_4 \cdot 3\text{H}_2\text{O}$ . Combining stoichiometric mixtures of calcined MgO with magnesium and/or calcium chlorides, sulfates and carbonates can produce modified magnesium oxychloride/magnesium oxysulfate cement that on reaction with water produces cement that can be retarded and is stable at temperatures up to 100°C, provides good compressive strength and is > 98% acid soluble (Sweatman and Scoggins, 1990). Magnesium oxychloride and modified magnesium oxychloride/oxysulfate cements also bond well to a variety of inorganic materials and can be formulated with microspheres for low density or barite and hematite for high density.

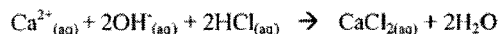
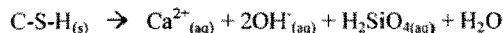
Conventional well cements are soluble in acid as evidenced by field studies on wells submitted to acidizing operations (Brady et al. 1989, Carpenter and Edwards 1994, Miranda and Gold 1997). How soluble the cement is depends on the acid used and its concentration. For normal Class G cement 12%/3% HCl/HF acid is more effective than 15% HCl acid. Conventional well cements are composed of a composite mixture of tricalcium silicate ( $\text{C}_3\text{S}$ ) and dicalcium silicate ( $\text{C}_2\text{S}$ ) embedded in a matrix of tricalcium aluminate ( $\text{C}_3\text{A}$ ) and tetracalcium aluminoferrite ( $\text{C}_4\text{AF}$ ). These components react with water to form hydrated products according to the following chemical equations:



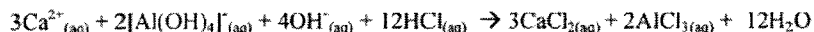
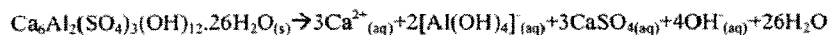
The calcium silicate hydrate (C-S-H) hydration product is the principal component of set cement and is the binder that gives set cement its compressive strength. The Ca/Si ratio and the Si/H ratio of the C-S-H are variable and range between 1.7 – 2.0 and 1.5 – 2.1 respectively. Gypsum ( $3\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ) is added to cement during manufacture to retard the aluminate hydration by forming ettringite ( $\text{Ca}_6\text{Al}_2(\text{SO}_4)_3(\text{OH})_{12} \cdot 26\text{H}_2\text{O}$ ) or monosulfate ( $\text{Ca}_6\text{AlFe}(\text{SO}_4)_3(\text{OH})_{12} \cdot 26\text{H}_2\text{O}$ ). Dissolution of set cement in hydrochloric acid (HCl) is caused by reaction of the acid with  $\text{Ca}(\text{OH})_2$  to form calcium chloride which is soluble in water:



This reaction essentially causes leaching of  $\text{Ca}(\text{OH})_2$  from the set cement. As  $\text{Ca}(\text{OH})_2$  is leached out of the cement the C-S-H and ettringite start to decompose, with release of  $\text{Ca}^{2+}$  to counteract the loss in  $\text{Ca}(\text{OH})_2$  and the set cement starts to disintegrate accelerating the dissolution. A number of potential ionic silicate species can exist and the following is a simplified equation showing potential effect of  $\text{Ca}^{2+}$  leaching from C-S-H.

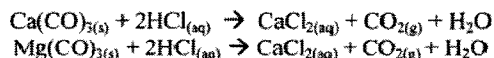


The potential breakdown of ettringite on leaching of  $\text{Ca}^{2+}$  ions and continued reaction with HCl is shown as follows. In this case both released  $\text{Ca}^{2+}$  and hydrous alumina can react with the acid.



Normal Class G cement can be almost completely dissolved if 12%/3% hydrochloric/hydrofluoric (HCl/HF) acid is used under dynamic conditions and there is enough acid employed (Brady et al., 1989). In general with 15% HCl acid and under static conditions the reaction is slow and it is believed that as the  $\text{Ca}^{2+}$  is leached from the C-S-H the siliceous component remaining forms a protective coating around the cement that prevents further dissolution. The siliceous component is soluble in the HF component of the 12%/3% HCl/HF acid and hence in this acid it is less likely to form a protective coating.

The solubility of conventional cement can be increased by adding an acid soluble component, usually magnesium or calcium carbonate (Carpenter et al., 1996). The carbonate acts in a number of ways. First it dissolves rapidly in acid increasing the permeability of the set cement and allows the acid to penetrate further into the set cement and aid in disintegration and dissolution.



Second, some of the carbonate is incorporated into the C-S-H and ettringite and increases the solubility of these phases to acid. The increased solubility on adding calcium carbonate to Class G cement is shown in Table 1. Data show the effect of increasing the calcium carbonate content from 5 to 200 % by weight of cement as compared to a set sample of Class G cement without addition of calcium carbonate when dissolved in 15% HCl as a function of time. The compressive strength of the set cement notably decreases as the carbonate content and the resulting acid solubility increases.

### Test Methods

Although there are a number of papers on the solubility of set ASC in acid, whether for dissolution of cement during acidizing operations or for developing an acid soluble plug for lost circulation in producing zones, there is no standard test method to determine ASC solubility. Test methods reported are both qualitative and quantitative but due to the variable parameters involved have led to marked differences in procedures and results.

Qualitative testing, usually involves curing 5.1 x 5.1 x 5.1 cm cubes of cement at temperature in a water bath for a given time period and then determining the weight loss of the set cement on placing it in acid at temperature for a pre-selected time. The choice and variability of the parameters in this test that affect the final results are considerable:

- Time and temperature of cure
- Method of removal of grease on the curing mold- if used
- Drying or desiccating the cured cement after demolding
- Length of time from demolding to acid testing and storage conditions
- Type and concentration of acid used
- Volume of acid used
- Static testing or agitated – method of agitation
- Cement cube suspended or placed in acid
- Temperature of the acid
- Length of time cement in acid
- Treatment (washing/drying) of cement cube after removing from acid

An attempt was made to develop a methodology for cement acid solubility in relation to acidizing operations to minimize on the inconsistencies arising from this test (Carpenter and Edwards, 1994). No methodology was adopted in testing ASCs for lost circulation plugs and as well as cement cubes, samples of solid material of unknown size and weight and even pulverized samples have been used for determining solubility.

Quantitative testing of ASCs typically uses a rotating-disk method that is commonly applied to reservoir rock to measure the rate at which the acid reacts with the exposed surface. Disks of ASC are cured at temperature in a water bath for a given period of time, then transferred to the rotating disk assembly cell and brought up to temperature and a pressure of 6.89 MPa. The disk, one surface exposed, is rotated at constant angular velocity in acid and samples of the bulk solution taken for analysis at selected time intervals. As with the qualitative method the choice and variability of the parameters are considerable.

- Time and temperature of cure
- Method of removal of grease on the curing mold- if used
- Size of the disk
- Type and concentration of acid used
- Volume of acid used
- Temperature of the acid
- Length of time cement in acid
- Angular velocity
- Time interval for sampling the bulk solution
- Volume of bulk solution taken

Data collected from the rotating-disk method can be used to determine the reaction rate constant and the order of reaction, terms that can be used in acid simulator programs to provide usable data at treatment conditions. However, no standard methodology for testing ASCs with the rotating-disk was ever developed.

### Solubility of ASC

Laboratory tests were initially performed on cement cube solubility at 25°C and atmospheric conditions. The ASC was mixed and blended according to ISO-10426-2 specifications and cured in 5.1 x 5.1 x 5.1 cm mold in a water bath for 24 hours unless otherwise specified. The faces of the molds were not greased so as to prevent contamination of the cement cubes that could affect their solubility in acid. After demolding, the cement cubes were damp dried with a paper towel, weighed and then placed in acid solution. At predetermined time intervals the cement cubes were removed from the acid, dipped in water to remove the excess acid, damp dried with a paper towel, weighed and then replaced back in the acid, care taken to place the same surface on the base of the container. The percentage acid solubility was determined from the weight loss according to the following equation:

$$\text{Acid Solubility (\% wt. loss)} = \frac{(\text{Initial Weight} - \text{Final Weight})}{\text{Initial Weight}} \times 100$$

Compressive strengths of each of the ASC systems investigated were also determined on crushed cement cubes after demolding.

Several series of tests were performed on the cement cubes to obtain information on the test parameters and their influence on acid solubility of ASCs. To determine the effect of acid volume on solubility, cement cubes of Class G cement blended with 150 and 200 % BWOC calcium carbonate ( $\text{CaCO}_3$ ) were placed in 375, 500 and 1000 mL of 15% HCl. As expected, Table 2, the higher the volume of acid used the greater the solubility, however, even at 120 minutes there was still 20-30% of the cement cube remained undissolved. Most of the reaction was observed to occur during the first 20 minutes after which time the reaction progressively slowed down. This has been attributed to formation of a protective skin of reaction product limiting further reaction though this is questionable. Cement cubes that had been placed in 500 mL of 15% HCl for up to 1 hour and then placed in a fresh solution of acid for a second hour showed a renewed burst of reaction and the cement cubes were almost completely dissolved. If the cement cubes had a protective skin then this renewed reaction would not have occurred. It was observed that as soon as the ASC contacts acid an immediate and very vigorous reaction occurs creating an in-situ dynamic system. This reaction causes a significant turbulence around the cement cube that allows greater contact with acid. As the acid becomes spent, reactivity decreases and conditions become more static resulting in reduced solubility.

The next series of tests were designed to determine if the curing time of the cement has an impact on the solubility. It was considered that the longer the cement cured and developed strength that the porosity/permeability would decrease and as such the solubility would decrease. The data provided in Table 3 show that this is in fact not the case and that the solubility increased the longer the cement was cured with the greatest effect occurring between 1 to 4 days and a lesser effect between 4 and 7 days. The increased solubility is likely due to the incorporation of carbonate into the Class G cement hydration products, C-S-H and ettringite, making them more susceptible to acid attack.

Although the acid typically used in the Barnett Shale unconventional gas is 15% HCl, other concentrations and acids were investigated to determine their potential and impact on solubility. The results of this series of tests are provided in Table 4. Increasing HCl acid concentration increases the solubility of the cement as expected with 28% HCl showing the greatest effect. The 12%/3% HCl/HF which is considered to be more effective in dissolving the C-S-H in the cement shows no difference compared to 15% HCl on the ASC. Acetic acid is a weak acid and this is clearly seen by the minimal solubility.

The acid solubility of the different types of ASC was compared with normal Class G cement and is presented in Table 5. All the ASCs contain a percentage of  $\text{CaCO}_3$  as an acid soluble component and all show similar solubilities, however the 24 hour compressive strengths vary considerably.

Although the cement cube solubility gives some indication of acid solubility of ASCs, it can be seen that the actual solubility depends on the choice of parameters used. There is also a question as to how the cement cube test relates to the actual use of ASCs in horizontal laterals and a more dynamic system down hole.

### Dynamic Acid Solubility – Development of New Test Method

In order to obtain a better understanding between the laboratory testing and field application a method simulating the field dynamics was developed. This allows validation of 'bench top' testing such as placing 5.1 x 5.1 x 5.1 cm cement cubes in



acid. Also, creating a dynamic flow simulates actual field application providing a better indication of the solubility effects of acid on ASCs.

A common well bore design of 114.3 mm production casing and 73 mm tubing typical of lateral wells cemented with ASCs was scaled to 50%. A pump, valves and pipe were used to create a well bore flow loop, Fig.1. Transparent strings of pipe allowed for full qualitative analysis throughout the duration of the test before, during and after being subjected to 10 L of 15% HCl pumped at 50 L/min. A well bore configuration was designed using a simulated production string with an outer casing to mold the ASC around the outside of the casing. Holes were drilled both through the simulated production string and outer mold to create perforations. These perforations were also scaled to match typical well bore schematics and also incorporated 60° phasing for simulating typical perforation clusters, Fig.1A. The mold was assembled to the production string with fasteners and once removed after the cement sets would provide the simulation of perforated casing and cement. The ASC was poured in the annulus of the production string and outer mold and allowed to set for 24 hrs.

After 24 hours, the mold was removed leaving the production casing with ASC and perforations. The cement was then wrapped with fibreglass screening to prevent any sand, representing a permeable formation that was placed around the cement sheath, from entering into the pump. An outer well bore containment, Fig.1B was used to contain the acid and direct any flow back into the loop if excess acid was forced through the perforations and into the sand, Fig.1C.

The simulated casing string with the cement sheath and fibreglass screening, Fig 2A, was centralized in the containment pipe and was filled with sand. An inner string (tubing) was inserted and spotted off the bottom of the simulated production casing. Acid was circulated through the tubing string and up the annulus back into the reservoir simulating field dynamics. The flow loop was operated for 30 min with samples of acid taken every 5 minutes and analyzed for acid content. Changes in the acid concentration are indicative of the acidizing solubility of the cement sheath and are shown in Table 6 for concentrations of  $\text{CaCO}_3$  ranging from 0% to 200% BWOC - the lower the %HCl the greater the solubility.

The cement sheath prepared from Class G cement with 0%  $\text{CaCO}_3$  showed a slight initial decrease in pH indicating low solubility. This was verified at the end of the test when the simulated casing string was removed and found to still have an intact cement sheath around it, Fig 2B. The sheath appeared to have some sort of gel type skin on the outer surface. The ASC Class G/ $\text{CaCO}_3$  - 50% BWOC sheath showed a greater decrease in pH from 15.0 to 14.3 over a 15 minute period and was then constant, indicating some degree of reaction. Fig. 2C shows that there was some reaction. Test results, Table 7, are a measure of the changes in the perforation tunnels. The conventional cement shows <1% increase in hole diameter where as the Class G/ $\text{CaCO}_3$ -50% BWOC had noticeable increase in diameters. Interconnection between lower perforations were also noted and indicated by the boxed regions in Table 7. The Class G/ $\text{CaCO}_3$  - 200% BWOC ASC system dissolved completely within the well bore perforation clusters with cement isolated on the top and bottom of the cluster, Fig.2D. This is indicative of isolation of ASC along the laterals between the perforation clusters. The dynamic flow of acid past the perforations provided increased solubility without requiring the acid to be forced through the perforations. Flow rate, acid volume, acid concentration, cement volume and perforations sizes were all kept constant to ensure comparative results. The data obtained indicates that the dynamic flow loop provides a better assessment on ASC solubility for use in unconventional gas completions and allows for better design and formulation of ASC systems.

## Conclusion

A number of conclusions can be drawn from the testing performed.

1. The % solubility of ASC depends on testing procedures and on the concentration and volume of acid used. Strong acids are more effective than weak acids.
2. Class G cement with  $\text{CaCO}_3$  acts as an acid-soluble cement. At least 30%  $\text{CaCO}_3$  is required to give some solubility based on cement cubes. Compressive strength, however, decreases as the % of  $\text{CaCO}_3$  increases.
3. Acid solubility increases with longer cure time up to 4 days and is likely due to incorporation of the carbonate into the hydrated phases
4. Class G cement with 200%  $\text{CaCO}_3$  has similar solubility to oxychloride and oxysulphate cements containing  $\text{CaCO}_3$  though, compressive strengths of the different ASCs differ considerably.
5. The rate of solubility of ASCs in acid is highest during the initial 20 minutes and the reaction during this time period is very vigorous. Static cement cube acid solubility for most ASCs in 500 mL 15% HCl indicates at most about 60% solubility after 2 hours. The decrease in rate and limitation of acid solubility is considered to be due to the decrease in reactivity of the acid rather than formation of a protective silica gel.
6. Dynamic testing using a scaled version of a simulated production string in a well bore flow loop verified the results of the static cement cube test in that acid solubility increased with increasing carbonate content in Class G/ $\text{CaCO}_3$  systems. The solubility rate of the ASCs was greater in the dynamic testing and differentiation between ASCs was

more evident. In the dynamic test Class G/CaCO<sub>3</sub> - 50% BWOC showed significantly less solubility than in the cement cube test whereas the Class G/CaCO<sub>3</sub>- 200% BWOC showed greater solubility.

7. The dynamic testing method gives a better indication of how ASC would behave in field application in unconventional gas reservoirs. This shows not only the potential of having complete solubility and open hole in the area of the perforations but also the fact that isolation occurs in the laterals between perforations.

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### Nomenclature

ASC – acid-soluble cement  
HCl – hydrochloric acid  
HF – hydrofluoric acid  
C<sub>3</sub>S – 3CaO.SiO<sub>2</sub> or Ca<sub>3</sub>SiO<sub>5</sub>  
C<sub>2</sub>S – 2CaO.SiO<sub>2</sub> or Ca<sub>2</sub>SiO<sub>4</sub>  
C<sub>3</sub>A – 3CaO.Al<sub>2</sub>O<sub>3</sub> or Ca<sub>3</sub>Al<sub>2</sub>O<sub>6</sub>  
C<sub>4</sub>AF – 4CaO.Al<sub>2</sub>O<sub>3</sub>.Fe<sub>2</sub>O<sub>3</sub> or Ca<sub>2</sub>AlFeO<sub>5</sub>  
BWOC – by weight of cement

### Conversion Factors

m<sup>3</sup>/min x 6.29 = bpm  
m<sup>3</sup> x 264.2 = gal  
tonne x 2205 = lb  
m x 3.281 = ft  
cm x 0.394 = in  
mm x 0.0394 = in  
MPa x 145.04 = psi  
(°C x 1.8) + 32 = °F

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**TABLE 1**  
**Class G/CaCO<sub>3</sub> cement blend – Impact of CaCO<sub>3</sub> content**  
**on acid solubility in 15% HCl and on compressive strength**

CaCO <sub>3</sub> content (% BWOC)	Acid Solubility (% wt loss)						Compressive Strength (MPa)
	0 min	20 min	40 min	60 min	120 min	1080 min	
0	0	2.4	4.6	6.3	9.3	13.8	17.4
10	0	2.5	4.9	6.9	8.9	19.5	16.8
30	0	23.8	26.4	28.2	30.4	36.0	15.2
50	0	24.9	28.8	30.7	32.7	36.1	11.3
100	0	28.4	36.9	39.1	40.7	42.9	6.5
150	0	29.9	35.9	38.8	41.6	nd	3.0
200	0	38.2	44.1	47.9	52.0	nd	2.0

nd – not determined

**TABLE 2**  
**Acid solubility of Class G/CaCO<sub>3</sub> set cement**  
**in different volumes of 15% HCl acid**

CaCO <sub>3</sub> content (% BWOC)	Volume of Acid (mL)	Acid Solubility (% wt. loss)				
		0 min	20 min	40 min	60 min	120 min
150	375	0	25.3	29.3	31.3	33.0
150	500	0	29.9	35.9	38.8	41.6
150	1000	0	45.0	55.3	60.8	72.6
200	375	0	35.1	41.0	43.0	45.5
200	500	0	38.2	44.1	47.9	52.0
200	1000	0	53.4	65.9	69.9	82.2

**TABLE 3**  
**Effect of Class G/CaCO<sub>3</sub> blended cement curing time on acid solubility in 15% HCl**

Carbonate content (% BWOC)	Curing time (days)	Acid Solubility (% wt. loss)					Compressive Strength (MPa)
		0 min	20 min	40 min	60 min	120 min	
150	1	0	25.3	29.3	31.3	33.0	2.0
150	4	0	42.2	46.8	48.4	50.4	4.3
150	7	0	41.8	44.4	45.5	47.1	9.4
200	1	0	35.1	41.0	43.0	45.5	3.0
200	4	0	48.9	54.3	56.3	58.5	4.6
200	7	0	51.7	55.4	57.2	59.2	6.2

**TABLE 4**  
**Class G/CaCO<sub>3</sub> (200%) set cement solubility in acid**

Type of acid <sup>1</sup>	Acid Solubility (% wt. loss)				
	0 min	20 min	40 min	60 min	120 min
10% HCl	0	19.3	24.8	27.3	30.4
15% HCl	0	38.2	44.1	47.9	52.0
28% HCl	0	65.3	69.8	72.5	85.4
12%/3% HCl/HF	0	43.4	47.1	48.3	51.0
10% Acetic Acid	0	0.6	1.6	2.2	3.1

<sup>1</sup> 500 mL volume used in all tests

**TABLE 5**

**Acid solubility of different types of cement in 15% HCl**

Type of cement	Acid Solubility (% wt. loss)					Compressive Strength (MPa)
	0 min	20 min	40 min	60 min	120 min	
Normal Class G	0	2.4	4.6	6.3	9.3	17.4
Class G/CaCO <sub>3</sub> -50% BWOC	0	24.9	28.8	30.7	32.7	11.3
Class G/CaCO <sub>3</sub> - 200% BWOC	0	38.2	44.1	47.9	52.0	3.0
Oxychloride cement/CaCO <sub>3</sub>	0	33.0	43.1	46.9	48.1	46.1
Oxysulphate cement/CaCO <sub>3</sub>	0	28.4	36.9	39.1	40.7	1.2

**TABLE 6**

**Change in filtrate HCl concentration under dynamic conditions**

Type of cement	Filtrate (%HCl)						
	0 min	5 min	10 min	15 min	20 min	25 min	30 min
Normal Class G	15.3	15.1	15.1	15.1	15.1	15.1	15.0
Class G/CaCO <sub>3</sub> -50% BWOC	15.0	14.9	14.6	14.3	14.3	14.3	14.3
Class G/CaCO <sub>3</sub> - 200% BWOC	15.0	14.6	14.3	14.0	13.4	13.0	12.4

**TABLE 7**

**Change in perforation hole diameter for the Class G/CaCO<sub>3</sub> set cement before and after acidizing with 15% HCl**

Top to Bottom	Original Hole Diameter (mm)	Hole Diameter After Acidizing (mm)	
		Horizontal	Vertical
1	8.50	16.90	23.82
2	7.95	18.77	20.29
3	8.16	15.86	24.70
4	8.12	11.70	20.16
5	8.33	19.70	21.00
6	7.83	17.80	32.60
7	8.00	25.10	32.10
8	8.44	42.70	21.30
9	8.40	36.30	46.80

Boxed area indicates interconnectivity between perforation holes

Fig. 1. Simulated well bore flow loop: A- simulated production string with outer casing and 60° phase perforations. B- simulated production string

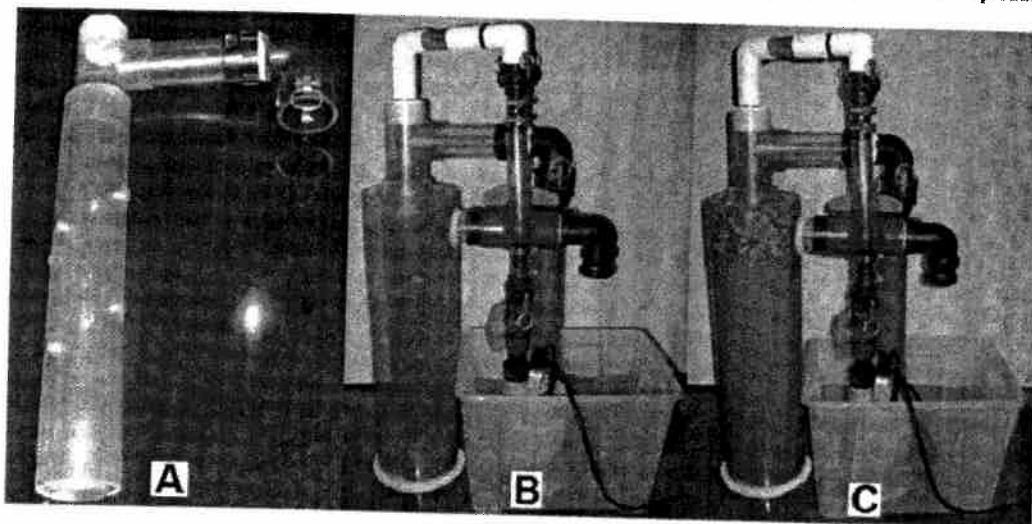


Fig.2. Cement sheath around the simulated production string: A-prior to testing with fibreglass screen, B- Class G cement after test, C- Class G/ $\text{CaCO}_3$  (50%) after test and D- Class C/ $\text{CaCO}_3$  (200%) after test.

