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SUMMARY OF CHEMICAL CEMENT

ALTERNATIVES PROTOCOLS

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EXECUTIVE SUMMARY

Data on Chemical Cement Alternatives were received from eight (8) Stakeholders in Calgary, Alberta and Riceboro, Georgia, USA. For a comprehensive document on the subject matter, three of the Stakeholders, Shell Canada, ConocoPhillips and William Butler from Alberta Energy Regulator, recommended a review of a United Kingdom (UK) Oil and Gas document titled "Guidelines on Qualification of Materials for the Abandonment of Wells". This document was authored by representatives from the following organizations:

1) Royal Dutch Shell
2) University of Dundee (UK)
3) Conoco Phillips UK
4) SINTEF Norway
5) TNO Netherlands (Netherlands Organisation for Applied Scientific Research)
6) Schlumberger UK
7) BP Norway
8) Halliburton UK
9) Oil and Gas UK

Protocols and properties to be tested were identified and reviewed from the Stakeholders' data received. Their details form the body of this Report. Some of the protocols were found to be applied across the industry, while some are company specific. There are some protocols and testing relevant to identifying the characteristics of Chemical Cement Alternatives that are not covered by the Stakeholders' practices or recommendation. Protocols to fill these shortfalls will be the subject of our next report.

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2) William Butler of Alberta Energy Regulator
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5) John Gould of SNF
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TABLE OF CONTENTS

Contents

EXECUTIVE SUMMARY 2
ACKNOWLEDGEMENTS 2
TABLE OF CONTENTS 3
INTRODUCTION 4
METHODOLOGY FOR WORK PACKAGES 1 & 2 5
PRESENTATION OF DATA AND RESULTS 8
NEXT MILESTONE DELIVERABLES 22
SCHEDULE "A" MILESTONE AND INVOICING SCHEDULE 23
REFERENCES 25
APPENDIX 26
METHODS OF TESTING 27
STAKEHOLDERS ENGAGED 33
STAKEHOLDER MEETING MINUTES 35

Table 1: Work Packages 4
Table 2: Stakeholder Participants 5
Table 3: Chemical Cement Alternatives [1] 6
Table 4 Properties of Chemical Cement Alternatives and Their Relevance [1] 7
Table 5: Safety and Toxicology Protocols for Chemical Cement Alternatives 8
Table 6: Leaching Toxicity Protocols for Chemical Cement Alternatives 8
Table 7: Groundwater Protection Protocols for Chemical Cement Alternatives 9
Table 8: Field Pilot Protocols for Chemical Cement Alternatives 10
Table 9: Testing Protocols for Modified Cements/ ceramics (non-setting) 10
Table 10: Testing Protocols for Grouts (non-setting) 11
Table 11: Testing Protocols for Thermosetting polymers and composites 13
Table 12: Testing Protocols for Thermoplastic polymers and composites 14
Table 13: Testing Protocols for Elastomeric polymers and composites 15
Table 14: Testing Protocols for Formation 15
Table 15: Testing Protocols for Gels 17
Table 16: Testing Protocols for Glass 17
Table 17: Testing Protocols for Metals 19
Table 18: Testing Protocols for Modified in-situ materials 19
Table 19: Protocols for Chemical Cement Alternatives Under Adverse Conditions 20
Table 20: Protocols for Field Deployment Verification of Chemical Cement Alternatives 21

INTRODUCTION

Wilkie Enterprises was contracted by PTAC to deliver the following:

- Identify, approach Stakeholders and develop standardized testing protocols for the approval of Chemical Cement Alternatives in the Province of Alberta
- Deliver a roadmap, complete with capable vendors and industry partners, to provide an achievable, repeatable, consistent testing process for Chemical Cement Alternatives.

The detailed scope of work comprises the following work packages:

Work Package	The Work
1	Verify current Chemical Cement Alternative protocols used by the major industry Stakeholders in Alberta
2	Research and identify Chemical Cement Alternative testing protocols used in the oil and gas industry of other provinces and major international markets
3	Review all identified protocols and recommend (and justify) standardised testing for each protocol based on: a) Industry best practice b) Test duration for each protocol c) Cost Estimate for each protocol
4	Upon review of all of the above information, recommend (and justify) a standard value for each property, where applicable, for all identified protocols
5	Propose criteria for approving laboratories for Chemical Cement Alternative protocols testing (if non-existent)
6	Identify and evaluate laboratories or facilities for conducting Chemical Cement Alternative protocol testing
7	Propose a list of "Approved Laboratories" for the above testing protocols
8	Propose a format for test reporting to the AER
9	Develop and submit Final Report

Table 1: Work Packages

This report covers the work done in Work Packages 1 and 2.

In executing Work Packages 1 and 2, Wilkie Enterprises engaged the Stakeholders to identify the existence of the following standardized testing protocols in Alberta, Canada, other Provinces of Canada and major international markets:

I. Safety and Toxicology during storage, handling and transportation
II. Leaching toxicity
III. Groundwater protection
IV. Field Pilot Protocol
V. Bonding to casing, cement and formation
VI. Effects of products on the wellbore (i.e. corrosion, limits wellbore access, etc.)
VII. Longevity of the product in wellbore conditions with evidence supporting the expected longevity
VIII. Product integrity under anticipated adverse conditions (example interaction with H2S or diesel products)
IX. Field deployment verification protocol

METHODOLOGY FOR WORK PACKAGES 1 & 2

A total of twenty-six (26) major Stakeholders in the Oil and Gas industry were identified and the contact information of their signoff authority (Focal Point) were obtained. The identified Stakeholders are broken down as follows:
Sixteen (16) Service based organizations.
Nine (9) Oil and Gas Producers.
One (1) Industry Committee: Drilling and Completion Committee (DACC) on Wellbore Remediation, An Industry Recommended Practice
One (1) Regulator: Alberta Energy Regulator.

Wilkie Enterprises contacted the Focal Point of each Stakeholder to sensitise him or her on the objectives of Project. Five (5) Stakeholders out rightly declined to participate and twenty-one (21) Stakeholders agreed to participate.
Thereafter, PTAC issued formal letters of introduction and official invitation to the twenty-one (21) Stakeholders to participate in the Project.

Following this, a detailed Stakeholder Questionnaire, constructed by Wilkie Enterprises, was emailed to the twenty-one (21) Stakeholders.
Thirteen (13) Stakeholders did not respond, either because they did not have the time or did not have the resources to deploy to answer the Questionnaire.
Only eight (8) Stakeholders as shown in Table 2: Stakeholder Participants, below, provided feedback on the Questionnaire.

Stakeholder Identifier Number	Stakeholder Name	Category	Chemical Cement Alternative Product
1	Matrix Drilling Solutions	Service based organisation	Resin (Type C)
2	National Silicates	Service based organisation	Silicate Gel (Type G)
3	SealWell	Service based organisation	Bismuth Alloy (Type I)
4	SNF	Service based organisation	Polymer Gel (Type G)
5	Shell Canada	Oil and Gas Producer	N/A
6	Conoco Phillips Canada	Oil and Gas Producer	N/A
7	Alberta Energy Regulator	Regulator	N/A
8	DACC	Industry Committee	N/A

Table 2: Stakeholder Participants

Shell Canada, Conoco Phillips and the William Butler from the Alberta Energy Regulator recommended a United Kingdom (UK) Oil and Gas document titled “Guidelines on Qualification of Materials for the Abandonment of Wells”. This document was authored by representatives from the following organizations:

1) Royal Dutch Shell
2) University of Dundee (UK)
3) Conoco Phillips UK
4) SINTEF Norway
5) TNO Netherlands (Netherlands Organisation for Applied Scientific Research)
6) Schlumberger UK
7) BP Norway
8) Halliburton UK
9) Oil and Gas UK

The document identified the following Chemical Cement Alternatives as listed in Table 3 below:

Type	Chemical Cement Alternatives	Examples
A	Modified Cements/ ceramics (non-setting)	Pozzolanic cements, slag, phosphate cements, hardening ceramics, geopolymers
B	Grouts (non-setting)	Sand or clay mixtures, bentonite pellets, barite plugs, calcium carbonate and other inert particle mixtures
C	Thermosetting polymers and composites	Resins, epoxy, polyester, vinylesters, including fibre reinforcements
D	Thermoplastic polymers and composites	Polyethylene, polypropylene, polyamide, PTFE, PEEK, PPS, PVDF and polycarbonate, including fibre reinforcements
E	Elastomeric polymers and composites	Natural rubber, neoprene, nitrile, EPDM, FKM, FFKM, silicone rubber, polyurethane, PUE and swelling rubbers, including fibre reinforcements
F	Formation	Claystone, shale, salt
G	Gels	Polymer gels, polysaccharides, starches, silicate-based gels, clay based gels, diesel/clay mixtures
H	Glass	Solid impermeable silicate glass
I	Metals	Bismuth Alloys
J	Modified in-situ materials	Chemical Cement Alternatives formed from casing and / or formation through thermal or chemical modification

Table 3: Chemical Cement Alternatives [1]

The document further listed properties in the Chemical Cement Alternatives to be tested for and their significance to the Alternatives' integrity. The properties are listed in Table 4 Properties of Chemical Cement Alternatives and Their Relevance

Property (Applicable Protocol Number)	Definition	Units	Importance of the Property to the Chemical Cement Alternative Testing
Permeability (V)	Measure of the ability of a porous material to transmit fluids under a pressure differential.	Darcy	Provides an estimate of the lag time between placement and breakthrough and release rate of fluid below a given length of material under a set pressure differential
Diffusion coefficient (V)	Proportionality constant between the gradient of concentration driving the diffusion process and the corresponding flux of the moving fluid	m^2s^{-1}	Provides an estimate of the lag time between placement and breakthrough and release rate of fluid below a given length of material under a set concentration differential
Absorption (V, VI)	Mass of fluid taken up by porosity within a substance	% mass/ % vol.	Allows an indication of swelling, from which resulting stresses may be projected
Chemical Resistance	Indication of reactivity of a material. Described with terms non resistant, limited resistance, resistant		Allows an indication of the degree to which properties of the material may change
Volume Change (V, VI)	Change in volume	strain or % by vol.	Variable required to calculate stresses from expansion or shrinkage
Modulus of elasticity (VII)	Uniaxial stress over uniaxial strain.	Pressure	Variable required to determined degree of deformation under a given pressure, and under temperature change

Poisson's ratio (VII)	Ratio of lateral strain to axial strain under uniaxial stress	None	Variable required to determine lateral deformation under a given pressure and under temperature change
Cohesion (VII)	Describes a granular material's cementation strength between grains under shear stress.	Pressure	Variable required to determine shear failure
Internal friction angle (VII)	Describes a granular material's ability to increase load-capacity or shear stress with confinement	Degrees	Variable required to determine reduction in ultimate compressive strength and loss in cohesion
Hydrostatic yield (VII)	Stress applied uniformly in all directions when plastic deformation happens	Pressure	Above this threshold material will undergo irreversible plastic deformation causing loss of cohesion and load-bearing capacity. Provides indication of pore collapse in granular materials
Tensile Strength (VII)	Threshold at which failure occurs under a tensile load	Pressure	Describes maximum tensile stress
Unconfined compressive strength (VII)	Threshold at which failure occurs under axial compressive stress	Pressure	Maximum compressive stress that a Chemical Cement Alternative can withstand
Hardness (VII)	Describes a material's resistance to surface deformation		QA/QC control test. For some materials, provides indication of yield strength in shear
Shear bond strength (V, VII)	Threshold at which bond between two materials fails under shear loading	Pressure	Variable required to calculate pressure differential value resulting in the movement of Chemical Cement Alternative
Tensile bond strength (V, VII)	Threshold at which bond between two materials fails under tensile loading	Pressure	Maximum tensile at the Chemical Cement Alternative casing interface prior to failure
Creep (VII)	Linear deformation over time at a set load	Strain rate/time %/s	Provides an estimate of the ultimate dimensional change of a Chemical Cement Alternative under a given pressure differential or other load
Fatigue life	Threshold number of stress cycles of a given property prior to failure		Provides an indication of longevity at a specified cyclical stress regime.
Decomposition temperature (VII)	Threshold temperature at which Chemical Cement Alternative begins to thermally decompose for a given pressure and environment composition	Temperature	Provides an indication of the degree of deterioration and gives a prediction of maximum operating temperature of the material
Density (II)	Mass per unit volume	Mass per unit volume	QA/QC test. Provides an indication of the likelihood of Chemical Cement Alternative moving due to differences between densities of Chemical Cement Alternative and well fluids

Table 4 Properties of Chemical Cement Alternatives and Their Relevance [1]

PRESENTATION OF DATA AND RESULTS

Review of existing protocols from participating Stakeholders are listed for Alberta, the United States and major oil and gas countries.

I. Safety and toxicology during storage, handling and transportation

Protocol Objective:

To ensure that toxic elements of the Chemical Cement Alternatives are safely and properly handled during transportation and storage.

Existing Protocol as used by SealWell	Protocol as recommended by DACC
- Follow Material Data Sheet directions on storage and transportation - Ensure adequate ventilation during product preparation	- Check the Material Data Sheet and identify the safety and toxicology protocol for the Chemical Cement Alternative. - When not available, follow the safety and toxicology protocol of the individual chemicals prior to and post-setting, by visiting list in section 26.8.1.3 of "An Industry Recommended Practice (IRP) for the Canadian Oil and Gas Industry, IRP 26 Wellbore Remediation" [2].

Table 5: Safety and Toxicology Protocols for Chemical Cement Alternatives

II. Leaching toxicity

Protocol Objective:

Chemical Cement Alternatives should not contaminate subsurface formations with harmful substances during deployment (curing) or through deterioration.

Existing Protocol as used by Matrix Drilling Solutions	Protocol as recommended by Shell, Conoco Phillips & Alberta Energy Regulator
- See leachate test method Revision 1b under "Methods of Testing" of Appendix below.	- Measurement of Density During Ageing Testing in "Guidelines on Qualification of Materials for the Abandonment of Wells" [1] - Measurement of Change in Dry Mass After Ageing Testing in "Guidelines on Qualification of Materials for the Abandonment of Wells" [1]

Table 6: Leaching Toxicity Protocols for Chemical Cement Alternatives

III. Groundwater Protection

<u>Protocol Objective:</u>

Chemical Cement Alternatives should not contaminate groundwater with harmful substances during deployment (curing) or through deterioration.

Existing Protocol as used by Matrix Drilling Solutions	Protocol as recommended by National Silicates
- See leachate test method – Revision 1b under "Methods of Testing" of Appendix below. -See Element Materials Technology "Water Analysis Testing" [3] for a list of constituents requiring measurement after Chemical Cement Alternative contacts groundwater in a lab setting (prior to field pilot tests)	- See PSAC and CAPP database of toxicity test results for mud additives for a list of constituents requiring measurement after Chemical Cement Alternative contacts groundwater in a lab setting (prior to field pilot tests)

Table 7: Groundwater Protection Protocols for Chemical Cement Alternatives

IV. Field Pilot Protocol

<u>Protocol Objective:</u>

To ensure that the Chemical Cement Alternative meets its design specifications during field tests.

Protocol as recommended by Shell, Conoco Phillips & Alberta Energy Regulator	Protocol as recommended by DACC
- Establish anticipated downhole conditions based on current knowledge - Function test as identified in Appendix 8 performed by Shell Global Solutions in "Guidelines of the Qualification of Materials Used in the Abandonment of Wells" [1]. See also lines 291-299 of same document. - Model placement techniques - Confirm effectiveness of placement techniques with previous yard trials - Determine the extent of material contamination during placement - Ensure that the material has appropriate properties that allow it to displace the existing fluids and form a continuous sealing medium even when contaminated - Conduct in situ testing and verification of the Chemical Cement Alternative - Model the effect of varying loads and external influences - Conduct long term monitoring - Utilize tools like acoustic transducers to determine extent of deterioration	- Document product operating envelope to prevent degradation as a result of future operations in the reservoir. - Consider retaining pre-mixed and blended post-set sample for future evaluation - Develop success/failure criteria for tests after placement - Consider collaborating with other organizations to share costs

- Ensure Chemical Cement Alternative can be removed in the event that well re-entry is required - Minimize setting time (when practical) and/or maintain sufficient surface pressure when transitioning from liquid into solid to prevent escape of fluid and loss of the integrity of the Chemical Cement Alternative	

Table 8: Field Pilot Protocols for Chemical Cement Alternatives

V. Bonding to casing, cement and formation

VI. Effects of products on the wellbore (i.e. corrosion, limits wellbore access, etc.)

VII. Longevity of the product in wellbore conditions with evidence supporting the expected longevity

Objectives for the three (3) Protocols above:
To ensure the integrity (and performance) of the Chemical Cement Alternatives will last over the entire abandonment life of the well.

Subject (Applicable Protocol Number)	Property	Protocol as recommended by Shell, Conoco Phillips & Alberta Energy Regulator
PERMEATION TESTING		
V	Nitrogen Permeability	Section 8.2.1 of "Guidelines on Qualification of Materials for the Abandonment of Wells" [1]
V	Diffusion coefficient	Not required
INTERACTION WITH FLUID		
II, VIII	Dry Mass	Measurement of mass after drying to constant mass at 105^0C
V, VI	Absorption	Not required
DIMENSIONAL STABILITY		
Expansion/Swelling		
V, VI	During hardening	API RP 10B-5 ring test
V, VI	Hardened	API RP 10B-5 ring test
Shrinkage		
V, VI	During hardening	API RP 10B-5 ring test
V, VI	Hardened	API RP 10B-5 ring test
V, VI	Differential thermal expansion	ASTM E228
V, VI, VII	creep	ASTM C512-10
MECHANICAL TESTING		
VII	Triaxial testing	Not required
VII	Cohesion	Not required
VII	Poisson's ratio	Not required
VII	Internal friction angle	Not required
VII	Hydrostatic compressive yield	Not required
VII	UCS	API RP 10B-2
VII	Tensile strength	ASTM C496
VII	Elastic modulus	ASTM C469
VII	Hardness	ASTM E384
OTHER		
V, VII	Shear bond strength	See Section 8.6 of "Guidelines on Qualification of Materials for the Abandonment of Wells" [1]
V, VII	Tensile bond strength	Not required
VII	Decomposition temperature	Not required
II	Density	ASTM C 138
V, VI, VII	Stress relaxation	Not required

Table 9: Testing Protocols for Modified Cements/ ceramics (non-setting)

Subject (Applicable Protocol Number)	Property	Protocol as recommended by Shell, Conoco Phillips & Alberta Energy Regulator
PERMEATION TESTING		
V	Nitrogen Permeability	See Section 8.2.1 of “Guidelines on Qualification of Materials for the Abandonment of Wells” [1]
V	Diffusion coefficient	Not required
INTERACTION WITH FLUID		
II, VIII	Dry Mass	Measurement of mass after drying to constant mass at 105^0C
V, VI	Absorption	Not required
DIMENSIONAL STABILITY		
Expansion/Swelling		
V, VI	During hardening	Not required
V, VI	Hardened	Not required
Shrinkage		
V, VI	During hardening	Not required
V, VI	Hardened	Non – identified
V, VI	Differential thermal expansion	ASTM E228
V, VI, VII	creep	Not required
MECHANICAL TESTING		
VII	Triaxial testing	Not required
VII	Cohesion	Not required
VII	Poisson’s ratio	Not required
VII	Internal friction angle	Not required
VII	Hydrostatic compressive yield	Not required
VII	UCS	Not required
VII	Tensile strength	Not required
VII	Elastic modulus	Not required
VII	Hardness	Not required
OTHER		
V, VII	Shear bond strength	See Section 8.6 of “Guidelines on Qualification of Materials for the Abandonment of Wells” [1], recommended that substrate rugosity measurements done as per ASTM D7172
V, VII	Tensile bond strength	Not required
VII	Decomposition temperature	Not required
II	Density	Pressurized mud balance
V, VI, VII	Stress relaxation	Not required

Table 10: Testing Protocols for Grouts (non-setting)

Subject (Applicable Protocol Number)	Property	Protocol as recommended by Shell, Conoco Phillips & Alberta Energy Regulator	Existing Protocol as used by Matrix Drilling Solutions
PERMEATION TESTING			
V	Nitrogen Permeability	Not required	See "Cement Plug Testing: Weight vs. Pressure Testing to Assess Viability of a Wellbore Seal Between Zones" [4] (VA, USA) for perm testing
V	Diffusion coefficient	See section 8.2.2 of "Guidelines on Qualification of Materials for the Abandonment of Wells" [1]	
INTERACTION WITH FLUID			
II, VIII	Dry Mass	Measurement of mass after drying to constant mass at 105^{0}C	
V, VI	Absorption	Not required	
DIMENSIONAL STABILITY			
Expansion/Swelling			
V, VI	During hardening	See section 8.4.1 of "Guidelines on Qualification of Materials for the Abandonment of Wells" [1]	
V, VI	Hardened	See section 8.4.1 of [1]	
Shrinkage			
V, VI	During hardening	See section 8.4.2 of "Guidelines on Qualification of Materials for the Abandonment of Wells" [1]	
V, VI	Hardened	See section 8.4.2 of [1]	
V, VI	Differential thermal expansion	ASTM E228	
V, VI, VII	creep	ISO 899-1	
MECHANICAL TESTING			
VII	Triaxial testing	Not required	API 10 A (AB, CA)
VII	Cohesion	Not required	
VII	Poisson's ratio	Not required	See "Cement Plug Testing: Weight vs. Pressure Testing to Assess Viability of a Wellbore Seal Between Zones" [4] (VA, USA)
VII	Internal friction angle	Not required	
VII	Hydrostatic compressive yield	Not required	
VII	UCS	API RP 10B-2	API 10 A "COSS 2018-18" [5] (AB, CA), ASTM D695 "Tensile, Flexural, and Compression Testing of Two-Part Polymer Resin" [6](GA, USA), See "The Effect of Waterborne Epoxy Resin Emulsion on the Physical Properties of Oil Well Cement" [7](China)
VII	Tensile strength	ISO 527-1	ASTM D638 "Tensile, Flexural, and

			Compression Testing of Two-Part Polymer Resin" [6] (GA, USA),
VII	Elastic modulus	ISO 527-1	API 10 A "COSS 2018-18" [5] (AB, CA), ASTM D638 "Tensile, Flexural, and Compression Testing of Two-Part Polymer Resin" [6] (GA, USA), See "Cement Plug Testing: Weight vs. Pressure Testing to Assess Viability of a Wellbore Seal Between Zones" [4] (VA, USA)
VII	Hardness	See section 8.4.2 of [1]	
OTHER			
VII	Flexural strength		ASTM D790 & D670, flexural strength "Tensile, Flexural, and Compression Testing of Two-Part Polymer Resin" [6] (GA, USA)
V, VII	Shear bond strength	See Section 8.6 of "Guidelines on Qualification of Materials for the Abandonment of Wells" [1], recommended that substrate rugosity measurements done as per ASTM D7172	"Cement Plug Testing: Weight vs. Pressure Testing to Assess Viability of a Wellbore Seal Between Zones" [4] (VA, USA), See "The Effect of Waterborne Epoxy Resin Emulsion on the Physical Properties of Oil Well Cement" [7](China) (China)
V, VII	Tensile bond strength	Not required	
VII	Decomposition temperature	TGA/DTA/DSC measurement	
II	Density	ISO 1183-1	
V, VI, VII	Stress relaxation	Not required	

Table 11: Testing Protocols for Thermosetting polymers and composites

Subject (Applicable Protocol Number)	Property	Protocol as recommended by Shell, Conoco Phillips & Alberta Energy Regulator
PERMEATION TESTING		
V	Nitrogen Permeability	Not required
V	Diffusion coefficient	See section 8.2.2 of "Guidelines on Qualification of Materials for the Abandonment of Wells" [1]
INTERACTION WITH FLUID		
II, VIII	Dry Mass	Measurement of mass after drying to constant mass at 105^{0}C
V, VI	Absorption	Not required
DIMENSIONAL STABILITY		
Expansion/Swelling		
V, VI	During hardening	See section 8.4.1 of "Guidelines on Qualification of Materials for the Abandonment of Wells" [1]
V, VI	Hardened	See section 8.4.1 of [1]
Shrinkage		

V, VI	During hardening	See section 8.4.2 of "Guidelines on Qualification of Materials for the Abandonment of Wells" [1], may need to investigate thermal shock
V, VI	Hardened	See section 8.4.2 of [1]
V, VI	Differential thermal expansion	ASTM E228
V, VI, VII	creep	ISO 899-1
MECHANICAL TESTING		
VII	Triaxial testing	Not required
VII	Cohesion	Not required
VII	Poisson's ratio	Not required
VII	Internal friction angle	Not required
VII	Hydrostatic compressive yield	Not required
VII	UCS	ISO 604
VII	Tensile strength	ISO 527-1
VII	Elastic modulus	ISO 527-1
VII	Hardness	ISO 868
OTHER		
V, VII	Shear bond strength	See Section 8.6 of "Guidelines on Qualification of Materials for the Abandonment of Wells" [1], recommended that substrate rugosity measurements done as per ASTM D7172
V, VII	Tensile bond strength	Not required
VII	Decomposition temperature	TGA/DTA/DSC measurement
II	Density	ISO 1183-1
V, VI, VII	Stress relaxation	Not required

Table 12: Testing Protocols for Thermoplastic polymers and composites

Subject (Applicable Protocol Number)	Property	Protocol as recommended by Shell, Conoco Phillips & Alberta Energy Regulator
PERMEATION TESTING		
V	Nitrogen Permeability	Not required
V	Diffusion coefficient	See section 8.2.2 of "Guidelines on Qualification of Materials for the Abandonment of Wells" [1]
INTERACTION WITH FLUID		
II, VIII	Dry Mass	Measurement of mass after drying to constant mass at 105^{0}C
V, VI	Absorption	Not required
DIMENSIONAL STABILITY		
Expansion/Swelling		
V, VI	During hardening	See section 8.4.1 of "Guidelines on Qualification of Materials for the Abandonment of Wells" [1]
V, VI	Hardened	See section 8.4.1 of [1]
Shrinkage		
V, VI	During hardening	See section 8.4.2 of "Guidelines on Qualification of Materials for the Abandonment of Wells" [1]
V, VI	Hardened	See section 8.4.2 of [1]
V, VI	Differential thermal expansion	ASTM E228
V, VI, VII	creep	ISO 899-1 / ASTM D395
MECHANICAL TESTING		
VII	Triaxial testing	Not required
VII	Cohesion	Not required
VII	Poisson's ratio	ISRM suggested method

VII	Internal friction angle	Not required
VII	Hydrostatic compressive yield	Not required
VII	UCS	BS EN ISO 604
VII	Tensile strength	BS EN ISO 527-1
VII	Elastic modulus	BS EN ISO 527-1
VII	Hardness	ISO 868
OTHER		
V, VII	Shear bond strength	See Section 8.6 of "Guidelines on Qualification of Materials for the Abandonment of Wells" [1], recommended that substrate rugosity measurements done as per ASTM D7172
V, VII	Tensile bond strength	Not required
VII	Decomposition temperature	TGA/DTA/DSC measurement
II	Density	ISO 1183-1
V, VI, VII	Stress relaxation	ASTM D395 and NORSOK M710

Table 13: Testing Protocols for Elastomeric polymers and composites

Subject (Applicable Protocol Number)	Property	Protocol as recommended by Shell, Conoco Phillips & Alberta Energy Regulator
PERMEATION TESTING		
V	Nitrogen Permeability	See Section 8.2.1 of "Guidelines on Qualification of Materials for the Abandonment of Wells" [1]
V	Diffusion coefficient	Not required
INTERACTION WITH FLUID		
II, VIII	Dry Mass	Measurement of mass after drying to constant mass at 105^0C
V, VI	Absorption	Not required
DIMENSIONAL STABILITY		
Expansion/Swelling		
V, VI	During hardening	Not required
V, VI	Hardened	ISRM suggested method
Shrinkage		
V, VI	During hardening	Not required
V, VI	Hardened	ISRM suggested method
V, VI	Differential thermal expansion	ASTM E228
V, VI, VII	creep	ASTM C512-10
MECHANICAL TESTING		
VII	Triaxial testing	ISRM suggested method
VII	Cohesion	ISRM suggested method
VII	Poisson's ratio	ISRM suggested method
VII	Internal friction angle	ISRM suggested method
VII	Hydrostatic compressive yield	ISRM suggested method
VII	UCS	ISRM suggested method
VII	Tensile strength	ASTM C496
VII	Elastic modulus	ASTM C469
VII	Hardness	Not required
OTHER		
V, VII	Shear bond strength	Not required
V, VII	Tensile bond strength	Not required
VII	Decomposition temperature	Not required
II	Density	Not required
V, VI, VII	Stress relaxation	Not required

Table 14: Testing Protocols for Formation

Subject (Applicable Protocol Number)	Property	Protocol as recommended by Shell, Conoco Phillips & Alberta Energy Regulator	Protocol as recommended by National Silicates
PERMEATION TESTING			
V	Nitrogen Permeability	See Section 8.2.1 of "Guidelines on Qualification of Materials for the Abandonment of Wells" [1]	
V	Diffusion coefficient	See Section 8.2.1 of "Guidelines on Qualification of Materials for the Abandonment of Wells" [1]	
INTERACTION WITH FLUID			
II, VIII	Dry Mass	Measurement of mass after drying to constant mass at 105^0C	
V, VI	Absorption	Absorption index	
DIMENSIONAL STABILITY			
Expansion/Swelling			
V, VI	During setting	Not required	
V, VI	Set	Not required	
Shrinkage			
V, VI	During setting	See Section 8.4.2 of "Guidelines on Qualification of Materials for the Abandonment of Wells"	
V, VI	Set	See Section 8.4.2 of "Guidelines on Qualification of Materials for the Abandonment of Wells"	
V, VI	Differential thermal expansion	ASTM E228	
V, VI, VII	creep	Not required	
MECHANICAL TESTING			
VII	Triaxial testing	Not required	
VII	Cohesion	Not required	
VII	Poisson's ratio	Not required	
VII	Internal friction angle	Not required	
VII	Hydrostatic compressive yield	Not required	
VII	UCS	Not required	
VII	Tensile strength	Not required	
VII	Elastic modulus	Not required	
VII	Hardness	Not required	
OTHER			
VI	Corrosion		API Recommended Practice 13B-1.
V, VII	Shear bond strength	See Section 8.6 of "Guidelines on Qualification of Materials for the Abandonment of Wells" [1], recommended that substrate rugosity measurements done as per ASTM D7172	See "Improving Cement Bond in the Appalachian Basin With Adjustments to Preflush and Spacer Design" [8] (OH, USA), See ""The Cement-to-Formation Interface in Zonal Isolation" [9] (KL, Malaysia)
V, VII	Tensile bond strength	Not required	
VII	Decomposition temperature	TGA / DTA / DSC	See ""High-Temperature Plug Formation With

			Silicates" [10] (TX, USA) & "Advanced Cements for Geothermal Wells" [11]
II	Density	Not required	
V, VI, VII	Stress relaxation	Not required	

Table 15: Testing Protocols for Gels

Subject (Applicable Protocol Number)	Property	Protocol as recommended by Shell, Conoco Phillips & Alberta Energy Regulator
PERMEATION TESTING		
V	Nitrogen Permeability	Not required
V	Diffusion coefficient	Not required
INTERACTION WITH FLUID		
II, VIII	Dry Mass	Measurement of mass after drying to constant mass at 105^{0}C
V, VI	Absorption	Not required
DIMENSIONAL STABILITY		
Expansion/Swelling		
V, VI	During hardening	See Section 8.4.1 of "Guidelines on Qualification of Materials for the Abandonment of Wells" [1]
V, VI	Hardened	See Section 8.4.1 of "Guidelines on Qualification of Materials for the Abandonment of Wells" [1]
Shrinkage		
V, VI	During hardening	See Section 8.4.2 of "Guidelines on Qualification of Materials for the Abandonment of Wells" [1]
V, VI	Hardened	See Section 8.4.2 of "Guidelines on Qualification of Materials for the Abandonment of Wells" [1]
V, VI	Differential thermal expansion	ASTM E228, may need to investigate thermal shock
V, VI, VII	creep	Not required
MECHANICAL TESTING		
VII	Triaxial testing	Not required
VII	Cohesion	Not required
VII	Poisson's ratio	Not required
VII	Internal friction angle	Not required
VII	Hydrostatic compressive yield	Not required
VII	UCS	API RP 10B-2
VII	Tensile strength	Not required
VII	Elastic modulus	ASTM C469
VII	Hardness	ASTM E384
OTHER		
V, VII	Shear bond strength	See Section 9.6 of "Guidelines on Qualification of Materials for the Abandonment of Wells" [1]
V, VII	Tensile bond strength	Not required
VII	Decomposition temperature	Not required
II	Density	ASTM C138
V, VI, VII	Stress relaxation	Not required

Table 16: Testing Protocols for Glass

Subject (Applicable Protocol Number)	Property	Protocol as recommended by Shell, Conoco Phillips & Alberta Energy Regulator	Existing Protocol as used by SealWell
PERMEATION TESTING			
V	Nitrogen Permeability	Not required	
V	Diffusion coefficient	Not required	
INTERACTION WITH FLUID			
II, VIII	Dry Mass	Measurement of mass after drying to constant mass at 105^{0}C	
V, VI	Absorption	Not required	
DIMENSIONAL STABILITY			
Expansion/Swelling			
V, VI	During hardening	See Section 8.4.1 of "Guidelines on Qualification of Materials for the Abandonment of Wells" [1]	
V, VI	Hardened	See Section 8.4.1 of [1]	
Shrinkage			
V, VI	During hardening	See Section 8.4.2 of "Guidelines on Qualification of Materials for the Abandonment of Wells" [1], may need to investigate thermal shock	
V, VI	Hardened	See Section 8.4.2 of [1]	
V, VI	Differential thermal expansion	ASTM E228	See Section 4 of "Remediating sustained casing pressure by forming a downhole annular seal with low melt point eutectic metal" [12] (AB, CA)
V, VI, VII	creep	ISO 204	
MECHANICAL TESTING			
VII	Triaxial testing	ISRM suggested method	
VII	Cohesion	Not required	
VII	Poisson's ratio	ISRM suggested method (triaxial) or ASTM E1876	
VII	Internal friction angle	Not required	
VII	Hydrostatic compressive yield	ISRM suggested method	
VII	UCS	ASTM E9	
VII	Tensile strength	ISO 6892-1	
VII	Elastic modulus	ISO 3312 or ASTM E9	
VII	Hardness	ASTM E18, ASTM E10 or ASTM E384	
OTHER			
Corrosion		ISO 1516/NACE MR0175	See "CCEMC Project C110113: Permanent Sealing of GHG Emitting Wells" [13] (AB, CA)
V, VII	Shear bond strength	See Section 8.6 of "Guidelines on Qualification of Materials for the Abandonment of Wells" [1], recommended that substrate rugosity measurements done as per ASTM D7172	See "Remediating sustained casing pressure by forming a downhole annular seal with low melt point eutectic metal" procedure conducted in

			Halliburton lab in Duncan [12] (OK, USA)
V, VII	Tensile bond strength	Not required	
VII	Decomposition temperature	TGA/DTA/DSC measurement	
II	Density	ISO 3369	
V, VI, VII	Stress relaxation	Not required	

Table 17: Testing Protocols for Metals

Subject (Applicable Protocol Number)	Property	Protocol as recommended by Shell, Conoco Phillips & Alberta Energy Regulator
PERMEATION TESTING		
V	Nitrogen Permeability	See Section 8.2.1 of "Guidelines on Qualification of Materials for the Abandonment of Wells" [1]
V	Diffusion coefficient	Not required
INTERACTION WITH FLUID		
II, VIII	Dry Mass	Measurement of mass after drying to constant mass at 105^{0}C
V, VI	Absorption	Not required
DIMENSIONAL STABILITY		
Expansion/Swelling		
V, VI	During hardening	Not required
V, VI	Hardened	ISRM suggested method
Shrinkage		
V, VI	During hardening	Not required
V, VI	Hardened	ISRM suggested method
V, VI	Differential thermal expansion	ASTM E228
V, VI, VII	creep	ASTM C512-10
MECHANICAL TESTING		
VII	Triaxial testing	ISRM suggested method
VII	Cohesion	ISRM suggested method
VII	Poisson's ratio	ISRM suggested method
VII	Internal friction angle	ISRM suggested method
VII	Hydrostatic compressive yield	ISRM suggested method
VII	UCS	ISRM suggested method
VII	Tensile strength	ASTM C496
VII	Elastic modulus	ASTM C469
VII	Hardness	Not required
OTHER		
V, VII	Shear bond strength	Not required
V, VII	Tensile bond strength	Not required
VII	Decomposition temperature	Not required
II	Density	Not required
V, VI, VII	Stress relaxation	Not required

Table 18: Testing Protocols for Modified in-situ materials

VIII. Product integrity under anticipated adverse conditions (example interaction with H2S or diesel products)

Protocol Objective:

To ensure lifetime integrity (and performance) of the Chemical Cement Alternatives in the worst downhole conditions

Stakeholder	Recommended Protocol
National Silicates	- See procedure described in Field Test Results of a New Silicate Gel System that is Effective in Carbon Dioxide Enhanced Recovery and Waterfloods” [14]
SealWell	- See comprehensive corrosion testing conducted by the University of Calgary in Appendix A of “CCEMC Project C110113: Permanent Sealing of GHG Emitting Wells” [13]
SNF	- See procedure described in “More Than 12 Years' Experience With a Successful Conformance-Control Polymer-Gel Technology” [15] and ““Acrylamide-Polymer/Chromium (III)-Carboxylate Gels for Near Wellbore Matrix Treatments” [16]
Shell, ConocoPhillips, AER	See Section 8.10 of “Guidelines on Qualification of Materials for the Abandonment of Wells” [1]

Table 19: Protocols for Chemical Cement Alternatives Under Adverse Conditions

IX. Field deployment verification

Protocol Objective:

To ensure that the Chemical Cement Alternative meets its design specifications during field deployment.

Stakeholder	Recommended Protocol
SealWell	- Conduct unsteady state heat transfer modelling with multiphysics program, such as COMSOL, and consult with firms such as AltaSim Technologies to execute program ““CCEMC Project C110113: Permanent Sealing of GHG Emitting Wells” [13] - See Section 6 of “CCEMC Project C110113: Permanent Sealing of GHG Emitting Wells” [13]
Shell, ConocoPhillips, AER	- Determine Displacement efficiency with relevant wellbore fluids - Evaluate and prevent slumping of plugging materials components in relevant wellbore fluids. Component size can be critical meaning tests may be required at different scales. Computer simulations may assist in understanding the behavior as long as the output is verified.

	- Minimize setting time (when practical) and/or maintain sufficient surface pressure when transitioning from liquid into solid to prevent escape of fluid and loss of the integrity of the Chemical Cement Alternative - Verify that the Chemical Cement Alternative has been successfully placed - Ensure Chemical Cement Alternative can be removed in the event that well re-entry is required - Account for thermal expansion differences between casing and material during setting reactions. Cracking and de-bonding may result post set if this is significant. For thermal expansion testing see ASTM E228 coefficient of thermal expansion.
DACC	- Verify that the product blend is not altered from what was tested or accepted for use - Review life expectancy of product against accepted wellbore materials such as casing cement, packers, cast iron, fiberglass, etc. - Document product operating envelope to prevent degradation as a result of future operations in the reservoir - Consider retaining pre-mixed and blended post-set sample for future evaluation - Develop success/failure criteria for testing after product placement - Consider collaborating with other organizations to share costs

Table 20: Protocols for Field Deployment Verification of Chemical Cement Alternatives

NEXT MILESTONE DELIVERABLES

As per the contract, the next Milestone is to complete Work Package-3 by February 28th, 2019. The objective of this Work Package is to review all identified protocols and recommend (and justify) standardised testing for each protocol based on:

a) Industry best practice,
b) Test duration for each protocol,
c) Estimated cost for each protocol.

SCHEDULE "A" MILESTONE AND INVOICING SCHEDULE

Payment Milestones	Work Package	Proposed Completion Date	Actual Completion Date <u>approved</u> by Technical Champion			Anticipated Completion Date				
1. Meetings with Stakeholders and development of categories and protocols required per category.	1	Nov 30, 2018			Jan 31, 2019					
2. Develop list of current Chemical Cement Alternative protocols per category in Alberta	1	Dec 31, 2018			Jan 31, 2019					
3. Develop list of current Chemical Cement Alternative protocols per category outside Alberta	2	Jan 31, 2019			Jan 31, 2019					
4. Propose best practice protocols	3	Feb 28, 2019				Feb 28, 2019				
5. Propose a standard value per property (where applicable)	4	Mar 31, 2019					Mar 31, 2019			
6. Develop criteria for approving of laboratories for Chemical Cement Alternative testing	5	Apr 30, 2019						Apr 30, 2019		
7. Develop list of "Approved Laboratories" for testing protocols	6 & 7	May 31, 2019							May 31, 2019	
8. Provide format for test reporting to the AER	8	May 31, 2019							May 31, 2019	

9. Final Report	9	Jun 30, 2019								Jun 30, 2019

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[16] R. D. Sydansk, "Acrylamide-Polymer/Chromium (III)-Carboxylate Gels for Near Wellbore Matrix Treatments," Society of Petroleum Engineers, 1993.

[17] M.McDonald et al., "Use of Silicate-Based Fluids to Mitigate Corrosion," SPE, Woodlands, TX, 2007.

APPENDIX

II. Leaching toxicity

INTER-OFFICE CORRESPONDENCE

TO: Catherine Evans, Alberta Energy Regulator
Gerry Boyer, Alberta Energy Regulator

FROM: Brent Warren, Matrix Specialty Chemicals

DATE: February 6, 2015

SUBJECT: Method for CORE TEST - Leachate Testing – Revision 1B

Following is a proposed method for doing a leachate test for a system that represents water above the base of the groundwater. The test method is based upon a document entitled "Current State of the JSCE Standard on Test Method for Leaching Trace Elements from Hardened Concrete" by T Sugiyama et al. Internet reference for this document is www.claisse.info/special%20papers/sugiyama_full_text.pdf.

While the cited document refers to the analysis of inorganic ions, the proposed analysis in this instance is an organic phenol compound. The test method used is based upon the Sugiyama paper as this is done with a similar material (hardened concrete) in a freshwater environment. The test method is described as a "Tank Leaching Test".

Proposed Test Method:

1. Sample Preparation:

- Samples will be prepared in a mold, as per the usual method of preparing samples. The sample will be typical of the formulation used in a remedial operation above the base of the groundwater. It is expected that the formulation will be made in one batch and poured into the appropriate number of molds required for the test.
- After fully hardening, the samples will be removed from the mold, exact dimensions of the samples will be measured (typically close to a cube in size with each side being ~ 50 mm in length). The weight of the samples will also be taken
- The samples shall be kept at the same temperature as the Leachant water, typically room temperature of 20±2 °C.

2. Leachant Preparation

- Water for the tests will be tap water.
- The amount of water used in each test intervention is based upon the dimensions of the sample that was prepared in Step 1 above. For each 100 mm^2 of surface area, 5 mL of water are to be used.

  As an example, a perfect 50 mm per side cube would require 750 mL of water per test intervention. (5mL *50 mm*50 mm*6 sides/100 mm^2).

- Temperature of the water will be room temperature, with the temperature recorded in ^oC.

3. Test Method

The test is based upon two different pH's and performing each test in triplicate. Therefore, a total of six (6) cubes will be required for the testing. One pH test condition will be 6.5 and the other pH test is to be run at 8.5. For any one individual test, analytical samples will be drawn after 24 hours and 72 hours of contact time with water sample.

Following is an example for the pH 6.5 sample

- Place leachant water in a glass vessel (with a lid so that evaporation will not occur during the test) sufficient to hold the required amount of water. Adjust the pH to 6.5
- Place test specimen in pH adjusted leachant water, such that the bottom of the sample has minimal contact with the bottom of the test chamber. For example, the corners of the test cube may be resting on small metallic plugs.
- The tank chamber is closed to prevent evaporation and the sample is allowed to sit for a 24 hour period.
- After 24 hours, the leachant is totally removed from the tank, the sample container and test specimen rinsed quickly with the pH 6.5 water, and the specimen repositioned in the test holder. New tap water leachant is added for a period of 72 hours, the leachant is totally removed from the tank and the test is completed.
- For each leachant that is removed from the tank, that leachant is filtered through a glass fiber filter or membrane filter of 1 micrometer (same as 1 micron).
- The leachant water is measured for the organic phenol concentration as specified.

Upon completion of the testing, there will be 12 samples collected which will have been exposed to the tap water

4. Summary of Samples to be Tested

Six sample cubes will be made and measured (dimensions and weight)
Testing of the sample cubes as per the following:

(1) Blank of water used in the test, tested for phenol content

(2) pH of 6.5, water tested for phenol content after 24 hours exposure and 72 hours exposure
(3) pH of 6.5, water tested for phenol content after 24 hours exposure and 72 hours exposure
(4) pH of 6.5, water tested for phenol content after 72 hours exposure

(5) pH of 8.5, water tested for phenol content after 24 hours exposure and 72 hours exposure
(6) pH of 8.5, water tested for phenol content after 24 hours exposure and 72 hours exposure
(7) pH of 8.5, water tested for phenol content after 72 hours exposure

Organic analysis:

- 4 leachant samples will be measured for phenol content after 24 hours exposure
- 6 leachant samples will be measured after 72 hours exposure.
- 1 blank sample (water used for test)

A total of 11 analyses will be done.

Therefore, there will be two samples to be kept in storage until after the test is completed. One sample will be 24 hour exposure at pH 6.5, the other will be 24 hour exposure at pH 8.5.