SS-25A Fluid Sampling and Analyses Protocol

Prepared For:

SS-25 RCA:

CPUC, DOGGR, SoCalGas

Purpose:
To document the fluid sampling and analyses protocol **for the SS-25A well** as part of the Phase 3 Aliso Canyon SS-25 well RCA.

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Version Record

Version No.	Issue Date	Issued As / Nature of Revision	Author	Checked By	Project
0	28-Apr-17	Draft for Review	RHH/BWAS	RLR, WSW	RMK
1	5-May-17	Final	RHH/BWAS	RLR	RMK
2	9-May-17	Final	RLR	RMK	RMK

Revision History

Revision	Date	Description of Change
1	5-May-17	Changed the number of samples collected from 3 to 4. Corrected typos. Added comments from SCG to Section 1.
2	9-May-17	Updated Section 1 to clarify Blade's authority.

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1 Foreword

This document is intended to address and clarify wellbore fluid and scale sampling procedures, and the subsequent chemical analysis protocol in support of the SS-25 AC-RCA.

Specifically addressed are the pertinent EPA guidelines and protocols as they relate to the RCA objectives.

The work described herein will be executed by Texas Oil Tech Laboratories[i] (TOL), under Blade supervision, in part on location and in part in the TOL laboratories in Houston, TX.

These protocols and procedures are subject to change as new information is provided to the Blade Energy Partners Root Cause Analysis (RCA) team from the field.

Blade has provisional authority as granted by the CPUC to conduct a Root Cause Analysis (RCA) on well SS-25. This authority includes overseeing RCA related work on the SS-25A well. During that work, the Blade Team and those parties under Blade's direction are responsible for directing the work of contractors retained to perform the extraction of Well SS-25A wellhead and tubing and the preservation and protection of associated evidence. The person in charge (PIC) of the extraction activities and the protection of evidence on-site is the Blade Team Lead, Ravi Krishnamurthy.

All well and wellbore equipment, including fluids, shall be considered potential evidence. Therefore, every effort shall be taken to improve the chance for recovery of fluids and to avoid inadvertent damage to equipment and/or potential evidence. Every attempt will be made to mitigate any potential damage as a result of fluid collection by careful attention to tool selection, operational procedures and process. This implies careful service equipment selection and adhering to procedures that emphasize care over speed when removing fluid.

The procedure may be further modified based on discussions with Texas Oil Tech Laboratories (TOL) (Houston, TX), a qualified water testing vendor.

[i] ISO certification: 9001-2008; 17025-2005.

2 Phase 3 – Chemical Analysis Protocol

2.1 Objective and Rational

The objective is to remove the tubing string from well SS-25A for the purpose of exposing the production casing. The production casing will be inspected and evaluated as part of the RCA.

The nature of the fluid found in the tubing and tubing/casing annulus may establish whether certain chemical vectors contributed to any observable-identifiable damage process.

The purpose of this document is to provide a protocol for the collection and documentation of fresh drilling and annular fluid samples collected at the **SS-25A** well located in the Aliso Canyon gas storage field operated by SoCal Gas. The instructions provided in this document are considered a minimal set of requirements. These protocols and procedures are subject to change as information is provided to Blade Energy Partners Root Cause Analysis (RCA) team from the field.

The procedure may be further modified based on discussions with Texas OilTech Laboratories (TOL) (Houston, TX), a qualified water testing vendor.

2.2 Background

The nature of the drilling and annular fluids will reveal important information with respect to its corrosivity, environmental safety, and microbiological content (if any). The major diagnostic criteria are pH, bicarbonate concentration (alkalinity), chloride, bacterial enumeration, and/or acid (e.g., CO_2 and H_2S) gases. Additionally, the mineral composition ought to contain clues as to the origin of the water/brine.

Blade intends to collect fluid samples of the **tubing/annulus** fluid circulated from the well. The actual sample locations will be documented as part of the evidence sample documentation.

Table 1 shows an example of the sample depths versus volume pumped. The number of pump strokes will be updated based on the pump liner size. It is important to note that in addition to a sample of the virgin workover fluid, a sample from the first effluent of the casing will be obtained. Subsequently, samples will be obtained every 50 bbl as measured by the pump strokes.

Finally, after all the original fluid in the annulus/tubing has been displaced, a final sample is obtained for verification.

A total of 11 samples will have been obtained at the end of the process as follows:

1 virgin (fresh) work-over fluid sample

1 sample of first returns from the casing

8 fluid samples at 50 bbl intervals

1 overflow sample

Table 1. Example of volume pumped versus sample depth. This example assumes the both entire tubing and annulus columns are full of fluid to surface.

Vol Pumped (bbls)		Interface	EOT	Sample	Sample	Note	Cumulative	Sample
Increment	Total	Depth	Location	From	Depth		Strokes	Number
---	---	---	---	*mud pits*	---	*displacement fluid*	0	1
0	0	0	7880 ft	Ann	0 ft	Start (Surf Sample)	10	2
50	50	2285 ft	6234 ft	Ann	1632 ft		360	3
50	100	4540 ft	4572 ft	Ann	3294 ft		719	4
50	150	6794 ft	2911 ft	Ann	4955 ft		1079	5
23.7	173.7	7880 ft	2122 ft	-	-	*Tubing displaced*	1250	
26.3	200	7022 ft	1250 ft	Ann	6616 ft		1439	6
38.5	238.5	5744 ft	0 ft	-	-	*EOT fluid at surface*	1716	
11.5	250	5361 ft	-	Tbg	7345 ft		1799	7
50	300	3700 ft	-	Tbg	5090 ft		2158	8
50	350	2038 ft	-	Tbg	2836 ft		2518	9
50	400	377 ft	-	Tbg	581 ft		2878	10
12.2	412.2	0 ft	-	-	-	*Full displacement*	2966	
5.0	417.2	-105 ft	-	*mud pits*	-	Overflow sample	3002	11

3 Overview of Sample Collection and Analytical Testing of Fluid Samples

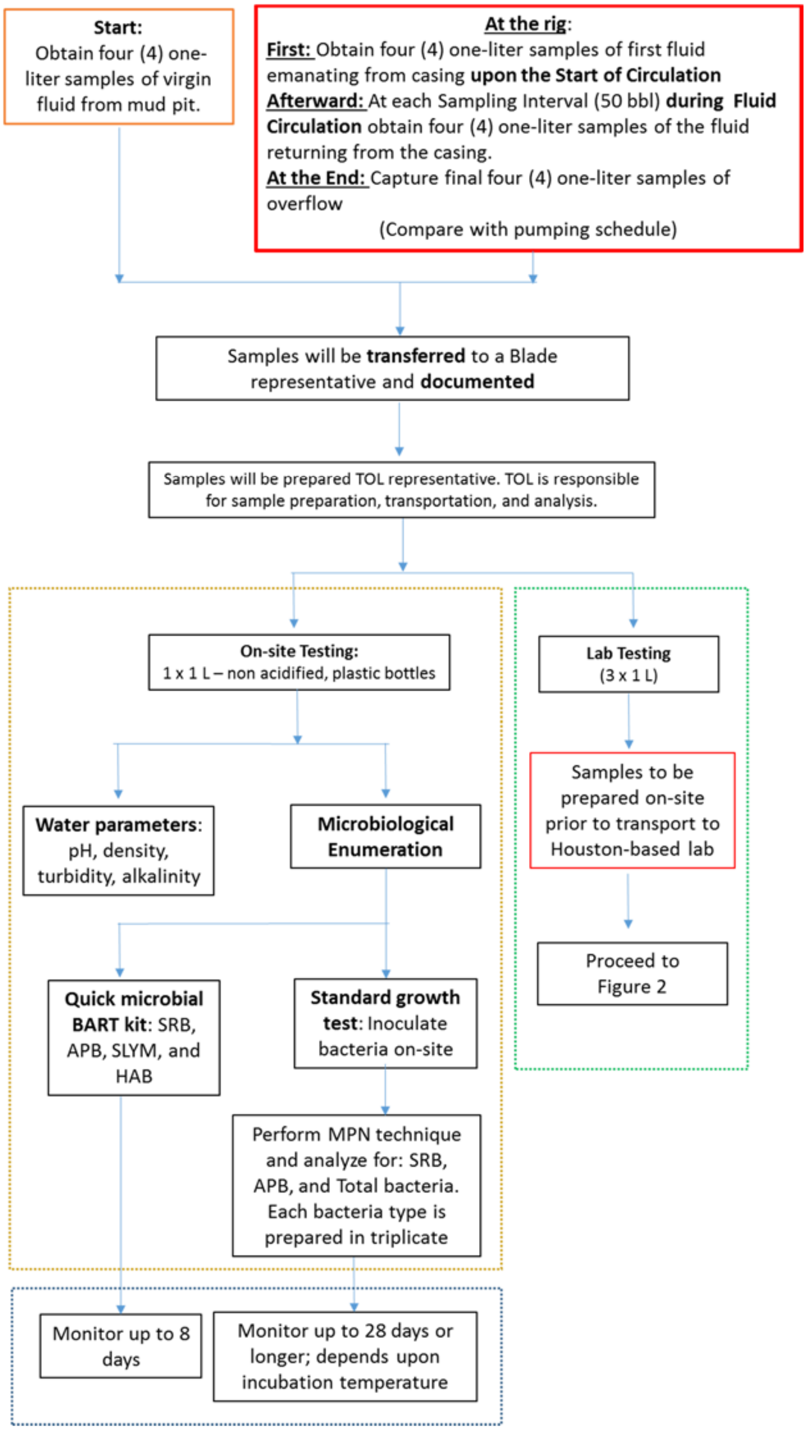

Figure 1. Flow chart for fluid sample collection and preparation. BART = biological activity reaction test.

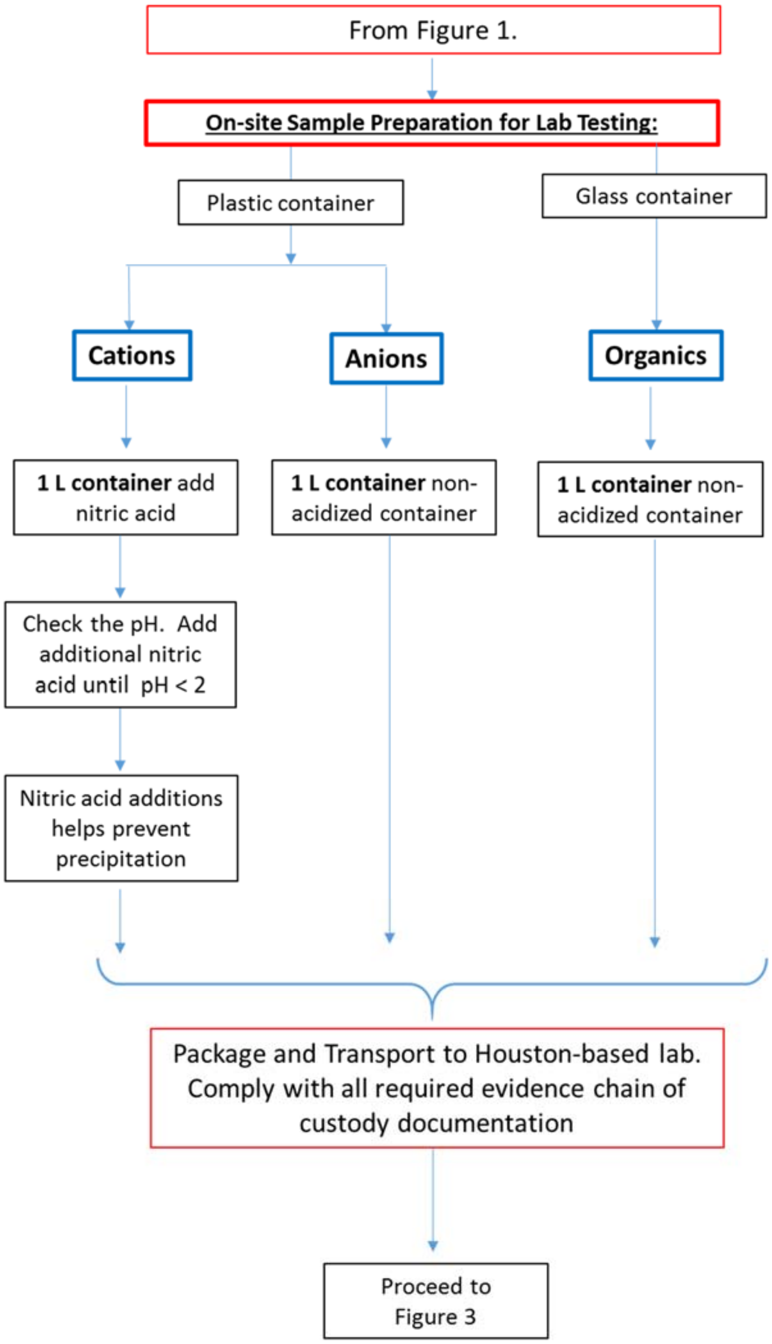

Figure 2. Flow chart for fluid sample collection and preparation.

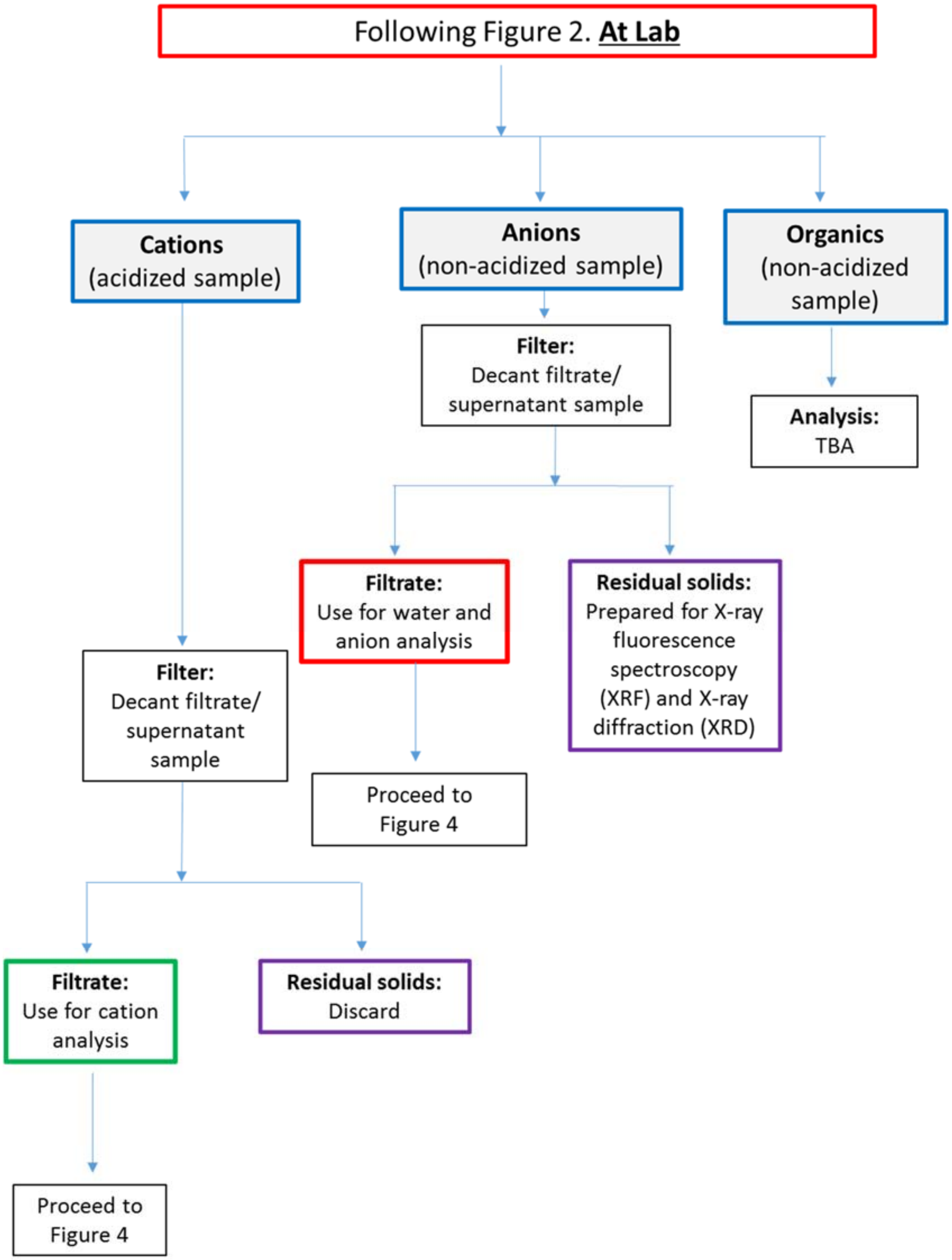

Figure 3. Flow chart for analyzing fluid samples at the Houston lab.

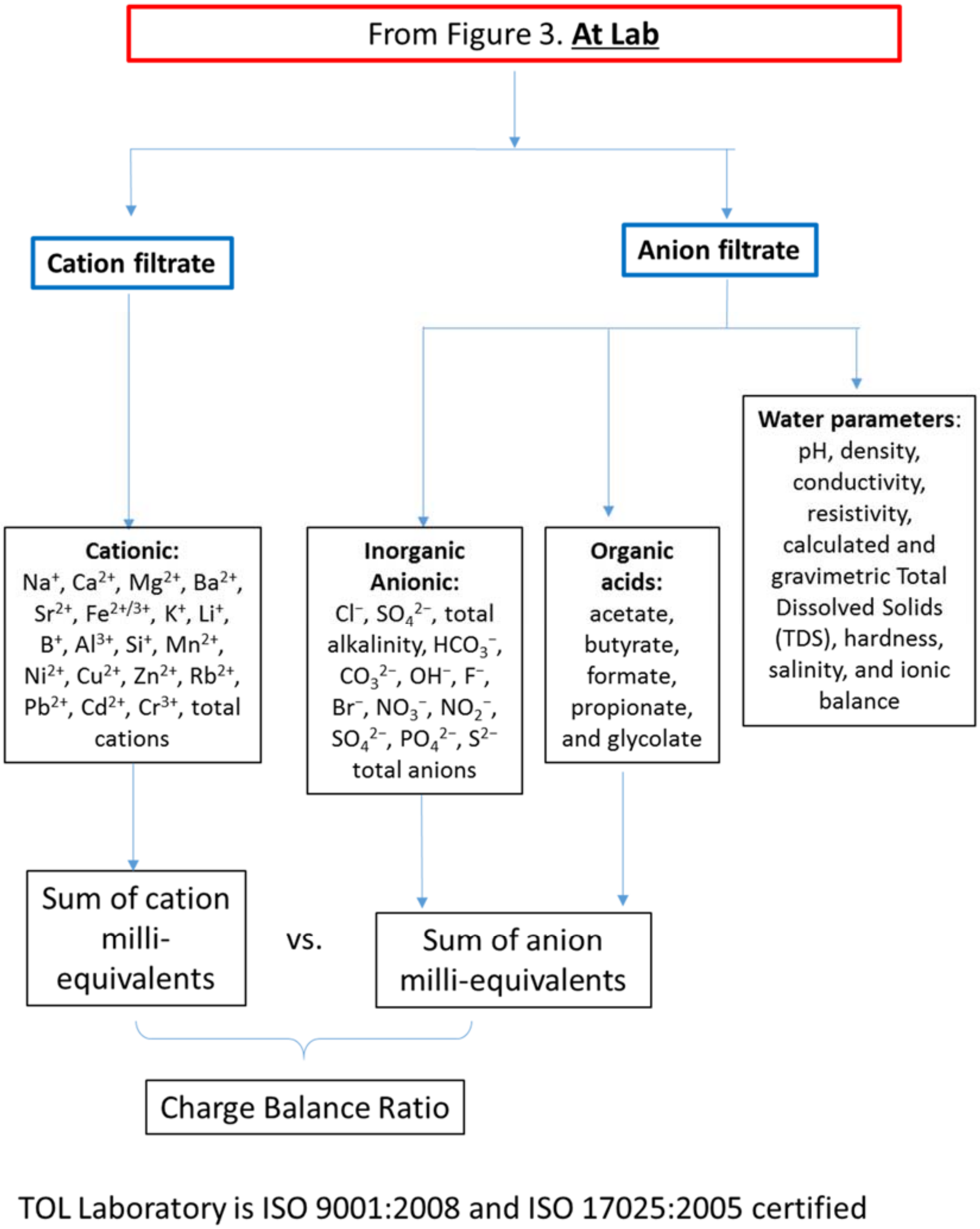

Figure 4. Flow chart for analyzing fluid samples at the Houston lab.

4 Proposed Fluid Collection Sequence

4.1 Fresh Drilling Fluid Collection

Fluid samples will be collected and documented according to the procedure outlined in document: *Aliso Canyon RCA SS-25 Site Evidence Collection and Documentation Protocol*, April 28, 2016, Version 008 (or latest version) by Blade.

4 one-liter samples of the fresh drilling fluid or other fluids will be collected each time fresh fluid is delivered to the drilling fluid tanks.

Analysis of the fresh fluids provides the basis for comparison with the fluids returned from the wellbore during operations. The analysis of the fresh fluids is performed for quality assurance and is carried out by a simplified protocol based on the fluid specs furnished by the fluid manufacturer.

4.2 Tubing/Annular Fluid Collection

Prior to the tubing extraction, the fluids will be circulated to exchange present **tubing/annular** fluids with fresh fluid. During circulation, 4 one- L samples will be taken every **50** bbl of fluid returns for analytical analysis.

At each 50 bbl circulated, 4 x 1 (one) liter sample of the returned fluid will be collected. The depth of the sample will be back calculated based on the volume circulated.

Approximately 9 **tubing/annulus** and 2 drilling fluid samples are anticipated to be collected.

4.3 Field Variances

As conditions in the field may vary, it may become necessary to implement minor modifications to the sampling protocol as presented in this plan. Blade reserves the right to deviate from these procedures and protocols as unique situations arise in the field. Any deviation from these procedures and protocols shall be documented and the appropriate entities notified.

5 Fluid Sampling Collection Procedure

5.1 Liquid Sample Collection

Fluid samples will be collected and put into EPA-approved sample containers. The return fluid samples will likely be obtained at the rig separator, however the exact location and hence method of obtaining the samples will be determined later when the physical arrangements will have become known.

Sample locations will be recorded and photographed and any physical reference points will be labeled.

Prepare samples for storage and shipment by TOL representative. Each sample bottle will be documented as described in the Evidence Collection and Documentation Protocol.

5.1.1 Sample Containers

Water samples will be collected in EPA-approved Nalgene™ Certified Wide-Mouth HDPE Bottle with Polypropylene Screw Closure containers.

Sample containers being ordered are as follows:

- 48 1L Amber Nalgene wide mouth plastic bottles,
- 24 950ml Amber tall wide mouth glass jars

Organic materials will be put in the glass jars and inorganic material will be put in the plastic Nalgene bottles.

Containers are to be pre-cleaned and will not be rinsed prior to sample collection. Preservatives, if required, will be added by a representative approved by the AC-RCA team to the containers prior to shipment of the samples to the laboratory.

6 Details of Chemical Analysis Protocol

The scope of the analytical work is consistent with API RP 45 - *Recommended Practice for Analysis of Oilfield Waters*. For each of the 4 one-L samples of fluid collected, a TOL representative will prepare samples for analytical analysis. Each container will serve a separate analytical function:

- On-site analysis:
  - 1 L – non-acidified sample for water parameters and bacteria enumeration (plastic container)
- Off-site analysis:
  - 1 L – acidified sample with concentrated nitric acid – for cation analysis (plastic container)
  - 1 L – non-acidified sample for anions (filtrate) and solids (residuals) (plastic container)
  - 1 L - non-acidified sample for organic material (glass container)

Sample preparation for off-site analysis will be performed in the field at Aliso Canyon.

6.1 On-Site Measurements

1 one-L fluid sample, collected in a plastic container, will be used to conduct water chemistry, quick microbiological tests, and inoculation of standard microbial growth tests in the field. The container is plastic.

6.1.1 Water Chemistry

Fluid Properties to be measured on-site are: pH, density, turbidity, and alkalinity. pH measurements will be made consistent with EPA Method 9040C – *pH Electrometric Measurement, part of Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*. These parameters need to be determined in the field immediately following the capture of a sample. This avoids a bias being generated through precipitation of certain minerals (calcite, iron oxy-hydroxide) on storage and transport of the sample to the laboratory. The pH should be measured with a properly calibrated pH electrode. Alkalinity titration will be performed with a burette and the appropriate acid to the methyl-orange endpoint.

6.1.2 Quick Microbiological Testing

Use Hach SRB-BART, APB-BART, SLYM-BART, and HAB-BART test kits to determine whether the fresh drilling and/or annular fluids are contaminated. BART kits contain a culture media which stimulates bacteria growth and which is observed for daily changes. Samples are inoculated on day one and are monitored for up to 8 days for indications of microbial growth (e.g. vial changes color). Refer to Figure 5.

BART – biological activity reaction test; SRB – sulfate reducing bacteria; APB – acid producing bacteria; SLYM – slime forming bacteria; HAB – heterotrophic aerobic bacteria.

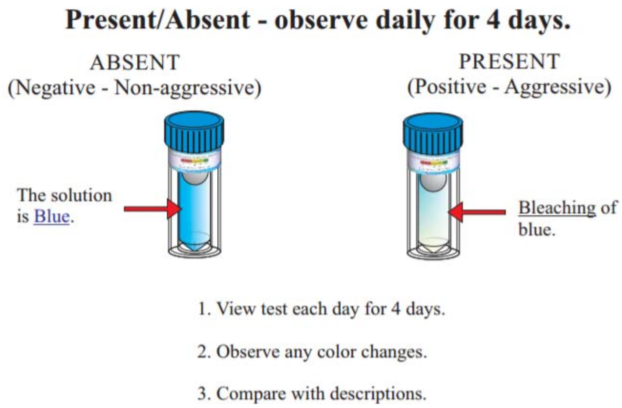

Figure 5. Example illustration of HAB-BART test kit.

6.1.3 Standard Microbiological Growth Tests

To assess the presence of microbes, the governing standard used for sampling will be NACE TM0194-2014, *Field Monitoring of Bacterial Growth in Oilfield Systems: Appendix B.* Bacteria culture vials are to be inoculated in the field using sterile containers. Sulfate reducing bacteria (SRB), acid producing bacteria (APB), and total (e.g., general heterotrophic) bacteria will be specifically monitored. The methodology to be used is the MPN (most probable number) dilute-to-extinction series approach (see Figure 6 and FDA, BAM Appendix 2 by R. Blodgett, 2010). From each fluid sample, 3 serial dilution series will be prepared for each of the three types of bacteria tests. Samples are monitored for up to 28 days.

The positive identification of bacteria is based on binomial statistics: growth (+) or no growth (–). The distribution of (+)/(–) for each dilution series is converted into an initial population density (cells/mL) from a statistical MPN table and assessed with ± 95% confidence.

Bacteria samples will be inoculated on-site, but the extended monitoring and statistical enumeration will be performed off-site.

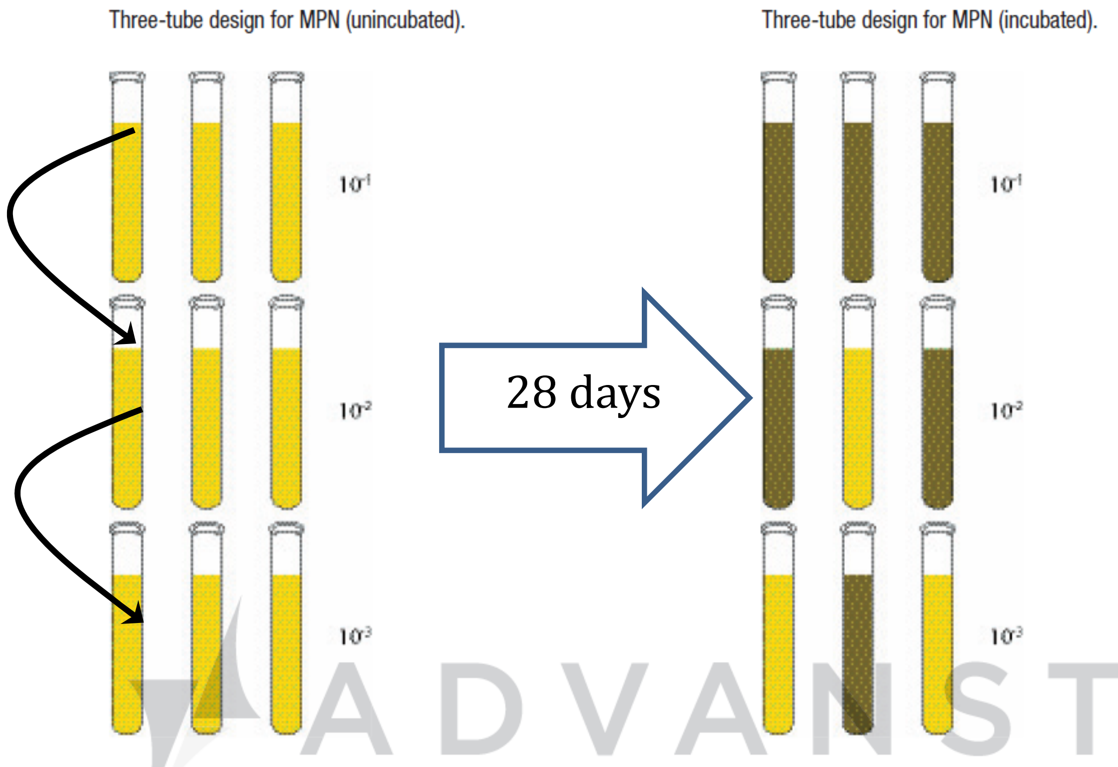

Figure 6. Illustration of the microbial growth test using the Most Probable Number (MPN) methodology.

6.2 Off-Site Laboratory Measurements

The remaining 3 one-L samples will be further sub-divided into individual sample containers in order to analyze for cations, anions, and organics.

6.2.1 Cation Determination

One (1) 1 L sample, collected in a plastic container, collected in a plastic container, will be used for cation analysis. The sample will be preserved by adding 50 mL of 10 wt % nitric acid (HNO_3) to the sample bottle. (This will prevent certain minerals from precipitating during storage and transport.) The bottle will be capped and lightly shaken to mix in the acid. A small quantity of sample will be poured into the bottle cap where the pH will be measured using pH paper. The pH must be ≤2. The sample in the cap will be discarded, and the pH of the sample will be adjusted further if necessary. The determination of the cation concentrations will be performed by the appropriate photo emission spectroscopic methods.

The filtrate will be analyzed following EPA Method 7000B – *FLAME ATOMIC ABSORPTION SPECTROPHOTOMETRY* and EPA Method 6010D – *INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROSCOPY* guidelines. Analysis is to be performed in the lab.

6.2.1.1 *Cations*

Cationic analytes: Na^+, Ca^{2+}, Mg^{2+}, Ba^{2+}, Sr^{2+}, $Fe^{2+/3+}$, K^+, Li^+, B^+, Al^{3+}, Si^+, Mn^{2+}, Ni^{2+}, Cu^{2+}, Zn^{2+}, Rb^{2+}, Pb^{2+}, Cd^{2+}, Cr^{3+}, and total cations.

6.2.2 Anion Determination

One (1) 1 L sample, collected in a plastic container, is to be used for inorganic anion, organic acids, and general chemistry analyses. This sample container is not acidized.

The filtrate will be analyzed following EPA Method 9056A – *DETERMINATION OF INORGANIC ANIONS BY ION CHROMATOGRAPHY* guidelines. Most of the anions will be determined by ion chromatography. Analyses are to be performed in the lab.

6.2.2.1 *Inorganic Anions*

Inorganic anionic analytes: Cl^-, SO_4^{2-}, F^-, Br^-, NO_3^-, NO_2^-, SO_4^{2-}, PO_4^{2-}, S^{2-} and total anions.

6.2.2.2 *Organic Acids*

Organic acid analytes: acetate, butyrate, formate, propionate, and glycolate.

6.2.2.3 *General Chemistry Properties*

Originating from the same sample container used to analyze for anions, the filtrate will be analyzed for the following:

- pH, density, conductivity, resistivity, calculated and gravimetric Total Dissolved Solids (TDS), hardness, salinity, and ionic balance.
- Alkalinity (e.g., carbonate, bicarbonate, carbonate, hydroxide, and total alkalinity) will be measured in the lab by titration. Alkalinity titration will be performed with a burette and the appropriate acid to the methyl-orange endpoint.

6.2.2.4 *Potential Solids in the Collected Annuluar Fluid Samples*

If the fluid sample is turbid or contains precipitates (deposits), it is proposed to filter the non-acidized sample and attempt to identify the solids if possible. A combination of X-ray diffraction (XRD) and/or X-ray Fluorescence (XRF) will be used to identify chemical speciation.

6.2.3 Organic Determination

One (1) 1 L sample, collected in a glass container, is to be used for organic material analyses. This sample container is not acidized.

There is a possibility that the fluid may contain organic material. Samples will be collected and shipped to the Houston-based lab. Analytical testing (if any) will be determined at a later date.

7 Potential Gases Measured at Separator During Displacement

During circulation, the return fluid will be directed to a separator.

As of April 19, 2017, there is no pressure reported on the tubing and A annulus (see AC-RCA Daily Site Report, 03-May-17).

It is anticipated that the separator pressure will remain atmospheric during circulation. In the unlikely event that pressure is detected, Tedlar bags and Summa canisters will be available to collect any discharge gas.

If gas samples are collected, the analytical protocol will be determined at a later date.

8 Potential Solids Retrieved From Recovered Pipe

Any solids or scale retrieved from the tubing or casing will be collected and stored for analytical laboratory analysis. Laboratory analysis will include X-ray diffraction (XRD) and qualitative polymerase chain reaction (qPRC).

XRD is useful to distinguish the identity of the chemical compound (e.g. calcium carbonate, iron oxide, or iron sulfide).

A subset of the sample will be preserved and will be analyzed by qualitative polymerase chain reaction (qPRC) which is a standard forensic fingerprinting technique to analyze for bacteria based on the DNA. This technique can differentiate between aerobic, anaerobic bacteria, and species associated with microbiologically induced corrosion (MIC).

Samples will be chilled to 4°C immediately upon collection.