Pacific Gas and Electric Company®

Utility Procedure: TD-4186P-100
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Internal Corrosion Control: Liquid and Solid Sampling and Testing

SUMMARY

This utility procedure describes steps for sampling and testing liquids and solids from Pacific Gas and Electric Company (Company) gas pipeline systems for internal corrosion evaluation. Chemical and bacteria analysis of the samples is used to evaluate internal corrosion activity and the effectiveness of internal corrosion control.

Level of Use: Information Use

TARGET AUDIENCE

Personnel engaged in or supervising liquid and solid sampling and testing from gas pipeline systems for internal corrosion evaluation.

SAFETY

Potential hazards impacting this work include, but are not limited to, the following:

- Chemical hazards
- Electrical hazards
- Traffic conditions
- Tripping and slipping hazards
- Environmental surroundings
- Construction sites

ABNORMAL OPERATING CONDITIONS (AOCs)

Abnormal operating conditions that could be encountered while performing this procedure may include, but are not limited to, the following:

- Confirmation that water is present
- Positive results from testing of bacteria inoculation vials (if bacteria testing is performed in the field)

Internal Corrosion Control: Liquid and Solid Sampling and Testing

BEFORE YOU START

Personal Protective Equipment (PPE): The following PPE must be worn by field personnel (or must be available for use if indicated) along with other applicable PPE as specified in the *Code of Safe Practices*:

- Gloves (latex or nitrile)
- Safety glasses
- Traffic vest
- Proper footwear
- Long sleeve shirt
- Long pants
- Respirator or mask (must be available)
- Hard hat (must be available)

Operator Qualifications (OQ): Field personnel conducting liquid and solid sampling must be trained for the task being performed. Operator qualification tasks to be developed.

Equipment: See Appendix A, "Equipment Checklist."

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PROCEDURE STEPS

1 General

Observe the following practices when performing liquid and solid sampling:

1.1 Dispose of hazardous liquid waste, solid waste, and sharps (needles, broken glass) properly.

1. Maintain separate containers for liquid waste, solid waste, and sharps throughout field testing.

2. Solid waste is not permitted in the liquid waste container, including ampoules containing liquid.

1.2 Keep sample bottle capped unless withdrawing a sample while performing liquid field testing, reducing the potential for contamination and limiting the quantity of dissolved gasses released.

1.3 IF deionized water is specified and not available,

THEN distilled water is an acceptable substitute. However, deionized water is preferred.

1.4 Sterilize and rinse equipment properly with deionized water to avoid contamination, before performing a liquid or solid field test.

1.5 When recording liquid levels, the liquid container must be level and the reading taken in the center of the meniscus at eye level.

1.6 Several tests rely on colorimetry to determine the concentration of corrosive species. Detecting a color change in darker samples may not be possible.

- IF a sample is determined to be too dark,

  THEN record the results for the test as “Sample too dark to perform test,” and perform the next test.

1.7 Check the expiration date of test kits and or chemicals before performing tests. Properly dispose of expired supplies and replace with unexpired items.

2 Liquid Sample Collection

2.1 General

1. Follow the steps in this section when sampling liquids from inside a pipeline.

2. Perform sampling at locations where liquids are present and a drip or other sampling location is present.

3. It is recommended that District or Division personnel obtain or draw the samples.

2.1 (continued)

4. Track the testing process with Form 62-1174 "Chain of Custody Record," and Gas Utility Form TD-4186P-100-F01, "Liquid Sampling Log."

2.2 Liquid Sample Collection Procedure

1. Prepare two new "Nalgene" or equivalent sample containers, caps, and labels:

   - One 60 mL container for bacteria testing
   - One 500 mL container for the remainder of the testing

2. Label each container with the same information. Labels can be obtained from Applied Technology Services (ATS). The label should include, but is not limited to:

   - Name of personnel performing the sampling
   - Sample Location
   - Date and time sampled
   - Pipeline number
   - Approximate mile point

3. Blow the system for several seconds into an appropriate container (e.g., truck, drum, bucket), removing striated material so the sample closely resembles the bulk fluid composition.

   - IF only a small amount of liquid is expected,

     THEN immediately collect the liquid as a sample.

4. IF needed,

   THEN retrofit the line so that the sample may be collected into the container.

   - IF an intermediate device is used to capture the liquids before depositing them into the sample container,

     THEN clean and sterilize the intermediate device with alcohol after each sample is taken to prevent contamination.

5. Fill both bottles to the top with liquid to eliminate air (if sufficient volume of liquid is present) AND secure the lids tightly.

2.2 (continued)

6. Field test the liquid in the 500 mL bottle for temperature and the presence of water per Appendix B, Section B.

   - Immediately take a temperature reading per Appendix B.
   - With the lid on, allow a few minutes for any liquid separation. Withdraw a small amount of liquid from the water layer (typically the bottom of the container) using a clean syringe or pipette. Place a few drops of sample on the water finding paper.
   - IF a positive indication results,

     THEN immediately take the pH of the water as described in Appendix B.
   - Record the data on Gas Utility Form TD-4186P-100-F01, "Liquid Sampling Log."
   - Perform any additional field tests as required by corrosion services in Appendix B.
   - Store remaining liquid for laboratory analysis.
     - IF the full amount of sample could not be collected,

       THEN perform as many required field tests as possible and return any remaining liquid for laboratory testing. A minimum sample volume of 25 mL is typically needed for laboratory analysis.

7. Reserve the 60 mL bottle sample for bacteria testing.

   - IF the sample cannot get to ATS within 24 hours, cannot be kept chilled, or both,

     THEN perform the field bacteria inoculation and fixation as described in Appendix D and transfer the test kit to ATS along with the chain of custody and remaining liquid sample.

8. Complete the Form 62-1174 "Chain of Custody Record," to transfer the samples to ATS. Refer to Section 4.

9. Deliver samples to ATS on ice with the completed Form 62-1174 "Chain of Custody Record," within 24 hours of taking the sample to obtain the most accurate test results.

2.2.9 (continued)

- Before delivery to ATS, store the bottles in a cool dark environment (approximately 40 degrees Fahrenheit). Do not freeze the bottles or store them in a location containing food. A dedicated sample refrigerator should be obtained and located in the shop facility.

3 Solid Sample Collection

3.1 General

1. Follow the steps in this section when sampling solids from inside the pipeline.
2. Types of solid samples include the following:
   - Solids in the form of debris
   - Scale or deposits
   - Sludge
   - Biofilms or slime
3. Isolate and collect in separate containers, when possible, samples that display different properties, such as color, texture, density, or composition.

3.2 Solid Sample Collection Procedure

1. Prepare a new, clean 250 mL sample container or plastic 8 ounce sample bag, such as a Whirl PakR.
2. Collect solids with a sterile tongue depressor or clean stainless steel spatula that has been sterilized with an alcohol swab.
3. Hard tenacious deposits may need to be scraped off the pipe or vessel surface.
4. IF the samples are originally moist,

   THEN protect them from drying out by completely filling an air-tight container with sample.
5. IF a plastic bag is being used to contain the sample,

   THEN squeeze the bag to eliminate the air after sampling AND seal the bag.
6. Identify where the sample is being collected and look carefully for any difference in color, texture, density, or composition in the solid materials present inside the piping. Multiple solid or sludge samples may be required if different materials are present.

3.2 (continued)

7. Label all samples, per Section 2.2.

8. Store the samples in a cool dark environment, such as a cooler with ice, if possible.

4 Laboratory Analysis

4.1 General

- After field testing is complete, solid and liquid samples should be kept in a cool environment, labeled, packaged and transported to a laboratory for analysis.
- Check for specific Company rules and regulations governing the transportation of the components.
- It is recommended to gather all samples in one day and transport them to the laboratory the following day at the latest. More accurate test results are achieved when minimal time elapses between sample collection and testing.

4.2 Labeling

1. Label each container with the same information. Labels can be obtained from ATS. The label should include, but is not limited to:
   - Name of personnel performing the sampling
   - Sample Location
   - Date and time sampled
   - Pipeline number
   - Approximate mile point
2. Complete the laboratory Form 62-1174 "Chain of Custody Record," with the following information:
   - Contact information for personnel delivering the sample
   - Facility sample is going to
   - Turnaround time
   - Sample information (up to 12 different samples can be included on this form). This is important so the test data can be identified with the proper sample.
   - Tests to be performed on each sample (refer to Section 4.3)

4.2.2 (continued)

- Proper accounting information for ATS
- Recipients of results. Include corrosion services at a minimum.

4.3 Required Analysis

1. Liquid Samples

Analyze liquid samples for the following information:

a. Water content (% by volume).

b. Iron (Fe), Manganese (Mn), Chloride (Cl^-).

c. Presence of bacteria.

d. CAM 17 metals (California Administrative Manual which lists 17 metals that can qualify waste as hazardous).

e. IF greater than 1% water by volume is detected in the sample,

THEN analyze for the following:

- pH
- Specific gravity
- Total dissolved solids (TDS)

2. Solid Samples

Analyze solid samples for the information listed below. Suggested techniques for performing the required analysis are provided; however, alternative techniques are permissible, provided similar information can be obtained.

a. Elemental composition.

- Energy dispersive spectroscopy

b. Crystalline material identification.

- X-ray powder diffraction

4.4 Transportation to the Laboratory

1. General

- Provide the laboratory with as much notice as possible before delivering samples for analysis.
- Laboratory personnel may pick up samples, or alternatively, the samples can be driven or shipped in an ice chest to the ATS facility.
- Ensure that labeling and packaging requirements are followed when shipping hazardous materials.
- The chilled samples should arrive to the laboratory within 24 hours after collection.
- Include the completed Form 62-1174 "Chain of Custody Record."

2. Contact Information

The contact information for the laboratory is as follows:

Main Contact:	Alternate Contact:
Redacted PG&E ATS Redacted Redacted Office: Redacted Mobile: Redacted E-mail: Redacted	Redacted PG&E ATS Redacted Redacted Office: Redacted E-mail: Redacted

4.5 Results

The laboratory will e-mail test results typically between 5 and 10 days after receipt of the samples. Bacteria testing results will be received approximately 1 month after receipt of the samples. To avoid delays, the lab should send bacteria testing results in a separate report. The lab should also include a summary of results at the beginning of the reports.

- Testing results that are outside the acceptable limits should be retested for confirmation.
  - IF the results are confirmed,

    THEN create an action plan, as needed, that includes additional testing, mitigative action, or both.
  - See Gas Utility Procedure TD-4186P-400, "Internal Corrosion Control: Mitigation," for more information.

5 Reporting

Report results in the appropriate form and retain for as long as the pipeline remains in service. The appropriate form depends on the tests performed and may include one of the following as indicated in procedure:

- Gas Utility Form TD-4186P-100-F01, "Liquid Sampling Log"
- Form 62-1174 "Chain of Custody Record"
- Laboratory testing reports

END of Instructions

Internal Corrosion Control: Liquid and Solid Sampling and Testing

DEFINITIONS

Acid-Producing Bacteria (APB): Bacteria that produce organic acids as an end product of their metabolism, which may be aerobic or anaerobic.

Alkalinity: A measure of water's ability to neutralize acids.

Aqueous: A liquid containing water.

Biocides: An additive used to kill or control bacteria.

Microbiologically Influenced Corrosion (MIC): Metal corrosion or deterioration which is aided by metabolic activity of microorganisms.

pH: The negative logarithm of the hydrogen ion activity written as:

pH = -log10(aH+)

Where: aH+ = hydrogen ion activity = the molar concentration of hydrogen ions multiplied by the mean ion-activity coefficient.

Planktonic Bacteria: Free-floating or free-swimming bacteria in bulk fluids.

Sessile Bacteria: Bacteria attached to a surface.

Sulfate Reducing Bacteria (SRB): A group of anaerobic bacteria that reduce sulfate to sulfide.

IMPLEMENTATION RESPONSIBILITIES

Corrosion services ensures all impacted personnel are aware of this utility procedure.

Superintendents and supervisors communicate this utility procedure to personnel who perform liquid and solid sampling and testing and ensure that personnel are trained and qualified to perform these tasks.

GOVERNING DOCUMENT

Gas Utility Standard TD-4186S, "Internal Corrosion Control of Gas Facilities"

COMPLIANCE REQUIREMENT / REGULATORY COMMITMENT

49 CFR 192.475 "Internal corrosion control: General"

49 CFR 192.477 "Internal corrosion control: Monitoring"

49 CFR 192.491 "Corrosion control records"

Internal Corrosion Control: Liquid and Solid Sampling and Testing

REFERENCE DOCUMENTS

Developmental References:

NACE SP0106-2006, "Control of Internal Corrosion in Steel Pipelines and Piping Systems"

TM0194, "Field Monitoring of Bacterial Growth in Oil and Gas Systems"

Supplemental References:

NA

APPENDICES

Appendix A, "Equipment Checklist"

Appendix B, "Liquid Sample Field Testing"

Appendix C, "Solid Sample Field Testing"

Appendix D, "Bacteria Testing"

ATTACHMENTS

NA

FORMS

Gas Utility Form TD-4186P-100-F01, "Liquid Sampling Log"

Internal Corrosion Control: Liquid and Solid Sampling and Testing

DOCUMENT RECISION

Gas Design Standard O-16, "Corrosion Control of Gas Facilities" is being replaced by the following document set:

- Gas Utility Standard TD-4180S, "General Corrosion Control of Gas Facilities"
- Gas Utility Standard TD-4181S, "External Corrosion Control of Gas Facilities"
- Gas Utility Standard TD-4186S, "Internal Corrosion Control of Gas Facilities"
- Gas Utility Standard TD-4188S, "Atmospheric Corrosion Control of Gas Facilities"
- Gas Utility Procedure TD-4181P-101, "Cathodic Protection Area (CPA) Design and Modification"
- Gas Utility Procedure TD-4181P-201, "Cathodic Protection Monitoring and Restoration"
- Gas Utility Procedure TD-4181P-202, "Cathodic Overprotection"
- Gas Utility Procedure TD-4181P-301, "Rectifier Maintenance and Adjustment"
- Gas Utility Procedure TD-4186P-100, "Internal Corrosion Control: Liquid and Solid Sampling and Testing"
- Gas Utility Procedure TD-4186P-200, "Internal Corrosion Control: Design Review"
- Gas Utility Procedure TD-4186P-300, "Internal Corrosion Control: Corrosion Rate Monitoring"
- Gas Utility Procedure TD-4186P-400, "Internal Corrosion Control: Mitigation"
- Gas Utility Procedure TD-4186P-500, "Internal Corrosion Control: Annual Program Review"

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Internal Corrosion Control: Liquid and Solid Sampling and Testing

REVISION NOTES

Where?	What Changed?
All	This is a new utility procedure, part of the document set replacing the internal corrosion control requirements portion of Gas Design Standard O-16, "Corrosion Control of Gas Facilities." O-16 is being updated and rewritten to comply with the new Company guidance document requirements, and is being reorganized and expanded into the following four standards with multiple procedures under each standard: • General Corrosion Control • External Corrosion Control • Internal Corrosion Control • Atmospheric Corrosion Control See the Guidance Document Analysis for details.

Appendix A, Equipment Checklist

Below is a list of equipment that may be needed depending on the types of tests performed.

A. Laboratory gloves ..... _____

B. Safety glasses ..... _____

C. Paper towels ..... _____

D. Sharps container ..... _____

E. 60 mL sample bottles (https://us.vwr.com/ Item# 16124-982) ..... _____

F. 500 mL sample bottles (https://us.vwr.com/ Item# 16124-988) ..... _____

G. Liquid waste container ..... _____

H. 8 oz. sample bags (optional) ..... _____

I. Cooler (with ice) ..... _____

J. Pipettes ..... _____

K. Tongue depressors or stainless steel spatula ..... _____

L. Alcohol swabs ..... _____

M. Deionized water ..... _____

N. Permanent marker ..... _____

O. Thermometer ..... _____

P. Water finding paper (https://www.microessentiallab.com/ Item# WF-130) ..... _____

Q. pH meter ..... _____

R. pH paper (https://www.microessentiallab.com/ Item# 140) ..... _____

S. CHEMetrics Carbon Dioxide (CO_2) 100-1000 ppm Test Kit (optional) (www.chemetrics.com , Item# K-1920) ..... _____

T. HACH Alkalinity test kit (optional) ..... _____

1. Check expiration dates on indicator pillows and sufficient number ..... _____
2. Sufficient amount of sulfuric acid

U. CHEMets H_2S test kit (optional) (www.chemetrics.com Item# K-9510) ..... _____

V. Lead acetate paper (optional) ..... _____

W. Bacteria testing kits (Contact corrosion services for ordering information.) ..... _____

1. Check expiration dates ..... _____
2. Ensure PBS vials are full ..... _____

Appendix B, Liquid Sample Field Testing
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A. General

1. Generally, begin field testing immediately after sample collection AND complete within 10 to 15 minutes.

2. At a minimum, immediately perform the following field tests described in Section B below:

   a Temperature.

   b Presence of water.

   c pH (if water is present).

3. IF Corrosion Engineering does not request additional field tests,

   THEN transport the sample to the laboratory.

4. IF Corrosion Engineering requests additional field tests,

   THEN perform the additional field tests in the order listed below. See Section B for field testing procedures, with the exception of bacteria inoculation, which is covered in Appendix D.

NOTE

Field tests (b) through (d) below are performed only on liquid samples that contain water. Samples collected will often contain a water phase and a hydrocarbon phase which separate into layers.

The term “sample,” in test procedures refers only to the layer that contains water.

   a Bacteria Inoculation.

   b Dissolved carbon dioxide (CO_2).

   c Alkalinity.

   d Dissolved hydrogen sulfide (H_2S).

5. When adding drops to a solution, hold the dropper vertical to keep the drop size consistent. Keep track of the number of drops.

B. Liquid Sample Field Tests (500 mL Sample)

1. Temperature

NOTE
Sample temperature can change depending on ambient conditions.

Immediately take temperature readings for the 500 mL liquid samples by completing the following steps:

a Obtain a clean thermometer or electronic temperature probe that has been sterilized with an alcohol swab.

b Insert the thermometer or probe into the liquid sample.

c Record the temperature in Fahrenheit (°F) when a stable reading is obtained on Gas Utility Form TD-4186P-100-F01, "Liquid Sampling Log."

d Clean the thermometer or probe with deionized water after each measurement.

2. Presence of Water

Test the 500 mL sample for the presence of water following the steps below to determine if additional field tests are needed.

- IF testing indicates that no water is present,

  THEN no further field testing is needed.

a Tear off at least 2 inches of water finding paper from the dispenser.

b Withdraw a small amount of liquid from the water layer (typically the bottom of the container) using a clean syringe or pipette. Place a few drops of sample on the paper.

c IF the sample is too dark or a water layer cannot be seen,

  THEN shake the sample AND dip the paper into the sample.

d A positive result for the presence of water is indicated by a lavender or pink color change on the water finding paper. Figure 1 shows a positive result for the presence of water.

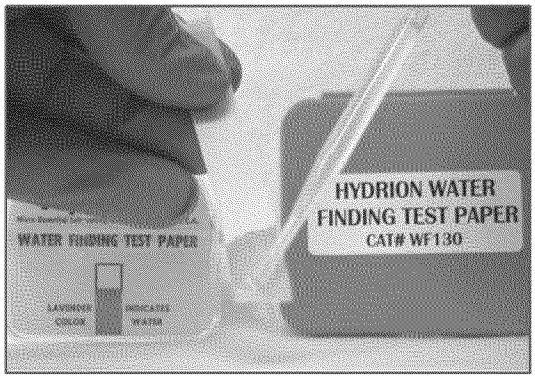

Figure 1. Positive Indication for the Presence of Water (Color Pink)

e Samples containing trace amounts of water will produce a less noticeable color change in the water finding paper compared to samples that are primarily water. The color change will typically be seen along the edge of the liquid front as shown in Figure 2.

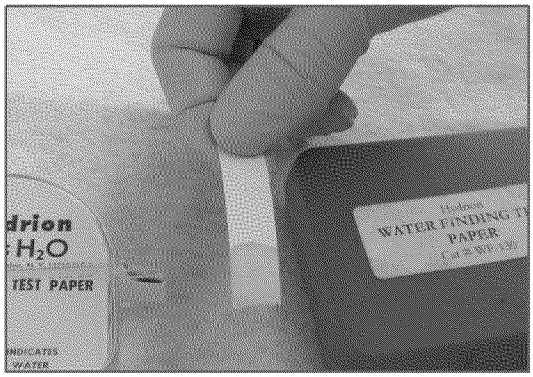

Figure 2. Positive Indication for the Presence of Water in a Sample Containing Trace Amounts of Water

3. pH

Determine the pH for liquid samples testing positive for the presence of water. Use either pH paper or a pH meter. pH meter readings are more reliable and preferred.

a Procedure – pH Meter.

CAUTION

Sensors on the pH meter are extremely delicate.
Do not touch with anything other than liquid samples.

(1) Calibrate the pH meter according to the manufacturer's specification. Calibrate the meter, at a minimum, before the first use of the day and every 4 hours afterwards while field testing is being performed.

(2) Rinse the pH meter with deionized water after calibration.

(3) Use a clean syringe or pipette to withdraw a small amount of sample and place a couple of drops on the meter's sensor.

(a) The sample must completely cover the meter's sensors.

(4) Once the reading has stabilized, record the pH on Gas Utility Form TD-4186P-100-F01, "Liquid Sampling Log."

(5) Rinse the pH meter with deionized water after each reading.

b Procedure – pH Paper.

CAUTION

Poor test results may result from touching the wet end of the pH paper being used for comparison or handling the paper with wet hands.

(1) Use a pipette to withdraw a portion of the sample and place it on a strip of pH paper.

(2) Compare the color change to the provided color code. An example color code is shown in Figure 3.

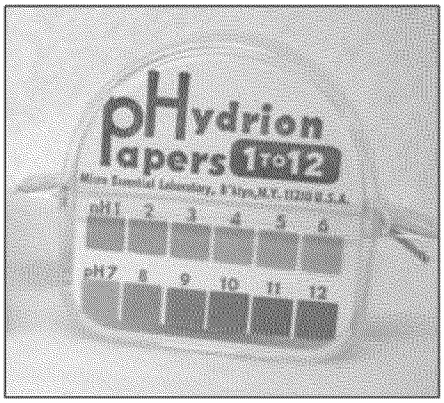

Figure 3. Example pH Paper Color Code

4. Dissolved Carbon Dioxide (CO_2)

These steps describe testing dissolved CO_2 concentration over the range 100-1000 mg/L using the CHEMetrics 1920 test kit. This is a colorimetric method of testing; handle the darker samples as described in Section 1.6.

- IF a different test kit is used,

  THEN follow the manufacturer's instructions for performing the test.

a Fill the test kit sample cup to the 20 mL mark with the sample as shown in Figure 4.

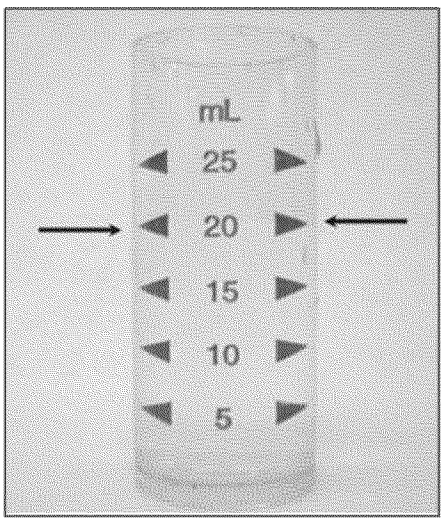

Figure 4. 20mL Mark on Sample Cup

b Add 2 drops of A-1900 activator solution to the cup.

c Stir the solution with the tip of the ampoule.

d Hold the valve assembly and ampoule in one hand. Use the other hand to snap the tip of the ampoule at the black score mark as shown in Figure 5.

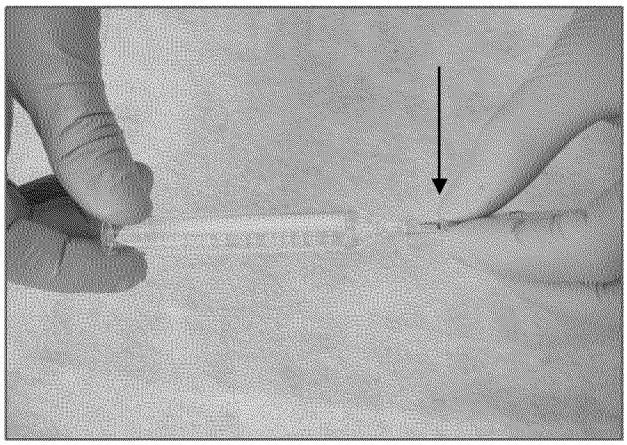

Figure 5. Snapping the Tip of the Ampoule

e Lift the control bar of the Titrettor AND insert the titret ampoule and valve assembly into the Titrettor as shown in Figure 6. The tip of the valve assembly should stick out of the Titrettor.

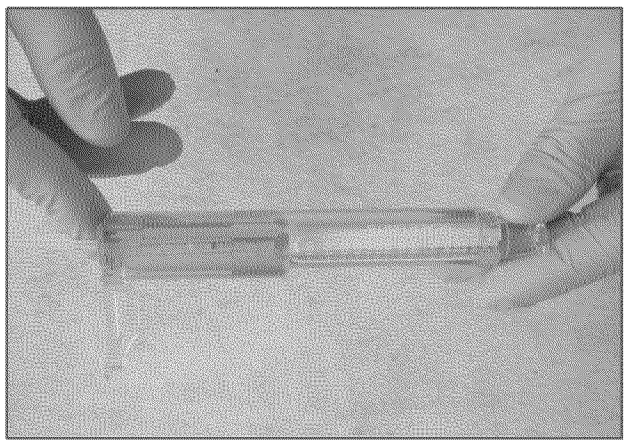

Figure 6. Inserting the Ampoule and Assembly into the Titrettor

f Hold the Titrettor over the sample cup with the valve assembly submerged in the sample.

NOTE

Never press the control bar when the valve assembly is not submerged in liquid.

g Press the control bar firmly and briefly, pulling a small amount of sample into the ampoule.

h Invert the Titrettor a few times to mix the sample. The solution inside the ampoule should turn a pink color.

i Repeat steps (g) and (h) until the solution inside the ampoule turns permanently from pink to colorless.

j Once a permanent color change (from pink to colorless) has occurred, remove the ampoule from the Titrettor.

k Hold the ampoule in a vertical position with the valve assembly point-up and read the scale at the center of the meniscus as shown in Figure 7. Record the results as mg/L of dissolved CO_2.

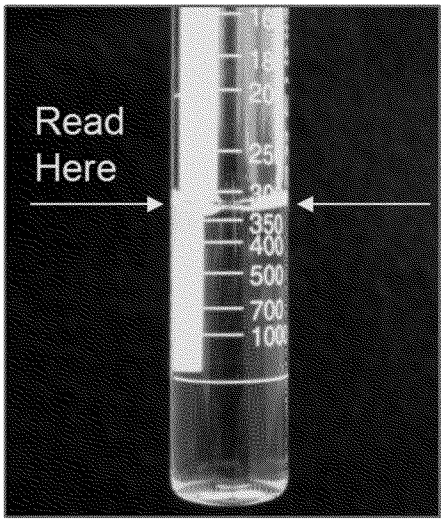

Figure 7. Reading the Liquid Level in the Ampoule

l IF a permanent color change, from pink to colorless, does not occur,

THEN the dissolved CO_2 concentration is below the range of the test kit. Record the dissolved CO_2 concentration as < 100 mg/L.

m IF the ampoule changes from colorless to pink to colorless immediately after the first amount of sample is added,

THEN the dissolved CO_2 concentration is beyond the range of the test kit. Record the dissolved CO_2 concentration as > 1,000 mg/L.

5. Alkalinity High and Low Range Test

The steps below describe testing alkalinity in two different ranges using the HACH Alkalinity Test Kit. The alkalinity ranges are:

- High Range Test: 20 – 400 mg/L
- Low Range Test: 5 – 100 mg/L

Perform the high range test first. The low range test is only used if the results from the high range test indicate an alkalinity of 100 mg/L or less. This is a colorimetric method of testing; handle darker samples as described in Section 1.6.

- IF a different test kit is used,

  THEN follow the manufacturer's instructions for performing the test.

a Prepare the mixing bottle with the sample to be tested using one of the following methods:

(1) For the High Range Test:

- Fill the plastic sample tube to the top with the sample to be tested.
- Place the mixing bottle upside-down on top of the sample tube AND invert to transfer the sample to the mixing bottle.

(2) For the Low Range Test:

- Fill the mixing bottle with the sample to the 23 mL mark.

b Tear open a Phenolphthalein indicator pillow and add it to the mixing bottle as shown in Figure 8.

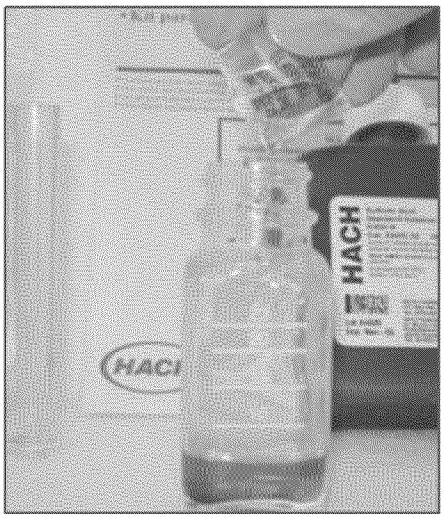

Figure 8. Adding Phenolphthalein indicator to a Mixing Bottle

c Gently swirl the bottle to mix the contents.

NOTE
In most cases the sample will remain colorless, as sample pH of 8.3 is necessary to turn the phenolphthalein indicator pink.

d IF the solution remains colorless after adding the indicator,

THEN the phenolphthalein alkalinity (or hydroxide alkalinity) is 0. Proceed to Step h. Otherwise, continue with the next step.

e IF the solution does not remain colorless,

THEN add a drop of 0.035 N sulfuric acid solution to the mixing bottle AND swirl the bottle. After swirling, check to see if the solution turns clear.

f Repeat Step e. until the solution turns clear. Count each drop as it is added.

g Record the number of drops required to reach a clear solution. The phenolphthalein alkalinity is calculated as:

- High Range: Phenolphthalein Alkalinity (mg/L) = drops of acid x 20
- Low Range: Phenolphthalein Alkalinity (mg/L) = drops of acid x 5

h Add the contents of a Bromocresol Green – Methyl Red indicator powder pillow, to the mixing bottle which contains the sample, the Phenolphthalein indicator powder, and any sulfuric acid added in the previous steps.

i Gently swirl the contents of the mixing bottle. The mixture should turn a green or green-blue color as shown in Figure 9.

- IF the solution turns a pink or red color after adding the indicator,

  THEN the total alkalinity is 0 mg/L. This usually occurs because the pH is less than 4.3.

Figure 9. Adding Bromocresol Green – Methyl Red Indicator to a Mixing Bottle.

j Add a drop of 0.035 N sulfuric acid solution to the mixing bottle, AND swirl the bottle after each drop.

(1) IF the solution turns a pink/red color as shown in Figure 10,

THEN proceed to Step k.

(2) IF the solution turns a purplish color as shown in Figure 11,

THEN repeat Step j. until the solution has changed completely to a pink/red color OR 20 drops of sulfuric acid have been added.

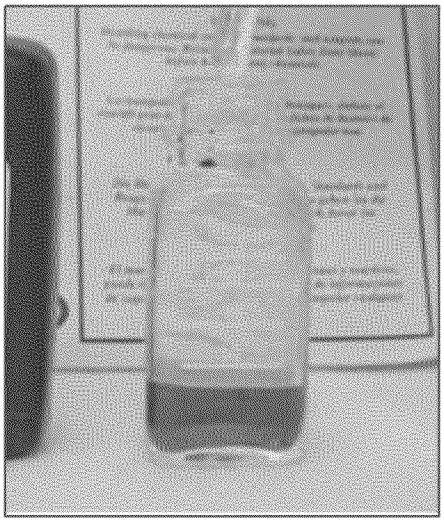

Figure 10. Color Change to Pink/Red after Adding Sulfuric Acid

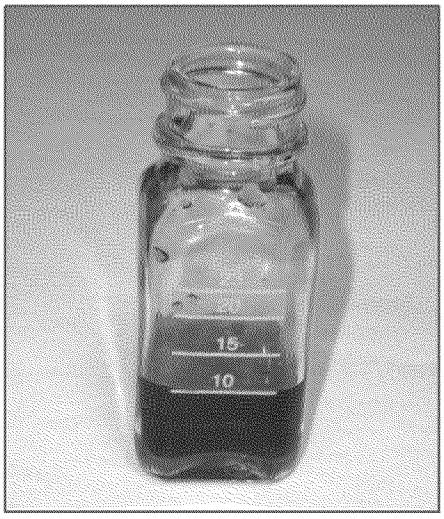

Figure 11. Incomplete Color Change – Additional Sulfuric Acid is Necessary

k IF the solution does not change to a pink/red color after adding 20 drops,

THEN record the alkalinity of the sample as >400 mg/L.

l IF the solution turns pink/red with 5 or fewer drops of sulfuric acid,

THEN perform the Low Range Test. See Step 5.a (2) above.

m Record the number of drops of sulfuric acid required to change the sample to a pink/red color. The bicarbonate/carbonate alkalinity is calculated as:

- High Range: Bicarbonate/carbonate Alkalinity (mg/L) = drops of acid x 20
- Low Range: Bicarbonate/carbonate Alkalinity (mg/L) = drops of acid x 5

n IF both the Phenolphthalein and Bromocresol indicators were used,

THEN record the Total Alkalinity as:

- Total Alkalinity (mg/L) = Phenolphthalein Alkalinity + Bicarbonate/carbonate Alkalinity

6. Dissolved Hydrogen Sulfide (H_2S)

The steps below describe the method for determining the amount of dissolved H_2S present in a liquid sample using the CHEMets (K-9510) test kit. This is a colorimetric method of testing; handle the darker samples as described in Section 1.6.

- IF a different test kit is used,

THEN follow the manufacturer's instructions for performing the test.

a Fill the test kit sample cup to the 25 mL mark with the sample to be tested as shown in Figure 12.

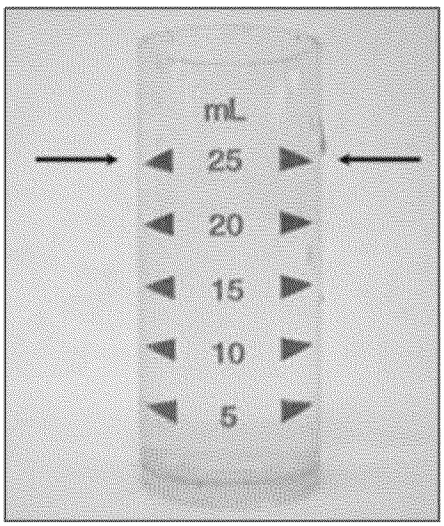

Figure 12. 25mL Mark on the Sample Cup

b Add 3 drops of A-9500 activator solution to the sample cup.

c Stir the sample with the tip of an ampoule to mix the contents of the sample cup.

WARNING

Use care when handling ampoule, as tip may be sharp from being snapped.

d Submerge the tip of the ampoule into the sample AND snap the tip of the ampoule against the side of the cup as shown in Figure 13. The ampoule should fill with sample, leaving a small bubble to help mix the ampoule contents.

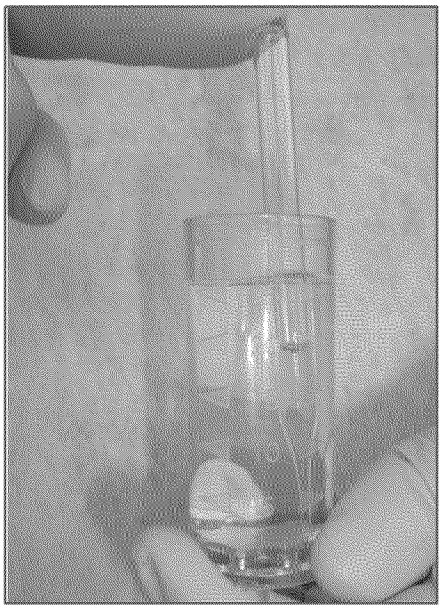

Figure 13. Snapping the Tip of the Ampoule on the Side of the Cup

e Mix the contents of the ampoule by inverting the ampoule several times causing the bubble to travel from one end of the ampoule to the other.

f Wipe off the liquid on the outside of the ampoule.

g Allow the ampoule to sit for exactly 5 minutes while color development occurs.

h Place the ampoule between the CHEMet color standard and move it from left to right along the comparator until the best color match is found as shown in Figure 14.

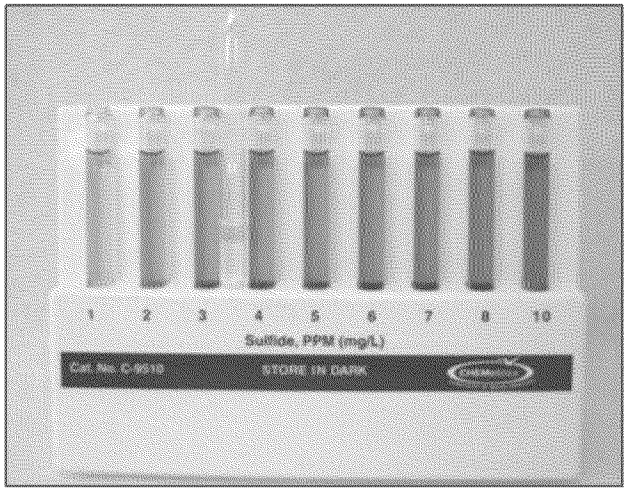

Figure 14. CHEMets H_2S Ampoule Color Standard (High Range)

i Record the H_2S concentration corresponding to the matching color. The value is listed on the base of the comparator in mg/L.

j IF the color of the ampoule appears to be between the colors of two adjacent standards,

THEN record an estimation of the concentration.

k IF the ampoule color is lighter than the high range comparator,

THEN place the flat end of the ampoule down in the center of the low range comparator.

l Point the top of the comparator towards a light source and view from the bottom as shown in Figure 15.

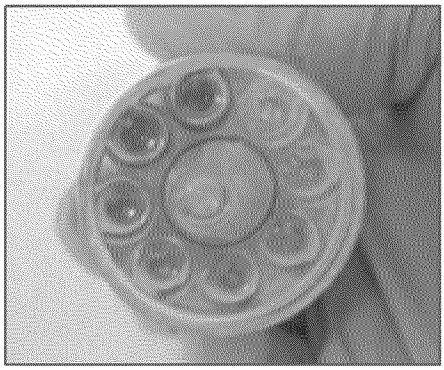

Figure 15. CHEMets H2S Ampoule Color Standard (Low Range)

m Move the comparator around in a circle until the color standard below the test ampoule is the closest match.

n Record the H_2S concentration corresponding to the matching color. The value is labeled on the side of the tube in mg/L.

Appendix C, Solid Sample Field Testing
Page 1 of 3

A. General

1. Perform the following field tests (as required by corrosion services) immediately after sample collection on each sample type collected from the pipeline:

   - pH
   - Presence of carbonates
   - Presence of sulfides
   - Bacteria inoculation

2. Procedures for performing these field tests are contained in this section, with the exception of bacteria inoculation, which is provided in Appendix D.

B. Solid Sample Field Tests

1. pH

   Determine the pH for any solid samples collected using either pH paper or a pH meter. pH meter readings are more reliable and preferred.

   a Procedure – pH Meter.

   (1) Calibrate the instrument according to the manufacturer's specification. The meter must be calibrated while field testing is being performed at least:

   - Before the first use of the day
   - At 4 hour intervals thereafter

   (2) Rinse the pH meter with deionized water following calibration.

   (3) Place a few drops of deionized water on the pH meter AND record the reading.

   (4) Place approximately 1 mL of solids or sludge in a clean test tube or sample vial, and add 1-2 mL of deionized water.

   (5) Mix the contents thoroughly by gently shaking the test tube or vial.

(6) Using a clean syringe or pipette, withdraw a small amount of sample and place a couple of drops on the meter's sensor.

- The sample must completely cover all of the meter's sensors.

(7) Once the reading has stabilized, record the pH.

(8) Rinse the pH meter with deionized water after each reading.

b Procedure – pH Paper

(1) Remove a strip or section of paper from the dispenser. Use a new piece of paper for each test.

(2) Moisten the pH paper with 2 drops of deionized water. Check and record the pH value.

(3) Wet the sample solid or sludge with 2-3 drops of deionized water.

(4) Place a new piece of pH paper on the sample for 1 minute and compare the paper color with the provided color code.

(5) Record the pH test results.

2. Presence of Carbonates and Sulfides

Test solid samples qualitatively for the presence of carbonates and sulfides as described below.

a Using a sterile instrument, place 1 mL of sample into a clean 15 mL centrifuge tube.

b Tear off 1 to 2 inches of lead acetate paper from the dispenser.

c Wet the test paper with deionized water.

d Add 1/2 to 1 mL of 1 Normal hydrochloric acid (HCl) to the test tube containing the solid sample ensuring the acid does not come in contact the tube sides.

e Insert the lead acetate paper into the tube and secure the paper by folding it over the edge of the test tube. Ensure that the paper does not stick to the inside of the test tube. The bottom of the paper should be approximately 1 inch above the acid/solid mixture.

f Place a cap loosely over the tube.

g Gently shake the centrifuge tube. Be careful to avoid direct contact of the acid mixture with the lead acetate paper.

- IF the lead acetate paper comes into contact with the solids or acid at any point during testing,

  THEN the results will be invalid. Discard the lead acetate paper AND return to Step e.

h Analyze Results

WARNING

To prevent health hazards, DO NOT put your nose directly over the centrifuge tube.

(1) Carbonates

- IF vigorous bubbling occurs when HCl is added to the sample,

  THEN the test is positive for the presence of carbonates.

- IF no bubbling occurs,

  THEN the test is negative and carbonates do not exist in the sample.

(2) Sulfides

- IF the lead acetate paper changes from white to a brownish or silver color after Step g,

  AND the result presents a rotten egg odor.

  THEN the test is positive for the presence of sulfides.

- IF there is no color change in the lead acetate paper,

  AND rotten egg odor is not present,

  THEN the test is negative and sulfides are not present in the sample.

Appendix D, Bacteria Testing
Page 1 of 9

WARNING

Personal injury may result from failure to exercise caution when using the extremely sharp syringes in this procedure.

NOTE

Test kit referenced in this section is one of two available kits.
Contact corrosion services for more information on bacteria test kits.

This appendix details the procedures used to perform bacteria inoculation and fixation. Bacteria inoculation and fixation must be performed for all liquid and solid samples collected as described below:

- IF the sample cannot be delivered to ATS with 24 hours, cannot be kept chilled, or both,

  THEN perform the field bacteria inoculation and fixation as described below and transfer the test kit to ATS along with the chain of custody and remaining liquid sample.

- IF the sample can be kept chilled and delivered to ATS with 24 hours,

  THEN ATS will perform the bacteria inoculation and fixation.

A. Sample Preparation

1. Liquid Samples

   - When performing inoculations for a liquid sample, pull the sample directly from the 60 mL sample container using a new syringe.
   - Ensure that the needle on the syringe does not contact the sample container.
   - IF the sample contains multiple layers,

     THEN insert the syringe into the aqueous layer.

2. Solid Samples

   a Use a sterile instrument to collect the solids.

   b Using the sterile instrument, collect approximately 1 mL amount (if present) of the solid.

c Remove the top from a tube or vial of nitrogen-purged Phosphate Buffer Solution (PBS) or Anaerobic Diluting Solution (ADS).

d Add the sample to the vial, AND replace the top.

e Shake the vial vigorously for 1 minute to homogenize as much material as possible into the solution and form a slurry. The slurry will be used to inoculate the media.

3. Scale, Biofilm or Swab Samples

a Remove the top from the tube or vial of nitrogen-purged Phosphate Buffer Solution (PBS) or Anaerobic Diluting Solution (ADS).

b IF the surface to be swabbed is dry,

THEN dip a sterile cotton-tipped swab in the vial to wet it.

c Swab a 1 inch square area on the surface to be sampled.

d Break off the tip of the swab in the vial. Do not touch any part of the applicator that will be broken off into the vial.

e Replace the top.

f Shake the vial vigorously for 1 minute to homogenize as much material as possible into the solution and form a slurry. The slurry will be used to inoculate the media.

B. Bacteria Inoculation

1. General

- Inoculate both Acid Producing Bacteria (APB) and Sulfate Reducing Bacteria (SRB) media vials.
- Vials containing red or reddish-orange (un-inoculated) media are used to test for APB.
- Vials containing a clear (un-inoculated) media are used to test for SRB.
- An additional bottle containing 2% formalin may also be inoculated to preserve the sample for further microscopic analysis and microbial enumeration.

1. Vial Sterilization

   a Using a permanent marker, label each bottle sequentially (i.e., 1, 2, 3, 4 and 5), for each type ("string") of media (APB and SRB).

   b IF the vials have a metal tab protecting the center of the rubber septum,

   THEN remove the tabs.

   c Sterilize the rubber stopper (septum) by wiping it with an alcohol pad as shown in Figure 16. Do not touch the vial tops after this step.

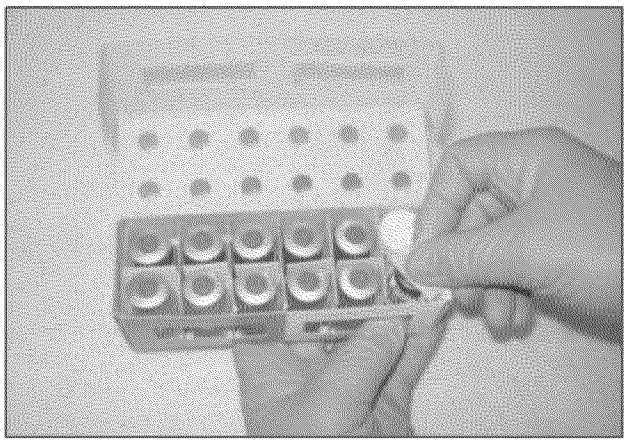

Figure 16. Sterilizing the Septum on the Top of the Vials

2. Inoculation Step

WARNING

Personal injury may result from touching needles.

CAUTION

Test results may be contaminated by touching needles,
or from loose or broken needles.

   a Remove a new syringe from its sterile packaging. Before removing the cap, ensure the needle is securely tightened to the body.

b Withdraw 2 mL of the sample.

c Hold the needle upright and gently tap the syringe body with your finger to work air bubbles toward the needle.

d Eliminate the air from the syringe by depressing the plunger while the syringe is in the upright position.

e Expel excess sample from the syringe into a waste container until 1 mL of sample remains in the syringe.

f Insert the needle through the center of the rubber septum and inject the 1 mL sample into the vial labeled #1 in the APB series as shown in Figure 17.

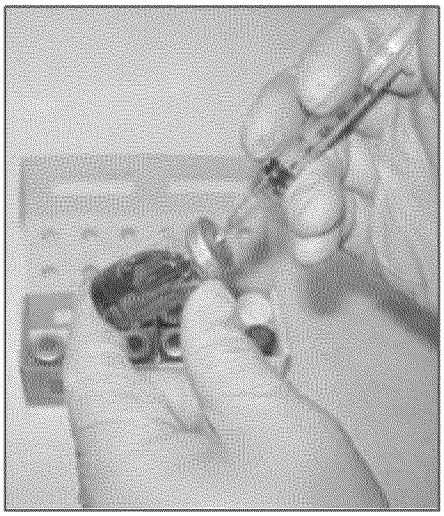

Figure 17. Inoculating Media Vial

g Dispose of the syringe in a sharps container without replacing the needle cover.

h Using a new syringe, repeat the inoculation Steps a. through g. for the vial labeled #1 of the SRB series.

3. Serial Dilution Step

a Begin the serial dilution with APB vial #1 and a new syringe.

b Withdraw between 2 and 3 mL of solution from the middle of the current vial, AND re-inject the withdrawn solution to mix the vial contents. Do not remove the syringe from the vial.

c Repeat Step (b) at least 3 times to thoroughly mix the solution.

d Using the syringe, withdraw 2-3 mL of solution from the middle of the vial while the vial is inverted. Work all gas bubbles in the syringe back into the vial and continue to expel fluid until the syringe contains 1 mL of solution that is free of bubbles.

e Withdraw the syringe from the current vial and inject the contents into the next vial in the series as shown in Figure 18. Do not remove the syringe from this vial.

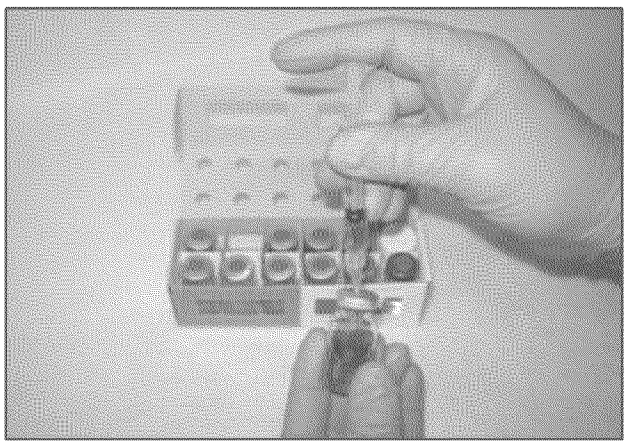

Figure 18. Inoculating the Next Vial in the Series

f Repeat Steps b through e using the same syringe until all vials in the series have been properly inoculated with 1 mL of mixture from the previous vial (vial #1 to vial #2, vial #2 to vial #3, vial #3 to vial #4, and vial #4 to vial #5). Dispose of the syringe in a sharps container without replacing the needle cover after inoculating vial #5.

g Immediately repeat Steps a. through f. with the SRB series. In Step a., APB vial #1 should be replaced by SRB vial #1.

C. Bacteria Fixation

Perform this step immediately after all bacteria inoculations and serial dilutions are complete, or, alternatively, after the first vial of both the APB and SRB series have been inoculated. This step also assumes that, for solid, scale, biofilm or swab samples the PBS vial has already been prepared.

WARNING

Personal injury may result from touching needles.

CAUTION

Test results may be contaminated by touching needles.

1. Remove a new syringe from its sterile packaging. Before removing the cap, ensure the needle is securely tightened to the body.
2. Withdraw 2 mL of the sample from the 60 mL sample container for liquid samples or from the PBS or ADS vial for all other sample types.
3. Hold the needle upright and gently tap the syringe body with your finger to work all air bubbles toward the needle.
4. Eliminate the air from the syringe by depressing the plunger while the syringe is in the upright position.
5. Expel excess sample from the syringe into a waste container until 1 mL of sample remains in the syringe.
6. Insert the needle through the center of the rubber septum and inject the 1 mL sample into the vial of 2% formalin solution.
7. Dispose of the syringe in a sharps container without replacing the needle cover.
8. Specify additional use of the sample in the PBS or ADS vial in a variance.

D. Results Interpretation

Follow these instructions when interpreting the results of bacteria inoculation vials. Shake vials prior to results interpretation.

1. Acid Producing Bacteria (APB) Vials

   The color change is an indication of the change in pH because the solution has become more acidic due to the organic acids produced by the microbes.

   APB vials contain a red or reddish-orange (un-inoculated) media. A positive indication for APB is confirmed when the media is cloudy or turbid and its color is yellow or orange-yellow.

   The turbidity of the media is an indication of the microbial growth in the media.

   Vials that are clear and yellow are not interpreted as "positive."

   Figure 19 shows positive and negative results for both APB and SRB.

   a APB False Indications

   - IF the #1 APB vial turns yellow immediately or within a few minutes after inoculation,

     THEN the sample has a low pH. This is a false indication. The indication of a change to yellow only results in a positive if subsequent vials show positive results after being incubated for 24 hours or more.

2. Sulfate Reducing Bacteria (SRB) Vials

   SRB vials contain a clear (un-inoculated) media. Sometimes grey or white solids are also present.

   A positive indication for SRB is confirmed when the medium in the vial turns black or black stringy material grows in the vial. The black solids that form in the media vials are produced when the SRB convert sulfate provided in the media to sulfide, which then reacts with iron in the media forming iron sulfide (FeS).

   Figure 19 shows positive and negative results for both APB and SRB.

a SRB False Indications

- IF the #1 SRB vial turns black immediately or within a few minutes after inoculation,

  THEN the sample contains an excessive amount of hydrogen sulfide (H_2S) or sulfide ions. This is a false indication. The indication of a change to black only results in a positive test after the sample has incubated for 24 hours or more. A false indication may also occur if the sample being inoculated is black or turbid. Injection of a black or turbid fluid will create a color change, introduce black solids which could be misinterpreted as a positive indication for SRB, or both. The color and consistency of the sample being injected should be noted.

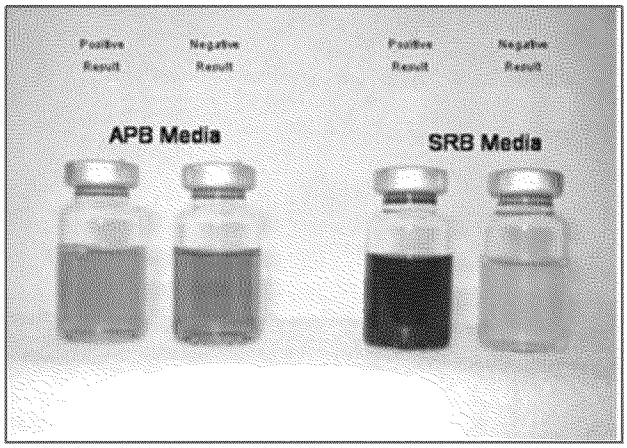

Figure 19. Positive and Negative APB and SRB Results

3. "Skipped" Vials

After incubation, a vial that has not changed color (indicating no growth) may be followed by one that has changed color, which indicates growth. For example, vial #3 may not have changed, while vials #1, #2, and #4 were positive for bacteria.

- Report only the number of positive vials and note which vials were skipped, as it is not possible to determine the reason for a "skipped" vial.

E. Reporting Bacteria Test Results

Table 1 provides the conversion between the number of vials turned positive, and the concentration of bacteria in the sample. Typically, five vials for each bacteria type are inoculated, although additional vials may be used when higher levels of bacteria are expected.

Table 1. Bacteria Results

Number of Positive Vials	Actual Dilution of Sample	Positive Vial Indicates (Bacteria per mL)	Reported Bacteria per mL of sample
1	1:10	1 to 9	10
2	1:100	10 to 99	100
3	1:1,000	100 to 999	1,000
4	1:10,000	1,000 to 9,999	10,000
5	1:100,000	10,000 to 99,999	100,000
6	1:1,000,000	100,000 to 999,999	1,000,000