WP 02-EM1005

Revision 5

Groundwater Serial Sample Analysis

Technical Procedure

EFFECTIVE DATE: 08/11/08

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APPROVED FOR USE

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INTRODUCTION [1]

This procedure provides the instructions necessary to perform a field analysis serial sample in support of the Groundwater Monitoring Program. Field analysis consists of the following:

- Eh (oxidation-reduction potential) (Redox) Measurement (Section 1.0)
- pH (hydrogen ion concentration) Measurement (Section 2.0)
- Temperature Measurement (Section 3.0)
- Specific Gravity Measurement (Section 4.0)
- Specific Conductance Measurement (Section 5.0)
- Alkalinity Analysis (Section 6.0)
- Chloride Analysis (Section 7.0)
- Chloride Spike Analysis (Section 8.0)
- Divalent Cation Concentration Analysis (Section 9.0)
- Divalent Cation Spike Analysis (Section 10.0)
- Total Iron Analysis (Section 11.0)

Serial samples will be performed at regular intervals in the mobile field laboratory for various physical and chemical parameters (called field indicator parameters). Serial sample data will be used to determine whether the sample is representative of undisturbed groundwater as a direct function of stabilization of field indicator parameters and the volume of the water being pumped from the well. As serial sample analyses are performed, analytical data will be entered into Excel spreadsheets which represent the sample Attachments 1 and 2, and 4 through 11 in this document; however, the Excel versions contain the mathematical formulas required to obtain the desired calculated values for parameters described herein.

Interpretation of serial sample data will enable the Team Leader (TL), or designee, to determine when conditions representative of undisturbed groundwater are attained in the pumped groundwater. A final sample will be collected and sent to a contract laboratory for analysis, in accordance with WP 02-EM1006, once the field indicator parameters have stabilized.

The following records are generated by the performance of this procedure:

- Attachment 1, Serial Sampling Report for Eh (Redox)
- Attachment 2, Serial Sampling Summary Sheet

- Attachment 3, Serial Sampling Check Printout Sheet
- Attachment 4, Serial Sampling Report for pH
- Attachment 5, Serial Sampling Report for Temperature
- Attachment 6, Serial Sampling Report for Specific Gravity
- Attachment 7, Serial Sampling Report for Specific Conductance
- Attachment 8, Serial Sampling Report for Alkalinity
- Attachment 9, Serial Sampling Report for Chlorides and Spike
- Attachment 10, Serial Sampling Report for Divalent Cations and Spike
- Attachment 11, Serial Sampling Report for Total Iron

REFERENCES

BASELINE DOCUMENTS

- Operation Manual, Hach Model DR 2000 Spectrophotometer
- Operation Manual, YSI Model 3200 Conductance Instrument
- Instruction Manual, Orion Model 720A pH Meter
- WIPP-DOE-215, Water Quality Sampling Plan
- WP 02-1, Groundwater Surveillance Program Plan
- WP 12-IH.01, WIPP Chemical Hygiene Plan

REFERENCED DOCUMENTS

- WP 02-EM1006, Final Sample and Serial Sample Collection
- WP 02-RC.01, Hazardous and Universal Waste Management Plan
- WP 10-AD3029, Calibration and Control of Monitoring and Data Collection Equipment

EQUIPMENT

TEST EQUIPMENT

- Hach DR 2000 spectrophotometer
- Orion 9678BN Platinum, Redox electrode
- Orion Model 720A pH/ISE meter
- Orion 9157BN pH electrode
- YSI Model 3200 conductance meter
- YSI Model 3252 conductivity cell, dip-type
- Hydrometer cylinder
- Hydrometer
- Digital thermometer
- Ag/AgCl reference electrode filling solution, Orion 900011

ADDITIONAL EQUIPMENT AND SUPPLIES

- Class A pipettes
- 50-mL (milliliters) Class A buret
- 50-mL beakers
- 250-mL beakers
- Coors beakers
- Tempering beaker
- 100-mL, 250-mL, 500-mL, and 1000-mL volumetric flasks
- Electrode holder
- Magnetic stir plate
- Teflon-coated magnetic stir bars
- Indelible pens
- Constant temperature bath

- Protective eyeglasses
- Laboratory coats
- Nitrile gloves, "powderless" or "powder-free"
- Industry standard 0.45 micron filters
- Laptop/printer/paper

REAGENTS

- Iron (Fe) Standard Reagent (1,000 µg/mL), Atomic Absorption Quality
- FerroVer Iron AccuVac Powder ampuls (4 each)
- Deionized (DI) water
- Hydroxylamine Hydrochloride Solution (7.5 g hydroxylamine hydrochloride dissolved in 100 mL deionized water and diluted with sufficient deionized water to make 250 mL of aqueous solution)
- Triethanolamine solution (30 mL triethanolamine diluted with 70 mL deionized water to make 100 mL of aqueous solution)
- Ammonium Hydroxide/Ammonium Chloride Buffer Solution (33.75 g ammonium chloride dissolved in 100 mL deionized water, with addition of 285 mL conc. [30%] ammonium hydroxide and diluted with sufficient deionized water to make 500 mL of aqueous solution)
- Calmagite Indicator Solution (0.5 g calmagite powder and 4.5 g hydroxylamine hydrochloride dissolved in 100 mL deionized water to make an aqueous solution)
- ZoBell Reference Solution (0.704 g potassium ferrocyanide, 0.549 g potassium ferricyanide and 3.728 g potassium chloride dissolved in 100 mL deionized water and diluted with sufficient deionized water to make 500 mL of aqueous solution)
- Lights' Reference Solution (19.605 g ferrous ammonium sulfate and 24.11 g ferric ammonium sulfate dissolved in 200 mL deionized water, with addition of 28.10 mL conc. sulfuric acid [H_2SO_4 Ultrex II, 89.4% by Wt., or equivalent] and diluted with sufficient deionized water to make 500 mL of aqueous solution)
- Sulfuric Acid (H_2SO_4) Solution, 0.02 N (Standardized, Commercially Prepared with Specific Value Certification, actual N value obtained from container = entry on Attachment 8)

- Silver Nitrate ($AgNO_3$) Solution, 0.0282 N (Standardized, Commercially Prepared with Specific Value Certification, 1 mL = 1 mg chloride (Cl), entry on Attachment 9 = 0.0282 N)

- EDTA Solution, 0.01 M (Standardized, Commercially Prepared with Specific Value Certification, 1 mL = 1 mg calcium carbonate ($CaCO_3$), entry on Attachment 10 = 0.01 M)

- pH Buffer Solution (NIST [National Institute of Science and Technology] -Traceable, pH 4.01, 7.00 and 10.01)

- 5% Potassium Chromate Solution (12.5 g potassium chromate dissolved in 100 mL deionized water and diluted with sufficient deionized water to make 250 mL of aqueous solution)

- YSI 3161 Conductivity Calibrator Solution (NIST-traceable, 1,000 microsiemens/cm [µS/cm])

- YSI 3163 Conductivity Calibrator Solution (NIST-traceable, 10,000 µS/cm)

- YSI 3165 Conductivity Calibrator Solution (NIST-traceable, 100,000 µS/cm)

PRECAUTIONS AND LIMITATIONS

- Only personnel with a current EM-14 Serial Sample Analysis qualification card, or trainees operating under the direct supervision of a qualified sampling staff, are authorized to perform the sample analysis activities specified in this procedure.

- Environmental Monitoring and Hydrology (EM&H) Manager shall be contacted If this procedure cannot be performed as written

- EM&H Manager shall be contacted if abnormal conditions are found during the performance of this procedure.

- Protective eye glasses and nitrile gloves are to be worn when performing this procedure (lab coat is optional). **WEAR LONG-SLEEVED SHIRTS AND FULL-LENGTH PANTS OR LAB COAT, AND CLOSED-TOP SHOES WHEN WORKING WITH CHEMICALS IN THE LABORATORY**.

- A spill control kit and eye wash station will be available and maintained appropriately (i.e., spill control kit to be kept stocked and eye wash station solution to be changed according to Industrial Safety & Hygiene (IS&H) specifications/frequency).

- All reagents listed above, especially those that contain toxic compounds, concentrated acids or concentrated bases, are to be handled with extreme

care. Ammonium Hydroxide/Ammonium Chloride Buffer Solution must be prepared and dispensed inside the fume hood.

- MSDS sheets for all chemicals used shall be available and shall be reviewed prior to performing this procedure.

- Instruments used for serial sampling analysis will be calibrated and maintained in accordance with WP 10-AD3029.

- Laboratory glassware must be washed after each use with a solution of "RCRA-free," phosphate-free detergent (e.g., Liquinox) and DI water, and triple-rinsed with DI water.

- The analytical procedures for Eh, pH, chloride, divalent cations, and iron produce liquid wastes which must be disposed of according to local, state and federal regulations; therefore, it is imperative that these wastes be properly segregated and stored in separate and suitable containers while awaiting disposal. An SAA (Satellite Accumulation Area) number designation of “26" has been assigned to the SAA in the mobile lab and all hazardous wastes generated by the analytical procedures described herein must be stored in this SAA until disposal can be arranged according to WP 02-RC.01.

- The frequency at which serial samples are collected and analyzed will be left to the discretion of the TL, but will be performed a minimum of three times during each sampling event.

- Stabilization for a well shall be determined by the Team Leader based on field work. The acceptance criteria for stabilization is when the majority of field indicator parameter measurements have stabilized within ± 5% of the average of analytical results for the field indicator parameter from the baseline groundwater quality for that well, or when a minimum of three well bore volumes have been purged from that well

- The Eh, pH, temperature, and specific gravity measurements shall be performed on an unfiltered sample collected from a dedicated stainless steel and Teflon sample line at atmospheric pressure.

- Specific conductance, alkalinity, chlorides, divalent cations, and total iron measurements shall be performed on a sample collected from a dedicated stainless steel and Teflon sample line at atmospheric pressure and filtered through an industry standard 0.45 micron membrane filter in a stainless steel filter holder.

- Check prints shall be completed with 45 working days of the last sample analysis for the associated well.

PREREQUISITE ACTIONS

- At least 90 days prior to commencement date of the next sampling round, gather M&DC equipment in mobile lab that needs to be sent out for calibration and repair (as required by WP 10-AD3029). Deliver equipment that needs to be calibrated/repaired to the measuring and testing equipment (M&TE) office in Building 459 (next to Tool Crib). M&TE will send the equipment out for calibration and/or repair, but a list of the items that were delivered to M&TE, along with Waste Isolation Pilot Plant (WIPP) equipment numbers, manufacturer's serial numbers (S/N), and the date delivered will need to be made by the EM&H representative.

- At least 90 days prior to commencement date of the next sampling round, check on-hand supply and condition of safety equipment/personal protective equipment (PPE) (safety glasses, nitrile gloves, etc.), chemical reagents and consumable sampling supplies ("certified clean" bottles, 0.45 µm filters, plastic pipettes, zip-lock bags, paper towels, Kim Wipes, etc.). Make a list of needed items, and request/reorder replacements at least 60 days prior to commencement date of the next sampling round.

- Check interior storage spaces of mobile lab to determine if required quantities of serial sampling and analysis equipment (including monitoring and data collection [M&DC] equipment) and supplies are present at least 30 days prior to commencement date of the next sampling round. Make a list of items needed for restocking.

- At least 10 days prior to commencement date of the next sampling round, notify IS&H representative that eyewash station in mobile lab needs to be serviced (solution needs to be changed and unit needs to be inspected and tested for proper operation).

- At least 10 days prior to commencement date of the next sampling round, pick-up M&DC equipment that was sent out for calibration and repair (as required by WP 10-AD3029) at the end of the last sampling round from the M&TE office in Building 459, and place in mobile lab.

- At least 5 days prior to commencement date of the next sampling round, restock any required items, including chemical reagents from Building 918B chemical storage cabinets; and sampling supplies (sample bottles, zip-lock bags, etc.), and equipment from the Porta Camp Connex.

- At least 3 days prior to commencement date of the next sampling round, check for required quantities of chemical reagents that need preparation (ZoBell and Lights' Reference Solutions, Hydroxylamine Hydrochloride Solution, Triethanolamine Solution, 5% Potassium Chromate Solution, etc.) and refer to REAGENTS section (above) of this document for mixing proportions. Prepare reagents as required to have on-hand, and make note of reagents that may need to be mixed in the near future.

- At least 3 days prior to commencement date of the next sampling round, prepare Excel templates for Attachments 1, 2, and 4 through 11.

- At least 3 days prior to commencement date of the next sampling round, print copies of Attachment 3.

DEFINITIONS

Serial Sample - A serial sample is a sample of groundwater taken at periodic intervals to establish stabilization of chemical and physical parameters prior to final sampling, based on results of past analytical results.

S/N - Serial Number designated by the equipment manufacturer.

Team Leader (TL) - Person responsible for assuring that the WIPP Groundwater Monitoring Program is being conducted in accordance with applicable plans and procedures.

PERFORMANCE

NOTE

The analysis of serial sample parameters may be performed in any sequence deemed appropriate by the technician performing the analysis; however, temperature, pH, alkalinity and Eh measurements should be performed as soon as possible after the samples collected for the determination of these parameters have been obtained.

1.0 Eh (REDOX) MEASUREMENT

NOTE

When taking data in this section, Eh and temperature are reported to nearest tenth. Time is reported in 24-hour format.

CAUTION

Used ZoBell and Lights' Reference Solutions must be segregated by placing in separate and suitable containers labeled "Hazardous Waste" and stored in the SAA located within the mobile lab until proper disposal can be arranged. Used Ag/AgCl Reference Electrode Filling Solution can be placed in the hazardous waste container designated for chloride waste because the chloride analysis procedure uses $AgNO_3$ titrant.

WARNING

ZoBell Reference Solution and Ag/AgCl Reference Electrode Filling Solution (Orion 900011) are toxic and Lights' Reference Solution is corrosive. Handle these solutions with care. Avoid spilling on exposed skin and clothing to prevent chemical burns or irritation. If spillage occurs on exposed skin and/or clothing, affected clothing should be removed, and skin area rinsed immediately with large quantities of water. If small amount is spilled on lab surfaces, they should be wiped and rinsed with water immediately. Spill control kits are to be used to neutralize and clean up large spills. Acid neutralizers are recommended for spills of Lights' Reference Solution.

1.1 Two-Point Check of Electrode System

1.1.1 Record the following on Attachment 1:

- Well
- Zone
- Round
- Serial Sample No.

- Meter S/N
- Thermometer S/N
- Thermometer Cal. Exp. Date
- ZoBell Lot No. (date prepared)
- Lights' Lot No. (date prepared)

1.1.2 Verify ZoBell and Lights' reference solutions have been stored in a temperature-controlled water bath set at 25 (24.5 to 25.5)°C.

NOTE

ZoBell and Lights' Reference Solutions must not be subjected to extreme heat. These solutions will last up to 90 days if stored at 4°C (39°F).

1.1.3 Flush inside filling solution chamber of Eh electrode with Ag/AgCl Reference Electrode Filling Solution (Orion 900011).

1.1.4 Fill Eh electrode solution chamber with Orion 900011 filling solution.

1.1.5 Rinse Eh electrode and thermometer with DI water.

1.1.6 Position Eh electrode and thermometer in ZoBell solution.

1.1.7 Allow electrode and thermometer to equilibrate.

1.1.8 Generate a printout from Eh meter that records Eh of ZoBell 1 Solution.

1.1.9 Record the following Equipment Check ZoBell 1 values on Excel version of Attachment 1:

- Time
- Temp (°C)
- Meter Value (mV)

1.1.10 Rinse Eh electrode and thermometer with DI water.

1.1.11 Position electrode and thermometer in Lights' solution.

1.1.12 Allow electrode and thermometer to equilibrate.

1.1.13 Generate a printout from Eh meter that records Eh of Lights' Solution.

1.1.14 Record the following Equipment Check Lights' values on Excel version of Attachment 1:

- Time
- Temp (°C)
- Meter Value (mV)

1.1.15 Rinse Eh electrode and thermometer with DI water.

1.2 Measurement of Serial Sample Eh

1.2.1 **GO TO** WP 02-EM1006 and collect a serial sample, and **RETURN TO** Step 1.2.2.

1.2.2 Record Measurement Time of Collection on Excel version of Attachment 1.

NOTE

The 250-mL plastic beaker will have holes cut in lid to accommodate electrode and thermometer.

1.2.3 Position electrode and thermometer in sample.

1.2.4 Generate a printout from Eh meter that records Eh of sample when electrode stabilization has occurred.

1.2.5 Record the following measurement values on Excel version of Attachment 1:

- Time of Measurement
- Temp (°C)
- Meter Value (mV)

1.2.6 Perform a post-measurement equipment check as follows:

NOTE

If gas bubbles form on electrode's platinum surface, they can be removed by gently tapping electrode.

[A] Rinse Eh electrode and thermometer with DI water.

[B] Recheck ZoBell reference solution.

1.2.7 **IF** Meter Value reading is not within 10 mV of ZoBell 1 value obtained in Step 1.1.9,
THEN repeat Steps 1.1.2 through 1.2.7.

1.2.8 Generate a printout from Eh meter that records Eh of ZoBell 2 Solution.

1.2.9 Record the following Equipment Check ZoBell 2 values on Excel version of Attachment 1:

- Time
- Temp (°C)
- Meter Value (mV)

1.2.10 Rinse Eh electrode and thermometer with DI water.

1.2.11 Empty filling solution and rinse inside of electrode with DI water.

1.2.12 Store electrode dry.

NOTE

The calculated values of Eh and expected Eh relative to Standard Hydrogen Electrode (SHE) should be within 10 mV of each other.

NOTE

Excel will calculate and record the following on Attachment 1:

- ZoBell 1 - Expected Temp Value Rel to SHE
- ZoBell 1 - Eh Relative to SHE (mV)
- Lights' - Expected Temp Value Rel to SHE
- Lights' - Eh Relative to SHE (mV)
- ZoBell 2 - Expected Temp Value Rel to SHE
- ZoBell 2 - Eh Relative to SHE (mV)
- Measurement (sample) Eh Relative to SHE (mV)

1.2.13 **IF** the difference between the calculated values of Eh relative to SHE and expected temperature values of Eh relative to SHE for ZoBell and Lights' solutions are ≤ 10 mV,
THEN GO TO Step 1.2.16.

1.2.14 **IF** the difference between the calculated values of Eh relative to SHE and expected values of Eh relative to SHE for ZoBell and Lights' solutions are >10 mV,
THEN perform the following:

[A] Change reference solutions.

[B] Change electrode filling solution.

[C] Polish tip of electrode.

[D] Repeat Steps 1.2.1 through 1.2.13.

1.2.15 **IF** the difference between the calculated values of Eh relative to SHE and expected values of Eh relative to SHE for ZoBell and Lights' solutions are still >10 mV,
THEN perform the following:

[A] Replace electrode.

[B] Repeat Steps 1.2.1 through 1.2.13.

1.2.16 Complete the Prepared By and Date on Excel version of Attachment 1.

NOTE

Excel spreadsheet will transfer the following to Attachment 2:

- Well
- Zone
- Round
- Serial Sample No.
- Eh Test Results
- Eh Analyst
- Eh Date/Time Tested

1.2.17 Complete the following on Attachment 3:

- Well
- Zone
- Round
- Sample

1.2.18 Glue and tape printout produced by Orion 720A pH/ISE Ion meter to Attachment 3. All related Eh calculations will be performed by embedded formulas in the Excel spreadsheet version of Attachment 1.

NOTE

Best results in Step 1.2.19 will be obtained if copies of Attachment 3 with printout produced by the Orion 720A meter are made as soon as possible following the serial sampling events because the thermally printed data generated by this device will gradually fade and become unreadable. The copies can be stamped "BEST AVAILABLE COPY" and the sheets with glued and taped printouts can be stamped "ORIGINAL."

1.2.19 After gluing the printout produced by Orion 720A meter to Attachment 3, position the tape on the edges of the printout paper because if the tape (including transparent tape) is placed over the printed information it will become unreadable.

1.2.20 Complete the Prepared By and Date on Attachment 3.

2.0 pH MEASUREMENT

NOTE

When taking data in this section, buffer temperature should be recorded to nearest tenth of a degree. Time should be recorded in 24-hour format. pH should be recorded to nearest hundredth of a Standard Unit (SU).

2.1 Two-Point Performance Check of pH/mV Meter and pH Electrode

NOTE

Two-point performance check is performed prior to taking the pH of a serial sample. The purpose of the check is to ensure electrode slope is 92-105%. An acceptable two-point performance check, and subsequent measurements, will only be obtained if the probe is continuously maintained in the solution being tested during the measurement period.

CAUTION

Buffer solutions should be stored below 30°C (86°F) to minimize the likelihood of error due to evaporation, to thermal degradation, or to microbial growth. **SOLUTIONS MUST NOT BE FROZEN**. They must be discarded if expiration date is past, or if color, turbidity, or visible microbial growth develops. Used solution must not be poured back into new solution container.

2.1.1 On the first day of serial sampling, and prior to performing the two-point performance check, change the Ag/AgCl reference electrode filling solution in the Orion 9157BN pH electrode, and check/reset the date and time function in the Orion 720A meter.

WARNING

Ag/AgCl Reference Electrode Filling Solution is toxic and must be handled with care to avoid injury. Used Ag/AgCl Reference Electrode Filling Solution must be segregated and stored in the SAA located within the mobile lab until proper disposal can be arranged. Used Ag/AgCl Reference Electrode Filling Solution can be placed in the hazardous waste container designated for Chloride Waste since the chloride analysis procedure uses $AgNO_3$.

Although buffer solutions are "not hazardous" by regulatory definition, they may be harmful to humans and animals; therefore, the used or "expired" solutions should be disposed of properly (e.g., as "nonhazardous" waste, or mixed with wash and rinse water waste and stored separately for proper disposal).

2.1.2 Record the following on Excel version of Attachment 4:

- Well
- Zone
- Round
- Serial Sample No.
- pH meter S/N
- pH Buffers Lot No. and Exp. Dates

NOTE

The first buffer should be at the isopotential point (pH 7.00) and the second buffer at pH 4.01 or pH 10.01. A separate digital thermometer is not required for pH measurements because the Orion 720A meter has Automatic Temperature Correction (ATC) capability when used with the Orion 9157BN electrode which has a "built-in" temperature probe.

2.1.3 Select two buffers that bracket expected sample pH.

2.1.4 Rinse electrode with DI water.

2.1.5 Place electrode in pH 7.00 buffer solution and stir on mixing plate.

2.1.6 Press "1st."

2.1.7 Press "Calibrate."

2.1.8 Enter "2" for number of buffers.

2.1.9 Press "Yes."

NOTE

In Steps 2.1.10 and 2.1.14, the ___.___ will be a number, such as 7.03 or 10.05, but it will never, or rarely, be the same.

2.1.10 Wait for "RDY CAL AS ___.___" to display.

2.1.11 Press "Yes."

2.1.12 Rinse electrode with DI water.

2.1.13 Place electrode in second buffer solution and stir on mixing plate.

2.1.14 Wait for "RDY CAL AS ___.___" to display.

2.1.15 Press "Yes."

2.1.16 **IF** slope reading is 92-105%,
THEN GO TO Step 2.1.19.

2.1.17 **IF** slope reading is < 92% or >105%,
THEN perform the following:

[A] Change buffer solutions.

[B] Change reference electrode solution.

[C] Repeat Steps 2.1.4 through 2.1.16.

2.1.18 **IF** slope reading is still <92% or >105%,
THEN perform the following:

[A] Replace reference electrode.

[B] Repeat Steps 2.1.4 through 2.1.16.

2.1.19 Record the following Performance Check values on Excel version of Attachment 4:

- pH 7.00 and pH 4.01 (or 10.01) Time
- pH 7.00 and pH 4.01 (or 10.01) Temp (°C)
- pH 7.00 and pH 4.01 (or 10.01) Meter Value
- Electrode Slope

2.2 Measurement of Serial Sample pH

2.2.1 **GO TO** WP 02-EM1006 and collect a serial sample, and **RETURN TO** Step 2.2.2.

2.2.2 Record sample Time of Collection on Excel version of Attachment 4.

2.2.3 Place stirring bar in sample and stir on stirring plate.

2.2.4 Position H electrode in beaker.

2.2.5 When "RDY" displays, press "Print."

2.2.6 Record the following measurement values on Excel version of Attachment 4.

- Time of Measurement
- Temp (°C)
- Meter Value

2.3 pH Buffer Recheck

2.3.1 **IF** pH meter will no longer be used for the remainder of the day, **THEN** perform pH Buffer Recheck as follows:

[A] Rinse electrode thermometer with DI water.

[B] Place electrode in pH 7.00 buffer.

[C] When "RDY" displays press "Print."

[D] **IF** pH of buffer solution is not within ± 0.1 log unit pH 7.00,
THEN GO TO Subsections 2.1 and 2.2, and
RETURN TO Step 2.3.1[E].

[E] Record the following on Excel version of Attachment 4:

- pH (7.00) Time
- pH (7.00) Temp (°C)
- pH (7.00) Meter Value

[F] Rinse electrode with DI water.

[G] Store electrode vertically in 4 M KCl storage solution.

[H] Complete the Prepared By and Date on Excel version of Attachment 4.

NOTE
Excel spreadsheet will transfer pH test results to Attachment 2.

[I] Glue and tape printout produced by Orion 720A pH/ISE Ion meter to Attachment 3.

NOTE
Best results will be obtained if copies of Attachment 3 with printout produced by the Orion 720A meter are made as soon as possible following the serial sampling events because the thermally printed data generated by this device will gradually fade and become unreadable. The copies can be stamped "BEST AVAILABLE COPY" and the sheets with glued and taped printouts can be stamped "ORIGINAL."

2.3.2 After gluing the printout produced by the Orion 720A meter to Attachment 3, position the tape on the edges of the printout paper because if the tape (including transparent tape) is placed over the printed information it will become unreadable.

3.0 TEMPERATURE MEASUREMENT

3.1 Record the following on Excel version of Attachment 5:

- Well
- Zone
- Round
- Serial Sample No.
- Thermometer S/N
- Thermometer Cal. Exp. Date

3.2 Initiate flow of sample water into tempering beaker allowing it to overflow.

3.3 Insert thermometer into tempering beaker.

3.4 **WHEN** water has overflowed for 2-3 minutes,
THEN observe temperature to nearest 0.1°C.

3.5 Record the following on Excel version of Attachment 5:

- Time of Sample Collection
- Time of Temperature Measurement
- Temperature (°C)

3.6 Stop flow of sample water through tempering beaker.

3.7 Drain tempering beaker.

3.8 Rinse tempering beaker and thermometer with DI water.

3.9 Complete the Prepared By and Date on Excel version of Attachment 5.

NOTE

Excel spreadsheet will transfer test results to Attachment 2.

4.0 SPECIFIC GRAVITY MEASUREMENT

4.1 Record the following on Excel version of Attachment 6:

- Well
- Zone
- Round
- Serial Sample No.
- Thermometer S/N
- Thermometer Cal. Exp. Date
- Hydrometer S/N
- Hydrometer Cal. Exp. Date

4.2 **GO TO** WP 02-EM1006 and collect a serial sample, and **RETURN TO** Step 4.3.

4.3 Fill hydrometer cylinder with unfiltered sample water.

NOTE

Degassing time will vary from well to well, but will take approximately 15 to 30 minutes. High carbonate water may require 30 to 60 minutes. Degassing has been accomplished when visible air bubbles inside of the cylinder containing the sample water have disappeared. This process can be accelerated by gently tapping on the outside of the cylinder with a plastic rod to help dislodge and float the bubbles to the top for faster removal.

4.4 Allow water sample to degas, using the guidelines stated above.

4.5 Record degassing time on Excel version of Attachment 6.

NOTE

Hydrometer float must not be allowed to contact the walls of the hydrometer cylinder during reading.

4.6 Place hydrometer in unfiltered sample water and read water line on stem. Temperature measurement of sample water can be performed right before or right after the hydrometer reading.

NOTE

Field density evaluations will be expressed in terms of specific gravity, which is a unitless measure. Specific gravity will be recorded to three decimal places and temperature to the nearest tenth of a degree Celsius.

4.7 Record the following on Excel version of Attachment 6:

- Time of Sample Collection
- Time of Measurement
- Observed Hydrometer Reading
- Temperature (°C)
- Degassing Time

4.8 Rinse thermometer, hydrometer, and hydrometer cylinder with DI water.

4.9 Complete the Prepared By and Date on Excel version of Attachment 6. Excel spreadsheet will transfer test results to Attachment 2.

5.0 SPECIFIC CONDUCTANCE MEASUREMENT

5.1 Two-Point Performance Check of Cell-Meter System with Automatic Temperature Correction (ATC) Capability

NOTE

Temperature has a large effect on conductivity. Calibration of the YSI 3200 meter should be performed as close as practical to 25°C by maintaining the calibration solutions in a water bath to control temperature. In addition, calibration solutions should be stored below 30°C to minimize the likelihood of error due to evaporation or to microbial growth. **SOLUTION MUST NOT BE FROZEN**.

Solution should be discarded if expiration date is past, or if color, turbidity or visible microbial growth develops. Used solution must not be poured back into new solution container.

WARNING

Although calibration solutions are "not hazardous" by regulatory definition, they may be harmful to humans and animals; therefore, the used or "expired" solutions should be disposed of properly (e.g., as nonhazardous waste, or mixed with wash and rinse water waste and stored separately for proper disposal).

5.1.1 Record the following on Excel version of Attachment 7:

- Well
- Zone
- Round
- Serial Sample No.
- Meter Model No.
- Meter S/N
- Probe Model and I.D. No.

NOTE

If expected specific conductance is greater than the highest value of available reference standards (pre-prepared standard solutions with known conductivity values in "µmhos/cm" or "µS/cm"), then the two highest standards will be used. Because 1 µmhos/cm = 1 µS/cm (equivalent units), either unit may be designated on the standard solutions.

Reference standards should be equilibrated in a constant temperature water bath at or near 25°C. The YSI 3252 conductivity cell has a "built-in" temperature probe; therefore, a digital thermometer is not required.

5.1.2 Select two reference standards that will bracket sample specific conductance, if possible.

5.1.3 Plug the power supply into the 12.0 V DC connector on the back of the YSI 3200 unit.

5.1.4 Press the power key briefly to turn on power supply.

NOTE

After momentarily displaying the "YSI" logo, the screen should display the "OPERATION" mode data with standard conductivity units of "µS/cm" to the right of the measured value in the upper left part of the screen indicating that the YSI 3200 is in the "conductivity measurement mode."

5.1.5 Rinse conductivity cell with DI water and immerse the cell in Reference Standard 1. Gently tap the cell to remove air bubbles and dip the cell up and down in the solution 2 or 3 times to ensure proper wetting.

NOTE

To avoid measurement error, the electrodes must be completely submerged and the electrode chamber must not contain trapped air.

5.1.6 Position conductivity cell in Reference Standard 1 so that the cell is not touching the sides or bottom of the container.

5.1.7 Allow cell to equilibrate with standard.

5.1.8 Check that the correct date and time are set by pressing the SETUP key, then the TIME/DATE key to enter the Time/Date menu. The cursor will be positioned on the first line in the bracket containing the time. Check that the "[24Hr]" time format is displayed on the third line, and that the "[MM/DD/YY]" date format is displayed on the fourth line.

5.1.9 If the [24Hr] time format is not displayed, press the NEXT key twice to position the cursor in the time format bracket, press the UP key to toggle to [24Hr].

5.1.10 If the [MM/DD/YY] date format is not displayed, press the NEXT key to position the cursor in the date format bracket, press the UP key to toggle to [MM/DD/YY]).

5.1.11 After checking and setting the time and date format fields, press the NEXT key to position the cursor in the time bracket and the DIGIT key to move the cursor to the right while using the UP and DOWN keys to set the time. Use the same technique to set the date. After the correct time and date have been entered, press the MODE key twice to save and exit to the OPERATION mode.

5.1.12 Record the following on Excel version of Attachment 7:

- Standards (Standard Solutions in µS/cm)
- Lot Nos.
- Known conductivity of Standard 1 (1K or 10K µS/cm)
- Standard 1 Time displayed on screen
- Standard 1 Temperature (°C) displayed on screen

NOTE

Cell calibration should be performed once daily at the beginning of each day of sampling.

5.1.13 In the OPERATION mode, press the CELL key once, then the CAL K key to enter the "CALIBRATE K" menu. Press the MULTI PT key to begin and the cursor will be positioned on the first line in the bracket containing the Standard 1 Value. Use the UP/DOWN and DIGIT keys to enter the value of Standard 1 at the temperature measured by the cell probe and displayed on line 2 of the screen.

NOTE

Temperature compensation is disabled during cell calibration; therefore, the standards should be maintained at or near 25°C in the constant temperature water bath for an accurate calibration.

5.1.14 Press ENTER to store the first cell constant. Stored values are numbered 0-4 (up to 5 standard solutions may be entered; however, only 2 are used for this procedure).

5.1.15 Rinse conductivity cell with DI water and immerse the cell in Reference Standard 2.

5.1.16 Gently tap the cell to remove air bubbles and dip the cell up and down in the solution 2 or 3 times. The electrodes must be submerged and cell must be positioned in reference Standard 2 so that the cell is not touching the sides or bottom of the container.

5.1.17 Allow cell to equilibrate with standard.

5.1.18 While allowing the cell to equilibrate, record the following on Excel version of Attachment 7:

- Known conductivity of Standard 2 (10K or 100K µS/cm)
- Standard 2 Time
- Standard 2 Temperature (°C)

5.1.19 Use the UP/DOWN and DIGIT keys to enter the value of Standard 2 at the temperature measured by the cell probe and displayed on line 2 of the screen. Press ENTER to store the second cell constant.

5.1.20 After entering the second cell constant, rinse conductivity cell with DI water, immerse, gently tap, dip 2-3 times and position conductivity cell in DI water sample with electrodes submerged. Enter 0.00 µS/cm for each remaining (unused) point, and press ENTER to store each.

5.1.21 Press MODE twice to exit to the "CELL" menu.

5.1.22 Press the CONFIGURE key to enter the "CONFIG" menu, press NEXT twice to move the cursor to the Cell Cal field.

5.1.23 Press UP key to toggle to [Multi], then press MODE twice to exit to the OPERATION screen. The conductivity will be a low value in DI water (x.xxx, xx.xx, or xxx.x).

5.1.24 Immerse/gently tap/dip 2-3 times and position conductivity cell in reference Standard 1 so that the cell is submerged and not touching the sides or bottom of the container, and allow cell to equilibrate with standard.

5.1.25 Read Standard 1 conductivity (G25) and temperature. If Standard 1 is 10K µS/cm, the conductivity may be displayed as xx.xx mS/cm, so it is converted to µS/cm by moving the decimal 3 places to the right (example: 10.71 mS/cm = 10,710 µS/cm). Record Standard 1 G25 measurement on Attachment 7 in µS/cm.

5.1.26 **IF** standard conductivity at 25°C is not within ± 10% of expected standard value,
THEN perform the following:

[A] Replace reference Standard 1 with fresh solution.

[B] Inspect meter and conductivity cell for defects or damage.

[C] Make necessary changes if meter or conductivity cell is defective, such as using backup equipment, so that a reliable conductivity reading can be obtained.

5.1.27 Rinse conductivity cell with DI water, immerse/gently tap/dip 2-3 times and position conductivity cell in reference Standard 2 so that the cell is not touching the sides or bottom of the container, and allow cell to equilibrate with standard.

5.1.28 Read Standard 2 conductivity (G25) and temperature. If Standard 2 is 100K µS/cm, the conductivity may be displayed as xx.xx mS/cm or xxx.x mS/cm, so it is converted to µS/cm by moving the decimal 3 places to the right (example: 103.6 mS/cm = 103,600 µS/cm). Record Standard 2 G25 on Attachment 7 in µS/cm.

5.1.29 **IF** standard conductivity at 25°C. is not within ± 10% of expected standard value,
THEN perform the following:

[A] Replace reference Standard 2 with fresh solution.

[B] Inspect meter and conductivity cell for defects or damage.

[C] Make necessary changes if meter or conductivity cell is defective, such as using backup equipment, so that a reliable conductivity reading can be obtained.

5.2 Measurement of Serial Sample Specific Conductance

5.2.1 **GO TO** WP 02-EM1006 and collect a filtered serial sample, and **RETURN TO** Step 5.2.2.

5.2.2 Record Time of Collection on Excel version of Attachment 7.

5.2.3 Rinse conductivity cell with DI water.

NOTE

The serial sample does not have to be at or near 25°C when measurement is performed because the meter and cell system with the built-in temperature probe will compensate automatically.

5.2.4 Immerse/gently tap/dip 2-3 times and position conductivity cell in sample so that the cell is not touching the sides or bottom of the container.

5.2.5 Allow cell to equilibrate with sample.

5.2.6 Read sample conductance and temperature and convert the value from mS/cm to µS/cm, as in Steps 5.1.25 or 5.1.28, depending upon the magnitude (1K, 10K or 100K range) of the measurement value.

5.2.7 Record the following measurement values on Excel version of Attachment 7:

- Time
- Temperature (°C)
- Meter Value (µS/cm) = G25

5.2.8 Rinse conductivity cell with DI water and position in Standard 1, as in Step 5.1.24.

5.2.9 Recheck Standard 1 conductivity (G25) and temperature to verify that the value is within ± 10% of the value obtained in Step 5.1.25.

5.2.10 **IF** value is not within ± 10% of initial value,
THEN perform the following:

[A] Replace reference Standard 1 with fresh solution.

[B] Inspect meter and conductivity cell for defects or damage.

[C] Make necessary changes if meter or conductivity cell is defective, such as using backup equipment, so that a reliable conductivity reading can be obtained.

5.2.11 Record the following Equipment Check values on Excel version of Attachment 7.

- Conductivity of Standard 1 (rechecked)
- Time
- Temperature (°C)

5.2.12 Complete the Prepared By and Date on Excel version of Attachment 7. Excel spreadsheet will transfer test results to Attachment 2.

6.0 ALKALINITY ANALYSIS

NOTE

When taking data in this section, time should be recorded in 24-hour format; pH, and titrant volume shall be recorded to the nearest hundredth of a unit. Measurement of alkalinity should be performed as soon as possible after sample collection.

WARNING

Sulfuric Acid (H_2SO_4) Solution, 0.02 N is corrosive and must be handled with care to avoid injury. Spilling on exposed skin and clothing should be avoided to prevent chemical burns or irritation. If spillage occurs on exposed skin and/or clothing, affected clothing should be removed, and skin area rinsed immediately with large quantities of water. If a small amount is spilled on lab surfaces, they should be wiped and rinsed with water immediately. Spill control kits are to be used to neutralize and clean up large spills. Acid neutralizers are recommended for spills of sulfuric acid solution.

Expired sulfuric acid solution must be segregated and stored in the SAA located within the mobile lab until proper disposal can be arranged. Although the waste solutions produced during alkalinity titration are not hazardous by regulatory definition, they may be harmful to humans and animals; therefore, the waste solutions should be disposed of properly (e.g., as nonhazardous waste, or mixed with wash and rinse water waste and stored separately for proper disposal).

6.1 **IF** Two-Point Performance Check of pH Meter and electrode has not been performed,
THEN GO TO Subsection 2.1 and perform check, and
RETURN TO Step 6.2.

6.2 Record the following on Excel version of Attachment 8:

- Well
- Zone
- Round
- Serial Sample No.

6.3 **GO TO** WP 02-EM1006 and collect a filtered serial sample, and
RETURN TO Step 6.4.

6.4 Pipet 100 mL of sample solution into a 250-mL beaker.

6.5 Carefully position pH electrode in solution.

6.6 Turn meter on and record sample time on Excel version of Attachment 8.

6.7 Stir solution slowly with Teflon stir bar.

6.8 When meter stabilizes, record Initial pH on Excel version of Attachment 8; if initial pH ≤8.1, skip to Step 6.10; if initial pH >8.1, then proceed to Step 6.9.

6.9 Perform phenolphthalein alkalinity titration as follows:

6.9.1 Record buret Initial Volume (mL) on Excel version of Attachment 8.

NOTE
Following each addition, solution is allowed to mix thoroughly.

6.9.2 Titrate solution with standard sulfuric acid solution (0.02 N) in increments of ≤ 0.5 until pH of 8.1 is reached.

NOTE
Net Titrant Used should be reported to nearest hundredth of a mL.

NOTE
Excel will calculate and record the Net Titrant Used in Attachment 8.

6.9.3 Record the following on Excel version of Attachment 8:

- Titrant N
- Titrant Lot #
- Buret Final Volume

6.10 Perform Total Alkalinity titration as follows:

6.10.1 Record Buret Initial Volume (mL) on Excel version of Attachment 8.

6.10.2 If initial pH ≤8.1, as determined in Step 6.8, titrate solution until a pH of 4.5 is reached. If initial pH >8.1, as determined in Step 6.8, continue titrating solution until a pH of 4.5 is reached.

NOTE

Net Titrant Used should be reported to nearest hundredth of a mL. Excel will calculate and record the Net Titrant Used on Attachment 8

6.10.3 Record the Buret Final Volume on Excel version of Attachment 8.

6.11 Pipet 100 mL of duplicate solution into a 250-mL beaker.

6.12 Carefully position pH electrode in solution.

6.13 Turn meter on and record duplicate sample time on Excel version of Attachment 8.

6.14 Stir solution slowly with Teflon stir bar.

6.15 When meter stabilizes, record Initial pH on Excel version of Attachment 8.

6.16 Perform phenolphthalein Alkalinity titration as follows:

6.16.1 When meter stabilizes, record "Initial pH" on Excel version of Attachment 8; if initial pH $\leq$8.1, skip to Step 6.17; if initial pH >8.1, proceed to Step 6.16.2.

NOTE

Following each addition, solution must be allowed to mix thoroughly.

6.16.2 Titrate solution with standard sulfuric acid solution (0.02 N) in increments of $\leq$ 0.5 until pH of 8.1 is reached.

NOTE

Net Titrant Used should be reported to nearest hundredth of a mL. Excel will calculate and record the Net Titrant Used on Attachment 8.

6.16.3 Record the Buret Final Volume on Excel version of Attachment 8.

6.17 Perform Total Alkalinity titration as follows:

6.17.1 Record Buret Initial Volume (mL) on Excel version of Attachment 8.

6.17.2 If initial pH $\leq$8.1, as determined in Step 6.16.1, titrate solution until a pH of 4.5 is reached; if initial pH >8.1, as determined in Step 6.16.1, continue titrating solution until a pH of 4.5 is reached.

NOTE

Net Titrant Used should be reported to nearest hundredth of a mL. Excel will calculate and record Net Titrant Used on Attachment 8.

6.17.3 Record Buret Final Volume on Excel version of Attachment 8.

6.18 If initial pH $\leq$8.1, calculate only bicarbonate alkalinity using only the equation below for sample and duplicate concentration of Bicarbonate (mg/L HCO_3). Excel will calculate sample and duplicate concentrations of carbonate, bicarbonate, and hydroxide (mg/L HCO_3, mg/L CO_3 and mg/L OH), as applicable based upon the value of "P" on the table below, using the following equations:

Equations for concentration of carbonate, bicarbonate, and hydroxide

- Bicarbonate

$$mg/L\ HCO_3 = \frac{ml\ acid * N\ acid * 61 * 1000}{mL\ sample}$$

- Carbonate

$$mg/L\ CO_3 = \frac{ml\ acid * N\ acid * 30 * 1000}{mL\ sample}$$

- Hydroxide

$$mg/L\ OH = \frac{ml\ acid * N\ acid * 17 * 1000}{ml\ sample}$$

Table with relationships to determine volume of acid in above equations

	Volume of Standard Acid Corresponding to:		
Results	Bicarbonate	Carbonate	Hydroxide
P is zero	T	0	0
P < 1/2T	T-2P	2P	0
P = 1/2T	0	2P	0
P > 1/2T	0	2 (T-P)	2P-T
P = T	0	0	T
where: T = total titration P = titration to pH 8.1 in mL			

6.19 Verify that the following has been recorded by Excel on Attachment 8, as applicable based upon the value of "P" on the Table above.

- Sample Alkalinity as Carbonate (mg/L)
- Sample Alkalinity as Bicarbonate (mg/L)
- Sample Alkalinity as Hydroxides (mg/L)
- Duplicate Alkalinity as Carbonate (mg/L)
- Duplicate Alkalinity as Bicarbonate (mg/L)
- Duplicate Alkalinity as Hydroxides (mg/L)

6.20 Complete the Prepared By and Date on Excel version of Attachment 8.

6.21 Verify that Excel spreadsheet has transferred test results to Attachment 2.

6.22 **IF** this is the last measurement taken for the day using the pH meter, **THEN GO TO** Subsection 2.3 and perform a pH buffer recheck of pH/mV meter and pH electrode.

7.0 CHLORIDE ANALYSIS

NOTE

A method blank will be carried through the entire process, along with sample and duplicate sample.

WARNING

Silver nitrate ($AgNO_3$) solution, 0.0282 N, and 5% potassium chromate solution are toxic and must be handled with care to avoid spilling, as irritation and staining may occur on exposed skin and clothing. If spillage occurs on exposed skin and/or clothing, clothing should be removed, and skin area rinsed immediately with large quantities of water. If small amount is spilled on laboratory surfaces, surface should be rinsed and wiped with water immediately. Spill control kits are to be used to clean up large spills.

Expired silver nitrate solution must be segregated and stored in the SAA located within the mobile laboratory until proper disposal can be arranged. Waste solutions produced during chloride titration must be segregated by placing in a suitable container labeled "Hazardous Waste" and stored in the SAA located within the mobile lab until proper disposal can be arranged.

7.1 Record the following on Excel version of Attachment 9:

- Well
- Zone
- Round
- Serial Sample No.

7.2 **GO TO** WP 02-EM1006 and collect a filtered serial sample, and **RETURN TO** Step 7.3.

NOTE

If normality of $AgNO_3$ titrant is not 0.05N, then dilution must be modified from dilution table proportionately.

7.3 Refer to specific gravity of current serial sample on Attachment 2.

7.4 Using the specific gravity of current serial sample on Attachment 2, dilute sample to concentration shown on Attachment 12, Dilution Tables, or modify proportionately as specified in the **NOTE** above (the modified dilutions are posted on a separate dilution table in the mobile lab for the corresponding normality [N] of the $AgNO_3$ titrant being used during the current sampling Round), and use diluted sample to prepare the sample, duplicate sample and spike sample, as described below.

7.5 Record Sample and Duplicate Dilution Factor (Flask/Pipet) on Excel version of Attachment 9.

7.6 Record Sample and Duplicate Aliquot of Sample (mL) on Excel version of Attachment 9.

7.7 Buret aliquot (10-50 mL depending on the salinity of the well water; refer to Dilution Tables described in Step 7.4) of diluted sample into a Coors beaker for the primary sample.

7.8 Pipet another aliquot (same amount as used in Step 7.7) of diluted sample solution into a second Coors beaker for the duplicate sample.

7.9 Add sufficient DI water to cover the stirring bar in each beaker.

7.10 Add DI water to a third beaker to cover stirring bar for a blank sample.

7.11 Add 1 mL of 5% Potassium Chromate solution to the sample, duplicate, and blank samples.

7.12 Stir resulting blank solution in Coors beaker on a magnetic stir plate.

7.13 Record Buret Initial Volume on Excel version of Attachment 9.

7.14 Record Titrant N and Lot No. on Excel version of Attachment 9.

NOTE

End point has been reached when solution shows a slightly red shade for 10 to 15 seconds.

7.15 Titrate solution with standardized silver nitrate until end point is reached.

7.16 Record Buret Final Volume and Net Titrant Used on Excel version of Attachment 9.

7.17 Stir sample solution in Coors beaker on a magnetic stir plate.

7.18 Record Buret Initial Volume on Excel version of Attachment 9.

NOTE

End point has been reached when solution shows a slightly red shade for 10 to 15 seconds.

7.19 Titrate solution with standardized silver nitrate until end point is reached.

7.20 Record Buret Final Volume and Net Titrant Used on Excel version of Attachment 9.

7.21 Stir duplicate sample in Coors beaker on a magnetic stir plate.

7.22 Record Buret Initial Volume on Excel version of Attachment 9.

NOTE

End point has been reached when solution shows a slightly red shade for 10 to 15 seconds.

7.23 Titrate solution with standardized silver nitrate until end point is reached.

7.24 Record Buret Final Volume and Net Titrant Used on Excel version of Attachment 9.

NOTE

Excel will calculate and record the sample and duplicate Cl concentrations on Attachment 9 using the following formula:

$$Cl\ (mg/L) = \frac{(C-D)\ (N\ AgNO_3)\ (35500)\ (B)}{A}$$

where: C = net titrant used to titrate sample (mL)
D = net titrant used to titrate blank (mL)
N $AgNO_3$ = normality of $AgNO_3$
B = dilution factor
A = sample volume (mL)

7.25 Record Sample and Duplicate Time of Analysis on Excel version of Attachment 9

7.26 Complete the Prepared By and Date on Excel version of Attachment 9.

7.27 Verify that Excel spreadsheet has transferred test results to Attachment 2.

8.0 CHLORIDE SPIKE ANALYSIS

NOTE

Spike analysis is performed only once during a sampling round at the discretion of the TL.

8.1 Prepare spike sample as follows:

8.1.1 Obtain the corresponding pre-prepared spike solution with a known true chloride concentration (in mg/L), as specified on the separate dilution table posted in the mobile lab (the same table that contains the modified dilutions, as described in Step 7.4).

8.1.2 Use diluted sample, prepared above in Step 7.4, and corresponding pre-prepared spike solution with a known true chloride concentration obtained in Step 8.1.1, to prepare the spike sample, as specified below.

8.1.3 Pipet a volume equal to one-half of that used in Step 7.7 of diluted sample solution into Coors beaker.

8.1.4 Pipet a volume equal to that used in Step 8.1.3 of pre-prepared spike solution into same Coors beaker.

8.1.5 Record the following on Excel version of Attachment 9:

- Aliquot of Sample for Spike (mL)
- Dilution Factor of Spike (Flask/Pipet)
- Volume of Spike (mL)
- True Chloride Concentration of Spike

8.1.6 Add 1 mL of 5% Potassium Chromate.

8.1.7 Adjust volume in Coors beaker using DI water to cover stirring bar.

8.2 Stir spike sample in Coors beaker on a magnetic stir plate.

8.3 Record Buret Initial Volume on Excel version of Attachment 9.

NOTE

End point has been reached when solution shows a slightly red shade for 10 to 15 seconds.

8.4 Titrate spike sample with standardized silver nitrate until end point is reached.

8.5 Record Buret Final Volume and Net Titrant Used on Excel version of Attachment 9.

8.6 Calculate Percent Spike Recovery using the following equation:

$$Percent\ Recovery = 100\ [\frac{Measured\ Chloride\ Conc.\ of\ Spike}{True\ Chloride\ Conc.\ of\ Spike}]$$

8.7 **IF** spike recovery is <90% or >110% of calculated value, **THEN** repeat Sections 7.0 and 8.0.

8.8 Verify that Excel has calculated and recorded the following on Attachment 9:

- Average Net Titrant Used for Serial Sample/Blank
- Net Titrant Used for Spike
- Measured Chloride Concentration of Spike
- Percent Recovery

9.0 DIVALENT CATION CONCENTRATION ANALYSIS

NOTE

A method blank solution will be carried through the entire process, along with sample solution and duplicate sample solution.

CAUTION

Expired EDTA solution must be segregated and stored in the SAA located within the mobile lab until proper disposal can be arranged.

WARNINGS

EDTA solution, 0.01 M must be handled with care. Spilling on exposed skin and clothing must be avoided to prevent irritation. If spillage occurs on exposed skin and/or clothing, user should remove affected clothing and immediately wash skin area with soap and rinse with large quantities of water. If small amount is spilled on laboratory surfaces, user should wipe up and rinse surface with water immediately. Spill control kits are to be used to clean up large spills.

Ammonium hydroxide/ammonium chloride buffer solution is toxic and corrosive and must be handled with care. Inhalation and prolonged exposure to vapors are to be avoided. PPE and vent hood must be used when dispensing. Avoid spilling on exposed skin and clothing to prevent burns and irritation. If spillage occurs on exposed skin and/or clothing, affected clothing should be removed, and skin area rinsed immediately with large quantities of water. If small amount is spilled on lab surfaces, the surface should be wiped and rinsed with water immediately. Spill control kits are to be used to neutralize and clean up large spills. Caustic neutralizers are recommended for spills of this reagent.

Hydroxylamine hydrochloride solution is corrosive to skin and potentially toxic, and must be handled with care. Spilling on exposed skin and clothing should be avoided to prevent burns and irritation. If spillage occurs on exposed skin and/or clothing, affected clothing should be removed, and skin area immediately rinsed with large quantities of water. If small amount is spilled on lab surfaces, they should be wiped and rinsed with water immediately. Spill control kits are to be used to clean up large spills.

Triethanolamine Solution must be handled with care. Spilling on exposed skin and clothing should be avoided to prevent irritation. If spillage occurs on exposed skin and/or clothing, affected clothing should be removed, and skin area immediately washed with soap and rinsed with large quantities of water. If small amount is spilled on lab surfaces, they should be wiped

and rinsed with water immediately. Spill control kits are to be used to clean up large spills.

Calmagite Indicator Solution is corrosive to skin and potentially toxic, and must be handled with care. Spilling on exposed skin and clothing should be avoided to prevent burns, irritation, and staining. If spillage occurs on exposed skin and/or clothing, affected clothing should be removed, and skin area immediately rinsed with large quantities of water. If a small amount is spilled on lab surfaces, they should be wiped and rinsed with water immediately. Spill control kits are to be used to clean up large spills.

Although the waste solutions produced during divalent cation titration are not hazardous by regulatory definition, they may be harmful to humans and animals; therefore, the waste solutions should be segregated during storage and disposed of properly (i.e., as nonhazardous waste).

9.1 Record the following on Excel version of Attachment 10:

- Well
- Zone
- Round
- Serial Sample No.

9.2 **GO TO** WP 02-EM1006 and collect a filtered serial sample, and **RETURN TO** Step 9.3.

NOTE

If normality of EDTA is not 0.025 M, then dilution must be modified from dilution table proportionately.

9.3 Refer to specific gravity of current serial sample on Attachment 2.

9.4 Using the specific gravity of current serial sample on Attachment 2, dilute sample to concentration shown on Attachment 12, Dilution Tables, or modify proportionately as specified in the **NOTE** above (the modified dilutions are posted on a separate table in the mobile lab for the corresponding molarity [M] of the EDTA titrant being used during the current sampling Round), and use diluted sample to prepare the sample, duplicate sample and spike sample, as described below.

9.5 Record sample and duplicate Dilution Factor on Excel version of Attachment 10.

9.6 Record sample and duplicate Aliquot Volume, and record Titrant M and Lot No. on Excel version of Attachment 10.

9.7 Pipet aliquot of diluted sample from appropriate dilution table into a 250-mL beaker for the sample.

9.8 Pipet another aliquot of diluted sample solution into a second 250-mL beaker for a duplicate sample.

9.9 Add sufficient DI water to cover the stirring bar.

9.10 Add DI water to a third beaker to cover stirring bar for a method blank.

9.11 Add 1 mL hydroxylamine hydrochloride solution to each beaker.

9.12 Add 3 drops triethanolamine solution to each beaker.

9.13 Add 5 mL ammonium chloride/ammonium hydroxide buffer solution to each beaker.

9.14 Add 5 drops Calmagite indicator solution to each beaker.

9.15 Stir blank solution in 250-mL beaker on a magnetic stir plate.

9.16 Record Blank Buret Initial Volume on Excel version of Attachment 10.

NOTE

The end reaction is slow and care must be taken to prevent titrating past end point. The blank will require only a few drops of titrant. End point is indicated by a change in color from red to true blue.

9.17 Titrate blank solution with EDTA until end point is reached.

9.18 Record Blank Buret Final Volume and Excel will calculate Net Titrant Used on Attachment 10.

9.19 Stir sample solution in beaker on a magnetic stir plate.

9.20 Record Sample Buret Initial Volume on Excel version of Attachment 10.

9.21 Titrate sample with EDTA until end point is reached.

9.22 Record Sample Buret Final Volume and Excel will calculate Net Titrant Used on Attachment 10.

9.23 Stir duplicate solution in 250-mL beaker on a magnetic stir plate.

9.24 Record duplicate Buret Initial Volume on Excel version of Attachment 10.

9.25 Titrate duplicate solution with EDTA until end point is reached.

9.26 Record Duplicate Buret Final Volume and Excel will calculate Net Titrant Used on Attachment 10.

9.27 Dispose of titrant residue.

NOTE

Excel will use data obtained and entered to calculate concentration (meq/L) of calcium and magnesium present in water according to the following equation:

(Ca + Mg) meq/L = (C-D) * (M EDTA) * (2,000) * (B)/(A)

where: A = sample aliquot (mL)
B = dilution factor (flask/Buret)
C = net titrant used for sample aliquot (mL)
D = net titrant used for the blank (mL)

NOTE

Divalent Cation concentration is reported to nearest tenth of a meq/L.

Excel will calculate and record the following on Attachment 8:

- Sample Divalent Cations (meq/L)
- Duplicate Divalent Cations (meq/L)

9.28 Record the following on Excel version of Attachment 10:

- Sample Time of Analysis
- Duplicate Time of Analysis

9.29 Complete the Prepared By and Date on Excel version of Attachment 10.

9.30 Verify that Excel spreadsheet has transferred test results to Attachment 2.

10.0 DIVALENT CATION SPIKE ANALYSIS

NOTE

Spike analysis is performed only once during a sampling round at the discretion of the TL.

10.1 Prepare a spike sample as follows:

10.1.1 Pipet a volume of diluted sample equal to 5 mL less than of that used in Step 9.7 into a 250-mL beaker.

10.1.2 Record Aliquot of Diluted Sample on Excel version of Attachment 10.

10.1.3 Pipet a volume of pre-prepared 0.01 M Calcium Carbonate spike solution into the same 250-mL beaker.

10.1.4 Record Volume of Spike Solution on Excel version of Attachment 10.

10.1.5 Record Dilution Factor of Spike on Excel version of Attachment 10.

NOTE

The following equation will be used by Excel to calculate the True Value of Spike; however, the result will not be displayed on Attachment 10:

$$True\ Value\ of\ Spike = (Volume\ of\ Spike)\ (M\ of\ CaCO_3)$$

10.1.6 Add 1 mL hydroxylamine hydrochloride solution to the beaker.

10.1.7 Add 3 drops triethanolamine solution.

10.1.8 Add 5 mL ammonium chloride/ammonium hydroxide solution.

10.1.9 Add 5 drops Calmagite indicator solution.

10.1.10 Add a stirring bar to beaker.

10.1.11 Add DI water to cover stirring bar.

10.2 Stir spike solution in beaker on magnetic stir plate.

10.3 Record spike Buret Initial Volume on Excel version of Attachment 10.

10.4 Titrate spike solution with EDTA until end point is reached.

10.5 Record spike Buret Final Volume and Excel will calculate Net Titrant Used on Attachment 10.

NOTE

Excel will use data obtained and entered to calculate Average Net Titrant (ANT) Used For Serial Sample/Blank for the sample and duplicate, minus the blank, using the following formula:

$$ANT = \frac{(Net\ Titrant\ Used\ for\ Sample) + (Net\ Titrant\ Used\ for\ Duplicate)}{2} - Net\ Titrant\ Used\ for\ Blank$$

10.6 Verify that Excel has recorded ANT used on Attachment 10.

NOTE

Excel will calculate Net Titrant Used for Spike (NTS) using the following formula:

$$NTS = NT - \frac{ANT}{2}$$

where: NT = Net Titrant Used

10.7 Verify that Excel has recorded NTS on Attachment 10.

NOTE

Excel will calculate the Measured Value of Spike using the following formula:

$$Measured\ Value\ of\ Spike = (Net\ Titrant\ Used\ for\ Spike)\ (M\ of\ Titrant\ (EDTA))$$

10.8 Verify that Excel has recorded the Measured Value of Spike on Attachment 10.

NOTE

Excel will calculate Percent Recovery using the following formula:

$$Percent\ Recovery = 100\ \frac{(Measured\ Value\ of\ Spike)}{(True\ Value\ of\ Spike)}$$

10.9 Verify that Excel has recorded the Percent Recovery on Attachment 10.

11.0 TOTAL IRON ANALYSIS

NOTE

Measurement of Total Iron should be performed as soon as possible after collection of sample.

WARNINGS

Iron (Fe) Standard Reagent (1,000 µg/mL) contains iron oxide and dilute nitric acid in an aqueous solution, is corrosive and potentially toxic, and must be handled with care. PPE and vent hood must be used. Spilling on exposed skin and clothing is to be avoided to prevent chemical burns or irritation. If spillage occurs on exposed skin and/or clothing, affected clothing should be removed, and the skin area rinsed immediately with large quantities of water. If small amount is spilled on lab surfaces, surface must be wiped up and rinsed with water immediately. Spill control kits are to be used to neutralize and clean up large spills. Acid neutralizers are recommended for spills of Iron Standard Reagent.

Expired Iron (Fe) Standard Reagent solution is hazardous by regulatory definition; therefore, it should be stored in the SAA located within the mobile lab until proper disposal can be arranged.

Although the waste solutions produced during iron analysis are not hazardous by regulatory definition, they may be harmful to humans and animals; therefore, the waste solutions should be placed in a suitable container, segregated during storage and disposed of properly (i.e. as nonhazardous waste).

Breaking the tips of the FerroVer Iron Reagent ampuls when filling them with the iron standard and samples in Steps 11.15 through 11.18 (and in Steps 11.34.3 and 11.34.4) produce sharp edges and glass shards. Extreme care must be used when handling these objects, as severe injury may result if mishandled.

Ferrover AccuVac Iron Reagent ampuls contain several dry chemicals (white powders) which may cause irritation and/or allergic reaction if spilled on exposed skin or inhaled (see WIPP MSDS #W0338 for Composition/ Information on Ingredients). Ampuls **MUST BE HANDLED WITH EXTREME CARE** to avoid accidental exposure. If spillage of powder or test sample solution (after filling) occurs on exposed skin and/or clothing, affected clothing is to be removed, and skin area rinsed immediately with large quantities of water. If allergic reaction is suspected, medical care should be sought immediately. If small amount is spilled on laboratory surfaces, they must be wiped and rinsed with water immediately.

11.1 Record the following on Excel version of Attachment 11:

- Well
- Zone
- Round
- Serial Sample No.
- Spectrophotometer Make
- Spectrophotometer S/N

11.2 **GO TO** WP 02-EM1006 and collect a filtered serial sample, and **RETURN TO** Step 11.3.

11.3 Prepare a one mg/L (nominal) iron standard as follows:

11.3.1 Pipet a 1-mL aliquot of AA iron standard (1000 mg/L [nominal]) into a 1-liter volumetric flask.

11.3.2 Dilute standard solution with DI water to 1 liter.

11.3.3 Record the following on Excel version of Attachment 11:

- Standard Fe 1000 ppm Manufacturer and Lot Number
- Aliquot of AA Iron Standard
- Aliquot DI water
- Concentration (mg/L) of Fe Standard

NOTE

No warmup time is required for the DR 2000 Spectrophotometer.

11.4 Turn on DR 2000 Spectrophotometer by pressing Power key.

11.5 Enter 267 (the stored program number for Iron FV AV).

11.6 Press Read/Enter.

11.7 Verify display reads: **Dial nm to 510**.

NOTE

Approach the required wavelength reading by dialing it in from the high side.

11.8 Adjust Wavelength dial located In upper right corner of instrument until digital LCD display marked "nm" reads **510 nm**.

11.9 Press Read/Enter.

11.10 Verify display reads **mg/L Fe FV AV**.

11.11 Transfer 40 mL of prepared iron standard solution into a 50 mL beaker and set aside.

11.12 Transfer 40 mL of DI water for blank sample into a 50 mL beaker and set aside.

11.13 Transfer 40 mL of filtered serial sample into a 50 mL beaker and set aside.

11.14 Transfer 40 mL of filtered duplicate serial sample into a 50 mL beaker and set aside.

WARNING

Breaking the tips of the FerroVer Iron Reagent ampuls when filling them with the iron standard and samples in Steps 11.15 through 11.18 (and in Steps 11.34.3 and 11.34.4) produces sharp edges and glass shards. Extreme care must be used when handling these objects, as severe injury may result if mishandled.

NOTE

In Steps 11.15 through 11.18 (and in Steps 11.34.3 and 11.34.4) the Ferrover AccuVac Iron Reagent ampuls are filled by using the following technique:

1. Ampul is inverted (pointed tip down) and lowered into the corresponding 50 mL beaker (prepared in Steps 11.11 through 11.14) so that the pointed tip of the ampul is submerged.

2. The tip of the ampul is gently pressed against the inside of the 50 mL beaker and the tip of the ampul breaks off. The vacuum inside of the ampul will cause it to fill.

3. Ampul is set aside when filled.

11.15 Fill a Ferrover AccuVac Ampul with prepared iron standard.

11.16 Fill a Ferrover AccuVac Ampul with DI water for blank sample.

11.17 Fill a Ferrover AccuVac ampul with filtered water sample.

11.18 Fill a Ferrover AccuVac ampul with duplicate sample.

NOTE

A three-minute period will begin for chemicals to react when timer has been started.

11.19 Press Shift, then Timer.

11.20 Quickly invert the Ferrover AccuVac ampuls to mix solutions.

11.21 Wipe off finger prints or any water from ampuls.

11.22 **WHEN** timer beeps,
VERIFY display reads **mg/L Fe FV AV.**

11.23 Place Ferrover AccuVac ampul containing blank (DI water) in cell holder of instrument.

11.24 Close light shelf on top of instrument.

11.25 Press Zero key.

11.26 Verify display reads **wait**.

11.27 Wait for display to read **0.00 mg/L Fe Fv Av**.

11.28 Remove Ferrover AccuVac ampul from instrument.

11.29 Record Blank Instrument Reading and Time on Excel version of Attachment 11.

11.30 Place AccuVac ampul containing sample in cell holder of instrument.

11.31 Close light shield.

11.32 Press Read/Enter and verify display reads **wait**.

11.33 Wait for instrument to read total iron concentration.

NOTE

Operation range constraints of instrument may require dilution of sample.

11.34 **IF** instrument reads **Over Range**,
THEN perform the following:

11.34.1 Dilute 50 mL of sample with 100 mL DI water (2:1 dilution).

11.34.2 Record Sample Dilution Factor on Excel version of Attachment 11.

11.34.3 Fill a Ferrover AccuVac Ampul with diluted sample.

11.34.4 Fill a Ferrover AccuVac Ampul with a duplicate diluted sample.

11.34.5 Place Ferrover AccuVac Ampul containing diluted sample in cell holder.

11.34.6 Close light shield.

11.34.7 Press Read/Enter and verify display reads **wait**.

11.34.8 Wait for instrument to read total iron concentration.

11.34.9 Record Sample Instrument Reading and Time on Excel version of Attachment 11. Excel will multiply Sample Instrument Reading by Dilution Factor and record Sample Result.

11.35 Remove Ferrover AccuVac ampul from instrument.

11.36 Place AccuVac ampul containing duplicate diluted sample in cell holder of instrument.

11.37 Close light shield.

11.38 Press Read/Enter and verify display reads **wait**.

11.39 Wait for instrument to read total iron concentration.

11.40 Record Duplicate Instrument Reading and Time on Excel version of Attachment 11. Excel will multiply Duplicate Instrument Reading by Dilution Factor and record Duplicate Result.

11.41 **IF** no dilution is required,
THEN remove Ferrover AccuVac ampul from instrument.

11.42 Place AccuVac Ampul containing duplicate sample in cell holder.

11.43 Close light shield.

11.44 Press Read/Enter and verify display reads **wait**.

11.45 Wait for instrument to read total iron.

11.46 Record Duplicate Instrument Reading and Time on Excel version of Attachment 11.

11.47 Remove Ferrover AccuVac ampul from instrument.

11.48 Place AccuVac ampul containing standard in cell holder of instrument.

11.49 Close light shield.

11.50 Press Read/Enter and verify display reads **wait**.

11.51 Wait for instrument to read total iron concentration.

11.52 Remove Ferrover AccuVac ampul from instrument.

11.53 Record Standard Instrument Reading and Time on Excel version of Attachment 11.

11.54 Verify reading of standard is within ± 0.1 mg/L of certified value of standard.

11.55 **IF** reading is not within ± 0.1 mg/L of certified value of standard, **THEN** repeat Steps 11.5 through 11.53.

11.56 **IF** reading is not within ± 0.1 mg/L of certified value of standard, **THEN**, prepare a new standard per Step 11.3, and repeat Steps 11.5 through 11.50.

11.57 **IF** reading is still not within ± 0.1 mg/L of certified value of standard, **THEN**, stop testing and notify TL.

NOTE

Excel will calculate % recovery of Iron standard using the following formula and record results on Attachment 11:

$$Percent\ Recovery = 100\ [\frac{Measured\ Value\ of\ Standard}{True\ Value\ of\ Standard}]$$

11.58 Remove Ferrover AccuVac ampul from instrument.

11.59 Dispose of glass ampuls in designated waste container.

11.60 Turn instrument off by pressing Power.

11.61 Complete the Prepared By and Date on Excel version of Attachment 11. Excel spreadsheet will transfer test results to Attachment 2.

12.0 DATA MANAGEMENT

12.1 TL/designee, perform the following:

12.1.1 Print Excel versions of completed Attachments 1 and 2, and 4 through 11.

12.1.2 Make check print copy of Attachments 1 through 11.

12.1.3 Stamp original attachments "ORIGINAL."

12.1.4 Stamp check print copies "CHECK PRINT."

12.1.5 Check all entries on check print copy of attachments.

12.1.6 Complete the Checked By and Date on check print copies of Attachments 1 through 11.

12.1.7 Enter sampling data into Sampling Round Data Book.

12.1.8 Store attachments in EM 1-hour fire rated files.

Attachment 1 - Sample Serial Sampling Report for Eh (Redox)

WELL:_____ PREPARED BY:_____ DATE:_____

ZONE:_____ ROUND:_____

SERIAL SAMPLE:_____

EQUIPMENT/REAGENTS

METER ID: ORION 720A SERIAL NO: _____

ELECTRODE AND FILLING SOLUTION: Orion 9678BN / Orion 90-00-11

THERMOMETER ID: SERIAL NO: _____ CAL. EXP. DATE: _____

STANDARDS: ZoBell LOT NO: _____ LIGHTS' LOT NO: _____

EQUIPMENT CHECK

SOLUTION	TIME	TEMP (°C)	* EXPECTED TEMP VALUE REL TO SHE	METER VALUE (mV)	Eh RELATIVE TO SHE (mV)*
ZoBell 1					
Lights'					
ZoBell 2					

MEASUREMENT

TIME OF COLLECTION	TIME OF MEASUREMENT	TEMP (°C)	METER VALUE (mV)	Eh RELATIVE TO SHE (mV) *

REMARKS:_____

_____

_____

_____

_____

CHECKED BY:_____ DATE:_____

Attachment 2 - Sample Serial Sampling Summary Sheet

WELL:_____ PREPARED BY:_____ DATE:_____

ZONE:_____ ROUND:_____

SERIAL SAMPLE:_____

PARAMETER	UNITS	TEST RESULTS	ANALYST	DATE/TIME TESTED
Eh	mV			
pH	S. U.			
Temperature	°C			
Sp. Gravity at °C	____			
Sp. Conductance at °C	µmhos/cm			
Bicarbonate Alkalinity as HCO_3	mg/L	Samp. _____ Dupl. _____		
Carbonate Alkalinity as CO_3	mg/L	Samp. _____ Dupl. _____		
Chloride	mg/L	Samp. _____ Dupl. _____		
Divalent Cations	meq/L	Samp. _____ Dupl. _____		
Iron (Total)	mg/L	Samp. _____ Dupl. _____		

REMARKS:_____

_____

_____

CHECKED BY:_____ DATE:_____

Attachment 3 - Serial Sampling Check Printout Sheet

WELL:_____ PREPARED BY:_____ DATE:_____

ZONE:_____ ROUND:_____

SERIAL SAMPLE:_____

CHECKED BY:_____ DATE:_____

Attachment 4 - Sample Serial Sampling Report for pH

WELL:_____ PREPARED BY:_____ DATE:_____

ZONE:_____ ROUND:_____

SERIAL SAMPLE:_____

EQUIPMENT/REAGENTS

METER ID: Orion 720A SERIAL NO:_____

ELECTRODE AND FILLING SOLUTION: Orion 9157BN/Orion 90-00-11

PH 7.00 BUFFER LOT NO:_____ EXP. DATE:_____

PH (4.00 OR 10.00) BUFFER LOT NO:_____ EXP. DATE:_____

PERFORMANCE CHECK

SOLUTION	TIME	TEMP (°C)	METER VALUE	ELECTRODE SLOPE
pH (7.00)				
pH (4.00 or 10.00)				
pH (7.00)				

MEASUREMENT

TIME OF COLLECTION	TIME OF MEASUREMENT	TEMP (°C)	METER VALUE

REMARKS:_____

_____

_____

CHECKED BY:_____ DATE:_____

Attachment 5 - Sample Serial Sampling Report for Temperature

WELL:_____ PREPARED BY:_____ DATE:_____

ZONE:_____ ROUND:_____

SERIAL SAMPLE:_____

EQUIPMENT

THERMOMETER ID: SERIAL NO:_____ CAL. EXP. DATE:_____

MEASUREMENT

TIME OF SAMPLE COLLECTION	TIME OF TEMPERATURE MEASUREMENT	TEMPERATURE (°C)

REMARKS:_____

_____

_____

_____

CHECKED BY:_____ DATE:_____

Attachment 6 - Sample Serial Sampling Report for Specific Gravity

WELL:_____ PREPARED BY:_____ DATE:_____

ZONE:_____ ROUND:_____

SERIAL SAMPLE:_____

EQUIPMENT

THERMOMETER: SERIAL NO:_____ CAL. EXP. DATE:_____

HYDROMETER: SERIAL NO:_____ CAL. EXP. DATE:_____

MEASUREMENT

TIME OF SAMPLE COLLECTION	TIME OF MEASUREMENT	OBSERVED HYDROMETER READING	TEMPERATURE (°C)	DEGASSING TIME

REMARKS:_____

_____

_____

_____

CHECKED BY:_____ DATE:_____

Attachment 7 - Sample Serial Sampling Report for Specific Conductance

WELL:_____ PREPARED BY:_____ DATE:_____

ZONE:_____ ROUND:_____

SERIAL SAMPLE:_____

EQUIPMENT

METER MODEL NO:_____ SERIAL NO:_____

PROBE MODEL & ID NO: YSI No 3252/ID NO:_____

STANDARD:_____ µmhos/cm LOT NO:_____

STANDARD:_____ µmhos/cm LOT NO:_____

TIME OF COLLECTION _____

METHOD OF TEMP. CORR: Automatic Temperature Correction (ATC)

EQUIPMENT CHECK

STANDARD	TIME	TEMPERATURE (°C)	METER VALUE (µmhos/cm) = G25

MEASUREMENT

TIME	TEMPERATURE (°C)	METER VALUE (µmhos/cm) = G25

REMARKS: _____

_____

_____

CHECKED BY:_____ DATE:_____

Attachment 8 - Sample Serial Sampling Report for Alkalinity

WELL:_____ PREPARED BY:_____ DATE:_____

ZONE:_____ ROUND:_____

SERIAL SAMPLE:_____

SAMPLE IDENTIFICATION NUMBER	SAMPLE	DUPLICATE
Sample Time		
Initial pH		
Sample Volume, mL	100 mL	100 mL
Titrant _____ N H_2SO_4 Titrant Lot No. _____		
PHENOLPHTHALEIN ALKALINITY (Titration to 8.1 pH)		
Buret Final Volume (mL)		
Buret Initial Volume (mL)		
Net Titrant Used (mL)		
TOTAL ALKALINITY (Titration to 4.5 pH)		
Buret Final Volume (mL)		
Buret Initial Volume (mL)		
Net Titrant Used (mL)		
Alkalinity as Carbonate (mg/L)		
Alkalinity as Bicarbonate (mg/L)		
Alkalinity as Hydroxides (mg/L)		

REMARKS:_____

_____

_____

_____

CHECKED BY:_____ DATE:_____

Attachment 9 - Sample Serial Sampling Report for Chlorides and Spike

WELL:_____ PREPARED BY:_____ DATE:_____

ZONE:_____ ROUND:_____

SERIAL SAMPLE:_____

PARAMETER	ANALYSIS INFORMATION	SAMPLE	DUPLICATE	BLANK
CHLORIDE	Dilution Factor (Flask/Pipet)			
	Aliquot of Sample (mL)			
	Titrant _____ N $AgNO_3$ Lot No. _____			
	Buret Final Volume (mL)			
	Buret Initial Volume (mL)			
	Net Titrant Used (mL)			
	Cl (mg/L)			
	Time of Analysis			
SPIKE	Aliquot of Sample for Spike (mL)			
	Dilution Factor of Spike (Flask/Pipet)			
	Volume of Spike (mL)			
	Buret Final Volume (mL)			
	Buret Initial Volume (mL)			
	Net Titrant Used (mL)			
	True Chloride Concentration of Spike			
	Average Net Titrant Used for Serial Sample/Blank			
	Net Titrant Used for Spike _____			
	Measured Chloride Concentration of Spike _____ _____			
	Percent Recovery _____			

REMARKS: _____

_____

_____

CHECKED BY:_____ DATE:_____

Attachment 10 - Sample Serial Sampling Report for Divalent Cations and Spike

WELL:_____ PREPARED BY:_____ DATE:_____

ZONE:_____ ROUND:_____

SERIAL SAMPLE:_____

PARAMETER	ANALYSIS INFORMATION	SAMPLE NUMBER		
		SAMPLE	DUPLICATE	BLANK
DIVALENT CATIONS	Dilution Factor (Flask/Pipet)			
	Aliquot of Sample (mL)			
	Titrant _____ M EDTA Lot No. _____			
	Buret Final Volume			
	Buret Initial Volume			
	Net Titrant Used (mL)			
	Divalent Cations (meq/L)			
	Time of Analysis			
SPIKE	Aliquot of Diluted Sample (mL)			
	Volume of Spike Solution (mL)			
	Dilution Factor of Spike			
	Buret Initial Volume (mL)			
	Buret Final Volume (mL)			
	Net Titrant Used (mL)			
	Average Net Titrant Used			
	Net Titrant Used for Spike			
	Measured Value of Spike			
	Percent Recovery			

REMARKS: _____

_____

_____

_____

CHECKED BY:_____ DATE:_____

Attachment 11 - Sample Serial Sampling Report for Total Iron

WELL:_____ PREPARED BY:_____ DATE:_____

ZONE:_____ ROUND:_____

SERIAL SAMPLE:_____

EQUIPMENT/REAGENT

SPECTROPHOTOMETER MAKE:_____ SERIAL NO.:_____

STANDARD Fe 1000 PPM (NOMINAL) MFG.:_____ LOT NO.:_____

PREPARED STANDARD:____ mL OF STANDARD INTO ____ DI WATER = ____ mg/L Fe STANDARD

MEASUREMENT

TEST	INSTRUMENT READING	DILUTION FACTOR	RESULT	TIME
Blank			mg/L	
Sample Iron (Total)			mg/L	
Duplicate Iron (Total)			mg/L	
Standard			mg/L	
% Recovery of _____ mg/L Fe Std. _____ %				

REMARKS:_____

_____

_____

_____

CHECKED BY:_____ DATE:_____

Attachment 12 - Dilution Tables

TABLE 1 - Aliquots that contain < 50 mg of chloride as estimated from the specific gravity.

SPECIFIC GRAVITY (mL)	DILUTION	ALIQUOT
1.000 - 1.002	None	100.000
1.003 - 1.004	None	50.000
1.005 - 1.012	Dilute 10 mL to 100 mL, take 50 mL	5.000
1.013 - 1.019	Dilute 10 mL to 100 mL, take 20 mL	2.000
1.020 - 1.032	Dilute 10 mL to 100 mL, take 10 mL	1.000
1.033 - 1.064	Dilute 25 mL to 500 mL, take 10 mL	0.500
1.065 - 1.087	Dilute 20 mL to 500 mL, take 10 mL	0.400
1.088 - 1.162	Dilute 10 mL to 500 mL, take 10 mL	0.200
> 1.163	Dilute 10 mL to 1000 mL, take 10 mL	0.100

TABLE 2 - Aliquot size for divalent cation determination.

SPECIFIC GRAVITY (mL)	DILUTION	ALIQUOT
1.000 - 1.010	None	50.000
1.010 - 1.025	None	25.000
1.025 - 1.050	Dilute 25 mL to 100 mL, take 50 mL	12.500
1.050 - 1.090	Dilute 25 mL to 100 mL, take 25 mL	6.250
1.090 - 1.120	Dilute 25 mL to 500 mL, take 25 mL	1.250
1.120 - 1.150	Dilute 25 mL to 1000 mL, take 25 mL	0.625