Appendix B
Sample Standard Operating Procedures

STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF pH

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SOP - pH

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Section 1: Control Information

The SOP is stored as a Microsoft Word document in the SOP folder as file named SOPpH.doc.

Section 2: Personnel Qualifications

Technician should assist in the test for at least 1 week and should have performed a witnessed, unassisted pH performance sample before initiating the procedure unsupervised.

Section 3: Summary of Method Used

The method used is Standard Methods for the Examination of Water and Wastewater, 20th edition, Section 4500-H^+-B . This method is applicable to drinking water and wastewater samples. The method consists of placing a glass pH electrode into a well mixed sample and recording the display on a calibrated pH meter.

Section 4: Materials and Equipment

Fisher Accumet 50
Combination glass electrode
General laboratory glassware
pH 4 buffer
pH 7 buffer
pH 10 buffer

Section 5: Personal Protection Equipment

Latex gloves and safety goggles should be worn at all times. Personal hygiene should include hand washing with disinfectant upon completion.

Section 6: Daily pH meter Calibration

The pH meter is used to measure the hydrogen ion concentration of a sample. For routine work, the pH meter must be accurate and reproducible to the nearest 0.1 pH unit. Sample pH and standard buffers should be measured at the same temperature, generally room temperature. The pH meter is normally very stable and needs to be calibrated only once daily. (generally within 3 hours of use). Calibrate more frequently if indicated.

pH Meter Calibration

6.1a Connect the pH meter and electrode to channel A. Remove the electrode from the KCl or pH 7 buffer storage solution; rinse with deionized water and blot dry. TIP: The bulb of the pH electrode must be below the water level. If the meter reading is erratic, check the water depth. If using the ATC, insert the ATC in the buffer as well. The meter will adjust for temperature differences automatically.

6.1b Press the **CHANNEL** button and select channel A. Press the **pH** button.

6.1c Press the **STANDARDIZE** button and press 2 to clear any previous data. Calibrate with at least 3 buffers. Select buffers that will bracket the typical sample pH. A fourth buffer can be used to validate the electrode response.

6.1d Place the electrode in pH 7 buffer. Press the **STANDARDIZE** button, and then press 1. Gently swirl the buffer, and then press **ENTER**. The large number

display should show 7.0. In the upper left-hand corner, a small 7.0 should appear; indicating the calibration point has been entered. **Record the lot #, expiration date, and calibration value on the pH Meter Calibration Log**

6.1e Rinse the electrode with deionized water and blot dry.

6.1f Place the electrode in pH 10 buffer. Press the **STANDARDIZE** button, then press 1. Gently swirl the buffer, and then press **ENTER**. The large number display should show 10.0. In the upper left-hand corner, a small 10.0 should appear; indicating the calibration point has been entered. **Record the lot #, expiration date, and calibration value on the pH Meter Calibration Log**

6.1g Rinse the electrode with deionized water and blot dry.

6.1h. Place the electrode in pH 4 buffer. Press the **STANDARDIZE** button, then press 1. Gently swirl the buffer, and then press **ENTER**. The large number display should show 4.01. In the upper left-hand corner, a small 4.01 should appear; indicating the calibration point has been entered. **Record the lot #, expiration date, and calibration value on the pH Meter Calibration Log**
Corrective Action: If the pH 4 buffer does not measure within 0.1 pH units, troubleshoot the electrode.

6.1i Press the SLOPE button. The slope should be 56 $\pm$ 3 mv or (>95% recovery) **Record the slope on the pH Meter Calibration Log. Press the slope button again to get back to the original pH display.**
Corrective Action: If the slope is not within tolerance, recalibrate. If the meter does not calibrate properly, replace the electrode and try again.

Section 7 Electrode Maintenance

7.1a The electrode must **NOT** be stored dry. The best solution to store the electrode is saturated KCl or pH 7 buffer. Tap water or deionized water should not be used because the electrolyte within the electrode will diffuse out and cause premature aging or additional maintenance.

7.1b Store the electrode in a solution of saturated KCl. Long term storage requires storage in a large test tube covered with parafilm.

Section 8 Troubleshooting

Meter

8.1a Disconnect the electrode and attach the shorting strap.

8.1b Press the millivolt button. The display should show zero.
If the display does not change, the meter needs repair.

Electrode

8.2a Gel electrodes generally require no maintenance if kept moist. However gel electrodes have a shelf life of around 2 years. Recording the date the electrode was placed in service helps with troubleshooting.

8.2b Glass electrodes fail because of scratches, deterioration, and accumulation of debris on the glass bulb. Rejuvenate the electrodes by cleaning in 0.1 M HCl and 0.1 M NaOH.
Corrective action: If the pH responds slowly or drifts after rejuvenation, replace the electrode.

8.2c Reference electrodes fail because of clogged liquid junctions. The liquid junction must remain open to avoid slow response and meter drift. If the liquid junction becomes clogged, place in hot deionized water for several hours. Some combination electrodes can be refilled with saturated KCl.

8.3 If the pH meter does not respond to different buffers (remains at pH 7 all the time), replace the electrode.

Section 9 Sample Handling

9.1 Sample collection is the responsibility of the customer. Clean plastic Nalge bottles are provided to the customer in the size indicated below. Glass containers are also acceptable.

9.2 Samples should be collected and analyzed within 15 minutes. No sample preservation is permitted. On site analysis is preferred.

9.3 Minimum sample volume should be 50 ml.

SAFETY: Use latex gloves when collecting and handling all wastewater samples

Section 10 Sample pH Measurement

10.1 Use a calibrated pH meter. The meter should be on channel A and in "pH" mode.

10.2 Carefully pour the sample into a 100 ml beaker. If the temperature is significantly different from room temperature, use the Automatic Temperature Compensating (ATC) Electrode if available.

10.3 Immerse the pH electrode and ATC into the solution. Be careful not to break the glass bulb.

10.4 Swirl the sample gently until the meter reading stabilizes. For poorly buffered samples such as deionized water, immerse in three or four consecutive portions of sample, and then immediately measure the pH in a fresh sample.

10.5 Report the answer to the nearest 0.1 pH units on the bench sheet.

Section 11 Data Management and Records Management

11.1 All records must be maintained in three ring binders and filed under the customer's name.

11.2 Maintain all calibration records for a minimum of 5 years.

Section 12 Quality Control

Decisions on the accuracy of the reported data will be based on the quality control information.

Sample QC

12.1 Sample holding time cannot exceed 15 minutes.
Corrective Action: Reject samples and request a resample.

12.2 Record sample date, time, type, sampler, date and time of analysis, analyst and method used.

12.3 Samples must be mixed well, poured quickly, and swirled constantly when measuring the pH

Equipment QC

12.4 Calibrate against a minimum of 2 buffers. Generally pH 7, pH 4 and pH 10 are used.

12.5 Buffers should bracket the expected sample pH.

12.6 Buffers used should be within the expiration date. Record date of purchase, lot number, and expiration date of buffers used.
Corrective Action: Expired buffers should be discarded and not used. Replace with fresh buffers. Purchase a 6 month supply and rotate stock frequently.

12.7 Store buffers in the dark, at room temperature, and in the chemical supply cabinet.

12.8 Performance evaluation samples should be run at least annually.

12.9 Split samples can be run

12.10 Duplicate sample pH results should be within 0.1 pH units.

Section 13 pH Calibration Log Sheet (Appendix 1)

pH METER CALIBRATION

Make and Model pH meter: _____
Date electrode place in service: _____

Date	Time	pH 4 buffer			pH 7 buffer			pH 10 buffer			Slope	Name
		Lot #	Exp. Date	pH	Lot #	Exp. Date	pH	Lot #	Exp. Date	pH		