

River Monitoring Program

Water Testing Procedures

Water testing of Salt Creek and East/West branches of DuPage River

Legend:

SC = Salt Creek

EB = DuPage River; East Branch

WB = DuPage River; West Branch

Sample sites are numbered as follows:

SC 1 = Prairie Path (Elmhurst)

SC 2 = Eldridge Park (Elmhurst)

EB 1 = Churchill Woods (Glen Ellyn)

EB 2 = Butterfield Rd. (Lombard)

EB 3 = Burlington Ave. (Lisle)

EB 4 = St. Joseph Creek (Lisle)

WB 1 = Beecher Ave. (Winfield)

WB 2 = Warrenville Grove (Warrenville)

WB 3 = Centennial Park (Naperville)

Traditionally, SC and EB sites are collectively referred to as “east branch”. EB4 is St. Joseph creek, a tributary to the East Branch of the DuPage River.

Samples are tested for pH and four analytes:

- **Phosphorus**
- **Nitrate**
- **Ammonia**
- **Conductivity (Chloride)**



When handling reagents, exercise the same caution and common sense you would with hazardous household chemicals. Use in a well ventilated area. Do not spill. Cap tightly after use. Keep away from eyes, food, children, and pets. Wash hands after contact. Some test chemicals will stain clothes and damage surfaces, so line work surface with newspapers or plastic (disposable table cloth or paint tarp).

Nomenclature

- *analyte* - compound being tested for (phosphorus, nitrate, ammonia, chloride)
- *cell* - tiny glass and plastic bottles in which tests are performed
- *electrode* - the “business end” (bottom) of the pH meter
- *jar* - large (one quart) glass jar of river water
- *method* - a particular analyte’s test method (reagents and procedure)
- *range* - the concentration range for which a method is accurate
- *reagent* - test chemical
- *sample* - river water
- *colorimeter* - instrument which calculates analyte concentration based on its color

General Testing Procedure

The test methods described herein involves using industry-standard reagents for colorimetric analysis with a Hach DR900 colorimeter. Together, very precise measurements (1 part in 10 million) are achievable - as long as test procedures are performed carefully and exactly!

For each analyte, you will test a BLANK (river sample or distilled water), a STANDARD (“standard” solution), and the river samples. The BLANK calibrates the colorimeter, and the STANDARD serves to verify your test procedures. Therefore, if either is prepared incorrectly, all test results for that analyte are jeopardized and must be repeated.

The new colorimeter will not require dilution of the river samples so the results can be recorded on the worksheets as shown by the colorimeter. Write final values in the worksheet’s rightmost column (marked “Result”) and also in the official log book. Double-check every calculation! Temperature and pH, must also be recorded on the worksheet and then copied to the log book. It may be easier to copy data from the worksheet to the log book at the end of the day.

Note any problems or test method deviations on the worksheet and log book. **Feel free to call me at 630-621-2877 with any questions.**

Test Preparation

- Upon receiving the test equipment, perform an inventory to verify sufficient supplies. New supplies can take up to four weeks to order, so plan ahead!
- Purchase at least two gallons of distilled water from the grocery store, which is used primarily for cleaning test equipment.
- Select a work area that is well ventilated, free of children and pets, and uncluttered. Avoid areas with vapors (such as laundry area) and open flames. Do not test in the vicinity of return vents (heating and air conditioning intake ducts). Table top should be flat and level, and covered with liner to protect against drips and spills.
- Review previous years’ site and analyte data. Your test results should jibe with them.

- Read this entire document, and the “Using Pipettes” and “Tri-valve Bulb” documents.
- Remove a blank East Branch and/or West Branch worksheet from the three-ring binder.

A Note on Distilled Water

In the past, ordinary grocery-store distilled water was sufficiently pure for both testing and cleaning. However, bottled water eventually became subject to federal drinking water regulations, and companies, in response, began treating distilled water with an inexpensive disinfection process which left a chemical residue. The residue, ironically, is not regulated, and River Prairie Group has detected high pH and/or ammonia concentrations greater than 0.50 mg/L in a variety of different brands. Since this level exceeds that which is found in the rivers, ordinary distilled water can no longer be used for testing, although it remains fine for cleaning.

Because the residues in ordinary distilled water interfere with the ammonia test (and possibly others), we now use laboratory-grade “ultra-pure” distilled water for phosphorus, nitrate, and ammonia tests. Due to the high cost of ultra-pure distilled water we DO NOT use it for cleaning!!

If ultra-pure distilled water is not available, then purchase a new bottle of brand-name distilled water from the store. Write a note on the worksheet (and log book) indicating the brand of distilled water you substituted for ultra-pure, and notify the test coordinator to determine if the test results are valid. If a small amount of ultra-pure distilled water is available, use it for the ammonia test, but don't use two different distilled waters in the same test.

In this document, *ordinary distilled water* refers to inexpensive distilled water purchased from a store, while *ultra-pure distilled water* refers to expensive, laboratory-grade distilled/deionized water purchased from a chemical supplier.

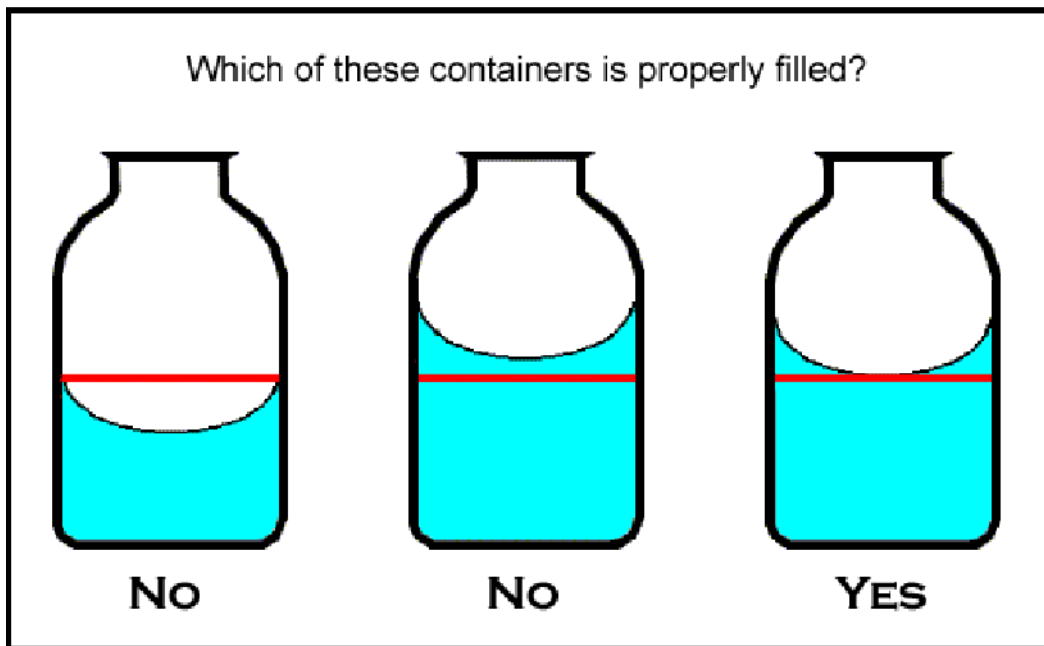
Keys to Accurate Tests

- Rushing invites mistakes. Reserve sufficient time so that testing is not rushed.
- Follow the directions in this manual carefully.
- Don't mix up sample cells. An adequately-sized work area allows sample jars to be neatly arranged in the same order they appear on the worksheet, with each cell placed in front of its jar. Handling cells one-at-a-time in this manner will help prevent mix-ups.
- Measurements are jeopardized if testing is not performed within a few hours of sample collection, as collection procedures and containers are not designed for long-term storage.
- Since the colorimeter measures analyte concentration by shining a precise light through one side of the cell and out the other side, any foreign material on the inside or outside of the cell will interfere and diminish accuracy. Therefore, cells must be very clean and free of scratches. Before testing, inspect cells, and if necessary, use a soft tissue and ordinary distilled water to gently wipe fingerprints, dirt, and water spots off of the outside of a cell, and a Q-tip to remove old residue on the inside. During testing, there should be no sediment floating in the liquid and no residue or bubbles on the walls (bubbles which form during a test can be eliminated by slowly tilting the cell back and forth so that the liquid washes them away without disturbing the bottom sediment; bubbles can cause a 10% error!) The outside of the cell should be dry, free of drips.

- Similarly, suspended solids, such as sediment and algae, can interfere with the colorimeter light. Record the clarity ('clear', 'cloudy', etc.) of each jar in the log book.
- Because the colorimeter uses a precise light, the cap must be securely placed on the colorimeter during tests.
- Measure liquids accurately. See following paragraph on the meniscus.

Meniscus

Proper measurement of liquids is crucial! The meniscus is the curved surface that forms on a liquid in a container such as a pipette or cell. The proper measurement is with the meniscus resting on the measurement line, viewed on a flat surface at eye-level:



Colorimeter Hints

- Place cells in colorimeter with the diamond marking toward you.
- If colorimeter reading exceeds the indicated test range by more than 20%, then a mistake may have been made. Carefully re-do that entire test, and if the reading is still high, then contact the test coordinator for instructions on further diluting the sample.

Piston Pipette Operation

The pipette is operated by means of the push button at the end of the handle. Maximum accuracy can be achieved by carrying out the following steps:

1. Move the piston up or down slowly and evenly.
2. Do not allow the push button to spring back into its original position.
3. Use light pressure to fit a new tip to the cone, turning the tip slightly as you do so.
4. Make sure that the tip is firmly in position on the tip cone and there are no foreign bodies between the tip and the cone.
5. Wet the tips with the liquid by repeatedly filling and emptying them.
6. Hold the pipette almost vertical (no more than 10° from the vertical) while liquid is being drawn into the tip.
7. The temperature of the pipette and the tip should match that of the liquid.

Standard Technique

1. Press the push button to the first pressure point.
2. Take the tip out of the liquid and touch it against the inside wall of the vessel to remove excess liquid.
3. Place the tip against the inside wall of the vessel and press lightly and evenly on the push button until the first pressure point is reached. Hold the pipette in this position. After about one second, press the push button until the second pressure point is reached. This completely empties the tip.
4. Allow the push button to return to its rest position.

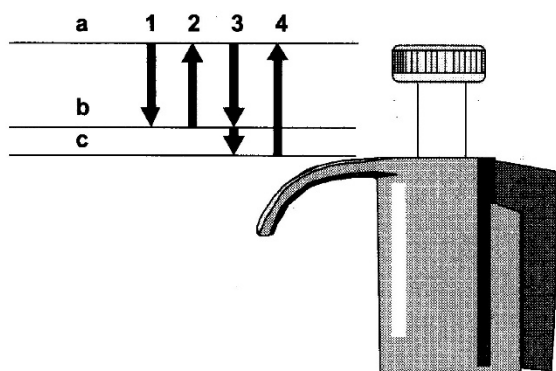


Figure 3 Standard technique

a Rest position	c Second pressure point
b First pressure point	

Water Testing Procedures

Revised 2018

Pretest Activities

Before conducting any tests, record from each sample the collection time, temperature, sample collector and sample appearance.

Also, remove conductivity meter from package and soak the electrode for at least 30 minutes in regular distilled water. Conductivity will be measured after the ammonia test is conducted to allow the samples to reach room temperature.

Temperature & pH

Temperature

The test temperature will be measured for each sample at the same time the conductivity is measured.

pH

1. Verify the accuracy of the pH meter:
 - a. Pour yellow "pH 7.0 Standard" liquid into the clean, dry, 100 ml plastic cup. The liquid is a pH-neutral buffer solution.
 - b. Turn pH meter On, uncap, and immerse electrode into liquid. Reading should eventually stabilize around 7.0 ± 0.3 . In the pH section of the worksheet, write the reading on the line marked "Standard".
 - c. Return liquid to its glass container and cap.
 - d. Rinse plastic cup with ordinary distilled water.
 - e. Thoroughly rinse the pH meter with ordinary distilled water and gently shake dry.
2. Measure the pH of each river sample:
 - a. For each sample, submerge electrode in jar. After the reading stabilizes (20-30 seconds), record it on the worksheet. River samples are unbuffered solutions, so if the reading continues to fluctuate slightly, record a median value. Readings will normally be between 6.9 - 8.1, and should never exceed 7.0 ± 1.5 . (Note that pH < 7.0 indicates acidity and pH > 7.0 indicates alkalinity.)
 - b. To avoid cross-contamination, rinse the bottom of the pH meter with ordinary distilled water and gently shake dry after each reading.
 - c. After the last sample, rise with ordinary distilled water and gently shake dry. Turn pH meter Off and recap when dry.

PHOSPHORUS

Equipment	
Cells	Test "N Tube vial (one use, disposable)
16 mm Cell Holder (only used for this test)	
Micro funnel	
Reagents:	
Liquid	Phosphate Standard Solution Ultra-pure distilled water Isopropyl alcohol
Powder Pillow	PhosVer 3 Phosphate Reagent

Orthophosphate, Method 8048. Range = 0.06 to 5.00 mg/L PO₄³⁻.
Test 'N Tube Vials

Procedure:

1. Turn on DR900 Colorimeter, the last test results will show on the screen. Press button under "Options", use down scroll arrow to highlight "Favorites/User Programs" and press button under Select. A list of test programs will be shown on screen.
2. Scroll down to highlight "**535 P React. PV TNT**", press select. The screen will show mg/l, PO₄, time and date.
3. Insert the 16-mm cell holder into colorimeter.

SAMPLE ANALYSIS:

(Note: each river sample will be used as its own blank prior to addition of the reagent and analysis)

4. Add 5 mL of river water (regardless of sample location) to the Reactive Phosphorus Test N' Tube Vial.
5. Put the cap on the vial, invert to mix, clean the vial with lens cloth. Label the vial using a Sharpie.
6. Insert the vial into the 16mm cell holder and push **ZERO**. The display should show 0.00 mg/L PO₄³⁻.
7. Remove the vial from the colorimeter, and using the micro funnel, add one PhosVer 3 Phosphate Reagent powder pillow to the vial, cap and shake for 20 seconds to mix. The powder will not dissolve completely.

8. Start the instrument timer by pressing the “options” button, “start timer” will be first on list of options, press “select” button, “Timer 1: 02:00” will be highlighted, press “select button to activate timer.
9. When the timer expires, clean the vial, insert into 16-mm cell holder, push READ button. Results show in mg/L PO_4^{3-} . Record reading on worksheet.

Preparation of Standard Solution: used to validate the test procedure, reagents and instrument.

10. Using 5 mL of 1mg/L phosphate standard solution follow steps 4 through 9 above. The standard reading should read between 1.10 and 0.9 mg/L. Consider redoing the test if this reading is not achieved.
11. Record the readings on the worksheet and dispose of the used Test “N Tube vials in the trash.

NITRATE

E q u i p m e n t	
Cells	20 mL glass sample cells
100-ml volumetric flask	
Reagents:	
Liquid	Nitrate Nitrogen Standard Solution Ultra-pure distilled water Isopropyl alcohol
Powder Pillow	NitraVer 5 Nitrate Reagent (2 per sample)

Cadmium Reduction method #8171. Range = 0.1 to 10.0 mg/L N NO₃⁻-N MR.

Standard Solution Preparation: The standard is used to validate the test procedure, reagents and instrument.

Prepare a 5.0 mg/L nitrate-nitrogen standard solution by adding 5.0 ml of 100 mg/l nitrate-nitrogen standard solution into the 100-ml volumetric flask. Dilute to the mark with ultra-pure distilled water, insert glass stopper, invert several times to mix.

Procedure:

1. Turn on DR900 Colorimeter, the last test results will show on the screen. Press button under “Options”, use down scroll arrow to highlight “Favorites/User Programs” and press button under Select. A list of test programs will be shown on screen.
2. Scroll down to highlight “**353 N Nitrate MR PP**”, press select. The screen will show mg/l, NO₃-N, time and date.
3. Remove the 16-mm cell holder that was used in the phosphorus test.

SAMPLE ANALYSIS:

(Note: Two 20 ml sample cells will be used for each river sample analysis, one for the blank and one for addition of reagents and analysis.)

4. Add 10 mL of river water (regardless of sample location) to each of the two 20 ml sample cells, label one BLANK and one with the sample site (e.g. WB 1) with a Sharpie. Do not get the ink on the white vial markings, isopropyl alcohol will not remove ink from these locations.

5. Add the contents of one NitraVer 5 Nitrate Reagent powder pillow to each sample vial marked for the river sample. Put the lid on the sample cell.
6. Start the instrument timer by pressing the “options” button, “start timer” will be first on list of options, press “select” button, “Timer 1: 01:00” will be highlighted, press “select” button to activate timer.
7. Shake the sample cells vigorously until the timer expires. Some of the solid material will not dissolve. Undissolved powder will not affect results.
8. Start the instrument timer (Timer 2) for a 5-minute reaction time. An amber color shows if nitrate is present.
9. When the timer expires, clean the BLANK vial, insert into colorimeter, make sure the “white diamond” on the sample vile is aligned with the notch on the bottom of the vial slot. Push ZERO button. The display should show 0.0 mg/L NO₃-N.
10. Clean the SAMPLE vial, insert into colorimeter, make sure the “white diamond” on the sample vile is aligned with the notch on the bottom of the vial slot. Push READ button. The display shows the results in mg/L NO₃-N. Record reading on worksheet.
11. **SATANDARD ANALYSIS:** Using the standard solution, follow steps 4 through 10 above. The standard reading should read between 4.8 and 5.2 mg/L. Consider redoing the test if this reading is not achieved.
12. Record the readings on the worksheet and pour contents of test cell into waste container.

At this time during the testing, move the conductivity meter from soaking in distilled water to 50 milliliters of 1413 uS Conductivity Standard solution.

A M M O N I A

E q u i p m e n t	
Cells	20 mL glass sample cells
100-ml volumetric flask	
Reagents:	
Liquid	Ammonia Nitrogen Standard Solution Ultra-pure distilled water Isopropyl alcohol
Powder Pillow	Ammonia Cyanurate Reagent Powder Pillow, 10-ml Ammonia Salicylate Reagent Powder Pillow, 10-ml

Salicylate method #8155. Range = 0.01 to 0.50 mg/L NH₃-N.

Standard Solution Preparation: The standard is used to validate the test procedure, reagents and instrument.

Prepare a 0.40 mg/L ammonia nitrogen standard solution by adding 4.0 ml of 10 mg/l Ammonia-nitrogen standard solution into the 100-ml volumetric flask. Dilute to the mark with ultra-pure distilled water, insert glass stopper, invert several times to mix. (don't forget to dial the pipette back to 5 ml before preparing samples)

Procedure:

- Turn on DR900 Colorimeter, the last test results will show on the screen. Press button under "Options", use down scroll arrow to highlight "Favorites/User Programs" and press button under Select. A list of test programs will be shown on screen.
- Scroll down to highlight "**385 N Ammonia, Salic**", press select. The screen will show mg/l, NH₃-N, time and date.

6. **Prepare the BLANK:** Fill a 20 mL sample cell with 10 mL of ultra-pure deionized water. Only one blank will be necessary for this analysis. Reagents will NOT be added to the BLANK.
7. **Prepare the SAMPLE:** Add 10 mL of river water to a 20 ml sample cells, label with a Sharpie if necessary.
8. Add the contents of one Ammonia Salicylate powder pillow to each sample cell. Put the lid on the sample cell and shake to dissolve the reagent.
9. Start the instrument timer for 3 minutes by pressing the first option, "Timer 1: 03:00".
10. After the timer expires, add the contents of one Ammonia Cyanurate powder pillow to each sample cell. Close the sample sell and shake to dissolve the reagent.
11. Start the instrument timer (Timer 2) for a 15-minute reaction time. A green color shows if ammonia is present.
12. When the timer expires, clean the BLANK vial, insert into colorimeter, make sure the "white diamond" on the sample vile is aligned with the notch on the bottom of the vial slot. Push ZERO button. The display shows 0.0 mg/L NH₃-N.
13. Clean the SAMPLE cell, insert into colorimeter, make sure the "white diamond" on the sample vile is aligned with the notch on the bottom of the vial slot. Push READ button. The display shows results in mg/L NH₃-N. Record reading on worksheet.
13. **SATANDARD ANALYSIS:** Using the standard solution, follow steps 4 through 10 above. The standard reading should read between 0.38 and 0.42 mg/L. Consider redoing the test if this reading is not achieved.
14. Record the readings on the worksheet and pour contents of test cell into waste container.

CONDUCTIVITY (CHLORIDE)

E q u i p m e n t	
Conductivity Meter	
Liquid	Standard Solution (1413 uS)

Calibration

1. At this point the meter should have been soaking in the 1413 uS standard solution. If not probe in beaker with 50 mL standard solution, allow to sit for 15 minutes.
2. Power the meter, it will cycle through several screens and stop in conductivity mode. uS should be in the upper left corner of screen with temperature in °C on bottom.
3. Press and hold the HLD/CAL button for at least 2 seconds. The conductivity value and the 'CAL' icon on the display will flash for a few moments then the 'CAL' will stop flashing.
4. Press the up or down arrow to adjust the displayed conductivity value so that it matches the standard solution value (1413 uS).
5. Press the SET button to set the calibration value.

Operation

1. After calibrating the meter, rinse the probe with regular distilled water and place in jar of river water sample.
2. When dipping the probe into the sample, be sure to eliminate air bubbles trapped in the probes slot by gently stirring the probe in the sample.
3. The uS icon will flash on the meter's display while stabilization is taking place in the measurement mode. When stabilization is achieved, the meter's icon will stop flashing.
4. Record this uS value on the work sheet next to the chloride results.
5. Record the temperature in °C at this time also.
6. Rinse probe with regular distilled water and proceed to next sample.

Test Wrap-Up

After all testing is complete:

1. Pour the remaining river water down the drain and rinse the sample jars and lids thoroughly with tap water. Make sure no sediments remain in the jars. The sample jars can be run through the dishwasher if **phosphate free** detergent is used.
2. Rinse all equipment in regular tap water then place in a wash tub or bucket with a small amount of liquinox (4-5 drops) and soak for a few minutes. Avoid scratching the sample cells.
3. Rinse again with tap water to remove soapy water then rinse with ordinary distilled water.
4. Air-dry in a dry area free of vapors before storing. Cells should literally be spotless; clean equipment is a courtesy to the next tester.
5. Practice good hygiene: wash hands after contact with reagents and river water. While river water is not toxic, it may contain pathogens from natural and manmade sources (river water in most cities contains treated sewage).
6. Discard table liner in the trash, not recycling. Wash table and sink thoroughly.
7. Copy all data from worksheet into official log book, using the exact format as prior entries, and then e-mail the data (scanning worksheets work best) to Bob Barbieri at bob-barbieri@comcast.net so that it can be posted on the River Prairie Group's Internet website:

Double-check data in email and log book against worksheet, and then file worksheet in three-ring binder.
8. Perform an inventory. Contact test coordinator regarding shortages of equipment, reagent, or ultra-pure distilled water. Also, if the next scheduled test is a quarterly one, verify that sufficient jars and lids (at least nine) are available for collectors (due to traditional staffing limitations, SC and EB sites are tested quarterly and WB sites are tested monthly).
9. Contact the next tester to make arrangements for equipment delivery. In the meantime, do not store chemicals in an area where they will be subjected to extreme heat or freezing temperatures (e.g., unheated garage in winter); 50° - 80° is ideal. Keep out of reach of children!