

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**
- **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
- *spectrophotometer* - computer which calculates analyte concentration based on its color

General Testing Procedure

The test methods described herein involve titration with industry-standard reagents, and colorimetric analysis with a Hach DR/2010 Spectrophotometer. 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** (distilled water), a **STANDARD** (“standard” solution), and the river samples. The **BLANK** calibrates the spectrophotometer, 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.

Since analyte concentrations in river water often exceed the spectrophotometer’s range, most of these tests involve diluting the river samples with distilled water and then multiplying them back up to their final values on the worksheet. Write final values in the worksheet’s rightmost column (marked “Result”) and also in the official log book. Double-check every calculation!

Other test results, such as 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.

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 (e.g., the black rubber caps for the 25 mL plastic cells leak slightly).
- 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, it is not used for the chloride test (which consumes a large amount of distilled water) nor 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.

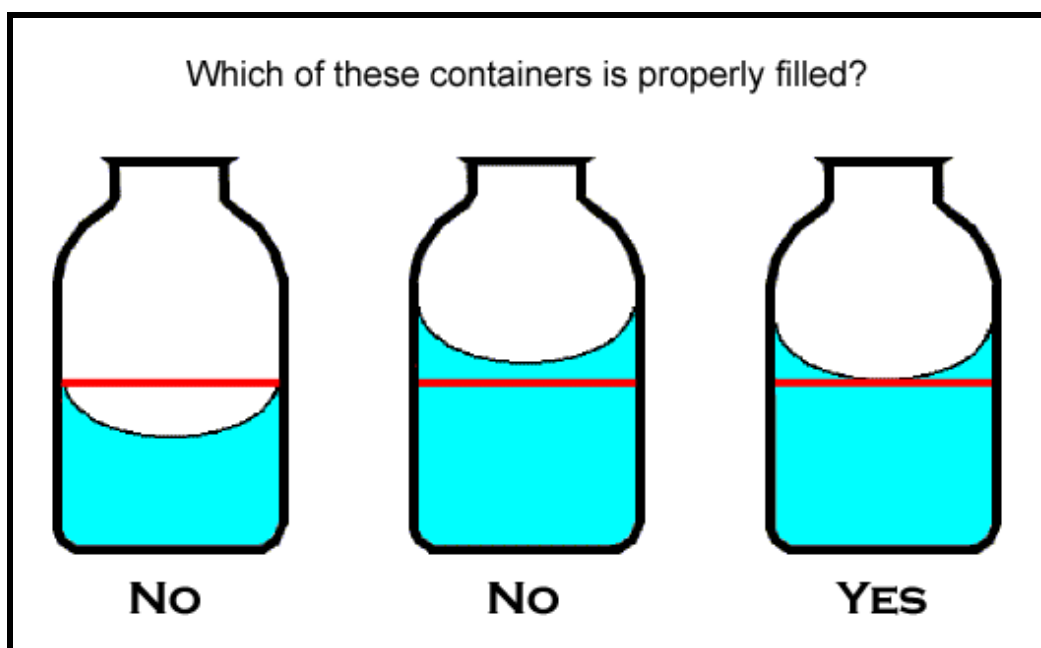
Keys to Accurate Tests

- 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 spectrophotometer 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 spectrophotometer's light. Record the clarity ('clear', 'cloudy', etc.) of each jar in the log book.
- Because the spectrophotometer uses a precise light, the lid must remain closed 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:



Spectrophotometer Hints

- Place cells in spectrophotometer with the marking to your left.
- In this manual, spectrophotometer keys are indicated by this special font **ABC**
- Tests can be performed in any sequence. However, the small, plastic 10 mL cell riser must remain in the spectrophotometer only for the phosphorus test.
- After turning the spectrophotometer on, allow it to warm up for a moment.
- Unlike a typewriter or computer, the spectrophotometer **SHIFT** key is pressed once and released. It is not pressed simultaneously with another key.
- If spectrophotometer 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.
- After pressing **ZERO**, the spectrophotometer will sometimes beep and display "Low Battery". If this occurs, simply press **ZERO** again.



Water Testing Procedures



Temperature & pH

Temperature

For each sample, insert thermometer into jar and then record temperature in the worksheet's Temperature section under the column marked "Test". In the same section, copy the river temperature (which the collector wrote on the jar) to the column marked "Collection". Always specify celsius or fahrenheit.

To avoid cross-contamination, rinse thermometer with ordinary distilled water and gently shake dry after each reading.

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 $\text{pH} < 7.0$ indicates acidity and $\text{pH} > 7.0$ 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	10 mL glass cells
Reagents:	
Liquid	Phosphate Standard Solution Ultra-pure distilled water
Powder Pillow	PhosVer 3 Phosphate Reagent

Orthophosphate/Ascorbic Acid method #8048. Range = 0 to 2.50 mg/L PO₄³⁻ PV.

Procedure:

- Enter the program number for phosphorus by pressing **SELECTPRGM** and then **490 ENTER**
- The display will read “dial nm to 890”. Rotate the wavelength dial until display reads “890 nm”. When the correct wavelength is dialed in, display will read “Zero Sample”, then “mg/L PO₄³⁻ PV”.
- Insert the small, black, plastic 10 mL cell riser into the spectrophotometer.
- Prepare all cells:
 - Add 10 mL of ultra-pure distilled water to a cell. This is the **BLANK**.
 - Add 10 mL of Phosphate Standard Solution to a cell. This is the **STANDARD**.
 - For each river sample:
 - All sites except EB4: add 2 mL of river water to a cell and then fill to 10 mL with ultra-pure distilled water
 - EB4: add 10 mL of river water to a cell, undiluted
 - Add one PhosVer 3 Phosphate Reagent powder pillow to each of the cells in 4a-4c and swirl immediately to mix.
- Press **SHIFT** then **TIMER** and a two-minute reaction period will begin. A blue color will form if phosphorus is present.
- After the timer beeps, insert the **BLANK** into the spectrophotometer and close the lid. Press **ZERO** and the display will read “Zeroing.....” then “0.00 mg/L PO₄³⁻ PV”. The spectrophotometer is now calibrated. Remove the **BLANK**.

7. Insert the **STANDARD** into the spectrophotometer, close the lid, and press **READ**. In the Phosphorus section of the worksheet, write the reading on the line marked "Standard". If the reading is greater than 1.10 or less than 0.90, consider re-doing the entire test.
8. Insert each river sample into the spectrophotometer, close the lid, press **READ**, and then write the reading on the worksheet. Ideally, all river readings should fall between the **BLANK** (zero) and the method's upper limit (2.50). If not, consider re-doing the sample, or contact the test coordinator.
9. On the worksheet, perform the specified arithmetic and write the answer in the column marked "Result" and also in the log book.
10. Rinse the 10 mL cells with ordinary distilled water until they are perfectly clean. A Q-tip will remove reagent sediment stuck to the bottom.
11. Remove the small, plastic 10 mL cell riser from the spectrophotometer and set aside.

NITRATE

E q u i p m e n t	
Cells	25 mL plastic cells
Reagents:	
Liquid	Nitrate Nitrogen Standard Solution Ultra-pure distilled water
Powder Pillow	NitraVer 5 Nitrate Reagent

Cadmium Reduction method #8171. Range = 0 to 4.5 mg/L N NO₃⁻ -N MR.

Procedure:

1. Enter the program number for nitrate by pressing **SELECTPRGM** and then **353**
ENTER
2. The display will read “dial nm to 400”. Rotate the wavelength dial until display reads “400nm”. When correct wavelength is dialed in, display will read “Zero Sample”, then “mg/L NO₃⁻ -N MR”.
3. Prepare all cells:
 - a. Add 25 mL of ultra-pure distilled water to a cell. This is the **BLANK**.
 - b. Add 25 mL of Nitrate Nitrogen Standard Solution to a cell. This is the **STANDARD**.
 - c. For each river sample:
 - All WB sites: add 5 mL of river water to a cell and then fill to 25 mL with ultra-pure distilled water
 - All SC & EB sites except EB4: add 10 mL of river water to a cell and then fill to 25 mL with ultra-pure distilled water
 - EB4: add 25 mL of river water to a cell, undiluted
 - d. Add one NitraVer 5 Nitrate Reagent powder pillow to each of the cells in 3a-3c. Cap with a black rubber stopper and shake for one minute.
4. Press **SHIFT** then **TIMER** for a one-minute “shake” timer.
5. Press **SHIFT** then **TIMER** and let cells rest for five minutes. An amber color will form if nitrate is present.

6. After the timer beeps, insert the **BLANK** into the spectrophotometer and close the lid. Press **ZERO** and the display will read “Zeroing.....” then “0.0 mg/L NO₃⁻ -N MR”. The spectrophotometer is now calibrated. Remove the **BLANK**.
7. Insert the **STANDARD** into the spectrophotometer, close the lid, and press **READ**. In the Nitrate section of the worksheet, write the reading on the line marked “Standard”. If the reading is greater than 1.1 or less than 0.9, consider re-doing the entire test.
8. Insert each river sample into the spectrophotometer, close the lid, press **READ**, and then write the reading on the worksheet. Ideally, all river readings should fall between the **BLANK** (zero) and the method’s upper limit (4.5). If not, consider re-doing the sample, or contact the test coordinator.
9. On the worksheet, perform the specified arithmetic and write the answer in the column marked “Result” and also in the log book.
10. Rinse the 25 mL cells and rubber stoppers with ordinary distilled water until they are perfectly clean. A Q-tip will remove reagent sediment stuck to the sides and bottom of the cells.

AMMONIA

E q u i p m e n t	
Cells	25 mL plastic cells
Reagents:	
Liquid	Nitrogen Ammonia Standard Solution Mineral Stabilizer Polyvinyl Alcohol Dispersing Agent Nessler Reagent Ultra-pure distilled water
Powder Pillow	none

Nessler method #8038. Range = 0 to 2.5 mg/L NH₃ -N Ness.

Procedure:

Do not allow more than 5 minutes to elapse between steps 3f and 7.

1. Enter the program number for ammonia by pressing **SELECTPRGM** and then **380 ENTER**
2. The display will read “dial nm to 425”. Rotate the wavelength dial until display reads “425nm”. When correct wavelength is dialed in, display will read “ Zero Sample”, then “mg/L NH₃ -N Ness”.
3. Prepare all cells:
 - a. Add 25 mL of ultra-pure distilled water to a cell. This is the **BLANK**.
 - b. Add 25 mL of Nitrogen Ammonia Standard Solution to a cell. This is the **STANDARD**.
 - c. For each river sample:
 - All sites: add 25 mL of river water to a cell, undiluted
 - d. Add 3 drops of Mineral Stabilizer to each of the cells in 3a-3c. Cap with a black rubber stopper and invert several times to mix.
 - e. Add 3 drops of Polyvinyl Alcohol Dispersing Agent to each of the cells in 3a-3c. Cap with a black rubber stopper and invert several times to mix.
 - f. Using its eyedropper, add 1 mL of Nessler Reagent to each of the cells in 3a-3c. The “1 mL” marking on the glass eyedropper may be difficult to see; typically, 1 mL

is drawn into the eyedropper if the rubber is pinched fully and released. Cap each cell with a black rubber stopper and invert several times to mix.

4. Press **SHIFT** then **TIMER** and a one-minute reaction period will begin. A yellow color will form if ammonia is present.
5. After the timer beeps, insert the **BLANK** into the spectrophotometer and close the lid. Press **ZERO** and the display will read "Zeroing....." then "0.00 mg/L NH₃ -N Ness". The spectrophotometer is now calibrated. Remove the **BLANK**.
6. Insert the **STANDARD** into the spectrophotometer, close the lid, and press **READ**. In the Ammonia section of the worksheet, write the reading on the line marked "Standard". If the reading is greater than 1.10 or less than 0.90, consider re-doing the entire test.
7. Insert each river sample into the spectrophotometer, close the lid, press **READ**, and then write the reading on the worksheet. Ideally, all river readings should fall between the **BLANK** (zero) and the method's upper limit (2.50). If not, consider re-doing the sample, or contact the test coordinator.
8. Rinse the 25 mL cells and rubber stoppers with ordinary distilled water until they are perfectly clean.

CHLORIDE

E q u i p m e n t	
Cells	25 mL plastic cells
Reagents:	
Liquid	Chloride Reference Standard Solution Mercuric Thiocyanate Solution Ferric Ion Solution Ordinary distilled water
Powder Pillow	none

Mercuric Thiocyanate method #8113. Range = 0 to 20 mg/L Cl⁻.

Procedure:

The pipette volumes in this test are tiny, requiring a steady hand and careful measurement. Use of the tri-valve bulb is recommended.

1. Enter the program number for chloride by pressing **SELECTPRGM** and then **70**
ENTER
2. The display will read “dial nm to 455”. Rotate the wavelength dial until display reads “455nm”. When correct wavelength is dialed in, display will read “Zero Sample”, then “mg/L Cl⁻”.
3. Prepare a diluted chloride standard solution:
 - a. The chloride standard solution must be diluted to fall within range.
 - b. Add 5 mL of Chloride Reference Standard Solution to the 500 mL flask (has very long neck). Then fill to 500 mL (line on neck) with ordinary distilled water. Cap the flask and swirl to mix.
4. Prepare all cells:
 - a. Add 25 mL of ordinary distilled water to a cell. This is the **BLANK**.
 - b. Add 25 mL of the diluted chloride standard solution (flask) to a cell. This is the **STANDARD**.
 - c. For each river sample:
 - All sites: add 1 mL of river water to a cell and then fill to 25 mL with ordinary distilled water

- d. Add 2 mL of Mercuric Thiocyanate Solution to each of the cells in 4a-4c. Cap with a black rubber stopper and invert several times to mix.
 - e. Add 1 mL of Ferric Ion Solution to each of the cells in 4a-4c. Cap with a black rubber stopper and invert several times to mix.
5. Press **SHIFT** then **TIMER** and a two-minute reaction period will begin. An orange color will form if chloride is present.
 6. After the timer beeps, insert the **BLANK** into the spectrophotometer and close the lid. Press **ZERO** and the display will read “Zeroing.....” then “0.0 mg/L Cl⁻”. The spectrophotometer is now calibrated. Remove the **BLANK**.
 7. Insert the **STANDARD** into the spectrophotometer, close the lid, and press **READ**. In the Chloride section of the worksheet, write the reading on the line marked “Standard”. If the reading is greater than 11.0 or less than 8.0, consider re-doing the entire test.
 8. Insert each river sample into the spectrophotometer, close the lid, press **READ**, and then write the reading on the worksheet. Ideally, all river readings should fall between the **BLANK** (zero) and the method’s upper limit (20.0). If not, consider re-doing the sample, or contact the test coordinator.
 9. On the worksheet, perform the specified arithmetic and write the answer in the column marked “Result” and also in the log book.
 10. Rinse the 25 mL cells and rubber stoppers with ordinary distilled water until they are perfectly clean.

Test Wrap-Up

After all testing is complete:

1. Rinse all equipment, including jars and lids, with ordinary distilled water and 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. Do not wash with soap or detergent!
2. 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).
3. Discard table liner in the trash, not recycling. Wash table and sink thoroughly.
4. Insert the small, plastic 10 mL cell riser back in the spectrophotometer.
5. Copy all data from worksheet into official log book, using the exact format as prior entries, and then e-mail the data to the following people so that it can be posted on the River Prairie Group's Internet website:

Paul Mack <umrgrad@sbcglobal.net>
Frank Orto <forto@aol.com>

Double-check data in email and log book against worksheet, and then file worksheet in three-ring binder.

6. 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).
7. 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!

Document revision history:

- original document by Duncan Wiedemann
- updated April 2001, January 2002, January 2003, and January 2009 by Paul Mack