LABORATORY TESTING

Sampling and laboratory analysis of source water and finished water are part of every water treatment plant operator's daily routine. Sampling and testing are necessary to gather data on pH, alkalinity, turbidity, temperature, chlorine residuals, hardness, tastes and odors, and bacteriological contamination. There are requirements, both regulatory and operational, that dictate how and when these samples are collected, analyzed, and reported.

GRAB SAMPLES

Grab samples are single point real-time samples. A temperature or pH reading would be a grab sample. A chlorine residual test would also be a grab sample. Grab samples give a snapshot of a specific condition at a specific point in time and at a specific point in the system.

COMPOSITE SAMPLES

Composite sampling consists of a collection of numerous individual discrete samples taken at regular intervals over a period of time, usually 24 hours. The material being sampled is collected in a common container over the sampling period. There are only a few instances where composite samples are collected in water treatment. It is sometimes done when large systems have multiple processes run in parallel. EPA designates what water parameters require composite sampling.

GENERAL LABORATORY SAFETY

The following items are some of the general issues that pertain to laboratory operations:

- Make sure all lab chemicals are properly labeled and have not exceeded their expiration dates.
- Make sure the MSDS information and safety equipment required for each different chemical is available.
- Combustible chemicals must be store in approved fire cabinets.
- Acids and Bases must also be stored in approved cabinets
- Some chemicals have brown glass bottles because they are light sensitive. Store them in closed cabinets.
- Make sure that all electrical equipment is properly grounded.
- Be careful when handling glassware, especially when connecting glassware to rubber hoses. Use water as a lubricant to avoid injury from breakage.
- Always dilute acids and bases by pouring them into the dilution water. Adding water to an acid or base can cause enough heat to boil the water.

pH METERS

A pH test can be run using a meter instead of laboratory chemical analysis. A pH meter has an electrode that contains a fluid called an electrolyte. The electrolyte is held in the electrode by a semi-permeable membrane. The membrane allows the chemical being analyzed to react with the electrolyte. This generates an electrical current proportional to the concentration of hydrogen ions in the sample. This current is converted to a numerical display on the meter.

A pH meter must be standardized before each use. They are standardized using buffer solutions of a known pH, usually 4, 7, and 10. A buffer solution has chemicals that neutralize acids and bases while maintaining a constant pH. If a drop of acid is added to a pH 7.0 buffer solution, the pH will remain at 7.0. These solutions allow the operator to correct any deviation in the pH meter by adjusting it to match the pH of the buffer solution. Fresh buffer solution should be used each time the unit is standardized. When the sample pH is below 7, the 4 and 7 pH buffers should be used. When the pH is above 7, the 7 and 10 pH buffers should be used. Electrodes must be rinsed with distilled water between readings. Some should be stored in distilled water when not in use. Others may require that the electrode be stored in a buffered pH 4.0 solution that contains potassium chloride. The electrode membrane can be cleaned with isopropyl alcohol when slime or scale builds up on it.

A field-test for pH may include color strips or indicator chemicals. Both contain chemical indicators that change color as the pH changes. Chemical indicators are also used in laboratory tests that require titration, like alkalinity and chlorine residual.

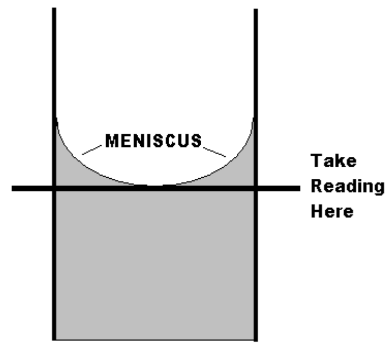
ALKALINITY TESTING

Alkalinity is the amount of carbonates, bicarbonates and hydroxides in water. Alkalinity acts as a buffer in water. It minimizes fluctuations in pH and plays an important role in maintaining chemical stability in water. The addition of water treatment chemicals can cause a change in the alkalinity. Coagulant chemicals remove hydroxides that result in a lower pH and alkalinity. The addition of chemicals like lime, calcium oxide (quick lime), or sodium carbonate (soda ash) will increase the alkalinity and pH of the water.

Alkalinity is determined by a chemical titration. A titration test involves adding a chemical indicator and titrating by adding a neutralizing chemical until a certain endpoint, pH, or color change is achieved. The chemical solution used in the titration must be standardized to a specific strength and the amount needed to reach the endpoint must be accurately measured. A burette is used to add the titrant chemical until the color change is achieved.

The strength of the titrant chemical is expressed as its “Normality”. The titrant for the alkalinity test is sulfuric acid. A standard solution of 0.02 Normal sulfuric acid is used (also written as 0.02*N*, 1/50*N* or *N*/50). The acid will neutralize the alkalinity and cause the pH to drop. The accuracy of the beginning and ending burette readings is important. A curved fluid surface, called a meniscus, is formed by the surface tension between the fluid and the glass burette tube. The reading must be taken at the bottom of the meniscus.

The indicators that are used for the titration are methyl orange and phenolphthalein. Both of these chemicals create a color change at a specific pH. Phenolphthalein has an endpoint or color change (pink to clear) at a pH of 8.3. Methyl orange has an endpoint (yellow to red) at pH 4.3. The amount of acid needed to reach each endpoint is recorded and used to calculate the alkalinity. The results are recorded as “P” (phenolphthalein) alkalinity and “T” (total) alkalinity. The relationship between the two values is used to calculate the alkalinity as hydroxide, carbonate, and bicarbonate.



Read the fluid level at the bottom of the meniscus

MICROBIOLOGICAL TESTING

Microbiological tests are conducted using one of three testing methods. The Multiple Tube Fermentation (MTF) test is also sometimes called the Most Probable Number (MPN) test. It is the oldest method for determining the probable number of coliform bacteria in a sample. It is seldom used today because it takes longer to get results and is more expensive than the other tests. The test uses 15 to 30 test tubes (in sets of five) that are inoculated with different decimal dilutions of the sample (like 10 ml, 1 ml, and 0.1 ml). Lactose lauryl sulphate tryptose (LST) broth is used as the incubation media. The MTF sample tubes are incubated at $35^{o} \pm 0.5$ C for up to 48 hours. Any tubes that produce gas are then transferred to EC (E. Coli) broth and incubated for another 24 hours at $44.5^{o} \pm 0.2$ C. Any tube that produces gas is noted and coded for each set of tubes. A table of most probable numbers is used with the coded results to determine the MPN index. The MPN table uses a geometric mean to determine the most probable number of bacteria.

The membrane filter test is easier to run and results are obtained by counting individual colonies on the membrane. These are visible without the need for magnification. A sample is drawn through the filter pad where the bacteria are trapped on the filter. The filter is then incubated in a nutrient broth at a temperature that allows the coliform bacteria to grow on the filter pad. After 24 hours, the individual bacteria that were caught on the filter will grow into colonies that can be visually counted using an optical scope. Although the procedure is similar total coliform and fecal coliform use different nutrient media and incubation temperatures.

Parameter	Total Coliform	Fecal Coliform
Media	M-Endo Broth	M-FC Broth
Incubation Time	24 $\pm$ 2 hours	24 $\pm$ 2 hours
Temperature	$35^o \pm 0.5$ C	$44.5^o \pm 0.2$ C
Colony Color	Red w/Grn. Metallic Sheen	Blue

The third test is the MMO-MUG or colilert test. It is not a test that can be used to quantify the number of colonies per sample. It simply indicates the presence or absence of coliform bacteria in the sample. The test is run by adding the 100 ml sample to a flask containing the media broth and incubating it at 35^o +/- 0.5C. The sample is incubated for 24 hours. A yellow color change indicates the presence of coliform. If the yellow color has a florescent glow under a UV light it is also fecal coliform positive.

JAR TESTING

Changes in water characteristics or chemistry can have a dramatic effect on a chemical precipitation treatment process. A jar test is used to simulate the coagulation, flocculation and sedimentation processes in the treatment plant. A jar test is done using six different samples of the raw water at the same time. A different dosage of coagulant is added to each of the jars. This provides the treatment plant operator with an opportunity to test several new chemical doses at one time. Mixers are used to create conditions similar to coagulation and flocculation. The sample with the best results can then be used to adjust the chemical feed systems to achieve the new dosage.

TURBIDITY TESTING

The effluent from the filtration process must be continuously monitored for turbidity. Turbidimeters, also called nephelometers, are used to monitor the effluent of each filter. A turbidimeter passes abeam of light through a sample of water. If there is no turbidity, the beam of light passes through the sample unaltered. A photometric cell on the opposite side of the sample measures the light intensity. If there are turbidity particles in the sample, they will reflect some of the light and reduce the intensity of the beam as it passes through the sample. The instrument then calculates the amount of turbidity (NTU's) by measuring the loss of beam intensity and the amount of light reflected off the turbidity particles.

ADVANCED STUDY QUESTIONS

1. What is a pH buffer used for?
2. What would you be doing if you were reading a meniscus?
3. What kind of test is the MPN method?
4. What are the two indicators used in the alkalinity test?
5. What does the term "normality" of a chemical solution mean?

ADVANCED SAMPLE TEST QUESTIONS

1. Which of these chemicals will not raise the alkalinity of water?

   A. Calcium carbonate
   B. Sodium hydroxide
   C. Sodium chloride
   D. Calcium oxide

2. Fecal coliform colonies on a membrane filter will be blue.

   A. True
   B. False

3. The endpoint for an alkalinity titration using phenolphthalein should be a pH of:

   A. 4.3
   B. 6.8
   C. 7.2
   D. 8.3

4. Always add acid to the water when diluting an acid.

   A. True
   B. False

5. What is the correct incubation temperature for total coliform testing?

   A. $35^{o} \pm 0.5$ C
   B. $44.5^{o} \pm 0.2$ C
   C. 103^{o} F
   D. 135^{o} F

6. Which media is used for fecal coliform in the membrane filter test?

   A. M-Endo Broth
   B. M-FC broth
   C. LST broth
   D. None of the above

7. What should the normality of the sulfuric acid be for an alkalinity titration?

   A. 0.02*N*
   B. 1/50*N*
   C. N/50
   D. All of the above

8. Which treatment process is not simulated with a jar test?

   A. Coagulation
   B. Flocculation
   C. Sedimentation
   D. Filtration