Water and Wastewater Test Methods for Operators

Introduction

A variety of test procedures and methods is available for use by water and wastewater operators. They run the gamut from colorimetric, titrimetric, electrometric (meter & probe), turbimetric and nephlometric through demonstrative methods. Often, more than one of these methods can be utilized to measure a single unknown parameter. For example, chlorine residuals can be measured colorimetrically, titrimetrically, or electrometrically. What is the best method for your application? First, let's define these methods, give examples of each, learn the limitations of each method, and then decide what method (and, therefore, test kit and procedure) is best suited for your needs.

The key issue is to decide upon the test procedure that best meets your requirements concerning:

a) accuracy,

b) cost (initial & cost per test),

c) skill level,

d) repeatability,

e) portability,

f) decision-making value of information obtained,

g) safety and reagent disposal and,

h) reportability of results

Remember, getting the most complete, reliable, and accurate information from your testing operations will provide you with the decision-making tools to you need monitor your water or wastewater systems, make operational changes and meet permit requirements and state mandates.

Colorimetric Method

This is defined as the measurement of a parameter when the concentration is directly proportional to color development and intensity after the addition of known volume of reagent chemicals. In cases like chlorine residual, the colorimetric reaction is almost immediate, and results can be determined right away. Other tests like those for nitrates and phosphates may require 5- to 10-minute waiting periods before full color development is obtained, due to the chemistry involved.

Some unique colorimetric tests react in reverse. That is, the greater the color development, the lower the concentration of a particular substance. Examples are fluoride, and some tests for ozone.

To determine concentration, the color developed in the sample is either compared visually with manufacturer supplied standards (color comparator) or inserted into a photometer, colorimeter, or spectrophotometer to give results directly on a meter scale, or digitally via a discrete readout. Results are expressed as parts per million (ppm), milligrams per liter (mg/L), grains per gallon (gpg), etc.

Visual Comparator Limitations:

– Individual differences in one's ability to discern color intensity

– Background lighting. Most manufacturers formulate their color standards using natural daylight. Incandescent, fluorescent or direct sunlight are unacceptable and may produce errors

– Color blindness is a definite problem with visual color comparison methods

– With certain colors, it is extremely difficult to discern fine variations. Example: yellows and some hues of blue

Even with the limitations described above, visual color comparison methods are inexpensive, generally easy to use, conveniently packaged and designed for simplicity. Some visual test results are reportable for permit purposes. Check with your local inspector.

Colorimetric methods using a photometer, colorimeter, or spectrophotometer offer a unique advantage. Many meters are battery powered and conveniently packaged for portability. To briefly describe their operation: a light beam is pointed at a the sample. Depending upon the amount of color present, light will pass through the sample and be detected by a photodiode. With the aid of electronics, the results are displayed on a meter, either directly in concentration or as a percentage of light transmitted. Advantages of such instrumentation are:

– It eliminates the need for visual interpretation by a human operator.

– It eliminates concern for background lighting.

– It ultimately offers greater accuracy.

Of course, using a meter to "read" color development can initially be more expensive.

Colorimetric test methods offer on-the-spot results and can test for a variety of common substances. Tests for chlorine, iron, manganese, copper, zinc, aluminum fluoride, ozone, nitrates, phosphates, sulfides, and many more materials are available. Weigh the advantages, disadvantages and overall requirements before you make your decision.

Titrimetric Method

In this procedure a sample is taken, and reagent known as an indicator reagent is added to produce a color. A "titrant" or a reacting reagent is added drop by drop until a color change occurs. The point at which the color changes is called the endpoint.

Titrimetric methods offer a number of titrant dispensing apparatuses:

– Drop count: a calibrated dropper dispenses drops of equal size. When the endpoint is reached, the number of drops that was required is counted and multiplied by a conversion factor. Example: one drop indicates 5 ppm.

– Laboratory burets: automatic burets which are not generally portable. This dispensing apparatus has a calibrated scale on its barrel. Titrant is dispensed until the endpoint is reached. The volume used is then read from the calibrated scale, in many cases, the number of milliliters used equals the test result in ppm.

– Direct Reading Microburet: a syringe-sized calibrated microburet dispenses the titrant until the endpoint is reached. Results are usually read directly off the calibrated scale in ppm. The apparatus used to perform this procedure is totally portable, as with the drop count method.

– Digital Titrators: titrant from a cartridge is inserted into a micro-dispensing device. The amount dispensed is read on a digital venier, usually in ppm.

Titration methods are generally quite inexpensive, and are the preferred method in many procedures. Tests for acidity, alkalinity, carbon dioxide, hardness, dissolved oxygen, and chlorine are among the most common. Here, too, convenient packaging and simplicity are the key to portability and accuracy. This method is preferred in determining corrosion to water supplies, and offers the operator an easy, inexpensive

approach to meeting lead/copper requirements.

Turbidimetric Method

Some test procedures do not use color as a way to determine results. Instead, a sample is taken and a reagent is added which produces turbidity or cloudiness in the sample. The greater the turbidity, the greater the concentration. As with the colorimetric method, turbidimetric results can be "read" using a visual comparator, or by the use of a colorimeter. Results are expressed in ppms or mg/L. Typical tests using this method are those for potassium and sulfates.

Again, the mechanism employing this method can be totally portable and conveniently packaged as a kit.

Electrometric Method

One of the most commonly used: an electrode is inserted into a sample. A small current or voltage is produced and electronically amplified and read on a meter scale. Typical tests of this sort are for pH and conductivity, but a variety of substances can be measured using ion specific electrodes (ISE), including calcium, nitrates, chlorine, etc.

Nearly every electrometric procedure requires meter calibration and/or sample pretreatment. Examples are the 4, 7 & 10 pH buffers used to calibrate pH meters.

Generally, electrometric methods are initially quite costly and they require a high degree of care and maintenance because of the electrode systems.

Today, inexpensive pocket pH, conductivity, and ORP meters are on the market. Even though they are designed to be disposable after a period of time, great care must be exercised in their use and maintenance. These pocket meters rival the costs of colorimetric or titrimetric methods. They generally are not acceptable for reporting purposes, but are ideal for quick system checks.

Nephlometric Method

This method is specifically for measuring water turbidity. Suspended matter within a sample is measured via a specially designed meter which sends a focused light beam through the water sample. Suspended solids, dirt, and silt scatter the light. The scattering is measured by a photodiode at a 90° angle incident to the light source. Results are expressed as Nephlometric Turbidity Units (NTU's), and are more qualitative than quantitative.

Portable battery-powered units are available for field use. Private and municipal water treatment systems using surface water supplies such as lakes, streams, etc. are required to measure turbidity routinely as a guide to monitoring various water treatment systems like settling basins and sand filter performance. Continuous-monitoring turbidity meters and recorders are becoming the rule rather than the exception.

Gravimetric Test Methods

These are essentially physical test procedures. They include monitoring settleable solids by means of settleability tests, and are primarily used as operational guideposts in both water and waste facilities.

A sample is taken (usually one liter) thoroughly mixed and allowed to settle. Imhoff cones and settlometers are the common containers of choice here. The samples are timed at various intervals to determine the ratio of solids and the volume of solids that settle. Results can be transferable to plant operations to determine proper flocculant doses, expected sludge volumes, adjusted waste and return sludge in wastewater facilities.

These are relatively simple test methods that require no chemical or reagent to perform (except when determining flocculant dosages), and they provide valuable data to a water or wastewater operator.

Sampling Technique

All of the test methods described above require proper samples. Accurate sample volumes for each test are important. Some other important points to remember:

– Choose the proper location in the water system from which to extract your sample.

– Let the spigot run a short period of time to obtain a representative sample. (**Note:** applies only to a first draw sample. For lead or copper, disregard this step.)

– Pour the correct volume of the sample into the test tube or jar. Accurate results require accurate sample volumes.

– Once the test is complete, dispose of both the waste reagent and the sample properly, and clean all test tubes thoroughly.

– Follow the test kit manufacturers' directions specifically. Do not alter the procedure to suit your needs or take shortcuts; this risks skewed results.

– Do not intermix different manufacturers' reagents, particulary colorimetric ones, unless they are the **exact** same concentration.

Summary

Briefly, we have looked at six water testing methods for use by water and wastewater operators. So which one is right for you?

– A careful review of the different kinds of test is needed.

– Choose the test method that suits your testing skill level.

– What degree of accuracy do you need? Know the test's limitations.

– Review the test kit and method costs versus expected results.

– Look to the marketplace for manufacturers of test equipment and kits. Review their products.

– Consider safety and reagent disposal requirements.

– Are the results reportable?
Does the procedure follow standard methods or the EPA manual?
Is it state approved?

Safety & Environmental Factors

Many test kits and instruments contain hazardous reagents. Read all safety related instructions. Thoroughly review test procedures before running the test. Use manufacturer supplied Material Safety Data Sheets (MSDS) to learn about specific hazards and proper waste reagent disposal. Know the shelf-life of specific reagents and replace when required.

Some heavy metal test kit reagents have been banned for water testing in the home. Tests for lead, cadmium, mercury, etc, may contain extremely hazardous materials like carbon tetrachloride and sodium cyanide. Leave those tests for an outside certified laboratory to perform.

Nearly all test methods defined and described here are common inorganic tests. Tests for pesticides, aromatic hydrocarbons (gasolines), PCBs and the like should also be left to a qualified certified laboratory that has the proper equipment.

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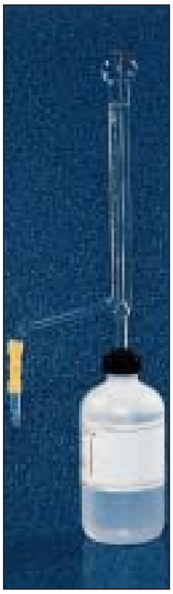
Water Testing Methods

Titrimetric

Titrimetric tests can be used to determine the concentration of a substance in a sample solution. After the sample is treated with an indicator, a standard titrant is added until a color change indicates a completed reaction. OMEGA offers four separate types of titration methods, allowing a choice of precision and convenience.



Automatic Buret
The self-zeroing automatic buret is calibrated from 0 to 10 mL in 0.1 mL increments. It is available with a squeeze valve (pinchcock), glass stopcock, or Teflon® stopcock.

Direct Reading Titrator
The direct reading titrator is a 1.0 mL microburet calibrated to allow direct reading of the test result. Each titrator has a specific range, but may be refilled to test higher concentrations.

Dropper Pipet
The drop count test uses a pipet to provide fast, precise measurements in the field. The number of drops used before the color change is multiplied by a fixed factor to provide the test result.

Dropper Bottle
The dropper bottle test uses bottle tips which deliver a consistent, standard drop size to add titrant to the sample. As with the drop count test, the number of drops used to complete the reaction is multiplied by a given equivalence factor to determine the concentration.

Colorimetric

There are two basic types of colorimetric tests:

1. Tests which determine the concentration of a substance are based on Beer's Law. Simply stated, this law says that, the higher the concentration of a substance, the darker the color developed in the test; as a result more light will be absorbed by the sample.

2. pH tests use an indicator which changes color with changes in the concentration of hydrogen ions, that is, according to the acidity of the solution.

Visual Methods

OMEGA offers several visual colorimetric comparators:

The Octet Comparator and Octa-Slide contain eight color standards with built-in filters which eliminate optical distortion. The color standards in the Octet Comparator are arranged so that the sample can be compared to four standards at once. In the Octa-Slide, the standards are placed in a bar so that they can be compared to the sample individually.

There are two accessories which can be used with an Octet Comparator. The Bi-Color Reader neutralizes sample color and/or turbidity to give more accurate readings. The Axial Reader uses a mirror to extend the view path and intensify the faint colors of low concentrations for easy distinguishing. Both accessories attach directly to the comparator and come with complete instructions for use.

Electronic Methods

Electronic colorimeters measure the amount of light which travels through the reacted sample, and convert the measurement to an analog or digital reading as ppm or %T.

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