

# Water Characteristics and Testing Methods

By Emily Bolda, CWS-VI and Greg Pronger

**M**any *WC&P* readers have analytical tests performed on water, whether it is necessary during the R&D process, for customer problem diagnosis, for product testing and certification or other reasons.

## *How should a sample be taken?*

Samples should be representative of the water to be analyzed. Care should be taken during the collection process to reduce contamination and to make it as representative as possible. To measure ambient concentrations in a drinking water sample in a home, it should be collected after running water until it is cold. Alternatively, for a 'worst-case' sample, a first-draw sample could be collected, which is the first liter of water that comes from a faucet that hasn't been used in at least six hours. Samples should always be collected in clean containers.

## *How should a sample be preserved?*

Different techniques require different preservation methods. Metals require preservation with nitric acid, mercury with gold chloride. VOC samples are preserved with hydrochloric acid, while semi-VOC samples are not preserved. There can also be additional requirements for temperature or time in storage (or 'hold time').

## *What can go wrong?*

There are things that can go wrong during analysis; not enough sample or other things in the sample that interfere with the measurement technique, though those things are mostly accounted for in instrument software. These methods are run with QC techniques to ensure data is of the highest quality, including control checks, analysis of known standards as samples, etc.

## *How reliable are the test kits and strips sold by others? How do they compare with real-world analyses?*

There are test kits available through several vendors that are great for quick and fairly reliable results in the field or in the lab. Generally, these techniques cannot measure concentrations that are very low, while the techniques described below can.

There are a variety of contaminants and characteristics that can be measured in water, including general characteristics and inorganic and organic analytes. General water characteristics include pH, alkalinity, turbidity, hardness and TDS. pH is, reported as the negative log of actual hydrogen concentration and is expressed as a unitless number on a scale of 0 to 14, neutral being 7.0. Values under are increasingly more acidic, and values of 7 and over are increasingly more basic or alkaline. The overall pH range of natural water is generally between 6 and 8. Variations in pH value from 7 are mainly due to hydrolysis of salts of strong

bases and weak acids, or vice versa, and dissolved gases, such as carbon dioxide, hydrogen sulfide and ammonia, among others. In the laboratory, pH is measured using a meter that measures the electro-chemical potential between a known liquid inside a glass electrode and an unknown liquid (the sample), separated by a membrane. The resulting pH value is expressed as the logarithm of the reciprocal of hydrogen ion activity in moles per liter.

Alkalinity is a measure of water's capacity to react with a strong acid to a designated pH. Highly alkaline waters are usually unpalatable. Components of alkalinity can also combine with both acids and bases, which act to buffer water and prevent sudden uncontrolled changes in pH. Alkalinity is measured using the titration technique, where a reagent is added (drop wise) to a known amount of sample until an indicative endpoint is reached. Using the volume of sample, volume of reagent and the strength of the reagent, the amount of alkalinity can be determined.

Turbidity is the reduction of clarity in water that is caused by dispersion of light by extremely tiny particles. Turbidity results from addition of clay, silt and organic or other material to water. It is measured using a turbidimeter on an arbitrary scale of nephelometric turbidity units (NTUs). During analysis, light is transmitted through the water sample. The amount of light that is scattered and absorbed rather than transmitted in straight lines through the sample is then related to the turbidity value.

Hardness in water, which is mostly calcium and magnesium, can be measured two ways. One technique is titration, as described above. Another is by adding separate concentrations of calcium and magnesium to obtain the hardness value.

Many dissolved substances are undesirable in water. TDS can be made up of dissolved minerals, gases and organic constituents and may produce aesthetically displeasing color, taste or odor. During analysis, samples are passed through a 0.4-micron glass fiber filter to remove non-dissolved substances, dried to constant mass in a dish and dried in an oven. After drying, the remaining residue is TDS. TDS can also be estimated by using the conductivity value with a conversion factor.

Another category of water characteristics is metals. The major cations (ions with a positive charge) are sodium, calcium, potassium and magnesium. Calcium and magnesium are common constituents in natural water; they often cause hardness in water and scale buildup in water heaters and pipes. Sodium and potassium can be present naturally, as well as from human contribution. Iron and manganese may cause metallic taste, stains on laundry and porcelain fixtures, and fouling of equipment. Copper and zinc can also cause stains on porcelain and fittings, and can result from corrosion and deterioration of plumbing materials. Silica, a naturally occurring dissolved mineral, produces a glassy scale in high temperature equipment, and can

be used to predict the life of certain water treatment media. At high levels, silica tends to form scale or colloidal silica, which can foul membranes and other components. Also included in this category are heavy metals (including lead, arsenic, selenium, chromium, cadmium and others), which are elements with documented adverse health effects.

Several methods can be used to analyze metals in water, each employing its own technology. The fundamental characteristic of water analysis techniques for metals is that each element emits and absorbs energy at specific wavelengths, particular to its chemical character. Intensity of the energy emitted or absorbed at the chosen wavelength is proportional to the amount of that element in the analyzed sample. Thus, by determining which wavelengths are emitted by a sample, and by determining their intensities, chemists can quantify concentrations of elements in the sample, relative to a set of reference standards.

Another method to analyze metals is flame atomic absorption, or FLAA. Samples are introduced into an extremely hot flame fueled by acetylene and compressed air. This produces atoms in a highly energized state, which absorb light at a particular wavelength. Light emitted at a frequency specific to the analyte of interest is passed through the flame chamber and reaches a detector that measures the amount of light absorbed by the sample, which is related to concentration. This technique is quick but not particularly sensitive, detecting only to the low-ppm and high-ppb range.

A more sensitive technique for measuring metals in water is called stabilized temperature platform graphite furnace atomic absorption spectroscopy, or GFAA. This technique is usually computer-controlled. In this method, a very small amount of prepared sample is introduced into a small furnace constructed of high-purity graphite, heated to a very high temperature and vaporized. Light is passed through the furnace chamber and through the sample vapor. This light is emitted from a special lamp, which produces a particular wavelength that is mostly exclusive to the analyte of interest. A sensitive detector measures the amount of light that is absorbed by the sample, and then related to the concentration in the sample. Using this technique, contaminants can be observed at ppb and sub-ppb levels. This technique is sensitive but slow and can analyze only one element at a time.

Another metals analysis method is inductively coupled plasma (ICP), a computer-controlled, optical emission spectrophotometric technique, in which excited electrons emit energy at a given wavelength as they return to ground state. ICP is an argon plasma that is maintained by the interaction of a radio frequency field and ionized argon gas. This creates extremely high temperatures (up to 10,000°K), which is comparable to the surface of the sun. Samples are first introduced to a nebulizer, which causes them to become aerosolized. Fine droplets pass through a spray chamber and are introduced into the plasma field. The aerosolized sample becomes energized in the plasma field and becomes ionized. Through the plasma field, ions emit their signature optical wavelength; sensitive detectors view the light emitted and relate the wavelengths and intensities to the particular elements and their concentrations. This technique is quick and fairly sensitive for many elements (into the low-ppm and mid-ppb range), and can measure many analytes simultaneously.

ICP technology can be used in conjunction with a mass spectrophotometer (MS) to provide a very powerful analytical instrument. This technique is based on each element becoming ions of known mass in ICP/MS conditions, and can thus be measured and related to concentration, functionally 'weighing' ions to determine concentration. The technique first operates the same as ICP described above. Ions in the plasma then exit and

**The Water Quality Association is an ANSI-accredited, third-party certifier of products to many NSF/ANSI and other standards. The organization's on-site product testing and analytical laboratories offer testing for performance, structural integrity, extraction and many other types related to product certification and R&D work. The laboratory also offers analytical services through its Water Analysis Program, which includes a basic water analysis and was designed to be used as a tool for diagnosing water problems.**

**WQA also works in conjunction with Suburban Laboratories, located in Hillside, Illinois, an independent commercial testing lab that serves hundreds of customers in a variety of disciplines. Suburban Labs, capable of tailoring methodology to meet non-routine testing requirements, has invested in personnel and the latest instrumentation required for quality analytical testing to maintain a high level of internal technical expertise. Full testing services are available, including physical, chemical and microbiological testing procedures currently used for analysis of drinking water quality by state and local water supply programs.**

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enter an interface region. Here they pass through a focusing lens and are pulled toward the mass spectrometer by a high vacuum. The MS has four long metal rods collectively called a quadrupole, which acts as a mass filter and operates by changing voltage in order to separate ions of different mass. The quadrupole allows only one mass to pass through to the detector at any time and sorts ions on the mass-to-charge ratio. After ions are sorted, they reach the detector at the end, which generates an electronic signal. The detector counts individual ions passing through the quadrupole and relates them to concentration. This technique is both quick and extremely sensitive, and can also measure many analytes simultaneously, well down to the sub-part-per-trillion (ppt) level.

On the other side of things are major anions (ions with a negative electrical charge). Some major anions (such as chloride, fluoride, phosphate, nitrate, nitrite and sulfate) can be analyzed by a technique called ion chromatography (IC) and is often computer-controlled. Some of these anions are nutrients but can contribute noticeable taste to water, corrosion, health effects in the case of nitrates and nitrites, and fluoride (traces of which can be found in natural waters), which is, for the most part, added into municipal water supplies.

IC-analysis technology is based on the migration of different anions through columns (of resin) at known rates. In this technique, a small amount of sample is introduced through a series of small columns containing a special ion exchange resin that separates all of the anions as they move. This generates a graph (a chromatogram) of the peaks of the different anions. The peak areas are then related to the anion concentration.

Analysis of organic compounds calls for several gas chromatography (GC) techniques. GC is used to analyze for many organic compounds, volatile and semi-volatile. These methods, generally computer-controlled, employ a technique similar to ion chromatography. During analysis, compounds leave the sample through a small syringe and are then introduced into a capillary column. Just as with IC, different compounds travel through the column at different rates, producing a chromatogram from which

concentrations can be calculated. This technique is very sensitive, and can see down to the low- and sub-ppb range.

Like ICP/MS, GC can be made more powerful when coupled with MS. Volatile organics can be analyzed by a GC/MS using a sampling device called a purge-and-trap, developed by the US EPA for the analysis of 84 chemicals identified as potential contaminants. The purge-and-trap passes helium through samples, stripping any organics from the system that boil below roughly 200°C, and are relatively water insoluble. Helium, after passing through the sample, passes through a trap, where the organics are absorbed. After purging is complete, the trap is heated to around 260°C and the components are transferred to the GC/MS for analysis. The organics must be sufficiently stable to desorb from the trap, and must also be amenable to gas chromatography. Typical chemicals measured by this technique are short-chained chlorinated solvents (trichloroethene and tetrachloroethene); aromatics (benzene, toluene and xylenes) and ketones (acetone, methyl ethyl ketone and methyl isobutyl ketone).

This technique can be used for analyses that require very high levels of sensitivity, both for identification and quantitation purposes. Some compounds are relatively water-soluble, and typically have poor response to purge and trap analysis. To address this problem, chemists employ a technique called selected ion monitoring (SIM), which greatly enhances the sensitivity of the instrument when applied for a small group of analytes. The SIM technique is based on soluble compounds breaking down into specific ions in GC, and those ions (particular to that compound) being monitored and used to relate to concentration

GC/MS can also be used to analyze semi-volatile organics. When analyzing these, analytes are first extracted from a water

sample with methylene chloride, followed by concentration of the solvent to one-milliliter volume. This concentrate is then analyzed.

There are organic compounds that are not amenable to GC/MS. Many of these can be examined by using high-performance liquid chromatography (HPLC), which is similar to GC, except analytes remain in the liquid phase during analysis. HPLC can also be paired with MS for a more powerful tool.

Water analysis is a powerful resource for many facets of the water industry. It can be used in part to determine compliance with regulatory and industry standards, help determine the best treatment for individual water problems, and is crucial during R&D processes.

### **About the authors**

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