



# On-Site Bottled Water Testing

*Editor's Note:* In view of the increased need for ensuring water quality, this article by Michael Neiheiser is a good reference for all involved in bottling water.

Continuous assessment of water quality is necessary to ensure a safe and consistent bottled water final product. This is the basis for the regulatory framework established throughout the bottling industry, including governmental agencies such as the US EPA, FDA, state agencies and industry-specific groups, such as the IBWA and other regional-based bottling associations.

Quality control programs implementing hazard analysis of critical control points (HACCP) and safe quality foods (SQF) guidelines are based on this principle of ongoing quality evaluation. But, it is neither cost effective nor timely to rely strictly on testing by an outside laboratory.

## Testing selection

So, what should bottlers be concerned with regarding their own on-site testing capabilities?

Overall, both on-site testing and outside, independent lab testing have their place in water bottling. Each can offer an array of parameters and cost effectiveness. An outside lab generally provides a higher degree of precision with a more expansive list of parameters than on-site testing can offer, plus is necessary for many regulatory purposes, such as full, annual source and product testing.

For routine purposes, such as everyday monitoring, however, on-site testing offers real-time results and gives plant personnel the data necessary to keep the process performing appropriately. Through on-site testing, bottlers can monitor their systems to mitigate problems immediately during production and significantly reduce revenue loss.

Bottlers should be monitoring quality parameters at all points in the process: water entering the plant, during production and as finished product. Testing by quality control personnel should evaluate the chemical, physical and microbial quality of various target parameters.

This monitoring can be used to detect the presence of agents that may indicate product contamination. These protection measures and other quality control programs help ensure that inferior products do not reach market.

## In-plant tests

The first step is to determine what tests are appropriate to run at your plant. Depending on the source of your water (municipal, spring, artesian well) and the type of product being produced, your list of on-site testing parameters will vary.

Referring to an implemented quality control program such as HACCP can help provide direction for what parameters to monitor at each point of the process. Once you have determined what parameters to test, you need to determine what range is appropriate.

The range is the level you expect to observe. You can get an idea of the expected level by looking at past analytical reports and criteria, and discussions with water treatment professionals, local health departments and the US Geological Survey (USGS).

Although this list is not meant to be all-inclusive, some of the basic on-site quality control monitoring parameters are necessary. These include: pH, temperature, conductivity, TDS, resistivity, ORP, ozone, chlorine, other specific ions and microbiological constituents (coliform and HPC).

## pH

Monitoring pH levels is one of the most important gauges of water quality because it impacts almost all water bottling treatment applications. It is important to note that to obtain an accurate pH reading, the testing should be done onsite, immediately after sample collection because pH can change almost instantly upon exposure to carbon dioxide from atmospheric conditions.

The use of a properly calibrated on-site meter can actually provide a more precise pH reading of the water before it changes due to atmospheric uptake. Monitoring pH is important to many stages of production, from particulate filtration/coagulation for particle removal at the beginning to bromate formation at the end. By evaluating pH levels throughout various points in the process, adjustments can be made that can seriously change the performance of the system.

## Temperature

As with pH, the temperature of water can have a huge impact on the treatment techniques being employed. Temperature affects the amount of dissolved species in solution (like calcium), the direction of chemical reactions (removal of certain contaminants), conversion potential of certain chemical processes, viability of microorganisms, etc.

As temperature increases, so does the relative solubility of most metals and other inorganic components. Calcium and magnesium can precipitate out of solution, for example, as a result of both elevating and decreasing temperatures.

## **Conductivity, TDS and resistivity**

Conductivity is observed at frequent intervals because it is a quick and economical way to determine the ionic strength of the water. It is basically a measurement of a substance's ability to conduct electricity.

Because conductivity is the measurement of ions that conduct electricity, it is often used to estimate dissolved solids in water. This is different than how an outside lab will test for TDS, which often employs a gravimetric method that is both time consuming and more costly.

Conductivity is measured in millisiemens per centimeter or microsiemens per centimeter, while TDS is measured in parts per million or milligrams per liter. An on-site meter uses a simple calculation to convert conductivity into TDS, based on the calibration solution used. Although some elements can be 'invisible' to the conductivity method of TDS determination (such as silica), it is a good day-to-day gauge.

Pure water has to have much lower conductivity than water that contains higher levels of salts, minerals, acids or bases. High conductivity may result in the need for additional analysis by an outside lab for common contaminants such as chlorides, silica, sodium, phosphates and hardness minerals.

Converse to conductivity, resistivity is the measure of how strongly a material opposes the flow of current. Resistivity is the inverse of conductivity and is measured in ohms.

The worse water is at conducting electricity (lower the conductivity), the higher the resistivity. This is the basis for using resistivity as the primary means of ion impurity detection.

This becomes applicable to purified waters that have very small amounts of dissolved ions. Some standards, such as those regulating highly purified waters for use in pharmaceutical or lab reagent waters, call for resistivity on the order of 18 M ohms per centimeter, whereas a typical purified (RO, DI, distilled) bottled water ranges between 0.05-2 M ohms/cm.

## **ORP**

Oxidation-reduction potential (ORP) gives an overall measure of the residual disinfectant strength of water. Oxidation-reduction refers to gain and loss of electrons and is helpful in determining what chemical reactions can occur. This becomes extremely important to ensure proper sanitation. Disinfectants such as chlorine, chloramine, bromine and ozone are powerful oxidizers, so monitoring ORP helps to determine when these oxidizers are used up.

In order to protect public health, monitoring ORP and pH helps maintain an adequate level of disinfectant. ORP is measured in units of millivolts.

## **Ozone**

As with ORP, measuring the residual ozone in both process water (after-contact tank) and final product is key to ensuring the control of microorganisms. It is recommended to monitor ozone residual every half hour because even a tiny spike in ozone levels can lead to product quality problems.

Since ozone generation is directly proportionate to the electricity being supplied to the unit, a spike in the electrical grid can cause over-production of ozone. Additional ozone can potentially cause inadvertent conversion of bromide to bromate and lead to an exceedance of a regulatory limit.

Too low of an ozone residual, on the other hand, will not provide the necessary microbial disinfection. Generally, ozone residual should remain between 0.1 and 0.4 ppm. The appropriate level for a particular water should be determined based on the water chemistry and regular microbiological testing. Various

adjustments coinciding with testing of ozone, microbiological constituents and bromate may be required to determine the appropriate levels.

## **Chlorine**

Chlorine is another test commonly done onsite. Chlorine should be tested immediately after a sample has been collected because it is a very reactive chemical that will readily combine with other elements already in the water to form other compounds referred to as disinfection byproducts.

Bottlers using a chlorinated source should test for chlorine onsite. This will ensure the removal of chlorine that can cause issues with the performance of membrane filters and the resulting taste profile of the final product.

## **Microbiological**

Because there are too many bacteria to feasibly test for every possible species, when we refer to bacteria in drinking water we are commonly looking for coliform, with emphasis on fecal and more specifically *E. coli* bacteria. Coliforms are indicator bacteria, meaning their presence indicates that conditions exist for the potential presence of other infectious, disease-causing bacteria.

Weekly coliform testing is required on source and product waters by an approved laboratory. Some bottlers have an in-house lab approved by their state(s) for coliform testing, but most send it to a certified laboratory.

In addition to satisfying regulatory requirements, these independent results offer a check and balance against inhouse test results. Generally, bottlers using a municipal source don't have to do weekly testing on the source as it is already tested by the municipality (of course this is on a case-by-case basis and some states may require further evaluation).

The finished product water should be tested in-house by the bottler daily. Testing may also be done more frequently in order to break up production runs into smaller groups.

A more general microbial test is the heterotrophic plate count (HPC). This is also referred to interchangeably as standard plate count (SPC).

The HPC testing is not as specific as coliform testing and is designed more so to monitor a system's overall microbiological load as opposed to specific human disease-causing species. Regularly reviewing HPC levels in conjunction with ORP and ozone residual allows you to see how a system's microbiological load is being controlled.

## **When and where**

Determining what points in the process to test and the testing intervals should coincide with the bottler's HACCP plan or their version of a similar quality control strategy. There are many points in the bottling process that could be monitored as a control point or a point in the process where product quality could be at risk. Critical control points represent the last point in the process where contamination can be prevented.

Frequency of testing performed to ensure that all equipment in the process is operating effectively should be determined based on manufacturer recommendations. Equipment and process monitoring are case-specific, as different plants have their own unique configurations and different types of finished products.

Checks should be performed at start-up each day (or at the beginning of each shift) and then at regular intervals throughout each production run. Beginning, middle and end testing is a good way to bracket product and more easily pinpoint quality problems.

Examples of critical control points for evaluation include, but are not limited to, filtration/demineralization processes (sand, carbon or particle filtration, distillation, reverse osmosis and deionization), UV, ozone contact tank, storage tanks and filter heads. Several other points are also possible for evaluation.

### **Types of on-site equipment**

On-site testing equipment and methods can include simple test strips for a single parameter, handheld meters that test dozens of parameters, hands-on wet chemistry titrations and microbiological plating. Most on-site testing for these parameters are best performed using well-maintained, handheld meters that employ either colorimetric or potentiometric techniques.

Colorimetric meters are used mainly for evaluation of inorganics such as iron, manganese, chlorine, alkalinity, hardness, etc. A colorimeter is used to determine an unknown concentration of a given substance through visual comparison against a known standard solution of the same substance. The standards may come pre-packaged in cuvettes or in packets to batch-up in the field.

Potentiometric meters are used for monitoring parameters, such as pH, ORP, conductivity, etc. These meters compare the known internal voltage from a reference probe against the charge of a probe in the unknown sample. The difference in voltage between the two probes is measured and reported as the value of the parameter for which you are testing.

Because different meters and calibration standards operate in various ranges it is important to select accordingly. For instance, if the expected TDS value of your product is 50 ppm, then you will need to select a meter and reagents that will test in a range that includes that value.

There are several options for on-site testing equipment and supplies to meet the needs of the various applications for which they will be used. Many distributors offer demonstrations, technical training or sometimes even trial units to make you comfortable with using their products. Take them up on these offers and find out which equipment and/or test method you are most comfortable running.

### **Calibration/maintenance**

Calibration is critical to ensure a high level of accuracy with any on-site testing meters. It is extremely important to properly maintain your testing meters with an appropriate cleaning and calibrating schedule, as inaccurate test data can result from inappropriately maintained equipment.

Suppliers or manufacturers should be able to give advice on what meters will meet your needs. This is often based on your applications and specifications and how to care for them to ensure a high level of accuracy. Calibration involves using a solution with a known concentration for comparison and adjustment of the meter. Meters should be calibrated based on how often they are used and the analysis required.

Some meters can automatically self-correct for certain vari-

ances and/or interferences such as temperature or turbidity. For instance, temperature can greatly affect a conductivity reading, so you can expect an error of two to five percent per one°C (33.8°F). As a result, many meters on the market contain sensors, which allow the instrument to correct for temperature.

Although each manufacturer's instructions are specific to the instruments they make, following are some general recommendations about instrument calibration and maintenance.

- A good rule of thumb for TDS and conductivity meters is to calibrate once a month for meters used on a daily basis.

- A pH meter should be calibrated regularly (daily or more frequently). Never return unused buffer back into the container of solution. Be sure to keep the calibration standard bottles tightly sealed and always adhere to the expiration date that should be clearly printed on each solution's label.

- An ORP meter does not require instrument adjustment as much as it needs electrode maintenance. To clean the electrode, simply dip the top of the electrode in a dilute (1:100) acidic solution for about two minutes; then rinse with clean water.

- The pH/ORP probe should never be stored dry. Always store the probes with proper storage solution.

Another matter to take into consideration is the use of the reagents (buffers, standards solutions) in the testing methods you have chosen. Reagents have a specific shelf life that can vary greatly depending on the reagent and storage conditions.

Keep in mind how quickly you can acquire reagents when you run out and how long they will be good. It's like buying enough milk to last, but not so much that it spoils before you drink it.

### **Valuable information**

There are hundreds of on-site testing methods available, all of which differ based on the contaminant being tested, the detection level or results you would like to achieve and the sample matrix. On-site testing for quality control purposes is a critical part of a bottled water business.

The data accumulated is an invaluable tool for detecting problems in the process, heading off potential problems and troubleshooting product quality complaints. On-site testing offers real-time results, so that any variance in a treatment system can be detected and corrected in a timely fashion to reduce product contamination and loss of revenue. Using on-site testing equipment will help you save time by giving you valuable information on the spot.

### **About the author**

◆ *Michael Neiheiser is Technical Services Representative with the Beverage Unit at National Testing Laboratories, where he has worked for the past four years. He has a double major in biology and chemistry from Wittenberg University, Springfield, Ohio. Neiheiser has established himself as a technical expert in the area of bottled water chemistry and process problem solving. He has extensive account management experience with bottled water, beverage and food customers.*