

# A Look Behind the Scenes of a Modern Drinking Water Laboratory

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By Stephen R. Tischler

## Introduction

A modern drinking water analysis laboratory is a complex place. The instruments are sophisticated, highly engineered and expensive. The technicians who run the instruments are highly educated and highly trained. Many have Ph.D. or Master's level education and a college degree is nearly mandatory even in most entry-level positions.

Federal and state regulations are constantly being written or modified, and laboratory executives and managers have to stay abreast of the latest changes if their businesses are to remain viable. Test methods, developed for and approved by the United States Environmental Protection Agency (U.S. EPA) or published in Standard Methods, provide the rigorous framework by which all modern drinking water labs operate. These methods drive the need for sophisticated quality control and assurance systems that are evaluated by a voluntary association of state and federal agencies providing third-party accreditation. Internal procedures are needed to control the flow of water samples through a complex process to a fast and accurate result. Customers demand faster results to aid in decision-making. Electronic reports are becoming the standard and sophisticated Laboratory Information Management Systems (L.I.M.S.) software is being developed and implemented to tie all of this together.

## Background

The genesis of the modern drinking water analysis laboratory has its roots in the Safe Drinking Water Act, which was originally passed by Congress in 1974 to protect the public health by regulating the nation's drinking water supply. The Act grants authority to the U.S. EPA to set national standards for drinking water based on sound science to protect against health risks, considering available technology and costs. These standards, as defined by the National Primary Drinking Water Regulations, set enforceable maximum contaminant levels (MCLs) for particular contaminants in drinking water and required methods to treat water to remove contaminants. The U.S. EPA prioritizes contaminants for potential regulations based on risk and how often they occur in water supplies.

There also exists a National Secondary Drinking Water Regulation. This is a non-enforceable guideline regarding contaminants that may cause cosmetic effects (such as skin or tooth

discoloration) or aesthetic effects (such as taste, odor or color) in drinking water. Between the primary and secondary regulations, there are over 100 contaminants routinely analyzed in drinking water.

These contaminants will be the focus further on in this article. Drinking water standards apply to public water systems, which provide water for human consumption through at least 15 service connections, or regularly serve at least 25 individuals. Public water systems include municipal water companies, homeowners' associations, schools, businesses, campgrounds and shopping malls.<sup>1</sup>

Bottled water is also regulated through a comprehensive system at the federal and state levels. Bottled water is regulated as a food product; as a result, the Food and Drug Administration (FDA) has jurisdiction over it as stipulated by the Food, Drug and Cosmetic Act. It is subject to extensive general food safety and labeling requirements. Mandatory laboratory testing of both source water and finished product is based in the same National Primary Drinking Water Regulation that controls the quality of tap water. By law, bottled water standards must be at least as stringent and protective of public health as tap water standards. States can also regulate bottled water as well as perform functions that include laboratory certification and approval of source water.<sup>2</sup>

With so many entities required to maintain systems in compliance with U.S. EPA and FDA enforceable regulations, the need for independent laboratories is significant.

## Laboratory certification and oversight

Just as providers of drinking water are required to meet stringent requirements, laboratories involved in the analysis of drinking water contaminants are also required to meet the highest standards of quality. That involves certification at federal and state levels, successful analysis of samples provided by third-party proficiency testing companies using promulgated methods and passing periodic on-site audits. Many laboratories hold certification that meets the requirements from the National Environmental Laboratory

Accreditation Conference (NELAC). NELAC is a voluntary association of state and federal agencies with full opportunity for input from the private sector. NELAC's purpose is to establish and promote mutually acceptable performance standards for the operation of environmental laboratories. The U.S. EPA's National Environmental Laboratory Accreditation Program (NELAP) office provides support to NELAC and evaluation of the accrediting authority programs.<sup>3</sup> Additional information on NELAC can be found on the U.S. EPA's website. Laboratories also hold multiple state certifications that allow them to analyze samples originating within that particular state. It is not unusual for large national laboratories to have 25 or more state certifications.

## Laboratory methods overview

The U.S. EPA, through the National Primary and Secondary Drinking Water Regulations, maintains a list of contaminants and their associated MCLs, treatment techniques or secondary standards. This list can be located at [www.epa.gov/safewater/mcl.html](http://www.epa.gov/safewater/mcl.html) and is divided into the following categories: microorganisms, disinfectants, disinfection by-products, inorganic chemicals, organic chemicals and radionuclides.<sup>1</sup> The analysis of these contaminants is controlled by very specific test methods generally developed for and approved by the EPA or published in Standard Methods jointly by the American Public Health Association, American Water Works Association and Water Environment Federation.

The methods are written as comprehensive procedures that typically specify:

- the type(s) of contaminants that can be analyzed;
- the appropriate analytical instrument to be used;
- working definitions;
- things that can interfere with the analysis;
- safety issues;
- equipment and supplies;
- reagents and standards;
- sample collection, preservation and storage;
- quality control requirements;
- the actual step-by-step procedure;
- data analysis and calculations.

Select contaminants and methods for their analysis are shown in Table 1. Many contaminants can be analyzed using more than one method, but this table shows only

<b>Table 1. Contaminants, methods and method descriptions</b>		
<b>Category and contaminants</b>	<b>Method identification</b>	<b>Method name and description</b>
<b>Microorganisms</b> Total coliforms	SM9222B	Membrane filter technique for members of the coliform group
<b>Disinfectants</b> Chlorine	SM4500 Cl (various)	Chlorine (residual)
<b>Disinfection by-products</b> Bromate	300.1	Determination of inorganic anions in drinking water by ion chromatography
Haloacetic acids	552.2	Determination of haloacetic acids and Dalapon in drinking water by liquid-liquid extraction, derivitization and gas chromatography with electron capture detection
Trihalomethanes	524.2	Measurement of purgeable organic compounds in water by capillary column gas chromatography / mass spectrometry
<b>Inorganic chemicals</b> Arsenic	200.8	Determination of trace elements in waters by inductively coupled plasma / mass spectrometry
Chromium	SM3113B	Metals in water by electrothermal atomic absorption spectrometry
<b>Organic chemicals</b> Dioxin	1613	Tetra- through octa-chlorinated dioxins and furans by isotope dilution HRGC/HRMS
Diquat	549.2	Determination of Diquat and Paraquat in drinking water by liquid-solid extraction and high-performance liquid chromatography with ultraviolet detection
Endothall	548.1	Determination of Endothall in drinking water by ion exchange extraction, acidic methanol methylation and gas chromatography/ mass spectrometry
Glyphosate	547	Determination of Glyphosate in drinking water by direct aqueous injection HPLC, post column derivitization and fluorescence detection
PCBs	505	Analysis of organochlorine pesticides and commercial polychlorinated biphenyl (PCB) products in water by microextraction and gas chromatography
Volatile organics	524.2	Measurement of purgeable organic compounds in water by capillary column gas chromatography / mass spectrometry
<b>Radionuclides</b> Gross alpha, beta	900.0	Gross alpha and gross beta radioactivity in drinking water

one method for each contaminant. As the table indicates, the analysis of drinking water contaminants included in the U.S. EPA's Primary and Secondary Regulations involves many complex methods using very sophisticated instrumentation.

### **The analysis of a drinking water sample**

There are many, many steps in the analysis of a drinking water sample. The following represents the general flow of a sample from collection to reporting results.

#### **Step 1: Collection, preservation, shipping and storage**

Collection, preservation, shipping and storage are critical to the analysis of a drinking water sample. For a successful outcome, samples must be taken in a manner that suits the particular analysis to be performed. This is a complex step because a battery of tests involving many methods will require different sizes and types of bottles to be returned to the laboratory in one or more coolers or packages. Each method specifies the particular type of container required,

preservative (if any) used to stabilize the sample, method of shipment (for samples requiring refrigeration, transportation in a cooler packed with ice) and the length of time the sample can be held prior to analysis. Some are considered short hold time samples that have to be analyzed within 48 hours of collection or sooner, while others can be analyzed months after being collected. This single step creates the most difficulty for the laboratory, because many samples are not collected properly. The result is often a need to resample in order to meet method requirements. In addition to sample collection, it is important for the customer to complete a proper Chain of Custody papers. This will ensure that the sample has been identified correctly and will be reported properly. This is an extremely important document in the analysis process.

#### **Step 2: Sample log in**

The sample log in procedure is critical to the successful outcome of

an analysis. Because samples arrive in many different containers for different tests, the log in process has to be done with the utmost care. Chain of Custody forms have to be inspected for completeness, so that sample identification is accurate. Samples must be uniquely identified and sent to the proper analysis station or work center for analysis. Some samples require refrigeration until analysis time. All samples must be checked for date and time collected information so that they don't run into hold time issues.

#### **Step 3: Sample preparation**

The amount of preparation required prior to analysis depends on the specific method. Some samples, like pH, require no preparation. Others require extensive pre-analysis preparation. For instance, the analysis of Haloacetic Acids (HAA's) by EPA 552.2 requires a complex liquid-liquid extraction using methyltert-butyl-ether (MTBE). The haloacetic acids that have been partitioned into the MTBE phase are then converted to their methyl esters (derivitization) by the addition of acidic methanol. The acidic extract is neutralized

Table 2: Method 552 Primary Standard	
Analyte	Concentration ug/ml
Monochloroacetic acid	60
Monobromoacetic acid	40
Dalapon	40
Dichloroacetic acid	60
Trichloroacetic acid	20
Bromochloroacetic acid	40
Dibromochloroacetic acid	20
Bromodichloroacetic acid	40
Chlordibromoacetic acid	100
Tribromoacetic acid	200
2,3 Dibromopropionic acid (surrogate)	100

by a back-extraction with a saturated solution of sodium bicarbonate and the target analytes are identified by gas chromatography in combination with an electron capture detector.<sup>5</sup> Complicated stuff!

#### Step 4: Quality control

Along with samples that are to be analyzed, the laboratory has to run quality control samples to determine if method analytes or other interferences are present in the laboratory environment, the reagents or the instruments.

Laboratory Reagent Blanks (reagent water treated exactly the same as samples) are used for this purpose. Laboratory Fortified Blanks (known quantities of method analytes added to reagent water) are also prepared and analyzed to determine whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements. Internal Standards and Surrogate Analytes are also used in the quality control process to assure that results are accurate. A detailed explanation of all the quality control steps required would be too lengthy but needless to say, the quality control procedures required to operate a modern drinking water analysis laboratory are extensive.

#### Step 5: Sample analysis

Before a sample can be analyzed, instruments need to be calibrated and standardized. This procedure can be fairly simple. For instance, a pH meter can be calibrated at a minimum of two points that bracket the expected pH range. Typically a pH meter is calibrated at four units and 10 units, as most of the measurements will be in the range of five to eight.

The calibration and standardization for Method 552.2 is much more complicated. For this method, a five-point calibration curve is prepared by diluting a primary standard at appropriate levels. A primary dilution standard for this method can have as many as eleven analytes at various concentrations. A typical primary

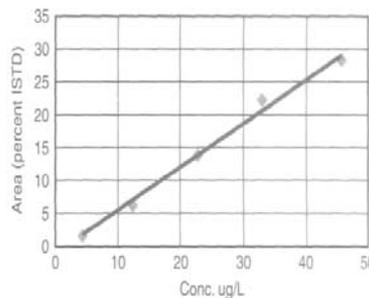
standard for this method can be seen in Table 2."

This primary dilution standard is used to prepare calibration standards, which comprise five concentration levels of each analyte with the lowest standard being at or near the method detection level for each analyte.<sup>7</sup> A typical calibration curve is shown in Figure 1.

Once the instrument is operating properly and all preliminary quality control work and calibrations have been completed, samples can be run, during which the quality of the instrument is checked continually; a quality control sample is generally run after every 10th sample. Most modern analytical equipment is configured to run using auto samplers. These can be loaded with many samples and then left unattended.

There are, of course, methods that do not use sophisticated

Figure 1: Calibration curve for monochloroacetic acid



analytical instrumentation. Most of these methods are for determining physical factors such as hardness, dissolved solids, color and odor.

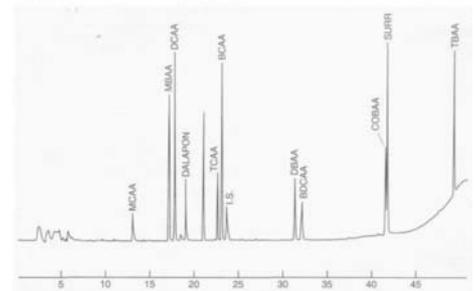
#### Step 6: Data analysis and calculations

After the analysis is complete, data is reviewed and results are calculated. This can be a simple matter, as in the recording of pH results. Staying with our Method 552.2 example, results are determined based on two criteria. First, compounds are identified after the chromatography run by peak retention time. Second, concentration is determined by measuring peak areas as compared to the calibration standards for each of the analytes. A typical chromatography run for Method 552.2 is shown in Figure 2.<sup>8</sup> The data analysis and calculation procedures are very specific for each method and most modern instrumentation makes use of sophisticated software for this purpose.

#### Step 7: Reporting

Reporting results is the culmination of all the previous steps in the process. Information gathered from the Chain of Custody along with

Figure 2: Chromatography run from Method 552.2



the results is formatted into a user-friendly report format. The report generally lists the customer contact information, sample identification, date and time collected, the report date, each contaminant, each method, the maximum contaminant level (as appropriate) and the results. All reports should indicate whether an individual contaminant has exceeded its MCL. Many laboratories make reports available electronically to help speed the results to the customer. Reports sent via email are quite common these days.

#### Conclusion

As was stated in the introduction, a modern drinking water laboratory is a complex place. The accurate reporting of contaminant levels in parts per million, parts per billion, and even parts per trillion, can only happen in an environment that is tightly controlled. This control is maintained by dedicated, highly educated and highly trained professionals that are committed to a system of rigid quality control and assurance procedures.

#### References

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