

Simplified Procedures for Water Examination

Sixth Edition



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Simplified Procedures for Water Examination

Sixth Edition

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Simplified Procedures for Water Examination

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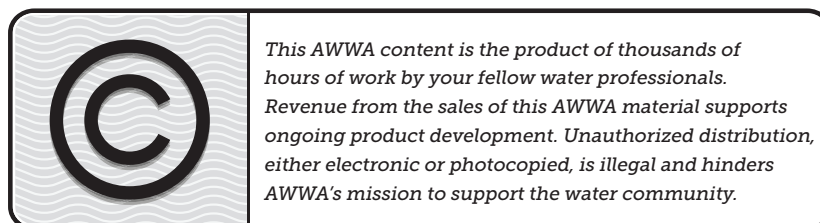
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Foreword

This publication is the sixth edition and the fourth revision of the original American Water Works Association (AWWA) Manual M12, Simplified Procedures for Water Examination Laboratory. The original manual, published in 1964, was revised in 1975, with a supplement on instrumental methods added in 1978. A complete revision was done in 1997 and updated in 2002. This 2014 edition includes basic quality control and disposal guidance that may be superseded by local or state regulations. The appendices were updated to include containers, preservatives, and holding times.

The original goal for the manual—to provide operators with simplified procedures for tests commonly needed for process control in drinking water production—remains the same. Monitoring plant processes through reliable, reproducible analyses enables plant operators to evaluate and optimize those processes to produce the highest possible quality drinking water.

The manual has been rewritten to include new and updated methods, laboratory equipment, safety procedures, and disposal guidelines. AWWA does not intend to recommend any one manufacturer, as most kits are available from a variety of manufacturers. This manual contains basic information on quantitative analysis, but beginners in the laboratory can gain helpful training from additional resources, such as short courses offered by state departments of health, AWWA sections, and chemical or equipment suppliers. Such classroom instruction, demonstrations by experienced teachers, and supervised laboratory work provide valuable additions to independent reading and practice.

The methods in this manual are simplified methods, and it is important to note when more sophisticated methods may be required for compliance monitoring. This manual is not designed to replace *Standard Methods for the Examination of Water and Wastewater*. M12 may serve as a tool to acquire laboratory skills that will eventually facilitate the use of *Standard Methods*. Refer to *Standard Methods* for more information on methods and water quality.

The methods included in M12 are based on the presumption of high-quality water of known and relatively constant composition. State health departments or other regulatory agencies that conduct bacteriological and chemical analyses of drinking waters should be consulted for advice of the applicability of methods to your situation. Some source waters may require the more complicated procedures from *Standard Methods*. Please note that there is a warning section for each procedure to ensure the applicability of the method.

Appendix A provides information regarding containers, recommended minimum volumes, preservation, and maximum holding times. These may be superseded by local or state regulations. Appendix B provides directions for the preparation of common types of reagents. Appendix C provides information on the safe storage of chemicals. A list of the compounds identified in this manual is available in Appendix D. References are presented in Appendix E with a glossary and periodic table at the end of the manual.

If you have any comments or questions about this manual, please email the AWWA Engineering and Technical Service support group at ETSupport@awwa.org.

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Chapter **1**

Water Quality Laboratory

INTRODUCTION

Reliable laboratory quality testing forms the basis for water quality control. Laboratory test results make it possible for operators to evaluate and optimize plant performance in the following ways:

- Results from water testing help the operator to know and to document water quality conditions throughout the plant.
- Laboratory analyses form a basis for selecting operational procedures and chemical treatment and then for evaluating the effectiveness of changes to the system.
- Logging data and reviewing trends provide the opportunity to identify potential problems before they affect water quality.
- Reliable water quality testing forms the basis for regulatory compliance and ensures the best possible quality drinking water for the community.

LABORATORY PROCEDURES

The reliability and quality of analytical data are dependent not only on the analytical procedure used but also the environmental conditions of the laboratory. Temperature, humidity, amount of available counter space, and cleanliness of the laboratory can impact the analytical process.

Temperature and humidity must be controlled in the laboratory. Temperature changes affect the reaction rates of certain test procedures, and high humidity adversely

affects the storage life of reagents and media. Storage in direct sunlight contributes to rapid deterioration of some reagents and most media. Unopened bottles of media and chemical reagents should be stored in dark, cool, dry locations, such as storage cabinets, separate from opened bottles. Inverting bottles and storing them cap-side down reduces caking. Consider storing opened bottles of media in a desiccator.

Laboratory facilities need enough counter space to adequately separate bench instrumentation and avoid constantly moving unused equipment to perform testing. Depending on the types of tests conducted, major pieces of equipment may require additional counter or floor space. Laboratory design should include tests currently conducted and accommodate future tests. Good overall laboratory design includes provisions for a vacuum line and natural gas lines, and areas for refrigerators, incubators, ovens, and autoclaves.

Maintaining a clean and efficient work environment is important for sound laboratory procedure. Equipment and supplies should be labeled and allocated a space in the laboratory. Chemicals should be kept in suitable containers and clearly labeled. The laboratory should be kept as clean and dust free as possible, and equipment should be protected from moisture and harmful fumes. There should be adequate lighting for color comparisons and ample facilities for lubricating, washing, and drying laboratory equipment, and for other laboratory activities. Testing equipment and supplies should be handled with care and respect. The laboratory should never be used as a lunchroom, kitchen, or miscellaneous storage facility.

Improper sampling or laboratory procedures in water testing are as bad as, or worse than, no testing at all. Inaccurate information obtained from contaminated samples or that result from incorrect procedures may lead to incorrect water treatment decisions. Accurate analysis depends on sampling procedures, careful maintenance and calibration of equipment, and attention to laboratory procedures.

SAMPLING TECHNIQUES

Water sampling seems to be a simple procedure. However, any time a small sample is withdrawn from a larger body of water such as source water, through the treatment plant or distribution system, there is a potential for error. Most major errors in water quality analysis are caused by poor sampling. When sampling is inadequate or not representative, water treatment decisions will be based on inaccurate information. It is essential to be particularly careful to obtain representative samples, use the proper sampling technique, and preserve samples until they are analyzed.

The location and frequency of sampling will be governed by the type of water available whether the samples are from an untreated water source such as a river or lake, from a treatment plant process unit, or from the distribution system. Samples may be obtained either as *grab* or *composite* samples.

Grab samples are most useful when an analysis is needed of the water characteristics at a single point in time. This is particularly important for water quality indicators that may change over time. These include dissolved gases, residual chlorine, pH, temperature, and coliform bacteria. Grab samples are most useful when the water has characteristics that remain relatively constant and flow is not continuous.

Where water characteristics change continuously such as in streams, composite sampling is most effective if done continually. However, in many cases this is impossible. In composite sampling, water samples are collected at regular intervals, perhaps hourly or every two hours. Portions of these samples are then combined and analyzed at the end of 24 hr. The size of the portion to be mixed with other sample portions is proportionate to the water flow at the time the sample was collected.

Representative Sampling

Because a large body of water is not uniform in quality throughout, the best procedure is to take samples from many sites, analyze them separately, and evaluate data as a group to determine the quality of that body. This procedure is more likely to be representative of the entire body than a single sample taken at one point. The more points from which samples are withdrawn, the more representative of actual water quality the overall data set will be. Good judgment should be used to select a sampling method, and several factors should be considered including:

- the character of the laboratory examinations to be made,
- how test results will be used (the objectives of the analysis),
- airflow that may create disturbances,
- the nature of the water and variations in its characteristics over time, and
- the variation of the flow rate over a sampling period.

Wells. Pumped water is usually the only source for well sampling. The mix in well water is generally good, and composite sampling is rarely needed. Pumps and casings can contribute to sample contamination, and the sample may not be representative, particularly if a pump is not used often. Changes in water quality, i.e., arsenic, nitrate, and some organic contaminants, may also occur in certain formations with the length of time a well is pumped and the well drawdown.

Lakes and reservoirs. Representative sampling of lakes and reservoirs is often difficult because of changes in water composition at various depths and temperatures. For this reason, a single sample represents only one area of the lake. Depending on the size of the impoundment, several samples must be collected at various areas and depths. The larger the impoundment, the more samples are needed if the objective is to fully characterize the system.

Rivers. Small- or medium-sized streams are usually more easily sampled than larger rivers because it is possible to find a place where the water is uniform or well mixed. If such a location cannot be found, it is advisable to collect samples from several locations. This is often necessary in larger streams and rivers.

In-plant sampling. Most sampling is done at the water treatment plant. Large plants may have continuous sampling provisions, but such systems may be too expensive for small plants. A sample tap must be located properly to ensure that samples are representative. A tap installed in an area that does not provide a representative sample is useless for obtaining accurate information.

Distribution system sampling. Obtaining representative samples throughout the distribution system is probably the best way to determine water quality throughout the system. Sampling sites should be selected to trace the course of finished water through mains and then through the major arteries of the system. A short, corrosion-resistant connection to the main is an ideal sampling site. If special sample taps are not available, samples may be collected from customers' faucets. Fire hydrants are generally not satisfactory sampling sites because the flow is erratic and they are not used often; therefore, often samples may become contaminated from corrosion or sediment. If hydrants are used, ensure sufficient flushing to remove sediment and obtain a representative sample.

Sampling Devices

Sampling may be done either automatically or manually. Automatic samplers save time in the sampling process, but they are expensive and may require frequent maintenance.

Staff must be aware of these possibilities and check regularly for possible problems to prevent sampler malfunctions. Dippers, weighted bottles, hand-operated pumps, and similar equipment are classified as manual sampling devices. A weighted bottle or other collection container is used to collect depth samples. Depth samplers are designed to be lowered in the open position with a mechanism that allows for a valve or stopper to be closed when the container is filled.

Sampling Procedures

Sample collection requires preparation. Please be sure to obtain the proper type and size of containers, pens for completing labels and chain-of-custody forms, and iced coolers. When using prepreserved containers that have had the preservative chemical added prior to sample collection, the container should not be rinsed or overfilled because of the potential loss of preservative. It is important to understand whether or not a sample requires zero headspace in the container. For example, bacteriological samples require headspace while many organic parameters, such as samples for disinfection-by products, must be collected with zero headspace in the container.

Surface sampling. Surface water may be sampled easily by using the following procedure:

1. Grasp the sample container at the base with one hand and submerge it, mouth down.
2. Position the mouth of the container into the current and away from the collector's hand.
3. If there is no current, create an artificial one by moving the container in the direction it is pointed.
4. Tip the bottle up to allow air to escape and water to enter the container.
5. Remove the bottle from the water.
6. Seal the container tightly.
7. Label the container with collection date, time, type of sample, location, name of collector, and water temperature. Fill out chain-of-custody form, if applicable.

If the sample must be taken from a bridge, walkway, or other structure above the water surface, first place the container in a frame that has enough weight to submerge the container. Attach a nylon or other nonrotting rope to the frame and remove the stopper. Lower the frame and container to just above the water level, facing the current. Swing it downstream slightly and drop it into the water. Pull the container upstream and out of the water. Seal and label it.

Water tap sampling. When sampling from water main connections, flush sediment and possible contaminants from the line before collecting the sample. Never take samples from leaking or corroded taps, from taps surrounded by dense vegetation, from public drinking fountains, restrooms, or from taps with aerators. To collect a sample:

1. Use a container that is clean and free from dust or other contaminants.
2. Clean the outer parts of the tap to prevent debris from falling into the container. Disinfect the tap if appropriate.
3. Flush the line by partially opening the tap and allowing the water in the line to run freely until it reaches the known temperature of distribution mains in the area. This may take 4 or 5 min. (Do not turn the water on full to save time. This may disturb incrustations and sediment in the line and contaminate the sample.)

4. Place the container close to the tap but do not allow it to touch the connection.
5. Collect the sample.
6. Seal and label the container. The label should show a date, time, type of sample, location, and name of collector. Fill out a chain-of-custody form.

Sample Preservation

Samples should be analyzed as quickly as possible after sampling. Some analyses, such as those for temperature, pH, chlorine, and sulfide, must be done immediately. Other analyses may be done as long as six months after the sample is taken (if the sample is properly preserved). However, the sooner the analysis is done, the more quickly any problems related to sample quality, test method, or water quality can be resolved.

USING THE METRIC SYSTEM

In a laboratory, chemicals are weighed in grams (g) and milligrams (mg), and liquids are measured in liters (L) and milliliters (mL). Temperature is measured in degrees Celsius or centigrade (°C) rather than in Fahrenheit (°F). Length is usually measured in meters (m) and centimeters (cm) rather than in inches (in.), feet (ft), or yards (yd). The metric system, like the decimal system, is based on units of 10. If a measurement is less than 1 meter, liter, or gram, a prefix signifies how much less. For example, the following are some common prefixes used in the laboratory: a centimeter equals one hundredth of a meter, or expressed in decimals, it is 0.01 m; a millimeter (mm) is one thousandth or 0.001 m; and a micrometer (μm) is one millionth of a meter or 0.000001 m. Measurements larger than meters, liters, or grams also use prefixes to signify the multiplier. For example, a kilometer (km) is 1,000 m.

Conversion Factors

Standard reports are made in milligrams per liter (mg/L). One liter of water weighs close to 1 million mg, so 1 mg of a substance in 1 L of water represents 1 part per million (ppm). The unit ppm used in water analysis always means by weight, never by volume. For example, 10 ppm hardness as calcium carbonate (CaCO_3) means 10 mg of CaCO_3 per 1 million mg water or 10 pounds of CaCO_3 per 1 million pounds water.

Table 1-1 provides a set of conversion factors useful in converting milligrams per liter to grains per gallon or milligrams per liter to pounds per 1,000 gal.

Table 1-1 Conversion factors

	Equivalent				
	<i>ppm or mg/L</i>	<i>gr/ US gal</i>	<i>lb/ 1,000 US gal</i>	<i>gr/ Imp gal</i>	<i>lb/ 1,000 Imp gal</i>
1 part per million	1	0.0583	0.00834	0.0700	0.100
1 grain per US gallon	17.1	1	0.143	1.20	0.172
1 pound per 1,000 US gallon	120	7	1	8.41	1.20
1 grain per Imperial gallon	14.3	0.833	0.119	1	0.143
1 pound per 1,000 Imperial gallon	99.8	5.83	0.833	7	1

WORKING WITH CHEMICALS

All prepared solutions should be of the best available quality. Chemicals labeled “ACS grade,” “primary standard grade,” or “analytical reagent grade” and dyes certified by the Biological Stain Commission yield the best results. The methods in this manual may have two names for the same chemical (such as potassium dihydrogen phosphate and potassium monobasic phosphate, both KH_2PO_4). These refer to the same substance and the chemical formula follows the name.

A *compound* is a substance composed of two or more elements. As a rule, all chemical compounds are divided into organic compounds, those that contain carbon (C), and inorganic compounds that have no carbon. However, there are a few exceptions. These include the inorganic compounds of carbon dioxide (CO_2), carbon monoxide (CO), bicarbonate (HCO_3^-), and carbonate (CO_3^{2-}). Table 1-2 provides a list of compounds commonly used in water treatment. For a more extensive listing of the compounds in this manual, see appendix D.

Many compounds can be created from the same two or three elements. This formula should be checked with the formula on the container label to confirm that the right chemical is used. A chemical symbol is often used in the laboratory as a symbol for the names of the elements. For example, in the periodic table of the elements (inside back cover), calcium is expressed as Ca and carbon as C. Because chlorine’s symbol is Cl and copper’s symbol is Cu, mistakes can easily be made in identifying chemicals in the laboratory. It is wise to double-check the name of the chemical and its formula to make sure the correct chemical is used. Chemicals behave very differently in solutions, and a mistake in the solution can result in errors and even hazards.

All bottles that contain standard solutions should be labeled with the name of the solution, its concentration, date of preparation, expiration date, name/initials of the person who prepared the solution, and, in some cases, the procedure for which the solution is intended.

The word *normal* (abbreviated *N*) in front of a reagent’s name indicates the concentration or strength. Normality is the amount of mole equivalents of a solute dissolved in one liter of solution. The word *standard* is also used, but means merely that the concentration is exactly known. The concentration of a standard solution is sometimes referred to as its *normality*. Thus, the normality of the standard acid used to determine the alkalinity of water is 0.02. This can be written 0.02N, N/50, or 1/50N, all of which have the same meaning. The important thing to remember about *normal* solutions is that, for example, 100 mL of a 0.02N sodium hydroxide (NaOH) solution will exactly neutralize 100 mL of a 0.02N

Table 1-2 Chemicals commonly used for water treatment

Chemical Name	Formula
Acetic acid	CH_3COOH
Aluminum sulfate	$\text{Al}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}$
Ammonium hydroxide	NH_4OH
Calcium carbonate	CaCO_3
Copper sulfate	CuSO_4
Ferric chloride	FeCl_3
Nitric acid	HNO_3
Phenylarsine oxide	$\text{C}_6\text{H}_5\text{AsO}$
Sodium bicarbonate	NaHCO_3
Sodium hydroxide	NaOH
Sulfuric acid	H_2SO_4

sulfuric acid (H_2SO_4) solution. See the appendix B for preparation of common types of reagents and solutions.

Some reagents are available in *molar* (abbreviated *M*) concentration. *Molarity* is the number of moles of solute dissolved in one liter of solution. For example 0.01M ethylenediaminetetraacetic acid (EDTA) used in the hardness procedure contains 0.01 moles of EDTA dissolved in one liter of water. Normality and molarity are related. Where molarity describes the moles of a complete substance per liter of solution, normality describes only the moles of reactive species per liter of solution. Normality is always a multiple of molarity. It describes the “equivalent” moles of reactants involved in chemical reactions.

With the high-quality reagents available from chemical supply sources, solutions can be prepared easily for analyses in the laboratory by adding distilled or deionized (reagent grade) water. However, they must be measured and standardized carefully before they are used in a laboratory procedure. The standardization of each solution is described in the 22nd edition of *Standard Methods*. Please note that *Standard Methods* is only one source for procedures for standardization of solutions. The reader is encouraged to refer to approved methods for the test of choice for proper preparation and standardization of reagents. Preparing reagent solutions requires more time and effort but is less expensive than using prepared solutions. Many standard solutions can be purchased from reliable supply houses, and their use is recommended for individuals who may not have the experience, time, or equipment to prepare them.

Each chapter includes instructions for preparation of reagents necessary for the particular method. Please refer to local, state or provincial, and federal regulations for any additional requirements. For example, chapter 9, Chlorine Residual, states that the stock potassium permanganate standard should be prepared at least annually. The state of South Carolina requires the stock potassium permanganate to be prepared at least every six months.

SAFETY PROCEDURES

Laboratory safety is important. There are many hazardous materials in the water laboratory, and everyone in the laboratory must remain alert and careful to avoid danger. For information on safe storage of chemicals, see appendix C. Take an extra moment or two to follow a safe practice rather than to risk injury. Be especially careful at the end of a shift or when tired, because most injuries occur at those times.

For specific safety questions, refer to state or provincial General Industrial Safety Orders or to federal Occupational Safety and Health Act (OSHA) regulations. Information on developing a program to manage hazardous chemicals may be found in US Environmental Protection Agency and OSHA publications.

OSHA has modified the Hazard Communication Standard (HCS) to adopt the international Global Harmonizing System (GHS) to improve safety and health of workers through more effective communications on chemical hazards. Since it was first promulgated in 1983, the HCS has provided employers and employees extensive information about the chemicals in their workplaces. The GHS provides a standardized approach to classifying the hazards and conveying the information including detailed criteria for determining what hazardous effects a chemical poses, as well as standardized label elements assigned by hazard class and category. This will enhance both employer and worker comprehension of the hazards, which will help to ensure appropriate handling and safe use of workplace chemicals. In addition, the safety data sheet (SDS) requirements establish an order of information that is standardized.

OSHA required employees to be trained on the new label elements (i.e., pictograms, hazard statements, precautionary statements, and signal words) and SDS format by Dec. 1,

2013, while full compliance with the final rule will begin in 2015. OSHA believes that U.S. workplaces will soon begin to receive labels and SDSs that are consistent with the GHS, because many U.S. and foreign chemical manufacturers have already begun to produce HazCom 2012/GHS-compliant labels and SDSs. Employers must ensure that when the new labels and SDSs appear in the workplace, the employees will be familiar with the labels, understand how to use them, and access the information effectively.

Chemical suppliers must provide SDS (previously known as material safety data sheets) for chemicals used in the laboratory and in water treatment. The SDSs must be readily available to all plant personnel and updated as chemicals are added or deleted (Figure 1-1). It is recommended that the laboratory order and keep only the amount of chemicals required for their routine testing needs to minimize the hazard and prevent disposal issues of unused expired reagents. Several chemical suppliers also offer smaller quantities of reagents to minimize the quantity of hazardous chemicals onsite and ensure freshness.

Safety and right-to-know information must be clearly posted for all plant employees. Figures 1-2 and 1-3 show how a medium-size water treatment plant provides safety and hazardous materials information to plant staff.

First-aid equipment should be readily available for laboratory use. Figure 1-4 shows a first aid station near the laboratory. It includes a first-aid kit, stocked regularly, and a 5-min minimum emergency oxygen supply.

Personal protective equipment (PPE) is essential when working in a laboratory or performing sample analysis. Examples of PPE include safety glasses, lab coats or aprons, and gloves.

Eyewash and shower stations illustrated in Figure 1-5 should be immediately accessible from the laboratory. Employees who might be exposed to chemical splash should be trained to

- remove protective equipment after activating the eyewash station,
- rinse any chemicals that may come in contact with eyes or skin,
- remove all affected clothing,
- remove contact lenses,
- hold eyelids open and roll the eyeballs so water flows on all surfaces of the eyes and in the folds surrounding the eyeballs.

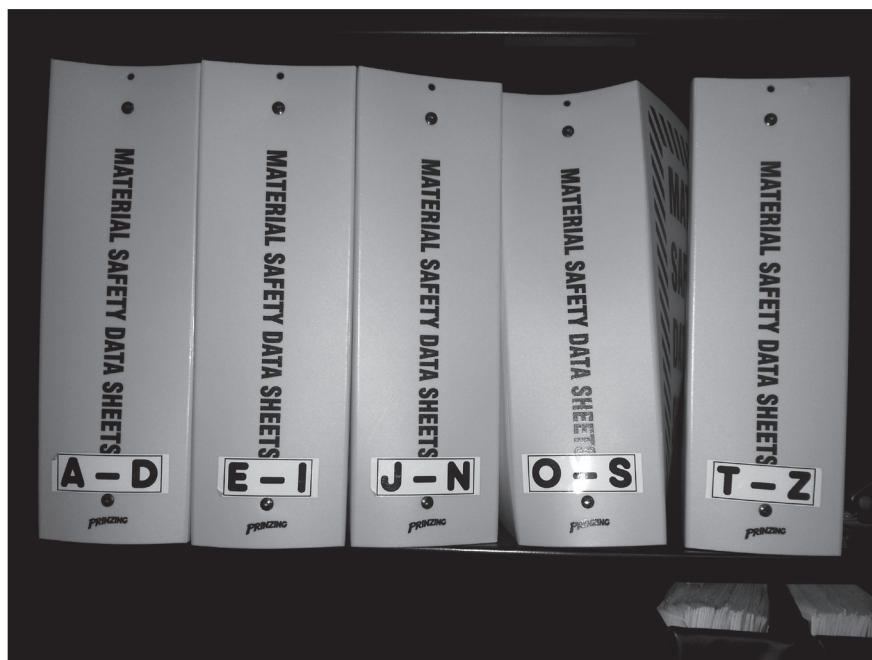


Figure 1-1 Material safety data sheets/safety data sheets



Figure 1-2 Plant information and safety center



Figure 1-3 Right-to-know information



Figure 1-4 First-aid station

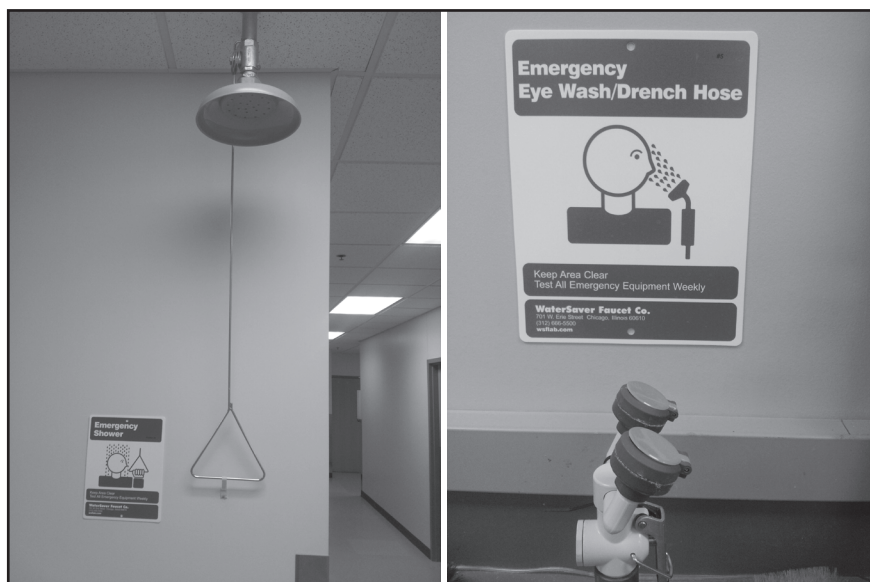


Figure 1-5 Safety shower and eyewash stations

Corrosive and Toxic Materials

To avoid injury, all chemical reagents should be handled with care. Use extreme caution when handling chemicals marked *poison*, *danger*, *caution*, or *flammable*. These chemicals should be kept in their original containers or in carefully marked containers used only for those solutions. Using a fume hood (shown in Figure 1-6) can prevent injury to laboratory employees by containing and removing hazardous fumes and hazardous chemicals while procedures are being performed. Any danger should be clearly posted on the hood. Most fume hoods pull air past the operator into the hood at about 50 to 100 ft per min as the sash opening is changed. Take care not to block the baffles at the rear of the hood. Standards are available to check the air flow against capacity recommendations. Most fume hoods also contain cabinets that are useful for storing hazardous materials (see Figure 1-7), but always guard against storing incompatible chemicals in the same place. Fume hoods are designed specifically to provide ventilation for the protection of lab occupants during chemical manipulations. The airflow that they provide is greatly in excess of that needed for storage of closed containers of even the most toxic of volatile materials. Storing materials in this way is, therefore, a misuse of an expensive piece of equipment. In general, the storage of chemicals in fume hoods is strongly discouraged.

Safety, automatic, and bulb-operated pipettes are recommended for measuring and transferring chemical solutions that can inflict injury by inhalation, absorption, or contact through the mouth, lungs, and skin. **Caution: Never pipette any liquids by mouth.**

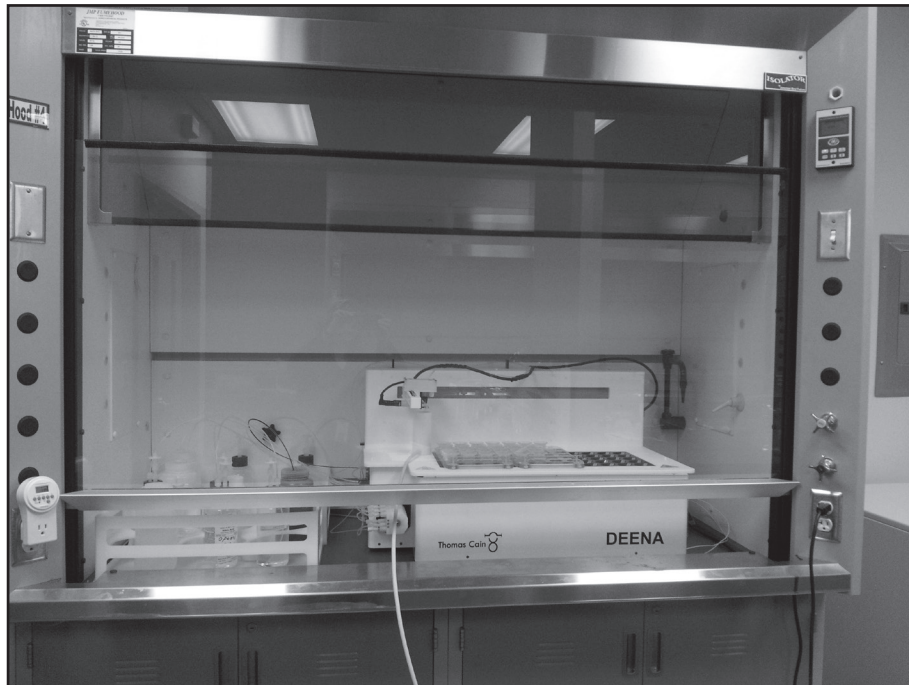


Figure 1-6 Fume hood



Figure 1-7 Safe storage and labeling for fume hood

Preventing Accidents

Work in the laboratory can be hazardous, but following basic precautions can reduce the risks. Figures 1-8 and 1-9 illustrate essential items for personal safety in the laboratory.

- If possible, do not work alone in the laboratory. If this situation is unavoidable, another person should check the laboratory regularly in case an accident occurs.
- Wear protective clothing, such as safety glasses, safety shoes, a laboratory coat, and a rubber apron to protect from spills.
- Clean up spills immediately with the proper spill kit.
- Wear insulated gloves or use tongs when handling hot materials. If material might erupt from a container, wear a face shield and goggles.
- Flush the outside of acid bottles with water before opening. Always pour acid into water (never water into acid) to avoid splashing. Never place stoppers or utensils on the counter after use. Always be careful of heat generated. Rinse the outsides of acid containers after use.
- Secure gas cylinders to prevent tipping or rolling using approved straps and clamps. Use a hand truck to move cylinders. Never roll a cylinder by its valve.
- Immediately wash off any chemicals that spatter with large quantities of water. Use vinegar to neutralize bases and baking soda to neutralize acids.
- **Never** eat, drink, smoke, chew gum, or apply cosmetics where laboratory chemicals are present. Always wash hands before doing any of these things.
- Do not smell or taste chemicals. Vent any apparatus that might discharge toxic chemicals, such as vacuum pumps and distillation columns, into local exhaust devices.
- Always use a fume hood with adequate air displacement when using hazardous or volatile chemicals. Have the laboratory building ventilation systems and fume hoods checked monthly. Some chemicals are volatile and require use of fume hoods. Some are safe to use without hoods. SDSs and other chemical references are used to determine which chemicals require hoods.
- Neutralize corrosive materials before disposing of them in corrosion-resistant sinks and sewers. Flush with large amounts of water.
- Do not force glass tubing, thermometers, or other glass objects through rubber connections. Wet or lubricate the joints to avoid breakage, and use thick protective gloves and goggles.
- Ensure that properly labeled fire extinguishers and a fire blanket are available at all times (Figure 1-9). Fire extinguishers are classified by the type of fire they will control. Be careful when using a fire extinguisher on small container fires because the force of the spray might knock over the container and spread the fire. Use a fire blanket to smother clothing fires. Fire extinguishers should be checked monthly and serviced annually.
- Ensure that a first-aid kit is available at all times. Space should be available to post proper emergency first-aid procedures in plain sight or store a manual in an accessible place.
- Do not store incompatible chemicals together.

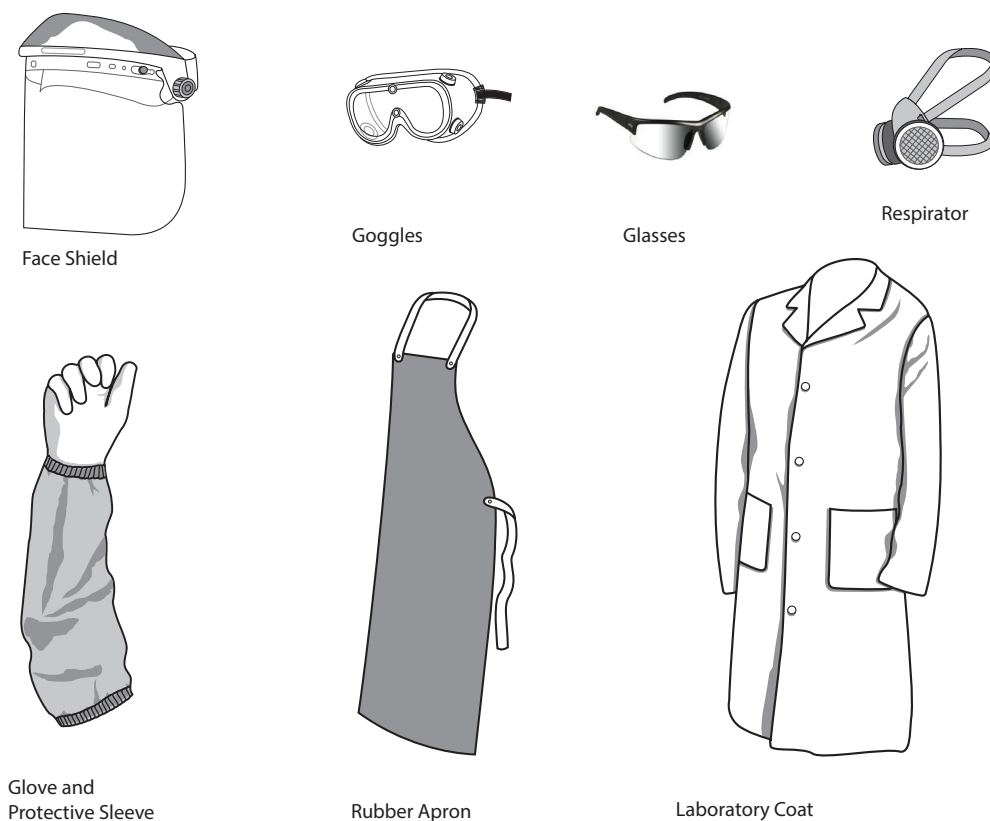


Figure 1-8 Personal safety equipment

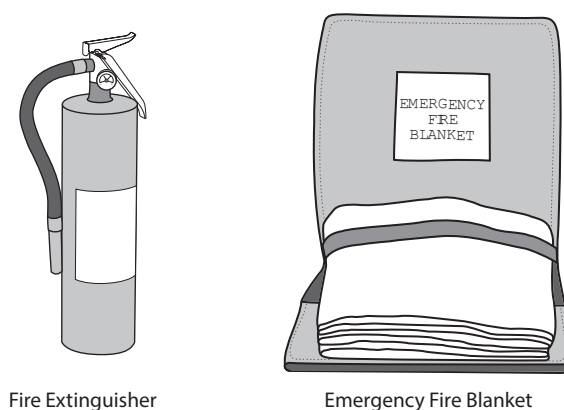


Figure 1-9 Laboratory safety essentials

EQUIPMENT

Laboratory equipment is divided into categories in the following sections to help readers locate equipment. Sections are arranged beginning with glassware, then weighing and measuring equipment, followed by equipment used for steps in a variety of procedures, and finally, test-specific equipment such as pH and conductivity meters.

Glassware

Glassware used in the laboratory should be heat resistant, borosilicate, class A glassware (trade names for class A glassware include Pyrex™ and Kimax™). Bottles, beakers, and stirring rods made of polyethylene are suitable for some operations, but plastic ware does not withstand high temperatures or strong oxidizing solutions. Disposable glassware, such as pipettes, test tubes, and bottles, may be used if the laboratory lacks time or equipment necessary for cleaning. Teflon-lined stoppers and caps should be available for some applications.

Pay special attention to ordering and using glassware intended for measuring liquids, because some vessels are designed to contain (TC) or to deliver (TD) the specified amount of fluid. All TC or TD beakers and pipettes must have clearly marked volumes with a 2.5 percent tolerance or less.

Beakers. Beakers, as shown in Figure 1-10, are perhaps the most common pieces of laboratory equipment. They are used to mix and measure chemicals. Measurements from a beaker are approximate volumes. Beakers range in size from 1 mL to 4 L.

Flasks. Flasks come in various shapes and sizes and are used to hold and mix reagents. Figure 1-11 illustrates a variety of flasks. Volumetric flasks, squat bottles with long, narrow necks, are used for measuring a specific volume. Their typical capacity ranges from 25 to 2,000 mL, indicated by an etched ring around the neck.

A filled volumetric flask should be viewed at eye level so the front and back sections of the ring around the neck merge into a straight line, and the bottom of the water level (the meniscus) touches this line. In using volumetric glassware, such as flasks, graduates, burettes, and pipettes, always read the quantity at the bottom of the curve (the meniscus). See Figure 1-16 under burette description for more information on the meniscus.

Volumetric flasks are used to prepare and dilute standard solutions. For example, 0.02N acid can be prepared by measuring with a transfer pipette 50 mL of stock 0.1N acid solution and placing the acid in a 250-mL volumetric flask. After filling to the mark with distilled water, mix the contents thoroughly by stoppering and inverting the flask 15 times or more. Because volumetric flasks are designed for measuring purposes, the contents should be poured into a clean storage bottle. Erlenmeyer flasks are not used for measuring but are useful for mixing and other laboratory processes.



Figure 1-10 Beakers

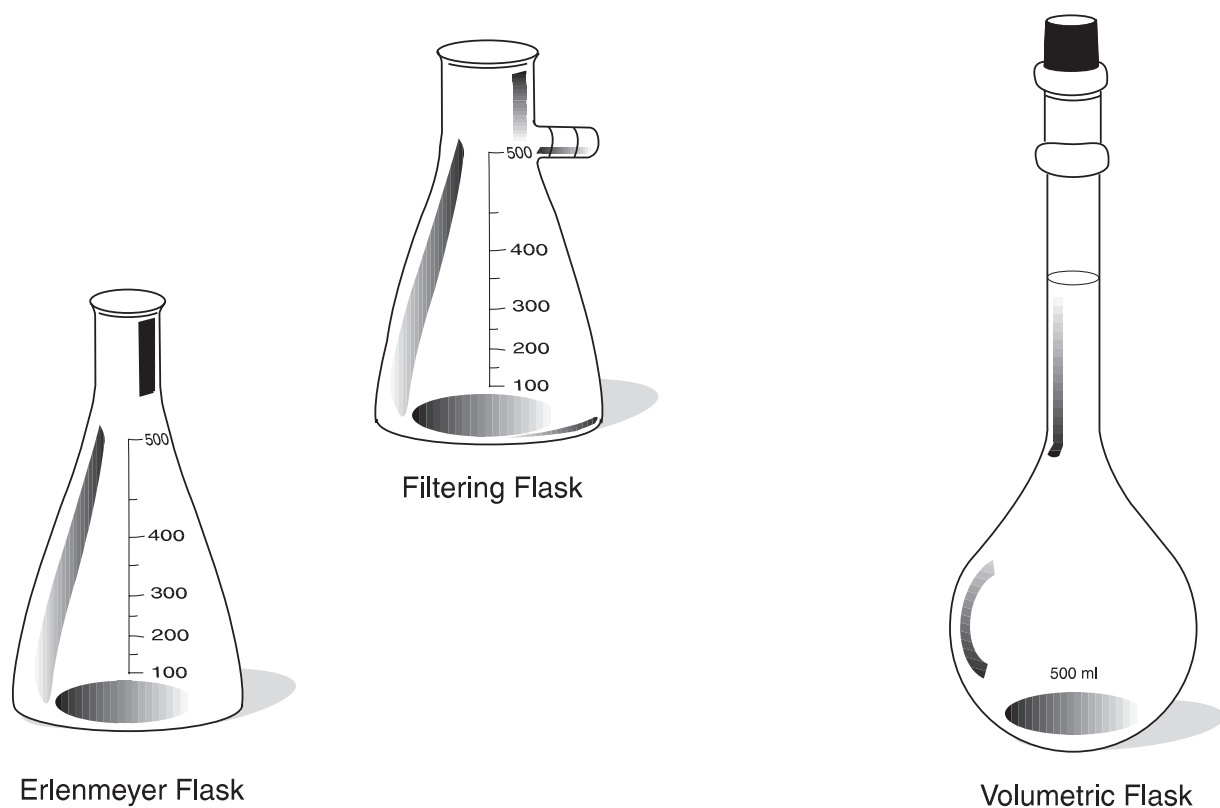


Figure 1-11 Varieties of flasks

Graduated cylinders. The workhorse of laboratory glassware is the graduated cylinder, popularly called the *graduate*. See Figure 1-12. Available in sizes as large as several liters, graduates are marked in milliliters, except for the 10-mL size, which is subdivided into fractions of a milliliter, and sizes of 250 mL or larger, which are etched at intervals of 5 or 10 mL.

Pipettes. **Warning: Never use mouth suction.** Two kinds of pipettes are in general use. See Figure 1-13 for the most common pipettes in the water process lab. Those with a graduated stem are called *graduated* or *Mohr* pipettes and can measure any volume up to the designated capacity of the pipette. Those with a single etched ring near the top are called *transfer* or *volumetric* pipettes.

One end of the pipette is tapered; the other end is fire polished so it can be easily closed by the pipette bulb. The small tapered end of the pipette is inserted in the bottle, the liquid drawn above the upper etched ring by suction from the pipette bulb (Figure 1-14). The pipette's tip is wiped dry with a clean cloth except during bacteriological examinations. The air is expelled from the bulb just enough to allow the liquid to gradually fall to the desired level. The measured liquid is allowed to flow freely into the receiving container by entirely removing the bulb from the top of the pipette.

When the graduation line denotes the volume contained in the pipette, the pipette is marked "TC". When the graduation line indicates the volume delivered from the pipette, the pipette is marked "TD". When a pipette is calibrated TD, it differs from a TC calibrated pipette in that a drainage holdback error, the amount of water required to wet the inner surface of the vessel in contact with the water, is added to the TC volume. The TD vessel then delivers the same volume as contained in a TC vessel. Operationally, the TC pipette's tip must be touched to the inner surface of the receiving container to remove the last drop from the pipette.

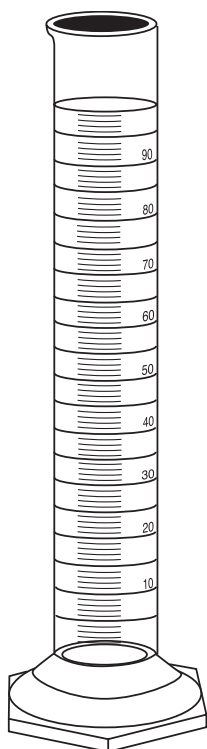


Figure 1-12 Graduated cylinder

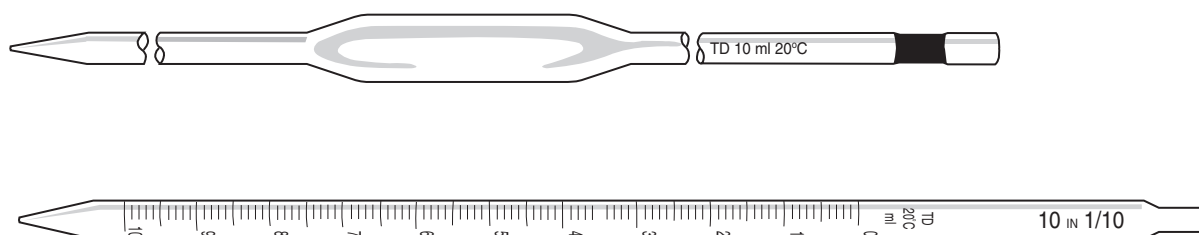


Figure 1-13 Transfer (volumetric) and measuring pipettes

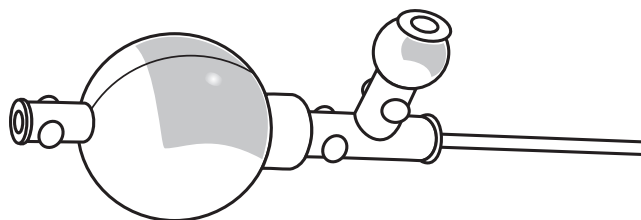


Figure 1-14 Pipette bulb

When drawing liquid into a pipette, the tip must be submerged as long as suction is being applied. Avoid vigorous suction during the filling operation because bubbles may form and rise to the surface, where they may take some time to break, making an exact

reading difficult. Medicine droppers are convenient to dispense small quantities from a few drops to 1 mL.

For accurate work, measure samples with volumetric pipettes. When speed is desired at a slight sacrifice in accuracy, carefully measure sample volumes with 100- or 50-mL graduated cylinders for results that are acceptable on an occasional basis. In the range below 50 mL, however, use volumetric pipettes to measure sample volumes because measuring errors can play a significant role in the final result.

Burettes. A burette is a glass tube graduated over part of its length. The most commonly used sizes are 10, 25, and 50 mL. The graduations in tenths of a milliliter help to estimate a fraction of a tenth or hundredths of a milliliter. Clamp the burette to a stand as shown in Figure 1-15 and fill from the top, usually through a funnel. Allow the excess solution above the zero line to drain to waste through the stopcock before starting a titration. Autozeroing burettes are also available.

When reading a burette, take care to read the level of liquid at eye level as in Figure 1-16, not from above or below. A burette that contains liquid shows a curvature in its upper surface, known as the *meniscus*. Mercury curves upward and water curves down.

Always read a burette twice. The difference between the readings represents the volume of titrant dispensed. Take precautions against air bubbles that arise from the failure of the titrant to wet the burette uniformly through its entire length. Also take care to avoid a slowly leaking stopcock, which allows the titrant to drain as the burette stands idle.

Teflon stopcocks are preferable because glass stopcocks need frequent lubrication to function satisfactorily. For best results with glass stopcocks, apply a lubricant, such as petroleum jelly, as a thin film on the dried surface of the stopcock. Use the lubricant sparingly, because an excess on the stopcock can clog the burette tip and contribute to the formation of air bubbles in the tip.

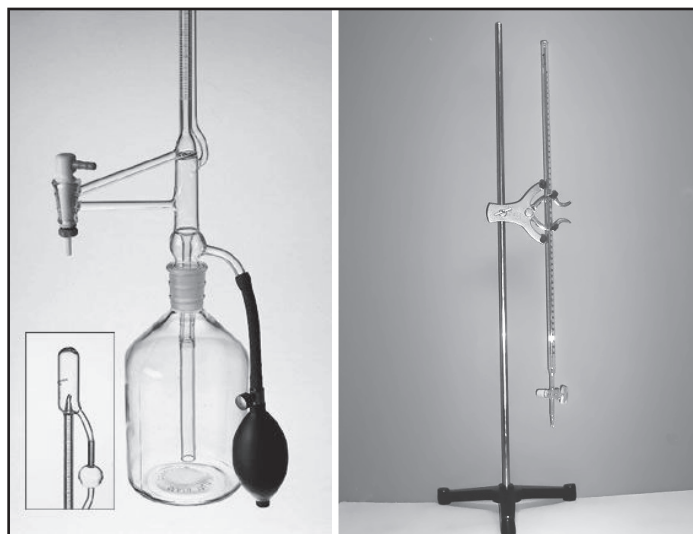


Figure 1-15 Autozeroing burette plus conventional burette, stand, and clamp

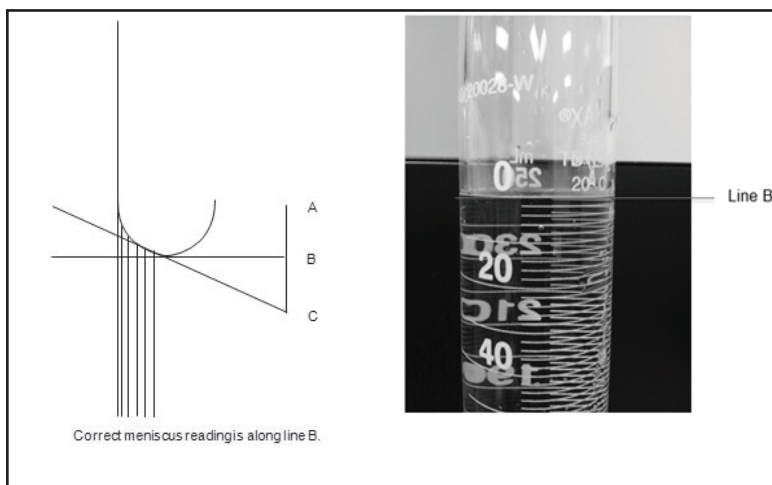
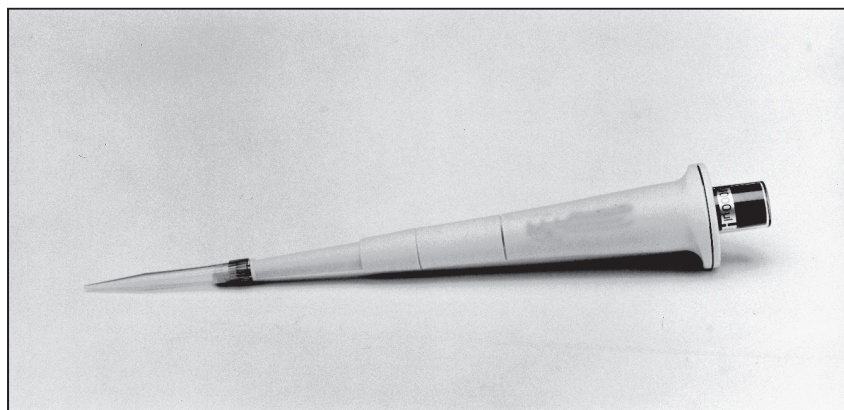


Figure 1-16 Reading of meniscus

A burette is specifically designed for dispensing a titrating solution, and individuals who find it difficult to control a pipette may find a burette a convenient substitute for measuring the accurate volumes of standard solutions needed in colorimetric work.

Automatic pipettes and dispensers. These convenient laboratory aids have the advantage of delivering a premeasured quantity of reagent solution quickly. Automatic pipettes and micropipettes (Figure 1-17) are particularly helpful in the exacting process of preparing standard curves and must be calibrated quarterly on the balance. Dispenser bottle tops (Figure 1-18) save time when repeatedly dispensing the same volume of reagent such as during the preservation of samples.



Courtesy of Brinkmann Instruments, Inc.

Figure 1-17 Automatic pipette



Courtesy of Brinkmann Instruments, Inc.

Figure 1-18 Dispenser bottle top

Automatic titrators. Improvements have been made in the classic burette with the intent to increase the speed of titrations. These titrators have a burette and quickly transfer the titrant. This type of burette system is available in several forms, two of which are shown in Figures 1-19 and 1-20.

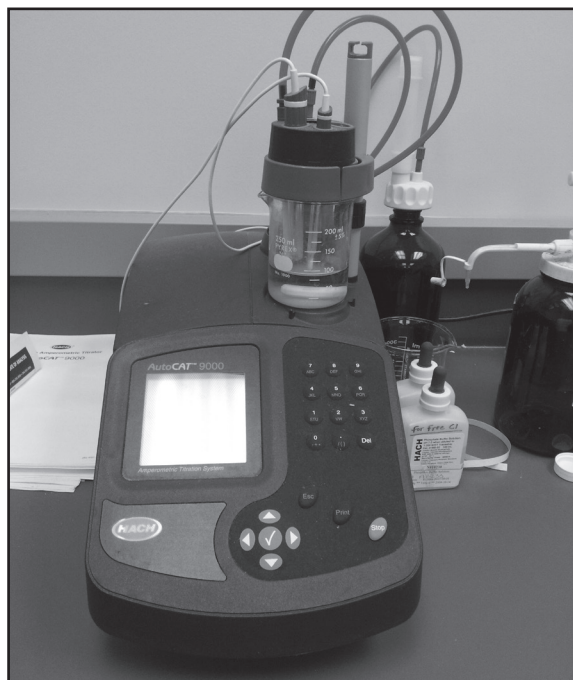


Figure 1-19 Amperometric titrator



Figure 1-20 Digital titrator

Bottles. Bottles commonly used for mixing, storing, and dispensing reagents and samples include glass and plastic sample and reagent bottles, biochemical oxygen demand (BOD) bottles, and milk dilution bottles, illustrated in Figure 1-21.

Crucibles. Crucibles are glass, porcelain, or metal dishes, shown in Figure 1-22, that are particularly useful for drying and mixing operations in titrations.

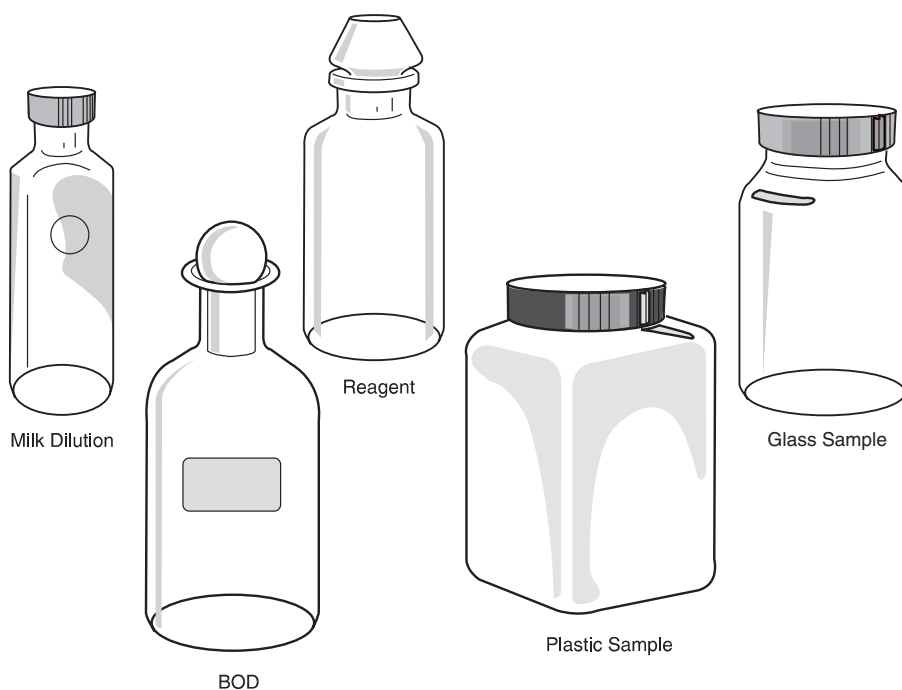


Figure 1-21 Bottles commonly used in the laboratory

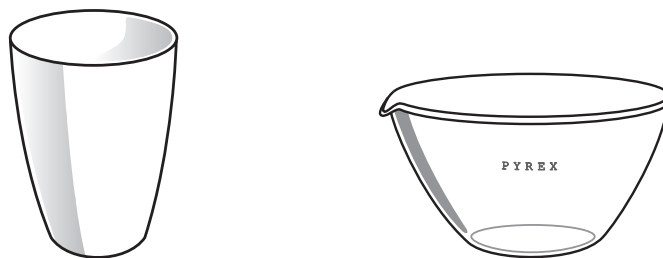


Figure 1-22 Crucibles

Funnels. Funnels are used to pour solutions or transfer chemicals from one container to another. Specialized funnels that separate solids from a mixture (Buchner funnel) or that separate one chemical mixture from another (filter funnel) are available. See Figure 1-23.

Petri dishes. These are shallow, flat dishes with low, straight sides and loose covers, illustrated in Figure 1-24. They are commonly used for microbiological cultures. Petri dishes may be made of Pyrex glass that can be sterilized in an autoclave or hot-air oven, or of disposable, presterilized plastic.

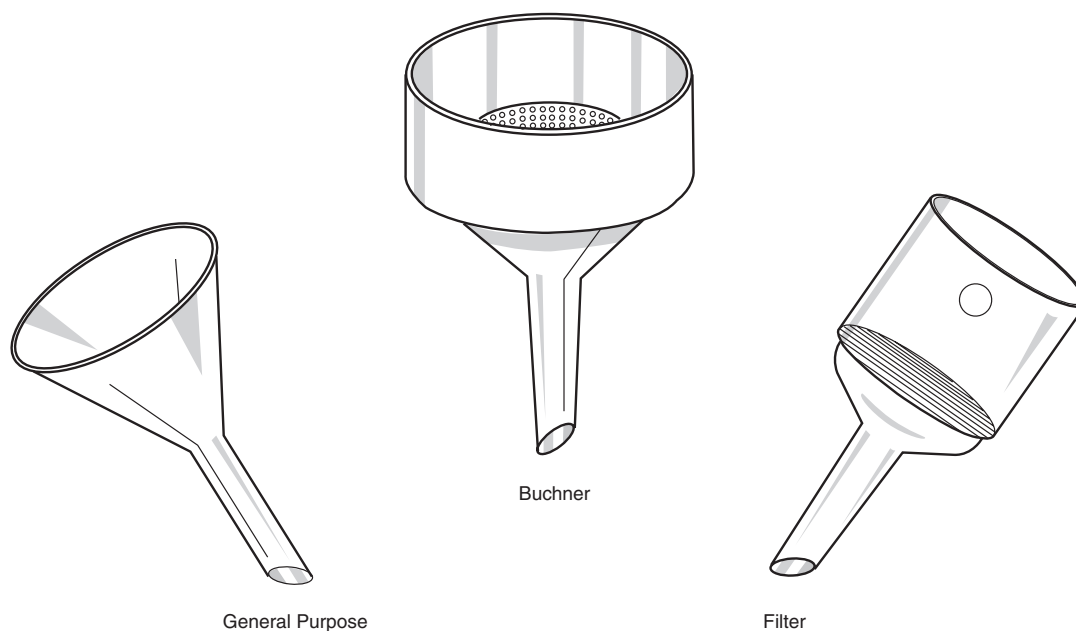


Figure 1-23 Funnels

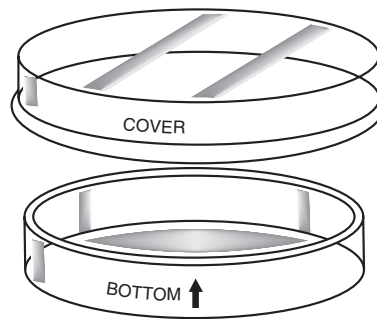


Figure 1-24 Petri dish

Test tubes. Test tubes come in many sizes and are stored in racks when in use (see Figure 1-25). They serve as containers for certain chemicals, liquids, or bacteriological media, or they can be used for mixing small quantities of chemicals. Commonly used tubes include the chemical, serological, and bacteriological. The Durham tube is a specialized tube inverted inside a larger tube that contains media for bacteriological testing. The Durham tube is inverted to trap fermentation gases from bacteriological growth. Several types of stoppers are available for test tubes.

Thermometers. Thermometers are calibrated for total or partial immersion. Total immersion thermometers must be completely immersed in water to yield the correct temperature. Partial immersion thermometers must be immersed only to the depth of the etched circle that appears around the stem just below the scale level. An all-metal dial thermometer may also be used but must be calibrated. A thermometer in an oven can be placed in a flask filled with sand to protect from breakage and from temperature changes when the door is opened. Please note that mercury thermometers are being phased out due to safety hazards. They are illegal in several states.

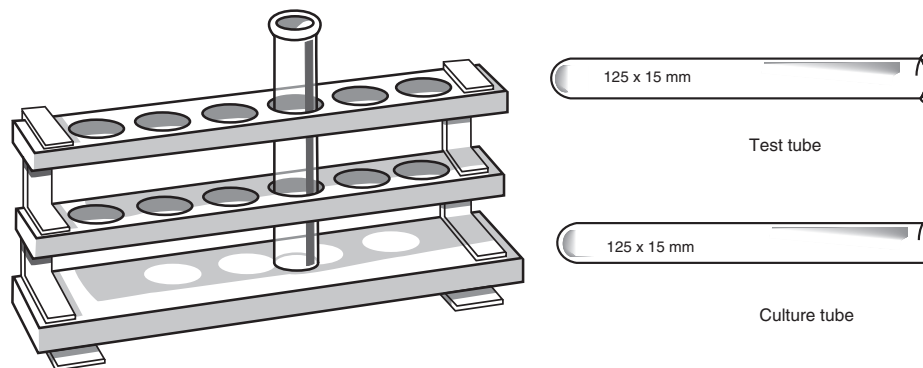


Figure 1-25 Test tubes and storage rack

An electronic thermometer uses a thermistor and a digital readout. This type of thermometer must also be calibrated. Follow the manufacturer's instructions for use and calibration. For best results, check the accuracy of the thermometer in routine use against a thermometer certified by the National Institute of Standards and Technology.

Aspirator. The T-shaped attachment (shown in Figure 1-26) to the sink faucet is a relatively inexpensive vacuum device used instead of a vacuum pump to filter samples.

Desiccators. A desiccator is a container of heavy glass with a removable top and a false bottom above the real base (see Figure 1-27). The space below the false bottom is filled with a drying agent to remove residual moisture while hot dishes cool to room temperature. A 12-in. diameter desiccator provides enough space for most water plant demands. Depending on the type of desiccator, a separate desiccant may be required.

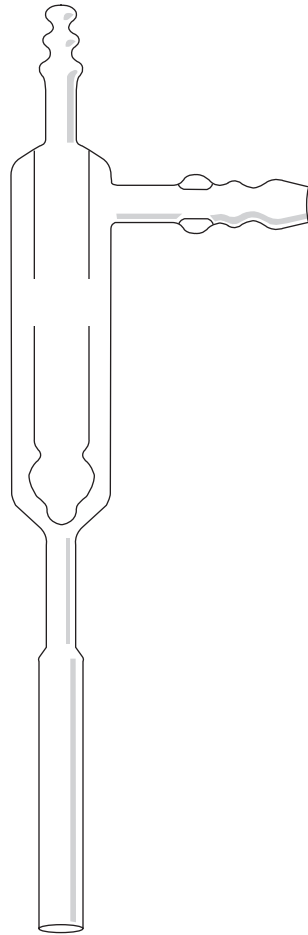


Figure 1-26 Aspirator

Weighing and Measuring Equipment

Many tools in the laboratory are used for specialized measuring or weighing. They are designed to give extremely accurate measurements and weights of chemicals and liquids. Such accuracy is essential in laboratory analysis.

Balance. An ordinary analytical balance (shown in Figure 1-28) weighs substances as heavy as 200 g with an accuracy of 0.0001 g or one-tenth of a milligram. Such careful measurement is essential for preparing standard solutions. Follow the manufacturer's directions carefully to ensure correct weight and balance sensitivity.

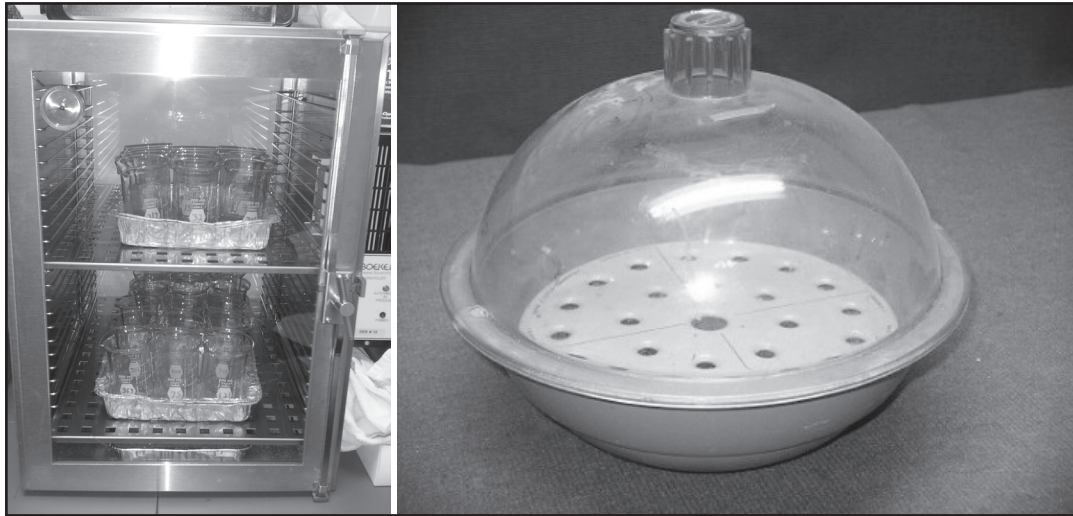
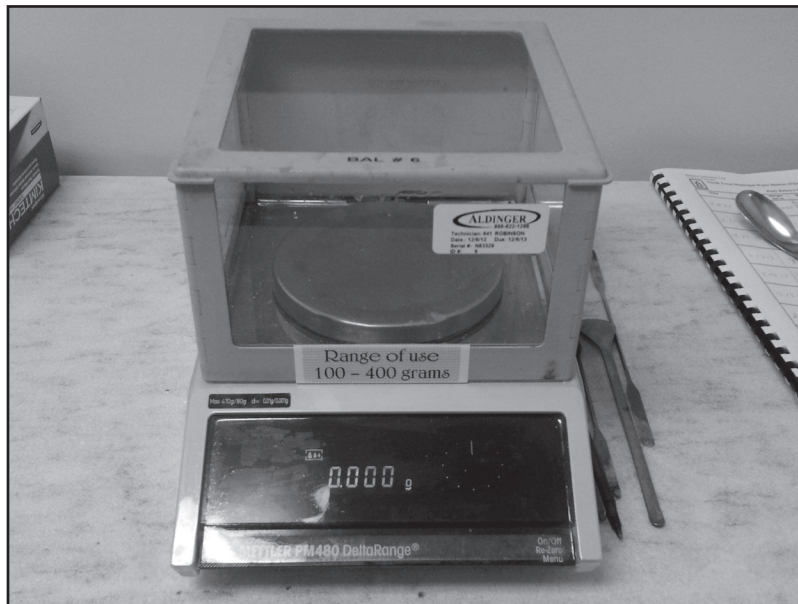


Figure 1-27 Desiccators



Courtesy of Denver Instrument.

Figure 1-28 Analytical balance

At least monthly, check the calibration of all balances using a set of S or S-1 weights with the range of checks that encompass the range of weights being measured. Please note that some programs require that balances be checked each day of use. Please be sure to check local, state or provincial, and federal regulations for any additional requirements. Routine checks should include a sensitivity check to confirm that the balance can determine the difference between 150.0 g and 150.1 g or a similar difference near the range of weights normally measured by the balance. If checks confirm a problem, balance maintenance is required. Place balances on heavy tables made to resist vibration (see Figure 1-29). Balances should be calibrated and certified annually by a third party.

The top-loading balance is also commonly used in laboratories to weigh larger quantities to less exacting standards than the analytical balance (see Figure 1-30).

A good housekeeping practice is to clean the balance pans with a camel-hair brush before and after using any balance.

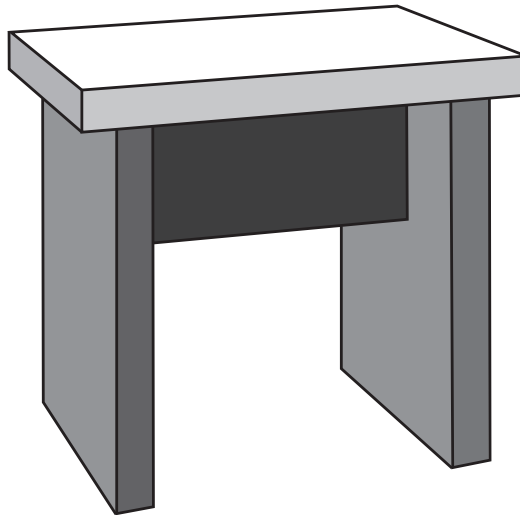


Figure 1-29 Balance table



Figure 1-30 Top-loading balance

A set of glass or plastic pans or weigh boats are desirable for weighing most solid chemicals needed to prepare standard solutions. The weigh boats save the metal pans of the balance from being attacked by corrosive chemicals. Place one weigh boat on the metal pan of the balance and adjust the weighing mechanism to exact zero (tare). Add the chemical with a spatula to the glass pan until it contains the correct quantity.

General Purpose Laboratory Equipment

The equipment discussed in this section is used for steps in a variety of laboratory procedures. For example, the oven is used for drying dissolved solids samples and for preparing glassware for specific analyses.

Autoclaves. Autoclaves, illustrated in Figure 1-31, are used to sterilize laboratory equipment, bacteriological media, and contaminated waste with pressurized steam. Use a maximum registering thermometer, autoclave tape, or spore strips with each autoclaving cycle to ensure sterilization temperatures have been reached.

Microscopes. Microscopes magnify extremely small objects so they can be seen (and often counted). Microscopic examination of water yields information on the presence and amounts of plant and animal life, such as algae, diatoms, protozoa, and crustacea.

Because microscopes are available in a variety of basic types, optional equipment, and prices, the purchase of a microscope requires research into the laboratory's needs and the best supplier to meet those needs. The manufacturer's representative should maintain the microscopes and instruct laboratory staff in proper use. The manufacturer's instructions should be followed carefully. Figure 1-32 shows a type of microscope typically found in water plant laboratories.

Muffle furnace. This high-temperature oven, shown in Figure 1-33, ignites and burns volatile solids. It is usually operated at temperatures near 550°C (1,022°F).

Oven. Ovens are used in the laboratory to dry, burn, or sterilize substances. An example of an oven typically found in laboratories is shown in Figure 1-34.

Vacuum pump. The pump in Figure 1-35 creates a vacuum for filtering laboratory samples. The membrane filter apparatus assembly (used with either a vacuum pump or aspirator) is shown in Figure 1-36.

Specific Purpose Laboratory Equipment

The laboratory equipment described in this section is used for specific procedures, for example, to measure conductivity, pH, for jar testing, and for color comparisons. Most need calibration to ensure the accuracy of their measurements.



Figure 1-31 Autoclave



Courtesy of Olympus Corporation.

Figure 1-32 Microscope



Figure 1-33 Muffle furnace



Figure 1-34 Oven



Courtesy of Gast Manufacturing Company.

Figure 1-35 Vacuum pump

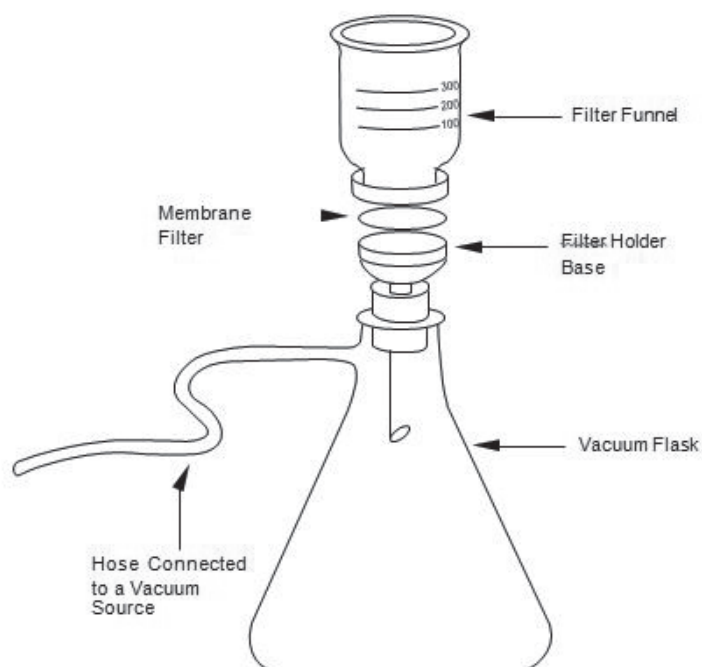


Figure 1-36 Membrane filter apparatus

Conductivity meters. Conductivity meters, such as the one shown in Figure 1-37, measure the ability of a solution to conduct an electric current. Generally, the lower the conductivity, the purer the water.

Jar-testing equipment. Essential to conventional coagulation/filtration water treatment plants, this equipment features six jars, either square or round, and variable speed paddles. Optional background light is particularly helpful when viewing small floc. See Figure 1-38.



Figure 1-37 Conductivity meter (or bridge)



Figure 1-38 Jar-testing equipment

pH meter. This essential piece of laboratory equipment is a voltmeter that indicates how basic or acidic a solution is by measuring the hydrogen-ion activity in a solution. Figure 1-39 shows a pH meter setup with the solution moving in the flask by means of a stir bar and magnetic stirrer. The manufacturer's instructions should be followed for calibrating the pH meter with standard buffers.

Turbidimeter. A turbidimeter (Figure 1-40) consists of a nephelometer with a light source to illuminate the sample and one or more photoelectric detectors with a readout device to indicate the intensity of light scattered at right angles to the path of the incident light.

Instrument sensitivity detects turbidity differences of 0.02 ntu or less in waters with a turbidity of less than 1 ntu. Instruments are available that can cover a range from 0 to 4,000 ntu.

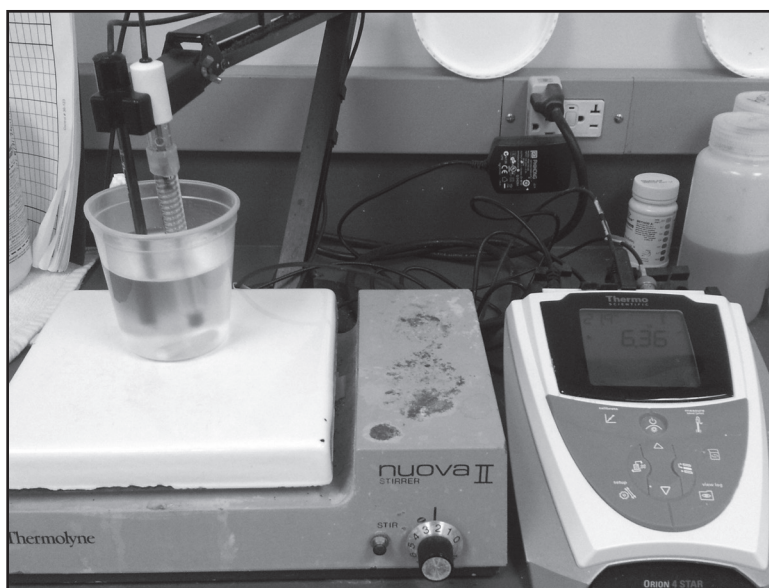


Figure 1-39 pH meter setup in a laboratory



Figure 1-40 Turbidimeter

Color comparisons. Several determinations in this manual specify the use of Nessler tubes for making color comparisons. For best results, place the sample in a tube identical to those of the color standards. The complete set of tubes should be matched; that is, they should be of the same size and have the same length of viewing path. Make the color comparison by looking into the tubes against a white surface to allow light to be reflected through the columns of liquid. Nessler tube sets are available in tall or short sizes. Generally, tall is preferable.

Comparators and test kits. Like standard solutions, permanent standards for color, turbidity, and many chemical substances are available commercially. The colorimetric kits come in two general forms: the disk type and slide types, which contains a color wheel or liquid standards in glass ampoules (Figure 1-41), and the photometer type, which is a simplified spectrophotometer (Figure 1-42). Please note that color wheels are not USEPA approved for compliance monitoring.

Check kits for reliability against a standard immediately after purchase and periodically thereafter.

Always be alert for any deterioration of the purchased reagents over time. If the reagents fail to yield reasonable results with the standard solutions, replace them immediately. When a test is performed at infrequent intervals, buy the needed solutions and reagents in small amounts, and date the bottles on delivery and when first opened so that inaccurate results based on use of out-of-date reagents may be avoided.

Test kits give rapid, acceptable, and consistent results. Their easy portability makes them useful for checking operations in the field. Many test kits are based on a simplified version of a test published in *Standard Methods*. The reagents come ready to use. Among the advantages of a test kit are freedom from having to prepare solutions and standards for every occasion; and an apparatus assembly designed specifically for the determination. However, the accuracy of tests run by kits may not equal that attainable in a good laboratory. Precision (the ability to reproduce the same result time after time) may approach the results obtained in the laboratory. Test kits should be used to monitor source and treated water only after trials demonstrate that the results on those particular waters equal or closely approximate values obtained by recognized and accepted standard methods. Do not use test kits for compliance with regulations unless the kit is USEPA approved.



Courtesy of Hach Company.

Figure 1-41 Color comparison kit with color wheels for several analyses