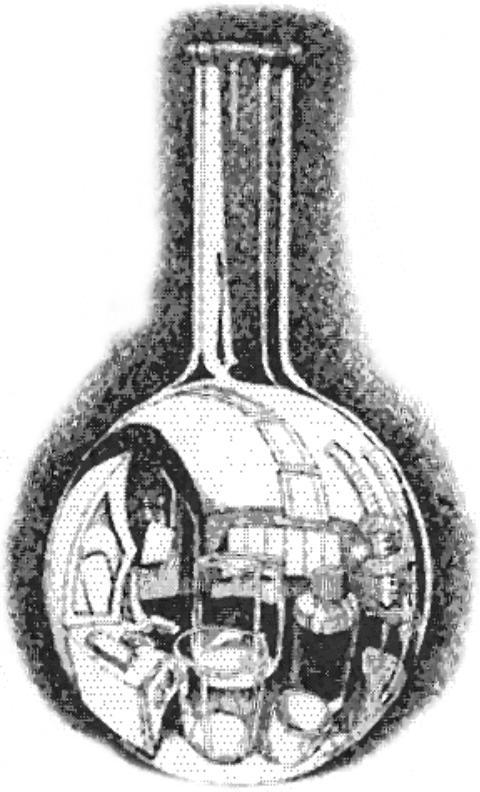
Basic Laboratory Skills

for Water and Wastewater Analysis



By Douglas W. Clark
Illustrated by Paul Lehrer

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Contents

Author and Illustrator

Mr. Clark is a laboratory consultant and editor of " The Bench Sheet, " a newsletter for water and wastewater analysts. He has authored numerous publications on water and wastewater laboratory methods, operations and administration, and was previously the supervisor of a municipal water quality laboratory.

Mr. Lehrer has been involved in the fine arts, illustration and design since moving to Albuquerque in 1968. His works have appeared in editions from Prentice Hall, the University of New Mexico Press and in private galleries and businesses throughout the southwest.

Acknowledgements

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This book is dedicated to the memory of John W. Clark, engineer, teacher, author, father and friend.

Preface

Mention laboratory analysis and you will almost certainly conjure up images in someone's mind of white-coated, absent-minded scientists, complex instruments studded with lights and dials, and mysterious solutions bubbling through a maze of tubing and glass. Such fantasies are reinforced by everything from Hollywood Frankensteins to press coverage of Nobel prize-winning chemists. Although day-to-day work in a water or wastewater laboratory obviously lacks this level of drama, it can nevertheless be difficult to shake the idea that lab work is an awesome task only to be conducted by some kind of 20th century wizard.

Laboratory analysis does require special equipment and skills. Most of the equipment and skills used in routine analyses, however, are relatively simple and can be mastered by individuals who make a conscientious effort to do so.

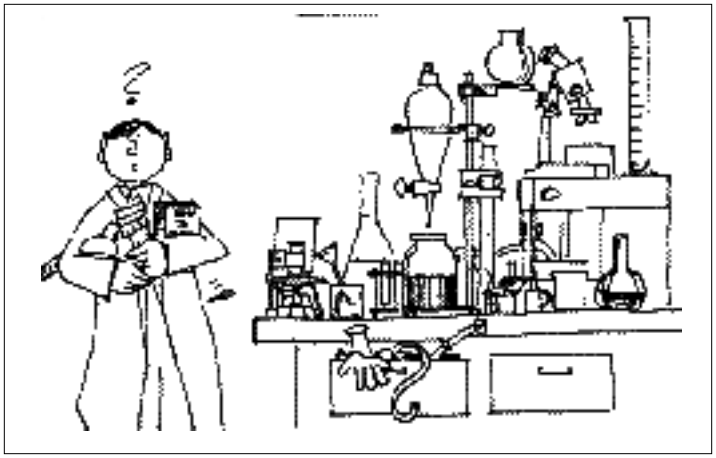
This handbook presents the basic skills necessary to function in a water or wastewater laboratory. It does not describe how to perform individual tests; rather it explains and illustrates the underlying techniques and principles involved. The emphasis is on correct use of routine equipment and proper laboratory procedures. The book is designed for anyone wishing to acquire an introductory knowledge of the subject, but who has not had formal training in laboratory skills. Considerable effort has gone into making the explanations and illustrations as clear and concise as possible. We have tried to strip away the aura of mystery surrounding laboratory analysis, while still retaining some of the sense of fun which should be part of any learning experience. We hope you will learn from this book, and we hope you will find the experience enjoyable.

Douglas W. Clark
Paul Lehrer

Introduction

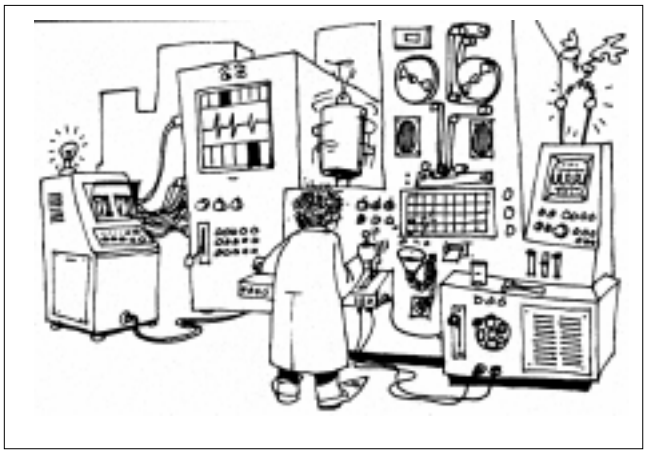
Laboratory analysis is a practical process. Through it, you can obtain information you need to operate your water or wastewater system more effectively. All you have to do is . . .

...have the right equipment and supplies and use them properly (chapter 1)...



...know how to use laboratory balances (chapter 2)...

...know how to use volumetric glassware (chapter 3)...



... be able to make other analytical measurements when they are called for (chapter 4)...

...conduct all procedures with minimum of damage and injury (chapter 5), and...

...keep proper records and ensure that your data is reliable (chapter 6)...

...all carried out with an understanding of the procedures involved and the principles behind them (appendix).

If you have problems in any of these areas, this book is designed to help.

General Techniques

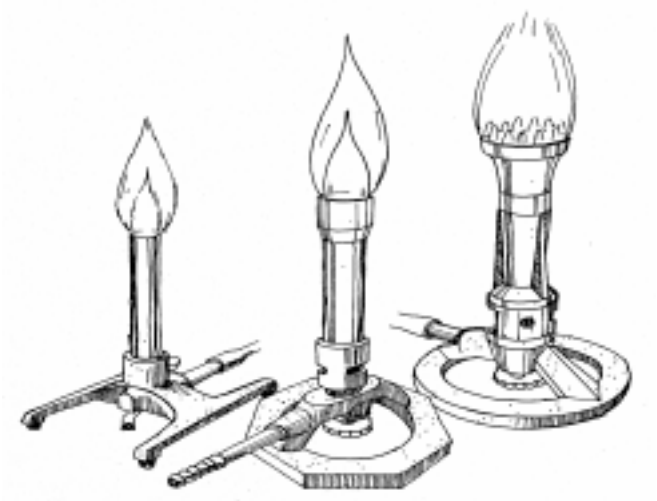
The purpose of laboratory analysis is to make measurements, and much of the equipment in the lab is designed to accomplish this task. For example, balances measure weights, volumetric glassware measures liquid volumes and pH meters measure electrochemical potentials. But many items in the laboratory provide support services, particularly temperature control. An understanding of how to use these items is critical if later measurements are to be performed properly. In addition, certain techniques must be followed when working with glass and when handling solid and liquid reagents. This chapter covers some of the general laboratory techniques and equipment which will be used later when making actual measurements.

LABORATORY BURNERS

Burners are used to heat solutions, dry samples, ignite substances and soften glass for glassworking. Although electric heating devices have taken over many of the functions of burners in recent years, they are still common laboratory items and must be used for certain applications. Bunsen and Tirrill burners produce cone-shaped flames and are the most widely used laboratory burners. Fisher burners provide much higher temperatures with a more concentrated flame.

Air flow to a burner flame is controlled by vents in the side of the burner near the base. The vents may be regulated either by turning the barrel of the burner, or by adjusting a sleeve around the barrel, depending upon the burner design. Most burners also have a gas control valve at the base of the burner which is used to regulate the height and intensity of the flame. The hottest part of a burner flame occurs just above the pale blue cone inside the base of the flame. (With a Fisher burner, there will be many of these small, pale blue cones inside the flame)

To ignite a burner, open the air vents and the burner gas valve about half way. Place a lit match or a striker next to the top edge of the burner and turn on the gas cock at the bench. If the gas is lit from above the burner, it may flare up or go out. If too much air and gas flow through the burner and the flame is extinguished, turn off the gas cock and partially close the air vents in the barrel of the burner. Then repeat the ignition procedure. After the flame is lit, keep the gas cock at the bench open all the way and regulate gas flow with the valve in the burner base. If a burner begins to flame around the base, immediately shut off the gas cock at the bench and wait for the burner to cool. Then reduce the amount of air reaching the flame by partially closing the air vents and ignite the burner again.



BURNERS (LEFT TO RIGHT): BUNSEN, TIRRILL AND FISHER

WORKING WITH GLASS

Normally, soft glass tubing is used for glassworking in the laboratory. To cut a piece of tubing, scratch it firmly once or twice at the desired point with a triangular file. Wrap the tubing near the scratch with a towel or wear gloves to protect your hands. Put your thumbs close together on the opposite side of the tubing from the scratch. Then push out with the thumbs while pulling the ends of the tubing apart. Fire glaze piece of cut glass in a burner flame to smooth any rough or jagged edges. Hold the cut end of the tubing in the flame and rotate it until the glass begins to soften visibly. Too much heating should be avoided as it will cause the glass to droop and sag.

When bending glass, attach a flame spreader to the burner top to obtain a broad, even flame. Adjust the width of the flame spreader if the flame does not burn evenly across the top at first. Roll the tubing back and forth near the top of the flame until it becomes very soft. Take it from the flame, still holding the tubing at both ends, and wait a few seconds for the heat to become uniform. Then bend the tubing quickly and hold it until it hardens. Do not set the glass on a cold surface while it is hot as the thermal shock may shatter or fracture the glass.

Tubing, thermometers, funnels and other glassware must often be inserted into rubber stoppers for use in the laboratory. Attempting to do this improperly can result in shattered glassware, ruined stoppers and personal injuries. Knit cotton, leather, or rubber- or plastic-coated cotton gloves should be worn when working with glass to protect your hands. If these are not available, wrap both hands in the opposite ends of a towel. Hold the stopper in one hand and the glass tubing (or other piece of glassware) in the other. Grasp the tubing near the end to be inserted and moisten it with glycerine or soapy water. Twist the tubing while pushing it gently into the stopper hole to the desired depth. Finally, rinse the glycerine or soap from the tubing and the stopper. Never try to withdraw glassware from a stopper once it has been inserted. If the glassware must be removed, carefully cut the stopper from around it and discard the stopper.

HOT PLATES

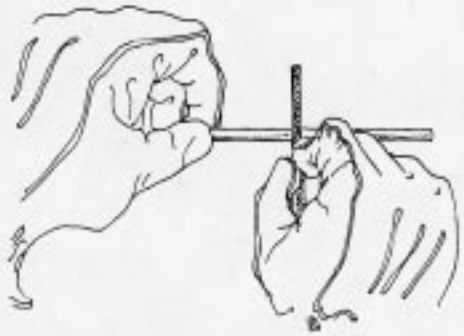
Electric heating devices have assumed many of the roles formerly filled by laboratory burners, particularly where constant, controlled heating is required over extended periods or where open flames present a serious hazard (as when working with flammable reagents). Electric hot plates, for example, are commonly used to heat solutions in flat -bottomed glassware such as Erlenmeyer flasks and beakers. They usually have a temperature range of about 40-350° C and heat up in 5-10 minutes. A burner should be used where higher temperatures or faster warm-up times are needed.

HOT PLATE/MAGNETIC STIRRER

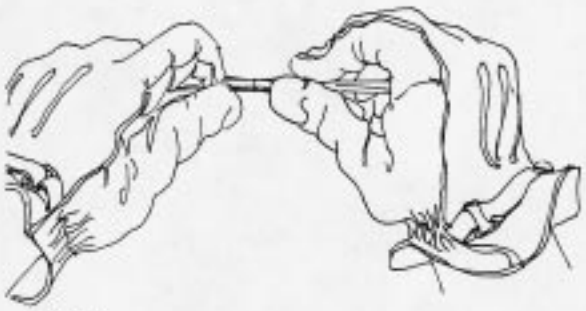
The amount of heat produced by a hot plate is controlled by an adjustable transformer or rheostat. This appears as a control knob on the face of the hot plate and is graduated in numbers, such as from one to five. If these numbers are not assigned specific temperatures in the manufacturer's operations manual, the hot plate can be calibrated in the laboratory. Set a beaker of a smokeless, high boiling, nonflammable oil, such as mineral oil, on the hot plate and suspend a thermometer that reads up to 300° C in the oil. Do not allow the thermometer to touch the beaker. Turn on the hot plate and set the control knob to the first number. Stir the oil occasionally, being careful not to damage the thermometer. When the temperature of the oil stops rising, record the temperature along with the control knob setting in a laboratory notebook. Then proceed to the next higher number, repeating the procedure until temperature values have been obtained for each number on the knob.

Working with Glass

Cutting

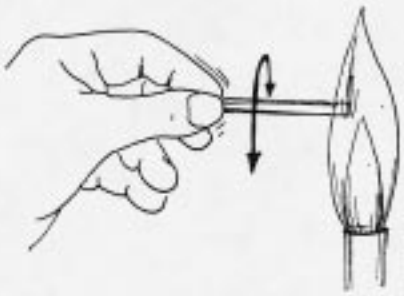


STEP #1 - SCRATCH ONCE WITH TRIANGULAR FILE.



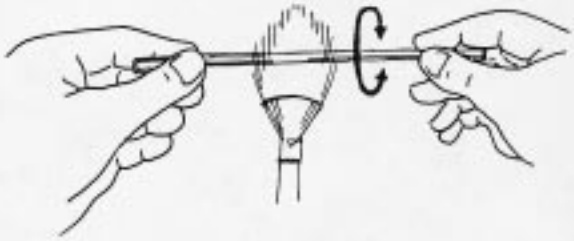
STEP #2 - PLACE THUMBS TOGETHER ON SIDE OPPOSITE SCRATCH.

STEP #3 - BEND TUBING WHILE PULLING ENDS APART.

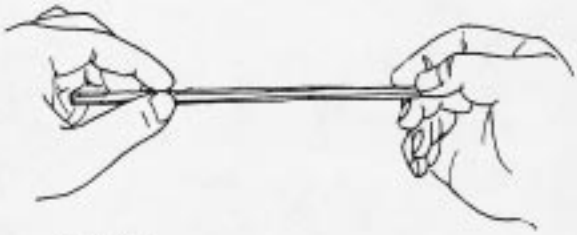


STEP #4 - FIRE GLAZE CUT ENDS TO ELIMINATE JAGGED EDGES.

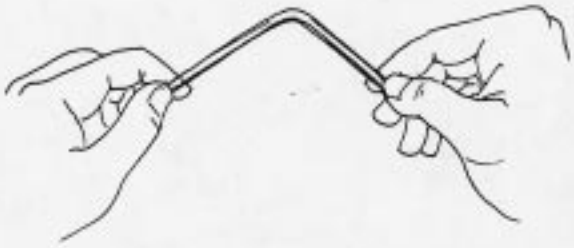
Bending



STEP #1 - ROLL TUBING IN A FLAT FLAME UNTIL GLASS IS SOFT.



STEP #2 - REMOVE FROM FLAME AND HOLD FOR A MOMENT TO LET HEAT BECOME UNIFORM.



STEP #3 - BEND QUICKLY AND HOLD UNTIL HARD.

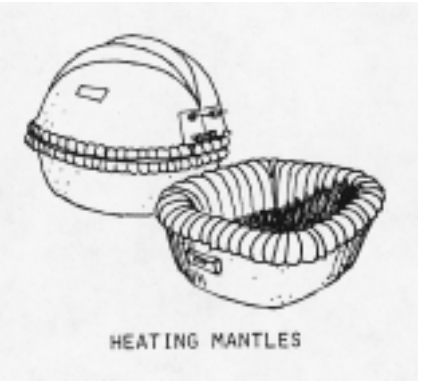
EXAMPLE BENDS - GOOD BEND (ABOVE) AND POOR BEND (BELOW).

MAGNETIC STIRRERS

Many laboratory hot plates also function as magnetic stirrers, although both items can be obtained separately. A combined hot plate-stirrer is very versatile since it can be used to stir solutions while they are being heated, or either the hot plate or the stirrer can be used independently. Teflon covered magnets are placed in a solution to be stirred by tilting the vessel and carefully allowing the magnet to slide down the side. To begin stirring, turn the speed control knob on the lowest setting and then switch on the unit. As the magnet begins slowly turning in the solution, increase the speed control knob gradually. Occasionally, the contents of the vessel may have to be swirled to dislodge the magnet if it fails to spin freely at first. If the magnet begins to wobble excessively or starts vibrating without spinning as the speed is increased, stop the stirrer and return the speed control to the lowest setting. Then begin the procedure again. Be careful not to stir solutions so violently that they splash out of their containers.

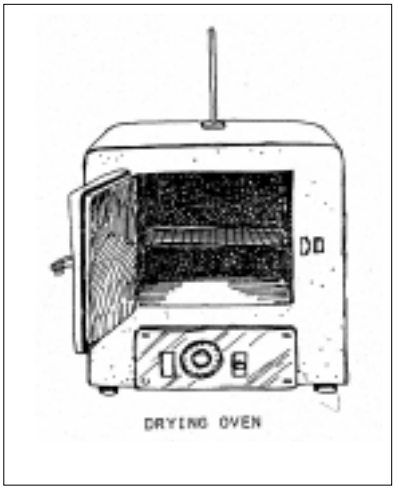
HEATING MANTLES

Heating mantles are essentially electric blankets specially designed to fit snugly around round-bottom flasks. The heating elements are covered with asbestos, which reduces damage to the glassware, while the molded form enables greater temperature control in the heated solution. The amount of heat produced is controlled by an adjustable transformer or rheostat, similar to those on hot plates. Again as with hot plates, the numbers shown on a heating mantle temperature control knob are usually arbitrary, but can be calibrated by heating a flask of oil in the mantle and reading the temperature of the oil at each number setting. These calibrations should be recorded in a laboratory notebook.


HEATING MANTLES

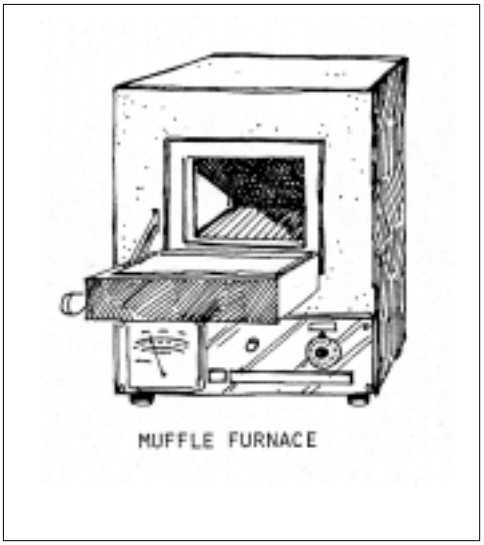
OVENS

Laboratory ovens are used for drying samples and reagents, sterilizing glassware and similar functions. Heating coils may be distributed throughout the bottom, sides and top of an oven and the heat is distributed either through convection or, in the more efficient models, by fan. Ovens should be equipped with friction latches so the pressure from an explosion inside the oven would be released without blowing off the door. The temperature in an wen is monitored by a thermometer extending through the top, with the bulb inside the oven and the temperature scale protruding above so it can be read without opening the door. It is recommended that the thermometer bulb be placed in a beaker of fine sand to reduce rapid fluctuations in the temperature reading when the oven door is opened or closed. A routine record should be kept of the oven temperature prior to its first use for the day for quality control purposes.


DRYING OVEN

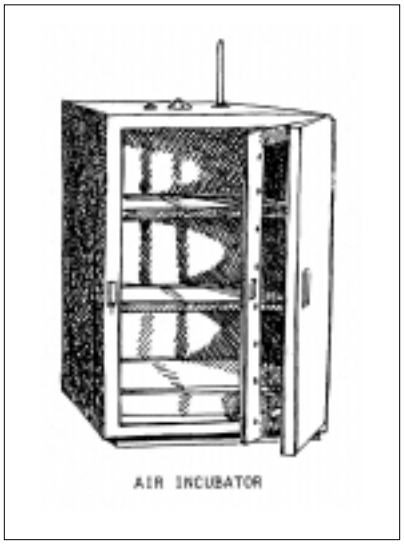
The temperature control knob on most ovens will be graduated with arbitrary numbers and should be calibrated against thermometer readings for the actual temperatures obtained with each knob setting. These calibration values should be recorded. If an oven is to be used for hot-air sterilization, it should first be checked to ensure that it will hold the required temperature of 170° C + or - 10° C for a two hour period. When in use, sufficient space should be left around the items in the oven to permit heat to be distributed evenly throughout the interior.

MUFFLE FURNACES


MUFFLE FURNACE

Muffle furnaces heat materials to temperatures from about 180° C to 1100°C. A common use of a muffle furnace is to burn off the organic portions of a sample, leaving only the inorganic or ash portion in the crucible or other container. Temperatures in a muffle furnace are measured by a pyrometer, which is part of the furnace, rather than by thermometer. The pyrometer scale will usually appear on the base of the instrument, along with the temperature control knob. If the temperature control knob has not already been calibrated for specific temperatures, this should be done and the results recorded. Because they heat up slowly, muffle furnaces should be turned on at least an hour before use. Be careful not to leave a muffle furnace turned up all the way or it may overheat and damage the heating elements. Always use asbestos gloves and tongs to place items in a muffle furnace or to remove them. Never touch the door or walls of a muffle furnace when it is operating as they will be extremely hot and might cause burns even through insulated gloves.

INCUBATORS

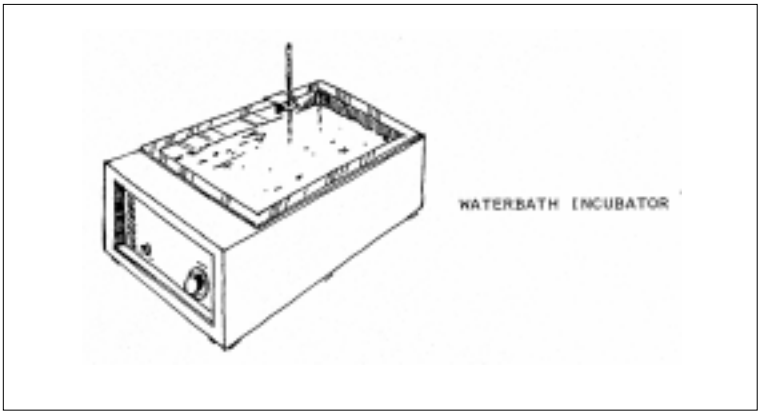

AIR INCUBATOR

Constant, known temperatures are critical for bacteriological work. If the temperature varies from the intended setting, or if the wrong temperature is selected, the results of a test may appear normal while in fact being completely invalid. Incubators therefore perform a crucial role in water quality analysis and careful attention must be paid to their operation.

Most incubators perform similarly to ovens, except that the temperature control is usually more precise. Water-jacketed incubators are preferred over those with other types of insulation. Incubators should be equipped with low-temperature heating elements, rather than high temperature ones, to prevent the formation of local hot spots during operation. Temperature control in incubators with mechanical air circulation is superior to that in incubators using only convection. Incubators equipped with recording thermometers are recommended. Where these are not available, temperature readings should be taken at least once, preferably twice daily during use. Thermometer bulbs should be immersed in a liquid (glycerine, water or mineral oil) to minimize the effects of sudden temperature changes when the incubator door is opened. Humidity in air incubators should be kept high to prevent culture media from drying. This can be achieved by partially submerging a towel in a beaker of water and placing them both in the incubator. Some incubators have built-in water reservoirs which can be used instead.

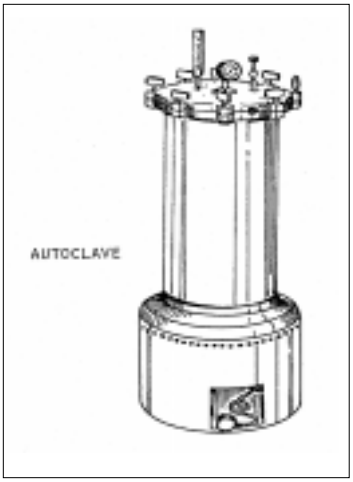
BOD incubators must be kept at 20° C + or - 1° C. Because of the relatively low temperature requirements of this test, a cooling system must be present in the incubator as well as heating elements. For this reason, BOD incubators are frequently made from converted refrigerators. Incubators for total coliform analysis must be capable of maintaining a temperature of 35° C + or - 0.5° C. Fecal coliform analysis requires a temperature of 44.5° C + or - 0.2° C, which cannot be maintained with an air incubator. Instead, either a water bath or a heat sink incubator (for example, one using an aluminum

block) should be used for this test. Although mechanical circulation of the water in a water bath incubator may not be essential to maintain the temperature within the required range, it does greatly improve temperature control. Water baths which lack means for mechanical circulation should at least be



provided with gabled covers to reduce water and heat losses.

AUTOCLAVES



Autoclaves are glorified pressure cookers. They sterilize equipment and culture media by exposing them to steam at 121° C for 15 minutes, which also requires the contents of an autoclave to be under pressure. In fact, household pressure cookers, as well as vertical autoclaves, can be used when necessary if they are equipped with pressure gauges and thermometers whose bulbs are placed one inch above water level. Generally, however, specially constructed autoclaves should be used instead. These should be large enough to allow plenty of room for the steam to circulate when the autoclave is loaded and should be provided with an accurate thermometer with its bulb located in the exhaust line to measure the minimum temperature in the sterilizing chamber. A recording thermometer is a desirable option. Autoclaves should be capable of reaching sterilization temperature within 30 minutes. They should be clean and inspected to prevent damage or deterioration.

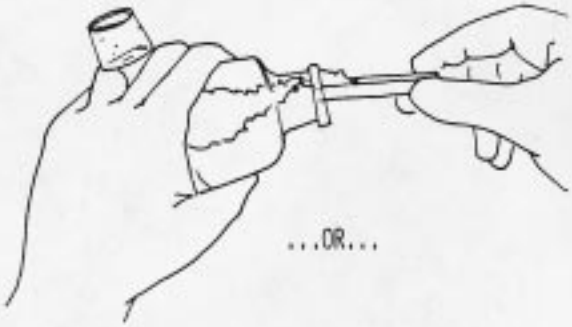
Labeling tapes having heat-sensitive inks which undergo color changes at sterilization temperature, or similar temperature indicators, should be used during sterilization to monitor heat penetration throughout the autoclave.

CHEMICAL REAGENTS & BACTERIOLOGICAL MEDIA

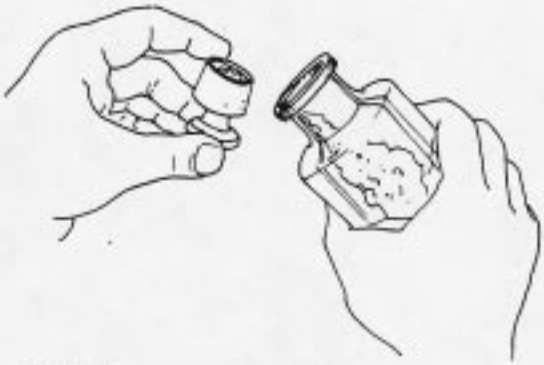
It is essential to maintain the purity of chemicals in reagent bottles and bacteriological media containers. For this reason, withdraw only as much of such material as is needed at a time and never return unused portions to the original containers. Avoid contamination of bottle stoppers. Usually, these should be held in one hand while a bottle is open rather than being placed on the bench top. If a stopper must be set aside, it should be placed upside down so no bench surface comes into contact with the inside portion of the stopper.

Handling Solid Reagents

STEP #1 - TILT BOTTLE AND ROLL UNTIL REAGENT FILLS GLASS STOPPER.



STEP #1 - USE A SPATULA TO REMOVE SUFFICIENT REAGENT.



STEP #2 - REMOVE STOPPER WITH REAGENT STILL IN IT.

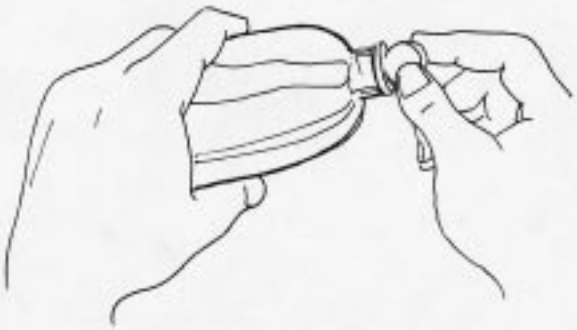
STEP #2 - TAP THE SPATULA UNTIL ENOUGH REAGENT HAS FALLEN OUT.

STEP #3 - TAP STOPPER WITH A PEN OR PENCIL UNTIL ENOUGH REAGENT HAS FALLEN OUT.

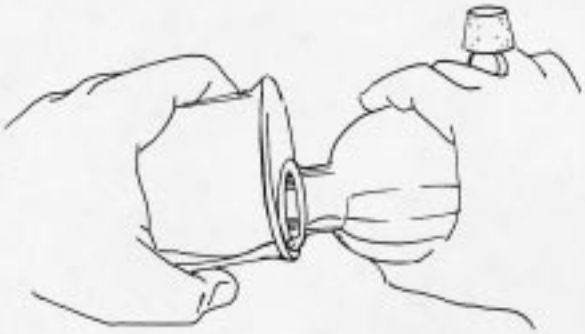
...OR...

SINGLE STEP - TILT BOTTLE AND ROLL UNTIL ENOUGH REAGENT FALLS OUT.

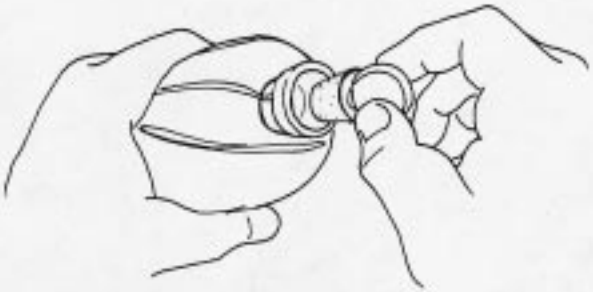
Handling Liquid Reagents



STEP #1 - HOLD THE STOPPER IN WHILE WETTING IT WITH CONTENTS OF THE BOTTLE.



STEP #4 - CONTINUE HOLDING STOPPER WHILE GENTLY POURING REAGENT INTO RECEIVING CONTAINER.



STEP #2 - USE WET STOPPER TO MOISTEN NECK AND LIP OF BOTTLE TO PREVENT LIQUID FROM GUSHING OUT WHEN POURED.

WHENEVER POSSIBLE, POUR LIQUIDS DOWN A GLASS ROD.

STEP #3 - REPLACE STOPPER AND WITHDRAW IT AGAIN WITH THE BACK OF THE HAND.

HOLD THE ROD THIS WAY TO POUR FROM A BEAKER.

Whenever possible, reagents should be of ACS (American Chemical Society) grade. Where this specification is unavailable, order reagents labeled “analytical reagent grade” or “spectral grade organic solvents. “

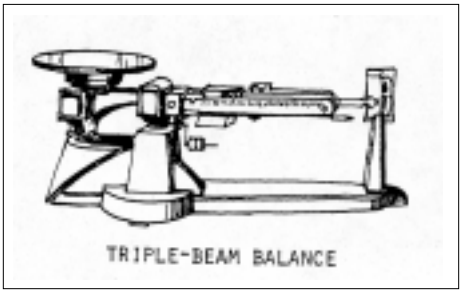
Anhydrous reagent chemicals needed for preparation of standard calibration solutions and titrants should be dried in an oven at 105-110° C for at least one or two hours, preferably overnight. Weigh the amount required promptly after the reagent has cooled to room temperature in a desiccator. For hydrated salts, drying in an efficient desiccator may be substituted for oven-drying. Some standardized chemical solutions change over time due to chemical or biological action. Other solutions may be chemically stable, but their concentrations may change if they are stored for extended periods of time. A standard cannot be considered valid for more than one year without restandardization even when evaporation is minimized. Concentrations of solutions in partially full bottles or bottles which are opened frequently may change within a few months.

Dehydrated bacteriological media should be stored in tightly closed bottles in the dark at less than 30° C. They should also be kept at low humidity. If the contents become discolored or caked, they should be discarded. Purchase dehydrated media in small quantities which will be used within six months after opening. Prepare culture media in batches of such size that an entire batch will be used in less than one week.

Measuring Weights

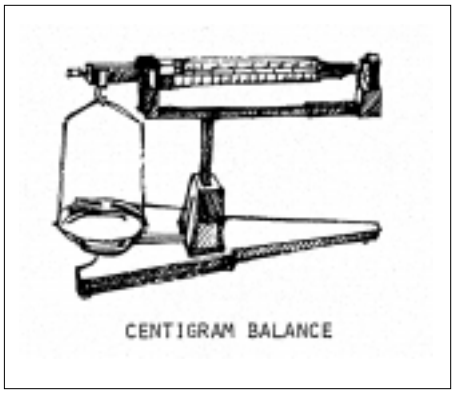
Weight is involved in almost every analysis conducted in a water or wastewater laboratory. In some analyses, weight is the primary measurement performed, as in suspended solids or total dissolved solids tests. In other cases, weight is a less obvious -- though no less significant -- measurement, as when it is used in preparing standard solutions and stock reagents. Weight is therefore one of the most basic and most important measurements performed in the laboratory.

Approximate Weights


TRIPLE-BEAM BALANCE

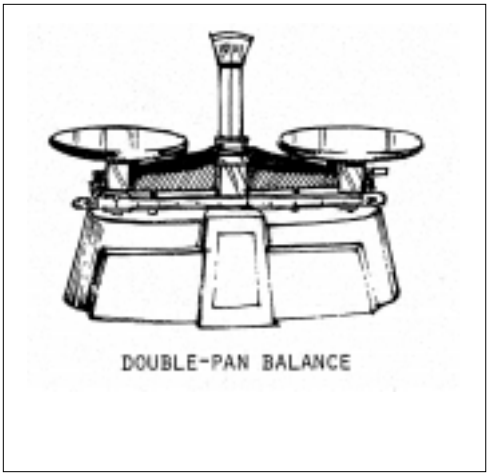
Sometimes only approximate weights of a substance or object are necessary. Such weights will be specified to the nearest gram, tenth of a gram or, at most, the nearest hundredth of a gram. Three or four decimal place accuracy is not required for these weighings and an analyst need not use delicate, precision instruments to make them. Instead, several types of cheaper, simpler balances are available for this work. They include triple-beam, centigram and double-pan balances. Triple-beam and double-pan balances weigh to the nearest 0.1 g. Triple-beam balances are capable of weighing objects up to about 600 g. Centigram balances weigh to the nearest 0.01 g and usually have a 110 g maximum capacity.

When an object to be weighed has been placed on the pan of a triple-beam balance, the beam pointer will swing up to the top of the scale. To determine the weight of the object, start with the largest reference weight (the one on the scale marked in 100 g increments) and move it along the beam one notch at a time. When a notch is reached where the beam pointer swings below the zero mark on the pointer scale, move the weigh back one notch. Then follow the same procedure with the second largest reference weight (on the scale marked in 10 g increments). Finally, slide the third and smallest reference weight along the beam until the pointer swings equal distances above and below the zero mark. The weight of the object is the total of all the values indicated by the reference weights on the beam. After recording this total, return the reference weights to zero and gently remove the object (lock the beam and pan arrests if these are present). Check to ensure that the balance is still zeroed. If it is not, the balance must be re-zeroed and the entire weighing procedure repeated.


CENTIGRAM BALANCE

A centigram balance is operated in much the same manner, except that the largest reference weight may be marked in 10 g increments because of the balance's lower total weight capacity. Also, the smallest reference weight will be divided into 0.019 increments rather than the 0.19 increments found on the smallest reference weight scale on triple beam balances.

When using a double-pan balance, the object to be


DOUBLE-PAN BALANCE

weighed should be placed on the left pan. The beam pointer is then brought back to zero by sliding the reference weights along the scale between the pans. If the object exceeds the capacity of the sliding weights on the balance, calibrated weights must be placed on the right pan to bring the difference between the pans to within the instrument's capacity for measurement. The weight of the object is then calculated as the total of the values indicated by the sliding weights plus the sum of the weights (if any) on the right pan. Because of this technique, a double-pan balance can be used to weigh heavier objects than can be weighed on either triple-beam or centigram balances.

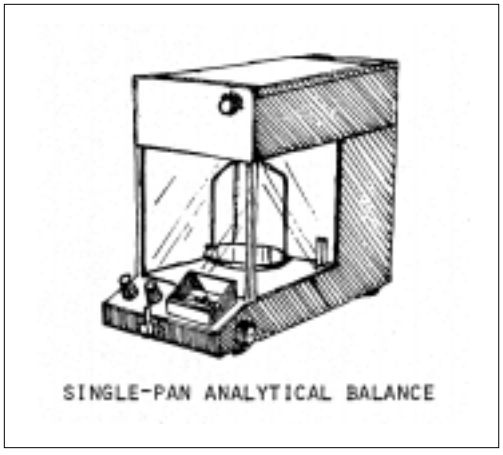
Analytical Balances

Where objects or substances must be weighed with greater accuracy than is obtainable from triple-beam, centigram or double-pan balances, an analytical balance should be used. Analytical balances generally have a maximum capacity of about 200 g and can weigh an object to the nearest 0.00019 (0.1 mg). These balances may be either of two kinds: double-pan or single-pan.

For many years, the classical double-pan, equal-arm analytical balance was the standard instrument for water quality analysis. They are still found in some laboratories. One disadvantage to this type of instrument, however, particularly where laboratory personnel have not had extensive formal training in quantitative analysis, is that the double-pan balance requires considerable time and skill on the part of the analyst. Most laboratories have therefore now abandoned the double-pan analytical balance in favor of the faster and much more convenient electric single-pan balance.

The Single-pan Balance

In a single-pan analytical balance, a set of calibrated weights is suspended from the balance beam, directly over the pan. These weights are generally hidden inside the instrument and are manipulated by control knobs on the outside of the balance case. When the balance is zeroed, the combined effect of the weights and the pan on one end of the beam is exactly offset by a fixed counterweight on the other end of the beam. Weighing is then accomplished by substitution: a sample object is placed in the pan and the beam becomes heavier on that side. The counterweight is not sufficient to offset the combined weight of the pan, object and calibrated weights. So calibrated weights are removed from the beam using the control knobs on the outside of the case until the total of the remaining weights, the pan and the sample object is again exactly offset by the counterweight. The weight of the object is equal to the


SINGLE-PAN ANALYTICAL BALANCE

sum of the weights removed from the beam; this sum shows up on indicator scales on the face of the balance.

A delicate knife edge supports the balance beam when the instrument is in use. This knife edge is protected when the instrument is not being used by a beam release knob on the outside of the case. The beam release knob has three positions. In the vertical position, the beam is arrested and out of contact with the knife edge. The balance pan should be loaded or unloaded in this position. When the knob is turned clockwise, the balance is partially released and the larger increments of the calibrated weights can be safely manipulated. When the knob is turned counterclockwise, the beam is fully released and very small weight adjustments can be made (as when the zero point is set) or the results of a weighing operation can be read.

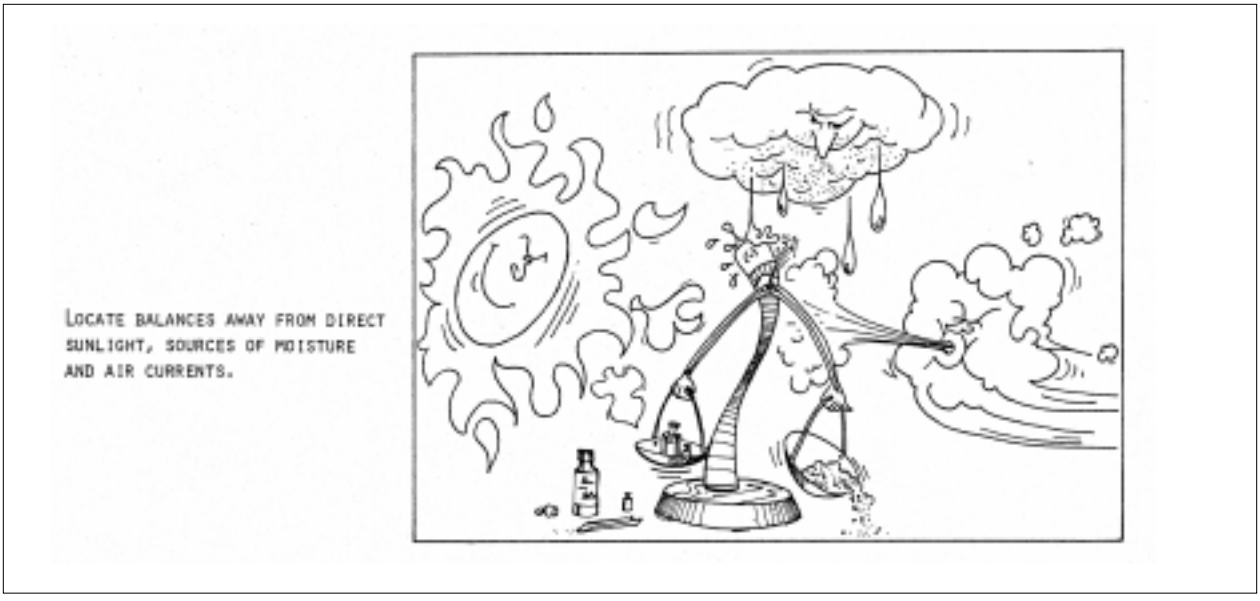
A special dampening device stops the beam from swinging when the balance is used. Because of this, the beam is not moving when the instrument is in use and the zero point can be rapidly set by means of a knob. The zero point and small fractional weights are read on a scale projected onto a ground glass screen by an optical system inside the balance.

Single-pan analytical balances can be operated very rapidly even by relatively unskilled personnel. With practice, a single weighing can be accomplished in less than a minute. Important sources of potential error are eliminated because rest points and total weight values need not be calculated by the analyst. While there may be a slight loss in sensitivity with a single-pan analytical balance as compared to the double-pan kind, this is minimal and not of any serious consequence in most water and wastewater laboratory applications. The convenience of the single-pan instrument more than makes up for this slight limitation.

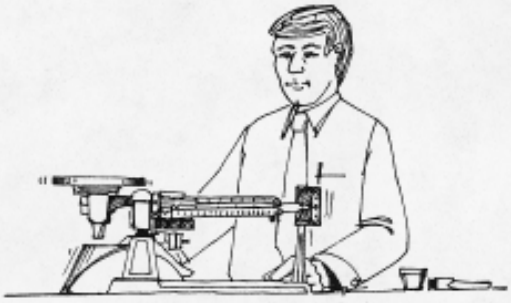
Using Laboratory Balances

The following rules should be observed in caring for and using any kind of laboratory balance:

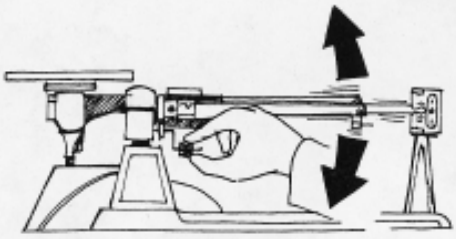
Balance location. Balances should be located away from direct sunlight, unusual heat, sources



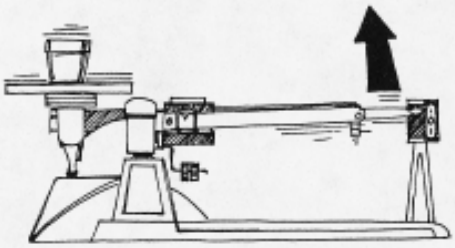
Using a Triple-Beam Balance



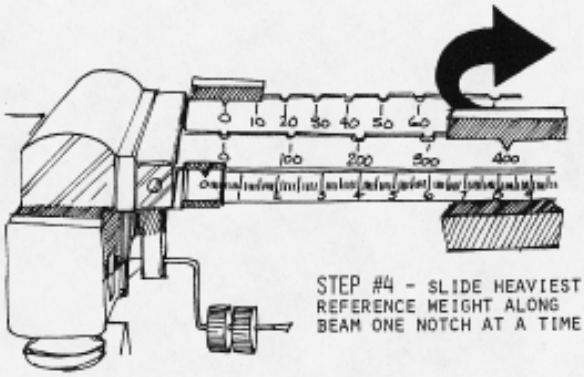
STEP #1 - MOVE BALANCE AROUND UNTIL STABLE.



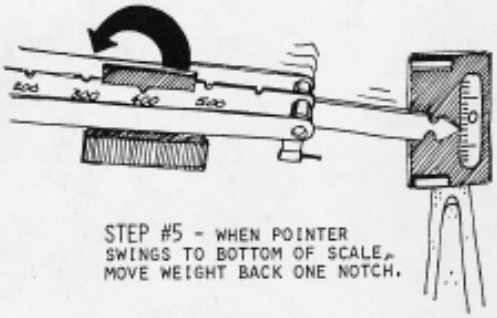
STEP #2 - WITH REFERENCE WEIGHTS AT THE FAR LEFT, ZERO THE BALANCE.



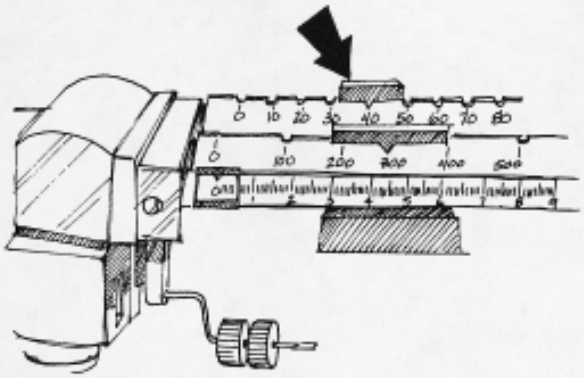
STEP #3 - PLACE OBJECT TO BE WEIGHED ON PAN. BEAM POINTER WILL SWING TO TOP OF SCALE.



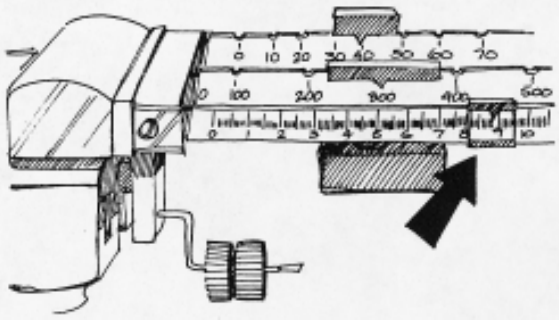
STEP #4 - SLIDE HEAVIEST REFERENCE WEIGHT ALONG BEAM ONE NOTCH AT A TIME.



STEP #5 - WHEN POINTER SWINGS TO BOTTOM OF SCALE, MOVE WEIGHT BACK ONE NOTCH.

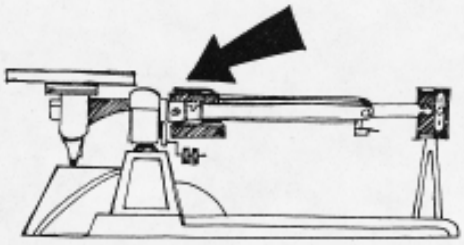


STEP #6 - REPEAT STEPS 4 AND 5 WITH NEXT HEAVIEST REFERENCE WEIGHT.

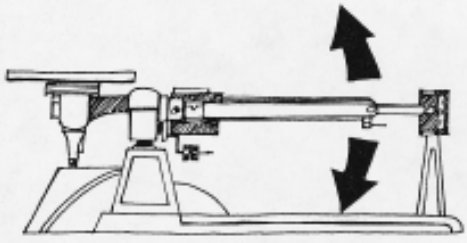


STEP #7 - SLIDE SMALLEST REFERENCE WEIGHT ALONG SCALE UNTIL POINTER IS AGAIN ZEROED.

STEP #8 - ADD VALUES INDICATED ON EACH SCALE AND RECORD TOTAL WEIGHT.



STEP #9 - RETURN ALL REFERENCE WEIGHTS TO ZERO AND REMOVE OBJECT FROM PAN.



STEP #10 - CHECK ZERO. IF BALANCE IS NOT STILL ZEROED, REZERO IT AND REPEAT ENTIRE WEIGHING PROCEDURE.

of moisture, air currents and heavy laboratory traffic. Analytical balances should be placed on sturdy tables, preferably ones designed specifically for this purpose.

Leveling the balance. Before using any balance, it should first be leveled. Many balances are provided with level indicators, which use air bubbles floating inside a fluid-filled glass dome. A circle, slightly larger than the diameter of the bubble, is etched into the glass. If the bubble is centered within that circle, the balance is level. If the bubble over laps the circle, the balance is out of level and should be adjusted. Most balances which have level indicators also have leveling screws for adjusting the instrument. In balances where these are lacking, the balance must be moved around on the bench top until it does not rock or wobble.

Zeroing the balance. Balances must also be zeroed before use. The beam pointer on a triple-beam, centigram or double-pan balance should be pointing directly at zero if the pointer is stationary (not moving). If the pointer is swinging back and forth, it should swing equal distances to either side of the zero mark. If the pointer is swinging further to one side of zero than to the other, slowly turn the zero control knob or screws until the distances are equal. On a single-pan analytical balance, the zero is set by adjusting a knob outside the balance case.

Beam and pan arrests. The more delicate balances are protected by beam and pan arrests. These should be in the locked position when the balance is not being used and when changing weights on the balance pans. With balances which have no beam or pan arrests, place objects or weights on the pans very carefully to avoid damaging the balance.

Manipulating weights. Calibrated reference weights should be manipulated only with forceps or some similar device, never with fingers. Moisture and oils from the skin can change the value of the weights. Objects to be weighed should also be handled with forceps or tongs for precise weighings.

Heavy weights and objects. Never overload a balance. Check the maximum rated capacity prior to use. Place heavy weights and objects in the center of the balance pans to keep the pans from swinging.

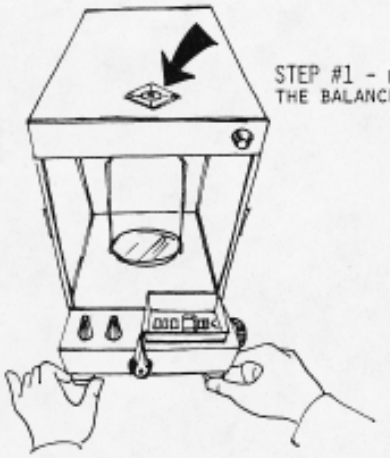
Weighing chemicals. Chemicals and corrosive materials should always be weighed in appropriate containers, never directly on the balance pans. Spilled chemicals should be removed and the balance cleaned immediately. Balance pans should also be cleaned daily with a camel's hair brush to remove dirt and dust.

Calibration and servicing. Balances, particularly analytical ones, should be calibrated and serviced annually. Calibrate analytical balances with National Bureau of Standards (NBS) Class S-1 weights.

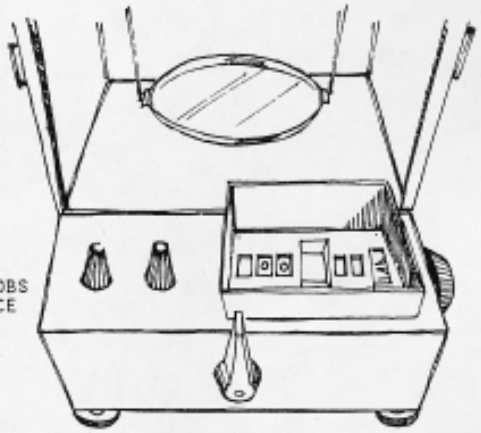
Sources of Error

Even when the above rules are followed, several potential sources of error may interfere with results. Errors can be caused by such factors as moisture, temperature and static electricity. Each of these sources of error must be considered when weighings are being made.

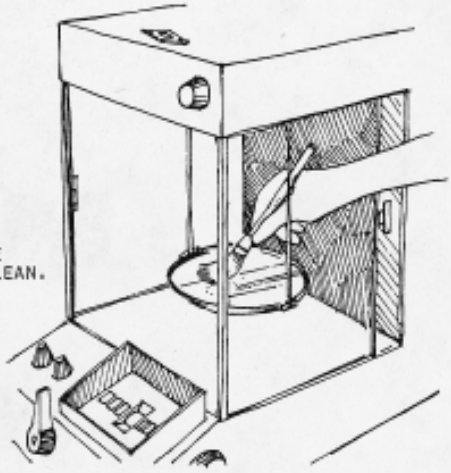
Using an Analytical Balance



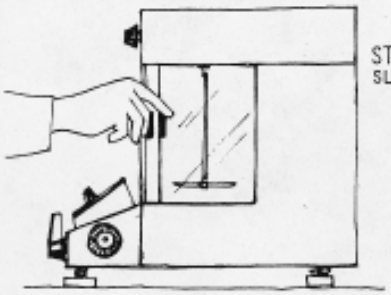
STEP #1 - LEVEL THE BALANCE.



STEP #2 - BE SURE KNOBS ARE ZEROED AND BALANCE IS ARRESTED.

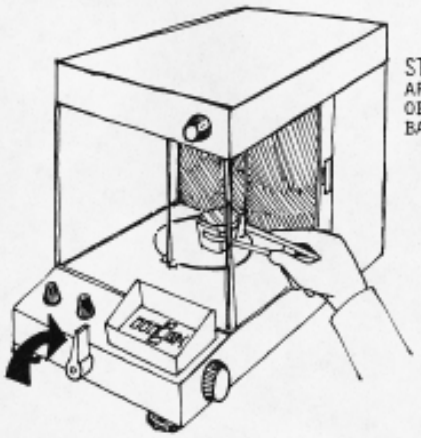


STEP #3 - BE SURE BALANCE PAN IS CLEAN.

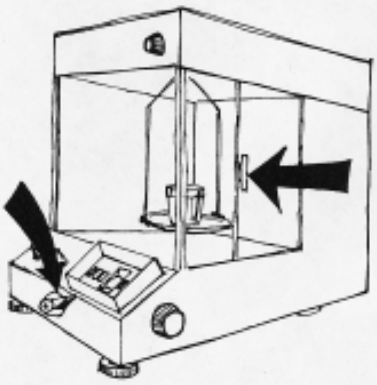


STEP #4 - CLOSE SLIDING DOORS.

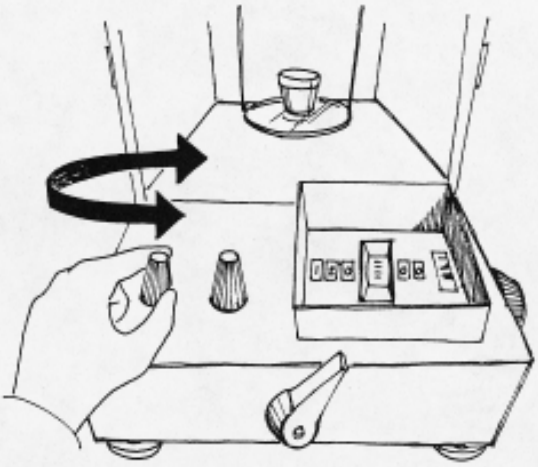
STEP #5 - TURN ARREST LEVER TO FULL RELEASE AND ZERO BALANCE.



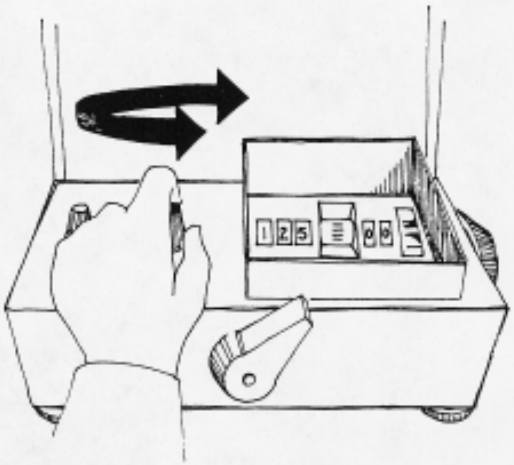
STEP #6 - RETURN LEVER TO ARREST POSITION AND PLACE OBJECT TO BE WEIGHED ON BALANCE PAN.



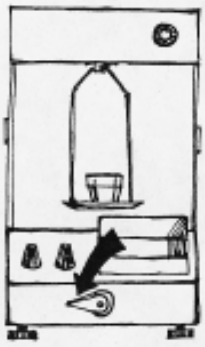
STEP #7 - CLOSE DOOR AGAIN AND TURN LEVER TO PARTIAL ARREST.



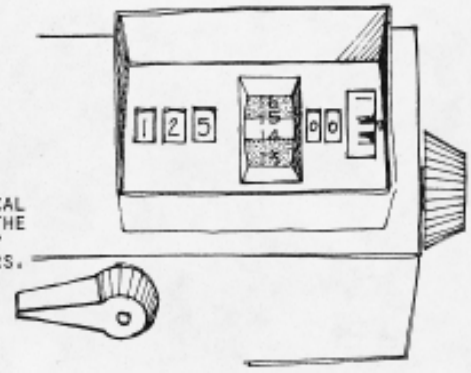
STEP #8 - TURN 10 GRAM CONTROL KNOB SLOWLY UNTIL OPTICAL SCALE DISPLAYS MINUS SIGNS. TURN KNOB BACK ONE POSITION.



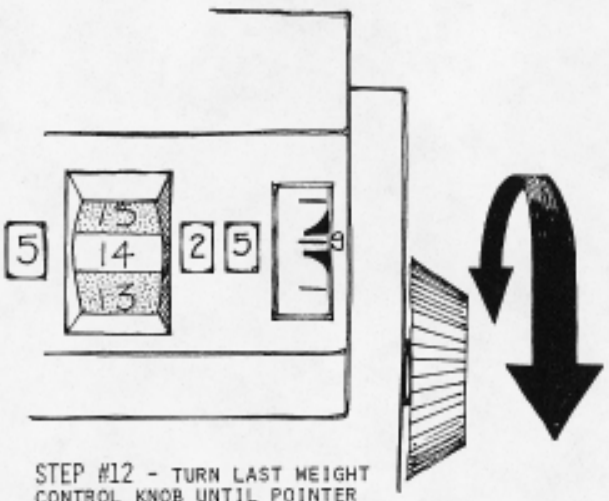
STEP #9 - REPEAT STEP #8 USING THE 1 GRAM WEIGHT CONTROL KNOB.



STEP #10 - TURN ARREST LEVER BACK TO FULL RELEASE.

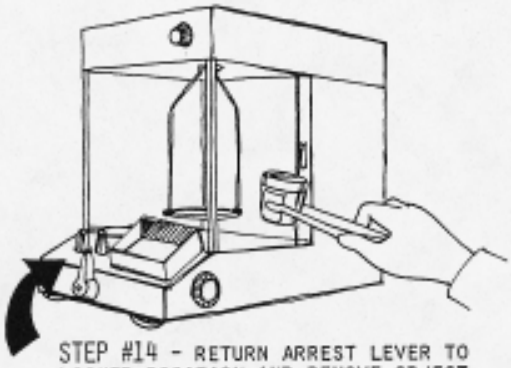


STEP #11 - WHEN OPTICAL SCALE STOPS MOVING, THE POINTER WILL PROBABLY BE BETWEEN TWO NUMBERS.

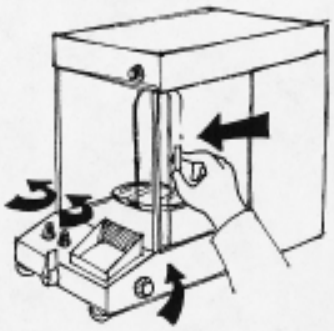


STEP #12 - TURN LAST WEIGHT CONTROL KNOB UNTIL POINTER EXACTLY ALIGNS WITH LOWER OF THE TWO NUMBERS. ADDITIONAL NUMBERS WILL BE DISPLAYED IN WINDOWS TO THE RIGHT.

STEP #13 - RECORD THE WEIGHT OBTAINED.

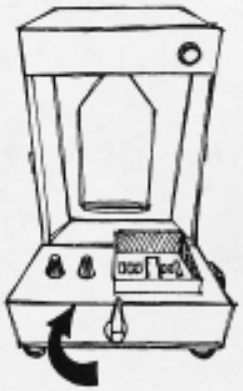


STEP #14 - RETURN ARREST LEVER TO LOCKED POSITION AND REMOVE OBJECT.



STEP #15 - CLOSE SLIDING DOORS AND RETURN WEIGHT CONTROL KNOBS TO ZERO.

STEP #16 - TURN ARREST LEVER TO FULL RELEASE AND CHECK ZERO. IF INSTRUMENT IS NOT STILL ZEROED, REZERO IT AND REPEAT ENTIRE WEIGHING PROCEDURE.



STEP #17 - RETURN ARREST KNOB TO LOCKED POSITION.

Moisture is a particularly difficult problem in water quality laboratories because many of the weighings are performed on various dried solids. Solids containing organic residues frequently yield residual moisture very slowly during drying operations and may still contain some water after the required drying time. It is therefore necessary to treat all samples in exactly the same manner to ensure that the extent of drying remains constant between different samples.

Porcelain crucibles used for weighing the samples will also retain moisture. Since the weight of the crucible (tare) is determined first and then subtracted from the combined weight of the crucible and sample, it is essential to subject the empty crucible to the same procedure before the initial weighing as it will later receive when it contains sample. Failure to dry the crucible sufficiently the first time will yield too high a tare weight, thereby lowering the apparent weight of the sample. Too much drying, on the other hand, may artificially raise the observed weight of the sample.

When highly hygroscopic (water absorbing) materials are weighed under humid conditions, the gain in weight due to water absorbed from the atmosphere can be observed on an analytical balance. A small tray of fresh desiccant kept inside the balance case will help eliminate this problem.

The temperature of the sample or object being weighed must be the same as that of the balance. If the material is too warm, convection currents formed within the balance case will push the pan upward, causing the observed weight to be less than the true value. Crucibles which have been ignited or exposed to a muffle furnace should be partially cooled in open air and then placed in a desiccator until they reach room temperature. They should be weighed as soon as possible to minimize absorption of moisture from the atmosphere.

Materials which are colder than the balance will form downward convection currents in the balance case, causing the observed weight to be higher than the true value.

A static charge can be established when a crucible is wiped with a dry cloth. If the crucible is then placed in contact with a balance, some of the charge will be transferred to the balance mechanism and will interfere with an accurate weighing. These charges may take as long as half an hour to dissipate on a dry day, shorter on a humid day.

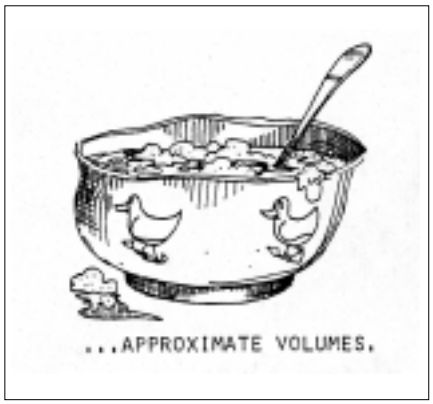
Air exerts a bouyant effect on objects just as does water. Normally, this effect is very small and can be disregarded. However, sometimes when a piece of volumetric glassware is being calibrated to determine the volume it contains or delivers, this effect must be taken into consideration. A calculation must be employed to estimate the truet weight of the item in a vacuum. Most water quality laboratories can rely on the original factory calibration of their volumetric glassware without resorting to this procedure.

Measuring Volumes

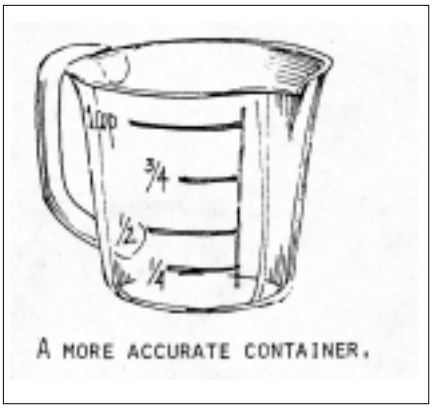
Water and wastewater laboratories deal almost entirely with liquid samples. While liquids can be weighed, generally it is simpler to measure them by volume. Most of the measurements made in water and wastewater labs are therefore volumetric. Volumetric measurements are probably the most important determinations made in these labs and it is critical for analysts to understand how to perform them properly.

SIMPLE CONTAINERS MEASURE...

Volumetric measurements begin with a simple container. Any teacup or coffee mug will do for starters. Even a child's cereal bowl could be used for some measurements. Markings can then be added to show how much fluid a particular container holds when filled to various levels. The teacup or coffee mug now becomes a kitchen measuring cup, with 1/4, 1/2 and full cup graduations shown on the side. In the laboratory, an equivalent piece of equipment would be the graduate cylinder, with volumes marked in portions of a liter.


...APPROXIMATE VOLUMES.

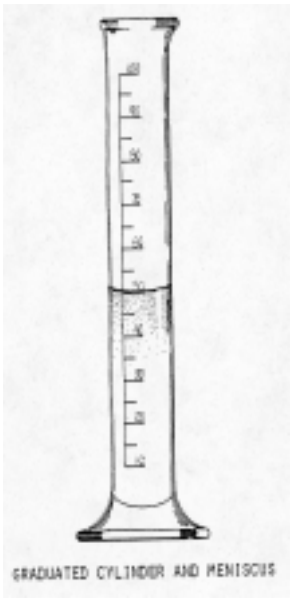
The more important a measurement becomes, however, the more restrictions must be placed on the container to be used and the way it is marked. For example, while the cereal bowl mentioned earlier could be used for measuring very crude volumes, it would not be suitable for precise work. For one thing, the materials it is made of might not be appropriate. If it's flexible plastic it could warp, thereby changing the amount of liquid it's capable of holding. Another problem is that its width makes a good reading of the fluid level difficult. Instead of looking across a wide bowl to read the volumes marked on the side, it is much easier and more accurate to observe a fluid level through a narrow column of glass. In this case, even small differences in volume result in noticeable changes in the level in the column, something which could never be achieved with a bowl or even a cup.



A MORE ACCURATE CONTAINER.

THE MENISCUS

Unfortunately, when water is placed in most containers, it tends to cling to the sides of the container and rises slightly around the edges. This gives a curved, distorted surface to the water. This surface is called the meniscus. When reading the level of water in a container, the bottom or lowest point on the meniscus is always taken as the point of reference. So if the upper edge of a water surface in a graduate cylinder is level with the 24.6 ml mark, but the lowest point on that water surface only reaches the 24.5 ml mark, the volume of liquid contained in the cylinder would be correctly read as 24.5 ml.



GRADUATED CYLINDER AND MENISCUS

Our simple container has now evolved into a more sophisticated piece of equipment, built of some rigid material that will hold its shape (usually glass) and with a relatively narrow column or neck for observing slight differences in fluid level. Volumes are indicated by markings on this column or neck. Now it is time to look at what containers are actually used to measure volumes in the lab and to consider some of the special requirements for using each of them.

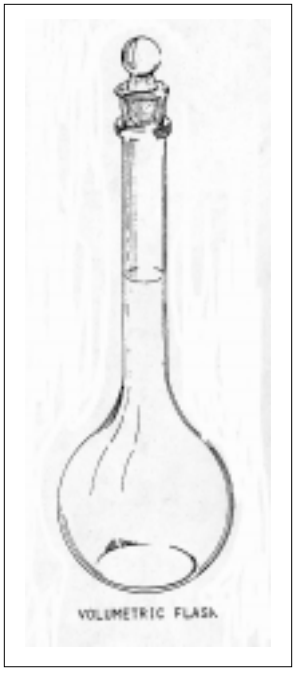
APPROXIMATE VOLUMES

Two of the most widely used types of glassware in the laboratory are beakers and Erlenmeyer flasks. They are used for such functions as transferring samples and reagents, mixing solutions and containing chemical reactions. Many beakers and flasks are marked with volume graduations, enabling them to also be used for measuring fluids. However, these graduations are only accurate to within about five percent and should therefore be used only for the most approximate measurements of noncritical reagents or samples.

BEAKER (FRONT) AND ERLENMEYER FLASK (REAR)

Graduated cylinders are also commonly used for measuring volumes in water quality labs. While graduated cylinders are more accurate than general flasks and beakers, even they are only calibrated to a tolerance of one percent. They are not as accurate as most analytical measurements require and therefore should not be used for measuring critical volumes. When using a graduated cylinder, it should be of such a size that the liquid being measured nearly fills it up.

Glassware which is calibrated with sufficient accuracy to truly be called “volumetric” includes volumetric flasks, transfer pipets and burets. They are of such importance in the laboratory that each of these items will be discussed individually.



VOLUMETRIC FLASK

VOLUMETRIC FLASKS

Most volumetric flasks are calibrated to contain a certain volume and have a single mark etched on the neck of the flask. Some flasks are also available having a second mark etched above the first, which is where the liquid level should be read if the flask is to be used to deliver that same volume. The difference between the two marks accounts for the volume of liquid which will remain on the walls of the flask when the contents are drained. These latter flasks are seldom used because the amount of fluid adhering to the sides of the flasks can only be estimated, which makes them relatively inaccurate.

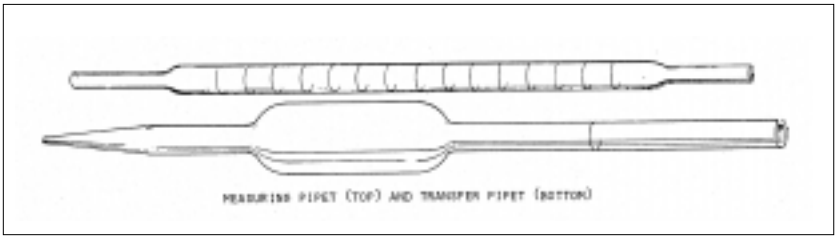
Commonly used sizes of volumetric flasks are those calibrated to contain 1000, 500, 250, 100, 50, 25 and 10 ml. These are often provided with ground-glass or plastic stoppers which prevent liquid from spilling when the contents are mixed.

When preparing or diluting a solution, use a beaker and funnel to empty the reagent involved into the flask. Rinse the funnel and beaker several times, with the rinse water also going into the flask. The solution should then be mixed by inverting the stoppered flask. With the flask upright again, add water until the liquid level is just below the mark. Stopper and invert the flask again, then continue adding water with a pipes until the meniscus of the liquid is exactly on the mark. Finally, thoroughly mix the contents of the flask by inverting numerous times.

To dispense the contents of a volumetric flask, gradually invert the flask until it is almost vertical. Allow the flask to drain for about 30 seconds, then touch the mouth of the flask to the side of the receiving vessel to remove the last adhering drop from the lip of the flask.

PIPETS

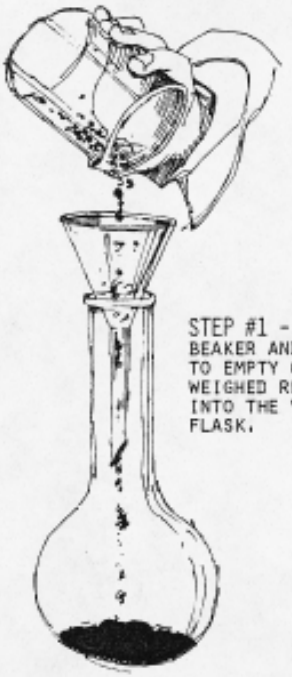
Pipets are of two general categories: transfer (also called volumetric) and measuring. Transfer pipets have an enlarged bulb at the center and are calibrated to deliver a single fixed volume. The opening at the tip of the pipet is sized to provide optimum outflow rates when dispensing the contents. Transfer pipets are used for precise work and their tolerance limits are specified by federal standards. Measuring pipets, also known as serological or Mohr pipets, are narrow straight tubes graduated to deliver variable amounts of liquids. They are not as accurate as transfer pipets and should not be used for precise work.



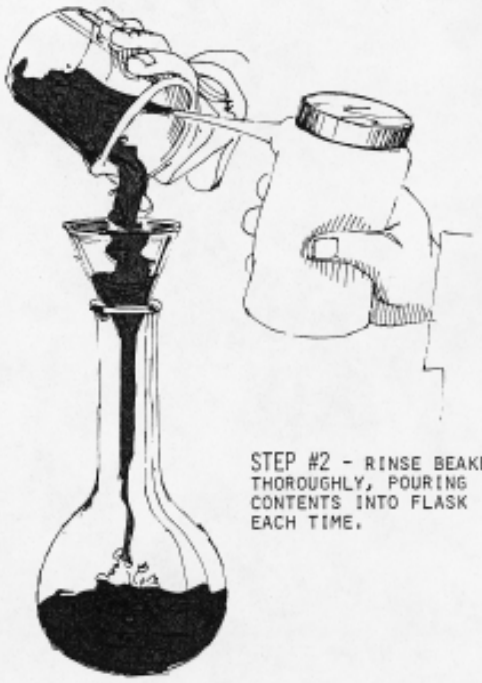
When using a transfer pipes for precise measurements, first rinse the pipes with the solution to be measured and discard the rinsings. For very precise work, repeat this procedure at least twice. Then, whether using a transfer or a measuring pipes, fill to above the calibration mark, hold the pipes nearly vertical and gradually drain the excess solution until the meniscus is exactly on the mark, keeping the tip in contact with thewall of a dry beaker to remove any drops of solution which would otherwise adhere to the tip. The solution is then ready to be dispensed. Dispensing should also be done with the pipes in a nearly vertical position and with the tip in contact with the wall of the receiving container. The liquid should be allowed to flow out freely and the pipes should be drained for five seconds or more (up to 20 seconds for precise work) after the liquid is out. For pipets calibrated to deliver (which constitutes most of the pipets used in water quality analysis), do not blow out or shake the pipes to remove the remaining drop from inside the tip. The presence of this drop is accounted for in the calibration of the pipes.

It is recommended that all pipeting in water quality laboratories be done with the aid of a pipeting bulb. This eliminates contact between the analyst's mouth and the pipes, thereby preventing accidental swallowing of dangerous reagents or contaminated samples.

Using Volumetric Flasks



STEP #1 - USE A BEAKER AND FUNNEL TO EMPTY CORRECTLY WEIGHED REAGENT INTO THE VOLUMETRIC FLASK.



STEP #2 - RINSE BEAKER THOROUGHLY, POURING CONTENTS INTO FLASK EACH TIME.

STEP #3 - RINSE FUNNEL INTO FLASK SEVERAL TIMES.

STEP #4 - STOPPER FLASK AND MIX BY INVERTING.

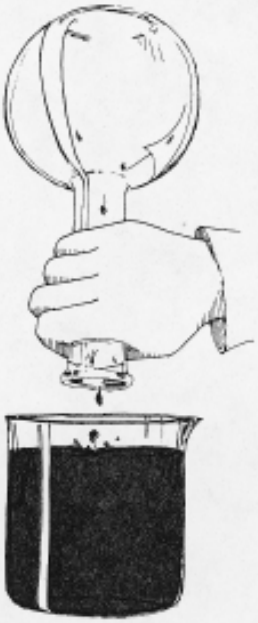
STEP #5 - ADD WATER TO JUST BELOW THE CALIBRATION MARK ON FLASK.

STEP #6 - STOPPER AND INVERT FLASK TO MIX.

STEP #7 - LINE UP MENISCUS WITH CALIBRATION MARK BY ADDING WATER SLOWLY FROM A PIPET.

STEP #8 - STOPPER AND INVERT FLASK TO MIX.

STEP #9 - TO DISPENSE, GRADUALLY INCLINE FLASK UNTIL IT IS ALMOST VERTICAL.



STEP #10 - DRAIN FLASK FOR 30 SECONDS.

STEP #11 - TOUCH FINAL DROP TO THE SIDE OF THE RECEIVING VESSEL.

Pipets, particularly transfer pipets, may be inappropriate for measuring samples containing large particulate matter as the opening at the tip may be too small to admit some of the particles. This would have the effect of filtering the sample prior to measurement.

BURETS

Burets are designed to dispense definite volumes and are made from accurate-bore glass tubing with glass or Teflon stopcocks and capillary tips of specific sizes. The most common buret sizes are those having 25 or 50 ml capacities. These are permanently marked in 0.1 ml increments, with the markings extending at least halfway around the tube. Other sizes of burets are also available, including larger ones having 100 ml capacities and smaller, micro-burets which generally have capacities of either 5 or 10 ml. These latter are graduated in 0.01 ml increments. Automatic burets can also be obtained with reservoir capacities ranging from 100 to 4000 ml.

The capillary tips restrict the outflow rates from the burets. With a 50 ml buret, the maximum outflow rate should not exceed 0.7 ml per second. An optimum flow rate when using the buret is about 0.5 ml per second. Flow rates in excess of the maximum will leave liquid adhering to the walls of the burets, resulting in errors in measurement.

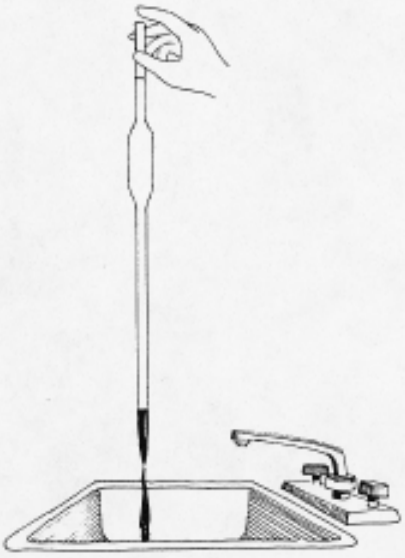
Before a buret is used, it should be rinsed two or three times with small volumes of the solution to be dispensed. It should then be filled to above the zero line, with the excess liquid withdrawn through the tip to remove any air bubbles trapped in the stopcock or capillary tip. Allow about 30 seconds for the sides to completely drain, then adjust the meniscus to the zero line. Remove any droplets clinging to the tip by touching the tip to the wall of a beaker.

The buret should be kept in an exactly vertical position during use. At the end of each titration, the last drop adhering to the tip of the buret should be removed by touching it to the wall of the receiving container. After making a reading at the end of a titration, allow 30 seconds for any additional drainage from the sides of the buret, then check the reading again. If droplets of liquid adhere to the sides of the buret, it needs to be thoroughly cleaned. If the solution is to be left in the buret for a few hours, place an inverted test tube or small beaker over the mouth of the buret to minimize changes in solution concentration due to evaporation. Before the buret is used again, withdraw some of the solution through the tip to flush it out. Never store alkaline solutions in a buret as they may cause glass stopcocks to “freeze” and can dissolve enough silicates from the glass to alter the buret’s capacity.

Titrations should be performed in good reflected light, but never in direct sunlight. A white background under the receiving container will help in observing color changes. The container used for the titration should be an Erlenmeyer flask if possible to reduce losses due to splashing. The end point of the titration has been reached when a fraction of one additional drop of the standard reagent in the buret causes a permanent color change. When all the titrations have been completed and the volumes recorded, discard the solution remaining in the buret;

Using Pipets

STEP #1 - USING A PIPET BULB, FILL THE PIPET WITH REAGENT TO RINSE THE PIPET.



STEP #2 - DISCARD THE RINSINGS. (REPEAT STEPS 1 AND 2 AT LEAST ONCE FOR VERY PRECISE WORK.)

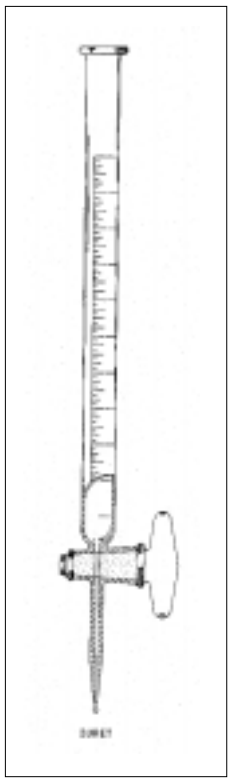
STEP #3 - FILL THE PIPET TO ABOVE THE CALIBRATION MARK.

STEP #4 - ADJUST REAGENT LEVEL TO CALIBRATION MARK. USE A DRY BEAKER TO REMOVE EXCESS DROPS FROM THE PIPET.

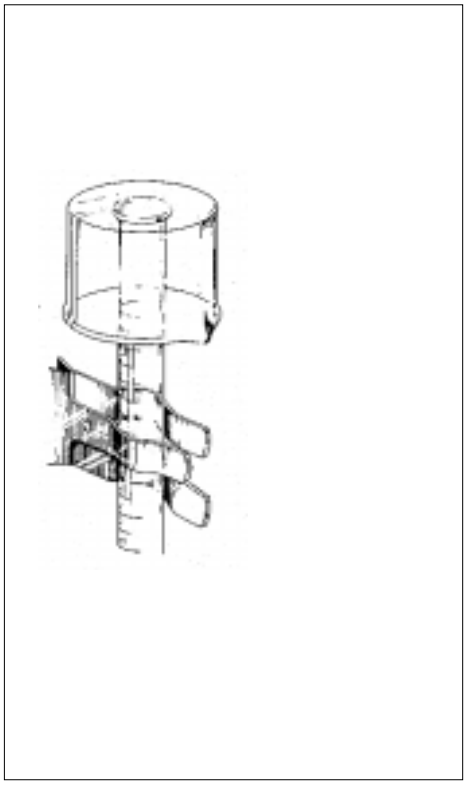
STEP #5 - TO DISPENSE, ALLOW THE REAGENT TO FLOW OUT FREELY. KEEP PIPET TIP IN CONTACT WITH WALL OF RECEIVING VESSEL AND DRAIN FOR 20 SECONDS.

STEP #6 - LIQUID REMAINING IN THE TIPS OF PIPETS CALIBRATED "TO DELIVER" SHOULD NOT BE BLOWN OR SHAKEN OUT.

never return this solution to the stock solution as it may contaminate the stock. Wash the buret immediately after use and invert it with the stopcock open to dry.

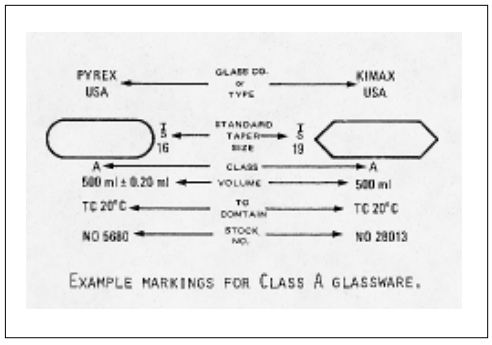


When storing solutions for short periods in a buret, cover the buret mouth with an inverted beaker to



GLASSWARE SPECIFICATIONS

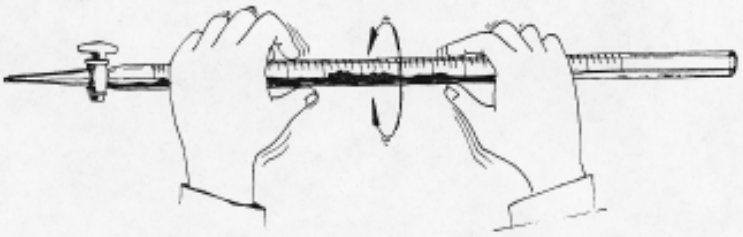
Federal specifications have been established for volumetric glassware and are listed in the National Bureau of Standards Circular 602, "Testing of Glass Volumetric Apparatus." Glassware which meets the NBS specifications is designated as Class A and all such items display a permanently marked "A." Most of these will be made of borosilicate glass such as that manufactured by Corning Glass Works under the name "Pyrex" or by Kimble Glass Co. under the name "Kimax." These items must also indicate the calibrated capacity of the vessel, the temperature at which the calibration was made and whether the vessel is designed to contain or deliver the specified volume (indicated as either TC for "to contain" or TD for "to deliver"). The name or trade-mark of the manufacturer and an identification number must also appear.



EXAMPLE MARKINGS FOR CLASS A GLASSWARE.

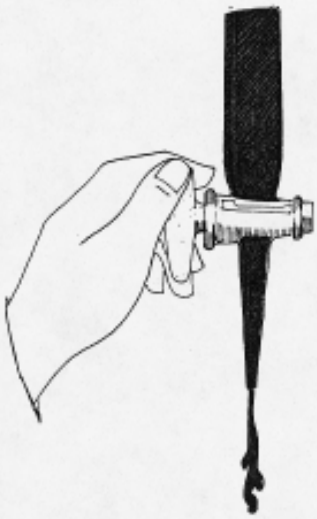
Actually, equipment for measuring volumes need not necessarily be glass in all cases; plastics, porcelain and metals have been used as satisfactory substitutes for some items and may even be preferred for particular applications. Teflon stopcock plugs have nearly replaced glass plugs in separatory funnels, burets and other instruments because they require no lubrication to prevent sticking or "freezing." Laboratory bottles, graduated cylinders, beakers and even volumetric flasks are

Using Burets



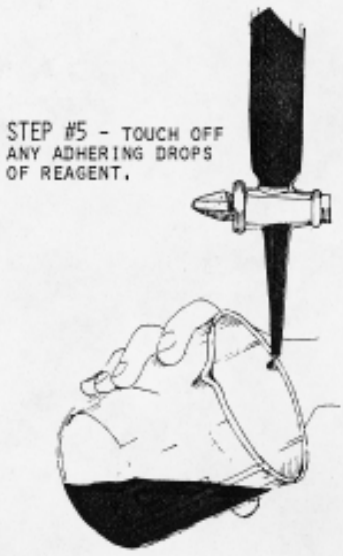
STEP #1 - RINSE BURET WITH A SMALL AMOUNT OF THE REAGENT TO BE USED IN THE TITRATION.

STEP #2 - DRAIN RINSE THROUGH BURET TIP.

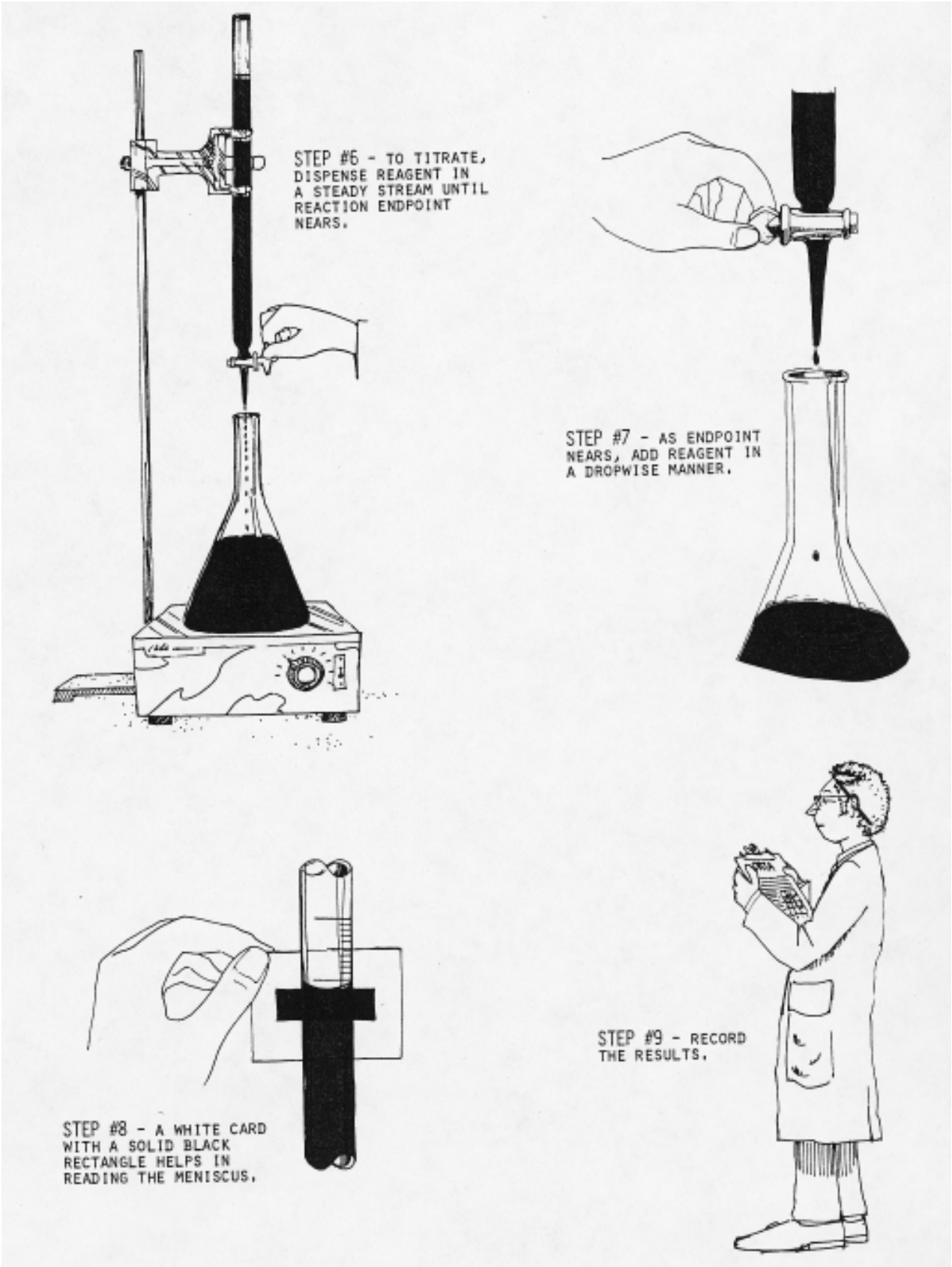


STEP #3 - REFILL BURET TO ABOVE ZERO MARK AND DRAIN EXCESS THROUGH STOPCOCK, BEING CERTAIN ALL BUBBLES ARE REMOVED.

STEP #4 - ADJUST MENISCUS TO ZERO MARK.



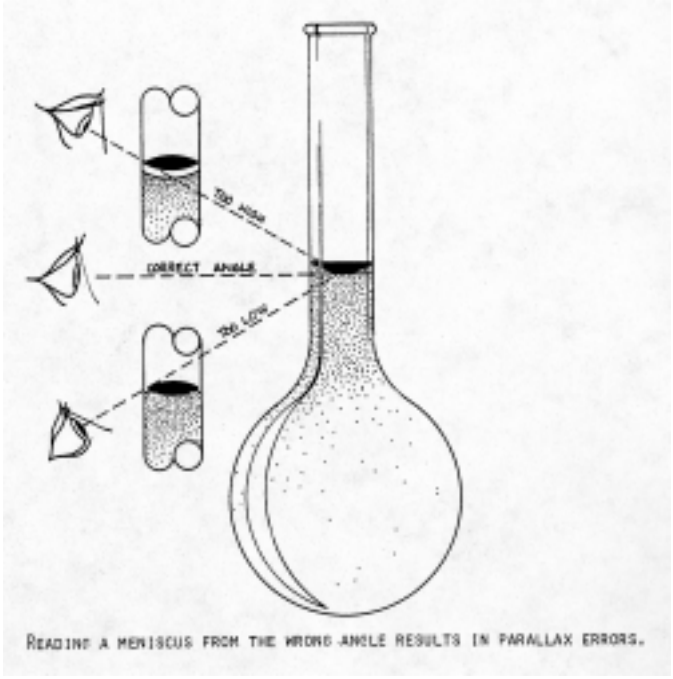
STEP #5 - TOUCH OFF ANY ADHERING DROPS OF REAGENT.


STEP #6 - TO TITRATE, DISPENSE REAGENT IN A STEADY STREAM UNTIL REACTION ENDPOINT NEARS.
STEP #7 - AS ENDPOINT NEARS, ADD REAGENT IN A DROPWISE MANNER.
STEP #8 - A WHITE CARD WITH A SOLID BLACK RECTANGLE HELPS IN READING THE MENISCUS.
STEP #9 - RECORD THE RESULTS.

sometimes made from a plastic called polypropylene. It is a clear, shatter-proof substance which can be autoclaved and which resists chemical attack. Another plastic, polystyrene, is used for some types of disposable bacteriological pipets.

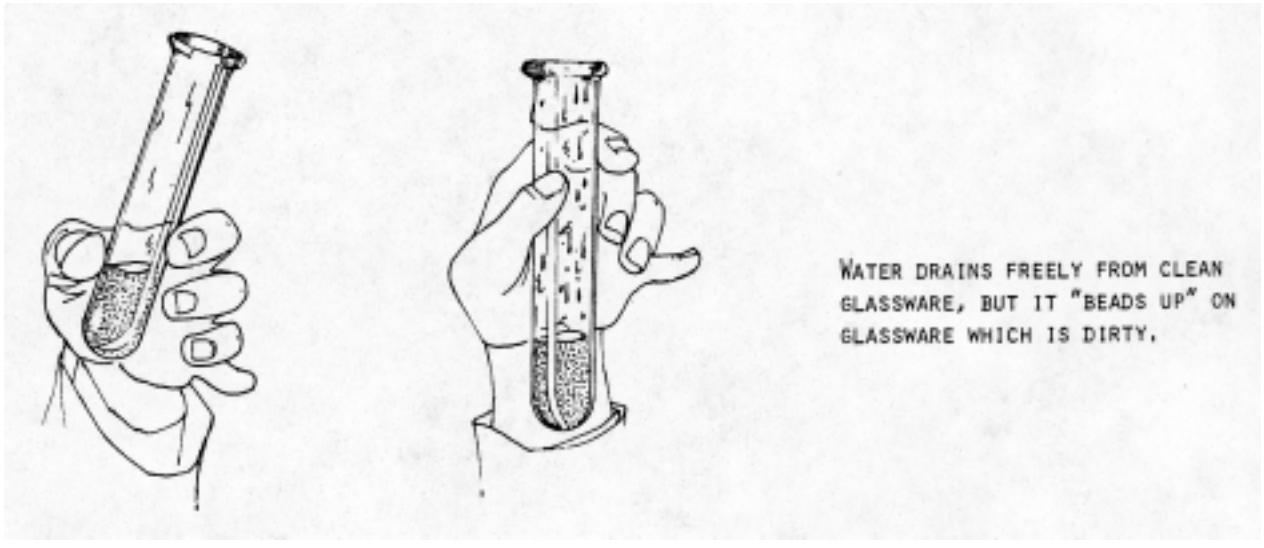
SOURCES OF ERROR

There is more to reading a meniscus level properly than merely knowing to line up the lowest point on the water surface with the calibration mark. It is also essential that the meniscus be read in the proper plane or it will result in a type of error called parallax. If the eye is above the plane of the meniscus and looking downward, the meniscus will appear higher and result in a high reading. Looking at the meniscus from too low a position will give a low reading. To avoid parallax, line up the bottom of the meniscus with the graduation mark where it appears both in front of the container and where it extends around to the other side. A white card with a black square painted on it can help in visualizing the meniscus for this.



READING A MENISCUS FROM THE WRONG ANGLE RESULTS IN PARALLAX ERRORS.

Another possible source of error stems from the temperatures of the glassware and the liquid being measured. Volumetric glassware is normally calibrated at 20°C. If the glassware is warmer, the glass will expand, resulting in greater volume. Water also expands with increasing temperature above 20°C, but not at the same rate as glass. Below room temperature, both glass and water contract, again


WATER DRAINS FREELY FROM CLEAN GLASSWARE, BUT IT "BEADS UP" ON GLASSWARE WHICH IS DIRTY.

at different rates, until the water approaches freezing (at which point it will once more expand). Therefore, for accurate measurements, both the glassware and the liquid should be as close to 20°C as possible.
Another potential source of error may arise when using improperly cleaned volumetric glassware. Not only can this cause contamination of the liquid being measured, but it can also result in false readings due to excess liquid clinging in beads to the sides of the vessel. This is especially important with apparatus calibrated to deliver, such as burets and pipets. Whenever water drains from the sides of glassware in other than a smooth, unbroken sheet, the glassware has been inadequately cleaned and should not be used for volumetric determinations.

CLEANING GLASSWARE

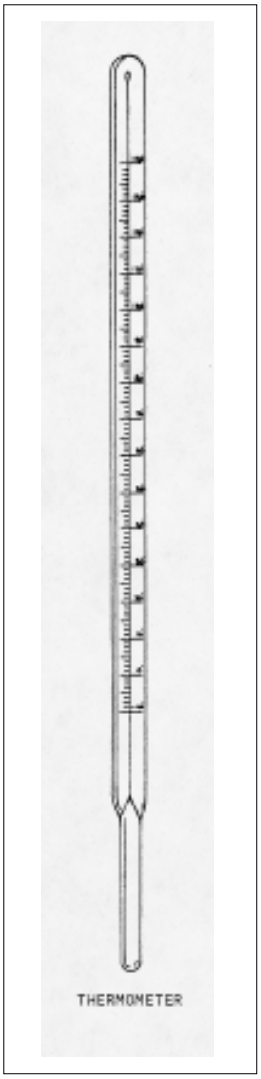
Most dirty glassware can be cleaned adequately by washing with a standard laboratory detergent and rinsing thoroughly first with tap water, then with distilled water. When a bottle brush is used, be sure there are no exposed metal edges or points which might scratch the glass. For more stubborn films or residues, stronger cleaning solutions may be necessary. A standard cleaning solution used in many labs consists of a mixture of concentrated sulfuric and dichromic acids. To make this solution, add a liter of concentrated sulfuric acid slowly, with stirring, to 35 ml of saturated sodium dichromate solution. Use this solution with extreme caution as it can cause severe burns.

Other Measurements

Most of the analyses performed in a water quality laboratory include measurements of weight, volume or both. The units typically used to express test results, mg/l, attest to the importance of these two types of measurement. But weight and volume are not the only categories of measurement performed, even in analyses whose results will be expressed in mg/l. Other types of measurement which are routinely made include temperature, electrical properties and color intensity. Instruments which monitor pH, selective ion concentrations and dissolved oxygen usually rely upon measurements of electrical potential. Colorimeters and spectrophotometers measure the intensity of particular wavelengths, or colors, of light. Each of these various types of measurement is considered briefly in this chapter.

TEMPERATURE

With few exceptions, temperature is measured in water and wastewater laboratories by means of mercury-filled thermometers. As a minimum, these thermometers should be graduated in intervals of 0.1° C. Accuracy should be checked against a thermometer certified to meet the requirements of the National Bureau of Standards (NBS), and readings taken from general lab thermometers should be corrected accordingly. A certified thermometer is provided with a plot verifying its accuracy; this plot is then used when establishing precise temperature measurements or when calibrating other lab thermometers. General thermometers should be checked for accuracy over the entire range of intended use because accuracy is not uniform over the full scale of a thermometer. Recheck thermometer calibrations periodically to detect inaccuracies caused by hairline breaks in the mercury columns. Certified thermometers are available either individually or in sets covering wide temperature ranges.



Every laboratory should have at least one certified thermometer, and a full set is recommended. Care should be taken to prevent thermometer breakage or separation of the mercury column. When long-stemmed thermometers are used in ovens and incubators so temperatures inside the instrument can be read without opening the door, the shelf space around the thermometer bulb should be left vacant to reduce the chance of breakage. Placing the thermometer bulb in a beaker of fine sand in an oven or in a beaker of liquid in an incubator not only helps provide a more accurate indication of temperature readings inside thick-walled glass containers in the units, but it also helps keep the mercury column from separating by buffering extreme temperature changes experienced when the door of the oven or incubator is opened and closed. Avoid placing a thermometer into solutions or environments with temperatures radically different from the initial temperature of the thermometer. The thermal shock may fracture the thermometer or cause the column to separate. Instead, allow the thermometer to gradually come within the temperature range to be measured prior to use. Never exceed the maximum temperature range of a thermometer. Thermometers which are to be used in the field should be protected by placing them in specially designed metal

cases to~reduce breakage. Discard thermometers if the graduations become illegible.

Recording thermometers may be used for processes which are highly temperature dependent, as in hot-air sterilizers, autoclaves and particularly incubators. Where recording thermometers are not available for incubators, a maximum and minimum registering thermometer should be used (in addition to a regular thermometer) so gross temperature variations over a 24-hour period can be determined. This additional thermometer should be placed on the middle shelf of the incubator.

ELECTRICAL PROPERTIES

When two electrodes are placed in a solution such as water or wastewater, an electrochemical cell is formed. The electrical potential of the cell, the current flowing through it and its resistance are all determined by the chemicals present in the solution. Measuring one or more of these electrical properties under controlled conditions enables an analyst to determine the chemical composition of the sample.

Conductivity meters measure how readily electrical current will pass through a solution, which depends upon the overall concentration of electrically active chemicals present. No attempt is made in this analysis to distinguish one chemical from another; rather it is the cumulative effect which is monitored.

Selective ion electrodes, on the other hand, measure the electrical activity of specific ions or groups of ions. The most familiar instrument of this kind is the pH meter, which measures the concentration of hydrogen ions in a sample. In addition to pH measurements, selective ion electrodes have also been developed to measure a wide variety of anions and cations. While only a few of these electrodes have been accepted by the Environmental Protection Agency for regulatory purposes, others are suitable for field investigations and plant control applications. Selective ion electrodes are typically used with pH meters having an expanded scale capability, in which a single pH unit can be expanded to occupy the full range of the scale.

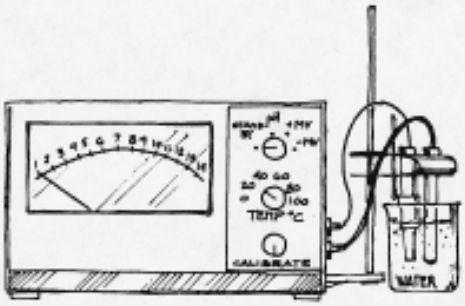
Related to selective ion electrodes are the various dissolved oxygen probes. These instruments are widely used to measure dissolved oxygen concentrations, despite considerable variability in instrument dependability and maintenance requirements. When using one of these probes, be sure to keep water flowing continuously over the face of the probe. Electrodes should be kept clean and membranes should be free of wrinkles.

One limitation with any of the electrical methods of analysis is that they all measure the electrical activity of a chemical or group of chemicals rather than actual concentration. When the chemical to be measured binds with another, the electrical activity of the first will be diminished, thereby lowering its apparent concentration in the sample.

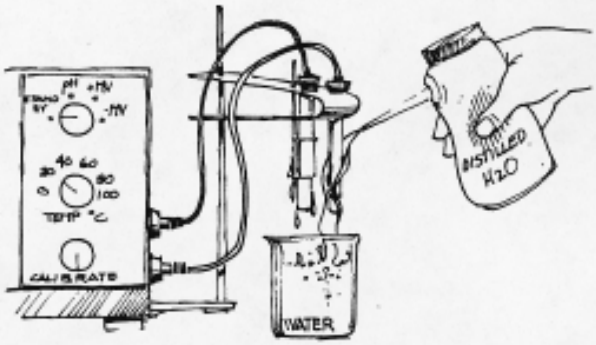
pH METERS

The glass electrode used on pH meters is subject to relatively little interference from turbidity, color, oxidants, reductants, colloids and high salinity, with the exception of a sodium error at

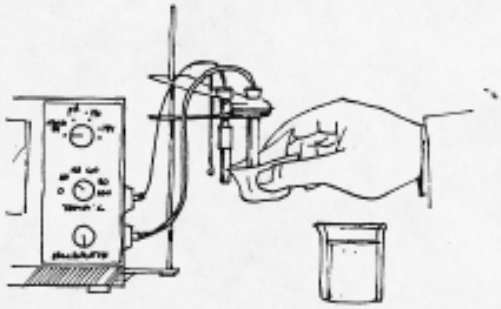
Using a pH Meter



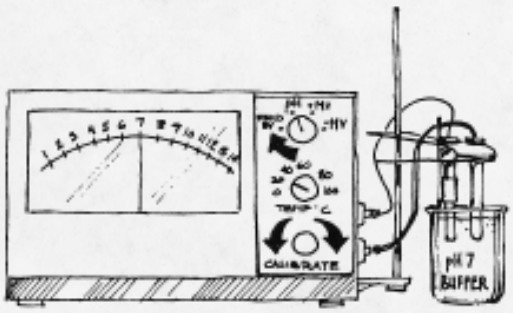
STEP #1 - KEEP THE ELECTRODES IMMERSED IN WATER BETWEEN USES.



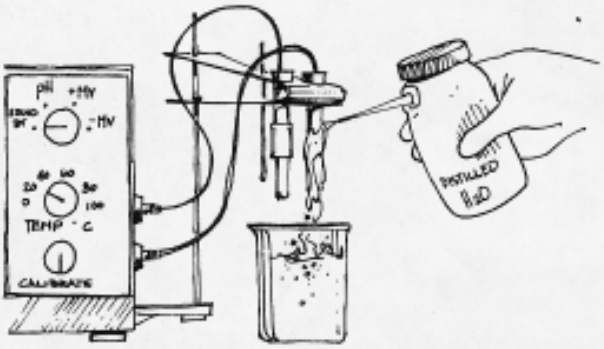
STEP #2 - BEFORE USE, RINSE ELECTRODES WITH DISTILLED WATER.



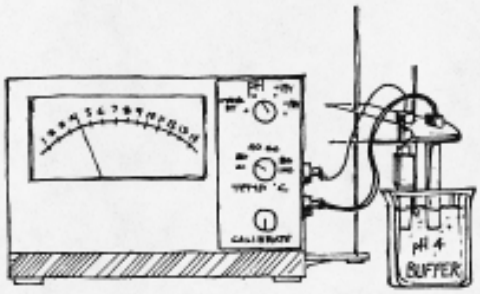
STEP #3 - DRY ELECTRODES GENTLY WITH A SOFT TISSUE.



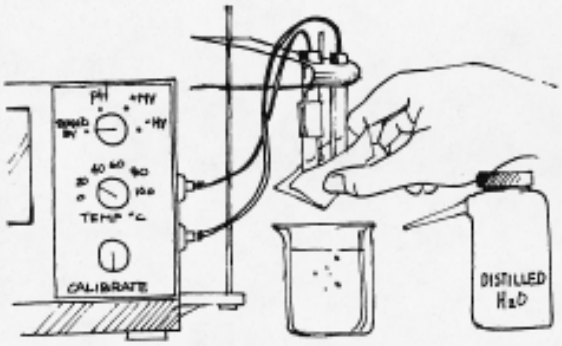
STEP #4 - STANDARDIZE METER WITH A BUFFER SOLUTION IN THE pH RANGE EXPECTED FOR THE SAMPLE.



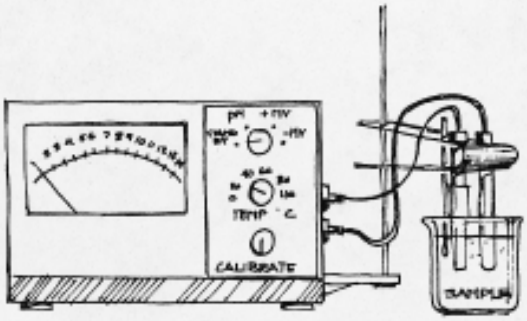
STEP #5 - REMOVE ELECTRODES FROM BUFFER, RINSE THOROUGHLY AND DRY.



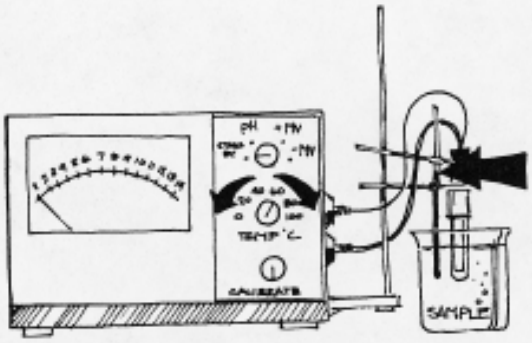
STEP #6 - CHECK CALIBRATION WITH A SECOND BUFFER.



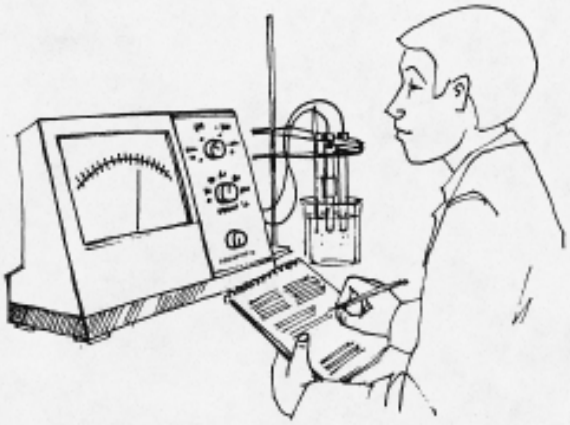
STEP #7 - RINSE ELECTRODES THOROUGHLY AND DRY.



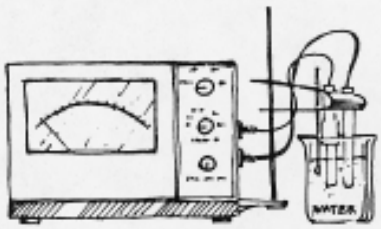
STEP #8 - IMMERSE ELECTRODES IN SAMPLE AND ALLOW TIME FOR TEMPERATURE TO EQUILIBRATE.



STEP #9 - MEASURE TEMPERATURE OF SAMPLE AND SET TEMPERATURE COMPENSATION KNOB ON METER.



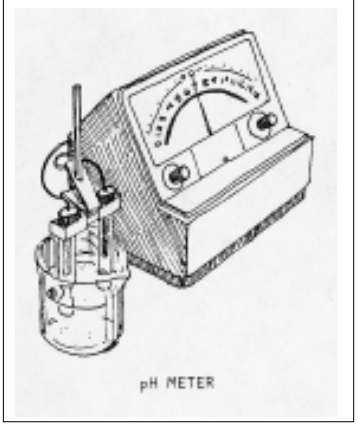
STEP #10 - READ pH AND RECORD.



STEP #11 - RINSE ELECTRODES AND RETURN THEM TO WATER AFTER USE.

pH levels above 10. For routine lab work, a sturdy, full-range combination electrode with either a plastic or glass body should be used. An electrode which contains a solid gel type filling material has the added convenience of not requiring the normal maintenance which a liquid-filled electrode does.

A glass electrode in combination with a calomel reference electrode is used to measure pH. Glass electrodes respond very rapidly in highly buffered solutions, but respond slowly in poorly buffered ones. This effect is compounded when changing the electrodes from buffered solutions to relatively unbuffered samples. Therefore, thoroughly rinse both electrodes with distilled water after each reading, then rinse with or dip the electrodes several times into the next sample to be measured before taking another reading. Stir weakly buffered samples during a measurement. Keep the electrodes immersed in water between readings to prevent them from drying out.

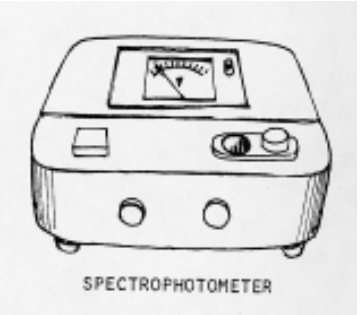

pH METER

Standardize a pH meter by rinsing the electrodes with distilled or deionized water, gently drying them with a soft tissue and placing them in a buffer solution having a pH value near that anticipated for the samples to be measured. Adjust the pH reading of the instrument to match the value of the buffer solution. Check electrode performance by removing them from the first buffer, rinsing and drying them again and placing them in a second buffer at least 4 pH units different from the first. Failure to read the pH value of this second buffer to within 0.1 units indicates a faulty electrode or other problem. Faulty electrodes may be dirty, cracked or contain insufficient KAI solution in the calomel electrode.

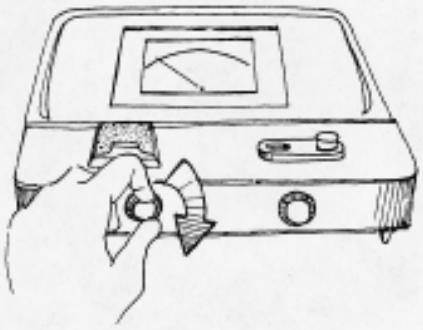
If a satisfactory value is obtained with the second buffer solution, rinse and dry the electrodes and proceed to the first sample solution. Allow the electrodes to reach the same temperature as the sample, then set the temperature calibration knob on the instrument. Temperature affects both the electrical response of a solution at a given pH value and the degree of ionization of the solution. The temperature of a sample must therefore be recorded along with the measured pH value. Measurements should be made to the nearest 0.1 PH unit.

COLOR INTENSITY

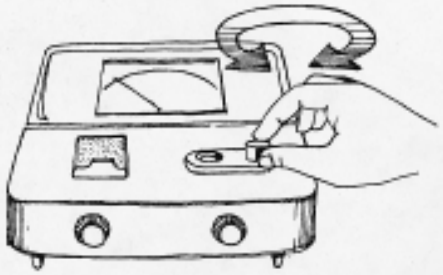
Some substances produce characteristic colors in solution, with the intensity of the color being proportional to the concentration of the substance. The color is formed by the substance's absorption of particular wavelengths of light passing through the solution. Measuring the intensity of the color enables the concentration of the substance to be determined. The simplest means of accomplishing this is through either Nessler tubes or a pocket comparator. The color developed in a sample is compared by the analyst to a series of color standards, each of which has a corresponding concentration value. The value for the standard most closely approximating the color intensity of the sample is taken as the concentration of the substance in that sample.


SPECTROPHOTOMETER

Using a Spectrophotometer

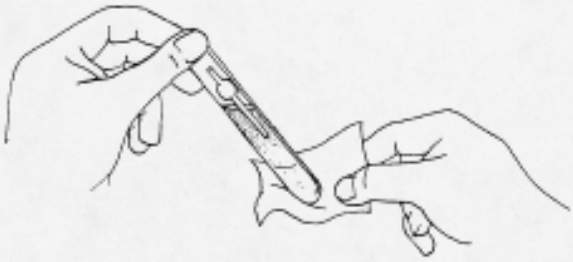


STEP #1 - TURN INSTRUMENT ON AND ALLOW 10-20 MINUTES TO WARM UP.



STEP #2 - SELECT DESIRED WAVELENGTH.

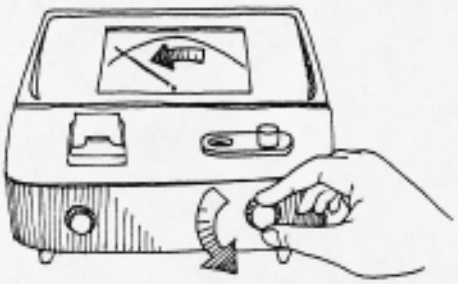
STEP #3 - TURN "ON" KNOB UNTIL SCALE READS INFINITE ABSORBANCE.



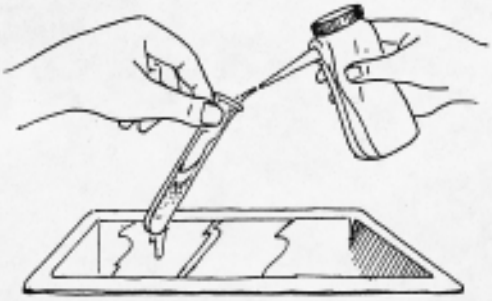
STEP #4 - RINSE CELL WITH BLANK SOLUTION 2-3 TIMES, THEN REFILL AND GENTLY DRY CELL.

STEP #5 - ALIGN MARK ON CELL WITH MARK ON SAMPLE HOLDER AND INSERT CELL IN INSTRUMENT.

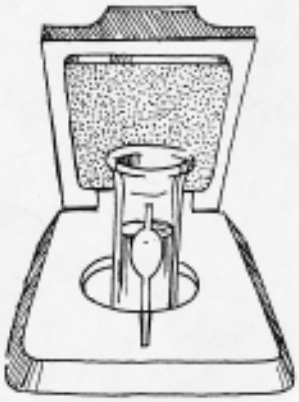
STEP #6 - CLOSE SAMPLE COVER.



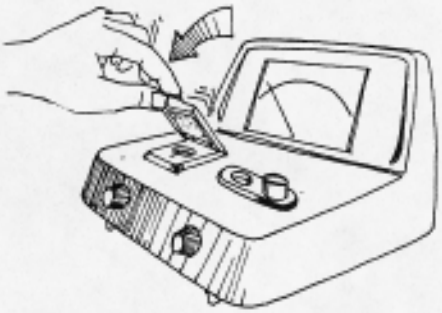
STEP #7 - ADJUST SCALE TO ZERO USING LIGHT CONTROL KNOB.



STEP #8 - REMOVE CELL AND DISCARD CONTENTS. RINSE CELL SEVERAL TIMES WITH DISTILLED WATER, THEN WITH SAMPLE TO BE MEASURED.



STEP #9 - DRY THE CELL AND INSERT INTO SAMPLE HOLDER, ALIGNING MARKS ON CELL AND HOLDER.



STEP #10 - CLOSE SAMPLE COVER.

STEP #11 - RECORD THE ABSORBANCE VALUE OBTAINED FROM THE SCALE.

More precise measurements may be made by passing a controlled beam of light of a suitable wavelength through the solution and measuring the amount of light which the solution absorbs. The absorbance is related to the concentration of the substance being measured. A simple, relatively inexpensive instrument based on this principle is the colorimeter or filter photometer, which uses colored filters to produce a beam of light of the desired wavelength. The intensity of the light passing through a vial containing the sample is measured by an electronic photodetector. Filter photometers are widely used for analysis of water and wastewater contaminants, particularly in field applications. One limitation of such instruments, however, is that filters produce a light beam composed of a fairly wide band of wavelengths, which limits sensitivity.

SPECTROPHOTOMETERS

Sensitivity of measurement is improved through the use of a spectrophotometer, which relies on a diffraction grating to produce a beam of light with more narrowly defined wavelength characteristics than does a filter. Simple spectrophotometers may measure light only in the visible spectrum, while more complex ones may also measure in the infrared and ultraviolet ranges as well.

Many organic compounds absorb characteristic wavelengths of light strongly enough to be measured spectrophotometrically. Most inorganic ions, however, do not. Therefore, before such ions can be measured, they must be reacted with some reagent (usually an organic molecule which is specific for the ion being sought) which will give an appropriately colored product. The product must not only possess the desired absorption characteristics itself, but must also absorb at a different wavelength than the original reagent so the two can be distinguished. An excess of reagent is then added to the sample so virtually all the substance being measured will be driven to react. If only a portion of the original substance reacts with the reagent, the concentration determined by the procedure will be low.

For any compound which is to be measured with a spectrophotometer (or a filter photometer! a calibration curve must first be prepared using a variety of known concentrations of the compound. These standards must be treated in the same fashion as will later sample solutions. When the absorbance values for the standards are determined and plotted on graph paper, a straight line should result. Comparison of the absorbance value obtained for a sample solution to the calibration curve will provide the concentration of the compound in the sample.

To use a spectrophotometer, turn on the power switch and wait at least 10 minutes (20-30 minutes for older instruments) for the instrument to warm up. Set the wavelength control to the proper setting and be sure the sample holder cover is closed. Turn the power switch until the needle on the scale reads infinite absorbance. Fill the spectrophotometer cell with the blank solution, rinsing it several times first and drying the outside of the cell. Slide the cell into the sample holder, being certain that the markings on the cell are aligned with the marking on the edge of the sample holder. Close the sample holder cover and adjust the light control knob until the needle reads zero absorbance. Remove the cell from the holder and replace the contents of the cell with the sample to be measured, rinsing the cell several times first with distilled water and then with sample. (Alternatively, use a matched cell for the sample.) Dry the cell and place it in the sample holder. Close the cover and record the absorbance value obtained for the sample. Compare this value to the. calibration curve for the particular substance sought to determine the substance's concentration in the sample.

Be sure the absorption cells are kept scrupulously clean, free of scratches, fingerprints, smudges and evaporated film residues. Hold the cells only from the top to avoid leaving prints or smudges in the area through which the light beam will pass. The absorbance of matched cells must be checked before use to ensure that they are equivalent.

Safety

Workers in water and wastewater laboratories are exposed to a wide variety of hazards, including fire and explosion, electrical shock, chemical burns, poisoning and bacteriological contamination. They must be protected adequately against these dangers. Too many laboratories either lack proper safeguards altogether or do not enforce their use. Operators in small labs are often unaware of correct safety techniques or believe safety presents a problem only in larger facilities. Those in larger labs may follow the more obvious safety procedures without realizing that many others are being ignored or violated. Even trained chemists and biologists are often at fault, for laboratory safety is rarely taught in college courses.

Accidents or injuries in the laboratory are always caused in some way, and can therefore be prevented. One of the principal reasons for laboratory accidents is the lack or misuse of equipment. The purpose of safety standards is to prevent an accident from occurring or to minimize the effects if one should take place.

SAFETY PROCEDURES

Besides sharing many dangers in common with other types of workplace situations, such as possible electrical shock, burns and injury from equipment, analysts must also contend with dangers specific to laboratory conditions. Predominant are the dangers imposed by working with hazardous chemicals and substances. A laboratory worker must be familiar with both the general safety procedures necessary for routine work and the specific procedures required for handling each of the various hazardous substances encountered.

Chemicals and samples received by a laboratory must be carefully handled at all times. This handling involves a number of precautions, several of which are important enough to become part of the formal regulations of the laboratory. These should include at least the following rules:

--Laboratory work is a serious and potentially dangerous endeavor and an analyst should conduct himself accordingly at all times.

--Anyone working in the laboratory should familiarize himself with the safety procedures relevant to each operation, as well as the locations of all safety equipment. These procedures should then be followed strictly and the necessary equipment used.

--Smoking, eating and drinking should never be permitted in the laboratory, nor should laboratory vessels ever be used to hold food or beverages.

--Pipetting of all hazardous substances should be done by mechanical means, never by mouth.

--Acids must always be added to water, never the reverse.
--Any work involving toxic or flammable gases, release of hazardous products or similar dan-

gers should be conducted under the fume hood and with appropriate personal protection.

--All chemical compounds should be considered toxic unless absolutely known to be otherwise.

Another critical laboratory activity besides general handling is labeling of chemicals and samples. Not only is proper labeling dictated by common sense, it is required by law. In general, any time a chemical is transferred from one container to another, a duplicate or facsimile of the original label should be firmly attached to the new container. Wax pencil and water soluble ink markings, abbreviations, formulas without names, codes and numbers should be avoided when making labels. Each label should include the name of the chemical; a word to indicate the severity of hazard if any), such as “Caution, “ ‘Warning” or “Danger;” a summary of those hazards beginning with the most serious; precautions to take when using, storing or handling the chemicals; and instructions to follow in the event of accidental exposure or contact. The date a sample is collected or a chemical is opened or prepared should also appear on the label so out-of-date materials can be disposed of. Finally, a label should also include the name or initials of the analyst who prepared the contents.

Storage of chemicals presents a number of problems to the laboratory worker because of the diverse characteristics of the reagents used. These problems can be reduced by limiting the types and amounts of chemicals kept on hand to the lowest practical levels. Excess, out-of-date or unused chemicals should be discarded. Storage containers should be checked frequently to ensure they are in satisfactory condition, and damaged containers replaced immediately.

Storage facilities should be appropriate for both the quantities and types of chemicals to be stored. They should consist of cool, well-lighted and ventilated rooms separated from the laboratory itself by fire walls. Automatic sprinkler systems should be provided. Avoid overcrowding or storage of incompatible chemicals together. Simple alphabetical arrangements of diverse and often incompatible chemicals should be avoided.

Following these guidelines will help ensure the general safety of the laboratory and those who work in it. However, many compounds exhibit hazards which require more detailed, specific attention. This is particularly true of those compounds which may be categorized as corrosive irritants, toxic chemicals, flammable liquids or compressed gases.

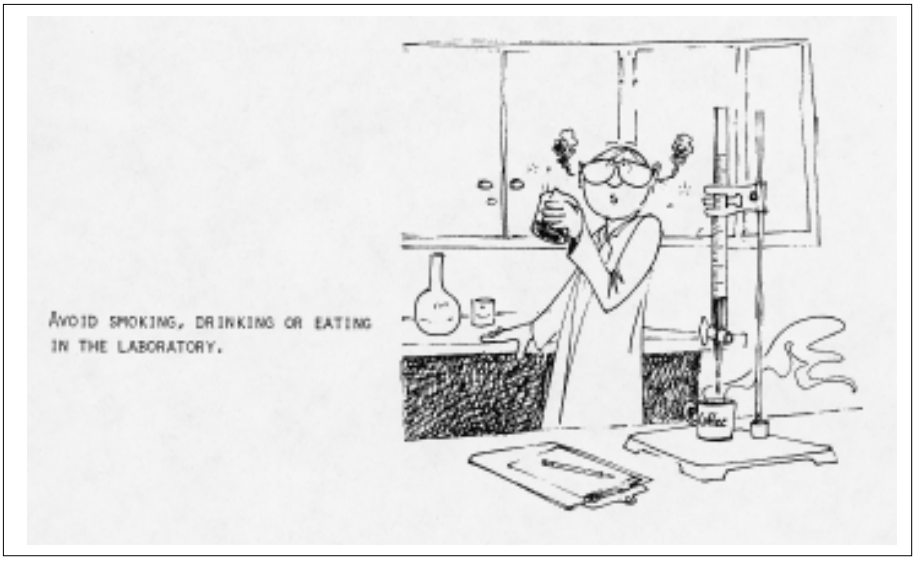
Corrosive irritants may be liquid, solid or gas. Concentrated liquids are the physical form of irritants most likely to cause immediate injury and therefore present the greatest danger of external damage, particularly to the eyes and skin. Protection against liquid corrosives is obtained by wearing sufficient protective clothing capable of preventing accidental contact with the irritant, especially rubber gloves, chemical safety goggles, face shield and rubber aprons.

Large bottles of corrosive liquids should be stored below head level and should be placed in chemically resistant pans or containers to catch the liquid should the original bottles break or leak. Suitable resistant materials for these pans are stainless steel, lead or rubber, among others. Similar trays or buckets should be used when transporting bottles of corrosives to or from storage.

In case of accidental contact with corrosive liquids, the exposed area should immediately be flushed with copious amounts of water. If the eyes are involved, this flushing should continue for at least 15 minutes. A physician should be called immediately.

Solid irritants generally present the least danger of the three types because the affected person usually has time to wash the material off before serious injury can occur. However, such materials can cause serious delayed irritation if they are not removed right away. Most are not immediately painful and if they remain in contact with the body until pain can be felt, serious injury generally has already taken place. Protection against finely divided solid irritants, such as fine powders, can be provided through adequate exhaust ventilation, plus gloves, respirators and protective clashing for particularly hazardous compounds. First aid for summoning a physician.

Generally, the most serious overall hazard from chemical irritants is presented by those in the gaseous state. Examples of these types of irritants are ammonia, hydrochloric acid vapors, formaldehyde, acetic acid, chlorine, bromine, iodine, sulfur dioxide and nitrogen dioxide. Precautions to follow when handling gaseous irritants includes the use of respiratory protective equipment and adequate exhaust ventilation, combined with additional protection for the skin and eyes in some cases.



Toxic chemicals cause widespread damage when taken into the body. Exposure may be through the skin and eyes, inhalation or swallowing. Contact with the skin and eyes is the most likely form to occur and may be prevented through use of appropriate protective clothing, goggles and face shields. Inhalation may occur with gases and vapors, with the compound being rapidly absorbed into the bloodstream and distributed throughout the body. Respiratory

protective equipment and adequate ventilation is necessary to safeguard against such contamination.

Swallowing is the least likely means of exposure to occur and can usually be controlled by not allowing food or beverages into the laboratory and never using laboratory containers for holding food and drinks.

SAFETY EQUIPMENT

Equipment for accident prevention or injury treatment purposes includes such items as first aid kits, safety shields, fire extinguishers, safety carrying containers for flammable or corrosive liquids, emergency showers, eyewashes, fume hoods and personal protective equipment. Several of these items deserve special attention.

EMERGENCY SHOWERS SHOULD BE EASY TO REACH FROM ANY PLACE IN THE LABORATORY.

Emergency showers should be located in conspicuous positions which can be readily reached from any work area. They should be located away from electrical or other hazardous equipment. Their locations should be clearly marked on the floor to help injured personnel find them. Stall or multiple head type showers are preferable if space permits. The activating pull device for a shower should not be higher than 6 ft 8 in. from the floor. Showers should be provided with plenty of water at a moderate temperature and pressure.

Eyewashes are often incorporated with emergency showers, but may be installed separately at sinks or other critical areas. The water provided by an eyewash should be at a moderate temperature, aerated and in soft streams sufficient to wash the eyes without damaging the tissues.

PROCEDURES INVOLVING HAZARDOUS GASES SHOULD BE PERFORMED UNDER A FUME HOOD.

Fume hoods should be located in low traffic areas away from doorways, ventilation outlets or other sources of air disturbance. They should be constructed of materials resistant to chemical and physical abuse, such as cast epoxy or epoxy-coated asbestos cement. The exhaust system should provide an air flow of at least 100 lineal fpm across the face of the hood and be capable of 24-hr continuous operation. All interior electrical fixtures, including the exhaust fan, should be explosion-proof. All switches, electrical outlets and water and gas controls should be located outside the hood

PERSONAL PROTECTION

Equipment necessary for personal protection is among the most important a laboratory will stock. Included should be items for the following purposes:

Body protection. Lab coats, smocks and other types of general body covering are adequate for routine chemical exposure situations. Some of the synthetic fibers are particularly recommended for this because of their resistance to corrosive chemicals. Where more complete

protection is required, such as against potential spills of particularly corrosive or hazardous liquids, more impermeable protection such as neoprene aprons should be used.

Eye and face protection. Some form of eye protection should be worn by everyone in the laboratory at all times. This protection may be in the form of safety glasses {preferably with side shields! chemical goggles or face shields. During particularly dangerous work, face shields should be worn in combination with goggles or safety glasses for more complete protection. The use of contact lenses in the laboratory should be discouraged because chemicals can become trapped between the lens and the surface of the eye, making it difficult to successfully remove these compounds.

Hand protection. Cotton, leather or leather-faced gloves are adequate for handling abrasives, sharp objects and glassware. Rubber or synthetic coated gloves should be used when handling chemicals. These may be a lightweight, surgical type for moderate conditions, or heavy duty for use with concentrated acids and other corrosives. The surfaces of the gloves should be roughened for safe handling of wet glassware. Asbestos gloves should be used for handling high-temperature equipment or materials.

Foot protection. Leather shoes are adequate for general laboratory work. However, rubber safety shoes should be worn where there is a possibility of considerable amounts of water, acid or other liquids spilling on the floor. Shoes with built-in steel toe caps are needed only where heavy objects are involved. Tennis shoes or open-weave footwear should never be worn in the laboratory.

Respiratory protection. Whenever fumes, gases or chemical dusts may be inhaled, some form of respiratory protection should be used. This may be a self-contained breathing apparatus or a filter canister. The self-contained breathing apparatus is preferred because of its greater reliability and effectiveness. Also, it is not limited by low oxygen concentrations in the air. This type of apparatus may use compressed or liquified air or oxygen, or it may contain its own oxygen generating unit. Where a filter canister is to be used, it should be appropriate for use with the particular chemical agents involved.

PREVENTING FIRE AND EXPLOSIONS

A laboratory fire requires three components: presence of a flammable gas or vapor at a critical concentration, an oxidant (normally air with at least a minimum concentration of oxygen) and a source of ignition suitable to initiate combustion. Removal of any one of these components will prevent a fire from occurring or extinguish an existing fire. This can best be achieved in the laboratory by preventing the coexistence of flammable vapors and any possible source of ignition.

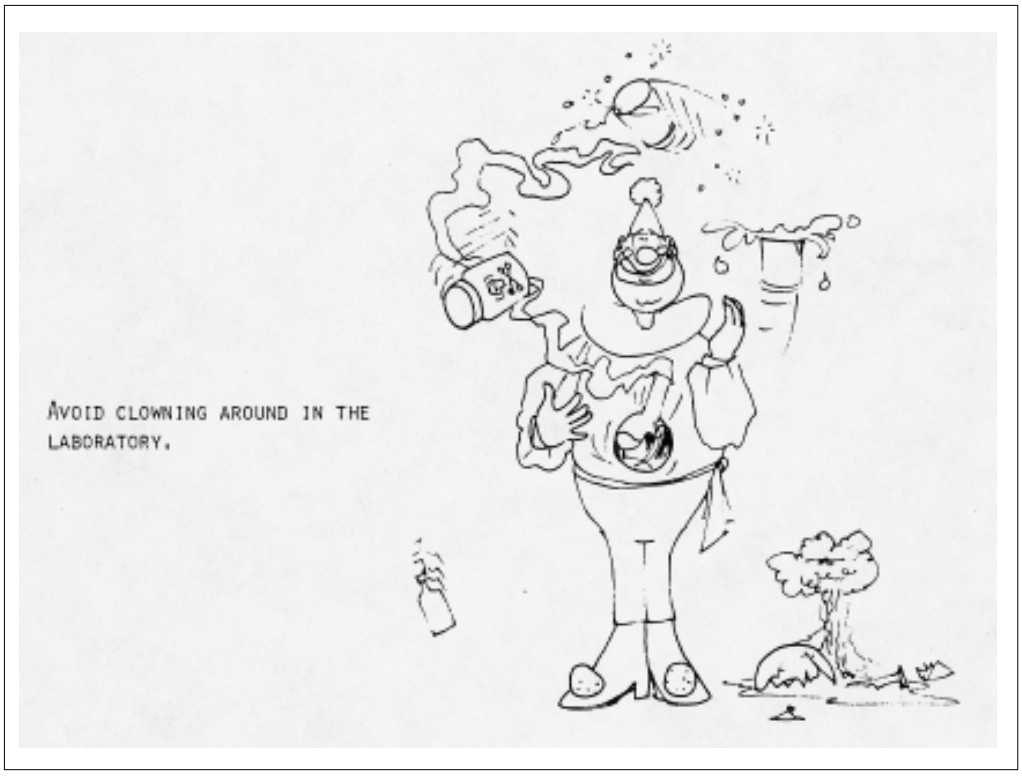
In order to do this, flammable materials should not be kept in glass containers if at all possible, due to the danger of breakage. The containers should be as small as is practical and there should be no more on hand than necessary. Where flammables must be kept in glass contain-

ers for purity, these should in turn be stored and carried in a padded metal bucket or covered metal container designed for such use.

It is particularly important not to store oxidizing agents near flammable materials (or any other potential fuel), as they may react at room temperature to produce a fire or explosion. Such oxidizing agents include oxides, peroxides, nitrates, nitrites, bromates, chromates, chlorates, dichromates, perchlorates and permanganates.

Cylinders of compressed gases present hazards in addition to those involved with the particular chemicals they contain. These cylinders should never be stored near a source of heat or corrosion and should be chained in place to prevent their falling over. Incompatible gases should be isolated from one another. Valve protection caps should never be removed before transporting cylinders. Appropriate hand trucks should be used for transporting cylinders rather than rolling them along the floor, and care should be exercised at all times not to drop the cylinders or strike them together, as this may cause them to rupture and explode or release hazardous contents.

Compressed gases, corrosives, toxic compounds and flammable liquids can all be used safely in the laboratory, as can other more general chemical reagents, only when their specific hazards are understood and countered through appropriate procedures. The laboratory analyst's greatest security lies in his familiarity with and use of these procedures. Failure to learn or use them leaves both the analyst himself and those around him open to dangerous and unnecessary risks which no amount of safety equipment can alleviate.



Records

Numbers are the only products of water and wastewater analysis. The numbers a lab turns out are derived from carefully controlled measurements and enable us to better understand and control our aquatic environment. But numbers tend to be slippery. They can't be held in the hand and examined for flaws or weaknesses. Knowing what a particular test result means may be difficult, and ensuring that the number itself is accurate is sometimes next to impossible.

Laboratory records are the essential link between making measurements and interpreting the results. Without adequate records, a lab's results are worthless. It is therefore necessary to understand what records should be kept, the purpose for keeping them and how they should be used.

QUALITY CONTROL

With demands for water quality data rising, an analyst must be almost superhuman at times to keep up. In the effort, labs run the risk of being reduced to mere random number generators, spewing out endless streams of meaningless data. Quality control programs help prevent this, and in so doing they determine many of the records which an analyst must keep.

Quality control is more than a collection of steps or procedures to be carried out blindly. It is a state of mind, a basic approach to the whole issue of lab analysis. Its purpose is to reduce the level of uncertainty associated with reported analytic results. Quality control eliminates some of the assumptions an analyst must otherwise make when performing a test, assumptions such as whether a balance, incubator or other piece of equipment is living up to its manufacturers claims or specifications. Can this general type of equipment perform the role demanded of it? How does this particular item actually perform in this specific setting? How has its performance changed over time? Such questions lead to one of the general rules which should guide development of a quality control program:

-Never assume equipment is operating the way it is supposed to.

The functioning of each piece of equipment which could affect the outcome of an analysis should be checked and calibrated. Wherever possible, the calibrations should descend in a direct line from standards issued or specified by the National Bureau of Standards (NBS). Where a manufacturer sells equipment listed as meeting NBS standards, such as Class A volumetric glassware or Class S-1 weights, these can be relied upon for most operations. But other items, such as general thermometers, should be calibrated against a certified standard thermometer (one meeting NBS specifications) regularly.

Reagents and bacteriological media also need to be checked for purity, concentration and conformance to standards. Distilled or deionized water should be tested for suitability for each of the various uses to which it will be put in the lab, particularly bacteriological analyses. This leads to another general rule of quality control:

--Never assume a reagent is suitably pure or at the proper concentration merely because the label says it is.

is measuring the desired parameter. Then the results from these check samples are evaluated statistically to determine whether the differences observed are due to normal variation in the test or whether they stem from some outside influence.

Many analysts feel uncomfortable with statistics, but the techniques necessary for a quality control program have been refined and simplified to the point that they usually need involve only simple arithmetic operations. Quality control charts of the type discussed in Standard Methods are actually fairly simple to establish and maintain. The major difficulty is in analyzing enough check samples to set up the initial chart. Once this is done, it is an easy matter to plot the performance of each successive analytic run and visually determine whether the results are acceptable or not. A quality control chart should be established for each parameter that each analyst performs routinely.

One final rule of quality control should be:

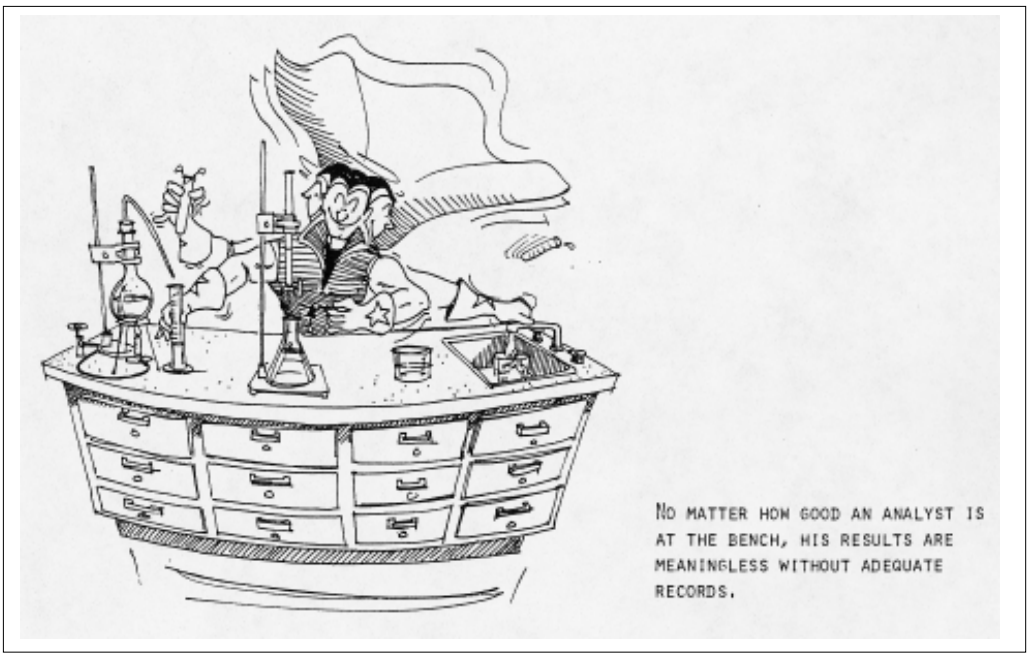
-Document everything.

When a thermometer is calibrated against a certified standard, tag the thermometer with that information. Include the analyst's name or initials and the date. Record it in a lab notebook. Record each pH meter calibration, each reading of an incubator or oven temperature, each cleaning of a still or deionizing system. Tape tables or charts to each piece of equipment which must be calibrated, next to a test tube for holding a pen. Make keeping records as simple as possible, but see that they are maintained. Without thorough records, even a bench wizard is playing a numbers game of chance with his results.'

BENCH SHEETS

A bench sheet is the first and ultimately the most important record of all those kept in a lab. It holds the key to understanding not only what happened during a particular analysis, but also what the results of that analysis signify.

A bench sheet is a work sheet for raw information, not a final, polished report. Comparisons or interpretations of results and data summaries should be kept separate, not combined with the bench sheet. The most important aspects of a bench sheet, of course, are the information it



contains and how this is arranged. Among the features which should be provided are:

-Spaces for complete sample identification. These include the date, time and location of sample collection and information on the manner of compositing, if this is done. There should also be room for brief notes on the appearance of the sample or unusual conditions which prevailed at the time it was collected.

--Logical development of test data. Each test included should be presented in a step-by-step manner reflecting the order in which data is obtained or calculations are made. Specific spaces should be included for vessel identification numbers, quantities of sample used, the amount of dilution, blank determinations, all primary readings or measurements, essential intermediate values and final test results. The data should be arranged vertically where possible so calculations can be more readily visualized.

-Space for calculations. There should be sufficient room on the bench sheet for any calculations which will be performed by hand or for recording any values that must be written down when using a calculator.


RECORD THE TEMPERATURES ON THE TOP AND MIDDLE SHELVES OF AN AIR INCUBATOR DAILY.

--Auxiliary information and formulas. Where solutions used in a procedure must be standardized routinely, the information should be recorded on the bench sheet. Formulas used should also be printed on the sheet.

--Space for comments. These may include important observations, unusual incidents, alterations in procedures or reference notes needed for proper interpretation of test results.

--A method of accountability. Where more than one person works in the lab, there should be some provision for each person to initial his or her own work, with all results checked by the person in charge. This ensures that each person takes full responsibility for his or her performance in the lab and allows later questions about technique or results to be directed to the appropriate person.

The best way to arrange a bench sheet for smaller labs often is to include all the tests normally performed on a sample on the same sheet. For instance, a basic sheet for a wastewater lab might include places for total and suspended solids, BOD, COD, pH and various other tests. A separate sheet would then be used for each sample analyzed. Each sheet would thus yield a complete description of an individual sample.

The example shows the front page of a bench sheet which incorporates the features discussed above. The top of the sheet identifies the sample and provides other information needed to interpret test results. Following is a box of miscellaneous single-reading determinations, such as dissolved oxygen (read from a probe in this case), temperature and pH. Space is provided

A Sample Bench Sheet

DATE: _____

SAMPLE LOCATION: _____ SAMPLED BY: _____

SAMPLE TIME: _____

Comments on Sample: _____

_____

_____

DISSOLVED OXYGEN (mg/l)			Cl_2 RES. (Tot.) (mg/l)		
TEMPERATURE (^{O}C)			Cl_2 RES. (Free) (mg/l)		
pH					
SETT. MATTER (ml/l)					

SOLIDS

Type of Solids					Average
Dish #					
Sample Volume (ml)					
Wt. Dry & Dish (gm) Wt. Dish (gm)					
Wt. Dry (gm)					
Solids (mg/l) = $\frac{\text{Wt. Dry x 1,000,000}}{\text{ml of sample}}$					
Wt. Dish & Dry Wt. Dish & Ash					
Wt. Volatile Matter					
% Volatile = $\frac{\text{Wt. Vol.}}{\text{Wt. Dry}}$ x 100					

Tested by: _____ Checked by: _____

Comments: _____

_____

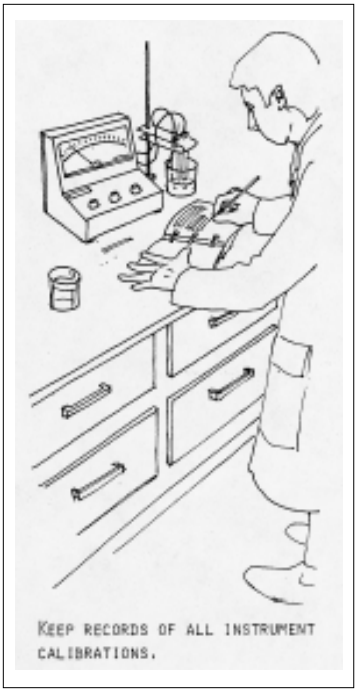
_____

_____

_____

for the measurement itself as well as the initials of the person making the reading. Blank sets of spaces are used for other determinations as needed.

The rest of the sheet is devoted to solids determinations. The columns are marked off in pairs so tests can be run in duplicate. Space is provided at the top to indicate suspended, dissolved or total solids. Most of the data are entered in the order in which they are accumulated, beginning with the number of the weighing dish and the volume of~sample used. The weights obtained are entered in reverse order, however, with that of the clean dish (the first weight obtained) going beneath that of the dish containing dried sample (the second weight). This makes the subtraction easier and provides a visual check on the answer. The weight of the dried solids is determined from this, followed by the concentration of solids in the sample. The formula necessary for this calculation is provided on the form. All calculations are to be done on a calculator, so room is not needed on the sheet for this.



Data for the percent volatile material is arranged similarly, with the weighings inverted for easier subtraction and with all calculations indicated on the form. This is followed by spaces for the initials of the person performing the test and those of the person checking the results. Lines are left at the bottom of the page for notes on any unusual incidents that occurred or procedures that were followed during the determinations.

The BOD test is the most complex analysis performed by many smaller laboratories. Here is a portion of a bench sheet designed for the BOD test. This could be used on the back of the bench sheet shown earlier. Basically, information for the test is divided into three parts. Two of these, the “seed” correction data and the actual sample BOD data, are included in a box just under the test heading. These two parts comprise the test itself. The third part is composed of miscellaneous information, such as who set up the test, who read it, who checked it, the behavior of the unseeded dilution water blank and appropriate comments. This is all located immediately under the data box.

The left half of the box contains the information used to correct for the seed content in the dilution water. This portion would only be used if seeding was necessary following effluent disinfection or some similar process. BODs are run on the seed material, using unseeded dilution water, and the average milligrams of oxygen consumed per milliliter of seed material during the five days is calculated. The form is designed for use with a dissolved oxygen meter having a BOD probe, so spaces for titration readings are not necessary in this case. The average oxygen demand obtained is then converted into the demand exerted by the specific amount of seed material present in each sample BOD bottle.

The data for determining the BOD of the sample follows much the same pattern as that for the solids determinations. However, the dissolved oxygen readings are not inverted, as were the solids weighings, because they are obtained in the order in which they are subtracted. The decrease in dissolved oxygen observed in each bottle is then corrected for the demand ex-

BIOCHEMICAL OXYGEN DEMAND

SEED CORRECTION DATA				SAMPLE BODs				
Bottle #				Bottle #				
ml Seed (v)				ml Sample (y)				
D.O. #1				D.O. #1				
D.O. #2				D.O. #2				
Difference (d)				Difference				
DO used/ml Seed (d/v)				Seed Correction				
Average (A)				Corrected Difference (D)				
Seed Ratio (P) = ____ ml/1000 ml = 0.				BOD (mg/l) = D x 300 ml / V				
Seed Correction = (A) x (P) x (300-V)				Ave. BOD (mg/l)				

Set Up By: _____ Read By: _____ Checked By: _____

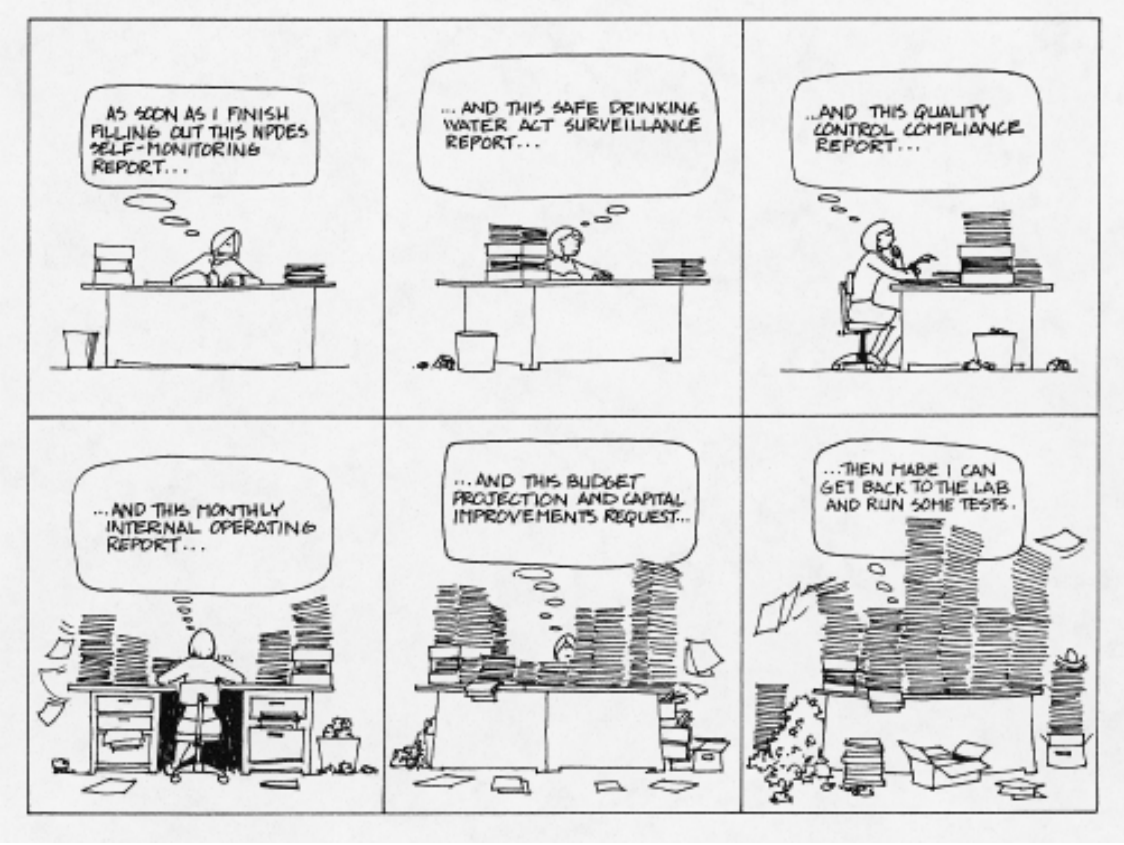
Unseeded Blanks: Day_0 = _____ mg/l Day_5 = _____ mg/l

Comments: _____

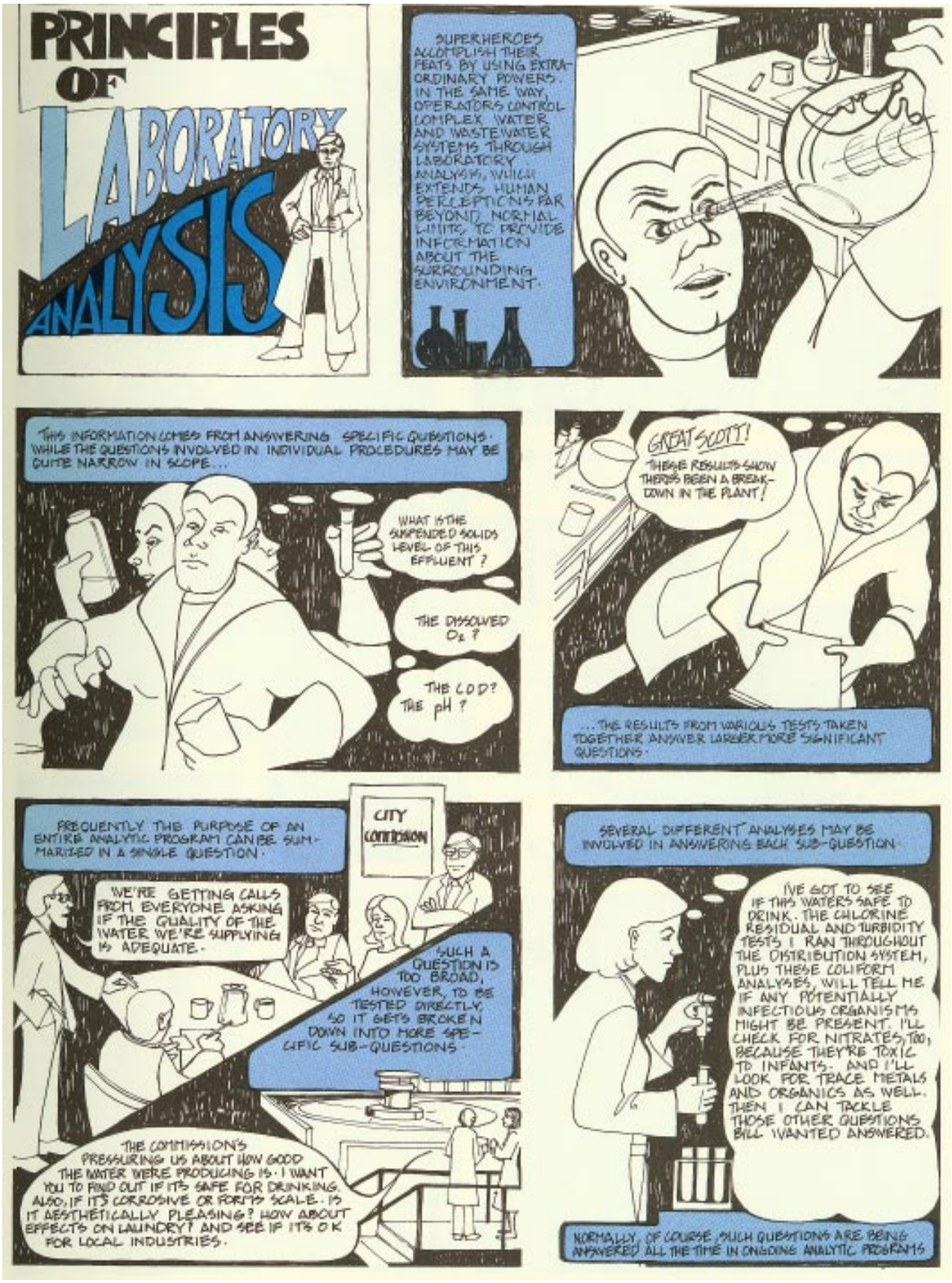
A PORTION OF A BENCH SHEET FOR RECORDING BIOCHEMICAL OXYGEN DEMAND DATA

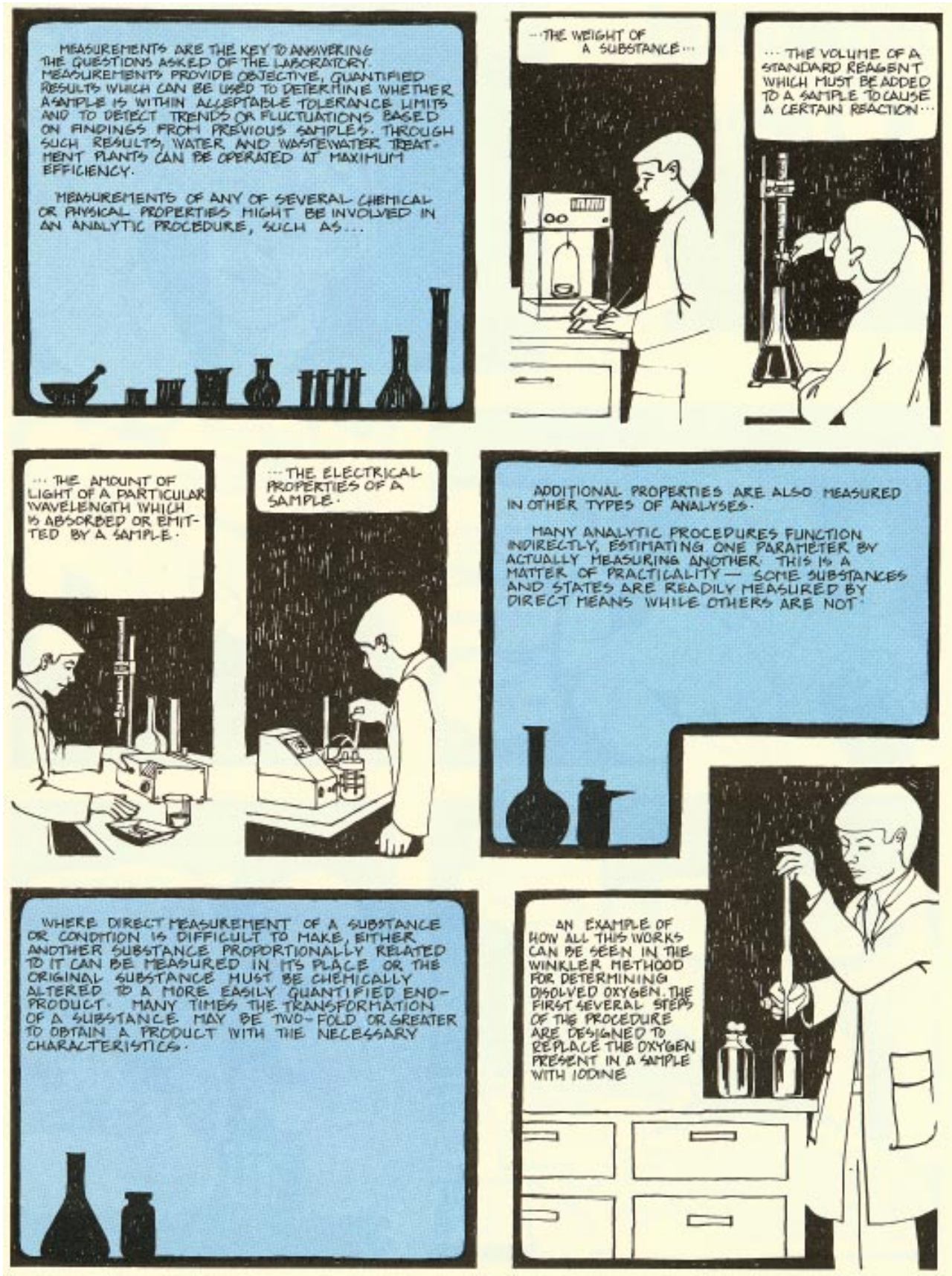
erted by the seed material, and the BOD is calculated from this corrected difference. Again, all formulas used in the calculations are presented on the form, and a space is provided for the average BOD obtained from the various dilutions.

When completely filled out, a bench sheet such as this is a thorough record of the condition of the effluent on a given day. While this particular sheet is primarily intended for analyzing wastewater treatment plant effluent samples, its features extend to all types of bench sheets used in the laboratory. The principles of proper data handling are the same whatever the particular test employed.



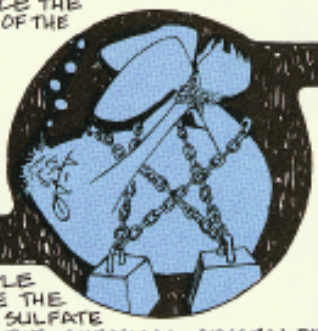
Appendix - Principles of Laboratory Analysis




MEASUREMENTS ARE THE KEY TO ANSWERING THE QUESTIONS ASKED OF THE LABORATORY. MEASUREMENTS PROVIDE OBJECTIVE, QUANTIFIED RESULTS WHICH CAN BE USED TO DETERMINE WHETHER A SAMPLE IS WITHIN ACCEPTABLE TOLERANCE LIMITS AND TO DETECT TRENDS OR FLUCTUATIONS BASED ON FINDINGS FROM PREVIOUS SAMPLES. THROUGH SUCH RESULTS, WATER AND WASTEWATER TREATMENT PLANTS CAN BE OPERATED AT MAXIMUM EFFICIENCY.
MEASUREMENTS OF ANY OF SEVERAL CHEMICAL OR PHYSICAL PROPERTIES MIGHT BE INVOLVED IN AN ANALYTIC PROCEDURE, SUCH AS...
...THE WEIGHT OF A SUBSTANCE...
...THE VOLUME OF A STANDARD REAGENT WHICH MUST BE ADDED TO A SAMPLE TO CAUSE A CERTAIN REACTION...
...THE AMOUNT OF LIGHT OF A PARTICULAR WAVELENGTH WHICH IS ABSORBED OR EMITTED BY A SAMPLE.
...THE ELECTRICAL PROPERTIES OF A SAMPLE.
ADDITIONAL PROPERTIES ARE ALSO MEASURED IN OTHER TYPES OF ANALYSES.
MANY ANALYTIC PROCEDURES FUNCTION INDIRECTLY, ESTIMATING ONE PARAMETER BY ACTUALLY MEASURING ANOTHER. THIS IS A MATTER OF PRACTICALITY — SOME SUBSTANCES AND STATES ARE READILY MEASURED BY DIRECT MEANS WHILE OTHERS ARE NOT.
WHERE DIRECT MEASUREMENT OF A SUBSTANCE OR CONDITION IS DIFFICULT TO MAKE, EITHER ANOTHER SUBSTANCE PROPORTIONALLY RELATED TO IT CAN BE MEASURED IN ITS PLACE OR THE ORIGINAL SUBSTANCE MUST BE CHEMICALLY ALTERED TO A MORE EASILY QUANTIFIED END-PRODUCT. MANY TIMES THE TRANSFORMATION OF A SUBSTANCE MAY BE TWO-FOLD OR GREATER TO OBTAIN A PRODUCT WITH THE NECESSARY CHARACTERISTICS.
AN EXAMPLE OF HOW ALL THIS WORKS CAN BE SEEN IN THE WINKLER METHOD FOR DETERMINING DISOLVED OXYGEN. THE FIRST SEVERAL STEPS OF THE PROCEDURE ARE DESIGNED TO REPLACE THE OXYGEN PRESENT IN A SAMPLE WITH IODINE


THE PRESENCE OF FREE IODINE CAN BE DETECTED WITH THE ADDITION OF A SMALL AMOUNT OF STARCH, WHICH REACTS WITH THE IODINE TO FORM A BLUE COLOR.
TITRATING THE SAMPLE WITH A STANDARD SOLUTION (THAT IS, A SOLUTION OF KNOWN CONCENTRATION) OF SODIUM THIOSULFATE BINDS THE IODINE, LEAVING IT UNABLE TO REACT WITH THE STARCH. THE BLUE COLOR DISAPPEARS.
AT THE EXACT POINT WHERE THE BLUE COLOR DISAPPEARS, THE AMOUNT OF SODIUM THIOSULFATE ADDED IS DIRECTLY PROPORTIONAL TO THE AMOUNT OF IODINE WHICH HAS BEEN REMOVED...
...WHICH IS IN TURN PROPORTIONAL TO THE AMOUNT OF OXYGEN ORIGINALLY REPLACED. KNOWING THESE PROPORTIONALITIES AND THE AMOUNT AND CONCENTRATION OF THE SODIUM THIOSULFATE USED, IT IS A RELATIVELY SIMPLE PROCESS TO CALCULATE THE ORIGINAL CONCENTRATION OF DISSOLVED OXYGEN IN THE SAMPLE.
1.7 mg/l DO
OFTEN CONSTITUENTS OTHER THAN THE ONE BEING MEASURED MAY INTERFERE WITH AN ANALYTIC PROCEDURE...
EITHER AN INTERFERING AGENT MAY INHIBIT A PARTICULAR REACTION, CAUSING A FALSE LOW VALUE TO BE OBTAINED FROM THE TEST...
MEASUREMENTS MADE HERE
...OR IT MAY REACT IN A MANNER SIMILAR TO THE SUBSTANCE BEING SOUGHT, IN WHICH CASE THE VALUE OBTAINED WOULD BE FALSELY HIGH.
IN EITHER CASE, THE EFFECTS OF SUCH INTERFERENCES MUST BE CONTROLLED.

ONE METHOD FOR ELIMINATING THE EFFECTS OF AN INTERFERING AGENT IS TO MASK THE SUBSTANCE'S PRESENCE. WHILE THE SUBSTANCE REMAINS IN THE SAMPLE, IT IS CHEMICALLY "BOUND" SO IT CAN NO LONGER INFLUENCE THE OUTCOME OF THE TEST.

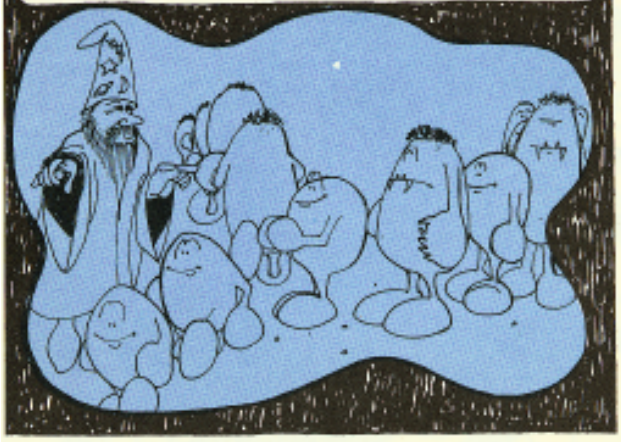


AN EXAMPLE WOULD BE THE MERCURIC SULFATE ADDED IN THE CHEMICAL OXYGEN DEMAND TEST. IT BINDS WITH ANY CHLORIDES PRESENT, PREVENTING THEM FROM INTERFERING WITH THE TEST

INTERFERING AGENTS MAY ALSO BE CHEMICALLY DESTROYED BY PRETREATING THE SAMPLE.

AN EXAMPLE OF THIS WOULD BE NITRITES IN THE WINKLER DISSOLVED OXYGEN TEST, WHICH ARE ELIMINATED THROUGH A REACTION WITH SODIUM AZIDE.

ANOTHER METHOD OF HANDLING INTERFERING AGENTS IS TO PHYSICALLY SEPARATE THEM FROM THE SUBSTANCE BEING ANALYZED.



A WIDE VARIETY OF SEPARATION TECHNIQUES EXIST, RANGING FROM SIMPLE FILTRATION FOR REMOVING PARTICULATE MATERIAL TO GAS CHROMATOGRAPHY FOR ISOLATION OF ORGANIC COMPOUNDS. IN ITS BROADEST SENSE, THIS CATEGORY ALSO INCLUDES SUCH TECHNIQUES AS ION EXCHANGE, SOLVENT EXTRACTION AND DISTILLATION. SOME PARTICULARLY INVOLVED ANALYTIC PROCEDURES MAY REQUIRE SEVERAL SEPARATION STEPS TO BE CARRIED OUT, EACH FOR THE REMOVAL OF A DIFFERENT KIND OF INTERFERING AGENT.

SOMETIMES THE CONCENTRATION OF A SAMPLE MUST ALSO BE ALTERED. DRYING MAY BE USED TO CONCENTRATE CONTAMINANTS WHICH OCCUR IN SUCH TRACE AMOUNTS THAT DIRECT ANALYSIS IS DIFFICULT.

AT OTHER TIMES, SAMPLES MUST BE DILUTED. THIS IS ROUTINELY DONE IN FECAL COLIFORM ANALYSIS OF WASTEWATER EFFLUENTS AND MAY OFTEN BE REQUIRED FOR SUCH PROCEDURES AS BIOCHEMICAL OXYGEN AND CHEMICAL OXYGEN DEMANDS.

THE ACTUAL MEASUREMENT INVOLVED IN AN ANALYTIC PROCEDURE IS RARELY DIFFICULT IN ITSELF — OBTAINING A SUBSTANCE FREE OF INTERFERENCES AND FOR WHICH MEASUREMENT IS PRACTICAL OFTEN IS. SEPARATIONS, CHEMICAL TRANSFORMATIONS AND OTHER SUCH PROCEDURES THEREFORE FREQUENTLY ACCOUNT FOR MOST OF THE STEPS INVOLVED IN A GIVEN TEST.