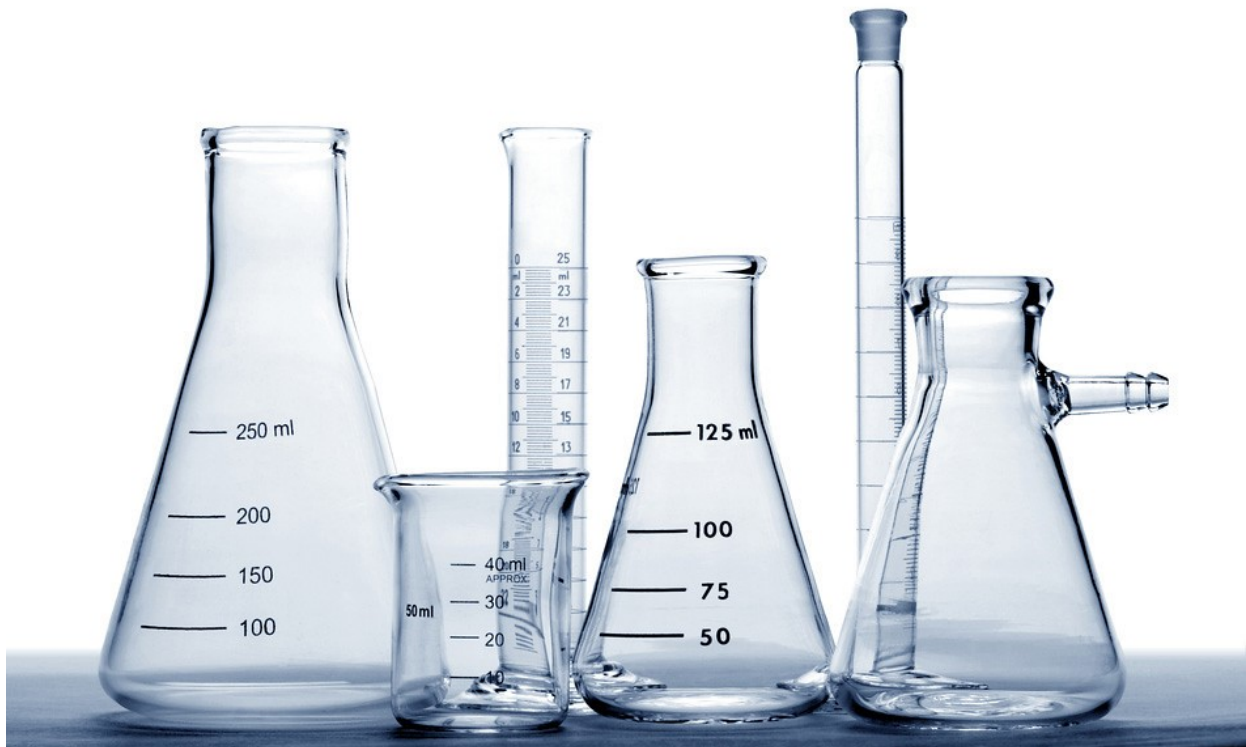


Introduction to Laboratory Methods for Operators

Course #2010



Department of
**Environment &
Conservation**

Introduction to Laboratory Methods for Operators Course #2010

February 1-5, 2020

Monday, February 1

- 8:30 Introduction and Welcome
- 8:45 Laboratory Policies and Safety
- 10:00 Identification of Laboratory Equipment
- 12:30 Lunch
- 2:00 Intro to Glassware and Pipetting Skills
- 3:00 Introduction to the Metric System and Conversions

Instructor: Sarah Snyder

Phone: 615-898-6506

Fax: 615-898-8064

E-mail: sarah.snyder@tn.gov

Tuesday, February 2

- 8:30 Analytical Balances and Weights
- 10:00 Introduction to Basic Chemistry
- 11:00 Lunch
- 12:00 Common Chemicals and Reagents
- 1:00 Temperature Measurement
- 1:30 Dissolved Oxygen Measurement

State of Tennessee

Dept. of Environment & Conservation

Fleming Training Center

2022 Blanton Dr.

Murfreesboro, TN 37129

Wednesday, February 3

- 8:30 Proper sample collection techniques, data collection, and documentation
- 9:30 Turbidity Measurement Procedure
- 10:30 pH Theory and Measurement Procedure
- 11:30 Lunch
- 12:30 Standard Operating Procedures (SOPs) and Standard Methods
- 2:30 Brief introduction to QA/QC

Thursday, February 4

- 8:30 Solutions Chemistry – dilutions and making standards
- 10:00 Chlorine Procedures
- 11:00 Lunch
- 12:00 Alkalinity Measurement Procedures
- 3:00 Equipment Maintenance and Troubleshooting

Friday, February 5

- 8:30 Exam Review
- 10:30 Exam and Course Evaluation



Introduction to Laboratory Methods for Operators

Course #2010

Section 1	Basic Laboratory Safety	3
Section 2	Laboratory Glassware and Equipment	17
Section 3	Metric System	39
Section 4	Balances and Pipetting	53
Section 5	Introduction to Basic Chemistry	67
Section 6	Temperature	83
Section 7	Dissolved Oxygen	89
Section 8	Sampling	131
Section 9	Turbidity	139
Section 10	pH	145
Section 11	Standard Methods, SOP's & QA/QC	181
Section 12	Chlorine	197
Section 13	Alkalinity	223
	Answers to Review Questions	228

Section 1
Laboratory Safety

Introduction to Laboratory Methods for Operators

Laboratory Safety



Safety

- Operators work around many different kinds of hazards on a daily basis
 - Electrical
 - Bacteriological/Viral
 - Confined space
 - Mechanical
 - Traffic



Safety

- Occupational Safety and Health Act (OSHA)
 - Demands that proper safety procedures be exercised in the lab at all times
 - "each employer has the general duty to furnish all employees with employment free from recognized hazards causing, or likely to cause, death or serious physical harm"
- TOSHA
 - Contact [local office](#) for specific questions

Laboratory Hazards

- Infectious Materials
- Poisons
- Explosions
- Cuts and Bruises
- Electric Shock
- Toxic Fumes
- Fire
- Burns



Be Aware

- Learn the lay-out of the lab
 - Emergency exits
 - Emergency routes
 - Emergency ventilation system
 - Fire-fighting equipment locations/ know how to use it
 - Eye wash station/Emergency shower
 - First Aid equipment
 - Emergency phone numbers

Infectious Materials

- Wastewater and sludge contain millions of microorganisms
- Some are infectious and can cause disease
 - Tetanus
 - Typhoid
 - Dysentery
 - Hepatitis
 - Parasitic worms



Infectious Materials

- Change out of work clothes before leaving
 - Prevent spread of infectious material into your home
- Inoculations from doctor/health dept.
 - Tetanus, polio, hepatitis A and B
 - Diseases contracted through breaks in skin, cuts, puncture wounds
 - Wastewater risk: breathing contaminated air

Infectious Materials

- Always wash hands with soap and water, especially before handling food or smoking
 - Hand sanitizer is not sufficient
- Never pipet by mouth
 - Could lead to serious illness or death
 - Use mechanical or rubber bulbs
- Never drink from a beaker or other lab glassware



Corrosive Chemicals

- Acids: a chemical substance that neutralizes alkalis, dissolves some metals; turns litmus paper red; typically a corrosive liquid
- Extremely corrosive to human tissue, metals, clothing, wood, cement, stone, concrete
- Sulfuric acid (H_2SO_4)
- Hydrochloric or muriatic (HCl)
- Nitric (NHO_3)
- Glacial acetic ($H_4C_2O_2$)

Corrosive Chemicals

- Bases: turn litmus paper blue, pH greater than 7
- Extremely corrosive to skin, clothing, and leather
- Sodium hydroxide, aka "caustic soda" or "lye" (NaOH)
- Potassium hydroxide (KOH)
- Chlorine (and other oxidants)

Corrosive Chemicals

- Commercially available chemical spill clean-up materials should be kept on hand
- Baking soda (bicarbonate) effectively neutralizes acids
- A jug of ordinary vinegar can be kept on hand to neutralize bases

Toxic Materials

- Solids:
 - Cyanide, chromium, heavy metals
- Liquids:
 - Chlorine, nitric acid, ammonium hydroxide, chloroform, organic solvents
- Gases:
 - Chlorine, ammonia, hydrogen sulfide, sulfur dioxide, and chlorine dioxide

Explosive or Flammable Materials

- Liquids:
 - Acetone, ethers, gasoline, benzene
- Gases:
 - Propane, hydrogen, acetylene

Personal Protective Equipment

- Known as **PPE**
- Safety Glasses
- Face Shield
- Lab Coat
- Lab Apron
- Gloves
 - Rubber, heat resistant
- Closed Toed Shoes
 - Steel-toed boots



Personal Safety and Hygiene

1. Never work alone in the lab
 - In case of accident or fire
 - If necessary, have someone check on you regularly
2. Wear protective goggles or safety glasses at all times
 - Fumes can seep between contact lens and eyeball



Personal Safety and Hygiene

3. Wear a face shield if there is danger of hot liquid erupting or flying glassware due to explosion
4. Wear protective or insulated gloves when handling hot or cold objects, or when handling liquids or solids that are skin irritants



Personal Safety and Hygiene

5. Always wear a lab coat or apron
6. Never pipet by mouth
7. Never eat or smoke in the lab
8. Do not keep food in a refrigerator that is used for chemical or sample storage



Personal Safety and Hygiene



Avoid using cell phones and other personal electronic devices in the laboratory! Remove gloves, wash hands, then use the device.




Partners and with permission of Cornell University
Original source: www.cornell.edu/ehp/ehp101

Nebraska
Department of Health & Senior Services
© 2012-13










Personal Safety and Hygiene

9. Use ventilated lab fume hoods when handling toxic chemicals
10. Maintain clear access to emergency eye wash stations/showers
 - Flush weekly
11. Practice good housekeeping to prevent accidents

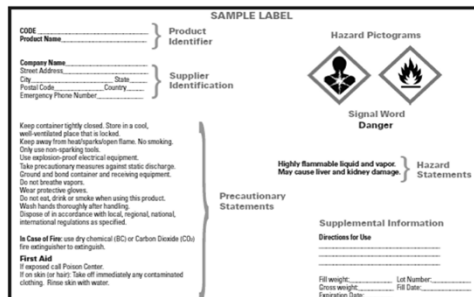


Manufacturer Label Requirements

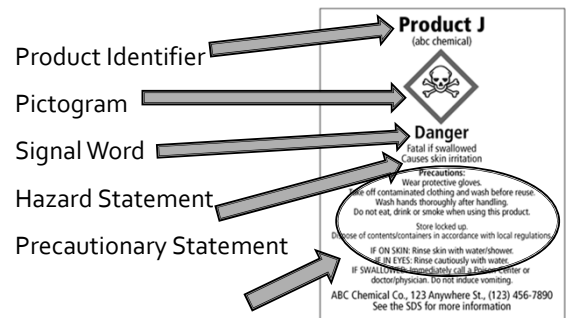
1. Product Identifier: The name used for a hazardous chemical on the label and in the SDS
2. Pictogram
3. Signal Word: Used to indicate the relative level of severity of hazard and alert the reader to a potential hazard
 - Danger—more severe hazard
 - Warning—less severe hazard
4. Hazard Statement: describes the nature of the hazard
5. Precautionary Statement: describes recommended measures that should be taken to minimize or prevent adverse effects resulting from exposure or improper storage or handling

HCS Pictograms and Hazards		
Health Hazard  <ul style="list-style-type: none"> ▪ Carcinogen ▪ Mutagenicity ▪ Reproductive Toxicity ▪ Respiratory Sensitizer ▪ Target Organ Toxicity ▪ Aspiration Toxicity 	Flame  <ul style="list-style-type: none"> ▪ Flammables ▪ Pyrophorics ▪ Self-Heating ▪ Emits Flammable Gas ▪ Self-Reactives ▪ Organic Peroxides 	Exclamation Mark  <ul style="list-style-type: none"> ▪ Irritant (skin and eye) ▪ Skin Sensitizer ▪ Acute Toxicity ▪ Narcotic Effects ▪ Respiratory Tract Irritant ▪ Hazardous to Ozone Layer (Non-Mandatory)
Gas Cylinder  <ul style="list-style-type: none"> ▪ Gases Under Pressure 	Corrosion  <ul style="list-style-type: none"> ▪ Skin Corrosion/Burns ▪ Eye Damage ▪ Corrosive to Metals 	Exploding Bomb  <ul style="list-style-type: none"> ▪ Explosives ▪ Self-Reactives ▪ Organic Peroxides
Flame Over Circle  <ul style="list-style-type: none"> ▪ Oxidizers 	Environment (Non-Mandatory)  <ul style="list-style-type: none"> ▪ Aquatic Toxicity 	Skull and Crossbones  <ul style="list-style-type: none"> ▪ Acute Toxicity (fatal or toxic)

Manufacturer Container Label



Manufacturer Container Label



Safety Data Sheets (SDS)

- Includes all information on chemical label and specific info pertaining to that chemical
- 16 sections
 - ID, Hazards, Ingredients, First Aid, Fire-Fighting, Accidental Release, Handling/Storage, PPE, Physical and Chemical Properties, Stability/Reactivity, Toxicological Info, Ecological Info, Disposal, Transport Info, Regulatory Info

SDS

- All chemicals in the facility currently:
 - In a labeled notebook or binder
 - Specific location near the entrance
 - Must be yellow or safety orange
- Must keep on file for all chemicals purchased
 - On file for at least 30 years

Chemical Storage

- Properly ventilated
- Well lit
- Laid out to segregate incompatible chemicals
 - Not in alphabetical order
- Order and cleanliness must be maintained
- Clearly label and date all chemicals and bottles of reagents
 - Chemicals transferred to different containers MUST be labeled

Chemical Storage

- Store heavy items on or as near to floor as possible
- Volatile liquids that may escape as a gas, such as ether, must be kept away from heat sources, sunlight, and electric switches
 - Flammable cabinet
- Cap and secure cylinders of gas in storage to prevent rolling or tipping

Chemical Storage

- Store acids and bases in separate storage cabinets
- If incompatible chemicals are inadvertently mixed a fire, explosion, or toxic release can easily occur
- For especially dangerous materials, use a secondary container (e.g. plastic tub) large enough to contain a spill of the largest container

Chemical Storage



Moving chemicals

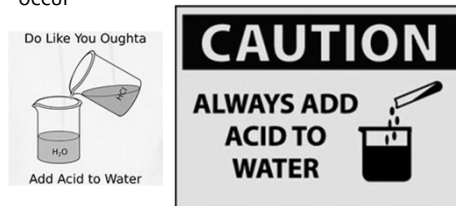
- Use cradles or tilters for carboys or other large chemical vessels
- Use a trussed hand truck for transporting cylinders of compressed gas
- Never roll a cylinder by its valve
- Clamp securely into place to prevent shifting or toppling
- Carry flammable liquids in safety cans
 - Gloves, safety shoes, rubber apron

Proper lab technique

- Acids and other corrosives
 - Flush outside of acid bottles with water before opening
 - Do not lay stopper/lid on counter where person may rest arm or hand
 - Keep all acids tightly stoppered when not in use
 - Immediately clean up spills

Proper lab technique

- Always add acid to water
 - If you pour water into acid, violent splashing may occur



Mercury

- Even a small amount of spilled mercury can poison the atmosphere in a room
- To clean up a small spill (amount in a thermometer):
 - Put on rubber, nitrile, or latex gloves
 - Use squeegee or cardboard to sweep mercury beads together
 - Use disposable dropper/pipet to suck up beads
 - Slowly squeeze mercury onto damp paper towel
 - Place in labeled zip lock bag for proper disposal



Electric shock

- Follow the usual "do's" and "do not's"
- Ground all apparatus with 3 prong plugs
- Do not continue to run a motor after liquid has spilled on it
 - Turn off immediately, clean and dry inside thoroughly before use
- Electrical units operated in an area exposed to flammable vapors should be explosion proof

Cuts

- Some lab glassware must be inserted through rubber stoppers
 - Glass tubing, thermometers, funnels
 - Ends should be flame polished and either wetted or lubricated
 - Never use oil or grease
 - Wear gloves
 - Hold tubing as close to end being inserted as possible to prevent bending/breaking
- Never force rubber tubing or stoppers from glassware

Cuts

- Examine all glassware before use
 - Discard any broken pieces in the appropriate sharps container
- Never store broken glassware in cabinets
 - Damaged glassware should either be sent for repair or disposed of properly
- Use gloves when sweeping up broken glass, do not use bare hands
 - Pick up fine glass particles with wet paper towel

Burns

- Immediately wash off splatterings of acids, caustics, and strong oxidizers with large amounts of water
- Every worker should have access to a sink and emergency deluge shower
- Keep vinegar and baking soda handy to neutralize bases and acids
 - Vinegar neutralizes bases
 - Baking soda neutralizes acids

Burns

- Heat resistant gloves
- Safety tongs to handle hot glassware
 - Do not juggle from hand to hand
- Most harmful and painful chemical burn = Eyes
 - Immediately flood eyes with water or special eyewash solution
 - Rinse within 1 minute of the burn
 - Flush at least 20 minutes
 - Consult doctor
- Alkali powder (such as lime) should be brushed off before adding water

First Aid

- First Aid box should:
 - Be easy to access
 - Be easy to identify
 - Be adequately stocked (re-stocked quickly after use)
 - Contain a copy of basic first aid instructions
- Call 911 for major accidents
- Notify manager ASAP



Toxic Fumes

- Use ventilated fume hood
 - Work at least 6 inches inside the hood
 - Annual maintenance
- Do not store chemicals in fume hood
 - Can impede proper air flow
 - Do not block rear exhaust slot
- When working with chlorine and other toxic substances, always wear a self-contained breathing apparatus

Waste Disposal

- Corrosive materials should never be poured down the sink
 - Corrode the drain pipe or trap
- Corrosive acids should be neutralized and poured down corrosive-resistant sinks
 - Use large amounts of water to dilute and flush
- Broken glassware goes into designated sharps container



Fire

- Lab should be equipped with a fire blanket
 - Smother clothing fires
- Small fires in evaporating dish or beaker can be extinguished with
 - Glass plate
 - Wet towel
 - Wet blanket
- Do not use fire extinguisher on small beaker fire

Fire

- You must use the proper fire extinguisher for each class of fire
- Ex: Never pour water onto grease fires, electrical fires, or metal fires
 - Increase the hazard – splattering the fire or electric shock
- Fires are classified according to the materials being consumed
 - A,B,C, or D

Fire

- **Class A** = Ordinary combustibles
 - Wood
 - Paper
 - Cloth
 - Rubber
 - Many plastics
 - Grass, hay
- Use foam, water, soda-acid, carbon dioxide gas, or almost any type of extinguisher



Fire

- **Class B** = Flammable and combustible liquids
 - Gasoline
 - Oil
 - Grease
 - Tar
 - Oil-based paint
 - Solvents
 - Flammable gases
- Use foam, carbon dioxide, or dry chemical extinguishers



Fire

- **Class C** = Energized electrical equipment
 - Starters
 - Breakers
 - Motors
- Use carbon dioxide or dry chemical extinguishers to smother the fire
 - Both types are nonconductors of electricity



Fire

- **Class D** = Combustible metals
 - Magnesium
 - Sodium
 - Zinc
 - Potassium
- Use a Class D extinguisher or use fine dry soda ash, sand, or graphite to smother the fire
- Operators rarely encounter this type of fire

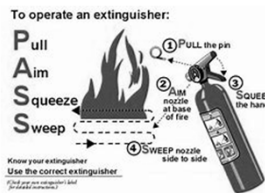


Fire Extinguishers

- A multipurpose ABC carbon dioxide extinguisher will handle most laboratory fires
 - Visual inspection – monthly
 - Maintenance check – annually
- Consult with your local fire dept. about best methods to use for specific hazards that exist at your facility

Fire Extinguishers

1. Pull the pin out
2. Aim the nozzle at the base of the fire
3. Squeeze the handle
4. Sweep the nozzle/spray from side to side
 - To contain the fire



Pregnancy

- Pregnant women should avoid teratogens
 - Teratogen = reproductive toxins that may cause damage to the fetus
- Ask supervisors to alter schedules/work assignments if the potential for exposure exists
- THM Plus method (Trihalomethanes) by Hach uses Chloroform, a teratogen

Key Points to Remember

- Be aware of the hazards around you
- Educate yourself
- Review safety procedures on a regular basis
- Take charge of your own safety
- Don't get complacent

Laboratory Safety – Review Questions

1. List at least 5 laboratory hazards.
2. Why should you never work alone in the laboratory?
3. You may add acid to water, but never add water to acid. True or False?
4. How would you dispose of a corrosive acid?
5. What does SDS stand for?
6. How long should SDS's be kept on file?
7. What is a signal word? List the 2 signal words that could be found on a chemical label and what each one represents.
8. What should you do if you get a chemical in your eyes?
9. What would you do if you spilled a concentrated acid on your hand?
10. List 4 types of Personal Protective Equipment.

11. Any work that has the potential to generate hazardous or toxic vapors or fumes should be conducted where?
12. What common household products should you keep on hand to neutralize acids and bases?
13. How often should (plumbed) eye wash stations and emergency safety showers be flushed?
14. Chemicals should be stored in alphabetical order for quick access. True or false?
15. Why must acids and bases be stored in separate cabinets?
16. How would you extinguish a small beaker fire?
17. List the 4 Classes of fire extinguishers mentioned in our presentation and the materials being consumed in each class.
18. What does P.A.S.S. stand for?
19. What is a teratogen?



U.S. Department of Labor

OSHA[®]
Occupational Safety
and Health Administration

Job Safety and Health IT'S THE LAW!

All workers have the right to:

- A safe workplace.
- Raise a safety or health concern with your employer or OSHA, or report a work-related injury or illness, without being retaliated against.
- Receive information and training on job hazards, including all hazardous substances in your workplace.
- Request an OSHA inspection of your workplace if you believe there are unsafe or unhealthy conditions. OSHA will keep your name confidential. You have the right to have a representative contact OSHA on your behalf.
- Participate (or have your representative participate) in an OSHA inspection and speak in private to the inspector.
- File a complaint with OSHA within 30 days (by phone, online or by mail) if you have been retaliated against for using your rights.
- See any OSHA citations issued to your employer.
- Request copies of your medical records, tests that measure hazards in the workplace, and the workplace injury and illness log.

This poster is available free from OSHA.

Contact OSHA. We can help.

Employers must:

- Provide employees a workplace free from recognized hazards. It is illegal to retaliate against an employee for using any of their rights under the law, including raising a health and safety concern with you or with OSHA, or reporting a work-related injury or illness.
- Comply with all applicable OSHA standards.
- Report to OSHA all work-related fatalities within 8 hours, and all inpatient hospitalizations, amputations and losses of an eye within 24 hours.
- Provide required training to all workers in a language and vocabulary they can understand.
- Prominently display this poster in the workplace.
- Post OSHA citations at or near the place of the alleged violations.

FREE ASSISTANCE to identify and correct hazards is available to small and medium-sized employers, without citation or penalty, through OSHA-supported consultation programs in every state.



Section 2

Laboratory Equipment Identification

LABORATORY GLASSWARE AND EQUIPMENT

1 Introduction to Laboratory Methods for Operators

LABORATORY GLASSWARE

2

OBJECTIVES

- Become familiar with the most common types of lab glassware you will encounter in the lab
- Understand the proper usage of each will ensure safe lab practices
- Learn to differential between the 3 main types of pipets and when to use each
- Be aware of the proper way to clean glassware



3

TYPES OF GLASSWARE

- There are different types of calibration marks and accuracy
- Glassware can be divided into 2 general categories:
 - General glassware
 - Markings are only approximate
 - Measuring glassware
 - Markings are more accurate

4

TYPES OF GLASSWARE

General Glassware

- Beakers
- Erlenmeyer flasks
- Any other glassware that is not used for precise measurements

Measuring Glassware

1. Volumetric Glassware
 - Pipets and Flasks
2. Graduated Cylinders
3. Burets

5

BEAKERS



- Used for:
 - Holding, Mixing, or heating liquids
 - Measuring approximate volumes
 - ~ 5 - 10% accuracy
 - Can be heated and cooled without concern for distortion of the graduation marks

6

FLASKS

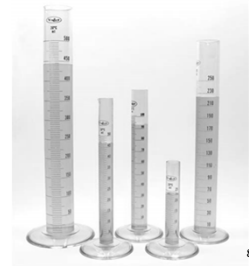


- Erlenmeyer Flasks
 - Used to hold and mix chemicals
 - The small neck prevents spilling while mixing
 - Can be heated and cooled without concern for distortion of the graduation marks

7

GRADUATED CYLINDERS

- Accurate to ~1%
- Measures liquid volumes more accurately than beakers, but still not the most accurate
- Measure quicker



8

VOLUMETRIC FLASKS



- Most accurate way to measure volume
- Used to prepare solutions to an accurate volume
- Disadvantage:
 - Only can measure one volume
 - Not used for storing or heating solutions

9

BURETTES AND TITRATIONS



- Burettes
 - Used for titrations
 - Used for dispensing an accurate volume of liquid
 - Treat like a Mohr pipet, do not let liquid completely drain out
 - Also, make sure to remove air bubble in tip before titrating

10

FLASKS



- Distilling Flask
 - Ammonia analysis
 - Distillation of samples before measurement is required unless analyst has data on file to prove the distillation step is unnecessary

11

FLASKS

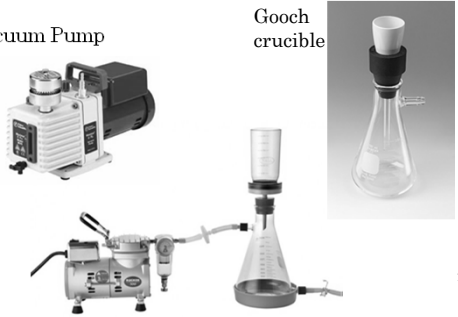


- Filter Flask
 - Attached hose barb for connecting a flexible hose/tubing for a vacuum pump
 - Used for TSS test or MLSS analysis


12

FILTER APPARATUS

- Vacuum Pump



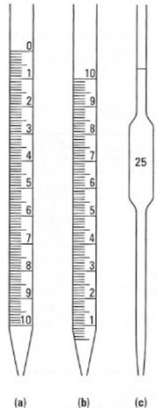
Gooch crucible



13

PIPETS

- Pipets are glass or plastic tubes, usually open at both ends, which are used to transfer specific amounts of liquid from one container to another
- 2 major categories:
 1. Volumetric (Transfer)
 2. Measuring (Mohr and Serological)




(a) Mohr
(b) Serological
(c) Volumetric

14

VOLUMETRIC PIPETS


- Used to deliver a single specific volume of liquid, usually between 1 and 100 ml
- Shaped like rolling pins with a large belly, one blunt end, the neck, and one tapering end, the tip



15

VOLUMETRIC PIPETS


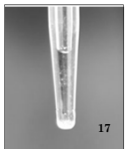
- Used for accurate measurements, since it is designed to deliver only one volume and is calibrated at that volume
- Should be used when accuracy and reproducibility are crucial, because these can achieve accuracy to four significant figures
- The most accurate pipet



16

VOLUMETRIC PIPETS

- When emptying a volumetric pipet, the liquid is allowed to drain out
 - It is NOT forced out
- After it is emptied, the small amount of liquid which remains in the tip should not be blown out
- Volumetric pipets are NOT blow-out pipets






17

MEASURING PIPETS

- 2 types:
 1. Mohr Pipet
 2. Serological Pipet
- Calibrated into small divisions so that various amounts of liquid can be measured with the same pipet
- Not as accurate as volumetric pipets

- Mohr Pipets
 - Graduations on these always end before the tip
- Serological Pipets
 - Graduation marks continue to the tip

18

Examine pipets A and B
Which is the serological and which is the Mohr?

19

SEROLOGICAL PIPETS

- Graduated in various divisions all the way to the end of the pipet
- Have a larger bore and work well with wastewater samples
- Serological pipets are TD = To Deliver
 - To accurately dispense the measured volume the last bit of liquid must be blown out.

20

MOHR PIPETS

- Graduated in various divisions that do not continue onto the end of the pipet
- No liquid should be delivered past the last graduation
 - Fluid is drawn up to a graduation mark and then dispensed to the zero mark, not completely evacuating the fluid from the pipet

21

MOHR PIPET

- To accurately transfer fluid with this type of pipet, the meniscus must be precisely on a calibration mark both at the beginning and at the end of a transfer.

22

10 mL IN 1/10 ML
TD 20°C

- Specifications on a pipet as shown above indicate that the pipet is calibrated in 1/10ml divisions and will deliver up to 10.0 ml within published tolerance levels at 20°C

23

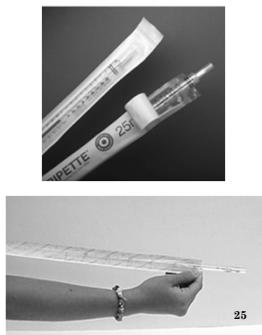
1 ML IN 1/100
TD 20°C

- These specifications indicate that the pipet is calibrated in 1/100 ml divisions and it will deliver up to 1.00 ml within published tolerance levels at 20°C

24

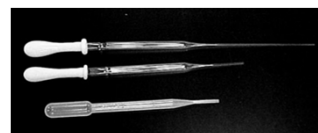
STERILE PIPETS

- When using sterile pipets, be sure to use proper sanitary techniques
- If you have a sterile package of disposable pipets, tear only a small corner of the package open and push one pipet out of this opening, then immediately close the package to prevent contamination



OTHER PIPET TYPES

- Transfer of uncalibrated volumes up to 2.5 ml can be accomplished using glass "transfer" or "Pasteur" pipets shown below. These may be sterilized before use
- Roughly calibrated volumes of 1 and 2 ml can be transferred with the one piece plastic transfer pipets which may be purchased as sterile or non-sterile units



BOTTLES

- Dilution Bottles
- Sample Bottles



WHAT TO DO WITH DIRTY PIPETS

- Place dirty pipets in soapy water solution in a tray or pipet washer
- Place disposable pipets in a cardboard holder
- Do not leave pipets on counters or sinks



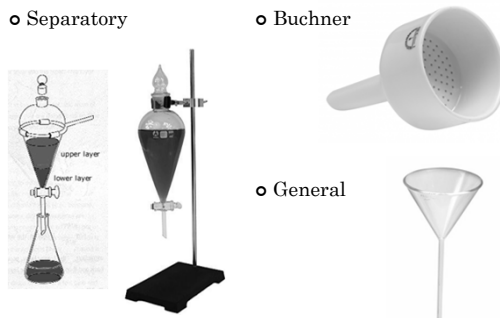
BOTTLES

- Reagent Bottles
- Weighing Bottles



FUNNELS

- Separatory
- Buchner
- General



CLEANING GLASSWARE

- Just because it looks clean does not mean residues are not left behind
- Results need to be accurate to use data for process control and/or reporting to the State
- Detergents should be phosphate free
 - Alconox contains phosphates!



31

CLEANING GLASSWARE

- Residues of minerals and other substances can build up on glassware, causing erroneous test results
- If the water beads up on the inside of glassware *after* it has been cleaned, there is residue present



32

STEPS FOR WASHING

- Clean glassware using laboratory detergent (phosphate-free)
- Rinse with tap water
- Rinse at least three times with distilled water
- Let air dry



33

STEPS FOR ACID-WASHING

- Clean glassware using laboratory detergent (phosphate-free)
- Rinse with tap water
- Rinse with 1:1 hydrochloric acid or nitric acid
 - 1:1 means equal parts distilled water and acid
 - *hydrochloric acid may interfere with TRC analysis
- Rinse well with distilled water
- Let air dry

34

Note: always use gloves and goggles when handling acids

LABORATORY EQUIPMENT

35

PETRI DISH/DESSICATOR

- Petri Dish
 - Culturing container
- Desiccators
 - Dust and moisture free



36

EVAPORATING DISH/CRUCIBLE

- Evaporating Dish
- Crucible



37

CENTRIFUGE



- Used to separate materials of different density
- Weight must be evenly distributed
 - Position test tubes across from each other to balance

38

AUTOCLAVE

- Pressure cooker used to sterilize glassware, bottles, membrane filter equip, culture media and contaminated material to be discarded
- Standard temperature is set at 121°C and 15 PSI



39

REFRIGERATORS



Walk-in cooler



- Sample storage should maintain between 1-5 °C
- Never store samples and chemicals together

40

INCUBATORS

- Artificially heated container used for growing bacteria cultures
- Dry-Heat types hold temperatures to $\pm 0.5^{\circ}\text{C}$
- For E. coli and Total coliform = $35 \pm 0.5^{\circ}\text{C}$
- Water Bath for fecal = $44.5 \pm 0.2^{\circ}\text{C}$



41

INCUBATORS

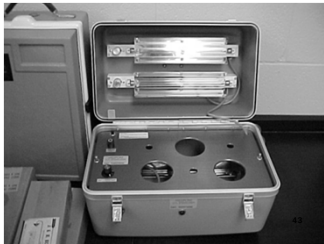


- For BOD incubation at $20 \pm 1^{\circ}\text{C}$
- Do not store chemical solutions and samples in same refrigerator

42

UV STERILIZER

- Use in Bacterial Lab to sterilize test equipment
- 3 minutes time

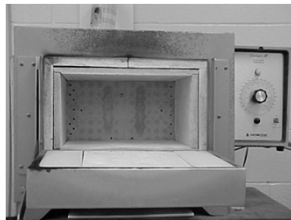


DRYING OVEN



- Used more often in wastewater labs
- For solids testing set oven at 103-105°C

MUFFLE FURNACE



- High temp oven used to ignite or burn solids
- Usually operate at temps of 550°C
- More often used in Wastewater lab work
 - MLVSS

45

FUME HOOD

- Can prevent serious accidents
- Use whenever heat is used in a test procedure
- Fumes vented out of lab
- Use when harmful smoke, gas, vapors, splashes or fumes are possible



46

WATER STILL



- Produces distilled water for lab tests and rinsing washed glassware
- Removes dissolved minerals, organic and inorganic nonvolatile compounds
- Does not sterilize

47


HEATING AND STIRRING SAMPLES

- Combo Heat/Stir Plate
 - Can be used to stir or heat and stir samples
 - Safer than heating with an open flame
- Gas Burner
 - Bunsen burner
 - Uses natural gas




BALANCES

- Top Loading
 - Weighs to the nearest 0.01 g
- Analytical
 - Precise to 0.0001 g



49

PH METER




- Use buffer solutions to calibrate
- Store electrodes properly
- Calibrate daily
- Maintain records on daily calibrations

50


SPECTROPHOTOMETER

- HACH DR 6000
 - Factory pre-set programs for lab chemical analysis
- Very versatile



51

COLORIMETERS




- Determine the concentration of many chemicals
- Most commonly used is chlorine type colorimeter
- Portable and battery powered

52


AMPEROMETRIC TITRATOR

- Chlorine analysis
- Accurate and unaffected by sample color or turbidity
- Takes greater skill use than DPD method with colorimetric devices



53

TURBIDIMETER




- Desk top and continuous on-line monitoring
- Position away from direct sunlight and have extra light bulb on hand
- Ensure sample bottles maintained; no scratches; acid clean if necessary

54

CHEMICAL STORAGE


- Do not store volatile chemicals together
- Have separate storage cabinets for acids and bases/caustics



55

FLAMMABLE CABINET

- Flammable chemicals should be kept in a flammable cabinet



56

SAFETY EQUIPMENT

Safety Glasses →

Lab Coat →


Covered Legs →

Closed Toed Shoes →

ALWAYS WEAR YOUR GLOVES

PPE (Personal Protective Equipment):

- Goggles
- Gloves
- Aprons
- Wear safety clothing



57

EYE WASH AND SHOWER

- Should be checked weekly



58

ANY QUESTIONS?

59

Laboratory Equipment – Review Questions

1. Which type of glassware is used for mixing and measuring approximate volumes?

2. Which type of glassware is calibrated to provide the most accurate volume measurement?

3. Give an example of when you would need to use the type of glassware from question #2 in the lab.

4. Mark whether or not the following types of pipets should be blown out (the last drop is forced out with the bulb).
 - a. Volumetric –
 - b. Mohr –
 - c. Serological –

5. What do you do with used glass pipets?

6. How long should glassware/equipment remain in the UV sterilization box in order to be properly sterilized?

7. What is the difference between a top loading balance and an analytical balance?

8. How often should eye wash stations and emergency showers be checked?

9. What type of detergent should be used to clean laboratory glassware?

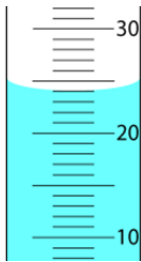
10. List the standard procedure for washing glassware.

11. What is the purpose of acid washing glassware?

12. List the steps that are required for acid washing glassware.

13. How often should laboratory grade water be tested and the (in house) system inspected?

How to Read a Meniscus



When using Graduated Cylinders, you must first learn how to read the meniscus. The meniscus is formed when the sides of the cylinder pull the water up the sides. This is due to adhesion. You should always read the bottom of the meniscus.

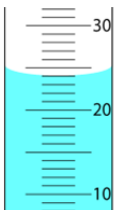
Steps to reading the meniscus in Graduated Cylinders:

1. Place the graduated cylinder on a level surface with the measurement lines facing you.
2. The water in a cylinder will form a curve called the meniscus
3. Your eye should be level with the top of the liquid
4. Find the bottom of the curved meniscus in the water. This should be in the center of the graduated cylinder.
5. Follow the lowest point at the surface of the water to the wall of the graduated cylinder. Read the volumetric scale at this point.

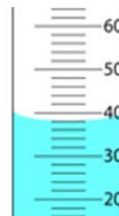
If you have trouble seeing the meniscus, try holding a black card against the opposite side of the graduated cylinder at the same height as the water.

Plastic cylinders may differ and the liquid level will have a flat surface. In that case you still read the center, not the edges.

Samples:

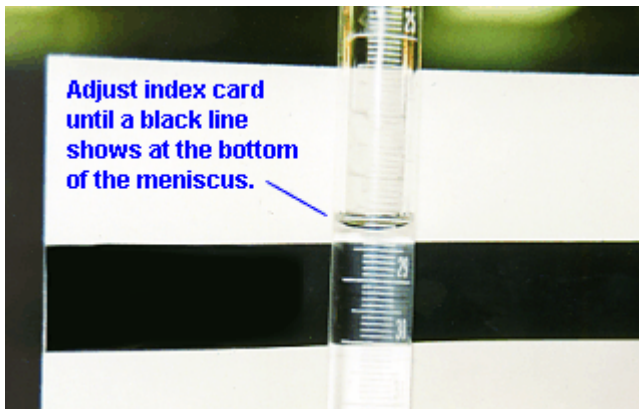


This reading would be 24



This reading would be 39

Sometimes it is helpful to use a piece of paper with a thick black line to hold behind the glassware in order to better see the meniscus.



Cleaning Glassware

We often overlook the importance of clean glassware in the lab. We think if it looks clean, it must be clean. But there may be residues on the glassware that can affect our results. Since we use those results for both running the plant and reporting water quality to the state, it is important that those results be as accurate as possible.

For many purposes in the water treatment lab, washing in a detergent such as Alconox is sufficient. However, some analyses and some glassware require special cleaning procedures to ensure removal of all residues. Residues of minerals and other substances can build up on glassware, causing erroneous test results. Always follow the recommendations for cleaning glassware and sample containers, and always use the suggested type of sample container.

The following is a partial list of special cleaning procedures for laboratory glassware used for chemical analyses:

Sample containers:

When collecting samples for metals analyses, special cleaning of the containers is necessary to prevent residues from affecting results. Clean the sample bottles by thoroughly washing with laboratory soap (preferably phosphate-free), followed by an acid wash and multiple rinses with distilled or deionized water. Do not use glass sample bottles for metals analysis.

Sample cells and cuvetts:

Wash thoroughly using laboratory soap (preferably phosphate-free), followed by an acid wash and multiple rinses with distilled or deionized water. Allow to air dry or wipe with a Kim-wipe, don't use paper towels.

Flasks, beakers, etc used for metals analysis:

Wash thoroughly using laboratory soap (preferably phosphate-free), followed by an acid wash and multiple rinses with distilled or deionized water.

Pipets:

Soak overnight in a solution of Alconox. Rinse thoroughly using a pipet washer.

Procedure for Acid Washing Glassware

If acid washing is required, follow these steps:

- Clean the glassware using laboratory detergent (phosphate-free)
- Rinse with tap water
- Rinse with 1:1 hydrochloric acid solution or 1:1 nitric acid solution
- Rinse well with distilled water
- Air dry

❖ Note: always use gloves and safety goggles when handling acids!

Basic Water and Wastewater Vocabulary

>	Greater than. (DO > 5 mg/L would be read: DO greater than 5 mg/L)
<	Less than. (DO < 5 mg/L would be read: DO less than 5 mg/L)
Aliquot	Portion of a sample
Ambient Temperature	Temperature of the surroundings
Amperometric	A method of measurement that records amount of electric current flowing or generated, rather than recording voltage. Amperometric titration is a means of measuring concentrations of certain substances in water.
Anaerobic Environment	A condition where “free” or dissolved oxygen is NOT present in aquatic environment.
Aseptic	Free from the living germs of disease, fermentation, or putrefaction. Sterile.
Blank	A bottle containing only dilution water or distilled water, but the sample being tested is not added. Tests are frequently run on a sample and a blank and the differences are compared.
Buffer	A solution or liquid whose chemical makeup neutralizes acids or bases without a great change in pH.
Buffer Capacity	A measure of the capacity of a solution or liquid to neutralize acids or bases. This is a measure of the capacity of water or wastewater for offering a resistance to changes in pH.
Colorimetric Measurement	A means of measuring unknown concentration of water quality indicators in a sample by measuring the sample’s color intensity. The color of the sample after the addition of specific chemicals (reagents) is compared with colors of known concentrations.

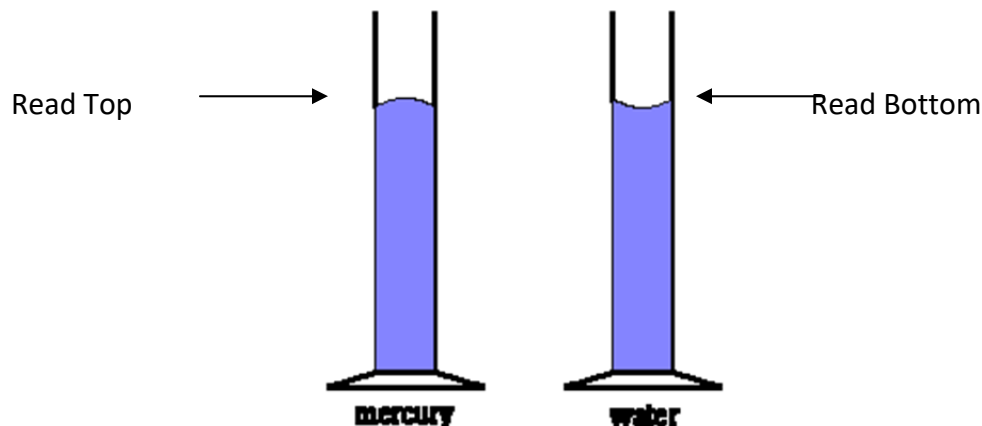
Composite (Proportional)	A collection of individual samples obtained at regular intervals, usually every one or two hours during a 24-hour time span. Each Samples individual sample is combined with the others in proportion to the flow when the sample was collected. The resulting mixture (composite sample) forms a representative sample and is analyzed to determine the average conditions during that sampling period.
Compound	A pure substance composed of two or more elements whose composition is constant. For example, table salt (sodium chloride, NaCl) is a compound.
Desiccator	A closed container that heated weighing or drying dishes are placed to cool in a dry environment. The dishes may be empty or they may contain a sample. Desiccators contain a substance, such as anhydrous calcium sulfate, which absorbs moisture and keeps the relative humidity near zero so that the dish or sample will not gain weight from absorbed moisture.
Distillate	In the distillation sample, a portion is evaporated; the part that is condensed afterwards is the distillate.
Element	A substance that cannot be separated into substances of other kinds by ordinary chemical means. For example, sodium (Na) is an element.
End Point	Samples are titrated to the end point. This means that a chemical is added, drop-by-drop, to a sample until a certain color change (blue to clear, for example) occurs that is called the end point of the titration. In addition to a color change, an end point may be reached by the formation of a precipitate or the reaching of a specified pH. An end point may be detected by the use of an electronic device such as a pH meter.
Flame Polished	Melted by a flame to smooth out irregularities. Sharp or broken edges of glass (such as the end of a glass tube) are rotated in a flame until the edge melts slightly and becomes smooth.
Grab Sample	A single sample of water or wastewater taken at neither a set time nor flow.
Gravimetric	A means of measuring unknown concentrations of water quality indicators in a sample by weighing a precipitate or residue of the sample.

Indicator (Chemical) A substance that gives a visible change, usually of color, at a desired point in a chemical reaction, generally at a specified end point.

M or Molar A molar solution consists of one gram molecular weight of a compound dissolved in enough water to make one liter of solution. A gram molecular weight is the molecular weight of a compound in grams. For example, the molecular weight of sulfuric acid (H_2SO_4) is 98. A 1 M solution of sulfuric acid would consist of 98 grams of H_2SO_4 dissolved in enough distilled water to make one liter of solution.

MPN Most Probable Number of coliform group organisms per unit volume. Expressed at a density of population of organisms per 100 mL.

Meniscus The curved top of a column of liquid (water, oil, mercury) in a small tube. When the liquid wets the sides of the container (as with water), the curve forms a valley. When the confining sides are not wetted (as with mercury), the curve forms a hill or upward bulge.



Molecular Weight The molecular weight of a compound in grams is the sum of the atomic weights of the elements in the compound. The molecular weight of sulfuric acid (H_2SO_4) in grams is 98.

ELEMENT	ATOMIC WEIGHT	NUMBER OF ATOMS	MOLECULAR WEIGHT
H	1	2	2
S	32	1	32
O	16	4	64
Total Weight			98

Molecule	The smallest portion of an element or compound that still retains or exhibits all the properties of the substance.
N or Normal	A normal solution contains one gram equivalent weight of reactant (compound) per liter of solution. The equivalent weight of an acid is that weight that contains one-gram atom of ionizable hydrogen or its chemical equivalent. For example, the equivalent weight of sulfuric acid (H_2SO_4) is 49 (98 divided by 2 because there are two replaceable hydrogen ions). A 1 <i>N</i> solution of sulfuric acid would consist of 49 grams of H_2SO_4 dissolved in enough water to make one liter of solution.
Oxidation	The addition of oxygen, removal of hydrogen, or the removal of electrons from an element or compound. The opposite of reduction.
Percent Strength	The amount of a substance that is dissolved in a solution compared with the amount that could be dissolved in the solution, expressed as a percent.
pH	An expression of the intensity of the alkaline or acid condition of a water. Mathematically, pH is the logarithm (base 10) of the reciprocal of the hydrogen ion concentration. The pH may range from 0-14, where 0 is most acidic, 14 most alkaline and 7 is neutral. Natural waters usually have a pH between 6.5 and 8.5.
Precipitate	To separate (a substance) out in a solid form from a solution, as by the use of a reagent.
Reagent	A substance that takes part in a chemical reaction and is used to measure, detect or examine other substances.
Reduction	The addition of hydrogen, removal of oxygen, or the addition of electrons to an element or compound. The opposite of oxidation.
Representative Sample	A portion of material or water identical in content to that in the larger body of material or water being sampled.
Solution	A liquid mixture of dissolved substances. In a solution, it is impossible to see all the separate parts.
Standard Solution	A solution that the exact concentration of a chemical or compound is known.

Titrate	A chemical solution of known strength is added on a drop by drop basis until a color change, precipitate or pH change in the sample is observed (end point). This is the process of adding the chemical solution to completion of the reaction as signaled by the end point.
Turbidity Units	Expressed in “Nephelometric Turbidity Units” (NTU) when measured by a nephelometric (reflected light) instrument.
Volumetric	A means of measuring unknown concentrations of water quality indicators in a sample by determining the volume of titrant or liquid reagent needed to complete particular reactions.

Section 3

Metric System

Metric System

Introduction to Laboratory Methods for Operators



{ 1 }

Is the English System Easier?

- 12 inches = 1 foot
- 3 feet = 1 yard
- 5280 feet = 1 mile
- 2 pints = 1 quart
- 4 quarts = 1 gallon
- 16 ounces = 1 pound
- 32 fluid ounces = 1 quart

- A foot determined by the size of a person's foot, there wasn't a standard
- Confusing numbers, nothing repeats

{ 2 }

History

- By the eighteenth century, dozens of different units of measurement were commonly used throughout the world
- Length, for example, could be measured in feet, inches, miles, spans, cubits, hands, furlongs, palms, rods, chains, leagues, and more
- The lack of common standards led to a lot of confusion and significant inefficiencies in trade between countries

{ 3 }

History

- At the end of the century, the French government sought to alleviate this problem by devising a system of measurement that could be used throughout the world
- In 1790, the French National Assembly commissioned the Academy of Science to design a simple decimal-based system of units; the system they devised is known as the metric system

{ 4 }

History

- In 1960, the metric system was officially named the *Système International d'Unités* (or SI for short) and is now used in nearly every country in the world except the United States
- The metric system is almost always used in scientific measurement

{ 5 }

Metric System Simplicity

- There is only one unit of measurement for each type of quantity measured
 - Length
 - Mass (weight)
 - Volume
 - Concentration
 - Temperature

{ 6 }

The Metric System

- The metric system is founded on base units.
 - The base unit of mass is the **gram**.
 - The base unit of length is the **meter**.
 - The base unit of volume is the **Liter**.
- To go from small to large quantities the base units are described by prefixes which represent a power of ten.

TDEC - Fleming Training Center 7

Metric System Simplicity

- The meter is a unit of length equal to 3.28 feet
- The gram is a unit of mass equal to approximately 0.0022 pounds
- The liter is a unit of volume equal to 1.05 quarts.
- Volume is always measured in liters, whether you are measuring how much water you need for a chlorine test or how much water is in your clarifier or sedimentation basin.

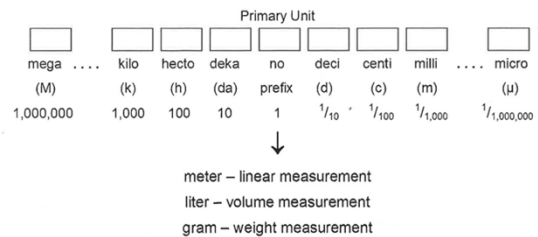
{ 8 }

Metric System

- Based on the decimal system
- All units of length, volume, and weight use factors of 10
- To express smaller amounts, prefixes are added to the names of the metric units
 - Milli- (1/1000th of or 0.001 times)
 - Centi- (1/100th of or 0.01 times)

{ 9 }

- All units can be converted into smaller or larger units by moving a decimal point



{ 10 }

Conversions

- Convert 1 meter to decimeters (dm)

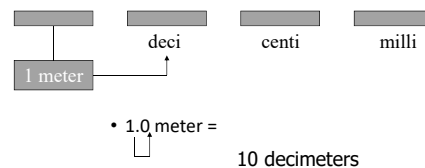


- Converting from meters to decimeters requires moving one place to the right, therefore, move the decimal point from its present position one place to the right as well.

{ 11 }

Conversions

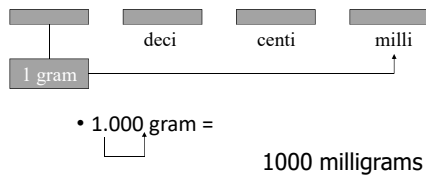
- Convert 1 meter to decimeters (dm)



{ 12 }

Conversions

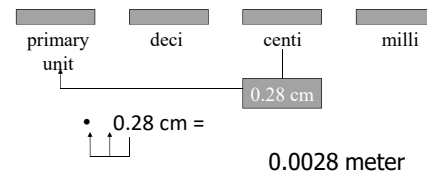
- Convert 1 gram to milligrams (mg)



[13]

Conversions

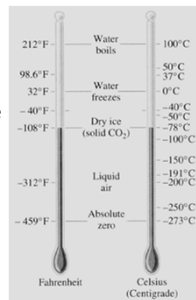
- Convert 0.28 cm to meters



[14]

Temperature – Fahrenheit

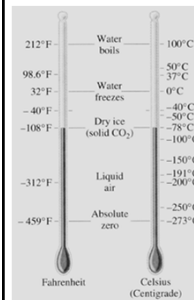
- The **Fahrenheit** scale is named for the 18th-century German physicist Daniel Fahrenheit.
- His scale is based on 32 for the freezing point of water and 212 for the boiling point of water
- The scale was in common use in English speaking countries until the 1970's when Europe and Canada adopted the centigrade (Celsius) scale.
- The U.S is the only country that still uses the Fahrenheit scale.



[15]

Temperature - Celsius

- The **Celsius** temperature scale is named for the Swedish astronomer Anders Celsius who invented the scale in 1742.
- The scale is based on 0 for the freezing point of water and 100 for the boiling point of water.
- It is sometimes called the **centigrade** scale because of the 100-degree interval between the defined points.



[16]

Temperature

- To convert Celsius ($^{\circ}\text{C}$) into Fahrenheit ($^{\circ}\text{F}$):

$$^{\circ}\text{F} = (^{\circ}\text{C})(1.8) + 32$$
- To convert Fahrenheit ($^{\circ}\text{F}$) into Celsius ($^{\circ}\text{C}$):

$$^{\circ}\text{C} = (0.556)(^{\circ}\text{F} - 32)$$

or

$$^{\circ}\text{C} = \frac{(^{\circ}\text{F} - 32)}{1.8}$$

[17]

Temperature Conversions

- You just won free tickets for an all-inclusive paid trip to Scotland! You are planning your wardrobe based on the weather forecast that predicts the temperature to be 21°C all week. Should you pack your wool sweaters or your t-shirts?

- $^{\circ}\text{F} = (^{\circ}\text{C})(1.8) + 32$
- $^{\circ}\text{F} = (21^{\circ}\text{C})(1.8) + 32$
- $^{\circ}\text{F} = 37.8 + 32$
- $^{\circ}\text{F} = 69.8$



[18]

Temperature Conversions

- You are recording your BOD incubator temperature for the day. Someone replaced your Celsius thermometer with a Fahrenheit thermometer. The temperature reading is 68 degrees F. What is the temperature in Celsius?

- $^{\circ}\text{C} = (0.556)(^{\circ}\text{F} - 32)$
- $^{\circ}\text{C} = (0.556)(68 - 32)$
- $^{\circ}\text{C} = (0.556)(36)$
- $^{\circ}\text{C} = 20.016$



[19]

Now we'll work some problems...

[20]

Basic Lab for Water and Wastewater - Metric Conversions

1. 1 m = _____ cm
 2. 1 g = _____ mg
 3. 1 kg = _____ g
 4. 1 cm = _____ mm
 5. 10 cm = _____ mm
 6. 50 cm = _____ mm
 7. 8 km = _____ m
 8. 19 km = _____ m
 9. 29 L = _____ mL
 10. 83 m = _____ mm
 11. 1.8 cm = _____ mm
 12. 2.5 mg = _____ g
 13. 2.6 km = _____ m
 14. 8.5 km = _____ m
 15. 80 mL = _____ L
 16. 150 mm = _____ cm
 17. 5000 m = _____ km
 18. 1300 g = _____ kg
 19. 17 mm = _____ cm
 20. 125 mm = _____ cm
 21. 170 L = _____ mL
 22. 155 m = _____ km
23. A particular pipe is delivered in sections 5 meters long. How many sections are required to span a distance of 1 kilometer?
24. You need to measure 34.6 milligrams of a chemical to make a solution. If the display on the scale only shows grams, what will the reading be?

25. During your last visit to the doctor, the nurse told you that you weighed 98 kilograms. Assuming that a nickel weighs approximately 5 grams, how many nickels would it take to equal your weight? If that were true, then how much is your weight worth in nickels?
26. Your favorite coffee mug at work holds about $\frac{1}{2}$ a liter. If you average about 8 milliliters each time you take a sip, how many sips does it take to get to the bottom of your mug?

Answers:

- | | | |
|--------------|---------------|---------------------------|
| 1. 100 cm | 10. 83,000 mm | 19. 1.7 cm |
| 2. 1000 mg | 11. 18 mm | 20. 12.5 cm |
| 3. 1000 g | 12. 0.0025 g | 21. 170,000 mL |
| 4. 10 mm | 13. 2600 m | 22. 0.155 km |
| 5. 100 mm | 14. 8500 m | 23. 200 sections |
| 6. 500 mm | 15. 0.08 L | 24. 0.0346 g |
| 7. 8000 m | 16. 15 cm | 25. 19,600 nickels, \$980 |
| 8. 19,000 m | 17. 5 km | 26. 62.5 sips |
| 9. 29,000 mL | 18. 1.3 kg | |

Basic Lab for Water and Wastewater – Temperature Conversions

1. Convert 60 degrees Fahrenheit to degrees Celsius.
2. What is 16°C expressed in terms of degrees Fahrenheit?
3. Convert 85°F into °C.
4. What is 29 degrees Celsius when converted to Fahrenheit?
5. The influent to a treatment plant has a temperature of 70°F. What is this temperature expressed in terms of Celsius?
6. The effluent of a treatment plant is 24°C. What is this temperature expressed in degrees Fahrenheit?
7. Your Canadian friend is coming to visit you for New Year's Day and they want to know what the average temperature is in Tennessee on January first. You look it up and tell them the average temp in January is 37.7°F. They immediately ask, "What is it Celsius?"

Answers:

- | | |
|--------------------|-----------|
| 1. 15.56°C or 16°C | 5. 21°C |
| 2. 60.8°F or 61°F | 6. 75°F |
| 3. 29.44°C or 29°C | 7. 3.17°C |
| 4. 84.2°F or 84°F | |

CONVERSION FACTORS AND USEFUL INFORMATION
 International Metric System - Le Systeme International d'Unites (SI Units)
 Base Units of the International Metric System (SI)

<u>Quantity</u>	<u>Name of the Unit</u>	<u>Symbol</u>
Length	Meter	m
Mass	Kilogram	kg
Time	Second	s
Temperature	Kelvin	K
Electric Current	Ampere	A
Luminous Intensity	Candela	cd
Amount of Substance	Mole	mol

Recommended Decimal Multiples and Submultiples and the Corresponding Prefixes and Names

<u>Factor</u>	<u>Prefix</u>	<u>Symbol</u>	<u>Meaning</u>
10^{12}	tera	T	One trillion times
10^9	giga	G	One billion times
10^6	mega	M	One million times
10^3	kilo	K	One thousand times
10^2	hecto	H	One hundred times
10	deca	D	Ten times
10^{-1}	deci	d	One tenth of
10^{-2}	centi	c	One hundredth of
10^{-3}	milli	m	One thousandth of
10^{-6}	micro	u	One millionth of
10^{-9}	nano	n	One billionth of
10^{-12}	pico	p	One trillionth of
10^{-15}	femto	f	One quadrillionth of
10^{-18}	atto	a	One quintillionth of

LENGTH	=	One kilometer	=	1,000 meters
		One meter (m)	=	1.0 meter
		One decimeter (dm)	=	0.1 meter
		One centimeter (cm)	=	0.01 meter
		One millimeter (mm)	=	0.001 meter

WEIGHT	=	One kilogram	=	1,000 grams
		One gram (g)	=	1.0 gram
		One decigram (dg)	=	0.1 gram
		One centigram (cg)	=	0.01 gram
		One milligram	=	0.001 gram

VOLUME	=	One kiloliter	=	1,000 liters
		One liter (L)	=	1.0 liter
		One deciliter (dL)	=	0.1 liter
		One centiliter (cL)	=	0.01 liter
		One milliliter (mL)	=	0.001 liter

AREA = One sq. kilometer (Km²) = 1,000 square meters

LENGTH CONVERSION FACTORS

1 inch (in)	=	2.54 centimeters	=	25.4 millimeters
1 foot (ft.)	=	12 inches	=	0.305 meters
1 yards(yd.)	=	3 feet	=	0.914 meters
1 mile (mi.)	=	5,280 feet	=	1,760 yards
1 meter (m.)	=	39.37 inches	=	3.261 feet
1 kilometer	=	0.62 miles	=	1,000 meters

1 square foot (ft. ²)	=	144 square inches (inch ²)
1 square yard (yd. ²)	=	9 square feet (ft. ²)
1 acre	=	43,560 square feet (ft. ²)
1 square mile (mi. ²)	=	640 acres or 1 section
1 square meter (m. ²)	=	10.8 square feet (ft. ²)
1 square meter (m. ²)	=	10,000 square centimeters
1 hectare	=	2.5 acres

VOLUME CONVERSION FACTORS

1 cubic foot (ft. ³)	=	1,728 cubic inches (inch ³)
1 cubic foot (ft. ³)	=	7.48 gallons
1 cubic yard (yd. ³)	=	27 cubic feet (ft. ³)
1 acre foot	=	43,560 cubic feet (ft. ³)
1 acre foot	=	325,851 gallons
1 gallon (gal.)	=	231 cubic inches (inch ³)
1 gallon (gal.)	=	4 quarts
1 cubic meter (m. ³)	=	35.3 cubic feet (ft. ³)
1 cubic meter (m. ³)	=	1.3 cubic yards (yd. ³)
1 liter	=	1.06 quarts
1 liter	=	1,000 milliliters

WEIGHT CONVERSION FACTORS

1 gallon	=	8.34 pounds (lbs.) of water
1 cubic foot	=	62.4 pounds (lbs.) of water
1 foot of water	=	0.434 PSI (pounds per square inch)
1 pound (lb)	=	0.454 kilograms (Kgs.)
1 kilogram (Kg)	=	2.2 pounds (lbs.)
1 kilogram (Kg)	=	1,000 grams
1 PSI	=	2.31 feet of water

TEMPERATURE

Section 3

TDEC Fleming Training Center

1. Convert Fahrenheit to Celsius $^{\circ}\text{C} = \frac{5(^{\circ}\text{F} - 32)}{9}$

$$0^{\circ}\text{F} = -17.8^{\circ}\text{C}$$

2. Convert Celsius to Fahrenheit $^{\circ}\text{F} = \frac{^{\circ}\text{C} \times 9}{5} + 32$

$$0^{\circ}\text{C} = 32^{\circ}\text{F}$$

$$100^{\circ}\text{C} = 212^{\circ}\text{F}$$

Remember: 100° between Ice/Steam = Celsius

180° between Ice/Steam = Fahr.

1. Convert Fahrenheit to Celsius:

$$^{\circ}\text{C} = (^{\circ}\text{F} + 40) \times \frac{5}{9} - 40$$

2. Convert Celsius to Fahrenheit:

$$^{\circ}\text{F} = (^{\circ}\text{C} + 40) \times \frac{9}{5} - 40$$

Quick Approximation:

$$(^{\circ}\text{C} \times 2) + 30 = ^{\circ}\text{F} \text{ (about)}$$

CONVERSION FACTORS FOR OPERATORS

TDEC Fleming Training Center

Section 3

<u>MULTIPLY</u>	<u>BY</u>	<u>TO OBTAIN</u>
Acres	43,560	Square feet
Acre-feet	43,560	Cubic feet
Acre-feet	325,851	Gallons
Centimeters	0.3937	Inches
Cubic feet	1728	Cubic inches
Cubic feet	7.48052	Gallons
Cubic feet	28.32	Liters
Cubic feet/second	448.831	Gal./min
Cubic feet/second	0.646317	Million gal/day
Cubic yards	27	Cubic feet
Degrees (angle)	60	Minutes
Feet	30.48	Centimeters
Feet	12	Inches
Feet	0.3048	Meters
Feet	1/3	Yards
Feet of water	0.4335	lbs/square in.
Gallons	0.1337	Cubic feet
Gallons	3.785	Liters
Gallons	4	Quarts (liquid)
Gallons, Imperial	1.20095	U.S. gallons
Gallons U.S.	0.83267	Imperial gallons
Gallons water	8.3453	Pounds of water
Gallons/min.	2.228×10^{-3}	Cubic ft/sec.
Gallons/min.	8.0208/area (sq.ft.)	Overflow rate (ft/hr)
Grains/U.S. gal.	17.118	Milligrams/liter
Grains/U.S. gal.	142.86	lbs/million gal.
Grams	0.3527	Ounces
Grams	2.205×10^{-3}	Pounds
Grams/liter	58.417	Grains/gal.
Grams/liter	1000	Milligrams/liter
Horse-power	33,000	foot-lbs./min.
Horse-power	0.7457	Kilowatts
Horse-power	745.7	Watts
Inches	2.540	Centimeters
Inches of mercury	1.133	Feet of water
Inches of mercury	0.4912	lbs/sq. inch
Inches of water	0.07355	In. of mercury
Inches of water	0.03613	lbs/sq. inch
Kilowatt-hours	1.341	Horse-power hrs.

<u>MULTIPLY</u>	<u>BY</u>	TDEC Fleming Training Center <u>TO OBTAIN</u>
Liters	0.03531	Cubic feet
Liters	0.2642	Gallons
Liters	1.057	Quarts (liquid)
<u>Width(in) x Thickness(in)</u> 12	Length (ft.)	Board feet
Meters	3.281	Feet
Meters	39.37	Inches
Miles	5280	Feet
Miles	1760	Yards
Milligrams/liter	1	Parts/million
Million gals./day	1.54723	Cubic ft/sec
Ounces	0.0625	Pounds
Ounces	28.349527	Grams
Overflow rate (ft/hr)	0.12468 x area (sq.ft.)	Gal/min
Milligrams/liter	0.0584	Grains U.S. gal.
Milligrams/liter	8.345	lbs/million gal.
Pounds	16	Ounces
Pounds	7000	Grains
Pounds	453.5924	Grams
Pounds of water	0.01602	Cubic feet
Pounds of water	0.1198	Gallons
Pounds/sq. inch	2.307	Feet of water
Pounds/sq. inch	2.036	In. of mercury
Revolutions	360	Degrees
Square feet	2.29 x 10 ⁻⁵	Acres
Square feet	144	Square inches
Square feet	1/9	Square yards
Square inches	6.452	Square centimeters
Square meters	10.76	Square feet
Square miles	640	Acres
Square yards	9	Square feet
Temp. EC + 17.78	1.8	Temp. EF
Temp. EF - 32	5/9	Temp. EC
Watts	1.34 x 10 ⁻³	Horse-power
Yards	3	Feet
Yards	36	Inches
Yards	0.9144	Meters

Section 4

Weight and Volume

Measurement

Balances and Pipetting

WEIGHT MEASUREMENT FOR GRAVIMETRIC ANALYSIS

Introduction to Laboratory Methods for
Operators

WEIGHT MEASUREMENT

- One of the oldest procedures in all of science, and a skill that you'll use throughout your entire career.
- Common lab practices include:
 - Weighing chemicals for standard and reagent preparation
 - Solids analysis (Ex: TSS, MLSS, TDS)
- Gravimetric = A means of measuring unknown concentrations of water quality indicators in a sample by weighing a precipitate or residue of the sample

WEIGHT MEASUREMENT

- Involved in almost every analysis in Water or Wastewater
- Accurate measurements of weight are important
- Must determine accuracy of scales needed for task at hand
- Most common units are grams or grains



TYPES OF SCALES

1. Triple-Beam Balance
2. Top Loading Balance
3. Analytical Balances

TRIPLE-BEAM BALANCE

- Three Beams, each containing a different reference weight
- The user manually weighs objects by adjusting the reference weights
- Weighs to the nearest 0.1 grams
- Weighs objects up to 600 grams



TOP LOADING BALANCES

- Has a digital readout for quick readings
- May be tared, or calibrated to zero after a container is placed on the pan
- Weighs to the nearest 0.01 gram



ANALYTICAL BALANCES

- Most accurate, requires most attention to detail
- Two types: Digital and Single Pan
- Weighs objects up to 160 grams
- Weighs to nearest 0.0001 grams



USING LABORATORY BALANCES

- Proper balance location
- Leveling the balance
- Zeroing the balance
- Placing objects on the balance
- Calibration, Sensitivity Checks, and Service
- Manipulation of Weights
- Weighing Chemicals
- Heavy weights or objects

PROPER BALANCE LOCATION



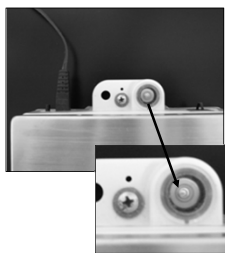
- Locate on a solid, level surface
- Never on a metal surface
- A solid marble table on a concrete floor is ideal
- Room should have a constant temperature, relative humidity
- Locate away from sunlight, drafts, moisture, and pedestrian traffic

THE TABLE

- Solid built, preferably made of stone
- Avoid causing the tabletop to sag or move
 - Don't prop your arm on it
- Vibration-free environment
 - No machines or engines that vibrate near it
- Put the table in the corner of a room
 - At least near a wall, there is less vibration this way
- Avoid direct sunlight or heat from heaters
- Place away from drafts, including air conditioner currents



LEVELING THE BALANCE



- Before use, level balance
- Most balances have a liquid bubble level indicator
- Ensure that bubble is located in the center of the center of the etched circle
- Adjust by using leveling screws on side of balance

ZEROING THE BALANCE

- Balances must be zeroed before use
- Triple-beam: The pointer should be on the zero point.
- Digital balances have a button labeled "Zero" that is pressed



PLACING OBJECTS ON THE BALANCE

- Place items carefully on the balance
- Do not drop items on balance
- Always use a weighing dish or pan
- Dish or pans prevent spillage on the balance



CALIBRATION, CHECKS, AND SERVICE

- Should be calibrated and inspected annually
- Some balances must be checked monthly
- Checks are for determining drift in measurement
- Use NIST Class S-1 or ANSI Class 1 Weights that bracket your needs
 - Daily before use
 - At least 2 weights
 - Calibrated every 5 years



MANIPULATION OF WEIGHTS

- Calibration weights should never be touched
- Moisture and oils can change the measured value of the weights
- Use forceps or tongs



WEIGHING CHEMICALS

- Weigh chemical and corrosive materials in appropriate containers, never on balance pan
- Spilled chemicals should be removed and the balance cleaned immediately
- Clean balance pan after use with camel's hair brush
- **When measuring chemicals, never put excess back into original container**

HEAVY WEIGHTS AND OBJECTS

- Never overload balance
- Check maximum rated capacity prior to use
- Place item to be weighed in the center of balance pan

SOURCES OF ERROR

- Moisture
- Temperature
- Static Charge
- Air



MOISTURE

- Measuring dried solids: all samples must be treated exactly the same to ensure the that samples have the same extent of drying
- Hygroscopic chemicals will absorb water if conditions are not controlled
- Porcelain crucibles will retain moisture

TEMPERATURE

- Temp of chemical must be the same as the balance
- Materials that are too warm will cause convection currents that will push pan upward causing item to weigh less
- Materials that are too cold will cause convection currents that will push down causing item to weigh more

STATIC CHARGE

- A static charge can be established when a crucible is wiped with a dry cloth
- If crucible touches balance, a transfer of charge will occur
- May take as long as half an hour to dissipate on dry day or shorter on a humid day

AIR

- Air exerts a buoyant effect on objects just as water does
- Not normally taken into account
- Negligible for most labs
- For extreme measurement or calibration of volumetric glassware

WEIGHING SAMPLES

- External environmental factors and improper handling can lead to inaccurate results
1. Choose a stable table in a quiet place
 2. Work in a climate controlled lab
 3. Ensure the balance is leveled and calibrated
 4. Use proper technique

WEIGHING SAMPLES – TABLE

- Solid built, preferably made of stone
- Avoid causing the tabletop to sag or move
 - Don't prop your arm on it
- Vibration-free environment
 - No machines or engines that vibrate near it
- Put the table in the corner of a room
 - At least near a wall, there is less vibration this way
- Avoid direct sunlight or heat from heaters
- Avoid drafts, including air conditioner currents

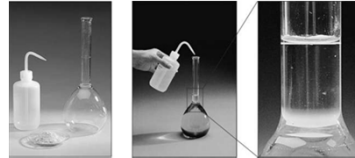
ANY QUESTIONS?

VOLUMETRIC ANALYSIS

- 1 Introduction to Laboratory Methods for Operators

VOLUMETRIC ANALYSIS

- The measurement of liquid
- Typical lab tech duties:
 - Measuring volumes
 - Transferring liquids
 - Preparing standard solutions
 - Analysis by titration and spectrophotometry



2

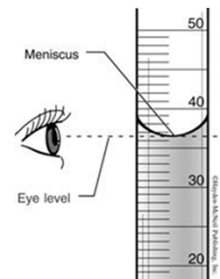
MEASUREMENT GLASSWARE

- All glassware designed for volumetric measurement is labeled either:
 1. TD = "To Deliver"
 2. TC = "To Contain"
- Glassware used "to deliver" consists of
 1. Graduated Cylinders
 2. Pipets
 3. Burets

3

THE MENISCUS

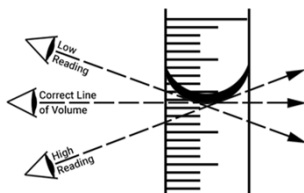
- There is a chemical attraction between water and its container
- Water tends to adhere to the sides of a container and "climb" the sides
- This climbing liquid gives a curved and distorted surface called the meniscus
- READ THE MENISCUS AT EYE LEVEL



4

THE MENISCUS

- The water level is measured at the bottom of the meniscus
 - "the belly"
- Parallax error will occur if you don't read at eye level



5

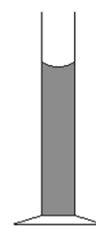
WATER VS. MERCURY

Read Top of Meniscus



Mercury

Read Bottom of Meniscus



Water

6

VOLUMETRIC GLASSWARE

- Used when accurate measurements are critical
- Include:
 - Flasks
 - Pipets
 - Burettes



7

HOW TO USE A VOLUMETRIC FLASK

8

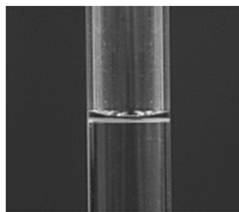
USE OF VOLUMETRIC FLASK



- Step 1
 - Begin with flask filled about $\frac{1}{3}$ way with distilled water
- Step 2
 - Dispense proper amount of liquid reagent or solids into the flask
 - If using solids, first rinse container well with distilled water to remove all solids

9

USE OF VOLUMETRIC FLASK



- Step 3
 - Add distilled water up to the neck of the flask and carefully fill it to the mark using a squeeze bottle or eye dropper

10

USE OF VOLUMETRIC FLASK



- Step 4
 - Stopper the flask and invert 26 times to mix
 - When inverting, allow the air bubble to go all the way to the top of the flask each time

11

HOW TO PIPET

The second type of glassware used "to deliver" various volumes of liquid.

12

HOW TO PIPET

- Filling any pipet requires suction
- A bulb is used to draw liquid up into the pipet
- Pipet bulbs are safety devices and eliminate contact between mouth and pipet
- NEVER PIPET BY MOUTH
- If liquid is pulled into the bulb, consider it contaminated
 - Clean the bulb promptly to avoid corroding the inside or spreading contamination to next pipet



13

TYPES OF PIPET BULBS



USE OF PIPET

- Step 1
 - If using a pipet bulb, start by squeezing the bulb in your preferred hand
 - Then place the bulb on the flat end of the pipet



15

USE OF PIPET

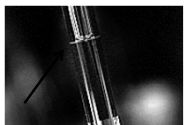
- Step 2
 - Place the tip of the pipet in the solution and release your grip on the bulb to pull solution into the pipet.
 - Draw solution in above the mark on the neck of the pipet.



16

USE OF PIPET

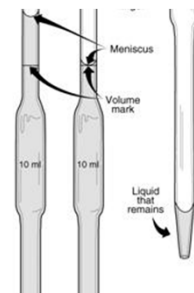
- Step 3
 - Quickly, remove the pipet bulb and put your index finger on the end of the pipet
 - Gently release the seal made by your finger until the level of the solution meniscus exactly lines up with the mark on the pipet



17

USE OF PIPET

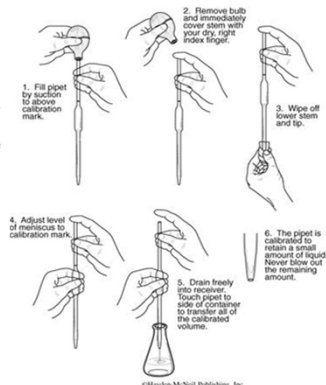
- Step 4
 - Touch the tip of the pipet to the wall of the receiving vessel to drain the last bit of liquid
 - Volumetric pipets are calibrated to have a drop of liquid remaining in the tip
 - DO NOT shake or blow out this drop of liquid



18

HOW TO PIPET

- Chipped and cracked pipets should be replaced as they are unsafe and may affect the accuracy of measurements.
- Hold the pipet by the upper third of the tube and keep the tip from touching anything



PIPETTING TIPS

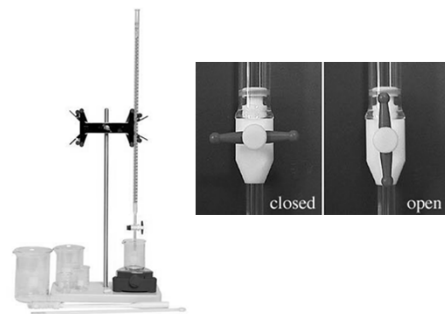
- Slow down and take your time
- Use the pipet that dispenses the closest volume to what you need
- Hold the pipet in a vertical position
- Wet pipette tips work best
 - Pull some liquid into it and then dispense before you get started
- Do not aspirate so quickly that bubbles form in the solution
 - The bubbles cause errors in volume measurement

20

HOW TO USE A BURET

21

HOW TO USE A BURET



22

HOW TO USE A BURET

Filling the Buret:



- Close the stopcock
- Pour ~ 10 mL of solution into buret
- Drain solution into waste beaker
- Fill the buret above the zero line
- Check for bubbles
 - Gently tap the sides to release bubbles

23

HOW TO USE A BURET

- Drain excess liquid through stopcock to remove any air bubbles in tip and adjust the meniscus to zero
 - A bubble in the tip is an error
- Adjust the meniscus to zero
 - At zero, the buret is saying "0 mL of the solution has been delivered."



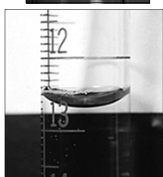
24

USE OF BURET



Begin analysis:

7. Place beaker with sample beneath buret and slowly open stopcock to allow 1 drop of liquid at a time
8. Perform titration and turn stopcock into closed position when endpoint is reached
9. Remove any drops clinging to the tip by touching to the wall of a waste beaker
10. Record final buret reading



25

HOW TO USE A BURET

- To calculate volume delivered, subtract the initial reading from the final reading
- Make sure to read at eye level to avoid parallax errors
- Wash buret immediately after use
 - Never store bases in buret
 - Bases will cause glass stopcocks to freeze and can etch the glass walls



26

**NOW IT'S TIME TO PRACTICE
YOUR SKILLS...**

27

Weight and Volume Measurement – Review Questions

1. Which type of scale (or balance) is the most accurate?
2. Why should you always use a weighing dish or pan when using any type of balance?
3. How often should your balance be calibrated and inspected?
4. You must document that you have verified the balance each day that you use it with weights that will bracket your anticipated sample weight. What type of weight is required? How often should they be calibrated?
5. Why is it important to never touch the weights that are used to check/verify your balance?
6. If you accidentally pour out too much when measuring chemicals, you are allowed to put the excess back into the original container. True or False
7. Which type of glassware is used when accurate measurements are critical (for example, when preparing standards)?
8. Which of the following statements regarding the use of volumetric flasks is not correct or incomplete?
 - a. Pour reagent directly into the flask
 - b. Add distilled or DI water up to neck of the flask and carefully fill to the mark
 - c. Stopper and invert to mix
 - d. When inverting, allow the air bubble to go all the way to the top each time to ensure a complete mix

9. Write out the basic steps to pipet a liquid.

1)

2)

3)

4)

10. When conducting a titration, the stopcock should be opened enough to allow for a steady flow of liquid until the pH endpoint has been reached. True or False

Section 5

Introduction to Basic Chemistry

Intro to Basic Chemistry & Solutions Chemistry

Introduction to Laboratory Methods for Operators

Basic Chemistry

- ▶ Matter = anything that has mass (weight) and occupies space
- ▶ Matter consists of Elements or a combination of elements

Basic Chemistry

- ▶ **Atom** = the smallest particle that still retains the characteristics of an element
 - If you took an element and divided it into smaller and smaller pieces...
- ▶ Atoms can be further broken down into even smaller pieces called **subatomic particles**

Basic Chemistry

- ▶ There are 3 fundamental **subatomic particles**:
 1. **Proton (+)** } Nucleus
 2. **Neutron** } Nucleus
 3. **Electron (-)** } Shells
- ▶ **Nucleus** = center of atom
 - Made up of protons and neutrons
- ▶ **Shell** = space around the nucleus
 - Contains electrons

Basic Chemistry

- ▶ **Carbon Atom**
 - 6 protons
 - 6 neutrons

Shell →

Carbon
6
C
12.011

● - Electron

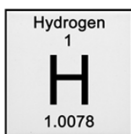
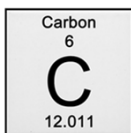
⊕ - Proton

○ - Neutron

Basic Chemistry

- ▶ **Atomic number** = the number of protons in the nucleus
 - This is the basic defining characteristic of the atoms of any one element
- ▶ **Atomic Mass (Weight)** = the sum of the number of protons and neutrons in the nucleus
 - Neutrons and protons have nearly identical weights and contain most of the mass of the atom

Atomic Weight

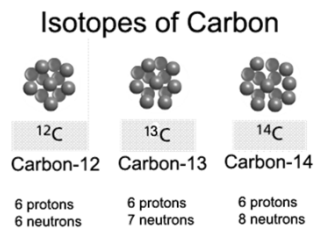


- The weight of Carbon is 12
- The weight of Hydrogen is 1
- Therefore, a carbon atom weighs 12 times more than a Hydrogen atom

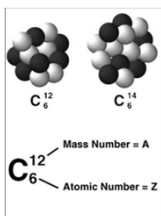
- Comparison

Isotopes

- ▶ Isotopes are atoms which have the same number of protons but different numbers of neutrons
- ▶ Isotopes will have the same atomic number but different mass numbers



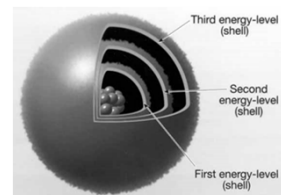
Isotopes of Carbon



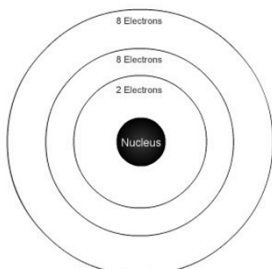
1. Carbon-12, which constitutes 99 % of all carbon atoms and serves as the standard for the atomic mass scale
 2. Carbon-14 is an isotope produced by cosmic rays bombarding the atmosphere and is radioactive, with a half-life of 5760 years
- ▶ The fact that they have varying numbers of neutrons makes no difference whatsoever to the chemical reactions of the carbon.

Electron Configuration

- ▶ Electrons are orbiting the nucleus just like the planets orbit the sun
- ▶ The orbits occur in a series of levels called energy levels
- ▶ Each energy level can only hold a certain number of electrons



Electron Energy Levels



Shell diagram of a neutral atom. The positively charged nucleus is surrounded by clouds of negatively charged electrons, arranged in successive shells. The first (inner) shell holds a maximum of 2 electrons; the second and third shells can hold 8 outer electrons each.

- The electrons will fill the lowest energy levels (closest to the nucleus) first
- The first level holds 2 electrons
- The second level holds 8 electrons
- The third level also holds 8 electrons

Basic Chemistry

- ▶ Valence Electrons
 - Valence electrons are located in the outermost shell of the atom
 - Since they are the electrons in the highest energy level, they are the most exposed of all the electrons, and consequently, they are the electrons that get involved in chemical reactions
 - The actual number of electrons that an atom gains or loses in bonding with one or more atoms is the **valence** of the atom
 - Gain or lose electrons

Periodic Table

KEY

Atomic Mass →	12.011		← Selected Oxidation States	-4
Symbol →	C			+2 +4
Atomic Number →	6			
Electron Configuration →	2-4			

Relative atomic masses are based on ¹²C = 12.000

Note: Mass numbers in parentheses are mass numbers of the most stable or common isotope.

TDEC - Fleming Training Center 1
3

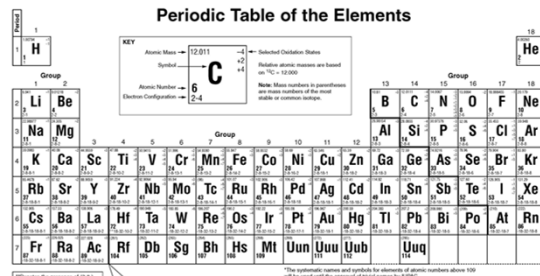
Periodic Table of the Elements

KEY

Atomic Mass →	12.011		← Selected Oxidation States	-4
Symbol →	C			+2 +4
Atomic Number →	6			
Electron Configuration →	2-4			

Relative atomic masses are based on ¹²C = 12.000

Note: Mass numbers in parentheses are mass numbers of the most stable or common isotope.



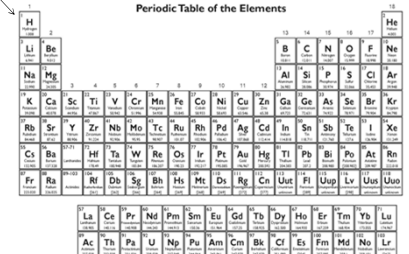
*Denotes the presence of (2+8) or elements (2) and above.

*The systematic names and symbols for elements of atomic numbers above 100 will be used until the approval of their names by IUPAC.

TDEC - Fleming Training Center 1
4

The Periodic Table

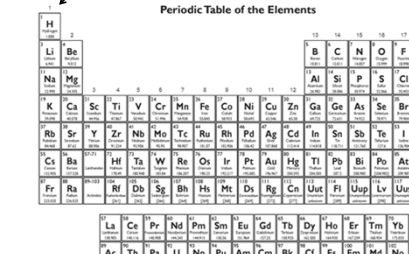
The **Period Number** is found at the beginning of a row. There are 7 periods. Atoms become larger in size as they move from left to right.



TDEC - Fleming Training Center

The Periodic Table

Group Number, found at the top of a column. Elements in the same group have similar reactivities and properties. There are 18 groups in the modern periodic table



TDEC - Fleming Training Center


Valence Electrons

- ▶ The reason that elements in groups have similar characteristics is because of their valence electrons
- ▶ Elements in the same group have the same number of valence electrons

		X		Valence electrons						Noble		
1 Ring →	H	X	X	X	X	X	X	X	X	X	X	← He
2 Rings →	Li	Be	B	C	N	O	F	X	X	X	X	← Ne
3 Rings →	Na	Mg	Al	Si	P	S	Cl	X	X	X	X	← Ar
4 Rings →	K	Ca	3	4	5	6	7	8				
			Valence electrons									

The Octet Rule

- ▶ The way that elements react with one another to form chemical bonds is based on their valence electrons.
- ▶ The noble gases have 8 electrons in their outer most energy level.
- ▶ Having 8 electrons in the outer energy level makes the atom happy, and non-reactive.
- ▶ Atom electrons are **lost, gained or shared** to form an octet



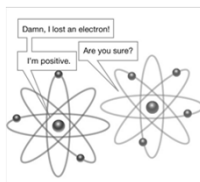
TDEC - Fleming Training Center

Chemical Bonds

There are two types of chemical bonds that we will discuss in this class.

1. **Ionic bonds** result from attraction between the opposite charges of an ion. One element in an ionic bond loses electrons, and another element must gain the electrons

2. **Covalent bonds** occur when the electrons are shared between the two atoms

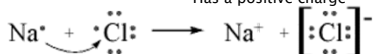


Ionic Bond

- ▶ Generally occur when a metal reacts with a non-metal
- ▶ One element in an ionic bond loses electrons, and another element must gain the electrons
- ▶ The atom that gains the electron becomes a negatively charged ion
- ▶ The atom that lost the electron becomes a positively charged ion

Ionic Bond

Sodium atom
Has 1 valence electron



Chlorine atom
Has 7 valence electrons

Sodium ion
Lost the electron
Has a positive charge

Chloride ion
Gained an electron
Now has a negative charge

The Octet Rule is satisfied.

Both the chlorine atom and the sodium atom now have eight electrons in their outermost energy level.

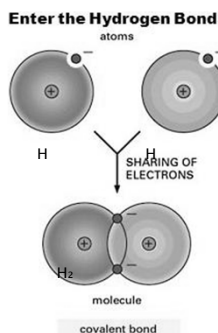
Covalent Bond

- ▶ As opposed to ionic bonding in which a complete transfer of electrons occurs, covalent bonding occurs when two (or more) elements share electrons
- ▶ Covalent bonding occurs because the atoms in the compound have a similar tendency to gain or lose electrons
- ▶ This most commonly occurs when two nonmetals bond together
- ▶ Because both of the nonmetals will want to gain electrons, the elements involved will share electrons in an effort to satisfy the octet rule

22

Covalent Bond

- ▶ Hydrogen (H) atoms have one valence electron in their first electron shell
- ▶ Since the capacity of this shell is two electrons, each hydrogen atom will "want" to pick up a second electron
- ▶ Then, by sharing, both atoms will have 2 electrons in their outermost energy level



23

Solutions

- A liquid mixture of dissolved substances
- A solution consist of two parts:
 - Solute
 - Solvent
- The solute part of the solution is dissolved in the solvent
- The most common solvent is water

TDEC - Fleming Training Center

2
4

Solutions

- Solutes added to water can be in solid, liquid, or gaseous form

Common solutes found in water treatment:

- Solids
 - Dry alum
 - Dry lime
 - Soda ash
- Liquids
 - Bleach
 - Sulfuric acid
- Gas
 - Chlorine gas
 - Carbon dioxide

TDEC - Fleming Training Center

2
7

Concentration

- The measure of a solution that describes the amount of solute in the solvent
- Listed below are expressions for concentration:
 - milligrams per liter (mg/L)
 - grains per gallon
 - percent strength
 - molarity (M)
 - normality (N)

TDEC - Fleming Training Center

2
6

Milligrams per Liter and Grains per Gallon

- These express weight per volume
- mg/L is the most commonly accepted measurement in water and wastewater industry
- 1 ppm (part per million) is equivalent to 1 mg/L

TDEC - Fleming Training Center

2
7

Percent Strength

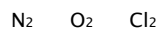
- Another way of expressing concentration
- % Strength = $\frac{\text{weight of solute}}{\text{weight of solution}} \times 100$
- Weight of Solution = Weight of solute + weight of solvent

TDEC - Fleming Training Center

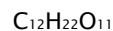
28

Compound

- Two or more different atoms “stuck” (chemically bonded) together
- When atoms of two or more elements are bonded together to form a compound, the resulting particle is called a **molecule**.



- Or a molecule may consist of several elements, with dozens of atoms bonded together



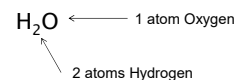
TDEC - Fleming Training Center

2
9

Chemical formulas

- Shorthand way of writing what elements are present in a molecule of a compound and how many atoms of each element are present in each molecule

H = Hydrogen
O = Oxygen

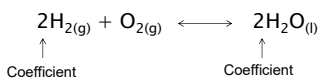


TDEC - Fleming Training Center

3
0

Chemical equations

- ▶ A shorthand way, using chemical formulas, to accurately represent what happens in a chemical reaction.
- ▶ The balanced chemical equation for water:



- ▶ The coefficients indicate the relative number of molecules (moles) that are involved in the reaction

TDEC - Fleming Training Center

3
1

Mole concept

- ▶ Individual atoms and molecules are very tiny. In order to count molecules, it is easier to use a quantity called a **mole**, abbreviated **mol**.
- ▶ A mole of anything contains 6.02×10^{23} atoms or molecules

TDEC - Fleming Training Center

3
2

Mole concept

- ▶ Ex: 1 dozen eggs weighs differently than 1 dozen donuts, but they have the same number of items.



- ▶ Ex: 1 mole of sugar weighs more than 1 mole of salt but they both contain 6.02×10^{23} molecules (because the sugar molecule is bigger.)

TDEC - Fleming Training Center

3
3

Molarity

- ▶ Perhaps the most accurate way of expressing the concentration of a solution
- ▶ Molarity = $\frac{\text{moles of solute}}{\text{liter of solution}}$

How much does 1 mole of water weigh?

- ▶ H₂O

	Number of Atoms	Atomic Weight	Total Weight
Hydrogen (H)	2	1 amu	2
Oxygen (O)	1	16 amu	16
Molecular Weight of H ₂ O			18 grams/mol

- ▶ 1 mol = 18 grams

TDEC - Fleming Training Center

3
5

Normality

- ▶ Another method for expressing the concentration of a solution
- ▶ Depends on Equivalent Weights
 - Think of "Equivalent" as "Equal Valence"
 - Equivalent weights level the playing field
- ▶ Normality = the number of equivalent weights of solute per liters of solution
- ▶ Normality = $\frac{\text{Equivalent weight}}{\text{Liters of solution}}$

Normality

- *Normal solutions* are solutions which have a specific number of equivalent masses of the acid or base dissolved in the solution per liter. A 1 N solution (a 1 normal solution) contains 1 equivalent mass per liter, a 2 N solution contains 2 equivalent masses per liter, and so on.
- Normality = $\frac{\text{number of equivalent weights of solute}}{\text{liters of solution}}$
- Number of equivalent weights = $\frac{\text{total weight of solute}}{\text{equivalent weight}}$
- Equivalent weight = $\frac{\text{molecular weight}}{\text{number of positive charges}}$

TDEC - Fleming Training Center

3
7

Standard Solutions

- ▶ A solution in which the exact concentration of a chemical or compound is known
- ▶ Used to determine the concentration in other solutions or to make dilutions of a solution
- ▶ You "standardize" by comparing with a standard
 - Set up an instrument or device to read a standard
 - This allows you to adjust the instrument so that it reads accurately, or enables you to apply a correction factor to the readings

TDEC - Fleming Training Center

3
8

Dilutions

- Weakening a stock solution
- Start with a stock solution, add an amount to the volumetric flask, and fill to mark
- Can use the following calculation with any expression of concentration or normality

Use this equation to make calculation:

$$C_1V_1 = C_2V_2$$

1 means stock or initial concentration or volume

2 is the concentration or volume you end up with

TDEC - Fleming Training Center

3
9

Dilution Examples

- Operator has 12% bleach on hand and wants to make 25 gallons of 8%.

$$C_1V_1 = C_2V_2$$

$$(0.12)(V_1) = (0.08)(25 \text{ gal})$$

$$V_1 = \frac{(0.08)(25 \text{ gal})}{0.12}$$

$$V_1 = 16.7 \text{ gals}$$

TDEC - Fleming Training Center

4
0

Dilution Examples

- Operator has 100 mL of 5N sulfuric acid on hand and wants to make 1N. How much water should be added?

$$C_1V_1 = C_2V_2$$

$$(5N)(100 \text{ mL}) = (1N)(V_2)$$

$$\frac{(5N)(100 \text{ mL})}{1N} = (V_2)$$

$$500 \text{ mL} = V_2$$

500 mL is final volume, you started with 100 mLs and added 400 mL to get a final volume of 500 mL

TDEC - Fleming Training Center

4
1

Periodic Table of the Elements

1 H Hydrogen 1.008																	2 He Helium 4.003
3 Li Lithium 6.941	4 Be Beryllium 9.012											5 B Boron 10.811	6 C Carbon 12.011	7 N Nitrogen 14.007	8 O Oxygen 15.999	9 F Fluorine 18.998	10 Ne Neon 20.180
11 Na Sodium 22.990	12 Mg Magnesium 24.305											13 Al Aluminum 26.982	14 Si Silicon 28.086	15 P Phosphorus 30.974	16 S Sulfur 32.066	17 Cl Chlorine 35.453	18 Ar Argon 39.948
19 K Potassium 39.098	20 Ca Calcium 40.078	21 Sc Scandium 44.956	22 Ti Titanium 47.867	23 V Vanadium 50.942	24 Cr Chromium 51.996	25 Mn Manganese 54.938	26 Fe Iron 55.845	27 Co Cobalt 58.933	28 Ni Nickel 58.693	29 Cu Copper 63.546	30 Zn Zinc 65.38	31 Ga Gallium 69.723	32 Ge Germanium 72.631	33 As Arsenic 74.922	34 Se Selenium 78.971	35 Br Bromine 79.904	36 Kr Krypton 84.798
37 Rb Rubidium 84.468	38 Sr Strontium 87.62	39 Y Yttrium 88.906	40 Zr Zirconium 91.224	41 Nb Niobium 92.906	42 Mo Molibdenum 95.95	43 Tc Technetium 98.907	44 Ru Ruthenium 101.07	45 Rh Rhodium 102.906	46 Pd Palladium 106.42	47 Ag Silver 107.868	48 Cd Cadmium 112.414	49 In Indium 114.818	50 Sn Tin 118.711	51 Sb Antimony 121.760	52 Te Tellurium 127.6	53 I Iodine 126.904	54 Xe Xenon 131.29
55 Cs Cesium 132.905	56 Ba Barium 137.328	57-71 Lanthanides	72 Hf Hafnium 178.49	73 Ta Tantalum 180.948	74 W Tungsten 183.84	75 Re Rhenium 186.207	76 Os Osmium 190.23	77 Ir Iridium 192.217	78 Pt Platinum 195.085	79 Au Gold 196.967	80 Hg Mercury 200.592	81 Tl Thallium 204.383	82 Pb Lead 207.2	83 Bi Bismuth 208.980	84 Po Polonium [208.982]	85 At Astatine 209.987	86 Rn Radon 222.018
87 Fr Francium 223.020	88 Ra Radium 226.025	89-103 Actinides	104 Rf Rutherfordium [261]	105 Db Dubnium [262]	106 Sg Seaborgium [266]	107 Bh Bohrium [264]	108 Hs Hassium [269]	109 Mt Meitnerium [268]	110 Ds Darmstadtium [269]	111 Rg Roentgenium [272]	112 Cn Copernicium [277]	113 Uut Ununtrium unknown	114 Fl Flerovium [289]	115 Uup Ununpentium unknown	116 Lv Livermorium [298]	117 Uus Ununseptium unknown	118 Uuo Ununoctium unknown

57 La Lanthanum 138.905	58 Ce Cerium 140.116	59 Pr Praseodymium 140.908	60 Nd Neodymium 144.243	61 Pm Promethium 144.913	62 Sm Samarium 150.36	63 Eu Europium 151.964	64 Gd Gadolinium 157.25	65 Tb Terbium 158.925	66 Dy Dysprosium 162.500	67 Ho Holmium 164.930	68 Er Erbium 167.259	69 Tm Thulium 168.934	70 Yb Ytterbium 173.055	71 Lu Lutetium 174.967
89 Ac Actinium 227.028	90 Th Thorium 232.038	91 Pa Protactinium 231.036	92 U Uranium 238.029	93 Np Neptunium 237.048	94 Pu Plutonium 244.064	95 Am Americium 243.061	96 Cm Curium 247.070	97 Bk Berkelium 247.070	98 Cf Californium 251.080	99 Es Einsteinium [254]	100 Fm Fermium 257.095	101 Md Mendelevium 258.1	102 No Nobelium 259.101	103 Lr Lawrencium [262]

Molarity

- Molarity is perhaps the most accurate way we have of expressing the concentration of a solution.
- A mole is the quantity of a compound that has a weight in grams equal to the compound's molecular weight.

1. How much does 1 mol of NaCl weigh?

Atomic weight of Na = 23

Atomic weight of Cl = 35.5

Molecular Weight of NaCl = 58.5 g/mol

2. How would you make a 1 molar (1M) solution of NaCl?

Step 1: Weigh out the solute, 1 mol = 58.5 g

Step 2: Pour solute into a 1 liter volumetric flask containing solvent

Step 3: Fill the flask to the liter mark. You now have a 1 M NaCl solution.

3. How would you make a 0.001 M NaCl solution?

Molecular weight of NaCl = 58.5 g/mol

0.001 M = 0.001 mol/L

$$\left(\frac{58.5\text{g}}{\text{mol}}\right) \left(\frac{0.001\text{ mol}}{\text{L}}\right) = \frac{0.0585\text{g}}{\text{L}} \text{ or } \frac{58.5\text{ mg}}{\text{L}}$$

Step 1: Weigh out solute (0.058 g on the analytical balance)

Step 2: Add the solute to the solvent. (Add the 58.5 mg to a volumetric flask that is 1/3 full of DI water)

Step 3: Fill the flask to the 1 Liter mark, paying close attention to the meniscus. This gives you a 0.001 M NaCl solution.

Normality

Normality is defined as the number of equivalent weights of solute per liter(s) of solution.

$$N = \frac{\text{Eq.Wt.}}{L}$$

1. If 2.1 equivalents of NaOH were used in making up 1.75L of solution, what is the normality of the solution?

$$N = \frac{\text{Eq.Wt.}}{L}$$

$$N = \frac{2.1 \text{ Eq.Wt.}}{1.75 \text{ L}}$$

$$N = 1.2 \text{ N NaOH}$$

2. How would you make a 1.2 N NaOH Solution?

Step 1: Determine the MW of NaOH.

$$\text{Molecular Weight} = 40 \text{ g/mol}$$

Step 2: Determine equivalent weight.

$$\frac{40 \text{ g}}{1} = 40 \text{ g/Eq. Wt.}$$

Step 3: Set up Ratio.

$$\frac{1 \text{ N}}{1.2 \text{ N}} = \frac{40 \text{ g/EqWt}}{X}$$

$$X = \frac{(40 \text{ g/Eq.Wt.})(1.2 \text{ N})}{1 \text{ N}}$$

$$X = 48 \text{ g}$$

Normality

Another important method for expressing the concentration of a solution is **normality**.

To understand normality, one must first understand **equivalent weights** (Eq.Wt.).

Equivalent Weights

*Think of Equivalent as Equal Valence.

Normality depends in part on the valence of an element or compound. Some elements have more than one valence, (i.e. Fe^{+2} , Fe^{+3}). Thus, when dealing with a solution of Iron it is not always clear which valence a given normality represents.

"Equivalent weights" level the playing -field.

$$\text{Equivalent Weight} = \frac{\text{Molecular wt.}}{\text{valence}}$$

Example:

$$\text{Eq.Wt. of Calcium} = \frac{\text{MW of Calcium}}{\text{Valence of Calcium}} = \frac{40 \text{ g}}{2} = \frac{20 \text{ g}}{\text{Eq.Wt.}}$$

$$\text{Eq.Wt. of Sodium} = \frac{\text{MW of Sodium}}{\text{Valence of Sodium}} = \frac{23 \text{ g}}{1} = \frac{23 \text{ g}}{\text{Eq.Wt.}}$$

$$\text{Eq.Wt. of Calcium Carbonate} = \frac{\text{MW of CaCO}_3}{\text{Valence of Calcium}} = \frac{100 \text{ g}}{2} = \frac{50 \text{ g}}{\text{Eq.Wt.}}$$

$$\text{Eq.Wt. of Ferric Hydroxide} = \frac{\text{MW of Fe(OH)}_3}{\text{Valence of Fe}^{+3}\text{(OH)}_3} = \frac{107 \text{ g}}{3} = \frac{35.6 \text{ g}}{\text{Eq.Wt.}}$$

$$\text{Eq.Wt. of Magnesium} = \frac{\text{MW of Mg}}{\text{Valence of Mg}} = \frac{24 \text{ g}}{2} = \frac{12 \text{ g}}{\text{Eq.Wt.}}$$

Equivalent Weight, Normality to determine Hardness

The total hardness in water is usually expressed in terms of CaCO_3 . For example a lab report might read:

Total Hardness 180 mg/L as CaCO_3

This means that the lab has not determined exactly what chemicals are causing the water's hardness, but that their combined effect is the same as if the water contained exactly 180 mg/L of CaCO_3 .

By expressing the hardness of every sample in terms of how much calcium carbonate it might contain, the hardness of any two samples can be **compared** more easily.

Example:

A lab report shows $\text{Mg} = 17 \text{ mg/L}$. How do you express Magnesium as CaCO_3 ?

Step 1: Determine MW

$$\begin{aligned} \text{MW Mg} &= 24\text{g/mol} \\ \text{MW CaCO}_3 &= 100\text{g/mol} \end{aligned}$$

Step 2: Determine Eq. Wt.

$$\text{Eq.Wt. Mg} = \frac{\text{MW}}{\text{Valence}} = \frac{24}{2} = 12\text{g/Eq.Wt.}$$

$$\text{Eq.Wt. CaCO}_3 = \frac{\text{MW}}{\text{Valence}} = \frac{100}{2} = 50\text{g/Eq.Wt.}$$

Step 3: Set up Ratio

$$\frac{\text{Eq.Wt. Mg}}{\text{Eq.Wt. CaCO}_3} = \frac{[\text{Mg}]}{[X]} \quad \frac{12\text{g/Eq.Wt. Mg}}{50 \text{ g/Eq.Wt. CaCO}_3} = \frac{17\text{mg/L Mg}}{X}$$

$$X = \frac{(17 \text{ mg/L Mg}) (50 \text{ g/Eq.Wt. CaCO}_3)}{12 \text{ g/Eq.Wt. Mg}}$$

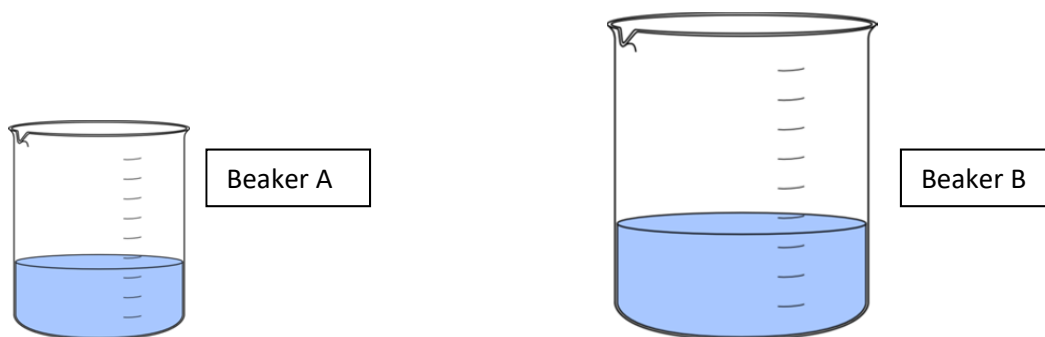
$$X = 71 \text{ mg/L as CaCO}_3$$

Dilution of Concentrated Acids and Bases to Prepare a 1N Solution

Compound	Formula	Molecular Weight	Approximate Spec. Grav. Of Concentrated Reagent	Approximate % Present in Concentrated Reagent	Normality of Concentrated Reagent	Approximate mL of Concentrated Reagent to dilute to 1 L
Acetic Acid	$\text{CH}_3 \bullet \text{COOH}$	60.054	1.05	99.6	17.4	58
Ammonium Hydroxide	NH_4OH	35.048	0.90	57.6	14.8	68
Hydrochloric Acid	HCl	36.465	1.19	37.0	12.1	83
Lactic Acid	$\text{CH}_3 \bullet \text{CHOH} \bullet \text{COOH}$	90.081	1.21	85.0	11.4	88
Nitric Acid	HNO_3	63.016	1.42	69.5	15.7	64
Perchloric Acid	HClO_4	100.465	1.67	70.0	11.6	87
Phosphoric Acid (ortho-)	H_3PO_4	97.999	1.69	85.0	44.0	23
Potassium Hydroxide	KOH	56.108	1.51	50.0	13.5	75
Sodium Hydroxide	NaOH	39.999	1.53	50.0	19.1	53
Sulfuric Acid	H_2SO_4	98.082	1.84	96.0	36.0	28

Intro to Basic Chemistry and Solutions Chemistry – Review Questions

1. What basic information is given in the Periodic Table?
 - a. Atomic weight
 - b. Elemental symbol
 - c. Name of the element
 - d. Atomic number
 - e. All of the above
2. Pure water is a chemical made up of two components. Each molecule of water is made up of two atoms of hydrogen and:
 - a. Two atoms of oxygen
 - b. One pound of oxygen
 - c. Three atoms of nitrogen
 - d. One atom of carbon
 - e. One atom of oxygen
3. What is a molecule?
 - a. The smallest portion of an atom
 - b. Two or more atoms joined together by a chemical bond
 - c. Two or more atoms joined together by physical attraction
 - d. A fundamental substance consisting of only one kind of atom
4. What is a compound?
 - a. Two or more elements bonded together by a chemical reaction
 - b. The smallest particle of an element that still retains the characteristics of that element
 - c. Two or more atoms joined together by physical attraction
 - d. Two or more atoms of the same element
 - e. Both c. and d.
5. Beaker A holds 250 mL and Beaker B holds 1000 mL. Two drops of food coloring are added to Beaker A, and 4 drops of food coloring are added to Beaker B.



- What is the solute?
- Which beaker has the greater amount of food dye?
- Which beaker has the greater concentration of food dye?

Section 6
Temperature

Temperature

Introduction to Laboratory Methods for Operators



Temperature

- One of the most frequently taken tests in the water industry
- Accurate temperature readings are important for:
 - Historical purposes
 - Chemical reaction rates
 - Biological growth
 - Dissolved gas concentrations (DO)
 - Water stability (calcium carbonate)

Temperature - Sampling

- Measurements should be taken where samples are collected for other tests
- Larger volume of sample = less change in temp as compared to a smaller sample
- Changes rapidly
- Performed immediately
- Do not touch the bottom or sides of the sample container

Temperature

- Always leave the thermometer in the liquid while reading the temperature
- Grab samples
- Record:
 - Temperature result
 - Time of analysis
 - Location of sample
 - Sampler's name

Thermometers

- Periodically check thermometer's bias against a reference thermometer certified by NIST
 - At least annually
 - Record of certification
- NIST = National Institute of Standards and Technology
 - Formerly NSB (National Bureau of Standards)

Thermometers

- After calibration, mark the necessary calibration correction factor on each device so that only calibrated/corrected temperature values are recorded.



Thermometers

- **Mercury filled**
 - Avoid due to danger of releasing mercury into atmosphere if thermometer breaks
 - Spill kit in lab
- **Spirit filled/Environmentally safe liquid**
 - Petroleum hydrocarbon
 - Clear odorless liquid with a petroleum odor, usually mixed with blue, red or green colored dye

Thermometers

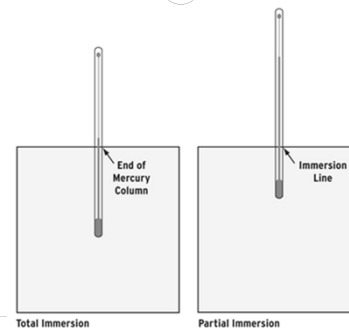
Styles of thermometers:

1. **Total Immersion** – must be totally immersed when read
 - Readings will change most rapidly when removed from the liquid to be recorded
 - Indicate an accurate temperature reading when the thermometer is immersed to the level of the liquid in the capillary column
 - There is a minimal emergent length that is for handling

Thermometers

2. **Partial Immersion** – will have a solid line (water-level indicator) around the stem below the point where the scale starts
 - Must be immersed to the depth of the etched circle around the stem to get the correct reading
 - Indicate accurate temperatures when the thermometer is immersed to the specific depth indicated by the immersion line, regardless of the liquid in the column
 - Appropriate for any application where total immersion is impractical or impossible, such as in a shallow water bath

Total Immersion vs. Partial Immersion



Thermometers

3. **Dial** – has a dial that can be easily read while the thermometer is still immersed
 - Should be checked (calibrated) against an NIST thermometer
 - Some can be recalibrated (adjusted) to read at a set temperature against the NIST thermometer



Thermometers

4. **Digital readout**



Note: Infra-red heat guns are used in laboratories for sample receiving, but are not allowed for compliance monitoring or as part of method process

Temperature Readings

- **In the laboratory:**
 - Twice daily
 - At least 4 hours apart
- **Can use a metal case to protect from breaking in the field**

Thermometers

- **If the liquid inside separates, no longer valid readings**
- **Two options to fix (spirit filled):**
- 1. Centrifugal Method:**
 - 1. Force the liquid down the capillary using a centrifuge with a cup deep enough to apply centrifugal force below the liquid column
 - 2. Insert the thermometer, bulb down, in the centrifuge. Pad the bottom of the cup to prevent damage to the bulb
 - 3. Turn on the centrifuge for several seconds to force all the liquid past the separation

Thermometers

2. Tapping Method:

- **A separated spirit-filled thermometer column can be reunited by brisk tapping until the separated liquid runs down to join the main column**
- 1. Hold the thermometer in an upright position and gently tap the stem above the liquid separation against the palm of the hand
- 2. Continue tapping until the liquid above the separation breaks away from the wall of the capillary and runs down to join the main column

Where is temperature used?

- **General laboratory operations**
- **Calculation of percent saturation of dissolved oxygen in the DO test**
- **Calculation of various forms of alkalinity**
- **Studies of saturation and stability with respect to calcium carbonate**
- **Calculation of salinity**
- **In a number of colorimetric tests**

Where is temperature used

- **Discharges of heated water may have significant ecological impact**
- **Detecting changes in raw wastewater quality**
 - Ex: influent temp drop may indicate large volumes of cold water from infiltration
- **Source of water supply, such as deep wells, often can be identified by temperature alone**

Any Questions?

**ALDRICH**[®]

Advancing Science

Aldrich is a member of the Sigma-Aldrich family

6000 N. Teutonia Ave., Milwaukee, WI 53209 Telephone: (800) 558-9160 • (414) 438-3850 Fax: (800) 962-9591 • (414) 273-4979 Website: <http://www.sigma-aldrich.com>
E-mail: aldrich@sial.com

Technical Bulletin

Reuniting Separated Thermometer Columns

Mercury-filled Thermometers

This technique applies to most mercury thermometers regardless of temperature range, except deep immersion types.

Dry-ice Method:

1. Hold the thermometer in an upright position and gradually immerse the bulb in a solution of dry-ice and alcohol so that the mercury column retreats slowly into the bulb. Do not cool the stem or mercury column.
2. Keep the bulb in the solution until the main column as well as the separated portion retreats into the bulb.
3. Remove and swing thermometer in a short arc, forcing all the mercury into the bulb.

Caution:

Do not touch the thermometer bulb until the mercury emerges from the bulb into the column or immerse the stem or mercury column in the dry ice solution as it will freeze the mercury in the column and fracture the bulb.

Spirit-filled Thermometers

Centrifugal Method:

1. Force the liquid down the capillary using a centrifuge with a cup deep enough to apply centrifugal force below the liquid column.
2. Insert the thermometer, bulb down, in the centrifuge. Pad the bottom of the cup to prevent damage to the bulb.
3. Turn on the centrifuge for several seconds to force all the liquid past the separation.

Caution:

If the applied centrifugal force is not below the entire column, the liquid column will split forcing part of the liquid down and the rest upwards filling the expansion chamber.

Tapping Method:

A separated spirit-filled thermometer column can be reunited by brisk tapping until the separated liquid runs down to join the main column.

1. Hold the thermometer in an upright position and gently tap the stem above the liquid separation against the palm of the hand.
2. Continue tapping until the liquid above the separation breaks away from the wall of the capillary and runs down to join the main column.

Caution:

Wear a pair of cut-resistant gloves while performing this procedure in the event of breakage.


Temperature – Review Questions

1. Why are temperature readings important?
2. It is better to choose a smaller volume of water to take your temperature sample because it will result in less change in temperature. True or False
3. Why should a thermometer remain immersed in liquid while being read?
4. Temperature readings should be taken on a composite sample. True or False
5. Which type of thermometer that we discussed is not approved for compliance monitoring or as part of a method process?
6. How often must temperature readings be taken in the laboratory?
7. Why must thermometers be calibrated against an NIST-certified thermometer? And how often does the State of TN recommend for calibration?

Section 7
Dissolved Oxygen

Dissolved Oxygen


Introduction to Laboratory Methods for Operators



1

What is Dissolved Oxygen (DO)?

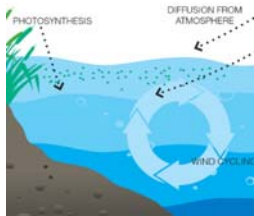
- * Measure of the amount of gaseous oxygen contained water
- * Necessity in water to support life
- * Max amount of DO is the saturation concentration



2

How does Dissolved Oxygen enter water?

- * Direct absorption from atmosphere
- * Rapid movement from winds, waves, currents or mechanical aeration
- * Photosynthesis



3

Why monitor Dissolved Oxygen?

- * Quality of Water
 - * Determines the quality of source water
 - * Without DO, water turns foul and unhealthy
 - * Can affect the quality of the environment, drinking water and other products
- * Regulatory Compliance
 - * Certain concentrations are required before discharged to stream
- * Process Control

4

Factors Influencing DO Saturation Concentration

- * Atmospheric Pressure
 - * The weight of air above a certain point
 - * Higher atmospheric pressure allows bodies of water to retain more dissolved oxygen
 - * Higher elevation, DO concentrations are lower
- * Temperature
 - * Lower temperature can contain more dissolved oxygen because they have less movement
 - * Increase in movement allows them to escape out of the water


5

Factors Influencing DO Saturation Concentration

- * Depth of Water
 - * Shallower water, higher the DO concentration
 - * Waves and movement will increase DO
 - * Aquatic plants will be able to create DO through Photosynthesis
- * Salinity
 - * Salts affect the solubility of gases, essentially driving them out of water
- * Bioactivity
 - * Microorganisms feeding on organics and decaying matter use up oxygen

6

Barometric Pressure

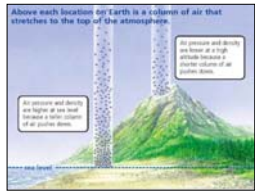


"Not now - I'm under a lot of atmospheric pressure."

7

Barometric Pressure

- * Barometric Pressure = the pressure of the column of air above us
- * The higher we go up, the less the pressure becomes
- * Lower barometric pressures are found at higher elevations



8

Barometric Pressure

- Where to find barometric pressure:
 - * Barometer (in lab), onboard barometer (built into meter), online (ex: weather app, airport, etc.)
- When you get a BP reading from the airport, it has been "corrected" to sea level
 - * You need to uncorrect the BP before calibrating your DO probe!
 - * In other words, you must determine the true barometric pressure

9

Why is BP important?

- * You must compensate for barometric pressure when calibrating the instrument
- * Weather condition variations are typically +/- 1% from week to week
 - * Extreme weather can cause variations up to 4x larger
- * You can see 1-2% variations in readings between weeks

10

How to Uncorrect a Barometric Pressure

1. Determine the altitude (in feet) of your lab/facility
2. Determine the correction factor for your lab/facility

$$CF = [760 - (\text{Altitude} \times 0.026)] \div 760$$
3. Look up the barometric pressure (BP) for the day
 - Ex: Airport website, Weather Channel or app
4. Multiply the corrected BP times your Correction Factor (CF)

11

Example

1. Fleming Training Center altitude = 543 feet
2. Plug that altitude into the Correction Factor equation:

$$CF = [760 - (\text{Altitude} \times 0.026)] \div 760$$

$$= [760 - (543 \times 0.026)] \div 760$$

$$= [760 - 14.1] \div 760$$

$$= 745.9 \div 760 = 0.9795 \text{ (rounds to 0.98)}$$
3. Use your cell phone to find the barometric pressure (Ex: Airport BP = 29.65 in Hg... remember, this is a corrected BP)
4. Multiply 29.65 x 0.98 = 29.05

Therefore, your uncorrected BP is 29.05 in Hg

12

Example (continued)

- * Once you have the uncorrected true BP, determine the oxygen solubility at that pressure and temperature
- * Use the DO Saturation Table to obtain the maximum O₂ solubility at that temperature

<http://water.usgs.gov/software/DOTABLES/>

USGS
Water Resources of the United States

DOTABLES Result
Oxygen Solubility Table

Solubility of oxygen in fresh water at various temperatures and pressures.
[Solubility shown in milligrams per liter. Values based on published equations by Benson and Krause (1980 and 1984).
°C, degrees Celsius; in Hg, inches of mercury.]

Temp. (°C)	22.44	22.83	33.22	33.61	24.00	24.39	24.78	25.17	25.56	25.95	26.34	26.73	27.12	27.51	27.90	28.29	28.68	29.07	29.46	29.85	
20	6.77	6.69	7.01	7.13	7.25	7.37	7.46	7.52	7.58	7.64	7.70	7.74	7.78	7.82	7.85	7.88	7.91	7.94	7.97	8.00	8.03
21	6.63	6.75	6.87	6.99	7.11	7.23	7.35	7.46	7.56	7.65	7.72	7.78	7.84	7.89	7.94	7.98	8.02	8.06	8.10	8.14	8.18
22	6.50	6.62	6.73	6.85	6.97	7.08	7.20	7.32	7.44	7.55	7.67	7.79	7.90	8.02	8.14	8.25	8.37	8.49	8.61	8.73	8.85
23	6.37	6.49	6.60	6.72	6.83	6.95	7.06	7.18	7.29	7.41	7.52	7.64	7.75	7.87	7.98	8.10	8.21	8.33	8.44	8.56	8.68
24	6.25	6.36	6.48	6.59	6.70	6.82	6.93	7.04	7.15	7.27	7.38	7.49	7.61	7.72	7.83	7.95	8.06	8.17	8.28	8.40	8.52
25	6.13	6.24	6.35	6.47	6.58	6.69	6.80	6.91	7.02	7.13	7.24	7.35	7.47	7.58	7.69	7.80	7.91	8.02	8.13	8.24	8.35
26	6.02	6.13	6.24	6.34	6.45	6.56	6.67	6.78	6.89	7.00	7.11	7.22	7.33	7.44	7.55	7.66	7.77	7.88	7.99	8.10	8.21
27	5.90	6.01	6.12	6.23	6.33	6.44	6.55	6.66	6.77	6.87	6.98	7.09	7.20	7.30	7.41	7.52	7.63	7.73	7.84	7.95	8.06
28	5.80	5.90	6.01	6.11	6.22	6.32	6.43	6.54	6.64	6.75	6.86	6.96	7.07	7.17	7.28	7.38	7.49	7.60	7.70	7.81	7.91
29	5.69	5.79	5.90	6.00	6.11	6.21	6.32	6.42	6.52	6.63	6.73	6.84	6.94	7.05	7.15	7.26	7.36	7.46	7.57	7.67	7.77
30	5.59	5.69	5.79	5.90	6.00	6.10	6.20	6.31	6.41	6.51	6.62	6.72	6.82	6.92	7.03	7.13	7.23	7.33	7.44	7.54	7.64

13

Example (continued)

USGS
Water Resources of the United States

DOTABLES Result
Oxygen Solubility Table

Solubility of oxygen in fresh water at various temperatures and pressures.
[Solubility shown in milligrams per liter. Values based on published equations by Benson and Krause (1980 and 1984).
°C, degrees Celsius; in Hg, inches of mercury.]

Temp. (°C)	22.44	22.83	33.22	33.61	24.00	24.39	24.78	25.17	25.56	25.95	26.34	26.73	27.12	27.51	27.90	28.29	28.68	29.07	29.46	29.85	
20	6.77	6.69	7.01	7.13	7.25	7.37	7.46	7.52	7.58	7.64	7.70	7.74	7.78	7.82	7.85	7.88	7.91	7.94	7.97	8.00	8.03
21	6.63	6.75	6.87	6.99	7.11	7.23	7.35	7.46	7.56	7.65	7.72	7.78	7.84	7.89	7.94	7.98	8.02	8.06	8.10	8.14	8.18
22	6.50	6.62	6.73	6.85	6.97	7.08	7.20	7.32	7.44	7.55	7.67	7.79	7.90	8.02	8.14	8.25	8.37	8.49	8.61	8.73	8.85
23	6.37	6.49	6.60	6.72	6.83	6.95	7.06	7.18	7.29	7.41	7.52	7.64	7.75	7.87	7.98	8.10	8.21	8.33	8.44	8.56	8.68
24	6.25	6.36	6.48	6.59	6.70	6.82	6.93	7.04	7.15	7.27	7.38	7.49	7.61	7.72	7.83	7.95	8.06	8.17	8.28	8.40	8.52
25	6.13	6.24	6.35	6.47	6.58	6.69	6.80	6.91	7.02	7.13	7.24	7.35	7.47	7.58	7.69	7.80	7.91	8.02	8.13	8.24	8.35
26	6.02	6.13	6.24	6.34	6.45	6.56	6.67	6.78	6.89	7.00	7.11	7.22	7.33	7.44	7.55	7.66	7.77	7.88	7.99	8.10	8.21
27	5.90	6.01	6.12	6.23	6.33	6.44	6.55	6.66	6.77	6.87	6.98	7.09	7.20	7.30	7.41	7.52	7.63	7.73	7.84	7.95	8.06
28	5.80	5.90	6.01	6.11	6.22	6.32	6.43	6.54	6.64	6.75	6.86	6.96	7.07	7.17	7.28	7.38	7.49	7.60	7.70	7.81	7.91
29	5.69	5.79	5.90	6.00	6.11	6.21	6.32	6.42	6.52	6.63	6.73	6.84	6.94	7.05	7.15	7.26	7.36	7.46	7.57	7.67	7.77
30	5.59	5.69	5.79	5.90	6.00	6.10	6.20	6.31	6.41	6.51	6.62	6.72	6.82	6.92	7.03	7.13	7.23	7.33	7.44	7.54	7.64

14

Conversions - BP

- * Your barometer may read in mm or in Hg (mercury)
- * Likewise, DO Tables can display either mm or in Hg
- * To convert inches of mercury (Hg) to mm of mercury (Hg):
inches of Hg X 25.4 = mm of Hg
- * To convert mm of mercury (Hg) to inches of mercury (Hg):
mm of Hg ÷ 25.4 = inches of Hg

15

Conversion - Temperature

- * To convert from Celsius to Fahrenheit:
$$^{\circ}\text{F} = [(^{\circ}\text{C})(1.8) + 32]$$
- * To convert from Fahrenheit to Celsius:
$$^{\circ}\text{C} = \frac{(^{\circ}\text{F} - 32)}{1.8}$$

16

Barometer on site

Easiest way to do this - Direct Read from Barometer & Thermometer in the lab to determine DO from USGS DO table

Barometric Pressure (BP) UNCORRECTED **Temperature °C** **USGS DO Table (mm Hg)**

17

Dissolved Oxygen Measurement

OPTICAL
• BLUE DETECTION LIGHT
• RED REFERENCE LIGHT
• PHOTOSENSOR
• FLUORESCENT DYE
• CW

POLAROGRAPHIC
• GOLD CATHODE
• SILVER ANODE
• HCl
• MEMBRANE

GALVANIC
• SILVER OR MERCURY CATHODE
• ZINC OR LEAD ANODE
• NaCl
• MEMBRANE

18

Approved Methods 40 CFR 136

Parameter	Methodology ⁶³	EPA ⁶²	Standard methods ⁶⁴	ASTM	USGS/AOAC/Other
46. Oxygen, dissolved, mg/L	Winkler (Azide modification)		4500-O (B-F)-2016	D888-12 (A)	973.458, ⁷ I-1575-78, ⁸
	Electrode		4500-O G-2016	D888-12 (B)	I-1576-78, ⁸
	Luminescence-Based Sensor		4500-O H-2016	D888-12 (C)	See footnote. ⁶³ See footnote. ⁶⁴

⁶³ Hach Method 10360, Luminescence Measurement of Dissolved Oxygen in Water and Wastewater and for Use in the Determination of BOD₅ and CBOD₅, Revision 1.2, October 2011. Hach Company. This method may be used to measure dissolved oxygen when performing the methods approved in Table 1B for measurement of biochemical oxygen demand (BOD) and carbonaceous biochemical oxygen demand (CBOD).

⁶⁴ In-Situ Method 1002-8-2009, Dissolved Oxygen (DO) Measurement by Optical Probe, 2009. In-Situ Incorporated.

19

Winkler Method

- * Classic wet chemistry procedure
- * Titration method
- * A series of reactions produces iodine
- * In proportion to the amount of DO

- * Sodium thiosulfate with starch as the indicator
- * Complicated method
- * Calibration check or meter check

20

Membrane Electrode Method SM 4500- O G

- * Polarographic Probe
- * Voltage across two electrodes is provided by the meter
- * Oxygen molecules are chemically changed to hydroxide ions
 - * DO is consumed by the probe
- * Oxygen diffuses through a plastic membrane
 - * Rate proportional to concentration
- * Always circulate water by the membrane
- * Proper maintenance is key

21

Optical Probe Method 4500-O H

- * Use luminescence to measure oxygen
- * Blue light is shone on a special dye
- * Light excites the atoms in the dye, emit a red light
- * Oxygen molecules interfere with the red light
- * The sensor correlates the red light returning with how much oxygen in sample
- * Probe caps must be replaced
- * Oxygen is not consumed

22

Sampling

23

Sampling 40 CFR 136

Parameter number/name	Container ¹	Preservation ^{2,3}	Maximum holding time ⁴
46. Oxygen, Dissolved Probe	G, Bottle and top	None required	Analyze within 15 minutes.

24

Collecting DO samples

- * Oxygen can dissolve from the atmosphere into the sample and change the reading
- * If using a wide-mouth dipper or a bucket, you're likely to catch mainly water from the upper layer
 - * Could have a higher DO than lower layers because it is in contact with the air

25

Collecting DO Samples

- * After collecting a sample, shield it from contact with the atmosphere
- * Oxygen will dissolve into a container of water sitting open on a lab bench
- * When taking DO readings in an aerated tank using a probe, do not place the probe directly over a diffuser
 - * You want to measure the DO in the water, not the oxygen in the air supply to the aerator

26

Any Questions?

27



Water Resources of the United States

DOTABLES Result

Oxygen Solubility Table

Solubility of oxygen in fresh water at various temperatures and pressures

[Solubility shown in milligrams per liter. Values based on published equations by Benson and Krause (1980 and 1984).

°C, degrees Celsius; in Hg, inches of mercury]

Temp. (°C)	Barometric Pressure (in Hg)																			
	22.44	22.83	23.22	23.61	24.00	24.39	24.78	25.17	25.56	25.95	26.34	26.73	27.12	27.51	27.90	28.29	28.68	29.07	29.46	29.85
0	10.95	11.14	11.33	11.52	11.71	11.90	12.10	12.29	12.48	12.67	12.86	13.05	13.24	13.44	13.63	13.82	14.01	14.20	14.39	14.59
1	10.64	10.83	11.01	11.20	11.39	11.57	11.76	11.95	12.13	12.32	12.51	12.69	12.88	13.06	13.25	13.44	13.62	13.81	14.00	14.18
2	10.35	10.53	10.71	10.89	11.08	11.26	11.44	11.62	11.80	11.98	12.16	12.35	12.53	12.71	12.89	13.07	13.25	13.43	13.62	13.80
3	10.07	10.25	10.43	10.60	10.78	10.96	11.13	11.31	11.49	11.66	11.84	12.02	12.19	12.37	12.55	12.72	12.90	13.08	13.25	13.43
4	9.81	9.98	10.15	10.32	10.50	10.67	10.84	11.01	11.18	11.36	11.53	11.70	11.87	12.04	12.22	12.39	12.56	12.73	12.90	13.08
5	9.55	9.72	9.89	10.06	10.22	10.39	10.56	10.73	10.89	11.06	11.23	11.40	11.57	11.73	11.90	12.07	12.24	12.40	12.57	12.74
6	9.31	9.47	9.64	9.80	9.96	10.13	10.29	10.45	10.62	10.78	10.95	11.11	11.27	11.44	11.60	11.76	11.93	12.09	12.25	12.42
7	9.08	9.24	9.40	9.55	9.71	9.87	10.03	10.19	10.35	10.51	10.67	10.83	10.99	11.15	11.31	11.47	11.63	11.79	11.95	12.11
8	8.85	9.01	9.16	9.32	9.48	9.63	9.79	9.94	10.10	10.26	10.41	10.57	10.72	10.88	11.04	11.19	11.35	11.50	11.66	11.81
9	8.64	8.79	8.94	9.10	9.25	9.40	9.55	9.70	9.86	10.01	10.16	10.31	10.47	10.62	10.77	10.92	11.08	11.23	11.38	11.53
10	8.43	8.58	8.73	8.88	9.03	9.18	9.33	9.47	9.62	9.77	9.92	10.07	10.22	10.37	10.52	10.67	10.81	10.96	11.11	11.26
11	8.24	8.38	8.53	8.67	8.82	8.96	9.11	9.25	9.40	9.55	9.69	9.84	9.98	10.13	10.27	10.42	10.56	10.71	10.86	11.00
12	8.05	8.19	8.33	8.47	8.62	8.76	8.90	9.04	9.19	9.33	9.47	9.61	9.75	9.90	10.04	10.18	10.32	10.47	10.61	10.75
13	7.86	8.00	8.14	8.28	8.42	8.56	8.70	8.84	8.98	9.12	9.26	9.40	9.54	9.68	9.81	9.95	10.09	10.23	10.37	10.51
14	7.69	7.83	7.96	8.10	8.23	8.37	8.51	8.64	8.78	8.92	9.05	9.19	9.33	9.46	9.60	9.74	9.87	10.01	10.14	10.28
15	7.52	7.65	7.79	7.92	8.06	8.19	8.32	8.46	8.59	8.72	8.86	8.99	9.12	9.26	9.39	9.53	9.66	9.79	9.93	10.06
16	7.36	7.49	7.62	7.75	7.88	8.01	8.14	8.28	8.41	8.54	8.67	8.80	8.93	9.06	9.19	9.32	9.45	9.58	9.72	9.85
17	7.20	7.33	7.46	7.59	7.72	7.84	7.97	8.10	8.23	8.36	8.49	8.61	8.74	8.87	9.00	9.13	9.26	9.38	9.51	9.64
18	7.05	7.18	7.30	7.43	7.56	7.68	7.81	7.93	8.06	8.19	8.31	8.44	8.56	8.69	8.81	8.94	9.07	9.19	9.32	9.44
19	6.91	7.03	7.15	7.28	7.40	7.52	7.65	7.77	7.90	8.02	8.14	8.27	8.39	8.51	8.64	8.76	8.88	9.01	9.13	9.25

Temp. (°C)	Barometric Pressure (in Hg)																			
	22.44	22.83	23.22	23.61	24.00	24.39	24.78	25.17	25.56	25.95	26.34	26.73	27.12	27.51	27.90	28.29	28.68	29.07	29.46	29.85
20	6.77	6.89	7.01	7.13	7.25	7.37	7.49	7.62	7.74	7.86	7.98	8.10	8.22	8.34	8.46	8.59	8.71	8.83	8.95	9.07
21	6.63	6.75	6.87	6.99	7.11	7.23	7.35	7.46	7.58	7.70	7.82	7.94	8.06	8.18	8.30	8.42	8.54	8.66	8.77	8.89
22	6.50	6.62	6.73	6.85	6.97	7.08	7.20	7.32	7.44	7.55	7.67	7.79	7.90	8.02	8.14	8.25	8.37	8.49	8.61	8.72
23	6.37	6.49	6.60	6.72	6.83	6.95	7.06	7.18	7.29	7.41	7.52	7.64	7.75	7.87	7.98	8.10	8.21	8.33	8.44	8.56
24	6.25	6.36	6.48	6.59	6.70	6.82	6.93	7.04	7.15	7.27	7.38	7.49	7.61	7.72	7.83	7.95	8.06	8.17	8.28	8.40
25	6.13	6.24	6.35	6.47	6.58	6.69	6.80	6.91	7.02	7.13	7.24	7.35	7.47	7.58	7.69	7.80	7.91	8.02	8.13	8.24
26	6.02	6.13	6.24	6.34	6.45	6.56	6.67	6.78	6.89	7.00	7.11	7.22	7.33	7.44	7.55	7.66	7.77	7.88	7.98	8.09
27	5.90	6.01	6.12	6.23	6.33	6.44	6.55	6.66	6.77	6.87	6.98	7.09	7.20	7.30	7.41	7.52	7.63	7.73	7.84	7.95
28	5.80	5.90	6.01	6.11	6.22	6.33	6.43	6.54	6.64	6.75	6.86	6.96	7.07	7.17	7.28	7.38	7.49	7.60	7.70	7.81
29	5.69	5.79	5.90	6.00	6.11	6.21	6.32	6.42	6.52	6.63	6.73	6.84	6.94	7.05	7.15	7.25	7.36	7.46	7.57	7.67
30	5.59	5.69	5.79	5.90	6.00	6.10	6.20	6.31	6.41	6.51	6.62	6.72	6.82	6.92	7.03	7.13	7.23	7.33	7.44	7.54

[Return](#) to the DOTABLES main page.

[USGS Home](#) :: [Biology](#) :: [Geology](#) :: [Geography](#) :: [Water Intranet](#) :: [USGS Intranet](#) :: [Site Map](#)

[U.S. Department of the Interior](#) | [U.S. Geological Survey](#)

URL: <http://water.usgs.gov/cgi-bin/dotables>

Page Contact Information: [Water Webserver Team](#)

Page Last Modified: Wednesday, 16-Jan-2013 at 10:03:49 EST

Dissolved Oxygen (DO) – Review Questions

1. What is the hold time for a DO sample?

2. List at least 5 reasons why DO levels are important with regards to water?

3. The amount of oxygen that a given volume of water can hold is a function of what 3 things?
 - a)
 - b)
 - c)

4. What is barometric pressure?

5. Convert the following numbers into in Hg:
 - a) 760 mm Hg
 - b) 732 mm Hg
 - c) 745 mm Hg

6. Convert the following numbers into mm Hg:
 - a) 23.61 in Hg
 - b) 25.56 in Hg
 - c) 29.85 in Hg

7. Convert the following temperatures into °F:
 - a) 17°C
 - b) 20°C
 - c) 29°C

8. Convert the following temperatures into °C:
 - a) 55°F
 - b) 65°F
 - c) 70.5°F

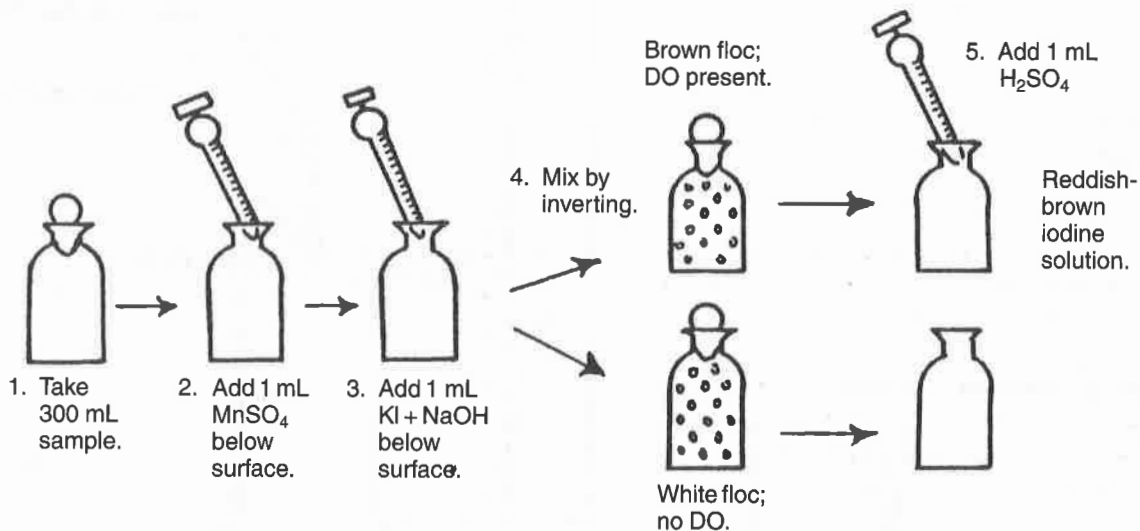
9. You checked the local airport website and they are reporting the current barometric pressure as 29.46 in Hg. You know that this is a “corrected” BP, so now you must “uncorrect” it to determine the oxygen solubility. Use the equation listed below to find the correction factor that will be used to get the uncorrected BP. (The elevation/altitude of Fleming Training Center is 543 feet above sea level.)

$$CF = [760 - (\text{Altitude} \times 0.026)] \div 760$$

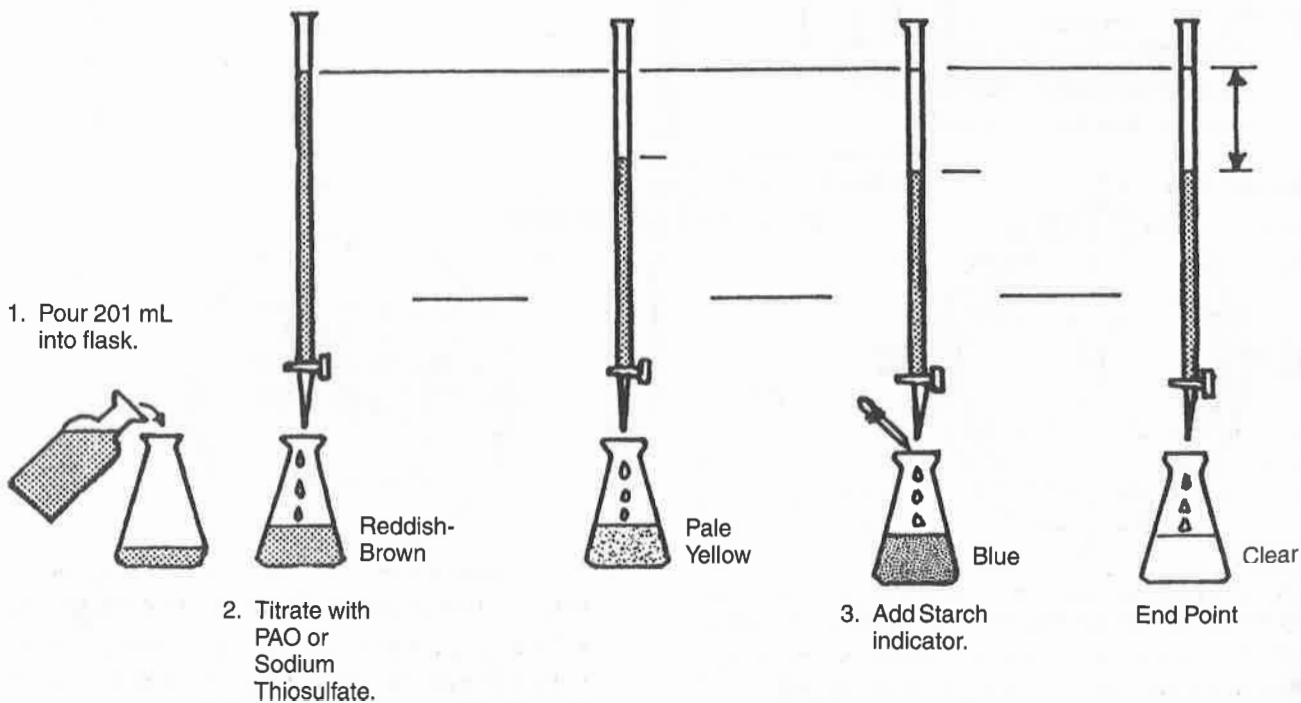
Use that correction factor to determine the uncorrected BP.

10. Now that you have calculated the uncorrected BP, you must use the USGS DOTABLE find the oxygen solubility. The room temperature is 68°F and must be converted to Celsius. Use that information, in conjunction with the uncorrected BP, to determine the oxygen solubility.

OUTLINE OF PROCEDURE FOR DO



Titration of Iodine Solution:



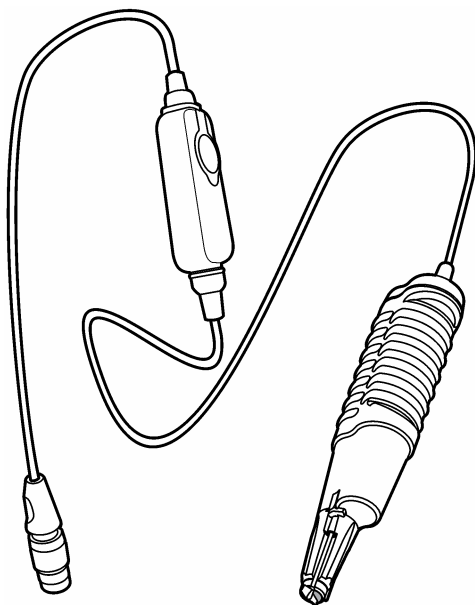


DOC022.53.80025

LBOD101

05/2021, Edition 4

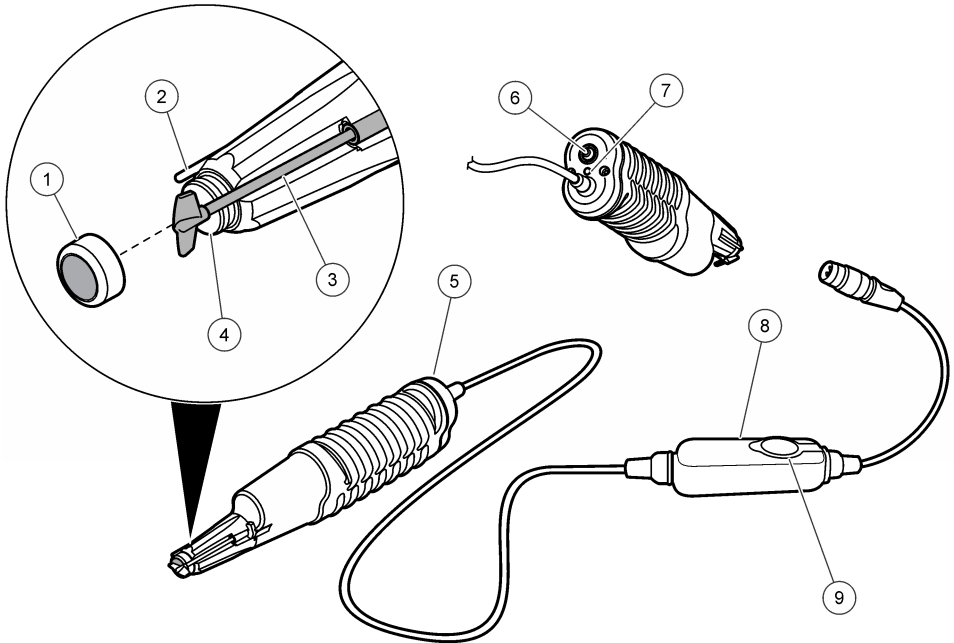
User Manual



Section 1 Product overview	3
Section 2 Specifications	3
Section 3 Safety information	4
3.1 Intended use	4
3.2 Use of hazard information	4
3.3 Precautionary labels	5
3.4 Product hazards	5
Section 4 Preparation for use	5
Section 5 Calibration	6
5.1 Calibration notes	6
5.2 LBOD water-saturated air (100%) calibration procedure	6
5.3 Zero-point calibration procedure	7
Section 6 Sample measurement	8
6.1 Sample measurement notes	8
6.2 Sample measurement procedure	8
6.3 Low-level measurements	9
Section 7 Maintenance	9
7.1 Clean the probe	9
7.2 Replace the sensor cap and iButton	9
7.3 Replace the stirrer	12
7.4 Storage	12
Section 8 Troubleshooting	13
Section 9 Consumables	14

The Intellical LBOD101 probe is a digital, luminescent dissolved oxygen sensor that measures the dissolved oxygen concentration of BOD (biochemical oxygen demand) samples. The probe has temperature and absolute air pressure sensors for accurate dissolved oxygen measurements. The probe stays on standard BOD bottles and stirs the sample during measurements. Refer to [Figure 1](#).

Figure 1 Probe overview



1 LBOD sensor cap	6 Stirrer on/off button
2 Thermistor	7 Power indicator LED
3 Stirrer assembly	8 Air pressure sensor module
4 Probe lens	9 iButton® compartment ¹
5 Probe body with stirrer assembly	

Section 2 Specifications

Specifications are subject to change without notice.

Specifications	Details
Probe type	Luminescent dissolved oxygen (LDO) sensor with integrated stirring system
Dissolved oxygen range	0.05 to 20.0 mg/L (ppm); 1 to 200% saturation
Dissolved oxygen accuracy	±0.05 mg/L for concentrations less than 10 mg/L O ₂ ±0.1 mg/L for concentrations more than 10 mg/L O ₂

¹ iButton® is a registered trademark of Maxim Integrated Products, Inc.

Specifications	Details	Section 7
% saturation resolution	0.1%	
% saturation accuracy	±0.59% of reading	
Stabilization time	T90% at 10 seconds (when the solution is stirred)	
Temperature resolution	0.1 °C (0.18 °F)	
Temperature accuracy	±0.3 °C (±0.54 °F)	
Pressure resolution	5 mbar (0.5 kPa)	
Pressure accuracy	±3 mbar (0.3 kPa)	
Operating temperature	0 to 50 °C (32 to 122 °F)	
Storage temperature	5 to 40 °C (41 to 104 °F)	
Minimum immersion depth	10 mm (0.394 in.)	
Dimensions	Diameter: 15.875 mm (0.625 in.) Length: 215 mm (8.46 in.) Cable length: 1.8 m (5.9 ft)	
Cable connection	M12 digital output and connector	
Warranty	1 year on the probe. This warranty covers manufacturing defects, but not improper use or wear.	
Certifications	CE, FCC/ISED	

Section 3 Safety information

3.1 Intended use

The Intellical probes are intended for use by individuals who measure water quality parameters in the laboratory or in the field. The Intellical probes do not treat or alter water.

3.2 Use of hazard information

▲ DANGER

Indicates a potentially or imminently hazardous situation which, if not avoided, will result in death or serious injury.

▲ WARNING

Indicates a potentially or imminently hazardous situation which, if not avoided, could result in death or serious injury.

▲ CAUTION

Indicates a potentially hazardous situation that may result in minor or moderate injury.


NOTICE

Indicates a situation which, if not avoided, may cause damage to the instrument. Information that requires special emphasis.



3.3 ^{Section 7} Precautionary labels

TDEC Fleming Training Center

Read all labels and tags attached to the instrument. Personal injury or damage to the instrument could occur if not observed. A symbol on the instrument is referenced in the manual with a precautionary statement.

	Electrical equipment marked with this symbol may not be disposed of in European domestic or public disposal systems. Return old or end-of-life equipment to the manufacturer for disposal at no charge to the user.
-----------------------------------------------------------------------------------	---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------

3.4 Product hazards

▲ CAUTION	
	Chemical exposure hazard. Obey laboratory safety procedures and wear all of the personal protective equipment appropriate to the chemicals that are handled. Refer to the current safety data sheets (MSDS/SDS) for safety protocols.
▲ CAUTION	
	Chemical exposure hazard. Dispose of chemicals and wastes in accordance with local, regional and national regulations.

Section 4 Preparation for use

Prepare the probe for calibration and measurement as follows. Do not touch the protective black layer on the LBOD sensor cap.

1. Open the pressure sensor module cap and make sure that the label on the iButton is on top.
2. Rinse the probe with deionized water. Blot dry with a lint-free cloth.
3. Make sure that the meter has the correct date and time settings. The service-life time stamp in the probe comes from the date and time settings in the meter.

Note: *Some meters automatically open the date and time settings when the meter starts for the first time, or after battery replacement.*

4. Connect the probe to the meter.

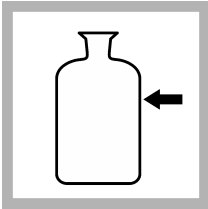
The procedure that follows is applicable to meters that can connect to Intellical LBOD probes. Refer to the applicable meter documentation for meter operation and probe-specific settings.

5.1 Calibration notes

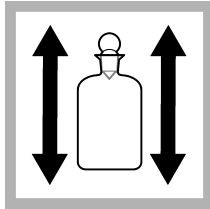
Read the notes that follow before calibration.

- Measure samples as soon as possible after collection.
- Use the single display mode for calibration when more than one probe is connected to the meter (if applicable).
- Calibrate the probes and verify the calibration regularly for best results. Use the meter to set calibration reminders.
- The calibration data is stored in the probe. When a calibrated probe is connected to a different meter with the same calibration options, a new calibration is not necessary.
- Air bubbles below the sensor when in solution can cause a slow response or error in the calibration. Make sure to remove air bubbles during calibration.
- The meter uses the slope value shown at the end of the calibration to monitor the condition of the sensor cap.

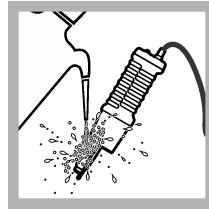
5.2 LBOD water-saturated air (100%) calibration procedure



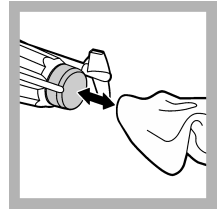
1. Fill a BOD bottle approximately $\frac{3}{4}$ full with water.



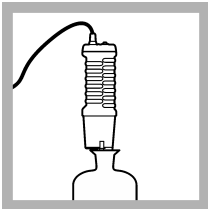
2. Put a stopper on the bottle and shake for 30 seconds.



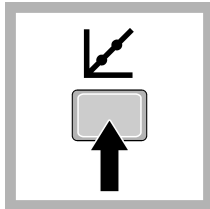
3. Rinse the probe with deionized water. Dry the probe with a lint-free cloth.



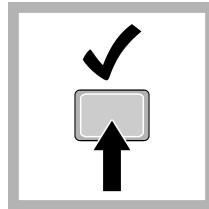
4. Carefully dry the LBOD sensor cap with a non-abrasive cloth.



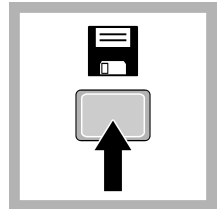
5. Put the probe in the bottle. Wait approximately 10 minutes for the contents to adjust to ambient temperature.



6. Go to the calibrate menu. Select the probe, if applicable.



7. Read the dissolved oxygen value. The display shows 100% when the reading is stable.



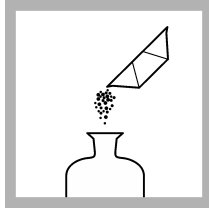
8. Save the calibration.

5.3 Zero-point calibration procedure

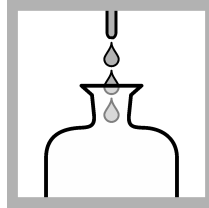
A zero-point calibration can increase the measurement accuracy of samples that have less than 1 mg/L O₂. A zero point calibration is not recommended for samples that have more than 1 mg/L O₂. Set the calibration mode to "100% with 0". The meter starts the calibration with the 100% measurement, then continues to the zero-point measurement.



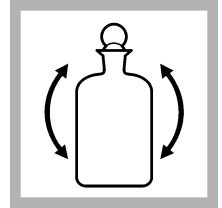
1. Fill a BOD bottle with deionized water.



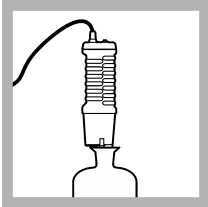
2. Add 300 mg of sodium sulfite to the bottle.



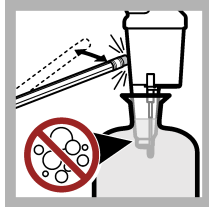
3. Add 2 mL of cobalt chloride solution to the bottle.



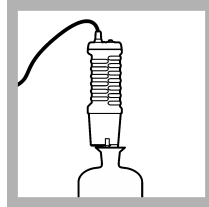
4. Put a stopper on the bottle and fully mix for 30 seconds.



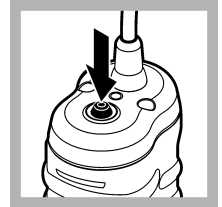
5. Remove the stopper. Put the probe in the bottle.



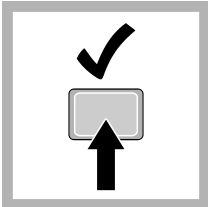
6. Tap the probe to remove air bubbles.



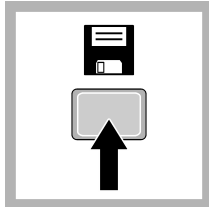
7. Wait approximately 10 minutes for the contents to adjust to ambient temperature.



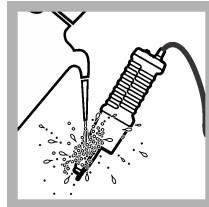
8. Start the stirrer.



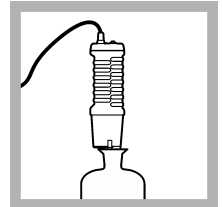
9. Read the dissolved oxygen value. Wait for the measurement to become stable.



10. Save the calibration.



11. Rinse the probe with deionized water. Dry the probe with a lint-free cloth.



12. Fill a BOD bottle with deionized water. Put the probe in the bottle and stir for 10 minutes to fully remove the sulfite from the probe.

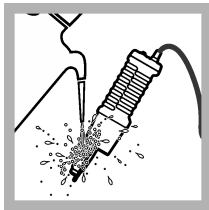
The procedure that follows is applicable to meters that can connect to Intellical LBOD probes. Refer to the applicable meter documentation for meter operation and probe-specific settings.

6.1 Sample measurement notes

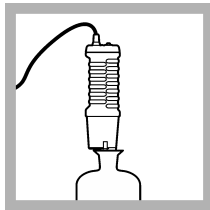
Read the notes that follow before sample measurements.

- Salinity changes the solubility of oxygen in water. Measure the sample salinity and enter the value in the probe settings of the meter.
- High concentrations (more than 1 molar) of acids or bases will decrease the service life of the LBOD sensor cap.
- Rinse the probe with deionized water and dry with a lint-free cloth between measurements to prevent contamination.
- If complete traceability is necessary, enter a sample ID and operator ID before measurement. Refer to the meter manual for instructions.
- The meter automatically saves the measurement data when the user manually reads each data point and when the meter is set to read at regular intervals. The user must manually save each data point when the meter is set to read continuously.
- Air bubbles below the sensor can cause a slow response or error in the measurement. Make sure to remove air bubbles before and during measurements.

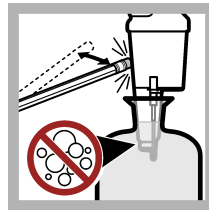
6.2 Sample measurement procedure



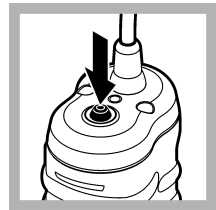
1. Rinse the probe with deionized water. Dry the probe with a lint-free cloth.



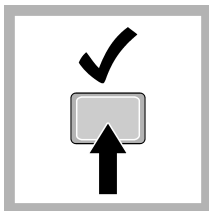
2. Put the probe in the BOD sample.



3. Tap the probe to remove air bubbles.



4. Start the stirrer.



5. Read the dissolved oxygen value of the sample. The display shows the dissolved oxygen value when the reading is stable.

6.3 Low-level measurements

Section 7

TDEC Fleming Training Center

If samples are expected to have less than 1 mg/L O₂, calibrate the probe with a zero-oxygen solution. A zero-point calibration is not recommended for samples that have more than 1 mg/L O₂. Use the guidelines and steps that follow only when the sample is expected to have less than 1 mg/L O₂.

- Calibrate the probe with a zero-oxygen solution. Use the "100% with 0" calibration option in the meter. Measure the 100% value first, then measure the zero-oxygen solution. Refer to [Zero-point calibration procedure](#) on page 7.
- Measure the salinity of the sample and enter the salinity correction factor in the probe settings.
- Wait approximately 10 minutes for the measurement contents to adjust to ambient temperature, then measure the sample. Measure the sample again to make sure the result is the same.
- Clean the probe regularly. Refer to [Clean the probe](#) on page 9.

Section 7 Maintenance

7.1 Clean the probe

NOTICE

The LBOD sensor cap has a protective black layer to increase the life of the LBOD sensor. Do not rub the black layer to clean the LBOD sensor cap. Do not use alcohol or other organic solvents to clean the LBOD sensor cap.

Keep the LBOD sensor cap clean for best results. Use only water and neutral detergents to clean the probe.

1. Put the probe in a neutral cleaning solution and stir the solution. Do not rub or remove the black layer on the LBOD sensor cap.
2. Rinse the probe with deionized water. Blot dry with a lint-free cloth.

7.2 Replace the sensor cap and iButton

NOTICE

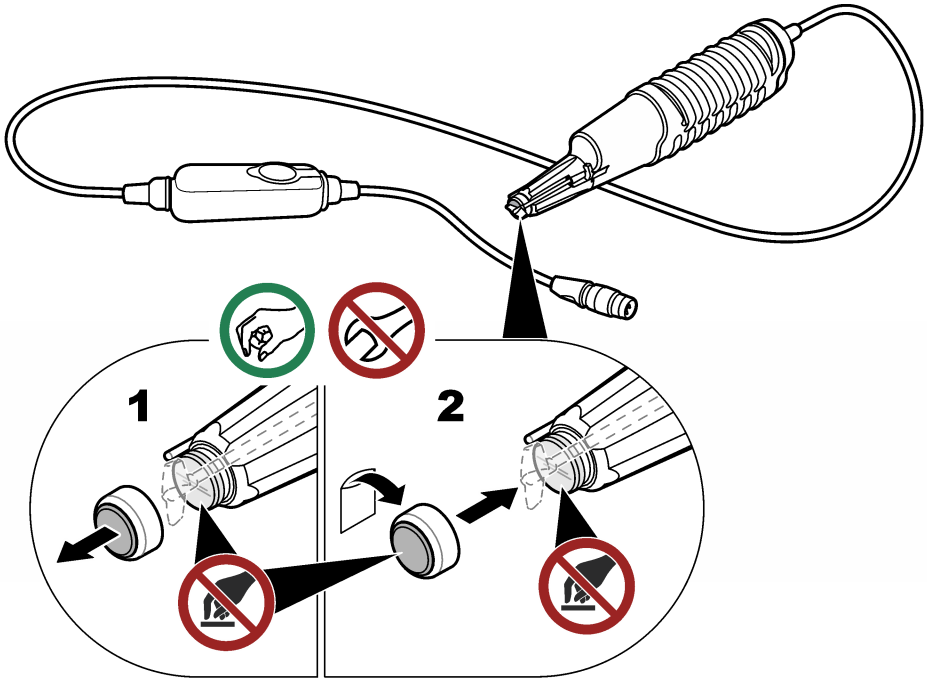
Do not use sharp metal tools to remove the LBOD sensor cap.

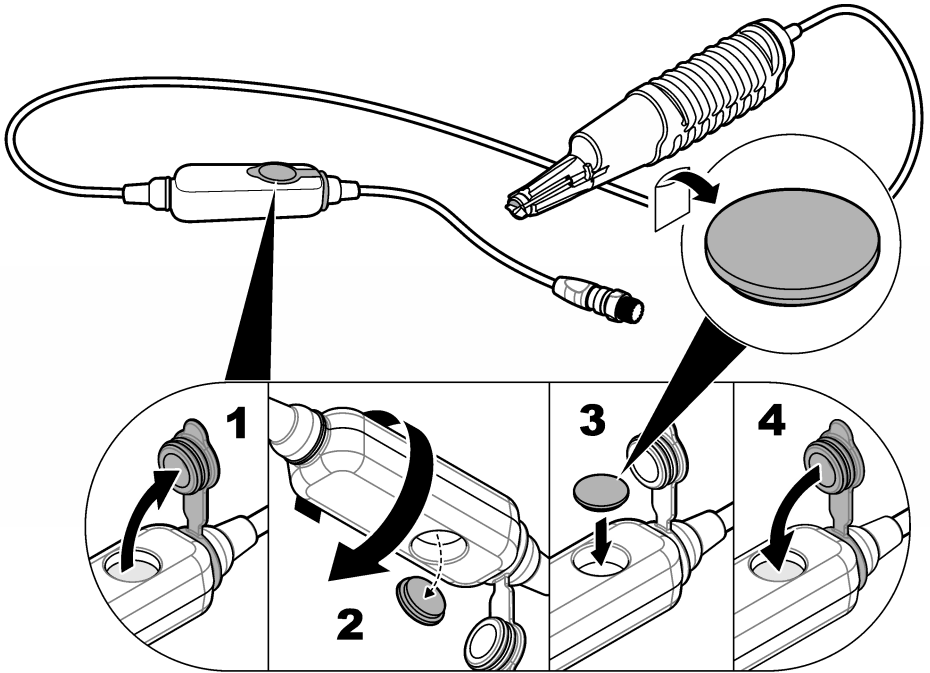
Replace the LBOD sensor cap after 365 days, or more frequently if the cap becomes damaged or dirty. The LBOD sensor cap and iButton operate together and must be replaced at the same time.

1. Use fingers to remove the LBOD sensor cap from the probe. Do not touch the probe lens. Refer to [Figure 2](#). Discard the used LBOD sensor cap.
Note: *The stirrer assembly can stay installed.*
2. If water is between the LBOD sensor cap and the probe lens, or if the probe lens looks dirty, rinse with dilute isopropyl alcohol (10% or less) or deionized water and blot dry with a non-abrasive cloth. Do not wipe the probe lens or use abrasive cleaners.
3. Hold the new LBOD sensor cap by the sides and install the new cap on the probe. Refer to [Figure 2](#). Make sure the LBOD sensor cap is on tightly.

Note: *Do not touch the protective black layer on the LBOD sensor cap.*

4. Install the new iButton® in the pressure sensor module. Refer to [Figure 3](#). Make sure that the label on the iButton is on top when the cap is open. Make sure to fully close the cap.

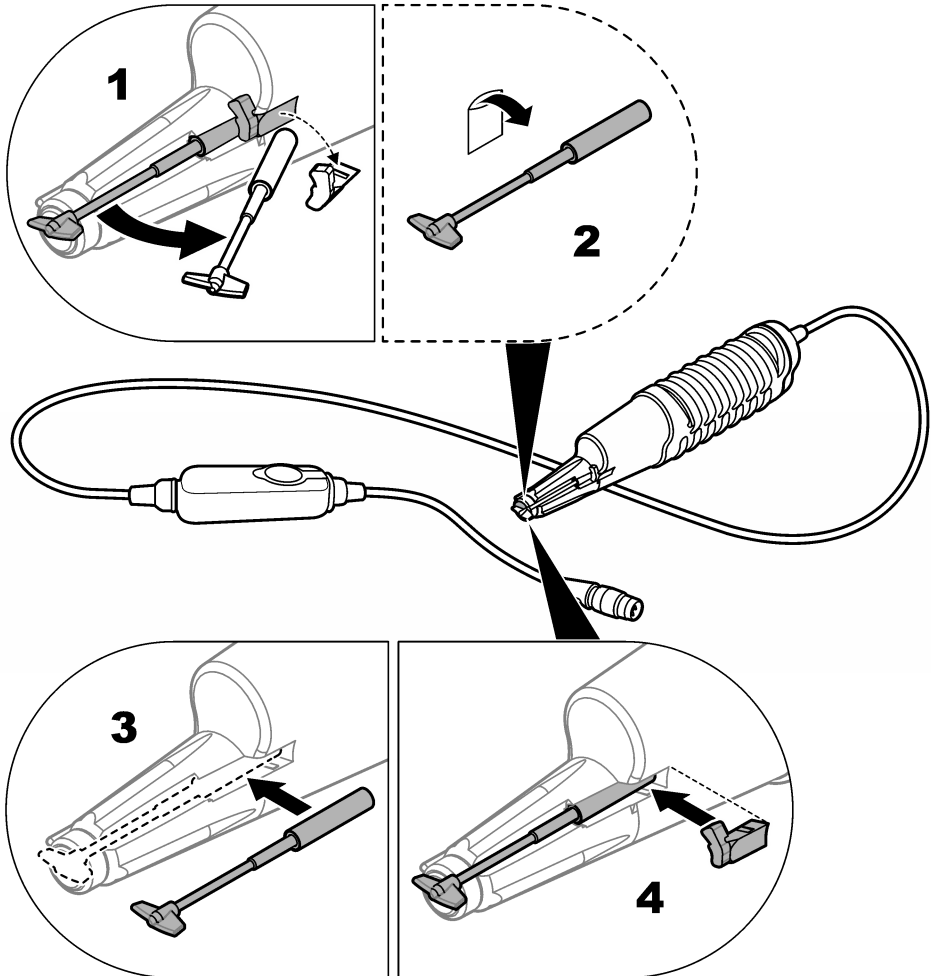




7.3 Replace the stirrer

Refer to [Figure 4](#) to replace the stirrer assembly.

Figure 4 Replace the stirrer



7.4 Storage

Keep the probe in a BOD bottle with some water (fill to $\frac{1}{4}$ minimum) when not in use.

Section 8 Troubleshooting

Section 7

TDEC Fleming Training Center

Problem	Possible cause	Solution
Decreased probe performance causes slow stabilization and prevents accurate calibrations or measurements.	The sensor cap is loose or damaged.	Tighten or replace the sensor cap. Always replace the sensor cap and the iButton at the same time. Refer to Replace the sensor cap and iButton on page 9.
	Water is between the sensor cap and the probe lens.	Remove the sensor cap and dry the probe lens. Refer to Replace the sensor cap and iButton on page 9.
	The sensor cap is not sufficiently conditioned.	Keep the probe in the BOD bottle with some water for more time, then try the calibration again.
	The temperature or pressure sensor does not operate correctly.	Compare the temperature and pressure readings of the probe with external measurements. The pressure sensor reads absolute pressure, which is not adjusted to sea level. If the measurements are not correct, contact technical support.
	The lot code on the iButton is not the same as the lot code on the sensor cap.	Replace the sensor cap and iButton.
The stirrer does not operate correctly.	The stirrer does not operate.	The stirrer uses the power from the meter to operate. Make sure to start the meter, then start the stirrer.
	The stirrer turns only when the probe is not in a BOD bottle.	Remove and install the stirrer assembly. Refer to Replace the stirrer on page 12.
	The stirrer makes a lot of noise.	Remove and install the stirrer assembly. Refer to Replace the stirrer on page 12.
Sample properties cause slow stabilization or inaccurate measurements.	The measurement was not adjusted for salinity in the sample.	Measure the salinity of the sample and enter the value as a salinity correction factor in the meter.
Procedure problem causes slow stabilization and prevents accurate calibrations or measurements.	Air bubbles are around or below the probe tip.	Carefully tap or shake the probe to remove air bubbles.
	The contents of the BOD bottle did not fully adjust to ambient temperature.	Wait more time for the contents of the BOD bottle to adjust to ambient temperature.

Section 9 Consumables

TDEC Fleming Training Center

Section 7

Note: Product and Article numbers may vary for some selling regions. Contact the appropriate distributor or refer to the company website for contact information.

Description	Quantity	Item no.
LBOD sensor cap replacement kit (includes iButton)	1	5838000
LBOD probe replacement stirrer assembly	1	5850800
LBOD stirrer bearing clip	1	5852800
BOD bottle with glass stopper, 300 mL	1	62100
Beaker, 250 mL, polypropylene	1	108046
Cobalt chloride solution	500 mL	1422249
Sodium sulfite	100 g	2386026
Disposable wipes, 11 x 22 cm	280/pkg	2097000
Wash bottle, polyethylene, 500 mL	1	62011

**HACH COMPANY World Headquarters**

P.O. Box 389, Loveland, CO 80539-0389 U.S.A.
Tel. (970) 669-3050
(800) 227-4224 (U.S.A. only)
Fax (970) 669-2932
orders@hach.com
www.hach.com

HACH LANGE GMBH

Willstätterstraße 11
D-40549 Düsseldorf, Germany
Tel. +49 (0) 2 11 52 88-320
Fax +49 (0) 2 11 52 88-210
info-de@hach.com
www.de.hach.com

HACH LANGE Sàrl

6, route de Compois
1222 Vézenaz
SWITZERLAND
Tel. +41 22 594 6400
Fax +41 22 594 6499

Hach Method 10360
Luminescence Measurement of Dissolved Oxygen in Water and Wastewater and
for Use in the Determination of BOD₅ and cBOD₅
Revision 1.2
September 2011

1.0 Scope and Application

- 1.1 This method is for the measurement of dissolved oxygen (DO) in surface and ground water, and municipal and industrial wastewater.
- 1.2 The method may be used as a replacement for the modified Winkler and membrane electrode procedures for the measurement of DO in wastewater treatment processes such as aeration and biological nutrient basins, effluent outfalls, receiving water, and in Biochemical Oxygen Demand (BOD) determinations where it is desired to perform nondestructive DO measurements.
- 1.3 The method is for use in the United States Environmental Protection Agency's (EPA's) survey and monitoring programs for the measurement of DO and for the determination of BOD and cBOD under the Clean Water Act.
- 1.4 This method is capable of measuring DO in the range of 0.20 to 20 mg/L.
- 1.5 Calibration is by single-point water-saturated air (100% saturation).

2.0 Summary of Method

- 2.1 This luminescence-based sensor procedure measures the light emission characteristics from a luminescence-based reaction that takes place at the sensor-water interface. A light emitting diode (LED) provides incident light required to excite the luminophore substrate. In the presence of dissolved oxygen the reaction is suppressed. The resulting dynamic lifetime of the excited luminophore is evaluated and equated to DO concentration.

3.0 Interferences

- 3.1 There are no known interferences at normal wastewater concentrations that interfere with DO detection and quantification with this method.

4.0 Safety

- 4.1 This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of any chemicals specified in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses. Additional information on laboratory safety can be found in References 15.5-15.6.

5.0 Equipment for the Measurement of Dissolved Oxygen

- 5.1 BOD bottle 300-mL with stoppers and plastic caps (Hach # 62016 and 241906)
- 5.2 Magnetic Stirring plate (optional)
- 5.3 Magnetic stirring device (optional)
- 5.4 BOD Bottle, glass stopper, 300-mL (Hach Catalog Number 62106)
- 5.5 Pipette, serological, 1-mL (Hach Catalog Number 919002)
- 5.6 Pipette, serological, 5-mL (Hach Catalog Number 53237)

- 5.7 Pipette, serological, 10-mL (Hach Catalog Number (53238)
- 5.8 Pipette Filler (Hach Catalog Number 1218900)
- 5.9 Meter and LBOD Probe (Hach Catalog Number HQDBOD01, for DO measurement in BOD bottles
- 5.10 Meter and LDO Probe (Hach Catalog Number 8506300 for DO measurement in open containers and water bodies
- 5.12 Temperature controlled environment for BOD bottle incubation, 20 ± 1 °C.

6.0 REAGENTS

- 6.1 Buffer Solution, APHA, for BOD, pH 7.2, Phosphate type, (Hach Catalog Number 43149)
- 6.2 Calcium Chloride Solution, APHA, for BOD (Hach Catalog Number 42849)
- 6.3 Ferric Chloride Solution, APHA, for BOD (Hach Catalog Number (42953)
- 6.4 Glucose-glutamic Acid, Standard Solution, Voluette™ Ampoule, 300-mg/L (150 mg/L glucose and 150 mg/L glutamic Acid), 10 mL (Hach Catalog Number 1486510) or, ezGGA Ampoules, 450 mg/L (225 mg/L Glucose and 225 mg/L Glutamic Acid, (Hach Catalog Number 25144-20)
- 6.5 Magnesium Sulfate Solution, APHA, (Hach Catalog Number (43094)
- 6.6 Nitrification Inhibitor (Hach Catalog Number 2845425)
- 6.7 Potassium Iodide Solution (Hach Catalog Number 1228949)
- 6.8 Sodium Thiosulfate Solution – 0.025 N (Hach Catalog Number 35253.
- 6.9 Sodium Hydroxide Solution, 1 N (Hach Catalog Number 104532)
- 6.10 Sodium Hydroxide Pellets, ACS (Hach Catalog Number 18734)
- 6.11 Starch Indicator (Hach Catalog Number 35253)
- 6.12 Sulfuric Acid Solution, 0.020 N (Hach Catalog Number 104532)
- 6.13 Sulfuric Acid Solution, 1.000 N (Hach Catalog Number 127053)

7.0 Standards

7.1 *Initial LDO/LBOD Probe Calibration*

- 7.1.1 Add approximately 1 inch (2.54 cm) of reagent water to a clean BOD bottle and stopper.
- 7.1.2 Shake vigorously for ~ 10 seconds.
- 7.1.3 Allow for the BOD bottle and its contents to equilibrate to room temperature.
- 7.1.4 The stopper may now be removed from the BOD bottle and the probe inserted for calibration purposes.

7.1.5 The luminescence technology for measuring dissolved oxygen is a superior technique from that of Winkler titration and membrane potentiometric measurement and has no interferences associated with oxygen detection process (EPA Validation study, 2004). Therefore, do not adjust the luminescence measurement to that of Winkler or membrane readings.

Note: Section 7.1 is a suggested procedure for the preparation of. Other procedures for the preparation of water-saturated air water-saturated air may be equally effective.

7.2 Calibration Verification, Initial Precision and Recovery, and On-going Precision and Recovery

7.2.1 Add approximately 1500 mL of organic-free water or BOD dilution water to a 2-L beaker or PET bottle.

7.2.2 Allow the water to equilibrate to room temperature ($\pm 2^\circ \text{C}$).

7.2.3 With a steady stream of filtered air ($\approx 10 - 40 \text{ mL per minute}$), aerate the water for a minimum of 30 minutes. Alternatively, vigorously shake the reagent water or BOD dilution water for several minutes.

7.2.4 At the completion of aeration, let water re-equilibrate to room temperature ($\pm 2^\circ \text{C}$) for 30 minutes and note the barometric pressure of the laboratory during preparation.

7.2.5 Transfer the aerated water to a BOD bottle until overflowing and stopper.

7.2.6 Calculate the theoretical dissolved oxygen concentration using a dissolved oxygen table such as Hitchman (1978).

Note: Section 7.2 is a suggested procedure for the preparation of air-saturated water. Other procedures for the preparation of air saturated-water may be equally effective.

8.0 Sample Collection Preservation and Storage

8.1 See Title 40 of the Code of Federal Regulations Part 136.3, Table II (Section 15.3) for information regarding required sample collection containers, preservation techniques and holding times for collection of water for measurement of DO and for the determination of BOD and cBOD.

9.0 Quality Control

9.1 It is recommended that each laboratory that uses this method be required to operate a formal quality assurance program (Reference 15.1). The minimum requirements of this program consist of an initial demonstration of laboratory capability and ongoing analyses of laboratory prepared water standards as a test of continued performance to assess accuracy and precision. Laboratory performance is compared to established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1.1 The analyst shall make an initial demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 9.2.

9.1.2 The laboratory shall, on an ongoing basis, demonstrate through calibration verification and analysis of the ongoing precision and recovery sample that the analysis system is in control. These procedures are described in Sections 9.3 and 9.4, respectively.

9.1.3 Accompanying QC for the determination of DO is required per analytical batch. An analytical batch is a set of samples processed during a contiguous 8-hour period. Each analytical

batch should be accompanied by a calibration verification and ongoing precision and recovery sample, resulting in a minimum of three analyses (1 CV, 1 sample, and 1 OPR).

9.2 Initial Demonstration of Laboratory Capability

9.2.1 Initial precision and recovery (IPR) - To establish the ability to generate acceptable precision and accuracy for the measurement of DO in water, the analyst shall perform the following operations:

9.2.1.1 Prepare and measure four samples of the IPR standard (Section 7.2) according to the procedure beginning in Section 10.

9.2.1.2 Using the results of the set of four analyses, compute the average percent recovery (X) and the standard deviation of the percent recovery (s) for DO. Use the following equation for calculation of the standard deviation of the percent recovery:

$$s = \sqrt{\frac{\sum x^2 - \frac{(\sum x)^2}{n}}{n-1}}$$

where:

n = Number of samples

x = Concentration in each sample

9.2.1.3 Compare *s* and *X* with the corresponding limits for initial precision and recovery in Tables 5 and 6. If *s* and *X* meet the acceptance criteria, system performance is acceptable and analysis of samples may begin. If, however, *s* exceeds the precision limit or *X* falls outside the range for recovery, system performance is unacceptable. In this event correct the problem, and repeat the test.

9.3 Calibration Verification

9.3.1 Upon air calibration, prepare a calibration verification standard (Section 7.2) with each analytical batch. Analyze according to the procedure beginning in Section 10 and compare the recovery results to those in Tables 5 and 6.

9.4 Ongoing Calibration and Precision and Recovery

9.4.1 To demonstrate that the analysis system is in control, and acceptable precision and accuracy is being maintained with each analytical batch, the analyst shall perform the following operations

9.4.2 Prepare a precision and recovery standard (Section 7.2) with each analytical batch according to the procedure beginning in Section 10.

9.4.3 Initially, at the end of each analytical batch of samples, analyze a precision and recovery standard and compare the concentration recovery with the limits for ongoing precision and recovery in Tables 5 and 6. If the recovery is in the range specified, measurement process is in control and analysis of samples may proceed. If, however, the recovery is not in the specified range, the analytical process is not in control. In this event, correct the problem,

recalibrate and verify the calibration and reanalyze analytical batch, repeating the ongoing precision and recovery test.

- 9.4.4 The laboratory should add results that pass the specification in Tables 5 and 6 to IPR and previous OPR data and update QC charts to form a graphic representation of continued laboratory performance. The laboratory should also develop a statement of laboratory data quality for each analyte by calculating the average percent recovery (R) and the standard deviation of the percent recovery (sr). Express the accuracy as a recovery interval from $R - 2sr$ to $R + 2sr$. For example, if $R = 95\%$ and $sr = 5\%$, the accuracy is 85% to 105%.
- 9.5 Depending upon specific program requirements, field replicates may be required to assess the precision and accuracy of the sampling and sample transporting techniques.
- 9.6 Glucose-Glutamic Acid Seed Strength Check
- 9.6.1 Many factors can influence the BOD analysis (toxicity from sample matrix, contaminated dilution water, poor quality seed, etc. In order to insure sufficient seeding in the BOD test, a glucose-glutamic acid check is performed in parallel with BOD₅ test. Well prepared dilution water and an active seed will produce a BOD₅ of 198 ± 30 mg/L BOD.

9.6.1.1 Prepare in triplicate a 300-mL BOD bottle with 3.0 mL of the 300 mg/L Standard Solution of GGA (Section 6.4) or 1 ampoule (2.0 mL) of ezGGA (Section 6.4).

9.6.1.2 When using ezGGA ampoules, place the ezGGA ampoule in the ampoule breaker (provided with ezGGA ampoules) and rinse the assembly with reagent water. Hold the ampoule and breaker over the rim of the BOD bottle, break and allow the ampoule to fall into the BOD bottle. Leave the ampoule in the BOD bottle during the incubation period and reading of DO.

Standard Solution

$$\frac{3.0 \text{ mL} \times 0.300 \text{ mg/mL GGA}}{300 \text{ mL final volume}} \times 1000 \text{ mL/L} = 3.0 \text{ mg/L GGA per bottle}$$

ezGGA Ampoule

$$\frac{1 \text{ ampoule (2.0 mL)} \times 0.450 \text{ mg/mL GGA}}{300 \text{ mL final volume}} \times 1000 \text{ mL/L} = 3.0 \text{ mg/L GGA per bottle}$$

9.6.1.2 Add seed at three different volumes (typically 4 mL, 6 mL, and 8 mL,) to the GGA bottles. Other volumes may be required, depending on the strength of the seed being used.

9.6.1.3 Bring to volume with dilution water and analyze as described in Sections 12.9 and 12.10.

Note: GGA BOD₅ recovery results outside of 198 ± 30 mg/L should be investigated as to causation. If toxicity of dilution water has been ruled out as a probable cause for low recovery, it is likely that the seed is of low activity or poor quality. Either increase the seed amount or use a seed of higher quality. High GGA recoveries are generally due to incorrect amount of GGA Standard Solution.

10.0 Calibration and Standardization

- 10.1 Because of the possible diversity of future LDO instrument hardware and, no detailed operating conditions are provided. The analyst is advised to follow the recommended operating conditions provided by the manufacturer. It is the responsibility of the analyst to verify that the instrument

configuration and operating conditions satisfy the analytical requirements of this method and to maintain quality control data verifying instrument performance and analytical results.

10.2 Water-saturated air (Section 7.1) is used for instrument calibration.

10.3 Calibration verification (Section 7.2) is performed with air-saturated water prior to any DO sample measurements to the method specifications.

11.0 Procedure for Measuring DO in Grab Samples, Outfalls, and Open Water Bodies

11.1 Instrument Setup

11.1.1 Follow the instrument manufacturer's instructions for instrument setup (Hach Document DOC022.53.80021 for Hach LDO IntelliCal™ Rugged and Standard Probes, Hach Catalog Number 5790018 for Hach LDO process probes.

Note: Manufacturer's instructions are only for instrument set and use. These instructions do not preclude the calibration and performance requirements in this method.

11.2 Measurement of DO

11.2.1 Insert the LDO probe into the water sample to be measured and stir gently probe with or add a stir bar. Do not put the probe on the bottom or sides of the container. Stir the sample at a moderate rate or put the probe in flowing conditions. Read sample. The display will show "Stabilizing" and a progress bar as the probe stabilizes in the sample. The display will show the lock icon when the reading stabilizes.

11.3.1 Insert the LBOD probe into the BOD bottle of the prepared samples for BOD or cBOD determination. Insure that there are no air bubbles that may have collected around the probe or sensor. Turn on the stir paddle and read sample. The display will show "Stabilizing" and a progress bar as the probe stabilizes in the sample. The display will show the lock icon when the reading stabilizes.

12.0 Procedure for the Preparation and Determination of BOD₅ and cBOD₅ Samples

12.1 Follow the instrument manufacturer's instructions for instrument setup for the LBOD probe (Hach Document DOC022.53.80025).

12.2 The BOD test is a 5-day test. Follow all steps carefully to make sure that the test does not have to be repeated.

12.3 The dilution water for this test must be fully air-saturated and not have an oxygen demand or any toxins. When incubated for 5 days at 20 °C, the dissolved oxygen concentration in the dilution water must not change by more than 0.2 mg/L.

12.4 Distilled Water Preparation

12.4.1 The dilution water must be prepared very carefully to make sure that no source of oxygen demand or toxins are added. The water that is used to prepare the dilution water must be of very high quality. The water must not have any organic compounds or any toxic compounds such as chlorine, copper, and mercury at a concentration level that would interfere would the BOD seed and inhibit microbiological growth of organisms.

- 12.4.2 For best results, use an alkaline permanganate distillation for preparing dilution water. Resin in ionization cartridges will occasionally release organic materials that have an oxygen demand.
- 12.4.3 Store the distilled water in clean jugs at a temperature of 20 °C. Fill the containers to about $\frac{3}{4}$ full and shake the jugs to saturate the water with air. Alternatively, saturate the water with air as described in Section 7.2.1. A small aquarium pump or air compressor can be used to saturate the water with air. Insure that the air is filtered and that the air filter does not grow bacteria.

12.5 Dilution Water Preparation

- 12.5.1 Using the distilled water prepared above, select a BOD nutrient buffer pillow from the BOD nutrient buffer pillows table.
- 12.5.2 Add the contents of the BOD Nutrient Buffer Pillow to the distilled water in a jug with ample headspace. Cap the jug and shake vigorously for one minute to dissolve the nutrients and to saturate the water with air.
- 12.5.3 Alternatively, prepare the dilution water by adding 1 mL each of the following solutions per liter of distilled water prepared in Section 12.4.
- Buffer Solution, APHA, for BOD, pH 7.2, Phosphate type, (Hach Catalog Number 43149)
- Calcium Chloride Solution, APHA, for BOD (Hach Catalog Number 42849)
- Ferric Chloride Solution, APHA, for BOD (Hach Catalog Number (42953)
- Magnesium Sulfate Solution, APHA, (Hach Catalog Number (43094)
- 12.5.4 Cap the jug and shake vigorously for one minute to dissolve the nutrients and to saturate the water with air.

12.6 Seed Preparation

- 12.6.1 Use raw sewage or other reliable sources for the bacterial seed that will yield 198 ± 30 mg/L BOD with the GGA check sample in Section 9.6. Potential seed sources include wastewater influent, primary effluent, soil, and domestic sewage.
- 12.6.2 Allow raw sewage to stand undisturbed at 20 °C for 24 to 36 hours before use.
- 12.6.3 When seeding samples with raw sewage, always pipette from the upper portion of the sewage.

12.7 Sample Size Selection Guide

- 12.7.1 Make an estimate of the sample volumes that are necessary for the test. At least 2.0 mg/L of DO should be consumed during the test and at least 1.0 mg/L of un-depleted DO should remain in the bottle.
- 12.7.2 Samples such as raw sewage will have a high BOD. Small sample volumes must be used because large samples will deplete all of the oxygen in the sample. Samples with a low BOD must use larger sample volumes to insure that adequate oxygen is depleted to give accurate results.

- 12.7.3 Refer to the Minimum Sample Volume Table to select the minimum sample volume. For example, if a sewage sample is estimated to contain 300 mg/L BOD, the minimum sample volume is 2 mL. For sewage effluent with an estimated BOD of 40 mg/L, the minimum sample volume is 15 mL.
- 12.7.4 Refer to the Maximum Sample Volume Table to select the maximum sample volume. At 1000 in elevation, with an estimated BOD of 300 mg/L, the largest sample volume is 8 mL. For a BOD of 40 mg/L, the maximum volume of sample is 60 mL.

12.8 *Sample Matrix Pretreatment*

- 12.8.1 Determine the pH of each sample prior to BOD sample preparation. For samples that of have pH of less than 6.0 or greater than 8.5, adjust the pH accordingly with a solution of sulfuric acid (H₂SO₄) or sodium hydroxide (NaOH). Strength of pH adjustment solution should be at a concentration that does not dilute the sample by greater than 0.5 percent.
- 12.8.2 For sample matrices that contain residual chlorine, de-chlorinate with a solution of (Na₂S₂O₃).
- 12.8.2.1 Measure 100 mL of sample into a 250 mL Erlenmeyer flask. Using a 10-mL serological pipette and pipette filler, add 10 mL of 0.020 N Sulfuric Acid Standard Solution and 10 mL of Potassium Iodide Solution, 100-g/L, to the flask.
- 12.8.2.2 Add three full droppers of Starch Indicator Solution and swirl to mix.
- 12.8.2.3 Fill a 25-mL burette with 0.025 N Sodium Thiosulfate Standard Solution and titrate the sample from dark blue to colorless.
- 12.8.2.4 Calculate the amount of 0.025 N Sodium Thiosulfate Solution to add to the sample:
- $$\text{mL } 0.025 \text{ N Sodium Thiosulfate required} = \text{mL titrant used} \times \text{volume of remaining sample divided by } 100$$
- 12.8.2.5 Add the required amount of 0.025 N Sodium Sulfate Standard Solution to the sample. Mix thoroughly and wait 10 to 20 minutes before performing the BOD test.

12.9 *Sample Preparation*

- 12.9.1 Select the sample volume as described in Section 12.7. Select a minimum of three different volumes for each sample.
- 12.9.1.1 If the minimum sample volume is 3 mL or more, determine the DO concentration in the undiluted sample; this determination can be omitted when analyzing sewage and settled effluents known to have a dissolved oxygen content near 0 mg/L.
- 12.9.2 Stir the sample gently with a pipette. Use the pipette to add the determined sample volumes to the BOD bottles.
- 12.9.3 Add the appropriate seed to the individual BOD bottles as described in Section 12.6.

- 12.9.3.1 Separately, with each batch of BOD samples, prepare a seed sample with dilution water. Measure the BOD of the seed for subtraction from the sample BOD.
- 12.9.3.2 A seed that has a BOD of 200 mg/L (a typical range for domestic sewage) will typically deplete at least 0.6 mg/L DO when added at a rate of 3 mL/L of dilution water.
- 12.9.4 If the test is for cBOD, add two portions of Nitrification Inhibitor (approximately 0.16 g) to each bottle. The oxidation of nitrogen-based compounds will be prevented.
- 12.9.5 Fill each bottle to just below the lip with dilution water.
- 12.9.5.1 Allow the dilution water to flow down the sides of the bottle to prevent air bottles from becoming entrapped in the bottle.
- 12.9.6 Fill an additional BOD bottle with only dilution water. This will be the dilution water blank.
- 12.9.7 Stopper the bottles carefully to prevent air bubbles from becoming entrapped.
- 12.9.7.1 Tightly twist the stopper and invert the bottles several times to mix.
- 12.10 *Sample Analysis*
- 12.10.1 Measure the initial dissolved oxygen concentration in each bottle with the LBOD probe.
- 12.10.2 After the initial DO measurement, stopper the bottles carefully to prevent air bubbles from becoming entrapped.
- 12.10.2.1 Add dilution water to the lip of each BOD bottle to make a water seal.
- 12.10.3 Place a plastic cap over the lip of each bottle and incubate at 20 ± 1 °C for five days.
- 12.10.4 After 5 days, measure the remaining dissolved oxygen concentration in each bottle with the LBOD probe.
- 12.10.4.1 At least 1.0 mg/L DO should have remained in each bottle.
- 12.10.4.2 Discard results of samples where the DO is depleted below 1. mg/L.

13.0 BOD and cBOD Calculations

13.1 *When Dilution Water Not Seed (generally influent and primary treated influent to treatment)*

$$\text{BOD}_5 \text{ or cBOD}_5, \text{ mg/L} = \frac{D_1 - D_2}{P}$$

where:

BOD₅ or cBOD₅ = BOD value from the 5-day test

D₁ = DO of diluted sample immediately after preparation, in mg/L

D₂ = DO of diluted sample after 5 day incubation at 20 °C, in mg/L

P = Decimal volumetric fraction of sample used

f = ratio of seed in diluted sample to seed in seed control (% seed in diluted sample/%seed in seed control) or, if seed material is added directly to sample or to seed-control bottles:

f = ((volume of seed in diluted sample/volume of seed in seed control)

13.2 When Dilution Water Requires Seed

$$\text{BOD}_5 \text{ or cBOD}_5, \text{ mg/L} = \frac{(D_1 - D_2) - (B_1 - B_2)f}{P}$$

where as defined above plus:

B_1 = DO of seed control before incubation, in mg/L

B_2 = DO of seed control after incubation, in mg/L

13.3 Averaged Results

13.3.1 Averaged results from different dilutions are acceptable if more than one sample dilution meets all of the following criteria:

13.3.1.1 The remaining un-depleted DO is at least 1 mg/L.

13.3.1.2 The final DO value is at least 2 mg/L lower than the initial prepared sample DO

13.3.1.3 There is no evidence of toxicity at higher sample concentrations

14.0 Method Performance for Dissolved Oxygen in Reference Water and GGA Recovery

Acceptance Criterion	Section	Limit
Initial DO Accuracy in Reagent Water	9.2.1	95% to 105%
Initial Precision in Reagent Water	9.2.1	2.1%
Average GGA Recovery	15.7	198 ± 30.5 mg/L BOD

15.0 Pollution Prevention

15.1 There are no standards or reagents used in this method when properly disposed of, pose any threat to the environment.

14.0 Waste Management

14.1 It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect air, water, and land by minimizing and control all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

14.2 For further information on waste management, consult "The Waste Management manual for Laboratory Personnel", and Less is Better: Laboratory Chemical Management for Waste Reduction", both available from the American Society's Department of Government relations and Science Policy, 1155 16th Street N.W., Washington, D.C. 20036.

15.0 References

15.1 Handbook of Analytical Quality Control in Water and Wastewater Laboratories," USEPA, EMSL-CI, Cincinnati, OH 45268, EPA-600-4-79-019, March 1979.

15.2 Hitchmen, M.L. (1978) *Chemical analysis*. Vol. 49. *Measurement of Dissolved Oxygen*. Wiley and sons, New York.

15.3 Title 40, Code of Federal Regulations (40 CFR), Part 136.

- 15.4 Protocol for EPA Approval of New Methods for Organic and Inorganic Analytes in Wastewater and Drinking Water (EPA-821-B-98-003, March 1999).
- 15.5 "OSHA Safety and Health Standards, General Industry," (29 CFR 1910), Occupational Safety and Health Administration, OSHA 2206 (Revised, January 1976)
- 15.6 "Safety in Academic Chemistry Laboratories," American Chemical Society, Committee on Chemical Safety, 3rd Edition, 1979.
- 15.7 "Standard Methods for the Examination of Water and Wastewater", 20th Edition; American Public health Association: 1015 Fifteenth Street, NW, Washington, D.C. 2005, 1998; Method 5210B.

16.0 Tables

16.1 Nutrient Buffer Preparation Options

Table 1 - BOD Nutrient Buffer Pillows

Volume of Dilution Water to Prepare	Hach BOD Nutrient Pillow Catalog Number
300 mL) add pillow to each BOD Bottle	1416066
3 liters	1486166
4 liters	2436466
6 liters	1486266
19 liters	1486398

Table 2 – Conventional Dilution Water Preparation Reagents

Hach Buffer Reagents	Hach Buffer Reagent Catalog Number
Buffer Solution, APHA, for BOD, pH 7.2, phosphate type, 500 mL	43149
Calcium Solution, Alpha, for BOD, 500 mL	42849
Ferric Chloride Solution, APHA, 1000 mL	42953
Magnesium Sulfate Solution, APHA, for BOD, 500 mL	43049

Note: The Phosphate Buffer Solution should be refrigerated to decrease the rate of biological growth.

16.2 Sample Volume Selection Guides

Table 3 - Minimum Sample Volume Selection Guide

Sample Type	Estimated BOD mg/L	Minimum Sample Volume (mL)
Strong Waste	600	1
Raw and Settled Sewage	300	2
	200	3
	150	4
	100	6
	75	8
	60	10
	50	12
Oxidized Effluents	40	15
	30	20
	20	30
	10	60
	6	100
Polluted River Water	4	200
	2	300

Table 4 – Maximum Sample Volume Selection Guide

BOD at Sea Level (mg/L)	BOD at 1000 ft Elevation (mg/L)	BOD at 5000 feet Elevation (mg/L)	Maximum Sample Volume (mL)
615	595	508	4
492	476	406	5
410	397	339	6
304	294	251	8
246	238	203	10
205	198	169	12
164	158	135	15
123	119	101	20
82	79	68	30
41	40	34	60
25	24	21	100
12	12	10	200
8	8	7	300

Note: Samples with higher concentrations of BOD should be pre-diluted.

16.3 Performance Criteria

Table 5 - Initial Precision and Recovery Method Performance

IPR Range	IPR DO Conc. (mg/L)	97.5% Lower Limit of Recovery (%)	97.5% Upper Limit of Recovery (%)	95% Upper Limit of Precision (%)
Low	1.72 – 1.74	95.0	104.0	2.07
High	7.22 – 9.23	95.8	104.8	1.26

Table 6 - Calibration Verification Performance

CV DO Concentration	Average % Recovery	% Standard Deviation	% Relative Standard Deviation
7.22 mg/L – 9.23 mg/L	100.1	2.5	2.5

17.0 Glossary of Definitions and Purposes

The definitions and purposes are specified to this method but have been conformed to common usage as much as possible.

17.1 Units of Weight and Measure and their Abbreviations

- 17.1.1 Symbols
°C degrees Celsius
- 17.1.2 Alphabetical characters
mg/L milligram per liter

17.2 Definitions, acronyms, and abbreviations

- 17.2.1 LDO[®] - Luminescence dissolved oxygen
- 17.2.2 BOD₅: Biological oxygen demand, 5-day test
- 17.2.3 cBOD₅: Carboneous biological oxygen demand, 5-day test
- 17.2.4 DO: Dissolved oxygen

17.2.5 CV: Calibration verification

17.2.6 IPR: Initial precision and recovery


17.2.7 OPR: On-going precision and recovery

Section 8

Sampling

SAMPLING

Basic Lab Class



1

Why Sample?

- Meet compliance requirements
- Process control
- Ensure public safety and protect the environment

2

Sampling Plan

- There are many questions to consider before actually collecting a sample
- The answer to these questions will help you put together a sampling plan

1. Why is the sample being collected?
2. What tests need to be run on the sample?
3. Where is the sample going to be collected from?
4. How is the sample going to be collected?
5. When does the sample need to be collected/analyzed?
6. Who is going to analyze the sample?

3

Considerations

- Collection
- Volume
- Storage and preservation
- Sample points
- Sampling frequency
- Include Sampling Plan in SOP

4

Grab Sample

- Single sample
- Represents portion of water or wastewater at any one time
 - NOT the average
- Residual chlorine, dissolved oxygen, coliforms, *E. coli*, pH and temperature

5

Composite Sample

- Collected at regular intervals
 - Once every 1-2 hours, over 24 hour period
 - Once collected, refrigerated at 4°C
- In proportion to existing flow
 - As flow increases, more samples are collected
- Combined to form sample representative of entire flow for period

6

Composite Sample

- Refrigerated and thoroughly mixed
- Measure flow and sample volume
- BOD, total N, settleable solids
- NEVER use composite sample for bacterial analysis

7

Sampling Guidelines

- Representative
- Proper container
- Do not contaminate the lid
- Preservative/ dechlorinating agent

8

Sampling Guidelines

- Hold by base
- Turn into current
- Avoid air bubbles
- Label containers with:
 1. Sample Location
 2. Date and Time of collection
 3. Name of collector
 4. Any other pertinent information

9

Sample Labeling

Location: 196 E. Main Street, Springfield, TN

Date / Time: 9/22/2018 @ 8:15 AM

Sampled by: BS (Bob Smith)

Comments: grab sample
pH < 2 with H₂SO₄ and stored at 4°C

10

Sample Labeling

- Special care must be taken not to contaminate samples.
 - This includes storing samples in a secure location to preclude conditions which could alter the properties of the sample. Samples shall be custody sealed during long-term storage or shipment.
- Documentation of field sampling is done in a bound logbook.

11

Chain of Custody

- Written record to trace possession and handling of samples from collection to reporting
 - In case of legal litigation
- Used when sending out samples to contract lab
- Should identify who handled sample from collection to transport to storage to analysis to destruction
 - Including dates, times, initials, addresses, etc.

12

Chain of Custody

- Collected samples are in the custody of the sampler or sample custodian until the samples are relinquished to another party.
 - If samples are transported by the sampler, they will remain under his/her custody or be secured until they are relinquished.

13

Sample Volume

- Depends on test procedure
- Headspace for mixing
- Preservative
- QA/QC comparisons

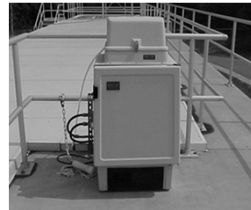
14

Sampling Point Selection

- Flow well mixed
- Exclude large particles (>1/4 inch)
- Exclude floating matter
- Readily accessible & in safe area

15

Sampling Devices



Automatic:

- Timesaver
- Composite: set to collect specific volumes over a period of time
- Clean intake line regularly to prevent growth of bacteria or algae

16

Sampling Devices

Manual:

- Dippers
- Weighted bottle sampler
- Whirl-pak® bags
- Jugs



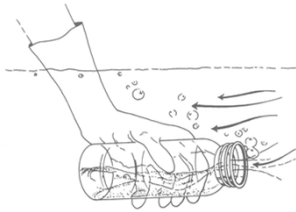
17

Sampling Devices

- Okay to improvise as long as the device can be properly cleaned
- Does this look like an approved device?

18

Subsurface Sampling

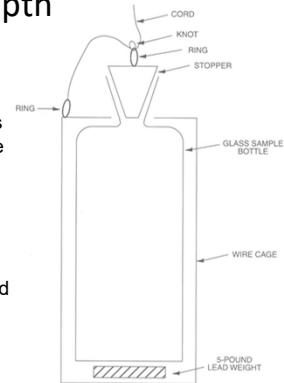


- Grasp container at base
- Plunge bottle mouth down into water
 - To avoid introducing any floating material
- Position mouth of bottle into current and away from hand
- Tip bottle slightly upward to allow air to exit so bottle can fill

19

Homemade Depth Sampler

- Collection from basins, tanks, lakes, reservoirs
- Pre-marked steel cable
- Pre-measured/marked rope
 - non-smearing ink/paint
- A jerk on the cord will remove the stopper and allow the bottle to fill



20

Sources of Error

- Improper sampling
- Poor or improper sample preservation
- Lack of sufficient mixing during compositing and testing

21

Preservation Techniques

- Refrigeration at 4°C
- pH<2:
 - * Using HCl
 - * Using H₂SO₄
 - * Using HNO₃
- pH>9 using NaOH
- pH>12 using NaOH

22

Preservation

- The less time that elapses between actual collection of sample and analysis = more reliable data
- Sample deterioration starts immediately after collection for most wastewaters
- Residual chlorine and temperature require immediate analysis

23

Drinking Water

- To collect samples from water main connections:
 - Flush the service line for a few minutes
- Do not take samples from drinking water fountains, restrooms, or taps that have aerators
 - Aerators can change pH, DO, or harbor bacteria
- Do not sample from taps with excessive foliage
- Never collect from a hose or other attachment
- Bottle must not come into contact with the faucet

24

Lead and Copper Rule

- Requires a “first draw” or “first flush” sample
- Water has stood motionless in pipe for at least 6 hours
- Take sample first thing in morning
- *Or* as soon as customer gets home from work

25

Any Questions?

26

Sampling – Review Questions

1. There are many things to take into consideration prior to collecting a sample. List 5 of those considerations.
2. What is the difference between a Grab sample and a Composite sample?
3. List some tests that require Grab samples.
4. List some tests that require Composite samples.
5. Automatic composite samplers must be periodically cleaned to prevent what?
6. What information should be included on the label that is attached to your sample container?

7. What is a Chain of Custody and when would you most likely encounter one as an operator?

8. Improvised sampling devices are allowed as long as they can be properly cleaned. True or False?

Section 9

Turbidity

TURBIDITY

Basic Lab Class



1

Turbidity

- ☐ A measure of the clarity of water
- ☐ It is an expression of the optical property that causes light to be scattered and absorbed in water
- ☐ It is caused by particulate, such as silt, clay, organic matter, algae, and other microorganisms
- ☐ Amount of light absorbed is proportional to the concentration of particulate in the sample

TDEC - Fleming Training Center

2

Turbidity

- ☐ Caused by suspended and colloidal matter in water
- ☐ It is an expression of light that is scattered or absorbed through a sample
- ☐ Does not indicate the number or size of particles in a sample
- ☐ General indicator of overall effluent water quality and a good process control test for operator

TDEC - Fleming Training Center

3

Importance

- ☐ Supports growth of microorganisms
- ☐ Reduces effectiveness of chlorination
- ☐ Interferes with chemical and microbiological analysis
- ☐ Is unacceptable for aesthetic reasons
- ☐ Is related to coagulation and filtration
- ☐ Is unacceptable for most industrial water

TDEC - Fleming Training Center

4

Measuring

- ☐ Use an instrument for measuring and comparing turbidity of liquids
- ☐ Nephelometers are instruments which measure turbidity by comparing the amount of light in a sample to the amount of light scattered by a standard
 - If the light detector is at an angle of 90° = Nephelometer
 - If the light detector is at an angle of 180° = Turbidimeter
- ☐ The amount of scattered light is measured and converted to units of turbidity or NTU's (Nephelometric Turbidity Units)

TDEC - Fleming Training Center

5

Instruments

Hach 2100N Laboratory
Benchtop Turbidimeter



Hach 2100Q
Portable Turbidimeter

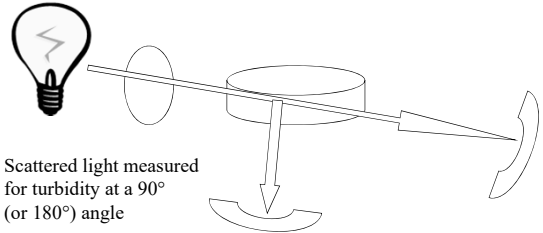
6

Instruments

Hach TU5 Series Benchtop
and Online Turbidimeter



Turbidimeters - 2100N

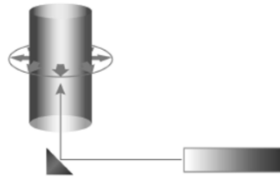


- Scattered light measured for turbidity at a 90° (or 180°) angle
- Light source from tungsten lamp passing through three precisely aligned lenses, the light is focused in a narrow, collimated beam

8

Turbidimeters - TU5 Series

- The light that is scattered at a 90° angle from the incident beam is reflected through a conical mirror in a 360° ring around the sample before it is captured by a detector
- The amount of light scattered is proportional to the turbidity of the sample.



Measuring Notes - 2100N

- Always cap the sample cell to prevent spillage into instrument
- Close the sample compartment lid during measurement
- Do not leave sample cell in the cell compartment for extended periods of time
- Leave the instrument on 24 hours a day if instrument is used regularly

TDEC - Fleming Training Center

10

Measuring Notes - 2100N

- Always use clean, scratch free sample cells and caps
- Always use silicone oil
- Measuring samples immediately to prevent changes in sample characteristics
- Remove air bubbles in sample cells
- Discard sample cells with scratches

TDEC - Fleming Training Center

11

TU5 Series Notes

- 98% less space to clean
- No need for indexing or using silicon oil
- Sealed vials reduce the time needed for calibration
- RFID
 - Allows easy comparison between lab and process (online) measurements
 - Provides chain of custody data - no need to manually record information
- System check feature

Calibrations

2100 N

- ☐ Primary Stable Cal Standards
 - Formazin Solution Primary Standards and Procedure for making solutions
- ☐ Use Gelex Secondary Turbidity Standards for periodic checks

TU5 SERIES

- ☐ Calibration
 - StablCal
 - Formazin Solution
- ☐ Verification
 - Glass verification rod (secondary turbidity standard)
 - StablCal
 - Formazin

TDEC - Fleming Training Center

13

Calibrations

- ☐ Record keeping requirements
 - Document everything on benchsheet
 - Date, time, operator initials, all relevant information
 - Maintenance records
 - Verification documentation
- ☐ Calibrate at least quarterly

Any Questions?

Turbidity – Review Questions

1. What is turbidity?
2. What causes turbidity?
3. A turbidity reading will give you an exact number of particles in the sample. True or False?
4. Turbidity is useful as an indicator of overall water quality. True or False?
5. List at least 4 reasons why high turbidity readings in your water sample could be problematic.
6. Turbidity readings are expressed in which unit of measurement?
7. Why should turbidity samples be read immediately?

Section 10

pH

*pH

Basic Lab Class



*pH = The intensity of the basic or acidic strength of water

*One of the most important and frequently used tests in water chemistry

*Logarithmic scale of 0 - 14 s.u. (standard units)

*pH

- *Electrometric measurement
- *Typically measured with a meter and a probe
- *Litmus paper
 - *Acid turns blue litmus paper red
 - *Base turns red litmus paper blue

*pH

*In a solution, both hydrogen ions [H+] and hydroxyl ions [OH-] are always present

*Acid

*More hydrogen ions (H+) in the solution

*Base

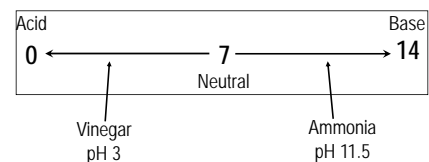
*More hydroxide ions (OH-) in the solution

*pH theory

*pH is defined as the negative log of the molar hydrogen ion concentration in aqueous solution

$$\text{pH} = -\log [\text{H}^+]$$

*pH

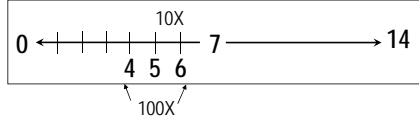


*pH Scale

- *At a pH of 7...
 - *The activity of both H^+ and OH^- are equal
- *When pH is below 7...
 - *The activity of H^+ ions is greater than the OH^- ions
- *When the pH is above 7...
 - *The activity of OH^- ions is greater than the H^+ ions

***pH**


- *pH is a negative logarithmic function
- *Each decrease in pH unit = 10X increase in acidity
 - *Solution at pH4 is 10X more acidic than solution at pH5
 - *Solution at pH 4 is 100X more acidic than pH6 solution



***pH Scale**

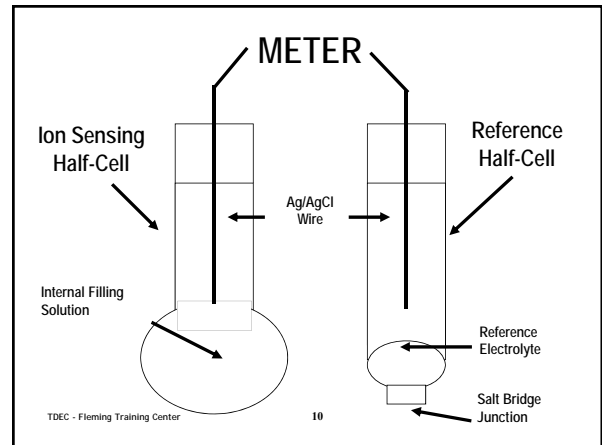
TDEC - Fleming Training Center 8

- *Probe measures hydrogen ion [H^+] concentration
- *Two electrodes in probe:
 - a) sensing half-cell
 - b) reference half-cell




***How Does a pH Probe Work?**

TDEC - Fleming Training Center 9



- *Dispenses reference solution which completes circuit for meter



***Reference Half-Cell**

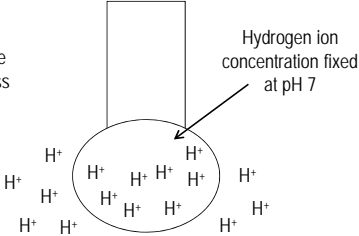
TDEC - Fleming Training Center 11

***Sensing Half-Cell**

pH 7 Solution

H^+ conc. the same both inside and outside glass bulb

*No potential develops



TDEC - Fleming Training Center 12

*Sensing Half-Cell

pH 7 Solution

H⁺ conc. the same both inside and outside glass bulb

*No potential develops



TDEC - Fleming Training Center

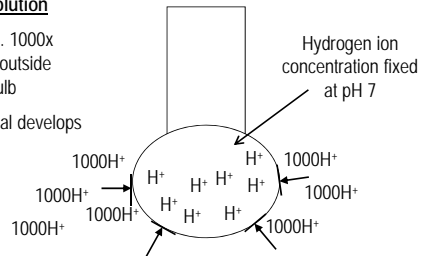
13

*Sensing Half-Cell

pH 4 Solution

H⁺ conc. 1000x greater outside glass bulb

*Potential develops



TDEC - Fleming Training Center

14

*Sensing Half-Cell

pH 4 Solution

H⁺ conc. 1000x greater outside glass bulb

*Potential develops



TDEC - Fleming Training Center

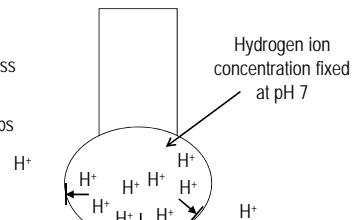
15

*Sensing Half-Cell

pH 10 Solution

H⁺ conc. 1000x greater inside glass bulb

*Potential develops



TDEC - Fleming Training Center

16

*Sensing Half-Cell

pH 10 Solution

H⁺ conc. 1000x greater inside glass bulb

*Potential develops

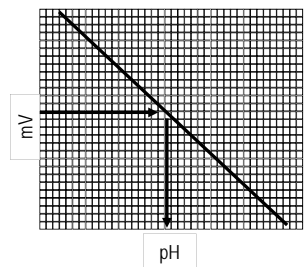


TDEC - Fleming Training Center

17

*A calibration curve allows the meter to convert a measured millivolt potential into a pH reading.

*Decade = one pH unit



*Calibration

TDEC - Fleming Training Center

18

- *Holding time = 15 minutes
- *Preservation = none
- *Sample container = glass or plastic
- *Grab sample
- *Continuous monitoring possible

*pH Sampling

TDEC - Fleming Training Center

19

- *Look in user manual
 - *Follow manufacturer's instructions
- *Calibrate Daily
- *pH meter, buffers, samples should all be at same temp
- *Use 3 fresh buffers
 - *4, 7, 10
- *Rinse with DI water, blot dry in between samples

*How to Calibrate a pH Probe

- *Accurate and reproducible to within 0.1 s.u.
- *Readings between standards should be within 0.1 s.u. (Using a probe and meter)
- *Record pH and temperature
- *Record time, date, initials
- *Optimal slope = -58 ± 3 mV/decade
 - *Check user manual

*How to Calibrate a pH Probe

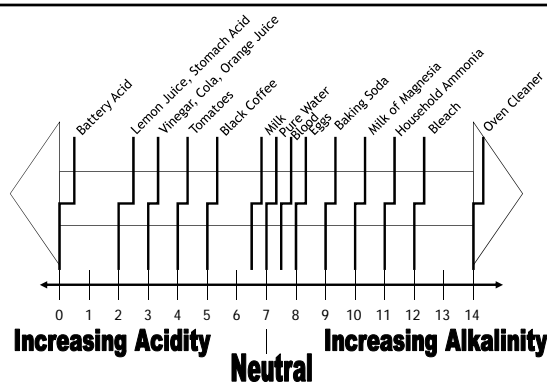
Drinking Water

- *Natural waters: pH 6.5 - 8.5
 - *Most are slightly basic
- *Rainwater usually slightly acidic
- *Alum coagulates most effectively at pH values near 6.8

Wastewater

- *pH 6 - 8 is acceptable for most organism activity
- *Influent or Raw Wastewater: pH 6.8 - 8
- *Wastewater Plant Effluent: pH 6.8 - 8

*Common pH Ranges



TDEC - Fleming Training Center

23

*Any Questions?

pH – Review Questions

1. pH is a measure of what?
2. What is the holding time for a pH sample?
3. How often should your pH meter be calibrated?
4. Why must you always use fresh buffers with each calibration?
5. An acid increases the _____ ion (H⁺) concentration in a solution.
6. A base increases the _____ ion (OH⁻) concentration in a solution.
7. Each decrease in pH unit equals a _____ times increase in acidity.
8. A solution with a pH of 3 is how many times more acidic than a solution with a pH of 7?

Fleming Training Center

Method: SM 4500-H+B-2011 pH Calibration Record

Date	Time	Temp of Buffers	Slope	Buffers Used			ICV	CCV	Analyst Initials	Remarks
				Lot#s						
				4	7	10				

Date	Time	Sample Name	Temp. Solution	Date of Last Calibration	Measured pH	Analyst Initials
		Vinegar				
		Coca Cola				
		Lemon Juice				
		Ammonia Cleaner				
		Alkaline Water				

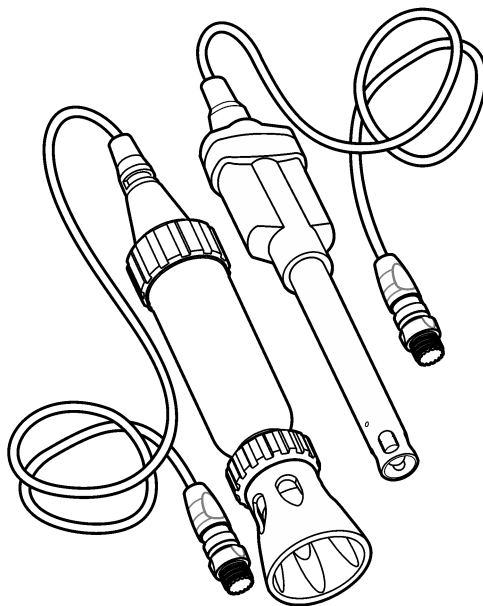


DOC022.53.80023

PHC101

06/2021, Edition 5

User Manual

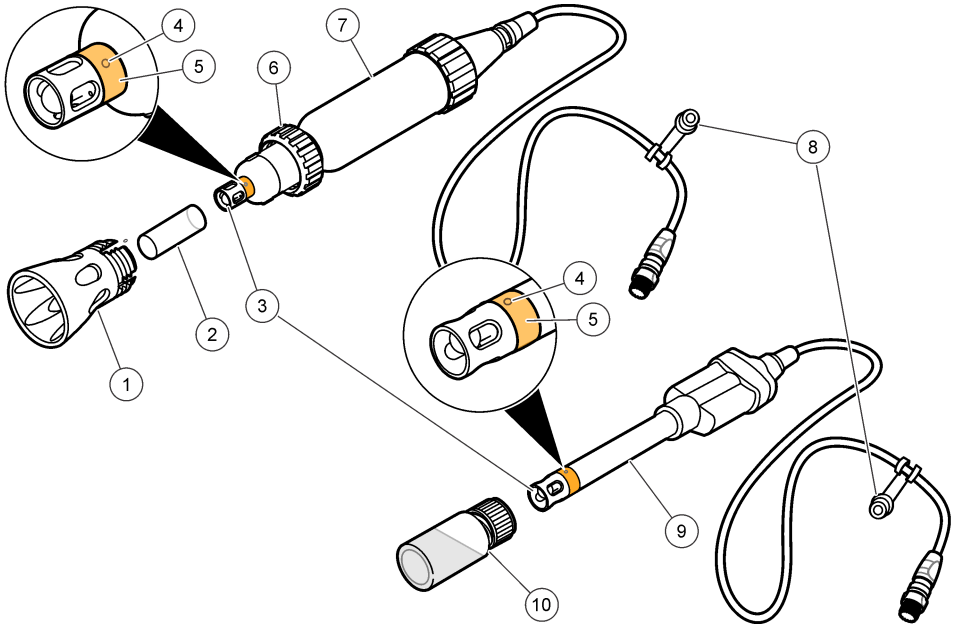


Section 1 Product overview	3
Section 2 Specifications	3
Section 3 Safety information	4
3.1 Intended use	4
3.2 Use of hazard information	5
3.3 Precautionary labels	5
3.4 Product hazards	5
Section 4 Preparation for use	5
Section 5 Calibration	6
5.1 Calibration notes	6
5.2 Calibration procedure	7
Section 6 Sample measurement	8
6.1 Sample measurement notes	8
6.2 Sample measurement procedure	8
Section 7 Verify the calibration	9
7.1 Verification procedure	9
Section 8 Maintenance	10
8.1 Clean the probe	10
8.2 Soak procedure for dry probes	11
8.3 Remove or install the shroud	11
8.4 Storage	12
Section 9 Troubleshooting	13
Section 10 Consumables	14
10.1 Recommended standards	14
10.2 Accessories	15

The Intellical PHC101 series pH probes are digital, combination electrodes that measure the pH of wastewater, drinking water and general water samples. The probes have a non-refillable, gel-filled electrolyte and a built-in temperature sensor. The open reference junction gives an optimum electrical connection between the sample and the electrolyte and does not become clogged. The standard probes are for laboratory use. The rugged probes are for field use. Refer to [Figure 1](#).

Note: Do not use the probe to measure the pH of organic solvents or samples with a pH less than 2.

Figure 1 Probe overview



1 Shroud (rugged model)	6 Locking ring (rugged model)
2 Probe storage cap	7 Rugged probe
3 Glass bulb and temperature sensor	8 Probe soaker bottle holder or storage cap
4 Reference junction	9 Standard probe
5 Protective tape	10 Probe soaker bottle with storage solution

Section 2 Specifications

Specifications are subject to change without notice.

Specifications	Details
Probe type	Digital combination pH probe with non-refillable gel electrolyte and built-in temperature sensor
pH range	2 to 14 pH
pH accuracy	±0.02 pH
Reference type	Ag/AgCl

Specifications	Details
Section 10	TDEC Fleming Training Center
Reference junction	Open
Slope	-59 mV/pH (90 to 110% at 25 °C (77 °F) per Nernstian theoretical value)
Isopotential point	0 (±30) mV at 7.0 (±0.5) pH
Sodium (alkalinity) error	-0.6 pH at pH 12.6 in 1 M NaOH
Temperature accuracy	±0.3 °C (±0.54 °F)
Temperature sensor type	30 kΩ NTC thermistor
Operating temperature	0 to 50 °C (32 to 122 °F)
Storage temperature	5 to 40 °C (41 to 104 °F)
Minimum immersion depth	20 mm (0.79 in.)
Body material (standard)	Epoxy
Body material (field rugged)	Epoxy/stainless steel
Electrolyte	Non-refillable gel reference element
Storage solution	Hach pH electrode storage solution ¹
Cable connection	M12 digital output and connector
Dimensions	Diameter: 12 mm (0.47 in.) Length: 175 mm (6.9 in.) total; 103 mm (4.1 in.) below head Cable length: PHC10101: 1 m (3.3 ft); PHC10103: 3 m (9.8 ft)
Dimensions (rugged)	Diameter: 46 mm (1.8 in.) Length: 223 mm (8.7 in.) Cable length: PHC10105: 5 m (16.4 ft); PHC10110: 10 m (32.8 ft); PHC10115: 15 m (49.2 ft); PHC10130: 30 m (98.4 ft)
Weight (includes cable)	PHCxxx01: ~0.4 kg (0.9 lb); PHCxxx03: ~0.45 kg (1 lb)
Weight (rugged, includes cable)	PHCxxx05: ~1.3 kg (2.9 lb); PHCxxx10: ~1.55 kg (3.4 lb); PHCxxx15: ~1.9 kg (4.2 lb); PHCxxx30: 3.0 kg (6.6 lb)
Warranty	6 months on the probe. This warranty covers manufacturing defects, but not improper use or wear.
Certifications	CE, FCC/ISED

Section 3 Safety information

3.1 Intended use

The Intellical probes are intended for use by individuals who measure water quality parameters in the laboratory or in the field. The Intellical probes do not treat or alter water.

¹ Use of other storage solutions can cause permanent damage to the probe.

▲ DANGER

Indicates a potentially or imminently hazardous situation which, if not avoided, will result in death or serious injury.

▲ WARNING

Indicates a potentially or imminently hazardous situation which, if not avoided, could result in death or serious injury.

▲ CAUTION

Indicates a potentially hazardous situation that may result in minor or moderate injury.

NOTICE

Indicates a situation which, if not avoided, may cause damage to the instrument. Information that requires special emphasis.

3.3 Precautionary labels

Read all labels and tags attached to the instrument. Personal injury or damage to the instrument could occur if not observed. A symbol on the instrument is referenced in the manual with a precautionary statement.



Electrical equipment marked with this symbol may not be disposed of in European domestic or public disposal systems. Return old or end-of-life equipment to the manufacturer for disposal at no charge to the user.

3.4 Product hazards

▲ CAUTION



Chemical exposure hazard. Obey laboratory safety procedures and wear all of the personal protective equipment appropriate to the chemicals that are handled. Refer to the current safety data sheets (MSDS/SDS) for safety protocols.

▲ CAUTION



Chemical exposure hazard. Dispose of chemicals and wastes in accordance with local, regional and national regulations.

▲ CAUTION



Personal injury hazard. Glass components can break. Handle with care to prevent cuts.

Section 4 Preparation for use

NOTICE

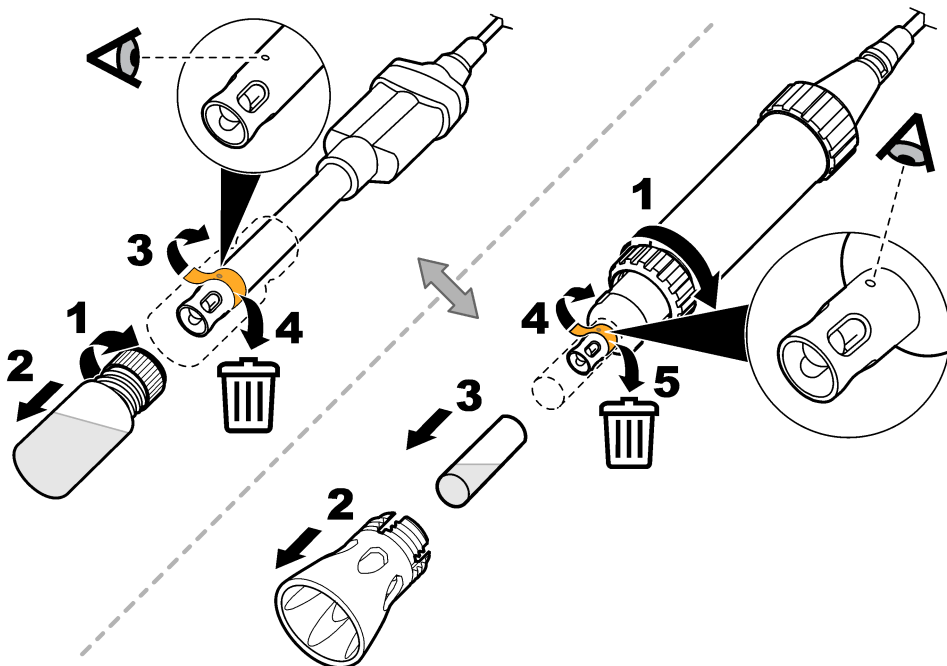
Make sure to remove the protective tape from the reference junction of new probes. A probe with a blocked reference junction will not operate correctly.

New probes come with protective tape and a soaker bottle that contains storage solution to keep the glass bulb and reference junction hydrated. Prepare the probe as follows.

1. Remove the protective tape from the reference junction. Refer to [Figure 2](#).
2. Rinse the reference junction and glass bulb with deionized water. Blot dry with a lint-free cloth.
3. For faster stabilization, soak the probe for 3 or more minutes in the sample before use.
4. Make sure that the meter has the correct date and time settings. The service-life time stamp in the probe comes from the date and time settings in the meter.

Note: Some meters automatically open the date and time settings when the meter starts for the first time, or after battery replacement.
5. Connect the probe to the meter.

Figure 2 Remove the protective tape



Section 5 Calibration

The procedure that follows is applicable to meters that can connect to Intellical pH probes. Refer to the applicable meter documentation for meter operation and probe-specific settings.

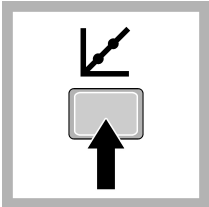
5.1 Calibration notes

Read the notes that follow before calibration:

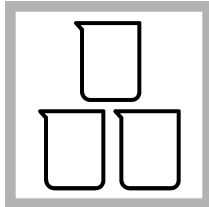
- Use prepared pH buffer solutions or mix pH buffer powder pillows with deionized water for calibration. Discard the prepared buffer solutions after each calibration.
- Use two or three buffer solutions for best results. Two buffer solutions are sufficient if the expected sample pH is between the pH of the two buffer solutions. The sequence in which the pH buffer solutions are used is not important. Use buffer solutions that are 2 or more pH units apart.
- For a one-point calibration, use a pH buffer near the expected sample pH.
- Use the default calibration options or change the options in the probe settings menu.
- Use the single display mode for calibration when more than one probe is connected to the meter (if applicable).

- Calibrate the probes and verify the calibration regularly for best results. Use the meter to set calibration reminders.
- The calibration data is stored in the probe. When a calibrated probe is connected to a different meter with the same calibration options, a new calibration is not necessary.
- Air bubbles below the sensor when in solution can cause a slow response or error in the calibration. Make sure to remove air bubbles during calibration.
- The pH buffer solutions have known pH values at different temperatures. The meter uses the mV and temperature readings of the probe in the pH buffer solutions to calculate a calibration slope. During measurements, the meter adjusts the slope for the sample temperature to determine the pH value of the sample.
- If the rugged probe does not easily go in the calibration container, remove the shroud. Refer to [Remove or install the shroud](#) on page 11.

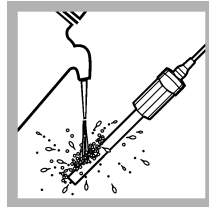
5.2 Calibration procedure



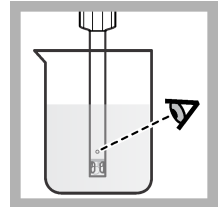
1. Go to the calibrate menu. Select the probe, if applicable. The display shows the pH buffer solutions to use for calibration.



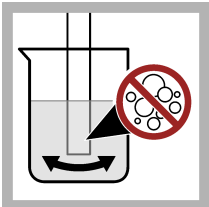
2. Prepare or pour the pH buffer solutions in different beakers.



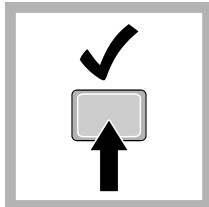
3. Rinse the probe with deionized water. Dry the probe with a lint-free cloth.



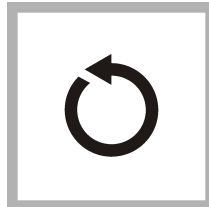
4. Put the probe in the first pH buffer solution. Make sure that the sensor and reference junction are fully in the solution. Do not put the probe on the bottom or sides of the beaker.



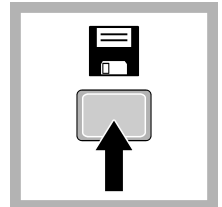
5. Shake the probe from side to side to refresh the reference junction and remove air bubbles.



6. Stir slowly, then read the pH value of the buffer solution. The display shows the temperature-corrected pH value when the reading is stable.



7. Continue with steps 3 through 6 for the remaining buffers or select Done.



8. Save the calibration.

Section 6 Sample measurement

Section 10

TDEC Fleming Training Center

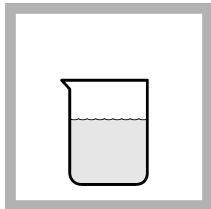
The procedure that follows is applicable to meters that can connect to Intellical pH probes. Refer to the applicable meter documentation for meter operation and probe-specific settings.

6.1 Sample measurement notes

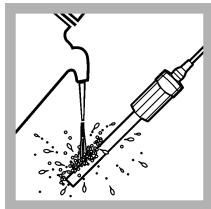
Read the notes that follow before sample measurements.

- Rinse the probe with deionized water and dry with a lint-free cloth between measurements to prevent contamination.
- If complete traceability is necessary, enter a sample ID and operator ID before measurement. Refer to the meter manual for instructions.
- The meter automatically saves the measurement data when the user manually reads each data point and when the meter is set to read at regular intervals. The user must manually save each data point when the meter is set to read continuously.
- Air bubbles below the sensor can cause a slow response or error in the measurement. Make sure to remove air bubbles before and during measurements.
- If the probe is a rugged type, make sure to install the shroud before field use to prevent damage to the sensing elements. Refer to [Remove or install the shroud](#) on page 11. The probe warranty does not include such damage.
- To deploy a rugged probe at a distance, toss the probe body with a slow underhand throw. Do not throw the probe by the cable to prevent damage to cable, the probe or the user.

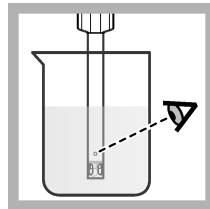
6.2 Sample measurement procedure



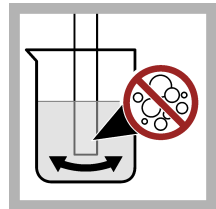
1. Collect the sample.



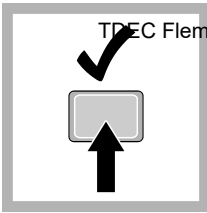
2. Rinse the probe with deionized water. Dry the probe with a lint-free cloth.
Rugged probes: install the shroud.



3. Put the probe in the sample with the sensor and reference junction fully in the sample. Do not put the probe on the bottom or sides of the beaker.



4. Shake the probe from side to side to refresh the reference junction and remove air bubbles.



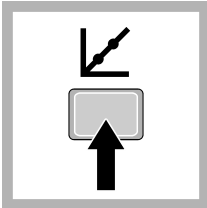
5. Stir gently, then read the pH value of the sample. The display shows the temperature-compensated pH value when the reading is stable.

Section 7 Verify the calibration

Measure the pH value of a fresh pH buffer solution to make sure the result is accurate. The meter compares the selected pH buffer value to the measured pH value and accepts or rejects the measurement. The user can change the pH buffer solution and acceptance criteria for verification in the probe-specific settings.

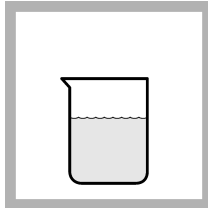
Note: Password protection may prevent access to the acceptance criteria.

7.1 Verification procedure

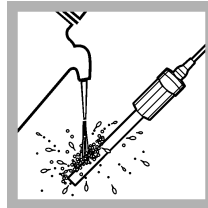


1. Go to the verification menu. The display shows the pH buffer solution to use for verification.

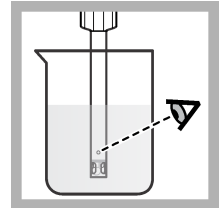
Note: Menu name for HQd meters: Run check standard.



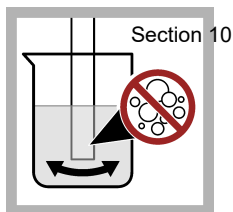
2. Prepare or pour the pH buffer solution into a beaker.



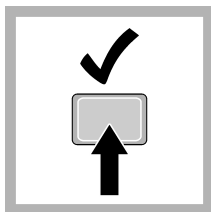
3. Rinse the probe with deionized water. Dry the probe with a lint-free cloth.



4. Put the probe in the pH buffer solution with the sensor and reference junction fully in the solution. Do not put the probe on the bottom or sides of the beaker.



5. Shake the probe from side to side to refresh the reference junction and remove air bubbles.



6. Stir gently, then read the pH value of the buffer solution. The meter accepts or rejects the result.

Section 8 Maintenance

Regular maintenance is necessary for the best accuracy, stabilization time and life of the probe. Keep the probe in the recommended storage solution between measurements.

8.1 Clean the probe

NOTICE

Probes with an open reference junction can become permanently damaged if the reference junction is soaked for a long time in a cleaning solution. Make sure to soak the probe below the reference junction only.

Clean the probe regularly to remove contamination and to keep the reference junction open. Symptoms of contamination:

- Incorrect or irregular readings
 - Slow stabilization times
 - Calibration errors
 - Sample material stays on the probe
1. Rinse the probe with deionized water. Use warm (35–45 °C (95–113 °F)) deionized water to remove storage solution that dries on the probe. Dry the probe body with a lint-free cloth.

Note: Remove the shroud on a rugged probe before cleaning. Install the shroud after the probe is clean. Refer to [Remove or install the shroud](#) on page 11.
 2. Soak the probe below the reference junction in the applicable cleaning solution for the specified time. Do not let the reference junction soak in the cleaning solution or the probe can become permanently damaged. Refer to [Figure 3](#), [Table 1](#) and [Consumables](#) on page 14.
 3. Rinse or soak the probe for 1 minute in deionized water. Dry the probe body with a lint-free cloth.
 4. Soak the probe in pH 4 buffer for 20 minutes.
 5. Rinse the probe with deionized water. Dry the probe body with a lint-free cloth.

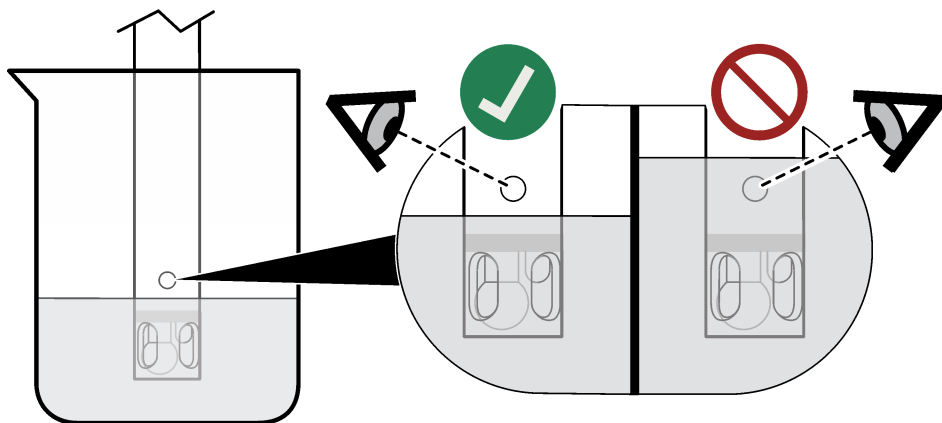


Table 1 Cleaning solution

Contamination	Cleaning solution	Active component	Soak time
General contamination	Electrode cleaning solution for regular maintenance	KATHON™ CG, DECONEX®11	12–16 hours
Minerals	Electrode cleaning solution for minerals/inorganic contamination	Phosphoric acid (~10%)	10–15 minutes
Fats, grease and oils	Electrode cleaning solution for fats, oils and grease contamination	KATHON™ CG, TRITON® X	2 hours maximum
Proteins	Electrode cleaning solution for proteins/organic contamination	Pepsin in HCl	3 hours maximum
Wastewater and organic compounds	Electrode cleaning solution, extra strong	Sodium hypochlorite	5–10 minutes

8.2 Soak procedure for dry probes

If the glass bulb becomes dry, complete the steps that follow to hydrate the probe.

1. Soak the probe tip in pH 4 and pH 7 buffer solutions for 5 minutes in each solution.
2. Rinse the probe with deionized water. Blot dry with a lint-free cloth.
3. Calibrate the probe.

8.3 Remove or install the shroud

Remove the shroud on the rugged probe during calibration and maintenance. Refer to [Figure 4](#). Keep the shroud installed on the rugged probe during sample measurements to prevent damage to the sensor. Refer to [Figure 5](#).

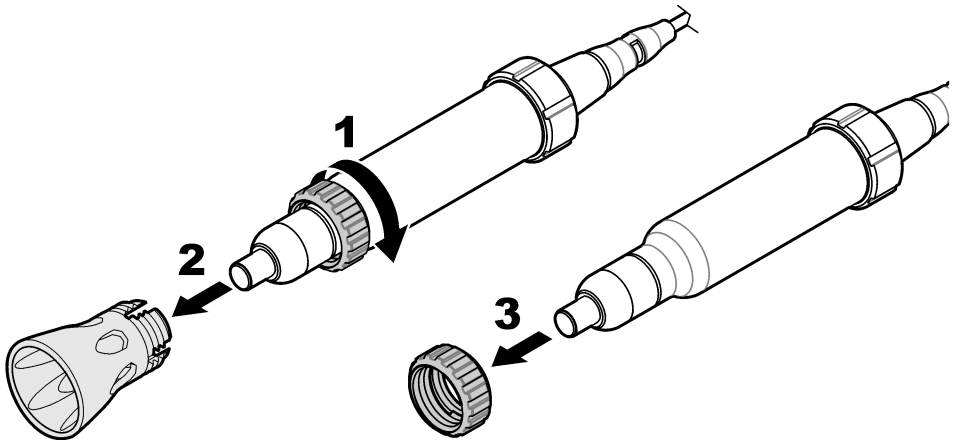
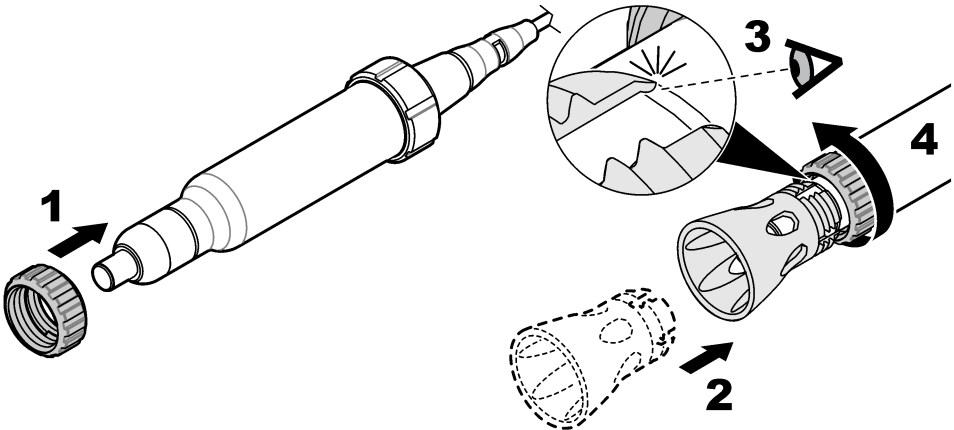


Figure 5 Install the shroud

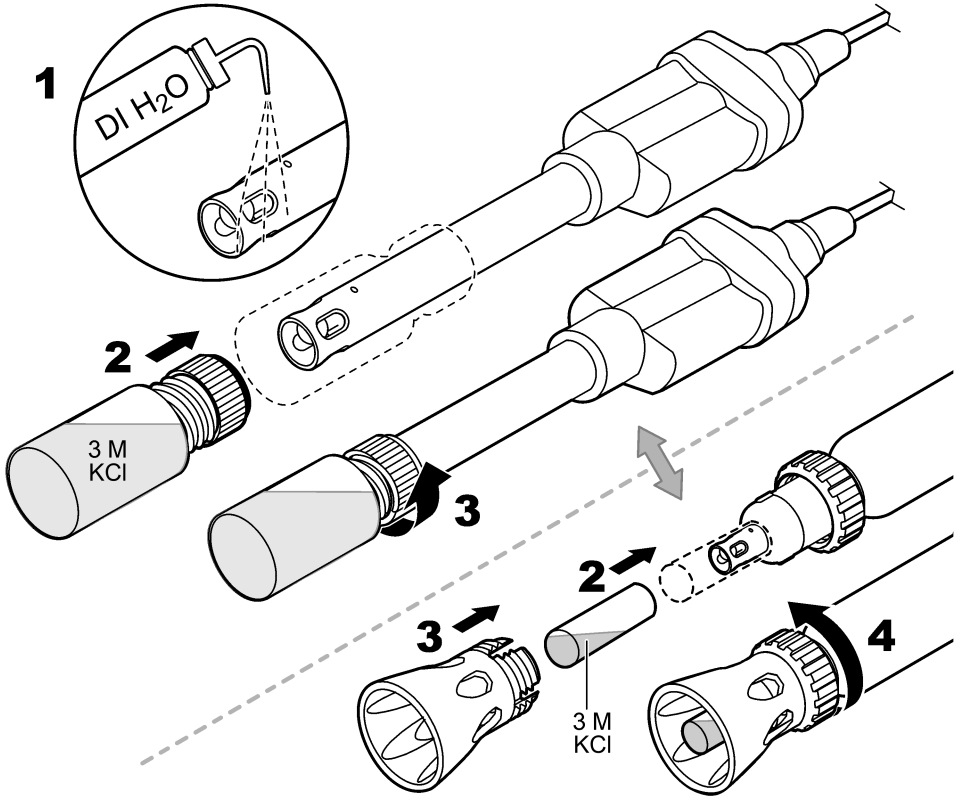


8.4 Storage

NOTICE

Probes can become permanently damaged if kept in a storage solution that is not specified by the manufacturer. Use only the specified storage solution (Hach pH electrode storage solution or 3 M KCl).

Do not store the probe in deionized water or in samples of low ionic strength. Put the soaker bottle that contains the storage solution on the probe when not in use. Make sure to only use the specified storage solution. Other solutions contaminate the non-replacement electrolyte gel through the open reference junction and the probe will not operate correctly. Refer to [Figure 6](#). Keep the probe in a vertical position with the sensor and reference junction below the liquid level in the soaker bottle. Add storage solution to the soaker bottle if necessary.



Section 9 Troubleshooting

Keep the probe clean and in the recommended storage solution when not in use for the best accuracy, stabilization time and life of the probe.

Problem	Possible cause	Solution
Decreased probe performance causes slow stabilization and prevents accurate calibrations or measurements.	The glass sensor is dirty.	Clean and condition the probe. Refer to Clean the probe on page 10.
	The glass sensor has become dry.	Clean and condition the probe. Refer to Maintenance on page 10.
	The calibration slope of the probe has changed.	Increase the accepted slope limit settings if possible, or contact technical support.

Problem	Section 10	Possible cause	TDEO	Solution	Fleming Training Center
Sample properties cause slow stabilization or inaccurate measurements.		The sample absorbs carbon dioxide (CO ₂) from the air, which causes the pH value to slowly decrease in low ionic strength (LIS) or high purity samples.		Use the LIS chamber for LIS/high purity samples to prevent CO ₂ absorption.	
		The sample temperature is low, or there is a large temperature difference between samples.		Increase the sample temperature or adjust the temperature of different samples to be the same (within 2 °C (3.6 °F)).	
Procedure problem causes slow stabilization and prevents accurate calibrations or measurements.		The probe is not conditioned to the sample.		Soak the probe in the sample before sample measurements. Refer to Preparation for use on page 5.	
		Air bubbles are around or below the probe tip.		Carefully tap or shake the probe to remove air bubbles.	
		The electrical connection through the reference junction is not sufficient.		Shake the probe in the solution from side to side to refresh the reference junction.	
		The stir speed is too slow or too fast.		Try a different stir speed.	
		An incorrect buffer solution was used or the buffer solution has contamination.		Use the specified buffer solutions of good quality.	

Section 10 Consumables

Note: Product and Article numbers may vary for some selling regions. Contact the appropriate distributor or refer to the company website for contact information.

Description	Quantity	Item no.
Hach pH electrode storage solution	500 mL	2756549
Electrode cleaning solution for regular maintenance	500 mL	2965249
Electrode cleaning solution for minerals/inorganic contamination	500 mL	2975149
Electrode cleaning solution for proteins/organic contamination	250 mL	C20C370
Electrode cleaning solution for fats, oils and grease contamination	500 mL	2964449
Electrode cleaning solution, extra strong	250 mL	S16M002

10.1 Recommended standards

Description	Unit	Item no.
pH 4.01 buffer solution, Singlet one-use packets, 20 mL each	20/pkg	2770020
pH 7.00 buffer solution, Singlet one-use packets, 20 mL each	20/pkg	2770120

10.1 Recommended standards (continued)

TDEC Fleming Training Center

Section 10

Description	Unit	Item no.
pH 10.01 buffer solution, Singlet one-use packets, 20 mL each	20/pkg	2770220
pH 4.01 and pH 7.00 buffer solution kit, Singlet one-use packets, 20 mL each	2 x 10/pkg	2769920
pH 7.00 and 10.01 buffer solution kit, Singlet one-use packets, 20 mL each	2 x 10/pkg	2769820
pH color-coded buffer solution kit (NIST), 500 mL, includes:	1	2947600
pH 4.01 ± 0.02 pH buffer (NIST)	500 mL	2283449
pH 7.00 ± 0.02 pH buffer (NIST)	500 mL	2283549
pH 10.01 ± 0.02 pH buffer (NIST)	500 mL	2283649
Powder pillows:		
pH 4.01 ± 0.02 pH buffer powder pillow (NIST)	50/pkg	2226966
pH 7.00 ± 0.02 pH buffer powder pillow (NIST)	50/pkg	2227066
pH 10.01 ± 0.02 pH buffer powder pillow (NIST)	50/pkg	2227166
Radiometer Analytical (IUPAC Series certified pH standards):		
pH 1.679 ± 0.010 at 25 °C (77 °F)	500 mL	S11M001
pH 4.005 ± 0.010 at 25 °C (77 °F)	500 mL	S11M002
pH 6.865 ± 0.010 at 25 °C (77 °F)	500 mL	S11M003
pH 7.000 ± 0.010 at 25 °C (77 °F)	500 mL	S11M004
pH 9.180 ± 0.010 at 25 °C (77 °F)	500 mL	S11M006
pH 10.012 ± 0.010 at 25 °C (77 °F)	500 mL	S11M007
pH 12.45 ± 0.05 at 25 °C (77 °F)	500 mL	S11M008
pH buffer 1.09, technical	500 mL	S11M009
pH buffer 4.65, technical	500 mL	S11M010
pH buffer 9.23, technical	500 mL	S11M011

10.2 Accessories

Description	Quantity	Item no.
Beaker, 30 mL, plastic, colorless	80/pkg	SM5010
Beaker, 30 mL, plastic, red	80/pkg	SM5011
Beaker, 30 mL, plastic, yellow	80/pkg	SM5012
Beaker, 30 mL, plastic, blue	80/pkg	SM5013
Beaker, 30 mL, plastic, green	80/pkg	SM5014
Beaker dispenser and holder, 30 mL	1	923-656
Beaker holder, 30 mL	1	923-556

pH

169

10.2 Accessories (continued)

Section 10

TDEC Fleming Training Center

Description	Quantity	Item no.
Beaker, 100 mL, polypropylene	1	108042
LIS (low ionic strength) chamber	1	5189900
Disposable wipes, 11 x 22 cm	280/pkg	2097000
Wash bottle, polyethylene, 500 mL	1	62011
Probe stand for standard Intellical probes	1	8508850
Soaker bottle for probe storage	1	5192900
Probe cable depth markers for rugged Intellical probes	5/pkg	5828610
Shroud kit for rugged probes	1	5825900
Storage caps for rugged PHC and MTC probes	5/pkg	5857305

**HACH COMPANY World Headquarters**

P.O. Box 389, Loveland, CO 80539-0389 U.S.A.
Tel. (970) 669-3050
(800) 227-4224 (U.S.A. only)
Fax (970) 669-2932
orders@hach.com
www.hach.com

HACH LANGE GMBH

Willstätterstraße 11
D-40549 Düsseldorf, Germany
Tel. +49 (0) 2 11 52 88-320
Fax +49 (0) 2 11 52 88-210
info-de@hach.com
www.de.hach.com

HACH LANGE Sàrl

6, route de Compois
1222 Vézenaz
SWITZERLAND
Tel. +41 22 594 6400
Fax +41 22 594 6499

pH

USEPA Electrode Method

Method 8156**pH electrode****Scope and application:** For drinking water¹, wastewater² and process water.¹ Based on Standard Method 4500-H+B, ASTM Method D1293-95 and USEPA Method 150.1² Based on Standard Method 4500-H+B, ASTM Method D1293-84(90)/(A or B) and USEPA Method 150.1

Test preparation

Instrument specific information

This procedure is applicable to the meters and probes that are shown in [Table 1](#).
Procedures for other meters and probes can be different.

Table 1 Instrument-specific information

Meter	Probe
HQ1100 and HQ11d portable one input, pH/ORP HQ4100, HQ2100 and HQ30d portable one input, multi-parameter HQ4200, HQ2200 and HQ40d portable two input, multi-parameter HQ4300 portable three input, multi-parameter HQ411d benchtop one input, pH/mV HQ430d benchtop one input, multi-parameter HQ440d benchtop two input, multi-parameter	Intellical PHC101, PHC201, PHC281 or PHC301 pH
Sension+ MM156 portable pH/EC/DO	Sension+ 5049 multi-parameter
Sension+ pH1 portable pH Sension+ MM110 portable pH/ORP Sension+ MM150 portable pH/ORP/EC	Sension+ 5050T, 5051T or 5052T combination pH Sension+ 5045, 5048 or 5059 multi-parameter
Sension+ pH3 lab pH Sension+ pH31 GLP lab pH Sension+ MM340 lab two input, pH/mV/ISE Sension+ MM374 lab two input, pH/mV/EC/ISE Sension+ MM378 lab two input, pH/ISE/EC/DO	Sension+ 5010T, 5011T, 5014T or 5021T combination pH

Before starting

Refer to the meter documentation for meter settings and operation. Refer to probe documentation for probe preparation, maintenance and storage information.

Prepare the probe before initial use. Refer to probe documentation.

When an Intellical probe is connected to an HQ meter or an HQd meter, the meter automatically identifies the measurement parameter and is prepared for use.

Condition the electrode for the best response time. To condition the electrode, soak the electrode for several minutes in a solution that has almost the same pH and ionic strength as the sample.

Calibrate the probe before initial use. Refer to [Calibration procedure](#) on page 3.

For rugged electrodes, it may be necessary to remove the shroud before measurement and calibration.

Air bubbles under the sensor tip can cause slow response or measurement errors. To remove the bubbles, carefully shake the probe.

To save data automatically, set the measurement mode to Press to Read or Interval. When the measurement mode is Continuous, select Store to save data manually.

Rinse the electrode between measurements to prevent contamination.

Keep the electrode in a pH storage solution when not in use. Refer to the probe documentation.

Review the Safety Data Sheets (MSDS/SDS) for the chemicals that are used. Use the recommended personal protective equipment.

Dispose of reacted solutions according to local, state and federal regulations. Refer to the Safety Data Sheets for disposal information for unused reagents. Refer to the environmental, health and safety staff for your facility and/or local regulatory agencies for further disposal information.

This procedure is specified for the HQ meters and HQd meters. The Sension+ meters can be used, but the menus and navigation will be different.

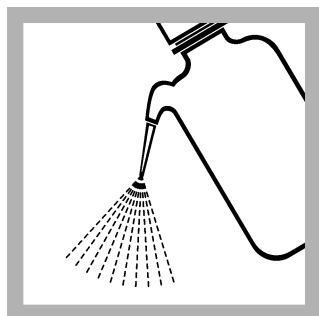
Items to collect

Description	Quantity
Beaker or sample containers	3
Wash bottle with deionized water	1
pH buffers (4.0, 7.0, 10.0)	3

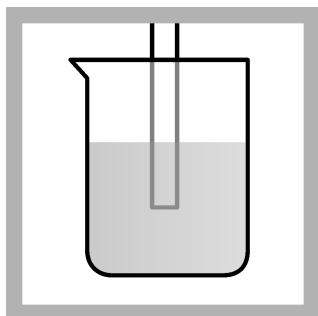
Sample collection

- Analyze the samples immediately. The samples cannot be preserved for later analysis.
- Collect samples in clean glass or plastic bottles.

Test procedure



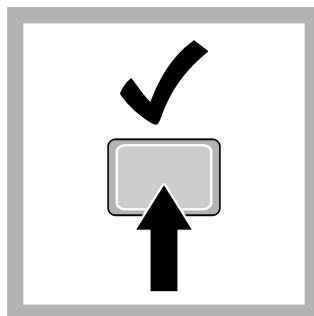
1. Rinse the probe with deionized water. Dry the probe with a lint-free cloth.



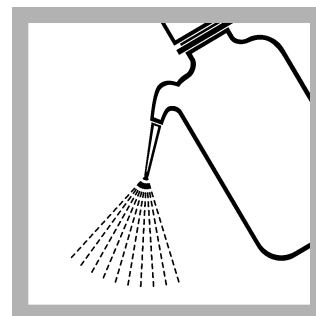
2. Laboratory test: Put the probe in a beaker that contains the solution. Do not let the probe touch the stir bar, bottom or sides of the container. Remove the air bubbles from under the probe tip. Stir the sample at a slow to moderate rate.

Field test: Put the probe in the sample. Move the probe up and down to remove bubbles from the electrode.

Make sure to put the temperature sensor fully in the sample.

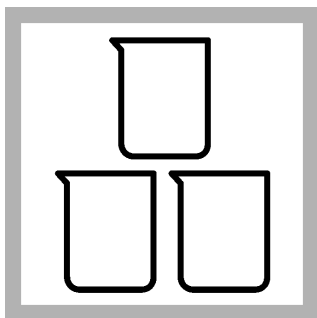


3. Push **Read**. A progress bar is shown. When the measurement is stable, the lock icon is shown.

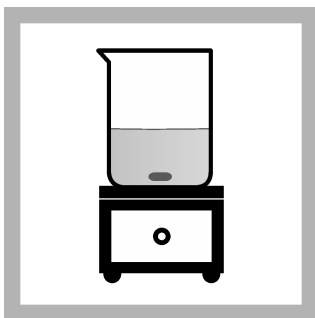


4. Rinse the probe with deionized water. Dry the probe with a lint-free cloth.

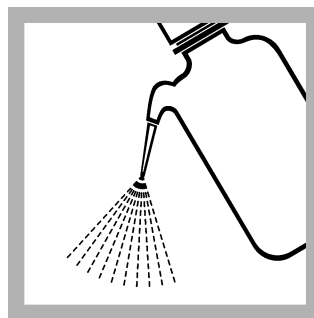
Calibration procedure



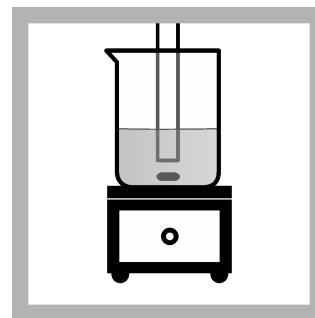
1. Prepare two or three fresh buffer solutions in separate beakers. If two buffers are used, use a 7.0 and a 4.0 or a 7.0 and a 10.0 pH buffer solution.



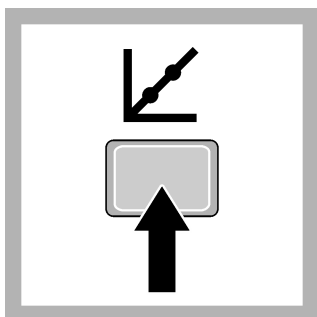
2. Add a stir bar and put the beaker on a magnetic stirrer. Stir at a moderate rate.



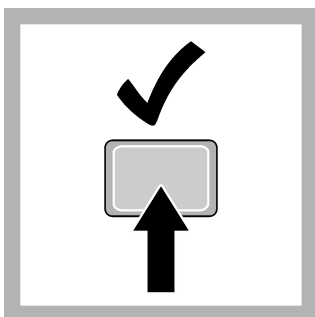
3. Rinse the probe with deionized water. Dry the probe with a lint-free cloth.



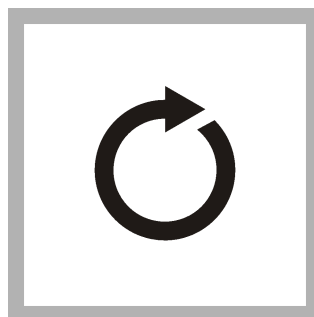
4. Put the probe in the solution. Do not let the probe touch the stir bar, bottom or sides of the container. Remove the air bubbles from under the probe tip.



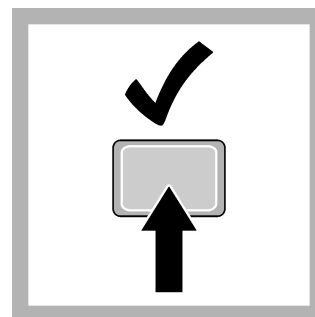
5. Push **Calibrate**. The standard solution value is shown.



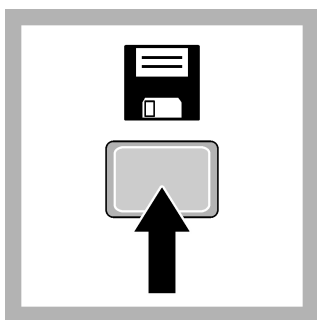
6. Push **Read**. A progress bar is shown. When the measurement is stable, the lock icon is shown.



7. Measure the remaining buffer solutions.



8. Push **Done**. A calibration summary is shown when the minimum number of calibration standards are measured.



9. Push **Store** to accept the calibration.

Low ionic strength or high-purity water measurements

NOTICE

Do not keep the probe in LIS samples for a long period of time because this can decrease the probe life. Put the probe in electrode storage solution or 3 M KCl when LIS measurements are complete.

Low ionic strength (LIS) solutions have very low buffering capacity and absorb carbon dioxide from the air. When a sample absorbs carbon dioxide from the atmosphere, carbonic acid forms. Carbonic acid decreases the sample pH, which causes inaccurate

readings. One solution to this problem is to measure the sample in a low volume, airtight sample chamber such as a low ionic strength chamber.

Use refillable or platinum series electrodes for measurement of pH in LIS or high purity waters.

Before an LIS sample is measured, condition the probe as follows:

1. Soak the probe in a solution equivalent to the sample in ionic strength and pH for 10 to 15 minutes.
2. Rinse the probe with deionized water.
3. Dry the probe with a soft paper towel.

Between measurements, keep the probe in the sample or a neutral LIS solution (e.g., tap water) for a maximum of 2 hours.

Interferences

The sodium error is low but increases at pH values that are higher than pH 11. The acid error is negligible. Refer to the electrode or the meter documentation.

Accuracy check

Slope test

The electrode operation is satisfactory when the calibration slope is within the specified range (typically $-58 \text{ mV} (\pm 3)$ at $25 \text{ }^\circ\text{C}$).

Calibration accuracy

Measure the pH of a fresh buffer solution. A calibration is satisfactory when the measured pH value agrees with the known pH value of the buffer solution.

Clean the probe

Clean the probe when:

- Drifting/inaccurate readings occur as a result of contamination on the sensing element or incorrect storage conditions.
- Slow response time occurs as a result of contamination on the sensing element.
- The slope is out of range as a result of contamination on the sensing element.

For general contamination, complete the steps that follow.

1. Rinse the probe with deionized water. Blot dry with a lint-free cloth.
2. Soak the glass bulb for 12 to 16 hours in Hach Electrode Cleaning Solution.
3. Rinse or soak the probe for 1 minute in deionized water.
4. Soak the probe in pH 4 buffer for up to 20 minutes, then rinse with deionized water.
5. Blot dry with a lint-free cloth.
6. If harsh contaminants are attached to the probe, polish the probe tip with a soft cloth or cotton swab to remove the contaminants.
7. Soak for up to 20 minutes in pH 4 buffer, then rinse with deionized water.

Method performance

The accuracy of the measurements is dependent on many factors that are related with the overall system, which includes the meter, the probe and calibration solutions. Refer to the meter or probe documentation for more information.

Summary of method

A combination pH electrode develops a potential at the glass/liquid interface. At a constant temperature, this potential varies linearly with the pH of the solution.

The pH is the hydrogen ion activity in a solution and is defined as $-\log_{10}a(\text{H}^+)$, where $a(\text{H}^+)$ is the activity of the hydrogen ion. The sample pH can change when carbon dioxide is absorbed from the atmosphere. In water that has a high conductivity, the buffer capacity is typically high and the pH does not change much.

Consumables and replacement items**HQ meters, HQd meters and probes**

Description	Unit	Item no.
HQ1110 portable one input, pH/ORP meter	each	LEV015.53.1110A
HQ2100 portable one input, multi-parameter meter	each	LEV015.53.2100A
HQ2200 portable two input, multi-parameter meter	each	LEV015.53.2200A
HQ4100 portable one input, multi-parameter meter	each	LEV015.53.4100A
HQ4200 portable two input, multi-parameter meter	each	LEV015.53.4200A
HQ4300 portable three input, multi-parameter meter	each	LEV015.53.4300A
HQ411d benchtop one input, pH/mV meter	each	HQ411D
HQ430d benchtop one input, multi-parameter meter	each	HQ430D
HQ440d benchtop two input, multi-parameter meter	each	HQ440D
Intellical pH gel probe, standard with 1 m cable	each	PHC10101
Intellical pH gel probe, standard with 3 m cable	each	PHC10103
Intellical pH gel probe, rugged with 5 m cable	each	PHC10105
Intellical pH gel probe, rugged with 10 m cable	each	PHC10110
Intellical pH gel probe, rugged with 15 m cable	each	PHC10115
Intellical pH gel probe, rugged with 30 m cable	each	PHC10130
Intellical pH gel probe, standard with 1 m cable	each	PHC20101
Intellical pH gel probe, standard with 3 m cable	each	PHC20103
Intellical pH gel probe, ultra with 1 m cable	each	PHC28101
Intellical pH gel probe, ultra with 3 m cable	each	PHC28103
Intellical pH liquid probe, standard with 1 m cable	each	PHC30101
Intellical pH liquid probe, standard with 3 m cable	each	PHC30103

Sension+ meters and probes

Description	Unit	Item no.
Sension+ pH3 lab pH meter	each	LPV2010T.97.002
Sension+ pH31 GLP lab pH meter	each	LPV2110T.97.002
Sension+ MM340 lab two input, pH/mV/ISE meter	each	LPV2200.97.0002
Sension+ MM374 lab two input, pH/mV/EC/ISE meter	each	LPV4110.97.0002
Sension+ MM378 lab two input, pH/ISE/EC/DO meter	each	LPV4130.97.0002
Sension+ 5010T combination pH probe	each	LZW5010T.97.002
Sension+ 5011T combination pH probe	each	LZW5011T.97.002
Sension+ 5014T combination pH probe	each	LZW5014T.97.002
Sension+ 5021T combination pH probe	each	LZW5021T.97.002
Sension+ 5050T combination pH probe	each	LZW5050T.97.002
Sension+ 5051T combination pH probe	each	LZW5051T.97.002
Sension+ 5052T combination pH probe	each	LZW5052T.97.002
Sension+ 5045 multi-parameter probe	each	LZW5045.97.0002

Sension+ meters and probes (continued)

Description	Unit	Item no.
Sension+ 5048 multi-parameter probe	each	LZW5048.97.0002
Sension+ 5049 multi-parameter probe	each	LZW5049.97.0002
Sension+ 5059 multi-parameter probe	each	LZW5059.97.0002

Recommended standards

Description	Unit	Item no.
pH 4.01 buffer solution, Singlet one-use packets, 20 mL each	20/pkg	2770020
pH 7.00 buffer solution, Singlet one-use packets, 20 mL each	20/pkg	2770120
pH 10.01 buffer solution, Singlet one-use packets, 20 mL each	20/pkg	2770220
pH 4.01 and pH 7.00 buffer solution kit, Singlet one-use packets, 20 mL each	2 x 10/pkg	2769920
pH 7.00 and 10.01 buffer solution kit, Singlet one-use packets, 20 mL each	2 x 10/pkg	2769820
pH color-coded buffer solution kit (NIST), 500 mL, includes:	1	2947600
pH 4.01 ± 0.02 pH buffer (NIST)	500 mL	2283449
pH 7.00 ± 0.02 pH buffer (NIST)	500 mL	2283549
pH 10.01 ± 0.02 pH buffer (NIST)	500 mL	2283649
Powder pillows:		
pH 4.01 ± 0.02 pH buffer powder pillow (NIST)	50/pkg	2226966
pH 7.00 ± 0.02 pH buffer powder pillow (NIST)	50/pkg	2227066
pH 10.01 ± 0.02 pH buffer powder pillow (NIST)	50/pkg	2227166
Radiometer Analytical (IUPAC Series certified pH standards):		
pH 1.679 ± 0.010 at 25 °C (77 °F)	500 mL	S11M001
pH 4.005 ± 0.010 at 25 °C (77 °F)	500 mL	S11M002
pH 6.865 ± 0.010 at 25 °C (77 °F)	500 mL	S11M003
pH 7.000 ± 0.010 at 25 °C (77 °F)	500 mL	S11M004
pH 9.180 ± 0.010 at 25 °C (77 °F)	500 mL	S11M006
pH 10.012 ± 0.010 at 25 °C (77 °F)	500 mL	S11M007
pH 12.45 ± 0.05 at 25 °C (77 °F)	500 mL	S11M008
pH buffer 1.09, technical	500 mL	S11M009
pH buffer 4.65, technical	500 mL	S11M010
pH buffer 9.23, technical	500 mL	S11M011

Accessories

Description	Unit	Item no.
Beaker, polypropylene, 50 mL, low form	each	108041
Beaker, polypropylene, 100-mL	each	108042
Bottle, wash, 500 mL	each	62011
Stir bar, magnetic, 2.2 x 0.5 cm (7/8 x 3/16 in.)	each	4531500
Stirrer, electromagnetic, 120 VAC, with electrode stand	each	4530001
Stirrer, electromagnetic, 230 VAC, with electrode stand	each	4530002

Accessories (continued)

Description	Unit	Item no.
Sample bottle with screw-top cap, polypropylene, 500-mL	each	2758101
Water, deionized	4 L	27256



FOR TECHNICAL ASSISTANCE, PRICE INFORMATION AND ORDERING:
In the U.S.A. – Call toll-free 800-227-4224
Outside the U.S.A. – Contact the HACH office or distributor serving you.
On the Worldwide Web – www.hach.com; E-mail – techhelp@hach.com

HACH COMPANY
WORLD HEADQUARTERS
Telephone: (970) 669-3050
FAX: (970) 669-2932

Section 11

Standard Methods, SOPs, & QA/QC

Standard Methods, SOP's & Introduction to QA/QC

Basic Lab Class



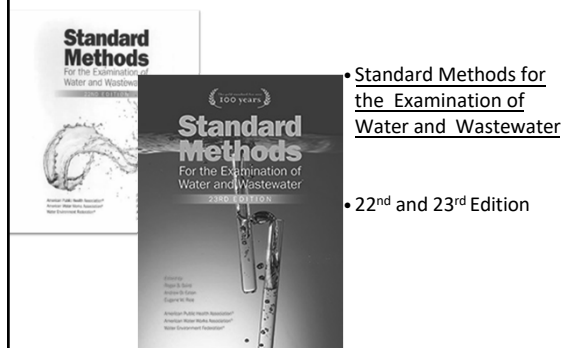
Standard Methods

- 1880's movement for "securing the adoption of more uniform and efficient methods of water analysis"
- Drinking water only until 1925
- 1933 joint publication
 - Standard Methods of the Examination of Water and Sewage

Standard Methods

- Methods believed to be best available
- Recommendations of specialists, ratified by large number of analysts and other experts
- Truly consensus standards
- Offers valid and recognized basis for control and evaluation

Standard Methods



- Standard Methods for the Examination of Water and Wastewater
- 22nd and 23rd Edition

Code of Federal Regulations (CFR)

- The purpose of the CFR is to present the official and complete text of agency regulations in one organized publication and to provide a comprehensive and convenient reference for all those who may need to know the text of general and permanent Federal regulations



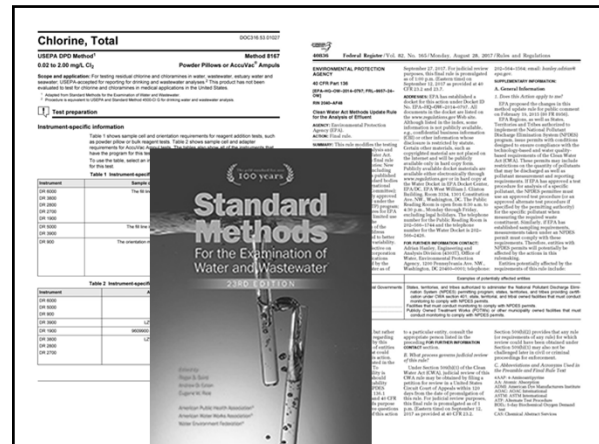
Code of Federal Regulations (CFR)

- The CFR is divided into 50 titles representing broad areas subject to Federal regulation
 - Each title divided into chapters
 - Each chapter divided into parts
 - Each part divided into sections
- Wastewater: 40 CFR 136
- Drinking Water: 40 CFR 141
- Sometimes CFR supersedes Standard Methods



Code of Federal Regulations (CFR)

- CFR will list approved methods for testing
- Includes:
 - Standard Methods
 - EPA methods
 - Hach methods
- Always check to make sure you are using an approved method!



Standard Operating Procedure (SOP)

- All procedures must be documented in some type of SOP
- It can be very simple but must provide the information necessary for someone who is not familiar with the test to perform it
 - Step by step instructions on how and where to collect the samples and then how to run the test

9

Standard Operating Procedure (SOP)

- Describes the analytical method
- Sufficient detail that someone unfamiliar with the method could perform it and get satisfactory results
- Can include pictures (Ex: where samples are collected)
- It must include the QC Acceptance Criteria, the definition of a "Batch" and the minimum frequency of QC checks

Standard Operating Procedure (SOP)

- Should include :
 - Title of reference
 - Method #
 - Summary
 - Definitions
 - Interferences
 - Safety considerations
 - Equipment and supplies
 - Preservation and storage requirements
 - QC information
 - Etc...

Standard Operating Procedure (SOP)

- Annually:
 - Review/Update
 - Make any necessary adjustments
 - Changes to facility?
 - Changes to staff?
 - Document new Revision
 - Training
 - Have all analysts review/read
 - Have analysts sign off that they have done refresher
 - Documentation

Standard Operating Procedure (SOP)

- Common documents in an SOP:
 - Copy of method from manufacturer on how to calibrate instrument, run samples, etc.
 - QA/QC information from TDEC
 - Step-by-step instruction for you plant on the 12 Steps that apply to that test
 - Where you grab your samples

Standard Operating Procedure (SOP)

Chlorine SOP

Testing Procedure for Total Chlorine Residual High Range (0.1 to 8.0 mg/L Cl₂)

Standard Operating Procedure

- What are some key points that are missing from this SOP?

QA/QC for Chlorine

DO: Dissolved Oxygen. DO is the amount of oxygen dissolved in water from atmospheric oxygen and other sources.

MBL: Most Biologically Limiting. The most limiting factor for growth.

Method Book: Contains detailed methods for water sampling.

ESP: Environmental Sampling Protocol. The standard for sampling and testing.

ESP: Methods: Detailed methods for sampling and testing.

ES/ACT: Environmental Sampling and Analysis. The standard for sampling and testing.

Documentation/Record Keeping

- Review of log books
 - Instrument calibration (daily)
 - Temperature
 - Maintenance
 - Sampler
 - Standard preparation
 - Calibration
- Lab instruments - yearly maintenance check (or more frequently)
 - including thermometers and weights
- Flow measurement devices – yearly maintenance check
- QA/QC that has been done

Record Keeping

- Maintain a complete and accurate list of exact locations of all sampling sites
- Maintain a complete and accurate list of all test procedures used
 - Record method numbers on bench sheets
- Write in pen
- Initial your entries
- Use a notebook that has numbered pages

Bench Sheets

- Where the analyst records the test results
- Even though data is transferred to the DMR, bench sheets are still an official record
- At a minimum, it should include:
 1. Date
 2. Time
 3. Analyst's initials
 4. Name of test/Method #
 5. Sample results
 6. Lot #s

Three QA Options

- A. ...follow equivalent EPA procedures
- B. Refer to QA/QC in consensus organization compendium. (Follow Standard Methods)
- C. Follow the 12 Steps where applicable.
- The 13th step requires an SOP (standard operating procedures)

12 Quality Control Elements

1. DOC – demonstration of capability
 2. MDL – method detection level
 3. LRB/MB – method blank
 4. LFB – laboratory fortified blank (standard)
 5. LFM/LFMD – laboratory fortified matrix/duplicate (spike)
 6. Internal standards, surrogate standards or tracer – *only applies to organic analysis and radiochemistry*
 7. Calibration- initial and continuing
 8. Control charts or other trend analysis
 9. Corrective action – root cause analysis
 10. QC acceptance criteria
 11. Definition of a batch (preparation and analytical)
 12. Minimum frequency for conducting all QC elements
 13. Unwritten 13th Step – SOP – Standard Operating Procedures need to be written and followed for all lab sampling and analyses
- Not all of these items apply to all tests, there are many exceptions!

Applicable tests for Drinking Water

- Total Residual Chlorine
- pH
- TSS
- Settleable Solids
- Aluminum
- Does your plant have a NPDES permit?

Can you defend what you do?

- How do you interpret your Permit language or the Rule?
- Can you defend that interpretation, will a judge or jury support you?
- What do Regulators say and what is written?
 - Is it clear?
 - Don't be afraid to ask Why?
 - Don't be afraid to ask for directives in writing



What You Are Already Doing

- Most Labs are doing lots of QA/QC stuff – especially contract labs
- Write down what you do....SOP
- Summarize QC Data
 - Table Form
 - Average, Max, Min.
 - Control Charts



Demonstration of Capability (DOC)

- DOC once for each analyst
- Standard Methods 1020.B.1
 - As a minimum, include a reagent blank and at least 4 LFBs at a concentration between 10 times the MDL and the midpoint of a calibration curve.
 - Something to keep along with these records is a signed form (documentation) that analyst has read and understands all appropriate SOPs and Methods.
- How often?
 - Once for each analyst
 - Recommended yearly for backup analyst who does not perform tests frequently
 - Each analyst should have a file kept on their training within and for the lab.
 - DW: chlorine, pH, TSS
 - WW: Ammonia, BOD/CBOD, Chlorine, pH, DO, Total phosphorus, TSS

Method Detection Level (MDL)

- Estimation of the detection limit for variety of physical and chemical methods
- EPA defines as: “the minimum measured concentration of a substance that can be reported with 99% confidence that the measured concentration is distinguishable from method blank results”
- What tests does this apply to?
 - DW: Chlorine
 - WW: Ammonia, Chlorine, and Total Phosphorus
- How often?
 - Annually – but at least every 13 months
- Ongoing data collection and MDL validation is now required quarterly

What IS an MDL study?

- It is a calculation that statistically gives the lowest concentration that a lab/facility can “see,” or detect an analyte
- Not practical for many analyses
- As detector sensitivity improves, the background contamination of the lab, consumable supplies, and equipment can be more important in determining the detection limit than the sensitivity of the instrument

Method Detection Level (MDL)

- Initial MDL
 - Process at least 7 spiked samples and 7 method blank samples
 - As a starting point for selecting the concentration to use when determining the MDL, use an estimate of five times the estimated true detection level
 - Ideally, use pooled data from several analysts
 - Must be prepared in at least 3 batches on 3 separate dates and analyzed on 3 separate dates
 - Preparation and analysis can be on the same day

Method Detection Level (MDL)

- Initial MDL continued...
 - If multiple instruments will be assigned the same MDL, sample analyses must be distributed across all the instruments
 - Minimum of 2 spiked and 2 blank samples per instrument, analyzed on different dates
 - Compute MDL_s – value based on spiked samples
 - Compute MDL_b – value based on blank samples
 - Use the MDL calculator on the FTC website
 - Whichever is greater is your MDL

Method Detection Level (MDL)

- Ongoing Data Collection:
 - MDL_s : Value calculated from the spiked samples
 - Minimum of 2 spiked samples on each instrument
 - Minimum of 8 per year (2 per quarter)
 - MDL_b : value calculated from the method blanks
 - No additional samples required – just use the routine method blanks

Method Detection Level (MDL)

- Samples used to calculate MDL should be performed throughout the year, not on a single date
 - Samples analyzed every quarter, but calculation performed only once a year
- Lab has the option to pool data from multiple instruments to calculate one MDL that represents multiple instruments

Method Detection Level (MDL)

- Annual Verification:
 - At least once every 13 months you need to re-calculate your MDL_s and MDL_b
 - Ideally, use all method blank results from the last 24 months for the MDL_b calculation
 - There is an option to use less data included in the rule
 - The verified MDL is the higher of the two numbers (either the MDL_s or the MDL_b)
 - Your existing MDL may be left unchanged if specific criteria are met
 - See 40 CFR 136 Appendix B

Method Detection Level (MDL)

- Where do I find the MDL Calculator?
 1. Go to Fleming Training Center's website
 - <https://www.tn.gov/environment/program-areas/wr-water-resources/fleming-training-center.html>
 2. On left side, click on "Course Books and Reference Material"
 3. In the drop-down menu, click on "Waste Water Information"
 4. Click on "Method Update Rule – Method Detection Level Math 2018"

Method Detection Level (MDL)

- Some things to remember...
 - Record values in multiple places so you don't lose them
 - You need to "save as" for each parameter
 - Save a copy of the calculator before you change the 13 cell date (every 13 months)
 - You need an electronic or a hardcopy on file
 - Play it safe – have both

Laboratory Reagent Blank (LRB)

- Also known as Method Blank
- Standard Methods 1020.B.5
 - A reagent blank (method blank) consists of reagent water and all reagents that normally are in contact with a sample during the entire analytical procedure (distillation, incubation, etc.)
- DW: Chlorine, TSS
- WW: Ammonia, BOD/cBOD, Chlorine, Total Phosphorus and TSS
- How often?
 - Depends on method QA/QC

Laboratory Fortified Blank (LFB)

- Standard Methods 1020.B.6
 - A laboratory-fortified blank is a reagent water sample to which a known concentration of the analyte of interest has been added
 - Sample batch = 5% basis = 1 every 20 samples
 - At least once a month
 - Use an added concentration of at least 10 times the MDL, or less than or equal to the midpoint of the calibration curve

Laboratory Fortified Blank (LFB) cont.

- For samples that need to be analyzed on a 5% basis or once for every 20 samples follow these criteria:
 - If a permit stated that 3 analyses per week, we would allow for a LFB to be analyzed at least once per month.
 - If a permit stated 5 analyses per week, we would suggest twice a month.
 - Once per month would be the minimum requirement
- DW: Chlorine, TSS
- WW: Ammonia, BOD/cBOD, Chlorine, Total Phosphorus, TSS

Laboratory Fortified Matrix and Duplicate (LFM/LFMD)

- Also known as a Spike and Spike dup
- Standard Methods 1020.B.7
 - A laboratory matrix (LFM) is an additional portion of a sample to which a known amount of the analyte of interest is added before sample preparation
 - The LFM is used to evaluate analyte recovery in a sample
 - Sample batch = 5% basis = 1 every 20 samples
- At least once a month
- Add a concentration less than or equal to the midpoint of the calibration curve
- Preferably the same concentration as the LFB (laboratory fortified blank)

Laboratory Fortified Matrix and Duplicate (LFM/LFMD)

- Also called a Matrix Spike/Matrix Spike Duplicate (MS/MSD)
- Shows if there are interferences in the effluent matrix
- WW: Ammonia and Total Phosphorus
- How often?
For samples that need to be analyzed on a 5% basis or once for every 20 samples follow these criteria:
 - If a permit stated that 3 analyses per week, we would allow for a LFB to be analyzed at least once per month.
 - If a permit stated 5 analyses per week, we would suggest twice a month.
 - Once per month would be the minimum requirement.

Duplicate (Dup)

- Not a part of the 12 Steps of QA, an addition from the State of TN
- Why is this important?
 - *Precision* refers to the closeness of two or more measurements to each other
- Standard Methods 1020.B.8
 - As a minimum, include one duplicate sample with each sample set or on a 5% basis
- Standard Methods 1020.B.12
 - Calculate the RPD (relative percent difference)
 - Equal to or less than 20% RPD

Duplicate (Dup)

- DW: Chlorine, pH, TSS, and Settleable Solids
- WW: BOD/CBOD, chlorine, pH, DO, TSS and Settleable Solids
- How often?
For samples that need to be analyzed on a 5% basis or once for every 20 samples follow these criteria: (10% would be once every 10 samples for TSS)
 - If a permit stated that 3 analyses per week, we would allow for a LFB to be analyzed at least once per month.
 - If a permit stated 5 analyses per week, we would suggest twice a month.
 - Once per month would be the minimum requirement.

Initial Calibration Verification (ICV)

- ICV
 - Standard Methods 1020.B.11.b
 - Perform Initial Calibration using at least 3 concentrations of standards for linear curve
 - Calibrate meter (DO, pH) or verify scale, colorimeter/spectrophotometer, and thermometer

Continuing Calibration Verification (CCV)

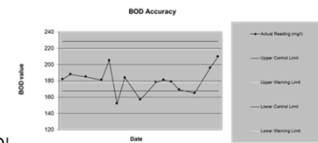
- CCV
 - Standard Methods 1020.B.11.c
 - Analysts periodically use a calibration standard to confirm that the instrument performance has not changed significantly since initial calibration.
 - Verify calibration by analyzing one standard at a concentration near or at the mid-point of the calibration range.
 - Verify the calibration (especially if preset by manufacturer) at beginning of day, after every 10 readings and at the end of the batch
 - **Daily**

Control Charts

- Accuracy Control Charts
- Standard Methods 1020 B.13.a
 - The accuracy chart for QC samples (e.g., reagent blanks, LFBs, calibration check standards and LFM) is constructed from the average and standard deviation of measurements.
 - The accuracy chart includes upper and lower warning levels (WL) and upper and lower control levels (CL).
 - Common practice is to use $\pm 2s$ and $\pm 3s$ limits for the WL and CL, respectively, where s represents standard deviation.
- Precision Control Charts
- Standard Methods 1020 B.13.b
 - The precision chart also is constructed on the average and standard deviation of a specified number of measurements (e.g., %RSD [relative standard deviation] or RPD) for a replicate or duplicate analyses of the analyte of interest.

Control Charts

- (2014 Update) Create and maintain control charts if you have 20-30 data points within 90 days
- If you do not meet the above criteria, follow QC Acceptance Criteria below.
 - Blanks < MDL
 - LFB $\pm 15\%$
 - ICV/CCV $\pm 10\%$
 - LFM/LFMD $\pm 20\%$
 - RPD < 20%
 - Reporting limit = MDL



Corrective Action

- Standard Methods 1020 B.15
 - QC data that are outside the acceptance limits or exhibit a trend are evidence of unacceptable error in the analytical process.
 - Take corrective action promptly to determine and eliminate the source of error.
 - Do not report data until the cause of the problem is identified and either corrected or qualified (see Table 1020:II)
- The corrective action plan needs to be in your SOP for each method on what to do if your QC tests fail or are out of range
- If you have a "boo boo", write down how you fixed it
- Any issues should be recorded and a sentence on how it can be prevented, if possible, in the future
- Common problems and their corrections should be covered in your Standard Operating Procedures (SOP)
 - If you see things frequently, you can give them qualifiers that are noted in your SOP
 - Ex: B = blanks failed, R = rain event

QC Acceptance

- Have in SOP for each method the acceptance ranges for standards, duplicates, spikes, etc. and make sure they match the method requirements.
- If not mentioned in method, these are the accepted criteria for QC:
 - Blank < reporting limit
 - LFB $\pm 15\%$
 - MS/MSD $\pm 20\%$
 - ICV/CCV $\pm 10\%$
 - RPD $\pm 20\%$

Batch Size & QC Frequency

- Each "Batch" could be daily, every 10 samples or every 20 samples
- Check method
- If you sample only once a month, need to run QC each time
- QC Frequency is usually lumped in with the definition of a "batch" and should be in the SOP of some kind

Any questions?



Standard Methods, SOPs, & QA/QC – Review Questions

1. What does CFR stand for?

2. List the CFR part numbers that contain approved test methods for both Drinking Water and Wastewater.

3. What does SOP stand for? What is an SOP?

4. List everything that should be included in the yearly review of your SOPs.

5. Record keeping can be conducted in pencil or pen. True or False

6. Why is it important to include reagent lot #s on your daily bench sheets?

7. Write out what the following abbreviations mean, as well as any alternate names:
 - a. DOC –
 - b. MDL –
 - c. LRB –
 - d. LFB –
 - e. LFM/LFMD –
 - f. Dup –
 - g. ICV –
 - h. CCV –

8. How often is data collected for the MDL? How often is the MDL validation required? How often is the MDL re-calculated?

9. What is the purpose of a duplicate?

NOTE: THIS IS FOR INSTRUCTIONAL PURPOSES ONLY. NOT AN OFFICIAL DOCUMENT. YOU MUST CREATE YOUR OWN SOP WITH SPECIFIC INFORMATION THAT PERTAINS TO YOUR FACILITY AND YOUR EQUIPMENT. THE FOLLOWING IS AN EXAMPLE ONLY.

STANDARD OPERATING PROCEDURE FOR
THE MEASUREMENT OF
pH

Prepared by:

Revision Date:

SOP-pH TABLE OF CONTENTS

Section 1:	Control Information, Computer Storage File.....	p
Section 2:	Personnel Qualifications.....	p
Section 3:	Summary of Method used.....	p
Section 4:	Materials and Equipment	p
Section 5:	Personal Protection Equipment.....	p
Section 6:	Daily pH Meter Calibration	p
Section 7:	Electrode Maintenance.....	p
Section 8:	Troubleshooting	p
Section 9:	Sample Handling	p
Section 10:	Sample pH Measurement.....	p
Section 11:	Data Management and Records Management.....	p
Section 12:	pH Quality Control	p
Section 13:	pH Calibration Log Sheet	p
	Appendix I.....	p

Section 1: Control Information

The SOP is stored as a Microsoft Word document in the SOP folder as file named SOPpH.doc.

Section 2: Personnel Qualifications

Technician should assist in the test for at least 1 week and should have performed a witnessed, unassisted pH performance sample (DOC) before initiating the procedure unsupervised. Each analyst must have run a known standard concentration at least 4 times and compared the limits listed in the method. (See QA/QC documentation.)

Section 3: Summary of Method Used

The method used is Standard Methods for the Examination of Water and Wastewater, 23rd edition, Section 4500-H+-B 2011. This method is applicable to drinking water and wastewater samples. The method consists of placing a glass pH electrode into a well-mixed sample and recording the display on a calibrated pH meter.

Section 4: Materials and Equipment

- Hach Q40d Meter
- Combination glass electrode
- 50 mL Beakers
- pH 4 buffer
- pH 7 buffer
- pH 10 buffer
- Kim wipes
- DI water

Section 5: Personal Protection Equipment

Latex gloves and safety goggles should be worn at all times. Personal hygiene should include hand washing with disinfectant upon completion.

Section 6: Daily pH meter Calibration

The pH meter is used to measure the hydrogen ion concentration of a sample. For routine work, the pH meter must be accurate and reproducible to the nearest 0.1 pH unit. Sample pH and standard buffers should be measured at the same temperature, generally room temperature. The pH meter is normally very stable and needs to be calibrated only once daily. (Generally within 3 hours of use). Calibrate more frequently if indicated.

pH Meter Calibration (ICV)

(Enter in the calibration instructions that apply to your specific probe/meter/instrument.)

Section 7: Electrode Maintenance

- The electrode must **NOT** be stored dry. The best solution to store the

electrode is saturated KCl or pH 7 buffer. Tap water or deionized water should not be used because the electrolyte within the electrode will diffuse out and cause premature aging or additional maintenance.

- Store the electrode in a solution of saturated KCl. Long term storage requires storage in a large test tube covered with parafilm.

Section 8 Troubleshooting

Meter:

- Disconnect the electrode and attach the shorting strap.
- Press the millivolt button. The display should show zero.
- If the display does not change, the meter needs repair.

Electrode:

- Gel electrodes generally require no maintenance if kept moist. However gel electrodes have a shelf life of around 2 years. Recording the date the electrode was placed in service helps with troubleshooting.
- Glass electrodes fail because of scratches, deterioration, and accumulation of debris on the glass bulb. Rejuvenate the electrodes by cleaning in 0.1 M HCl and 0.1 M NaOH.
- Corrective action: If the pH responds slowly or drifts after rejuvenation, replace the electrode.
- Reference electrodes fail because of clogged liquid junctions. The liquid junction must remain open to avoid slow response and meter drift.
- If the liquid junction becomes clogged, place in hot deionized water for several hours. Some combination electrodes can be refilled with saturated KCl.
- If the pH meter does not respond to different buffers (remains at pH 7 all the time), replace the electrode.

Section 9: Sample Handling

- Sample collection is the responsibility of the customer. Clean plastic Nalge bottles are provided to the customer in the size indicated below. Glass containers are also acceptable.
- Samples should be collected and analyzed within 15 minutes. No sample preservation is permitted. On site analysis is preferred.
- Minimum sample volume should be 50 ml.

SAFETY: Use latex gloves when collecting and handling all wastewater samples

Section 10: Sample pH Measurement

Section 11: Data Management and Records Management

- All records must be maintained in three ring binders and filed under the customer's name.
- Maintain all calibration records for a minimum of 5 years.

Section 12: Quality Control

Section 13: pH Calibration Log Sheet (Appendix 1)

State Regulations – Water Quality Reports and Publications

<https://www.tn.gov/environment/program-areas/wr-water-resources/water-quality/water-quality-reports---publications.html>

Wastewater:

1. Division of Water Resources Pollution Control - 0400-40-01
2. Design Criteria for Sewage Works
3. National Pollutant Discharge Elimination System (NPDES) Permit

Drinking Water:

1. Public Water Systems - Chapter 0400-45-01

All certifications:

1. Rules Governing Water and Wastewater Operator Certification – 0400-49-01

Be aware of:

2. Tennessee Code Annotated
<https://www.tn.gov/environment/permit-permits/water-permits1/water-and-wastewater-operator-certification.html>
 - o TCA section 68-221-101 et seq: Water and Sewerage
 - o TCA section 68-221-901 et seq: Water Environmental Health

Section 12

Chlorine

Chlorination

Basic Lab Class



• 1

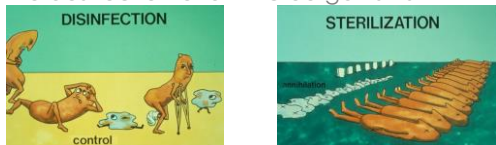
Disinfection

- Water is our single most important natural resource
 - Without water we could not exist
 - Need safe water to drink
- Water is the universal solvent and therefore, carries all types of dissolved materials
 - Including biological life
- Pathogens transmitted by water:
 - Cholera
 - Bacillary Dysentery
 - Gastroenteritis
 - Typhoid Fever
 - Cryptosporidiosis
 - Giardiasis
 - Infectious Hepatitis

• 2

Disinfection

- **Disinfection** is the selective destruction or inactivation of pathogens (disease-causing organisms)
 - This is usually through the addition of chlorine
- Do not confuse this with **sterilization**, which is the destruction of *all* microorganisms



•

Agents of disinfection

- Chemical Disinfectants
 - Chlorine -- Cl_2
 - 100% pure
 - gas
 - Calcium hypochlorite -- $\text{Ca}(\text{OCl})_2$
 - 65% pure
 - solid
 - HTH – high test hypochlorite
 - Sodium hypochlorite -- NaOCl
 - 5-15% pure
 - Liquid
 - Bleach



• 4

Chlorine Gas

- Properties of Chlorine Gas:
 - Yellowish green in color
 - Slightly soluble in water
 - 2.5 times heavier than air
 - High coefficient of expansion (460)
 - one liter of liquid will expand to 460 liters of gas
 - Highly toxic



• 5

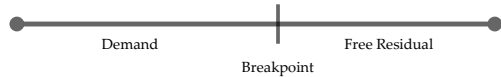
Chlorine Disinfection

- Exact mechanism is unknown, but the demonstrated effects are what matter
- When chlorine is added to water containing organic and inorganic material (reducing agents), it will combine with these materials and form chlorine compounds
 - i.e. chlororganics and chloramines
 - Some of these compounds have disinfecting properties, some do not

• 6

Breakpoint Chlorination

- The process of adding chlorine to water until the chlorine demand has been satisfied
 - Further additions of chlorine will result in a chlorine residual that is directly proportional to the amount of chlorine added beyond the breakpoint



- Total chlorine dose = residual + demand

• 7

Breakpoint Chlorination

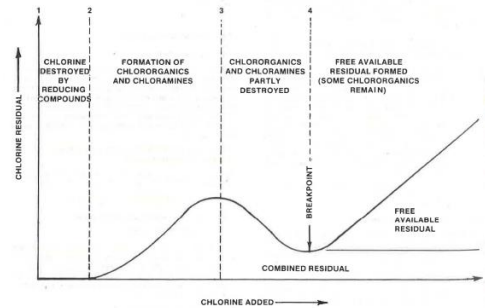


Fig. 7.2 Breakpoint chlorination curve

• 8

Chlorine Disinfection

- If you continue to add chlorine, you will reach a point where the reaction with organic/inorganic materials stops
- At this point, you have satisfied the **Chlorine Demand**
- Demand** = the difference between the amount of chlorine added and the amount of (residual) chlorine remaining after a given contact time

$$\text{Demand} = \text{Dosage} - \text{Residual}$$

• 9

Chlorine Disinfection

- Chlorine Residual** = the total of all the compounds with disinfecting properties plus any remaining free (uncombined) chlorine

$$\text{Residual} = \text{Dosage} - \text{Demand}$$

- Total Chlorine** = The total concentration of chlorine in water, including the combined chlorine and the free available chlorine

• 10

Chlorine Disinfection

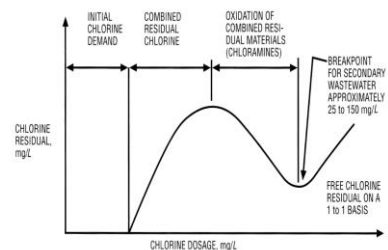
- Chlorine Dose** = the amount of chlorine needed to satisfy the chlorine demand and the amount of chlorine residual needed for disinfection

$$\text{Chlorine Dose} = \text{Demand} + \text{Residual}$$

• 11

Breakpoint Chlorination

- Adding chlorine to water until the chlorine demand has been satisfied
- Free available residual chlorine begins to form at the breakpoint



• 12

Breakpoint Chlorination

- <https://www.youtube.com/watch?v=Auz0cpObjl8>

• 13

Factors influencing disinfection

• • •

• 14

Factors influencing Disinfection

- pH
 - Chlorine disinfects faster at pH of 7 than at pH > 8
 - Hypochlorous acid disassociates at a higher pH
- Temperature
 - Higher temperature means more efficient disinfection
 - Longer contact time required at lower temperatures
 - Chlorine will dissipate faster in warmer waters
- Microorganisms
 - Number and type greatly influence disinfection effectiveness
 - Cysts and viruses can be very resistant to disinfection

• 15

Factors influencing Disinfection

- Turbidity
 - Excessive turbidity greatly reduces disinfection efficiency
 - Organic Matter
 - Organics can consume great amounts of disinfectants while forming unwanted compounds such as disinfection by-products
 - Reactions with organics and other reducing agents will significantly reduce the amount chemical available for disinfection
 - Inorganic matter
 - Ammonia can combine with disinfectant chemical to form side compounds

• 16

Factors influencing disinfection

- Reducing Agents
 - Any substance that will readily donate electrons
 - Demand for chlorine by reducing agents must be met before chlorine becomes available to accomplish disinfection
 - Inorganic reducing agents
 - Hydrogen sulfide gas (H_2S)
 - Ferrous ion (Fe^{2+})
 - Manganous ion (Mn^{2+})
 - Ammonia (NH_3)
 - Nitrite ion (NO_2^-)

• 17

Process of Disinfection

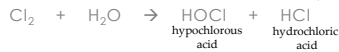
• • •

• 18

Chlorine (Cl₂)

Lowers pH

- Reaction with Water



- Free chlorine combines with water to form hypochlorous acid

- Most effective disinfectant
- Dissociates at higher pH (greater than 7)



hypochlorous acid hypochlorite ion

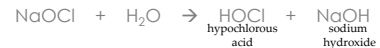
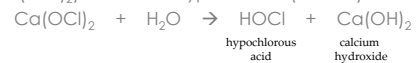
- Hypochlorous acid has a much higher disinfection potential than hypochlorite ion
- At pH = 7.5, of the chlorine present 50% will be HOCl and 50% will be OCl⁻

• 19

Hypochlorite (OCl⁻)

- Reactions with Water

- May be applied in the form of calcium hypochlorite (Ca(OCl)₂) or sodium hypochlorite (NaOCl)



- Raises pH due to OH⁻ ion
- If is Ca(OCl)₂ injected at the same point of as sodium fluoride, a severe crust can form at injection point

• 20

Reaction with Water

- The species formed is controlled by the pH of the water
 - Lower pH = more Hypochlorous Acid
 - Higher pH = more Hypochlorite ion
- Hypochlorous acid and Hypochlorite ion differ in disinfection ability
 - Hypochlorous acid has a much greater disinfection potential than Hypochlorite ion

• 21

Critical Factors

- Effectiveness of upstream processes
 - Solids can inhibit the disinfection process
- Injection point and mixing
 - Extremely important
- Temperature
 - Higher temp = more rapid rate of disinfection
- Dosage and type of chemical
- pH
 - Lower the better
- **Contact time**
 - Longer the better
- **Residual Chlorine**

• 22

Importance to Drinking Water

- Surface Water Treatment Rule (SWTR) requires disinfection of all surface water supply systems as protection against exposure to viruses, bacteria, and Giardia
- Residual chlorine in drinking water is required to prevent the growth of pathogens as water is moving through distribution system
- When chlorine comes in contact with organics, Disinfection By-Products (DBPs) are formed
 - Potential carcinogens

• 23

Importance to Wastewater

- Plant effluent is usually disinfected before discharge
- NPDES permits have a limit on *E.coli* or fecal coliforms
- Water must then be dechlorinated prior to discharge into receiving waters
 - Chlorine could harm the aquatic organisms
 - Sulfur dioxide is common dechlorinating agent

• 24

Approved Methods

- Amperometric – SM 4500-Cl D. & E. 2011
- Iodometric (TRC) – SM 4500-Cl B. 2011
- Back titration ether endpoint (TRC) – SM 4500-Cl C. 2011
- DPD-FAS – SM 4500-Cl F. 2011
- Spectrophotometric, DPD – SM 4500-Cl G. 2011

Note: Color Comparator is **NOT** an approved method

• 25

DPD Method

- Standard Method 4500-Cl G
- Grab sample, no preservative
- Analyze samples immediately (holding time is **15 minutes**)
 - After adding the reagent, a pink color will develop if chlorine is present
 - Wipe the outside of the sample cell with a wet then a dry towel to remove fingerprints
 - *DPD - N,N Diethyl-1,4 Phenylenediamine Sulfate*

DPD Method - continued

- Hach Procedure:
 - Add DPD to sample and swirl for 20 seconds to mix
 - Total Chlorine: Wait for a three-minute reaction period
 - Use a timer
 - Within three minutes after timer has ended, read sample
 - Free Chlorine: Read immediately (within 1 minute)

DPD Method - continued

- Interferences
 - Alkalinity > 300 mg/L as CaCO₃
 - Extreme pH: adjust to 6-7 using sulfuric acid or sodium hydroxide (1N)
- Sampling
 - Avoid plastic containers
 - If sampling from a tap, let the water flow at least 5 minutes to ensure a representative sample
 - Avoid agitating sample

Amperometric Titration

- Most sensitive and most complex method
- Least affected by interferences
- Training in proper determination technique
- Titrant initially verified and periodically checked
- Fresh titrant and proper buret
- Titrant storage – dark and cool



Amperometric Titration

- Apparatus
 - Amperometric Titrator (Wallace & Tiernan)
 - Buret with 0.01 mL increments
- Reagents
 - Phenylarsine oxide (PAO) titrant, 0.00564N
 - Potassium Iodide solution (KI solution)
 - Acetate Buffer solution

Amperometric Titration - Procedure

- Fill burette with 0.0056N phenylarsine oxide solution (PAO)
- Measure 200 mL of sample into the cell and place in the holder on the titrator
- Add 1 mL Potassium Iodide (KI) solution (5% solution)
- Add 1 mL acetate buffer solution
- Turn on stirrer and adjust control knob until the meter reads the maximum on the scale

Amperometric Titration - Procedure

- Add phenylarsine oxide in 0.01 mL increments
 - This should cause the meter reading to deflect downward
 - Adjust the control knob as needed to keep the pointer on the scale
 - The end-point is reached when the addition of titrant no longer results in a downward deflection.
- Read the burette, subtracting the amount of the last addition (which did not cause a downward deflection)
 - The burette reading in mL equals the free chlorine residual in mg/L

Common Deficiencies

- Sampling and analyses times were not documented for field parameters
- Standards weren't analyzed to verify the accuracy of the chlorine meter
- Measuring free residual chlorine instead of total (and vice versa)
- Non-approved method being used to measure TRC
- TRC was being measured on the composite sample

Chlorine Problems

Oh no, not math problems!!



Chlorine Problems

- A chlorinator is set to feed 50 pounds of chlorine per 24 hours. The wastewater flow rate is 0.85 MGD. The chlorine measured after 30 minutes of contact time is 0.5 mg/L. Find the chlorine dosage and demand in mg/L.

$$\text{dose, mg/L} = \frac{\text{chlorine, lb/day}}{(Q, \text{MGD})(8.34 \text{ lb/day})}$$

$$\text{dose, mg/L} = \frac{50 \text{ lbs/day}}{(0.85 \text{ MGD})(8.34 \text{ lbs/gal})}$$

$$\text{dose, mg/L} = 7.1 \text{ mg/L}$$

Chlorine Problems

- A chlorinator is set to feed 50 pounds of chlorine per 24 hours. The wastewater flow rate is 0.85 MGD. The chlorine measured after 30 minutes of contact time is 0.5 mg/L. Find the chlorine dosage and demand in mg/L.

$$\text{Demand, mg/L} = \text{Cl}_2 \text{ Dose, mg/L} - \text{Cl}_2 \text{ Residual, mg/L}$$

$$\text{Demand, mg/L} = 7.1 \text{ mg/L} - 0.5 \text{ mg/L}$$

$$\text{Demand, mg/L} = 6.6 \text{ mg/L}$$

Chlorine Problems

- The chlorine demand is determined to be **5 mg/L** and the plant flow rate is 8 MGD. How many pounds per day of gas chlorine should be fed? Include a **1 mg/L** residual.

$$\text{Cl}_2, \text{ lbs/day} = (\text{Dose, mg/L})(Q, \text{ MGD})(8.34 \text{ lbs/gal})$$

$$\text{Cl}_2, \text{ lbs/day} = (**6 \text{ mg/L}**)(8 \text{ MGD})(8.34 \text{ lbs/gal})$$

$$\text{Cl}_2, \text{ lbs/day} = 400 \text{ lbs/day}$$

Chlorine Problems

- The chlorine demand is determined to be 5 mg/L and the plant flow rate is 8 MGD. How many pounds per day of HTH (65% chlorine) should be fed? Include a 1 mg/L residual.

$$\text{Cl}_2, \text{ lbs/day} = \frac{(\text{Dose, mg/L})(Q, \text{ MGD})(8.34 \text{ lbs/gal})}{\text{HTH, chlorine percent as decimal}}$$

$$\text{Cl}_2, \text{ lbs/day} = \frac{(6 \text{ mg/L})(8 \text{ MGD})(8.34 \text{ lbs/gal})}{0.65}$$

$$\text{Cl}_2, \text{ lbs/day} = 616 \text{ lbs/day}$$

Chlorination – Review Questions

1. Water is the universal _____.
2. The destruction of all pathogenic microorganisms is called _____, which is not to be confused with _____, in which all microorganisms (pathogenic **and** nonpathogenic) are destroyed.
3. What is meant by “breakpoint chlorination?”
4. How do you determine the Chlorine Dose?
5. Explain why each of these factors that influence disinfection are important:
 - a. pH
 - b. Temperature
 - c. Microorganisms
 - d. Turbidity
 - e. Reducing agents
6. When chlorine is added to water, it breaks down into what two products? Which of these products is a more effective disinfectant?

7. Why are contact time and residual chlorine levels considered critical factors in the disinfection process?
8. Which of the following is not an approved method for chlorine analysis?
- Amperometric titration
 - DPD Colorimetric
 - DPD Color Comparator
 - DPD Titrimetric
 - Ion Specific Electrode
9. When analyzing Total Chlorine using the Hach procedure (method 8167), you can read the sample immediately (within 1 minute) of adding the DPD pillow. True or False
10. A water sample is tested and found to have a chlorine demand of 1.7 mg/L. If the desired chlorine residual is 0.9 mg/L, what is the desired chlorine dose (in mg/L)?
11. The chlorine dosage for water is 2.7 mg/L. If the chlorine residual after a 30 minute contact time is found to be 0.7 mg/L, what is the chlorine demand (in mg/L)?
12. What should the chlorinator setting be (in pounds per day) to treat a flow of 2.35 MGD if the chlorine demand is 3.2 mg/L and a chlorine residual of 0.9 mg/L is desired?

Chlorine, Free

USEPA DPD Method¹

0.02 to 2.00 mg/L Cl₂

Method 8021

Powder Pillows or AccuVac[®] Ampuls

Scope and application: For testing free chlorine (hypochlorous acid and hypochlorite ion) in water, treated waters, estuary and seawater. USEPA accepted for reporting for drinking water analyses.² This product has not been evaluated to test for chlorine and chloramines in medical applications in the United States.

¹ Adapted from Standard Methods for the Examination of Water and Wastewater.

² Procedure is equivalent to USEPA and Standard Method 4500-Cl G for drinking water.



Test preparation

Instrument-specific information

Table 1 shows sample cell and orientation requirements for reagent addition tests, such as powder pillow or bulk reagent tests. Table 2 shows sample cell and adapter requirements for AccuVac Ampul tests. The tables also show all of the instruments that have the program for this test.

To use the table, select an instrument, then read across to find the applicable information for this test.

Table 1 Instrument-specific information for reagent addition

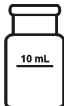



Instrument	Sample cell orientation	Sample cell
DR 6000 DR 3800 DR 2800 DR 2700 DR 1900	The fill line is to the right.	2495402 
DR 5000 DR 3900	The fill line is toward the user.	
DR 900	The orientation mark is toward the user.	2401906 

Table 2 Instrument-specific information for AccuVac Ampuls

Instrument	Adapter	Sample cell
DR 6000 DR 5000 DR 900	—	2427606 
DR 3900	LZV846 (A)	
DR 1900	9609900 or 9609800 (C)	
DR 3800 DR 2800 DR 2700	LZV584 (C)	2122800 

Before starting

Samples must be analyzed immediately after collection and cannot be preserved for later analysis.

Install the instrument cap on the DR 900 cell holder before ZERO or READ is pushed.

Do not use the same sample cells for free and total chlorine. If trace iodide from the total chlorine reagent is carried over into the free chlorine determination, monochloramine will interfere. It is best to use separate, dedicated sample cells for free and total chlorine measurements.

If the test result is over-range, or if the sample temporarily turns yellow after the reagent addition, dilute the sample with a known volume of high quality, chlorine demand-free water and do the test again. Some loss of chlorine may occur due to the dilution. Multiply the result by the dilution factor. Additional methods are available to measure chlorine without dilution.

For the best results, measure the reagent blank value for each new lot of reagent. Replace the sample with deionized water in the test procedure to determine the reagent blank value. Subtract the reagent blank value from the sample results automatically with the reagent blank adjust option.

An AccuVac Ampule for Blanks can be used to zero the instrument in the AccuVac test procedure.

Review the Safety Data Sheets (MSDS/SDS) for the chemicals that are used. Use the recommended personal protective equipment.

Dispose of reacted solutions according to local, state and federal regulations. Refer to the Safety Data Sheets for disposal information for unused reagents. Refer to the environmental, health and safety staff for your facility and/or local regulatory agencies for further disposal information.

The SwifTest Dispenser for Free Chlorine can be used in place of the powder pillow in the test procedure.

Items to collect

Powder pillows

Description	Quantity
DPD Free Chlorine Reagent Powder Pillows, 10-mL	1
Sample cells. (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	2

Refer to [Consumables and replacement items](#) on page 6 for order information.

AccuVac Ampuls

Description	Quantity
DPD Free Chlorine Reagent AccuVac Ampuls	1
Beaker, 50-mL	1
Sample cells (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	1
Stopper for 18-mm tubes and AccuVac Ampuls	1

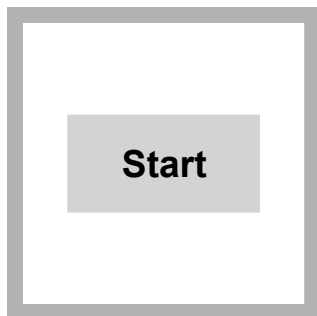
Refer to [Consumables and replacement items](#) on page 6 for order information.

Sample collection

- Analyze the samples immediately. The samples cannot be preserved for later analysis.
- Chlorine is a strong oxidizing agent and is unstable in natural waters. Chlorine reacts quickly with various inorganic compounds and more slowly with organic compounds. Many factors, including reactant concentrations, sunlight, pH, temperature and salinity influence the decomposition of chlorine in water.
- Collect samples in clean glass bottles. Do not use plastic containers because these can have a large chlorine demand.

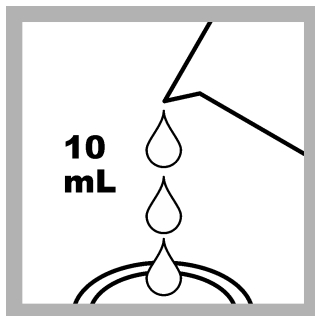
- Pretreat glass sample containers to remove chlorine demand. Soak the containers in a weak bleach solution (1 mL commercial bleach to 1 liter of deionized water) for at least 1 hour. Rinse fully with deionized or distilled water. If sample containers are rinsed fully with deionized or distilled water after use, only occasional pretreatment is necessary.
- Make sure to get a representative sample. If the sample is taken from a spigot or faucet, let the water flow for at least 5 minutes. Let the container overflow with the sample several times and then put the cap on the sample container so that there is no headspace (air) above the sample.

Powder pillow procedure

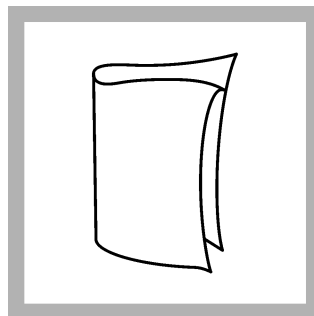


1. Start program **80 Chlorine F&T PP**. For information about sample cells, adapters or light shields, refer to [Instrument-specific information](#) on page 1.

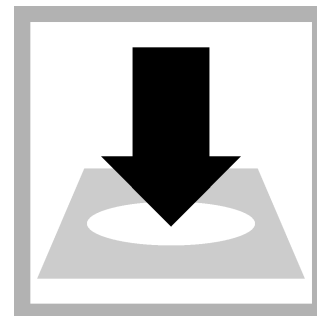
Note: Although the program name can be different between instruments, the program number does not change.



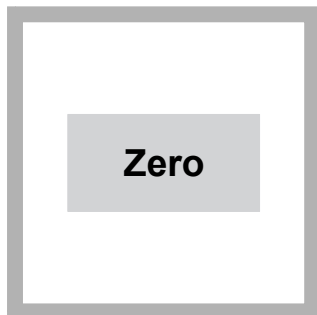
2. **Prepare the blank:** Fill the sample cell with 10 mL of sample.



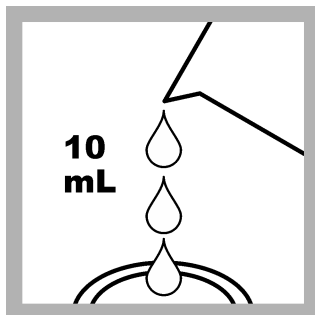
3. Clean the prepared sample cell.



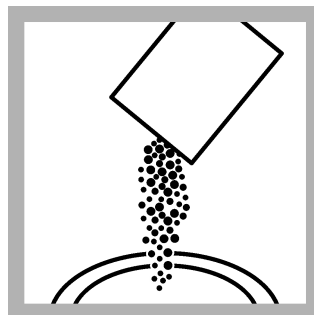
4. Insert the blank into the cell holder.



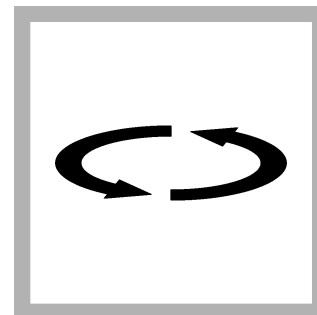
5. Push **ZERO**. The display shows 0.00 mg/L.



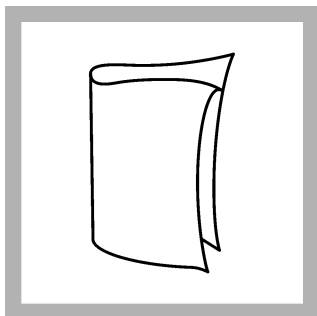
6. **Prepare the sample:** Fill a second sample cell with 10 mL of sample.



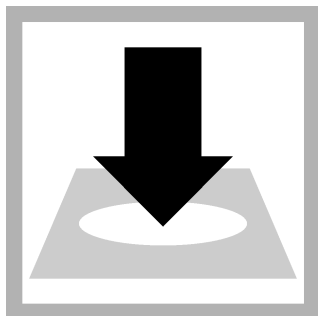
7. Add the contents of one powder pillow to the sample cell.



8. Swirl the sample cell for 20 seconds to mix. A pink color will develop if chlorine is present. Proceed to the next step immediately.



9. Clean the prepared sample cell.

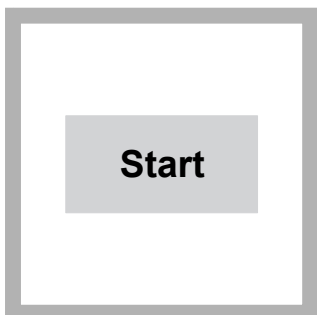


10. Within 60 seconds of the reagent addition, insert the prepared sample into the cell holder.



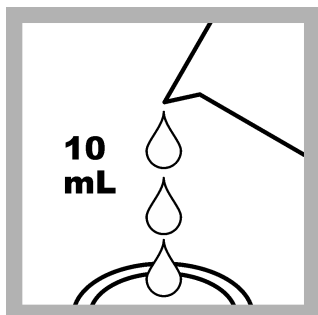
11. Push **READ**. Results show in mg/L Cl₂.

AccuVac Ampul procedure

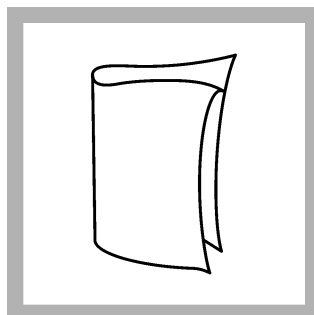


1. Start program **85 Chlorine F&T AV**. For information about sample cells, adapters or light shields, refer to [Instrument-specific information](#) on page 1.

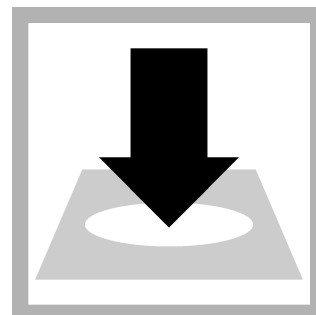
Note: Although the program name can be different between instruments, the program number does not change.



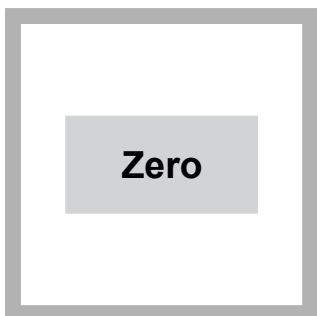
2. **Prepare the blank:** Fill the sample cell with 10 mL of sample.



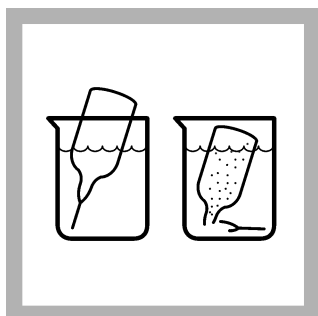
3. Clean the blank sample cell.



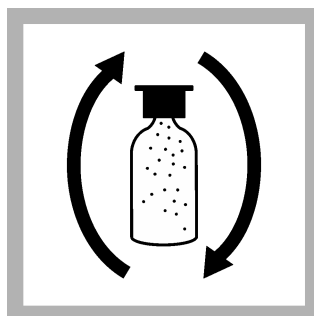
4. Insert the blank into the cell holder.



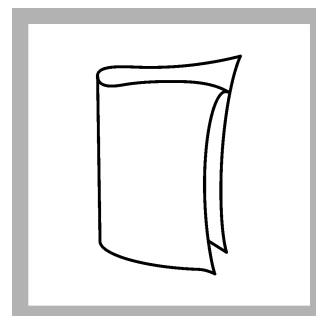
5. Push **ZERO**. The display shows 0.00 mg/L.



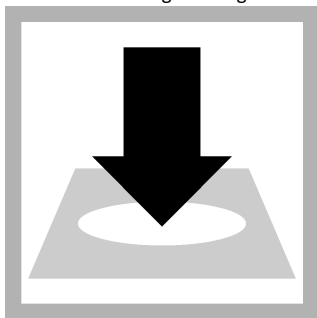
6. **Prepare the sample:** Collect at least 40 mL of sample in a 50-mL beaker. Fill the AccuVac Ampul with sample. Keep the tip immersed while the AccuVac Ampul fills completely.



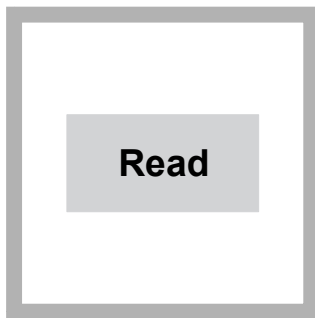
7. Quickly invert the AccuVac Ampul several times to mix.



8. Clean the AccuVac Ampul.



9. Within 60 seconds of the reagent addition, insert the prepared sample AccuVac Ampul into the cell holder.



10. Push **READ**. Results show in mg/L Cl_2 .

Interferences

Interfering substance	Interference level
Acidity	More than 150 mg/L CaCO_3 . The full color may not develop or the color may fade instantly. Adjust to pH 6–7 with 1 N Sodium Hydroxide. Measure the amount to add on a separate sample aliquot, then add the same amount to the sample that is tested. Correct the test result for the dilution from the volume addition.
Alkalinity	More than 250 mg/L CaCO_3 . The full color may not develop or the color may fade instantly. Adjust to pH 6–7 with 1 N Sulfuric Acid. Measure the amount to add on a separate sample aliquot, then add the same amount to the sample that is tested. Correct the test result for the dilution from the volume addition.
Bromine, Br_2	Positive interference at all levels
Chlorine Dioxide, ClO_2	Positive interference at all levels
Inorganic chloramines	Positive interference at all levels
Chloramines, organic	May interfere
Hardness	No effect at less than 1000 mg/L as CaCO_3
Manganese, Oxidized (Mn^{4+} , Mn^{7+}) or Chromium, Oxidized (Cr^{6+})	Pre-treat the sample as follows: <ol style="list-style-type: none"> 1. Adjust the sample pH to 6–7. 2. Add 3 drops of Potassium Iodide (30-g/L) to 10 mL of sample. 3. Mix and wait 1 minute. 4. Add 3 drops of Sodium Arsenite (5-g/L) and mix. 5. Use the test procedure to measure the concentration of the treated sample. 6. Subtract this result from the result without the treatment to obtain the correct chlorine concentration.
Monochloramine	Causes a gradual drift to higher readings. When read within 1 minute after reagent addition, 3 mg/L monochloramine causes less than a 0.1 mg/L increase in the reading.
Ozone	Positive interference at all levels
Peroxides	May interfere
Highly buffered samples or extreme sample pH	Can prevent the correct pH adjustment of the sample by the reagents. Sample pre-treatment may be necessary. Adjust to pH 6–7 with acid (Sulfuric Acid, 1.000 N) or base (Sodium Hydroxide, 1.00 N).

Accuracy check

Standard additions method (sample spike)

Use the standard additions method (for applicable instruments) to validate the test procedure, reagents and instrument and to find if there is an interference in the sample.

Items to collect:

- Chlorine Standard Solution, 2-mL PourRite® Ampule, 25–30 mg/L (use mg/L on label)
- Breaker, PourRite Ampules
- Pipet, TenSette®, 0.1–1.0 mL and tips

1. Use the test procedure to measure the concentration of the sample, then keep the (unspiked) sample in the instrument.
2. Go to the Standard Additions option in the instrument menu.
3. Select the values for standard concentration, sample volume and spike volumes.
4. Open the standard solution.
5. Prepare three spiked samples: use the TenSette pipet to add 0.1 mL, 0.2 mL and 0.3 mL of the standard solution, respectively, to three 10-mL portions of fresh sample. Mix well.

Note: For AccuVac® Ampuls, add 0.4 mL, 0.8 mL and 1.2 mL of the standard solution to three 50-mL portions of fresh sample.

6. Use the test procedure to measure the concentration of each of the spiked samples. Start with the smallest sample spike. Measure each of the spiked samples in the instrument.
7. Select **Graph** to compare the expected results to the actual results.

Note: If the actual results are significantly different from the expected results, make sure that the sample volumes and sample spikes are measured accurately. The sample volumes and sample spikes that are used should agree with the selections in the standard additions menu. If the results are not within acceptable limits, the sample may contain an interference.

Method performance

The method performance data that follows was derived from laboratory tests that were measured on a spectrophotometer during ideal test conditions. Users can get different results under different test conditions.

Program	Standard	Precision (95% Confidence Interval)	Sensitivity Concentration change per 0.010 Abs change
80	1.25 mg/L Cl ₂	1.23–1.27 mg/L Cl ₂	0.02 mg/L Cl ₂
85	1.25 mg/L Cl ₂	1.21–1.29 mg/L Cl ₂	0.02 mg/L Cl ₂

Summary of method

Chlorine in the sample as hypochlorous acid or hypochlorite ion (free chlorine or free available chlorine) immediately reacts with DPD (N,N-diethyl-p-phenylenediamine) indicator to form a pink color, the intensity of which is proportional to the chlorine concentration. The measurement wavelength is 530 nm for spectrophotometers or 520 nm for colorimeters.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Item no.
DPD Free Chlorine Reagent Powder Pillow, 10-mL	1	100/pkg	2105569
OR			
DPD Free Chlorine Reagent AccuVac® Ampul	1	25/pkg	2502025

Required apparatus

Description	Quantity/Test	Unit	Item no.
AccuVac Snapper	1	each	2405200
Beaker, 50-mL	1	each	50041H
Stoppers for 18-mm tubes and AccuVac Ampuls	2	6/pkg	173106

Recommended standards

Description	Unit	Item no.
Chlorine Standard Solution, 2-mL PourRite [®] Ampules, 25–30 mg/L	20/pkg	2630020

Optional reagents and apparatus

Description	Unit	Item no.
AccuVac [®] Ampul vials for sample blanks	25/pkg	2677925
Ampule Breaker, 2-mL PourRite [®] Ampules	each	2484600
Ampule Breaker, 10-mL Voluette [®] Ampules	each	2196800
Water, Chlorine-demand Free	500 mL	2641549
Mixing cylinder, graduated, 25-mL	each	2088640
Mixing cylinder, graduated, 50-mL	each	189641
Chlorine Standard Solution, 2-mL PourRite [®] Ampules, 50–75 mg/L	20/pkg	1426820
Chlorine Standard Solution, 10-mL Voluette [®] Ampule, 50–75 mg/L	16/pkg	1426810
DPD Free Chlorine Reagent Powder Pillows, 10-mL	1000/pkg	2105528
DPD Free Chlorine Reagent Powder Pillows, 10-mL	300/pkg	2105503
DPD Free Chlorine Reagent, 10-mL, SwifTest [™] Dispenser refill vial	250 tests	2105560
Paper, pH, 0–14 pH range	100/pkg	2601300
Pipet, TenSette [®] , 0.1–1.0 mL	each	1970001
Pipet tips for TenSette [®] Pipet, 0.1–1.0 mL	50/pkg	2185696
Pipet tips for TenSette [®] Pipet, 0.1–1.0 mL	1000/pkg	2185628
Potassium Iodide, 30-g/L	100 mL	34332
Sodium Arsenite, 5-g/L	100 mL	104732
Sodium Hydroxide Standard Solution, 1.0 N	100 mL MDB	104532
SpecCheck [™] Secondary Standard Kit, Chlorine DPD, 0–2.0 mg/L Set	each	2635300
Sulfuric Acid Standard Solution, 1 N	100 mL MDB	127032



FOR TECHNICAL ASSISTANCE, PRICE INFORMATION AND ORDERING:
In the U.S.A. – Call toll-free 800-227-4224
Outside the U.S.A. – Contact the HACH office or distributor serving you.
On the Worldwide Web – www.hach.com; E-mail – techhelp@hach.com

HACH COMPANY
WORLD HEADQUARTERS
Telephone: (970) 669-3050
FAX: (970) 669-2932

Chlorine, Total

USEPA DPD Method¹

0.02 to 2.00 mg/L Cl₂

Method 8167

Powder Pillows or AccuVac[®] Ampuls

Scope and application: For testing residual chlorine and chloramines in water, wastewater, estuary water and seawater; USEPA-accepted for reporting for drinking and wastewater analyses.² This product has not been evaluated to test for chlorine and chloramines in medical applications in the United States.

¹ Adapted from Standard Methods for the Examination of Water and Wastewater.

² Procedure is equivalent to USEPA and Standard Method 4500-Cl G for drinking water and wastewater analysis.



Test preparation

Instrument-specific information

Table 1 shows sample cell and orientation requirements for reagent addition tests, such as powder pillow or bulk reagent tests. Table 2 shows sample cell and adapter requirements for AccuVac Ampul tests. The tables also show all of the instruments that have the program for this test.

To use the table, select an instrument, then read across to find the applicable information for this test.

Table 1 Instrument-specific information for reagent addition





Instrument	Sample cell orientation	Sample cell
DR 6000 DR 3800 DR 2800 DR 2700 DR 1900	The fill line is to the right.	2495402 
DR 5000 DR 3900	The fill line is toward the user.	
DR 900	The orientation mark is toward the user.	2401906 

Table 2 Instrument-specific information for AccuVac Ampuls

Instrument	Adapter	Sample cell
DR 6000 DR 5000 DR 900	—	2427606 
DR 3900	LZV846 (A)	
DR 1900	9609900 or 9609800 (C)	
DR 3800 DR 2800 DR 2700	LZV584 (C)	2122800 

Before starting

Analyze the samples immediately. The samples cannot be preserved for later analysis.

Install the instrument cap on the DR 900 cell holder before ZERO or READ is pushed.

If the test result is over-range, or if the sample temporarily turns yellow after the reagent addition, dilute the sample with a known volume of high quality, chlorine demand-free water and do the test again. Some loss of chlorine may occur due to the dilution. Multiply the result by the dilution factor. Additional methods are available to measure chlorine without dilution.

For chloramination disinfection control, use one of the available Chloramine (Mono) methods.

For the best results, measure the reagent blank value for each new lot of reagent. Replace the sample with deionized water in the test procedure to determine the reagent blank value. Subtract the reagent blank value from the sample results automatically with the reagent blank adjust option.

Review the Safety Data Sheets (MSDS/SDS) for the chemicals that are used. Use the recommended personal protective equipment.

Dispose of reacted solutions according to local, state and federal regulations. Refer to the Safety Data Sheets for disposal information for unused reagents. Refer to the environmental, health and safety staff for your facility and/or local regulatory agencies for further disposal information.

The SwifTest Dispenser for Total Chlorine can be used in place of the powder pillow in the test procedure. One dispensation is equal to one powder pillow for 10-mL samples.

An AccuVac Ampul for Blanks can be used to zero the instrument in the AccuVac test procedure.

Items to collect

Powder pillows

Description	Quantity
DPD Total Chlorine Reagent Powder Pillow, 10-mL	1
Sample cells. (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	2

Refer to [Consumables and replacement items](#) on page 6 for order information.

AccuVac Ampuls

Description	Quantity
DPD Total Chlorine Reagent AccuVac [®] Ampul	1
Beaker, 50-mL	1
Sample cells (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	1
Stopper for 18-mm tubes and AccuVac Ampuls	1

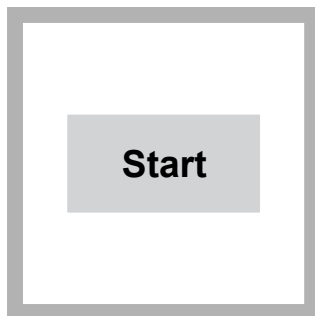
Refer to [Consumables and replacement items](#) on page 6 for order information.

Sample collection

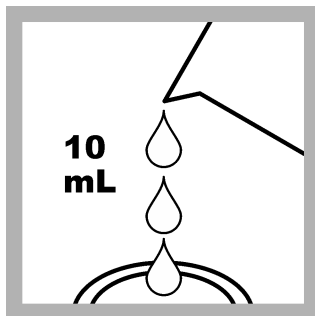
- Analyze the samples immediately. The samples cannot be preserved for later analysis.
- Chlorine is a strong oxidizing agent and is unstable in natural waters. Chlorine reacts quickly with various inorganic compounds and more slowly with organic compounds. Many factors, including reactant concentrations, sunlight, pH, temperature and salinity influence the decomposition of chlorine in water.
- Collect samples in clean glass bottles. Do not use plastic containers because these can have a large chlorine demand.

- Pretreat glass sample containers to remove chlorine demand. Soak the containers in a weak bleach solution (1 mL commercial bleach to 1 liter of deionized water) for at least 1 hour. Rinse fully with deionized or distilled water. If sample containers are rinsed fully with deionized or distilled water after use, only occasional pretreatment is necessary.
- Make sure to get a representative sample. If the sample is taken from a spigot or faucet, let the water flow for at least 5 minutes. Let the container overflow with the sample several times and then put the cap on the sample container so that there is no headspace (air) above the sample.

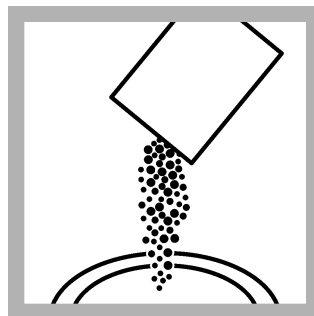
Powder pillow procedure



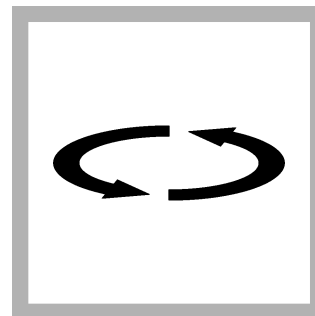
1. Start program **80 Chlorine F&T PP**. For information about sample cells, adapters or light shields, refer to [Instrument-specific information](#) on page 1.



2. Fill a sample cell with 10 mL of sample.



3. **Prepare the sample:** Add the contents of one powder pillow to the sample cell.

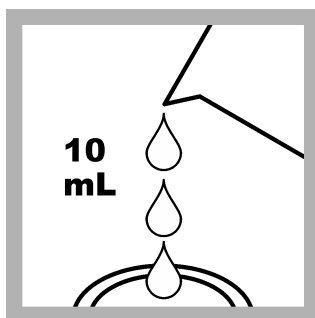


4. Swirl the sample cell for 20 seconds to mix. A pink color shows if chlorine is present in the sample.

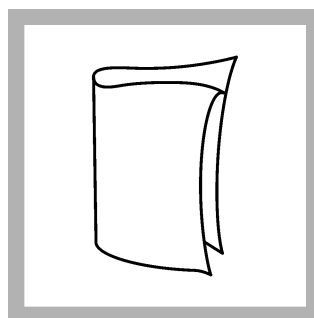


5. Start the instrument timer. A 3-minute reaction time starts.

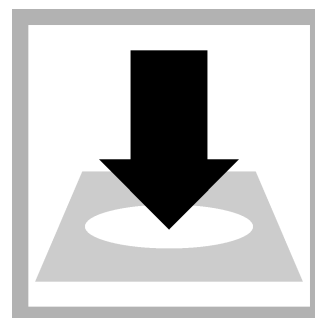
Prepare the sample blank and set the instrument to zero during the reaction time.



6. **Prepare the blank:** Fill a second sample cell with 10 mL of sample.



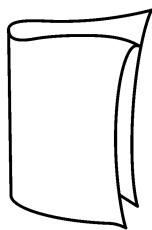
7. Clean the blank sample cell.



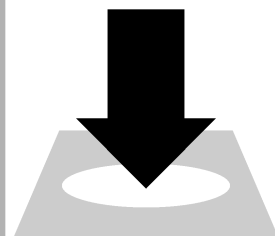
8. Insert the blank into the cell holder.

Zero

9. Push **ZERO**. The display shows 0.00 mg/L Cl₂.



10. Clean the prepared sample cell.



11. Within 3 minutes after the timer expires, insert the prepared sample into the cell holder.

Read

12. Push **READ**. Results show in mg/L Cl₂.

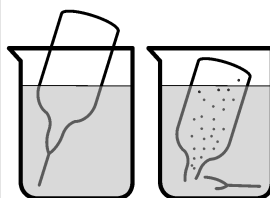
AccuVac Ampul procedure

Start

1. Start program **85 Chlorine F&T AV**. For information about sample cells, adapters or light shields, refer to [Instrument-specific information](#) on page 1.

10 mL

2. **Prepare the blank:** Fill the sample cell with 10 mL of sample.



3. **Prepare the sample:** Collect at least 40 mL of sample in a 50-mL beaker. Fill the AccuVac Ampul with sample. Keep the tip immersed while the AccuVac Ampul fills completely.

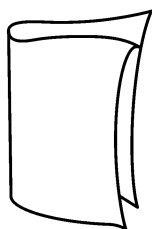


4. Quickly invert the AccuVac Ampul several times to mix.

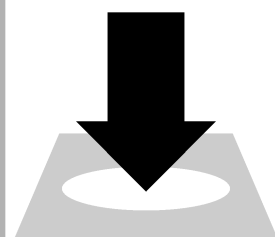
03:00

5. Start the instrument timer. A 3-minute reaction time starts.

Prepare the sample blank and set the instrument to zero during the reaction time.



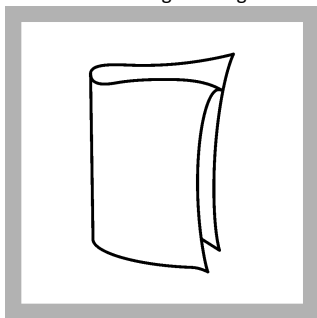
6. Clean the blank sample cell.



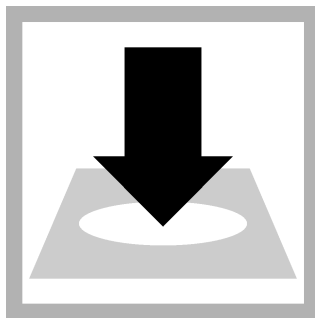
7. Insert the blank into the cell holder.

Zero

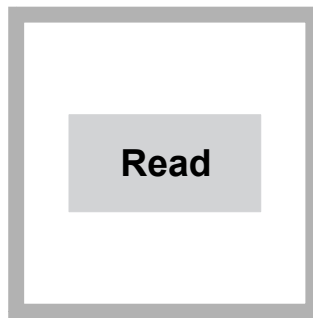
8. Push **ZERO**. The display shows 0.00 mg/L Cl₂.



9. Clean the AccuVac Ampul.



10. Within 3 minutes after the timer expires, insert the prepared sample AccuVac Ampul into the cell holder.



11. Push **READ**. Results show in mg/L Cl_2 .

Interferences

Interfering substance	Interference level
Acidity	More than 150 mg/L CaCO_3 . The full color may not develop or the color may fade instantly. Adjust to pH 6–7 with 1 N Sodium Hydroxide. Measure the amount to add on a separate sample aliquot, then add the same amount to the sample that is tested. Correct the test result for the dilution from the volume addition.
Alkalinity	More than 250 mg/L CaCO_3 . The full color may not develop or the color may fade instantly. Adjust to pH 6–7 with 1 N Sulfuric Acid. Measure the amount to add on a separate sample aliquot, then add the same amount to the sample that is tested. Correct the test result for the dilution from the volume addition.
Bromine, Br_2	Positive interference at all levels
Chlorine Dioxide, ClO_2	Positive interference at all levels
Inorganic chloramines	Positive interference at all levels
Chloramines, organic	May interfere in the result for total chlorine analysis
Hardness	No effect at less than 1000 mg/L as CaCO_3
Manganese, Oxidized (Mn^{4+} , Mn^{7+}) or Chromium, Oxidized (Cr^{6+})	Pre-treat the sample as follows: <ol style="list-style-type: none"> 1. Adjust the sample pH to 6–7. 2. Add 3 drops of Potassium Iodide (30-g/L) to 10 mL of sample. 3. Mix and wait 1 minute. 4. Add 3 drops of Sodium Arsenite (5-g/L) and mix. 5. Use the test procedure to measure the concentration of the treated sample. 6. Subtract this result from the result without the treatment to obtain the correct chlorine concentration.
Ozone	Positive interference at all levels
Peroxides	May interfere
Highly buffered samples or extreme sample pH	Can prevent the correct pH adjustment (of the sample) by the reagents. Sample pretreatment may be necessary. Adjust to pH 6–7 with acid (Sulfuric Acid, 1.000 N) or base (Sodium Hydroxide, 1.00 N).

Accuracy check

Standard additions method (sample spike)

Use the standard additions method (for applicable instruments) to validate the test procedure, reagents and instrument and to find if there is an interference in the sample.

Items to collect:

- Chlorine Standard Solution, 2-mL PourRite® Ampule, 25–30 mg/L (use mg/L on label)
- Breaker, PourRite Ampules
- Pipet, TenSette®, 0.1–1.0 mL and tips

1. Use the test procedure to measure the concentration of the sample, then keep the (unspiked) sample in the instrument.
2. Go to the Standard Additions option in the instrument menu.
3. Select the values for standard concentration, sample volume and spike volumes.
4. Open the standard solution.
5. Prepare three spiked samples: use the TenSette pipet to add 0.1 mL, 0.2 mL and 0.3 mL of the standard solution, respectively, to three 10-mL portions of fresh sample. Mix well.

Note: For AccuVac® Ampuls, add 0.4 mL, 0.8 mL and 1.2 mL of the standard solution to three 50-mL portions of fresh sample.

6. Use the test procedure to measure the concentration of each of the spiked samples. Start with the smallest sample spike. Measure each of the spiked samples in the instrument.
7. Select **Graph** to compare the expected results to the actual results.

Note: If the actual results are significantly different from the expected results, make sure that the sample volumes and sample spikes are measured accurately. The sample volumes and sample spikes that are used should agree with the selections in the standard additions menu. If the results are not within acceptable limits, the sample may contain an interference.

Method performance

The method performance data that follows was derived from laboratory tests that were measured on a spectrophotometer during ideal test conditions. Users can get different results under different test conditions.

Program	Standard	Precision (95% Confidence Interval)	Sensitivity Concentration change per 0.010 Abs change
80	1.25 mg/L Cl ₂	1.23–1.27 mg/L Cl ₂	0.02 mg/L Cl ₂
85	1.25 mg/L Cl ₂	1.21–1.29 mg/L Cl ₂	0.02 mg/L Cl ₂

Summary of method

Chlorine can be present in water as free chlorine and as combined chlorine. Both forms can exist in the same water and be determined together as total chlorine. Free chlorine is present as hypochlorous acid and/or hypochlorite ion. Combined chlorine exists as monochloramine, dichloramine, nitrogen trichloride and other chloro derivatives. The combined chlorine oxidizes iodide in the reagent to iodine. The iodine and free chlorine react with DPD (N,N-diethyl-p-phenylenediamine) to form a pink color which is proportional to the total chlorine concentration.

To find the concentration of combined chlorine, run a free chlorine test and a total chlorine test. Subtract the results of the free chlorine test from the total chlorine test to obtain the combined chlorine concentration. The measurement wavelength is 530 nm for spectrophotometers or 520 nm for colorimeters.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Item no.
DPD Total Chlorine Reagent Powder Pillow, 10 mL	1	100/pkg	2105669
OR			
DPD Total Chlorine Reagent AccuVac® Ampul	1	25/pkg	2503025

Required apparatus

Description	Quantity/Test	Unit	Item no.
AccuVac Snapper	1	each	2405200
Beaker, 50 mL	1	each	50041H
Stoppers for 18-mm tubes and AccuVac Ampuls	2	6/pkg	173106

Recommended standards

Description	Unit	Item no.
Chlorine Standard Solution, 10-mL Voluette [®] Ampule, 50–75 mg/L	16/pkg	1426810
Chlorine Standard Solution, 2-mL PourRite [®] Ampules, 50–75 mg/L	20/pkg	1426820
Chlorine Standard Solution, 2-mL PourRite [®] Ampules, 25–30 mg/L	20/pkg	2630020

Optional reagents and apparatus

Description	Unit	Item no.
AccuVac [®] Ampul vials for sample blanks	25/pkg	2677925
Ampule Breaker, 2-mL PourRite [®] Ampules	each	2484600
Ampule Breaker, 10-mL Voluette [®] Ampules	each	2196800
Water, Chlorine-demand Free	500 mL	2641549
Mixing cylinder, graduated, 25-mL	each	2088640
Mixing cylinder, graduated, 50 mL	each	189641
DPD Total Chlorine Reagent Powder Pillows, 10 mL	1000/pkg	2105628
DPD Total Chlorine Reagent Powder Pillows, 10 mL	300/pkg	2105603
DPD Total Chlorine Reagent, 10-mL, SwifTest [™] Dispenser refill vial	250 tests	2105660
Paper, pH, 0–14 pH range	100/pkg	2601300
Pipet, TenSette [®] , 0.1–1.0 mL	each	1970001
Pipet tips for TenSette [®] Pipet, 0.1–1.0 mL	50/pkg	2185696
Pipet tips for TenSette [®] Pipet, 0.1–1.0 mL	1000/pkg	2185628
Potassium Iodide, 30-g/L	100 mL	34332
Sodium Arsenite, 5-g/L	100 mL	104732
Sodium Hydroxide Standard Solution, 1.0 N	100 mL MDB	104532
SpecCheck [™] Secondary Standard Kit, Chlorine DPD, 0–2.0 mg/L Set	each	2635300
Sulfuric Acid Standard Solution, 1 N	100 mL MDB	127032
Water, deionized	4 L	27256



FOR TECHNICAL ASSISTANCE, PRICE INFORMATION AND ORDERING:
In the U.S.A. – Call toll-free 800-227-4224
Outside the U.S.A. – Contact the HACH office or distributor serving you.
On the Worldwide Web – www.hach.com; E-mail – techhelp@hach.com



HACH COMPANY
WORLD HEADQUARTERS
Telephone: (970) 669-3050
FAX: (970) 669-2932

Section 13

Alkalinity

ALKALINITY

BASIC LAB CLASS

ALKALINITY

- Defined as the measurement of a water's capacity to neutralize an acid
- An acid releases H^+
- The alkalinity in the water will absorb H^+
- Most common ions that add alkalinity are
 - OH^-
 - CO_3^{2-}
 - HCO_3^-
 - the major form of alkalinity in natural waters

TDEC - Fleming Training Center 2

ALKALINITY

- Alkalinity caused by OH^- is called
 - *hydroxide alkalinity*
- Alkalinity caused by CO_3^{2-} is called
 - *carbonate alkalinity*
- Alkalinity caused by HCO_3^- is called
 - *bicarbonate alkalinity*
- The combined effect of all three types is called
 - *total alkalinity*

TDEC - Fleming Training Center 3

INTRODUCTION

- Alkalinity is a general measure of the ionic characteristics of water
- Others: pH, oxidation-reduction potential (a.k.a. redox potential), hardness, and conductivity
- Not normally a compliance-monitoring requirement

TDEC - Fleming Training Center 4

IMPORTANCE TO WASTEWATER

- Chemical and biological treatment systems
 - Better able to survive an acidic discharge
 - pH stabilization
- Biological Nutrient Removal
 - Nitrification/Denitrification
- Anaerobic Digester Control
 - Volatile Acids/Alkalinity Relationship
- Ammonia Removal by Air Stripping
- Organism Nutrition
- Potential to Affect Chlorine Demand

IMPORTANCE TO DRINKING WATER

- Coagulation process
 - Alum needs alkalinity to work
- Lime/Soda softening
- Buffering changes in pH in the system
- Hard water complaints
 - Lime scale build-up

ALKALINITY DETERMINATION

- Titration against a standard acid:
 - Color change of standard indicator
 - pH meter
- Results expressed as total alkalinity, mg/L as calcium carbonate (CaCO₃)
- Buret Titration Method, SM 2320 B

TDEC - Fleming Training Center 7

ALKALINITY DETERMINATION

- Measured by determining the amount of acid needed to drop the pH of a sample to a certain endpoint
 - Phenolphthalein alkalinity is measured by titrating to a pH of 8.3
 - Total alkalinity is measured by titrating to a pH of 4.5

TDEC - Fleming Training Center 8

ALKALINITY DETERMINATION

- Color Indicators:
 - Phenolphthalein
 - Metacresol purple

} Titration to 8.3
- Bromcresol green
- Mixed bromcresol green-methyl red

} Titration to 4.5

TDEC - Fleming Training Center 10

PH END POINTS (HACH METHOD 8221)

Sample Composition	End Point pH	
	Total Alkalinity	Phenolphthalein Alkalinity
Alk ~ 30 mg/L	pH 4.9	pH 8.3
Alk ~ 150 mg/L	pH 4.6	pH 8.3
Alk ~ 500 mg/L	pH 4.3	pH 8.3
Silicates or Phosphates present	pH 4.5	pH 8.3
Industrial Waste or Complex System	pH 4.5	pH 8.3
Routine or Automated Process	pH 4.5	pH 8.3

TDEC - Fleming Training Center 10


OTHER METHODS AVAILABLE

- Standard Method 2320 B. Titration method
- Hach Method 8221
- Hach Method 8203
- Orion pH probe
- Hach method 10239, TNT plus 870

TDEC - Fleming Training Center 11

APPARATUS

- Buret and stand
- Beaker, 250 mL
- Stir plate
- Stir bar



TDEC - Fleming Training Center 12

SAMPLING AND STORAGE

- Collect samples in clean plastic or glass bottles
- Avoid excessive agitation or prolonged exposure to air
- Analyze as soon as possible
 - May be stored for 24 hrs at 4°C
- Warm to room temperature before analysis.

TDEC - Fleming Training Center

13

INTERFERENCES

- Highly colored or turbid samples may mask the color change at the end point.
 - Use a pH meter for these samples.
- Chlorine may interfere with indicators.
 - Add one drop 0.1N sodium thiosulfate to eliminate this interference.

TDEC - Fleming Training Center

14

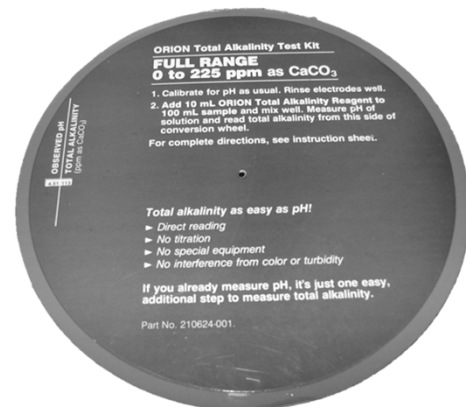
ORION PH PROBE

- Very simple
- No color change to watch
- Must have a properly calibrated pH meter
- By adding a reagent, the determination of alkalinity is made by measuring the drop in pH and comparing the measurement to a calibration chart that show the relationship between pH and alkalinity
- **Not an approved method for reporting.**



TDEC - Fleming Training Center

15



TDEC - Fleming Training Center

16

Alkalinity – Review Questions

1. Alkalinity is defined as what?
2. What are the most common ions that add alkalinity to water?
3. List at least 4 reasons why alkalinity is important to wastewater treatment.
4. List 3 reasons why alkalinity is important to drinking water treatment.
5. Phenolphthalein alkalinity is titrated to a pH endpoint of what?
6. Total alkalinity is titrated to a pH endpoint of what?
7. Alkalinity results are expressed in what unit?
8. What apparatus/equipment is needed to do an alkalinity titration?
9. What are 2 interferences with the alkalinity test?

Answers to Review Questions

Laboratory Safety

1. Poisons, explosions, cuts and bruises, electric shock, toxic fumes, fire, burns
2. Someone should always be there to help you in case you should have an accident that blinds you, leaves you unconscious, or starts a fire you cannot handle. If necessary, have someone check on you regularly.
3. True
4. You can dispose of small amounts of corrosive acids by pouring the neutralized acid down a corrosion-resistant sink (to a sewer) and using large quantities of water to dilute and flush the acid.
5. Safety Data Sheet
6. 30 years
7. Danger – more severe hazard, Warning – less severe hazard
8. Within one minute of contact, flush with copious amounts of water for at least 20 minutes. Consult doctor.
9. Immediately wash the area with water and neutralize the acid with sodium bicarbonate (baking soda) or bicarbonate
10. Gloves, safety glasses, apron, lab coat, face shield, closed toed shoes
11. Fume hood
12. Vinegar neutralizes bases, Baking soda neutralizes acids
13. Weekly
14. False – should be laid out to segregate incompatible chemicals
15. If incompatible chemicals are inadvertently mixed a fire, explosion, or toxic release can easily occur
16. Glass plate, wet towel, wet blanket
17. A – ordinary combustibles
B – Flammable and combustible liquids
C – Energized Electrical Equipment
D – Combustible metals
18. Pull, Aim, Squeeze, Sweep
19. Teratogen = reproductive toxins that may cause damage to the fetus

Laboratory Equipment

1. Beakers
2. Volumetric glassware
3. When you are making standards
4.
 - Volumetric – do not blow out
 - Mohr – do not blow out
 - Serological – Yes, blow out
5. Place them into soapy water, tip up in a pipet cleaner. Or lay them into a dish filled with soapy water. Phosphate free, lab grade soap.

6. 3 minutes
7. Analytical is more precise, can weigh down to 0.0001g vs. 0.01g for top loading.
8. Weekly
9. Phosphate free (Ex: Liquinox)
10. Detergent, tap water, rinse 3 times with DI water, air dry
11. To remove any built-up residue that could be causing water to bead up. To ensure glassware is as clean as possible.
12.
 - Clean glassware using laboratory detergent (phosphate-free)
 - Rinse with tap water
 - Rinse with 1:1 hydrochloric acid or nitric acid
 - 1:1 means equal parts distilled water and acid
 - Rinse well with distilled water
 - Let air dry
13. Monthly

Metric System and Conversions

See end of section for answers.

Weight and Volume Measurement

1. Analytical balance
2.
 - It prevents contamination of the weighed material
 - Chemicals placed directly on the pan may damage or corrode the balance pan itself
 - Using a tare allows chemicals to be easily and accurately transferred from the pan
3. Annually by an outside contractor who specializes in laboratory equipment
4. NIST Class S-1 or ANSI Class 1 weights, calibrated every 5 years
5. Moisture and oils from your fingers will change the measured value of the weights
6. False
7. Volumetric glassware: flasks, pipets, burettes
8. Pour reagent directly into the flask
 Add distilled or DI water up to neck of the flask and carefully fill to the mark
 Stopper and invert to mix
 When inverting, allow the air bubble to go all the way to the top each time to ensure a complete mix
9.
 - 1) Squeeze bulb in your hand and place bulb onto flat end of pipet
 - 2) Put tip of pipet in liquid and release your grip slowly to pull solution into the pipet. Draw liquid above the mark on the neck of the pipet
 - 3) Quickly remove bulb and replace with your finger, gently release finger until liquid meniscus lines up with fill mark

- 4) Touch tip of pipet to wall and allow liquid to drain out (or blow out if you are using a serological pipet)

10. True – it should be a steady stream of individual drops

Basic Chemistry and Solutions Chemistry

1. E
2. E
3. B
4. A
5.
 - The solute is the food dye
 - Beaker B
 - Beaker A
6. 3.85%
7. 2.34%
8. 85.7 mL of 1.4 N H₂SO₄
9. 2 mL of 1N H₂SO₄
10. 83.3 mL of 6N NaOH

Temperature

1.
 - Historical purposes – record keeping
 - Temperature influences chemical reaction rates, biological growth, dissolved gas concentrations, and water stability with respect to calcium carbonate
 - Used for many process control tests
 - Ecological impact of discharge in receiving streams
2. False – larger volume of sample will result in less change in temperature
3. To get the most accurate results, because the reading will change immediately when removed from the liquid. The only exception to this is a max-registering thermometer in the autoclave
4. False
5. Digital readout or temperature/heat guns
6. Twice daily, at least 4 hours apart
7. To check for accuracy because some poorer quality thermometers are substantially inaccurate

Dissolved Oxygen

1. 15 minutes
2.
 - Useful for maintaining a stream fit for swimming, fishing and/or as a source of potable water
 - DO must be present for fish and aquatic life
 - DO level must be kept high

- DO levels must be kept above permit minimum limit
 - The flavor of water is improved by DO
 - Low DO levels can have harmful effects on receiving waters; causes suffocation of fish & promotes growth of harmful bacteria
 - Presence of DO in drinking water can contribute to corrosion of piping systems
 - Low or zero DO levels at the bottom of lakes or reservoirs often cause flavor and odor problems in drinking water
3. Barometric pressure, temperature, other substances dissolved in the water
 4. The pressure of the column of air above us
 5.
 - a. 29.92 in Hg
 - b. 28.82 in Hg
 - c. 29.33 in Hg
 6.
 - a. 599.69 mm Hg
 - b. 649.22 mm Hg
 - c. 758.19 mm Hg
 7.
 - a. 62.6 degrees F
 - b. 68 degrees F
 - c. 84.2 degrees F
 8.
 - a. 12.78 degrees C
 - b. 18.34 degrees C
 - c. 21.39 degrees C
 9. 28.87 in Hg
 10. 8.77 mg/L solubility

Sampling

1.
 - Collection
 - Volume
 - Storage and preservation
 - Sample points
 - Sampling frequency
2. A grab sample is a single sample that represents a portion of the water at the time of collection. A composite sample is smaller aliquots collected at regular intervals and then combined, the composite sample is representative of entire flow for a period of time.
3. Residual chlorine, DO, all bacterial tests, pH, temperature
4. BOD, total N, settleable solids, alkalinity
5. Growth of algae and bacteria

6. Sample Location, Date and Time of collection, Name of collector, Any other pertinent information
7. Written record to trace possession and handling of samples from collection to reporting. In case of legal litigation or used when sending out samples to contract lab
8. True

Turbidity

1. The measure of the clarity of water; also an expression of the optical property that causes light to be scattered and absorbed in water.
2.
 - Particulates, such as silt, clay, organic matter, algae, or other microorganisms
 - Suspended and colloidal matter in the water
3. False
4. True
5.
 - Supports growth of microorganisms
 - Reduces effectiveness of chlorination
 - Interferes with chemical and microbiological analysis
 - It's unacceptable for aesthetic reasons
 - It affects coagulation and filtration
 - It is unacceptable for most industrial water
6. Nephelometric Turbidity Units (NTU's)
7. To prevent changes in sample characteristics; and also because the solids will settle out.

pH

1. The intensity of the basic or acidic strength of water; and the concentration of hydrogen ions
2. 15 min
3. Daily
4.
 - Buffer solutions may deteriorate as a result of mold growth or contamination.
 - The buffers will entrain carbon dioxide from the air, which will lower the pH. This especially affects the pH 10 buffer. This is also the reason why you want to stir buffers gently (to avoid entrainment)
5. Hydrogen
6. Hydroxide
7. 10
8. 10,000 times more acidic

Standard Methods, SOP's, and QA/QC

1. Code of Federal Regulations
2. Wastewater = 40 CFR 136, Drinking Water = 40 CFR 141

3. Standard Operating Procedure = A document that thoroughly describes every step in an analytical method. It should have enough detail that someone unfamiliar with the method could perform it after reading the SOP. It is often helpful to include a copy of the manufacturer's instructions/method, as well as the locations of where samples are collected. QA/QC info should also be included.
4.
 - Review and update anything that has changed within the last year (Upgrades to facility? Changes in staff?)
 - Document a new revision
 - Document training, which should include that all analysts have read/reviewed the SOPS, all analysts sign off that they have done this refresher
5. False – pen only
6. In the event that a lot# of reagent is recalled, you can easily determine if the reagents you used were affected. This could impact the quality of the data.
7.
 - a. DOC – Demonstration of capability
 - b. MDL – Method Detection Level
 - c. LRB – Lab Reagent Blank, Method Blank, or just Blank
 - d. LFB – Lab Fortified Blank, or Standard
 - e. LFM/LFMD – Lab Fortified Matrix/Duplicate, or Spike/Spike Dup
 - f. Dup - Duplicate
 - g. ICV- Initial Calibration Verification
 - h. CCV – Continuing Calibration Verification
8.
 - Ongoing data collection (numbers are continually plugged into the MDL calculator) and MDL validation is done quarterly
 - MDL is calculated annually, or at least every 13 months
9. Duplicates check for precision – the closeness of two or more measurements to each other. Do not confuse with accuracy (how close the measurement is to the known value, the purpose of standards)

Disinfection

1. Solvent
2. Disinfection, sterilization
3. Adding chlorine to the water until the chlorine demand has been satisfied or met
4. Dose = Demand + Residual
 Residual = Dose – Demand
 Demand = Dosage – Residual

5.
 - a. pH

Chlorine disinfects faster at pH of 7 or below
Hypochlorous acid disassociates at a higher pH
 - b. Temperature

Higher temp = more efficient disinfection
Longer contact time is required at lower temps
Chlorine will dissipate faster in warmer water
 - c. Microorganisms

Number and type of microorganisms affects the effectiveness of the disinfection
Cysts (Ex: Cryptosporidium) and viruses are very resistant to disinfection
 - d. Turbidity

Excessive turbidity reduces disinfection efficiency
Organics will react with the chlorine and cause disinfection by-products
Ammonia can combine to form chloramines
 - e. Reducing agents

They create a demand that will react with and use up the chlorine
6. Hypochlorous acid and hydrochloric acid; Hypochlorous is the most effective disinfectant
7. Contact time is critical to allow sufficient time for the chlorine to react (longer the better)
Residual chlorine levels are critical to protect water in distribution system (0.2 -4.0 mg/L)
8. C
9. False – Total Chlorine requires a 3 minute reaction period, whereas Free chlorine can be read immediately (within 1 minute, after 20 seconds of swirling)
10. 2.6 mg/L
11. 2.0 mg/L
12. 80.4 lb/day

Alkalinity

1. The measurement of a water's capacity to neutralize an acid
2.
 - Hydroxide alkalinity (OH^-)
 - Carbonate alkalinity (CO_3^-)
 - Bicarbonate alkalinity (HCO_3^-)
3.
 - Chemical and biological treatment systems need alkalinity to survive an acidic discharge, also for pH stabilization

- Biological nutrient removal – Nitrification/denitrification
- Anaerobic digesters need alkalinity to prevent them from going sour – you monitor the volatile acids/alkalinity relationship
- Ammonia removal by air stripping involves using an alkaline material to bring the pH up to 11 to convert the ammonia into the gaseous form so it can be released into the environment
- Organism nutrition (Nitrosomonas and Nitrobacter use Carbonate as their food source)
- Potential to affect chlorine demand (Partial nitrification could result in too much nitrite, which has a large chlorine demand – 1 mg/L NO₂ consumes 5 mg/L chlorine)

4.

- Coagulation process – coagulants will decrease the alkalinity of water, which will lower the pH. The operator may have to add supplemental alkalinity in the form of lime (calcium carbonate), soda ash, or caustic soda to buffer the pH drop.
- Lime or soda ash softening – both of which will bring the pH up
- Buffering changes in pH in the system
- Hard water complaints – lime scale build-up

5. 8.3

6. 4.5

7. mg/L calcium carbonate (CaCO₃)

8. Buret and stand, beaker, stir plate, stir bar, graduated cylinder

9.

- Highly colored or turbid samples can mask the color change at the end point – use a pH meter to eliminate this
- Chlorine can interfere with indicators – add sodium thiosulfate to eliminate this