Introduction to Laboratory Methods for Operators

Course #2010
# 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

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### 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

### 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

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**State of Tennessee**  
Dept. of Environment & Conservation  
Fleming Training Center  
2022 Blanton Dr.  
Murfreesboro, TN 37129
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

- 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 (H₂SO₄)
  - Hydrochloric or muriatic (HCl)
  - Nitric (HNO₃)
  - Glacial acetic (H₄C₂O₂)

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

- No Food or Drink in Lab
- No Pipetting by Mouth

Laboratory Safety
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

Manufacturer Container Label

Product Identifier
Pictogram
Signal Word
Hazard Statement
Precautionary Statement

Safety Data Sheets (SDS)

- Includes all information on chemical label and specific info pertaining to that chemical
- 16 sections
**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

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**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

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**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

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**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

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**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 spatterings 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

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

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

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

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?
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.

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.

This poster is available free from OSHA.

Contact OSHA. We can help.

1-800-321-OSHA (6742) • TTY 1-877-889-5627 • www.osha.gov
Section 2

Laboratory Equipment Identification
**LABORATORY GLASSWARE AND EQUIPMENT**

*Introduction to Laboratory Methods for Operators*

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**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

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**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

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**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
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

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

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

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

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

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

○ Vacuum Pump

Gooch crucible

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)

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

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

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

MEASURING PIPETS

○ 2 types:
  1. Mohr Pipet
  2. Serological Pipet

• Mohr Pipets
  a. Graduations on these always end before the tip

• Serological Pipets
  a. Graduation marks continue to the tip

Calibrated into small divisions so that various amounts of liquid can be measured with the same pipet

Not as accurate as volumetric pipets
Examine pipets A and B
Which is the serological and which is the Mohr?

Serological

Mohr

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.

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

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.

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

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
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!

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

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
  - Note: always use gloves and goggles when handling acids

LABORATORY EQUIPMENT

PETRI DISH/DESSICATOR

- Petri Dish
  - Culturing container
- Desiccators
  - Dust and moisture free
**Evaporating Dish/Crucible**
- Evaporating Dish
- Crucible

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

**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

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

**Incubators**
- Artificially heated container used for growing bacteria cultures
- Dry-Heat types hold temperatures to ± 0.5°C
- For E. coli and Total coliform = 35 ± 0.5°C
- Water Bath for fecal = 44.5 ± 0.2°C

**Incubators**
- For BOD incubation at 20 ± 1°C
- Do not store chemical solutions and samples in same refrigerator
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

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

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

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

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

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

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

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

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

**FLAMMABLE CABINET**
- Flammable chemicals should be kept in a flammable cabinet

**SAFETY EQUIPMENT**
- PPE (Personal Protective Equipment):
  - Goggles
  - Gloves
  - Aprons
  - Wear safety clothing

**EYE WASH AND SHOWER**
- Should be checked weekly

**ANY QUESTIONS?**
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 cuvets:**
- 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 chemicals 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 (H₂SO₄) is 98. A 1 M solution of sulfuric acid would consist of 98 grams of H₂SO₄ 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 (H₂SO₄) in grams is 98.

<table>
<thead>
<tr>
<th>ELEMENT</th>
<th>ATOMIC WEIGHT</th>
<th>NUMBER OF ATOMS</th>
<th>MOLECULAR WEIGHT</th>
</tr>
</thead>
<tbody>
<tr>
<td>H</td>
<td>1</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>S</td>
<td>32</td>
<td>1</td>
<td>32</td>
</tr>
<tr>
<td>O</td>
<td>16</td>
<td>4</td>
<td>64</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Total Weight 98</td>
</tr>
</tbody>
</table>
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 ($\text{H}_2\text{SO}_4$) is 49 (98 divided by 2 because there are two replaceable hydrogen ions). A 1 N solution of sulfuric acid would consist of 49 grams of $\text{H}_2\text{SO}_4$ 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
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

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

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

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

Metric System Simplicity

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

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.

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)

Conversions

• Convert 1 meter to decimeters (dm)

  1 meter → deci → centi → milli

• 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.

Conversions

• Convert 1 meter to decimeters (dm)

  1 meter → deci → centi → milli

    • 1.0 meter = \[ \underline{10} \] decimeters
Conversions

- Convert 1 gram to milligrams (mg)

<table>
<thead>
<tr>
<th>gram</th>
<th>deci</th>
<th>centi</th>
<th>milli</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1000</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

• 1.000 gram = 1000 milligrams

Conversions

- Convert 0.28 cm to meters

<table>
<thead>
<tr>
<th>primary unit</th>
<th>deci</th>
<th>centi</th>
<th>milli</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.28 cm</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

• 0.28 cm = 0.0028 meter

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.

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.

Temperature

- To convert Celsius (°C) into Fahrenheit (°F):
  °F = (°C)(1.8) + 32

- To convert Fahrenheit (°F) into Celsius (°C):
  °C = (°F − 32) / 1.8

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?

  °F = (°C)(1.8) + 32
  °F = (21°C)(1.8) + 32
  °F = 37.8 + 32
  °F = 69.8
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?

\[
°C = (0.556)(°F - 32)
\]

\[
°C = (0.556)(68 - 32)
\]

\[
°C = (0.556)(36)
\]

\[
°C = 20.016
\]

Now we’ll work some problems...
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 ½ 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
2. 1000 mg
3. 1000 g
4. 10 mm
5. 100 mm
6. 500 mm
7. 8000 m
8. 19,000 m
9. 29,000 mL
10. 83,000 mm
11. 18 mm
12. 0.0025 g
13. 2600 m
14. 8500 m
15. 0.08 L
16. 15 cm
17. 5 km
18. 1.3 kg
19. 1.7 cm
20. 12.5 cm
21. 170,000 mL
22. 0.155 km
23. 200 sections
24. 0.0346 g
25. 19,600 nickels, $980
26. 62.5 sips
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
2. 60.8°F or 61°F
3. 29.44°C or 29°C
4. 84.2°F or 84°F
5. 21°C
6. 75°F
7. 3.17°C
CONVERSION FACTORS AND USEFUL INFORMATION

International Metric System - Le Systeme International d'Unites (SI Units)
Base Units of the International Metric System (SI)

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

Recommended Decimal Multiples and Submultiples and the Corresponding Prefixes and Names

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

Metric System 47
EXAMPLES

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 (Km\(^2\)) = 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
### AREA CONVERSION FACTORS

<table>
<thead>
<tr>
<th>Unit</th>
<th>Equivalent</th>
</tr>
</thead>
<tbody>
<tr>
<td>A square foot (ft(^2))</td>
<td>144 square inches (inch(^2))</td>
</tr>
<tr>
<td>1 square yard (yd(^2))</td>
<td>9 square feet (ft(^2))</td>
</tr>
<tr>
<td>1 acre</td>
<td>43,560 square feet (ft(^2))</td>
</tr>
<tr>
<td>1 square mile (mi(^2))</td>
<td>640 acres or 1 section</td>
</tr>
<tr>
<td>1 square meter (m(^2))</td>
<td>10.8 square feet (ft(^2))</td>
</tr>
<tr>
<td>1 square meter (m(^2))</td>
<td>10,000 square centimeters</td>
</tr>
<tr>
<td>1 hectare</td>
<td>2.5 acres</td>
</tr>
</tbody>
</table>

### VOLUME CONVERSION FACTORS

<table>
<thead>
<tr>
<th>Unit</th>
<th>Equivalent</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 cubic foot (ft(^3))</td>
<td>1,728 cubic inches (inch(^3))</td>
</tr>
<tr>
<td>1 cubic foot (ft(^3))</td>
<td>7.48 gallons</td>
</tr>
<tr>
<td>1 cubic yard (yd(^3))</td>
<td>27 cubic feet (ft(^3))</td>
</tr>
<tr>
<td>1 acre foot</td>
<td>43,560 cubic feet (ft(^3))</td>
</tr>
<tr>
<td>1 acre foot</td>
<td>325,851 gallons</td>
</tr>
<tr>
<td>1 gallon (gal.)</td>
<td>231 cubic inches (inch(^3))</td>
</tr>
<tr>
<td>1 gallon (gal.)</td>
<td>4 quarts</td>
</tr>
<tr>
<td>1 cubic meter (m(^3))</td>
<td>35.3 cubic feet (ft(^3))</td>
</tr>
<tr>
<td>1 cubic meter (m(^3))</td>
<td>1.3 cubic yards (yd(^3))</td>
</tr>
<tr>
<td>1 liter</td>
<td>1.06 quarts</td>
</tr>
<tr>
<td>1 liter</td>
<td>1,000 milliliters</td>
</tr>
</tbody>
</table>

### WEIGHT CONVERSION FACTORS

<table>
<thead>
<tr>
<th>Unit</th>
<th>Equivalent</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 gallon</td>
<td>8.34 pounds (lbs.) of water</td>
</tr>
<tr>
<td>1 cubic foot</td>
<td>62.4 pounds (lbs.) of water</td>
</tr>
<tr>
<td>1 foot of water</td>
<td>0.434 PSI (pounds per square inch)</td>
</tr>
<tr>
<td>1 pound (lb)</td>
<td>0.454 kilograms (Kgs.)</td>
</tr>
<tr>
<td>1 kilogram (Kg)</td>
<td>2.2 pounds (lbs.)</td>
</tr>
<tr>
<td>1 kilogram (Kg)</td>
<td>1,000 grams</td>
</tr>
<tr>
<td>1 PSI</td>
<td>2.31 feet of water</td>
</tr>
</tbody>
</table>
Temperature

Section 3

TDEC Fleming Training Center

1. Convert Fahrenheit to Celsius

\[ ^\circ C = \frac{5 \left( {^\circ F} - 32 \right)}{9} \]

\[ 0^\circ F = -17.8^\circ C \]

2. Convert Celsius to Fahrenheit

\[ ^\circ F = \frac{\left( ^\circ C \right) \times 9}{5} + 32 \]

\[ 0^\circ C = 32^\circ F \]

\[ 100^\circ C = 212^\circ F \]

Remember: 100\(^\circ\) between Ice/Steam = Celsius

180\(^\circ\) between Ice/Steam = Fahr.

1. Convert Fahrenheit to Celsius:

\[ ^\circ C = \left( ^\circ F + 40 \right) \times \frac{5}{9} - 40 \]

2. Convert Celsius to Fahrenheit:

\[ ^\circ F = \left( ^\circ C + 40 \right) \times \frac{9}{5} - 40 \]

Quick Approximation:

\[ (^\circ C \times 2) + 30 = ^\circ F \text{ (about)} \]
<table>
<thead>
<tr>
<th>MULTIPLY</th>
<th>BY</th>
<th>TO OBTAIN</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acres</td>
<td>43,560</td>
<td>Square feet</td>
</tr>
<tr>
<td>Acre-feet</td>
<td>43,560</td>
<td>Cubic feet</td>
</tr>
<tr>
<td>Acre-feet</td>
<td>325,851</td>
<td>Gallons</td>
</tr>
<tr>
<td>Centimeters</td>
<td>0.3937</td>
<td>Inches</td>
</tr>
<tr>
<td>Cubic feet</td>
<td>1728</td>
<td>Cubic inches</td>
</tr>
<tr>
<td>Cubic feet</td>
<td>7.48052</td>
<td>Gallons</td>
</tr>
<tr>
<td>Cubic feet</td>
<td>28.32</td>
<td>Liters</td>
</tr>
<tr>
<td>Cubic feet/second</td>
<td>448.831</td>
<td>Gal./min</td>
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<td>Minutes</td>
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</tr>
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<td>0.4335</td>
<td>lbs/square in.</td>
</tr>
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<td>Gallons</td>
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<td>Gallons</td>
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<td>Quarts (liquid)</td>
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<td>U.S. gallons</td>
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<td>Overflow rate (ft/hr)</td>
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<td>Grains/U.S. gal.</td>
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<td>Pounds</td>
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<td>33,000</td>
<td>foot-lbs./min.</td>
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<tr>
<td>Inches</td>
<td>2.540</td>
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<td>lbs/sq. inch</td>
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<td>0.07355</td>
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<tr>
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<td>1.341</td>
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<tr>
<td>MULTIPLY</td>
<td>BY</td>
<td>TO OBTAIN</td>
</tr>
<tr>
<td>------------------------------</td>
<td>---------------------</td>
<td>--------------------------------</td>
</tr>
<tr>
<td>Liters</td>
<td>0.03531</td>
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<td>1.057</td>
<td>Quarts (liquid)</td>
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<tr>
<td>Width (in) x Thickness (in)</td>
<td>Length (ft.)</td>
<td>Board feet</td>
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<td>12</td>
<td></td>
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<td>Overflow rate (ft/hr)</td>
<td>0.12468 x area (sq.ft.)</td>
<td>Gal/min</td>
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<td>Square feet</td>
<td>1/9</td>
<td>Square centimeters</td>
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<td>Square inches</td>
<td>6.452</td>
<td>Square feet</td>
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<td>10.76</td>
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<td>Square yards</td>
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<tr>
<td>Temp. EC + 17.78</td>
<td>1.8</td>
<td>Temp. EF</td>
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<tr>
<td>Temp. EF - 32</td>
<td>5/9</td>
<td>Temp. EC</td>
</tr>
<tr>
<td>Watts</td>
<td>1.34 x 10^-3</td>
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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 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**

Introduction to Laboratory Methods for Operators

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

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**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

---

**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**

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**Water vs. Mercury**

- Read Top of Meniscus
- Read Bottom of Meniscus

---

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

HOW TO USE A VOLUMETRIC FLASK

USE OF VOLUMETRIC FLASK
- Step 1
  - Begin with flask filled about 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
- 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
- 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

HOW TO PIPET
The second type of glassware used “to deliver” various volumes of liquid.
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

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

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

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

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
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

HOW TO USE A BURET

Filling the Buret:
1. Close the stopcock
2. Pour ~ 10 mL of solution into buret
3. Drain solution into waste beaker
4. Fill the buret above the zero line
5. Check for bubbles
   - Gently tap the sides to release bubbles

HOW TO USE A BURET

- Adjust the meniscus to zero
- At zero, the buret is saying "0 mL of the solution has been delivered."
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

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

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

- Carbon Atom
  - 6 protons
  - 6 neutrons

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

- 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
- Symbol: C
- Atomic Number: 6
- Electron Configuration: 2-4

Periodic Table of the Elements

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.

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.

The Periodic Table

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.

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.
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.

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.

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
**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

**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)

**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

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

**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. 
  \[ \text{N}_2 \quad \text{O}_2 \quad \text{Cl}_2 \]
- Or a molecule may consist of several elements, with dozens of atoms bonded together
  \[ \text{C}_{12}\text{H}_{22}\text{O}_{11} \]

**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
- \[ \text{H} = \text{Hydrogen} \]
- \[ \text{O} = \text{Oxygen} \]
- \[ \text{H}_2\text{O} \quad 1 \text{ atom Oxygen} \]
- \[ \text{H}_2\text{O} \quad 2 \text{ atoms Hydrogen} \]
Chemical equations

- A shorthand way, using chemical formulas, to accurately represent what happens in a chemical reaction.
- The balanced chemical equation for water:
  \[ 2\text{H}_2\text{(g)} + \text{O}_2\text{(g)} \rightleftharpoons 2\text{H}_2\text{O}\text{(l)} \]
- The coefficients indicate the relative number of molecules (moles) that are involved in the reaction.

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 \(6.02 \times 10^{23}\) atoms or molecules.

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 \(6.02 \times 10^{23}\) molecules (because the sugar molecule is bigger).

How much does 1 mole of water weigh?

- **H₂O**

<table>
<thead>
<tr>
<th>Number of Atoms</th>
<th>Atomic Weight</th>
<th>Total Weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen (H)</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>Oxygen (O)</td>
<td>1,6</td>
<td>16</td>
</tr>
<tr>
<td>Molecular Weight of H₂O</td>
<td>18 grams/mol</td>
<td></td>
</tr>
</tbody>
</table>

- \(1\text{ mol} = 18\text{ grams}\)

Molarity

- Perhaps the most accurate way of expressing the concentration of a solution:

\[ \text{Molarity} = \frac{\text{moles of solute}}{\text{liter of solution}} \]

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
- \(\text{Normality} = \frac{\text{Equivalent weight}}{\text{liters of solution}}\)
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 = number of equivalent weights of solute / liters of solution
- Number of equivalent weights = total weight of solute / equivalent weight
- Equivalent weight = molecular weight / number of positive charges

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.

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.

\[ C_1V_1 = C_2V_2 \]

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

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

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

\[ V_1 = 16.7 \text{ gals} \]

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

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

\[ (5N)(100 \text{ mL}) = (V_2) \]

\[ 500 \text{ mL} = V_2 \]

500 mL is final volume, you started with 100 mLs and added 400 mL to get a final volume of 500 mL.
# Periodic Table of the Elements

<table>
<thead>
<tr>
<th>Column 1</th>
<th>Column 2</th>
<th>Column 3</th>
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<tr>
<td>H</td>
<td>He</td>
<td>Li</td>
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<td>B</td>
<td>C</td>
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<td>Tc</td>
<td>Ru</td>
<td>Rh</td>
<td>Pd</td>
<td>Ag</td>
<td>Cd</td>
<td>In</td>
<td>Sn</td>
<td>Sb</td>
<td>Te</td>
<td>I</td>
<td>Xe</td>
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<td>Cs</td>
<td>Ba</td>
<td>La</td>
<td>Cm</td>
<td>Ac</td>
<td>Th</td>
<td>Pa</td>
<td>U</td>
<td>Np</td>
<td>Pu</td>
<td>Am</td>
<td>Cm</td>
<td>Bk</td>
<td>Cf</td>
<td>Es</td>
<td>Fm</td>
<td>Md</td>
<td>No</td>
</tr>
</tbody>
</table>

### Introduction to Basic Chemistry

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sciencenotes.org
**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

   \[ 0.001 \text{ M} = 0.001 \text{ mol/L} \]

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

   **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.

\[ N = \frac{\text{Eq.Wt.}}{\text{L}} \]

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

\[ N = \frac{\text{Eq.Wt.}}{\text{L}} \]

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

\[ N = 1.2 \text{ N NaOH} \]

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

**Step 1**: Determine the MW of NaOH.

Molecular Weight = 40 g/mol

**Step 2**: Determine equivalent weight.

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

**Step 3**: Set up Ratio.

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

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

\[ X = 48 \text{ g} \]
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.

**Equivalent Weight** = \( \frac{\text{Molecular wt.}}{\text{valence}} \)

**Example:**

\[
\begin{align*}
\text{Eq.Wt. of Calcium} &= \frac{\text{MW of Calcium}}{\text{Valence of Calcium}} = \frac{40 \text{ g}}{2} = 20 \text{ g Eq.Wt.} \\
\text{Eq.Wt. of Sodium} &= \frac{\text{MW of Sodium}}{\text{Valence of Sodium}} = \frac{23 \text{ g}}{1} = 23 \text{ g Eq.Wt.} \\
\text{Eq.Wt. of Calcium Carbonate} &= \frac{\text{MW of CaCO}_3}{\text{Valence of Calcium}} = \frac{100 \text{ g}}{2} = 50 \text{ g Eq.Wt.} \\
\text{Eq.Wt. of Ferric Hydroxide} &= \frac{\text{MW of Fe(OH)}_3}{\text{Valence of Fe}^{3+}\text{(OH)}_3} = \frac{107 \text{ g}}{3} = 35.6 \text{ g Eq.Wt.} \\
\text{Eq.Wt. of Magnesium} &= \frac{\text{MW of Mg}}{\text{Valence of Mg}} = \frac{24 \text{ g}}{2} = 12 \text{ g Eq.Wt.}
\end{align*}
\]

Source: Basic Water Chemistry for Water and Wastewater Operators by Richard Blodgett
Equivalent Weight, Normality to determine Hardness

The total hardness in water is usually expressed in terms of CaCO₃. For example a lab report might read:

| Total Hardness 180 mg/L as CaCO₃ |

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 Ca(O3).

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 Mg = 17 mg/L. How do you express Magnesium as CaCO₃?

Step 1: Determine MW

\[
\begin{align*}
\text{MW Mg} &= 24 \text{g/mol} \\
\text{MW CaCO}_3 &= 100 \text{g/mol}
\end{align*}
\]

Step 2: Determine Eq. Wt.

\[
\begin{align*}
\text{Eq. Wt. Mg} &= \frac{\text{MW}}{\text{Valence}} = \frac{24}{2} = 12 \text{g/Eq.wt.} \\
\text{Eq. Wt. CaCO}_3 &= \frac{\text{MW}}{\text{Valence}} = \frac{100}{2} = 50 \text{g/Eq.wt.}
\end{align*}
\]

Step 3: Set up Ratio

\[
\begin{align*}
\frac{\text{Eq. Wt. Mg}}{\text{Eq. Wt. CaCO}_3} &= \frac{[\text{Mg}]}{[X]} = \frac{12 \text{g/Eq.wt. Mg}}{50 \text{ g/Eq.wt. CaCO}} \\
X &= \frac{(17 \text{ mg/L Mg}) (50 \text{ g/Eq.wt. CaCO}_3)}{12 \text{ g/Eq.wt. Mg}} \\
X &= 71 \text{ mg/L as CaCO}_3
\end{align*}
\]
Dilution of Concentrated Acids and Bases to Prepare a 1N Solution

<table>
<thead>
<tr>
<th>Compound</th>
<th>Formula</th>
<th>Molecular Weight</th>
<th>Approximate Spec. Grav. Of Concentrated Reagent</th>
<th>Approximate % Present in Concentrated Reagent</th>
<th>Normality of Concentrated Reagent</th>
<th>Approximate mL of Concentrated Reagent to dilute to 1 L</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acetic Acid</td>
<td>CH₃ • COOH</td>
<td>60.054</td>
<td>1.05</td>
<td>99.6</td>
<td>17.4</td>
<td>58</td>
</tr>
<tr>
<td>Ammonium Hydroxide</td>
<td>NH₄OH</td>
<td>35.048</td>
<td>0.90</td>
<td>57.6</td>
<td>14.8</td>
<td>68</td>
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<tr>
<td>Hydrochloric Acid</td>
<td>HCl</td>
<td>36.465</td>
<td>1.19</td>
<td>37.0</td>
<td>12.1</td>
<td>83</td>
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<tr>
<td>Lactic Acid</td>
<td>CH₃ • CHOH • COOH</td>
<td>90.081</td>
<td>1.21</td>
<td>85.0</td>
<td>11.4</td>
<td>88</td>
</tr>
<tr>
<td>Nitric Acid</td>
<td>HNO₃</td>
<td>63.016</td>
<td>1.42</td>
<td>69.5</td>
<td>15.7</td>
<td>64</td>
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<tr>
<td>Perchloric Acid</td>
<td>HClO₄</td>
<td>100.465</td>
<td>1.67</td>
<td>70.0</td>
<td>11.6</td>
<td>87</td>
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<tr>
<td>Phosphoric Acid (ortho-)</td>
<td>H₃PO₄</td>
<td>97.999</td>
<td>1.69</td>
<td>85.0</td>
<td>44.0</td>
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<tr>
<td>Potassium Hydroxide</td>
<td>KOH</td>
<td>56.108</td>
<td>1.51</td>
<td>50.0</td>
<td>13.5</td>
<td>75</td>
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<tr>
<td>Sodium Hydroxide</td>
<td>NaOH</td>
<td>39.999</td>
<td>1.53</td>
<td>50.0</td>
<td>19.1</td>
<td>53</td>
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<tr>
<td>Sulfuric Acid</td>
<td>H₂SO₄</td>
<td>98.082</td>
<td>1.84</td>
<td>96.0</td>
<td>36.0</td>
<td>28</td>
</tr>
</tbody>
</table>
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?
6. If 20 lbs of chemical is added to 500 lb of water, what is the percent strength by weight?

7. If 60 lbs of chemical is added to 300 gal of water, what is the percent strength of the solution by weight?

8. You have a 1.4 N H₂SO₄ standard solution. How many milliliters of the standard solution must be added to water to make 100 mL of a 1.2 N H₂SO₄ solution?

9. You need to make a 100 mL solution of a 0.02 N H₂SO₄ for an alkalinity titrant. You only have a 1 N H₂SO₄ standard solution. How could you make the 0.02 N solution?

10. You need to make 500 mL of 1 N NaOH, but you only have 6 N NaOH in the lab. How many mL of 6 N NaOH will it take?
Section 6

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

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 of the liquid in the column
   - Appropriate for any application where total immersion is impractical or impossible, such as in a shallow water bath

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

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
  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

## Any Questions?

- 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
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

What is Dissolved Oxygen (DO)?
- Measure of the amount of gaseous oxygen contained in water
- Necessity in water to support life
- Max amount of DO is the saturation concentration

How does Dissolved Oxygen enter water?
- Direct absorption from atmosphere
- Rapid movement from winds, waves, currents or mechanical aeration
- Photosynthesis

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

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
- Temperature
  - Lower temperature can contain more dissolved oxygen because they have less movement
  - Increase in movement allows them to escape out of the water

Factors Influencing DO Saturation Concentration
- Depth of Water
  - Shallower water, higher the DO concentration
  - Waves and movement will increase DO
- Salinity
  - Salts affect the solubility of gases, essentially driving them out of water
- Bioactivity
  - Microorganisms feeding on organics and decaying matter use up oxygen
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

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

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

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 − (Altitude x 0.026)] ÷ 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)

Example
1. Fleming Training Center altitude = 543 feet
2. Plug that altitude into the Correction Factor equation:
   CF = [760 − (543 x 0.026)] ÷ 760
      = [760 − (543 x 0.026)] ÷ 760
      = [760 − 14.1] ÷ 760
      = 745.9 ÷ 760 = 0.9795 (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
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 $O_2$ solubility at that temperature
  
  [Link to DO Saturation Table](http://water.usgs.gov/software/DOTABLES/)

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):
  
  \[ \text{inches of Hg} \times 25.4 = \text{mm of Hg} \]

- To convert mm of mercury (Hg) to inches of mercury (Hg):
  
  \[ \text{mm of Hg} \div 25.4 = \text{inches of Hg} \]

Conversion - Temperature

- To convert from Celsius to Fahrenheit:

  \[ ^\circ F = [(^\circ C)(1.8) + 32] \]

- To convert from Fahrenheit to Celsius:

  \[ ^\circ C = \left( \frac{^\circ F - 32}{1.8} \right) \]

Barometer on site

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

[Images of barometer, thermometer, and USGS DO table]

Dissolved Oxygen Measurement

[Image of dissolved oxygen measurement equipment]
**Approved Methods 40 CFR 136**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Methodology</th>
<th>Standard method</th>
<th>ASTM</th>
<th>USES/IDAC/Other</th>
</tr>
</thead>
<tbody>
<tr>
<td>Oxygen, dissolved</td>
<td>Winkler method</td>
<td>4500-0-0.06</td>
<td>D588-12</td>
<td></td>
</tr>
<tr>
<td>(mg/L)</td>
<td>(0.06)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Classic wet chemistry procedure</td>
<td>4500-0-0.16</td>
<td>D588-12</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Titration method</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>A series of reactions produces</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>iodine</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>In proportion to the amount of</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>DO</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Sodium thiosulfate with starch</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>as the indicator</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Complicated method</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Calibration check or meter check</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**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

**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

**Sampling 40 CFR 136**

- **Parameter number**: 48
- **Container**: 600 mL wide-mouth glass
- **Preservatives**: None required
- **Maximum holding time**: Analyze within 15 minutes.
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.

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.

Any Questions?

94  Dissolved Oxygen
Solubility of oxygen in fresh water at various temperatures and pressures
°C, degrees Celsius; in Hg, inches of mercury]

<table>
<thead>
<tr>
<th>Temp. (°C)</th>
<th>Barometric Pressure (in Hg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>18</td>
<td>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</td>
</tr>
<tr>
<td>19</td>
<td>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</td>
</tr>
<tr>
<td>20</td>
<td>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</td>
</tr>
<tr>
<td>Temp. (°C)</td>
<td>22.44</td>
</tr>
<tr>
<td>-----------</td>
<td>-------</td>
</tr>
<tr>
<td>20</td>
<td>6.77</td>
</tr>
<tr>
<td>21</td>
<td>6.63</td>
</tr>
<tr>
<td>23</td>
<td>6.37</td>
</tr>
<tr>
<td>24</td>
<td>6.25</td>
</tr>
<tr>
<td>26</td>
<td>6.02</td>
</tr>
<tr>
<td>27</td>
<td>5.90</td>
</tr>
<tr>
<td>28</td>
<td>5.80</td>
</tr>
<tr>
<td>29</td>
<td>5.69</td>
</tr>
<tr>
<td>30</td>
<td>5.59</td>
</tr>
</tbody>
</table>

Return to the DOTABLES main page.
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.)

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

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.
Sodium Azide Modification of the Winkler Method

OUTLINE OF PROCEDURE FOR DO

1. Take 300 mL sample.
2. Add 1 mL MnSO₄ below surface.
3. Add 1 mL KI + NaOH below surface.
4. Mix by inverting.
5. Add 1 mL H₂SO₄

Brown floc; DO present.
Reddish-brown iodine solution.
White floc; no DO.

Titration of Iodine Solution:

1. Pour 201 mL into flask.
2. Titrate with PAO or Sodium Thiosulfate.
3. Add Starch indicator.

Reddish-Brown
Pale Yellow
Blue
Clear

End Point

Source: Operation of Wastewater Treatment Plants, Vol II, p. 571 Dissolved Oxygen
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  3.2  Use of hazard information ........................................................ 4
  3.3  Precautionary labels ................................................................. 5
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<th>Page</th>
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<tr>
<td>7</td>
<td>103</td>
</tr>
</tbody>
</table>

Dissolved Oxygen
Section 1 Product overview

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

Section 2 Specifications

Specifications are subject to change without notice.

<table>
<thead>
<tr>
<th>Specifications</th>
<th>Details</th>
</tr>
</thead>
<tbody>
<tr>
<td>Probe type</td>
<td>Luminescent dissolved oxygen (LDO) sensor with integrated stirring system</td>
</tr>
<tr>
<td>Dissolved oxygen range</td>
<td>0.05 to 20.0 mg/L (ppm); 1 to 200% saturation</td>
</tr>
<tr>
<td>Dissolved oxygen accuracy</td>
<td>±0.05 mg/L for concentrations less than 10 mg/L O₂</td>
</tr>
<tr>
<td></td>
<td>±0.1 mg/L for concentrations more than 10 mg/L O₂</td>
</tr>
</tbody>
</table>

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

#### ▲ D A N G E R

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

#### ▲ W A R N I N G

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

#### ▲ C A U T I O N

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

#### N O T I C E

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.

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.
Section 5 Calibration

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 ¾ 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.
Section 6: Sample measurement

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

If samples are expected to have less than 1 mg/L O\textsubscript{2}, calibrate the probe with a zero-oxygen solution. A zero-point calibration is not recommended for samples that have more than 1 mg/L O\textsubscript{2}. Use the guidelines and steps that follow only when the sample is expected to have less than 1 mg/L O\textsubscript{2}.

• 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

\begin{table}[h]
\centering
\begin{tabular}{|l|}
\hline
\textbf{NOTICE} \\
\hline
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. \\
\hline
\end{tabular}
\end{table}

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

\begin{table}[h]
\centering
\begin{tabular}{|l|}
\hline
\textbf{NOTICE} \\
\hline
Do not use sharp metal tools to remove the LBOD sensor cap. \\
\hline
\end{tabular}
\end{table}

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.
\textbf{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.
\textbf{Note:} Do not touch the protective black layer on the LBOD sensor cap.
4. Install the new iButton\textsuperscript{®} 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.
Figure 2  Replace the LBOD sensor cap

Dissolved Oxygen

TDEC Fleming Training Center
Figure 3  Replace the iButton

Section 7

Dissolved Oxygen
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 ¼ minimum) when not in use.
<table>
<thead>
<tr>
<th>Problem</th>
<th>Possible cause</th>
<th>Solution</th>
</tr>
</thead>
<tbody>
<tr>
<td>Decreased probe performance causes slow stabilization and prevents</td>
<td>The sensor cap is loose or damaged.</td>
<td>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.</td>
</tr>
<tr>
<td>accurate calibrations or measurements.</td>
<td>Water is between the sensor cap and the probe lens.</td>
<td>Remove the sensor cap and dry the probe lens. Refer to Replace the sensor cap and iButton on page 9.</td>
</tr>
<tr>
<td></td>
<td>The sensor cap is not sufficiently conditioned.</td>
<td>Keep the probe in the BOD bottle with some water for more time, then try the calibration again.</td>
</tr>
<tr>
<td></td>
<td>The temperature or pressure sensor does not operate correctly.</td>
<td>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.</td>
</tr>
<tr>
<td></td>
<td>The lot code on the iButton is not the same as the lot code on the sensor cap.</td>
<td>Replace the sensor cap and iButton.</td>
</tr>
<tr>
<td>The stirrer does not operate correctly.</td>
<td>The stirrer does not operate.</td>
<td>The stirrer uses the power from the meter to operate. Make sure to start the meter, then start the stirrer.</td>
</tr>
<tr>
<td></td>
<td>The stirrer turns only when the probe is not in a BOD bottle.</td>
<td>Remove and install the stirrer assembly. Refer to Replace the stirrer on page 12.</td>
</tr>
<tr>
<td></td>
<td>The stirrer makes a lot of noise.</td>
<td>Remove and install the stirrer assembly. Refer to Replace the stirrer on page 12.</td>
</tr>
<tr>
<td>Sample properties cause slow stabilization or inaccurate measurements.</td>
<td>The measurement was not adjusted for salinity in the sample.</td>
<td>Measure the salinity of the sample and enter the value as a salinity correction factor in the meter.</td>
</tr>
<tr>
<td>Procedure problem causes slow stabilization and prevents accurate</td>
<td>Air bubbles are around or below the probe tip.</td>
<td>Carefully tap or shake the probe to remove air bubbles.</td>
</tr>
<tr>
<td>calibrations or measurements.</td>
<td>The contents of the BOD bottle did not fully adjust to ambient temperature.</td>
<td>Wait more for the contents of the BOD bottle to adjust to ambient temperature.</td>
</tr>
</tbody>
</table>
### Section 9 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.

<table>
<thead>
<tr>
<th>Description</th>
<th>Quantity</th>
<th>Item no.</th>
</tr>
</thead>
<tbody>
<tr>
<td>LBOD sensor cap replacement kit (includes iButton)</td>
<td>1</td>
<td>5838000</td>
</tr>
<tr>
<td>LBOD probe replacement stirrer assembly</td>
<td>1</td>
<td>5850800</td>
</tr>
<tr>
<td>LBOD stirrer bearing clip</td>
<td>1</td>
<td>5852800</td>
</tr>
<tr>
<td>BOD bottle with glass stopper, 300 mL</td>
<td>1</td>
<td>62100</td>
</tr>
<tr>
<td>Beaker, 250 mL, polypropylene</td>
<td>1</td>
<td>108046</td>
</tr>
<tr>
<td>Cobalt chloride solution</td>
<td>500 mL</td>
<td>1422249</td>
</tr>
<tr>
<td>Sodium sulfite</td>
<td>100 g</td>
<td>2386026</td>
</tr>
<tr>
<td>Disposable wipes, 11 x 22 cm</td>
<td>280/pkg</td>
<td>2097000</td>
</tr>
<tr>
<td>Wash bottle, polyethylene, 500 mL</td>
<td>1</td>
<td>62011</td>
</tr>
</tbody>
</table>
Hach Method 10360
Luminescence Measurement of Dissolved Oxygen in Water and Wastewater and for Use in the Determination of BOD$_5$ and cBOD$_5$
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 127053)
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 (± 2°C).

7.2.3 With a steady stream of filtered air (≈ 10 – 40 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 (± 2°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:

\[
s = \sqrt{\frac{\sum x^2 - (\sum x)^2}{n}}
\]

where:

\[
n = \text{Number of samples}
\]

\[
x = \text{Concentration in each sample}
\]

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

\[
\begin{align*}
3.0 \text{ mL} & \times 0.300 \text{ mg/mL GGA} \times 1000 \text{ mL/L} = 3.0 \text{ mg/L GGA per bottle} \\
300 \text{ mL final volume} \\
\text{ezGGA Ampoule} & \\
1 \text{ ampoule (2.0 mL)} & \times 0.450 \text{ mg/mL GGA} \times 1000 \text{ mL/L} = 3.0 \text{ mg/L GGA per bottle} \\
300 \text{ mL final volume} \\
\end{align*}
\]

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 gentle 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 ¾ 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:

\[
\text{mL 0.025 N Sodium Thiosulfate required} = \text{mL titrant used} \times \frac{\text{volume of remaining sample divided by 100}}{}
\]

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 potions 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 1.0 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:

- \( \text{BOD}_5 \text{ or cBOD}_5 = \) BOD value from the 5-day test
- \( D_1 = \) DO of diluted sample immediately after preparation, in mg/L
- \( D_2 = \) 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:
- \( f = (\text{volume of seed in diluted sample/volume of seed in seed control}) \)
13.2 When Dilution Water Requires Seed

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

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

<table>
<thead>
<tr>
<th>Acceptance Criterion</th>
<th>Section</th>
<th>Limit</th>
</tr>
</thead>
<tbody>
<tr>
<td>Initial DO Accuracy in Reagent Water</td>
<td>9.2.1</td>
<td>95% to 105%</td>
</tr>
<tr>
<td>Initial Precision in Reagent Water</td>
<td>9.2.1</td>
<td>2.1%</td>
</tr>
<tr>
<td>Average GGA Recovery</td>
<td>15.7</td>
<td>198±30.5 mg/L BOD</td>
</tr>
</tbody>
</table>

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.


15.0 References


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

15.5 “OSHA Safety and Health Standards, General Industry,” (29 CFR 1910), Occupational Safety and Health Administration, OSHA 2206 (Revised, January 1976)


16.0 Tables

16.1 Nutrient Buffer Preparation Options

**Table 1 - BOD Nutrient Buffer Pillows**

<table>
<thead>
<tr>
<th>Volume of Dilution Water to Prepare</th>
<th>Hach BOD Nutrient Pillow Catalog Number</th>
</tr>
</thead>
<tbody>
<tr>
<td>300 mL add pillow to each BOD Bottle</td>
<td>1416066</td>
</tr>
<tr>
<td>3 liters</td>
<td>1486166</td>
</tr>
<tr>
<td>4 liters</td>
<td>2436466</td>
</tr>
<tr>
<td>6 liters</td>
<td>1486266</td>
</tr>
<tr>
<td>19 liters</td>
<td>1486398</td>
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**Table 2 – Conventional Dilution Water Preparation Reagents**

<table>
<thead>
<tr>
<th>Hach Buffer Reagents</th>
<th>Hach Buffer Reagent Catalog Number</th>
</tr>
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<tbody>
<tr>
<td>Buffer Solution, APHA, for BOD, pH 7.2, phosphate type, 500 mL</td>
<td>43149</td>
</tr>
<tr>
<td>Calcium Solution, Alpha, for BOD, 500 mL</td>
<td>42849</td>
</tr>
<tr>
<td>Ferric Chloride Solution, APHA, 1000 mL</td>
<td>42953</td>
</tr>
<tr>
<td>Magnesium Sulfate Solution, APHA, for BOD, 500 mL</td>
<td>43049</td>
</tr>
</tbody>
</table>

*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**

<table>
<thead>
<tr>
<th>Sample Type</th>
<th>Estimated BOD mg/L</th>
<th>Minimum Sample Volume (mL)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Strong Waste</td>
<td>600</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td>300</td>
<td>2</td>
</tr>
<tr>
<td></td>
<td>200</td>
<td>3</td>
</tr>
<tr>
<td></td>
<td>150</td>
<td>4</td>
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<tr>
<td>Raw and Settled Sewage</td>
<td>100</td>
<td>6</td>
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<tr>
<td></td>
<td>75</td>
<td>8</td>
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<td></td>
<td>60</td>
<td>10</td>
</tr>
<tr>
<td>Oxidized Effluents</td>
<td>50</td>
<td>12</td>
</tr>
<tr>
<td></td>
<td>40</td>
<td>15</td>
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<td></td>
<td>10</td>
<td>60</td>
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<tr>
<td>Polluted River Water</td>
<td>6</td>
<td>100</td>
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<td></td>
<td>4</td>
<td>200</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>300</td>
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</table>
Table 4 – Maximum Sample Volume Selection Guide

<table>
<thead>
<tr>
<th>BOD at Sea Level (mg/L)</th>
<th>BOD at 1000 ft Elevation (mg/L)</th>
<th>BOD at 5000 feet Elevation (mg/L)</th>
<th>Maximum Sample Volume (mL)</th>
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</thead>
<tbody>
<tr>
<td>615</td>
<td>595</td>
<td>508</td>
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<td>492</td>
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<td>200</td>
</tr>
<tr>
<td>8</td>
<td>8</td>
<td>7</td>
<td>300</td>
</tr>
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</table>

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

16.3 Performance Criteria

Table 5 - Initial Precision and Recovery Method Performance

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

Table 6 - Calibration Verification Performance

<table>
<thead>
<tr>
<th>CV DO Concentration</th>
<th>Average % Recovery</th>
<th>% Standard Deviation</th>
<th>% Relative Standard Deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>7.22 mg/L – 9.23 mg/L</td>
<td>100.1</td>
<td>2.5</td>
<td>2.5</td>
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</tbody>
</table>

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
Why Sample?

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

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?

Considerations

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

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

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
Composite Sample

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

Sampling Guidelines

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

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

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

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.

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.
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.

Sample Volume

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

Sampling Point Selection

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

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

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

Sampling Devices

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

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

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

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

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

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
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

Any Questions?
**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

---

**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

---

**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

---

**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)

---

**Instruments**

- Hach 2100N Laboratory Benchtop Turbidimeter
- Hach 2100Q Portable Turbidimeter

---

**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
**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

**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

**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

**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
    - Procedure for making solutions
- Use Gelex Secondary Turbidity Standards for periodic checks

TU5 SERIES
- Calibration
  - StabiCal
    - Formazin Solution
- Verification
  - Glass verification rod (secondary turbidity standard)
  - StabiCal
  - Formazin

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 = 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)

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

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

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

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

\*How Does a pH Probe Work?

\*Dispenses reference solution which completes circuit for meter

\*Reference Half-Cell

\*Sensing Half-Cell

\*pH 7 Solution
H\(^+\) conc. the same both inside and outside glass bulb
\*No potential develops

Hydrogen ion concentration fixed at pH 7
**Sensing Half-Cell**

**pH 7 Solution**
H⁺ conc. the same both inside and outside glass bulb
*No potential develops

**pH 4 Solution**
H⁺ conc. 1000x greater outside glass bulb
*Potential develops

**pH 4 Solution**
H⁺ conc. 1000x greater outside glass bulb
*Potential develops

**pH 10 Solution**
H⁺ conc. 1000x greater inside glass bulb
*Potential develops

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

*Decade = one pH unit
*Holding time = 15 minutes
*Preservation = none
*Sample container = glass or plastic
*Grab sample
*Continuous monitoring possible

*pH Sampling

*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

*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?
### pH Calibration Record

<table>
<thead>
<tr>
<th>Date</th>
<th>Time</th>
<th>Temp of Buffers</th>
<th>Slope</th>
<th>Buffers Used</th>
<th>ICV</th>
<th>CCV</th>
<th>Analyst Initials</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>4 7 10</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Lot#s</th>
</tr>
</thead>
<tbody>
<tr>
<td>4</td>
</tr>
<tr>
<td>7</td>
</tr>
<tr>
<td>10</td>
</tr>
</tbody>
</table>

### Sample Name

<table>
<thead>
<tr>
<th>Date</th>
<th>Time</th>
<th>Sample Name</th>
<th>Temp. Solution</th>
<th>Date of Last Calibration</th>
<th>Measured pH</th>
<th>Analyst Initials</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Vinegar</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>Coca Cola</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>Lemon Juice</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>Ammonia Cleaner</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>Alkaline Water</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
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Section 10

TDEC Fleming Training Center

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pH
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

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Shroud (rugged model)</td>
</tr>
<tr>
<td>2</td>
<td>Probe storage cap</td>
</tr>
<tr>
<td>3</td>
<td>Glass bulb and temperature sensor</td>
</tr>
<tr>
<td>4</td>
<td>Reference junction</td>
</tr>
<tr>
<td>5</td>
<td>Protective tape</td>
</tr>
<tr>
<td>6</td>
<td>Locking ring (rugged model)</td>
</tr>
<tr>
<td>7</td>
<td>Rugged probe</td>
</tr>
<tr>
<td>8</td>
<td>Probe soaker bottle holder or storage cap</td>
</tr>
<tr>
<td>9</td>
<td>Standard probe</td>
</tr>
<tr>
<td>10</td>
<td>Probe soaker bottle with storage solution</td>
</tr>
</tbody>
</table>

### Section 2  Specifications

Specifications are subject to change without notice.

<table>
<thead>
<tr>
<th>Specifications</th>
<th>Details</th>
</tr>
</thead>
<tbody>
<tr>
<td>Probe type</td>
<td>Digital combination pH probe with non-refillable gel electrolyte and built-in temperature sensor</td>
</tr>
<tr>
<td>pH range</td>
<td>2 to 14 pH</td>
</tr>
<tr>
<td>pH accuracy</td>
<td>±0.02 pH</td>
</tr>
<tr>
<td>Reference type</td>
<td>Ag/AgCl</td>
</tr>
<tr>
<td>Specifications</td>
<td>Details</td>
</tr>
<tr>
<td>----------------</td>
<td>---------</td>
</tr>
<tr>
<td>Reference junction</td>
<td>Open</td>
</tr>
<tr>
<td>Slope</td>
<td>–59 mV/pH (90 to 110% at 25 °C (77 °F) per Nernstian theoretical value)</td>
</tr>
<tr>
<td>Isopotential point</td>
<td>0 (±30) mV at 7.0 (±0.5) pH</td>
</tr>
<tr>
<td>Sodium (alkalinity) error</td>
<td>–0.6 pH at pH 12.6 in 1 M NaOH</td>
</tr>
<tr>
<td>Temperature accuracy</td>
<td>±0.3 °C (±0.54 °F)</td>
</tr>
<tr>
<td>Temperature sensor type</td>
<td>30 kΩ NTC thermistor</td>
</tr>
<tr>
<td>Operating temperature</td>
<td>0 to 50 °C (32 to 122 °F)</td>
</tr>
<tr>
<td>Storage temperature</td>
<td>5 to 40 °C (41 to 104 °F)</td>
</tr>
<tr>
<td>Minimum immersion depth</td>
<td>20 mm (0.79 in.)</td>
</tr>
<tr>
<td>Body material (standard)</td>
<td>Epoxy</td>
</tr>
<tr>
<td>Body material (field rugged)</td>
<td>Epoxy/stainless steel</td>
</tr>
<tr>
<td>Electrolyte</td>
<td>Non-refillable gel reference element</td>
</tr>
<tr>
<td>Storage solution</td>
<td>Hach pH electrode storage solution&lt;sup&gt;1&lt;/sup&gt;</td>
</tr>
<tr>
<td>Cable connection</td>
<td>M12 digital output and connector</td>
</tr>
<tr>
<td>Dimensions</td>
<td>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)</td>
</tr>
<tr>
<td>Dimensions (rugged)</td>
<td>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)</td>
</tr>
<tr>
<td>Weight (includes cable)</td>
<td>PHCxxx01: ~0.4 kg (0.9 lb); PHCxxx03: ~0.45 kg (1 lb)</td>
</tr>
<tr>
<td>Weight (rugged, includes cable)</td>
<td>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)</td>
</tr>
<tr>
<td>Warranty</td>
<td>6 months on the probe. This warranty covers manufacturing defects, but not improper use or wear.</td>
</tr>
<tr>
<td>Certifications</td>
<td>CE, FCC/ISED</td>
</tr>
</tbody>
</table>

### 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.

---

<sup>1</sup> Use of other storage solutions can cause permanent damage to the probe.
3.2 Use of hazard information

 نفس التأسيس للمادة

| **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

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.
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.
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.

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

Figure 6 Probe storage
<table>
<thead>
<tr>
<th>Problem</th>
<th>Possible cause</th>
<th>Solution</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample properties cause slow stabilization or inaccurate measurements.</td>
<td>The sample absorbs carbon dioxide (CO₂) from the air, which causes the pH value to slowly decrease in low ionic strength (LIS) or high purity samples.</td>
<td>Use the LIS chamber for LIS/high purity samples to prevent CO₂ absorption.</td>
</tr>
<tr>
<td>Procedure problem causes slow stabilization and prevents accurate calibrations or measurements.</td>
<td>The probe is not conditioned to the sample.</td>
<td>Soak the probe in the sample before sample measurements. Refer to Preparation for use on page 5.</td>
</tr>
<tr>
<td></td>
<td>Air bubbles are around or below the probe tip.</td>
<td>Carefully tap or shake the probe to remove air bubbles.</td>
</tr>
<tr>
<td></td>
<td>The electrical connection through the reference junction is not sufficient.</td>
<td>Shake the probe in the solution from side to side to refresh the reference junction.</td>
</tr>
<tr>
<td></td>
<td>The stir speed is too slow or too fast.</td>
<td>Try a different stir speed.</td>
</tr>
<tr>
<td></td>
<td>An incorrect buffer solution was used or the buffer solution has contamination.</td>
<td>Use the specified buffer solutions of good quality.</td>
</tr>
</tbody>
</table>

### 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.*

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

### 10.1 Recommended standards

<table>
<thead>
<tr>
<th>Description</th>
<th>Unit</th>
<th>Item no.</th>
</tr>
</thead>
<tbody>
<tr>
<td>pH 4.01 buffer solution, Singlet one-use packets, 20 mL each</td>
<td>20/pkg</td>
<td>2770020</td>
</tr>
<tr>
<td>pH 7.00 buffer solution, Singlet one-use packets, 20 mL each</td>
<td>20/pkg</td>
<td>2770120</td>
</tr>
</tbody>
</table>
## 10.1 Recommended standards (continued)

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

## 10.2 Accessories

<table>
<thead>
<tr>
<th>Description</th>
<th>Quantity</th>
<th>Item no.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Beaker, 30 mL, plastic, colorless</td>
<td>80/pkg</td>
<td>SM5010</td>
</tr>
<tr>
<td>Beaker, 30 mL, plastic, red</td>
<td>80/pkg</td>
<td>SM5011</td>
</tr>
<tr>
<td>Beaker, 30 mL, plastic, yellow</td>
<td>80/pkg</td>
<td>SM5012</td>
</tr>
<tr>
<td>Beaker, 30 mL, plastic, blue</td>
<td>80/pkg</td>
<td>SM5013</td>
</tr>
<tr>
<td>Beaker, 30 mL, plastic, green</td>
<td>80/pkg</td>
<td>SM5014</td>
</tr>
<tr>
<td>Beaker dispenser and holder, 30 mL</td>
<td>1</td>
<td>923-656</td>
</tr>
<tr>
<td>Beaker holder, 30 mL</td>
<td>1</td>
<td>923-556</td>
</tr>
</tbody>
</table>
### 10.2 Accessories (continued)

<table>
<thead>
<tr>
<th>Description</th>
<th>Quantity</th>
<th>Item no.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Beaker, 100 mL, polypropylene</td>
<td>1</td>
<td>108042</td>
</tr>
<tr>
<td>LIS (low ionic strength) chamber</td>
<td>1</td>
<td>5189900</td>
</tr>
<tr>
<td>Disposable wipes, 11 x 22 cm</td>
<td>280/pkg</td>
<td>2097000</td>
</tr>
<tr>
<td>Wash bottle, polyethylene, 500 mL</td>
<td>1</td>
<td>62011</td>
</tr>
<tr>
<td>Probe stand for standard Intellical probes</td>
<td>1</td>
<td>8508850</td>
</tr>
<tr>
<td>Soaker bottle for probe storage</td>
<td>1</td>
<td>5192900</td>
</tr>
<tr>
<td>Probe cable depth markers for rugged Intellical probes</td>
<td>5/pkg</td>
<td>5828610</td>
</tr>
<tr>
<td>Shroud kit for rugged probes</td>
<td>1</td>
<td>5825900</td>
</tr>
<tr>
<td>Storage caps for rugged PHC and MTC probes</td>
<td>5/pkg</td>
<td>5857305</td>
</tr>
</tbody>
</table>
USEPA Electrode Method  
Method 8156  
pH electrode

Scope and application: For drinking water\(^1\), wastewater\(^2\) and process water.

\(^1\) Based on Standard Method 4500-H+B, ASTM Method D1293-95 and USEPA Method 150.1
\(^2\) 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

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

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

<table>
<thead>
<tr>
<th>Description</th>
<th>Quantity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Beaker or sample containers</td>
<td>3</td>
</tr>
<tr>
<td>Wash bottle with deionized water</td>
<td>1</td>
</tr>
<tr>
<td>pH buffers (4.0, 7.0, 10.0)</td>
<td>3</td>
</tr>
</tbody>
</table>

### 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 mV (±3) at 25 °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 \(-\log_{10} a(H^+)\), where \(a(H^+)\) 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**

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

**Sension+ meters and probes**

<table>
<thead>
<tr>
<th>Description</th>
<th>Unit</th>
<th>Item no.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sension+ pH3 lab pH meter</td>
<td>each</td>
<td>LPV2010T.97.002</td>
</tr>
<tr>
<td>Sension+ pH31 GLP lab pH meter</td>
<td>each</td>
<td>LPV2110T.97.002</td>
</tr>
<tr>
<td>Sension+ MM340 lab two input, pH/mV/ISE meter</td>
<td>each</td>
<td>LPV2200.97.0002</td>
</tr>
<tr>
<td>Sension+ MM374 lab two input, pH/mV/EC/ISE meter</td>
<td>each</td>
<td>LPV4110.97.0002</td>
</tr>
<tr>
<td>Sension+ MM378 lab two input, pH/ISE/EC/DO meter</td>
<td>each</td>
<td>LPV4130.97.0002</td>
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<tr>
<td>Sension+ 5010T combination pH probe</td>
<td>each</td>
<td>LZW5010T.97.002</td>
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<tr>
<td>Sension+ 5011T combination pH probe</td>
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<td>LZW5011T.97.002</td>
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<tr>
<td>Sension+ 5014T combination pH probe</td>
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<td>Sension+ 5021T combination pH probe</td>
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<td>Sension+ 5050T combination pH probe</td>
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<td>Sension+ 5051T combination pH probe</td>
<td>each</td>
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<tr>
<td>Sension+ 5052T combination pH probe</td>
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<td>Sension+ 5045 multi-parameter probe</td>
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Sension+ meters and probes (continued)

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<thead>
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<th>Item no.</th>
</tr>
</thead>
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<tr>
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<tr>
<td>Sension+ 5049 multi-parameter probe</td>
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<td>LZW5049.97.0002</td>
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<td>Sension+ 5059 multi-parameter probe</td>
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Recommended standards

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<tr>
<th>Description</th>
<th>Unit</th>
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</tr>
</thead>
<tbody>
<tr>
<td>pH 4.01 buffer solution, Singlet one-use packets, 20 mL each</td>
<td>20/pkg</td>
<td>2770020</td>
</tr>
<tr>
<td>pH 7.00 buffer solution, Singlet one-use packets, 20 mL each</td>
<td>20/pkg</td>
<td>2770120</td>
</tr>
<tr>
<td>pH 10.01 buffer solution, Singlet one-use packets, 20 mL each</td>
<td>20/pkg</td>
<td>2770220</td>
</tr>
<tr>
<td>pH 4.01 and pH 7.00 buffer solution kit, Singlet one-use packets, 20 mL each</td>
<td>2 x 10/pkg</td>
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</tr>
<tr>
<td>pH 7.00 and 10.01 buffer solution kit, Singlet one-use packets, 20 mL each</td>
<td>2 x 10/pkg</td>
<td>2769820</td>
</tr>
<tr>
<td>pH color-coded buffer solution kit (NIST), 500 mL, includes:</td>
<td>1</td>
<td>2947600</td>
</tr>
<tr>
<td>pH 4.01 ± 0.02 pH buffer (NIST)</td>
<td>500 mL</td>
<td>2283449</td>
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<tr>
<td>pH 7.00 ± 0.02 pH buffer (NIST)</td>
<td>500 mL</td>
<td>2283549</td>
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<tr>
<td>pH 10.01 ± 0.02 pH buffer (NIST)</td>
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<td>2283649</td>
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<td>Powder pillows:</td>
<td></td>
<td></td>
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<tr>
<td>pH 4.01 ± 0.02 pH buffer powder pillow (NIST)</td>
<td>50/pkg</td>
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<tr>
<td>pH 7.00 ± 0.02 pH buffer powder pillow (NIST)</td>
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<td>pH 10.01 ± 0.02 pH buffer powder pillow (NIST)</td>
<td>50/pkg</td>
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<td>Radiometer Analytical (IUPAC Series certified pH standards):</td>
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<td></td>
</tr>
<tr>
<td>pH 1.679 ± 0.010 at 25 °C (77 °F)</td>
<td>500 mL</td>
<td>S11M001</td>
</tr>
<tr>
<td>pH 4.005 ± 0.010 at 25 °C (77 °F)</td>
<td>500 mL</td>
<td>S11M002</td>
</tr>
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<td>pH 6.865 ± 0.010 at 25 °C (77 °F)</td>
<td>500 mL</td>
<td>S11M003</td>
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<td>pH 7.000 ± 0.010 at 25 °C (77 °F)</td>
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<td>S11M004</td>
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<td>pH 9.180 ± 0.010 at 25 °C (77 °F)</td>
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<td>pH 10.012 ± 0.010 at 25 °C (77 °F)</td>
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<td>pH 12.45 ± 0.05 at 25 °C (77 °F)</td>
<td>500 mL</td>
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<tr>
<td>pH buffer 1.09, technical</td>
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<td>S11M009</td>
</tr>
<tr>
<td>pH buffer 4.65, technical</td>
<td>500 mL</td>
<td>S11M010</td>
</tr>
<tr>
<td>pH buffer 9.23, technical</td>
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Accessories

<table>
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<tr>
<th>Description</th>
<th>Unit</th>
<th>Item no.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Beaker, polypropylene, 50 mL, low form</td>
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</tr>
<tr>
<td>Beaker, polypropylene, 100-mL</td>
<td>each</td>
<td>108042</td>
</tr>
<tr>
<td>Bottle, wash, 500 mL</td>
<td>each</td>
<td>62011</td>
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<tr>
<td>Stir bar, magnetic, 2.2 x 0.5 cm (7/8 x 3/16 in.)</td>
<td>each</td>
<td>4531500</td>
</tr>
<tr>
<td>Stirrer, electromagnetic, 120 VAC, with electrode stand</td>
<td>each</td>
<td>4530001</td>
</tr>
<tr>
<td>Stirrer, electromagnetic, 230 VAC, with electrode stand</td>
<td>each</td>
<td>4530002</td>
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</table>
## Accessories (continued)

<table>
<thead>
<tr>
<th>Description</th>
<th>Unit</th>
<th>Item no.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample bottle with screw-top cap, polypropylene, 500-mL</td>
<td>each</td>
<td>2758101</td>
</tr>
<tr>
<td>Water, deionized</td>
<td>4 L</td>
<td>27256</td>
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</tbody>
</table>

pH, USEPA electrode method
Section 11

Standard Methods, SOPs, & QA/QC
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

- 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

- 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

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

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

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

- DOC – demonstration of capability
- MDL – method detection level
- LRB/MB – method blank
- LFB – laboratory fortified blank (standard)
- LFMLFMD – laboratory fortified matrix/duplicate (spike)
- Internal standards, surrogate standards or tracer – only applies to organic analysis and radiochemistry
- Calibration initial and continuing
- Control charts or other trend analysis
- Corrective action – root cause analysis
- QC acceptance criteria
- Definition of a batch (preparation and analytical)
- Minimum frequency for conducting all QC elements
- 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/COBOD, 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 23 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<sub>1</sub> – value based on spiked samples
  - Compute MDL<sub>b</sub> – 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<sub>1</sub>: Value calculated from the spiked samples
    - Minimum of 2 spiked samples on each instrument
    - Minimum of 8 per year (2 per quarter)
  - MDL<sub>b</sub>: 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 23 months you need to recalculate your MDL₁ and MDL₂.
  - Ideally, use all method blank results from the last 24 months for the MDL₂ calculation.
  - There is an option to use less data included in the rule.
  - The verified MDL is the higher of the two numbers (either MDL₁ or the MDL₂).
  - Your existing MDL may be left unchanged if specific criteria are met.
  - See 40 CFR 136 Appendix B.

### 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 LFMs) 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 ±2σ and ±3σ limits for the WL and CL, respectively, where σ 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.

**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 1020.11)

- 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**

- (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 ± 10%
    - ICV/CCV ± 10%
    - LFM/LFMD ± 20%
    - RPD < 20%
    - Reporting limit = MDL

**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?**

KEEP CALM AND LET QA/QC HANDLE IT
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

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<tr>
<td>Section 13</td>
<td>pH Calibration Log Sheet</td>
<td>p</td>
</tr>
<tr>
<td>Appendix 1</td>
<td></td>
<td>p</td>
</tr>
</tbody>
</table>
**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](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:**


**Be aware of:**

2. Tennessee Code Annotated
   - TCA section 68-221-101 et seq: Water and Sewerage
   - TCA section 68-221-901 et seq: Water Environmental Health
Section 12

Chlorine
Chlorination

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

Agents of disinfection

- Chemical Disinfectants
  - Chlorine -- Cl₂
    - 100% pure
    - gas
  - Calcium hypochlorite -- Ca(OCl)₂
    - 65% pure
    - solid
    - HTH – high test hypochlorite
  - Sodium hypochlorite -- NaOCl
    - 5-15% pure
    - Liquid
    - Bleach

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

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
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

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
    \[ \text{Demand} = \text{Dosage} - \text{Residual} \]

Chlorine Disinfection

- Chlorine Residual = the total of all the compounds with disinfecting properties plus any remaining free (uncombined) chlorine
  \[ \text{Residual} = \text{Dosage} - \text{Demand} \]

Chlorine Disinfection

- Chlorine Dose = the amount of chlorine needed to satisfy the chlorine demand and the amount of chlorine residual needed for disinfection
  \[ \text{Chlorine Dose} = \text{Demand} + \text{Residual} \]
Breakpoint Chlorination

Factors influencing disinfection

Factors influencing disinfection

Factors influencing Disinfection

Factors influencing Disinfection

Factors influencing disinfection

Process of Disinfection

• pH
  o Chlorine disinfects faster at pH of 7 than at pH > 8
  o Hypochlorous acid disassociates at a higher pH

• Temperature
  o Higher temperature means more efficient disinfection
  o Longer contact time required at lower temperatures
  o Chlorine will dissipate faster in warmer waters

• Microorganisms
  o Number and type greatly influence disinfection effectiveness
  o Cysts and viruses can be very resistant to disinfection

• Turbidity
  o Excessive turbidity greatly reduces disinfection efficiency

• Organic Matter
  o Organics can consume great amounts of disinfectants while forming unwanted compounds such as disinfection by-products
  o Reactions with organics and other reducing agents will significantly reduce the amount of chemical available for disinfection

• Inorganic Matter
  o Ammonia can combine with disinfectant chemical to form side compounds

• Reducing Agents
  o Any substance that will readily donate electrons
  o Demand for chlorine by reducing agents must be met before chlorine becomes available to accomplish disinfection
  o Inorganic reducing agents
    • Hydrogen sulfide gas (H₂S)
    • Ferrous ion (Fe²⁺)
    • Manganous ion (Mn²⁺)
    • Ammonia (NH₃)
    • Nitrite ion (NO₂⁻)
Chlorine ($\text{Cl}_2$)

- **Reaction with Water**
  \[ \text{Cl}_2 + \text{H}_2\text{O} \rightarrow \text{HOCl} + \text{HCl} \]
  
  - Free chlorine combines with water to form hypochlorous acid
    - Most effective disinfectant
    - Dissociates at higher pH (greater than 7)
  
  - 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 OCI⁻

Lowers pH

Hypochlorite (OCI⁻)

- **Reactions with Water**
  - May be applied in the form of calcium hypochlorite ($\text{Ca(OCI)}_2$) or sodium hypochlorite ($\text{NaOCl}$)
    \[ \text{Ca(OCI)}_2 + \text{H}_2\text{O} \rightarrow \text{HOCl} + \text{Ca(OH)}_2 \]
    \[ \text{NaOCl} + \text{H}_2\text{O} \rightarrow \text{HOCl} + \text{NaOH} \]
  
  - Raises pH due to OH⁻ ion
  
  - If is Ca(OCI)₂ injected at the same point of as sodium fluoride, a severe crust can form at injection point

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

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

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

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
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

DPD Method

- Standard Method 4500-Cl G
- Grab sample, no preservative
- Anayze 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)

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

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
  o This should cause the meter reading to deflect downward
  o Adjust the control knob as needed to keep the pointer on the scale
  o 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)
  o 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.

\[

dose_{mg/L} = \frac{chlorine_{lb/day}}{(Q_{MGD})(8.34 \text{ lb/day})} \cdot \frac{50 \text{ lbs/day}}{(0.85 \text{ MGD})(8.34 \text{ lbs/gal})}
\]

\[
dose_{mg/L} = 7.1 \text{ mg/L}
\]

Demand, mg/L = \[ Cl_2 \text{ Dose, mg/L} - Cl_2 \text{ Residual, mg/L} \]
Demand, mg/L = 7.1 mg/L - 0.5 mg/L
Demand, mg/L = 6.6 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.

\[
\text{Cl}_2, \text{ lbs/day} = (\text{Dose, mg/L})(Q, \text{ MGD})(8.34 \text{ lbs/gal})
\]

\[
\begin{align*}
\text{Cl}_2, \text{ lbs/day} &= (6 \text{ mg/L})(8 \text{ MGD})(8.34 \text{ lbs/gal}) \\
&= 400 \text{ lbs/day}
\end{align*}
\]

### 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.

\[
\text{Cl}_2, \text{ lbs/day} = \frac{(\text{Dose, mg/L})(Q, \text{ MGD})(8.34 \text{ lbs/gal})}{\text{HTH, chlorine percent as decimal}}
\]

\[
\begin{align*}
\text{Cl}_2, \text{ lbs/day} &= \frac{(6 \text{ mg/L})(8 \text{ MGD})(8.34 \text{ lbs/gal})}{0.65} \\
&= 616 \text{ lbs/day}
\end{align*}
\]
**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?
   a. Amperometric titration
   b. DPD Colorimetric
   c. DPD Color Comparator
   d. DPD Titrimetric
   e. 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  Method 8021
0.02 to 2.00 mg/L Cl₂  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>
<thead>
<tr>
<th>Instrument</th>
<th>Sample cell orientation</th>
<th>Sample cell</th>
</tr>
</thead>
<tbody>
<tr>
<td>DR 6000</td>
<td>The fill line is to the right.</td>
<td>![Image]</td>
</tr>
<tr>
<td>DR 3800</td>
<td>The fill line is toward the user.</td>
<td>2495402</td>
</tr>
<tr>
<td>DR 2800</td>
<td>The orientation mark is toward the user.</td>
<td>2401906</td>
</tr>
<tr>
<td>DR 2700</td>
<td></td>
<td>![Image]</td>
</tr>
<tr>
<td>DR 1900</td>
<td></td>
<td>![Image]</td>
</tr>
</tbody>
</table>

Table 2 Instrument-specific information for AccuVac Ampuls

<table>
<thead>
<tr>
<th>Instrument</th>
<th>Adapter</th>
<th>Sample cell</th>
</tr>
</thead>
<tbody>
<tr>
<td>DR 6000</td>
<td></td>
<td>2427606</td>
</tr>
<tr>
<td>DR 5000</td>
<td></td>
<td></td>
</tr>
<tr>
<td>DR 900</td>
<td></td>
<td></td>
</tr>
<tr>
<td>DR 3900</td>
<td>LZV846 (A)</td>
<td>![Image]</td>
</tr>
<tr>
<td>DR 1900</td>
<td>9609900 or 9609800 (C)</td>
<td>![Image]</td>
</tr>
<tr>
<td>DR 3800</td>
<td>LZV584 (C)</td>
<td>![Image]</td>
</tr>
<tr>
<td>DR 2800</td>
<td></td>
<td>![Image]</td>
</tr>
<tr>
<td>DR 2700</td>
<td></td>
<td>![Image]</td>
</tr>
</tbody>
</table>
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

<table>
<thead>
<tr>
<th>Description</th>
<th>Quantity</th>
</tr>
</thead>
<tbody>
<tr>
<td>DPD Free Chlorine Reagent Powder Pillows, 10-mL</td>
<td>1</td>
</tr>
<tr>
<td>Sample cells. (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)</td>
<td>2</td>
</tr>
</tbody>
</table>

Refer to Consumables and replacement items on page 6 for order information.

### AccuVac Ampuls

<table>
<thead>
<tr>
<th>Description</th>
<th>Quantity</th>
</tr>
</thead>
<tbody>
<tr>
<td>DPD Free Chlorine Reagent AccuVac Ampuls</td>
<td>1</td>
</tr>
<tr>
<td>Beaker, 50-mL</td>
<td>1</td>
</tr>
<tr>
<td>Sample cells (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)</td>
<td>1</td>
</tr>
<tr>
<td>Stopper for 18-mm tubes and AccuVac Ampuls</td>
<td>1</td>
</tr>
</tbody>
</table>

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 Cl₂.

### Interferences

<table>
<thead>
<tr>
<th>Interfering substance</th>
<th>Interference level</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acidity</td>
<td>More than 150 mg/L CaCO₃. 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.</td>
</tr>
<tr>
<td>Alkalinity</td>
<td>More than 250 mg/L CaCO₃. 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.</td>
</tr>
<tr>
<td>Bromine, Br₂</td>
<td>Positive interference at all levels</td>
</tr>
<tr>
<td>Chlorine Dioxide, ClO₂</td>
<td>Positive interference at all levels</td>
</tr>
<tr>
<td>Inorganic chloramines</td>
<td>Positive interference at all levels</td>
</tr>
<tr>
<td>Chloramines, organic</td>
<td>May interfere</td>
</tr>
<tr>
<td>Hardness</td>
<td>No effect at less than 1000 mg/L as CaCO₃</td>
</tr>
</tbody>
</table>
| Manganese, Oxidized (Mn⁴⁺, Mn⁷⁺) or Chromium, Oxidized (Cr⁶⁺) | Pre-treat the sample as follows:  
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.

<table>
<thead>
<tr>
<th>Program</th>
<th>Standard</th>
<th>Precision (95% Confidence Interval)</th>
<th>Sensitivity Concentration change per 0.010 Abs change</th>
</tr>
</thead>
<tbody>
<tr>
<td>80</td>
<td>1.25 mg/L Cl₂</td>
<td>1.23–1.27 mg/L Cl₂</td>
<td>0.02 mg/L Cl₂</td>
</tr>
<tr>
<td>85</td>
<td>1.25 mg/L Cl₂</td>
<td>1.21–1.29 mg/L Cl₂</td>
<td>0.02 mg/L Cl₂</td>
</tr>
</tbody>
</table>

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**

<table>
<thead>
<tr>
<th>Description</th>
<th>Quantity/Test</th>
<th>Unit</th>
<th>Item no.</th>
</tr>
</thead>
<tbody>
<tr>
<td>DPD Free Chlorine Reagent Powder Pillow, 10-mL</td>
<td>1</td>
<td>100/pkg</td>
<td>2105569</td>
</tr>
<tr>
<td>OR</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>DPD Free Chlorine Reagent AccuVac® Ampul</td>
<td>1</td>
<td>25/pkg</td>
<td>2502025</td>
</tr>
</tbody>
</table>
### Required apparatus

<table>
<thead>
<tr>
<th>Description</th>
<th>Quantity/Test</th>
<th>Unit</th>
<th>Item no.</th>
</tr>
</thead>
<tbody>
<tr>
<td>AccuVac Snapper</td>
<td>1</td>
<td>each</td>
<td>2405200</td>
</tr>
<tr>
<td>Beaker, 50-mL</td>
<td>1</td>
<td>each</td>
<td>50041H</td>
</tr>
<tr>
<td>Stoppers for 18-mm tubes and AccuVac Ampuls</td>
<td>2</td>
<td>6/pkg</td>
<td>173106</td>
</tr>
</tbody>
</table>

### Recommended standards

<table>
<thead>
<tr>
<th>Description</th>
<th>Unit</th>
<th>Item no.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chlorine Standard Solution, 2-mL PourRite® Ampules, 25–30 mg/L</td>
<td>20/pkg</td>
<td>2630020</td>
</tr>
</tbody>
</table>

### Optional reagents and apparatus

<table>
<thead>
<tr>
<th>Description</th>
<th>Unit</th>
<th>Item no.</th>
</tr>
</thead>
<tbody>
<tr>
<td>AccuVac® Ampul vials for sample blanks</td>
<td>25/pkg</td>
<td>2677925</td>
</tr>
<tr>
<td>Ampule Breaker, 2-mL PourRite® Ampules</td>
<td>each</td>
<td>2484600</td>
</tr>
<tr>
<td>Ampule Breaker, 10-mL Voluette® Ampules</td>
<td>each</td>
<td>2196800</td>
</tr>
<tr>
<td>Water, Chlorine-demand Free</td>
<td>500 mL</td>
<td>2641549</td>
</tr>
<tr>
<td>Mixing cylinder, graduated, 25-mL</td>
<td>each</td>
<td>2088640</td>
</tr>
<tr>
<td>Mixing cylinder, graduated, 50-mL</td>
<td>each</td>
<td>189641</td>
</tr>
<tr>
<td>Chlorine Standard Solution, 2-mL PourRite® Ampules, 50–75 mg/L</td>
<td>20/pkg</td>
<td>1426820</td>
</tr>
<tr>
<td>Chlorine Standard Solution, 10-mL Voluette® Ampule, 50–75 mg/L</td>
<td>16/pkg</td>
<td>1426810</td>
</tr>
<tr>
<td>DPD Free Chlorine Reagent Powder Pillows, 10-mL</td>
<td>1000/pkg</td>
<td>2105528</td>
</tr>
<tr>
<td>DPD Free Chlorine Reagent Powder Pillows, 10-mL</td>
<td>300/pkg</td>
<td>2105503</td>
</tr>
<tr>
<td>DPD Free Chlorine Reagent, 10-mL, SwifTest™ Dispenser refill vial</td>
<td>250 tests</td>
<td>2105560</td>
</tr>
<tr>
<td>Paper, pH, 0–14 pH range</td>
<td>100/pkg</td>
<td>2601300</td>
</tr>
<tr>
<td>Pipet, TenSette®, 0.1–1.0 mL</td>
<td>each</td>
<td>1970001</td>
</tr>
<tr>
<td>Pipet tips for TenSette® Pipet, 0.1–1.0 mL</td>
<td>50/pkg</td>
<td>2185696</td>
</tr>
<tr>
<td>Pipet tips for TenSette® Pipet, 0.1–1.0 mL</td>
<td>1000/pkg</td>
<td>2185628</td>
</tr>
<tr>
<td>Potassium Iodide, 30-g/L</td>
<td>100 mL</td>
<td>34332</td>
</tr>
<tr>
<td>Sodium Arsenite, 5-g/L</td>
<td>100 mL</td>
<td>104732</td>
</tr>
<tr>
<td>Sodium Hydroxide Standard Solution, 1.0 N</td>
<td>100 mL MDB</td>
<td>104532</td>
</tr>
<tr>
<td>SpecCheck™ Secondary Standard Kit, Chlorine DPD, 0–2.0 mg/L Set</td>
<td>each</td>
<td>2635300</td>
</tr>
<tr>
<td>Sulfuric Acid Standard Solution, 1 N</td>
<td>100 mL MDB</td>
<td>127032</td>
</tr>
</tbody>
</table>
Chlorine, Total

USEPA DPD Method\textsuperscript{1}  
Method 8167  
0.02 to 2.00 mg/L Cl\textsubscript{2}  
Powder Pillows or AccuVac\textsuperscript{®} 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.\textsuperscript{2} This product has not been evaluated to test for chlorine and chloramines in medical applications in the United States.

\textsuperscript{1} Adapted from Standard Methods for the Examination of Water and Wastewater.  
\textsuperscript{2} Procedure is equivalent to USEPA and Standard Method 4500-Cl G for drinking water and wastewater analysis.

\section*{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**

<table>
<thead>
<tr>
<th>Instrument</th>
<th>Sample cell orientation</th>
<th>Sample cell</th>
</tr>
</thead>
<tbody>
<tr>
<td>DR 6000</td>
<td>The fill line is to the right.</td>
<td>2495402</td>
</tr>
<tr>
<td>DR 3800</td>
<td>The fill line is toward the user.</td>
<td></td>
</tr>
<tr>
<td>DR 2800</td>
<td>The orientation mark is toward the user.</td>
<td>2401906</td>
</tr>
<tr>
<td>DR 2700</td>
<td></td>
<td></td>
</tr>
<tr>
<td>DR 1900</td>
<td></td>
<td></td>
</tr>
<tr>
<td>DR 5000</td>
<td></td>
<td></td>
</tr>
<tr>
<td>DR 3900</td>
<td></td>
<td></td>
</tr>
<tr>
<td>DR 900</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Table 2  Instrument-specific information for AccuVac Ampuls**

<table>
<thead>
<tr>
<th>Instrument</th>
<th>Adapter</th>
<th>Sample cell</th>
</tr>
</thead>
<tbody>
<tr>
<td>DR 6000</td>
<td>—</td>
<td>2427606</td>
</tr>
<tr>
<td>DR 5000</td>
<td>LZV846 (A)</td>
<td></td>
</tr>
<tr>
<td>DR 900</td>
<td>9609900 or 9609800 (C)</td>
<td></td>
</tr>
<tr>
<td>DR 3900</td>
<td>LZV584 (C)</td>
<td>2122800</td>
</tr>
<tr>
<td>DR 1900</td>
<td></td>
<td></td>
</tr>
<tr>
<td>DR 3800</td>
<td></td>
<td></td>
</tr>
<tr>
<td>DR 2800</td>
<td></td>
<td></td>
</tr>
<tr>
<td>DR 2700</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
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

<table>
<thead>
<tr>
<th>Description</th>
<th>Quantity</th>
</tr>
</thead>
<tbody>
<tr>
<td>DPD Total Chlorine Reagent Powder Pillow, 10-mL</td>
<td>1</td>
</tr>
<tr>
<td>Sample cells. (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)</td>
<td>2</td>
</tr>
</tbody>
</table>

Refer to Consumables and replacement items on page 6 for order information.

AccuVac Ampulls

<table>
<thead>
<tr>
<th>Description</th>
<th>Quantity</th>
</tr>
</thead>
<tbody>
<tr>
<td>DPD Total Chlorine Reagent AccuVac® Ampul</td>
<td>1</td>
</tr>
<tr>
<td>Beaker, 50-mL</td>
<td>1</td>
</tr>
<tr>
<td>Sample cells (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)</td>
<td>1</td>
</tr>
<tr>
<td>Stopper for 18-mm tubes and AccuVac Ampulls</td>
<td>1</td>
</tr>
</tbody>
</table>

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.

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.**
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.

12. 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.

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.

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.

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 Cl₂.

### Interferences

<table>
<thead>
<tr>
<th>Interfering substance</th>
<th>Interference level</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acidity</td>
<td>More than 150 mg/L CaCO₃. 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.</td>
</tr>
<tr>
<td>Alkalinity</td>
<td>More than 250 mg/L CaCO₃. 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.</td>
</tr>
<tr>
<td>Bromine, Br₂</td>
<td>Positive interference at all levels</td>
</tr>
<tr>
<td>Chlorine Dioxide, ClO₂</td>
<td>Positive interference at all levels</td>
</tr>
<tr>
<td>Inorganic chloramines</td>
<td>Positive interference at all levels</td>
</tr>
<tr>
<td>Chloramines, organic</td>
<td>May interfere in the result for total chlorine analysis</td>
</tr>
<tr>
<td>Hardness</td>
<td>No effect at less than 1000 mg/L as CaCO₃</td>
</tr>
</tbody>
</table>
| Manganese, Oxidized (Mn⁴⁺, Mn⁷⁺) or Chromium, Oxidized (Cr⁶⁺) | Pre-treat the sample as follows:
  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.

<table>
<thead>
<tr>
<th>Program</th>
<th>Standard</th>
<th>Precision (95% Confidence Interval)</th>
<th>Sensitivity Concentration change per 0.010 Abs change</th>
</tr>
</thead>
<tbody>
<tr>
<td>80</td>
<td>1.25 mg/L Cl₂</td>
<td>1.23–1.27 mg/L Cl₂</td>
<td>0.02 mg/L Cl₂</td>
</tr>
<tr>
<td>85</td>
<td>1.25 mg/L Cl₂</td>
<td>1.21–1.29 mg/L Cl₂</td>
<td>0.02 mg/L Cl₂</td>
</tr>
</tbody>
</table>

**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**

<table>
<thead>
<tr>
<th>Description</th>
<th>Quantity/Test</th>
<th>Unit</th>
<th>Item no.</th>
</tr>
</thead>
<tbody>
<tr>
<td>DPD Total Chlorine Reagent Powder Pillow, 10 mL</td>
<td>1</td>
<td>100/pkg</td>
<td>2105669</td>
</tr>
<tr>
<td>OR</td>
<td></td>
<td></td>
<td>2503025</td>
</tr>
</tbody>
</table>

Chlorination Chlorine, Total, DPD Method (2.00 mg/L)
### Required apparatus

<table>
<thead>
<tr>
<th>Description</th>
<th>Quantity/Test</th>
<th>Unit</th>
<th>Item no.</th>
</tr>
</thead>
<tbody>
<tr>
<td>AccuVac Snapper</td>
<td>1</td>
<td>each</td>
<td>2405200</td>
</tr>
<tr>
<td>Beaker, 50 mL</td>
<td>1</td>
<td>each</td>
<td>50041H</td>
</tr>
<tr>
<td>Stoppers for 18-mm tubes and AccuVac Ampuls</td>
<td>2</td>
<td>6/pkg</td>
<td>173106</td>
</tr>
</tbody>
</table>

### Recommended standards

<table>
<thead>
<tr>
<th>Description</th>
<th>Unit</th>
<th>Item no.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chlorine Standard Solution, 10-mL Voluette® Ampule, 50–75 mg/L</td>
<td>16/pkg</td>
<td>1426810</td>
</tr>
<tr>
<td>Chlorine Standard Solution, 2-mL PourRite® Ampules, 50–75 mg/L</td>
<td>20/pkg</td>
<td>1426820</td>
</tr>
<tr>
<td>Chlorine Standard Solution, 2-mL PourRite® Ampules, 25–30 mg/L</td>
<td>20/pkg</td>
<td>2630020</td>
</tr>
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</table>

### Optional reagents and apparatus

<table>
<thead>
<tr>
<th>Description</th>
<th>Unit</th>
<th>Item no.</th>
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<tbody>
<tr>
<td>AccuVac® Ampul vials for sample blanks</td>
<td>25/pkg</td>
<td>2677925</td>
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<tr>
<td>Ampule Breaker, 2-mL PourRite® Ampules</td>
<td>each</td>
<td>2484600</td>
</tr>
<tr>
<td>Ampule Breaker, 10-mL Voluette® Ampules</td>
<td>each</td>
<td>2196800</td>
</tr>
<tr>
<td>Water, Chlorine-demand Free</td>
<td>500 mL</td>
<td>2641549</td>
</tr>
<tr>
<td>Mixing cylinder, graduated, 25-mL</td>
<td>each</td>
<td>2088640</td>
</tr>
<tr>
<td>Mixing cylinder, graduated, 50 mL</td>
<td>each</td>
<td>189641</td>
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<tr>
<td>DPD Total Chlorine Reagent Powder Pillows, 10 mL</td>
<td>1000/pkg</td>
<td>2105628</td>
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<td>DPD Total Chlorine Reagent Powder Pillows, 10 mL</td>
<td>300/pkg</td>
<td>2105603</td>
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<tr>
<td>DPD Total Chlorine Reagent, 10-mL, SwifTest™ Dispenser refill vial</td>
<td>250 tests</td>
<td>2105660</td>
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<tr>
<td>Paper, pH, 0–14 pH range</td>
<td>100/pkg</td>
<td>2601300</td>
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<tr>
<td>Pipet, TenSette®, 0.1–1.0 mL</td>
<td>each</td>
<td>1970001</td>
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<tr>
<td>Pipet tips for TenSette® Pipet, 0.1–1.0 mL</td>
<td>50/pkg</td>
<td>2185696</td>
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<tr>
<td>Pipet tips for TenSette® Pipet, 0.1–1.0 mL</td>
<td>1000/pkg</td>
<td>2185628</td>
</tr>
<tr>
<td>Potassium Iodide, 30-g/L</td>
<td>100 mL</td>
<td>34332</td>
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<tr>
<td>Sodium Arsenite, 5-g/L</td>
<td>100 mL</td>
<td>104732</td>
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<tr>
<td>Sodium Hydroxide Standard Solution, 1.0 N</td>
<td>100 mL MDB</td>
<td>104532</td>
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<tr>
<td>SpecCheck™ Secondary Standard Kit, Chlorine DPD, 0–2.0 mg/L Set</td>
<td>each</td>
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<tr>
<td>Sulfuric Acid Standard Solution, 1 N</td>
<td>100 mL MDB</td>
<td>127032</td>
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<tr>
<td>Water, deionized</td>
<td>4 L</td>
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</table>
Section 13

Alkalinity
**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⁻
- CO₃⁻
- HCO₃⁻
- the major form of alkalinity in natural waters.

**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.

**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

- 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

PH END POINTS (HACH METHOD 8221)

<table>
<thead>
<tr>
<th>Sample Composition</th>
<th>End Point pH</th>
<th>Total Alkalinity</th>
<th>Phenolphthalein Alkalinity</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alk ~ 30 mg/L</td>
<td>pH 4.9</td>
<td>pH 8.3</td>
<td></td>
</tr>
<tr>
<td>Alk ~ 150 mg/L</td>
<td>pH 4.6</td>
<td>pH 8.3</td>
<td></td>
</tr>
<tr>
<td>Alk ~ 500 mg/L</td>
<td>pH 4.3</td>
<td>pH 8.3</td>
<td></td>
</tr>
<tr>
<td>Silicates or Phosphates present</td>
<td>pH 4.5</td>
<td>pH 8.3</td>
<td></td>
</tr>
<tr>
<td>Industrial Waste or Complex System</td>
<td>pH 4.5</td>
<td>pH 8.3</td>
<td></td>
</tr>
<tr>
<td>Routine or Automated Process</td>
<td>pH 4.5</td>
<td>pH 8.3</td>
<td></td>
</tr>
</tbody>
</table>

OTHER METHODS AVAILABLE

- Standard Method 2320 B, Titration method
- Hach Method 8221
- Hach Method 8203
- Orion pH probe
- Hach method 10239, TNT plus 870

APPARATUS

- Buret and stand
- Beaker, 250 mL
- Stir plate
- Stir bar
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.

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.

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.
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 weighs
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 (CO₃⁻)  
   - Bicarbonate alkalinity (HCO₃⁻)  
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