

Introduction to Laboratory Methods for Operators

Course # 2010



July 13-17, 2020



Introduction to Laboratory Methods for Operators Course #2010

July 13-17, 2020

Monday, July 13

- 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, July 14

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

State of Tennessee

Dept. of Environment & Conservation
Bureau of Environment

Fleming Training Center

2022 Blanton Dr.

Murfreesboro, TN 37129

Wednesday, July 15

- 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, July 16

- 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, July 17

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



Introduction to Laboratory Methods for Operators

Course# 2010

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Section 1
Laboratory Safety

Introduction to Laboratory Methods for Operators

Laboratory Safety



Safety

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



Safety

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

Laboratory Hazards

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



Be Aware

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

Infectious Materials

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



Infectious Materials

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

Infectious Materials

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



Corrosive Chemicals

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

Corrosive Chemicals

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

Corrosive Chemicals

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

Toxic Materials

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

Explosive or Flammable Materials

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

Personal Protective Equipment

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



Personal Safety and Hygiene

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



Personal Safety and Hygiene

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



Personal Safety and Hygiene

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



Personal Safety and Hygiene



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



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Original source: www.cornell.edu/ehp/ehp101

Nebraska
Department of Health & Senior Services
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SDS

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

Chemical Storage

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

Chemical Storage

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

Chemical Storage

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

Chemical Storage



Moving chemicals

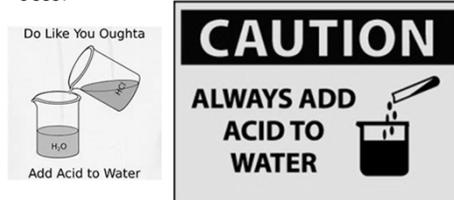
- Use cradles or tilters for carboys or other large chemical vessels
- Use a trussed hand truck for transporting cylinders of compressed gas
- Never roll a cylinder by its valve
- Clamp securely into place to prevent shifting or toppling
- Carry flammable liquids in safety cans
 - Gloves, safety shoes,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

Proper lab technique

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

Proper lab technique

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



Mercury

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



Electric shock

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

Cuts

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

Cuts

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

Burns

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

Burns

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

First Aid

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



Toxic Fumes

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

Waste Disposal

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



Fire

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

Fire

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

Fire

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



Fire

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



Fire

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



Fire

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



Fire Extinguishers

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

Fire Extinguishers

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



Pregnancy

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

Key Points to Remember

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

Laboratory Safety – Review Questions

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

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



U.S. Department of Labor



Job Safety and Health IT'S THE LAW!

All workers have the right to:

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

This poster is available free from OSHA.

Contact OSHA. We can help.

Employers must:

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

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



Section 2

Laboratory Equipment Identification

LABORATORY GLASSWARE AND EQUIPMENT

1 Introduction to Laboratory Methods for Operators

LABORATORY GLASSWARE

2

OBJECTIVES

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



3

TYPES OF GLASSWARE

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

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TYPES OF GLASSWARE

General Glassware

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

Measuring Glassware

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

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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

6

FLASKS



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

7

GRADUATED CYLINDERS

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



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VOLUMETRIC FLASKS



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

9

BURETTES AND TITRATIONS



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

10

FLASKS



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

11

FLASKS



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

12

FILTER APPARATUS

- Vacuum Pump



Gooch crucible



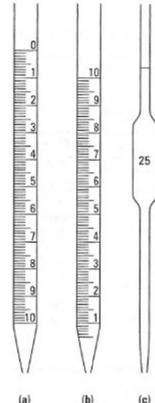
13

PIPETS

- Pipets are glass or plastic tubes, usually open at both ends, which are used to transfer specific amounts of liquid from one container to another

- 2 major categories:

1. Volumetric (Transfer)
2. Measuring (Mohr and Serological)



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

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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



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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



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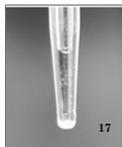
VOLUMETRIC PIPETS

- 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



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MEASURING PIPETS

- 2 types:

1. Mohr Pipet
2. Serological Pipet

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

- Not as accurate as volumetric pipets

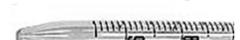
- Mohr Pipets

- Graduations on these always end before the tip



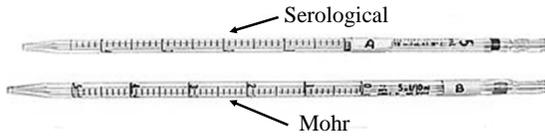
- Serological Pipets

- Graduation marks continue to the tip



18

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



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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.



KIMAX-51 USA
10ml in 1/10
TD 20°C NO. 37034

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MOHR PIPETS

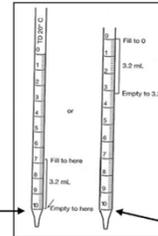
- Graduated in various divisions that do not continue onto the end of the pipet
- No liquid should be delivered past the last graduation
 - Fluid is drawn up to a graduation mark and then dispensed to the zero mark, not completely evacuating the fluid from the pipet



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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.



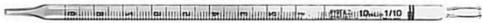
Do not empty below this line!

Do not empty below this line!

Dispensing 3.2 mL from a Mohr Pipet

22

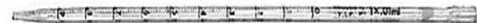
10 mL IN 1/10 ML
TD 20°C



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

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1 ML IN 1/100
TD 20°C



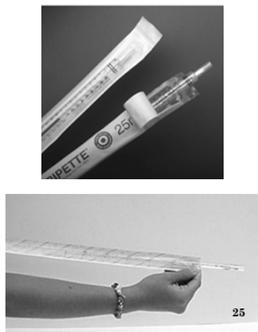
BELLCO 1.0
V. T. P. 20°C 1 x .01ml

- 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

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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



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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

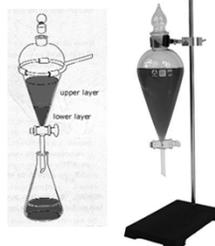


- Weighing Bottles



FUNNELS

- Separatory



- Buchner



- General



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CLEANING GLASSWARE

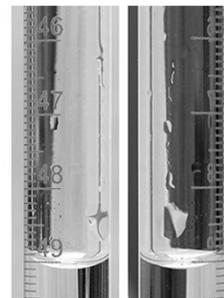
- Just because it looks clean does not mean residues are not left behind
- Results need to be accurate to use data for process control and/or reporting to the State
- Detergents should be phosphate free
 - Alconox contains phosphates!



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CLEANING GLASSWARE

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



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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



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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

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LABORATORY EQUIPMENT

35

PETRI DISH/DESSICATOR

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



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EVAPORATING DISH/CRUCIBLE

- Evaporating Dish
- Crucible



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CENTRIFUGE



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

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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



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REFRIGERATORS



Walk-in cooler



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

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INCUBATORS

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



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INCUBATORS



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

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UV STERILIZER

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

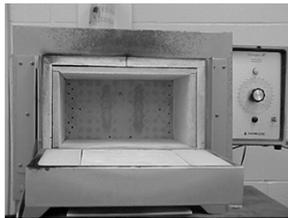


DRYING OVEN



- 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

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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



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WATER STILL



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

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HEATING AND STIRRING SAMPLES

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



BALANCES

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



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PH METER

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



50

SPECTROPHOTOMETER

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



51

COLORIMETERS

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



52

AMPEROMETRIC TITRATOR

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



53

TURBIDIMETER

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



54

CHEMICAL STORAGE

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



55

FLAMMABLE CABINET

- Flammable chemicals should be kept in a flammable cabinet



56

SAFETY EQUIPMENT



PPE (Personal Protective Equipment):

- Goggles
- Gloves
- Aprons
- Wear safety clothing

57

EYE WASH AND SHOWER

- Should be checked weekly



58

ANY QUESTIONS?

59

Laboratory Equipment – Review Questions

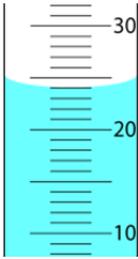
1. Which type of glassware is used for mixing and measuring approximate volumes?
2. Which type of glassware is calibrated to provide the most accurate volume measurement?
3. Give an example of when you would need to use the type of glassware from question #2 in the lab.
4. Mark whether or not the following types of pipets should be blown out (the last drop is forced out with the bulb).
 - a. Volumetric –
 - b. Mohr –
 - c. Serological –
5. What do you do with used glass pipets?
6. How long should glassware/equipment remain in the UV sterilization box in order to be properly sterilized?
7. What is the difference between a top loading balance and an analytical balance?
8. How often should eye wash stations and emergency showers be checked?
9. What type of detergent should be used to clean laboratory glassware?
10. List the standard procedure for washing glassware.

11. What is the purpose of acid washing glassware?

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

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

How to Read a Meniscus



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

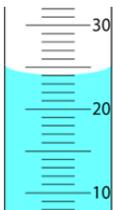
Steps to reading the meniscus in Graduated Cylinders:

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

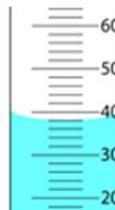
If you have trouble seeing the meniscus, try holding a black card against the opposite side of the graduated cylinder at the same height as the water.

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

Samples:

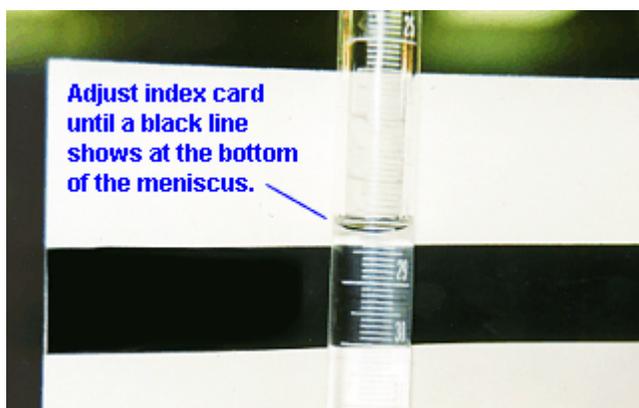


This reading would be 24



This reading would be 39

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



Cleaning Glassware

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

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

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

Sample containers:

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

Sample cells and cuvetts:

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

Flasks, beakers, etc used for metals analysis:

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

Pipets:

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

Procedure for Acid Washing Glassware

If acid washing is required, follow these steps:

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

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

Basic Water and Wastewater Vocabulary

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

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

**Indicator
(Chemical)**

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

M or Molar

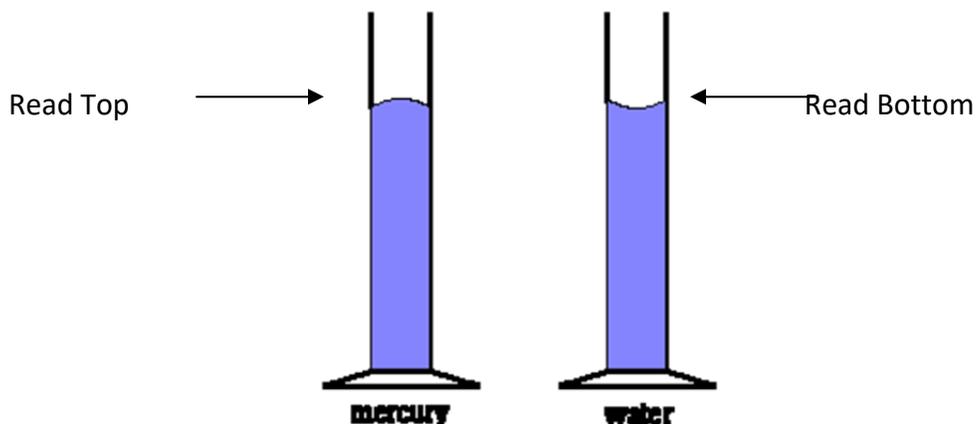
A molar solution consists of one gram molecular weight of a compound dissolved in enough water to make one liter of solution. A gram molecular weight is the molecular weight of a compound in grams. For example, the molecular weight of sulfuric acid (H_2SO_4) is 98. A 1 M solution of sulfuric acid would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

MPN

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

Meniscus

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



**Molecular
Weight**

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

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

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

Titrate A chemical solution of known strength is added on a drop by drop basis until a color change, precipitate or pH change in the sample is observed (end point). This is the process of adding the chemical solution to completion of the reaction as signaled by the end point.

Turbidity Units Expressed in “Nephelometric Turbidity Units” (NTU) when measured by a nephelometric (reflected light) instrument.

Volumetric A means of measuring unknown concentrations of water quality indicators in a sample by determining the volume of titrant or liquid reagent needed to complete particular reactions.

Section 3

Metric System

Metric System

Introduction to Laboratory Methods for Operators



{ 1 }

Is the English System Easier?

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

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

{ 2 }

History

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

{ 3 }

History

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

{ 4 }

History

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

{ 5 }

Metric System Simplicity

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

{ 6 }

The Metric System

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

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Metric System Simplicity

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

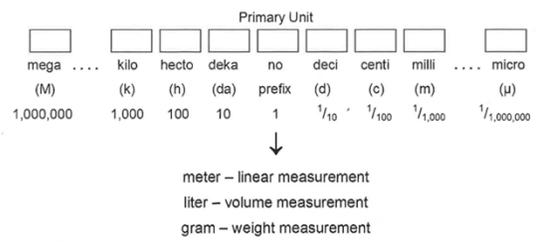
{ 8 }

Metric System

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

{ 9 }

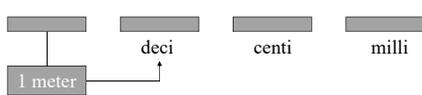
- All units can be converted into smaller or larger units by moving a decimal point



{ 10 }

Conversions

- Convert 1 meter to decimeters (dm)

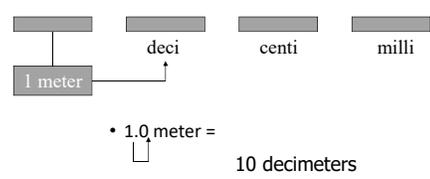


- Converting from meters to decimeters requires moving one place to the right, therefore, move the decimal point from its present position one place to the right as well.

{ 11 }

Conversions

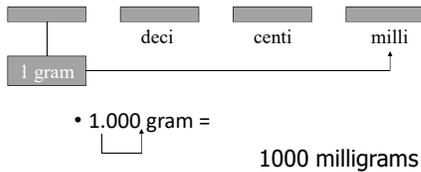
- Convert 1 meter to decimeters (dm)



{ 12 }

Conversions

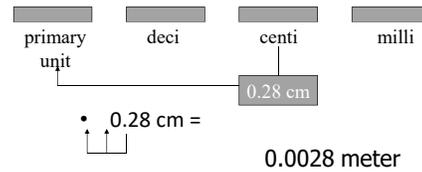
- Convert 1 gram to milligrams (mg)



{ 13 }

Conversions

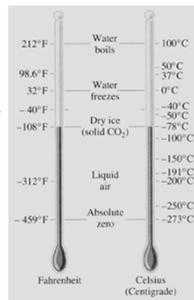
- Convert 0.28 cm to meters



{ 14 }

Temperature – Fahrenheit

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



{ 15 }

Temperature - Celsius

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



{ 16 }

Temperature

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

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

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

or

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

{ 17 }

Temperature Conversions

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

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



{ 18 }

Temperature Conversions

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

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



{ 19 }

Now we'll work some problems...

{ 20 }

Basic Lab for Water and Wastewater - Metric Conversions

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

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

Answers:

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

Basic Lab for Water and Wastewater – Temperature Conversions

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

Answers:

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

CONVERSION FACTORS AND USEFUL INFORMATION

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

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

Recommended Decimal Multiples and Submultiples and the Corresponding Prefixes and Names

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

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 (Km²) = 1,000 square meters

LENGTH CONVERSION FACTORS

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

AREA CONVERSION FACTORS

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

VOLUME CONVERSION FACTORS

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

WEIGHT CONVERSION FACTORS

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

TEMPERATURE

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

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

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

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

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

Remember: 100° between Ice/Steam = Celsius

180° between Ice/Steam = Fahr.

1. Convert Fahrenheit to Celsius:

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

2. Convert Celsius to Fahrenheit:

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

Quick Approximation:

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

CONVERSION FACTORS FOR OPERATORS

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

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

Section 4

Weight and Volume

Measurement

Balances and Pipetting

WEIGHT MEASUREMENT FOR GRAVIMETRIC ANALYSIS

Introduction to Laboratory Methods for
Operators

WEIGHT MEASUREMENT

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

WEIGHT MEASUREMENT

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



TYPES OF SCALES

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

TRIPLE-BEAM BALANCE

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



TOP LOADING BALANCES

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



ANALYTICAL BALANCES

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



USING LABORATORY BALANCES

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

PROPER BALANCE LOCATION



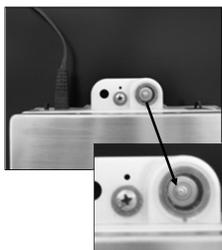
- Locate on a solid, level surface
- Never on a metal surface
- A solid marble table on a concrete floor is ideal
- Room should have a constant temperature, relative humidity
- Locate away from sunlight, drafts, moisture, and pedestrian traffic

THE TABLE

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



LEVELING THE BALANCE



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

ZEROING THE BALANCE

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



PLACING OBJECTS ON THE BALANCE

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



CALIBRATION, CHECKS, AND SERVICE

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



MANIPULATION OF WEIGHTS

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



WEIGHING CHEMICALS

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

HEAVY WEIGHTS AND OBJECTS

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

SOURCES OF ERROR

- Moisture
- Temperature
- Static Charge
- Air



MOISTURE

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

TEMPERATURE

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

STATIC CHARGE

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

AIR

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

WEIGHING SAMPLES

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

WEIGHING SAMPLES – TABLE

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

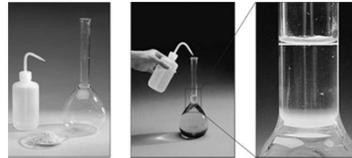
ANY QUESTIONS?

VOLUMETRIC ANALYSIS

1 Introduction to Laboratory Methods for Operators

VOLUMETRIC ANALYSIS

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



2

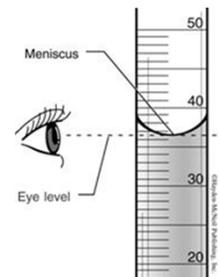
MEASUREMENT GLASSWARE

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

3

THE MENISCUS

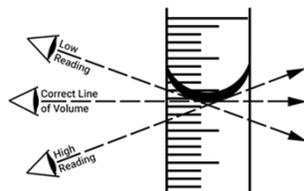
- There is a chemical attraction between water and its container
- Water tends to adhere to the sides of a container and "climb" the sides
- This climbing liquid gives a curved and distorted surface called the meniscus
- READ THE MENISCUS AT EYE LEVEL



4

THE MENISCUS

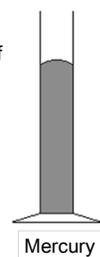
- The water level is measured at the bottom of the meniscus
 - "the belly"
- Parallax error will occur if you don't read at eye level



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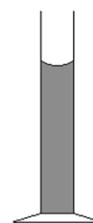
WATER VS. MERCURY

Read Top of Meniscus



Mercury

Read Bottom of Meniscus



Water

6

VOLUMETRIC GLASSWARE

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



7

HOW TO USE A VOLUMETRIC FLASK

8

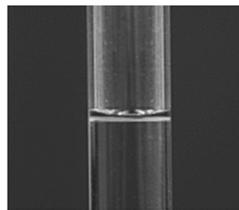
USE OF VOLUMETRIC FLASK



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

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USE OF VOLUMETRIC FLASK



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

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USE OF VOLUMETRIC FLASK



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

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HOW TO PIPET

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

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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



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TYPES OF PIPET BULBS



USE OF PIPET

○ Step 1

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



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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.

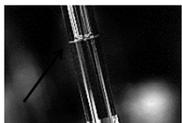


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USE OF PIPET

○ 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

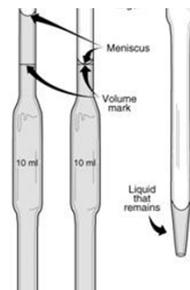


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USE OF PIPET

○ Step 4

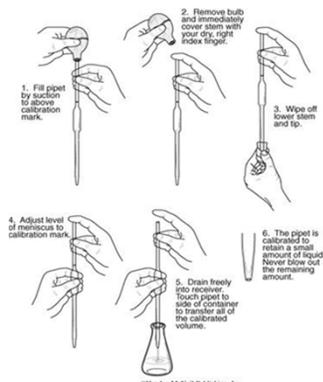
- Touch the tip of the pipet to the wall of the receiving vessel to drain the last bit of liquid
- Volumetric pipets are calibrated to have a drop of liquid remaining in the tip
 - DO NOT shake or blow out this drop of liquid



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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

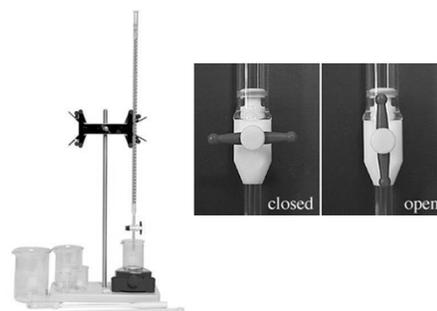
- Slow down and take your time
- Use the pipet that dispenses the closest volume to what you need
- Hold the pipet in a vertical position
- Wet pipette tips work best
 - Pull some liquid into it and then dispense before you get started
- Do not aspirate so quickly that bubbles form in the solution
 -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

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HOW TO USE A BURET

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HOW TO USE A BURET



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HOW TO USE A BURET

Filling the Buret:



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

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HOW TO USE A BURET

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



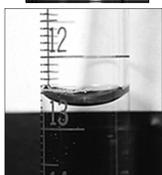
24

USE OF BURET



Begin analysis:

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



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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



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

27

Weight and Volume Measurement – Review Questions

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

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

1)

2)

3)

4)

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

Section 5

Introduction to Basic Chemistry

Intro to Basic Chemistry & Solutions Chemistry

Introduction to Laboratory Methods for Operators



Basic Chemistry

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

Basic Chemistry

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



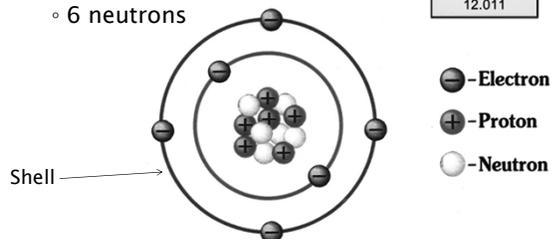
Basic Chemistry

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

Basic Chemistry

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

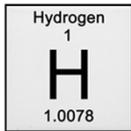
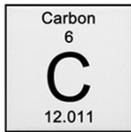
Carbon 6 C 12.011



Basic Chemistry

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

Atomic Weight

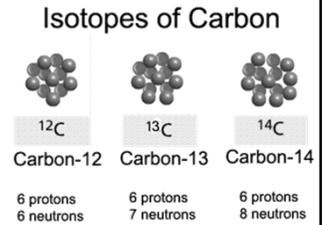


- The weight of Carbon is 12
- The weight of Hydrogen is 1
- Therefore, a carbon atom weighs 12 times more than a Hydrogen atom

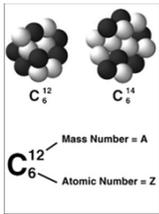
- Comparison

Isotopes

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



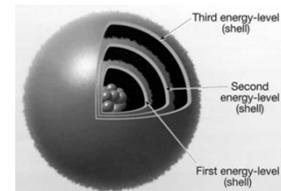
Isotopes of Carbon



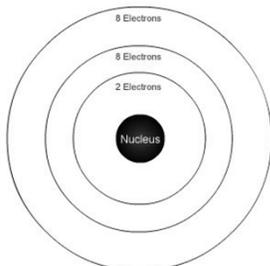
1. Carbon-12, which constitutes 99 % of all carbon atoms and serves as the standard for the atomic mass scale
 2. Carbon-14 is an isotope produced by cosmic rays bombarding the atmosphere and is radioactive, with a half-life of 5760 years
- ▶ The fact that they have varying numbers of neutrons makes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

Electron Configuration

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



Electron Energy Levels



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

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

Basic Chemistry

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

Periodic Table

KEY

Atomic Mass	12.011	-4	Selected Oxidation States
Symbol	C	+2 +4	
Atomic Number	6		Relative atomic masses are based on ¹² C = 12.000
Electron Configuration	2-4		

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

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Periodic Table of the Elements

KEY

Atomic Mass	12.011	-4	Selected Oxidation States
Symbol	C	+2 +4	
Atomic Number	6		Relative atomic masses are based on ¹² C = 12.000
Electron Configuration	2-4		

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

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

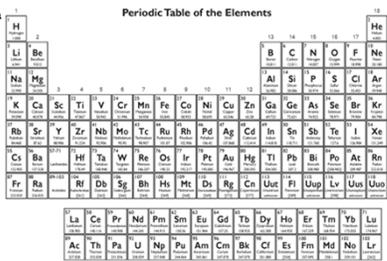
*The systematic names and symbols for elements of atomic numbers above 100 will be used until the approval of their names by IUPAC.

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The Periodic Table

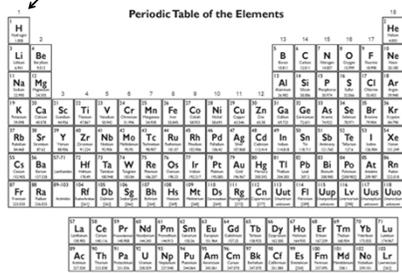
The **Period Number** is found at the beginning of a row. There are 7 periods. Atoms become larger in size as they move from left to right.



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The Periodic Table

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



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

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

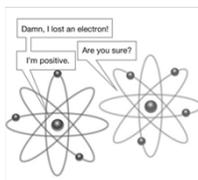


Chemical Bonds

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

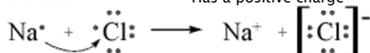


Ionic Bond

- ▶ 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

Ionic Bond

Sodium atom
Has 1 valence electron



Chlorine atom
Has 7 valence electrons

Sodium ion
Lost the electron
Has a positive charge

Chloride ion
Gained an electron
Now has a negative charge

The Octet Rule is satisfied.

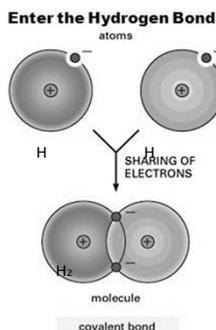
Both the chlorine atom and the sodium atom now have eight electrons in their outermost energy level.

Covalent Bond

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

Covalent Bond

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



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

- Solute 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

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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)

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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

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Percent Strength

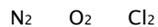
- Another way of expressing concentration
- % Strength = $\frac{\text{weight of solute}}{\text{weight of solution}} \times 100$
- Weight of Solution = Weight of solute + weight of solvent

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Compound

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



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



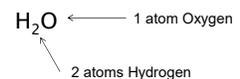
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Chemical formulas

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

H = Hydrogen
O = Oxygen

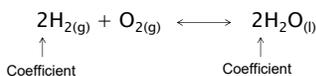


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Chemical equations

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



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

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Mole concept

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

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Mole concept

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



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

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Molarity

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

How much does 1 mole of water weigh?

- ▶ H₂O

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

- ▶ 1 mol = 18 grams

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Normality

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

Normality

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

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Standard Solutions

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

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Dilutions

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

$$C_1V_1 = C_2V_2$$

1 means stock or initial concentration or volume

2 is the concentration or volume you end up with

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Dilution Examples

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

$$C_1V_1 = C_2V_2$$

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

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

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

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Dilution Examples

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

$$C_1V_1 = C_2V_2$$

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

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

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

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

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Periodic Table of the Elements

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

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

Molarity

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

1. How much does 1 mol of NaCl weigh?

Atomic weight of Na = 23

Atomic weight of Cl = 35.5

Molecular Weight of NaCl = 58.5 g/mol

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

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

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

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

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

Molecular weight of NaCl = 58.5 g/mol

0.001 M = 0.001 mol/L

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

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

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

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

Normality

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

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

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

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

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

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

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

Step 1: Determine the MW of NaOH.

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

Step 2: Determine equivalent weight.

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

Step 3: Set up Ratio.

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

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

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

Normality

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

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

Equivalent Weights

*Think of Equivalent as Equal Valence.

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

"Equivalent weights" level the playing -field.

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

Example:

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

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

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

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

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

Equivalent Weight, Normality to determine Hardness

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

Total Hardness 180 mg/L as CaCO_3

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

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

Example:

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

Step 1: Determine MW

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

Step 2: Determine Eq. Wt.

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

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

Step 3: Set up Ratio

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

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

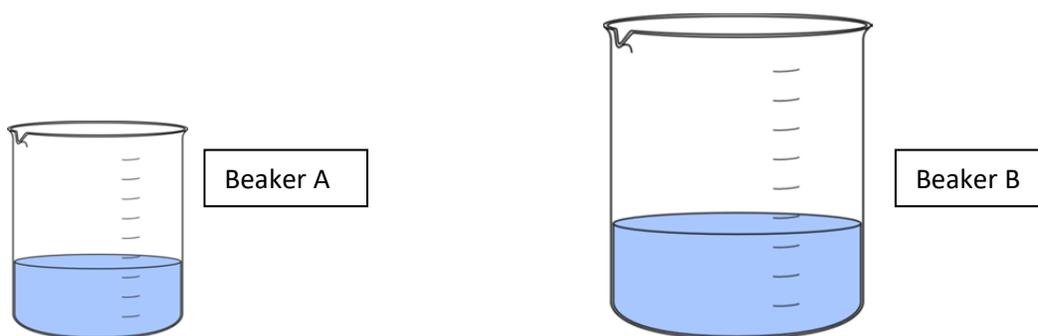
$$X = 71 \text{ mg/L as CaCO}_3$$

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

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

Intro to Basic Chemistry and Solutions Chemistry – Review Questions

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



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

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 H_2SO_4 standard solution. How many milliliters of the standard solution must be added to water to make 100 mL of a 1.2 N H_2SO_4 solution?

9. You need to make a 100 mL solution of a 0.02 N H_2SO_4 for an alkalinity titrant. You only have a 1 N H_2SO_4 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

Temperature

Introduction to Laboratory Methods for Operators



Temperature

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

Temperature - Sampling

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

Temperature

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

Thermometers

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

Thermometers

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



Thermometers

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

Thermometers

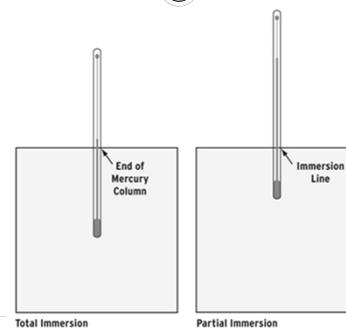
Styles of thermometers:

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

Thermometers

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

Total Immersion vs. Partial Immersion



Thermometers

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



Thermometers

4. **Digital readout**



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

Temperature Readings

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

Thermometers

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

Thermometers

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

Where is temperature used?

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

Where is temperature used

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

Any Questions?

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 Measurement

Introduction to Laboratory Methods for Operators



Definition

- Dissolved Oxygen
 - “DO” for short
 - measurement of the amount of oxygen dissolved in a unit volume of water
 - indicator of usefulness of water for a specific application



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Hold Times

- 15 minutes – note time of collection & time of analysis!



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Applications

- 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



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Applications

- 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

Applications

Sewage treatment:

- Specialized bacteria (also known as activated sludge) is added to solids in waste water treatment plants
- optimum level of DO necessary for process
- too low DO = bacteria die & decompose
- too high DO = process becomes costly

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DO Theory

- Amount of Oxygen that a given volume of water can hold is a function of:
 - The pressure the atmospheric oxygen is exerting at the air-water interface....
 - The temperature of the water
 - The amount of other substances dissolved in the water
- The higher the atmospheric pressure and the lower the temperature and conductivity, the more oxygen can be dissolved in the water

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

Temp °C	Atmospheric pressure, in millimeters of mercury																			
	795	790	785	780	775	770	765	760	755	750	745	740	735	730	725	720	715	710	705	700
15.0	10.5	10.5	10.4	10.3	10.3	10.2	10.1	10.1	10.0	9.9	9.8	9.7	9.7	9.6	9.5	9.5	9.4	9.3	9.3	9.2
15.5	10.4	10.4	10.3	10.2	10.2	10.1	10.0	9.9	9.8	9.7	9.6	9.6	9.5	9.4	9.4	9.3	9.2	9.2	9.1	9.0
16.0	10.3	10.2	10.2	10.0	10.0	9.9	9.8	9.7	9.7	9.6	9.5	9.5	9.4	9.4	9.3	9.2	9.2	9.1	9.1	9.0
16.5	10.2	10.1	10.1	10.0	9.9	9.8	9.7	9.7	9.6	9.5	9.5	9.4	9.4	9.3	9.2	9.2	9.1	9.1	9.0	8.9
17.0	10.1	10.0	10.0	9.9	9.8	9.8	9.7	9.6	9.6	9.5	9.4	9.4	9.3	9.3	9.2	9.1	9.1	9.0	8.9	8.9
17.5	10.0	9.9	9.9	9.8	9.7	9.7	9.6	9.5	9.5	9.4	9.4	9.3	9.3	9.2	9.2	9.1	9.0	9.0	8.9	8.8
18.0	9.9	9.8	9.8	9.7	9.6	9.6	9.5	9.4	9.4	9.3	9.3	9.2	9.2	9.1	9.1	9.0	9.0	8.9	8.8	8.7
18.5	9.8	9.7	9.7	9.6	9.5	9.5	9.4	9.3	9.3	9.2	9.2	9.1	9.1	9.0	8.9	8.9	8.8	8.7	8.7	8.6
19.0	9.7	9.6	9.6	9.5	9.4	9.4	9.3	9.2	9.2	9.1	9.1	9.0	8.9	8.9	8.8	8.8	8.7	8.6	8.6	8.5
19.5	9.6	9.5	9.5	9.4	9.3	9.3	9.2	9.1	9.1	9.0	9.0	8.9	8.9	8.8	8.7	8.7	8.6	8.5	8.5	8.4
20.0	9.5	9.4	9.4	9.3	9.2	9.2	9.1	9.1	9.0	8.9	8.9	8.8	8.8	8.7	8.6	8.6	8.5	8.4	8.4	8.3
20.5	9.4	9.3	9.3	9.2	9.1	9.1	9.0	8.9	8.9	8.8	8.7	8.7	8.6	8.6	8.5	8.4	8.4	8.3	8.3	8.2
21.0	9.3	9.2	9.2	9.1	9.0	9.0	8.9	8.8	8.7	8.7	8.6	8.6	8.5	8.4	8.4	8.3	8.3	8.2	8.2	8.1
21.5	9.2	9.1	9.1	9.0	8.9	8.9	8.8	8.7	8.7	8.6	8.5	8.5	8.4	8.4	8.3	8.3	8.2	8.1	8.1	8.0
22.0	9.1	9.0	9.0	8.9	8.8	8.8	8.7	8.7	8.6	8.5	8.5	8.4	8.4	8.3	8.2	8.2	8.1	8.1	8.0	7.9
22.5	9.0	8.9	8.9	8.8	8.7	8.7	8.6	8.5	8.5	8.4	8.4	8.3	8.3	8.2	8.2	8.1	8.1	8.0	8.0	7.9
23.0	8.9	8.8	8.8	8.7	8.6	8.6	8.5	8.4	8.4	8.3	8.2	8.2	8.1	8.1	8.0	8.0	7.9	7.8	7.8	7.7
23.5	8.8	8.7	8.7	8.6	8.5	8.5	8.4	8.4	8.3	8.2	8.2	8.1	8.1	8.0	8.0	7.9	7.8	7.8	7.7	7.6
24.0	8.7	8.6	8.6	8.5	8.4	8.4	8.3	8.3	8.2	8.1	8.1	8.0	8.0	7.9	7.8	7.8	7.7	7.7	7.6	7.5
24.5	8.7	8.6	8.5	8.5	8.4	8.4	8.3	8.3	8.2	8.1	8.1	8.0	8.0	7.9	7.8	7.8	7.7	7.7	7.6	7.4
25.0	8.6	8.5	8.5	8.4	8.3	8.3	8.2	8.2	8.1	8.1	8.0	8.0	7.9	7.8	7.8	7.7	7.7	7.6	7.6	7.4
25.5	8.5	8.4	8.4	8.3	8.2	8.2	8.1	8.1	8.0	8.0	7.9	7.8	7.8	7.7	7.7	7.6	7.6	7.5	7.4	7.3
26.0	8.5	8.4	8.3	8.3	8.2	8.1	8.1	8.0	8.0	7.9	7.8	7.8	7.7	7.7	7.6	7.6	7.5	7.5	7.4	7.3
26.5	8.4	8.3	8.2	8.2	8.1	8.1	8.0	8.0	7.9	7.8	7.8	7.7	7.7	7.6	7.6	7.5	7.4	7.4	7.3	7.2
27.0	8.3	8.2	8.2	8.1	8.0	8.0	7.9	7.8	7.8	7.7	7.7	7.6	7.6	7.5	7.4	7.4	7.3	7.3	7.2	7.1
27.5	8.2	8.1	8.1	8.0	8.0	7.9	7.8	7.8	7.7	7.7	7.6	7.5	7.5	7.4	7.4	7.3	7.3	7.2	7.2	7.1
28.0	8.2	8.1	8.0	8.0	7.9	7.8	7.7	7.7	7.6	7.6	7.5	7.4	7.4	7.3	7.3	7.2	7.2	7.1	7.1	7.0
28.5	8.1	8.0	8.0	7.9	7.8	7.8	7.7	7.7	7.6	7.5	7.5	7.4	7.4	7.3	7.3	7.2	7.2	7.1	7.1	7.0
29.0	8.0	8.0	7.9	7.8	7.8	7.7	7.7	7.6	7.5	7.5	7.4	7.4	7.3	7.3	7.2	7.2	7.1	7.1	7.0	7.0
29.5	8.0	7.9	7.8	7.8	7.7	7.7	7.6	7.5	7.5	7.4	7.4	7.3	7.3	7.2	7.2	7.1	7.1	7.0	7.0	6.9

Barometric Pressure (BP)

- Barometric pressure = The pressure of the column of air above us
- The higher we go up, the less that pressure becomes
- Lower barometric pressures are found at higher elevations
- When you get a BP from the airport, it has been "corrected" to sea level

Common Conversions w/ BP

- Ex: The local airport provides you with a "corrected" barometric pressure of 29.65 inches of Hg.

To UNcorrect this measurement:

- Determine the altitude (in feet) of your facility/lab (you can use the altitude of the city/town/village).
 - Blueprints
 - Internet
 - GPS
- Elevation of Fleming Training Center is 543 ft.

Common Conversions w/ BP

- NOTE:** Pressure drops by 26 millimeters (mm, about 1 inch) for every 1000 feet above sea level
 $26 \div 1000 = 0.026$
- That's why during the process, we multiply the altitude in feet by 0.026

Common Conversions w/ BP

- Determine the correction factor (CF):
 $CF = [760 - (\text{Altitude} \times 0.026)] \div 760$
 $= [760 - (543 \times 0.026)] \div 760$
 $= [760 - 14.1] \div 760$
 $= 745.9 \div 760 = 0.9795$
- Therefore, true uncorrected barometric pressure = 29.65 x 0.9795 = 29.04

(Remember: The "corrected" BP was given as 29.65 inches of Hg. And FTC elevation = 543 ft)

Common Conversions w/ BP

4. To convert inches of mercury (Hg) to mm of mercury (Hg):
inches of Hg X 25.4 = mm of Hg
Therefore 29.04 X 25.4 = 737.6

Your uncorrected BP is either 29.04 inches of Hg or 737.6 mm of Hg.

BP Conversion Example

- Example:
Weather.com reports a stable 29.88* in of Hg.
We are at 543 ft and our water sample is at 21°C.
 - CF = $[760 - (\text{Altitude} \times 0.026)] \div 760$
= $745.8 \div 760 = 0.98$
 - Therefore, **true uncorrected** barometric pressure = $29.88 \times 0.98 = 29.32$ in of Hg
 - Or $29.32 \times 25.4 = 744.7$ mm of Hg

*Weather.com reports values at sea level, this needs to be corrected for your elevation.

BP Conversion Example

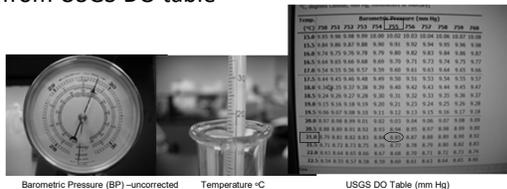
- The pressure of oxygen at sea level is **160 mm of Hg** because oxygen is about 21% of the earth's atmosphere
 - 21% of 760 (average sea level barometric pressure) is about 160 mm of Hg.
 - $(0.21)(760) = 159.6$
- https://www.fondriest.com/pdf/yysi_do_handbook.pdf

BP Conversion Example

- Find % Saturation Calibration Value:
 - $\frac{(0.21)(745)(100)}{160} = \frac{15,645}{160} = 97.8\%$
- Find Concentration Calibration Value:
 - At 21°C, the oxygen solubility table reports 8.915 mg/L
 - $(8.915)(.978) = 8.72$ mg/L

BP Conversion Example

Easy way to do this - Direct Read from Barometer & Thermometer to determine DO from USGS DO table



Conversion - Temperature

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

Effect of Partial Pressure of Oxygen on Dissolved O₂

- Water in contact with air will absorb air (O₂)
 - until the pressure at air-water interface is equal...
 - said to be saturated, which is about 5 to 10 parts of oxygen to one million parts of water



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Effect of Temperature on Dissolved Oxygen

- Pot of boiling water...
 - bubbles form on bottom & sides of pot...
 - number & size of bubbles increase with temperature...
 - These are air bubbles that have been dissolved in water



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Two Types of Measurement

- Electrode
- LDO probe

- Winkler titration

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Electrodes

- An electrode system where DO reacts at the cathode producing a measurable electrochemical effect.
- Effect can be galvanic (ability to conduct an electrical current), polarographic (electrochemical), or potentiometric (measurement of voltages).

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Dissolved Oxygen

Electrochemical method

- Two metal electrodes in contact with electrolyte and separated from test solution by gas permeable membrane and a constant voltage created
- Oxygen diffuses through membrane and is reduced at the cathode by the voltage
- This process produces a current flow, which is detected by the meter, and is proportional to the partial pressure of oxygen

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Dissolved Oxygen

- Meter calibrated in saturated air
- Winkler test may be used as a QC check
- Correction for altitude or barometric pressure
- Membrane checked and changed regularly
- Temperature calibration data verified

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Dissolved Oxygen Analysis

- Analyze immediately
 - Maximum holding time = 15 minutes
- Preservation = none
- Sample should be measured in situ
- May be collected as a grab sample with minimal aeration into a BOD bottle with a glass stopper

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Common Deficiencies

- Samples for dissolved oxygen were collected in a bucket and then poured into the BOD bottle
- The D.O. probe was immersed in the water during calibration
- The D.O. probe had a water droplet on the end during calibration
- There was an air bubble under the membrane on the probe
- The meter was air calibrated by placing the probe on the counter

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Luminescent Dissolved Oxygen



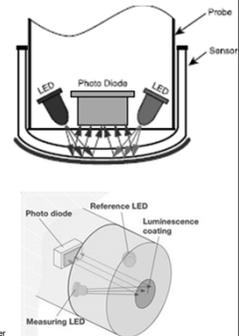
- No membrane
- No electrolyte to foul or poison
- Won't affect readings
- Accurate & stable readings

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Luminescent Dissolved Oxygen

- The sensor is coated with a luminescent material, called luminophore, which is excited by blue light from an internal LED.
- As the luminescent material relaxes, it emits a red light, and this luminescence is proportional to the dissolved oxygen present.
- The luminescence is measured both in terms of its maximum intensity and its decay time.



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Winkler

- Winkler method (azide modification)
- SM 4500-OC
- Titrimetric wet chemistry test that measures the amount of oxygen present based on conversion of oxygen to iodine



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Winkler

- Titrate with 0.025M Sodium Thiosulfate
 - Use a burette with 0.5 mL increments
- Titrate until a pale straw color



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We will now calibrate an LDO probe
in the lab...



Water Resources of the United States

DOTABLES Result

Oxygen Solubility Table

Solubility of oxygen in fresh water at various temperatures and pressures

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

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

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

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

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Dissolved Oxygen (DO) – Review Questions

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

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

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

4. What is barometric pressure?

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

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

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

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

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

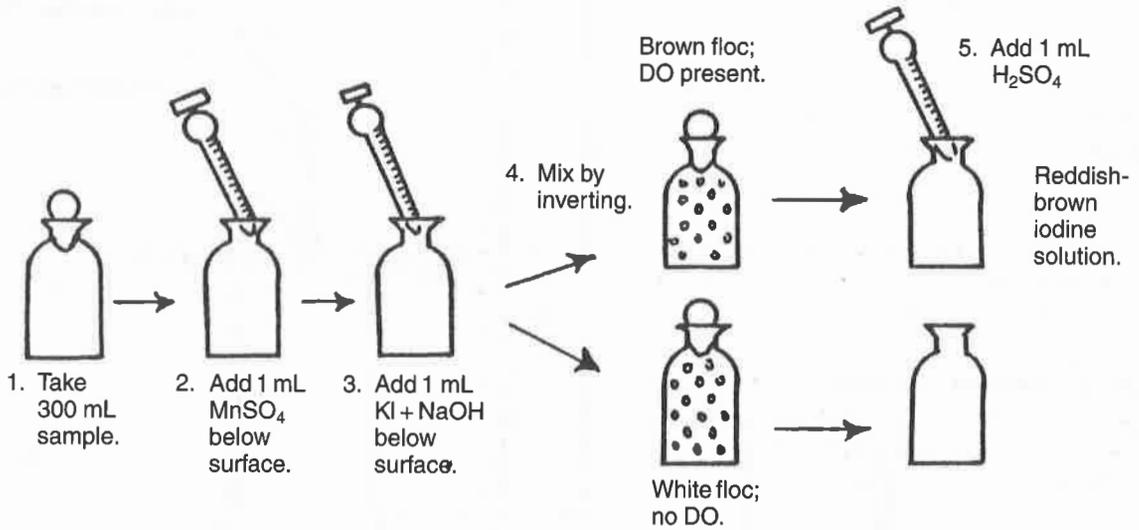
$$CF = [760 - (\text{Altitude} \times 0.026)] \div 760$$

Use that correction factor to determine the uncorrected BP.

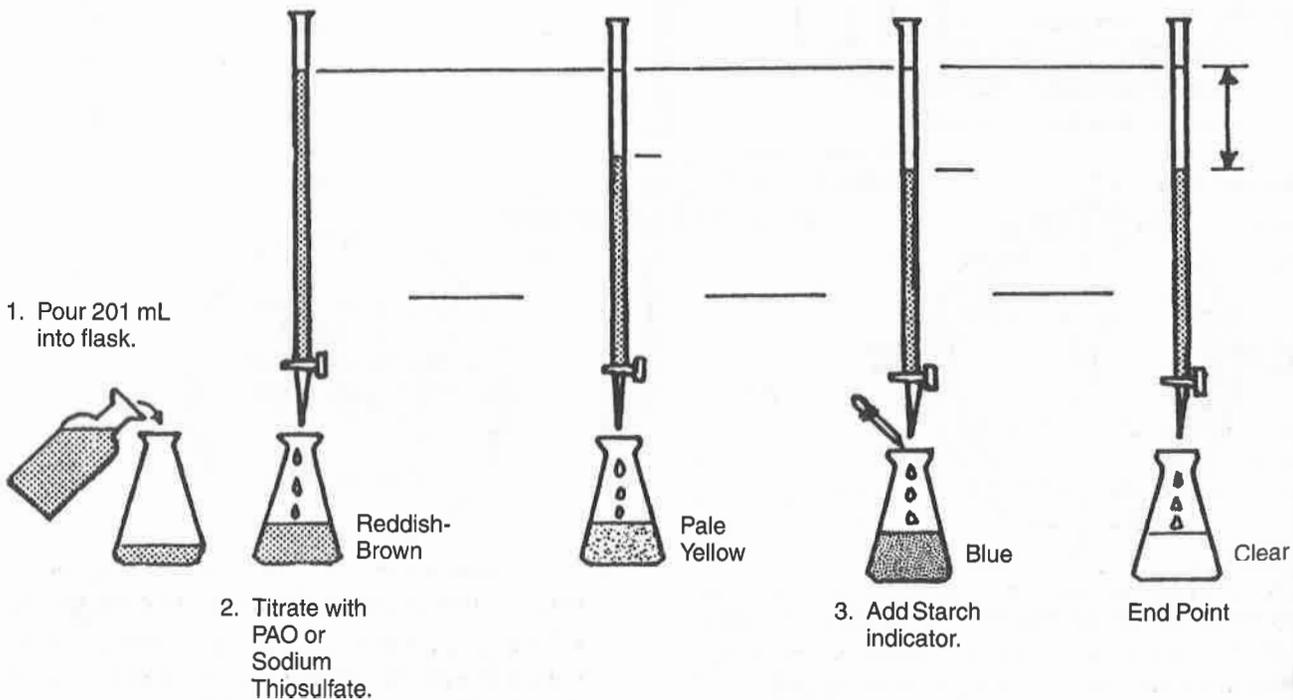
10. Now that you have calculated the uncorrected BP, you must use the USGS DOTABLE find the oxygen solubility. The room temperature is 68°F and must be converted to Celsius. Use that information, in conjunction with the uncorrected BP, to determine the oxygen solubility.

Sodium Azide Modification of the Winkler Method

OUTLINE OF PROCEDURE FOR DO



Titration of Iodine Solution:



Section 8

Sampling

SAMPLING

Introduction to Laboratory Methods for Operators



1

Why Sample?

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

2

Sampling Plan

- There are many questions to consider before actually collecting a sample
- The answer to these questions will help you put together a sampling plan
 1. Why is the sample being collected?
 2. What tests need to be run on the sample?
 3. Where is the sample going to be collected from?
 4. How is the sample going to be collected?
 5. When does the sample need to be collected/analyzed?
 6. Who is going to analyze the sample?

3

Considerations

- Collection
- Volume
- Storage and preservation
- Sample points
- Sampling frequency

- Include Sampling Plan in SOP

4

Grab Sample

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

5

Composite Sample

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

6

Composite Sample

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

7

Sampling Guidelines

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

8

Sampling Guidelines

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

9

Sample Labeling

Location: 196 E. Main Street, Springfield, TN

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

Sampled by: BS (Bob Smith)

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

10

Sample Labeling

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

11

Chain of Custody

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

12

Chain of Custody

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

13

Sample Volume

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

14

Sampling Point Selection

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

15

Sampling Devices



Automatic:

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

16

Sampling Devices

Manual:

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



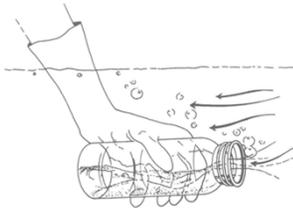
17

Sampling Devices

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

18

Subsurface Sampling

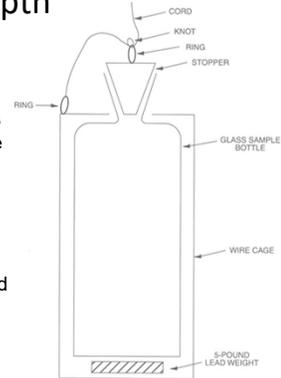


- Grasp container at base
- Plunge bottle mouth down into water
 - To avoid introducing any floating material
- Position mouth of bottle into current and away from hand
- Tip bottle slightly upward to allow air to exit so bottle can fill

19

Homemade Depth Sampler

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



20

Sources of Error

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

21

Preservation Techniques

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

22

Preservation

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

23

Drinking Water

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

24

Lead and Copper Rule

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

25

Any Questions?

26

Sampling – Review Questions

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

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

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

Section 9

Turbidity

TURBIDITY

Introduction to Laboratory Methods for Operators



1

Turbidity

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

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2

Turbidity

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

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3

Importance

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

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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)

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5

Instruments

Hach 2100N Laboratory
Benchtop Turbidimeter



Hach 2100Q
Portable Turbidimeter

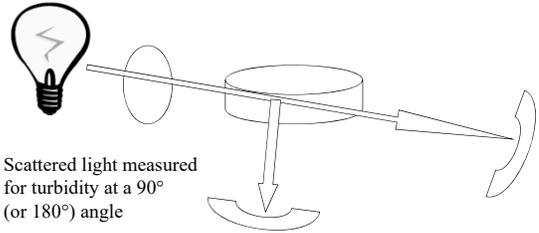
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Instruments

Hach TU5 Series Benchtop
and Online Turbidimeter



Turbidimeters - 2100N

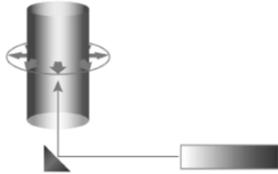


- ☐ Scattered light measured for turbidity at a 90° (or 180°) angle
- ☐ Light source from tungsten lamp passing through three precisely aligned lenses, the light is focused in a narrow, collimated beam

8

Turbidimeters - TU5 Series

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



Measuring Notes - 2100N

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

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10

Measuring Notes - 2100N

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

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11

TU5 Series Notes

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

Calibrations

2100 N

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

TU5 SERIES

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

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13

Calibrations

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

Any Questions?

Turbidity – Review Questions

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

Section 10

pH

*pH

Introduction to Laboratory Methods for Operators



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

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

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

*pH

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

*pH

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

*Acid

*More hydrogen ions (H+) in the solution

*Base

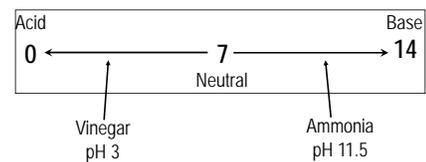
*More hydroxide ions (OH-) in the solution

*pH theory

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

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

*pH

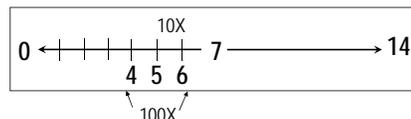


*pH Scale

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

*pH

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



*pH Scale

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8

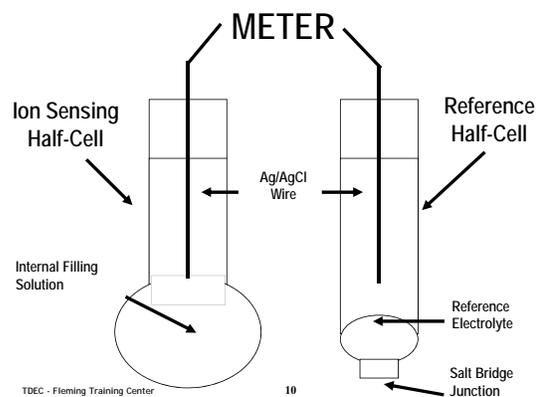
- *Probe measures hydrogen ion $[H^+]$ concentration
- *Two electrodes in probe:
 - sensing half-cell
 - reference half-cell



*How Does a pH Probe Work?

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9



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10

- *Dispenses reference solution which completes circuit for meter



*Reference Half-Cell

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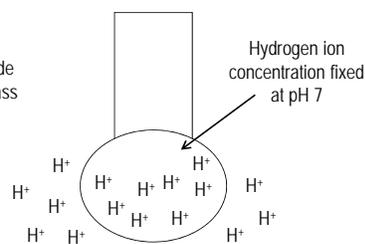
11

*Sensing Half-Cell

pH 7 Solution

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

*No potential develops



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12

*Sensing Half-Cell

pH 7 Solution

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

*No potential develops



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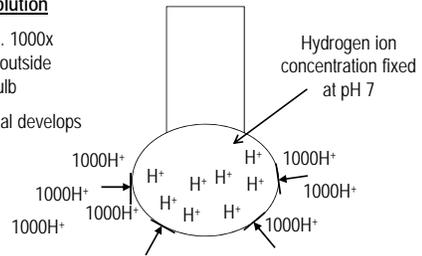
13

*Sensing Half-Cell

pH 4 Solution

H⁺ conc. 1000x greater outside glass bulb

*Potential develops



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14

*Sensing Half-Cell

pH 4 Solution

H⁺ conc. 1000x greater outside glass bulb

*Potential develops



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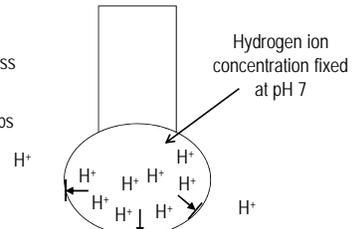
15

*Sensing Half-Cell

pH 10 Solution

H⁺ conc. 1000x greater inside glass bulb

*Potential develops



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16

*Sensing Half-Cell

pH 10 Solution

H⁺ conc. 1000x greater inside glass bulb

*Potential develops

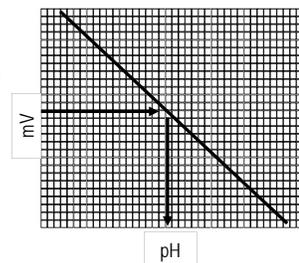


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17

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

*Decade = one pH unit



*Calibration

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18

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

*pH Sampling

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19

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

*How to Calibrate a pH Probe

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

*How to Calibrate a pH Probe

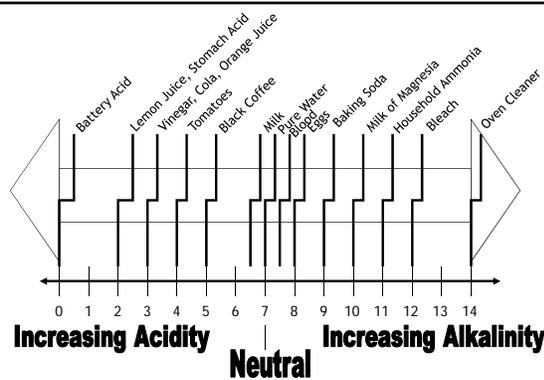
Drinking Water

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

Wastewater

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

*Common pH Ranges



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23

*Any Questions?

pH – Review Questions

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

Section 11

Standard Methods, SOPs, &

Introduction to QA/QC

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

Introduction to Lab Methods for Operators



1

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

2

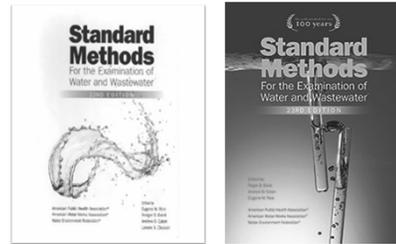
Standard Methods

- 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

3

Standard Methods

- Standard Methods for the Examination of Water and Wastewater



4

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



5

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



6

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!

7

Chlorine, Total (Method 8167)
 USEPA CDS Method: 8167
 Powder Pillows or AccuVol® Ampuls

Scope and application: For testing residual chlorine and chloramines in water, wastewater, surface water, and seawater. USEPA is not responsible for testing for chlorine and chloramines in industrial applications or the United States.

Instrument-specific information: Table 1. Instrument methods for Chlorine, Total. Table 2. Instrument methods for Chlorine, Total.

Standard Methods For the Examination of Water and Wastewater (100 years anniversary graphic)

8

Can you defend what you do?

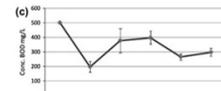
- How do you know that your data is good?
 - To be valuable or useful, data must:
 - Be representative
 - Accurately describe the characteristics and concentrations of constituents in the samples
 - Be reliable
 - Approximate or incorrect results are worse than no result at all
 - Lead to faulty interpretations



9

What do you do with data?

- Data reported to State
- Data collected for Process Control
 - Improve operations
 - Right now and also in the future
 - Example of "right now" = COD test
 - Example of "future" = historical trends, plant modifications



10

Quality Assurance

- QA refers to a total program for ensuring the reliability of data
 - A plan for lab operations that specifies the measures used to produce good data



11

Quality Assurance Programs

- 2 Primary Functions:
 - Continually monitor the reliability (accuracy and precision) of the results reported
 - "How good (accurate and precise) are the results obtained?"
 - The *determination* of quality
 - Control of quality – to meet the program requirements for reliability

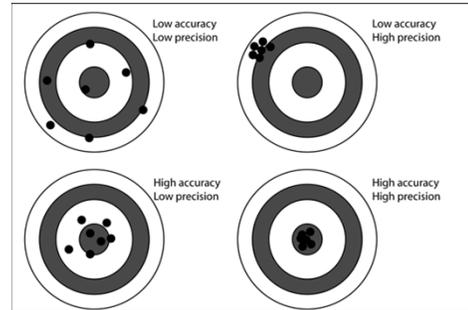
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Accuracy and Precision

- A QA/QC program consists of procedures that ensure the precision and accuracy of tests performed on a daily basis.
- **Precision** – repeatability; being able to get the same results time after time
 - Shooting at a target and hitting the same spot repeatedly
 - Duplicate samples
- **Accuracy** – closeness of test results to the correct (known) value
 - Shooting at a target and hitting bull's eye
 - Lab Fortified Blanks or Standards

13

Accuracy vs. Precision



14

Quality Control

- QC is a function of QA
 - The routine application of procedures for controlling the accuracy and precision of the measurement process
 - Includes:
 - Proper calibration of instruments
 - Use of the appropriate analytical procedures
 - The use of analytical grade reagents



15

Demonstration of Capability (DOC)

- Read the SOP and perform the method to demonstrate proficiency
- Proves that you can get acceptable results
 - *This shows that you know what you are doing*
- At a minimum, you must:
 1. Run a reagent blank
 2. Run at least 4 LFBs*
 - At a concentration between 10 times the MDL and the midpoint of the calibration curve

16

Method Detection Limit (MDL)

- EPA definition: The minimum measured concentration of a substance that can be reported with 99% confidence that the measured concentration is distinguishable from method blank results
- *The lowest concentration of an analyte that a lab can "see" or detect*
- *Can your instrument differentiate the analyte from the "background noise?"*

17

Method Detection Limit (MDL)

1. Estimate Initial MDL
2. Determine Initial MDL
 - Process at least 7 spiked samples and 7 method blank samples
 - Must be prepared in at least 3 batches on 3 separate dates and analyzed on 3 separate dates



Monday



Tuesday



Wednesday

Method Detection Limit (MDL)

- Ongoing Data Collection
 - MDL_s: Value calculated from the spiked samples
 - Minimum of 2 spiked samples on each instrument
 - Minimum of 8 per year (2 per quarter)
 - MDL_b: value calculated from the method blanks
- Annual Verification
 - *See EPA Definition and Procedure for the Determination of the Method Detection Limit, Revision 2 for full instructions on MDL procedure*

19

Method Detection Limit (MDL)

- Where do I find the MDL Calculator?
 - Go to Fleming Training Center's website
 - <https://www.tn.gov/environment/program-areas/wr-water-resources/fleming-training-center.html>
 - Middle row of boxes, left side, click on "Course Books and Reference Material"
 - In the drop-down menu on left, click on "Waste Water Information"
 - Click on "Method Update Rule – Method Detection Limit Math 2019"

20

Laboratory Reagent Blank (LRB)

- aka Reagent Blank, Method Blank, or Blank
- SM: Consists of reagent water and all reagents that are normally in contact with a sample during the procedure (distillation, incubation, etc.)
- Frequency depends on the method
 - At a minimum, include one reagent blank with each sample set



21

Laboratory Fortified Blank (LFB)

- aka Standard, or Laboratory Control Standard
- SM: A reagent water sample to which a known concentration of the analyte of interest has been added
- It is used to evaluate lab performance and analyte recovery in a blank matrix
- Process through all sample preparation and analysis steps



22

Laboratory-Fortified Matrix (LFM) and Laboratory-Fortified Matrix Duplicate (LFMD)

- aka Matrix Spike/Matrix Spike Duplicate or Spike/Spike Duplicate
- SM: An additional portion of a sample to which a known amount of the analyte of interest is added before sample preparation
- You are adding a standard to your actual sample
- This is used to evaluate analyte recovery in a sample matrix
 - Are there interferences in the sample water?

23

Duplicate

- Duplicate samples are analyzed randomly to assess precision on an ongoing basis
 - Precision = A measure of the degree of agreement among replicate analyses of a sample
 - How close is your duplicate to the sample?
- Not a part of the 12 Steps of QA, it's an addition from the State of TN (or in the approved method)



24

Initial and Continuing Calibration Verification

- Initial Calibration Verification (ICV) = perform initial calibration using at least 3 different standard concentrations
 - Calibrate the meter (DO, pH, or ISE) or verify balance, thermometer, and colorimeter/spectrophotometer
- Continuing Calibration Verification (CCV) = verify the calibration by analyzing one standard at the mid-point of the calibration range

25

Initial and Continuing Calibration Verification

- Example (pH):
 - ICV = Calibrate your pH probe/meter daily with fresh 4, 7, 10 buffers
 - CCV = Verify the calibration is still holding by reading a 7 buffer after every 10 samples or at the end of the sample run
 - Must be within +/- 0.1 pH



26

ICV – Balances/Scales

- Check instrument balances daily
 - Check balances routinely, preferably daily before use, with at least two working weights that bracket the normal usage range (e.g. ANSI/ASTM Class 1 or NIST Class S accompanied by appropriate certificate) for accuracy, precision and linearity.
 - Record results along with date and technicians initials
 - Recertify reference weights as specified in the certificate of calibration or at least every 5 years.



27

ICV – Temperature

- Check accuracy of all working temperature sensing devices (at the used temperature) against a certified National Institute of Standards and Technology (NIST) thermometer or one traceable to NIST and conforming to NIST specifications.
 - Keep records of calibration
 - Verify accuracy of the reference certified thermometer as specified on the certificate of calibration or at least every 5 years.
 - State of TN recommends annual verification.



28

Corrective Action

- If something goes wrong, 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;
 - R = rain event
 - D = bad dilution, etc.

29

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

30

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

31

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

32

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...

33

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

34

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 instructions for the tests that apply to your plant
 - Where you grab your samples

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Standard Operating Procedure (SOP)

Chlorine SOP

1. Insert cap
2. Invert bottle until water fills to High Range - (0.1 mg/L to 8.0 mg/L) - (0.1 mg/L to 8.0 mg/L)
3. Wait 30 seconds (if not on 0.00, Press Menu button, 0.00 will appear)
4. Fill sample cell (if not on 0.00, Press Menu button, 0.00 will appear)
5. Press Enter

Testing Procedure for Total Chlorine Residual High Range (0.1 to 8.0 mg/L Cl₂)
General Method - 18110-100

1. Fill a 100 mL plastic sample cell up to the fill mark (The Blank)
2. Place cap on the 100 mL plastic sample cell and place into the meter. (Downward mark facing the back of cell holder)
3. Press Enter
4. Fill another sample cell to the fill line with sample. Add the contents of 0.10 DPD tablet (chlorine residual powder pellets) to the sample cell and shake gently for 20 seconds. (Gently shaking dispenses bubbles.)

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Standard Operating Procedure



QA/QC for Chlorine
1000: Determination of quality. Each instrument is checked and zeroed before use. The instrument is checked for accuracy by comparing it to a known standard. The instrument is checked for accuracy by comparing it to a known standard. The instrument is checked for accuracy by comparing it to a known standard.

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

 1. The reference from which they are pulling these instructions
 2. The Method Number
 3. Equipment needed
 4. A brief summary
 5. Safety considerations
 6. Interferences – this is especially relevant for wastewater facilities who are running chlorine since color/turbidity will interfere

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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

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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



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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

40

Applicable tests for Drinking Water

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

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Remember!

- This is just a *very brief introduction* to QA/QC!
- Some of these procedures are very detailed (far more than what is shown here)
- The approved methods ,40 CFR 136, and your own lab will have more information
- Or, you can come back for one of our more in-depth lab classes! 😊

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Any questions?



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Standard Methods, SOPs, & QA/QC – Review Questions

1. What does CFR stand for?

2. List the CFR part numbers that contain approved test methods for both Drinking Water and Wastewater.

3. What does SOP stand for? What is an SOP?

4. List everything that should be included in the yearly review of your SOPs.

5. Record keeping can be conducted in pencil or pen. True or False

6. Why is it important to include reagent lot #s on your daily bench sheets?

7. Write out what the following abbreviations mean, as well as any alternate names:
 - a. DOC –
 - b. MDL –
 - c. LRB –
 - d. LFB –
 - e. LFM/LFMD –
 - f. Dup –
 - g. ICV –
 - h. CCV –

8. How often is data collected for the MDL? How often is the MDL validation required? How often is the MDL re-calculated?

9. What is the purpose of a duplicate?

NOTE: THIS IS FOR INSTRUCTIONAL PURPOSES ONLY. NOT AN OFFICIAL DOCUMENT. YOU MUST CREATE YOUR OWN SOP WITH SPECIFIC INFORMATION THAT PERTAINS TO YOUR FACILITY AND YOUR EQUIPMENT. THE FOLLOWING IS AN EXAMPLE ONLY.

STANDARD OPERATING PROCEDURE FOR
THE MEASUREMENT OF
pH

Prepared by:

Revision Date:

SOP-pH TABLE OF CONTENTS

Section 1:	Control Information, Computer Storage File.....	p
Section 2:	Personnel Qualifications.....	p
Section 3:	Summary of Method used.....	p
Section 4:	Materials and Equipment	p
Section 5:	Personal Protection Equipment.....	p
Section 6:	Daily pH Meter Calibration	p
Section 7:	Electrode Maintenance.....	p
Section 8:	Troubleshooting	p
Section 9:	Sample Handling	p
Section 10:	Sample pH Measurement.....	p
Section 11:	Data Management and Records Management.....	p
Section 12:	pH Quality Control	p
Section 13:	pH Calibration Log Sheet	p
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Section 1: Control Information

The SOP is stored as a Microsoft Word document in the SOP folder as file named SOPpH.doc.

Section 2: Personnel Qualifications

Technician should assist in the test for at least 1 week and should have performed a witnessed, unassisted pH performance sample (DOC) before initiating the procedure unsupervised. Each analyst must have run a known standard concentration at least 4 times and compared the limits listed in the method. (See QA/QC documentation.)

Section 3: Summary of Method Used

The method used is Standard Methods for the Examination of Water and Wastewater, 23rd edition, Section 4500-H+-B 2011. This method is applicable to drinking water and wastewater samples. The method consists of placing a glass pH electrode into a well-mixed sample and recording the display on a calibrated pH meter.

Section 4: Materials and Equipment

- Hach Q40d Meter
- Combination glass electrode
- 50 mL Beakers
- pH 4 buffer
- pH 7 buffer
- pH 10 buffer
- Kim wipes
- DI water

Section 5: Personal Protection Equipment

Latex gloves and safety goggles should be worn at all times. Personal hygiene should include hand washing with disinfectant upon completion.

Section 6: Daily pH meter Calibration

The pH meter is used to measure the hydrogen ion concentration of a sample. For routine work, the pH meter must be accurate and reproducible to the nearest 0.1 pH unit. Sample pH and standard buffers should be measured at the same temperature, generally room temperature. The pH meter is normally very stable and needs to be calibrated only once daily. (Generally within 3 hours of use). Calibrate more frequently if indicated.

pH Meter Calibration (ICV)

(Enter in the calibration instructions that apply to your specific probe/meter/instrument.)

Section 7: Electrode Maintenance

- The electrode must **NOT** be stored dry. The best solution to store the

electrode is saturated KCl or pH 7 buffer. Tap water or deionized water should not be used because the electrolyte within the electrode will diffuse out and cause premature aging or additional maintenance.

- Store the electrode in a solution of saturated KCl. Long term storage requires storage in a large test tube covered with parafilm.

Section 8 Troubleshooting

Meter:

- Disconnect the electrode and attach the shorting strap.
- Press the millivolt button. The display should show zero.
- If the display does not change, the meter needs repair.

Electrode:

- Gel electrodes generally require no maintenance if kept moist. However gel electrodes have a shelf life of around 2 years. Recording the date the electrode was placed in service helps with troubleshooting.
- Glass electrodes fail because of scratches, deterioration, and accumulation of debris on the glass bulb. Rejuvenate the electrodes by cleaning in 0.1 M HCl and 0.1 M NaOH.
- Corrective action: If the pH responds slowly or drifts after rejuvenation, replace the electrode.
- Reference electrodes fail because of clogged liquid junctions. The liquid junction must remain open to avoid slow response and meter drift.
- If the liquid junction becomes clogged, place in hot deionized water for several hours. Some combination electrodes can be refilled with saturated KCl.
- If the pH meter does not respond to different buffers (remains at pH 7 all the time), replace the electrode.

Section 9: Sample Handling

- Sample collection is the responsibility of the customer. Clean plastic Nalge bottles are provided to the customer in the size indicated below. Glass containers are also acceptable.
- Samples should be collected and analyzed within 15 minutes. No sample preservation is permitted. On site analysis is preferred.
- Minimum sample volume should be 50 ml.

SAFETY: Use latex gloves when collecting and handling all wastewater samples

Section 10: Sample pH Measurement

Section 11: Data Management and Records Management

- All records must be maintained in three ring binders and filed under the customer's name.
- Maintain all calibration records for a minimum of 5 years.

Section 12: Quality Control

Section 13: pH Calibration Log Sheet (Appendix 1)

State Regulations – Water Quality Reports and Publications

<https://www.tn.gov/environment/program-areas/wr-water-resources/water-quality/water-quality-reports---publications.html>

Wastewater:

1. Division of Water Resources Pollution Control - 0400-40-01
2. Design Criteria for Sewage Works
3. National Pollutant Discharge Elimination System (NPDES) Permit

Drinking Water:

1. Public Water Systems - Chapter 0400-45-01

All certifications:

1. Rules Governing Water and Wastewater Operator Certification – 0400-49-01

Be aware of:

2. Tennessee Code Annotated
<https://www.tn.gov/environment/permit-permits/water-permits1/water-and-wastewater-operator-certification.html>
 - TCA section 68-221-101 et seq: Water and Sewerage
 - TCA section 68-221-901 et seq: Water Environmental Health

Section 12

Chlorine

Chlorination

Introduction to Laboratory Methods for Operators



• 1

Disinfection

- Water is our single most important natural resource
 - Without water we could not exist
 - Need safe water to drink
- Water is the universal solvent and therefore, carries all types of dissolved materials
 - Including biological life
- Pathogens transmitted by water:
 - Cholera
 - Bacillary Dysentery
 - Gastroenteritis
 - Typhoid Fever
 - Cryptosporidiosis
 - Giardiasis
 - Infectious Hepatitis

• 2

Disinfection

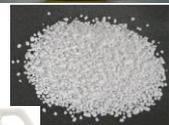
- **Disinfection** is the selective destruction or inactivation of pathogens (disease-causing organisms)
 - This is usually through the addition of chlorine
- Do not confuse this with **sterilization**, which is the destruction of *all* microorganisms



•

Agents of disinfection

- Chemical Disinfectants
 - Chlorine -- Cl_2
 - 100% pure
 - gas
 - Calcium hypochlorite -- $\text{Ca}(\text{OCl})_2$
 - 65% pure
 - solid
 - HTH – high test hypochlorite
 - Sodium hypochlorite -- NaOCl
 - 5-15% pure
 - Liquid
 - Bleach



• 4

Chlorine Gas

- Properties of Chlorine Gas:
 - Yellowish green in color
 - Slightly soluble in water
 - 2.5 times heavier than air
 - High coefficient of expansion (460)
 - one liter of liquid will expand to 460 liters of gas
 - Highly toxic



• 5

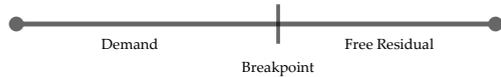
Chlorine Disinfection

- Exact mechanism is unknown, but the demonstrated effects are what matter
- When chlorine is added to water containing organic and inorganic material (reducing agents), it will combine with these materials and form chlorine compounds
 - i.e. chlororganics and chloramines
 - Some of these compounds have disinfecting properties, some do not

• 6

Breakpoint Chlorination

- The process of adding chlorine to water until the chlorine demand has been satisfied
 - Further additions of chlorine will result in a chlorine residual that is directly proportional to the amount of chlorine added beyond the breakpoint



- Total chlorine dose = residual + demand

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Breakpoint Chlorination

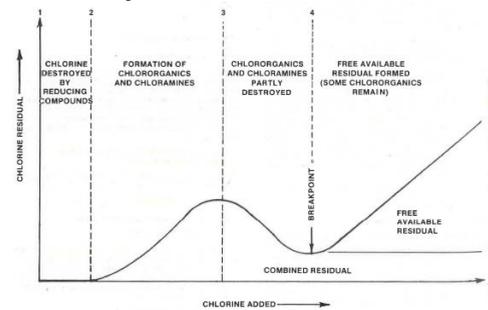


Fig. 7.2 Breakpoint chlorination curve

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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

$$\text{Demand} = \text{Dosage} - \text{Residual}$$

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Chlorine Disinfection

- Chlorine Residual** = the total of all the compounds with disinfecting properties plus any remaining free (uncombined) chlorine

$$\text{Residual} = \text{Dosage} - \text{Demand}$$

- Total Chlorine** = The total concentration of chlorine in water, including the combined chlorine and the free available chlorine

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Chlorine Disinfection

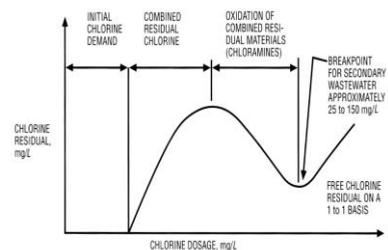
- Chlorine Dose** = the amount of chlorine needed to satisfy the chlorine demand and the amount of chlorine residual needed for disinfection

$$\text{Chlorine Dose} = \text{Demand} + \text{Residual}$$

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Breakpoint Chlorination

- Adding chlorine to water until the chlorine demand has been satisfied
- Free available residual chlorine begins to form at the breakpoint



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Breakpoint Chlorination

- <https://www.youtube.com/watch?v=Auz0cpObjl8>

• 13

Factors influencing disinfection

• • •

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Factors influencing Disinfection

- pH
 - Chlorine disinfects faster at pH of 7 than at pH > 8
 - Hypochlorous acid disassociates at a higher pH
- Temperature
 - Higher temperature means more efficient disinfection
 - Longer contact time required at lower temperatures
 - Chlorine will dissipate faster in warmer waters
- Microorganisms
 - Number and type greatly influence disinfection effectiveness
 - Cysts and viruses can be very resistant to disinfection

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Factors influencing Disinfection

- Turbidity
 - Excessive turbidity greatly reduces disinfection efficiency
 - Organic Matter
 - Organics can consume great amounts of disinfectants while forming unwanted compounds such as disinfection by-products
 - Reactions with organics and other reducing agents will significantly reduce the amount chemical available for disinfection
 - Inorganic matter
 - Ammonia can combine with disinfectant chemical to form side compounds

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Factors influencing disinfection

- Reducing Agents
 - Any substance that will readily donate electrons
 - Demand for chlorine by reducing agents must be met before chlorine becomes available to accomplish disinfection
 - Inorganic reducing agents
 - Hydrogen sulfide gas (H_2S)
 - Ferrous ion (Fe^{2+})
 - Manganous ion (Mn^{2+})
 - Ammonia (NH_3)
 - Nitrite ion (NO_2^-)

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Process of Disinfection

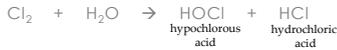
• • •

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Chlorine (Cl₂)

Lowers pH

- Reaction with Water



- Free chlorine combines with water to form hypochlorous acid

- Most effective disinfectant
- Dissociates at higher pH (greater than 7)



hypochlorous acid hypochlorite ion

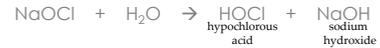
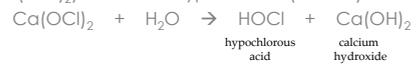
- Hypochlorous acid has a much higher disinfection potential than hypochlorite ion
- At pH = 7.5, of the chlorine present 50% will be HOCl and 50% will be OCl⁻

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Hypochlorite (OCl⁻)

- Reactions with Water

- May be applied in the form of calcium hypochlorite (Ca(OCl)₂) or sodium hypochlorite (NaOCl)



- Raises pH due to OH⁻ ion
- If is Ca(OCl)₂ injected at the same point of as sodium fluoride, a severe crust can form at injection point

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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

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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**

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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

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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

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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

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DPD Method

- Standard Method 4500-Cl G
- Grab sample, no preservative
- Analyze samples immediately (holding time is **15 minutes**)
 - After adding the reagent, a pink color will develop if chlorine is present
 - Wipe the outside of the sample cell with a wet then a dry towel to remove fingerprints
 - DPD - *N,N* Diethyl-1,4 Phenylenediamine Sulfate

•

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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)

•

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DPD Method - continued

- Interferences
 - Alkalinity > 300 mg/L as CaCO_3
 - 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

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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



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Amperometric Titration

- Apparatus
 - Amperometric Titrator (Wallace & Tiernan)
 - Buret with 0.01 mL increments
- Reagents
 - Phenylarsine oxide (PAO) titrant, 0.00564N
 - Potassium Iodide solution (KI solution)
 - Acetate Buffer solution

•

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Amperometric Titration - Procedure

- Fill burette with 0.0056N phenylarsine oxide solution (PAO)
- Measure 200 mL of sample into the cell and place in the holder on the titrator
- Add 1 mL Potassium Iodide (KI) solution (5% solution)
- Add 1 mL acetate buffer solution
- Turn on stirrer and adjust control knob until the meter reads the maximum on the scale

Amperometric Titration - Procedure

- Add phenylarsine oxide in 0.01 mL increments
 - This should cause the meter reading to deflect downward
 - Adjust the control knob as needed to keep the pointer on the scale
 - The end-point is reached when the addition of titrant no longer results in a downward deflection.
- Read the burette, subtracting the amount of the last addition (which did not cause a downward deflection)
 - The burette reading in mL equals the free chlorine residual in mg/L

Common Deficiencies

- Sampling and analyses times were not documented for field parameters
- Standards weren't analyzed to verify the accuracy of the chlorine meter
- Measuring free residual chlorine instead of total (and vice versa)
- Non-approved method being used to measure TRC
- TRC was being measured on the composite sample

Chlorine Problems

Oh no, not math problems!!



Chlorine Problems

- A chlorinator is set to feed 50 pounds of chlorine per 24 hours. The wastewater flow rate is 0.85 MGD. The chlorine measured after 30 minutes of contact time is 0.5 mg/L. Find the chlorine dosage and demand in mg/L.

$$dose, \frac{mg}{L} = \frac{\text{chlorine, lb/day}}{(Q, MGD)(8.34 \text{ lb/day})}$$

$$dose, \frac{mg}{L} = \frac{50 \text{ lbs/day}}{(0.85 \text{ MGD})(8.34 \text{ lbs/gal})}$$

$$dose, \frac{mg}{L} = 7.1 \text{ mg/L}$$

Chlorine Problems

- A chlorinator is set to feed 50 pounds of chlorine per 24 hours. The wastewater flow rate is 0.85 MGD. The chlorine measured after 30 minutes of contact time is 0.5 mg/L. Find the chlorine dosage and demand in mg/L.

$$\text{Demand, mg/L} = \text{Cl}_2 \text{ Dose, mg/L} - \text{Cl}_2 \text{ Residual, mg/L}$$

$$\text{Demand, mg/L} = 7.1 \text{ mg/L} - 0.5 \text{ mg/L}$$

$$\text{Demand, mg/L} = 6.6 \text{ mg/L}$$

Chlorine Problems

- The chlorine demand is determined to be **5 mg/L** and the plant flow rate is 8 MGD. How many pounds per day of gas chlorine should be fed? Include a **1 mg/L** residual.

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

$$\text{Cl}_2, \text{ lbs/day} = (6 \text{ mg/L})(8 \text{ MGD})(8.34 \text{ lbs/gal})$$

$$\text{Cl}_2, \text{ lbs/day} = 400 \text{ lbs/day}$$

Chlorine Problems

- The chlorine demand is determined to be 5 mg/L and the plant flow rate is 8 MGD. How many pounds per day of HTH (65% chlorine) should be fed? Include a 1 mg/L residual.

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

$$\text{Cl}_2, \text{ lbs/day} = \frac{(6 \text{ mg/L})(8 \text{ MGD})(8.34 \text{ lbs/gal})}{0.65}$$

$$\text{Cl}_2, \text{ lbs/day} = 616 \text{ lbs/day}$$

Chlorination – Review Questions

1. Water is the universal _____.
2. The destruction of all pathogenic microorganisms is called _____, which is not to be confused with _____, in which all microorganisms (pathogenic **and** nonpathogenic) are destroyed.
3. What is meant by “breakpoint chlorination?”
4. How do you determine the Chlorine Dose?
5. Explain why each of these factors that influence disinfection are important:
 - a. pH
 - b. Temperature
 - c. Microorganisms
 - d. Turbidity
 - e. Reducing agents
6. When chlorine is added to water, it breaks down into what two products? Which of these products is a more effective disinfectant?

7. Why are contact time and residual chlorine levels considered critical factors in the disinfection process?
8. Which of the following is not an approved method for chlorine analysis?
- Amperometric titration
 - DPD Colorimetric
 - DPD Color Comparator
 - DPD Titrimetric
 - Ion Specific Electrode
9. When analyzing Total Chlorine using the Hach procedure (method 8167), you can read the sample immediately (within 1 minute) of adding the DPD pillow. True or False
10. A water sample is tested and found to have a chlorine demand of 1.7 mg/L. If the desired chlorine residual is 0.9 mg/L, what is the desired chlorine dose (in mg/L)?
11. The chlorine dosage for water is 2.7 mg/L. If the chlorine residual after a 30 minute contact time is found to be 0.7 mg/L, what is the chlorine demand (in mg/L)?
12. What should the chlorinator setting be (in pounds per day) to treat a flow of 2.35 MGD if the chlorine demand is 3.2 mg/L and a chlorine residual of 0.9 mg/L is desired?

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 1 Instrument-specific information for reagent addition

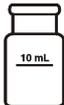
Instrument	Sample cell orientation	Sample cell
DR 6000 DR 3800 DR 2800 DR 2700 DR 1900	The fill line is to the right.	2495402 
DR 5000 DR 3900	The fill line is toward the user.	
DR 900	The orientation mark is toward the user.	2401906 

Table 2 Instrument-specific information for AccuVac Ampuls

Instrument	Adapter	Sample cell
DR 6000 DR 5000 DR 900	—	2427606 
DR 3900	LZV846 (A)	
DR 1900	9609900 or 9609800 (C)	
DR 3800 DR 2800 DR 2700	LZV584 (C)	2122800 

Before starting

Samples must be analyzed immediately after collection and cannot be preserved for later analysis.

Install the instrument cap on the DR 900 cell holder before ZERO or READ is pushed.

Do not use the same sample cells for free and total chlorine. If trace iodide from the total chlorine reagent is carried over into the free chlorine determination, monochloramine will interfere. It is best to use separate, dedicated sample cells for free and total chlorine measurements.

If the test result is over-range, or if the sample temporarily turns yellow after the reagent addition, dilute the sample with a known volume of high quality, chlorine demand-free water and do the test again. Some loss of chlorine may occur due to the dilution. Multiply the result by the dilution factor. Additional methods are available to measure chlorine without dilution.

For the best results, measure the reagent blank value for each new lot of reagent. Replace the sample with deionized water in the test procedure to determine the reagent blank value. Subtract the reagent blank value from the sample results automatically with the reagent blank adjust option.

An AccuVac Ampule for Blanks can be used to zero the instrument in the AccuVac test procedure.

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

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

The SwiftTest Dispenser for Free Chlorine can be used in place of the powder pillow in the test procedure.

Items to collect

Powder pillows

Description	Quantity
DPD Free Chlorine Reagent Powder Pillows, 10-mL	1
Sample cells. (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	2

Refer to [Consumables and replacement items](#) on page 6 for order information.

AccuVac Ampuls

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

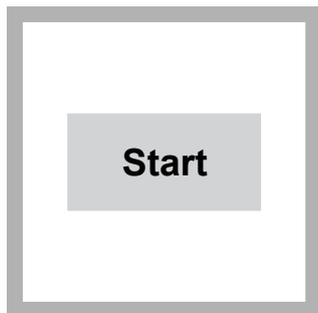
Refer to [Consumables and replacement items](#) on page 6 for order information.

Sample collection

- Analyze the samples immediately. The samples cannot be preserved for later analysis.
- Chlorine is a strong oxidizing agent and is unstable in natural waters. Chlorine reacts quickly with various inorganic compounds and more slowly with organic compounds. Many factors, including reactant concentrations, sunlight, pH, temperature and salinity influence the decomposition of chlorine in water.
- Collect samples in clean glass bottles. Do not use plastic containers because these can have a large chlorine demand.

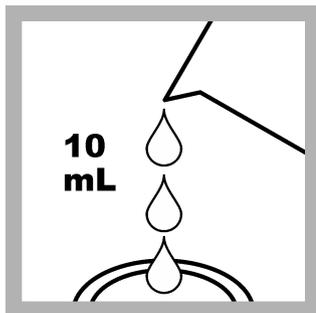
- Pretreat glass sample containers to remove chlorine demand. Soak the containers in a weak bleach solution (1 mL commercial bleach to 1 liter of deionized water) for at least 1 hour. Rinse fully with deionized or distilled water. If sample containers are rinsed fully with deionized or distilled water after use, only occasional pretreatment is necessary.
- Make sure to get a representative sample. If the sample is taken from a spigot or faucet, let the water flow for at least 5 minutes. Let the container overflow with the sample several times and then put the cap on the sample container so that there is no headspace (air) above the sample.

Powder pillow procedure

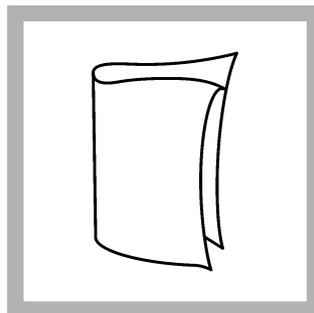


1. Start program **80 Chlorine F&T PP**. For information about sample cells, adapters or light shields, refer to [Instrument-specific information](#) on page 1.

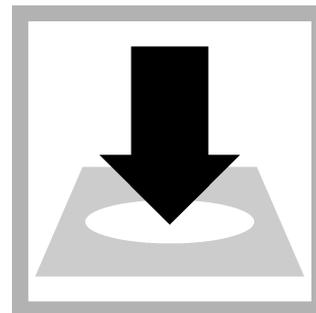
Note: Although the program name can be different between instruments, the program number does not change.



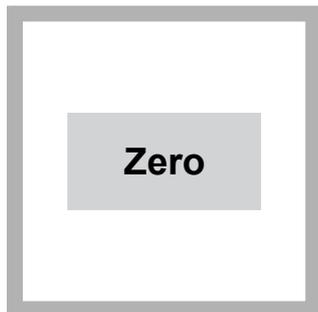
2. **Prepare the blank:** Fill the sample cell with 10 mL of sample.



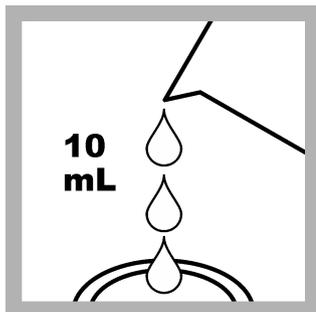
3. Clean the prepared sample cell.



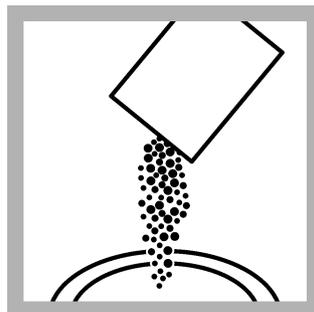
4. Insert the blank into the cell holder.



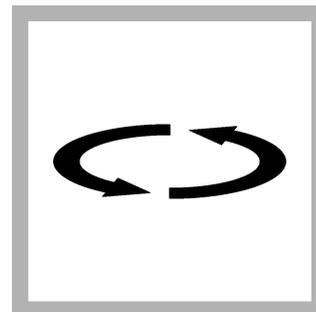
5. Push **ZERO**. The display shows 0.00 mg/L.



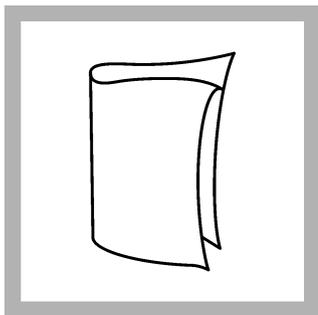
6. **Prepare the sample:** Fill a second sample cell with 10 mL of sample.



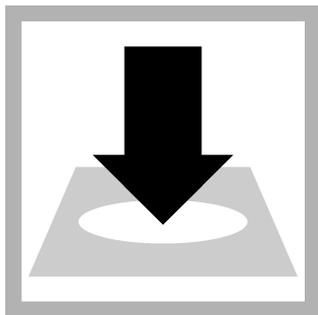
7. Add the contents of one powder pillow to the sample cell.



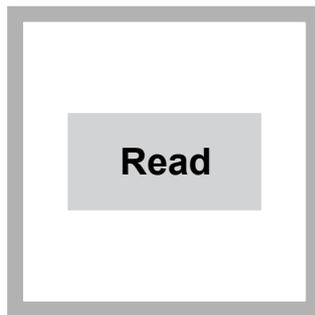
8. Swirl the sample cell for 20 seconds to mix. A pink color will develop if chlorine is present. Proceed to the next step immediately.



9. Clean the prepared sample cell.

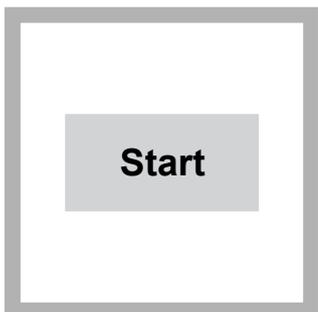


10. Within 60 seconds of the reagent addition, insert the prepared sample into the cell holder.



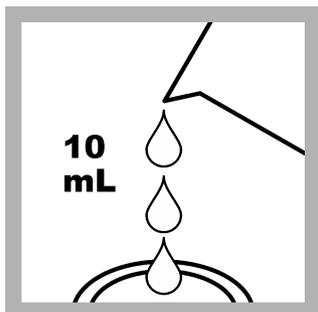
11. Push **READ**. Results show in mg/L Cl₂.

AccuVac Ampul procedure

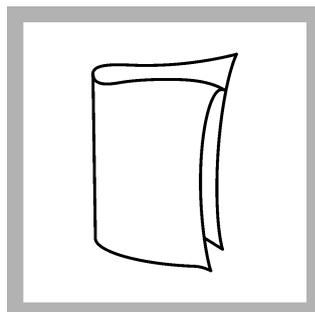


1. Start program **85 Chlorine F&T AV**. For information about sample cells, adapters or light shields, refer to [Instrument-specific information](#) on page 1.

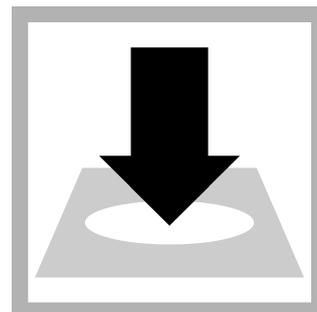
Note: Although the program name can be different between instruments, the program number does not change.



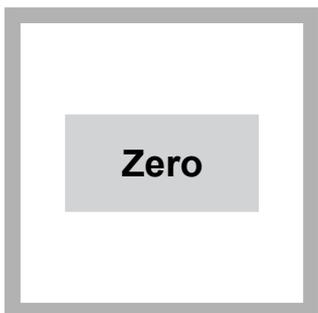
2. **Prepare the blank:** Fill the sample cell with 10 mL of sample.



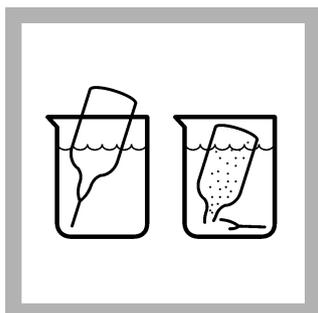
3. Clean the blank sample cell.



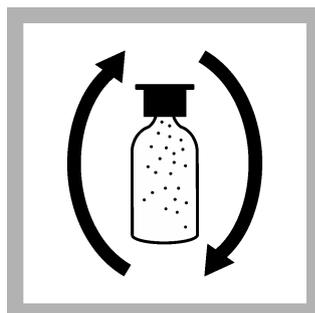
4. Insert the blank into the cell holder.



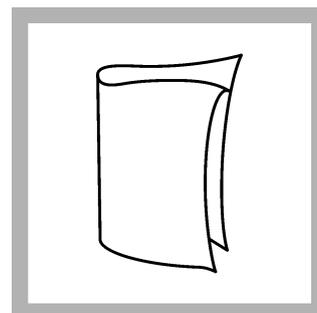
5. Push **ZERO**. The display shows 0.00 mg/L.



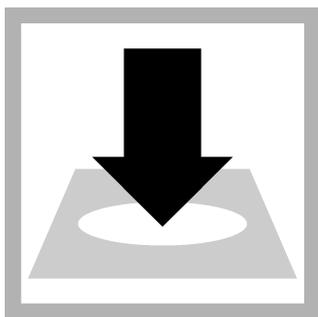
6. **Prepare the sample:** Collect at least 40 mL of sample in a 50-mL beaker. Fill the AccuVac Ampul with sample. Keep the tip immersed while the AccuVac Ampul fills completely.



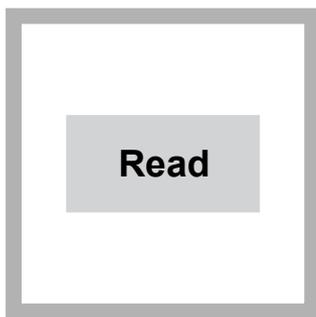
7. Quickly invert the AccuVac Ampul several times to mix.



8. Clean the AccuVac Ampul.



9. Within 60 seconds of the reagent addition, insert the prepared sample AccuVac Ampul into the cell holder.



10. Push **READ**. Results show in mg/L Cl₂.

Interferences

Interfering substance	Interference level
Acidity	More than 150 mg/L CaCO ₃ . The full color may not develop or the color may fade instantly. Adjust to pH 6–7 with 1 N Sodium Hydroxide. Measure the amount to add on a separate sample aliquot, then add the same amount to the sample that is tested. Correct the test result for the dilution from the volume addition.
Alkalinity	More than 250 mg/L CaCO ₃ . The full color may not develop or the color may fade instantly. Adjust to pH 6–7 with 1 N Sulfuric Acid. Measure the amount to add on a separate sample aliquot, then add the same amount to the sample that is tested. Correct the test result for the dilution from the volume addition.
Bromine, Br ₂	Positive interference at all levels
Chlorine Dioxide, ClO ₂	Positive interference at all levels
Inorganic chloramines	Positive interference at all levels
Chloramines, organic	May interfere
Hardness	No effect at less than 1000 mg/L as CaCO ₃
Manganese, Oxidized (Mn ⁴⁺ , Mn ⁷⁺) or Chromium, Oxidized (Cr ⁶⁺)	Pre-treat the sample as follows: <ol style="list-style-type: none"> 1. Adjust the sample pH to 6–7. 2. Add 3 drops of Potassium Iodide (30-g/L) to 10 mL of sample. 3. Mix and wait 1 minute. 4. Add 3 drops of Sodium Arsenite (5-g/L) and mix. 5. Use the test procedure to measure the concentration of the treated sample. 6. Subtract this result from the result without the treatment to obtain the correct chlorine concentration.
Monochloramine	Causes a gradual drift to higher readings. When read within 1 minute after reagent addition, 3 mg/L monochloramine causes less than a 0.1 mg/L increase in the reading.
Ozone	Positive interference at all levels
Peroxides	May interfere
Highly buffered samples or extreme sample pH	Can prevent the correct pH adjustment of the sample by the reagents. Sample pre-treatment may be necessary. Adjust to pH 6–7 with acid (Sulfuric Acid, 1.000 N) or base (Sodium Hydroxide, 1.00 N).

Accuracy check

Standard additions method (sample spike)

Use the standard additions method (for applicable instruments) to validate the test procedure, reagents and instrument and to find if there is an interference in the sample.

Items to collect:

- Chlorine Standard Solution, 2-mL PourRite® Ampule, 25–30 mg/L (use mg/L on label)
- Breaker, PourRite Ampules
- Pipet, TenSette®, 0.1–1.0 mL and tips

1. Use the test procedure to measure the concentration of the sample, then keep the (unspiked) sample in the instrument.
2. Go to the Standard Additions option in the instrument menu.
3. Select the values for standard concentration, sample volume and spike volumes.
4. Open the standard solution.
5. Prepare three spiked samples: use the TenSette pipet to add 0.1 mL, 0.2 mL and 0.3 mL of the standard solution, respectively, to three 10-mL portions of fresh sample. Mix well.

Note: For AccuVac® Ampuls, add 0.4 mL, 0.8 mL and 1.2 mL of the standard solution to three 50-mL portions of fresh sample.

6. Use the test procedure to measure the concentration of each of the spiked samples. Start with the smallest sample spike. Measure each of the spiked samples in the instrument.

7. Select **Graph** to compare the expected results to the actual results.

Note: If the actual results are significantly different from the expected results, make sure that the sample volumes and sample spikes are measured accurately. The sample volumes and sample spikes that are used should agree with the selections in the standard additions menu. If the results are not within acceptable limits, the sample may contain an interference.

Method performance

The method performance data that follows was derived from laboratory tests that were measured on a spectrophotometer during ideal test conditions. Users can get different results under different test conditions.

Program	Standard	Precision (95% Confidence Interval)	Sensitivity Concentration change per 0.010 Abs change
80	1.25 mg/L Cl ₂	1.23–1.27 mg/L Cl ₂	0.02 mg/L Cl ₂
85	1.25 mg/L Cl ₂	1.21–1.29 mg/L Cl ₂	0.02 mg/L Cl ₂

Summary of method

Chlorine in the sample as hypochlorous acid or hypochlorite ion (free chlorine or free available chlorine) immediately reacts with DPD (N,N-diethyl-p-phenylenediamine) indicator to form a pink color, the intensity of which is proportional to the chlorine concentration. The measurement wavelength is 530 nm for spectrophotometers or 520 nm for colorimeters.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Item no.
DPD Free Chlorine Reagent Powder Pillow, 10-mL	1	100/pkg	2105569
OR			
DPD Free Chlorine Reagent AccuVac® Ampul	1	25/pkg	2502025

Required apparatus

Description	Quantity/Test	Unit	Item no.
AccuVac Snapper	1	each	2405200
Beaker, 50-mL	1	each	50041H
Stoppers for 18-mm tubes and AccuVac Ampuls	2	6/pkg	173106

Recommended standards

Description	Unit	Item no.
Chlorine Standard Solution, 2-mL PourRite® Ampules, 25–30 mg/L	20/pkg	2630020

Optional reagents and apparatus

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



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Telephone: (970) 669-3050
FAX: (970) 669-2932

USEPA DPD Method¹
Method 8167
0.02 to 2.00 mg/L Cl₂
Powder Pillows or AccuVac[®] Ampuls

Scope and application: For testing residual chlorine and chloramines in water, wastewater, estuary water and seawater; USEPA-accepted for reporting for drinking and wastewater analyses.² This product has not been evaluated to test for chlorine and chloramines in medical applications in the United States.

¹ Adapted from Standard Methods for the Examination of Water and Wastewater.

² Procedure is equivalent to USEPA and Standard Method 4500-Cl G for drinking water and wastewater analysis.



Test preparation

Instrument-specific information

Table 1 shows sample cell and orientation requirements for reagent addition tests, such as powder pillow or bulk reagent tests. Table 2 shows sample cell and adapter requirements for AccuVac Ampul tests. The tables also show all of the instruments that have the program for this test.

To use the table, select an instrument, then read across to find the applicable information for this test.

Table 1 Instrument-specific information for reagent addition

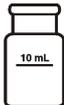
Instrument	Sample cell orientation	Sample cell
DR 6000 DR 3800 DR 2800 DR 2700 DR 1900	The fill line is to the right.	2495402 
DR 5000 DR 3900	The fill line is toward the user.	
DR 900	The orientation mark is toward the user.	2401906 

Table 2 Instrument-specific information for AccuVac Ampuls

Instrument	Adapter	Sample cell
DR 6000 DR 5000 DR 900	—	2427606 
DR 3900	LZV846 (A)	
DR 1900	9609900 or 9609800 (C)	
DR 3800 DR 2800 DR 2700	LZV584 (C)	2122800 

Before starting

Analyze the samples immediately. The samples cannot be preserved for later analysis.

Install the instrument cap on the DR 900 cell holder before ZERO or READ is pushed.

If the test result is over-range, or if the sample temporarily turns yellow after the reagent addition, dilute the sample with a known volume of high quality, chlorine demand-free water and do the test again. Some loss of chlorine may occur due to the dilution. Multiply the result by the dilution factor. Additional methods are available to measure chlorine without dilution.

For chloramination disinfection control, use one of the available Chloramine (Mono) methods.

For the best results, measure the reagent blank value for each new lot of reagent. Replace the sample with deionized water in the test procedure to determine the reagent blank value. Subtract the reagent blank value from the sample results automatically with the reagent blank adjust option.

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

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

The SwifTest Dispenser for Total Chlorine can be used in place of the powder pillow in the test procedure. One dispensation is equal to one powder pillow for 10-mL samples.

An AccuVac Ampul for Blanks can be used to zero the instrument in the AccuVac test procedure.

Items to collect

Powder pillows

Description	Quantity
DPD Total Chlorine Reagent Powder Pillow, 10-mL	1
Sample cells. (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	2

Refer to [Consumables and replacement items](#) on page 6 for order information.

AccuVac Ampuls

Description	Quantity
DPD Total Chlorine Reagent AccuVac [®] Ampul	1
Beaker, 50-mL	1
Sample cells (For information about sample cells, adapters or light shields, refer to Instrument-specific information on page 1.)	1
Stopper for 18-mm tubes and AccuVac Ampuls	1

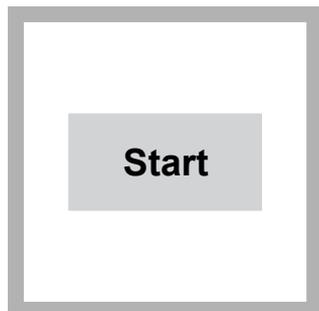
Refer to [Consumables and replacement items](#) on page 6 for order information.

Sample collection

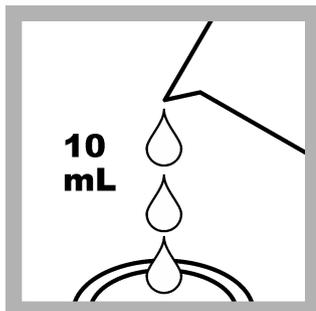
- Analyze the samples immediately. The samples cannot be preserved for later analysis.
- Chlorine is a strong oxidizing agent and is unstable in natural waters. Chlorine reacts quickly with various inorganic compounds and more slowly with organic compounds. Many factors, including reactant concentrations, sunlight, pH, temperature and salinity influence the decomposition of chlorine in water.
- Collect samples in clean glass bottles. Do not use plastic containers because these can have a large chlorine demand.

- Pretreat glass sample containers to remove chlorine demand. Soak the containers in a weak bleach solution (1 mL commercial bleach to 1 liter of deionized water) for at least 1 hour. Rinse fully with deionized or distilled water. If sample containers are rinsed fully with deionized or distilled water after use, only occasional pretreatment is necessary.
- Make sure to get a representative sample. If the sample is taken from a spigot or faucet, let the water flow for at least 5 minutes. Let the container overflow with the sample several times and then put the cap on the sample container so that there is no headspace (air) above the sample.

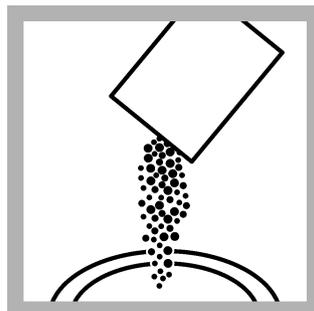
Powder pillow procedure



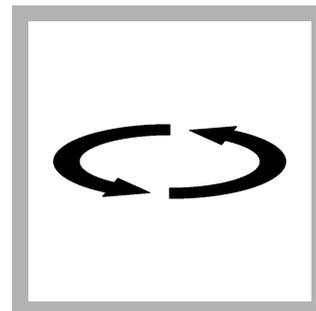
1. Start program **80 Chlorine F&T PP**. For information about sample cells, adapters or light shields, refer to [Instrument-specific information](#) on page 1.



2. Fill a sample cell with 10 mL of sample.



3. **Prepare the sample:** Add the contents of one powder pillow to the sample cell.

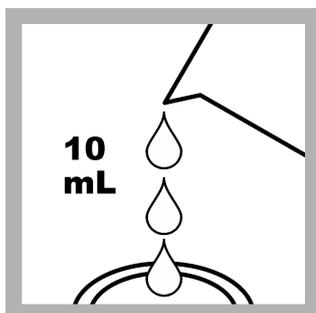


4. Swirl the sample cell for 20 seconds to mix. A pink color shows if chlorine is present in the sample.

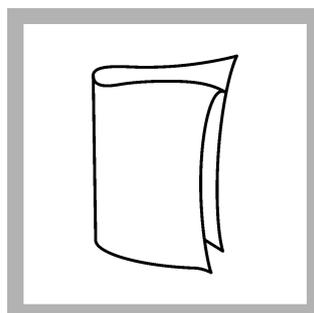


5. Start the instrument timer. A 3-minute reaction time starts.

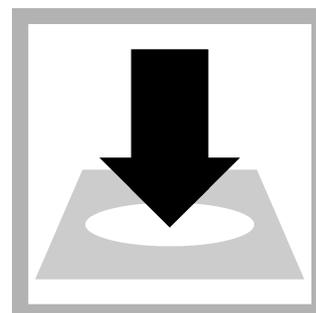
Prepare the sample blank and set the instrument to zero during the reaction time.



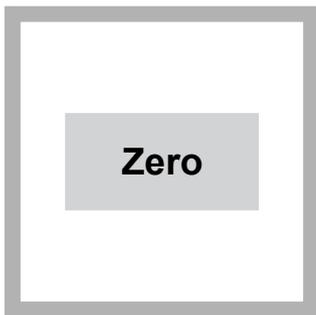
6. **Prepare the blank:** Fill a second sample cell with 10 mL of sample.



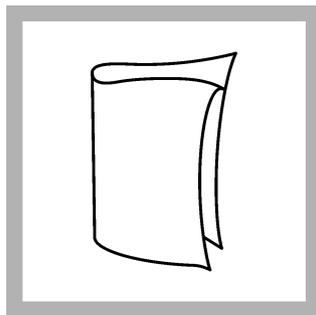
7. Clean the blank sample cell.



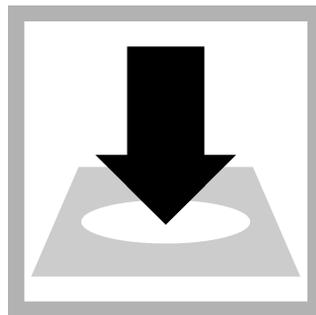
8. Insert the blank into the cell holder.



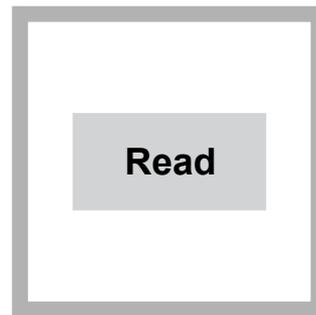
9. Push **ZERO**. The display shows 0.00 mg/L Cl₂.



10. Clean the prepared sample cell.

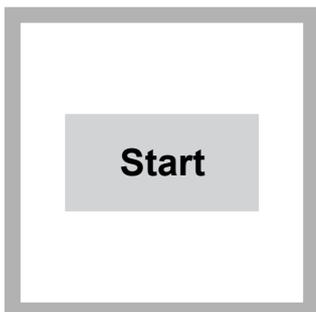


11. Within 3 minutes after the timer expires, insert the prepared sample into the cell holder.

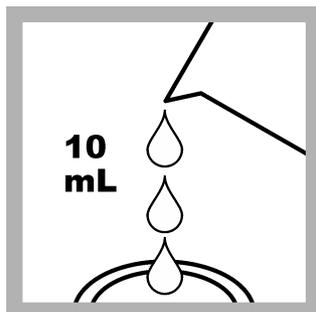


12. Push **READ**. Results show in mg/L Cl₂.

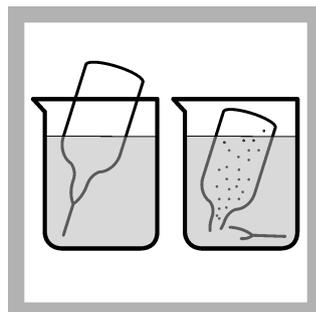
AccuVac Ampul procedure



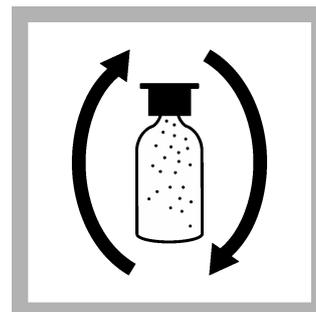
1. Start program **85 Chlorine F&T AV**. For information about sample cells, adapters or light shields, refer to [Instrument-specific information](#) on page 1.



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.

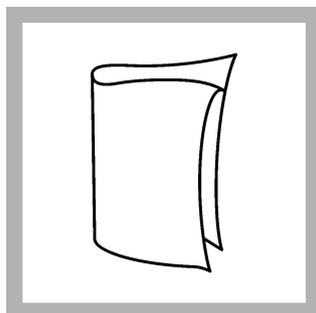


4. Quickly invert the AccuVac Ampul several times to mix.

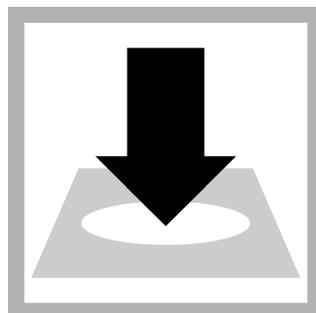


5. Start the instrument timer. A 3-minute reaction time starts.

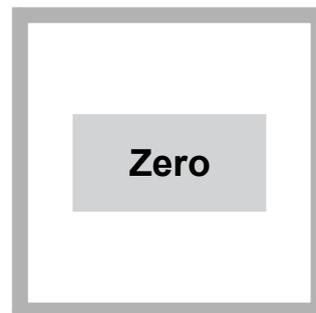
Prepare the sample blank and set the instrument to zero during the reaction time.



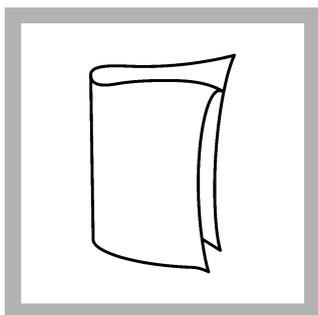
6. Clean the blank sample cell.



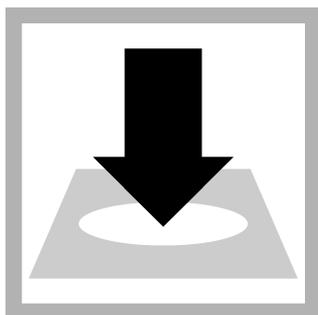
7. Insert the blank into the cell holder.



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

Interfering substance	Interference level
Acidity	More than 150 mg/L CaCO ₃ . The full color may not develop or the color may fade instantly. Adjust to pH 6–7 with 1 N Sodium Hydroxide. Measure the amount to add on a separate sample aliquot, then add the same amount to the sample that is tested. Correct the test result for the dilution from the volume addition.
Alkalinity	More than 250 mg/L CaCO ₃ . The full color may not develop or the color may fade instantly. Adjust to pH 6–7 with 1 N Sulfuric Acid. Measure the amount to add on a separate sample aliquot, then add the same amount to the sample that is tested. Correct the test result for the dilution from the volume addition.
Bromine, Br ₂	Positive interference at all levels
Chlorine Dioxide, ClO ₂	Positive interference at all levels
Inorganic chloramines	Positive interference at all levels
Chloramines, organic	May interfere in the result for total chlorine analysis
Hardness	No effect at less than 1000 mg/L as CaCO ₃
Manganese, Oxidized (Mn ⁴⁺ , Mn ⁷⁺) or Chromium, Oxidized (Cr ⁶⁺)	Pre-treat the sample as follows: <ol style="list-style-type: none"> 1. Adjust the sample pH to 6–7. 2. Add 3 drops of Potassium Iodide (30-g/L) to 10 mL of sample. 3. Mix and wait 1 minute. 4. Add 3 drops of Sodium Arsenite (5-g/L) and mix. 5. Use the test procedure to measure the concentration of the treated sample. 6. Subtract this result from the result without the treatment to obtain the correct chlorine concentration.
Ozone	Positive interference at all levels
Peroxides	May interfere
Highly buffered samples or extreme sample pH	Can prevent the correct pH adjustment (of the sample) by the reagents. Sample pretreatment may be necessary. Adjust to pH 6–7 with acid (Sulfuric Acid, 1.000 N) or base (Sodium Hydroxide, 1.00 N).

Accuracy check

Standard additions method (sample spike)

Use the standard additions method (for applicable instruments) to validate the test procedure, reagents and instrument and to find if there is an interference in the sample.

Items to collect:

- Chlorine Standard Solution, 2-mL PourRite® Ampule, 25–30 mg/L (use mg/L on label)
- Breaker, PourRite Ampules
- Pipet, TenSette®, 0.1–1.0 mL and tips

1. Use the test procedure to measure the concentration of the sample, then keep the (unspiked) sample in the instrument.
2. Go to the Standard Additions option in the instrument menu.
3. Select the values for standard concentration, sample volume and spike volumes.
4. Open the standard solution.
5. Prepare three spiked samples: use the TenSette pipet to add 0.1 mL, 0.2 mL and 0.3 mL of the standard solution, respectively, to three 10-mL portions of fresh sample. Mix well.

Note: For AccuVac® Ampuls, add 0.4 mL, 0.8 mL and 1.2 mL of the standard solution to three 50-mL portions of fresh sample.

6. Use the test procedure to measure the concentration of each of the spiked samples. Start with the smallest sample spike. Measure each of the spiked samples in the instrument.
7. Select **Graph** to compare the expected results to the actual results.

Note: If the actual results are significantly different from the expected results, make sure that the sample volumes and sample spikes are measured accurately. The sample volumes and sample spikes that are used should agree with the selections in the standard additions menu. If the results are not within acceptable limits, the sample may contain an interference.

Method performance

The method performance data that follows was derived from laboratory tests that were measured on a spectrophotometer during ideal test conditions. Users can get different results under different test conditions.

Program	Standard	Precision (95% Confidence Interval)	Sensitivity Concentration change per 0.010 Abs change
80	1.25 mg/L Cl ₂	1.23–1.27 mg/L Cl ₂	0.02 mg/L Cl ₂
85	1.25 mg/L Cl ₂	1.21–1.29 mg/L Cl ₂	0.02 mg/L Cl ₂

Summary of method

Chlorine can be present in water as free chlorine and as combined chlorine. Both forms can exist in the same water and be determined together as total chlorine. Free chlorine is present as hypochlorous acid and/or hypochlorite ion. Combined chlorine exists as monochloramine, dichloramine, nitrogen trichloride and other chloro derivatives. The combined chlorine oxidizes iodide in the reagent to iodine. The iodine and free chlorine react with DPD (N,N-diethyl-p-phenylenediamine) to form a pink color which is proportional to the total chlorine concentration.

To find the concentration of combined chlorine, run a free chlorine test and a total chlorine test. Subtract the results of the free chlorine test from the total chlorine test to obtain the combined chlorine concentration. The measurement wavelength is 530 nm for spectrophotometers or 520 nm for colorimeters.

Consumables and replacement items

Required reagents

Description	Quantity/Test	Unit	Item no.
DPD Total Chlorine Reagent Powder Pillow, 10 mL	1	100/pkg	2105669
OR			
DPD Total Chlorine Reagent AccuVac® Ampul	1	25/pkg	2503025

Required apparatus

Description	Quantity/Test	Unit	Item no.
AccuVac Snapper	1	each	2405200
Beaker, 50 mL	1	each	50041H
Stoppers for 18-mm tubes and AccuVac Ampuls	2	6/pkg	173106

Recommended standards

Description	Unit	Item no.
Chlorine Standard Solution, 10-mL Voluette [®] Ampule, 50–75 mg/L	16/pkg	1426810
Chlorine Standard Solution, 2-mL PourRite [®] Ampules, 50–75 mg/L	20/pkg	1426820
Chlorine Standard Solution, 2-mL PourRite [®] Ampules, 25–30 mg/L	20/pkg	2630020

Optional reagents and apparatus

Description	Unit	Item no.
AccuVac [®] Ampul vials for sample blanks	25/pkg	2677925
Ampule Breaker, 2-mL PourRite [®] Ampules	each	2484600
Ampule Breaker, 10-mL Voluette [®] Ampules	each	2196800
Water, Chlorine-demand Free	500 mL	2641549
Mixing cylinder, graduated, 25-mL	each	2088640
Mixing cylinder, graduated, 50 mL	each	189641
DPD Total Chlorine Reagent Powder Pillows, 10 mL	1000/pkg	2105628
DPD Total Chlorine Reagent Powder Pillows, 10 mL	300/pkg	2105603
DPD Total Chlorine Reagent, 10-mL, SwifTest [™] Dispenser refill vial	250 tests	2105660
Paper, pH, 0–14 pH range	100/pkg	2601300
Pipet, TenSette [®] , 0.1–1.0 mL	each	1970001
Pipet tips for TenSette [®] Pipet, 0.1–1.0 mL	50/pkg	2185696
Pipet tips for TenSette [®] Pipet, 0.1–1.0 mL	1000/pkg	2185628
Potassium Iodide, 30-g/L	100 mL	34332
Sodium Arsenite, 5-g/L	100 mL	104732
Sodium Hydroxide Standard Solution, 1.0 N	100 mL MDB	104532
SpecCheck [™] Secondary Standard Kit, Chlorine DPD, 0–2.0 mg/L Set	each	2635300
Sulfuric Acid Standard Solution, 1 N	100 mL MDB	127032
Water, deionized	4 L	27256



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In the U.S.A. – Call toll-free 800-227-4224
Outside the U.S.A. – Contact the HACH office or distributor serving you.
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HACH COMPANY
WORLD HEADQUARTERS
Telephone: (970) 669-3050
FAX: (970) 669-2932

Section 13

Alkalinity

ALKALINITY

Introduction to Laboratory Methods for Operators




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

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ALKALINITY

- Alkalinity caused by OH⁻ is called
 - *hydroxide alkalinity*
- Alkalinity caused by CO₃⁻ is called
 - *carbonate alkalinity*
- Alkalinity caused by HCO₃⁻ is called
 - *bicarbonate alkalinity*
- The combined effect of all three types is called
 - *total alkalinity*

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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

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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 (CaCO_3)
- Buret Titration Method, SM 2320 B

ALKALINITY DETERMINATION

- Measured by determining the amount of acid needed to drop the pH of a sample to a certain endpoint
 - Phenolphthalein alkalinity is measured by titrating to a pH of 8.3
 - Total alkalinity is measured by titrating to a pH of 4.5

ALKALINITY DETERMINATION

- Color Indicators:
 - Phenolphthalein
 - Metacresol purple
 - Bromcresol green
 - Mixed bromcresol green-methyl red
- } Titration to 8.3
 } Titration to 4.5

PH END POINTS (HACH METHOD 8221)

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

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.

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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.

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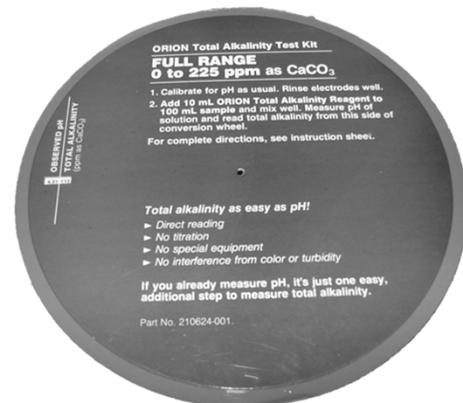
ORION PH PROBE

- 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.**



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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?