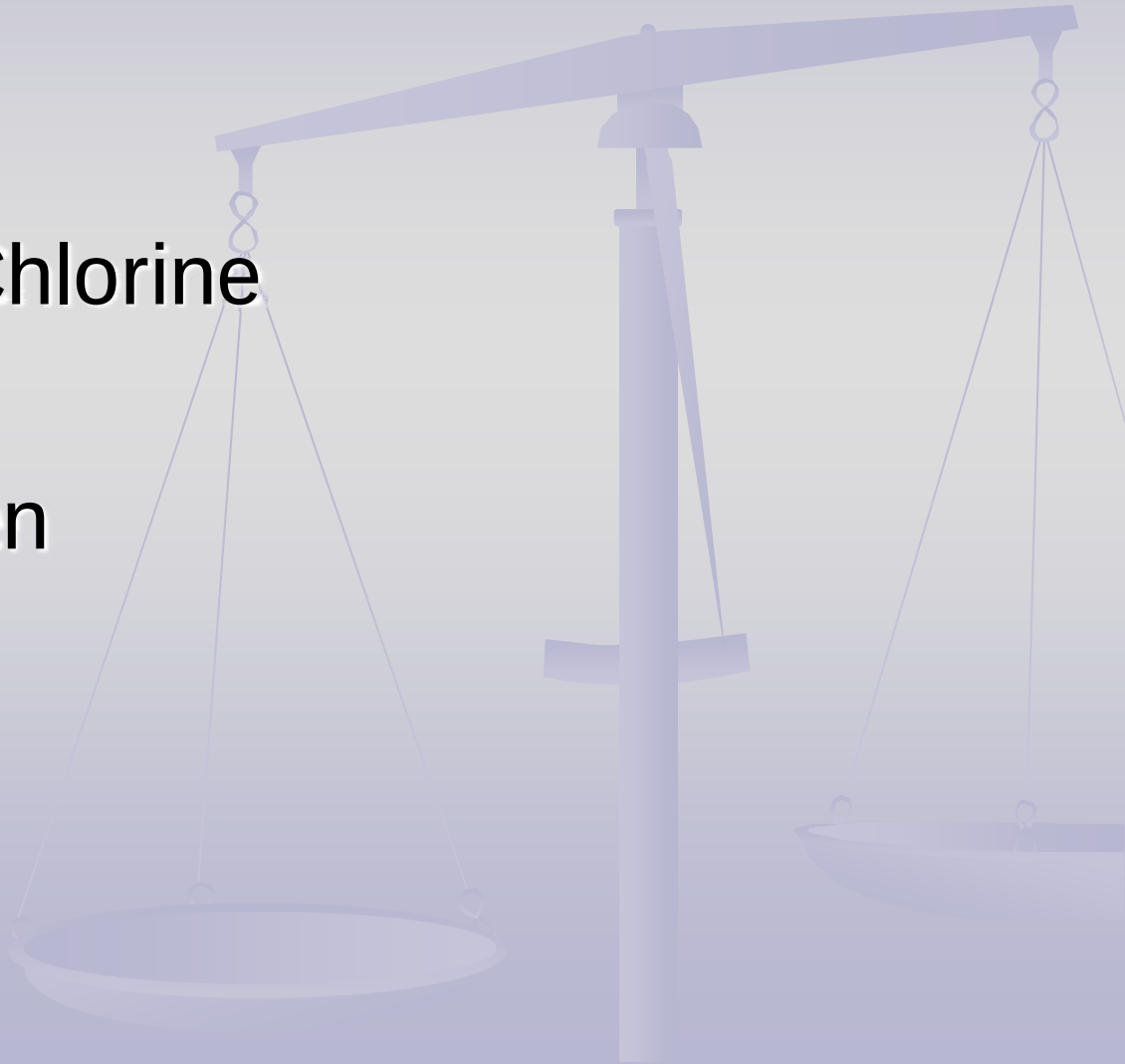
Wastewater Analyses

New Mexico Rural Water Association

2010

Field Tests

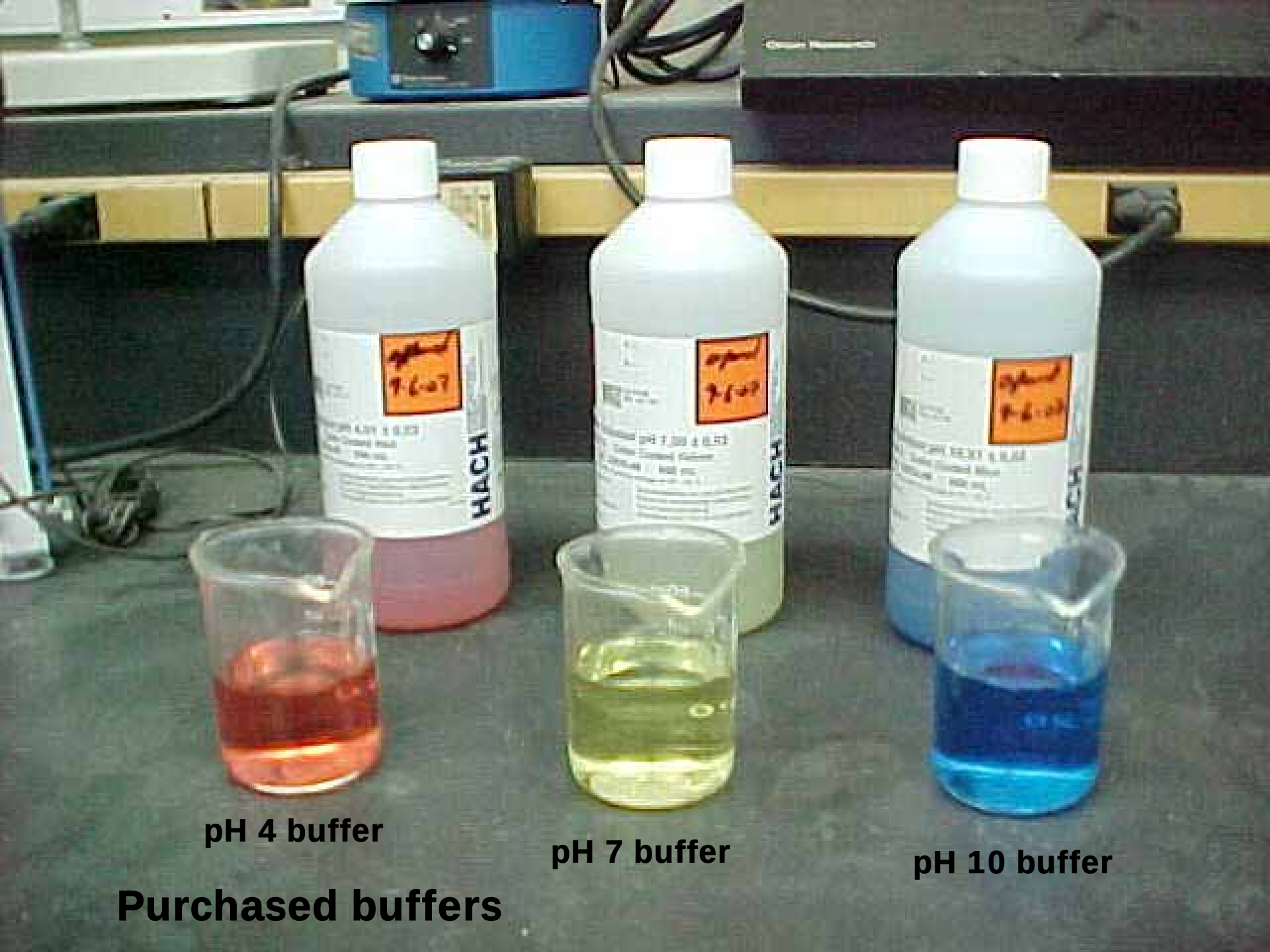
- pH
- Total Residual Chlorine
- Dissolved oxygen
- Temperature



How to Analyze for pH

- Calibrate meter ***daily*** when in use
- Use at least 2 buffers that bracket the expected pH of the sample (choose 7 and 4 or 10)
- Check calibration with the 3rd buffer
- Compensate for temperature differences (ATC)
- Document the calibration

- Collect the sample
- Analyze within 15 minutes
- Report answer to the nearest 0.1 pH units



pH 4 buffer

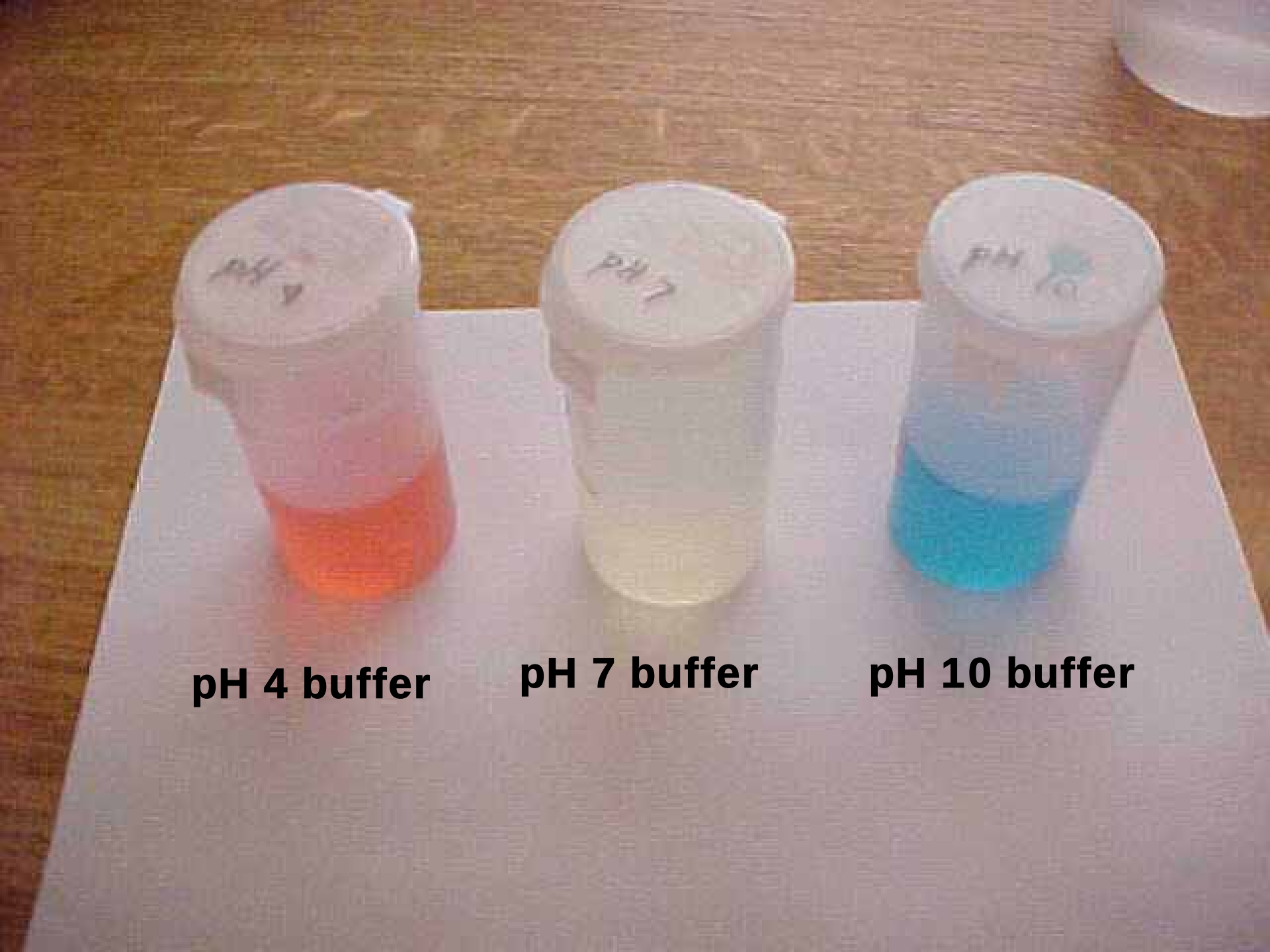
pH 7 buffer

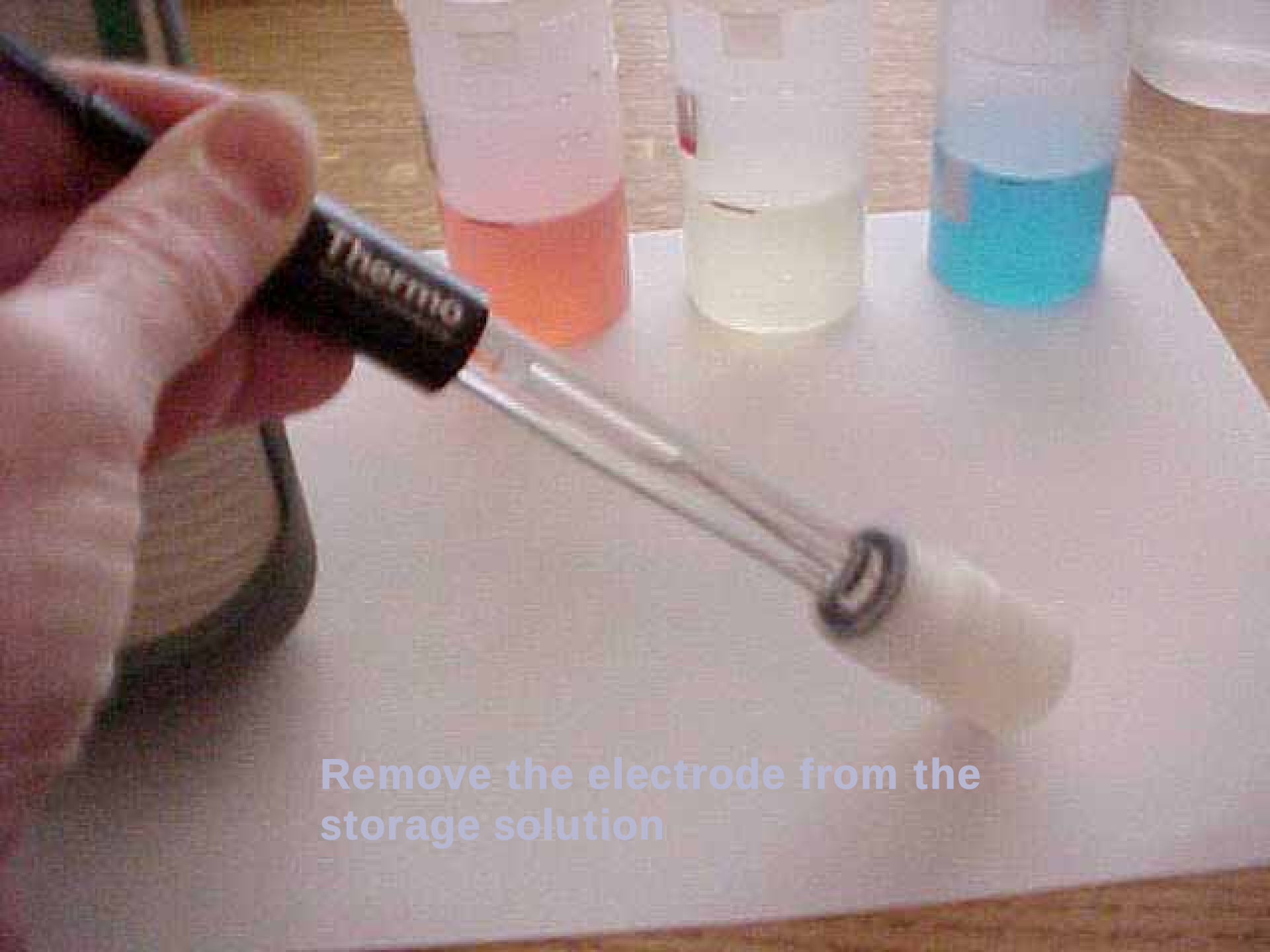
pH 10 buffer

Purchased buffers


Preparing pH 4
Buffer with powder
pillows…


...and distilled water
Distilled
H_2O
pH 4


pH 4 buffer
pH 7 buffer
pH 10 buffer



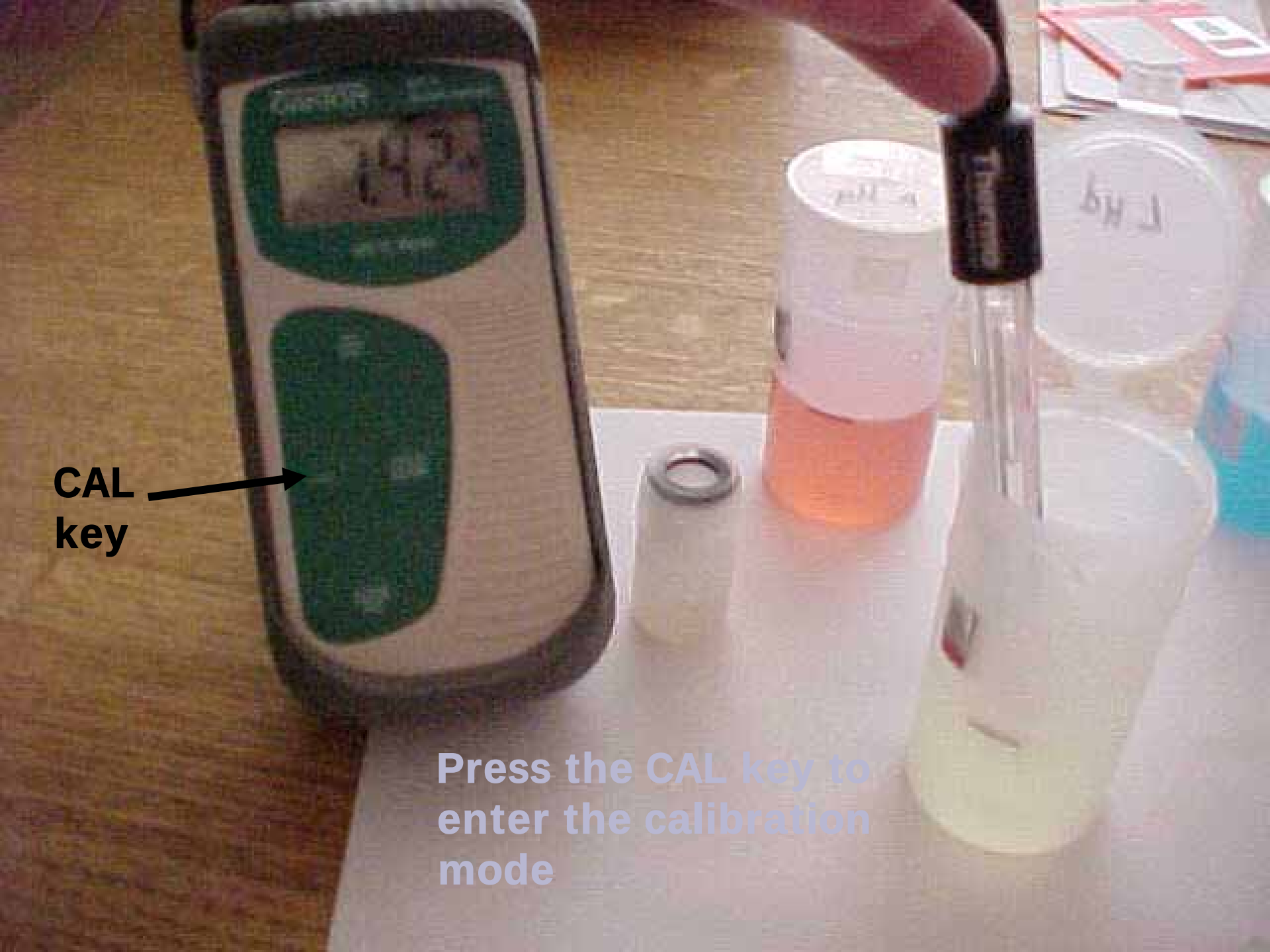
Remove the electrode from the storage solution


Turn the meter ON

Rinse the electrode


Blot the electrode dry
DO NOT wipe!

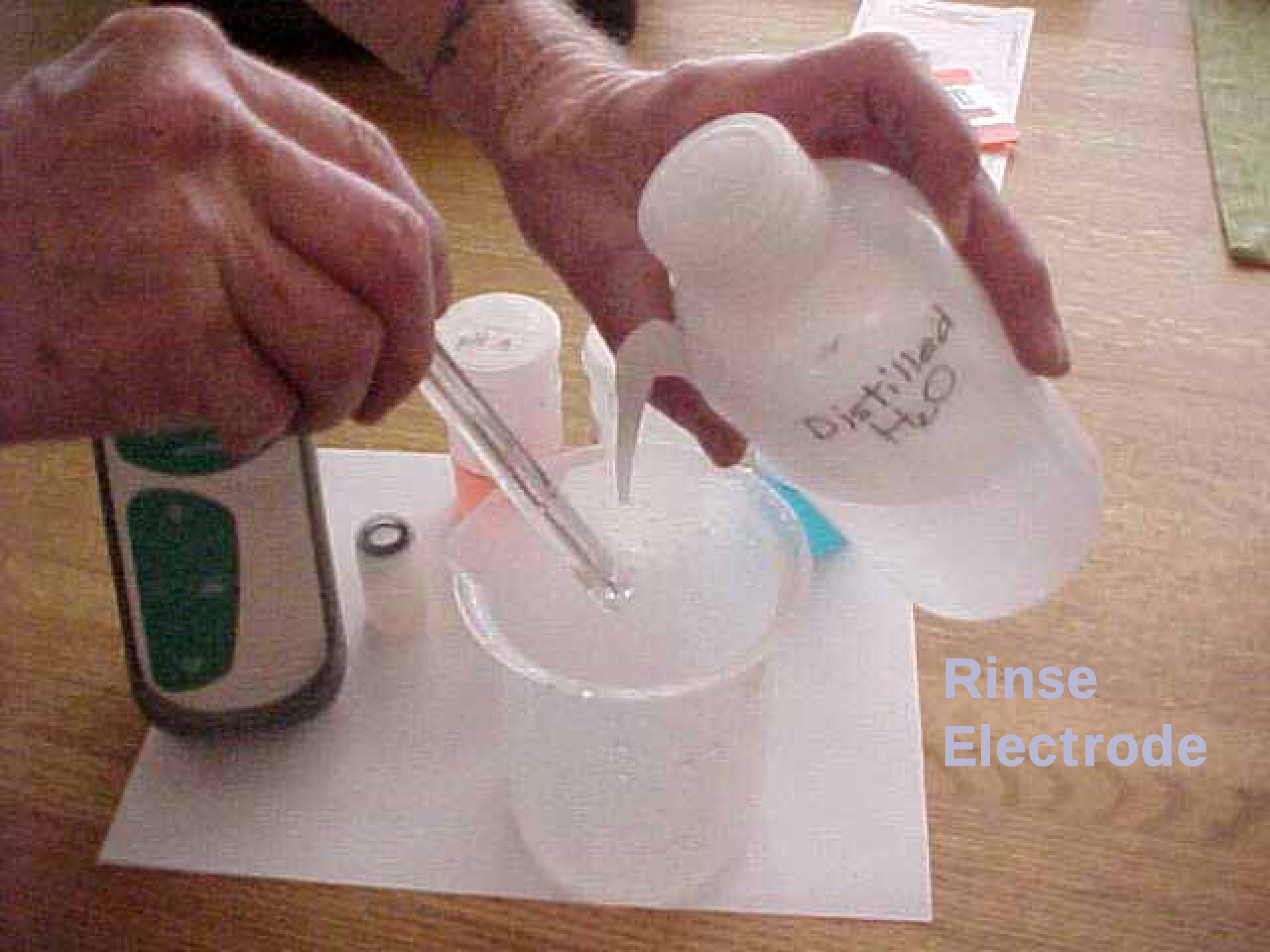

Dip electrode with ATC into pH 7 buffer.
Stir gently. Wait for reading to stabilize.



Press the CAL key to enter the calibration mode


ENTER
key
Allow reading to
Stabilize. Press the
ENTER key.


MODE
key
Press MODE key, read and record
buffer temperature

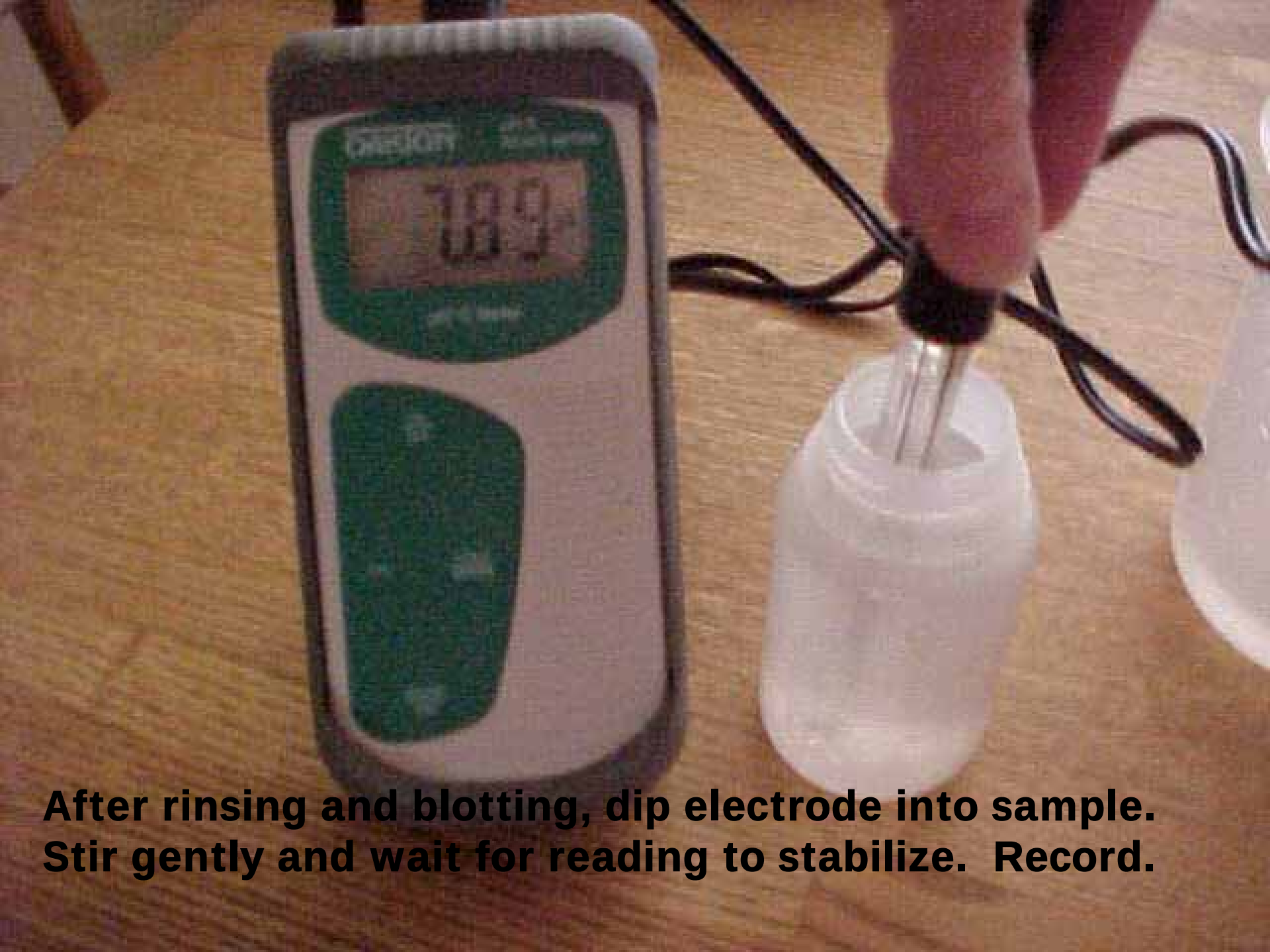


Rinse
Electrode


Blot electrode dry

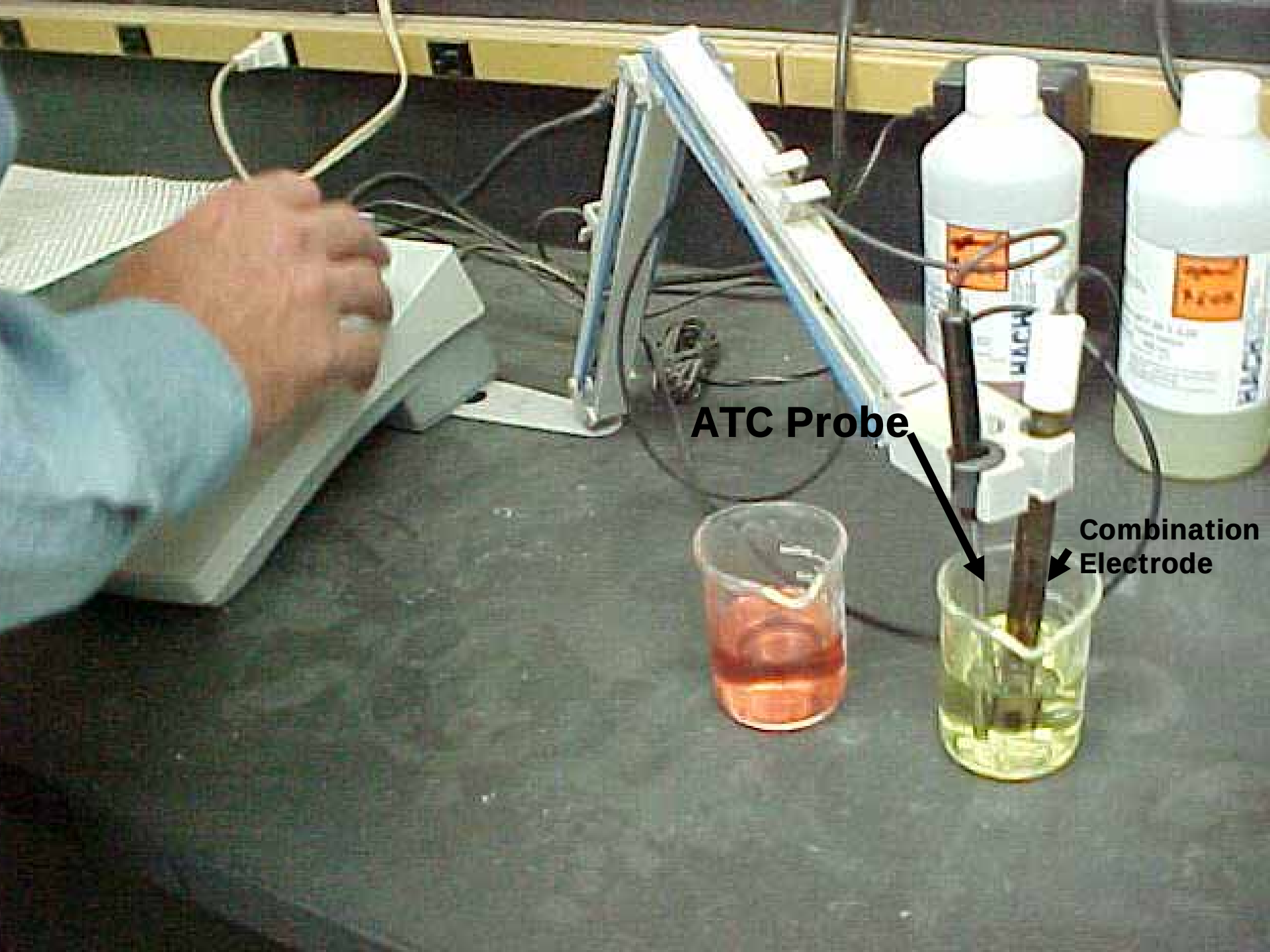

Repeat calibration
Procedure for buffers 4...


...and pH 10
Calibration is complete



After rinsing and blotting, dip electrode into sample. Stir gently and wait for reading to stabilize. Record.


Built-in ATC
Press MODE key, note temperature and record.


ATC Probe
Combination Electrode

pH Analysis Record

Date _____

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pH Equipment

Meter_____ Probe _____ [ ] ATC

pH Meter Calibration

Time _____ Analyst _____

STD # 1_____ Cal. Reading _____ Temp_____C
STD # 2_____ Cal. Reading _____ Temp_____C
STD # 3_____ Cal. Reading _____ Temp_____C

Slope _____

Measurement with 2nd Source 7 Buffer _____ Temp _____C

[ ] Buffers are prepared fresh daily when in use
[ ] Using one-use buffer packets or commercially prepared buffers

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Sample Collection

Time _____ Sampler _____

Exact Sampling Location _____

_____

Sample Analysis

Time _____Analyst_____

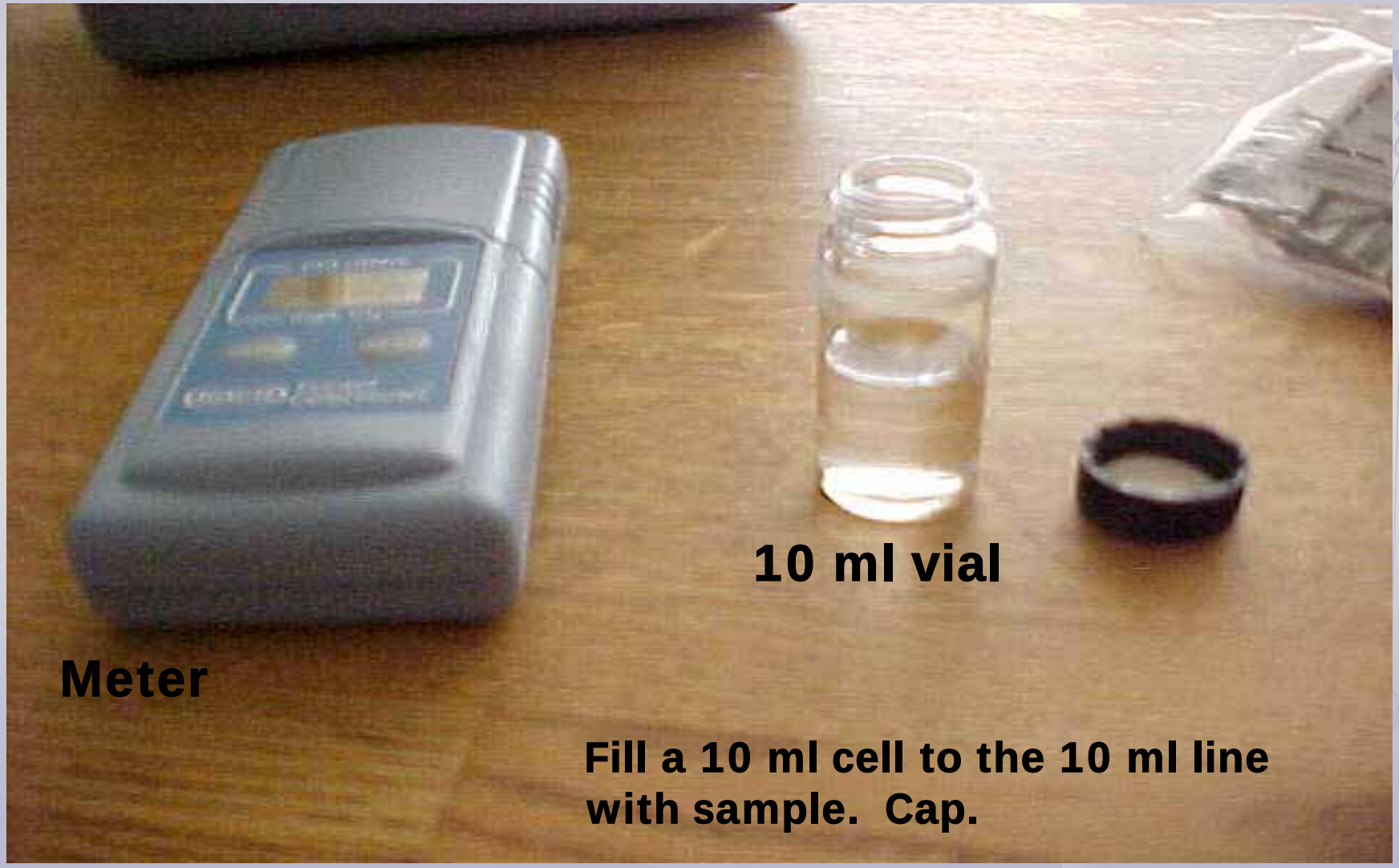
Sample pH _____ Temp _____C (Record to nearest 10th)

Duplicate pH _____ Temp _____C (for QC only)

Method used:

[ ] *Standard Methods*, 4500 H+ B.
[ ] ASTM D1293-84 (90),99 (A or B)
[ ] USGS I-1586-85
[ ] AOAC 973.41

Total Residual Chlorine
DPD Method




Remove the instrument cap
and place the blank (sample) in the
cell holder, with the diamond mark
facing you.
CHLORINE
mg/L Cl2
ZERO
READ
HACH

Press


Add the contents of one DPD Total Chlorine powder pillow to the sample cell.


Cap and gently shake for 20 seconds
Place vial in meter


Wait at least 3 minutes

Press READ

Record chlorine residual in mg/l

total chlorine

0.40 mg/l TRC

Total Residual Chlorine

Date_____ Sampler_____
Time_____

Low Range Total Chlorine (0-2.0 mg/l)

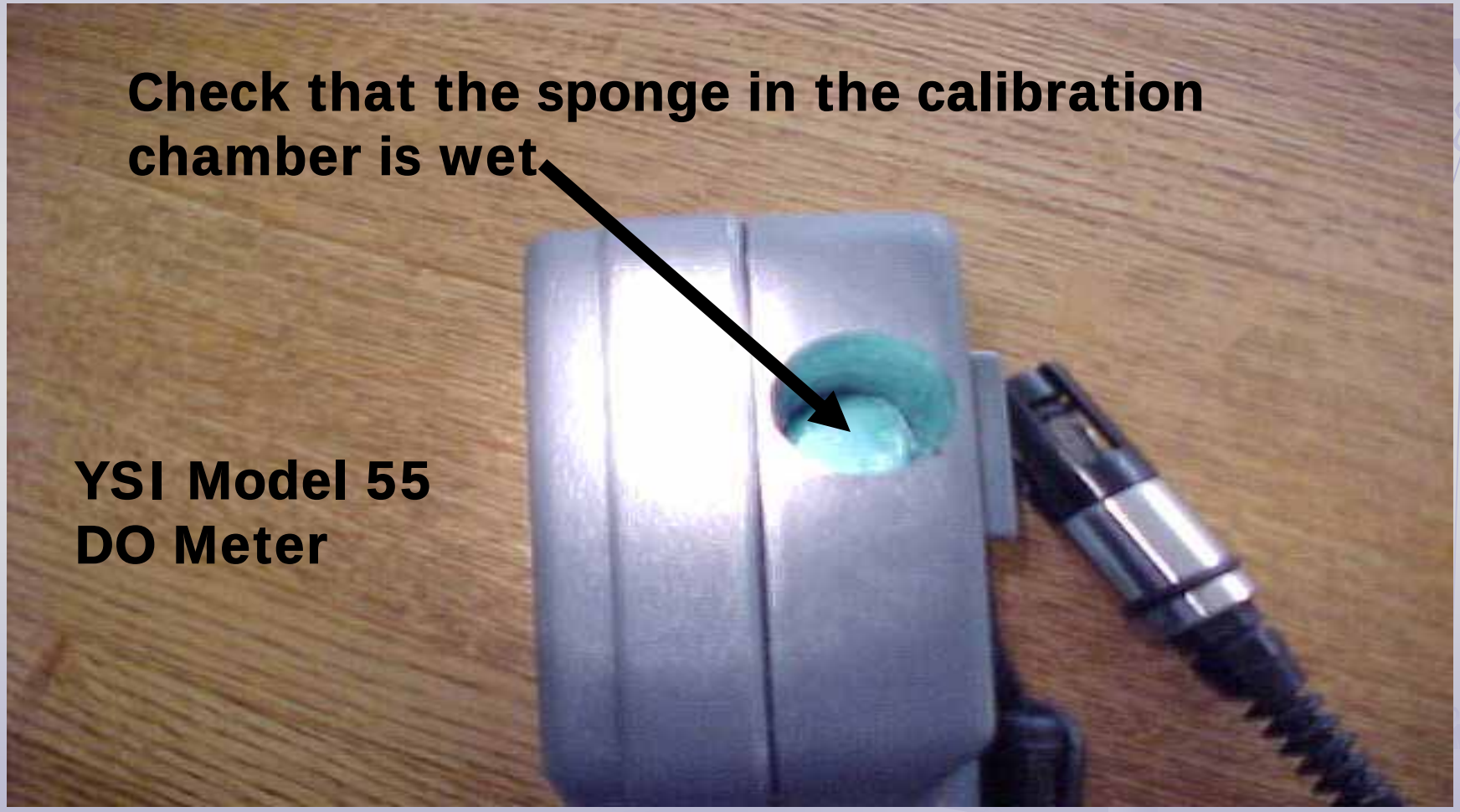
[ ] Zero/blank colorimeter using sample to be tested
[ ] Add 1 powder packet of DPD Total Residual reagent
[ ] Cap and shake for 20 seconds
[ ] Wait 3 minutes. Read.
[ ] Dilute overrange sample with distilled water
[ ] Repeat above procedure and multiply result by dilution factor

Analyst_____
Effluent (at V-notch)_____ Time _____

(Prior to Dechlor _____ Time _____)

Method used: Hach Method 8167 (which is equivalent to ~~USEPA Method 330.5~~) Standard Methods, Method_____

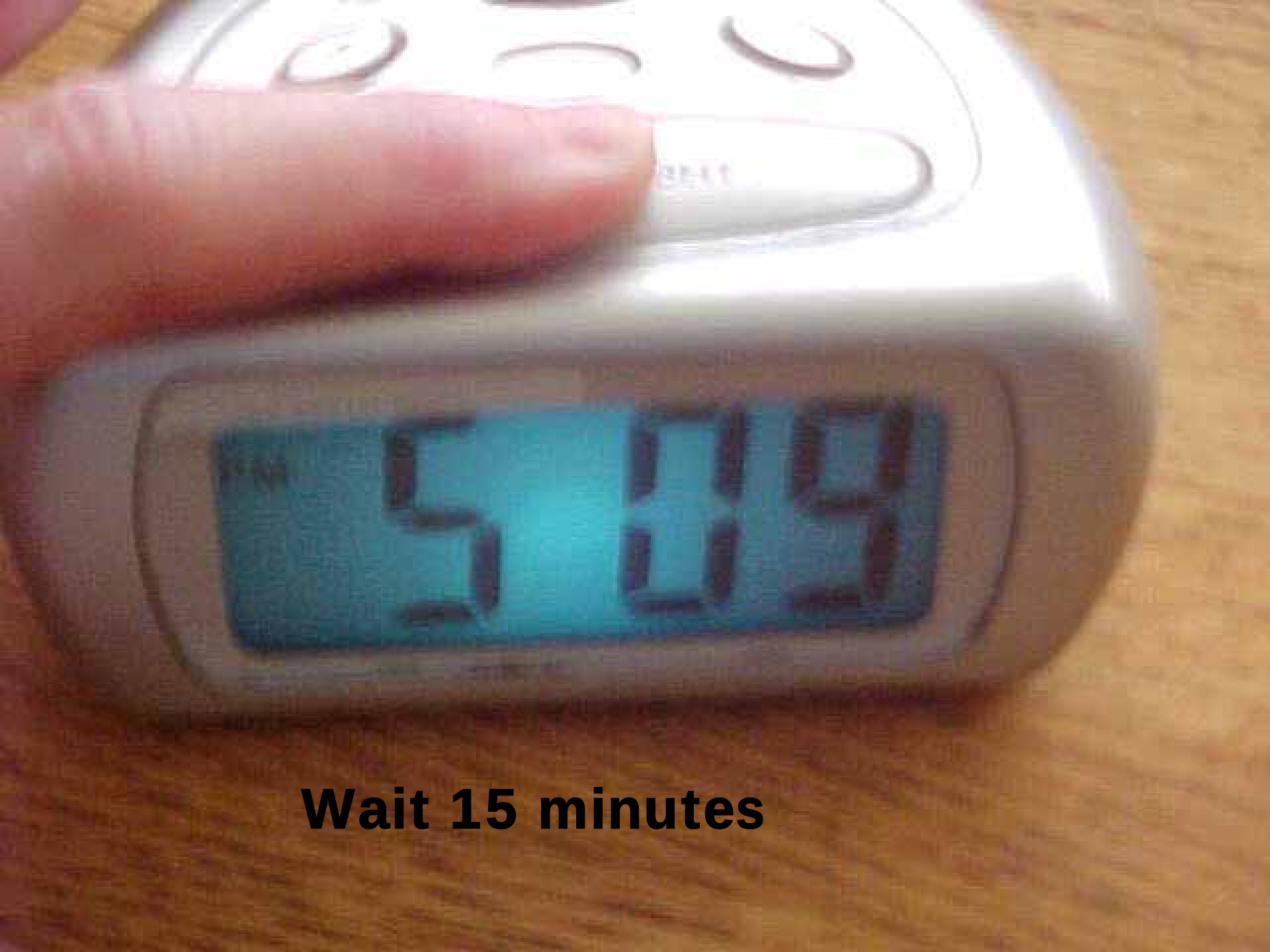
Dissolved Oxygen Analysis




Check that the probe membrane is wrinkle free with no air bubbles


Place the probe in the
calibration chamber


Turn the meter
ON
ON/OFF
LIGHT
MODE
ENTER



Wait 15 minutes

At the end of 15 minutes……


Press up and down at the same time....
6900 feet
ON/OFF
LIGHT
Verify altitude or enter new number

Press ENTER

Large display DO reading STABLE

Press ENTER

Verify salinity or enter new number

Press ENTER


55
DISSOLVED OXYGEN
YSI
ON/OFF
LIGHT
MODE

Stir probe in sample at 1 foot per second until reading stabilizes

Read DO in mg/l.....3.23 mg/l

- DO meter must be calibrated **DAILY** when in use
- Typical probe membrane life is 2 -4 weeks depending on use
- **ALWAYS** follow the manufacturer's instructions for calibration, care and use of your instrument

Temperature

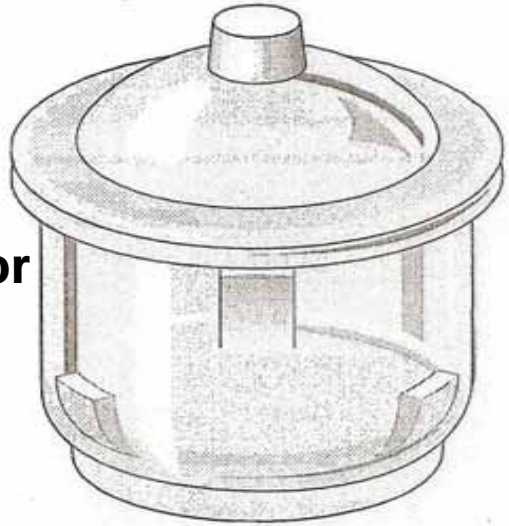
- To meet effluent permit limits, analysis must be ***in situ***
- Thermometer must be a good mercury model or an electronic thermistor which has been checked against a NIST or NIST-traceable thermometer annually and documented
- Thermometer must be immersed to the appropriate depth
- Thermometer must have a scale appropriate to the intended use

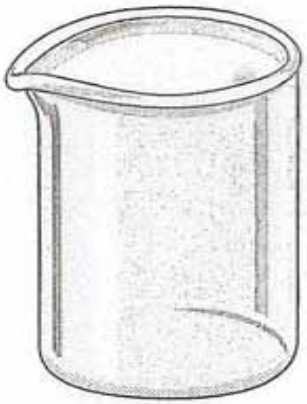
Wastewater Laboratory

- Equipment overview
- Lab Safety
- Common test procedures
- QA/QC
- Recordkeeping

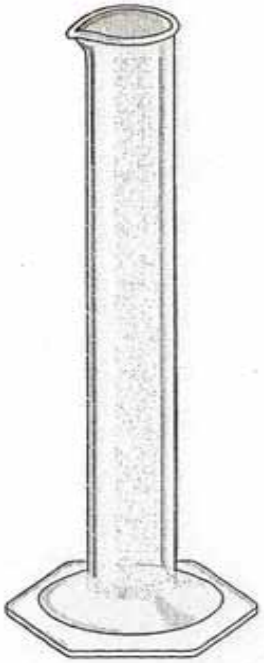
Equipment Overview

Desiccator





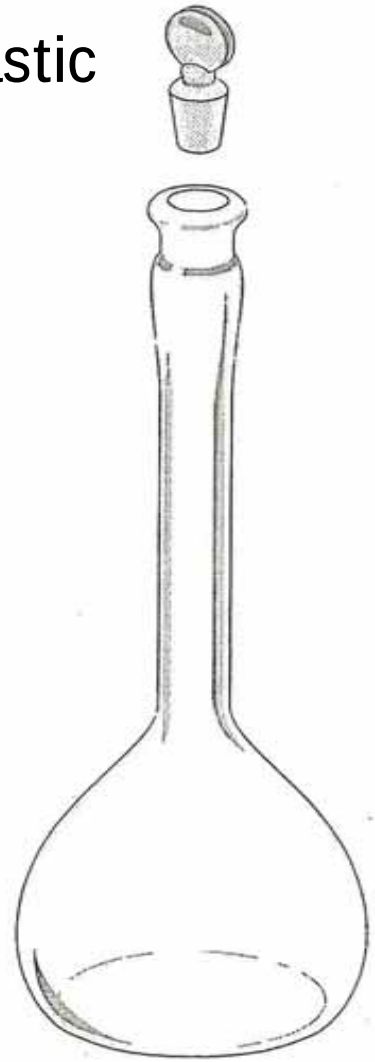
Beaker



Graduated Cylinder

BOD Bottle 300 mls

Borosilicate
Glass
Or Plastic

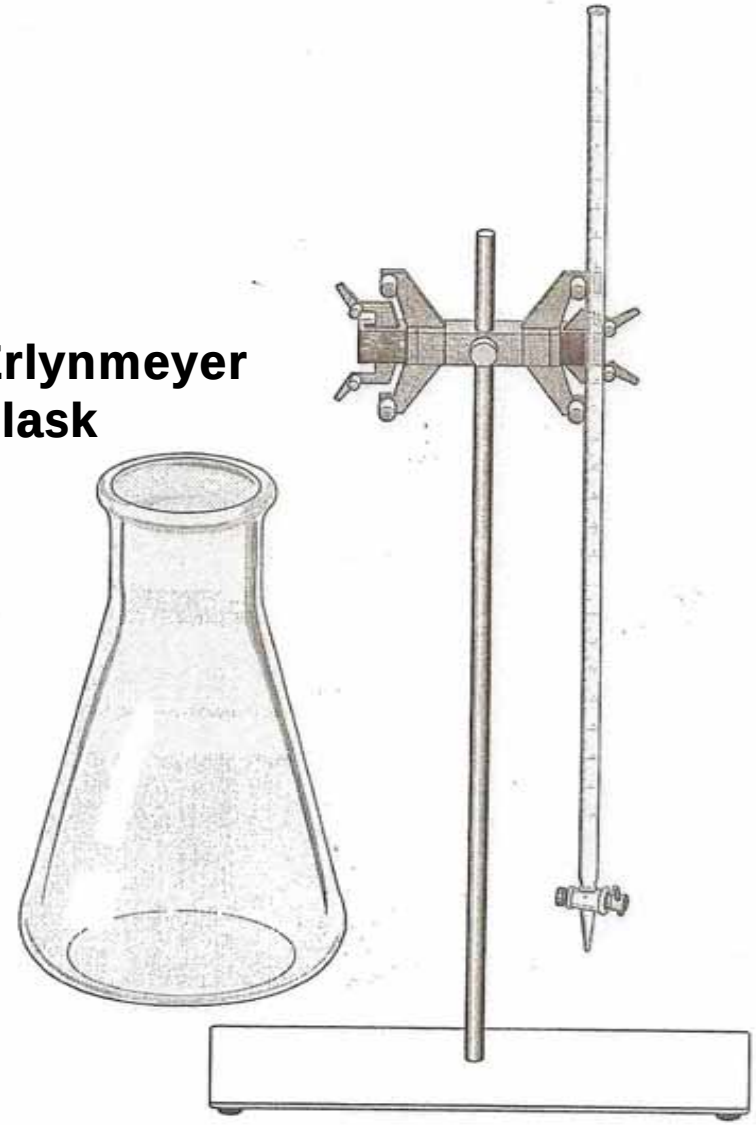


Volumetric Flask

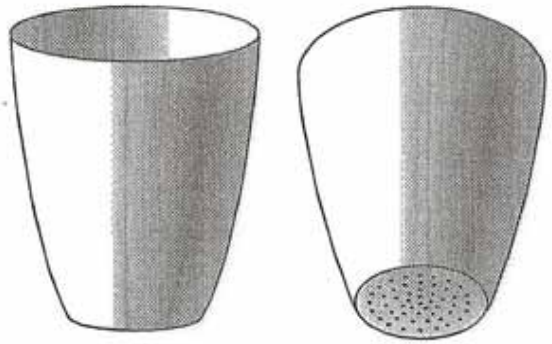
Pipette

Titrator or Buret

Erlynmeyer Flask

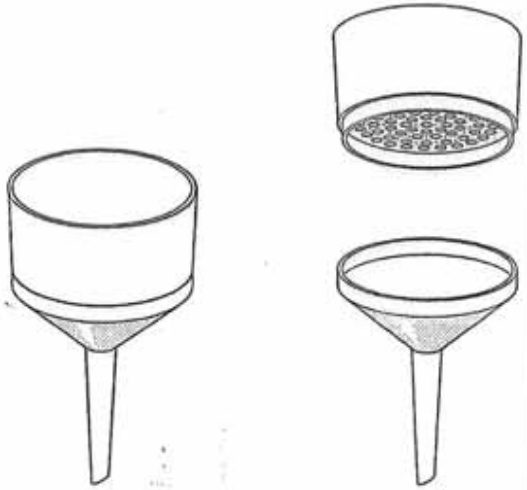


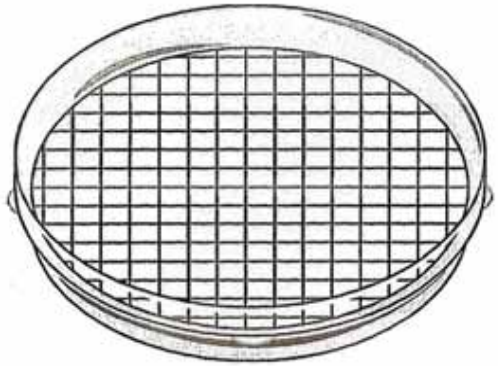
Filter Apparatus for TSS, Fecals and *E. coli*



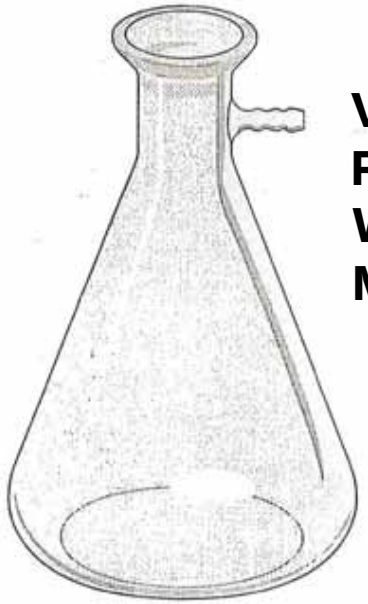
Gooch Crucibles for TSS

Buchner funnel

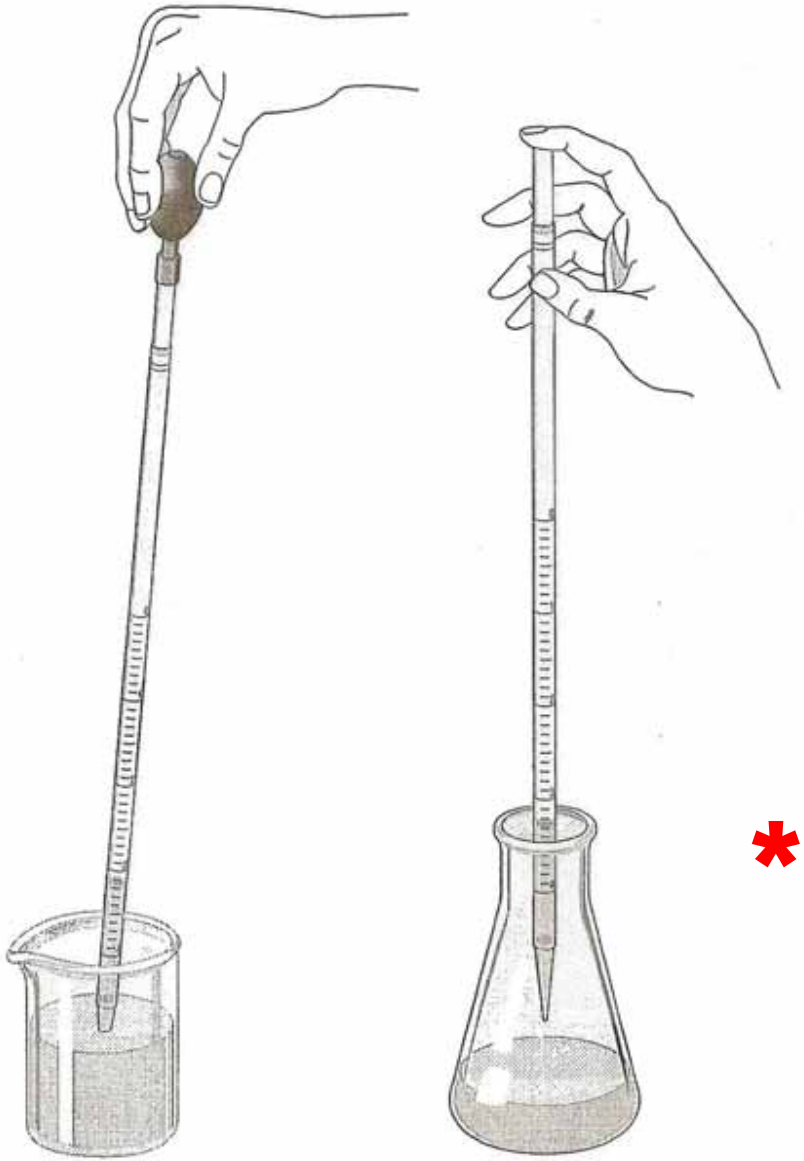


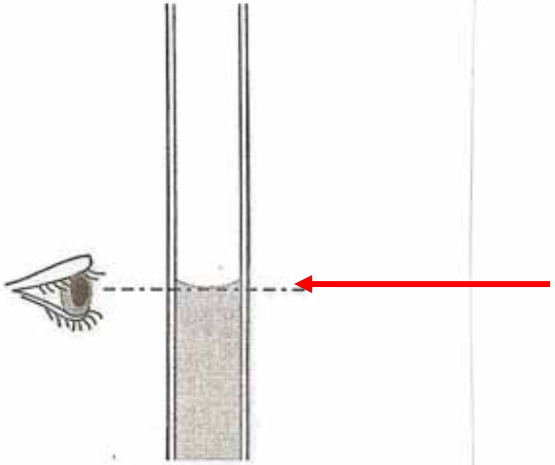


Petri dish



Vacuum Flask –used With TSS & MF tests





* Read the meniscus
  At the bottom of the concave curve

* **Pipetting**
  A process of transferring small precise volumes of liquids

Weights & Measures

Weighing

- For high precision, use an analytical balance
- For less accuracy, use a pan balance or triple beam balance
- All equipment should be kept in good working order and checked for accuracy regularly, once a year is recommended

Choosing & Using Equipment

1. First understand the level of accuracy required for the analysis
2. Know which equipment will deliver the desired accuracy
3. Master the proper techniques and procedures associated with the equipment

Lab Safety

- Use proper equipment
- When connecting glassware to rubber hoses, use water as a lubricant
- Check Material Safety Data Sheets (MSDS)
  - Chemical handling
  - Spill clean-up
- Use personal protective equipment
- Properly ground all electrical equipment

- Store combustible chemicals in fire approved cabinets
- Store acids and bases in separate approved cabinets
- Store light sensitive chemicals in closed cabinets (usually found in brown jars)
- NEVER add water to an acid
- ALWAYS add acid to water **A → W**
- Don’t eat, drink or smoke in a lab
- **Wash hands often**

Common Lab Safety Equipment

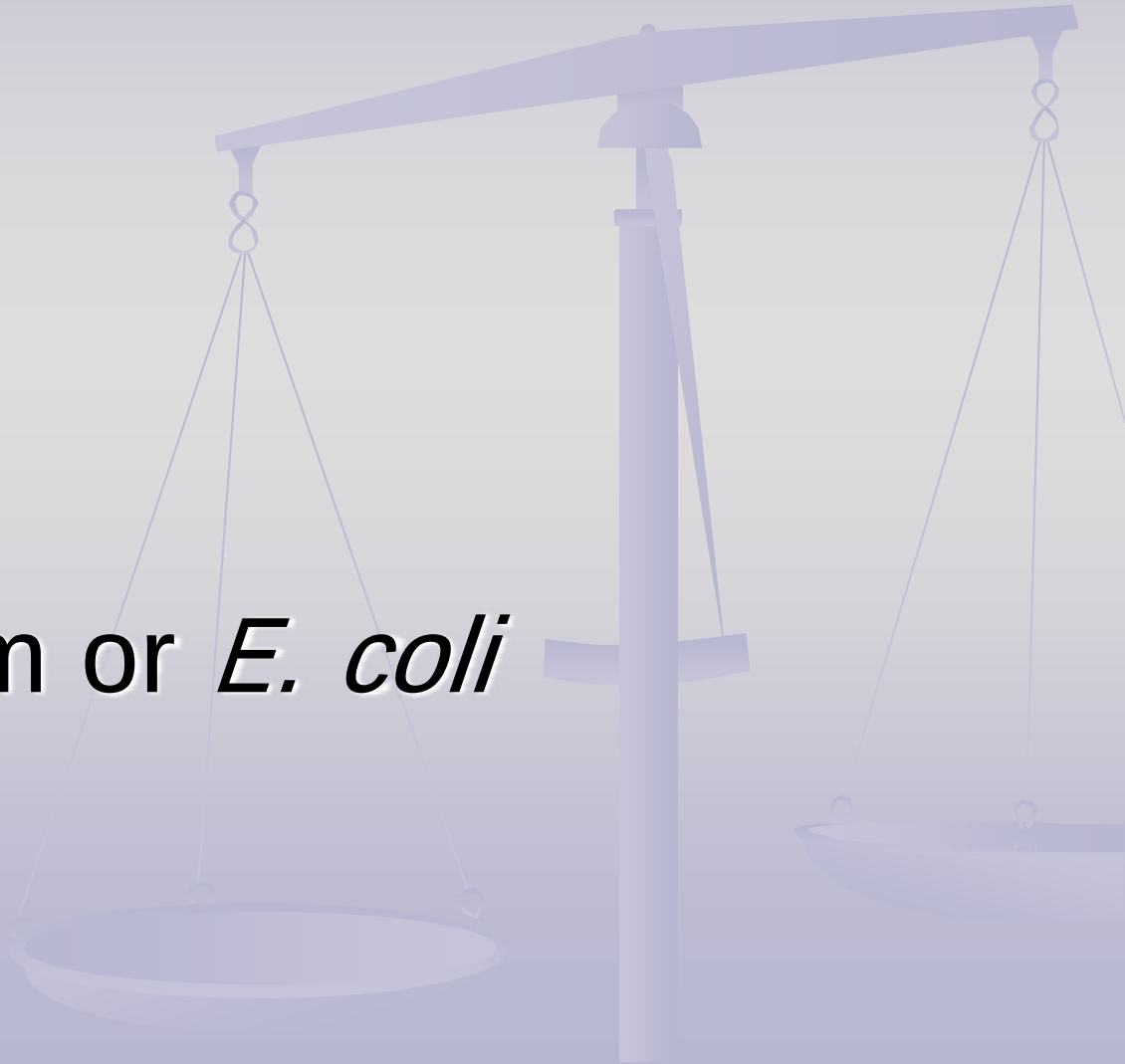
- Fume hood
- Emergency shower
- Eyewash
- Fire extinquishers
- First aid kit
- Hazardous materials storage cabinets
- Spill kits

Personal Protective Equipment

- Safety glasses
- Goggles
- Face shield
- Gloves
- Lab coat

Common Laboratory Analyses

- BOD
- TSS
- Fecal coliform or *E. coli*



Review

1 Person Yields 0.2 lbs. BOD/day

- **BOD** stands for **Biochemical Oxygen Demand**
- Lab test used to measure the **strength** or **organic load** on the treatment plant and on the receiving stream

Initial DO

Final DO

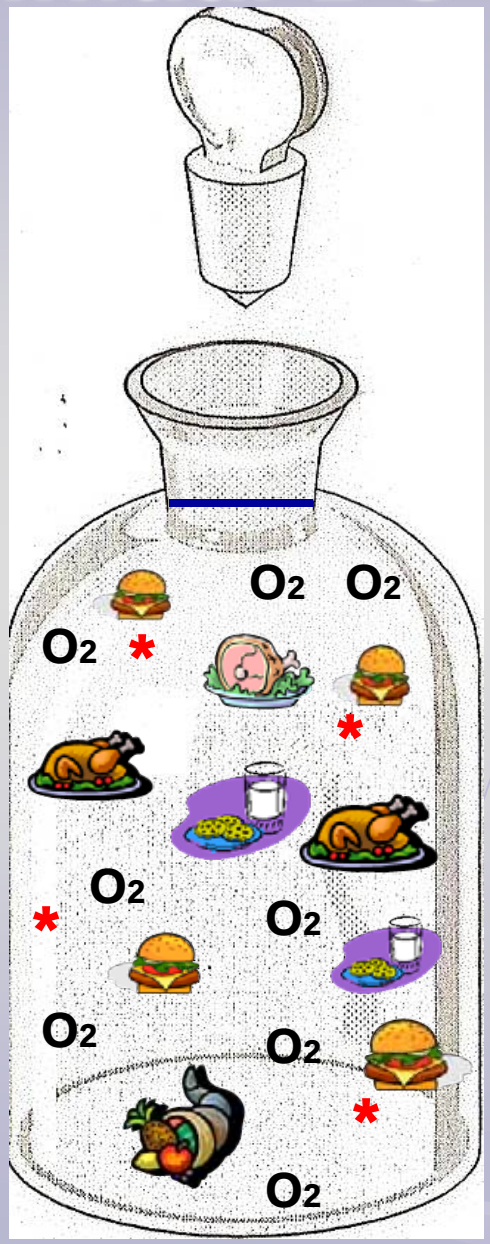
Add

Sample (food)
Seed (bugs)
Dilution water
- **Oxygen**
- **Nutrients**
- **pH balanced**

Measure
IDO

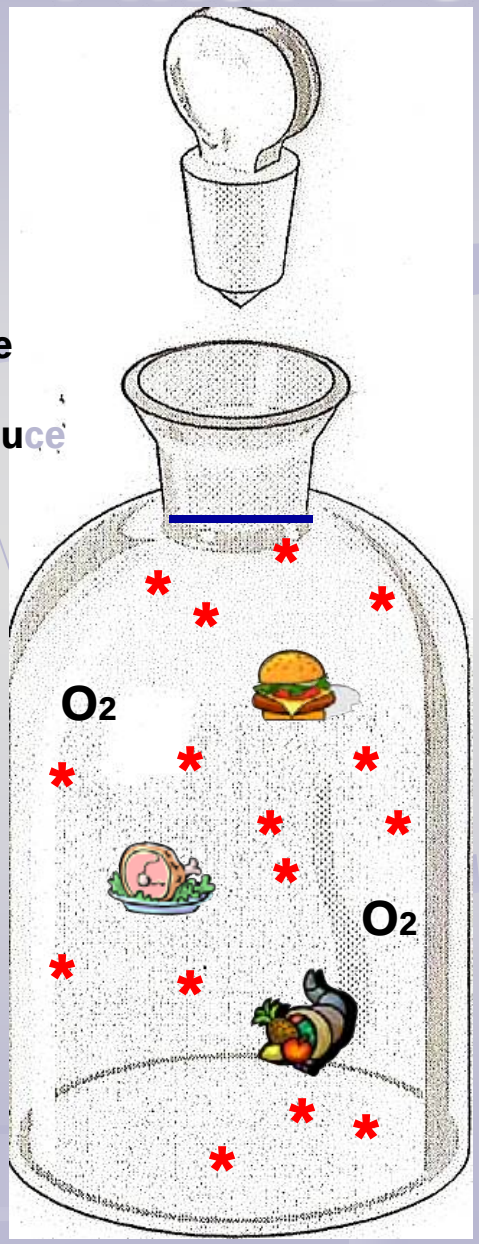
Incubate
- **5 days**
- **In dark**
- **20 C ±1 C**

Measure
Final DO



*

Breathe
Eat
Reproduce



Calculate BOD

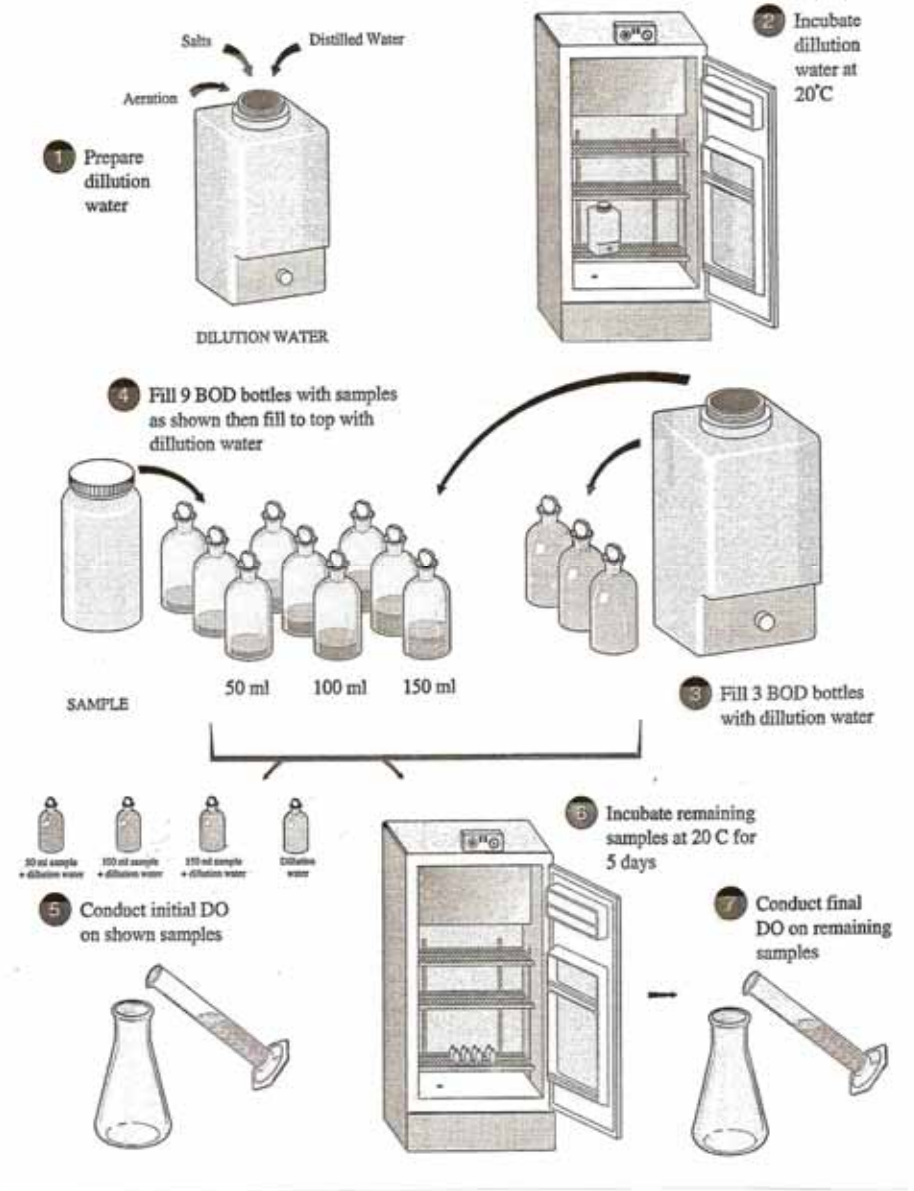
Subtract Final DO from IDO
Plug into equation

Estimate

Amount of BOD (food) in wastewater based on the oxygen consumed

BOD Procedure

- Use BOD to measure the **organic load** to the plant, a particular process unit or to the receiving stream



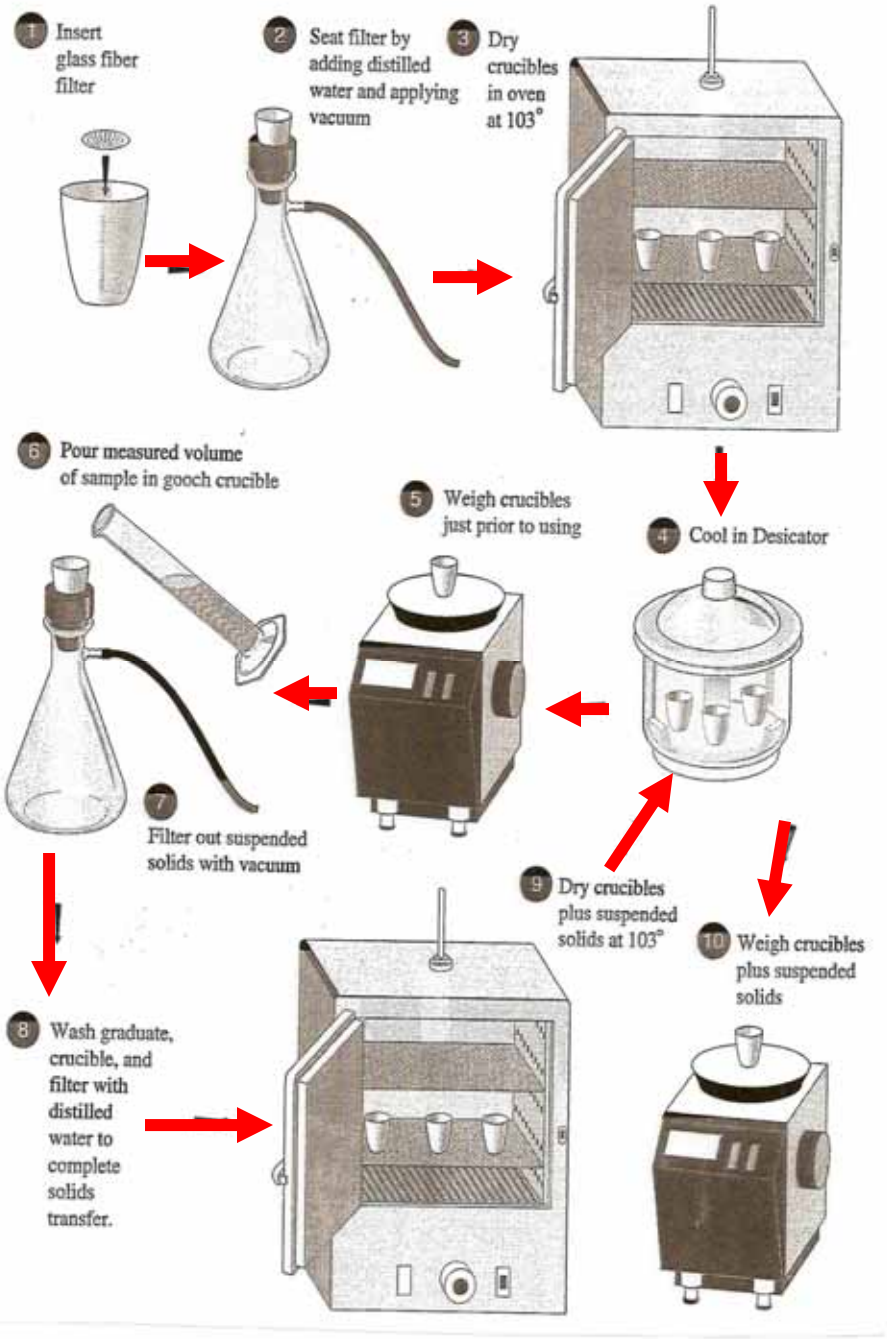
TSS

- **Non-filterable residue**
- What's left on the filter after drying at 103 – 105 C
- Subtract weights:

Wt. of Dry Solids, g =

(Residue + Filter) – Filter

Perform the Calculation to get TSS as mg/L



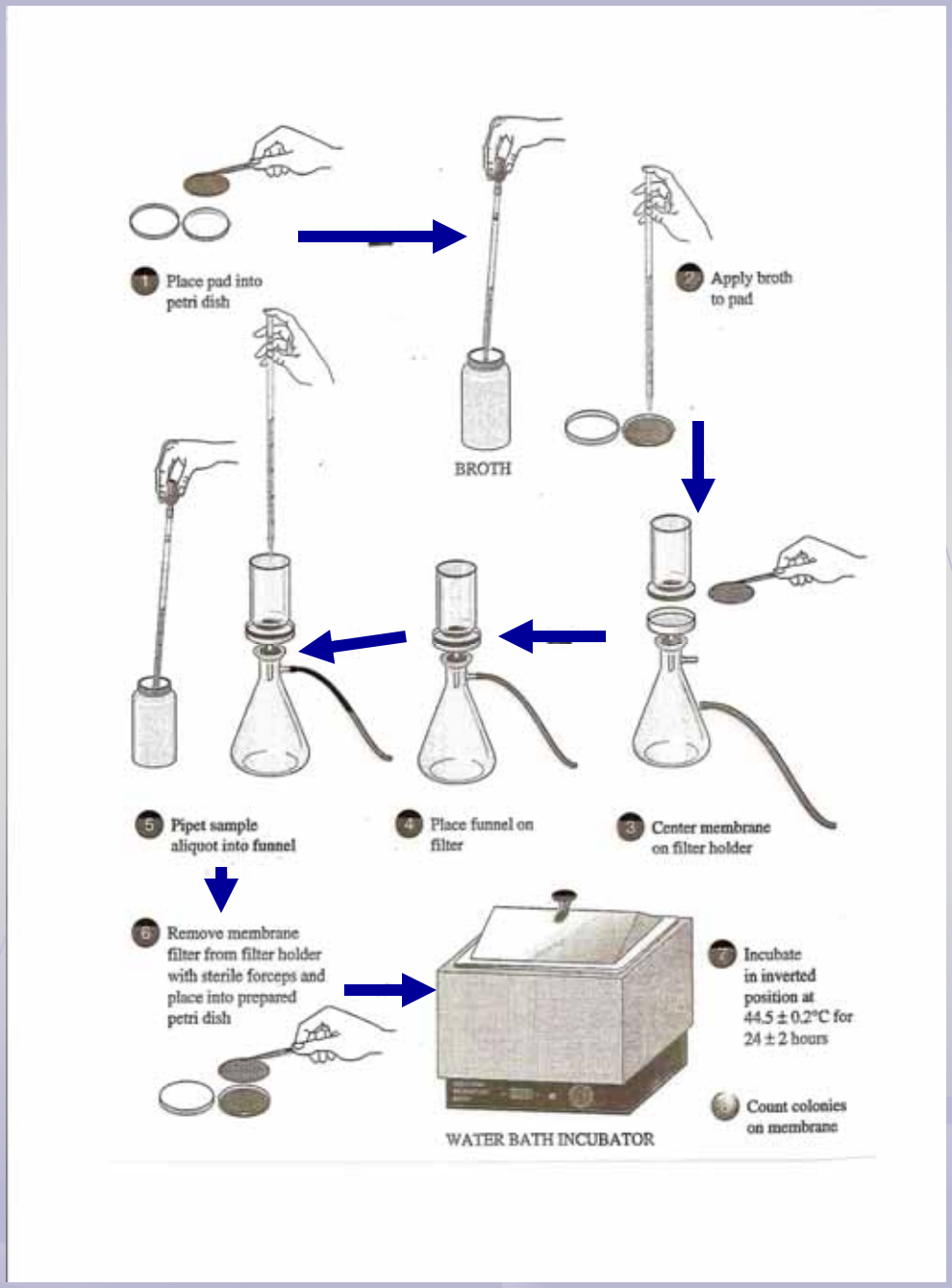
Fecal Coliform

Membrane Filtration

- Note water bath temperature **44.5 C ± .2 C**
- Count blue colonies

- ***E. coli*** HACH m-ColiBlue24 method uses same basic procedure

- Except
  - Incubation temperature is 35 C ± .5 C in dry air incubator
  - Count blue colonies



Quality Assurance/Quality Control (QA/QC)

- The methods and procedures used in the lab to guarantee the validity of the numbers reported on monitoring reports

- Includes
  - Checking or calibrating lab thermometers to NIST standard thermometers
  - Determining the BOD of a glucose/glutamic acid standard that has a known BOD

- Reweighing TSS samples to determine dryness within acceptable limits
- Incubating a sample of raw sewage (which is teeming with fecal bacteria) with the fecal coliform test to assure that coliforms will grow on a given media
- Duplicates and spikes run on 10% of all samples per year
- Each lab must have its own SOP (Standard Operating Procedures)

Lab Questions?