

BOD/CBOD, TSS pH OPERATOR BASICS

Presented by:
Marcy Bolek - Alloway

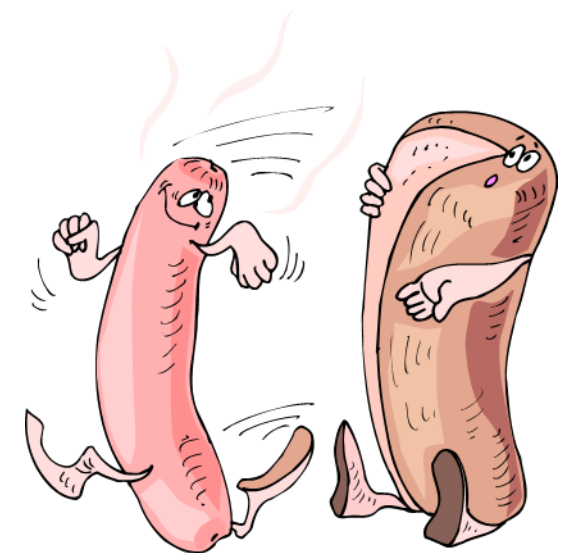
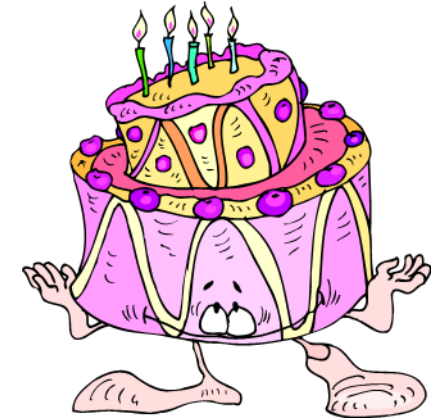


CONTENTS

- BOD/CBOD ANALYSIS
- TSS ANALYSIS
- pH ANALYSIS

BIOCHEMICAL OXYGEN DEMAND (BOD)

- mg/L oxygen bacteria use to oxidize organic matter (sugars, starch, proteins, petroleum hydrocarbons and other organic materials)
- Bacteria in water live and multiply when organic matter is available for food and OXYGEN is available for oxidation
- Bacteria oxidize mainly soluble organic matter



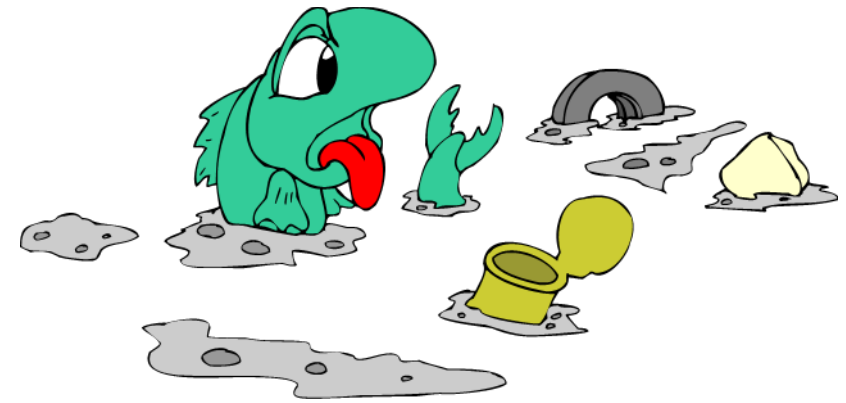
SIGNIFICANCE

- Measurement of BOD has long been the basic means for determining the degree of water pollution
- By comparing the BOD of incoming sewage and the BOD of the effluent water leaving the plant, the efficiency and effectiveness of sewage treatment can be judged



FISH KILL

- If water of a high BOD value flows into a river, the bacteria in the river will oxidize the organic matter, consuming oxygen from the river faster than it dissolves back in from the air. If this happens, fish will die from lack of oxygen, a consequence known as a fish kill



PRINCIPLE OF THE METHOD

- The test measures the oxygen utilized during a specified incubation period for the biochemical degradation of organic material
 - Carbonaceous demand and the oxygen used to oxidize inorganic material such as sulfides and ferrous iron.
 - It may also measure the oxygen used to oxidize reduced forms of nitrogen (Nitrogenous demand) unless their oxidation is prevented by an inhibitor.



Theory

PROCEDURE

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“When you’re done ruining the sofa, I want you to start clawing the new stereo speakers. After that, you need to leave your tongue prints in the butter, then take a nap on a pile of clean laundry.”

DILUTION METHOD

- The dilution method is conducted by placing various incremental portions of the sample into bottles and filling the bottles with dilution water (pH of the sample 6.0 – 8.0)



25 mL



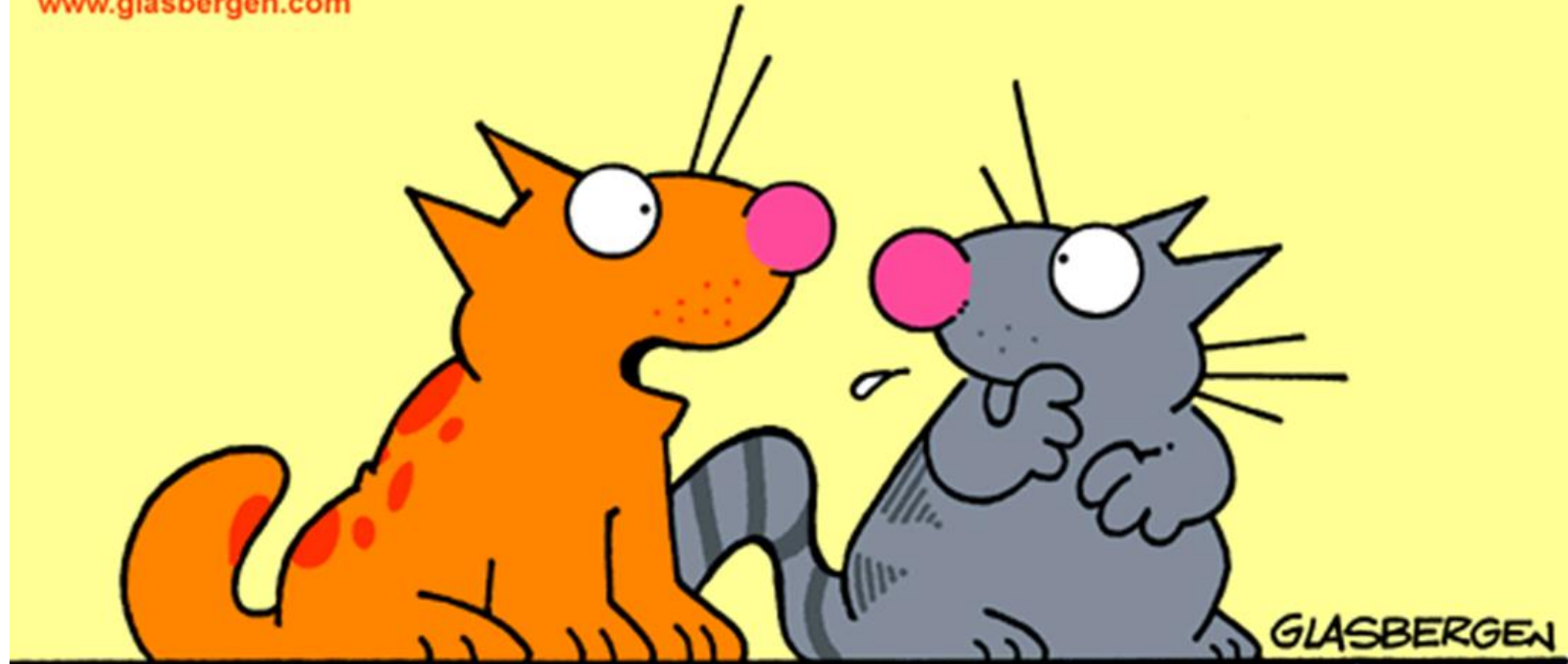
100 mL



300 mL

CLEAN GLASSWARE

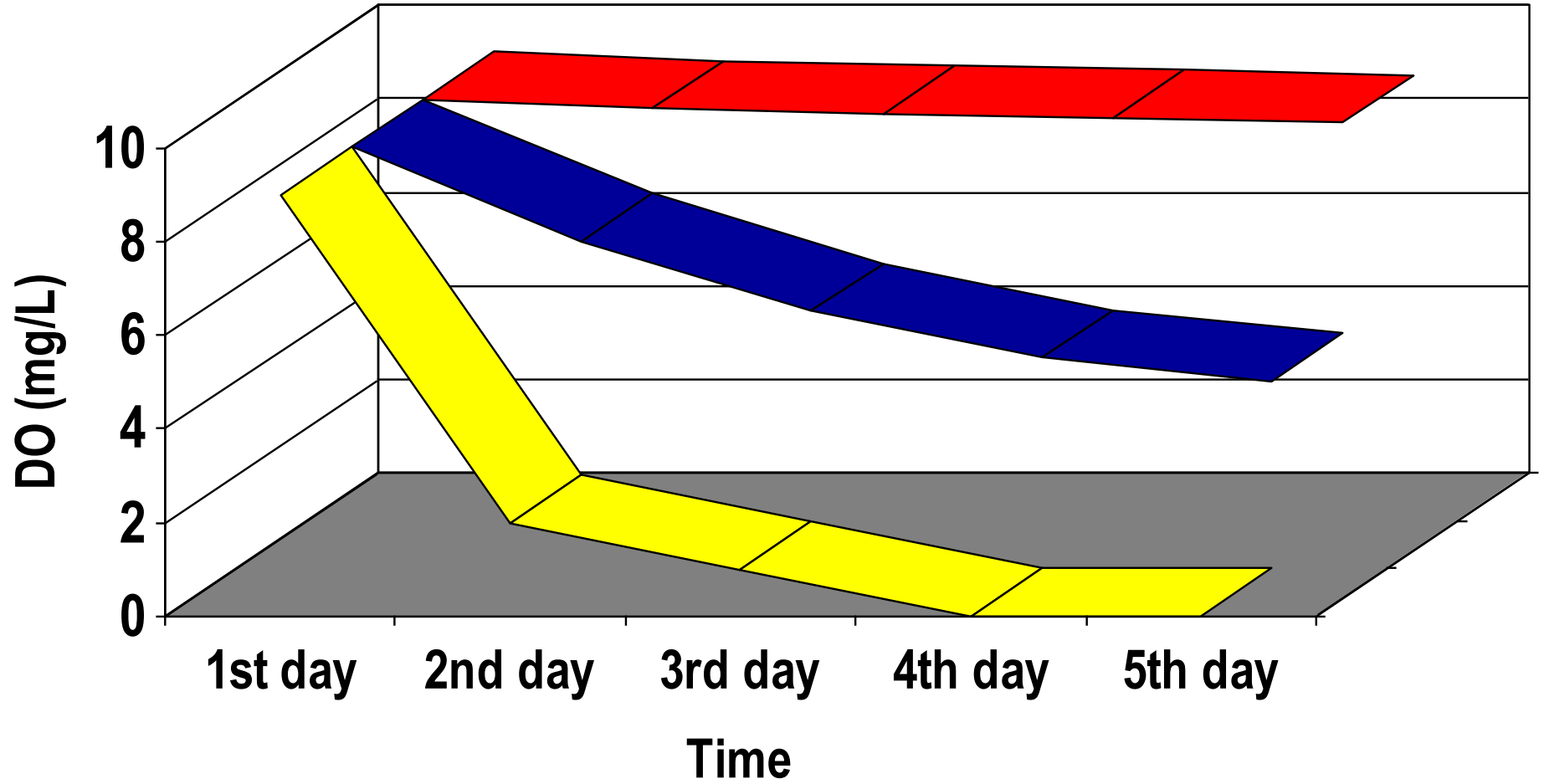
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**“Licking your paws is just the first step.
After that, you need to use a good antibacterial
body wash, then an exfoliating herbal facial scrub,
followed by avocado moisturizing cleanser....”**



RIGHT DILUTION

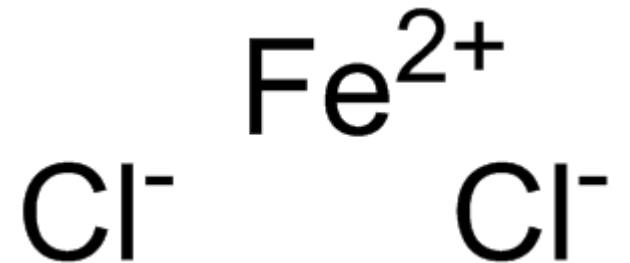
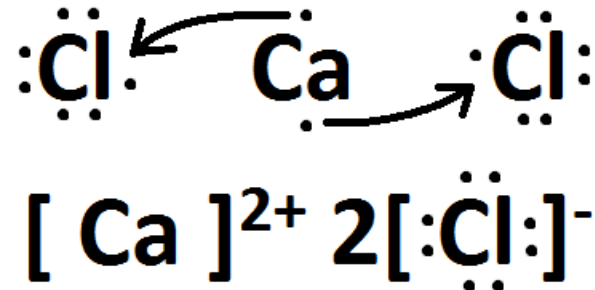
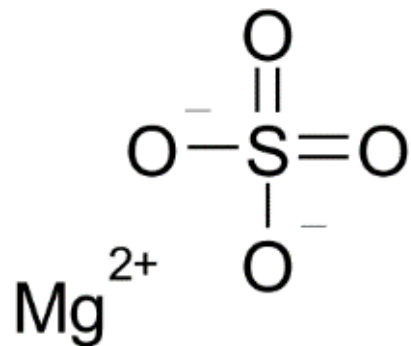


NEED FOOD



DILUTION WATER

- Dilution water contains a portion of inorganic nutrients
 - MgSO_4 , CaCl_2 , FeCl_2 & Phosphate Buffer
- Initial D.O. is measured
- Bottles are completely filled, freed of bubbles and sealed
- Bottles are incubated for 5 Days at 19-21 °C in the dark



DILUTION WATER QUALITY

- If the dilution water is of poor quality
 - Result = BOD detected in the sample
 - The amount will be amplified by dilution factors
 - Results will be biased high
- D.O. depletion must be ≤ 0.2 mg/L
- If requirement is not met consider
 - Changing source of water
 - Use one time use disposable bottles



AT THE END

- After the 5-day incubation
 - Measure the remaining dissolved oxygen (Final D.O.)
- The relationship of oxygen that was consumed during the incubation period and the volume of sample increment is then used to calculate the BOD



Calculations Part I

- When dilution water is not seeded

$$BOD(5)mg / L = \frac{(D_1 - D_2)}{P}$$

$$P = \frac{V}{300}$$

Where:

D_1 = initial D.O. of diluted sample, mg/L

D_2 = D.O. after 5 day incubation, mg/L

P = decimal volumetric fraction of sample used

V = volume of sample used, mL

(300 mL – volume of BOD bottle)

Calculations Part II

- When dilution water is seeded

$$BOD(5)mg / L = \frac{(D_1 - D_2) - (B_1 - B_2) \times f}{P}$$

Where:

D_1 = initial D.O. of diluted sample, mg/L

D_2 = D.O. after 5 day incubation, mg/L

P = decimal volumetric fraction of sample used

B_1 = D.O. of seed control before incubation, mg/L

B_2 = D.O. of seed control after incubation, mg/L

f = ratio of seed in diluted sample to seed in seed control

EXAMPLE

SEED CONTROL

$$f = \frac{2}{V}$$

mls seed (V)	Initial DO mg/L (B ₁)	Final DO mg/L (B ₂)	Depletion mg/L (B ₁ -B ₂)	DO uptake of 2 mls of seed (B ₁ -B ₂)xf	Average
10	8.4	5.0	3.4	0.68	0.64 (0.6)
15	8.4	3.5	4.9	0.65	
20	8.4	2.4	6.0	0.58	

EXAMPLE CONTINUED

mls of sample	mls of seed used	Initial DO mg/L	Final DO mg/L	Depletion mg/L
5.0	2.0	8.1	7.5	0.6
50	2.0	8.1	6.8	1.3
100	2.0	8.1	5.2	2.9
300	2.0	8.1	1.1	7.0

EXAMPLE CONTINUED

For 300 mLs:

$$BOD(5)mg / L = \frac{(8.1 - 1.1) - (0.6)}{\frac{300}{300}} = 6.4$$

For 100 mLs:

$$BOD(5)mg / L = \frac{(8.1 - 5.2) - (0.6)}{\frac{100}{300}} = 6.9$$

Final Result:

$$BOD(5)mg / L = \frac{(6.9 + 6.4)}{2} = 6.65$$

EXAMPLE CONTINUED

- Why were the 5.0 mL dilution and 50.0 mL dilution not used?
- The depletion for each one was $< 2.0 \text{ mg/L}$



TOTAL SUSPENDED SOLIDS

- Total Suspended Solids – TSS
 - Total Non-Filterable Solids
 - Residue, Non-Filterable
 - Solids/Residue retained on filter after filtration
- Analysis is important in the control of wastewater treatment plant process



HOLDING TIME/PRESERVATION

- Obtained from Code of Federal Regulations
 - 40 CFR 136.3 Table II

Analyte	Bottle Type	pH Preservation	Temperature	Holding Time
TSS	P, FP or G	None	Cool, $\leq 6^{\circ}\text{C}^{18}$	7 Days

P = Polyethylene
FP = Fluoropolymer
G = Glass



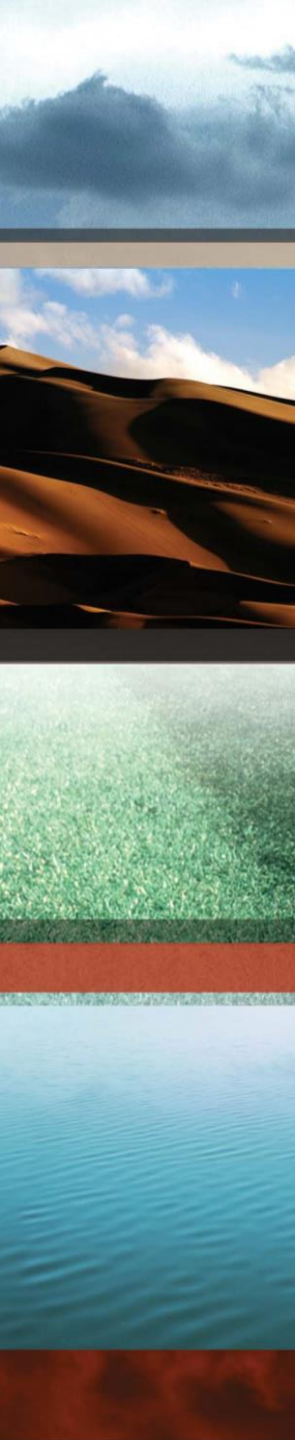
HOLDING TIME/PRESERVATION

- Obtained from Code of Federal Regulations
 - 40 CFR 136.3 Table II

¹⁸Aqueous samples must be preserved at ≤ 6 °C, and **should not be frozen** unless data demonstrating that sample freezing does not adversely impact sample integrity is maintained on file and accepted as valid by the regulatory authority. Also, for purposes of NPDES monitoring, the specification of “ \leq °C” is used in place of the “4 °C” and “<4 °C” sample temperature requirements listed in some methods. It is not necessary to measure the sample temperature to three significant figures (1/100th of 1 degree); rather, three significant figures are specified so that rounding down to 6 °C may not be used to meet the ≤ 6 °C requirement. **The preservation temperature does not apply to samples that are analyzed immediately (less than 15 minutes).**

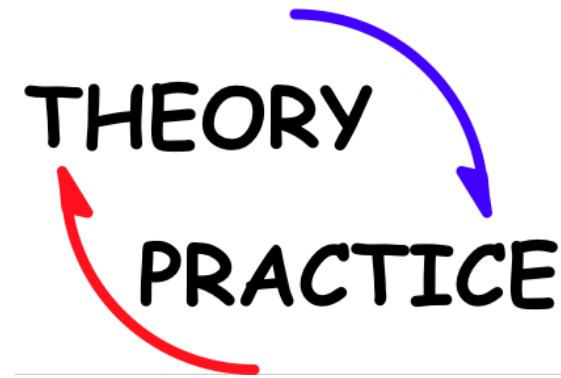
SIGNIFICANCE

- Sources of Error
 - Samples not homogenous
 - uniform in composition
 - Biological Decomposition
 - Hygroscopic nature of solids – collects moisture



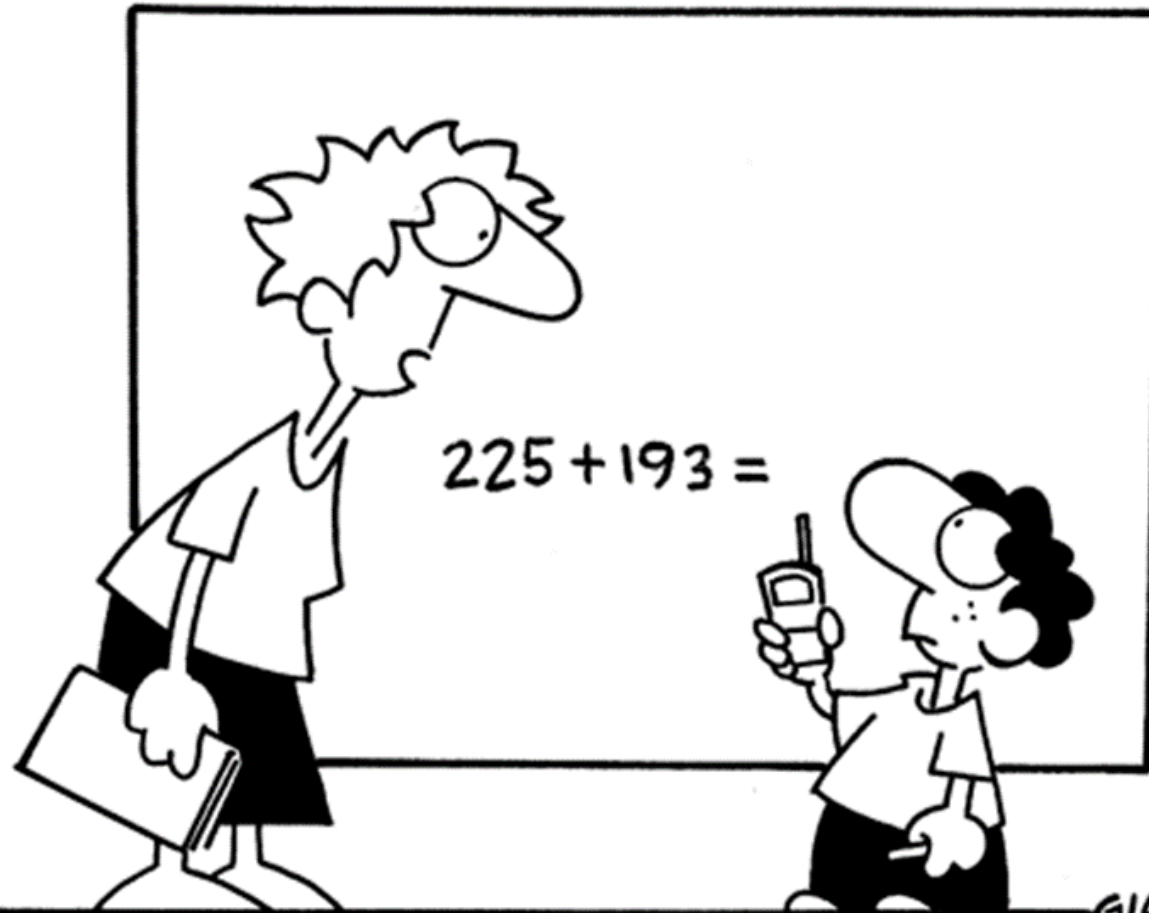
PRINCIPLE OF THE METHOD

- Sample is filtered through a glass fiber filter
- Residue is retained on filter and dried to constant weight
 - Temperature 103 – 105 °C
- Optimum residue amount on filter is 10 – 200 mg
 - 0.0100 g – 0.2000 g
- Sample aliquot must represent sample (homogenous)
- Filter must be dried at least one hour
- Constant weight (weight loss ≤ 0.5 mg)
 - 0.0005 g



PROCEDURE

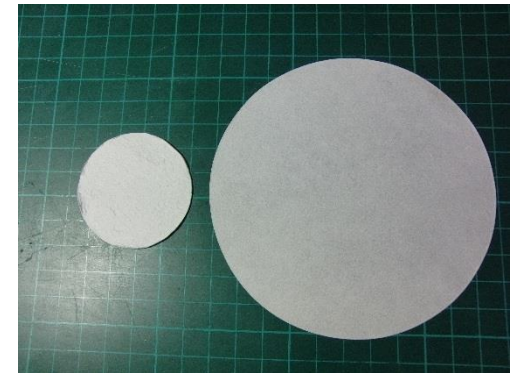
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“You have to solve this problem by yourself. You can’t call tech support.”

PREPARE FILTER

- Place filter on filter apparatus with wrinkled side up
- Apply vacuum and wash with 3 successive 20 mL portions of reagent water
- Remove from filter apparatus
- Dry in oven at 103 – 105 °C at least 1 hour
- Remove from oven and desiccate until cool
- Store in desiccator until needed



SELECTION OF SAMPLE VOLUME



- An attempt should be made to filter at least 100 mL if practical
- If weight difference between final and initial weights is less than 0.0010 grams increase sample volume to achieve at least 0.0010 grams of residue
- If filtration rate slows & takes more than 5 – 10 minutes attempt filtration again using smaller sample volume

SAMPLE FILTRATION & ANALYSIS



- Assemble filtration apparatus and begin vacuum
- Wet filter with reagent water to seat it against membrane support
- Mix sample vigorously and immediately transfer pre-determined volume to filter using a graduated cylinder (TD)
- Wash graduated cylinder, filter residue on filter and filter funnel walls with 3 portions of reagent water.

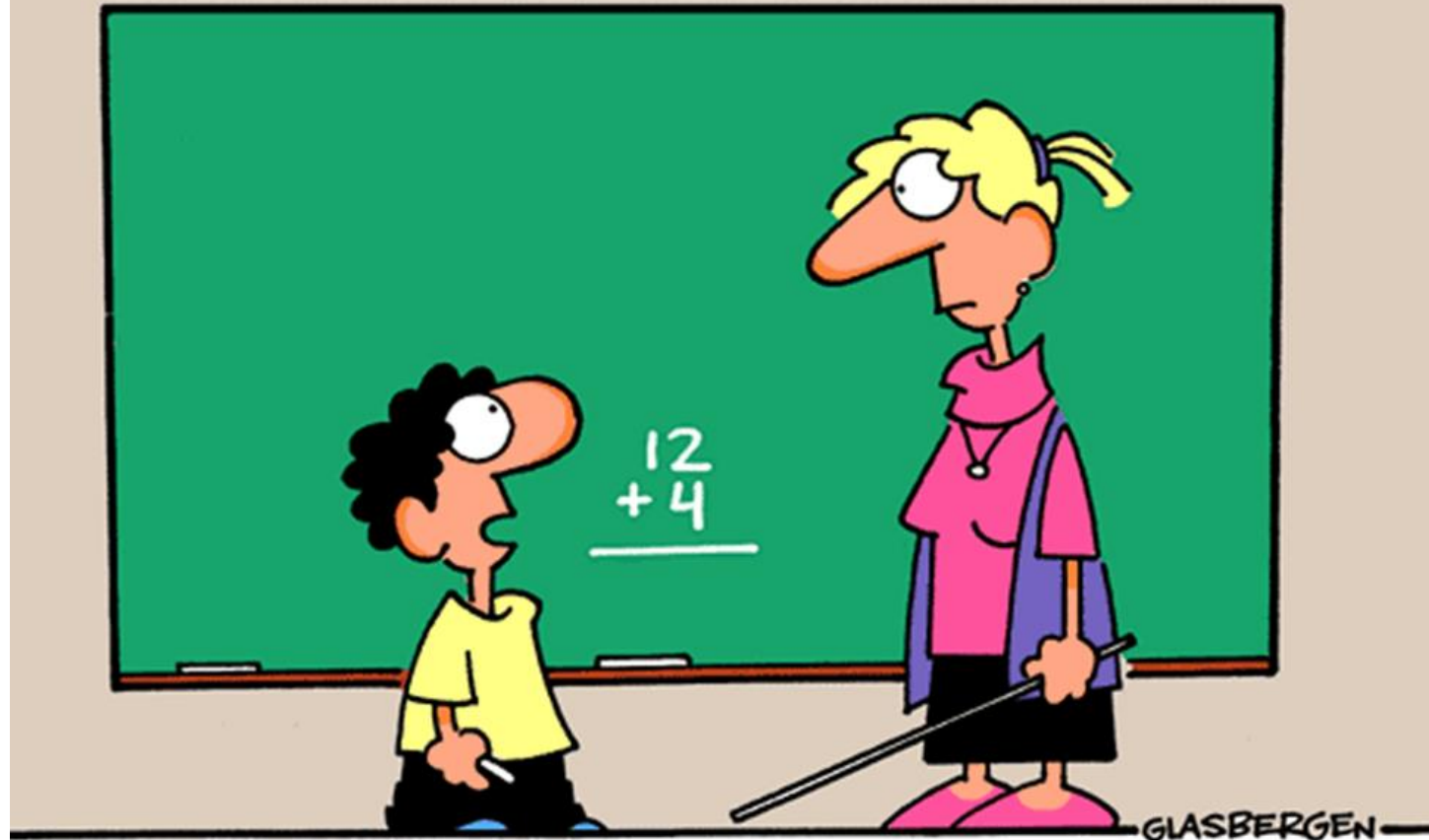


SAMPLE FILTRATION & ANALYSIS

- Continue vacuum until no additional drops come through the filter
- Stop vacuum and remove filter
- Dry in oven at 103 – 105 °C for minimum of 1 hour
- Remove from oven and transfer to desiccator to cool

CALCULATIONS

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“Do I get partial credit for simply having the courage to get out of bed and face the world again today?”

TSS FINAL RESULT

$$TSS \text{ mg} / L = \frac{A - B \times 1000}{mLs}$$

Where:

A = weight of dried residue and dish, mg

B = weight of dish, mg



pH

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—GLASBERGEN—

"I TURNED IN MY HOMEWORK TWO DAYS LATE,
BUT NORMALLY IT'S FOUR DAYS LATE,
SO TECHNICALLY IT'S EARLY!"

HOLDING TIME/PRESERVATION

- Obtained from Code of Federal Regulations
 - 40 CFR 136.3 Table II

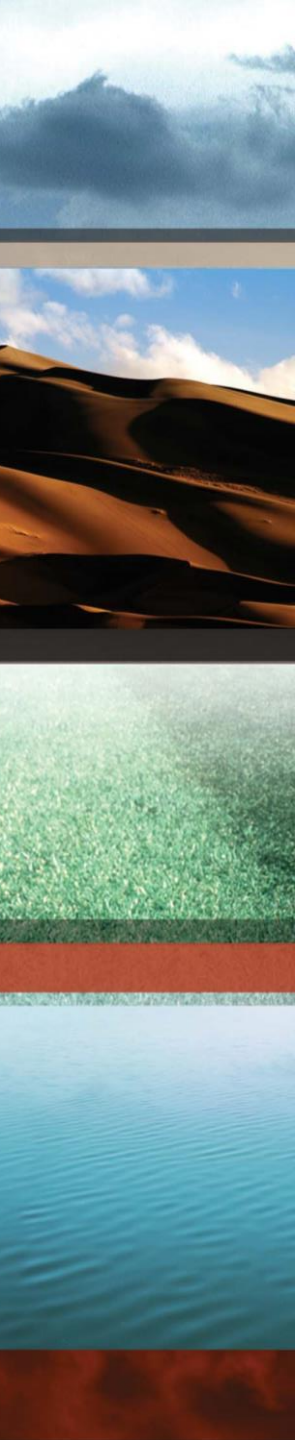
Analyte	Bottle Type	pH Preservation	Temperature	Holding Time
pH	P, FP or G	None	None	15 minutes

P = Polyethylene
FP = Fluoropolymer
G = Glass

SIGNIFICANCE



- pH important to
 - Plants using biological methods of secondary treatment
 - Plants using chemical treatment processes (chlorination)
 - Used to determine if process operates within acceptable range





SIGNIFICANCE

- Biological systems acceptable pH range
6.5 – 8.0
- Chemical treatment process acceptable pH
as close to pH 7.0 as possible
- Why?
Chlorination is most effective at a pH of 7.0



PRINCIPLE OF METHOD

- Commercially available meter is calibrated with known buffers
- Meter must have Automatic Temperature Compensation (ATC)
- pH is determined electrometrically using a glass electrode in combination with a reference potential or combination electrode

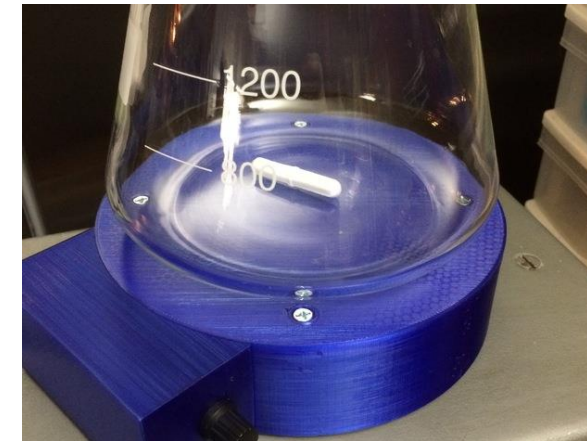
CALIBRATION – TWO POINT

- Calibrate each day of use and each shift if more than 8 hours exceed the last calibration
- Rinse electrode with reagent water and blot dry
- Transfer fresh 7.0 buffer to beaker
- Submerge electrode in buffer – covering sensing element and apply stirring
- Accept 7.0 calibration point when meter stabilizes



CALIBRATION – TWO POINT

- Rinse electrode with reagent water and blot dry
- Transfer fresh 10.0 buffer to beaker
- Submerge electrode in buffer to cover sensing element
- Apply stirring
- Accept 10.0 calibration point when meter stabilizes
- Document slope and ensure within meter limits



CALIBRATION CHECK

- Verify calibration using 2nd source buffer
- Use buffer that falls between 7.0 & 10.0
 - Suggest using a 9.0 buffer
- Analyze the check buffer the same way a sample is measured
- The check buffer must be ± 0.10 pH units
- For a 9.0 buffer
 - 8.90 - 9.10 acceptance criteria



PROCEDURE

- Rinse electrode with reagent water and blot dry
- Transfer portion of well mixed sample to a beaker
- Submerge electrode to cover sensing element
- Apply stirring
- Allow electrode to stabilize
- Record result





FINAL RESULT

- The pH of each sample must be bracketed by two pH buffers used for calibration
- If calibration buffers bracket the sample result, the sample result may be reported.
- If the calibration buffers do not bracket the sample value, the sample must be reanalyzed after a new calibration is performed using buffers that bracket the sample result.