Lab Exam Review

April 28, 2014

Why are we here?

A brief history of lab exam results

How to best prep for an exam?

Why should we care?

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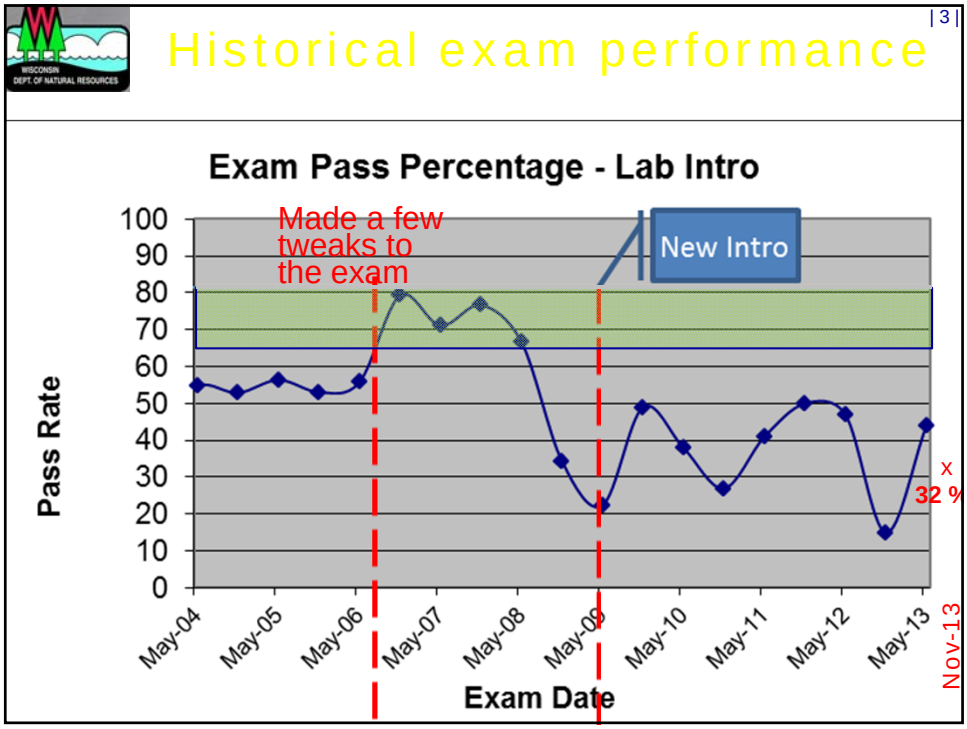
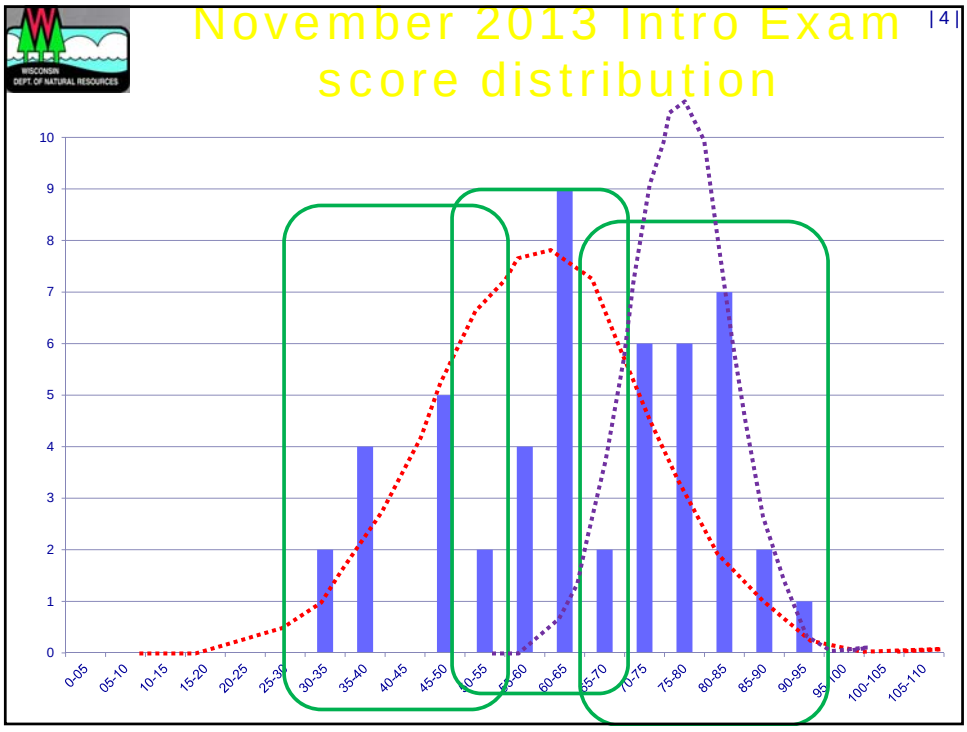
WI DNR Lab Certification

Score required to PASS

75%

- That's 30 of 40 questions
- You can miss no more than 10 questions


| 3 |
Historical exam performance
Exam Pass Percentage - Lab Intro
Made a few tweaks to the exam
New Intro
Pass Rate
100
90
80
70
60
50
40
30
20
10
0
May-04
May-05
May-06
May-07
May-08
May-09
May-10
May-11
May-12
May-13
Exam Date
X
32 %
Nov-13


| 4 |
November 2013 Intro Exam score distribution
10
9
8
7
6
5
4
3
2
1
0
0-05
05-10
10-15
15-20
20-25
25-30
30-35
35-40
40-45
45-50
50-55
55-60
60-65
65-70
70-75
75-80
80-85
85-90
90-95
95-100
100-105
105-110

| 5 |

EPIC FAIL

Seriously, how the heck did you manage that?

| 6 |

Evaluating Corrective Action

INTRO EXAM - NOV 2013

50 took the exam, 16 passed = **32% pass rate**.

Those who attended the 10/7/13 training

3 of 9 taking passed (**33%**),

(**9.5 pts better** than all)

Those that took exam previously & attended 10/7:

1 person doubled their previous test score.

Overall, a **16 point improvement in scores** from the previous attempt (**6 or 7 questions**).

WHY are people failing this exam?

[8]

- What's hard about it?
- Did you look at the Study Guide first?
- Did you take a prep class?
- If you took a class…do you think it helped?
- No different than corrective action in the lab
- Is it **you**, the **ammo**, or the **rifle**?
- Can we rule out things one at a time?
- But **if you repeat the same process, you should expect the same results**.
- If it's not you or the rifle…maybe switch ammo

Mentality issue

- Some think that one needs to pass the test BEFORE one does any lab testing.
- Often lab supervisors wont let people do the testing until they pass the OpCert exam (as part of an “IDC”).

- You need to know the lab testing and methods FIRST.

Exam Review: Good News 1st

INTRO Knowledges with >75% Pass

- 100% can define BOD!
- 99% can explain the function of a desiccator.
- 97% understand proper handling of weights.
- 95% know conditions that affect drying ovens.
- 95% know how and why records need to be permanent.
- 94% know when seeding is required for BOD.
- 92% understand super-saturation. **BUT…**
- 91% know when to use wide-bore pipets.

Now the Not-so-Good News....

INTRO Knowledges with 50 - 75% Pass

- 60%: Know what bias and precision are.
- 59%: Understand the concept of seeding BOD.
- 57%: Know the acceptance criteria for ISE
- 56%: Know the difference between arithmetic and geomean.
- 53%: Understand why, with ISE, samples and standards must be at the same temperature.
- 52%: understand how sample dilution volume affects the LOD for BOD.
- 51%: Given sample data, can calculate BOD.

...and the REALLY not-so-good news

INTRO Questions with < 50% Pass…

- **56%** still don't know the purpose of NaOH buffer for ammonia by ISE.
- **60%** don't know requirements for certified weights.
- **60%** don't know the best concentration to determine LODs.
- **62%** don't know the required LOD for TSS.
- **74%** don't know about balances.
- **82%** don't know understand LOD/LOQ.
- **85%** don't know calibration requirements for TP.
- **86%** don't know critical conditions for TP color development.

| 13 |

Post-Exam QA/QC

- We DO look at Exam results!
- A Quality Assurance review to ensure that the exam is properly measuring operator knowledge.
- Ask the questions:
  - Is the passing rate acceptable?
  - Particular question(s) too easy?
  - Particular question(s) too difficult?
  - Is there more than one perceived correct answer?
- Additionally, review operator comments/concerns about the exams

| 14 |

Don't just slap a bandaid on it!

- DO NOT go study only the concepts just presented!
- Remember: Exams are generated randomly from a bank of questions.
  - There are over 120 questions in each exam bank. Less than 2/3 have appeared so far.
- There are questions that have yet to appear tied to OTHER knowledges that operators lack.
- READ the Study Guide.
- …and did we mention...**READ the Study Guide!**

DON'T BE A SHERBY!

- Kaffee: You gotta trust me, Sherby, you read the Study Guide and your chances of passing the exam increase by a factor of 10

READ THE STUDY GUIDES!

How to effectively study/prepare for an exam:

Reading for Comprehension

Reading for Comprehension

| 17 |

- **Highlight** important ideas.
- Circle or bracket **key terms**.
- Identify the main point(s) of the info.
- Can you eliminate "extraneous" information?
- Look for "telling" words: "always, must, require"
- What question(s) would YOU create?

Reading for Comprehension isolating critical info

| 18 |

5.1.04 Define super-saturation.

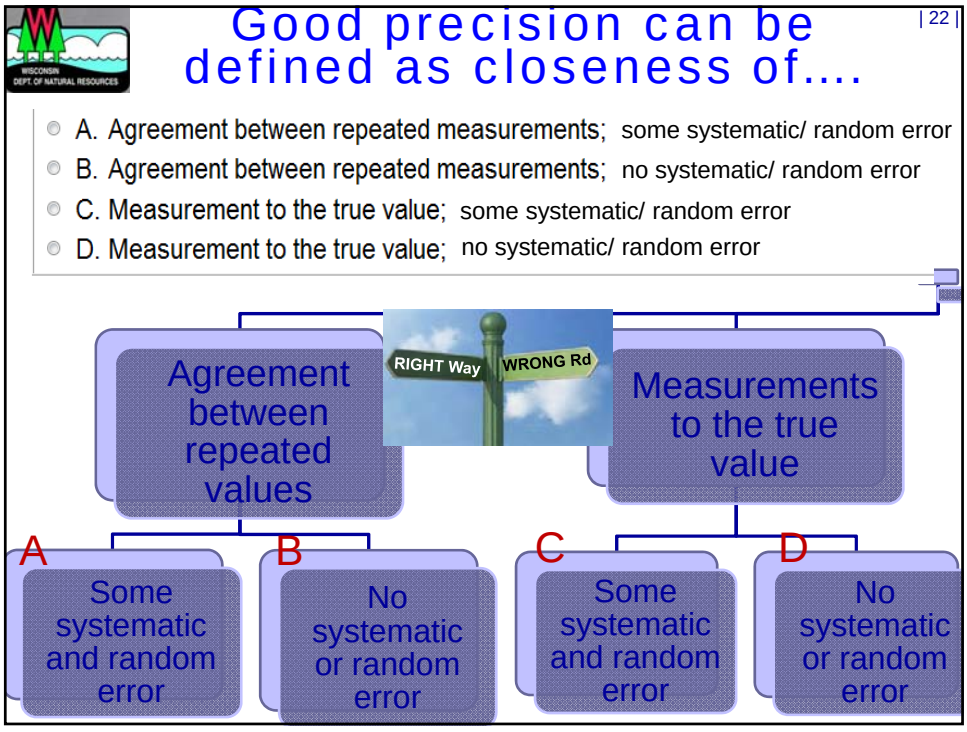
- Supersaturation means that the water contains more DO than it SHOULD contain according to physical tables.
- According to tables, the saturation point of oxygen in water at 20° and 760 mm pressure -which is standard temperature and pressure at sea level- is 9.06 mg/L. So, yes, at sea level and 20°C, anything over 9.06 mg/L represents supersaturation.
- The method kind of "defines" super-saturation as anything above 9.0 mg/L. However, in reality saturation will vary with temperature and pressure. Consult a DO saturation table.

Multiple Choice Test Taking Tips

- Read the question before you look at the answer.
- Come up with the answer in your head before looking at the possible answers, this way the choices given on the test won't throw you off or trick you.
- Eliminate answers you know aren't right.
- Read all the choices before choosing your answer.
- Don't keep on changing your answer; usually your first choice is the right one, unless you misread the question.

Many multiple choice answers have a dichotomy, or branching, to them. It's almost like a fork in the road and at the end of each fork are two doors.

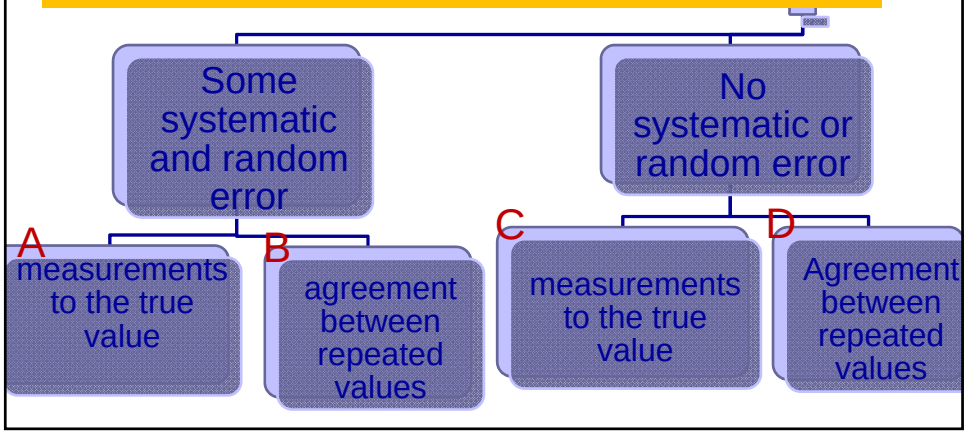

| 21 |
1
2
1
2
RIGHT Way
WRONG Rd


| 22 |
Good precision can be defined as closeness of....
A. Agreement between repeated measurements; some systematic/ random error
B. Agreement between repeated measurements; no systematic/ random error
C. Measurement to the true value; some systematic/ random error
D. Measurement to the true value; no systematic/ random error
Agreement between repeated values
RIGHT Way
WRONG Rd
Measurements to the true value
A
Some systematic and random error
B
No systematic or random error
C
Some systematic and random error
D
No systematic or random error

Good precision can be defined as closeness of....

m error
rror

Or come at it from the other direction



Is the answer in the question?

- Sometimes the right answer can be found right in the question itself.
- If an exam is developed allowing trickery (*these tempting answers are called "distractors", or "foils"),* this can be used to throw you off.
- But if trickery is not part of the design, use the words of the question to help find the right answer.

Answer in Question example

- **A sample obtained by taking portions of wastewater at a collection point in proportion to the flow is called _____**
- a. A time proportional composite.
- b. A routine sample.
- c. A Kemmmer-Sechi sample.
- d. A flow proportional composite

Let's look at some sample Lab related questions from other exam sources

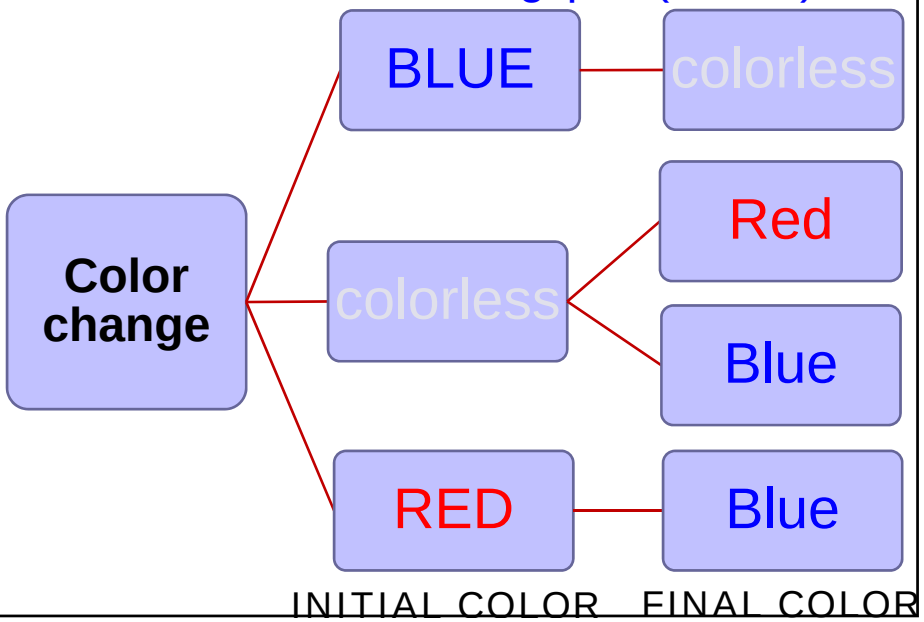
PHOSPHORUS

Between the pH range of 8.0 - 9.6 s.u., the indicator phenolphthalein undergoes what color change with increasing pH?

- A. Colorless to blue
- B. Colorless to red
- C. Red to blue
- D. Blue to colorless

- Break this question down to its basics.
- 3 of the 4 answers involve the color blue
- Does phenolphthalein ever give a blue color?
- If so, then at least you've eliminated 25% of the choices
- If not, then you've identified the correct answer

Phenolphthalein color change with increasing pH (8-9.6)



Thanks, Wikipedia

<8.2 colorless

8.2-12 pink

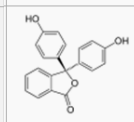
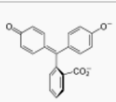
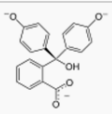
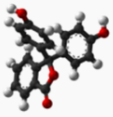
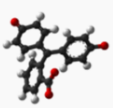
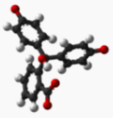
>12 colorless

Phenolphthalein

From Wikipedia, the free encyclopedia

"phph" redirects here. For Ph-Ph, see biphenyl.

Phenolphthalein /ˌfiːnɒlˈθeɪliːn/[1] is a chemical compound with the formula $C_{20}H_{14}O_4$ and is often written as "**HIn**" or "**phph**" in shorthand notation. Often used in titrations, it turns colorless in acidic solutions and pink in basic solutions. If the concentration of indicator is particularly strong, it can appear purple. In strongly basic solutions, phenolphthalein's pink color undergoes a rather slow fading reaction and becomes colorless again. The molecule has four forms:

Species	H_2In	In^{2-}	$In(OH)^{3-}$
Structure			
Model			
pH	0–8.2	8.2–12.0	>12.0
Conditions	acidic or near-neutral	basic	strongly basic
Color	colorless	pink to fuchsia	colorless
Image			

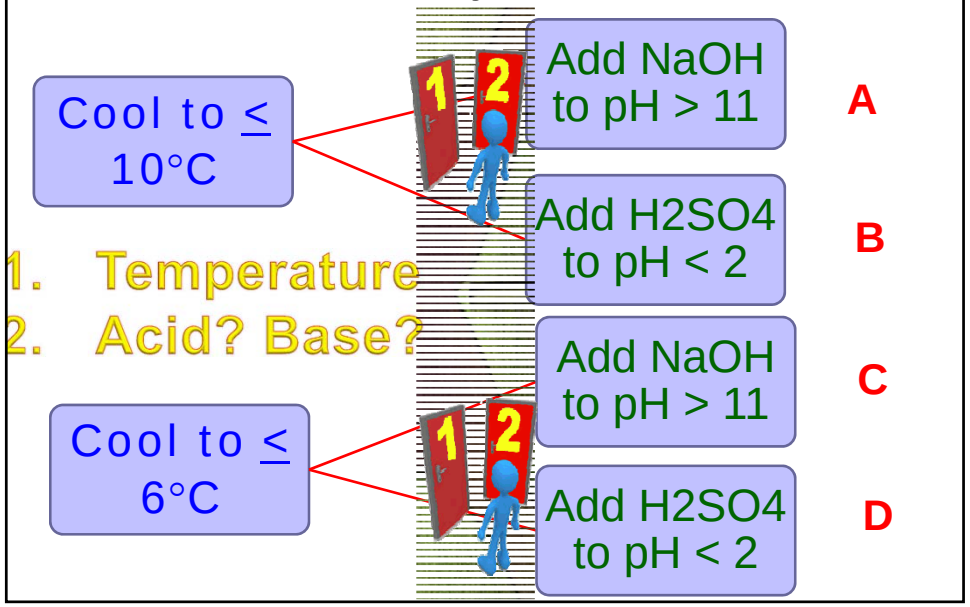
Calibration

When calibrating an instrument that uses a linear curve, what is the minimum number of concentrations that must be used?

- A. One
- B. Two
- C. Three
- D. Five

- Read carefully! Do not leap to conclusion or decide on the answer without reading the question fully.
- It says calibrate…not CHECK the calibration.
- MINIMUM…not how many standards YOU use in your lab.

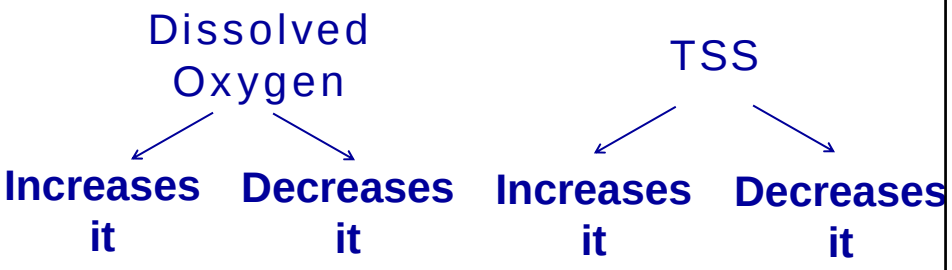
How do you preserve a sample for ammonia if it cannot be analyzed when collected ?



BASIC ?s

Agitating a sample before measuring DO for BOD …

a. Decreases the DO.
b. Increases the DO.
c. Decreases the SS.
d. Increases the SS.



Example of a bad question…why?

BASIC - BOD

The BOD of wastewater determines the milligrams per liter of oxygen required…

a. During stabilization of decomposable organic matter by aerobic bacterial action.

b. To produce an equilibrium between the oxygen of the wastewater and atmospheric oxygen.

c. To unite chemically with the inorganic matter present in the sample.

d. For the oxidation of sulfites and thiosulfates to sulfates.

Minimal rewording of answers

BASIC - BOD

The BOD of wastewater determines the milligrams per liter of oxygen required…

a. Bacterial action on organic matter

b. Equalize wastewater Oxygen with air

c. Bond with inorganic matter

d. Oxidize sulfites and thiosulfates

Minimal rewording of answers

BASIC - BOD

BOD incubation is at:

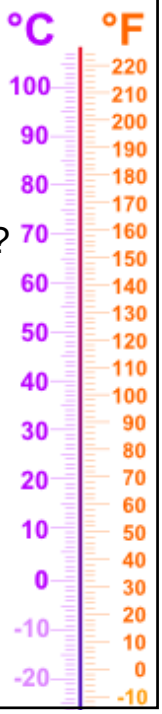
~~a. 37° C.~~

~~b. 98° F.~~

c. 20° F.

d. 20° C.

Incubate? or Hibernate?



- 37 °C equals about 98 °F…so since both can't be right, neither is!
- Where would you prefer to incubate? At 20 °F or 20 °C?

You're guessing!

The reporting limit for residual chlorine must not exceed:

- A. 0.380 mg/L [21% chose this]
- B. 0.100 mg/L [26% chose this]
- C. 0.200 mg/L [18% chose this]
- D. 1.00 mg/L [35% chose this]

Retired INTRO question

You're guessing!

The temperature preservation requirement for most wastewater tests is that they not be frozen and must be stored at:

- A. ≤ 4 °C [10% chose this]
- B. 4 ± 2 °C [45% chose this]
- C. ≤ 6 °C [43% chose this]
- D. 6 ± 2 °C [2% chose this]

Retired ADVANCED question

Creating Flashcards

- Summarize the CRITICAL information from the study guide and copy to index cards.
- Use THESE to study

Writing your own questions

- If you were quizzing someone on the topic, what questions would you ask?
- Writing the question and correct answer is the easy part.
- Coming up with 3 "wrong" answers without using all/none of the above and not being too tricky is a challenge!

What questions would YOU write for this?

5.4.10 Explain why temperature is so critical when using the ion-selective electrode.

- Ammonia electrodes function according to the physical constraints of the Nernst equation, and in that equation temperature is the only variable. Each one degree (°C) change in temperature is associated with a 1-2% error due to changes in the electrode slope. Therefore, calibration standards and samples must be at the same temperature.

How about these....

- What is the principle that governs electrode operation: **the Nernst equation.**
- What is the only variable in the Nernst equation: **Temperature**
- 1 degree C temp change = **1-2% error.**
- Standards & samples must be: **same temp.**

It's time to take a look at the Study Guides

How to "study" the Study Guides

Identify "buzz" words:

shall, must, require, only always, every, **however**

Take note of any numbers/values (criteria)

Ignore extraneous words

Find the point(s) being made.

Try to separate informative but non-critical info

Frequently the 1st sentence contains critical info

Create flashcards

? What question(s) would **you** create?

INTRO 2.2.7

Explain how to sub sample or split a wastewater sample.

| 43 |

Historical: 71%
November 2013: 66 % pass

Most composite autosamplers collect wastewater into a single large jug, generally 2-3 gallons in size. When the facility is required to test their wastewater for more than one constituent that requires a different preservative (e.g., metals, phosphorus, suspended solids), it is necessary to sub-sample or "split" the wastewater into one or more containers so the appropriate preservatives may be added. This process must be done in such a way to assure that each sub-sample is identical to the other. Mixing is the most critical step in the sub-sampling process. When the following procedure is followed precisely sub-samples will be homogeneous.

The rest of the key knowledge content is simply a protocol.

INTRO 2.3.1

Identify the maximum holding times and preservation methods for the following samples:

| 44 |

Historical: 77%
November 2013: 66 % pass

A. Sample: BOD

Preservation Method: Cool to 6°C, or less without freezing

Maximum Holding Time: 48 hours after the end of the compositing period

B. Sample: TSS

Preservation Method: Cool to 6°C, or less without freezing

Maximum Holding Time: 7 days

C. Sample: Ammonia

Preservation Method: Add sulfuric acid(H_2SO_4) to pH<2 and cool to 6°C, or less without freezing

Maximum Holding Time: 28 days

INTRO 2.3.1

Identify the maximum holding times and preservation methods for the following samples:

Historical: 77%
November 2013: 66 % pass

D. Sample: Total Phosphorus

Preservation Method: Add sulfuric acid(H2SO4) to pH<2 and cool to 6°C, or less without freezing

Maximum Holding Time: 28 days

E. Sample: Fecal Coliform

Preservation Method: Add sodium thiosulfate if sample was chlorinated and cool to 6°C, or less without freezing

Maximum Holding Time: 6 hours

Samples should be refrigerated at a temperature not to exceed 6°C and must not be frozen.

INTRO 3.3.11

Discuss the selection of weights used for verification of calibration of an analytical balance.

Historical: 71%
November 2013: 54 % pass

The laboratory certification program requires labs to verify the calibration of their analytical balance at least monthly using a minimum of one Type 1 weight in the gram range, and a second Type 1 weight in the milligram range.

Select the weight close to the weight that you typically measure.

For example:
If using a Gooch crucible, use a 20-g or 50-g Type 1 weight.
If using a filter pad and pan, use a 100mg Type 1 weight.

Which you should NOT be!

INTRO 4.2.2

Discuss the proper use of a volumetric pipet using a bulb type pipet aid. | 47 |

November 2013: 60 % pass

1. Evacuate the pipet bulb by squeezing.
2. Immerse the tip of the pipet into solution to be delivered.
3. Seat the bulb opening over the top opening of the pipet.
4. Hold the bulb in place while slowly releasing the squeezing pressure.
5. Continue to release the pressure while the solution is drawn into the pipet.
6. Draw the solution up well past the calibration line.
7. Quickly remove the bulb and seal the top of the pipet with the index finger.
8. Keeping the index finger in place, remove the tip from the solution.

INTRO 4.2.2

Discuss the proper use of a volumetric pipet using a bulb type pipet aid. | 48 |

November 2013: 60 % pass

9. Rest the tip of the pipet on the side of the container that held the solution.
10. Slowly release finger; allow solution level (meniscus) to drop to calibration line.
11. Place tip of the pipet over the receiving vessel and completely release the finger.
12. Keep the pipet upright and allow to drain completely.

(Note: Many Class A pipets have the drain time imprinted adjacent to the "TD" designation.)

When the draining is complete, touch any remaining droplet at the tip of the pipet to the inside wall of the vessel.

INTRO 5.2.28

Historical pass rate: 44.1%
November 2013: 46%

Discuss the testing differences between BOD and cBOD.

The only difference between samples analyzed for BOD and those analyzed for cBOD is the addition of a chemical inhibitor to all samples for which cBOD is determined.

If nitrification was occurring in the original sample, the cBOD result is expected to be lower than a BOD result. The difference between the results represents the amount of oxygen which is utilized during the nitrification process. Since the BOD test only measures oxygen utilized, performing both BOD and cBOD is the only way to distinguish the amount of oxygen utilized during decomposition of organic waste from that which is utilized during nitrification.

INTRO 5.2.29

Given data, calculate BOD for a sample.

Historical: 2 questions, 53%, 49%
November 2013: 52% & 56 % pass

1. The formula for the calculation is:

   BOD (mg/L) =[(iDO - fDO) - SCF] × DF
   iDO = Initial DO (mg/L)
   fDO = Final DO (mg/L)
   SCF = Seed Correction Factor (if applicable)
   DF (Dilution Factor) = 300 mL ÷ [sample volume (mL)]

2. Given the following data:

Dilution	IDO	FDO	Sample Volume
A	8.30 mg/L	0.80 mg/L	300 mL
B	8.30 mg/L	1.30 mg/L	250 mL
C	8.30 mg/L	4.25 mg/L	200 mL
D	8.30 mg/L	5.90 mg/L	100 mL
E	8.30 mg/L	7.40 mg/L	50 mL

INTRO 5.2.29

Given data, calculate BOD for a sample.

Historical: 2 questions, 53%, 49%
November 2013: 52% & 56 % pass

3. Calulate BOD of individual dilutions:

BOD #A: Depletion = (8.30 - 0.80) - 0 = 7.5 mg/L
Excess depletion. DO NOT USE

BOD #B: Depletion = (8.30 - 1.30) - 0 = 7.0 mg/L
BOD= 7.0 mg/L × (300 ÷ 250)
BOD= 7.0 mg/L × (1.2)
BOD = 8.4 mg/L

BOD #C: Depletion = (8.30 - 4.25) - 0 = 4.05 mg/L
BOD= 4.05 mg/L × (300 ÷ 200)
BOD= 4.05 mg/L × (1.5)
BOD = 6.075 mg/L

BOD #D: Depletion = (8.30 - 5.90) - 0 = 2.4 mg/L
BOD= 2.4 mg/L × (300 ÷ 100)
BOD= 2.4 mg/L × (3)
BOD = 7.2 mg/L

INTRO 5.2.29

Given data, calculate BOD for a sample.

Historical: 2 questions, 53%, 49%
November 2013: 52% & 56 % pass

3. Calulate BOD of individual dilutions:

BOD #E: Depletion = (8.30 - 7.40) - 0 = 0.9 mg/L
Insufficient depletion. DO NOT USE

4. Average all useable results to obtain a BOD for the whole sample.

Average BOD = (8.4 + 6.075 + 7.2) ÷ 3
Average BOD = (21.675) ÷ 3
Average BOD = 7.225
Report BOD = 7

INTRO 5.2.32

Historical: 68%
November 2013: 54 % pass

Discuss procedures for reporting results when all dilutions over-deplete (final DO less than 1.0 mg/L).

| 53 |

Each sample dilution must meet the minimum requirement of 2 mg/L DO depletion. There must also be at least 1 mg/L of DO remaining in each sample dilution after 5 days. If there is more than one acceptable dilution, these results must be averaged.

Example: Given the following data:

Sample Bottle #	A	B	C
Sample Size (mL)	300	200	100
Initial D.O. (mg/L)	8.48	8.50	8.47
Final D.O. (mg/L)	< 1.0	< 1.0	< 1.0
Depletion (mg/L)	> 7.48	> 7.50	> 7.47
Dilution Factor	1	1.5	3
BOD (mg/L)	>7.5	>11.3	>22.4

INTRO 5.2.32

Historical: 68%
November 2013: 54 % pass

Discuss procedures for reporting results when all dilutions over-deplete (final DO less than 1.0 mg/L).

| 54 |

In the example, all three dilutions fail to meet the residual DO requirement of 1.0 mg/L.

Report result as "> 22 mg/L"

NOTE: If this represents an unusual situation, then a comment should be included on the DMR indicating a non-routine event. Additional dilutions may be required over the next several days until the plant settles down. If, however, this situation occurs regularly, then the lab should be routinely using higher dilutions when preparing samples.

INTRO 5.2.35

Discuss the QC sample types and frequencies required for BOD testing.

Historical: 56%
November 2013: 62 % pass

- **Blanks**: Required on each day of analysis
- **Seed controls**: Required on each day that any samples/QC are seeded
- **LCS(GGA)**: Required at least once per week, OR after every 20 samples (whichever is more frequent)
- **PT samples**: Required for each matrix+technology+analyte combination at least once annually.

NOTE: A "WP" PT is sufficient for BOD.

INTRO 5.2.38

Explain the potential reasons why GGA results could be unacceptably high or low.

Historical: 50%, 87%
November 2013: 62 % pass

HIGH BIAS of GGA results is caused by:

- Nitrification
- Cold GGA solution
- Contamination: Organic matter (yucky stuff)
- Contamination: Microorganisms ("bugs")

NOTE: Contamination from either "bugs" or BOD material alone will cause high bias in GGA but is not likely to cause an exceedance in blanks. There must be contamination from BOTH "bugs" AND waste material for contamination to result in blank exceedances. This explains a common statement from lab analysts that "my GGA is failing high, but my blanks are fine".

LOW BIAS of GGA results is caused by:

- Not enough seed
- Seed materials too weak or variable
- GGA too old or contaminated

INTRO 5.3.2

Identify the critical requirements associated with the drying oven. | 57 |

Historical: 48.5% pass
November 2013: 60% pass

Drying ovens used for TSS determinations must be able to consistently maintain a temperature of 103-105°C. The purpose of the method temperature is to drive off water but not lose volatile solids.

Drying ovens should be vented properly as a health and safety precaution. Do not position a drying oven directly beneath an HVAC blower vent because drafts can be forced through the top of the oven. Direct contact with blowing cold air prevents the ability of the oven to maintain constant temperature. Ovens can be vented to, but should not be situated inside any hood.

Check and document the oven temperature daily when samples are being dried.

INTRO 5.3.3

Discuss the required filters for TSS testing. | 58 |

Historical: 44.1% pass
November 2013: 60% pass

Standard Methods 2540 D requires glass fiber filters without organic binders such as Whatman 934AH or equivalent.

All filter papers are NOT alike! Standard Methods cites the following brands of filters as being equivalent:

- Millipore AP40,
- Gelman type A/E, ED
- Scientific Specialties Grade 161.

Standard Methods 2540D states that, " Practical filter diameters are 2.2 cm [22 mm] to 12.5 cm [125 mm]".

INTRO 5.3.9 Discuss minimum and maximum solids capture weight requirements for TSS testing. | 59 |

2 ?s : 8.8% pass, 37.2% pass

32% passed in Nov 2013

A residue of at least 1 mg, and not more than 200 mg, must be captured. If at least 500 mL of sample volume is filtered and 1 mg of residue is not obtained, the analyst is not required to repeat the analysis using more sample volume.

A minimum capture weight of 1 mg and 500 mL of sample filtered is required to report an LOD of 2 mg/L, the required LOD for Discharge Monitoring Report (DMR) reporting for effluent samples.

Residue amounts greater than 200 mg on a filter can lead to "flash" surface drying and the formation of a salt crust layer that traps moisture beneath it. This can cause sample results to be biased high. This is generally expected to be a problem related to process control samples with heavier solids loading.

INTRO 5.3.9 Discuss minimum and maximum solids capture weight requirements for TSS testing. | 60 |

2 ?s : 8.8% pass, 37.2% pass

Flashcard

MUST capture at least 1 mg, and not > 200 mg solids

A minimum capture 1 mg and 500 mL of sample filtered is required to report an LOD of 2 mg/L,

2 mg/L = the required LOD for Discharge Monitoring Report (DMR) reporting for effluent samples.

WHY? > 200 mg residue can lead to formation of a salt crust layer that traps moisture beneath it. This can cause sample results to be biased high.

INTRO 5.4.9

Historical: 44%, 94% pass

Discuss the symptoms which indicate the need to replace the ammonia probe membrane.

Membrane failure will cause a shift in electrode potential, drift and poor response.

Replace the membrane module if:

- The electrode response becomes very slow
- The results are not reproducible
- The slope becomes too low or shifts
- Visual inspection reveals dark spots or discoloration of the membrane

INTRO 5.4.11

Historical: 35.3% pass
November 2013: 60% pass

Discuss the requirement to distill samples prior to performing ammonia determinations.

All wastewater samples must be distilled. An exemption is provided for domestic municipal wastewater effluents that do not have any significant industrial or waste component that are analyzed using ion selective electrode.

↑ **AND**

also Hach TNT+

INTRO 5.5.3

Historical: 71%, 65%

Discuss critical reagent and standard requirements for total phosphorus testing.

November 2013: 66% passed; 4, 12 and 18% for each possible choice.

- If standards are not purchased, dry chemicals, such as potassium dihydrogen phosphate must be dried at 105°C for at least an hour before preparing standards. This process ensures that bias is not introduced by the absorption of moisture from the air by the dry chemical, thereby increasing its mass.
- The ascorbic acid solution, one component used in preparing the color development solution, must be prepared fresh weekly and stored at 4°C in the dark.
- Combined color reagent, once prepared, must be used within 4 hours or discarded.

INTRO 5.5.7

Historical: 35.3% / 14.7% pass

Discuss critical calibration requirements for the total phosphorus test.

| 64 |

November 2013: 34% passed; 18-36% for each possible choice.

- Lab certification code requires at least three calibration standards plus a calibration blank.

  The more calibration standards used to develop the relationship between phosphorus concentration and absorbance, the more accurate the calibration.
- Calibration standards must bracket sample concentrations.
- Phosphorus calibrations are evaluated using the correlation coefficient which must be greater than or equal to 0.995.

INTRO 5.7.1 Discuss the importance of pH in process control. | 65 |

Historical: 85%, 86% pass

November 2013: 54% passed; 0, 18, 28% for each possible choice.

pH of the biological mass in treatment facilities must be monitored to determine if the levels are too acidic or caustic for the microbes in the plant or the receiving water. Most wastewater plants in Wisconsin have an effluent discharge limit of 6.0 to 9.0 standard units (s.u.) in their permit, but pH levels of 6.8 to 7.2 are optimum for biological activity of most aerobic organisms. Laboratories may use bench-top pH meters for the analysis, using two or more calibration pH buffers that bracket the pH levels measured. The pH meter should be calibrated daily using fresh buffers. There is no holding time for pH analysis, so all analyses for permit use must be done on-site.

INTRO 5.7.1 Discuss the importance of pH in process control. | 66 |

Historical: 85%, 86% pass

November 2013: 54% passed; 0, 18, 28% for each possible choice.

Generally, the lower the pH of wastewater, the greater the disinfection capacity with chlorine compounds, to a point. Below the pH of 4.0, very little disinfection takes place. The higher the pH, the lower the disinfection capability of chlorine. A pH of 5.5 to 7.5 is optimal.

The optimum pH for nitrification is 7.5 to 8.5 su. As ammonia is converted to nitrite and nitrate, alkalinity decreases and pH of the wastewater may drop.

INTRO 6.1.3 Define Bias and Precision

Historical: 53%, 67% pass
November 2013: 64% passed; 8, 10, 18% for each possible choice.

Bias (Accuracy) is the systematic or persistent error in an analysis which results in the expected sample measurement being consistently different than the sample's true value.

A systematic bias is a bias resulting from a flaw integral to the system within which the bias arises (for example, an incorrectly calibrated thermostat may consistently read - *that is, 'biased'* - several degrees hotter or colder than actual temperature). As a consequence, systematic bias commonly leads to systematic errors, as opposed to random errors, which tend to cancel one another out.

INTRO 6.1.3 Define Bias and Precision

Historical: 53%, 67% pass
November 2013: 64% passed; 8, 10, 18% for each possible choice.

Precision is a measure of how closely multiple determinations performed on the same sample will agree with each other.

Accuracy is the degree of closeness to the actual value while precision is the degree of reproducibility.

Using the typical "target" analogy, if a large number of arrows are fired at the target, the size of the arrow cluster on the target represents the shooter's precision. When all arrows are grouped tightly together, the cluster is considered precise since they all struck close to the same spot. Note that the cluster may be very precise, by virtue of a tight grouping, but if the cluster is nowhere near the bullseye, then the shooter is precise but not accurate.

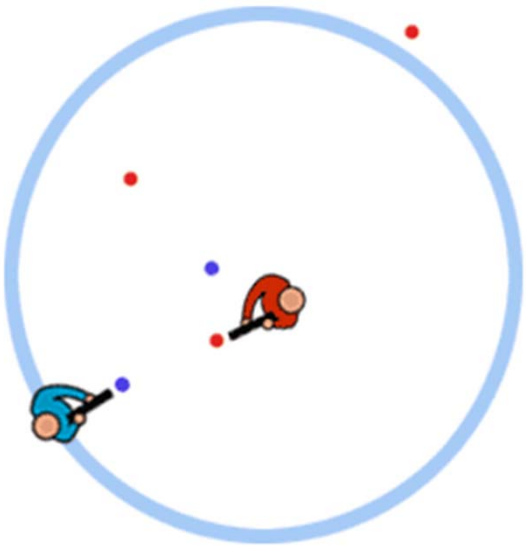
Accuracy v. Precision

- Accuracy: Can I get the right answer
- Precision: Can I get that answer again

Precision in practice

- Measuring precision requires multiple attempts….more than one check. That forces it to relate to duplicates or replicates

Is either accurate? Precise?



| 71 |

Accuracy & Precision recap

- We've tried the target and hunting analogy.
- Let's try something else.

	Accuracy?	Precision?
GGA = 200		
GGA = 160,231,172,225,158		
GGA = 224,226,221,228,222		
GGA = 196,202,199,203,200		

| 72 |

INTRO 6.1.5

Historical: 56%
Nov. 2013: 48% correct.
12%, 12% , and 28% other options

Define the following terms:
A. Arithmetic Mean.
B. Geometric Mean..

A. Arithmetic Mean.

The arithmetic mean (or simply the mean) of a list of numbers is the sum of all the values divided by the number of values.

If four (4) values are 20, 10, 80, and 10,
the arithmetic mean is (20 + 10 + 80 + 10) ÷ 4 = 30.

= 120 /4

INTRO 6.1.5

Historical: 56%
Nov. 2013: 48% correct.
12%, 12% , and 28% other options

Define the following terms:
A. Arithmetic Mean.
B. Geometric Mean..

B. Geometric Mean

The geometric mean of a set of positive data is defined as the 'n'th root of the product of all the values, where 'n' is the number of values. The geometric mean of a data set is always smaller than or equal to the set's arithmetic mean (the two means are equal if and only if all values of the data set are identical).

If four (4) values are 20, 10, 80, and 10, the geometric mean is the 4th root of (20 × 10 × 80 × 10) or the 4th root of 160,000. The 4th root of 160,000 is 20. [Note that the arithmetic mean is 30]

The geometric mean is used for microbiological analysis due to the unpredictability of their exponential growth rate.

INTRO 6.1.9

Historical: 32.4% pass

Define Laboratory Control Standard (LCS)

"Laboratory Control Standard" or "LCS" means a sample of reagent water spiked with known amount of the analyte of interest. The purpose of an LCS is to determine whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements.

In many EPA methods, the term "lab-fortified blank" is substantially equivalent to a laboratory control sample.

INTRO 6.1.10 Define Quality Control Standard (QCS).

Historical: 32.4% pass **November 2013: 60% pass**

Quality Control Standard or "QCS" means a solution or sample containing method analyte of known concentration, AND accompanied by specified analytical acceptance limits, AND obtained from a source external to the lab AND different from the source of calibration standards. These samples are distinguished from PT samples in that the acceptance limits are provided with the sample, rather than after analysis. QCS are used to check either lab or instrument performance.

These types of samples are important indicators of quality because instantaneous feedback on performance is provided. This offers the analyst an opportunity to correct any analytical problems in a timely manner. QCS are analyzed every 4 months as a check on analytical performance. Appropriate measures are taken to investigate problems when the result of a QCS analysis is inconsistent with past results.

Unlike PT samples, whose results are not received for some time after the testing is performed, QCS samples can be used at any time there is concern about the control of a specific analysis. Having immediate access to the validated concentrations allows the analyst to take immediate action to identify and correct the problem.
QCS standards are often referred to as "blind standards". QCS sample analysis is not required for tests in which the lab incorporates second source standards.

From the Advanced Study Guide ADV 6.2.10

QC samples used to assess accuracy:

Accuracy is a measure of the proximity of an unknown to the "true value" or the expected result.

PT (an external unknown standard; goal: determine true value)

QCS (an external known standard; goal: determine true value)

ICV (an internal known standard; goal: determine true value, validate calibration)

LCS (an internal known standard; goal: determine true value)

Matrix Spikes (an internal known addition; goal: recover true value)

Split samples [sent to a contract lab] (goal: determine which lab is correct) [range or RPD] (goal: reproducibility)

INTRO 6.1.18 Define LOD and LOQ.

| 77 |

Historical: 17.7% pass. November 2013 only 40% passed

Limit of detection (LOD) means the lowest concentration or amount of analyte that can be identified, measured, and reported with confidence that the concentration is statistically different from a blank (or, not a false positive value). For department purposes, the LOD approximates the "method detection limit" (MDL, the term used by the EPA and in many methods). The LOD is determined using the procedure in 40 CFR Part 136 Appendix B, Revision 1.11.

Limit of quantitation or (LOQ) means the lowest concentration or amount of an analyte for which quantitative results can be obtained with a specified degree of confidence. The limit of quantitation is typically considered to be a value 10/3 or 3.333 times the limit of detection (LOD).

INTRO 6.1.18 Define LOD and LOQ.

| 78 |

17.7% pass

Flashcard

LOD= Limit of detection ≅ MDL

- =lowest concentration …statistically different from a blank
- Statistically not a false positive value.
- determined by 40 CFR Part 136 App. B, Rev. 1.11.

LOQ= Limit of QUANTITATION ≅ 3.333 x LOD

- = lowest concentration …QUANTITATIVE results at a specified degree of confidence.
- LOD is to be a value 10/3 or 3.333 times the limit of LOD.

INTRO 6.2.10

Discuss what is meant by a "second source" standard and the rationale for using second source standards.

November 2013: 30% pass

Second source standards are actually a form of QCS sample, analyzed more frequently. The greater frequency helps to identify preparation errors made during dilution of stock standards to prepare working standards. It is recommended that stock standards be purchased from each of two different suppliers or different lots of the same solution may be purchased from a single supplier.

- Stock A= primary source used to prepare calibration standards.
- Stock B= secondary source used to verify the prepared calibration standards.

Note that second source standards will also help to identify discrepancies in the concentration of original stock standards. Second source standards are frequently used to prepare QC samples, including laboratory control standards (LCS) and matrix spikes.

INTRO 6.2.10

Discuss what is meant by a "second source" standard and the rationale for using second source standards.

November 2013: 30% pass

If there is no independent verification (such as second source standards) and the same solution that is used to prepare calibration standards is also used to prepare spiked QC samples, errors made in the preparation of the stock standard cannot be easily identified.

Second source standards need only be analyzed any time that a new dilution of a stock standard is prepared which will then be used to prepare calibration standards.

Many labs, however, routinely include a second source standard, by using second source standards to prepare LCS or matrix spikes. This practice provides a much more frequent, independent verification that standard solutions used to generate calibrations are accurate.

INTRO 6.3.3

Historical: 39.5% pass
November 2013: 34% pass
Other choices selected by 8%, 20%, and 38%

Discuss selection of an appropriate spike concentration for determining the limit of detection (LOD).

The best spiking level to determine the LOD, as specified in the EPA procedure, is 1 to 5 times the estimated detection level. An estimated detection level can be obtained from the analytical method referenced or from direct analytical experience. Particularly for ammonia, due to slow response of very low concentration samples, a spike level below 0.2 mg/L is ill advised.

- For ammonia (by the ISE method), a concentration between 0.2 and 0.5 mg/L is appropriate.
- For phosphorus, a spike concentration between 0.1 and 0.2 mg/L should result in a valid LOD.