INTRODUCTION TO LABORATORY MANAGEMENT AND METHOD PERFORMANCE

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OUTLINE

- Laboratory Management
- Method Performance
  - Accuracy and Precision
- Method Precision
- Method Detection Limit

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TAKE HOME MESSAGES – LABORATORY MANAGEMENT

- Communication, documentation, and training are essential to good laboratory management.

- Keep it simple - Try to document the essentials, without recording extraneous information.

- Standards are a useful tool for evaluating analyst, instrument, and reagent performance.

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HOW IS WATER TESTED?

- Steps of Analysis
  - Sample Collection
  - Sample Preparation
  - Use of Standards
  - Procedure
  - Interpretation

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The analysis is only as good as the sample

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LABORATORY MANAGEMENT

- Who should be involved in laboratory management and method performance?

LABORATORY MANAGEMENT

- **Everyone** involved with the lab:
  - Person sampling
  - Person running the test
  - Person washing the glassware
  - Person doing maintenance on the instruments
  - Person interpreting the results

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ANALYST AND USER

- People involved with lab management can usually be categorized in one of two groups:
    - Analysts
    - Users

- Analyst and user could be the same person!

ANALYST AND USER

- Analyst
  - Person or group providing the analytical results

- User
  - Person or group using (managing or interpreting) the analytical results

ANALYST AND USER RESPONSIBILITIES

- Good communication must exist between analyst and user
  - The user must define what information is required.
  - It is the analyst's task to provide required information.

ANALYST CONCERNS

- What do I need for this application?
  - Pretreatment required?
  - Screening test or reporting results?
  - Required sensitivity?
  - Digital instruments or test kits?
- How many samples and how much sample?
- How many tests are necessary?

USER CONCERNS

- How much is it going to cost?
- How long is it going to take to sample and get results?
- How can I realistically balance analytical requirements with resources?
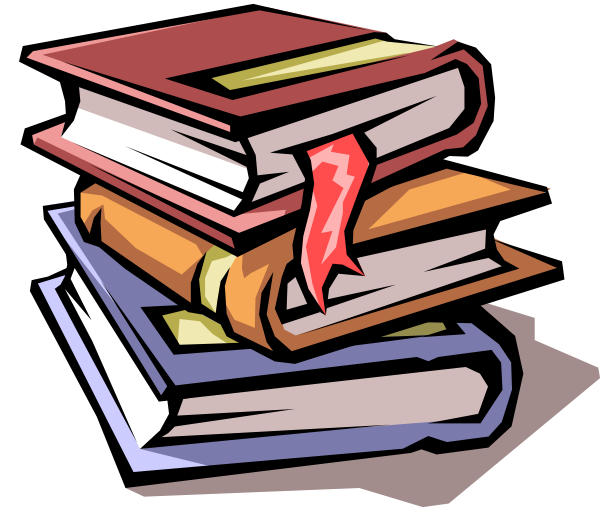
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LABORATORY MANAGEMENT

- Keys to Laboratory Management
  - Communicate
  - Document
  - Train
  - Cross-Train
  - Update

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DOCUMENT AND TRAIN

- Record keeping
- Cleanliness
- Labware
- Maintenance
- Use of standards
- Stability of reagents
- Procedures – Choice and training

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RECORD KEEPING

- A record keeping system (paper trail, chain of custody) should track samples <u>before</u>, <u>during</u>, and <u>after</u> analysis.

- Everyone involved needs to understand and utilize the system.

RECORD KEEPING

- Efficiently process information through lab system while minimizing actual time spent recording data

- Keep it simple!
  - Collect only the information you need

SUGGESTED INFORMATION - SAMPLE

Site	Hayfield Site Influent	Date	04-15-02 8am
Code	HS IN 1	Collected By	Jim S.
Conditions	Sunny, 75F		

Comments:

pH adjusted to <2 with nitric acid

Grab sample

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SUGGESTED INFORMATION - LAB

- Date of analysis

- Laboratory technicians performing the analysis

- Results (including units)

- Analytical comments: based on need to know
  - Dilutions
  - Interferences encountered

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CLEANLINESS

- Labware cleaning procedures should be documented and all persons involved should be trained.

ROUTINE CLEANING PROCEDURE

- Rinse glassware with tap water.
- Clean glassware with a solution of water and laboratory detergent.
- Rinse the glassware with an acidic solution
  - 1.0 N HCl
  - 6N HNO_3 for regulatory reporting of heavy metals
- Rinse glassware at least 3X with DI water.

ROUTINE CLEANING PROCEDURE (CONT.)

- Glassware should be stored in a manner that prevents contamination from dust particles.

- Prior to analysis, rinse the glassware with sample to prevent contamination or dilution.

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ROUTINE CLEANING

- Nitrate/ammonia – do not clean with nitric acid
- Phosphates – use phosphate-free detergent
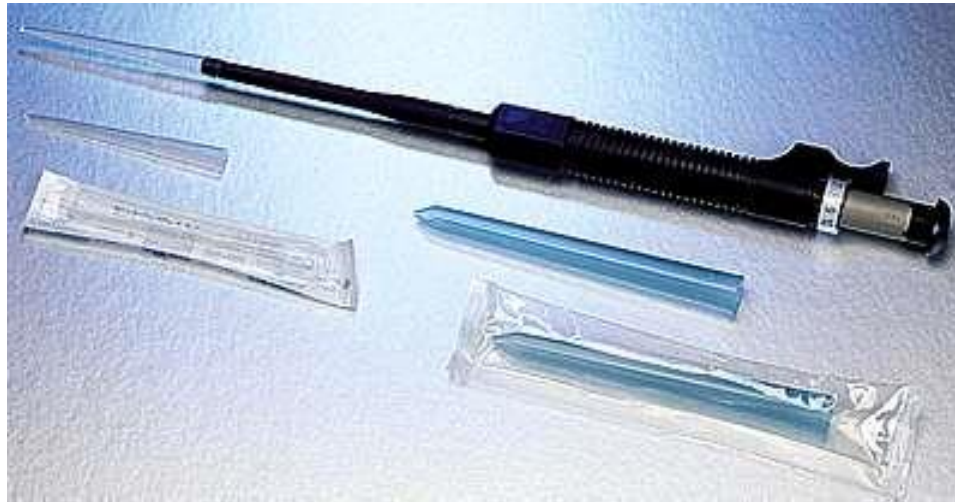 - use Liqui-Nox or hydrochloric acid
- Dedicate glassware

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LABWARE

- Use the highest quality glassware that you can, that best fits your application.
- Dilutions - clean Class A glassware
  - Volumetric flasks
  - Volumetric pipets

LABWARE

- An alternative to Class A glass pipets is an accurate volumetric dispenser such as Hach's Tensette Pipet.

MAINTENANCE

- Preventative maintenance is the key to optimal instrument performance.
  - Follow any maintenance program and guidelines suggested by the instrument manufacturer.
  - Instrument manual

MAINTENANCE

- Check the performance of instruments by using internal diagnostic programs
  - DR/6000, DR/3900 have self-diagnostic check
- Check the condition of analytical system (instruments, reagents and technique) with standards.

STANDARDS

- How are standards used?
    - Instrument calibration
    - Instrument verification/accuracy check

CALIBRATION

- Hach instruments - built-in calibration curves, not necessary to calibrate
- Instrument <u>without</u> preprogrammed calibration curves
  - Prepare curve daily - OR
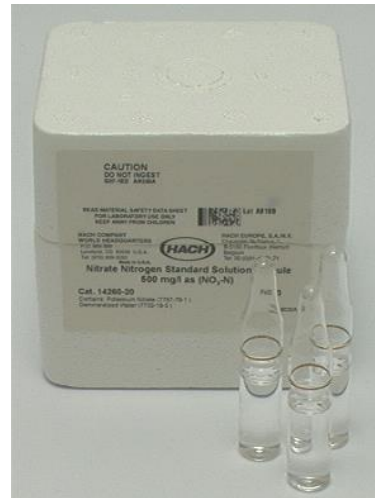 - Whenever a new lot of reagents is prepared

STANDARDS

- Standard solution – Am I running the test correctly?
  - Verifies instrument, technique, and reagents
  - Control charts

STANDARDS

- Standard additions – Is my sample compatible with the test?
  - Identifying interferences and percent recovery

REAGENT STABILITY

- Running a standard can help assess reagent performance.
  - Reagents should be checked routinely with a standard to insure that they have not deteriorated.
  - You can't always tell by the expiration date
    - Storage conditions

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REAGENT STORAGE

- Reagents should be stored properly
  - Maximum shelf life depends on storage in a cool, dry location (refrigeration necessary if indicated on the packaging)

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PROCEDURES

- Be sure that the correct procedure is chosen for:
  - Analytical range and necessary precision
  - Sample type
  - Regulatory acceptance
  - Chemical form

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PROCEDURES

- Procedures should be:
    - Understood and followed <u>exactly</u> by all technicians involved.
    - Based on sound chemical principles.
    - Be safe for the technicians performing the test.

PROCEDURES

- Practice new procedures using <u>standard solutions</u> in order to verify the analytical system.
  - Train and instill confidence in the technicians.
- If interferences are suspected, run a <u>standard additions</u>.

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OUTSIDE LAB COMPARISONS

- Confidence comes from within - not by comparison to outside labs.
- If you compare with outside labs, remember:
  - Paying for results doesn't necessarily make them accurate.
  - A true comparison means the same test is being run on the same sample.
  - 3 different labs could see greater than +/- 25% in results.

TAKE HOME MESSAGES – LABORATORY MANAGEMENT

- Communication, documentation, and training are essential to good laboratory management.

- Keep it simple - Try to document the essentials, without recording extraneous information.

- Standards are a useful tool for evaluating analyst, instrument, and reagent performance.

METHOD PERFORMANCE

TAKE HOME MESSAGES – METHOD PERFORMANCE

- Accept the fact that analytical errors happen.

- Know and control the amount of error in measurements so it can be taken into account when making decisions.

- High quality measurements are possible with attention to detail and technique.

WHY TEST WATER?

- To answer a question
  - Am I in compliance?
  - Is my process in control?

- Enough data is required so numbers can be accurately compared with historical data or MCL.

WHY TEST WATER?

- The measurement, together with consideration of any other relevant factors, is often the basis for decision making.
  - Accuracy is essential

ACCURACY IS ESSENTIAL

- Factors that could influence accuracy should always be carefully considered.
  - Representative sample, sample interferences, equipment quality, correct procedure, number of people involved
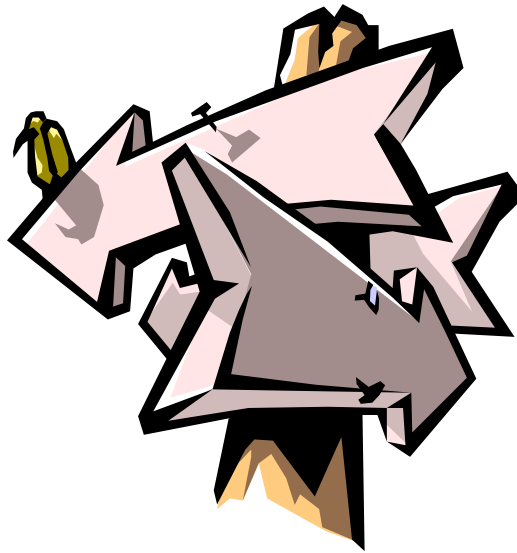- The accuracy of analytical results is a primary issue in any analytical program.

ERRORS HAPPEN

- Error – Difference between analytical results and the true concentration.
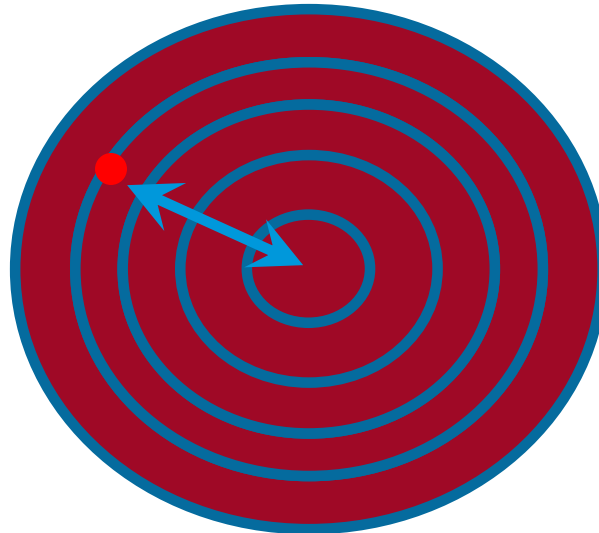
ERRORS HAPPEN

- Analytical error affects the validity of any decisions made on the basis of the results.

ERRORS HAPPEN

- In a perfect world, every analytical result would always be equal to the true concentration.

- This is impossible to achieve!

ERROR HAPPENS

- Since there's no way to avoid it – accept the fact that error happens!

IF YOU CAN'T BEAT THEM…..

- Since error can't be avoided in chemical analysis, there are a few ways to work with it:
    - Minimize error to ensure meaningful results
    - Be sure the magnitude of error is known, controlled, and quantified
    - Take error into account when decision-making

CHLORINATION – AN EXAMPLE

- To ensure adequate residual at the tap, water must leave your plant with 2.00 – 2.50 mg/L chlorine.
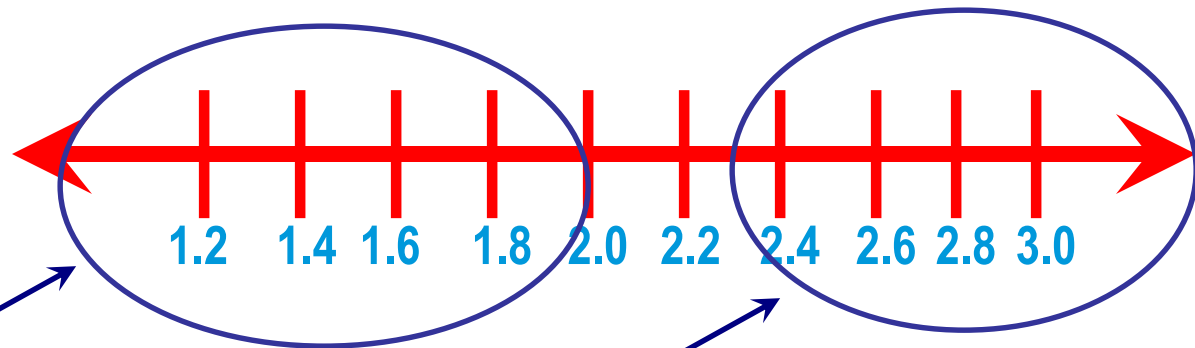
CHLORINATION – AN EXAMPLE

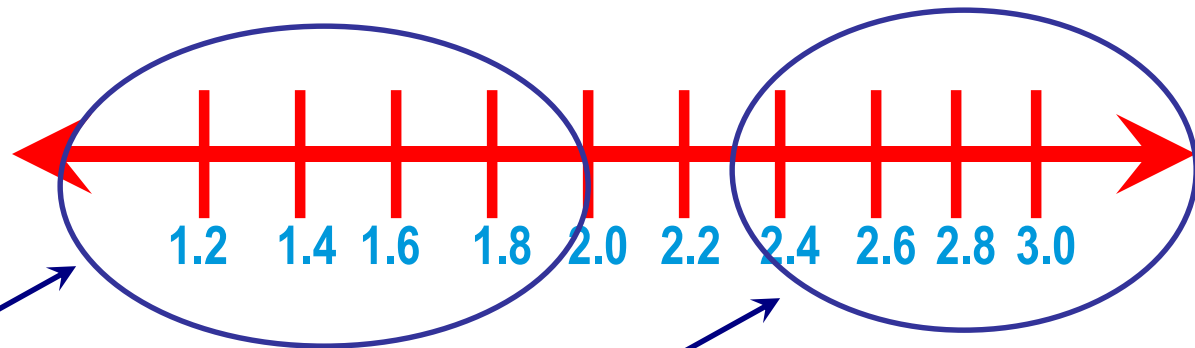- Duplicate analyses of your effluent indicate 1.3 and 1.4 mg/L chlorine.

1.2 1.4 1.6 1.8 2.0 2.2 2.4 2.6 2.8 3.0

Increase Feed

Decrease Feed

CHLORINATION – AN EXAMPLE

- Increasing precision will not affect the decision-making process.

1.2   1.4 1.6   1.8 2.0 2.2   2.4   2.6 2.8 3.0

Increase Feed

Decrease Feed

DECHLORINATION – AN EXAMPLE

- Dechlorinated wastewater effluent is discharged into a wetland and must be dechlorinated to less than 0.026mg/L.

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DECHLORINATION – AN EXAMPLE

- Results of two chlorine tests are:
  - 0.02 and 0.03mg/L

   (remember limit is 0.026mg/L)

DECHLORINATION – AN EXAMPLE

- A more precise test is necessary in order to make an accurate treatment decision!

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DIGITS VS. DECISIONS

- When choosing a method and collecting data, consider how accurate and how close the results must be in order to make a correct decision.
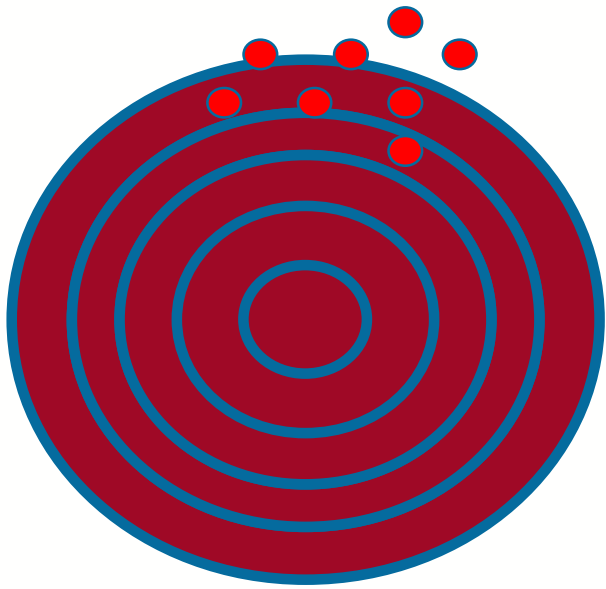
WHAT IS ACCURACY?

- <u>Accuracy</u> is the nearness of a test result to the true value.
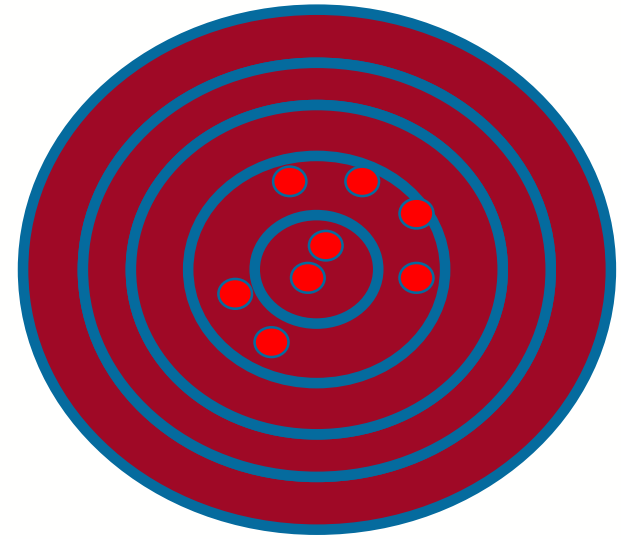

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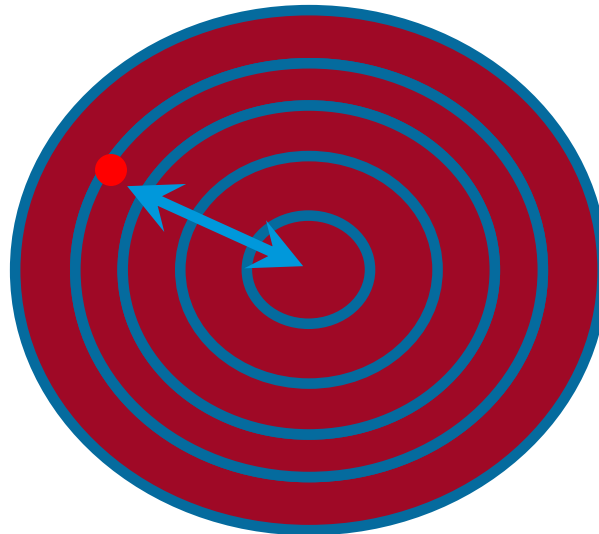
WHAT IS PRECISION?

- Precision is how closely repeated measurements agree with each other.

- Although good precision suggests good accuracy, precise results can be inaccurate.
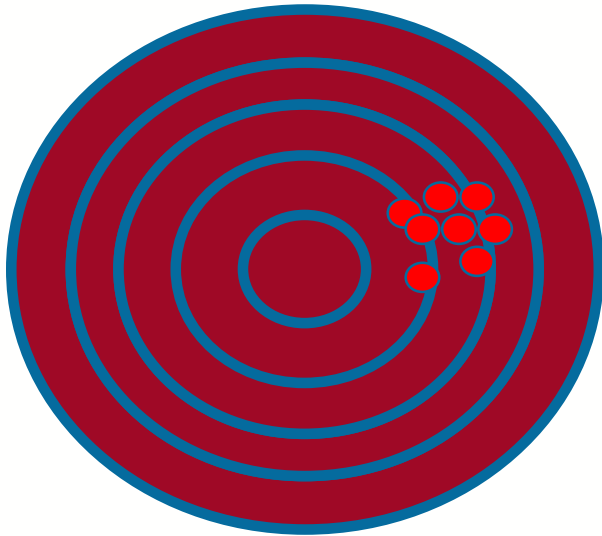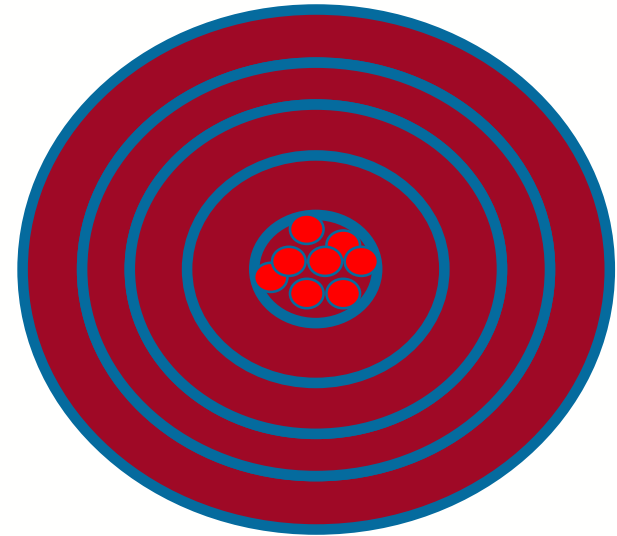
Imprecise and inaccurate

Accurate but imprecise

Precise but inaccurate

Precise and accurate

MEASURE TWICE, CUT ONCE

- Don't make judgments based on one analysis!
- Run multiple tests and get an average.
  - The amount of variation in those value gives you an idea of the precision.

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WHAT ARE THE SOURCES OF UNCERTAINTY?

- Systematic Error
- Random Error

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SYSTEMATIC ERRORS

- An error that is repeated for every measurement, causing bias in the same direction.

  – Reagent blank can cause consistently high results

  – Pipet that is out of calibration and dispenses low volume

  – Balance out of calibration and weighs high

RANDOM ERRORS

- Errors that are different for each test - add either positive or negative bias.

- Random errors result due to variation in technique
  - Washing glassware, dust on glassware
  - Rinsing sample cell
  - Improper use of pipet or TenSette
  - Monday morning or Friday afternoon syndrome

ACCURACY

- High quality measurements <u>are</u> possible with attention to detail and technique.

MEASUREMENT ISSUES

- Instrument

- Procedure

- Preparation

- Reagents

- Technique

- Interferences

INSTRUMENT

- Can the instrument do what I want it to?

- What is the current condition of the instrument?
  - Wavelength accuracy
  - Noise
  - Stray light
  - Absorbance check: tests the lamp, monochromator and photodetector as a system

PROCEDURE (METHOD PERFORMANCE)

- Be sure the procedure is correct for:
  - Analyte
  - Analysis range
  - Precision and sensitivity required

METHOD PERFORMANCE

- Determining the Method Detection Limit (MDL)
- Determining the Sensitivity
- Determining the Precision
- Using Control Charts

TAKE HOME MESSAGES – METHOD PERFORMANCE

- Accept the fact that analytical errors happen.

- Know and control the amount of error in measurements so it can be taken into account when making decisions.

- High quality measurements are possible with attention to detail and technique.

METHOD PRECISION

# TAKE HOME MESSAGES – PRECISION AND CONTROL CHARTS

- Every method has some degree of variability in measurements.
- The degree of variability in a method can be quantified by calculating method precision.
- Measurement precision can be visualized and controlled using a control chart.

PRECISION

- <u>Every</u> measurement has some degree of uncertainty.

PRECISION

- Chemical measurements have some degree of uncertainty, similar to the way a ruler with 1/16" markings leaves some doubt as to the exact length.
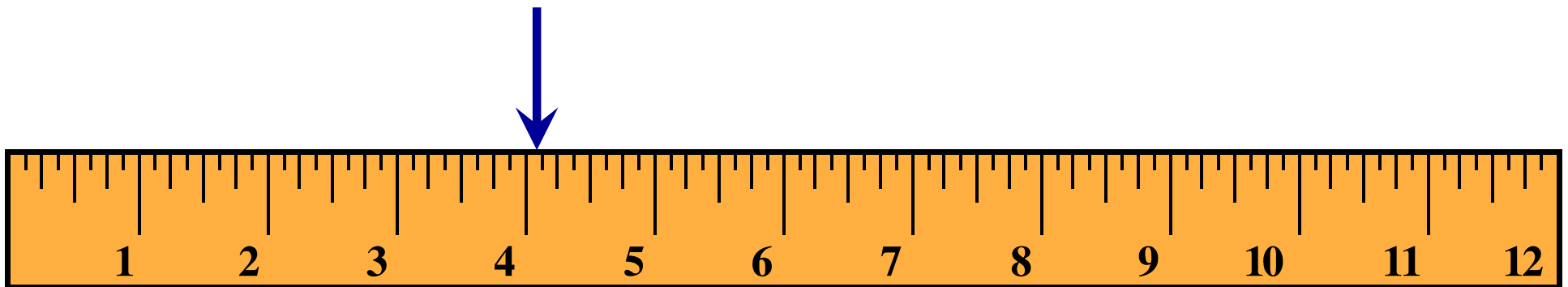
PRECISION

- Precision is:
  - An estimate of the average response variation.
  - The 95% confidence interval for the stated concentration.

PRECISION

- ## 95% Confidence Interval (2s)

  - Any <u>single</u> reading may fall outside of the range, but the average of several readings should fall within the range <u>95 times out of 100</u>.

- ## These values hold only for a DI water matrix

  - Ranges may vary depending on the sample matrix.

PRECISION LABORATORY PROCEDURE

- Analyze 7 replicates of a 1.000 mg/L iron standard and record results

- Calculate the mean and standard deviation

- The 95% confidence interval is determined from 2s

METHOD DETECTION LIMIT (MDL)

TAKE HOME MESSAGES - MDL

- There is a finite lower concentration limit to every chemical analysis method.

- The lower limit of a test can be quantified by determining the method detection limit for a particular method and analyst.

- Precision, MDL, and sensitivity are all factors which affect your choice of analytical methods.

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METHOD DETECTION LIMIT

- USEPA defines MDL as the minimum concentration that can be determined with 99% confidence that the true concentration is greater than zero.

METHOD DETECTION LIMIT

- MDL varies from analyst to analyst.
  - Each analyst must determine their own MDL based on their own unique operating conditions.
- MDL does not account for variations in sample matrix and can only be achieve under ideal conditions.

METHOD DETECTION LIMIT

- An idea of the estimated detection limit (EDL) is required in order to determine MDL.
- EDL – the upper 99% confidence limit for zero concentration based on calibration data used to prepare a calibration curve.
    - Many Hach procedures contain EDLs.

MDL DETERMINATION

- Estimate (or look up) the detection limit.

- Prepare a laboratory standard of the analyte in DI water that is 1-5 times the EDL.

- Analyze at least 7 portions of the standard and record each result.

MDL DETERMINATION

- Calculate the mean and standard deviation of the results.

- Compute MDL

  – MDL = Student's "t" x standard deviation

  – Student's "t" is obtained from a statistical table.

EXAMPLE – MDL DETERMINATION

- Method – FerroZine Iron method
- EDL = 0.003 mg/L (from procedures manual)
- Prepare 1 liter of 0.010 mg/L standard (1-5X EDL).
- Analyze 8 replicates of standard and record results.

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WHAT IS SENSITIVITY?

- Sensitivity is quantified as the change in concentration for a 0.010 change in absorbance.

TAKE HOME MESSAGES - MDL

- There is a finite lower concentration limit to every chemical analysis method.

- The lower limit of a test can be quantified by determining the method detection limit for a particular method and analyst.

- Precision, MDL, and sensitivity are all factors which affect your choice of analytical methods.

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FOR MORE INFORMATION…..

- "Standard Methods for the Examination of Water and Wastewater" is an excellent source to begin a QA/QC program in your laboratory.

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