Laboratory Quality Control, Statistics, and Measurement Uncertainty

Est. Length: up to 6 hours

Objective Review

- EO 1.1 Define quality assurance.
- EO 1.2 Describe the elements of a laboratory quality assurance plan.
- EO 1.3 Discuss the importance of a laboratory mission statement.
- EO 1.4 Explain how laboratory safety is incorporated in a quality assurance plan.

Objective Review

- EO 2.1 Explain what a quality control program is and why it is important.
- EO 2.2 Define and apply the following terms associated with a laboratory quality control program:

  Quality Control Samples, Mean, Standard Deviation, Control Charts, Method Blanks, Relative Percent Deviation, Matrix Spike and Percent Recovery
- EO 2.3 Given a data set, calculate the mean and standard deviation.

Objective Review

EO 2.4 State how confidence levels are used to produce a quality control chart.

EO 2.5 Given a data set, create a quality control chart showing the 2 and 3 sigma data lines.

EO 2.6 Given a quality control chart, identify out of limit data.

EO 2.7 Identify and differentiate random error and systematic error.

EO 2.8 Given a quality control chart, identify and differentiate shift and trend in a data plot.

EO 2.9 Describe the ways in which a laboratory technician is involved in a laboratory control program.

Objective Review

- EO 3.1 State why radiochemistry measurement uncertainty is calculated differently.
- EO 3.2 Describe what factors may be included in a radiochemistry uncertainty calculation.
- EO 3.3 Describe how radioactive measurement uncertainty can be improved.

Quality Assurance

- QA ensures the desired level of quality is obtained
- QA vs. QC
  - QC is only a part of QA
- QA also includes:
  - Mission Statement
  - Organization
  - Training
  - Safety

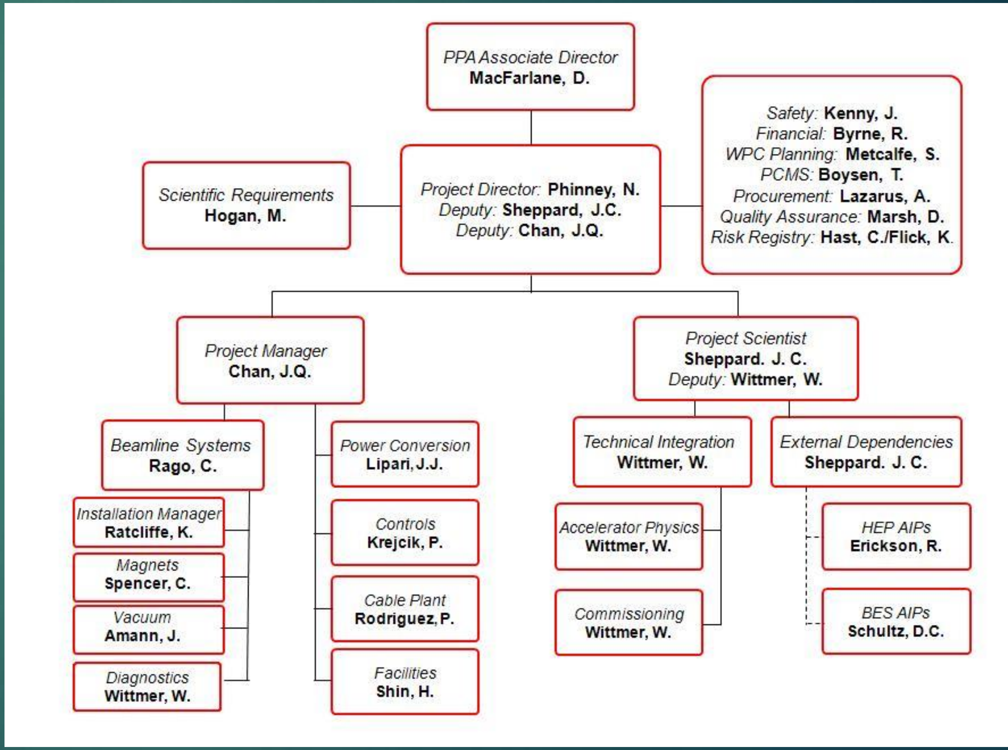
Mission Statement

- The Who and What of the organization
- Establishes the culture and ethics
- "It is Saybolt's mission to provide a fast, accurate, professional, independent and efficient service in a manner that reflects total commitment to today's most exacting standards."

From http://www.corelab.com/saybolt/mission-vision

Typical Organizational Chart

- Who is Who?
- Where you are and Where you Want to go…..
- Responsibility



Lab Safety


Lab Safety
don't touch the animals
wear safety goggles
wear lab coat
wear gloves when necessary
don't eat at your workstation
clean up your workspace
Anyone not following the rules will be denied access to the lab room

Assessments

- Internal and External
- Sometimes required
- Ensures quality and integrity

QC Program

- Set of procedures and processes
- Several methods
  - Training and Procedures
  - Standards
  - Calibrations
  - Data Confidence Measures
  - Assessments
- Labs must produce accurate, consistent, and reliable data for customers.

QC Samples

- Analysis typically done in "batches" which include:
  - Several samples
  - Method Blank
  - QC standard
  - Duplicate
  - Spike
- Data from QCs is use to ensure accuracy and precision

Mean

- Mean = Average
- Sum of data/# of data points

$$\overline{X} = \frac{\sum X}{N}$$

What is the mean of the following data sets?

- Data Set 1: 165, 145, 152, 135, 128, 146, 146
- Data Set 2: 4.1, 4.2, 5.0, 4.4, 4.0, 5.1, 3.9, 4.0
- Data Set 3: 11.25, 11.68, 11.00, 10.86, 12.10, 10.91, 11.36, 11.50, 12.10, 11.42, 11.53, 10.98

Standard Deviation

Also known as precision

- How close together are they?
- Are they consistent?
- What should the next value be?

Data should be from same source using same technique

Provides the 1 sigma (σ) value

$$S = \sqrt{\frac{\Sigma(X - \bar{X})^2}{N}}$$

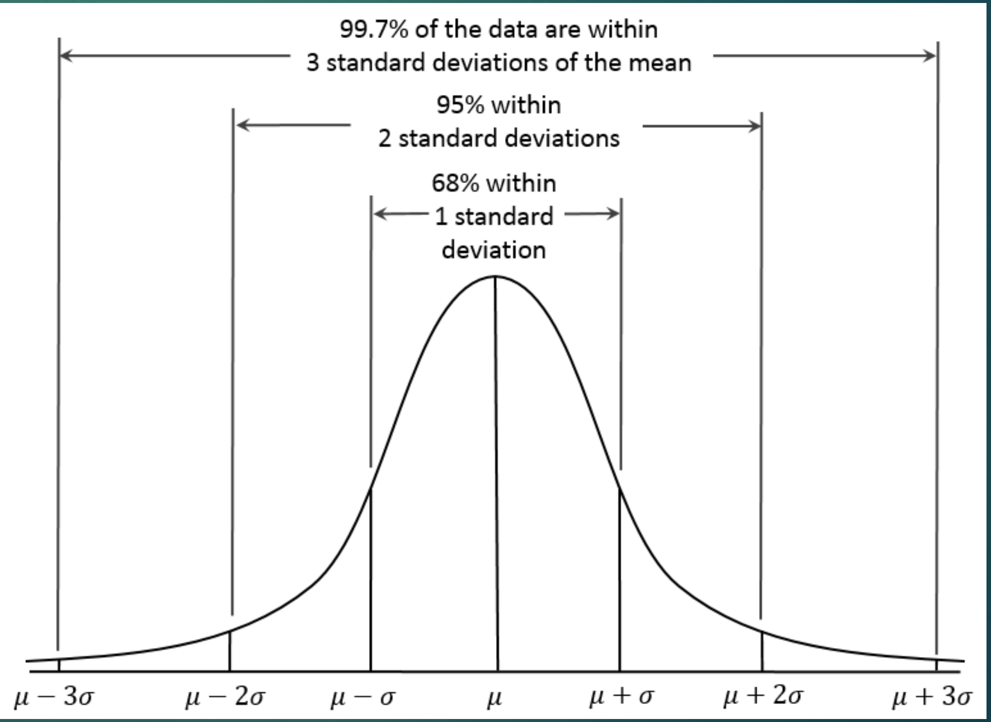
where S = the standard deviation of a sample,
Σ means "sum of,"
X = each value in the data set,
$\bar{X}$ = mean of all values in the data set,
N = number of values in the data set.

What is the standard deviation of the following data sets?

- Data Set 1: 165, 145, 152, 135, 128, 146, 146
- Data Set 2: 4.1, 4.2, 5.0, 4.4, 4.0, 5.1, 3.9, 4.0
- Data Set 3: 11.25, 11.68, 11.00, 10.86, 12.10, 10.91, 11.36, 11.50, 12.10, 11.42, 11.53, 10.98

Confidence Levels

- 2σ and 3σ values
- Used to establish control limits
- Normal distribution
  - 68% within 1σ
  - 95% within 2σ
  - 99.7% within 3σ
- Also called Gaussian



What are the 2σ and 3σ confidence levels for the following data sets?

- Data Set 1: 165, 145, 152, 135, 128, 146, 146
- Data Set 2: 4.1, 4.2, 5.0, 4.4, 4.0, 5.1, 3.9, 4.0
- Data Set 3: 11.25, 11.68, 11.00, 10.86, 12.10, 10.91, 11.36, 11.50, 12.10, 11.42, 11.53, 10.98

Sample Batching

- Samples run with other samples
- Batch includes samples and QC samples necessary
- 10 or 20 samples per batch
- Along with samples, batch can include:
  - method blank
  - QC sample
  - duplicate
  - matrix spike
  - Others (e.g. Matrix Spike Duplicate, Background, Reagent Blank)

Method Blank

- Run along with other samples
- Usually one per batch
- Detects loss of sample or cross-contaminaton
- Limits vary, but usually <10% of highest sample

Duplicate

- Measured in Relative Percent Deviation (% RPD)
- Precision
- 1 per batch
- Why is this important?

Duplicate Calculation

$$\% RPD = \left(\frac{|X1 - X2|}{\bar{X}} \right) \times 100\%$$

where:

X1 = assayed value of a given nuclide from the first measurement
X2 = assayed value of a given nuclide from the duplicate measurement
$\bar{X}$ = mean value of X1 and X2.

- Given the below, what is the % RPD?

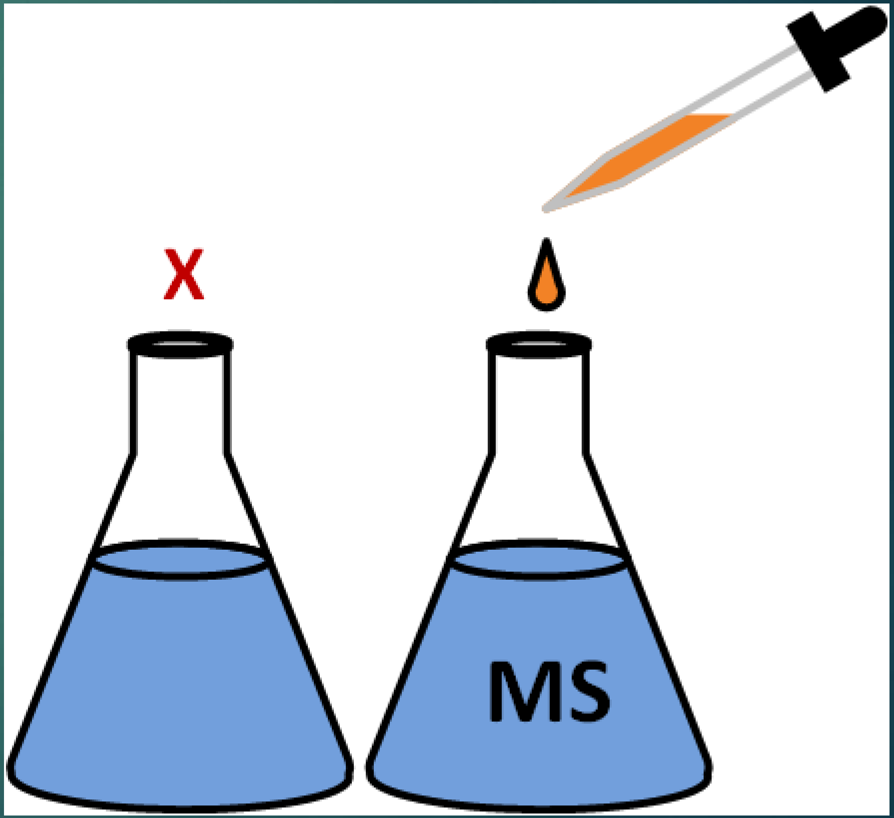
Sample = 1.35E-03 uCI/mL

Duplicate = 1.41E-03 uCi/mL

- Is this acceptable?

Matrix Spike

- Known amount added to sample
- Looking for recovery of spiked amount



Matrix Spike Calculation

- *% Spike Recovery =*
- *[(Spiked Sample Result - Unspiked Sample Result) * 100%] / [Amount of Spike Added]*

- Given the below, what is the %Recovery?

Original Sample = 124 ppm

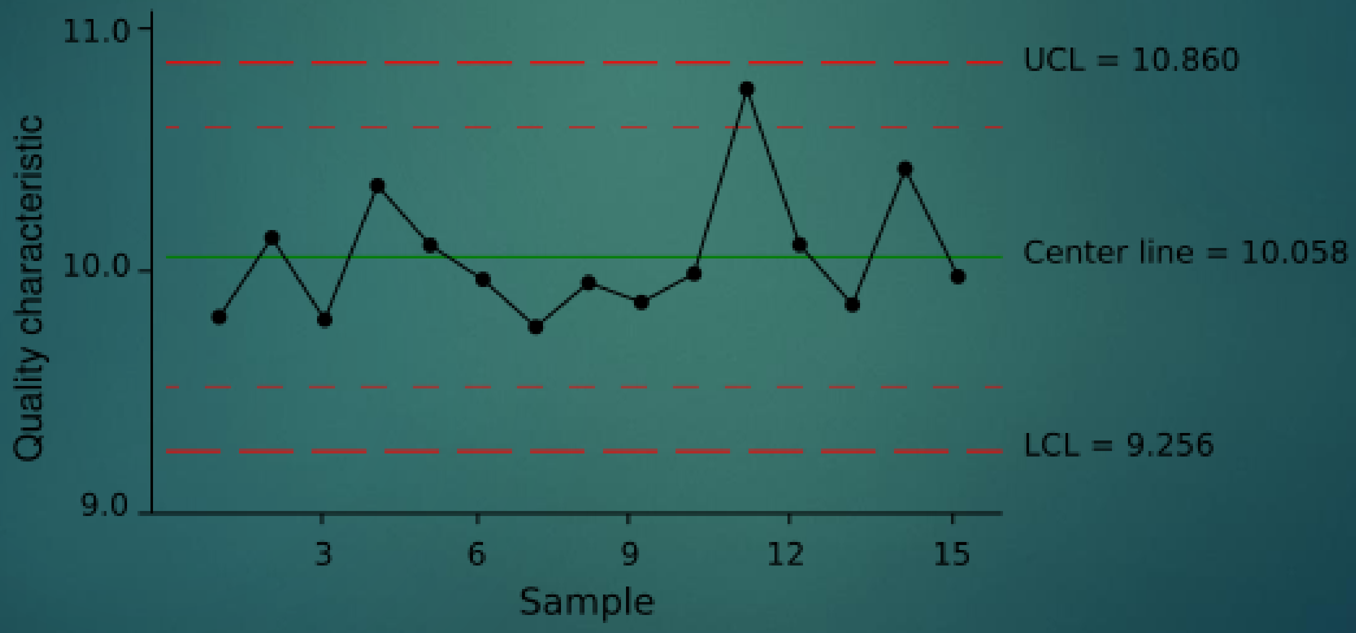
Spike Sample = 145 ppm

Spike Added = 25 ppm

- Is this acceptable?

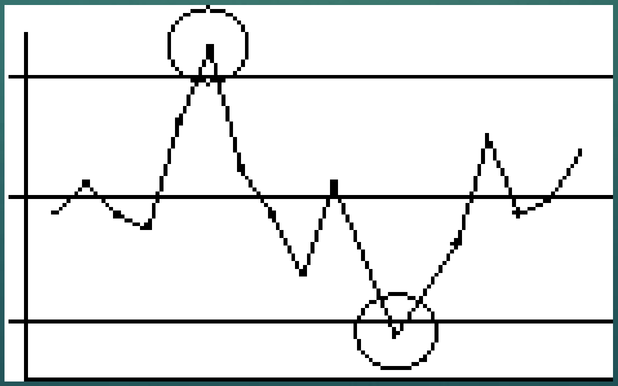
Typical Control Chart

- Identifies mean and confidence levels
- Specific for a measurement
- Used to track performance from day to day (or time to time)



Analysis Error: Random

- Random error is expected
- Always present
- Unpredictable
- Follows normal distribution (bell curve)
- Out of control
  - Consecutive points between 2σ and 3σ on same side of mean
  - Any point outside 3σ
- Back in control
  - Two consecutive acceptable results

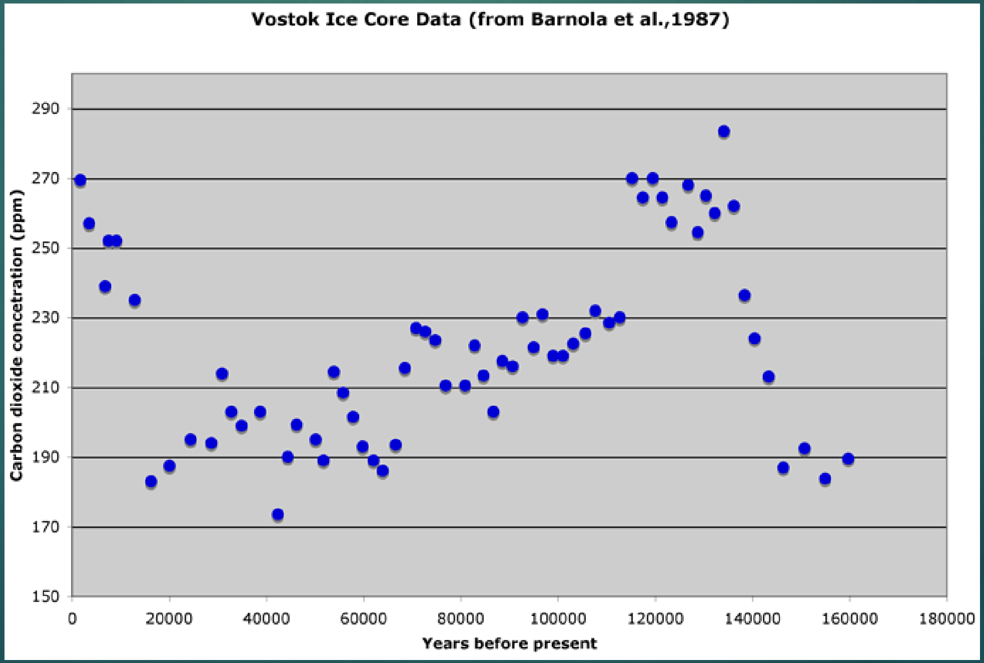


Analysis Error: Systematic

- Predictable
- Constant
- Easily viewable on a control chart
- Can (and must) be corrected
- Trends and Shifts

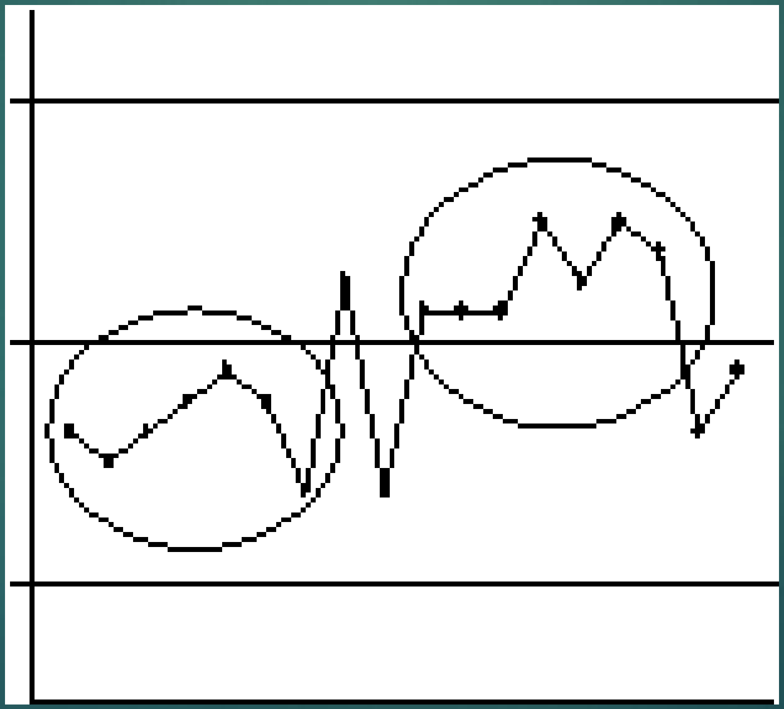
Trend

- Subtle
- Increasing or decreasing
- 7 or more points in same direction (usually but not necessarily consecutive)
- Out of Control



Shift

- Abrupt
- Sometime dramatic
- 7 or more points above or below the mean



Laboratory Technicians

- Con Ops
  - Procedure Process
- Well-trained
  - OJT in three steps
- Troubleshooting

Measurement Uncertainty

- Remember random error?
- Not always true for radiochemistry labs
  - Additional errors to account for
- One time analysis without a standard to follow
  - Multiple dilutions
  - Human performance errors
  - Variable count times
  - Error in radiochemistry counting
- Total propagated error is a better indicator of true error.

Total Propagated Error

Error_Analysis_Review.pdf

Propagation of Error

Most physical quantities cannot be measured in a single direct measurement. Instead, they are calculated from quantities measured in different steps. For example, to measure the density of a substance one would measure its volume, its mass, and then calculate the density (ρ) from the definition:

$$\rho = \frac{mass}{Volume} = \frac{m}{V}$$

To calculate the uncertainty in the density, one must first determine the uncertainty in the measured quantities, m and V, and **propagate** through the calculation.

Suppose that x, …, z are measured with uncertainties δx, …,δz, and the measured values are used to compute a function $q(x, \ldots, z)$. If the uncertainties in x, …, z are independent and random, then the uncertainty in q is

$$\delta q = \sqrt{\left(\frac{\partial q}{\partial x}\delta x\right)^2 + \ldots + \left(\frac{\partial q}{\partial z}\delta z\right)^2}$$

In this equation, the term $\frac{\partial q}{\partial x}$ represents the partial derivative of q with respect to x. If you need help calculating partial derivatives please is Appendix II or a general calculus book. You will need to be able to calculate partial derivatives in order to understand the reminder of this section.

Questions??

Review

- Laboratory QC provides the basis for acceptable results.
- We will review the learning objectives to ensure understanding.