Interpreting Laboratory Data as a Non-Chemist

Presentation to AWMA Young Professionals Workshop

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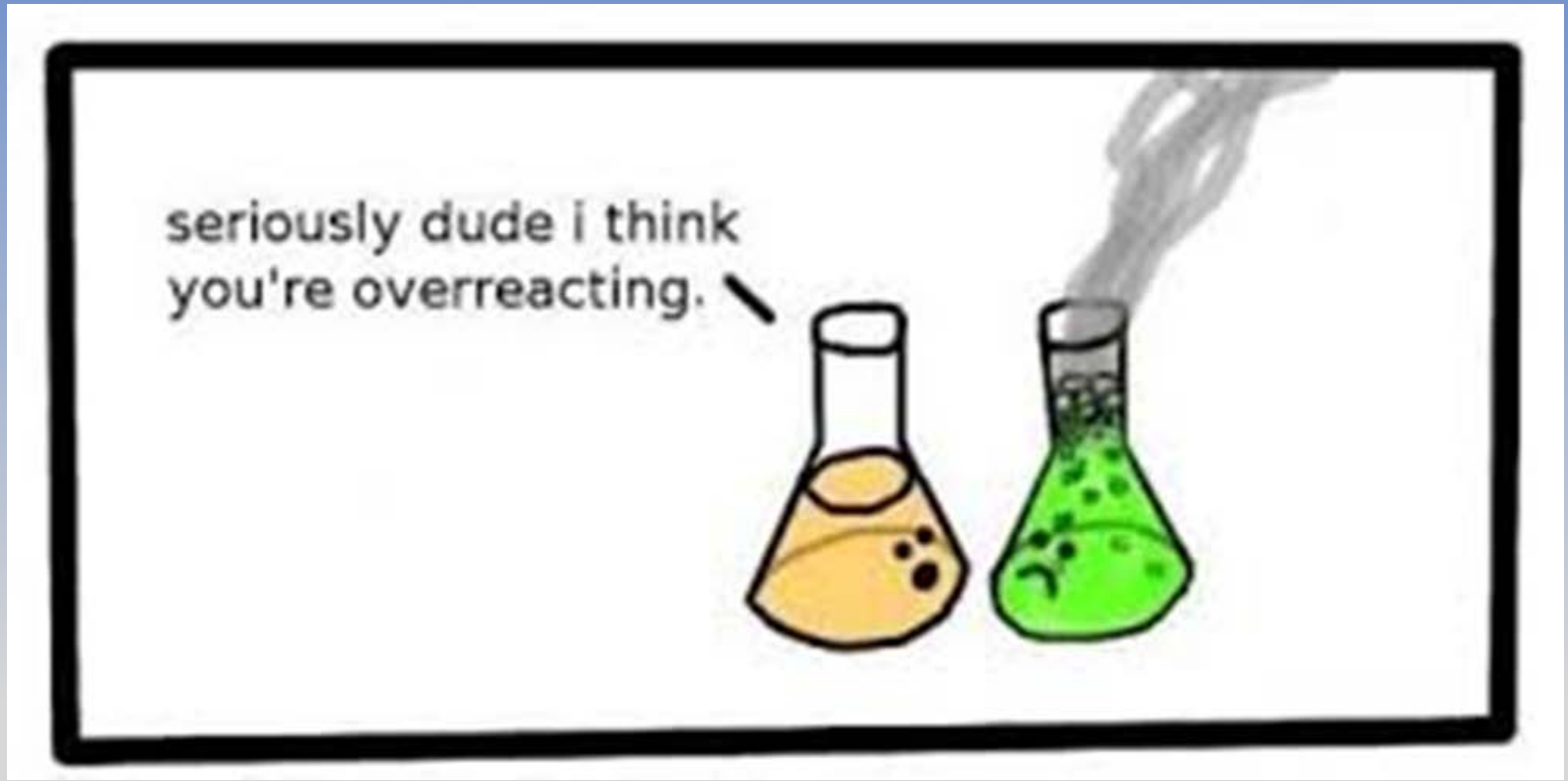
Let’s Avoid This!



Glossary of Terms

- Method Blank – A clean reference matrix sample (i.e., reagent water or purified sodium sulfate) spiked with internal standards and surrogate standards that is carried throughout the entire analytical procedure. The method blank is used to define the level of contamination associated with the processing and analysis of samples.
- Trip Blank – A blank used to provide information about contaminants that may be introduced during sample transport.
- Field Blank – A blank used to provide information about contaminants that may be introduced during sample collection.
- Rinsate/Equipment Blank: A sample of analyte free water poured over or through decontaminated field sampling equipment prior to the collection of environmental samples. Used to assess the adequacy of the decontamination process.

Glossary of Terms

- Laboratory Control Sample (LCS) – A matrix spiked at a known concentration. LCSs are analyzed using the same sample preparation, reagents, and analytical methods employed for the EPA samples received.

Glossary of Terms

- Matrix Spike (MS) – Aliquot of the sample spiked with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.
- Matrix Spike Duplicate (MSD) – A second aliquot of the same sample that is fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to determine precision of the method.
- Relative Percent Difference (RPD) – The relative percent difference is based on the mean of the two values, and is reported as an absolute value (i.e., always expressed as a positive number or zero).

Glossary of Terms

- Internal Standards – Compounds added to every volatile and semivolatile standard, blank, sample, or sample extract, including the Laboratory Control Sample (LCS), at a known concentration, prior to analysis. Internal standards are used to monitor instrument performance and quantitation of target compounds.
- Surrogates (Surrogate Standard) – For organic compounds, a similar organic compound is added to every blank, sample [including Laboratory Control Sample (LCS)], Matrix Spike/Matrix Spike Duplicate (MS/MSD), and standard; used to evaluate analytical efficiency by measuring recovery. Surrogates are compounds not expected to be detected in environmental media.

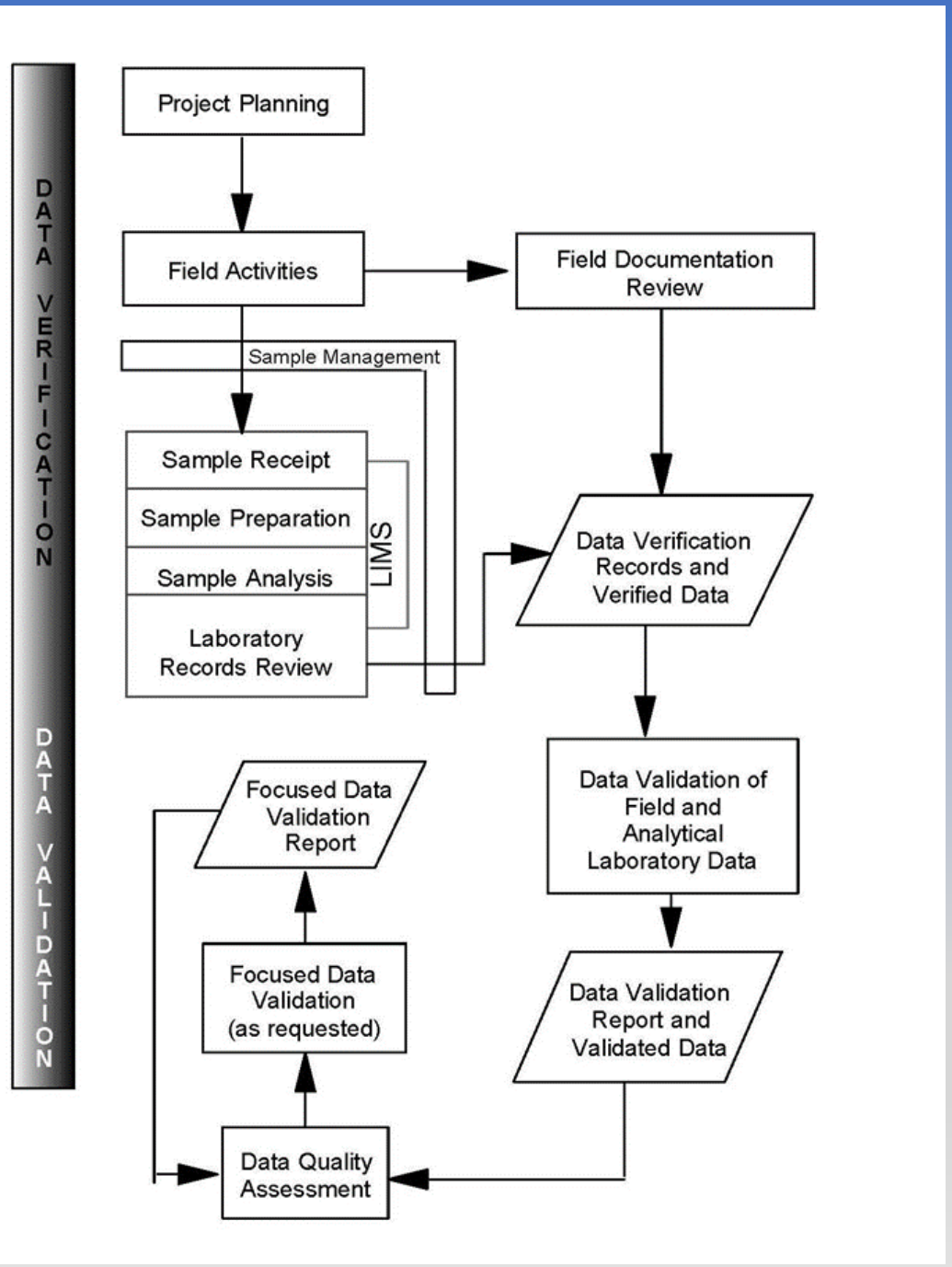
Before Requesting Laboratory Services

- Data Quality Objectives (DQOs)
  - Parameters
  - Methodologies (e.g. – 6010 vs. 6020)
  - Reporting limits
  - Regulatory Considerations (MCLs, RSKs, DTLs, RSLs)
  - QA/QC Requirements (e.g. Level II, Level III, Level IV)
  - Field Quality Control Samples – duplicates, blanks, site-specific MS/MSDs
  - Sampling Procedures
  - Field Sheets, Field Notes, Chain-of-Custody

Communication

- Communicate with the Laboratory Contact!
  - Don’t assume lab will automatically achieve all required DQOs
  - Review laboratory log-in confirmation emails
  - Let laboratory know if there are errors

- Communicate with Field Staff!
  - Don’t assume field staff will automatically understand Work Plan/QAPP
  - Encourage field staff to contact you from field if they have questions


DATA VERIFICATION
DATA VALIDATION
Project Planning
Field Activities
Field Documentation Review
Sample Management
Sample Receipt
Sample Preparation
Sample Analysis
Laboratory Records Review
LIMS
Data Verification Records and Verified Data
Data Validation of Field and Analytical Laboratory Data
Data Validation Report and Validated Data
Focused Data Validation Report
Focused Data Validation (as requested)
Data Quality Assessment

Laboratory Report Received, Now What?

Complete Cursory Review ASAP

- Samples
- Issues with CoC(s)
- Field QC (duplicates, field, trip, and/or rinsate blanks)
- Parameters (requested compound lists)
- Sample holding time/preservation issues
- Reporting Limits (dilutions, etc.)
- Permit Limit Exceedances
- Regulatory Exceedances
- Major QA/QC Issues (rejected data, holding times, blank contamination)

Potential QA/QC Issues Identified?

- Talk to Field Staff
- Contact the Laboratory
- Is Resampling Required?
- Can the laboratory rerun the sample?

Trouble Shooting

- Incomplete Parameter List
  - Contact laboratory – often can reissue report with missing parameters if method already analyzed (e.g. missing VOCs or metals)
- Holding Time Exceedances
  - Determine nature of exceedance/how far out of hold
  - Parameter dependent
  - Compare with historical data, if available

Trouble Shooting Continued

- Inadequate Preservation
  - Determine likely cause of inadequate preservation (e.g. highly buffered leachate sample)
  - Parameter Dependent
    - Unpreserved VOCs 7-day vs Preserved VOCs 14-day holding time
    - Method substitution: Nitrate/nitrite vs Nitrate
  - Compare with historical results

In-Depth Review of Data Results

- Reporting Limits (dilutions, etc.)
- Method Blanks
- Laboratory Duplicates
- Laboratory Control Samples
- Matrix Spike Samples
- Surrogates
- Other Laboratory QC (calibration, internal standards, etc.)

Guidance Documents

- **U.S. EPA, August 2014, National Functional Guidelines for Inorganic Data Review.**

- **U.S. EPA, August 2014, National Functional Guidelines for Organic Data Review.**

Analytical Reporting Limits

There are two distinct types of reporting limits: detection and quantitation

Method Detection Limit (MDL)

- Detection limits refer to the minimum concentration of an analyte that can be measured above the instrument background noise.
- When detection limits are used as reporting limits, a non-detect means that the analyte is not present at or above the value given.
- It may be present at a lower concentration, but cannot be "seen" by the instrument.
- How is the detection limit determined?
  - Limit is based on a statistically significant signal-to-noise ratio or a statistically determined confidence level
  - There is a correction for the effects of sample handling and preparation.
  - There is a correction for sample matrix and dilution
- Detection limits are only a measure of the ability of the test procedure to generate a positive response and **do not indicate the accuracy** of that response.

MDL Continued

Purpose of MDL

- An MDL is an estimated value that cannot be reported without qualification as such.
- An MDL is a statistically calculated number that has not been physically proven on the instrument. It is qualitative, not quantitative.
- Because Analytical Methods do not require method blanks to be evaluated to MDLs, possibility of false positives is high.
- Caution should always be used when using MDLs to make site decisions.

Practical Quantitation Limit

PQL or RL

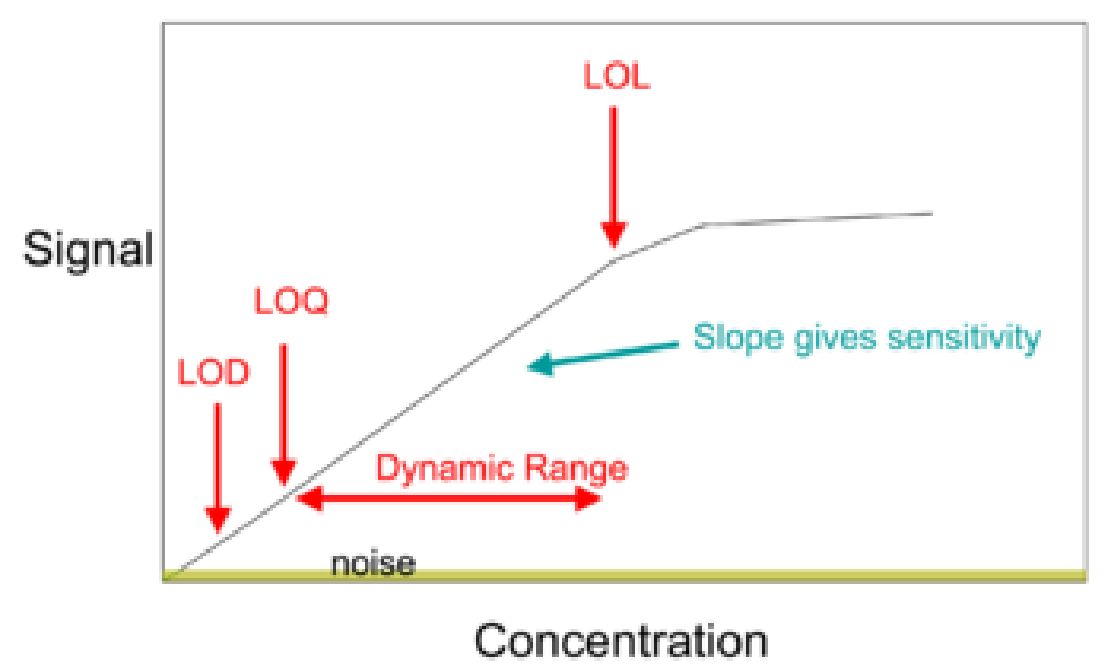
- Concentration of an analyte that can be measured within specified limits of precision and accuracy.
- Generally 2X to 10X the MDL.
- Where PQL = RL, a non-detect result indicates an analyte is not present at a concentration reliably quantified above the quantitation limit.
- The analyte may be present at a lower concentration, i.e. above the MDL.
- Unlike an MDL, there are both **lower and upper quantitation** limits. These limits represent the **calibration range** of the instrument .

Standard PQLs vs Project-Specific RLs

- In many cases, the laboratory can obtain a lower detection limit, which can be reported if specially requested for a project.

- In some cases, analyte concentrations detected between the MDL and the PQL will be flagged as estimated with a “J” qualifier.

Calibration Curve (Range)



Calibration range: Concentration where linearity is not compromised

Increase upper limit of calibration Ą lower reporting limit is higher, but dilutions are not as frequent

Decrease lower limit of calibration Ą Upper calibration is lower and therefore requires more dilutions

Meeting RLs is easy when samples are non-detect (clean)
Meeting RLs for **contaminated samples is more difficult:**

- May Require Dilutions
- May have matrix interference
- Usually cause carryover into the next sample
- Compromise the ability to achieve reporting limits for some analytes

Dilutions

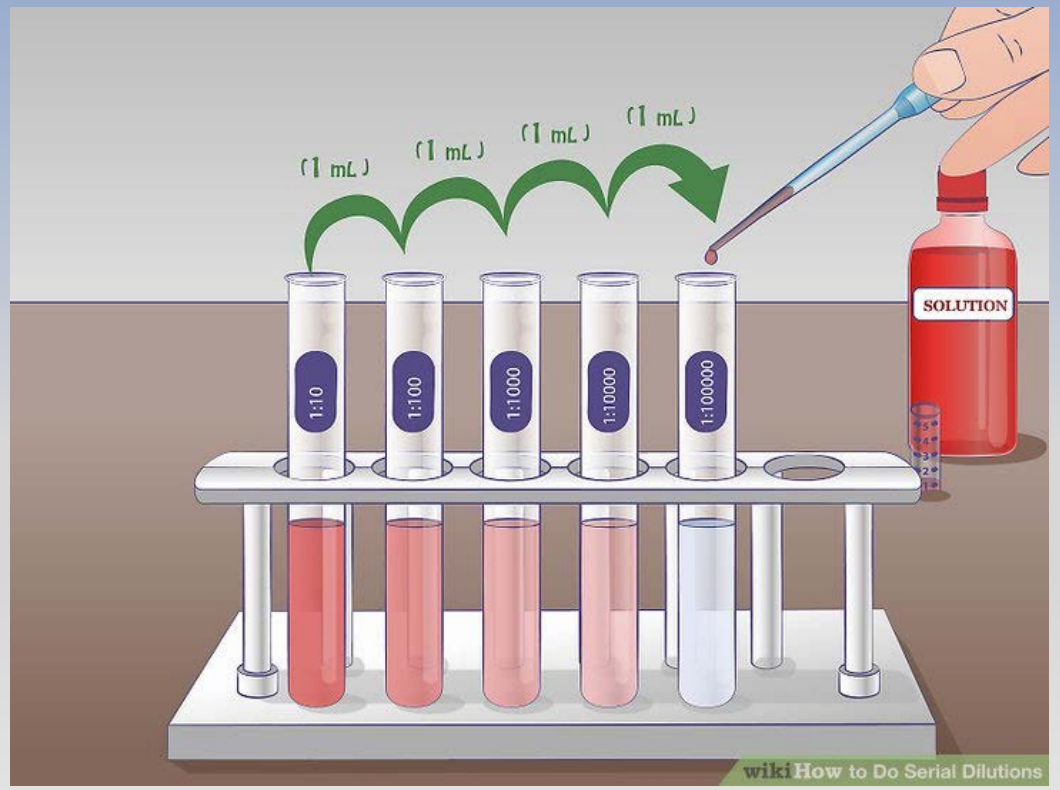
In order to have legally defensible data it must be reported within the calibration range (i.e.: no qualifiers)

Causes of Dilutions:

- Interferences present in sample resulting in failing internal standards or surrogates. The interference must be diluted out to meet method criteria. May dilute some analytes to concentration below detection limit.
- High concentrations of target analytes which can “swamp” a detector or overload a chromatographic column, resulting in expensive repairs and downtime for lab.
- Odd sample matrixes that foam, emulse, or are extremely light.

Resolving Reporting Limit Issues

- Can the laboratory rerun the sample?
  - using a different method (6010 vs 6020)
  - Using a different dilution factor



Blank Contamination Evaluation

- Generally follow same guidelines for all blanks (field, trip, rinsate blanks and laboratory method blanks)
- Parameter Dependent (i.e. handle common laboratory contaminants differently)
- Compare to Historical Data Results

Blank Contamination Evaluation

Previous Guidance

- “B-flag” or “R-flag” if less than 5x blank concentration
- “B-flag” or “R-flag” if less than 10x blank concentration for common laboratory contaminants (acetone, methylene chloride, MEK)

Blank Contamination Evaluation

Current Guidance – more complicated

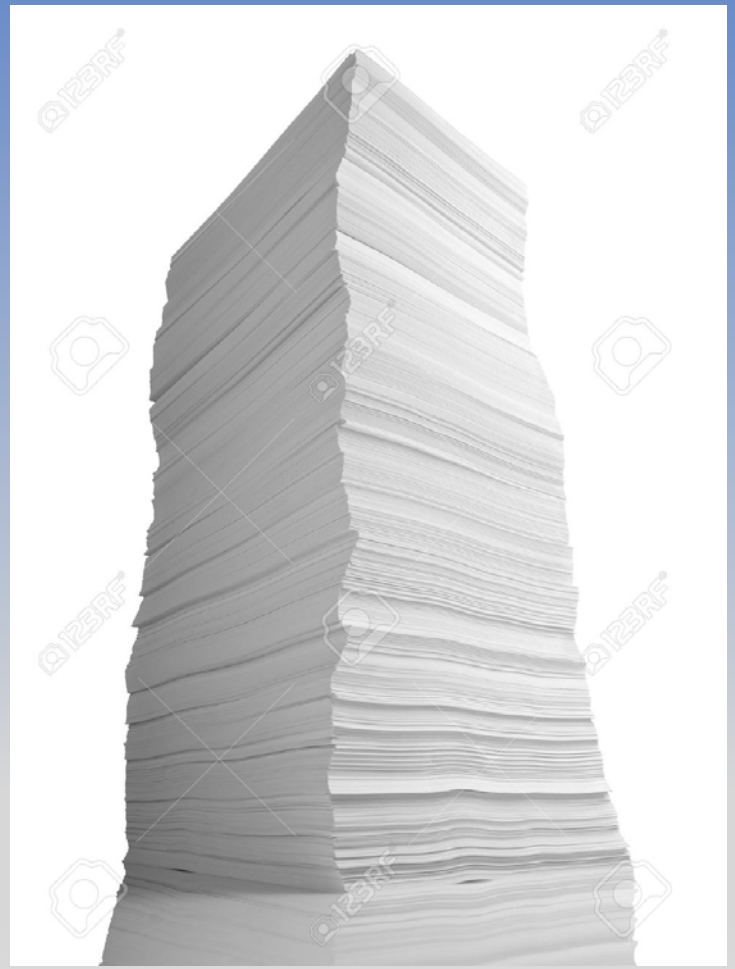
Table 7. Blank Actions for Trace Volatile Analysis

Blank Type	Blank Result	Sample Result	Action
Method, Storage, Field, Trip, Instrument*	Detect	Non-detect	No qualification
	< CRQL	< CRQL	Report at CRQL and qualify as non-detect (U)
		≥ CRQL or ≥ 2x Blank Result for Methylene Chloride, Acetone, and 2-Butanone	Use professional judgment
	≥ CRQL	< CRQL	Report at CRQL and qualify as non-detect (U)
		≥ CRQL but < Blank Result	Report at sample result and qualify as non-detect (U) or unusable (R)
		≥ CRQL and ≥ Blank Result or ≥ 2x Blank Result for Methylene Chloride, Acetone, and 2-Butanone	Use professional judgment
	Gross contamination*	Detect	Report at sample result and qualify as unusable (R)
	TIC > 0.5 µg/L	Detect	Use professional judgment

Additional QA/QC

- Usually this level of evaluation is not necessary
- Unless laboratory qualifies data as “R”, your data should be adequate for most projects….

Data Validation



- Data Validation is an analyte- and sample-specific process that extends the evaluation of data beyond method, procedural, or contractual compliance (i.e., data verification) to determine the analytical quality of a specific data set.

If data validation is required….

- Surrogates
  - Usually do not qualify data unless more than one or all surrogates are outside control limits
  - Depends on whether biased high or biased low
- Laboratory Control Samples
  - Usually do not qualify data unless both LCS and LCSD are outside control limits
  - Don’t qualify just on LCS/LCSD RPD
  - Depends on whether biased high or biased low

Matrix Spikes

- MS/MSDs (bonus for data evaluation; not usually required)
  - Usually do not evaluate unless site-specific
  - Usually do not qualify data unless both MS and MSD are outside control limits
  - Do not qualify just on MS/MSD RPD
  - Depends on whether biased high or biased low
  - Qualify only affected sample or qualify all samples with same matrix

Laboratory Duplicates

- Laboratory Duplicate
  - Do not qualify unless site-specific
  - Qualify only affected sample or qualify all samples with same matrix?

Table 7. Duplicate Sample Actions for ICP-AES Analysis

Duplicate Sample Results	Action for Samples
Both original sample and duplicate sample > 5x the CRQL and RPD > 20%*	Qualify those results that are ≥ MDL that professional judgment determines to be affected as estimated (J) and non-detects as estimated (UJ)
Original sample or duplicate sample ≤ 5x the CRQL (including non-detects) and absolute difference between sample and duplicate > CRQL*	Qualify those results that are ≥ MDL that professional judgment determines to be affected as estimated (J) and non-detects as estimated (UJ)

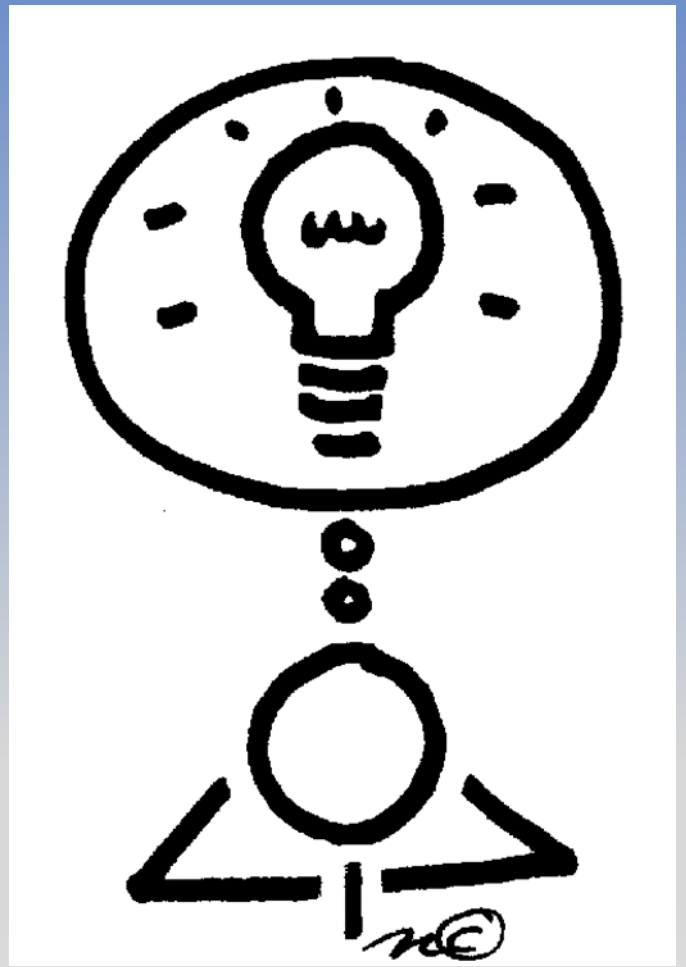
* The above control limits are **method requirements** for duplicate samples, regardless of the sample matrix type. However, it should be noted that laboratory variability arising from the sub-sampling of non-homogenous soil samples is a common occurrence. Therefore, for **technical review purposes only**, Regional policy or project Data Quality Objectives (DQOs) may allow the use of less restrictive criteria (e.g., 35% RPD, 2x the CRQL) to be assessed against duplicate soil samples.

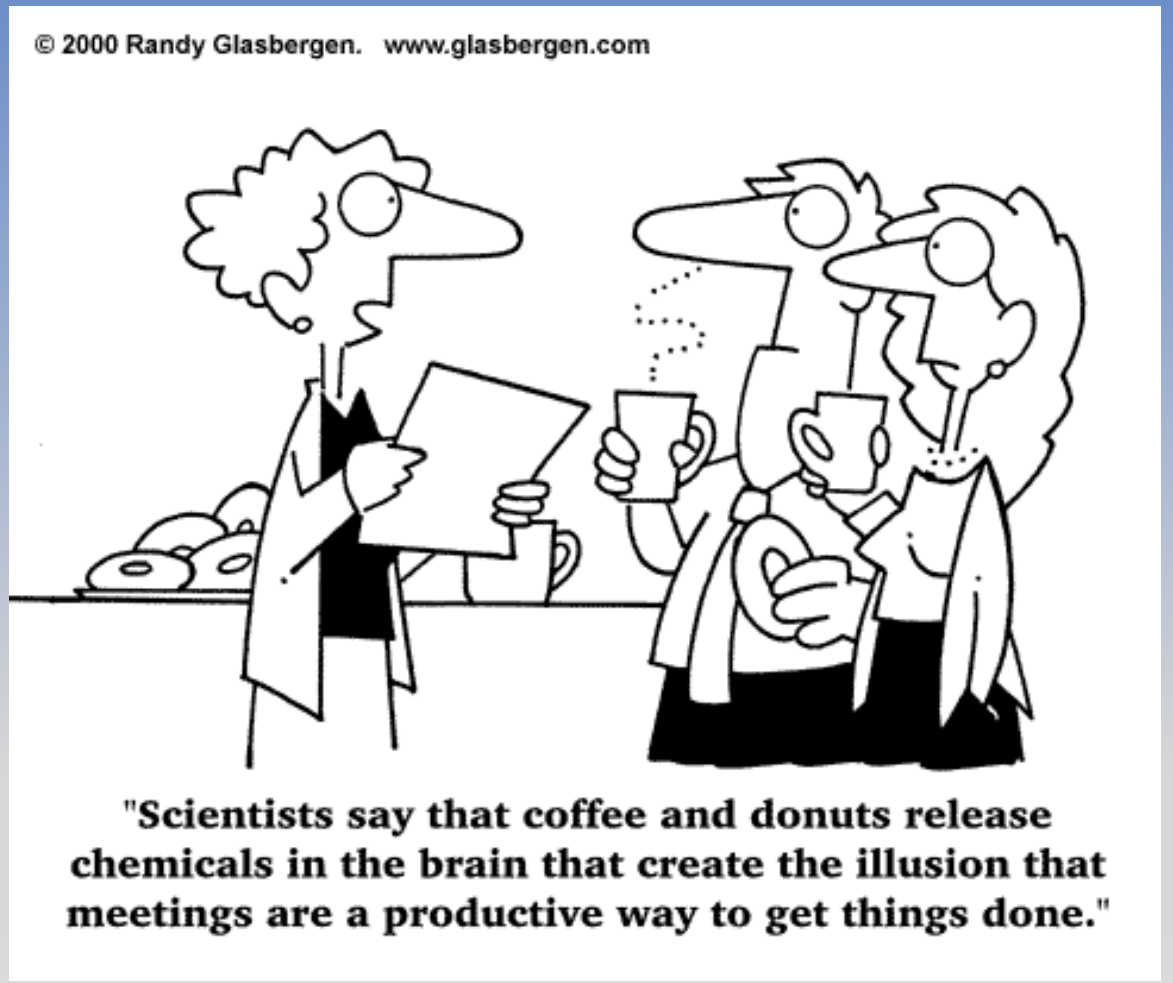
Beyond the scope of this presentation……

- Other Laboratory QC (calibration, internal standards, etc.)
- Refer to Guidance Documents…..

The Name’s Bond, Ionic Bond, Taken, Not Shared

Questions?

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"Scientists say that coffee and donuts release chemicals in the brain that create the illusion that meetings are a productive way to get things done."

Contact

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