Laboratory Analytical Data

NEWMOA

Data Collection & Interpretation: State of Practice & Lessons Learned

Jim Occhialini
jocchialini@alphalab.com

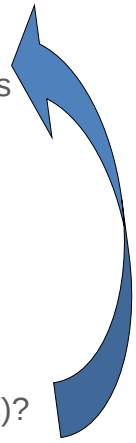
"Interface"

- *– verb meaning to blend, ally, coalesce, combine, come together, consolidate, incorporate, integrate, intermix, join together, meld, merge, pool, team up, or unite*
- Labs are an integral component of your projects
  - You need to interact with them before, during and after your sampling programs
  - **Why is this important?**
    - **BECAUSE RE-SAMPLING IS VERY EXPENSIVE, TIME CONSUMING AND POTENTIALLY EMBARISING**

Data Collection Process

Communication

1. Planning
   - Why are you collecting samples?
   - involve all data users / project applications
     - Everyone ***"on the same page"*** including lab?
2. Execution
   Collect & analyze samples
3. Evaluation
   Is data usable for the intended purpose(s)?
   Then interpret the results



Upfront Planning

- Logistical considerations
  - Scope & schedule?
    - Laboratory turnaround time
      - Discuss w/lab in advance, accelerated TATs?
    - Container delivery and sample pick up
- Hold times
  - Your samples have a shelf life
  - Beware of short hold time analyses
    - Dissolved metals (lab filtered), ferrous iron, hex chrome, nitrates, microbiology…

Upfront Planning

- Regulatory oversight drives choice of analytical method
  - State program (CAM / RCP / DKQP), EPA ***(TSCA?)*,** DoD, NPDES (RGP?), POTW (i.e. MWRA), etc.
- Certifications
- Discuss with lab project-specific requirements
  - Target compound list (TCL)
    - Compound(s) of interest not on the list?
    - Anything else?
- Share all applicable project documents
  - QAPPs, SAPs, etc.

Upfront Planning - Reporting Limits

- Specify regulatory criteria requirements
- Any "problem analytes" that are contaminates of concern?
  - Common considerations
    - EPH w/target PAHs (GW-1)
    - EDB & DCBP (GW-1)
    - 1,4 Dioxane (GW-1)
    - Other example(s) – vinyl chloride at dry cleaner site?
- Other confounding issues
  - Moisture content, volume/mass limitations, sample matrix (i.e. tissue), grossly contaminated samples
  - *Regulatory criteria not always achievable…*

Sample Extraction & Impact on Reporting Limits

1000 to 1

1 mg/L instru
concent
= 1 ug/L me

6

Anatomy of a Basic Laboratory Report

- Cover page / certification page*
- Case narrative
- Sample results pages
  - Includes sample - specific QC information
- Batch QC section

- Laboratory deliverables package?
- Online data summaries
- Electronic data deliverables (EDD)

7

What's so Important about Data Usability?

It's all about managing uncertainty…

and incorporating that uncertainty into your decision making

9

Relationship Between Risk Tolerance & Uncertainty

- Is all laboratory data treated the same way???
  - Final clean up verification samples vs. initial site screening?
- Level of scrutiny and interpretation applied to laboratory data commensurate with what it will be used for
  - Risk assessment?
  - Locate “hot spots”?

Regulatory Approaches to Managing Uncertainty

- **EPA**
  - Program wide approach
    - CERCLA (“superfund”)
      - Contractor laboratory program (CLP) - ***PRESCRIPTIVE***
  - Project specific approach
    - RCRA
      - SW-846 ***GUIDANCE***
      - Quality Assurance Project Plans (QAPPs)
  - Data Quality Objectives (DQOs) for RI/FS ~1984
- **States**
  - Program wide approach
    - CT Reasonable Confidence Protocols (RCP) ~2006
      - RCP DQA/DUE
    - MA Compendium of Analytical Methods (CAM) ~2003
      - MCP REDUA 2007
    - NJ DKQP Technical Guidance 4/2014

How Do You Evaluate Data Quality?

PARCCS

QA / QC

- Quality Control – (2 components)
  1. "QC infrastructure"
  2. Continuing monitoring / documenting data quality
     1. Internal lab system control & project- specific DQI info
- Quality Assurance
  - *Assures* the QC is performed, "enforcer"
  - Systematic & performance audits
    - Does the lab perform internal audits?
      - Follow up on corrective actions?

Quality System

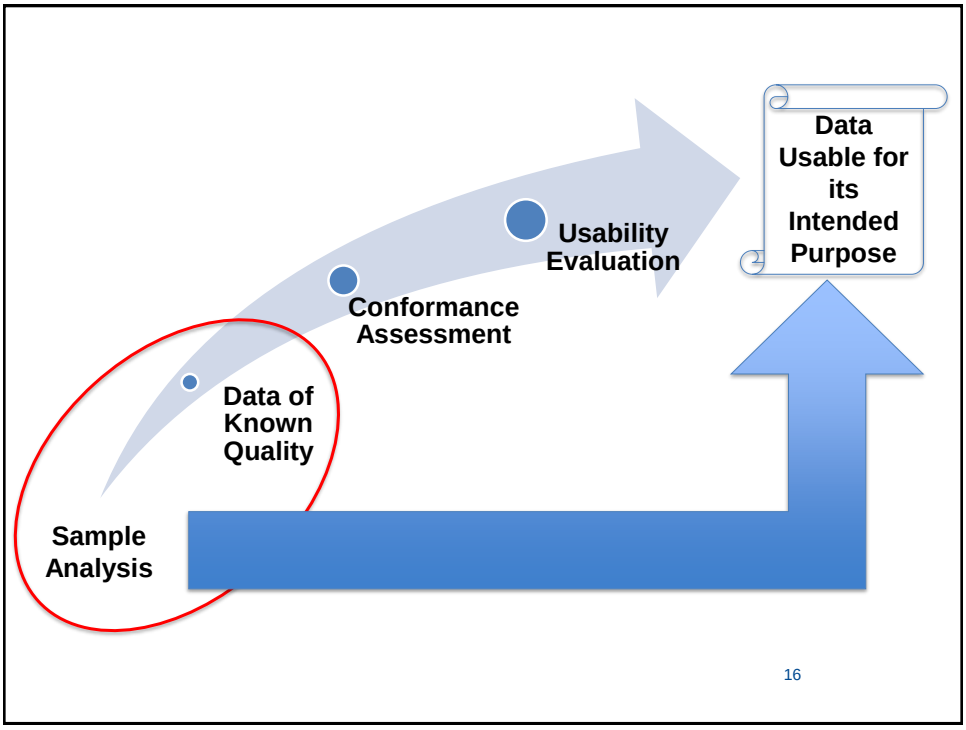
14

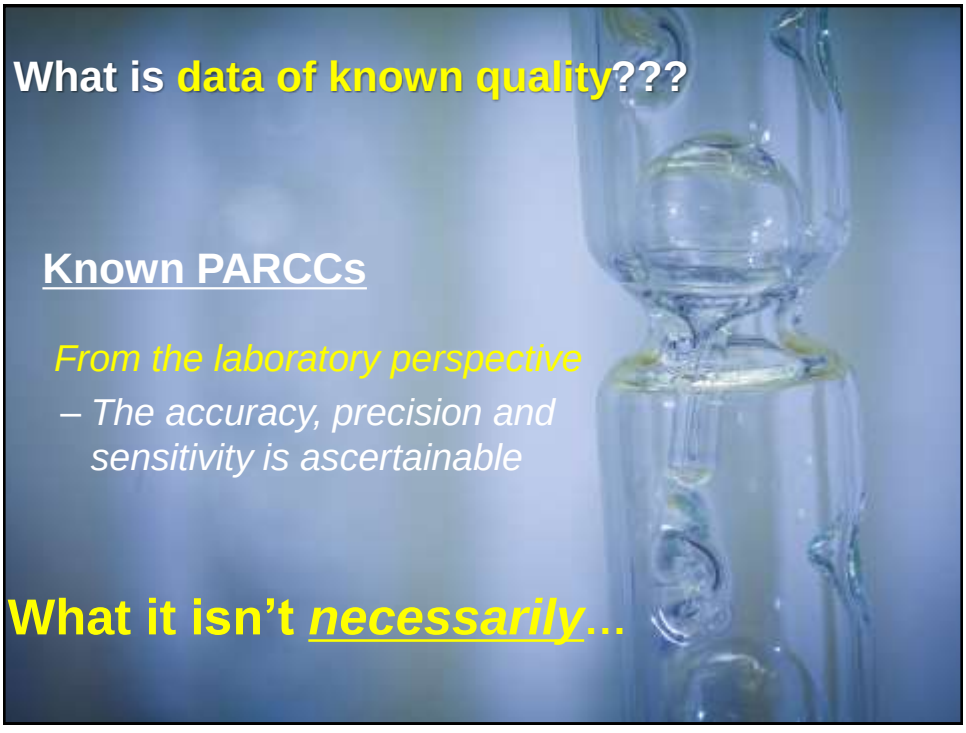
…so a Quality System means everything is in place to produce ***"data of known and ascertainable quality"***

Doesn't mean that all data generated by the lab is of known quality

…or the data in your specific report

15


Data Usable for its Intended Purpose
Usability Evaluation
Conformance Assessment
Data of Known Quality
Sample Analysis
16


What is data of known quality???
Known PARCCs
From the laboratory perspective
– The accuracy, precision and sensitivity is ascertainable
What it isn't necessarily…

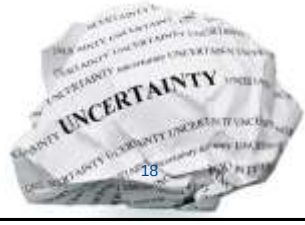
How Do You Get Data of Known Quality?

- Level of uncertainty is known*… **HOW?***

1. Data generated & reported in accordance with a "state data quality protocol" (i.e. CAM, RCP, DKQP)

   "presumptive certainty", "reasonable confidence" & "data of known quality"

2. Data generated & reported with a full data deliverables package & incorporating a comprehensive QAPP & complete data validation
3. Subset of #2…



18

State Data Quality Programs

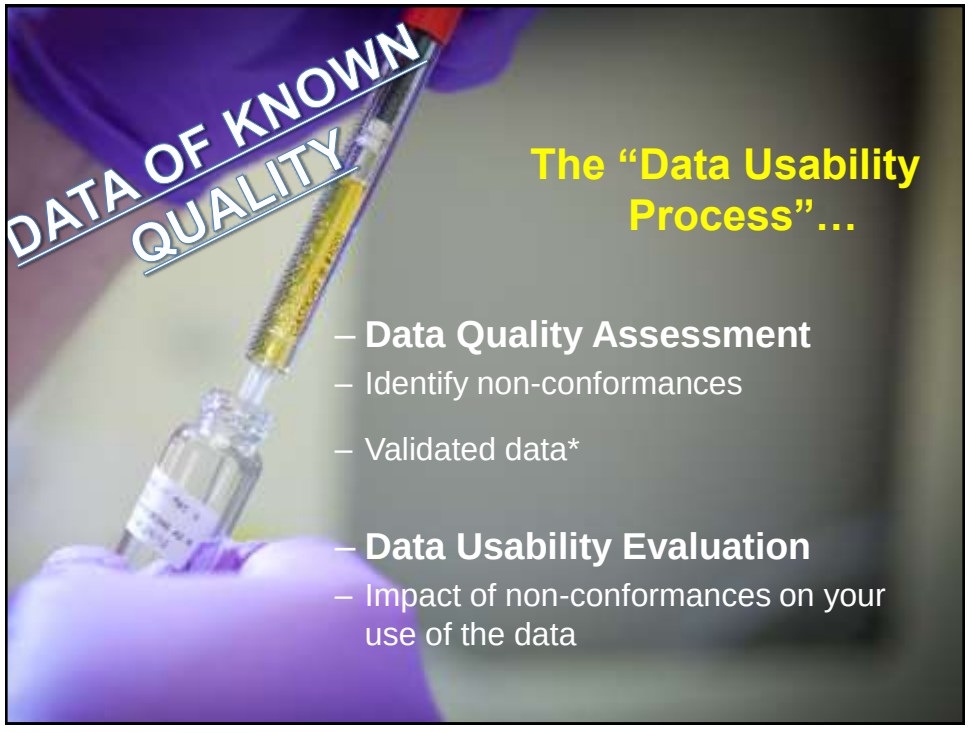
- Existing EPA RCRA methods “tightened up”
  - Specific performance standards & QA/QC criteria
  - Calibration and reporting limit determination
- Required laboratory report content
  - Required documentation to be kept on file
    - Information available to generate a complete “CLP-like data validation package” if requested
- Certification page questionnaire
  - Laboratory “certifies” compliance
  - Comprehensive narrative

Example QA/QC Requirements & Performance Standards

Quality Control Requirements and Performance Standards for the ***Analysis of Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)*** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)

Table II A-1: Specific QC Requirements and Performance Standards for VOCs (SW-846 8260B) Using WSC-CAM-II A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350?	Required Corrective Action	Required Analytical Response Action
Initial Demonstration of Proficiency	Laboratory Analytical Accuracy & Precision	(1) Must be performed prior to using method on samples. (2) Must be performed for each matrix. (3) Must contain all target analytes. (4) Must follow procedure in Section 8.4 of SW-846 8000B.	No	NA	Refer to Section 8.4 of SW-846 8000B and Section 3.1.2 of this protocol.	NA
GC/MS Tunes with BFB	Inter-laboratory Consistency & Comparability	(1) Criteria listed in Table 4 of SW-846 8260B (the same criteria must be used for all analyses). (2) Every 12 hours prior to sample analysis.	No	NA	Perform instrument maintenance as necessary; retune instrument.	Suspend all analyses until tuning non-compliance is rectified.
Initial Calibration	Laboratory Analytical Accuracy	(1) Must be analyzed at least once prior to analyzing samples, when initial calibration verification or continuing calibration does not meet the performance standards, and when major instrument maintenance is performed. (2) Minimum of 5 standards (or 6 if non-linear regression used). (3) Low standard must be ≤RL. (4) %RSD ≤20%, r ≥0.99 (linear regression), or r^2 ≥0.99 (non-linear regression) for each target analyte. (5) If %RSD >20, linear or non-linear regression must be used. (6) Minimum RFs as per Table 4 of SW-846 8260C for lowest concentration standard and for average RF. (7) Must contain all target analytes. (8) Calibration must be performed under the same conditions as the samples (e.g., heated purge).	No	RF <0.05; affects quantitative results for affected analyte in all samples analyzed under this initial calibration.	(1) Recalibrate if <10% of target analytes exceed %RSD, "r", or "r^2" criteria. (2) If ≥10% of compounds exceed criteria, recalibration is not required as long as %RSD ≤40, r ≥0.98, or r^2 ≥0.98. (3) If recalculated concentrations from the lowest calibration standard are outside of 70-130% recovery range, either: • The RL must be reported as an estimated value[1], or • The RL must be raised to the concentration of the next highest calibration standard that exhibits acceptable recoveries when recalculated using the	Samples analysis cannot proceed without a valid initial calibration. Report non-conforming compounds (%RSD >20, r <0.99, r^2 <0.99 or minimum RF not met) in laboratory narrative. If non-linear regression (i.e., quadratic equation) is used for calibration, this must be noted in the laboratory narrative along with the compounds affected.

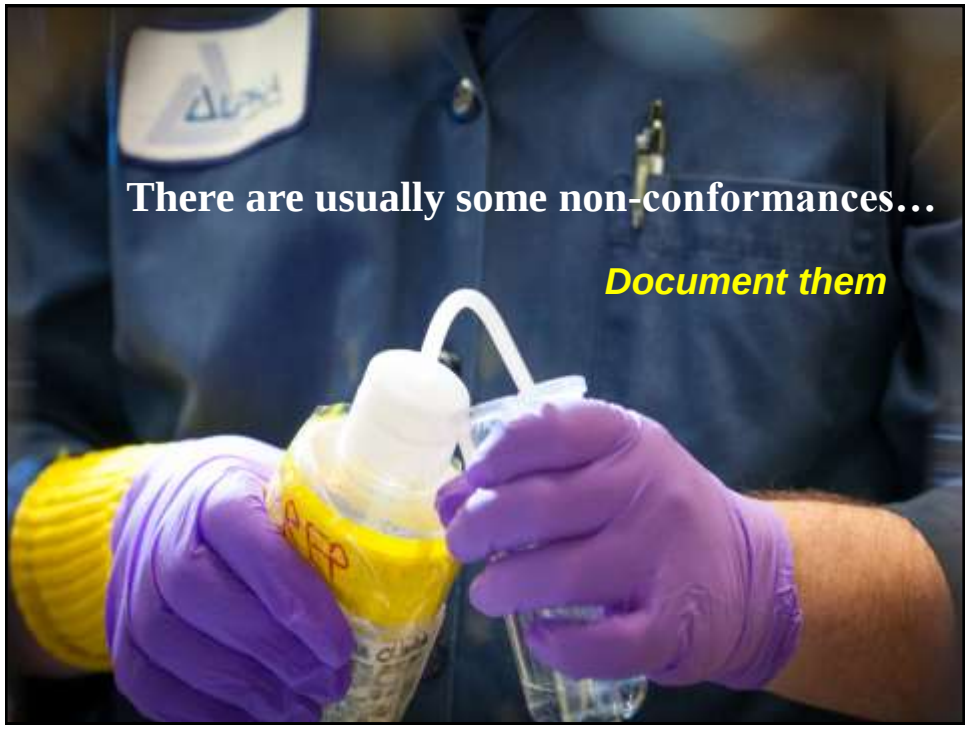


Data Quality Assessment-
Starting the Process
(w/ CAM, RCP, DKQP compliant data)

- "Presumptive Certainty", Reasonable Confidence and/or "Data of Known Quality"…
  - **QUESTION H* *(CAM)*** "*Were all QC performance standards for the specified methods achieved?*" ***"YES"***

A	Were all samples received in a condition consistent with those described on the Chain-of-Custody, properly preserved (including temperature) in the field or laboratory, and prepared/analyzed within method holding times?	☐ Yes ☐ No
B	Were the analytical method(s) and all associated QC requirements specified in the selected CAM protocol(s) followed?	☐ Yes ☐ No
C	Were all required corrective actions and analytical response actions specified in the selected CAM protocol(s) implemented for all identified performance standard non-conformances?	☐ Yes ☐ No
D	Does the laboratory report comply with all the reporting requirements specified in CAM VII A, "Quality Assurance and Quality Control Guidelines for the Acquisition and Reporting of Analytical Data"?	☐ Yes ☐ No
E	VPH, EPH, APH, and TO-15 only a. VPH, EPH, and APH Methods only: Was each method conducted without significant modification(s)? (Refer to the individual method(s) for a list of significant modifications). b. APH and TO-15 Methods only: Was the complete analyte list reported for each method?	☐ Yes ☐ No ☐ Yes ☐ No
F	Were all applicable CAM protocol QC and performance standard non-conformances identified and evaluated in a laboratory narrative (including all "No" responses to Questions A through E)?	☐ Yes ☐ No
Responses to Questions G, H and I below are required for "Presumptive Certainty" status		
G	Were the reporting limits at or below all CAM reporting limits specified in the selected CAM protocol(s)?	☐ Yes ☐ No[1]
Data User Note: ***Data that achieve "Presumptive Certainty" status may not necessarily meet the data usability and***		
H	Were **all** QC performance standards specified in the CAM protocol(s) achieved?	☐ Yes ☐ No[1]
I	Were results reported for the complete analyte list specified in the selected CAM protocol(s)?	☐ Yes ☐ No[1]

[1] *All negative responses must be addressed in an attached laboratory narrative.*



Data Quality Assessment

- **Where do I start? *(looking for non-conformances)***
  - **LAB NARRATIVE** *(exception report)*
    - Includes all issues of significance to data user: method performance problems, QA/QC outliers, etc.
  - Lab report **BATCH QC** summary data section
  - Lab report **SAMPLE SPECIFIC QC** data pages
- **What do I need to know?**
  - Data quality indicators
    - Accuracy
    - Precision
    - Sensitivity (reporting limits)

Data Quality Indicators *(information in your lab report)*

- Three Levels of Information

Field Generated QC *(submit blind, compile it)*
- Trip/field blanks (accuracy), field duplicates (precision)
- matrix spike / matrix spike duplicate (accuracy, precision)

Lab Batch Specific QC
- Method blanks (accuracy), LCS / LCSD (accuracy & precision)

Sample Specific QC
- surrogates, fractionation surrogates (accuracy)
- holding times, sample preservation & handling (accuracy)

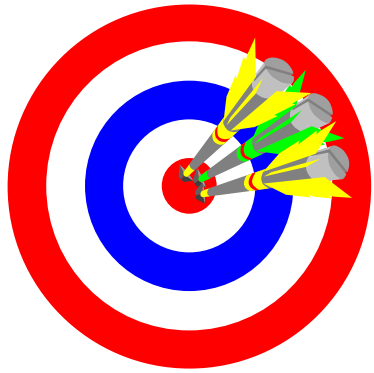
Accuracy – Evaluation of Bias that Exists in the Measurement System

- Is there bias?
  - Lab measurement system in control?
  - Sample - specific interferences?

Spike recovery:

$$\frac{MV}{TV} \times 100 = \%R$$

Where MV = Measured Value & TV = True value



Data quality indicators - measurement tool:

blanks & spikes

%R can indicate positive or negative bias

26

Accuracy - Lab Data Quality Indicators

Lab Batch QC

- Lab control sample (LCS) *if done in duplicate*… (LCS / LCSD)
  - Baseline accuracy determination, entire TCL
  - **Potential POSITIVE or NEGATIVE bias**
- Laboratory method blank
  - False positive indicator, **potential POSITIVE bias**

Sample Specific QC

- **Surrogate Spikes**
  - Chemically similar subset of analytes
    - Added to every sample (organics analysis)

27

Accuracy –
Additional Data Quality Indicators

- Hold times (sample & parameter specific QC element)
  - False negative indicator, **potential NEGATIVE bias**

Field QC

- Matrix spike/matrix spike duplicate (MS/MSD)
  - Same as LCS/LCSD w/spike added to actual sample
  - Organics analysis – applies to spiked sample only
  - Inorganic samples- applies to all samples in batch

- Field, trip, and/or equipment blank (field QC samples)
  - False positive indicator, **potential POSITIVE bias**

Evaluating Accuracy

Where does the criteria come from? What's in your report?

Example %R	Example Acceptance Criteria	Recommendation
55	70 – 130	Negative bias
147	70 – 130	Positive bias

Bias can be positive or negative, expressed as %R

- %R used for surrogates, LCS/LCSD & MS/MSD
  - ***Don't do the math!***

- *A word about positive bias…*

Interpreting Accuracy Bias

result	spike %R	action level	acceptance criteria
50	22%	55	75 – 110 %
50	47%	1	75 – 110 %

Interpretation:

Positive / negative bias

vs.

Relationship of data point to the action level

vs.

Specific use of the data

Precision – Expression of Reproducibility & Variability

Precision measurement tool: replicate analyses

Evaluated using relative percent difference (RPD)

- How reproducible is the lab measurement system?
- Sample homogeneity?

$$\frac{|R_1 - R_2|}{(R_1 + R_2)/2} \times 100 = \%RPD$$

% RPD = the absolute value of the range divided by the mean times 100

Precision - Expression of Reproducibility & Variability

Laboratory generated precision information:

- (LCS / LCSD)
  - Two analyses –> results compared (%RPD) for precision
- Laboratory batch duplicates

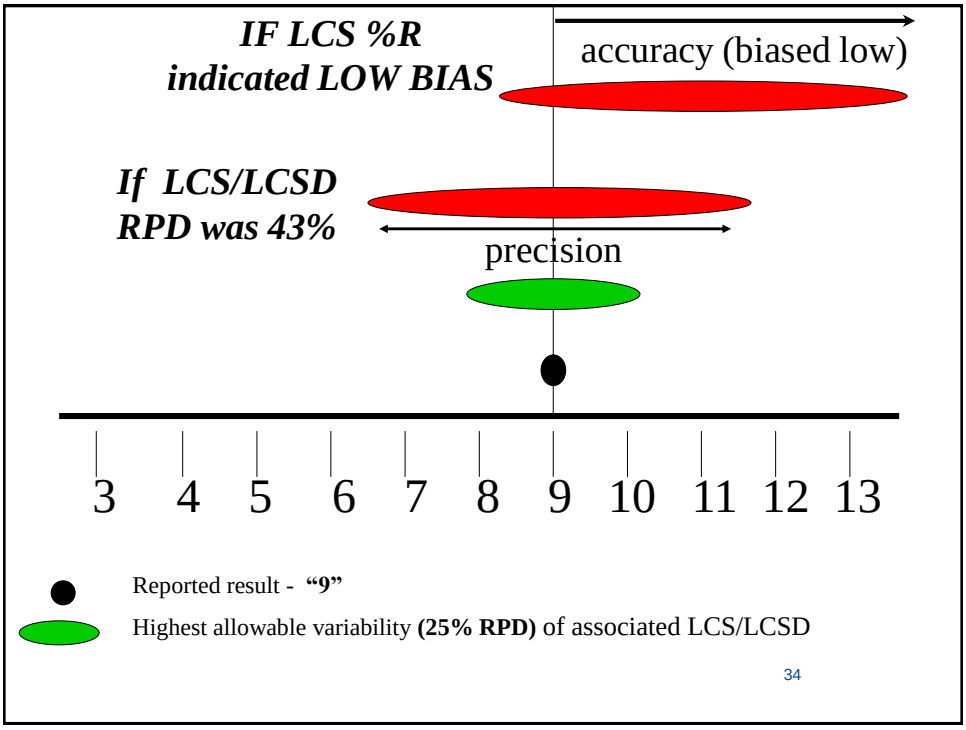
Field generated precision information:

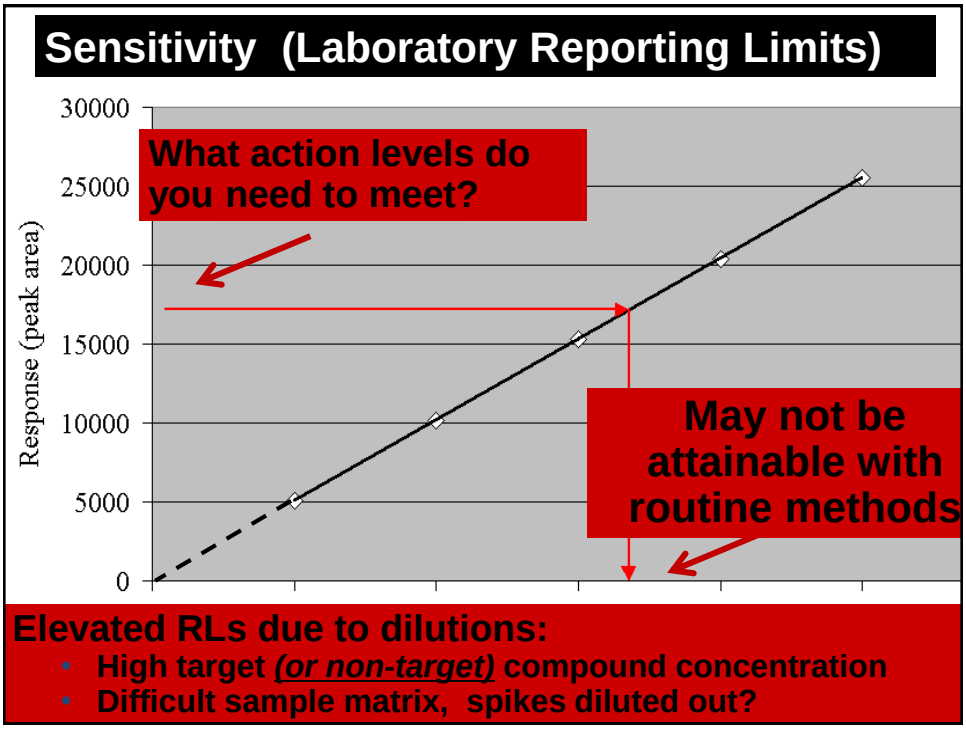
- Field duplicates, co-located samples, MS/MSD
  - Submit "blind", calculate RPD

Evaluating Precision

Example RPD	Example Acceptance Criteria (%RPD Upper Limit)	Recommendation
14	25	Precision **within** acceptable range
35	25	Precision **outside** acceptable range

- %RPD acceptance criteria represents an upper limit
  - Greater the RPD, more variability (less precision)
- %RPD used for LCS/LCSD, MS/MSD, lab/field duplicates


IF LCS %R
indicated LOW BIAS
accuracy (biased low)
If LCS/LCSD
RPD was 43%
precision
3 4 5 6 7 8 9 10 11 12 13
Reported result - "9"
Highest allowable variability (25% RPD) of associated LCS/LCSD
34


Sensitivity (Laboratory Reporting Limits)
30000
25000
20000
15000
10000
5000
0
Response (peak area)
What action levels do you need to meet?
May not be attainable with routine methods
Elevated RLs due to dilutions:
High target (or non-target) compound concentration
Difficult sample matrix, spikes diluted out?

So…Is the Data Usable?

Data with non-conformances … usable?

Focus…

- Why did the report get a "NO" on Questionnaire and/or what else did your review find?
  - Isolate analysis
    - Isolate analytes
  - This is the data that needs to be evaluated
- Everything else is OK to use "as is"…
  - **Still need sensitivity evaluation**

Data Usability Evaluation Process

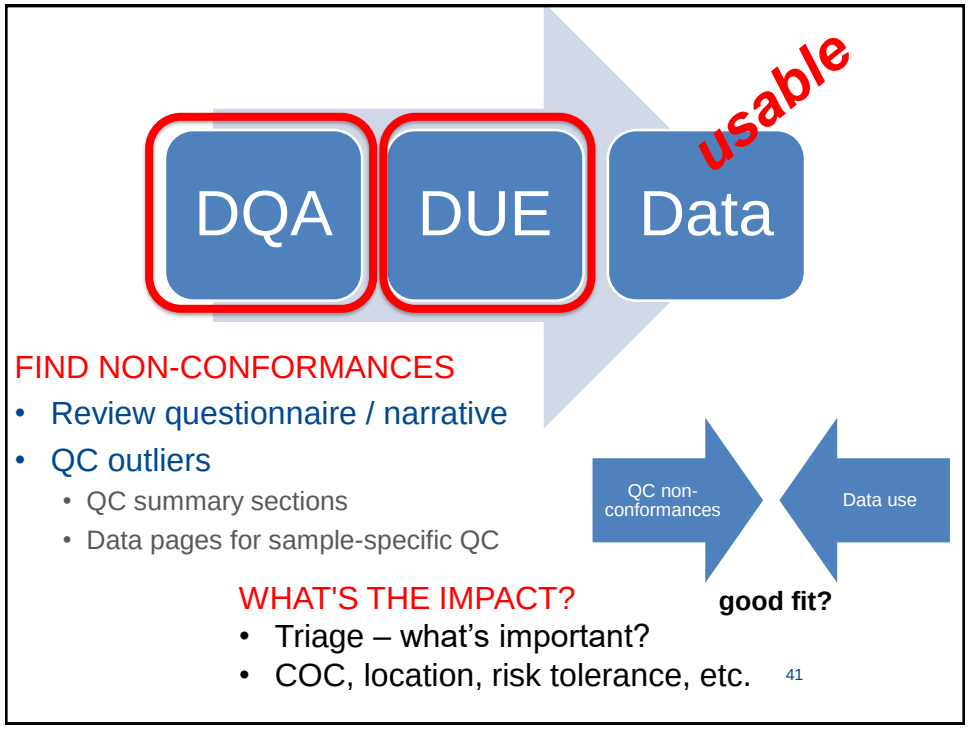
- Summary of non-conformances
  what does it mean for my project application?
- **Evaluate relevancy**
  - Contaminant of concern? Sample location?
  - Bias: +, - or indeterminate?
    - **Relationship of result to regulatory criteria**
- Incorporate uncertainty into decision-making
  - Does this non-conformance impact my use of the data?
  - **RISK TOLERANCE**

Additional Considerations

- Multiple lines of evidence
  - Batch QC DQIs / sample specific DQIs
    - Additive or contradictory effect?
  - Bring in info beyond current lab report
    - Historical data, field data, other samples (EPC), CSM, etc.
- Trade offs
  - Non-conformance severity (17% R or 70% R)
    - Importance of this data point / risk tolerance?
  - Is the non-conformance tempered by facts?
    - (dilution, co-elution, obvious sample matrix issues…)

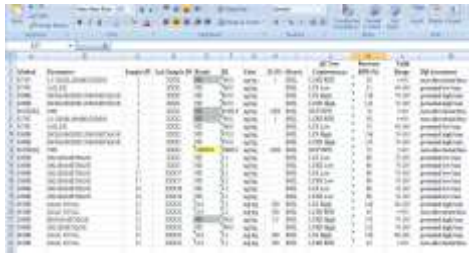
39


So…is the data usable?
Can you justify it?


DQA
DUE
Data
usable
FIND NON-CONFORMANCES
• Review questionnaire / narrative
• QC outliers
• QC summary sections
• Data pages for sample-specific QC
QC non-conformances
Data use
good fit?
WHAT'S THE IMPACT?
• Triage – what's important?
• COC, location, risk tolerance, etc.
41

Managing Usability Information

- Summarize your data qualifications
  - Table summary (Exception Report)
    - Integrate into project data base
  - Use data usability -qualified data for all decision making



- Reminder
  - you really should have an understanding of data limitations **ongoing** as decisions are made

42