Conducting Data Review For State Drinking Water Programs

Richard M. Amano
Laboratory Data Consultants, Inc.

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Thanks!!!

- Linda Geddes, Andy Eaton
  MWH Labs, Monrovia, CA

- Pei Geng, Ming Hwang
  LDC, Carlsbad, CA

Workshop Outline

- Analytical methods
- Data review

  - UCMR
- Formal data validation
- Electronic data review

  - ADR
- Data usability

What do end user's of data want?

- "Reliable" data
- Data of known quality
- Low detection limits
- Legally defensible data
- Organized reports
- Easy to understand reports
- Supporting QA/QC data for all final results
- ???????

Test Methods

- Test Methods for Evaluating Solid Waste - EPA SW-846
- Water and Wastewater Methods - EPA 300/600/900 Series
- Drinking Water Methods - EPA 200/500 Series
- Standard Methods
- CLP Methods

Types of Parameters

- Organics (Volatile and Semivolatile)
- Inorganics (Metals and Wet chemistry parameters)
- Radiochemistry
- Microbiological

Common Analyses

- EPA Method 8260B
- EPA Method 8270C
- EPA Method 8081/8082
- EPA Method 8015B
- EPA Method 6010B/7000
- EPA Method 9000 series
- EPA Method 1600 series
- EPA method 200/300/500/600 series

UCMR2 Methodology

- Method 521 – Nitrosamines by CI/MS/MS
- Method 525.2 – Acetanilide Pesticides by GC/MS
- Method 527 – Flame Retardants by GC/MS
- Method 529 – Explosives by GC/MS
- Method 535 – Acetanilide Degradates by LC/MS/MS

The Laboratory Process

Also
Surrogates
Continuing Calibration Verification (CCV)
Internal Standards
Other method required measurements

Areas of Report Review

- Chain of Custody
- Case Narrative
- Form 1s (or results summary)
- Initial Calibration
- Calibration Verification (Continuing Calibration)
- Blanks (Method, Instrument, Field, Trip, Equipment, Ambient)
- Surrogates

Areas of Report Review (cont)

- Matrix Spike (Lab Fortified Matrix)
- Matrix Spike Duplicate
- Lab Fortified Blank (Lab Control Sample)
- Lab Control Sample Duplicate
- Internal Standards (GC/MS, ICP/MS, ICP)

Areas of Report Review (cont)

- Compound Identification
- Compound Quantitation
- Detection Limits (IDL, MDL, MRL, PQL, RDL)
- TIC verification with GC/MS

Manual Integration Review

- Labs must date and initial changes
- Past problems in lab industry with “improper practices”
- Primarily identified through review of electronic data
- Indicators may be identified through raw data review

UCMR2-What is It?

- Unregulated Contaminant Rule-Part 2
- Continuing Series-UCMR1 was conducted from 2001-2005

  25 compounds & *Aeromonas*
- Part of the Contaminant Candidate List (CCL) process to evaluate future contaminants for regulation.

Environmental Protection Agency

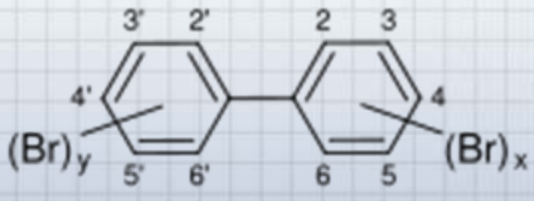
40 CFR Parts 9, 141, and 142

Unregulated Contaminant Monitoring Regulation (UCMR) for Public Water Systems Revisions; Final Rule

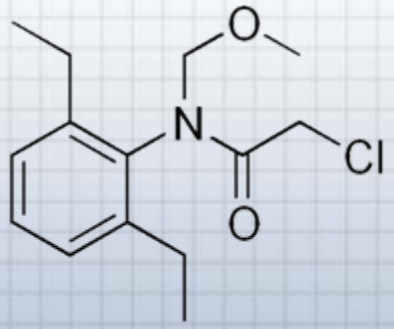
- First Posted in August, 2005
- Published for comment October 2005
- Promulgated on January 4, 2007

Purpose

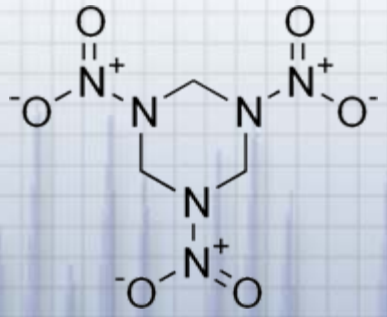
- Collect occurrence data for contaminants that may be in drinking water but have no health-based standards in SDWA



2,2', 4,4',5,5'-hexabromobiphenyl (HBB)



Alachlor



RDX

What is Being Monitored?

- Targets new compounds including pesticides, flame retardants, and explosives
- Requires specialized laboratory certification from EPA with specific turnaround and reporting requirements
- Specifies minimum reporting levels for each compound
- Compliance point is the point of entry to the distribution system at each plant and Max Residence Time location for List 2 selected method

The 2 Lists

- List 1-Assessment Monitoring (AM)
  - 10 compounds by 2 methods
    - Explosives 3 compounds (EPA 529)
    - Insecticides 2 compounds (EPA 527)
    - Flame Retardants 5 compounds (EPA 527)
    - Perchlorate was on original list but removed
- List 2- Screening Survey (SS)
  - 15 compounds by 3 methods
    - Nitrosamines – 6 compounds (EPA 521)
    - Acetanilide Parents- 3 compounds (EPA 525.2)
    - Acetanilide Degradates- 6 compounds (EPA 535)

List 1- Compounds & MRL's

Class	Compound	Method	MRL (ppb)
Insecticides	Dimethoate	527	0.7
	Terbufos sulfone	527	0.4
Flame Retardants	BDE-47	527	0.3
	BDE-99	527	0.9
	BDE-153	527	0.8
	BDE-100	527	0.5
	HBB	527	0.7
Explosives	2,4,6-Trinitrotoluene (TNT)	529	0.8
	1,3-dinitrobenzene	529	0.8
	RDX	529	1.0

List 2- Compounds & MRL's

Class	Compound	Method	MRL (ppb)
Acetanilide parents	Acetochlor	525.2	2.0
	Alachlor	525.2	2.0
	Metolachlor	525.2	1.0
Acetanilide degradates	Acetochlor ethane sulfonic acid (ESA)	535	1.0
	Acetochlor oxanilic acid (OA)	535	2.0
	Alachlor ESA	535	1.0
	Alachlor OA	535	2.0
	Metolachlor ESA	535	1.0
	Metolachlor OA	535	2.0

List 2- Compounds & MRL's (cont.)

Class	Compound	Method	MRL (ppb)
Nitrosamines	N-nitroso-diethylamine (NDEA)	521	0.005
	N-nitroso-dimethylamine (NDMA)	521	0.002
	N-nitroso-di-n-butylamine (NDBA)	521	0.004
	N-nitroso-di-n-propylamine (NDPA)	521	0.007
	N-nitroso-methylethylamine (NMEA)	521	0.003
	N-nitroso-pyrrolidine (NPYR)	521	0.002

Sample Collection Procedures

Requirement	EPA Method	Minimum Volume	Preservative
List 1 (AM)	527	1 Liter Glass *	0.1 g Ascorbic Acid 0.35 g EDTA 9.4 G KH_2PO_4 Cool 6oC
	529	1 Liter Glass *	0.5 g $CuSO_4*H_2O$ 5.0 g Trizma Pre-Set pH 7 Buffer Cool 6oC
List 2 (SS)	521	0.5 Liter *	50 mg $Na_2S_2O_3$ Cool 6oC
	525.2	1 Liter *	0.05 g Na_2SO_3 (field acidified) Cool 6oC
	535	250 mL *	25 mgNH_4Cl Cool 6oC

Lab QC Requirements

- Must meet MRLs

  Demonstrated with:

  - Calibration Checks
    - After every 10th Field sample and End of a Batch
  - Daily Performance Checks
    - LFM at or Below MRL Level

- Lab Fortified Blank (<MRL)
- LFM, with each extraction batch or with every 20 samples

Data Quality

- Surrogate recoveries must be between 70-130% for all samples or resampling is required for UCMR2.

- While the methods state that closing Continuing Calibration Verifications may be out high if the samples are “ND,” the UCMR QA Manual does not allow that practice.

Exercise 1

UCMR Data Review

- Worksheet
- Data Package

Method vs Data Validation

- Method Compliance
- Contract Compliance
- QAPP Compliance
- Method Quality Objectives (MQOs) and Data Quality Objectives (DQOs)

Goals and Objectives of Data Validation

- **Why data are validated?**

  The main objective of data validation is to provide data of known quality. Data of known quality is essential for the decision making process, supporting regulatory enforcement, litigation, and policy decisions regarding human health and safety.

- **How do you obtain data of known quality?**

  To obtain data of known quality requires upfront planning, communication, and a rigorous QA/QC program prior to sample collection and analysis. These program requirements are considered in the final data review process which involves data validation by well trained and experienced personnel.

Understanding Data Validation Qualifiers

- Why are data qualified?

  Data are qualified to bring to the user's attention the increased uncertainty in a measurement. This means that qualified data will have uncertainty or bias in excess of our specified limits. The qualifier is a flag (typically an alpha character) which denotes the level of uncertainty.

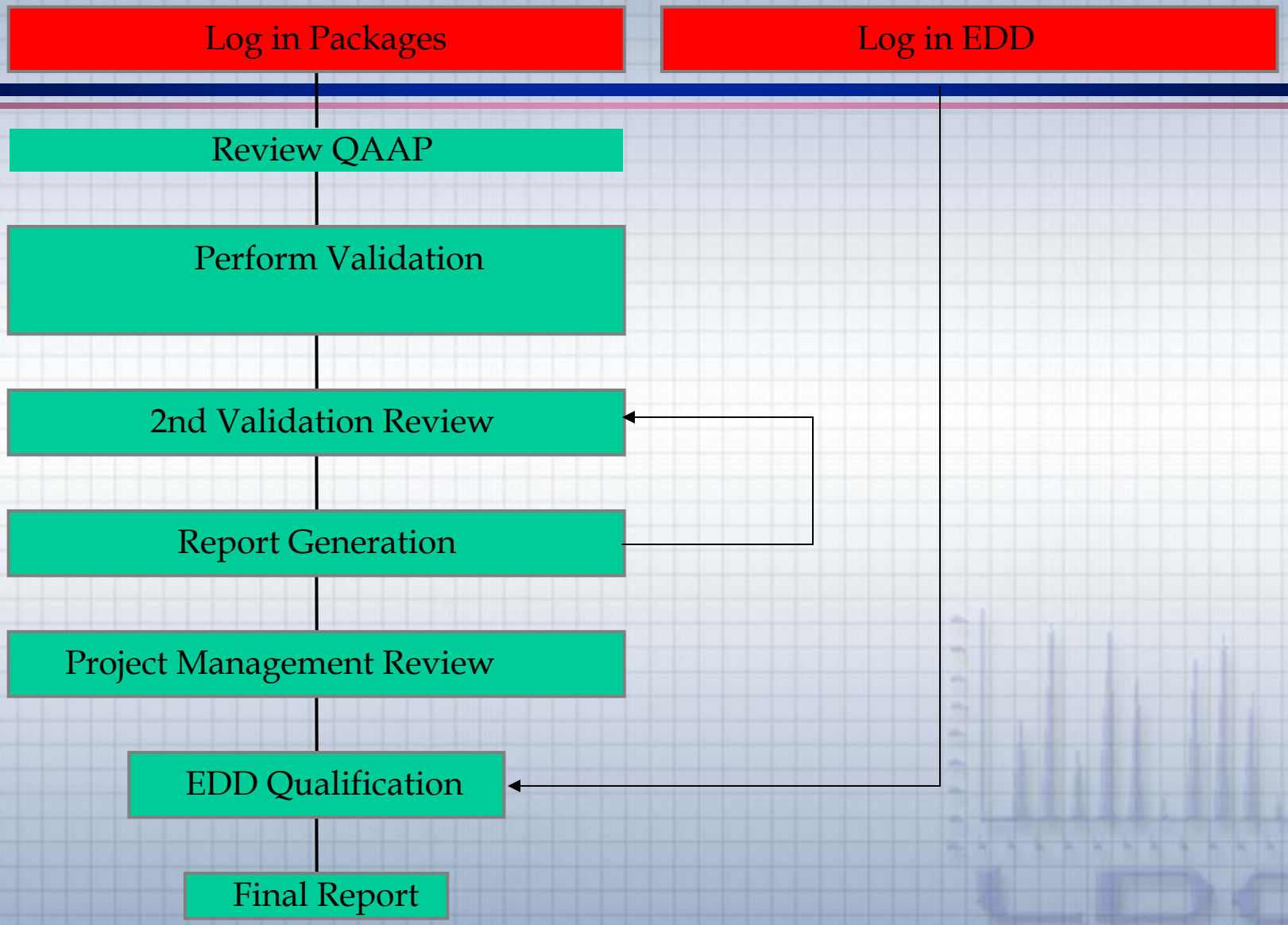
Data Deliverables/Validation

- EPA Level 2
- EPA Level 3
- EPA Level 4
- Cursory Review
- Full/Complete Review
- EPA Region IX, Tier 1A/1B and Tier 3
- AFCEE Level 1, Level 2

Tiered Data Review/Validation

- 90% Level 3 / 10% Level 4
- 80% Level 3 / 20% Level 4
- 10% Level 4 only
- 100% Level 3
- 100% Level 2
- 90% level 2/ 10% Level 4

Data Validation Process



EPA Qualifiers

- **U** Indicates the compound or analyte was analyzed for but not detected at or above the stated limit.
- **J** Indicates an estimated value.
- **R** Quality control indicates the data is not usable.
- **N** Presumptive evidence of presence of the constituent.
- **UJ** Indicates the compound or analyte was analyzed for but not detected. The sample detection limit is an estimated value.

Special Qualifiers

- Bias, positive (+) and minus (-)
- Region 3, L for low estimated
- AFCEE, M for matrix interference
- Florida DEP, Q holding times

Reason Codes

- Reason codes are flags linked to the validation qualifiers that associated the basis for the qualification
- For example, a MS outlier may require J flag a detect compound and the reason may be noted as #10, which means MS outlier

Reasons for Qualifying Data

- Results near the detection limit
- Calibration error
- QC samples with high or low spike recoveries
- Misidentification of compounds
- Results associated with method blank contamination
- Improper quantitation of concentration

Common reasons for "J" qualified data

- Results between the MDL and CRQL/RDL/MRL
- Instrument calibration does not meet criteria
- Samples exceed holding time
- Low or high MS or LCS spike recoveries
- Low or high surrogate recoveries

Usable or Unusable Data

- The Big “R”, rejected

Understanding Data Validation

- Data validators can be very conservative.
- Data validation requires professional judgement.
- Data validation is not data usability.
- DQOs should dictate the validation guidelines.
- Data validation does not make bad data good
- Data validation is not well understood by industry and regulators.

Chain of Custody

- Verify sample identification
- Verify sampling dates
- Verify sample preservation
- Confirm sample analyses requested
- Verify custody receipt and signatures
- Verify temperature of cooler upon receipt

Cooler Temperature

- Criteria generally 4 degrees C +/- 2 degrees
- Use professional judgment
- Greater than 10 degrees C, qualify
- Reject at 15 degrees C
- Less than 2 degrees C, no qualification
- ENCORE VOC samples or tissues, frozen if required

Preservation

- Confirm proper preservation
- Preservation may impact holding time
- Use professional judgment
- Data may be estimated or rejected

Holding Times

- Check date of collection vs date of analysis
- Verify holding time based on preservation if applicable
- Qualify as J detects/UJ non-detects
- Rejected (R) if gross outlier, use judgment

Misc Holding Times

			Soil		14 days to extract, 40 days to analyze
8081A	GC	Organochlorine Pesticide	Water Soil	Cool to 4°C	7 days to extract, 40 days to analyze 14 days to extract, 40 days to analyze
8082	GC	PCB	Water Soil	Cool to 4°C	7 days to extract, 40 days to analyze 14 days to extract, 40 days to analyze
8141A	GC	Organophosphorus Pesticide	Water Soil	Cool to 4°C	7 days to extract, 40 days to analyze 14 days to extract, 40 days to analyze
8151A	GC	Herbicide	Water Soil	Cool to 4°C	7 days to extract, 40 days to analyze 14 days to extract, 40 days to analyze
8260B	GCMS	Volatile	Water Soil	HCl to pH < 2; Cool to 4°C $HNaSO_4$; Cool to 4°C	14 days
8270C	GCMS	Semivolatile	Water Soil	Cool to 4°C	7 days to extract, 40 days to analyze 14 days to extract, 40 days to analyze
8310	HPLC	Polynuclear Aromatic Hydrocarbons	Water Soil	Cool to 4°C	7 days to extract, 40 days to analyze 14 days to extract, 40 days to analyze
8330	HPLC	Explosives	Water	Cool to 4°C	7 days to extract, 40 days to analyze

Instrument Tuning

- For GC/MS analyses
- Check ion abundance criteria
- BFB (VOC), DFTPP (SVOC)
- Make judgment on qualification based upon finding
- Typically NJ or R qualification

GC/MS Tuning Criteria

TABLE 4

BFB (4-BROMOFLUOROBENZENE) MASS INTENSITY CRITERIA[a]

m/z	Required Intensity (relative abundance)
50	15 to 40% of m/z 95
75	30 to 60% of m/z 95
95	Base peak, 100% relative abundance
96	5 to 9% of m/z 95
173	Less than 2% of m/z 174
174	Greater than 50% of m/z 95
175	5 to 9% of m/z 174
176	Greater than 95% but less than 101% of m/z 174
177	5 to 9% of m/z 176

[a] Alternate tuning criteria may be used, (e.g. CLP, Method 524.2, or manufacturers"

Instrument Calibration

- Initial Calibration
- Initial Calibration Verification
- Calibration Verification

Initial Instrument Calibration

- Multi point calibration
- 3-6 five point calibration, typical
- Evaluate linearity
- Percent Relative Standard Deviation (%RSD)
- Correlation Coefficient (r) or Coefficient of Determination (r-squared)
- Relative Response Factor (RRF) evaluation for GCMS

GC/MS System Check Compounds

m/z 95 may improve bromoform response.

7.3.5.3 Tetrachloroethane and 1,1-dichloroethane are degraded by contaminated transfer lines in purge-and-trap systems and/or active sites in trapping materials.

7.3.5.4 The minimum mean response factors for the volatile SPCCs are as follows:

Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

7.3.6 Calibration check compounds (CCCs)

7.3.6.1 The purpose of the CCCs are to evaluate the calibration from the standpoint of the integrity of the system. High variability for these compounds may be indicative of system leaks or reactive sites on the column. Meeting the CCC criteria is not a substitute for successful calibration of the target analytes using one of the approaches described in Sec. 7.0 of Method 8000.

7.3.6.2 Calculate the standard deviation (SD) and relative standard deviation

Linearity Calculation

To evaluate the linearity of the initial calibration, calculate the mean CF (external standard calibration) or RF (internal standard calibration), the standard deviation (SD), and the RSD as follows:

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (CF_i - \overline{CF})^2}{n-1}} \qquad SD = \sqrt{\frac{\sum_{i=1}^{n} (RF_i - \overline{RF})^2}{n-1}}$$

$$\text{mean CF} = \overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n} \qquad \text{mean RF} = \overline{RF} = \frac{\sum_{i=1}^{n} RF_i}{n}$$

$$RSD = \frac{SD}{\overline{CF}} \times 100 \qquad RSD = \frac{SD}{\overline{RF}} \times 100$$

where *n* is the number of calibration standards and RSD is expressed as a percentage (%).

11.5.1.1 If the RSD of the calibration or response factors is less than or equal to 20% over the calibration range, then the slopes of the lines for each standard are

Linear Regression

where SD is the standard deviation of the replicate results at each individual standard concentration.

The regression will produce the slope and intercept terms for a linear equation in the form:

$$y = ax + b$$

where:

- y = Instrument response (peak area or height)
- a = Slope of the line (also called the coefficient of x)
- x = Concentration of the calibration standard
- b = The intercept

The analyst should not force the line through the origin, but have the intercept calculated from the five data points. Otherwise, the problems noted with the RSD value will occur, i.e., a line through the origin will not meet the QC specifications. In addition, do not include the origin (0,0) as a sixth calibration point. The use of a linear regression may not be used as a rationale for reporting results below the calibration range demonstrated by the analysis of the standards. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.99.

In calculating sample concentrations by the external standard method, the regression equation is rearranged to solve for the concentration (x), as shown below.

$$x = \frac{(y - b)}{}$$

Initial Calibration Verification

- Verify initial compliance against the initial calibration
- Verify use of secondary source
- Evaluate percent difference
- Relative Response Factor (RRF) evaluation for GCMS
- Proper frequency, 12 hr, daily, every 10 samples

Calibration Verification

- Verify on-going compliance against the initial calibration
- Same source as initial calibration
- Evaluate percent difference
- Relative Response Factor (RRF) evaluation for GCMS

Percent Difference CCV

of the standard using the % difference. The criteria for acceptability based upon the additional check would have a similar impact upon the usability of a calibration for quantitation as is discussed in the above paragraph.

11.5.5.1 Re-fitting the calibration data back to the model or calculating the % difference is determined by using the following equation:

$$\% \text{ Difference} = \frac{C_c - C_e}{C_e} \times 100$$

where:

C_c = Calculated amount of standard, in mass or concentration units.
C_e = Expected amount of standard, in mass or concentration units.

The absolute value of the percent difference between these two amounts for every calibration level should be less than or equal to 20%.

NOTE: If every point in a 5-point calibration were off by 20%, then the RSD from an average CF or RF would be over 20%, and an r/COD/r^2 value from a least squares regression would be less than 0.99. It is more likely that for any calibration model chosen, all levels will not be close to 20%. It is also more likely that just one or two levels will exceed the 20% criterion, while still meeting the RSD/r/COD/r^2 criteria. Thus, when RSD/r/COD/r^2 statistics are used in conjunction with the inspection of the curve, more control is placed on the calibration model

Method Specific QC Checks

- ICSA/ICSAB, 80-120% recovery
- Pesticide DDT/Endrin breakdown, ± 15%
- ICP Serial Dilution, ± 10%
- GC/MS Semivolatile Tailing Factor, PCP< 5, Benzidine <3

DDT/Endrin Breakdown

NOTE: If during the initial calibration, linearity was determined based on peak area for the compound, then the midpoint CF must be based on peak area. If during the initial calibration, the linearity for the compound was determined based on peak height for the compound, then the midpoint CF must be based on peak height.

EQ. 6

$$\%\text{Breakdown DDT} = \frac{\text{Amount found (ng) (DDD+DDE)}}{\text{Amount (ng) of DDT injected}} \times 100$$

EQ. 7

$$\%\text{Breakdown Endrin} = \frac{\text{Amount found (ng) (endrin aldehyde + endrin ketone)}}{\text{Amount (ng) of endrin injected}} \times 100$$

EQ. 8

$$\text{Combined \%Breakdown} = \%\text{Breakdown DDT} + \%\text{Breakdown Endrin}$$

9.2.4.9 Calculate the percent difference for each pesticide and surrogate in the PEM using Equations 5 and 9.

EQ. 9

$$\%D = \frac{C_{calc} - C_{nom}}{C_{nom}} \times 100$$

Where:

%D = Percent Difference.

C_{nom} = Nominal concentration of each analyte.

C_{calc} = Calculated concentration of each analyte from the analysis of the standard.

9.2.4.10 Calculate the resolution between the analytes in the Resolution Check Mixture, PEM and the midpoint concentrations of Individual

Method Blanks

- Evaluate concentrations in the method blank against the associated samples in the prep batch
- Use NFG 5 or 10 times rule
- 10X for common contaminants
- Factor in dilution of samples

Field QC Blanks

- Evaluate concentrations in the trip blank, ambient blank, field blank and equipment blank against the associated samples in the prep batch
- Use NFG 5 or 10 times rule
- 10X for common contaminants
- Factor in dilution of samples

Surrogates

- Evaluate surrogate recovery against validation criteria
- Recoveries below lower limit, low bias
- Low bias, J (detects), UJ (non-detects)
- Recoveries above upper limit, high bias
- High bias, J (detects), UJ (no qual)
- Matrix interference may dilute surrogates

Surrogate Percent Recovery Calc.

percent recovery in the matrix spike (or matrix spike duplicate) falls outside the designated range for recovery, the laboratory should determine if there is a matrix effect or a laboratory performance problem. A matrix effect is indicated if the LCS data are within limits but the matrix spike data exceed the limits. The surrogate recovery data (Sec. 9.6) should also be used to evaluate the data. Recoveries of both matrix spike compounds and surrogates that are outside of the acceptance limits suggest more pervasive analytical problems than problems with the recoveries of either matrix spikes or surrogates alone.

9.6 Surrogate recoveries

9.6.1 It is necessary that the laboratory evaluate surrogate recovery data from individual samples versus surrogate recovery limits developed in the laboratory. The general considerations for developing in-house acceptance criteria for surrogate recoveries are described in Sec. 9.7.

9.6.2 Surrogate recovery is calculated as:

$$\text{Recovery (\%)} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) added}} \times 100$$

Lab Fortified Matrix Samples

- Evaluate LFM sample recovery against validation recovery criteria
- Recoveries below lower limit, low bias
- Low bias, J (detects), UJ (non-detects)
- Recoveries above upper limit, high bias
- High bias, J (detects), UJ (no qual)
- Evaluate laboratory control duplicate sample RPD against validation RPD criteria
- RPD outside limits, J/UJ associated samples
- Qualify all samples and associated analytes in the batch

Percent Recovery Calc.

Analyze the MS/MSD samples using the same procedures employed for the original sample, and calculate the concentration of each analyte in the matrix spike and matrix spike duplicate. Likewise, analyze the LCS samples using the same procedures employed for the original sample, and calculate the concentration of each analyte in the LCS.

9.5.3.1 Calculation of recovery

Accuracy is estimated from the recovery of spiked analytes from the matrix of interest. Laboratory performance in a clean matrix is estimated from the recovery of analytes in the LCS. Calculate the recovery of each spiked analyte in the matrix spike, matrix spike duplicate (if performed) and LCS according to the following formula.

$$\text{Recovery} = \%R = \frac{C_s - C_u}{C_n} \times 100$$

where:

C_s = Measured concentration of the spiked sample aliquot.
C_u = Measured concentration of the unspiked sample aliquot (use 0 for the LCS).
C_n = Nominal (theoretical) concentration increase that results from spiking the sample, or the nominal concentration of the spiked aliquot (for LCS).

9.5.3.2 Calculation of precision

Precision is estimated from the relative percent difference (RPD) of the concentrations (*not* the recoveries) measured for matrix spike/matrix spike duplicate

Matrix Spike Samples

- Evaluate matrix spike sample recovery against validation recovery criteria
- Recoveries below lower limit, low bias
- Low bias, J (detects), UJ (non-detects)
- Recoveries above upper limit, high bias
- High bias, J (detects), UJ (no qual)
- Evaluate matrix spike duplicate sample RPD against validation RPD criteria
- RPD outside limits, J/UJ associated samples
- Qualify all samples and associated analytes in the batch

Relative Percent Difference Calc.

pairs, or for duplicate analyses of unspiked samples. Calculate the RPD according to the formula below.

$$RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100$$

where:

C_1 = Measured concentration of the first sample aliquot.
C_2 = Measured concentration of the second sample aliquot.

9.5.4 Recommended QC acceptance criteria for matrix spike samples and LCS

It is necessary for the laboratory to develop single-laboratory performance data for accuracy and precision in the matrices of interest (see Sec. 9.7). In addition, laboratories should monitor method performance in each matrix, through the use of control charts and other techniques.

Many methods may not contain recommended acceptance criteria for LCS results. The laboratory should use 70 - 130% as interim acceptance criteria for recoveries of spiked analytes, until in-house LCS limits are developed (see Sec. 9.7). Where in-house limits have been developed for matrix spike recoveries, the LCS results should fall within those limits, as the LCS is prepared in a clean matrix.

Even where the determinative methods provide performance criteria for matrix spikes

Internal Standards

- Evaluate internal standard response against validation recovery criteria
- Recoveries below lower limit, low bias
- Low bias, J (detects), UJ (non-detects)
- Recoveries above upper limit, high bias
- High bias, J (detects), UJ (no qual)
- For GC/MS, +100% upper limit, - 50% lower limit
- For ICP/MS,
- Qualify sample specific analytes

Compound Identification

- Evaluate criteria for identification such as retention time for GC analyses, mass spectra for GC/MS and second column analysis for GC analyses
- For retention times, peak must fall within the window established in the SOP.
- For mass spectra, primary peaks (i.e., top 3-5) much match within 20% of library spectra.
- For second column analysis, criteria same as the primary column

Relative Retention Time

response is calculated relative to that of the internal standard.

The advantages of internal standard calibration include the fact that it can be used to account for routine variation in the response of the chromatographic system as well as variations in the exact volume of sample or sample extract introduced into the chromatographic system. In addition to normalizing the response (peak area) of the target compound to the response of the internal standard in that sample or extract for that injection, the retention times of the target compound and the internal standard may be used to calculate the relative retention time (RRT) of the target compound.

The RRT is expressed as a unitless quantity:

$$\text{RRT} = \frac{\text{Retention time of the analyte}}{\text{Retention time of the internal standard}}$$

The RRT of each target analyte in each calibration standard should agree within ± 0.06 RRT units. It is recognized here that with increasing retention times of the internal standard, target analytes will be able to more easily meet this criterion. Thus, care should be exercised when selecting the appropriate internal standards by retention times. The process of selecting internal standards to quantify target analytes should also include consideration of retention times as they should be similar.

The RRT of the sample component should be within ± 0.06 RRT units of the RRT of the standard component. If this criterion is not met and unless there are no other indicators of a component's identification such as a very unique but a high probability mass spectral match then that component may not be considered as identified by relative retention time.

The RRT evaluation allows the analyst to compensate for modest shifts in the

Compound Quantitation

- Evaluate criteria for quantitation such as formulas and calculations used for calibration response factors, linearity assessment, and final analyte concentration
- Must use all contributing data and attain the data from raw data printouts

Compound Quantitation

the circumstances and specific project requirements there may be exceptions for other options such as data qualification and alternative means of target analyte quantitation.

11.5.1.4 Calculation of sample amounts

If all of the conditions in Secs. 11.5.1.1 and 11.5.1.2 are met, then the average calibration or response factor may be used to determine sample concentrations, as described in Sec. 11.10. It is recommended that the curve generated by the average calibration or response factor be examined for acceptability using the re-fitting check described in Section 11.5.5.1. The calculations for the amount introduced into the instrument, x_s, are:

$$x_s = \frac{A_s}{\overline{CF}} \quad \text{and} \quad x_s = \frac{A_s}{\overline{RF}} \times \frac{C_{is}}{A_{is}}$$

where:

x_s = Calculated mass of the analyte or surrogate in the sample aliquot introduced into the instrument (in nanograms).

A_s = Peak area (or height) of the analyte or surrogate in the sample.

A_{is} = Peak area (or height) of the internal standard in the sample.

C_{is} = Mass of the internal standard in the sample aliquot introduced into the instrument (in nanograms).

$\overline{CF}$ = The average calibration factor from the most recent initial calibration.

$\overline{RF}$ = The average response factor from the most recent initial calibration.

The units for the mass of analyte should be the same units used to calculate the calibration or response factors. If alternate units are used for the amount (e.g., µg/L),

GC/MS Compound Quantitation

- Concentration = $\frac{(Ax)(Is)(Vt)(DF)(2.0)}{(Ais)(ave\ RRF)(Vo)(Vi)(\%S)}$

Ax =	Area of the characteristic ion (EICP) for the compound to be measured
Ais =	Area of the characteristic ion (EICP) for the specific internal standard
Is =	Amount of internal standard added in nanograms (ng)
Vo =	Volume or weight of sample extract in milliliters (ml) or grams (g).
VI =	Volume of extract injected in microliters (ul)
Vt =	Volume of the concentrated extract in microliters (ul)
Df =	Dilution Factor.
%S =	Percent solids, applicable to soil and solid matrices only.

Field Duplicates

- Evaluate field duplicate sample relative percent difference against validation RPD criteria
- RPD outside limits, maybe J/UJ associated samples
- Use professional judgment to assess whether or not to qualify samples

Overall Assessment

- Review all individual qualifiers and determine the additive impact of the outliers
- Communicate impact of qualifiers to the data user
- Use professional judgment as required and discuss reasons

Reference Documents

- **EPA National Functional Guidelines, Oct 1999 (Organics)**
- **EPA National Functional Guidelines, Oct 2004 (Inorganics)**
- **EPA National Functional Guidelines for Chlorinated Dioxin/Furan Data Review, Sept 2005**
- **Guidance on Environmental Data Verification and Data Validation, EPA QA/G-8, November 2002**
- **EPA National Functional Guidelines for Low Concentration Organics, June 2001**
- **EPA National Functional Guidelines, Draft, Jan 2005 (Organics)**

Reference Documents

- **EPA Region III Innovative Approaches to Data Validation, June 1995**
- **EPA Region III Modifications to NFG for Organics, Sept 1994**
- **Region III Modifications to NFG for Inorganics, April 1993**
- **EPA Region 1 Functional Guidelines for Organics, December 1996**
- **EPA Region 1 Functional Guidelines for Inorganics, February 1989**
- **Region 9 Superfund Data Evaluation/Validation Guidance, December 2001Draft**

Data Integrity

- Data integrity has become critical to successful lab operations
- NELAC Chapter 5, Quality Systems discusses a laboratory data integrity program
- EPA investigations have become more prominent and a routine way of life

Thanks to OIG Office

- Bruce Woods, OIG Chemist
  USEPA
  Office of Inspector General
  1200 Pennsylvania Avenue, N.W.
  Washington, D.C. 20460

Where LAB Fraud Occurs

Organics – 54%

Entire Lab – 17 %

Inorganics- 14 %

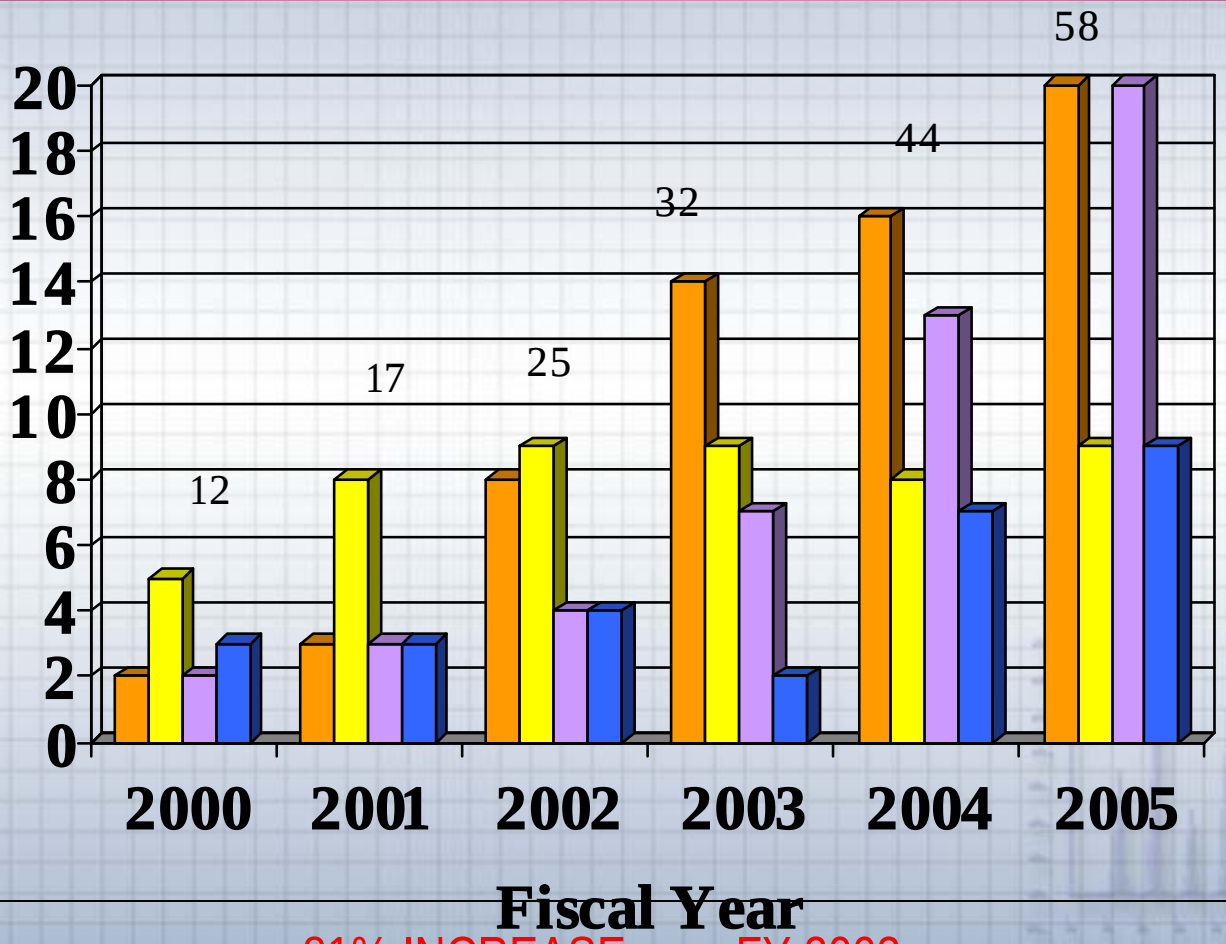
Microbiology – 6%

Air – 3 %

Asbestos – 3 %

Other – 3 %

Pending Cases by Region



OIG Evaluation Report

- Promising Techniques Identified to Improve Drinking Water laboratory Integrity and Reduce Public Health Risks

  USEPA OIG

  Report No. 2006-P-00036

  September 21, 2006

Going Beyond the Data Package

- Fraud Issues made it mandatory for labs to implement a system that verified raw data files, independently reproduce results and compare to hardcopy.
- Only a small percentage of data can be reviewed in such manner, typically by QA.
- There became need to verify authenticity of data integrity at the bench level, hence the need for a Data Integrity Program.

Definitions of Improper Practice

- A scientifically unsound or technically unjustified omission, manipulation or alteration of analytical procedures.
- Data by passing quality control parameters, making the results appear acceptable
- Any alteration of data such that the data are untrue representations of the test performed
- Altering or otherwise compromising analytical reports without authorization

Data Integrity Problems

- **Time traveling**
- **Peak “shaving”**
- **Juicing**
- **Cut and paste**
- **Dry labbing**
- **Manipulation of documentation**

Types of Inappropriate Practices

- Fabrication of data or supporting information
- Misrepresentation of QC samples results
- Improper date/time setting
- Improper peak integration
- Improper GC/MS tuning
- Improper calibration and verification

Types of Inappropriate Practices(cont.)

- Data file substitution or modification
- Unwarranted sample dilution
- Deletion of non-compliant data
- Improper alteration of analytical conditions
- Unwarranted manipulation of computer software
- Concealment of a known problem

Fabrication

DO NOT :

- **Fabricate or make up data (dry lab)**
- **Create data or information that is not true**
- **Intentionally use incorrect analyte concentrations for standards or QC standards with the intent to change apparent recoveries**
  - ➢ **"Juicing"- manipulating a sample prior to analysis by fortifying the sample with additional analyte to achieve good response. Another term is "over-spiking": adding more than the prescribed spike volume.**
    - ➢➢**Indication is a jump to high response from historical low response.**

Fabrication (cont.)

DO NOT :

- **Create data for an analysis that was not performed**
- **Create information for a sample that was not collected**
- **Cut and paste data or information into reports or data that are not true**
- **Intentionally subcontract PT samples and representing data or results as your own**

Misrepresentation

DO NOT:

- Claim ownership for work performed by external analysts, equipment, or facilities
- Misrepresent QC Samples as digested or extracted when it has not been prepped
- Report post-digested QC as pre-digested
- Prepare or analyze QC samples, PTs, or Standards under analytical conditions different from samples
- Add surrogates into samples after sample extraction unless specifically called for by the method or procedure
- Use more or less surrogate to already extracted samples to enhance recovery
- Use more or less than the prescribed amount of spike solution without adjusting the calculation

Improper Date/Time-Setting

DO NOT:

- Record date or time incorrectly or set clocks improperly (Time Travel)
- Alter recorded times that samples were collected, extracted or analyzed
- Set instrument clocks to make it appear that samples were analyzed within holding time
- Alter dates/time on printouts or screen to meet 12 hour tuning windows

Improper Peak Integration

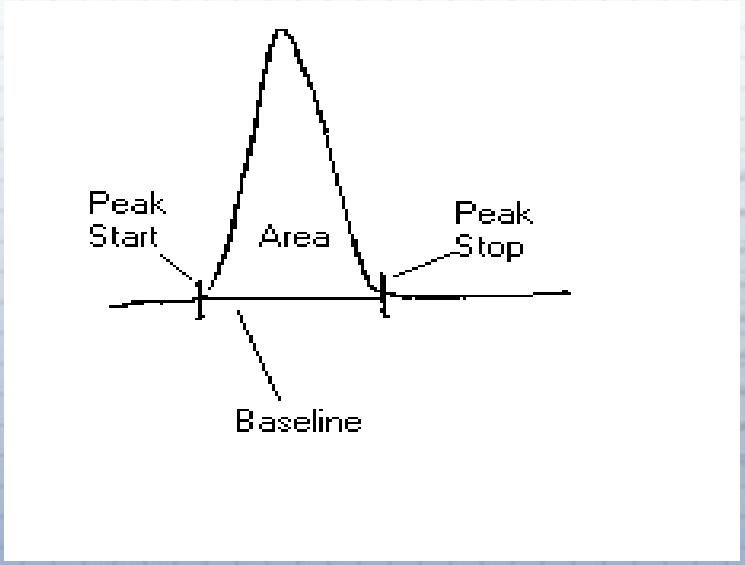
DO NOT:

- **Perform technically unsound or improper peak integrations in order to meet QC criteria**
- **Inappropriately subtract an interfering peak for the purpose of passing calibration or QC**
- **Intentionally integrate a compound and represent it as another compound for the purpose of passing calibration or QC**

➢**It is imperative that the lab establishes a Manual integration SOP based on the most stringent policies for appropriate and acceptable techniques; supervisory review; presentation of before and after data, and consistency. Data audit trails are essential to this objective.**

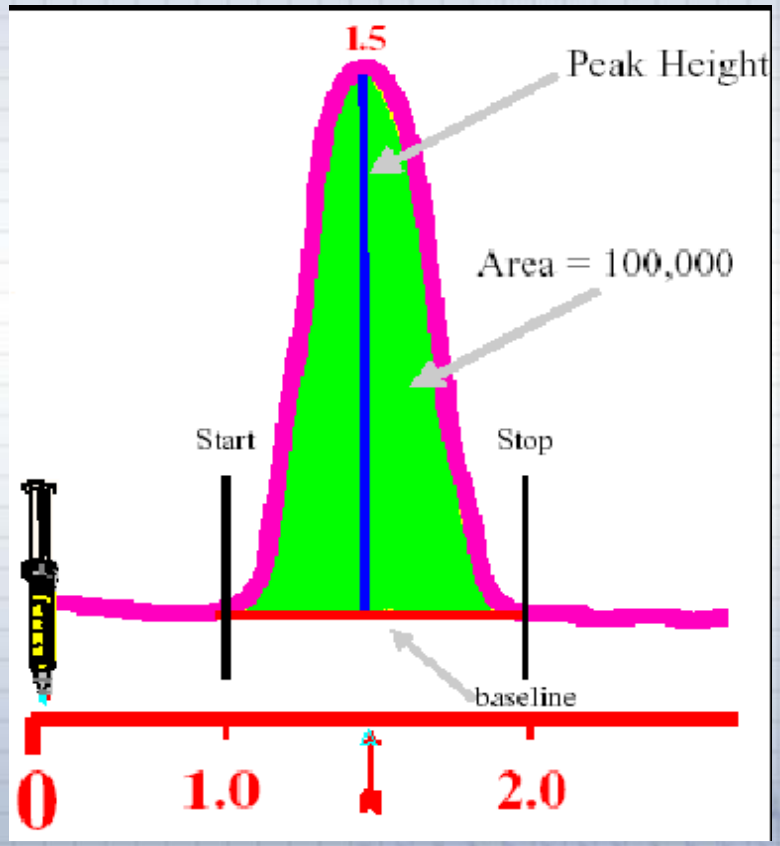
Examples of Peak shape and Proper integration Documenting Manual Integrations

Ideal peak
(Gaussian Shape)
The start of integration is where the peak begins to rise from the background, and the end is where the peak returns to background



Normal Peak Integration

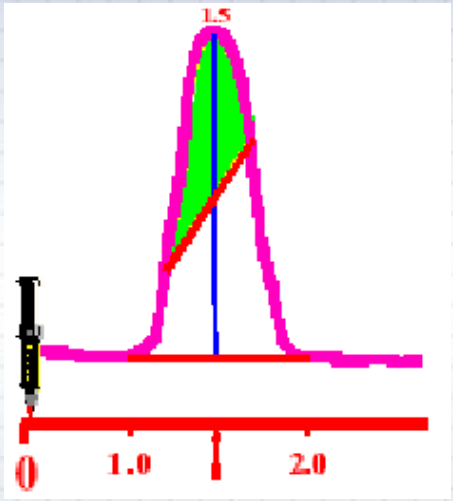
Name	RT		Area	Amount
8) 2-Chlorophenol ug/L	7.12	128	634775	67.57
10) 1,4-Dichlorophenol ug/L	7.33	146	250996	24.25
17) N-Nitroso-di-propylamine ug/L	7.87	70	253477	38.19



Peak Shaving: Less Area

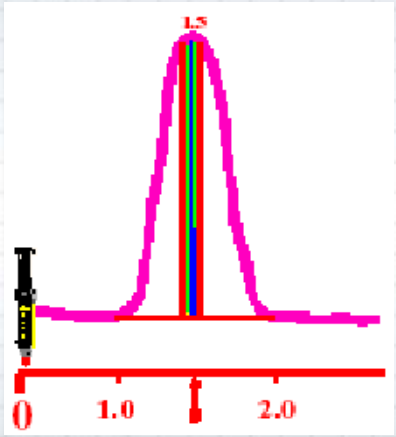
Std Area = 100,000

Sample Result = 1,000 ug/Kg



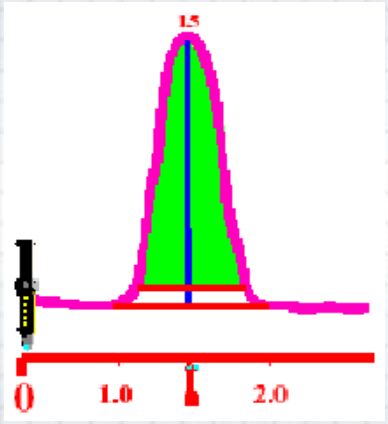
Std Area = 40,000

Sample Result = 2,500 ug/Kg



Std Area = 5,000

Sample Result = 20,000 ug/Kg



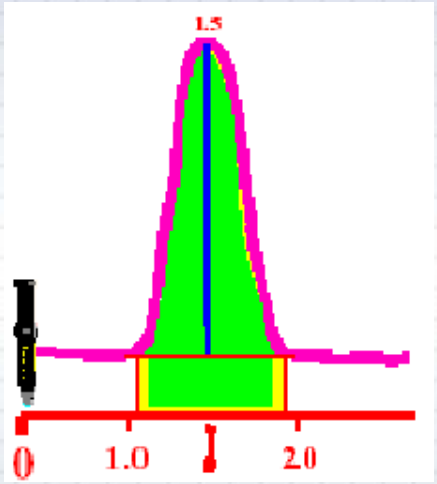
Std Area = 90,000

Sample Result = 1,111 ug/Kg

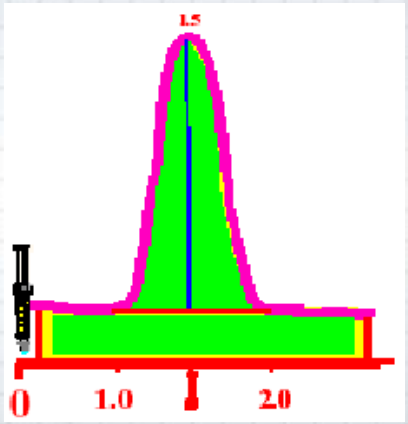
Name	RT		Area	Amount	
Target Compounds					
5) Phenol	7.02	94	79878m	62.71	ug/L

Peak Addition: More Area

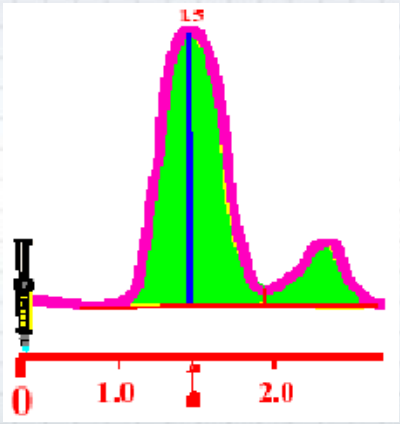
Std Area = 100,000
Sample Result = 1,000 ug/Kg



Std Area = 130,000
Sample Result = 769 ug/Kg



Std Area = 200,000
Sample Result = 500 ug/Kg



Std Area = 130,000
Sample Result = 769 ug/Kg

Name	RT		Area	Amount	
Target Compounds					
5) Phenol	7.02	94	79878m	62.71	ug/L

Improper GC/MS Tuning

DO NOT:

- **Manipulate GC/MS tuning data to artificially produce an ion abundance that appear to meet specified criteria when it did not**
- **Choose non-representative scan(s) for evaluation**
- **Perform incorrect background subtraction**
- **Inject incorrect amounts of BFB surrogate in CCV**
- **Copy and rename files in order to make an instrument appear to pass tuning criteria when in fact it has not**
- **Add spectra from two different files**

Improper Calibration/Verification

DO NOT:

- **Deviate from proper technically sound calibration practices**
- **Manipulate instruments or software to make calibrations to appear to pass acceptance criteria**
- **Perform multiple calibration runs until finally passing without corrective action or maintenance in between**
- **Delete or disregard analyte responses from the center of a calibration curve without solid technical justification**
- **Insert calibration levels or CCVs run well after the initial calibration curve**

Data File Substitution/Modification

DO NOT:

- **Substitute data files from a previous generated analytical run to make it appear that the calibration or QC were within acceptance limits**
- **Substitute data files from a previous generated analyses for non-compliant calibration, QC or samples runs**
- **Reuse historical initial calibration data and represent it as current**
- **Improperly change sample IDs in data files**

Unwarranted Sample Dilution

DO NOT:

- **Dilute samples or blanks, without documentation, explanation and technical justification, in order to eliminate target analyte responses**
- **Do not dilute samples or blanks to reduce the appearance of contamination**
- **Do not dilute sample extracts of failing surrogate recovery in order to narrate incalculable percent recovery due to dilution**

Deletion of Non-Compliant Data

DO NOT:

- **Delete non-compliant analytical results for calibrations, QC samples or blanks with the intent of concealing significant instrument problems**
- **Fail to record non-compliant data for these samples**
- **Delete non-compliant contaminants from method blanks**
- **Record only analyses that pass in logbooks or other records**

Improper Alteration of Analytical Conditions

DO NOT:

- **Alter analytical conditions between samples, standards, QC samples without appropriate, documented and technical justification**
- **Change analytical or instrument conditions from those stated in the method or SOP without documentation and approval**
- **Fail to run standards, samples and QC samples under the same conditions unless specifically called for in the methods**
- **Attempt to conceal instrument problems by failing to allow instruments to equilibrate and reach stable conditions prior to analyses**

Unwarranted Software Manipulation

DO NOT:

- **Disable automated data trail or flagging systems**
- **Remove operational codes to eliminate or hide manipulations or manual integration flags**
- **Inappropriately adjust baselines**
- **Improperly change calculations or algorithms for the purpose of passing calibration or QC**
- **Use screen printing to hide inappropriate editing of data such as failing %RSDs or manually integration “m” flags**

Improper Practices Deterrence

- **QA/QC requirements “Known”**
- **Pre-award audits**
- **PT samples**
- **Split sample analysis**
- **Data validation**
- **Third party electronic tape audits**
- **Internal lab electronic audits**
- **Strong QA/QC officer**

Mint Miner

Original File: C:\DATA\SAMPLES\EQMS\SMPL03.D
Acquired: 2/13/2000 11:38:00 PM
Operator: CDI
Instrument: GCMS6
Sample: 20ng #12345 (VOA CAL #2)

Status: Scanned
Attention level: 22
Alert Codes: MP(3), M(3), MM(1), MT(1)

Flagged Events | All Events | Quant Report

#	Analyte	Code	Event	Time	Alert
1	Chloromethane	MP	Changed peak amount for Chloromethane from 12.8259ng to 17.59ng	Feb-15-00 16:06	Manual integration of an SPCC
2		M	Changed peak amount for Chloromethane from 12.8259ng to 17.59ng	Feb-15-00 16:06	Manual integration (non-specific)
3		MM	Changed peak amount for Chloromethane from 17.59ng to 18.1884ng	Feb-16-00 16:29	More than 1 manual integrations of the same analyte
4		MT	Changed peak amount for Chloromethane from 17.59ng to 18.1884ng	Feb-16-00 16:29	Multiple manual integrations of the same analyte >1 minutes apart
5		MP	Changed peak amount for Chloromethane from 17.59ng to 18.1884ng	Feb-16-00 16:29	Manual integration of an SPCC
6		M	Changed peak amount for Chloromethane from 17.59ng to 18.1884ng	Feb-16-00 16:29	Manual integration (non-specific)
7		MP	Changed peak amount for Chloromethane from 18.1884ng to 20.9616ng	Feb-16-00 16:29	Manual integration of an SPCC
8		M	Changed peak amount for Chloromethane from 18.1884ng to 20.9616ng	Feb-16-00 16:29	Manual integration (non-specific)

Laboratory Data Consultants, Inc

Mint Miner

Original File: C:\DATA\SAMPLES\EQMS\SMPL10.D
Acquired: 2/18/2000 6:34:00 PM
Operator: COL
Instrument: GCMS6
Sample: 20ng #12345 (CCAL)

Status: Scanned
Attention level: 23
Alert Codes: MB(2), M(6), MM(2), MT(1)

Flagged Events | All Events | Quant Report

#	Event	Description	Analyte	Time	RES File
1	Quantitation	Calculation using initial calibration	VOA.RES	Feb-19-00 13:27	VOA.RES
2	Report Generation	Generated Report using option: S		Feb-19-00 13:27	VOA.RES
3	Manual Integration	Changed peak amount for 2-Butanone (MEK) from 10.6867ng to 16.6611ng	2-Butanone (MEK)	Mar-04-00 11:10	VOA.RES
4		Changed peak amount for 2-Butanone (MEK) from 16.6611ng to 17.6583ng	2-Butanone (MEK)	Mar-04-00 11:13	VOA.RES
5		Changed peak amount for Dibromomethane from 14.1029ng to 14.7062ng	Dibromomethane	Mar-04-00 11:14	VOA.RES
6		Changed peak amount for Dibromomethane from 14.7062ng to 37.6746ng	Dibromomethane	Mar-04-00 11:14	VOA.RES
7		Changed peak amount for Dibromomethane from 37.6746ng to 14.7066ng	Dibromomethane	Mar-04-00 11:14	VOA.RES
8		Changed peak amount for 4-Methyl-2-pentanone (MIBK) from 10.8435ng to 10.9779ng	4-Methyl-2-pentanone (MIBK)	Mar-04-00 11:17	VOA.RES

Laboratory Data Consultants, Inc.
Data Integrity Training
MintMiner: Scanned CCal Report- Manual Integration

Improper Practices Detection

- **Poor internal corrective action program**
- **Poor PT results**
- **Lack of responsiveness**
- **Lack of management commitment**
- **Unexplained lack of access to records or staff during audits**
- **General poor data quality (i.e. unstable calibration in GC or GC/MS areas)**
- **Staff hesitant to answer questions during audits**
- **Perfect QC results especially for difficult compounds**

Improper Practices Detection

- **No-fault QA mechanism for staff missing**
- **Production over quality**
- **Inadequate system for peer and supervisor review of data**
- **Inadequate staff training**
- **Inadequate expertise by the staff**
- **Data retrieval from archives difficult**
- **Selected data internally validated**
- **Disabled audit trail functions**

ASSESSMENT PROCESS FLOW CHART

PRE-ASSESSMENT ACTIVITIES

ON-SITE ASSESSMENT PROCESS

DATA ASSESSMENT

Determine Scope of Assessment

Select Assessment Team

Notify Laboratory & Request Records

Conduct Pre-Assessment Records Review

Plan On-Site Assessment

Determine Team Assignments

On-Site Assessment

Opening Meeting & Laboratory Tour

Quality Systems and Management Assessment

Quality Systems Documentation

Organization and Functions

Training Records

Management Interviews

Records Management & Archives

Computer Access & Security (LIMS, Data Systems)

Data Integrity Program (Records/ Policy)

Internal Audits

Corrective Action

Document Control

Sample Receipt and Storage Assessment

SOPs

Process Demonstration

Sample Receipt and Storage Records

Sample Custodian Interviews

Security

Sample Storage

Glassware and Bottle Preparation

Technical Area Assessments

SOPs

Process Demonstration

Logbooks, Bench Sheets, Maintenance Records

Analyst Interviews

Access and Computer Security

Data Reduction and Review (Process)

Equipment and Facilities

Process Waste Disposal and Material Throughput

ELECTRONIC DATA

Audit Trail

Data Transfer (from technical areas to reporting)

Summary Reports

Case Narratives

HARD COPY DATA

Archives - Original - Copies

RED FLAGS: PRE-ASSESSMENT

- Lack of documented assessment follow-up or corrective action
- Questionable proficiency
- Questionable technical depth
- Recent, significant, organizational changes
- Lack of responsiveness
- Lack of management commitment to data integrity

RED FLAGS: OPENING MEETING & LAB TOUR

Time wasters (e.g. lengthy health and safety overview, non-relevant briefings (e.g. history, sales)

Selected staff not available for interviews

Housekeeping practices

Unexplained, restricted access

RED FLAGS: QUALITY SYSTEMS AND MANAGEMENT ASSESSMENT

Lack of data integrity policy

Unclear communication of either expected behavior or improper practices

Lack of a "no-fault" reporting mechanism

Emphasis on production

Inadequate back-up (personnel and equipment)

Poor coordination for accepting new work

Excessive overtime

"Bottleneck" departments

Lack of technical depth

High staff turnover

Gaps in training records

QA Staff lacks direct access to senior management

Unclear roles and responsibilities

QA staff performing competing functions

Inadequate internal assessments

Lack of data surveillance

Incomplete assessment files

Repeat assessment findings

Inadequate procedures for handling complaints

Corrective action fails to address root cause

Lack of timely follow-up or documentation of corrective action

Lack of record verification in archiving procedures

Inadequate procedures to ensure data retrieval

RED FLAGS: SAMPLE RECEIPT & TECHNICAL AREA ASSESSMENTS

Denials of access or conditional access

Attempts to distract the assessment team

Records not readily accessible

Discrepancies between method and SOP

Referenced methods out of date

Presence of pencils, white-out

Extremely clean, neat logbooks

Uncontrolled records

Logbook entries all aligned

Discrepancies between SOP and practice

Expired standards

Disabled audit trails

Shared log-on and password access

Unlabeled containers or illegible labels

Sample volume discrepancies

Materials inventory does not match throughput

Sample throughput exceeds time required to process the samples

Managers translating or clarifying analysts' responses to questions

Emphasis on production

Inadequate sample-handling procedures for evening or weekend deliveries

No mechanism for reporting problems

Analysts not allowed to use their judgment

Analysts unable to describe data review and/or oversight

RED FLAGS: DATA ASSESSMENT

- Incomplete case narratives
- Unexpected sample results
- Too-perfect QC results
- Reports missing secondary reviewer signatures or dates
- Discrepancies between CoC and reports
- Unexplained gaps/changes in records
- Discrepancies in QC performance between analysts
- Calibration records missing reviewer signature/dates
- "Reappearing" quality control results
- Screen-printed reports
- Routine dead time on auto-injection run logs
- Samples or blanks diluted without apparent justification
- Too few quality control records to support data output
- Chronological chromatograms that do not display prevalent background

What is Automated Data Review?

Automated Data Review (ADR)

- Software developed by LDC for the Sacramento District of the US Army Corps of Engineers and commercial clients in the late 1990s
- Designed to automate, streamline and establish consistency in data review and data validation processes on 100% of data
- Produces reports that summarize QC outliers and qualified data
- Exports qualified data for upload to an environmental database

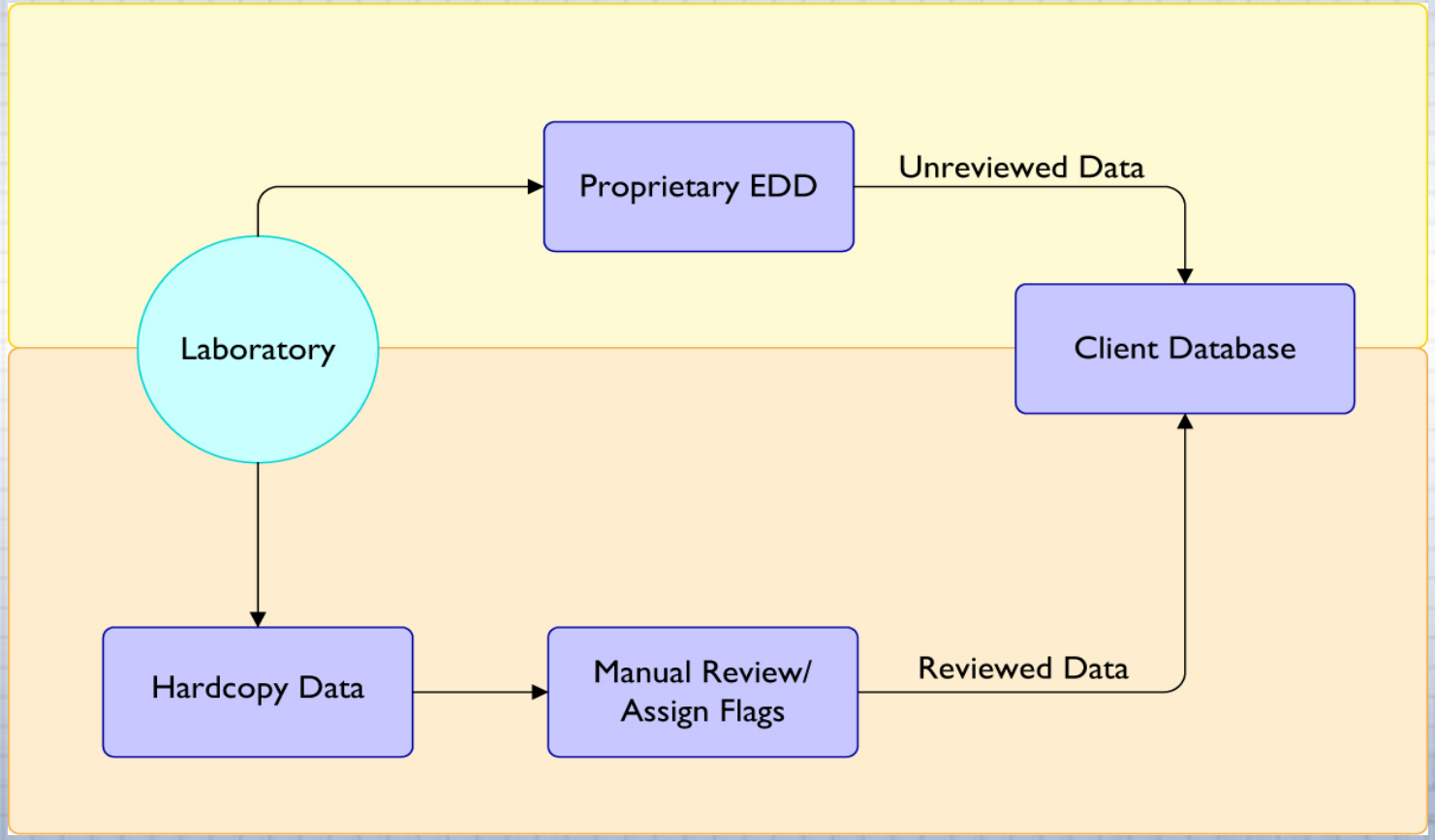
What is Automated Data Review?

Automated Data Review (ADR)

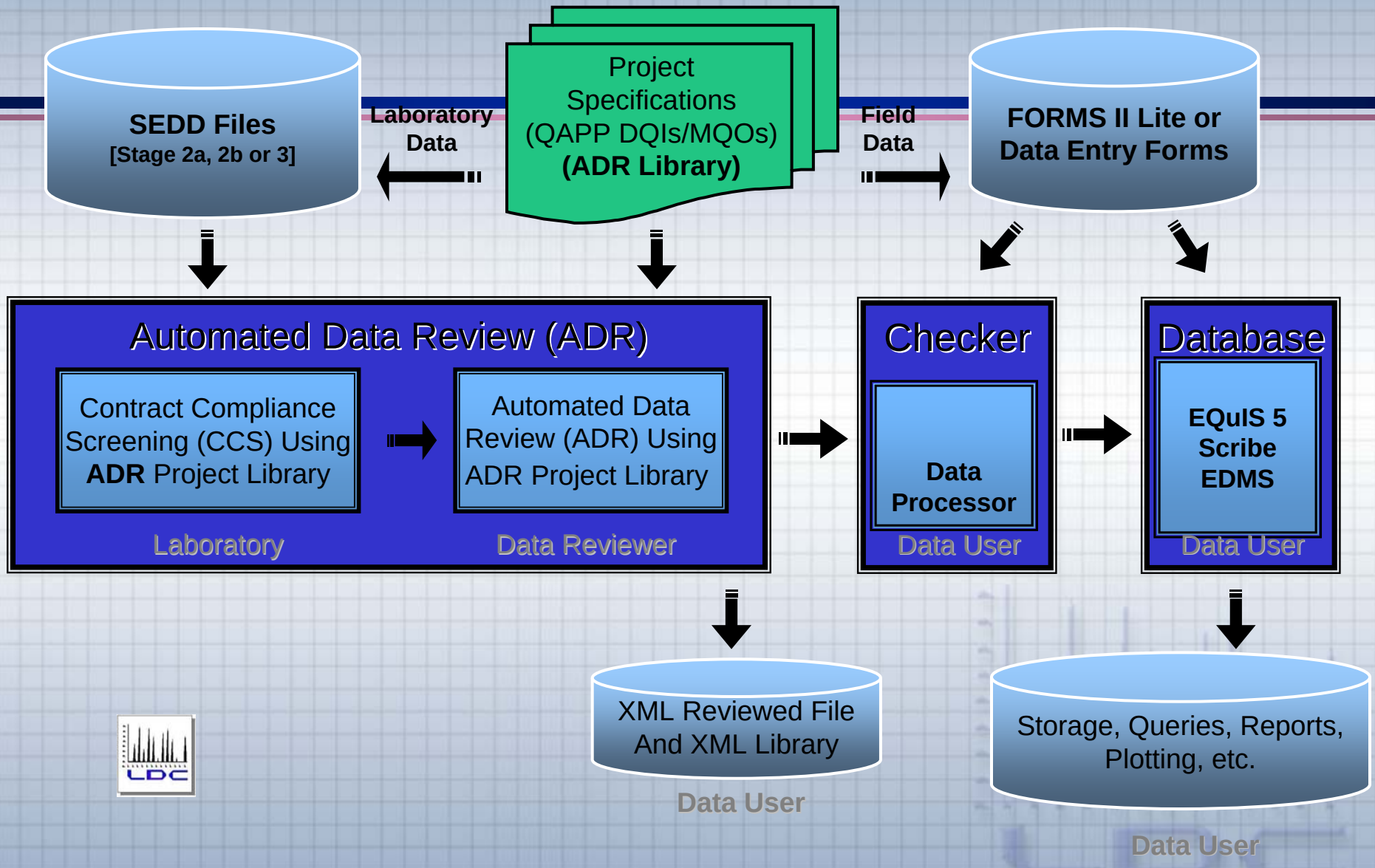
Consists of:

- **Library Tool (ADR Library) – Allows users to easily build an electronic version (.txt or .xml file) of the project specific QC objectives**
- **Laboratory Tool (CCS) – Allows laboratories to verify EDD completeness and contract compliance against the ADR project library**
- **Data Review Tool (ADR) – Allows data users to perform an automated data review against a ADR project library**

Typical Analytical Data Review Model



Overall Electronic Data Flow



Project Planning

Data Quality Indicators (DQIs), Measurement Quality Objectives (MQOs)

- Sample Preservation and Holding Times
- Project Reporting Limits
- Field and Laboratory Contamination
  - » - Method Blank, Trip Blank, Equip Rinsate, etc.
- Precision and Accuracy
  - » - LCS/LCSD, MS/MSD, Field and Lab Duplicates, Surrogates, Tracers
- Calibration (IC, ICV, CC/CCV, GC/MS Tune, etc.)

ADR Project Library (e-QAPP)

ADR performs automated review of laboratory data relative to project goals

ADR Project Library (e-QAPP)

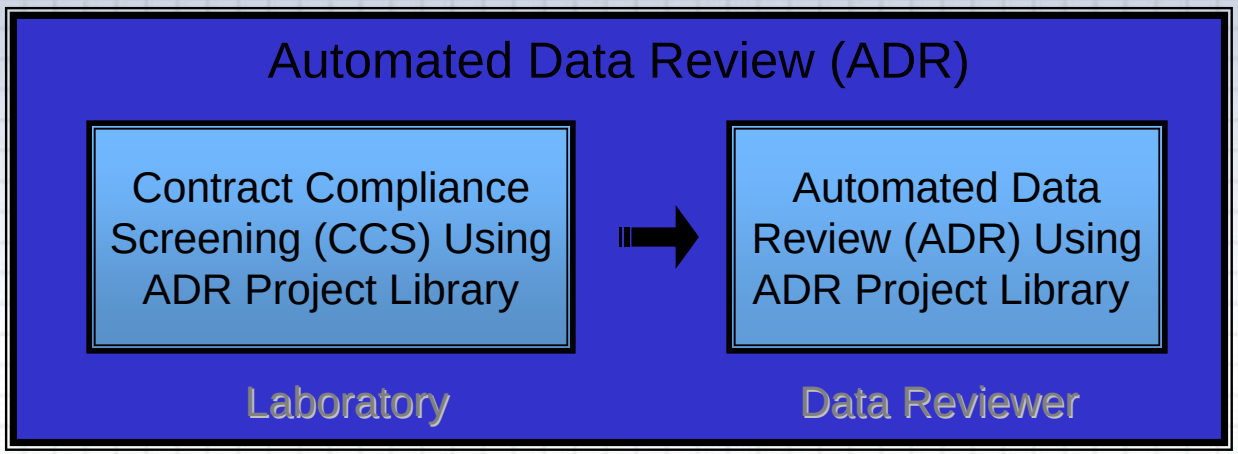
Lab QC data review criteria — Library: AcmeWasteMonitoring — Method: 6010B

Analyte Name	Client Analyte ID	Reporting Limits			LCS and LCSD Accuracy and Precision Criteria			
		Type	Value	Units	Rejection Point	% Recovery Lower Limit	% Recovery Upper Limit	%RPD/RER
ALUMINUM	7429-90-5	MRL	10	mg/Kg	50.00	75.00	120.00	35.00
IRON	7439-89-6	MRL	50	mg/Kg	50.00	75.00	120.00	35.00
MAGNESIUM	7439-95-4	MRL	500	mg/Kg	50.00	75.00	120.00	35.00
MANGANESE	7439-96-5	MRL	5	mg/Kg	50.00	75.00	120.00	35.00
NICKEL	7440-02-0	RDL	10	mg/Kg	50.00	75.00	120.00	35.00
POTASSIUM	7440-09-7	RDL	200	mg/Kg	50.00	75.00	120.00	35.00
SODIUM	7440-23-5	RDL	500	mg/Kg	50.00	75.00	120.00	35.00
BARIUM	7440-39-3	RDL	2	mg/Kg	50.00	75.00	120.00	35.00
CHROMIUM	7440-47-3	RDL	10	mg/Kg	50.00	75.00	120.00	35.00
COBALT	7440-48-4	RDL	10	mg/Kg	50.00	75.00	120.00	35.00
COPPER	7440-50-8	RDL	10	mg/Kg	50.00	75.00	120.00	35.00
VANADIUM	7440-62-2	RDL	5	mg/Kg	50.00	75.00	120.00	35.00
ZINC	7440-66-6	RDL	10	mg/Kg	50.00	75.00	120.00	35.00
CALCIUM	7440-70-2	PQL	1000	mg/Kg	50.00	75.00	120.00	35.00

Data Quality Indicators, Measurement Quality Objectives

CCS Component and ADR Component

A Laboratory Tool and a Data Review Tool

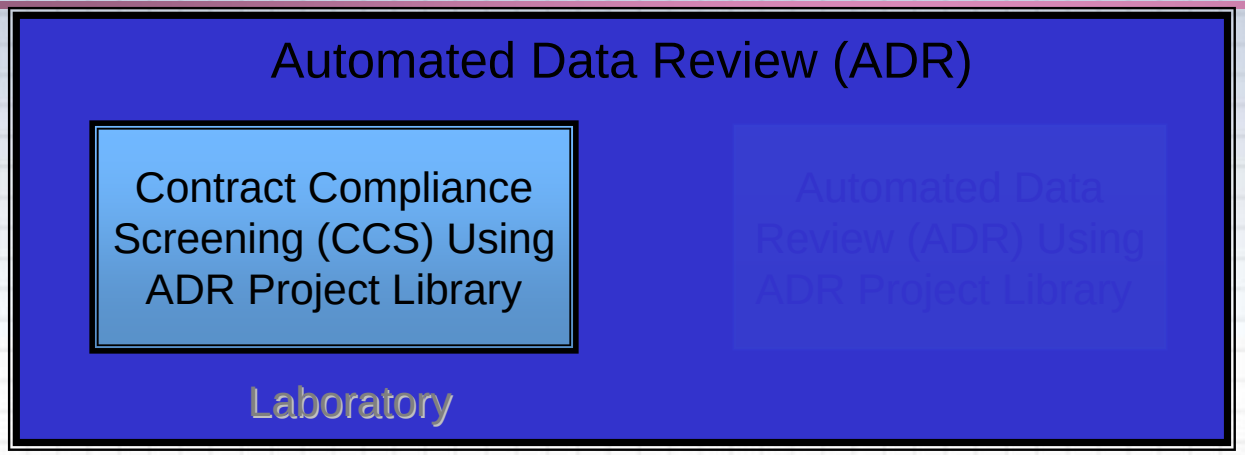


CCS and ADR performed relative to project requirements.

- Verifies Compliance with Data Deliverable Requirements
- Evaluates Data against Project Requirements
- Applies Data Review Qualifiers

ADR Software: Contract Compliance Screening (CCS)

A Laboratory Tool . . .

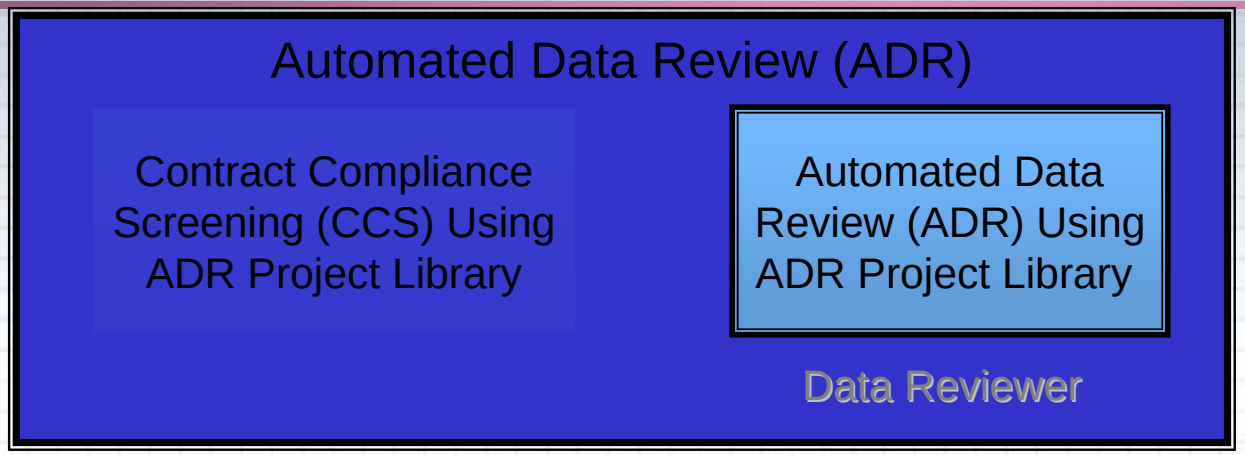


CCS and ADR performed relative to project requirements.

- Verifies Reported Results against Project Requirements
  - Methods and Analyte lists (CAS #s and Analyte Names)
  - Reporting Limits and Units
  - QC Data Provided and Linked to Samples
  - Correct Surrogate and Spike Analytes Added
  - Calibration Data for All Analytes Provided and Linked to Samples

ADR Software: Automated Data Review (ADR)

….. A Data Review Tool

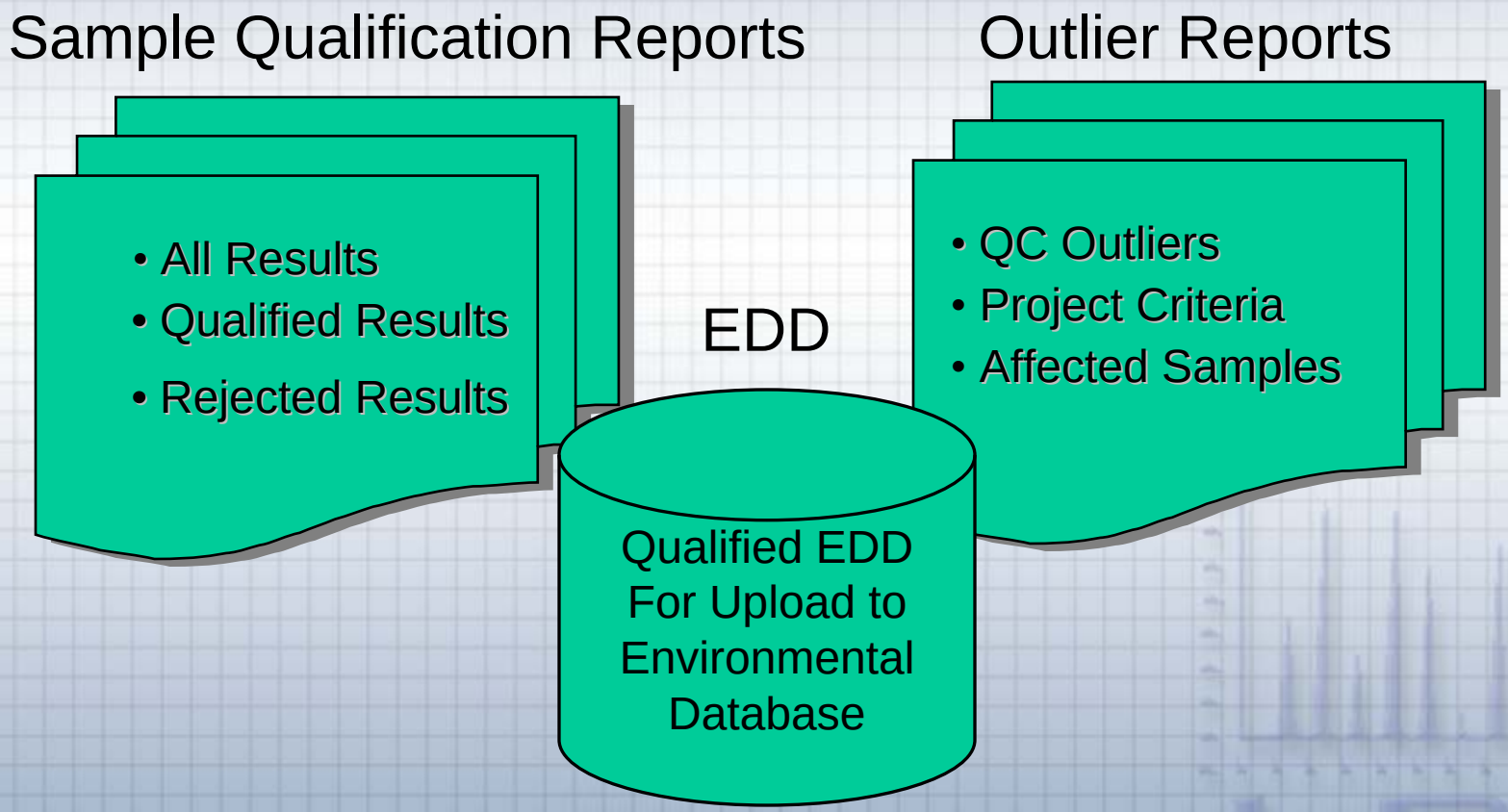


CCS and ADR performed relative to project requirements.

- Evaluates Reported Results against Project Requirements
  - Identifies QC Outliers (Holding Times, Precision, Accuracy)
  - Identifies Calibration Outliers
  - Identifies Field and Laboratory Contamination
  - Identifies Results below the Reporting Limit
  - Automatically Assigns Data Review Qualifiers
  - Allows Qualifier Modification Based on Professional Judgment

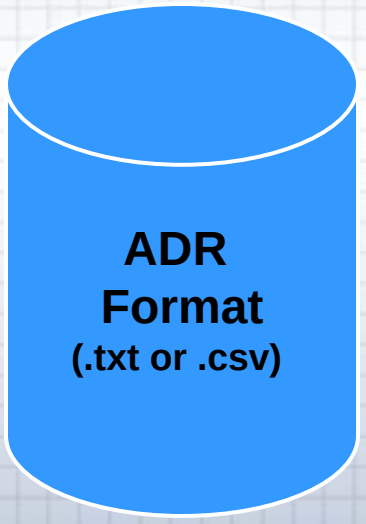
ADR Software: Reports and Qualified EDD

ADR produces hard copy reports as well as qualified electronic data



ADR Laboratory Electronic Data Deliverable

ADR Lab EDD Comprised of three types of information reported in three separate files



- **- Result information (A1 file)**
- **- Calibration Information (A2 file) (optional)**
- **- Sample information (A3 file)**

ADR Lab EDD – Sample Analysis (A3)

ADR - [qryHoldSampleAnalysis : Select Query]

File Edit View Insert Format Records Tools Window Help

Type a question for help

Unique Rec	Project Number	ProjectName	Client Sample ID	Sample Date	Sample Mat	Lab Sample ID	QC Type	Ship Batch ID	Temperature	Lab Method ID
1	1001.312	ACME Waste Monitoring - 2001	MW-01	10/06/2001 0:00:00	AQ	810095-01		C0003	4.50	8270C
2	1001.312	ACME Waste Monitoring - 2001	MW-01	10/06/2001 0:00:00	AQ	810095-01		C0001	4.50	8260B
3	1001.312	ACME Waste Monitoring - 2001	MW-02	10/06/2001 0:00:00	AQ	810095-02		C0001	4.50	8260B
4	1001.312	ACME Waste Monitoring - 2001	MW-03	10/06/2001 0:00:00	AQ	810095-03		C0001	4.50	8260B
5	1001.312	ACME Waste Monitoring - 2001	MW-04	10/06/2001 0:00:00	AQ	810095-04		C0001	4.50	8260B
6	1001.312	ACME Waste Monitoring - 2001	MW-01	10/06/2001 0:00:00	AQ	810095-01		C0006	4.50	9056
7	1001.312	ACME Waste Monitoring - 2001	MW-02	10/06/2001 0:00:00	AQ	810095-02		C0006	4.50	9056
8	1001.312	ACME Waste Monitoring - 2001	MW-03	10/06/2001 0:00:00	AQ	810095-03		C0006	4.50	9056
9	1001.312	ACME Waste Monitoring - 2001	MW-04	10/06/2001 0:00:00	AQ	810095-04		C0006	4.50	9056
10	1001.312	ACME Waste Monitoring - 2001	VBLKW10129801		AQ	VBLKW10129801	MB			8260B
11	1001.312	ACME Waste Monitoring - 2001	VLCSW10129801		AQ	VLCSW10129801	LCS			8260B
12	1001.312	ACME Waste Monitoring - 2001	MW-01MS	10/06/2001 0:00:00	AQ	810095-01MS	MS	C0001	4.50	8260B
13	1001.312	ACME Waste Monitoring - 2001	MW-01MSD	10/06/2001 0:00:00	AQ	810095-01MSD	MSD	C0001	4.50	8260B
14	1001.312	ACME Waste Monitoring - 2001	SBLKW101398		AQ	SBLKW101398	MB			8270C
15	1001.312	ACME Waste Monitoring - 2001	SLCSW101398		AQ	SLCSW101398	LCS			8270C
16	1001.312	ACME Waste Monitoring - 2001	MW-01MS	10/06/2001 0:00:00	AQ	810095-01MS	MS	C0003	4.50	8270C
17	1001.312	ACME Waste Monitoring - 2001	MW-01MSD	10/06/2001 0:00:00	AQ	810095-01MSD	MSD	C0003	4.50	8270C
18	1001.312	ACME Waste Monitoring - 2001	IBLK100998		AQ	IBLK100998	MB			9056
19	1001.312	ACME Waste Monitoring - 2001	ILCS100998		AQ	ILCS100998	LCS			9056
20	1001.312	ACME Waste Monitoring - 2001	MW-01MS	10/06/2001 0:00:00	AQ	810095-01MS	MS	C0006	4.50	9056
21	1001.312	ACME Waste Monitoring - 2001	MW-01DUP	10/06/2001 0:00:00	AQ	810095-01DUP	DUP	C0006	4.50	9056
22	1001.312	ACME Waste Monitoring - 2001	MW-02	10/06/2001 0:00:00	AQ	810095-02		MSB100698		6010B
23	1001.312	ACME Waste Monitoring - 2001	MW-01MS	10/06/2001 0:00:00	AQ	810095-01MS	MS	CLR01	5.00	8310
24	1001.312	ACME Waste Monitoring - 2001	MW-01MSD	10/06/2001 0:00:00	AQ	810095-01MSD	MSD	CLR01	5.00	8310
25	1001.312	ACME Waste Monitoring - 2001	8310BLK101098W		AQ	8310BLK101098W	MB			8310
26	1001.312	ACME Waste Monitoring - 2001	MW-02	10/06/2001 0:00:00	AQ	810095-02		CLR01	5.00	8310
27	1001.312	ACME Waste Monitoring - 2001	MW-01	10/06/2001 0:00:00	AQ	810095-01		CLR01	5.00	8310

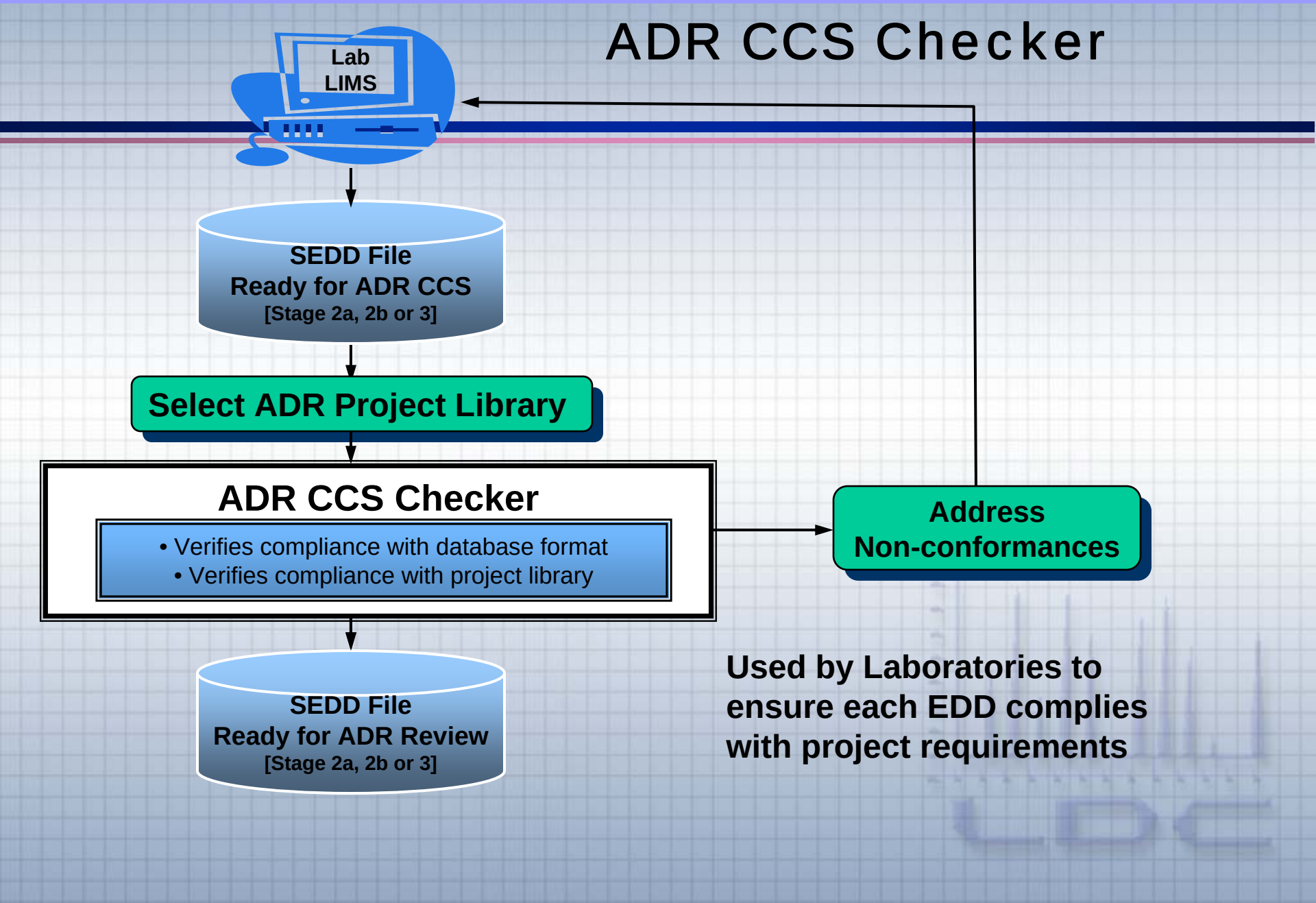
ADR Lab EDD –Analytical Results (A1)

ADR - [qryHoldAnalyticalResult : Select Query]

File Edit View Insert Format Records Tools Window Help

Unique Re	Client Sample ID	Lab Method ID	Analysis Type	Lab Sample ID	Lab ID	Client Analyte ID	Analyte Name	Result	Result Units	Lab Qualifiers
1	MW-04	8260B	RES	810095-04	LDC	74-95-3	Dibromomethane	5	UG/L	U
2	MW-04	8260B	RES	810095-04	LDC	75-00-3	Chloroethane	10	UG/L	U
3	MW-04	8260B	RES	810095-04	LDC	75-01-4	Vinyl chloride	10	UG/L	U
4	MW-04	8260B	RES	810095-04	LDC	75-09-2	Methylene chloride	10	UG/L	U
5	MW-04	8260B	RES	810095-04	LDC	75-25-2	Bromoform	5	UG/L	U
6	MW-04	8260B	RES	810095-04	LDC	75-27-4	Bromodichloromethane	5	UG/L	U
7	MW-04	8260B	RES	810095-04	LDC	75-34-3	1,1-Dichloroethane	5	UG/L	U
8	MW-04	8260B	RES	810095-04	LDC	75-35-4	1,1-Dichloroethene	9.0	UG/L	
9	MW-04	8260B	RES	810095-04	LDC	75-69-4	Trichlorofluoromethane	5	UG/L	U
10	MW-04	8260B	RES	810095-04	LDC	75-71-8	Dichlorodifluoromethane	5	UG/L	U
11	MW-04	8260B	RES	810095-04	LDC	78-87-5	1,2-Dichloropropane	5	UG/L	U
12	MW-04	8260B	RES	810095-04	LDC	79-00-5	1,1,2-Trichloroethane	5	UG/L	U
13	MW-04	8260B	RES	810095-04	LDC	79-01-6	Trichloroethene	5	UG/L	U
14	MW-04	8260B	RES	810095-04	LDC	79-34-5	1,1,2,2-Tetrachloroethane	5	UG/L	U
15	MW-04	8260B	RES	810095-04	LDC	87-68-3	Hexachlorobutadiene	5	UG/L	U
16	MW-04	8260B	RES	810095-04	LDC	91-20-3	Naphthalene	5	UG/L	U
17	MW-04	8260B	RES	810095-04	LDC	95-47-6	ortho-Xylene	5	UG/L	U
18	MW-02	8260B	RES	810095-02	LDC	110-75-8	2-Chloroethyl vinyl ether	10	UG/L	U
19	MW-04	8260B	RES	810095-04	LDC	95-50-1	1,2-Dichlorobenzene	5	UG/L	U
20	MW-04	8260B	RES	810095-04	LDC	96-12-8	1,2-Dibromo-3-chloropropane	5	UG/L	U
21	MW-04	8260B	RES	810095-04	LDC	96-18-4	1,2,3-Trichloropropane	5	UG/L	U
22	MW-04	8260B	RES	810095-04	LDC	98-82-8	Isopropylbenzene	5	UG/L	U
23	MW-02	8260B	RES	810095-02	LDC	1330-20-7	Total Xylenes	5	UG/L	U
24	MW-02	8260B	RES	810095-02	LDC	591-78-6	2-Hexanone	10	UG/L	U
25	MW-02	8260B	RES	810095-02	LDC	67-64-1	Acetone	10	UG/L	U
26	MW-02	8260B	RES	810095-02	LDC	75-15-0	Carbon disulfide	5	UG/L	U
27	MW-02	8260B	RES	810095-02	LDC	78-93-3	2-Butanone	10	UG/L	U

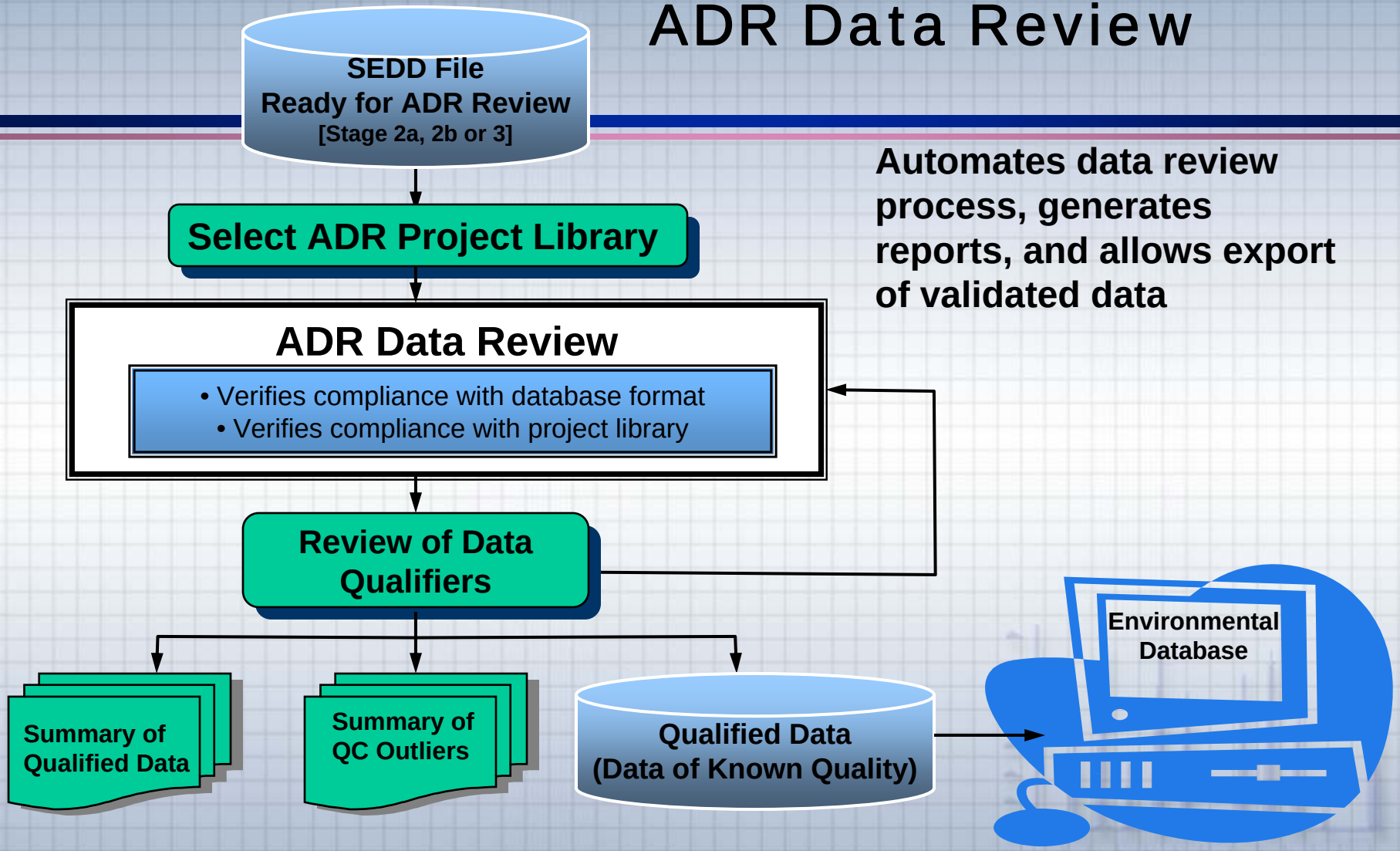
Automated Checking Tools (Laboratory Use)



Used by Laboratories to ensure each EDD complies with project requirements

Automated Checking Tools (Agency or Contractor Use)

ADR Data Review

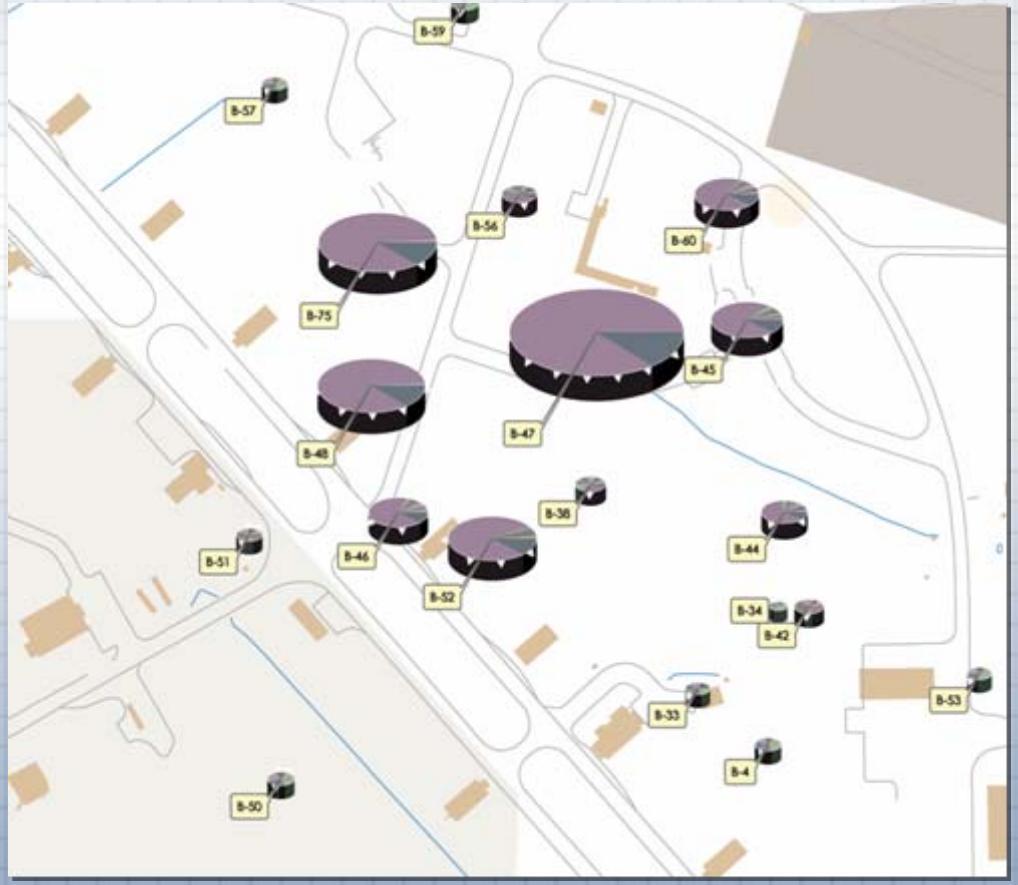


Automates data review process, generates reports, and allows export of validated data

Environmental Database

Contaminant Correlations

- Represent multiple chemical concentrations (based on data of known quality) using the EDMSi EZView LayerBuilder



EQuIS Layer Builder

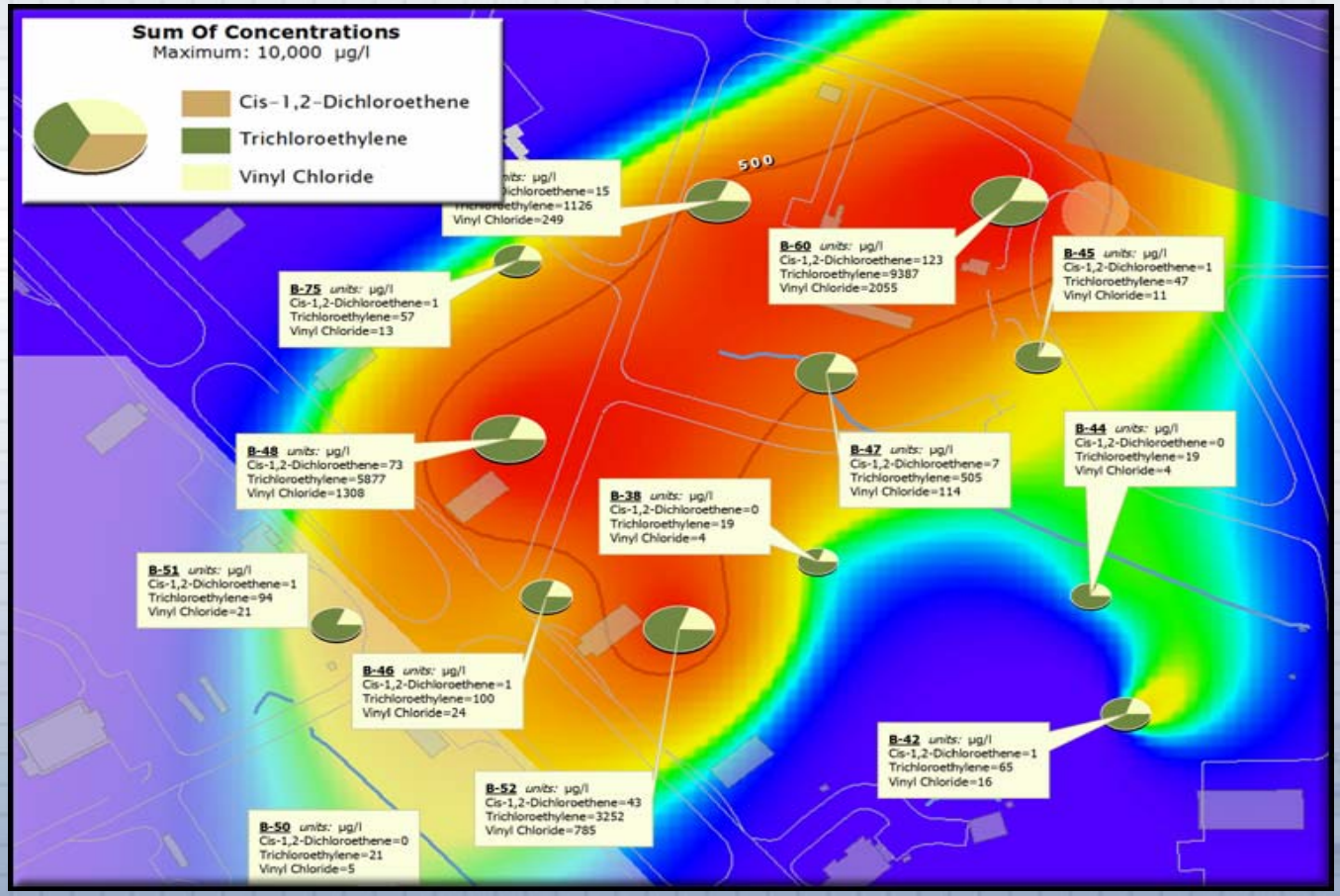
Layer Criteria
- Format: Layer
- Output Query: **LayerQuery**
- Type: **Analytical Concentration**
- Locations
- Sample
  - Type: **N**
  - Date: <ALL>
    - Start: 25 Jul 2001
  - Depth: <ALL>
  - Matrix: **W**
  - Task: <ALL>
- Result
  - Type: <ALL>
  - Total or Dissolved: <ALL>
  - Column Number: <ALL>
  - Analytical Method: <ALL>
  - Analyte: **Acenaphthene**
    - Sort By: **Chemical Name**
    - Select By: **Individual**
      - Operation: **Avg**
  - Action Level: <NONE>
    - Exceedance: **Greater Than (>=)**
      - Include ONLY Exceedances: **No**
  - Approval Code: <ALL>
  - Hits Only: **No**
  - Non-Detect Multiplier: **1.0**
  - Exclude Qualifier: <
    - AND Any Conta
  - Units: **ug/l**

interpreted_qualifiers	organic_yn
	True
	False
*	False
+	False
<	True
<	False
A	True
B	True

Cancel | Next >

Contaminant Correlations

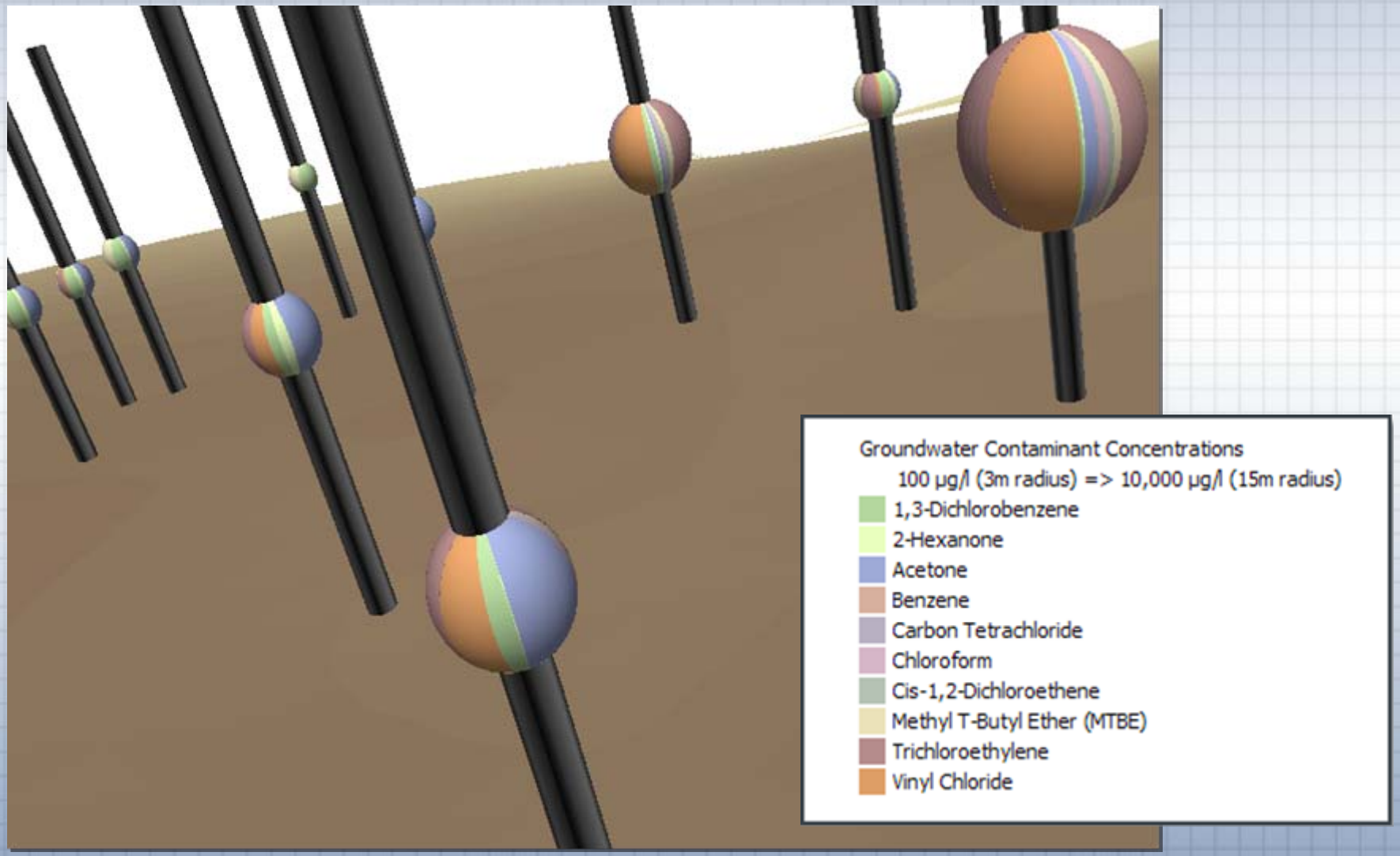
- Visualize in ArcMap using pie chart symbology
  - » Easily create complex labels and annotation



Environmental Database

Contaminant Correlations

Visualize in 3D using Multivariate Sphere Multipatch “Beach Balls”



Case studies

- PCB misidentification
- Improper manual integration
- Using wrong initial calibration
- Dropping calibration points
- Corrective action for method nonconformance

Summary

- Data review and validation are tools to be integrated into data usability.
- All data validation flags are not created equal.
- DQOs and method compliance should be used to develop validation criteria.
- Data review/validation should be performed and managed by professional chemists with hands-on laboratory experience.
- UCMR is a good example where a strict QA program generates high quality data.
- Data integrity must be a planned program in the laboratory.

Contact Info

- Richard M. Amano, Stella Cuenco
  Laboratory Data Consultants, Inc (LDC)
  7750 El Camino Real, Ste 2L
  Carlsbad, CA 92009
  (760) 634-0437
  (760) 634-1674 fax
  e-mail – ramano@lab-data.com