ALS Environmental
QC Parameters on Lab Reports

Presentation to Explain Quality Control Parameters on a Laboratory Report

ALS Life Sciences | Environmental - Rochester

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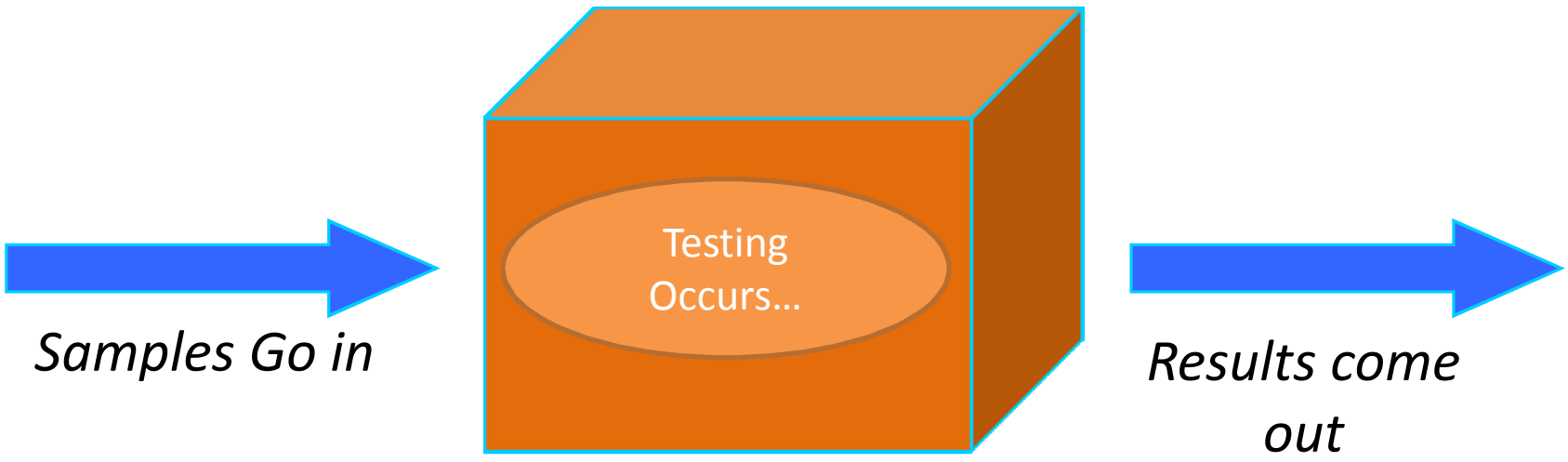
Explaining QC Parameters on Laboratory Reports

Purpose of this Presentation

To Explain:

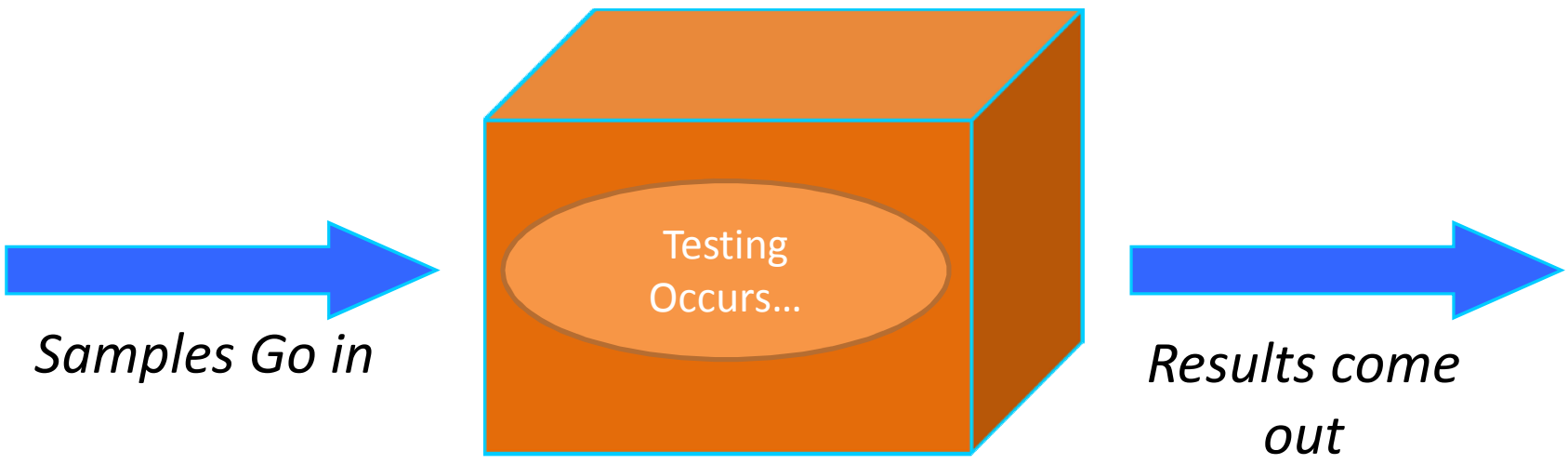
- How Data Quality Objectives impact data and method choice.
- Various Quality Control Parameters on a Lab Report
- Possible Qualifiers due to non-conformances
- Impact of non-conformances on data

Black Box Theory



Black Box Theory

What are the data quality objectives?



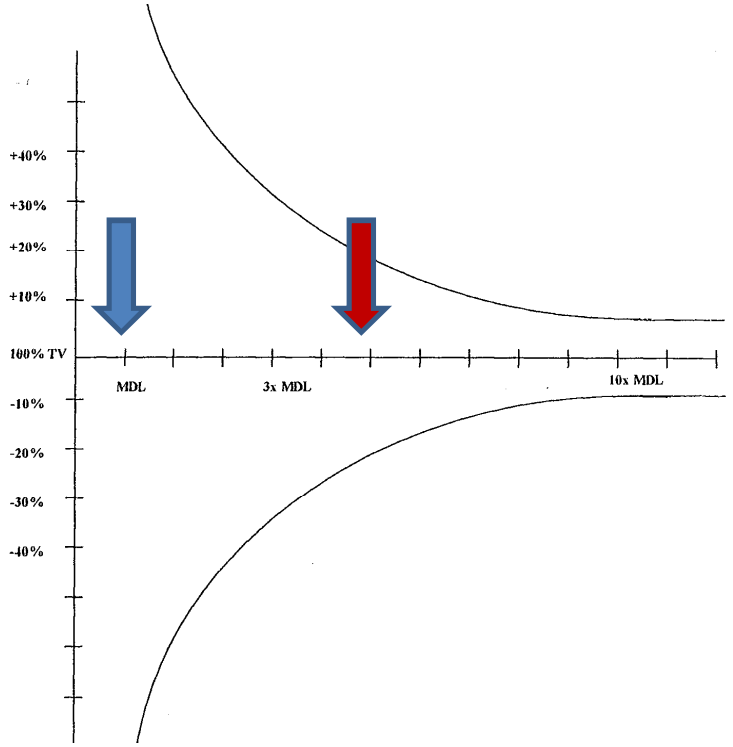
Laboratory must follow promulgated rules as set by EPA and States.

Quality Control Parameters

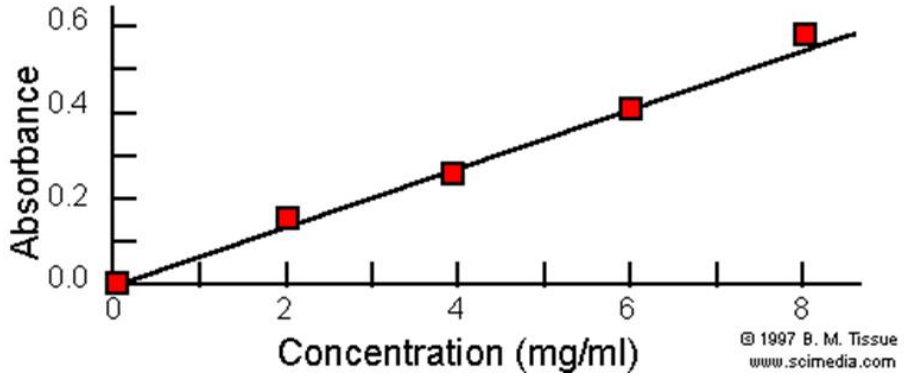
Method	Batch	Sample
• Sensitivity • Linear Range • Calibration	• Method Blank • LCS • MS/MSD • Duplicates	• Surrogates • MS/MSD • Internal standards

Method Sensitivity and Reporting Limit

- Method Detection Limit (MDL)
  - Statistically derived limit based 7 replicates (40CFR part 136 B).
  - Statistically different than a blank. Qualitative, not quant.
- **Reporting Limit (RL)**
  - RL is the low point on the curve. Accuracy and precision can be determined. Within linear range.

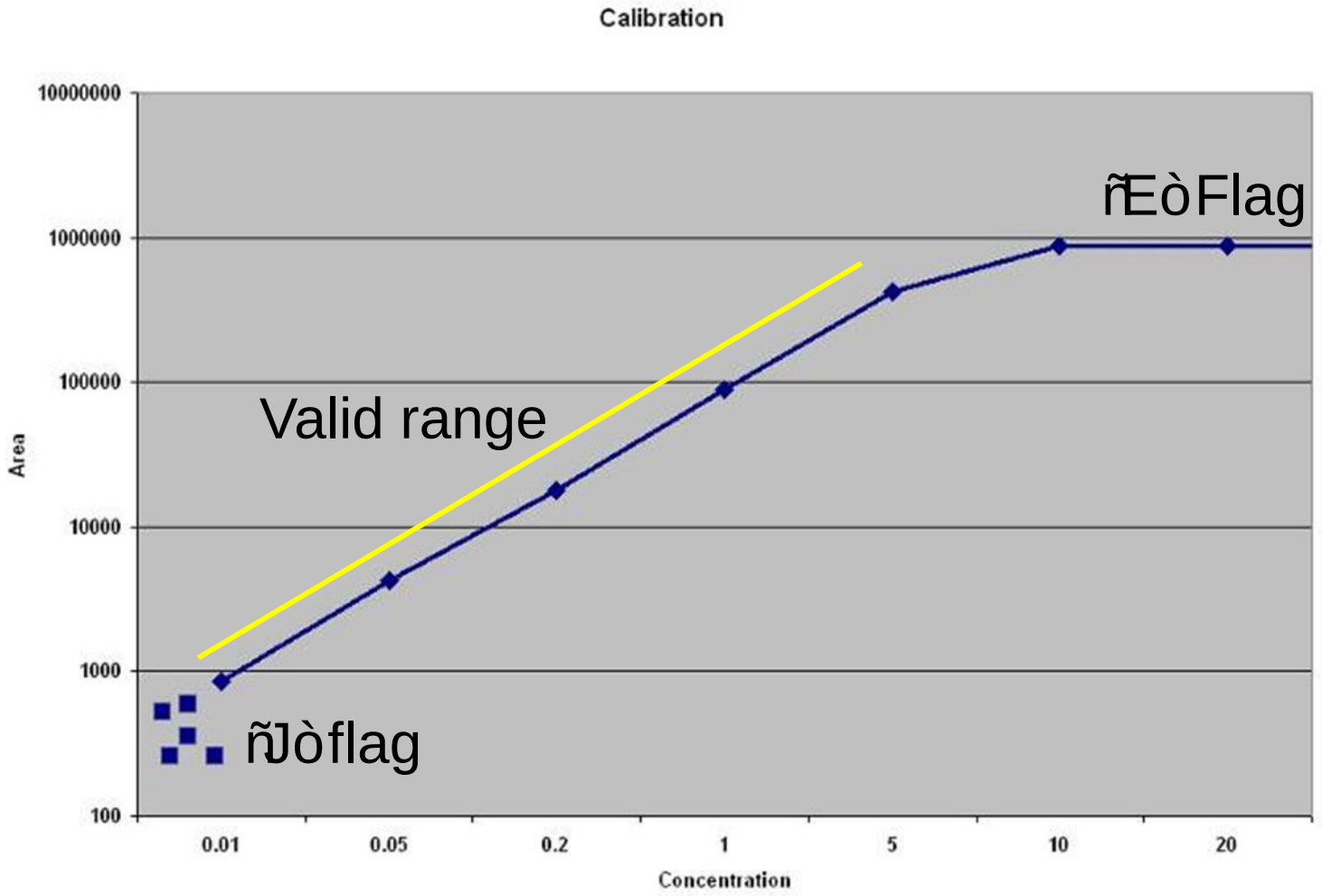


Method - Calibration and Verification

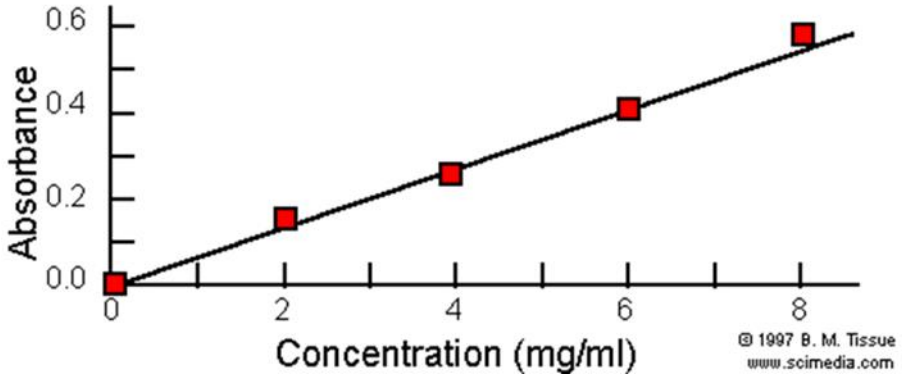


- A calibration curve plots known concentration versus instrument response. Linearity of the method determines the concentration range.

Method - Linear Range



Method - Calibration and Verification



QC associated with the Calibration:

- Linearity – limits on how much the points of the curve deviated from expected (RSD limits from average RF) or from a line (CC).
- ICV – a different source of standard – to verify the standard used for the initial calibration curve. Performed immediately after initial calibration.

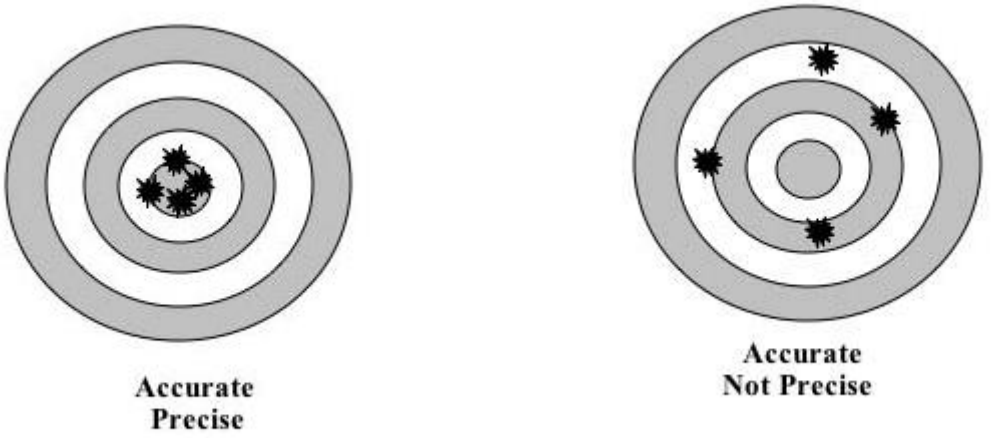
Method - Calibration and Verification

Continuing Calibration Verification (CCV)

- The Calibration Verification Standard (CCV) is performed at defined times to verify that the instrument is still producing the expected response compared to the initial calibration.
- Initial calibrations may be good for hours, days, months, or even years depending on the stability of the system.
- CCV is expected to perform within a defined criteria. A CCV that exceeds the expected range indicates a potential bias and requires re-calibration and sample analysis.

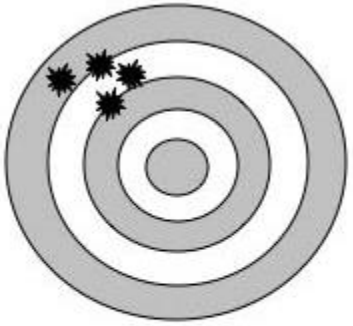
Batch Quality Control

- Quality Control (QC) are the analytical processes used to determine accuracy and precision of data.
  - Accuracy (Laboratory Control Samples, surrogates)
    - Measure of the closeness of a measurement to the actual value.
    - Determined from control samples each batch.

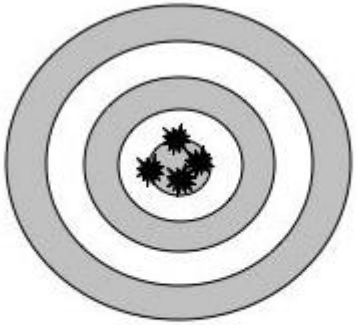


Quality Control

- Precision (Duplicates, MS/MSD, LCS/LCSD))
  - The ability of an analytical method to reproduce its own measurement (i.e. a measure of variability). Aka Bias.
  - Measured using replicate sample.



Not Accurate
Precise



Accurate
Precise

What should I expect from the lab?

Choose methods based on data needs.

Batch Quality Control

- Batch requirements
  - Defined by SOP and method

  Generally:

  - Method blank (MB)
  - Laboratory Control Sample (LCS)
  - Matrix spike (MS)
  - One of the following: Matrix spike duplicate (MSD), sample duplicate (DUP), or LCS duplicate (LCSD)

  May be others that are method-defined or project-specific.

Method Blank (MB)

- Purpose: to check for possible analyte contamination.
- Procedure: an aliquot of reagent water or other analyte-free matrix is prepared and carried through the entire sample preparation and analysis.

- Method blank analysis with all samples
  - Demonstrates that the analytical system itself does not introduce contamination into the analysis
  - Demonstrates that positive sample results are not false positives.
  - Is reported to Reporting Limit, unless requested to MDL

Method Blank

Impact on Data:

- If target analytes are detected in the MB above the Reporting Detection Limit, detections in the samples may be due to laboratory contamination.
- If an analyte is detected in the MB but NOT in the sample, data is not impacted.

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Analytical Report

Client:	Phenova
Project:	WP Organics/WP0715 Organics
Sample Matrix:	Water
Sample Name:	Method Blank
Lab Code:	RQ1507624-01

Low Level Semivolatile Organic Compounds by GC/MS

Analysis Method:	8270D
Prep Method:	EPA 3510C

Analyte Name	Result	MRL	MDL	Dil.	Date
Acenaphthene	0.200 U	0.200	0.0500	1	07/1
Acenaphthylene	0.200 U	0.200	0.0500	1	07/1
Anthracene	0.200 U	0.200	0.0500	1	07/1
Benz(a)anthracene	0.200 U	0.200	0.0500	1	07/1
Benzo(b)fluoranthene	0.200 U	0.200	0.0500	1	07/1
Benzo(k)fluoranthene	0.200 U	0.200	0.0500	1	07/1
Benzo(g,h,i)perylene	0.200 U	0.200	0.0510	1	07/1
Benzo(a)pyrene	0.200 U	0.200	0.0500	1	07/1
Chrysene	0.200 U	0.200	0.0500	1	07/1
Dibenz(a,h)anthracene	0.200 U	0.200	0.0500	1	07/1
Fluoranthene	0.200 U	0.200	0.0500	1	07/1
Fluorene	0.200 U	0.200	0.0500	1	07/1
Indeno(1,2,3-cd)pyrene	0.200 U	0.200	0.0500	1	07/1
Naphthalene	0.200 U	0.200	0.0500	1	07/1
Phenanthrene	**0.0700 J**	0.200	0.0500	1	07/1
Pyrene	0.200 U	0.200	0.0250	1	07/1

Method Blank

- If the concentration of a sample analyte is greater than 10x the concentration on the MB, the affect of any potential contamination is judged to be non-significant from a laboratory perspective.

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Analytical Report

Client:	Phenova	**Servic**
Project:	WP Organics/WP0715 Organics	**Date**
Sample Matrix:	Water	**Date**
Sample Name:	PT-PAH-WP 8158-37	
Lab Code:	R1505435-010	

Low Level Semivolatile Organic Compounds by GC/MS

Analysis Method:	8270D
Prep Method:	EPA 3510C

Analyte Name	Result	MRL	MDL	Dil.	Date An
Acenaphthene	**14.3 E**	0.189	0.0500	1	07/13/15
Acenaphthylene	**12.9 E**	0.189	0.0500	1	07/13/15
Anthracene	**3.19**	0.189	0.0500	1	07/13/15
Benz(a)anthracene	**1.45**	0.189	0.0500	1	07/13/15
Benzo(b)fluoranthene	**1.12**	0.189	0.0500	1	07/13/15
Benzo(k)fluoranthene	**2.93**	0.189	0.0500	1	07/13/15
Benzo(g,h,i)perylene	**2.59**	0.189	0.0510	1	07/13/15
Benzo(a)pyrene	**4.07**	0.189	0.0500	1	07/13/15
Chrysene	**1.12**	0.189	0.0500	1	07/13/15
Dibenz(a,h)anthracene	**3.08**	0.189	0.0500	1	07/13/15
Fluoranthene	**2.84**	0.189	0.0500	1	07/13/15
Fluorene	**1.92**	0.189	0.0500	1	07/13/15
Indeno(1,2,3-cd)pyrene	**1.76**	0.189	0.0500	1	07/13/15
Naphthalene	**5.97**	0.189	0.0500	1	07/13/15
Phenanthrene	**2.96**	0.189	0.0500	1	07/13/15
Pyrene	**1.36**	0.189	0.0250	1	07/13/15

Laboratory Control Sample (LCS)

- Purpose: to document laboratory performance.
- Procedure: an aliquot of analyte-free matrix is spiked with a known concentration of target compounds and carried through the entire sample preparation and analysis procedure as part of the analytical batch.
- Evaluation: recovered concentrations are compared to the true spiking concentrations and evaluated against predetermined acceptance criteria.

Laboratory Control Sample

- Impact on Data:
  - Recoveries above acceptance limits indicate the potential for a high bias with analytical results and recoveries below acceptance limits indicate the potential for a low bias with analytical results.
  - If the LCS recovery is above acceptance limits but the target analyte is not detected in the sample, data is not impacted.

		Lab Control Sample RQ1507590-02			Duplicate Lab Control Sample RQ1507590-03					
Analyte Name	**Analytical Method**	**Result**	**Spike Amount**	**% Rec**	**Result**	**Spike Amount**	**% Rec**	**% Rec Limits**	**RPD**	**RPD Limit**
4-Chloroaniline	8270D	94.7	100	95	98.2	100	98	40-111	3	30
1-Methylnaphthalene	8270D	81.5	100	82	86.3	100	86	49-105	5	30
2-Nitroaniline	8270D	87.4	100	87	93.9	100	94	60-119	8	30
3-Nitroaniline	8270D	87.2	100	87	92.9	100	93	49-110	7	30
4-Nitroaniline	8270D	98.6	100	99	107	100	107	61-122	8	30
Pyridine	8270D	45.2	100	45	43.7	100	44	10-123	2	30
1,2,4,5-Tetrachlorobenzene	8270D	62.9	100	63	69.1	100	69	31-100	9	30

Surrogate

- Purpose: to identify chemical or physical interferences in a sample matrix.
- Procedure: Surrogates are analyzed as part of most organic methods. Surrogates are compounds which are similar to the target analytes in chemical composition and behavior, but which are not normally found in environmental samples. Surrogate compounds are not present in the collected sample; they are added to the sample at the time of preparation or analysis by the laboratory and are used and included for quality assurance purposes only.

Surrogate

- Evaluation: Recovered surrogate concentrations are compared to the true spiking concentrations and evaluated against predetermined acceptance criteria.

Surrogate Name	% Rec	Control Limits	Date Analyzed
2,4,6-Tribromophenol	87	10 - 212	07/13/15 12:10
2-Fluorobiphenyl	82	27 - 133	07/13/15 12:10
2-Fluorophenol	46	10 - 104	07/13/15 12:10
Nitrobenzene-d5	78	31 - 167	07/13/15 12:10
Phenol-d6	30	11 - 63	07/13/15 12:10
p-Terphenyl-d14	114	38 - 166	07/13/15 12:10

Surrogate

- Impact on Data:
  - Recoveries above acceptance limits indicate there could potentially be a high bias with analytical results and recoveries below acceptance limits indicate there could potentially be a low bias with analytical results.
  - Because non-conformances related to surrogate recoveries are the result of chemical or physical interferences present in the sample, the lab may not be able to eliminate the bias. The lab attempts to reduce or eliminate interferences by cleanups and/or dilution.

Matrix Spike and/or Matrix Spike Duplicate (MS/MSD)

- Purpose: to identify chemical or physical interferences inherit in a sample matrix.
- Procedure: an aliquot of sample is spiked with a known concentration of target compounds and analyzed.
- Evaluation: recovered concentrations are compared to the true spiking concentrations and evaluated against predetermined acceptance criteria.

Matrix Spike

R1505433-006MS

Analyte Name	Method	Sample Result	Result	Spike Amount	% Rec	% Rec Limits
Alkalinity, Total as CaCO3	SM 2320 B-1997(2011)	108	144	40.0	91	69-114
Chloride	300.0	258	333	79.9	93	90-110
Chloride	9056A	258	333	79.9	93	80-120
Chloride	SM 4500-Cl-E-1997(2011)	263	356	99.9	93	72-130
Sulfate	300.0	120	205	80.0	106	90-110
Sulfate	9056A	120	205	80.0	106	80-120

Matrix Spike and/or Matrix Spike Duplicate

- Impact on Data:
  - Recoveries above acceptance limits indicate an interference that could potentially result in a high bias with analytical results and recoveries below acceptance limits indicate an interference that could potentially result in a low bias with analytical results.
  - Because non-conformances related to spike recoveries are the result of chemical or physical interferences present in the sample, these interferences must be identified and removed to obtain acceptable spike recoveries. In some instances, this may not be possible.

Relative Percent Difference (RPD)

- Purpose: to evaluate the reproducibility or precision associated with the sample analysis.
- Procedure: the relative percent difference between a sample and sample duplicate or a matrix spike and matrix spike duplicate

$$\%\ RPD = \frac{|(R1 - R2)|}{(R1 + R2) \div 2} \times 100$$

where: R_1 = sample or spike result

R_2 = duplicate or spike duplicate result

Relative Percent Difference

- Evaluation: calculated relative percent differences are evaluated against predetermined acceptance criteria.

Replicate Sample Summary
General Chemistry Parameters

Sample Name: PT-S2-WP 8158-22 **Units:** mg/L
Lab Code: R1505433-016 **Basis:** NA

Analyte Name	Analysis Method	MRL	MDL	Sample Result	Duplicate Sample R1505433-016DUP Result	Average	RPD	RPD Limit
Sulfide, Acid-Soluble	9034	1.0	0.4	4.6	4.5	4.55	3	20

			Lab Control Sample RQ1507621-02			Duplicate Lab Control Sample RQ1507621-03				
Analyte Name	Analytical Method	Result	Spike Amount	% Rec	Result	Spike Amount	% Rec	% Rec Limits	RPD	RPD Limit
2,4-D	8151A	2.56	2.50	102	2.52	2.50	101	29-146	2	30
Dicamba	8151A	2.18	2.50	87	2.25	2.50	90	39-111	3	30
2,4,5-T	8151A	2.23	2.50	89	2.17	2.50	87	22-137	3	30
2,4,5-TP	8151A	2.00	2.50	80	1.97	2.50	79	40-121	1	30
Pentachlorophenol (PCP)	8151A	2.05	2.50	82	2.10	2.50	84	10-112	2	30

Relative Percent Difference

- Impact on Data:

➢ Relative percent differences above the acceptance limit indicate a potential matrix effect, such as sample non-homogeneity, that may be affecting the ability to reproduce analytical results.

➢ Because non-conformances related to relative percent difference are the result of interferences present in the sample, these interferences must be identified and removed to obtain acceptable spike recoveries. In some instances, this may not be possible.

Non-Conformances

- Non-conformances and Corrective Action
  - There will be times when things don't go as planned. Each analytical SOP details procedures to deal with non-conforming events (where analyses failed, QC failed, etc.).
  - Analysts involve the Project Manager
  - Documentation of non-conformances is required.

Qualifiers

Symbols used as a reference to a table of explanations.

REPORT QUALIFIERS AND DEFINITIONS

U	Analyte was analyzed for but not detected. The sample quantitation limit has been corrected for dilution and for percent moisture, unless otherwise noted in the case narrative.
J	Estimated value due to either being a Tentatively Identified Compound (TIC) or that the concentration is between the MRL and the MDL. Concentrations are not verified within the linear range of the calibration. For DoD: concentration >40% difference between two GC columns (pesticides/Arclors).
B	Analyte was also detected in the associated method blank at a concentration that may have contributed to the sample result.
E	Inorganics- Concentration is estimated due to the serial dilution was outside control limits.
E	Organics- Concentration has exceeded the calibration range for that specific analysis.
D	Concentration is a result of a dilution, typically a secondary analysis of the sample due to exceeding the calibration range or that a surrogate has been diluted out of the sample and cannot be assessed.
*	Indicates that a quality control parameter has exceeded laboratory limits. Under the "Notes" column of the Form I, this qualifier denotes analysis was performed out of Holding Time.
H	Analysis was performed out of hold time for tests that have an "immediate" hold time criteria.
#	Spike was diluted out.
+	Correlation coefficient for MSA is <0.995.
N	Inorganics- Matrix spike recovery was outside laboratory limits.
N	Organics- Presumptive evidence of a compound (reported as a TIC) based on the MS library search.
S	Concentration has been determined using Method of Standard Additions (MSA).
W	Post-Digestion Spike recovery is outside control limits and the sample absorbance is <50% of the spike absorbance.
P	Concentration >40% (25% for CLP) difference between the two GC columns.
C	Confirmed by GC/MS
Q	DoD reports: indicates a pesticide/Aroclor is not confirmed (≥100% Difference between two GC columns).
X	See Case Narrative for discussion.
MRL LOQ	Method Reporting Limit. Also known as: Limit of Quantitation (LOQ) The lowest concentration at which the method analyte may be reliably quantified under the method conditions.
MDL	Method Detection Limit. A statistical value derived from a study designed to provide the lowest concentration that will be detected 99% of the time. Values between the MDL and MRL are estimated (see J qualifier).
LOD	Limit of Detection. A value at or above the MDL which has been verified to be detectable.
ND	Non-Detect. Analyte was not detected at the concentration listed. Same as U qualifier.

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QC Parameters on Laboratory Reports

Any Questions?