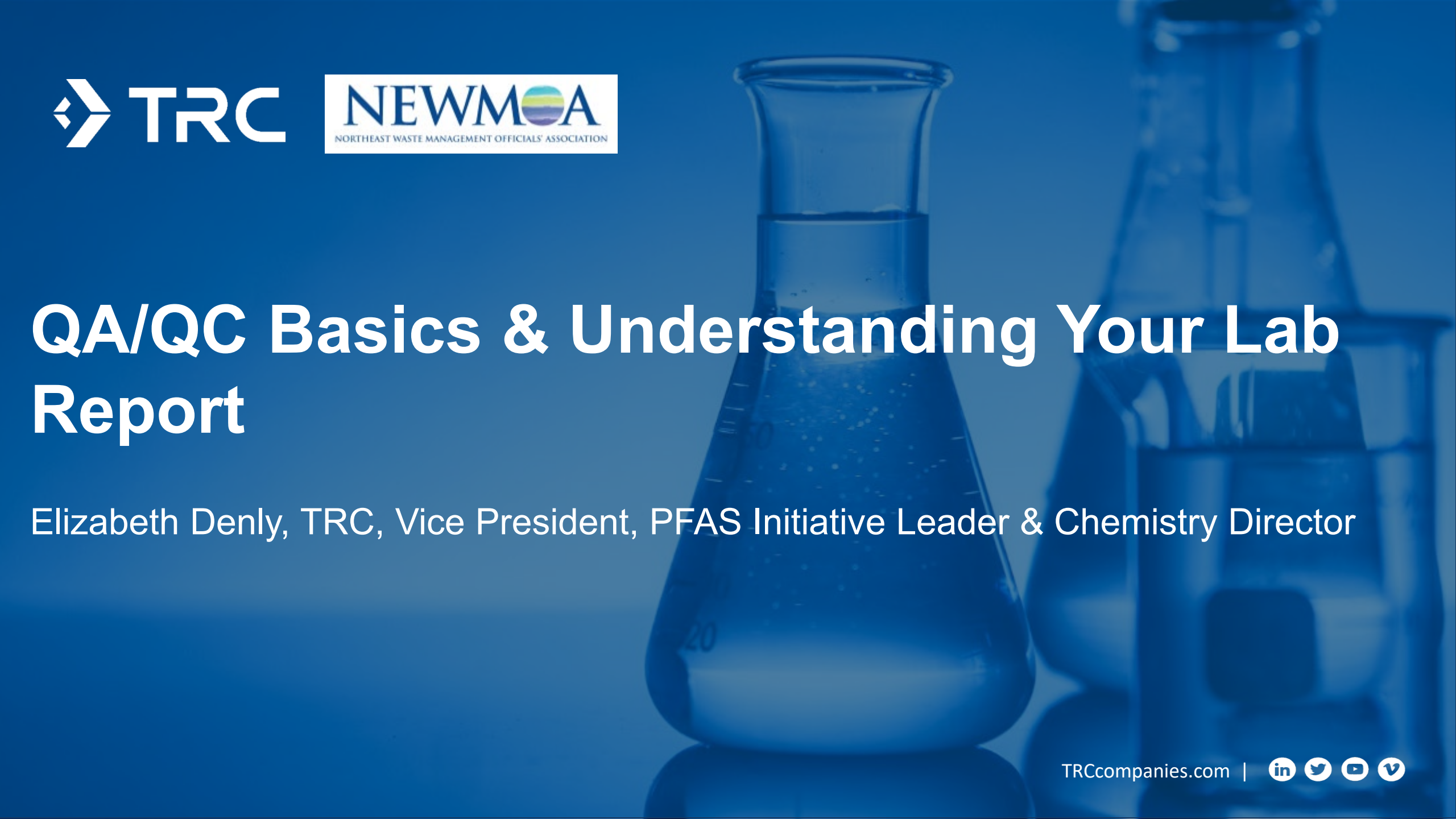


QA/QC Basics & Understanding Your Lab Report

Elizabeth Denly, TRC, Vice President, PFAS Initiative Leader & Chemistry Director

Today's Training Topics

- Communicating with the Analytical Laboratory
- Evaluating the Analytical Data

What Questions Do I Need to Answer While Preparing The Lab Scope of Work?

Why Am I Collecting This Sample?

- Is it a permit requirement?
- Is it for waste characterization?
- Will a human health or ecological risk assessment be performed?
- Are you evaluating nature & extent of contamination?
- Are you measuring effectiveness of a remediation system?

What Analytes are Needed?

- VOCs, SVOCs, Pesticides, Metals
  - What is the lab's full list? Every lab is different.
  - Should the lab report the entire list?
- Waste Characterization Parameters
  - TCLP VOCs, TCLP SVOCs, TCLP metals, TCLP pesticides, TCLP herbicides, ignitability, reactivity, corrosivity
- NPDES Permit Parameters
- Remediation or Investigation Parameters
- Air Emission Standard Permit

METHOD 8260D
VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY

Compound	CAS No.[a]
Acetone	67-64-1
Acetonitrile	75-05-8
Acrolein (Propenal)	107-02-8
Acrylonitrile	107-13-1
Allyl alcohol	107-18-6
Allyl chloride	107-05-1
t-Amyl ethyl ether (TAEE, 4,4-Dimethyl-3-oxahexane)	919-94-8
t-Amyl methyl ether (TAME)	994-05-8
Benzene	71-43-2
Benzyl chloride	100-44-7
Bromoacetone	598-31-2
Bromobenzene	108-86-1
Bromochloromethane	74-97-5
Bromodichloromethane	75-27-4
Bromoform	75-25-2
Bromomethane	74-83-9
n-Butanol (1-Butanol, *n*-Butyl alcohol)	71-36-3
2-Butanone (MEK)	78-93-3
t-Butyl alcohol	75-65-0
n-Butylbenzene	104-51-8
sec-Butylbenzene	135-98-8
tert-Butylbenzene	98-06-6
Carbon disulfide	75-15-0
Carbon tetrachloride	56-23-5
Chloral hydrate	302-17-0
Chlorobenzene	108-90-7
1-Chlorobutane	109-69-3
Chlorodibromomethane (Dibromochloromethane)	124-48-1
Chloroethane	75-00-3
2-Chloroethanol	107-07-3

Compound	CAS No.[a]
1,1,2-Trichloroethane	79-00-5
Trichloroethene (Trichloroethylene)	79-01-6
1,1,2-Trichloro trifluoroethane	76-13-1
1,1,1-Trichlorotrifluoroethane	354-58-5
Trichlorofluoromethane	75-69-4
1,2,3-Trichloropropane	96-18-4
1,2,3-Trimethylbenzene	526-73-8
1,2,4-Trimethylbenzene	95-63-6
1,3,5-Trimethylbenzene	108-67-8
Vinyl acetate	108-05-4
Vinyl chloride	75-01-4
m-Xylene	108-38-3
o-Xylene	95-47-6
p-Xylene	106-42-3

Compound	CAS No.[a]
2-Chloroethyl vinyl ether	110-75-8
Chloroform	67-66-3
1-Chlorohexane	544-10-5
Chloromethane	74-87-3
Chloroprene (2-Chloro-1,3-butadiene)	126-99-8
2-Chlorotoluene	95-49-8
4-Chlorotoluene	106-43-4
Crotonaldehyde	4170-30-3
Cyclohexane	110-82-7
1,2-Dibromo-3-chloropropane (DBCP)	96-12-8
1,2-Dibromoethane (EDB, Ethylene dibromide)	106-93-4
Dibromomethane	74-95-3
1,2-Dichlorobenzene	95-50-1
1,3-Dichlorobenzene	541-73-1
1,4-Dichlorobenzene	106-46-7
cis-1,4-Dichloro-2-butene	1476-11-5
trans-1,4-Dichloro-2-butene	110-57-6
Dichlorodifluoromethane	75-71-8
1,1-Dichloroethane	75-34-3
1,2-Dichloroethane	107-06-2
1,1-Dichloroethene (Vinylidene chloride)	75-35-4
cis-1,2-Dichloroethene	156-59-2
trans-1,2-Dichloroethene	156-60-5
1,3-Dichloropropane	142-28-9
1,2-Dichloropropane	78-87-5
2,2-Dichloropropane	594-20-7
1,3-Dichloro-2-propanol	96-23-1
1,1-Dichloropropene	563-58-6
cis-1,3-Dichloropropene	10061-01-5
trans-1,3-Dichloropropene	10061-02-6
1,2,3,4-Diepoxybutane	1464-53-5
Diethyl ether	60-29-7
Diisopropyl ether (DIPE)	108-20-3
1,4-Dioxane	123-91-1
Epichlorohydrin	106-89-8
Ethanol	64-17-5
Ethyl acetate	141-78-6
Ethyl benzene	100-41-4
Ethyl methacrylate	97-63-2
Ethyl *t*-butyl ether (ETBE)	637-92-3
Ethylene oxide	75-21-8

Compound	CAS No.[a]
Hexachlorobutadiene	87-68-3
Hexachloroethane	67-72-1
2-Hexanone	591-78-6
Iodomethane (Methyl iodide)	74-88-4
Isobutyl alcohol	78-83-1
Isopropylbenzene	98-82-8
p-Isopropyltoluene	99-87-6
Malononitrile	109-77-3
Methacrylonitrile	126-98-7
Methanol	67-56-1
Methyl acetate	79-20-9
Methyl acrylate	96-33-3
Methyl methacrylate	80-62-6
Methyl tert-butyl ether (MTBE)	1634-04-4
Methylcyclohexane	108-87-2
Methylene chloride (DCM)	75-09-2
4-Methyl-2-pentanone (MIBK)	108-10-1
Naphthalene	91-20-3
Nitrobenzene (NB)	98-95-3
2-Nitropropane	79-46-9
N-Nitroso-di-*n*-butylamine (*N*-Nitrosodibutylamine)	924-16-3
Paraldehyde	123-63-7
Pentachloroethane	76-01-7
Pentafluorobenzene	363-72-4
2-Pentanone	107-87-9
2-Picoline (2-Methylpyridine)	109-06-8
1-Propanol (*n*-Propyl alcohol)	71-23-8
2-Propanol (Isopropyl alcohol)	67-63-0
Propargyl alcohol	107-19-7
β-Propiolactone	57-57-8
Propionitrile (Ethyl cyanide)	107-12-0
n-Propylamine	107-10-8
n-Propylbenzene	103-65-1
Pyridine	110-86-1
Styrene	100-42-5
1,1,1,2-Tetrachloroethane	630-20-6
1,1,2,2-Tetrachloroethane	79-34-5
Tetrachloroethene	127-18-4
Toluene	108-88-3
o-Toluidine	95-53-4
1,2,3-Trichlorobenzene	87-61-6
1,2,4-Trichlorobenzene	120-82-1
1,1,1-Trichloroethane	71-55-6

What Detection Limits are Needed?

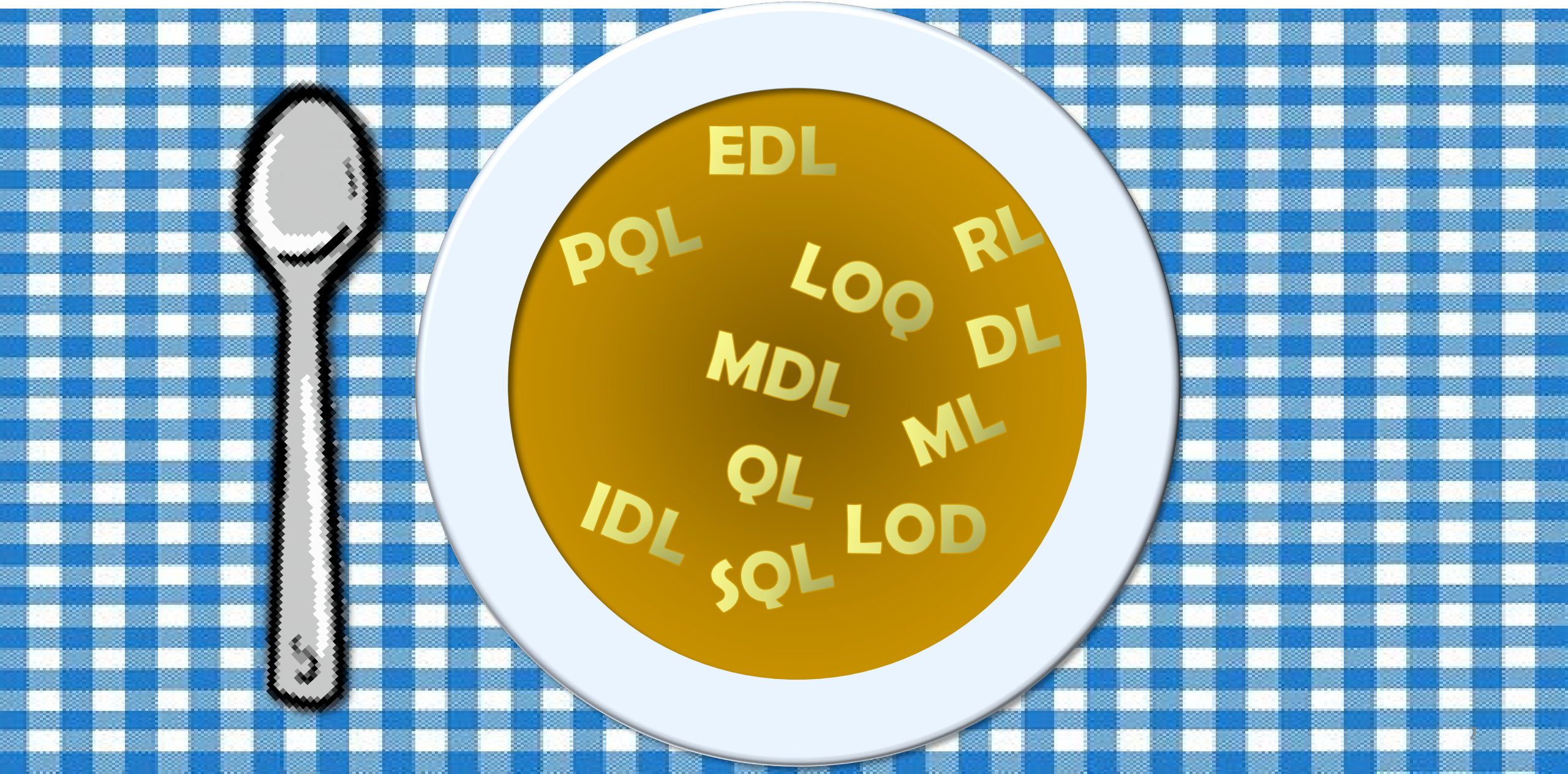
- What regulatory or screening criteria needs to be achieved?
- Are the lab's reporting limits below the regulatory or screening criteria?
- Do I need to request special methods to achieve the regulatory or screening criteria?

Soil Samples (mg/kg)			
Analyte	**Sample RL (method)**	**EPA RSL/Residential**	**EPA RSL/Industrial**
Benzo(a)pyrene	0.33 (8270) 0.033 (8270 SIM)	0.11	2.1
Arsenic	1.0 (6010) 0.1 (6020)	0.68	3.0

RSL = Regional Screening Level
SIM = Selective Ion Monitoring

If Reporting Limits (RLs) above screening criteria, may not be able to achieve objectives.

Detection Limits



Detection Limit Terminology

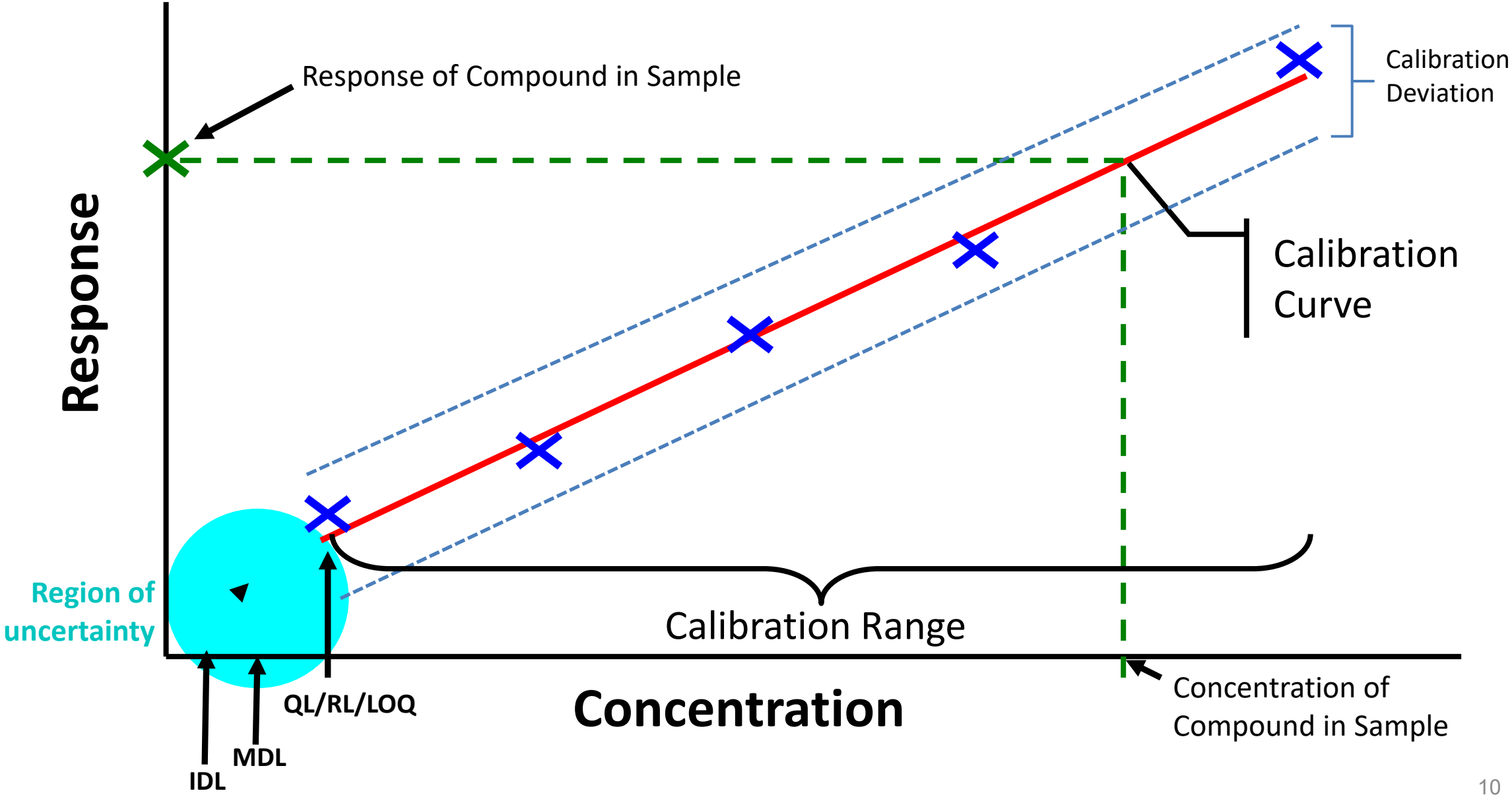
Acronym	Definition
IDL	Instrument Detection Limit
EDL	Estimated Detection Limit
DL	Detection Limit
MDL	Method Detection Limit
LOD	Limit of Detection
PQL	Practical Quantitation Limit
RL	Reporting Limit
QL	Quantitation Limit
SQL	Sample Quantitation Limit
LOQ	Limit of Quantitation
LLOQ	Lower Limit of Quantitation

Different Detection Limits

Detection Limit	Accurate?	Precise?	Use to Demonstrate Below Cleanup Standards?	Use Values in Risk Assessment?
IDL	No	Yes	No	No
EDL[1]	No	Yes	Yes	Yes
MDL / DL	No	Yes	No	No
LOD[2]	No	Yes	Yes	Yes
PQL	Yes	Yes	No	No
RL / QL / SQL / LOQ/LLOQ	Yes	Yes	Yes	Yes

[1]Specific to dioxins/furans
[2]Specific to DoD projects

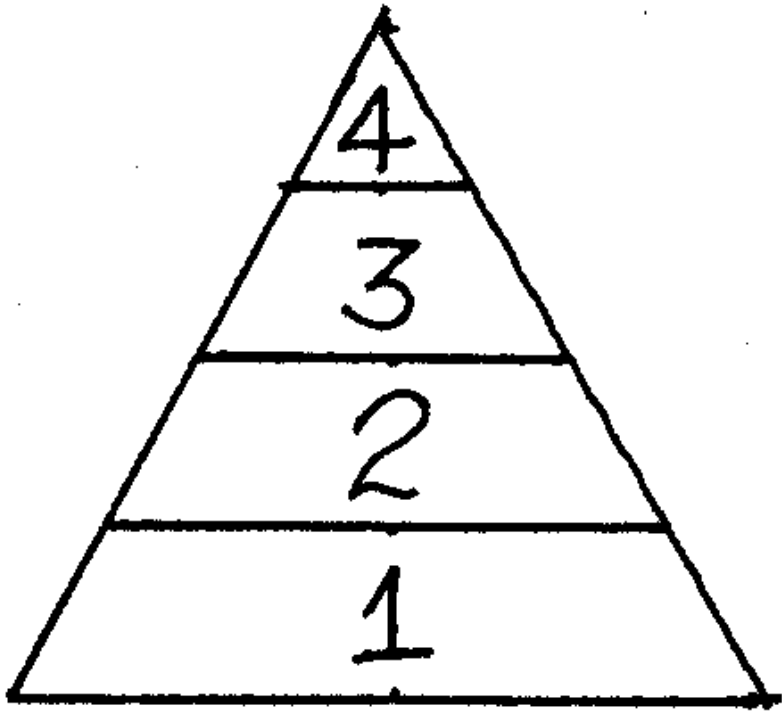
Calibration



What Type of Laboratory Deliverable do I Need?

- **Level 1, Level 2, Level 3, or Level 4** data package?

- **Level 1**: Sample results only: NEVER Request this.
- **Level 2: Sample results, QC sample results, laboratory narrative**
- **Level 3:** Level 2 plus calibration results
- **Level 4:** Level 3 plus all associated raw data



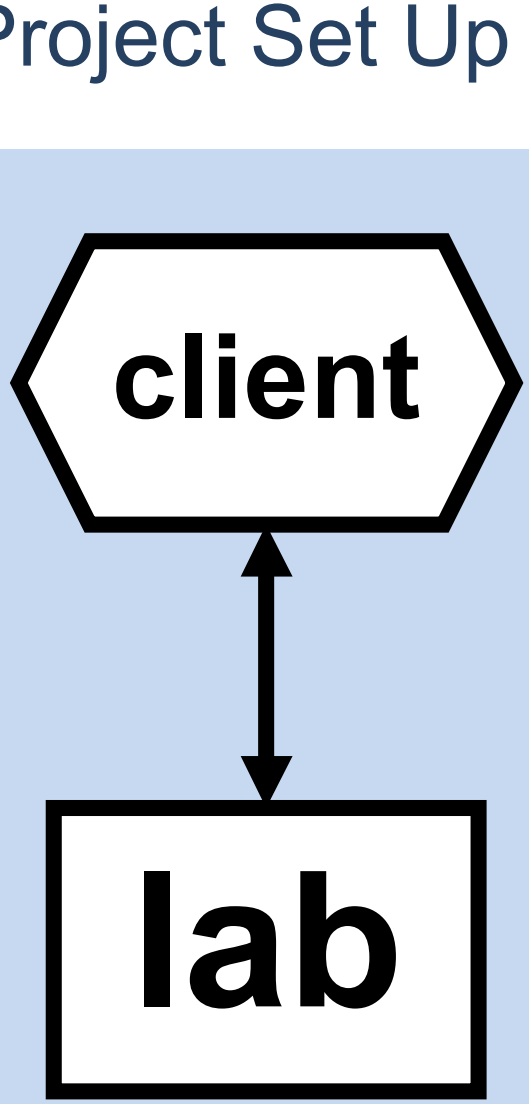
- **Level 2** will suffice in most cases.
- **Level 3 or 4:** consider for critical decision-making (risk assessment, remediation effectiveness, potential litigation)

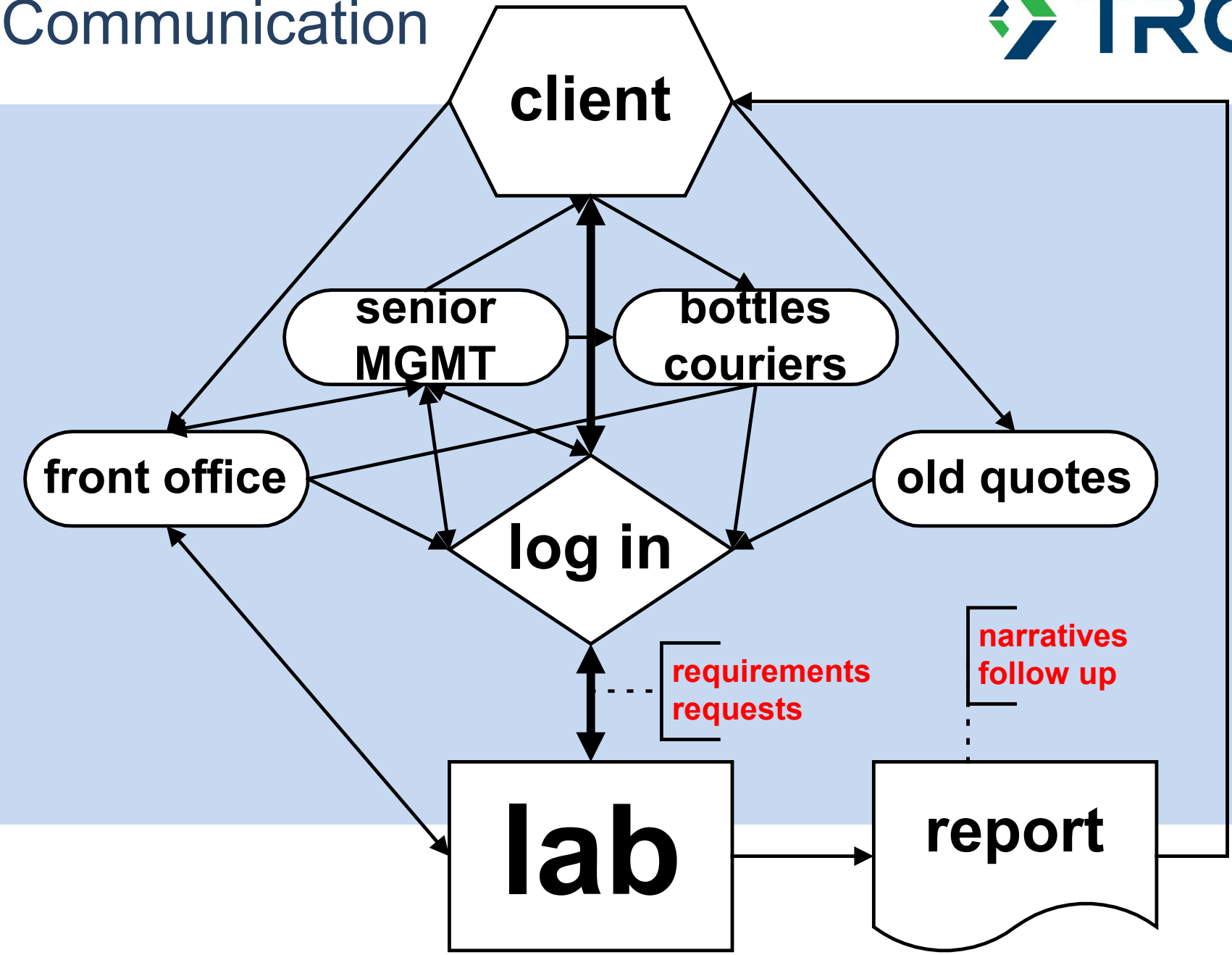
Field Quality Control: What are the Options?

QC Sample	Why Should I Collect?	How Often Should I Collect?
Trip Blank	To evaluate VOC contamination during shipment of samples	1 per every cooler containing samples for VOCs
Field Blank	To evaluate presence of contaminants in ambient air at the site	1 per day per parameter
Equipment Blank	To evaluate presence of contaminants on equipment after decontamination	1 per day per matrix and parameter
Field Duplicate	To evaluate sampling and analytical precision	1 per 20 samples per matrix and parameter
Matrix spikes/matrix spike duplicates (MS/MSDs)	To evaluate matrix-specific bias	1 per 20 samples per matrix and parameter
Cooler Temperature Blank	To ensure proper preservation of samples maintained during shipment	1 per each cooler

Communicating With the Laboratory

Project Set Up - Communication



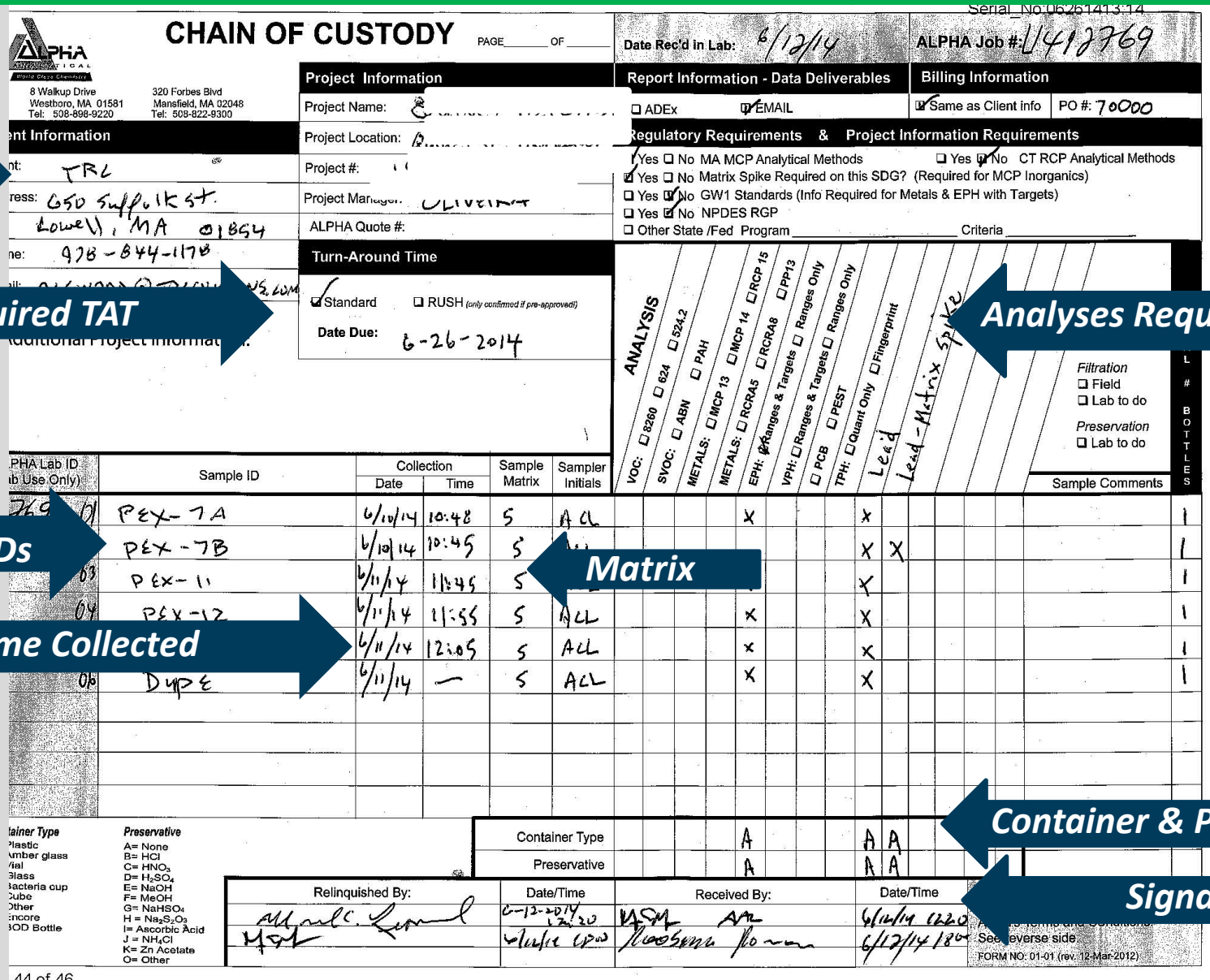


What Do We Need to Discuss With the Lab Before Samples Arrive?

- **State/federal agency involved; ensure lab has required certifications**
- No. of samples for each matrix, including QC samples
- Analyses required for each matrix
  - Analyte lists
  - Special analytical needs (e.g., SIM)?
- Project screening criteria
  - Lab's RLs below criteria?
- Which detection limits should be used?
  - MDLs, RLs, LODs?
- Sampling schedule
  - Courier pickups?
  - Any short holding times?
  - How will samples be delivered?
  - Hazardous materials shipping?
- Turnaround Time (TAT) and required deliverables
  - Hardcopy and electronic data deliverable (EDD)
  - Surcharges for quick TAT
  - Any analyses that cannot be done rush
- Special reporting requirements
  - Soils on dry weight basis
  - **"J" values needed?**
- Unusual sample or project-specific info
- Bottles and preservatives needed for each matrix and parameter
- Pre-printed sample labels or COCs possible?
- Quality Assurance Project Plan (QAPP)/Sampling & Analysis Plan (SAP)
- **Canister sampling: Batch or individual certification needed?**

Sample Traceability/Chain-of-Custody

Were COC forms Appropriately Completed?



CHAIN OF CUSTODY

ALPHA Job #: L1418769

Date Rec'd in Lab: 6/12/14

Project Information

Report Information - Data Deliverables

Billing Information

Turn-Around Time

Standard ☐ RUSH (only confirmed if pre-approved)

Date Due: 6-26-2014

Regulatory Requirements & Project Information Requirements

ANALYSIS

Sample ID | Collection Date | Collection Time | Sample Matrix | Sampler Initials | Sample Comments

PEX-7A 6/10/14 10:48 S ACL

PEX-7B 6/10/14 10:45 S

PEX-11 6/11/14 11:45 S

PEX-12 6/11/14 11:55 S ACL

6/11/14 12:05 S ACL

DUPE 6/11/14 — S ACL

Container Type

Preservative

Relinquished By: | Date/Time | Received By: | Date/Time

6/12/14 1220

6/13/14 1800

FORM NO: 01-01 (rev. 12-Mar-2012)

Company Name & Information

Required TAT

Analyses Requested; MS Designation

Sample IDs

Matrix

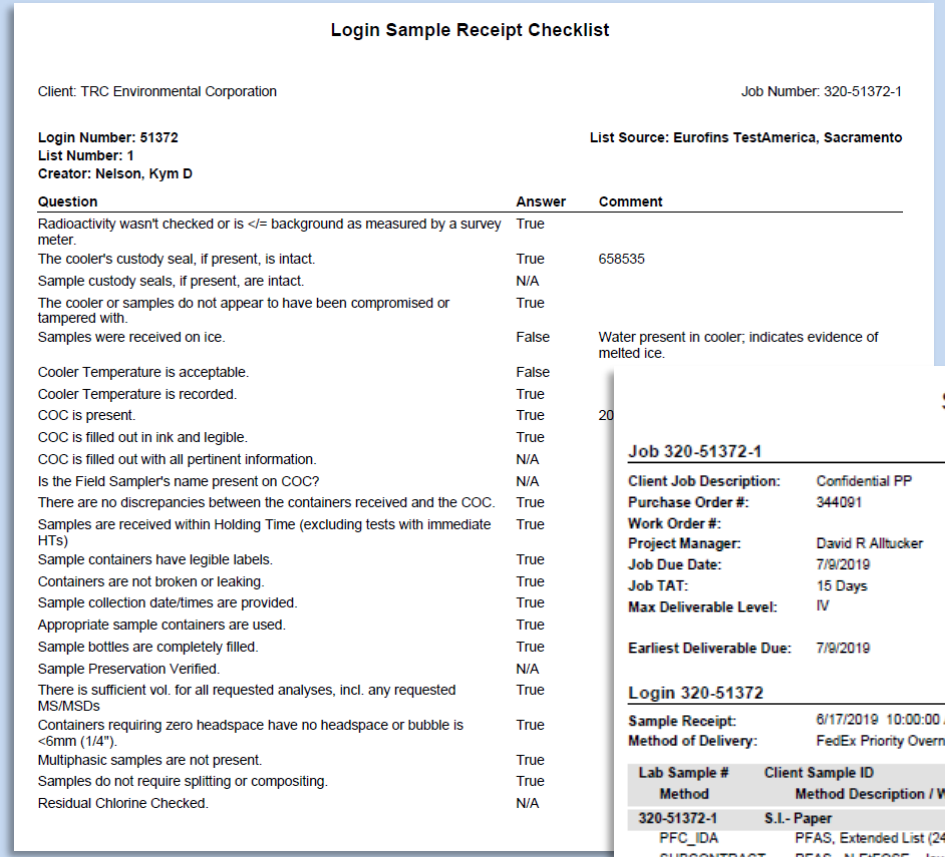
Date/Time Collected

Container & Preservation

Signatures

Communication After Samples are Received by the Lab

- Make sure lab sends sample receipt confirmations
  - Did lab transcribe sample IDs properly?
  - Did lab log in samples for the correct analyses?
  - Were there any issues with samples (e.g., broken bottles)?

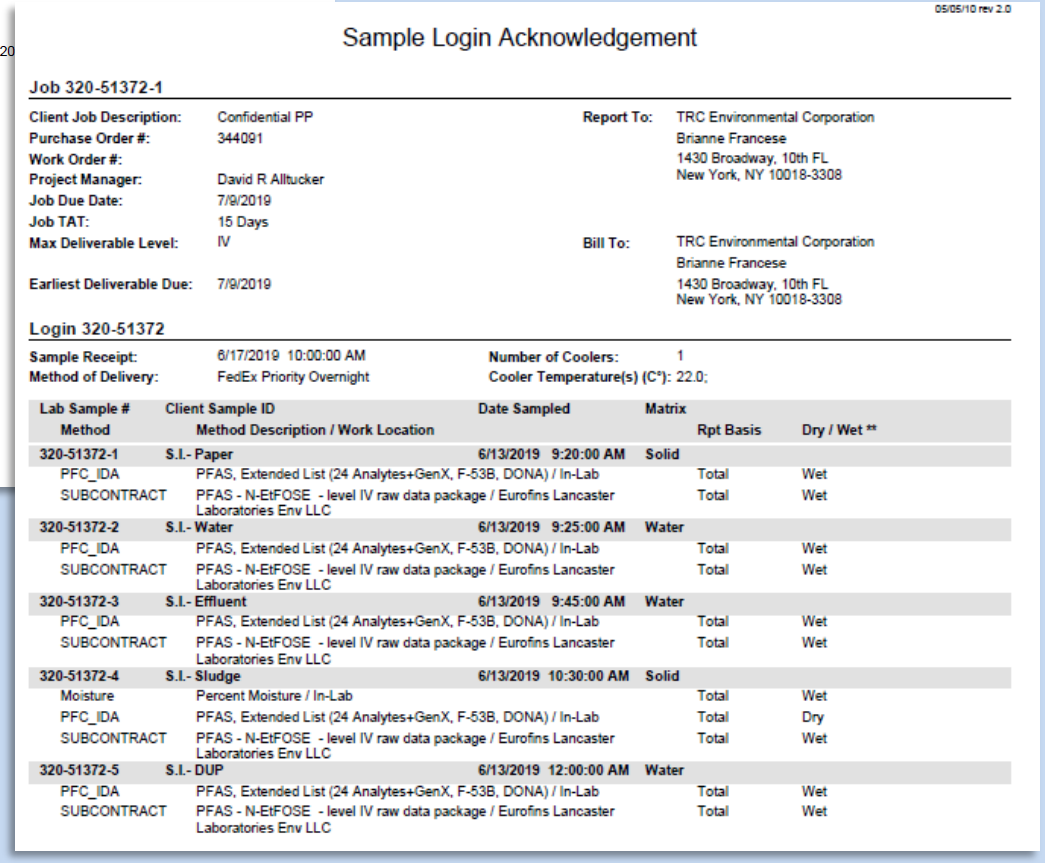


Login Sample Receipt Checklist

Client: TRC Environmental Corporation — Job Number: 320-51372-1

Login Number: 51372
List Number: 1
Creator: Nelson, Kym D
List Source: Eurofins TestAmerica, Sacramento

Question	Answer	Comment
Radioactivity wasn't checked or is </= background as measured by a survey meter.	True	
The cooler's custody seal, if present, is intact.	True	658535
Sample custody seals, if present, are intact.	N/A	
The cooler or samples do not appear to have been compromised or tampered with.	True	
Samples were received on ice.	False	Water present in cooler; indicates evidence of melted ice.
Cooler Temperature is acceptable.	False	
Cooler Temperature is recorded.	True	
COC is present.	True	20
COC is filled out in ink and legible.	True	
COC is filled out with all pertinent information.	N/A	
Is the Field Sampler's name present on COC?	N/A	
There are no discrepancies between the containers received and the COC.	True	
Samples are received within Holding Time (excluding tests with immediate HTs)	True	
Sample containers have legible labels.	True	
Containers are not broken or leaking.	True	
Sample collection date/times are provided.	True	
Appropriate sample containers are used.	True	
Sample bottles are completely filled.	True	
Sample Preservation Verified.	N/A	
There is sufficient vol. for all requested analyses, incl. any requested MS/MSDs	True	
Containers requiring zero headspace have no headspace or bubble is <6mm (1/4").	True	
Multiphasic samples are not present.	True	
Samples do not require splitting or compositing.	True	
Residual Chlorine Checked.	N/A	



05/05/10 rev 2.0

Sample Login Acknowledgement

Job 320-51372-1

Client Job Description: Confidential PP
Purchase Order #: 344091
Work Order #:
Project Manager: David R Alltucker
Job Due Date: 7/9/2019
Job TAT: 15 Days
Max Deliverable Level: IV
Earliest Deliverable Due: 7/9/2019

Report To: TRC Environmental Corporation, Brianne Francese, 1430 Broadway, 10th FL, New York, NY 10018-3308

Bill To: TRC Environmental Corporation, Brianne Francese, 1430 Broadway, 10th FL, New York, NY 10018-3308

Login 320-51372

Sample Receipt: 6/17/2019 10:00:00 AM
Method of Delivery: FedEx Priority Overnight
Number of Coolers: 1
Cooler Temperature(s) (C°): 22.0;

Lab Sample #	Client Sample ID	Date Sampled	Matrix		
Method	Method Description / Work Location			Rpt Basis	Dry / Wet **
320-51372-1	S.I.- Paper	6/13/2019 9:20:00 AM	Solid		
PFC_IDA	PFAS, Extended List (24 Analytes+GenX, F-53B, DONA) / In-Lab			Total	Wet
SUBCONTRACT	PFAS - N-EtFOSE - level IV raw data package / Eurofins Lancaster Laboratories Env LLC			Total	Wet
320-51372-2	S.I.- Water	6/13/2019 9:25:00 AM	Water		
PFC_IDA	PFAS, Extended List (24 Analytes+GenX, F-53B, DONA) / In-Lab			Total	Wet
SUBCONTRACT	PFAS - N-EtFOSE - level IV raw data package / Eurofins Lancaster Laboratories Env LLC			Total	Wet
320-51372-3	S.I.- Effluent	6/13/2019 9:45:00 AM	Water		
PFC_IDA	PFAS, Extended List (24 Analytes+GenX, F-53B, DONA) / In-Lab			Total	Wet
SUBCONTRACT	PFAS - N-EtFOSE - level IV raw data package / Eurofins Lancaster Laboratories Env LLC			Total	Wet
320-51372-4	S.I.- Sludge	6/13/2019 10:30:00 AM	Solid		
Moisture	Percent Moisture / In-Lab			Total	Wet
PFC_IDA	PFAS, Extended List (24 Analytes+GenX, F-53B, DONA) / In-Lab			Total	Dry
SUBCONTRACT	PFAS - N-EtFOSE - level IV raw data package / Eurofins Lancaster Laboratories Env LLC			Total	Wet
320-51372-5	S.I.- DUP	6/13/2019 12:00:00 AM	Water		
PFC_IDA	PFAS, Extended List (24 Analytes+GenX, F-53B, DONA) / In-Lab			Total	Wet
SUBCONTRACT	PFAS - N-EtFOSE - level IV raw data package / Eurofins Lancaster Laboratories Env LLC			Total	Wet

Evaluation of Analytical Data

Why Do We Need to Evaluate the Lab's Data?

- Data may be used to make costly decisions
- Data may have potential to impact human health
- Need to confirm quality data available and appropriate to support decisions
- Need to determine potential low or high biases, potential uncertainties, potential false positive or false negative results

Even if the lab follows all method-required procedures, there can still be data quality/usability issues.

Effective Post-Sampling Laboratory Communications

- Read the Report / Review the Data
  - As soon as you get it!

- Why?
  - Information is extremely time-sensitive – Anything requiring the laboratory to re-analyze samples dependent on hold time
  - The longer you wait, the shorter the memories become, both in the lab and with the field personnel that collected the samples
  - The longer you wait, the longer it takes a laboratory to produce requested documentation

- ❑ Right list of analytes?
- ❑ All samples analyzed?
- ❑ Required reporting limits achieved?
- ❑ Were "J" values reported?
- ❑ Does anything look unusual?

Data Completeness

Results for all requested analytical parameters?	Check vs. COC, QAPP
Results reported for all samples submitted?	Check vs. COC
Requested analytical methods used?	Check vs. COC, scope of work, QAPP, etc.
Results for requested target analytes?	Check vs. scope of work or QAPP, etc.
Soil/sediment results reported on dry weight basis?	
Were “J” values reported/requested? (gas chromatography/mass spectrometry [GC/MS], Inductively coupled plasma[ICP]/MS)	
Did we receive the required deliverables?	Check vs. scope of work or QAPP, etc.

Evaluation Categories

- Laboratory Performance
- Field Performance
- Matrix Interferences

Laboratory Performance	Field Performance	Matrix Interferences
Method Blanks	Field Blanks	Surrogates
Lab Control Samples	Sample Preservation	Internal Standards*
Holding Times	Field Duplicates	Matrix Spikes
Calibrations*		Laboratory Duplicates
Tunes*		

**Not typically included in Level 2 deliverables*

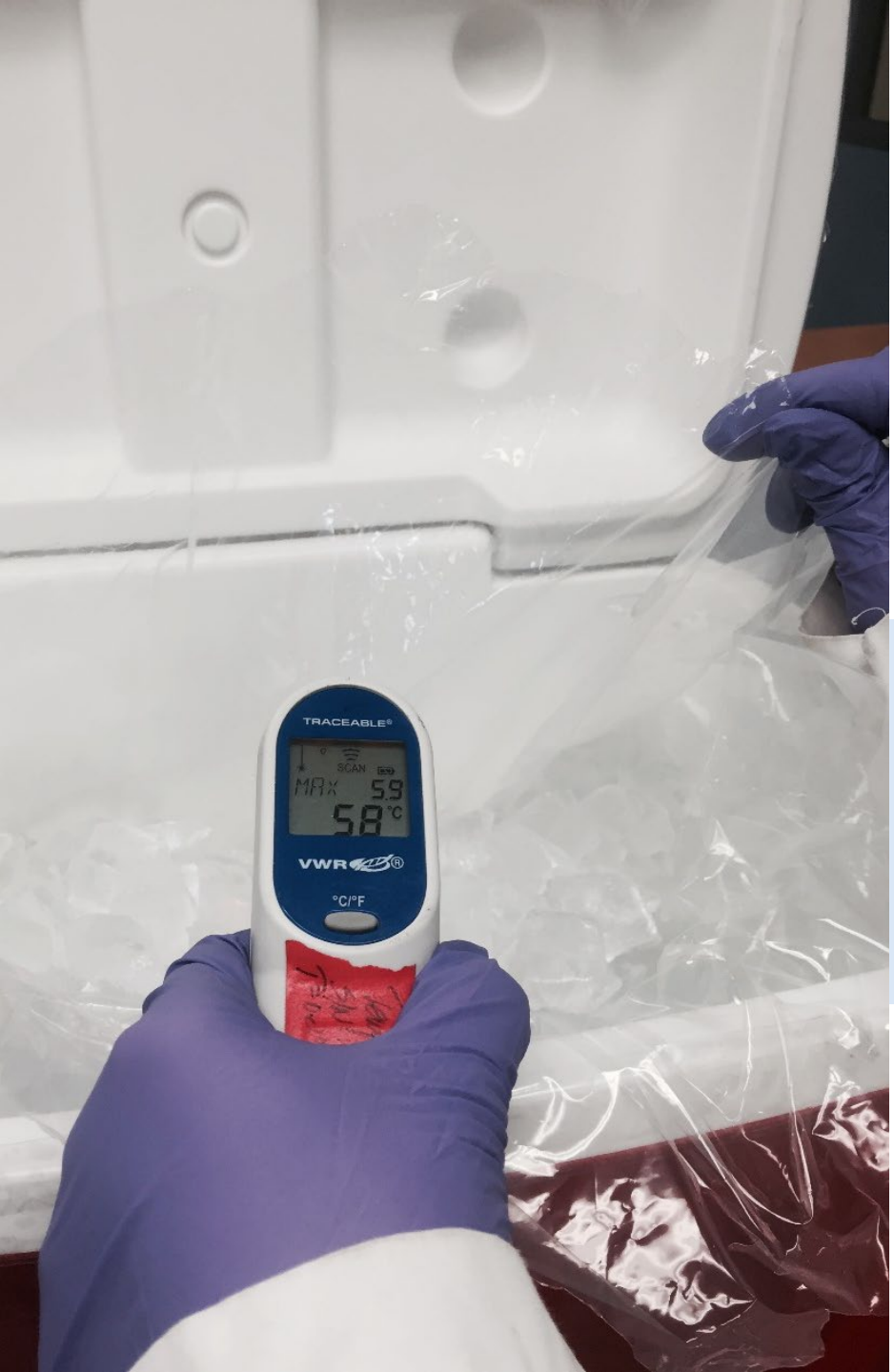
What is Affected by Each Parameter?

Sample-Specific	Batch-Specific
Holding Time	Method Blanks
Sample Preservation	Lab Control Samples
Field Duplicates	Calibrations*
Surrogates	Tunes*
Internal Standards*	Field Blanks
Matrix Spikes	
Laboratory Duplicates	

**Not typically included in Level 2 deliverables*

Organic Versus Inorganic Methods: *Examples*

Organic	Inorganic
Volatile Organic Compounds (VOCs)	Metals
Semivolatile Organic Compounds (SVOCs) Polycyclic Aromatic Hydrocarbons (PAHs)	Cyanide
Pesticides	Hexavalent Chromium
Polychlorinated Biphenyls (PCBs)	Anions
Dioxins/Furans	Hardness
Total Petroleum Hydrocarbons (TPH) Extractable Petroleum Hydrocarbons (EPH) Volatile Petroleum Hydrocarbons (VPH)	
Per- and Polyfluoroalkyl Substances (PFAS)	



Sample Preservation & Integrity

Was the Cooler Temperature ≤ 6° C?

Typically noted on COC or separate cooler receipt form.

What if samples are delivered to the laboratory on the same day of collection and temperatures are outside of the acceptance criteria but samples are on ice?

Temperature too low: likely okay as long as waters not frozen
Temperature too high: use professional judgment

- Metals
- PCBs or Dioxins
- VOCs (potential low bias)

Sample Preservation & Integrity

Was the Proper Chemical Preservative Used?

- Chemical Preservation of Aqueous Samples (examples)
  - **Metals** (nitric acid; pH <2)
  - **VOCs** (hydrochloric acid; pH <2)
  - **TOC** (sulfuric acid; pH <2)
  - **Cyanide** (sodium hydroxide; pH >12)
- Chemical Preservation of Soil/Sediment Samples
  - Most common: **VOCs**
    - ✓ **Low-level:** 5 grams with sodium bisulfate/DI water and magnetic stir bar
    - ✓ **Low-level**: 5 grams with DI water and magnetic stir bar
    - ✓ **High-level:** 5 grams with methanol

Most labs will note on cooler receipt form or in narrative IF there is an issue.

Option to chemical preservation is EnCore™ samplers.

Potential Issues with Lack of Preservation

Water VOCs not acid-preserved	Holding time reduced to 7 days
Water: other parameters not properly preserved	If >1 day to preservation, may have low bias
Soil VOCs not preserved	Detected results low bias Nondetect results rejected

Significant Data Quality Issue:
Lack of Preservation for Soil Samples for VOCs

Sample Preservation & Integrity

Air Samples

- Canister Vacuums
- Flow Controller Calibrations

Flow controller relative percent differences (RPDs) should be <20.

Want canister vacuum to be between 5-10” Hg; don’t want it to be 0.

Canister and Flow Controller Information

Samplenum	Client ID	Media ID	Media Type	Date Prepared	Bottle Order	Cleaning Batch ID	Can Leak Check	Initial Pressure (in. Hg)	Pressure [illegible] Receipt [illegible]. Hg)	Flow Controler Leak Chk	Flow Out mL/min	Flow In mL/min	% RPD
L1414095-01	5ALLB-1	0383	#20 AMB	06/23/14	104424		-			Pass	3.3	3.4	3
L1414095-01	5ALLB-1	999	6.0L Can	06/23/14	104424	L1413371-04	Pass	-29.2	-6.0	-	-	-	-
L1414095-02	5ALLB-2	0256	#16 SV	06/23/14	104424		-	-	-	Pass	3.1	3.2	3
L1414095-02	5ALLB-2	969	6.0L Can	06/23/14	104424	L1413442-02	Pass	-29.6	-8.0	-	-	-	-
L1414095-03	9ALLB-1	0201	#16 AMB	06/23/14	104424		-	-	-	Pass	3.2	3.4	6
L1414095-03	9ALLB-1	1526	6.0L Can	06/23/14	104424	L1413371-03	Pass	-29.5	-7.4	-	-	-	-
L1414095-04	9ALLB-2	0201	#16 AMB	06/23/14	104424		-	-	-	Pass	3		
L1414095-04	9ALLB-2	639	6.0L Can	06/23/14	104424	L1411852-01	Pass	-29.1	-4.6	-	-	-	-
L1414095-05	252-1	0240	#16 AMB	06/23/14	104424		-	-	-	Pass	3.0	0.4	153
L1414095-05	252-1	1799	6.0L Can	06/23/14	104424	L1413293-01	Pass	-29.3	-22.1	-	-	-	-

Sample Preservation & Integrity

Were Preparation & Analytical Holding Times Met?

- Need to check for ***every*** sample and ***every*** analysis
- Make sure lab provides both **PREPARATION** and **ANALYSIS** dates, when applicable.

Holding Times for Some Common Analyses

Holding Times and Preservation Requirements			
Parameter	**Reference Method**	**Acceptance Criteria**	
		Aqueous	**Solid**
VOCs	EPA 8260B	14 days (pH <2 with HCl) 7 days (un-preserved)	Low-level in 5 mL water: 48 hours to storage in freezer; 14 days to analysis Low-level in 5 mL NaHSO4: 14 days to analysis High-level in methanol: 14 days to analysis EnCore samplers: 48 hours to preservation for low-level and high-level analyses; above holding times for analysis apply
SVOCs	EPA 8270D	7 days to extraction; 40 days from extraction to analysis	14 days to extraction; 40 days from extraction to analysis
Metals (ICP-AES)	EPA 6010C	6 months to analysis (pH <2 with HNO_3)	6 months to analysis
Mercury	EPA 7470A, EPA 7471B	28 days to analysis (pH <2 with HNO_3)	28 days to analysis
Metals (GFAA)	EPA 7010	6 months to analysis (pH <2 with HNO_3)	6 months to analysis
Metals (ICP-MS)	EPA 6020A	6 months to analysis (pH <2 with HNO_3)	6 months to analysis
TPH	EPA 8015B	GRO: 14 days to analysis (pH <2 with HCl) DRO: 7 days to extraction; 40 days from extraction to analysis	GRO: 14 days to analysis (methanol) DRO: 14 days to extraction; 40 days from extraction to analysis
PCBs	EPA 8082A	1 year to extraction; 40 days from extraction to analysis	1 year to extraction; 40 days from extraction to analysis
Pesticides	EPA 8081B	7 days to extraction; 40 days from extraction to analysis	14 days to extraction; 40 days from extraction to analysis
Herbicides	EPA 8151A	7 days to extraction; 28 days to methylation; analysis immediately after methylation	14 days to extraction; 28 days to methylation; analysis immediately after methylation

Example Holding Time (HT) Check

PAHs in Soil

Collected 6/11/23

The Lab provides **dates prepared** and **dates analyzed**.
The **collection** dates are on the COC.

Therefore, YOU can calculate whether holding times were met.

Extracted 6/16/23

- 5 days lapse
- (HT = 14 days from collection)

Analyzed 6/23/23

- 7 days lapse
- (HT = 40 days from extraction)

If Holding time missed: *Data potentially biased low*

If Holding time missed by >2x holding time: *Data may not be usable for project objectives*

Evaluate Holding Times

VOCs: 14 days to analysis
SVOCs: 14 days to extraction; 40 days from extraction to analysis

Typical sample result summary form

- Number of VOCs reported
- Results, RLs, units
- Dilution results
- Collection date, prepared date, analysis date
- Percent solids (dry weight)
- Surrogate recoveries

VOCs in soil

Client Sample ID: -SS-01-TOP
Date Collected: 01/29/20 09:50
Date Received: 01/30/20 09:30

Lab Sample ID: XX XXX-1
atrix: Solid
Percen olids: 79.9

Method: 8260C - Volatile Organic Compounds by GC/MS

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
1,1,1-Trichloroethane	ND		5.75		ug/Kg	☼	01/30/20 13:45	01/31/20 12:23	1
1,2-Dichlorobenzene	ND		5.75		ug/Kg	☼	01/30/20 13:45	01/31/20 12:23	1
1,1,2,2-Tetrachloroethane	ND		5.75		ug/Kg	☼	01/30/20 13:45	01/31/20 12:23	1
1,1,2-Trichloroethane	ND		5.75		ug/Kg	☼	01/30/20 13:45	01/31/20 12:23	1

SVOCs in soil

Client Sample ID: -SS-01-TOP
Date Collected: 01/29/20 09:50
Date Received: 01/30/20 09:30

Method: 8270D - Semivolatile Organic Compounds (GC/MS)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Biphenyl	ND		2120	312	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
bis (2-chloroisopropyl) ether	ND		2120	424	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
2,4,5-Trichlorophenol	ND		2120	574	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
2,4,6-Trichlorophenol	ND		2120	424	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
2,4-Dichlorophenol	ND		2120	225	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
2,4-Dimethylphenol	ND		2120	511	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10

If RLs Above Screening Criteria, Why?

Analytical Limitation

- **EPA RSLs or MCLs** needed for PAHs? Request SIM
- Metals: may need ICP/MS (6020/200.8) instead of ICP/atomic emission spectroscopy (AES) (6010/200.7)

Dilutions Performed

- Is dilution reasonable?
- Does the lab note if it is due to non-target compounds?
- Is it due to an elevated extract volume?

Is It Obvious Why Lab Performed Dilution?

Method: 8270D - Semivolatile Organic Compounds (GC/MS)

Analyte	Result	Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Biphenyl	ND		2120	312	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
bis (2-chloroisopropyl) ether	ND		2120	424	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
2,4,5-Trichlorophenol	ND		2120	574	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
2,4,6-Trichlorophenol	ND		2120	424	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
2,4-Dichlorophenol	ND		2120	225	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
2,4-Dimethylphenol	ND		2120	511	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
2,4-Dinitrophenol	ND		20700	9780	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
2,4-Dinitrotoluene	ND		2120	437	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
2,6-Dinitrotoluene	ND		2120	249	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
2-Chloronaphthalene	ND		2120	349	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
2-Chlorophenol	ND		4120	387	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
2-Methylnaphthalene	ND		2120	424	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
2-Methylphenol	ND		2120	249	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
2-Nitroaniline	ND		4120	312	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
2-Nitrophenol	ND		2120	599	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
3,3'-Dichlorobenzidine	ND		4120	2490	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
3-Nitroaniline	ND		4120	586	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
4,6-Dinitro-2-methylphenol	ND		4120	2120	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
4-Bromophenyl phenyl ether	ND		2120	299	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
4-Chloro-3-methylphenol	ND		2120	524	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
4-Chloroaniline	ND		2120	524	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
4-Chlorophenyl phenyl ether	ND		2120	262	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
4-Methylphenol	ND		4120	249	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
4-Nitroaniline	ND		4120	1110	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
4-Nitrophenol	ND		4120	1480	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
Acenaphthene	ND		2120	312	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
Acenaphthylene	ND		2120	274	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
Acetophenone	ND		2120	287	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
Anthracene	ND		2120	524	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
Atrazine	ND		2120	736	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
Benzaldehyde	ND		2120	1680	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
Benzo[a]anthracene	882	J	2120	212	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
Benzo[a]pyrene	1020	J	2120	312	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
Benzo[b]fluoranthene	1220	J	2120	337	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
Benzo[g,h,i]perylene	881	J	2120	225	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
Benzo[k]fluoranthene	540	J	2120	274	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
Bis(2-chloroethoxy)methane	ND		2120	449	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
Bis(2-chloroethyl)ether	ND		2120	274	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
Bis(2-ethylhexyl) phthalate	ND		2120	724	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
Butyl benzyl phthalate	ND		2120	349	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
Caprolactam	ND		2120	636	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
Carbazole	ND		2120	249	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10
Chrysene	844	J	2120	474	ug/Kg	☼	01/31/20 14:48	02/03/20 21:31	10

10-fold dilution

Is there an explanation in the narrative?

Method 8270D: The following samples were diluted due to color and appearance: TRACON-SS-01-TOP (XXXXXX), TRACON-SS-01-BTM (XXXXXX-2), TRACON-SS-02-TOP (XXXXXX-3), TRACON-SS-03-TOP (XXXXXX-5), TRACON-SS-03-BTM (XXXXXX-6), TRACON-SS-04-TOP (XXXXXX-7), TRACON-SS-04-TOP (XXXXXX-7[MS]), TRACON-SS-04-TOP (XXXXXX-7[MSD]), TRACON-SS-04-BTM (XXXXXX-8), TRACON-SS-05-TOP (XXXXXX-9), TRACON-SS-05-BTM (XXXXXX-10) and TRACON-SS-06-TOP (XXXXXX-11). Elevated reporting limits (RL) are provided.

"J" flag: result is below RL but above MDL; is estimated value

Reminders

Laboratory Performance	Field Performance	Matrix Interferences
Method Blanks	Field Blanks	Surrogates
Lab Control Samples	Sample Preservation	Internal Standards*
Holding Times	Field Duplicates	Matrix Spikes
Calibrations*		Laboratory Duplicates
Tunes*		

* Not typically provided in Level 2 deliverables

Sample-Specific	Batch-Specific
Holding Times	Method Blanks
Sample Preservation	Lab Control Samples
Field Duplicates	Calibrations*
Surrogates	Tunes*
Internal Standards*	Field Blanks
Matrix Spikes	
Laboratory Duplicates	

* Not typically provided in Level 2 deliverables

Blanks: Method Blanks, Field Blanks, & Equipment Blanks

- Purposes:
  - **Method Blank:**
    To check for potential lab contamination in the sample preparation and analysis step
  - **Field/Equipment/Trip Blanks:**
    To check for potential contamination from ambient field conditions, equipment, or shipping/storage

- Does each prep batch have its own method blank?
- Were field/equipment blanks collected at the required frequency?

Method Blank Data in Your Data Package

Method: 8260C - Volatile Organic Compounds by GC/MS

Lab Sample ID: MB 480-515744/2-A
Matrix: Solid
Analysis Batch: 515698

Client Sample ID: Method Blank
Prep Type: Total/NA
Prep Batch: 515744

Analyte	MB Result	MB Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
1,1,1-Trichloroethane	ND		5.00		ug/Kg		01/31/20 10:30	01/31/20 11:48	1
1,2-Dichlorobenzene	ND		5.00		ug/Kg		01/31/20 10:30	01/31/20 11:48	1
1,1,2,2-Tetrachloroethane	ND		5.00		ug/Kg		01/31/20 10:30	01/31/20 11:48	1
1,1,2-Trichloroethane	ND		5.00		ug/Kg		01/31/20 10:30	01/31/20 11:48	1
1,1,2-Trichloro-1,2,2-trifluoroethane	ND		5.00		ug/Kg		01/31/20 10:30	01/31/20 11:48	1
1,1-Dichloroethane	ND		5.00		ug/Kg		01/31/20 10:30	01/31/20 11:48	1
1,1-Dichloroethene	ND		5.00		ug/Kg		01/31/20 10:30	01/31/20 11:48	1
1,2,4-Trichlorobenzene	ND		5.00		ug/Kg		01/31/20 10:30	01/31/20 11:48	1
1,2-Dibromo-3-Chloropropane	ND		5.00		ug/Kg		01/31/20 10:30	01/31/20 11:48	1
1,2-Dichloroethane	ND		5.00		ug/Kg		01/31/20 10:30	01/31/20 11:48	1
1,2-Dichloropropane	ND		5.00		ug/Kg		01/31/20 10:30	01/31/20 11:48	1
1,3-Dichlorobenzene	ND		5.00		ug/Kg		01/31/20 10:30	01/31/20 11:48	1

Method: 6010C - Metals (ICP)

Lab Sample ID: MB 480-515713/1-A
Matrix: Solid
Analysis Batch: 516280

Client Sample ID: Method Blank
Prep Type: Total/NA
Prep Batch: 515713

Analyte	MB Result	MB Qualifier	RL	MDL	Unit	D	Prepared	Analyzed	Dil Fac
Antimony	ND		14.7		mg/Kg		01/31/20 10:43	02/04/20 13:07	1
Arsenic	ND		1.96		mg/Kg		01/31/20 10:43	02/04/20 13:07	1
Barium	ND		0.490		mg/Kg		01/31/20 10:43	02/04/20 13:07	1
Beryllium	ND		0.196		mg/Kg		01/31/20 10:43	02/04/20 13:07	1
Cadmium	ND		0.196		mg/Kg		01/31/20 10:43	02/04/20 13:07	1
Chromium	ND		0.490		mg/Kg		01/31/20 10:43	02/04/20 13:07	1
Lead	ND		0.979		mg/Kg		01/31/20 10:43	02/04/20 13:07	1
Nickel	ND		4.90		mg/Kg		01/31/20 10:43	02/04/20 13:07	1
Selenium	ND		3.92		mg/Kg		01/31/20 10:43	02/04/20 13:07	1
Silver	ND		0.588		mg/Kg		01/31/20 10:43	02/04/20 13:07	1
Molybdenum	ND		0.979		mg/Kg		01/31/20 10:43	02/04/20 13:07	1
Thallium	ND		5.88		mg/Kg		01/31/20 10:43	02/04/20 13:07	1

Method blanks will be reported in data package.

- Each method
- Each day of analysis/extraction
- Results for all target analytes reported in samples

Blank Evaluation

Method Blanks, Field Blanks, & Equipment Blanks

- Is anything detected in the blank?
- Are there any potential **false positives**?
- **General Rule of Thumb:**
  If concentration in sample <2x the blank concentration, the result is potentially a false positive
- **Common contaminants:**
  methylene chloride, acetone, phthalates, 2-butanone

2x Blank = 4 ug/L	2x Blank = 4 ug/L
Sample conc = 120 ug/L	Sample conc = 3 ug/L
Real Hit	False Positive

Acetone in Blank = 2 ug/L

Question

The lab method blank contains 130 ug/kg benzene. One of the field samples associated with this blank contains 250 ug/kg benzene. How would you evaluate the benzene result in this field sample?

A. Report as is since it is highly unlikely benzene is a laboratory contaminant.

B. Flag the benzene result as a potential false positive since the concentration is <2x the blank concentration.

C. Report as is since the benzene result is > the blank concentration.

Reminders

Laboratory Performance	Field Performance	Matrix Interferences
Method Blanks	Field Blanks	Surrogates
Lab Control Samples	Sample Preservation	Internal Standards*
Holding Times	Field Duplicates	Matrix Spikes
Calibrations*		Laboratory Duplicates
Tunes*		

* Not typically provided in Level 2 deliverables

Sample-Specific	Batch-Specific
Holding Times	Method Blanks
Sample Preservation	Lab Control Samples
Field Duplicates	Calibrations*
Surrogates	Tunes*
Internal Standards*	Field Blanks
Matrix Spikes	
Laboratory Duplicates	

* Not typically provided in Level 2 deliverables

Laboratory Control Samples (LCS)

- **Purposes:** To check the accuracy of the method in the absence of any matrix effects
- What are LCSs?
- Does each analytical or prep batch have its own LCS?

LCS Data in Your Data Package

Lab Sample ID: LCS 480-515790/2-A
Matrix: Solid
Analysis Batch: 515974

Client Sample ID: Lab Control Sample
Prep Type: Total/NA
Prep Batch: 515790

Analyte	Spike Added	LCS Result	LCS Qualifier	Unit	D	%Rec	%Rec. Limits
Biphenyl	1660	1527		ug/Kg		92	59 - 120
bis (2-chloroisopropyl) ether	1660	1519		ug/Kg		92	44 - 120
2,4,5-Trichlorophenol	1660	1456		ug/Kg		88	59 - 126
2,4,6-Trichlorophenol	1660	1402		ug/Kg		85	59 - 123
2,4-Dichlorophenol	1660	1431		ug/Kg		86	61 - 120
2,4-Dimethylphenol	1660	1390		ug/Kg		84	59 - 120
2,4-Dinitrophenol	3320	2503		ug/Kg		75	41 - 146
2,4-Dinitrotoluene	1660	1626		ug/Kg		98	63 - 120
2,6-Dinitrotoluene	1660	1598		ug/Kg		96	66 - 120
2-Chloronaphthalene	1660	1399		ug/Kg		84	57 - 120
2-Chlorophenol	1660	1480		ug/Kg		89	53 - 120
2-Methylnaphthalene	1660	1468		ug/Kg		88	59 - 120
2-Methylphenol	1660	1483		ug/Kg		89	54 - 120

Lab Sample ID: LCSSRM 480-515713/2-A
Matrix: Solid
Analysis Batch: 516280

Client Sample ID: Lab Control Sample
Prep Type: Total/NA
Prep Batch: 515713

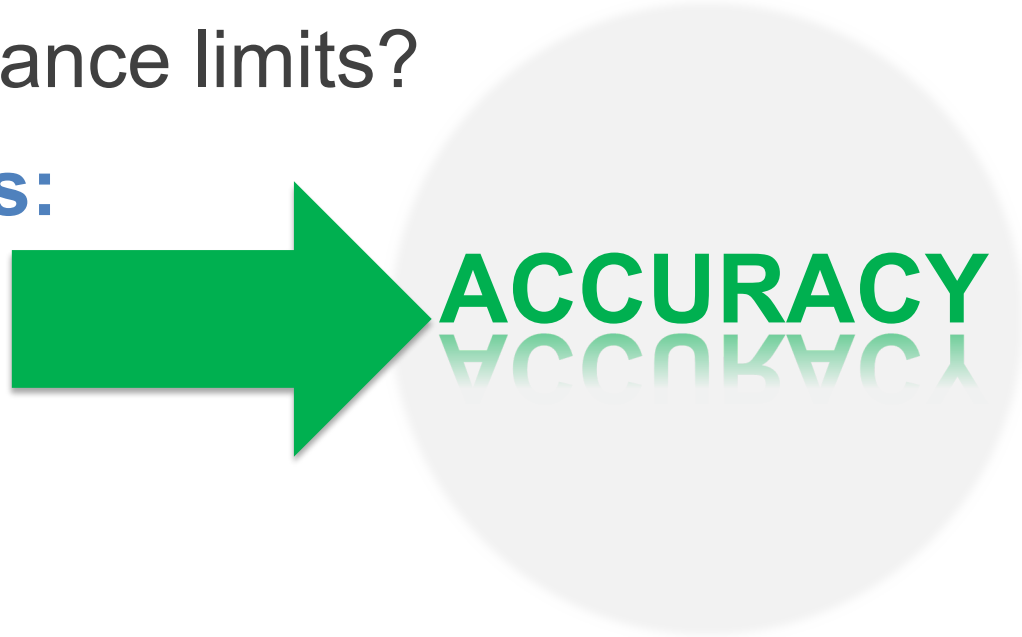
Analyte	Spike Added	LCSSRM Result	LCSSRM Qualifier	Unit	D	%Rec	%Rec. Limits
Antimony	282	98.17		mg/Kg		34.8	10.0 - 129.8
Arsenic	155	117.1		mg/Kg		75.5	64.5 - 120.0
Barium	439	343.6		mg/Kg		78.3	70.8 - 118.2
Beryllium	192	156.5		mg/Kg		81.5	69.8 - 116.7
Cadmium	61.5	49.35		mg/Kg		80.2	68.6 - 114.3
Chromium	104	84.94		mg/Kg		81.7	67.8 - 126.0
Lead	126	113.5		mg/Kg		90.1	70.9 - 127.

LCSs will be reported in data package.

- Each method
- Each day of analysis/extraction
- Percent recoveries for all target analytes reported in samples

Laboratory Control Sample (LCS) Evaluation

- Were **all** target analytes reported?
- Were all recoveries within the acceptance limits?
- **If LCS recoveries are outside limits:**
  - POTENTIAL LOW BIAS or
  - POTENTIAL HIGH BIAS



Affects the whole batch of samples prepared with the LCS.

Potentially unusable results:

- Organic methods: if recoveries are <10%
- Inorganic methods: if recoveries are <30%

Question

If naphthalene recovers at 25% in the LCS (criteria are 40-140%),
which of the following is true?

A. Naphthalene concentrations in the associated samples are biased high.

B. Naphthalene concentrations in the associated samples are biased low.

C. Naphthalene results are unusable.

Reminders

Laboratory Performance	Field Performance	Matrix Interferences
Method Blanks	Field Blanks	Surrogates
Lab Control Samples	Sample Preservation	Internal Standards*
Holding Times	Field Duplicates	Matrix Spikes
Calibrations*		Laboratory Duplicates
Tunes*		

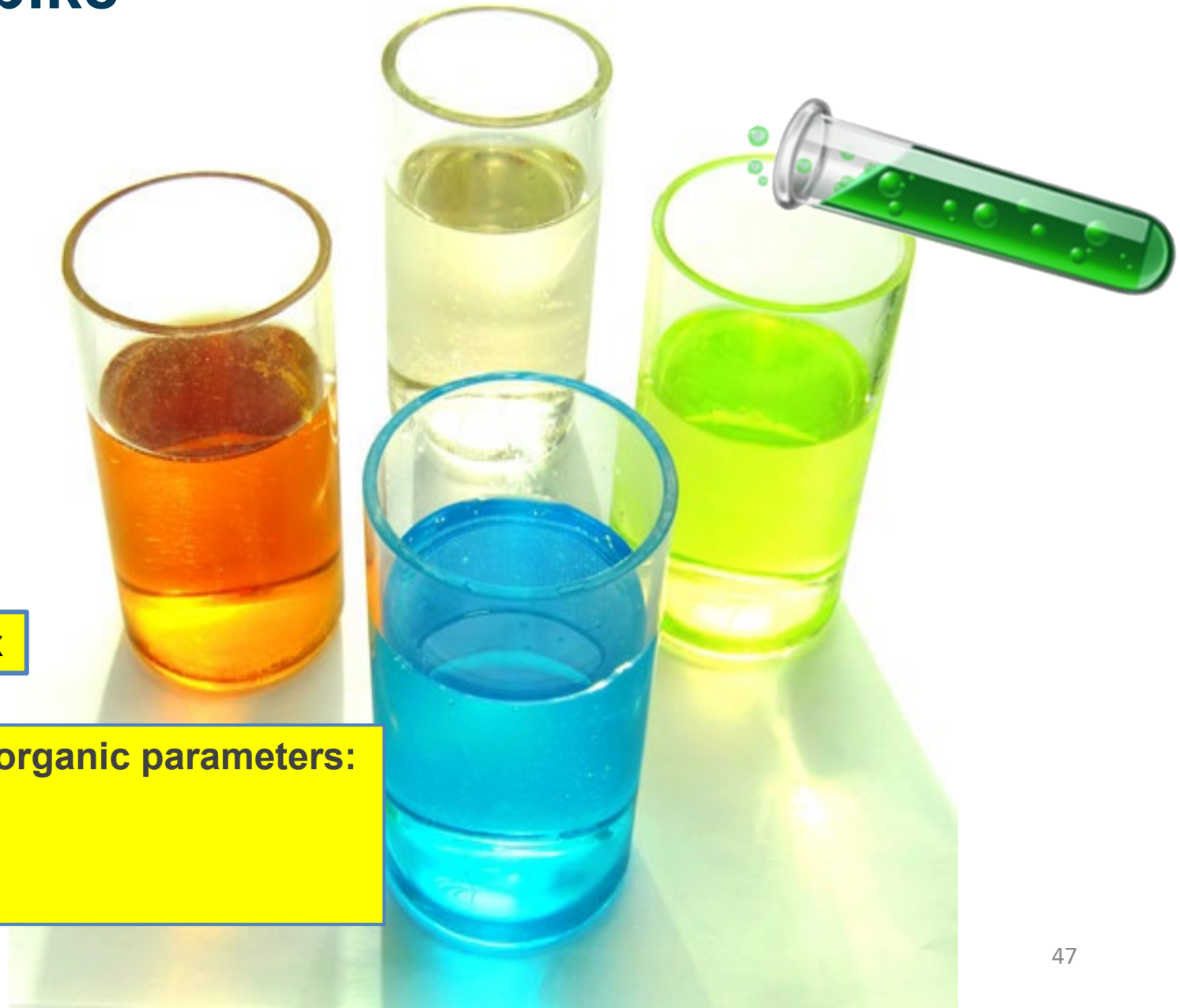
*Not typically provided in Level 2 deliverables

Sample-Specific	Batch-Specific
Holding Times	Method Blanks
Sample Preservation	Lab Control Samples
Field Duplicates	Calibrations*
Surrogates	Tunes*
Internal Standards*	Field Blanks
Matrix Spikes	
Laboratory Duplicates	

*Not typically provided in Level 2 deliverables

Matrix Spikes/Matrix Spike Duplicates (MS/MSDs)

- What are these?
- Were these analyses performed on a project sample?



MS/MSDs not applicable to the air matrix

Matrix spikes especially important for inorganic parameters:

- **Metals**
- **Hexavalent chromium**
- **Total cyanide**

MS/MSD Data in Your Data Package

Lab Sample ID: XXXXXX-7 MS
Matrix: Solid
Analysis Batch: 516280

Client Sample ID: -SS-04-TOP Prep
Type: Total/NA
Prep Batch: 515713

Analyte	Sample Result	Sample Qualifier	Spike Added	MS Result	MS Qualifier	Unit	D	%Rec	%Rec. Limits
Antimony	ND	F1	51.8	39.06	F1	mg/Kg	☼	74	75 - 125
Arsenic	5.34		51.8	50.51		mg/Kg	☼	87	75 - 125
Barium	46.1	F1	51.8	113.6	F1	mg/Kg	☼	130	75 - 125
Beryllium	0.431		51.8	47.64		mg/Kg	☼	91	75 - 125
Cadmium	0.391		51.8	47.00		mg/Kg	☼	90	75 - 125
Chromium	17.8		51.8	67.55		mg/Kg	☼	96	75 - 125
Lead	60.0		51.8	105.9		mg/Kg	☼	89	75 - 125
Nickel	10.8		51.8	60.19		mg/Kg	☼	95	75 - 125
Selenium	ND		51.8	45.63		mg/Kg	☼	86	75 - 125
Silver	ND		12.9	11.59		mg/Kg	☼	90	75 - 125
Molybdenum	ND		51.8	49.26		mg/Kg	☼	94	75 - 125
Thallium	ND		51.8	49.67		mg/Kg	☼	96	75 - 125
Vanadium	26.2		51.8	77.90		mg/Kg	☼	100	75 - 125
Zinc	140		51.8	182.5		mg/Kg	☼	82	75 - 125

Lab Sample ID: XXXXXX-7 MS
Matrix: Solid
Analysis Batch: 515698

Client Sample ID: -SS-04-TOP Prep
Type: Total/NA
Prep Batch: 515744

Analyte	Sample Result	Sample Qualifier	Spike Added	MS Result	MS Qualifier	Unit	D	%Rec	%Rec. Limits
1,1,1-Trichloroethane	ND	F2	163	141.0		ug/Kg	☼	86	77 - 121
1,2-Dichlorobenzene	ND	F1 F2	163	68.64	F1	ug/Kg	☼	42	75 - 120
1,1,2,2-Tetrachloroethane	ND	F1 F2	163	96.08	F1	ug/Kg	☼	59	80 - 120
1,1,2-Trichloroethane	ND	F1 F2	163	112.8	F1	ug/Kg	☼	69	78 - 122
1,1,2-Trichloro-1,2,2-trifluoroethane	ND	F2	163	125.3		ug/Kg	☼	77	60 - 140
1,1-Dichloroethane	ND	F2	163	140.2		ug/Kg	☼	86	73 - 126
1,1-Dichloroethene	ND	F2	163	138.5		ug/Kg	☼	85	59 - 125
1,2,4-Trichlorobenzene	ND	F1	163	35.79	F1	ug/Kg	☼	22	64 - 120
1,2-Dibromo-3-Chloropropane	ND	F1 F2	163	77.10	F1	ug/Kg	☼	47	63 - 124
1,2-Dichloroethane	ND	F2	163	132.4		ug/Kg	☼	81	77 - 122
1,2-Dichloropropane	ND	F2	163	130.8		ug/Kg	☼	80	75 - 124
1,3-Dichlorobenzene	ND	F1 F2	163	70.89	F1	ug/Kg	☼	43	74 - 120

Was it performed on a sample from your project?

MS/MSDs will be reported in data package.

- Each method, if performed
- Percent recoveries for all target analytes reported in samples

Matrix Spikes/Matrix Spike Duplicates (MS/MSDs)

- Were **all** target analytes reported?
- Were all recoveries within the acceptance limits? ➡ **ACCURACY**

- Were all RPDs within the acceptance limits? ➡ **PRECISION**

- **If MS recoveries are outside limits:**
  - POTENTIAL LOW BIAS or
  - POTENTIAL HIGH BIAS

Organics: Affects the sample that was spiked.

Inorganics: Affects all samples in data set of similar matrix (e.g., all groundwater)

Potentially unusable results:
- Organic methods: if recoveries are <10%
- Inorganic methods: if recoveries are <30%

Question

If arsenic recovers at 25% in the MS (criteria are 75-125%), which of the following is true?

A. Arsenic concentration in the associated sample is biased high.

B. Arsenic concentration in the associated sample is biased low.

C. Arsenic result in the associated sample is unusable.

Reminders

Laboratory Performance	Field Performance	Matrix Interferences
Method Blanks	Field Blanks	Surrogates
Lab Control Samples	Sample Preservation	Internal Standards*
Holding Times	Field Duplicates	Matrix Spikes
Calibrations*		Laboratory Duplicates
Tunes*		

*Not typically provided in Level 2 deliverables

Sample-Specific	Batch-Specific
Holding Times	Method Blanks
Sample Preservation	Lab Control Samples
Field Duplicates	Calibrations*
Surrogates	Tunes*
Internal Standards*	Field Blanks
Matrix Spikes	
Laboratory Duplicates	

*Not typically provided in Level 2 deliverables

Field Duplicates

- Were relative percent differences (RPDs) within the acceptance criteria?
- What are the acceptance criteria?
- Typical (when results are >5x the RL):
  - RPD ≤30 for aqueous and indoor air samples
  - RPD ≤50 for solid and soil gas samples

RPD = |X1 – X2| / (Average X1, X2) * 100
RPD = |Difference between two results|/ (Average of two results)* 100

Relative Percent Difference (RPD)

- RPD = |X1 – X2| / (Average X1, X2) * 100
- RPD = |Difference between two results|/ (Average of two results)* 100

NOT Percent Difference:

Percent Difference = (X2 – X1)/X1 (where X1 is the true value)

Some Potential Field Duplicate Scenarios

Both results <5x the RL: aqueous or air samples

Compound	RL (ug/L)	Sample (ug/L)	Field Dup (ug/L)	RPD
Benzene	1	1.8	0.3 J	52.6

When both results are <5x the RL, look at the absolute difference between the results.
Acceptable absolute difference: <RL
1.8 – 0.3 = 1.5: UNACCEPTABLE

Both results <5x the RL: soil/sediment samples

Analyte	RL (mg/kg)	Sample (mg/kg)	Field Dup (mg/kg)	RPD
Lead	2	3.9	2.1	60

When both results are <5x the RL, look at the absolute difference between the results.
Acceptable absolute difference: <2x the RL
3.9 – 2.1 = 1.8: ACCEPTABLE

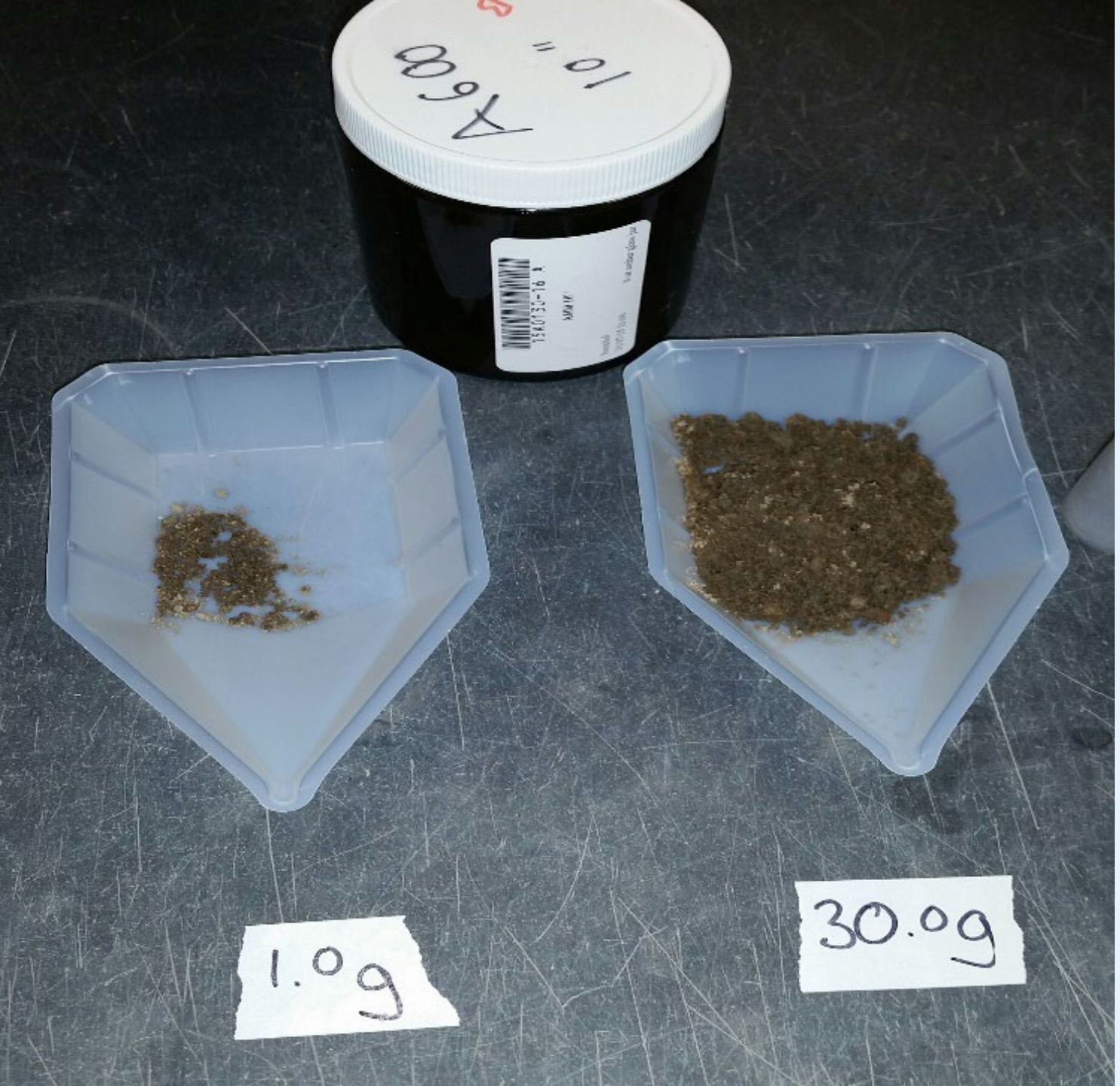
One detected result and one nondetect result

Analyte	RL (mg/kg)	Sample (mg/kg)	Field Dup (mg/kg)	RPD
Benzene	0.01	0.2	0.01 U	NC
Lead	2	3	2.0 U	NC

Benzene: absolute difference: 0.2 – 0.01 = 0.19 (>2xRL so UNACCEPATABLE)
Lead: 3 – 2 = 1 (<2x RL so ACCEPTABLE)

Field Duplicate Reporting Recommendations

Scenario	Reporting Recommendations
Both results nondetect	Use the lower of the two RLs
One result detected and one result not detected	Use the detected result
Both results detected and precision acceptable	Use the average or higher of the two results
Both results detected and precision not acceptable	Use the higher of the two results



Where is Variability Coming From?

- Sample Non-homogeneity
- Sampling Variability
- Laboratory Variability

Keep in Mind

Reminders

Laboratory Performance	Field Performance	Matrix Interferences
Method Blanks	Field Blanks	Surrogates
Lab Control Samples	Sample Preservation	Internal Standards*
Holding Times	Field Duplicates	Matrix Spikes
Calibrations*		Laboratory Duplicates
Tunes*		

*Not typically provided in Level 2 deliverables

Sample-Specific	Batch-Specific
Holding Times	Method Blanks
Sample Preservation	Lab Control Samples
Field Duplicates	Calibrations*
Surrogates	Tunes*
Internal Standards*	Field Blanks
Matrix Spikes	
Laboratory Duplicates	

*Not typically provided in Level 2 deliverables

Surrogates (Organic Analyses Only)

What are they?

Examples:
VOCs: Toluene-d8
SVOCs: Phenol-d5
PCBs: Decachlorobiphenyl

Surrogate Data in Your Data Package

Method: 8260C - Volatile Organic Compounds by GC/MS (Continued)

Analyte	Result	Qualifier	RL	MDL
Isopropylbenzene	ND		8.52	
Methyl acetate	ND		42.6	
Methyl tert-butyl ether	ND		8.52	
Methylcyclohexane	ND		8.52	
Methylene Chloride	ND		8.52	
Styrene	ND		8.52	
Tetrachloroethene	ND		8.52	
Toluene	ND		8.52	
trans-1,2-Dichloroethene	ND		8.52	
trans-1,3-Dichloropropene	ND		8.52	
Trichloroethene	ND		8.52	
Trichlorofluoromethane	ND		8.52	
Vinyl chloride	ND		8.52	
Xylenes, Total	ND		17.0	

Surrogate	*%Recovery*	*Qualifier*	*Limits*
Toluene-d8 (Surr)	*98*		*71 - 125*
1,2-Dichloroethane-d4 (Surr)	*106*		*64 - 126*
4-Bromofluorobenzene (Surr)	*95*		*72 - 126*
Dibromofluoromethane (Surr)	*107*		*60 - 140*

Actual %Rs

Acceptance limits

Method: 8270D - Semivolatile Organic Compounds (GC/MS) (Co

Analyte	Result	Qualifier	RL
Di-n-octyl phthalate	ND		1090
Dibenz(a,h)anthracene	ND		1090
Dibenzofuran	ND		1090
Diethyl phthalate	ND		1090
Dimethyl phthalate	ND		1090
Fluoranthene	305	J	1090
Fluorene	ND		1090
Hexachlorobenzene	ND		1090
Hexachlorobutadiene	ND		1090
Hexachlorocyclopentadiene	ND		1090
Hexachloroethane	ND		1090
Indeno[1,2,3-cd]pyrene	175	J	1090
Isophorone	ND		1090
N-Nitrosodi-n-propylamine	ND		1090
N-Nitrosodiphenylamine	ND		1090
Naphthalene	ND		1090
Nitrobenzene	ND		1090
Pentachlorophenol	ND		2120
Phenanthrene	ND		1090
Phenol	ND		1090
Pyrene	278	J	1090

Surrogate	*%Recovery*	*Qualifier*	*Limits*	
2,4,6-Tribromophenol	*105*		*54 - 120*	Acid
2-Fluorobiphenyl	*94*	B/N	*60 - 120*	
2-Fluorophenol	*92*		*52 - 120*	Acid
Nitrobenzene-d5	*75*	B/N	*53 - 120*	
p-Terphenyl-d14	*106*	B/N	*79 - 130*	
Phenol-d5	*88*		*54 - 120*	Acid

If Surrogate Recoveries are Outside Criteria

- Lab typically is required to re-prep and/or re-analyze the sample.
- Not uncommon to have two sets of data for one sample.

Biases Due to Surrogate Recoveries

- Were all recoveries within the acceptance limits?
- **If surrogate recoveries are outside limits:**
  - POTENTIAL LOW BIAS or
  - POTENTIAL HIGH BIAS
- **Sample-specific**
- **Effect on results dependent on method**

Method	Effect on Data
VOCs	If 1 surrogate out: affects all target VOCs
SVOCs	If ≥ 2 B/N surrogates out but ≥10%: affects all B/N compounds If ≥ 2 acid surrogates out but ≥10%: affects all acid compounds If 1 B/N or 1 acid surrogate <10%: affects associated compounds
Pesticides & PCBs	Affects all target pesticides or PCBs: dependent on which column surrogate is outside limits

- **Potentially unusable results: if recoveries are <10%**

Serial_No:06261413:14

Project Name: [redacted]

Project Number: 192019.PHASE10

Lab Number: L1412769

Report Date: 06/26/14

SAMPLE RESULTS

Lab ID: L1412769-03

Client ID: PEX-11

Sample Location: [redacted]

Date Collected: 06/11/14 11:45

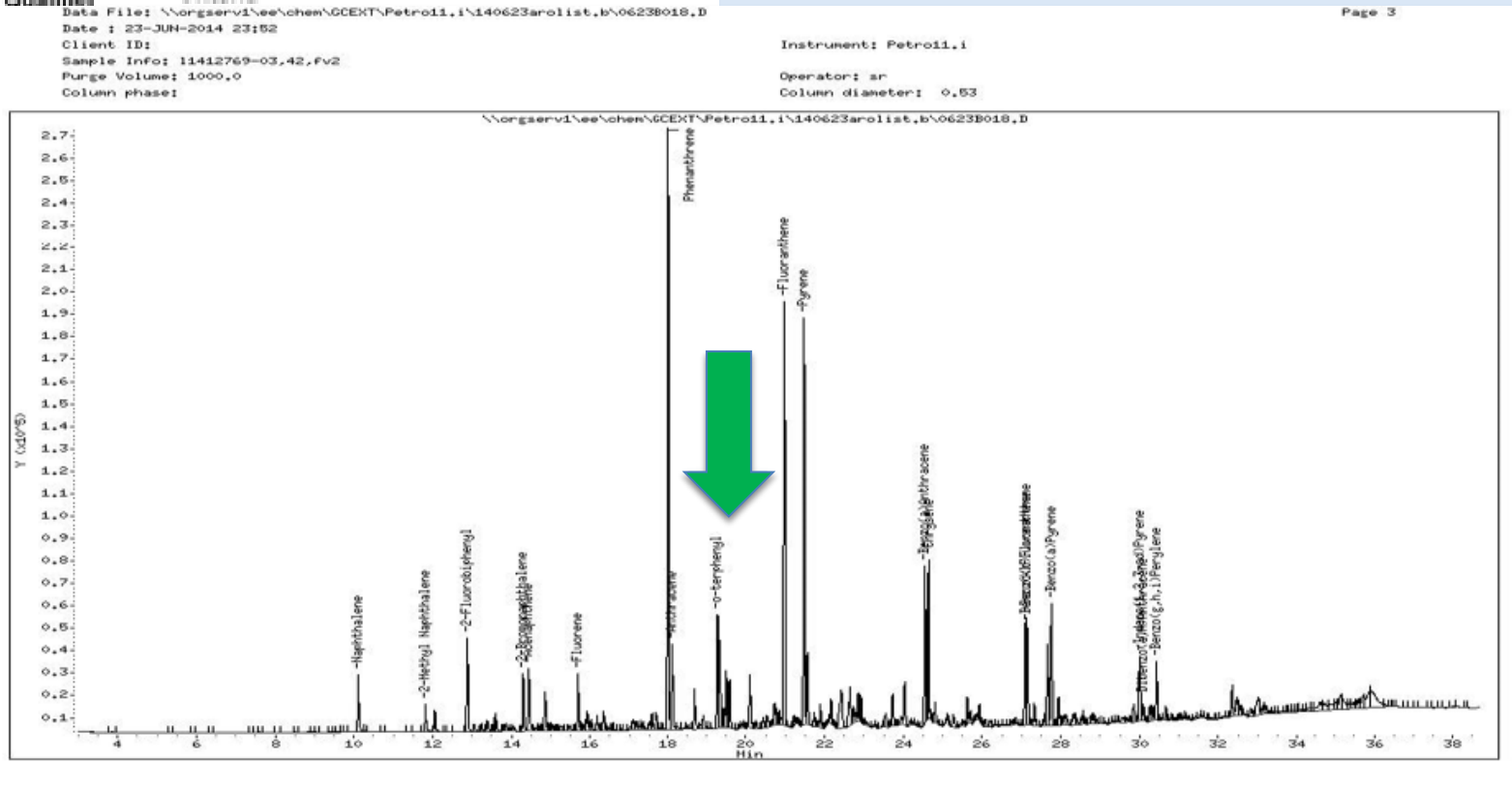
Date Received: 06/12/14

Field Prep: Not Specified

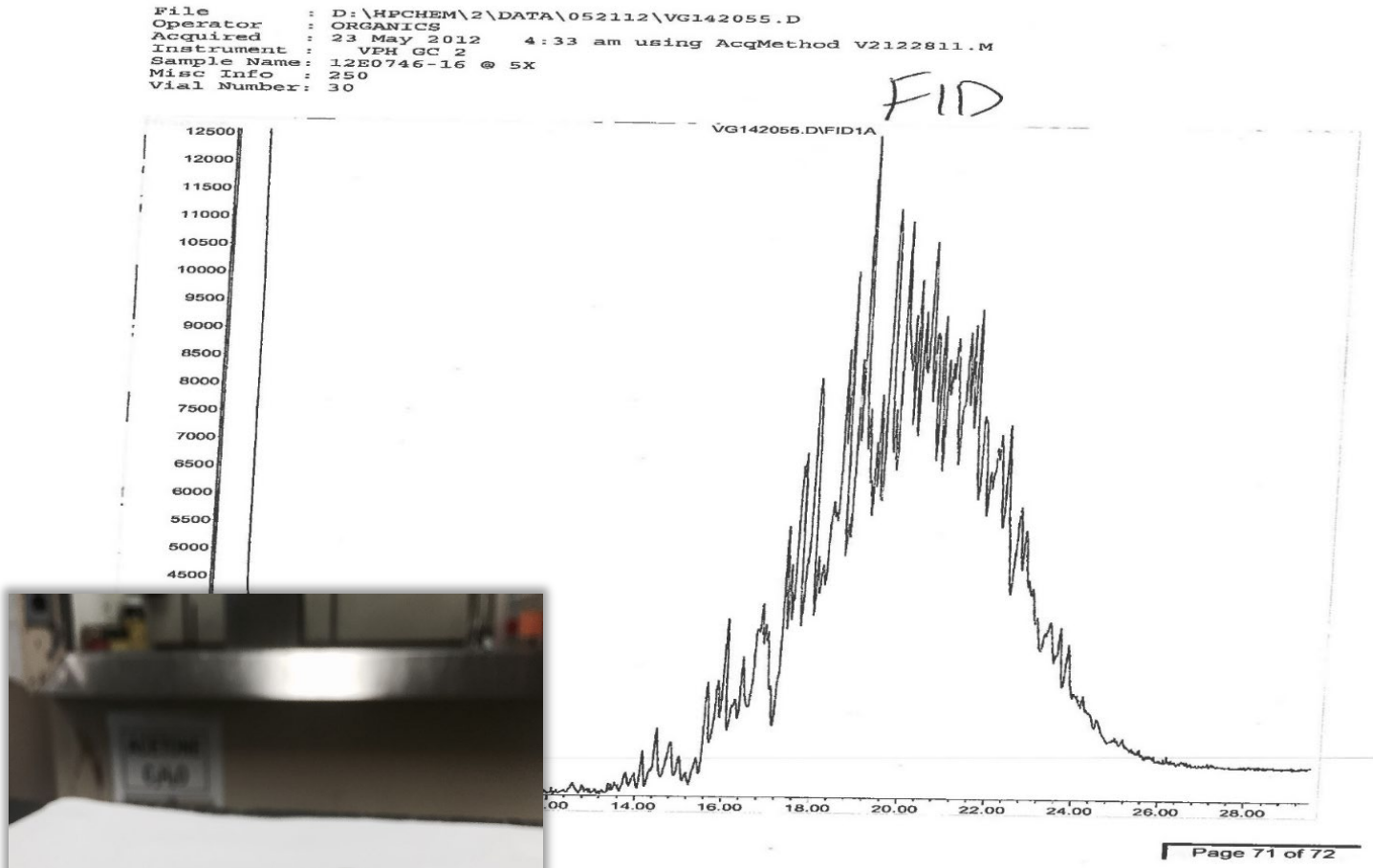
Parameter	Result	Qualifier	Units	RL	MDL	Dilution Factor

Extractable Petroleum Hydrocarbons - Westborough Lab

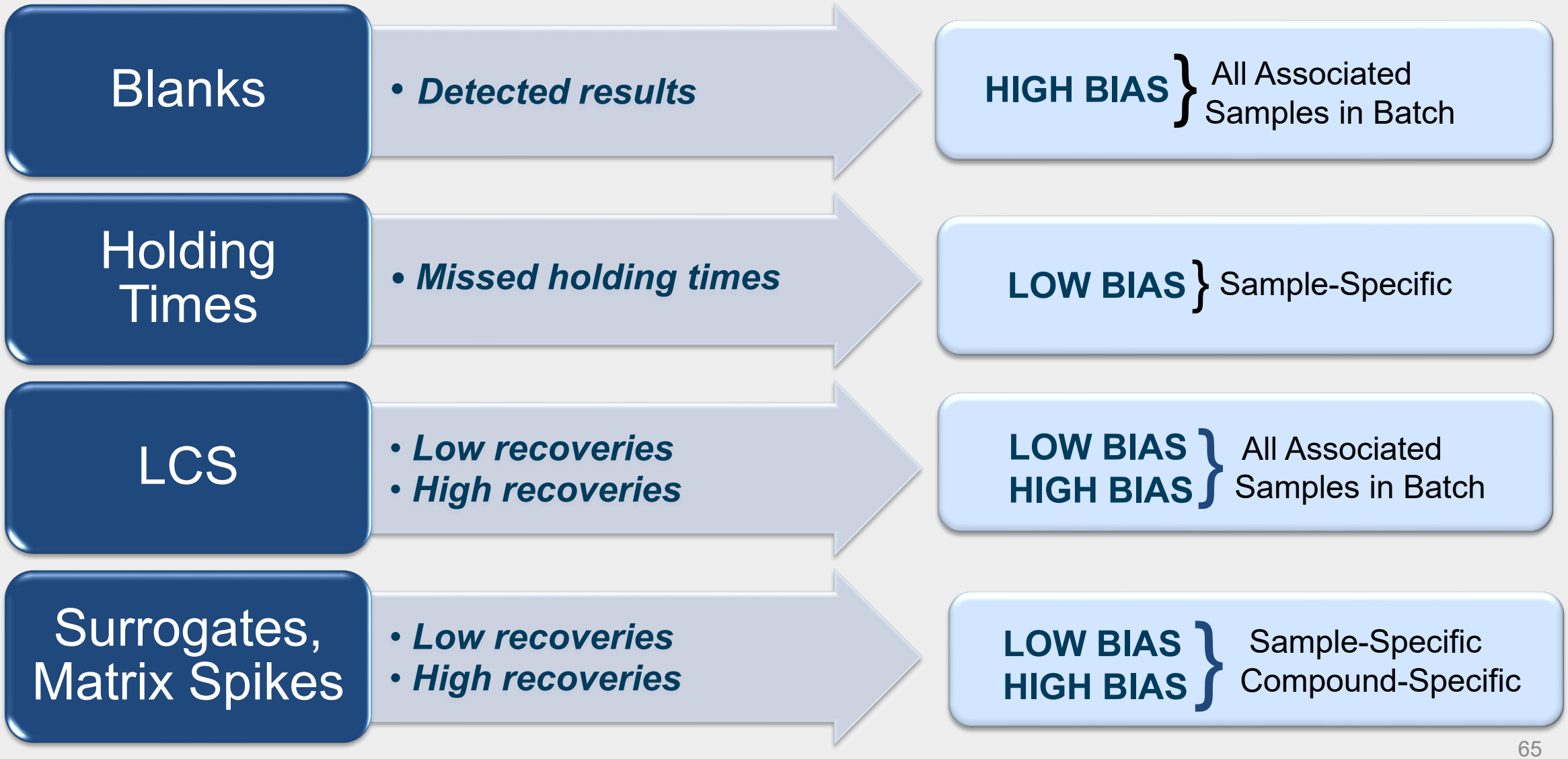
Surrogate	% Recovery	Qualifier	Acceptance Criteria
Chloro-Octadecane	70		
o-Terphenyl	191		
2-Fluorobiphenyl	81		
2-Bromonaphthalene	76		



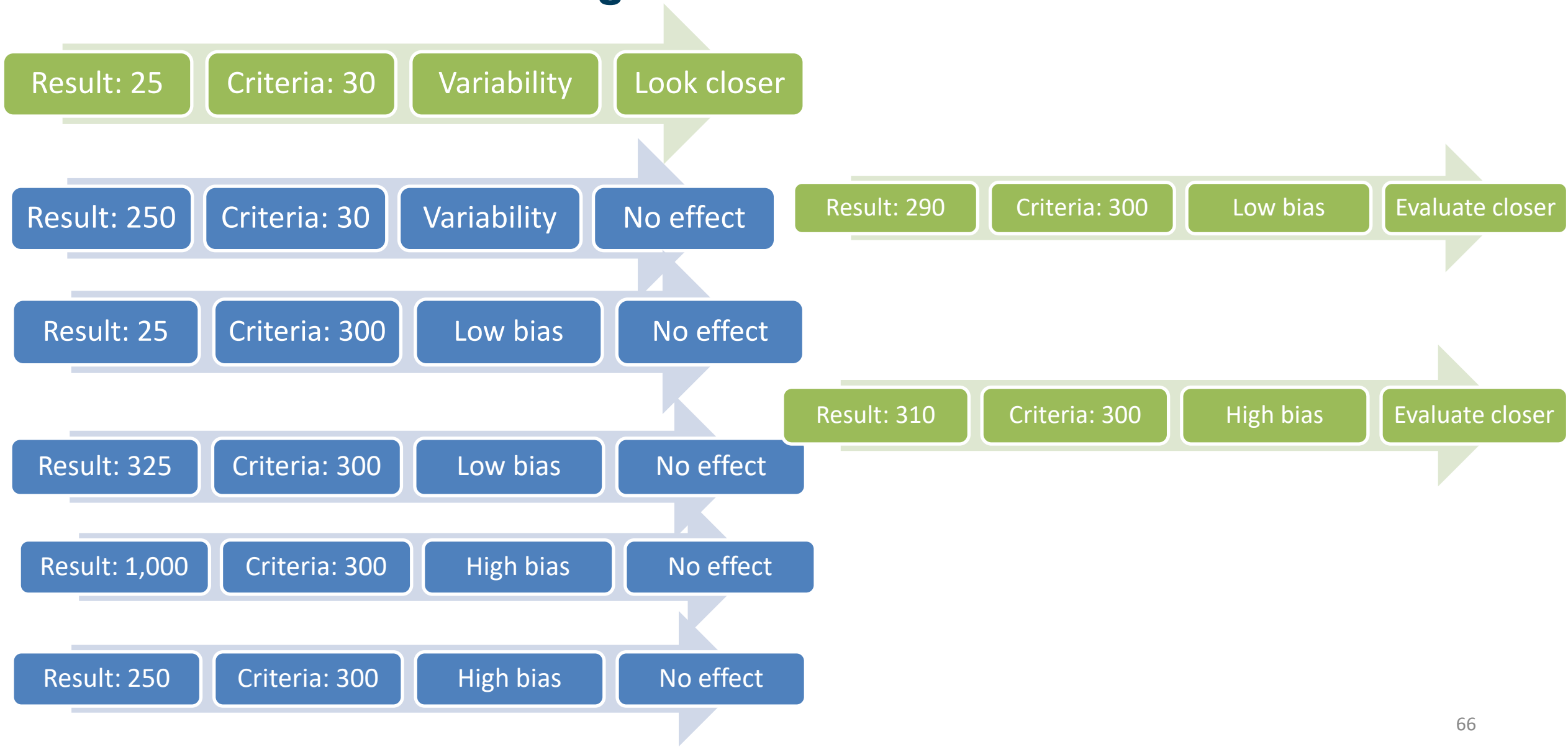
Matrix Interference



Let's Summarize Potential Biases

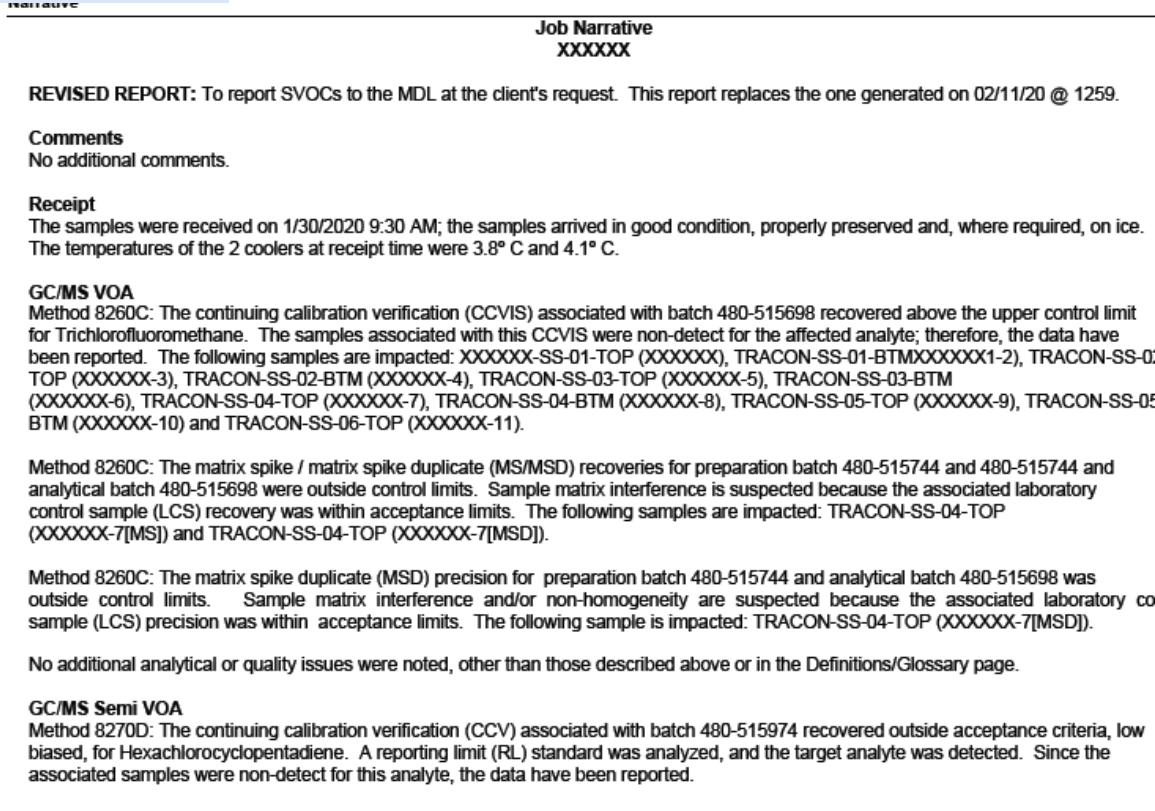


If There is Bias or Uncertainty in Result, How Does This Affect Decision-making?



What Else?

- Does the case narrative note any other anomalies with the data?
- Do any results look questionable?
- Has the EDD been compared with the lab report?



Job Narrative
XXXXXX

REVISED REPORT: To report SVOCs to the MDL at the client's request. This report replaces the one generated on 02/11/20 @ 1259.

Comments
No additional comments.

Receipt
The samples were received on 1/30/2020 9:30 AM; the samples arrived in good condition, properly preserved and, where required, on ice. The temperatures of the 2 coolers at receipt time were 3.8° C and 4.1° C.

GC/MS VOA
Method 8260C: The continuing calibration verification (CCVIS) associated with batch 480-515698 recovered above the upper control limit for Trichlorofluoromethane. The samples associated with this CCVIS were non-detect for the affected analyte; therefore, the data have been reported. The following samples are impacted: XXXXXX-SS-01-TOP (XXXXXX), TRACON-SS-01-BTMXXXXXX1-2), TRACON-SS-0
TOP (XXXXXX-3), TRACON-SS-02-BTM (XXXXXX-4), TRACON-SS-03-TOP (XXXXXX-5), TRACON-SS-03-BTM
(XXXXXX-6), TRACON-SS-04-TOP (XXXXXX-7), TRACON-SS-04-BTM (XXXXXX-8), TRACON-SS-05-TOP (XXXXXX-9), TRACON-SS-05
BTM (XXXXXX-10) and TRACON-SS-06-TOP (XXXXXX-11).

Method 8260C: The matrix spike / matrix spike duplicate (MS/MSD) recoveries for preparation batch 480-515744 and 480-515744 and analytical batch 480-515698 were outside control limits. Sample matrix interference is suspected because the associated laboratory control sample (LCS) recovery was within acceptance limits. The following samples are impacted: TRACON-SS-04-TOP (XXXXXX-7[MS]) and TRACON-SS-04-TOP (XXXXXX-7[MSD]).

Method 8260C: The matrix spike duplicate (MSD) precision for preparation batch 480-515744 and analytical batch 480-515698 was outside control limits. Sample matrix interference and/or non-homogeneity are suspected because the associated laboratory co sample (LCS) precision was within acceptance limits. The following sample is impacted: TRACON-SS-04-TOP (XXXXXX-7[MSD]).

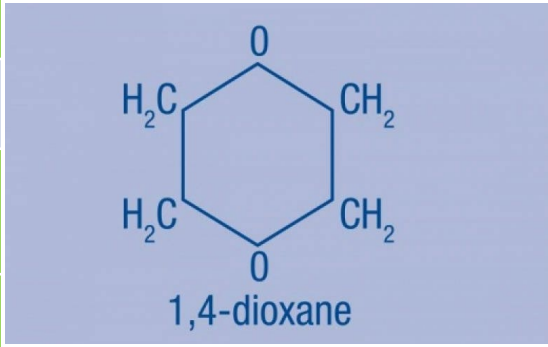
No additional analytical or quality issues were noted, other than those described above or in the Definitions/Glossary page.

GC/MS Semi VOA
Method 8270D: The continuing calibration verification (CCV) associated with batch 480-515974 recovered outside acceptance criteria, low biased, for Hexachlorocyclopentadiene. A reporting limit (RL) standard was analyzed, and the target analyte was detected. Since the associated samples were non-detect for this analyte, the data have been reported.

Some Analyte-Specific Issues

1,4-Dioxane

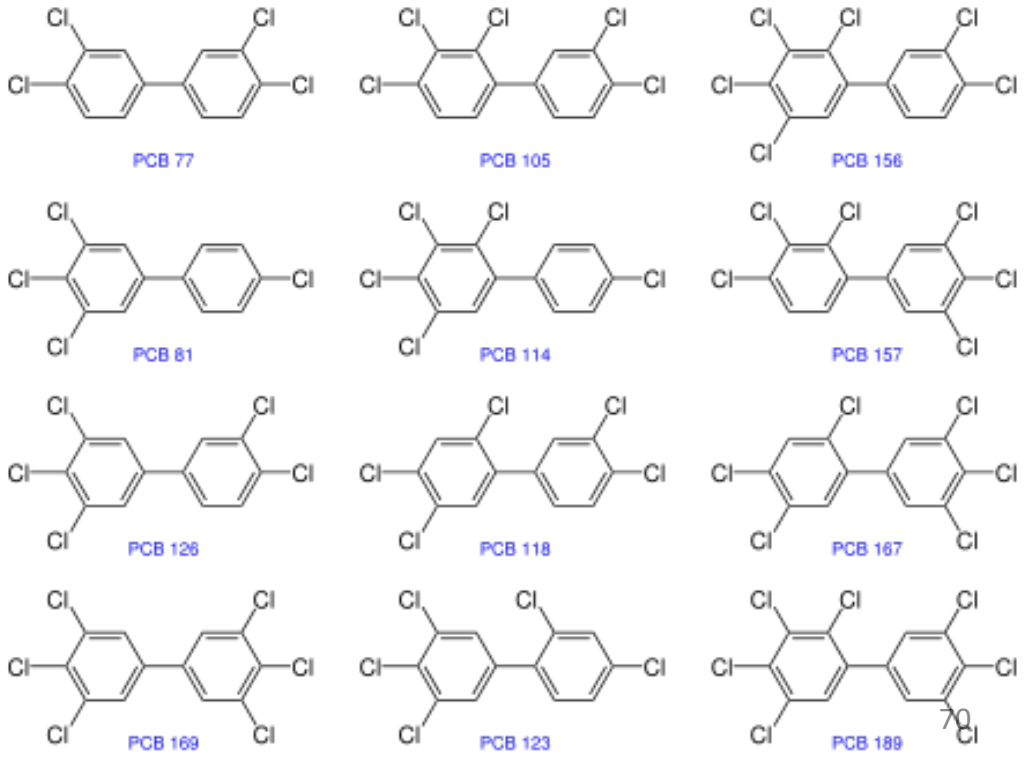
Method	Technique	RLs	Comments
8260 (VOC): Aqueous	Ambient purge & trap (P&T) with full scan GC/MS	200-500 µg/L	1,4-dioxane-d8 Internal Standard (IS)
	Heated P&T with SIM GC/MS	2-5 µg/L	1,4-dioxane-d8 IS Prone to interferences
8270 (SVOC): Aqueous	Full scan GC/MS	5-10 µg/L	Poor extraction efficiency
	Isotope dilution with SIM GC/MS	0.15-0.4 µg/L	1,4-dioxane-d8 IS Improved precision & accuracy
8260 (VOC): Solid	Ambient P&T with full scan GC/MS	0.2-0.5 mg/kg	1,4-dioxane-d8 IS
	Heated P&T with SIM GC/MS	0.002-0.005 mg/kg	1,4-dioxane-d8 IS Not routinely needed
8270 (SVOC): Solid	Full scan GC/MS	0.05-0.2 mg/kg	Poor extraction efficiency
	Isotope dilution with SIM GC/MS	0.05-0.2 mg/kg	1,4-dioxane-d8 IS Improved precision & accuracy
EPA 522: Drinking water	Solid Phase Extraction (SPE) with SIM GC/MS	0.05-0.1 µg/L	



1,4-dioxane

Polychlorinated Biphenyls (PCBs)

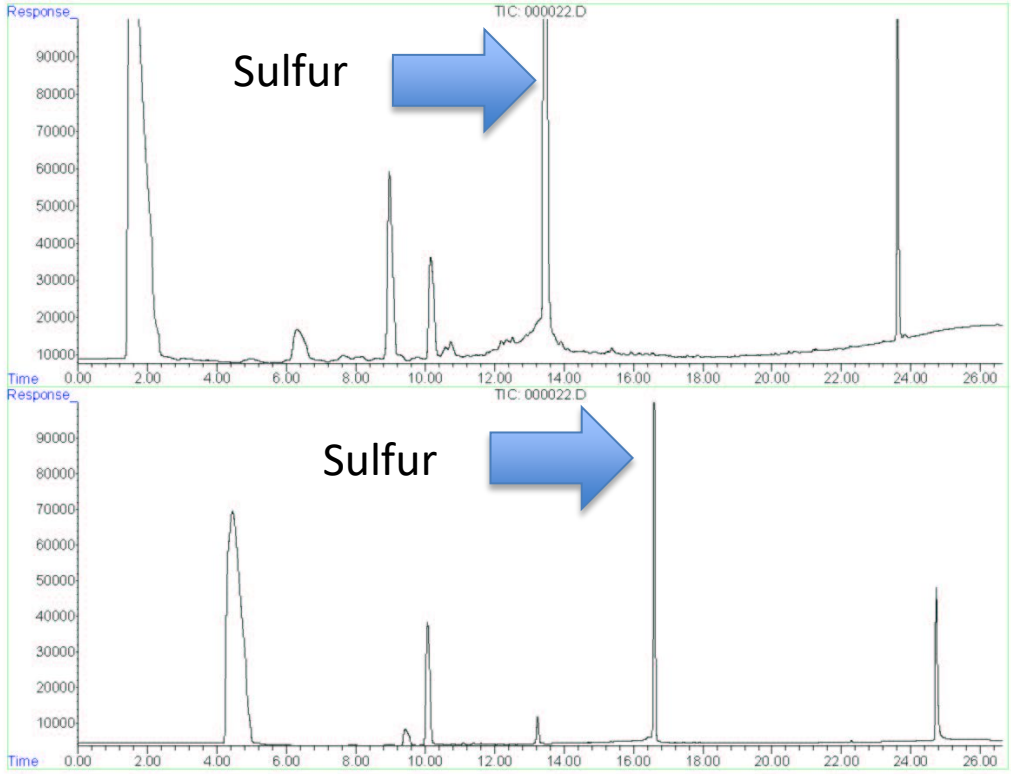
- Labs should always perform acid cleanup on all samples and sulfur cleanup on sediment samples.
- PCB Aroclors or PCB Congeners or PCB Homologues??
- Nondetect PCB Aroclor results do not necessarily mean no PCBs present in sample!
  - Weathered Aroclors
  - Results close to RL
- Dual Column Analyses:
  - Higher result reported (8000B)
  - Lower result reported (8000D)
- Holding Time: One year to extraction



Sulfur Cleanup Before and After: Affects Pesticides & PCBs

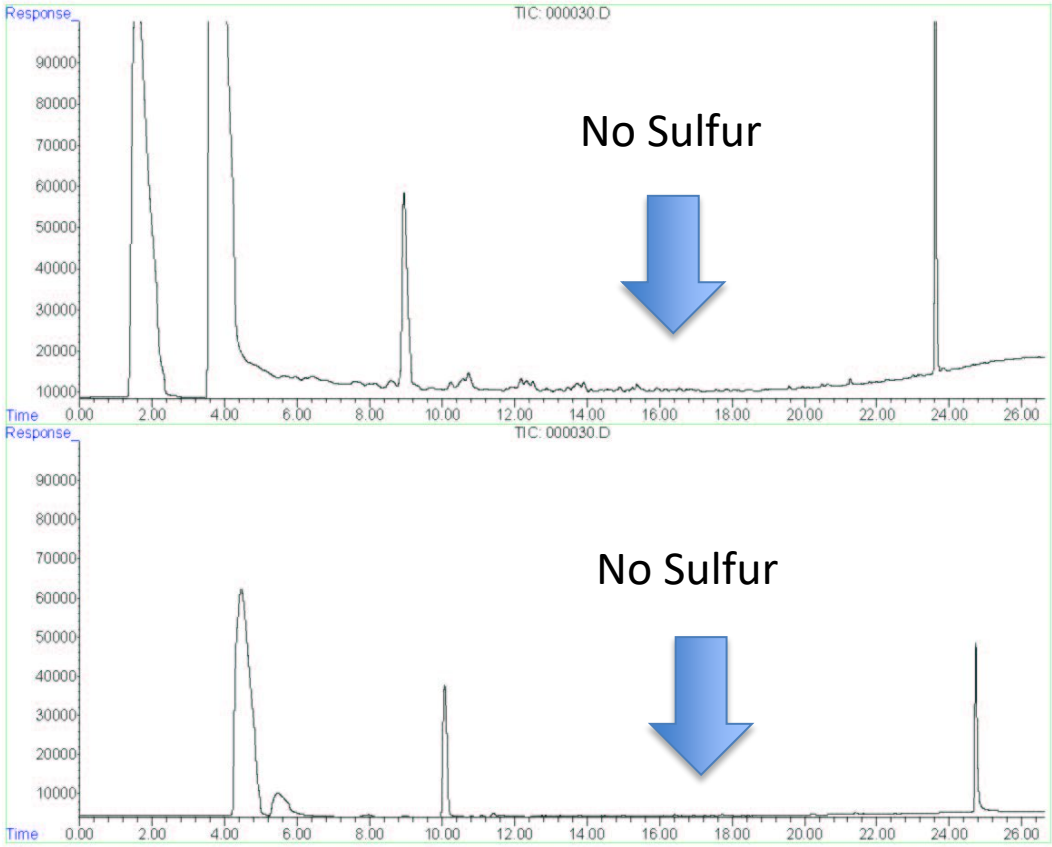
Before Sulfur Cleanup

```
File       :C:\Documents and Settings\Labtest\Desktop\pcb data\000022.D
Operator   : JB
Acquired   : 11 Jan 2006  12:36 pm using AcqMethod PCBRUN.M
Instrument :    DUAL ECD2
Sample Name: 00491
Misc Info  :
Vial Number: 11
```



After Sulfur Cleanup

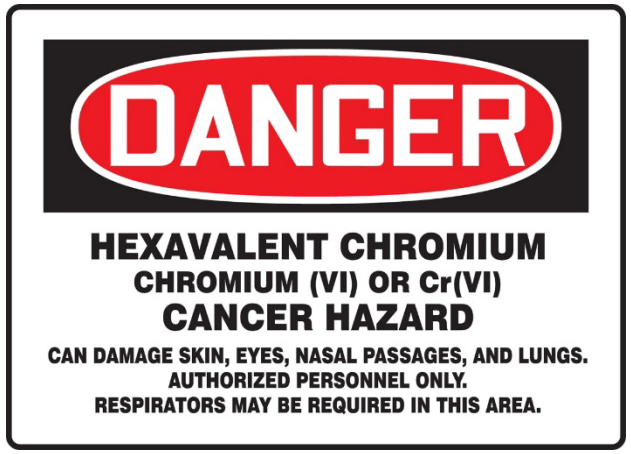
```
File       :C:\Documents and Settings\Labtest\Desktop\pcb data\000030.D
Operator   : JB
Acquired   : 11 Jan 2006   2:55 pm using AcqMethod PCBRUN.M
Instrument :    DUAL ECD2
Sample Name: 491 tba
Misc Info  :
Vial Number: 15
```

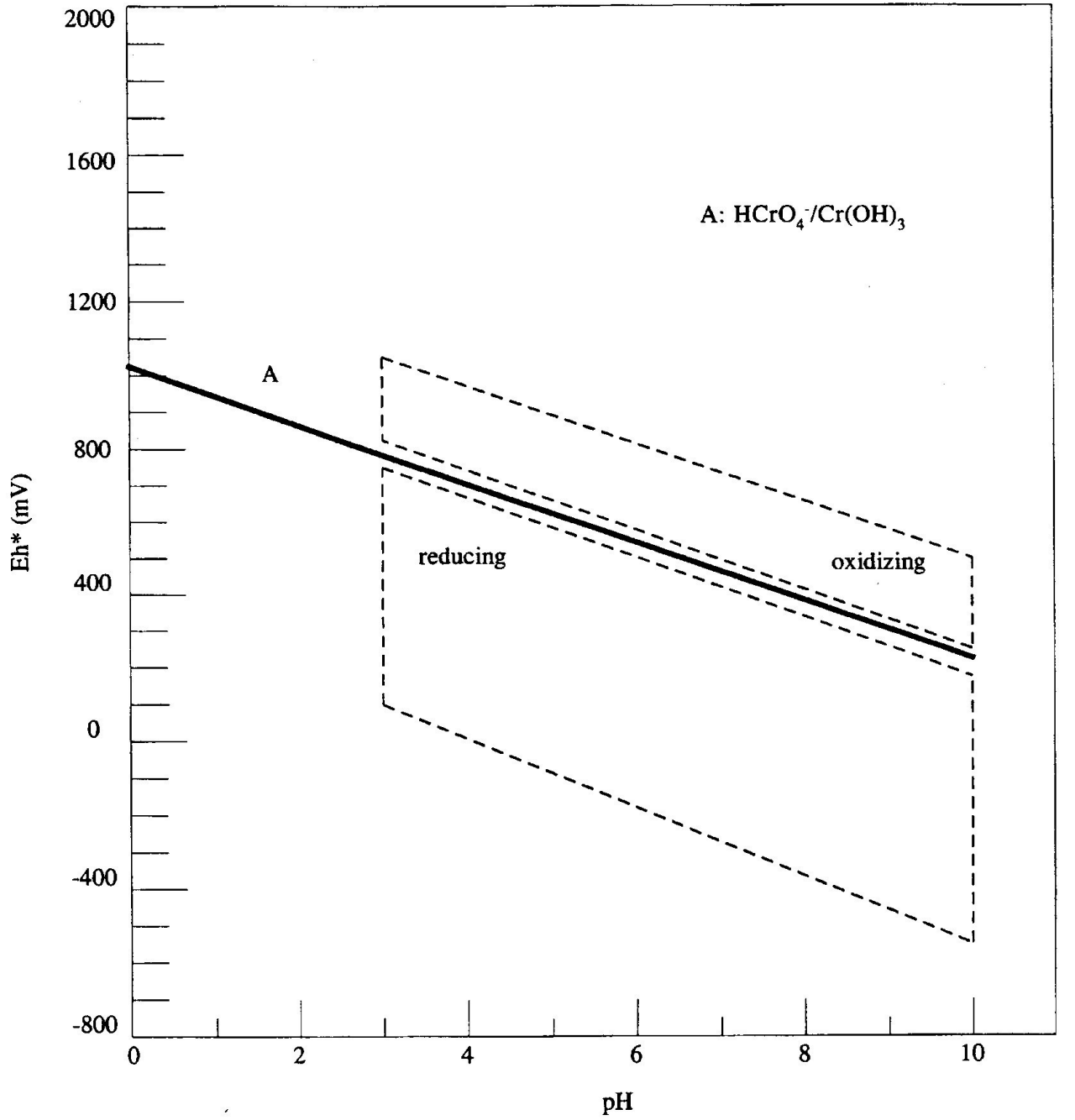


Hexavalent Chromium

- Make sure lab reports “analysis time” for aqueous samples so 24-hour hold time can be verified.
- Soil/sediment samples should also be submitted for pH and ORP analyses.

pH and oxidation-reduction potential (ORP) allow data user to determine if reducing environment exists



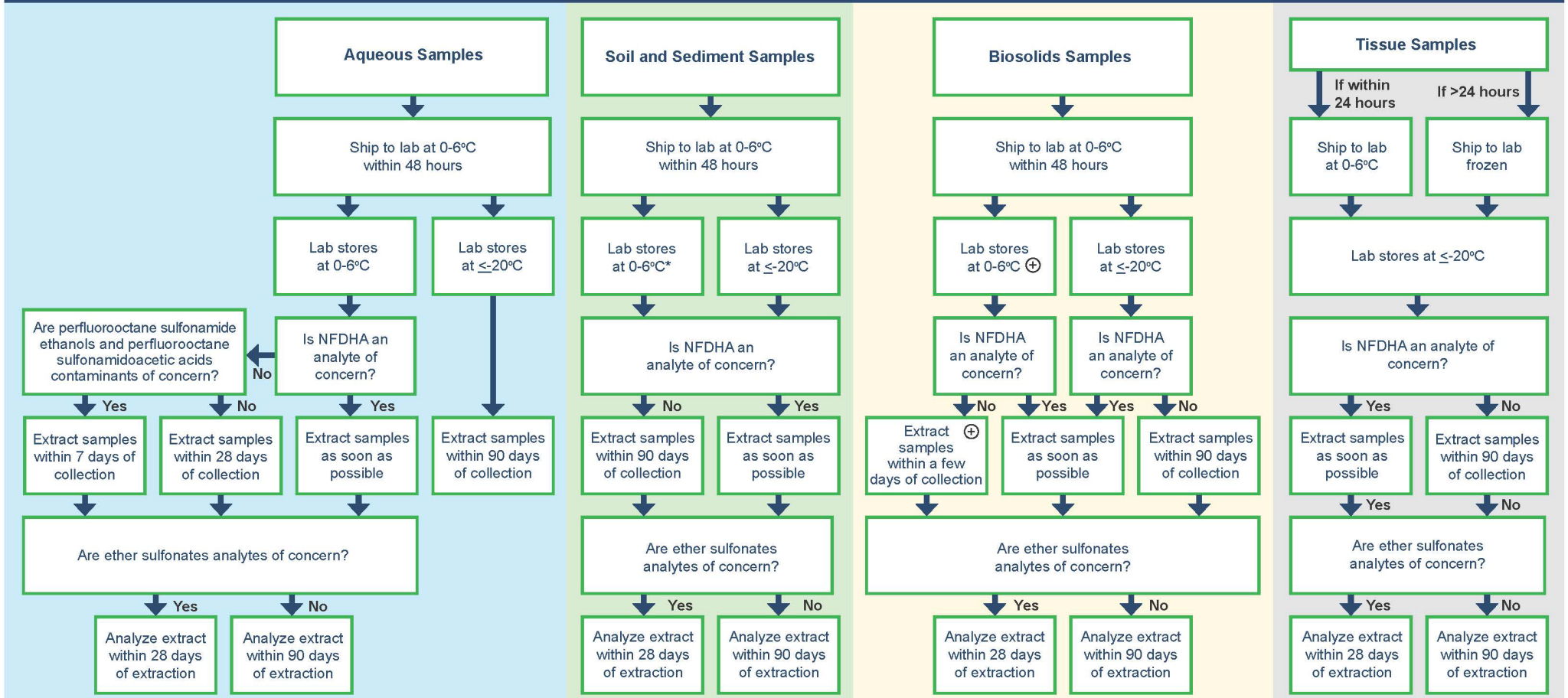


* Note the Eh values plotted on this diagram are corrected for the reference electrode voltage: 244 mV units must be added to the measured value when a separate calomel electrode is used, or 199 mV units must be added if a combination platinum electrode is used.

Thallium

- Iron interference on thallium may not be adequately corrected in all matrices by ICP interelement correction factors.
  - False positives for thallium using ICP-AES (EPA 6010)
  - Confirm by an alternate method, such as ICP-MS (EPA 6020)

PFAS Holding Times Using EPA 1633



* Some soil/sediments may exhibit microbial growth when stored at 0-6°C

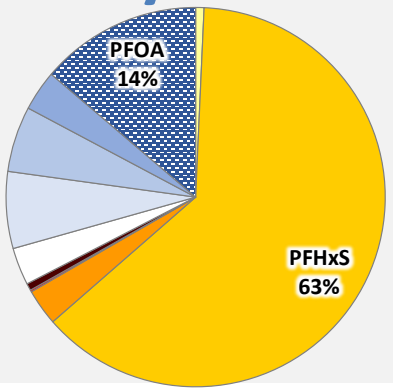
⊕ Biosolids samples at 0-6°C may exhibit microbiological activity that can lead to production of gases which may cause sample to be expelled from container when opened as well as producing noxious gases.

PFAS

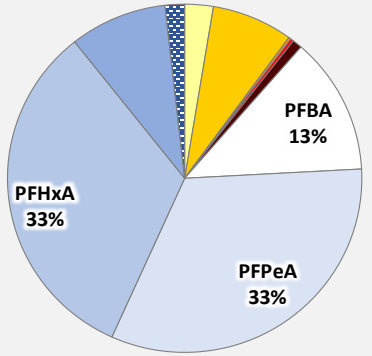
PFAS: Aqueous Samples with Suspended Solids: Lab Procedural Solutions vs Project Objectives

"Total" PFAS measurement of aqueous samples; may be acceptable or needed in certain instances depending on project objectives

Turbidity 1000 NTU



Turbidity 25 NTU



Total:

- Lab centrifuges sample to separate aqueous and particulate phases.
- Lab extracts aqueous phase.
- Lab extracts remaining particulate phase and combines extract with aqueous phase extract.

Dissolved:

- Lab centrifuges sample to separate aqueous and particulate phases.
- Lab extracts aqueous phase only.

When Should We Be Requesting "Total" versus "Dissolved"?

Reminder: We Need to Evaluate the Lab's Data!

- Data may be used to make costly decisions
- Data may have potential to impact human health
- Need to confirm quality data available and appropriate to support decisions
- Need to determine potential low or high biases, potential uncertainties, potential false positive or false negative results

Even if the lab follows all method-required procedures, there can still be data quality/usability issues.

Questions?

Elizabeth Denly, ASQ CMQ/OE

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