# INCREMENTAL SAMPLING

Review of PAH Surrogate Study & ISM Implementation

*William Corl Ph.D.*
*Chemist & Deputy Director, NAVSEA LQAO*
*William.e.corl.civ@us.navy.mil*



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## INCREMENTAL SAMPLING

- Brief Introduction
- EDQW Historical perspective on ISM in ELAP
- Introduction on EDQW ISM PAH Project- Purpose & Objectives
- Review of Results and Statistical Evaluation
- Conclusions and ISM PAH QSM Table Requirements for Version 6.0
- Path forward and Further EDQW Recommendations



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## What do we really know about ISM ?

The first known use of *ISM* was in 1680

- March 30 – A total eclipse of the Sun
- July 8 – The first documented tornado in America kills a servant at Cambridge, Massachusetts.
- October 9 – A massive 9.0 magnitude earthquake destroys part of Málaga



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## What do we really know about ISM ?

"ISM" (ironically) are the behavioral manifestation of bias, conscious or unconscious, that reinforce oppression and inequities in our culture. A distinctive doctrine, cause, or theory mostly linked to an oppressive and especially discriminatory attitude or belief …..Merriam-Webster

Examples: Alcoholism, criticism, plagiarism



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## Incremental sampling

"*We all have got to come to grips with our isms"*

Joycelyn Elders.



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## Incremental sampling

*"Incremental sampling is the physical realization of the Central Limit Theorem. It "normalizes" the effects of spatial and compositional heterogeneity through superior field coverage and aggressive laboratory processing."*

*ISM-ism number 1*



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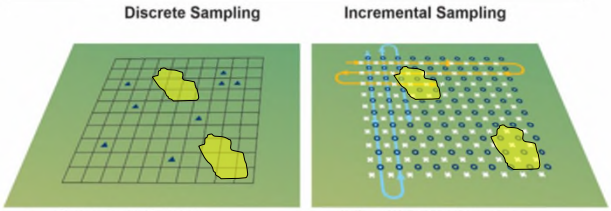
## ISM Overview- Example 1




- The dicrete samples have missed representing the areas with the higher concentrations
- ISM has captured the affects of the higher areas. But we dont know “hot” and we dont know “spot”

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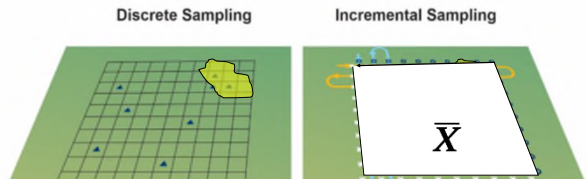
## ISM Overview- Example 2




- The dicrete and ISM sampling designs have both captured the area with the higher concentrations
- Discrete samples give us spatial concentration behavior. But they are only point estimates.
- “Hot-spots” do exist, but are not relevant when using ISM

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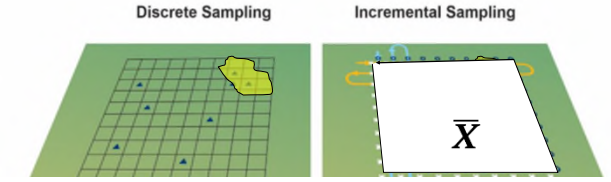
## ISM Overview- Example 2




- Nature and extent determinations using ISM can be difficult
- May require smaller and numerous Decision Units (DU’s)
- CSM plays a huge role in selection of the best sampling design
- Be careful about exit strategies



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## Incremental sampling

*“CSM and DQO’s are even more critical with ISM sample designs because you only get one data point of a larger area.”*

*ISM-ism number 2*



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## Laboratory Processing

ISM has only been fully validated for explosives (8330b).

- Positive and Negative Bias
- Some Method Performance Criteria for EPA methods were NOT calculated considering ISM Processing
- Is drying and milling always required?
- Surrogates!



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## Quality Control

What is the purpose of adding surrogates to samples?

- “.. Therefore, it is CRITICAL that any compounds added to a sample, including the surrogates, are added to the sample aliquot PRIOR TO any additional processing steps. The recovery of the surrogate standard is used to monitor for unusual matrix effects, *gross sample processing errors*, etc.” **(EPA 3500C - 4 Revision 3 Organic Extraction and Sample Preparation, February 2007)**



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## Quality Control

What is the purpose of adding surrogates to samples?

- … in order to evaluate the effects of matrix *on overall method performance*….surrogates should be subjected to the same analytical procedures (Sec. 11.0) as those used on field samples. **(SW-846 Update VI 8270E - 1 Revision 6 June 2018)**

Surrogates are added to each sample for organic methods, to evaluate potential bias caused by the matrix and/or **sample preparation**.



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## Recall-

- Table B-23. Incremental Sampling Methodology (ISM) Soil Preparation for Large Volume (1 kg or greater) Samples.
- Removed from QSM- "Surrogates must be spiked prior to any preparation steps performed such as drying, grinding, sieving, or extraction.
- EDQW completed PAH surrogate efficacy study in 2020



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## EDQW PAH ISM Surrogate Project

- Solid fortification standard can be used
- Sample drying/milling does affect recoveries
- Additional Study is needed
- SERDP/ESTCP ER Program tentatively agreed to funding additional validation study
- Project Management: NAVFAC EXWC
- 5 ELAP Accredited Laboratories Participating
- ER22-7947 New Start Project Overview 2022



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## ISM PAH Surrogates

- ACENAPHTHYLENE-D8 (B.P. 280 °C)
- BENZ (A) ANTHRACENE-D12 (B.P. 438 °C)
- BENZO (A) PYRENE-D12 (B.P. 495 °C)
- FLUORENE-D10 (B.P. 298 °C)



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## Phase1- Design and Objectives

- Bias Evaluation- Pre-fortified surrogates in blank soil, 10 replicates for each treatment
  - Treatment 1, No drying or milling
  - Treatment 2, 5x1 min milling, no drying
  - Treatment 3, 16-24 hr air drying, no milling
  - Treatment 4, drying and milling
  - ANOVA between laboratories and within laboratories for treatments
  - Surrogate recovery criteria (QSM V6.0)
  - Milling Blanks (evaluation of potential carry-over)



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## Phase 2- Design and Objectives

- Evaluation of ISM replicates. Laboratories add solid surrogate standards to soil. No drying or milling
  - Treatment 1, 30 increments
  - Treatment 2, 50 increments
  - ANOVA between laboratories and within laboratories for treatments
  - Difference between 3, 5, and 10 replicates
  - RSD Criteria for Subsampling replicates (QSM V6.0)
  - Any difference between the recoveries of pre-fortified soil from provider and the laboratories fortifying the soil?



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## Statistical Evaluations

- NAVFAC EXWC
  - –Dr. Anthony Danko, (Tony) NFEXWC
  - –Dr. Nicolette E Andrzejczyk (Nikki) NFEXWC



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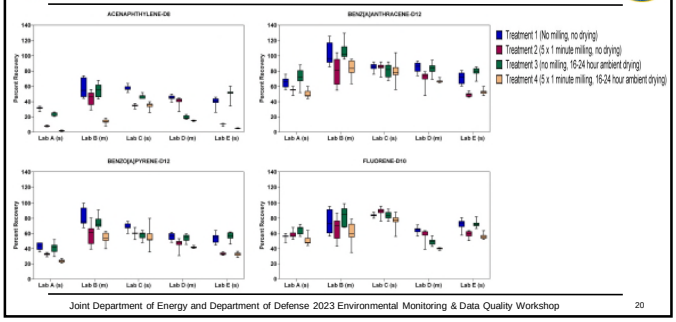
## Phase1 Inter-Laboratory Comparison



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## Phase1 Inter-Laboratory Comparison

- In almost all instances, there were significant differences in percent recovery among treatment groups within a given laboratory (ANOVA)
  - –Only no significant difference in recovery in benz[a]anthracene for Lab C
- Treatment 4 (5 x 1 min milling, 16-24 hr drying) tended to have lowest analyte recovery
- Treatments 1 (no milling/drying) and 3 (no milling, 16-24 hr drying) tended to have highest analyte recovery



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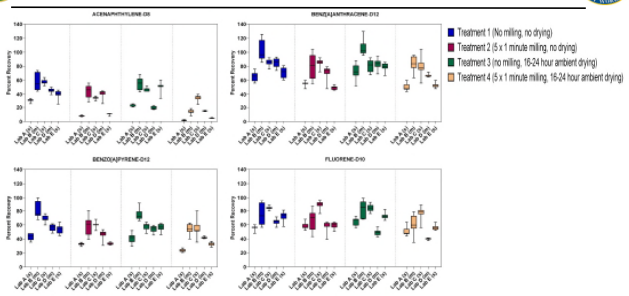
## Phase1 Intra-Laboratory Comparison



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## Phase1 Intra-Laboratory Comparison

- There were significant differences in percent recovery in the same treatment group across all laboratories (ANOVA)
- No clear trend in better analyte recovery among labs using sonication vs microwave



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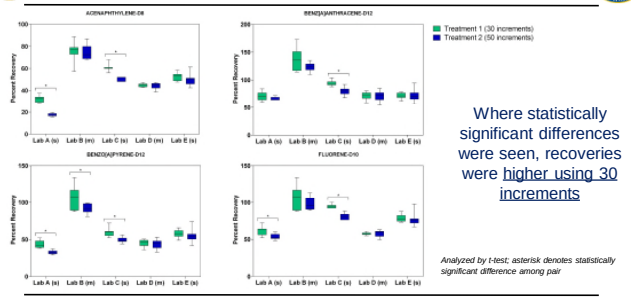
## Phase2: 30 vs 50 Increments




Where statistically significant differences were seen, recoveries were higher using 30 increments

*Analyzed by t-test; asterisk denotes statistically significant difference among pair*



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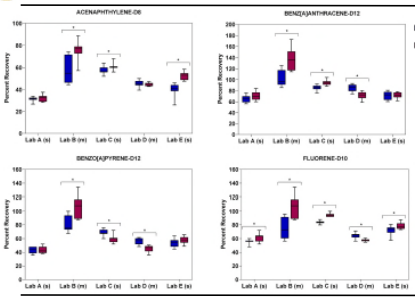
## Phase 1 Treatment 1 vs Phase 2 Treatment 1




Phase 1 the soils came pre-fortified from the vendor and in Phase 2 the labs fortified the blank soils

*Analyzed by t-test; asterisk denotes statistically significant difference among pair*



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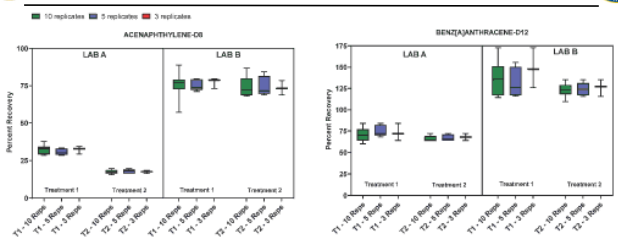
## Phase2: Replicate Comparison




**No significant differences among replicate size**

*Analyzed by ANOVA with Sidak's multiple comparison test*



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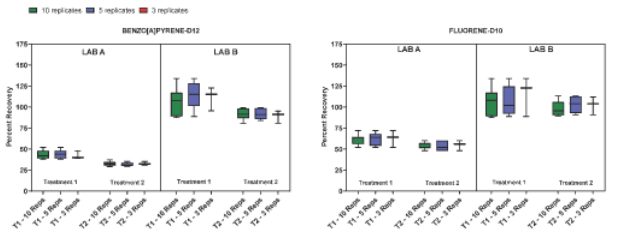
## Phase2: Replicate Comparison




*Analyzed by ANOVA with Sidak's multiple comparison test*



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## Incremental Sampling

ISM PAH Surrogate Study

Conclusions and Path Forward

*William Corl Ph.D.*

*Chemist & Deputy Director*

*NAVSEA LQAO*



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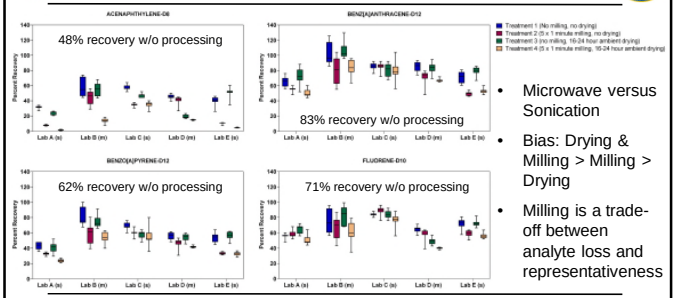
## Phase1 Inter-Laboratory Comparison




- Microwave versus Sonication
- Bias: Drying & Milling > Milling > Drying
- Milling is a trade-off between analyte loss and representativeness



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## ISM PAH Surrogates-Conclusions

- Procedural differences between laboratories likely play a role in precision and bias- evaluation of the process
- Recoveries were statistically higher with 30 increments than with 50 increments. (takes longer to sample?)
- % moisture and correction for surrogate calculations- (applicability of splitting and homogeneity)



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## ISM PAH Surrogates-Conclusions

- Statistical comparison between lab fortified and vendor fortified soils showed statistically significant differences. Lab fortified soils generally had higher recoveries. No appreciable differences in subsampling precision.
- 3 replicates for the determination of subsampling replicates
- There was no detection of carry-over contamination in the grinding blanks



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## Setting Recovery and Precision Criteria

EPA United States Environmental Protection Agency

Office of Water

www.epa.gov

**Protocol for Review and Validation of New Methods for Regulated Organic and Inorganic Analytes in Wastewater Under EPA's Alternate Test Procedure Program**

**February 2018**



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## Phase1 Results

| ACENAPHTHYLENE-D8 | BENZO (A) ANTHRACENE-D12 | BENZO (A) PYRENE-D12 | FLUORENE-D10 |
|---|---|---|---|
| QC Recovery Acceptance | QC Recovery Acceptance | QC Recovery Acceptance | QC Recovery Acceptance |
| 10 - 65.4 | 35.79 - 97.22 | 14.21 - 68.77 | 29.26 - 84.54 |
| 10 - 49.97 | 19.74 - 113.24 | 10 - 82.02 | 16.20 - 97.60 |
| Pooled RSD = 0.85 | Pooled RSD = 0.23 | Pooled RSD = 0.33 | Pooled RSD = 0.25 |
| Max RSD Criteria = 0.41 | Max RSD Criteria = 0.30 | Max RSD Criteria = 0.38 | Max RSD Criteria = 0.34 |

Criteria based upon EPA Protocol for Samples Processed with Drying and Grinding

Mean +/- 3 σ

10% Recovery is considered *de minimis* level, 120% Recovery upper limit will be applied



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## Draft Table B-30 QSM Requirements

**Table B-30. PAH in Soil (Sample Processing Requirements for Incremental Sampling Methodology)**

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action and Qualification Criteria |
|---|---|---|---|
| **Grinding** | Each field sample. | The entire field sample shall be ground. Drying is typically performed in order to aid in appropriate grinding and is therefore optional for dry soils. The need for grinding is a project decision and is therefore optional.<br><br>Splitting of the field sample is only allowed to determine the percent moisture for surrogate spikes. The field sample shall be well mixed. The split sample shall not exceed 10 g or 1% whichever is greater. | The laboratory shall indicate in the case narrative whether grinding was performed. |
| **Grinding Procedure for Incremental Sampling Methodology (ISM)** | When sample grinding is performed, initial method validation and any time major equipment is changed or when a reduction in the number or time of grinding cycles occur. | The laboratory shall demonstrate that grinding procedure is capable of reducing the particle size to <75 microns by passing representative portions of ground demonstration samples through a 200-mesh sieve. | Correct problem. |



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## Draft Table B-30 QSM Requirements

**Table B-30. PAH in Soil (Sample Processing Requirements for Incremental Sampling Methodology)**

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action and Qualification Criteria |
|---|---|---|---|
| **Grinding Cycle** | When sample grinding is performed using a puck mill, each field sample. | When a puck mill is used to grind the samples, grinding cycles shall not exceed 60 seconds and shall be followed by a 2-minute or longer cool down period between the grind cycles.<br><br>Other grinding apparatus may be used. | Correct problem and reprepare samples.<br><br>Qualification of data is not appropriate. |
| **Grinding Blank**<br><br>For batch preparation, the Grinding Blank may serve as the Method Blank. | When sample grinding is performed, a grinding blank is required. One per preparatory batch using Ottawa sand or a verified clean soil. A Grinding Blank shall be performed immediately after a customer-identified sample with suspected high target analyte concentration or after the LCS. | No analytes detected > ½ LOQ or > 1/10$^{th}$ the amount measured in the associated sample(s) whichever is greater. | Correct problem.<br><br>If required, reprepare and analyze Grinding Blank and all QC samples and field samples processed with the contaminated blank if sufficient sample material is available.<br><br>If the samples cannot be reprepared and analyzed, apply qualifier to specific analyte(s) in all samples in the associated preparatory batch and explain in the case narrative. |



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## Draft Table B-30 QSM Requirements

**Table B-30. PAH in Soil (Sample Processing Requirements for Incremental Sampling Methodology)**

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action and Qualification Criteria |
|---|---|---|---|
| **ISM Laboratory subsampling Replicates**<br><br>The laboratory shall prepare and analyze three or more replicates, each consisting of a minimum of 30 increments of the same mass and depth. | At the analytical subsampling step prior to extraction.<br><br>When grinding is not performed, each ISM field sample.<br><br>When grinding is performed, one ISM sample in each preparation batch. | RSD shall not exceed 30%. | RSD failure indicates the sample is not representative. If available, reprepare an additional field sample utilizing grinding procedures. Otherwise, apply qualifier and explain in the case narrative. |



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## Draft Table B-30 QSM Requirements

Table B-30. PAH in Soil (Sample Processing Requirements for Incremental Sampling Methodology)

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action and Qualification Criteria |
|---|---|---|---|
| **Laboratory Control Sample (LCS)**<br><br>A laboratory-prepared solid LCS may be used. The fortification shall be performed prior to any preparation steps.<br><br>The LCS shall be prepared and analyzed in exactly the same manner as the field samples, including all drying and grinding steps. | One per preparatory batch.<br><br>Shall contain all surrogates and all analytes to be reported.<br><br>A Standard Reference Material (SRM) that is used for a LCS can be ground as a single batch and subsampled repeatedly as long as the SRM is within expiration date. | Use customer-provided acceptance criteria. If customer-provided acceptance criteria are not available, use laboratory-developed acceptance criteria. | Correct problem.<br><br>If required, reprepare and analyze the LCS and all affected QC samples and field samples in the associated preparatory batch for failed analytes if sufficient sample material is available. For ISM samples, repreparation will be performed beginning at the subsampling step.<br><br>If the samples cannot be reprepared and analyzed, apply qualifier to specific analyte(s) in all affected samples in the associated preparatory batch and explain in the case narrative. |



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## Draft Table B-30 QSM Requirements

Table B-30. PAH in Soil (Sample Processing Requirements for Incremental Sampling Methodology)

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action and Qualification Criteria |
|---|---|---|---|
| **Matrix Spike (MS)**<br><br>Spiking is performed after ISM preparation prior to extraction. | One per preparatory batch if sufficient material is provided. | Same as the LCS acceptance criteria. | If MS results are not within the acceptance criteria, the data shall be evaluated to determine if the source(s) of failure is analytical error. If so, reprepare and analyze the MS if sufficient sample material is available. Otherwise, report data with qualification.<br><br>Qualify specific analyte(s) in the parent sample if results are not within acceptance criteria and explain in the case narrative. |



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## Draft Table B-30 QSM Requirements

Table B-30. PAH in Soil (Sample Processing Requirements for Incremental Sampling Methodology)

| QC Check | Minimum Frequency | Acceptance Criteria | Corrective Action and Qualification Criteria |
|---|---|---|---|
| **Surrogate Spike**<br><br>Surrogates shall be added to each field sample, blank, and LCS.A solid surrogate fortification standard shall be added prior to any processing (e.g. prior to drying/grinding or extraction) | All field samples, blanks and LCS. | The laboratory shall use at a minimum the following PAH surrogates and respective recovery criteria unless customer-provided limits are otherwise specified.<br><br>Recovery:<br>Acenaphthylene-d8 within 10% to 120%.<br>Benz[a]anthracene-d12 within 38% to 120%.<br>Benzo[a]pyrene-d12 within 14% to 120%<br>Fluorene-d10 within 29% to 120% | If surrogate results are outside the acceptance criteria, the data shall be evaluated to determine if the source(s) of failure is an analytical error. If so, reprepare and analyze if sufficient sample volume is available.<br><br>If obvious chromatographic interference is present, reanalysis may not be necessary, but the client shall be notified prior to reporting data.<br><br>If sample(s) with surrogate recoveries outside acceptance criteria cannot be reprepared and analyzed, apply qualifier to analyte(s) associated with the surrogate(s) outside acceptance criteria in the affected sample(s) and explain in the case narrative. |



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## Final Comments & Path Forward

- Project-specific requirements can over-ride QSM
- As Table B-30 is a new requirement, project teams can decide if they require a B-30 accredited laboratory for their project until QSM 6.0 accreditation.
- The EDQW does not recommend ISM for other methods until appropriate validation and appropriate use of performance indicators are available and used.



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## Final Comments & Path Forward

- Milling- Highly controversial! Trade-off between potential analyte loss and fundamental error. If the project decides not to mill the sample, subsampling replicates are mandatory and meet 30% RSD criteria.
- Splitting of a small portion for % moisture determination should be acceptable if no-milling is also acceptable.



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## Final Comments & Path Forward

- Milling should be performed for 8330B, inorganics, and matrices where compositional heterogeneity is assumed to be high.
- Surrogates for 8330B (3,4-DNT/1,2-DNB) are not commercially available yet. They should be added prior to ISM preparation.
- EPA is currently considering a stand-alone method for ISM.



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## Final Comments & Path Forward

### Collaboration

Tom Georgian Ph.D., USACE
Alison M. Suess, Ph.D., Chemist EM CX
Tony Danko Ph.D., NAVFAC EXWC
Nikki Andrzejczyk, Ph.D., NAVFAC EXWC



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## Final Comments & Path Forward

*"Proper implementation of ISM is complicated and will require project teams, chemists, and supporting contractors and laboratories to carefully consider all quality aspects of its use."*

*ISM-ism number 3*

*William Corl Ph.D.*
*Chemist & Deputy Director*
*NAVSEA LQAO*
*William.e.corl.civ@us.navy.mil*



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# Guidelines on Validation of Non-Regulatory Chemical and Radiochemical Methods

**Dr. Anand R. Mudambi**
**US EPA**
**Office of Research and Development**

2023 DOE/DoD EMDQ Workshop
Thursday, September 21, 2023

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## Disclaimer

- Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not constitute or imply its endorsement, recommendation, or favoring by the United States government.
- The views and opinions of the author expressed herein do not necessarily state or reflect those of the United States government or the United States Environmental Protection Agency and shall not be used for advertising or product endorsement purposes.

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## Background

- EPA develops methods for both regulatory and non-regulatory purposes
  - Regulatory method validation follows program/statutory-specific requirements and guidance
- Non-regulatory method development and validation is generally done to meet current and evolving Agency needs (e.g., emerging contaminants)

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## Issue

There was a lack of Agency-wide guidance for consistent non-regulatory method validation

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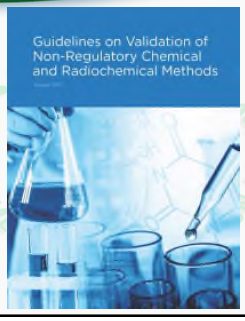
## Solution

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## Guidelines Overview

- Developed by an internal cross-Agency workgroup, with representatives from the following EPA offices:
  - OAR, OCSPP, OLEM, ORD, OW, Region 7, Region 10
- Document does NOT provide prescriptive or step-by-step guidance on conducting method validation studies

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## Guidelines Document Overview

- Addresses newly developed, adopted, or modified chemical and radiochemical methods
- Document provides:
  - An overview of the general principles and important areas of consideration for method validation including method performance characteristics
  - Lists and links to more detailed method validation resources (e.g., Agency documents, international standards, other guidance documents, etc.)
- Builds on concepts developed by the EPA Regional Laboratories and other parts of the Agency
- Introduces 3 new concepts

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## New Concepts

- Document introduces 3 new concepts to promote consistent method development and communication of validation results:
  - Method Life Cycle
  - Validation Descriptor
  - Method Validation Summary

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## New Concept #1: Method Lifecycle

- Illustrates the steps and processes involved with a method, from its beginning to its retirement
  - Initiates with the need, purpose, and method development
  - Validation is central to determination of method performance
  - Post-release, modifications made outside accepted method flexibilities may require "re-validation"


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## New Concept #2: Validation Design

- Standardized descriptor to concisely convey extent of validation performed
- Based on number of participating laboratories and different matrices
  - Noted as **[aL,bM]** where "**a**" is number of laboratories (**L**) and "**b**" is the number of different matrices (**M**)
  - For example, Validation Design [3L,2M] conveys that 3 laboratories and 2 matrices were included in the method validation

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## New Concept #3: Method Validation Summary

- Purpose
  - Concise overview of method validation presented in a consistent format
  - Easy access to pertinent and important information
  - Convenient comparison of similar validation studies
  - Facilitates sharing across the Agency

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## Document Content

- Reviews major Method Performance Characteristics
- Provides Additional Information on other method validation considerations

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## Method Performance Characteristics

- Guidelines cover typical method performance characteristics:
  - Bias/Trueness, Detection and Quantification Capability, Instrument Calibration, Measurement Uncertainty, Precision, Range, Ruggedness, and Selectivity
- For each characteristic, the document provides:
  - Definition(s)*
  - Short descriptions on its use
  - Useful resources/references

* Generally based on consensus standards

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## Additional Information

- In main document:
  - Guidance on interlaboratory validation study designs
  - Suggested resources for use in understanding and implementing statistical assessment of method validation results
- In appendices:
  - Discussion of method validation matrix variability considerations, with examples of matrices used/suggested from individual EPA offices
  - Compilation of detection and quantitation limit definitions

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# Method Validation Summary Overview

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## Method Validation Summary

- Designed to be placed at the front/introduction to the full Method Validation Report
- Does **NOT** replace the full Method Validation Report, which should be prepared in accordance with expectations and guidelines/protocols of individual offices and/or programs

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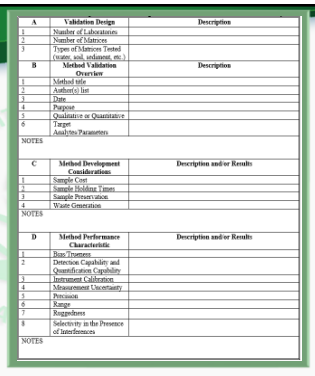
## Method Validation Summary

- Approximately 2-pages with 4 sections
  - Validation Design
  - Method Validation Overview
  - Method Development Considerations
  - Method Performance Characteristics



| A | Validation Design | Description |
|---|---|---|
| 1 | Number of Laboratories | |
| 2 | Number of Matrices | |
| 3 | Types of Matrices Tested (water, soil, sediment, etc.) | |

| B | Method Validation Overview | Description |
|---|---|---|
| 1 | Method title | |
| 2 | Author(s) list | |
| 3 | Date | |
| 4 | Purpose | |
| 5 | Qualitative or Quantitative | |
| 6 | Target Analytes/Parameters | |

NOTES

| C | Method Development Considerations | Description and/or Results |
|---|---|---|
| 1 | Sample Cost | |
| 2 | Sample Holding Times | |
| 3 | Sample Preservation | |
| 4 | Waste Generation | |

NOTES

| D | Method Performance Characteristic | Description and/or Results |
|---|---|---|
| 1 | Bias/Trueness | |
| 2 | Detection Capability and Quantification Capability | |
| 3 | Instrument Calibration | |
| 4 | Measurement Uncertainty | |
| 5 | Precision | |
| 6 | Range | |
| 7 | Ruggedness | |
| 8 | Selectivity in the Presence of Interferences | |

NOTES

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## Benefits to Using the New Concepts

- Method Lifecycle – Promotes a consistent approach to link and integrate method activities from identifying needs to revision/retirement
- Validation Descriptor [aL,bM] – Provides "one glance" overview of the extent of validation
- Method Validation Summary – Concisely communicates Validation Study information in a consistent format

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## Guidelines: Where to find them

On the EPA website at:

- EPA National Program Manager for Regional Laboratories
  (click the link to follow)

Direct Link to Document at:

- Guidelines on Validation of Non-Regulatory Chemical and Radiochemical Methods
  (click the link to follow)

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## New Document on Method Terms

- Wondered about what terms like "regulated", "promulgated", etc. mean with regard to EPA Methods?
- Never Fear - the document "Terms Used to Describe the Standing of US EPA Methods" is here!
- Hot off the press – or rather cold on the Internet at:
  - "Terms Used to Describe the Standing of U.S. EPA Methods"

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## Document Purpose

- Compile terms (e.g., regulatory, promulgated, approved) used by EPA to describe/designate the standing of its methods
- Promote a better understanding of these terms for both EPA personnel and external parties (e.g., states, laboratories and testing organizations, etc.).

Note: This document does not include terms that describe technical characteristics of a method (e.g., detection limits).

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## Document Layout

- Introduction, Purpose and Scope
- Background
- Publications Associated with Method Terms
- Method Standing and Related Terms (by EPA Program)
- Appendices

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## Questions?

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## Example Method Validation Summary Section A

A. Section for Validation Design

- Descriptions include enough detail for a "quick glance" summary of validation
- Number and types of matrices are the focus

24

## Method Validation Summary for SW-846 Methods 3512 and 8327

| A | Validation Design | Description |
|---|---|---|
| 1 | Number of Laboratories | 8 |
| 2 | Number of Matrices | 3 |
| 3 | Types of Matrices Tested(water, soil, sediment, etc.) | Ground water, surface water, wastewater |

25

## Example Method Validation Summary- Section B

B. Section for Method Validation Overview

- Title, authors, date
- Purpose including analytes

26

## Method Validation Summary for SW-846 Methods 3512 and 8327

| B | Method Validation Overview | Description |
|---|---|---|
| 1 | Method Table | • SW-846 Method 3512: Solvent Dilution of Non-Potable Waters<br>• SW-846 Method 8327: Per- and Polyfluoroalkyl Substances (PFAS) by Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) |
| 2 | Organization | EPA/Office of Land and Emergency Management/Office of Resource Conservation and Recovery |
| 3 | Date | July 2021 |
| 4 | Purpose | To validate the preparation and analysis of non-potable water samples for select PFAS by LC/MS/MS |
| 5 | Qualitative or Quantitative | Quantitative |
| 6 | Target Analytes/Parameters | Validated for 24 PFAS target analytes, including:<br>• C4-C14 perfluorinated carboxylic acids<br>• C4-C10 perfluorinated sulfonic acids,<br>• 4:2, 6:2, and 8:2 Fluorotelomer Sulfonic Acids (FTS)<br>• perfluoro_octane sulfonamide<br>• N-ethyl and N-methyl perfluoro_octane sulfonamido_acetic acids<br>Please refer to Method 8327 for more detail |

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## Example Method Validation Summary- Section C

C. Section for Method Development Considerations

- Practical findings and details considered during development that will be important during implementation

28

## Method Validation Summary for SW-846 Methods 3512 and 8327

| C | Method Development Considerations | Descriptions and/or Results |
|---|---|---|
| 1 | Sample Cost | Not Determined |
| 2 | Sample Holding Times | Sample collection to preparation: 14 days Sample preparation to analysis: 30 days |
| 3 | Sample Preservation | Refrigerate at 0-6°C |
| 4 | Waste Generation | These methods generate relatively small amounts of waste due to:<br>• Small recommended sample volumes;<br>• Low volumes of reagents and solvents used for sample preparation; Liquid chromatography columns with particle sizes ≤ 2 µm achieve efficient separations at low flow rates |

Notes on Section C:
C2 Holding times are published as guidelines and were based on holding time studies conducted for other PFAS methods, including EPA methods 533 and 537.1 and ASTM D7979-20.

29

## Example Method Validation Summary- Section D

D. Sections for Method Performance Characteristics and Results

- Provides the guidance used to validate specific method parameters
- Briefly summarizes results and data findings
- Notes section available in each section for any additional comments or items of note

30

## Method Validation Summary for SW-846 Methods 3512 and 8327

| D | Method Performance Characteristic | Description and/or Results |
|---|---|---|
| 1 | Bias/Trueness | Average (median) recovery across eight laboratories ranged from 80-118% at 95% confidence for every target analyte except 6:2 FTS in each matrix type and prepared concentration level. |
| 2 | Detection Capability and Quantification Capability | Lower limits of quantitation (LLOQs) across eight laboratories were verified at nominal concentrations of 10-20 ng/L at 95% confidence for all target analytes except for 8:2 FTS (40 ng/L) and 6:2 FTS (160 ng/L). |
| 3 | Instrument Calibration | Target analytes were calibrated by external standard using weighted regression. |
| 4 | Measurement Uncertainty | Not Applicable |
| 5 | Precision | Relative standard deviation (RSD) of measured concentrations in spiked samples was <50% in every matrix and spike level combination in each laboratory except for PFOS in one laboratory/matrix/spike level and 6:2 FTS in three laboratory/matrix/spike level combinations. |

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## Method Validation Summary for SW-846 Methods 3512 and 8327 cont.

| D | Method Performance Characteristic | Description and/or Results |
|---|---|---|
| 6 | Range | LC/MS/MS initial calibration range was from 5 to 200 ng/L (nominal). No attempt was made to determine an upper limit for quantitative analysis |
| 7 | Ruggedness | Formal ruggedness testing was not performed as part of the validation study |
| 8 | Selectivity in the Presence of Interferences | No major sources of interferences were observed that impacted qualitative identification of target analytes |

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## Notes on Section D

| Notes on Section D |
|---|
| D1- Validation study samples were tested unspiked or spiked at nominal concentrations of 60 and 200 ng/L. Each laboratory tested 5 replicate spiked samples of each matrix at each prepared concentration. Samples were prepared centrally and shipped to laboratories, which were blind to the identities of the samples and their prepared concentrations. Half of the participating laboratories reported background contamination with 6:2 FTS. |
| D2- High verified LLOQs for 6:2 FTS were attributed to background contamination in half of participating laboratories. Methods 3512 and 8327 are performance-based, and laboratories are required to establish and periodically verify LLOQs at which they can routinely meet the acceptance criteria for all categories of quality controls. Refer to Method 8000D Section 9.7 and Method 8327 Section 9.9 for more information about establishing and verifying LLOQs https://www.epa.gov/hw-sw846/sw-846-compendium |
| D4- Methods 3512 and 8327 are performance-based, and, like many other SW-846 methods, they recommend applying statistically-based or project-defined acceptance limits for recovery and precision in field samples and associated prepared quality control samples |

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## Notes on Section D cont.

| Notes on Section D |
|---|
| D5- PFOS imprecision was attributed to variability in background concentrations in wastewater samples, and 6:2 FTS imprecision was attributed to laboratory contamination in half of participating laboratories. |
| D7- Laboratory deviations from the method validation study protocol led to the addition cautionary measures to the methods related to ruggedness, including:<br>1) avoiding aqueous subsampling prior to adding sufficient organic solvent; and 2) avoiding storage of prepared samples and standards in glass containers |
| D8- Some PFAS target analytes do not make secondary product ions with sufficient relative abundance to be useful for supporting qualitative identification. |

Related Links:
1. Executive summary: https://www.regulations.gov/document/EPA-HQ-OLEM-2018-0846-0111
2. Quality Control summary report: https://www.regulations.gov/document/EPA-HQ-OLEM-2018-0846-0005

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# Data Review and Management Subgroup

Melinda McClellan, Ph.D.
Chemist
Huntsville Environmental and Munitions Center of Expertise
Melinda.S.Mcclellan@usace.army.mil



1

## DR&M Subgroup

- 8 members from across Army, Navy, and Air Force
- Meet every other week
- Major Efforts
  - Data Validation Guidelines and Modules
  - Data Usability Guidelines
  - Webinars and Trainings
    - As requested or available
  - Question and Answer
    - As questions are received



2

## Data Validation Guidelines

- Not requirements, but a set of guidelines for validating data
- Does not address performance of field work
- Cannot determine usability of data
- Modules are technology or method specific
  - Module 1: GC/MS
  - Module 2: ICP-OES
  - Module 3: PFAS by B-15
  - Module 4: GC
  - Module 5: ICP-MS
  - Module 6: PFAS by Draft EPA Method 1633



3

## Data Validation Guidelines

| Module | Topic | Published | Status Notes |
|---|---|---|---|
| General | Overview | 05 Nov 2019 | |
| 1 | GC/MS | 18 May 2020 | Updating currently |
| 2 | ICP-OES | 18 May 2020 | |
| 3 | PFAS B-15 | 07 May 2020 | Will not be updated |
| 4 | GC | 11 Mar 2021 | |
| 5 | ICP-MS | 09 Nov 2022 | |
| 6 | PFAS 1633 | 01 Nov 2022 | Will be updated after final 1633 |

Revised table for Sample Qualification in the Presence of Blank Contamination
- Published 2/9/2022
- Applies to all modules (replaces table in 1-4, included in 5 and 6)



4

## Data Review Comparison

| | Verification | Validation | Usability Assessment |
|---|---|---|---|
| Purpose | Ensure presence and completeness | Ensure compliance with underlying specifications (e.g., SOPs, Methods, QSM) | Adequate quality and quantity for decisions (DQOs, PARCCS) |
| Responsible Party | Varies, usually contractor | 3rd Party* Validators | Entire Project Team |
| Covers | Field records and laboratory data | Laboratory data (should have field data, but not always included) | All project records and data |
| Timeline | Following collection | Following data receipt or laboratory report issuance | End of sampling event, prior to decision making |



5

## Data Validation in a Nutshell

**Purpose**
- An analyte- and sample-specific process that determines the analytical quality of a specific data set (EPA G-8).
- The systematic review of laboratory data deliverables which can help identify laboratory and field sample analytical uncertainty. … Evaluation of data with respect to the project measurement quality objectives (MQOs). (DoD General Data Validation Guidelines)

**Scope**
- Performed on Analytical Laboratory Data
- Could and should be performed on field data, (but often is not).

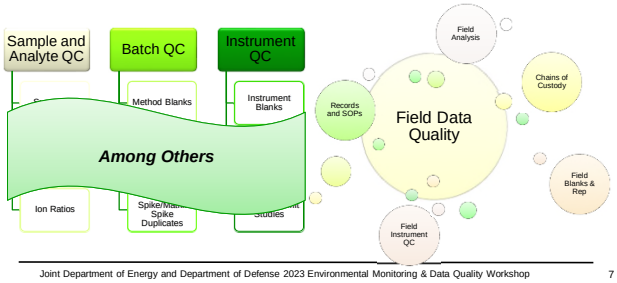
**Question**
How often is your field data or records formally validated or how often do you validate field data or records?



6

## Inputs to Validation

**Evaluating Laboratory Data Quality** **Evaluating Field Data Quality**






7

## Stages of Validation

- Stage 1:
  - Review of sample results forms, sample receipt summaries (chain of custody), and field QC data (field blank, field duplicates)
- Stage 2A:
  - Review of sample and batch summary forms
- Stage 2B:
  - Review of instrument summary forms
- Stages 3 and 4
  - Review of raw data with some recalculation

- Required for all stages:
  - Cover Sheet
  - Table of Contents
  - Laboratory Case Narrative
  - Results Summary




8

## Responsibilities

- 3rd Party – a definition
  - "It is anticipated that data validation is performed by a party independent of the laboratory. Project teams may identify a government quality assurance validation as necessary. A government quality assurance validation is defined as being independent of the prime contractor and performed by a government representative or services directly contracted by the government agency independent of the prime contractor." (DoD General Data Validation Guidelines)

**Independent of the Laboratory**
Contractor Chemists
Validators Subcontracted to Prime
Government Project Chemist

**Independent of the Prime Contractor**
Validators Contracted to Government
Government Project Chemist



9

## Lessons Learned

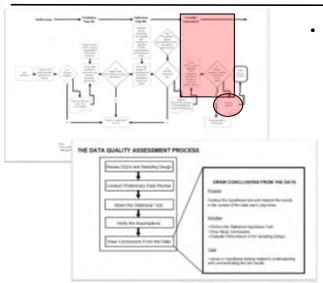
- Validators can and should give detail in their validation reports
- Guidelines cover almost all QC outlier situations, but can't cover all of them
- Projects are free to supplement the guidelines where they feel appropriate

**Question**
What are your lessons-learned?
What has worked well for projects?
What has not worked well and how can we improve?



10

## DUA Guidelines




Top Figure from IDQTF, UFP-QAPP Manual V1, March 2005, Figure 37. Data Review Process. Bottom Figure from EPA QA/G-9, Chapter 5.

- What
  - Data Usability Assessment is the final step in data review, involving the entire project team evaluating data with respect to project DQOs to determine whether data are of adequate quality and quantity to make project decisions

Why

- Project teams expressed a need for process-oriented guidance for how to perform a DUA. This guidance complements existing EPA guidance, such as EPA QA/G-9.

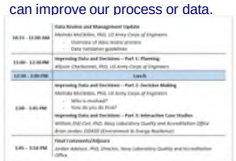


11

## DUA Guidelines

- Who
  - Document is joint effort with IDQTF
- When
  - Development is ongoing, with a sneak-peek webinar planned for later this year.
- Where
  - Document is intended to be published as an IDQTF-approved product

- How
  - Join us in just a few moments to hear how to develop strong DQOs. Join us after lunch to learn how to evaluate data with respect to those DQOs in a DUA. Then tell us how you think we can improve our process or data.





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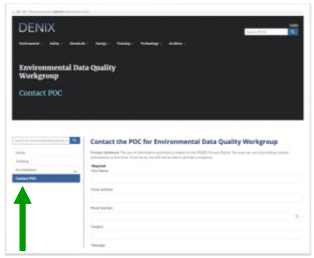
# Thank You

Questions or Comments

Submit through DENIX Portal at:
https://www.denix.osd.mil/edqw/

Or

Contact:
Melinda McClellan
Melinda.S.McClellan@usace.army.mil



# Improving Data and Decisions

## Part 1: Planning

Allyson Charbonnet, Ph.D.,
U.S. Army Corps of Engineers



1

## Outline

- Why are we here?
- Introduction to EPA G4.
- What should I bring to the SPP?
- The Process
- Evaluating the Execution
- What should we do now?



2

## Why are we here?

Isn't there a pool outside…



3

## Why are we here?

Because every Environmental Project decision is based on data.

Is there a problem? What is the problem? When is the problem solved?

If we aren't planning for, collecting, and basing our decisions on legally defensible data of known quality, none of the other project factors matter. We might as well toss a coin.



4

## Why are we here?

Why does legally defensible data of known quality matter?

Look at the communities you serve…

Consider the impact on risk to the Federal Government and our national security…

The work you do matters! Make sure the data matters!



5

## Why are we here?

Are you **EXCITED**

about **Data Quality**?

We hope so!

Or this is gonna be a long 90 minutes.



6

## Introduction to EPA G4

Where do we start?

We start with the basics!



7

## Poll: Vacation Planning Style?

The Planner

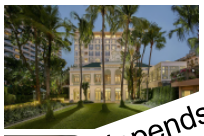
The Non-Planner



8

## Poll: Highest QUALITY Vacation?

The 5-Star Resort



The Rustic Cabin

It depends! What are your vacation quality objectives?



9

## The Ultimate Planning Guide

Taking an environmental remediation trip?

Use EPA G4 to plan your best vacation yet!

Guidance on Systematic Planning Using the Data Quality Objectives Process (February 2005)

It is EPA's recommended planning process when environmental data are used to select between two alternatives or to derive an estimation of contamination.

Through Systematic Planning, a project team determines the appropriate type, quantity, and quality of data necessary to support project decisions.



10

## What Are Data Quality Objectives?

Within the Systematic Planning Process (SPP) there is the Data Quality Objective (DQO) Process.

The DQO process consists of seven (7) steps that result in quantitative and qualitative criteria that:

- Clarify Study Objectives
- Define appropriate data types
- Specify tolerable levels of potential decision errors

1. State the Problem
2. Identify the Decision
3. Identify Inputs to the Decision
4. Define the Study Boundaries
5. Develop a Decision Rule
6. Specify Limits on Decision Errors
7. Optimize the Design for Obtaining Data



11

## More About the DQO Process

Series of logical steps

Used to plan for resource-effective acquisition of environmental data

It is flexible and iterative

Applies to both decision-making (e.g., compliance or non-compliance with a standard) and estimating (e.g., ascertaining the mean concentration of a contaminant)

It provides defensibility – use of a documented decision-making process increases the likelihood that decisions will withstand legal challenges



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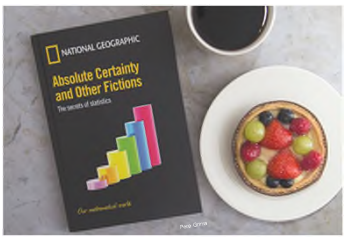
## Can Our Decisions be 100% Certain?




Don't like this one?

How about…



13

## This one…

Absolute certainty is a privilege of uneducated minds and fanatics.

It is, for scientific folk, an unattainable ideal.

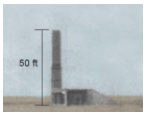
Cassius Jackson Keyser



14

## Let's take a trip…

Where are we going Pat?




The beautiful and balmy Former Air Force Base Incinerator and Landfill Site.

Site overview:

- Former incinerator constructed of bricks; 50' high stack measuring 9" internal diameter.
- The incinerator was demolished in 1999.
- Currently only a weathered concrete pad measuring 22-feet by 50-feet remains.
- Allegedly used to burn household and base waste products.
- Incinerator slag and bottom ash was dumped into the adjacent landfill disposal area.

Disclaimer / Credit where Credit is Due: This fictional FUDS is a synthesis of two real FUDS with data considerations based on work published by the Hawaii Department of Health through multiple venues.



15

## What should I bring to the SPP?

It's a party, right?

I'll bring the chips.



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## The Right Personnel

Entities

- Lead Organization/USACE
- Stakeholders/Regulators
- Contractor
- Subcontractors

Each entity brings their own personnel and subject matter experts (SMEs) to the table

Personnel

- Project Managers
- Technical Managers
- Resource Managers
- Geologists
- Hydrogeologist
- Geophysicists
- Risk Assessors
- Chemists
- Statisticians
- Analysts
- Field Samplers
- Safety Personnel

Look for ways to maximize these resources and collaborate with a common goal of protecting human health and the environment through decisions based on legally defensible data of known quality



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## The Right Information

Project Execution

- Budget
- Deadlines for collecting data
- Rights of Entry
- Available personnel / contracts
- Schedule
- Archeological / biological constraints

Initial Project Details

- Potential sources of contamination
- Potential location(s) of contaminants
- Media impacts
- Exposure scenarios / land use data
- Geologic and hydrogeologic data characteristic of site

Regulatory and Sociopolitical

- Regulatory requirements
- Organizations interested in the investigation
- Potential political issues
- Possible future uses of the data

Lessons Learned Similar Projects

- Performance of sampling designs
- Performance of analytical methods
- List of potential innovative technologies
- List of potential risks to scope and schedule



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## Our Former Incinerator TRIP

Soils: Grayish-yellow to yellowish-orange sand to silty sand
Average of 25% coarse sand and gravel

Previous investigations: Lead-contaminated soil throughout investigation area
Lead contamination extending to a depth of 10'

Prescreening of surface soil in current investigation area with a field XRF indicates concentrations of lead in excess of 400 mg/kg.

Current land use: Open recreational

Future land use: Residential

EPA Regional Screening Limit for Lead in Residential Soil: 400 mg/kg



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## The Process

Make it work!



20



## 1. STATE THE PROBLEM

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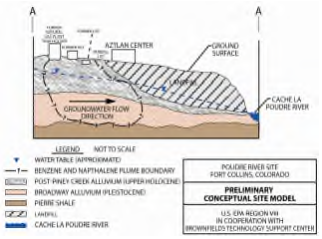
## Conceptual Site Model Foundation




Building a detailed Conceptual Site Model (CSM)

- Consolidation of historical site knowledge
- Details current site conditions / uses
- Documents knowns
- Reveals Data Gaps / unknowns

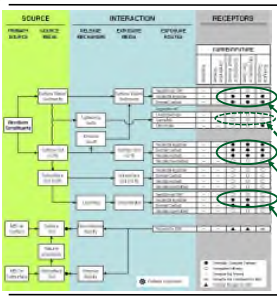
*The whole purpose of the investigation.*
*If we knew everything, we'd just decide!*

CSM Figure from: EPA 542-F-11-011 *Effective Use of Project Life Cycle CSM*, July 2011



22

## Starts with Detailed CSM




DQO to address each project element
– General contaminants
– Environmental media
Generally, relate to potentially complete exposure pathways

**DQO for contaminants in surface water**

***Ecological exposures typically addressed under other media***

**DQO for contaminants in surface soil**

**DQO for contaminants in groundwater**

Salisbury, James. FUDS Training Course: UFP-QAPP Overview



23

## 1. State the Problem – Incinerator

There is lead-contaminated soils at the site.

Is this a good problem statement?

There is evidence that former incinerator ash is present at the site resulting in lead-contaminated soils up to a depth of 10' below land surface which may pose health risks to current recreational land users and future residents and negative impacts to the surrounding environment.

Better?

Include available resources, budget and schedule constraints as known.



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25

## 2. IDENTIFY THE DECISION / ESTIMATION STATEMENT

25

## Start with Formulating a Question

Formulate the Problem into study question(s) based on the type of problem:

Decision problems
- Does the contaminant concentration in ground water exceed acceptable levels?
- Does the contaminant pose a human health or ecological risk?
- Is the contaminant concentration above background levels?

Estimation problems
- What is the distribution of pollutant air concentration over space and time?
- What is the largest concentration consistent with background?
- What is an upper bound estimate of the site mean?



26

## Outline Alternative Actions

Consider a range of potential answers to the study question(s).

For each possible answer, identify a logical course of action to take in response.

| Finding | Logical Course of Action |
|---|---|
| • No issue identified | • No Action |
| • Issue poses immediate threat | • Emergency Response Action |
| • Issue poses imminent threat | • Time-Critical Response Action |
| • Issue poses longer-term threat | • Remedial Action |



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## 2. Identify the Decision(s)

Develop the decision statement (template below):

Determine whether [unknown environmental issue] requires [taking one or more actions].


Determine whether lead contamination in surface and subsurface soils at the Former Incinerator and Landfill poses an unacceptable risk to human health or the environment that requires remediation.

Determine whether lead contamination in subsurface soils at the Former Incinerator and Landfill presents a source of groundwater contamination that requires remediation.



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## 3. IDENTIFY THE DECISION INPUTS

29

## 3. Identify Inputs to the Decision

- Focus on what information is needed for the decision.
  - Physical properties of media?
  - Chemical characteristics of matrix?
  - Existing data quality?
  - New data needed?
- Identify and evaluate appropriate sampling and analysis methods to minimize bias.
  - Representative sampling methods
  - Stability of samples during shipment
  - Matrix interferences / contamination
  - Calibration range / instrument sensitivity
- Identify the information needed to establish the screening or action level.
  - EPA Regional Screening Levels
  - Site-specific exposure concentrations
  - Other applicable regulatory requirements
  - Background concentrations



30

## 3. Identify Inputs - Incinerator

- Focus on what information is needed for the decision(s).
  - Lead concentration in surface soils
  - Lead concentration in subsurface soils
  - Lead leachability data
  - Other?
- Identify and evaluate appropriate sampling and analysis methods to minimize bias.
  - Discrete samples (#) vs Incremental Sampling
  - Onsite XRF vs Method 6010 vs Method 6020
  - Matrix interferences / contamination
  - Method 6020 ~ 2X more sensitive for lead in soil matrices
- Identify the information needed to establish the screening or action level.
  - EPA Regional Screening Levels
  - Site-specific exposure concentrations
  - Other applicable regulatory requirements
  - Background concentrations



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## 4. DEFINE THE BOUNDARIES OF THE STUDY

32

## 4. Define Study Boundaries

Activities to be completed:

- Define the target population
- Determine the spatial and temporal boundaries
- Identify practical constraints
- Define the scale of inference (i.e., decision unit or scale of estimation)



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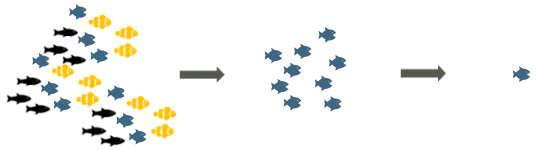
## Target Population

Target Population = total collection / universe of sampling units from which samples will be drawn

When the target population consists of "natural entities" (e.g., people, plants, fish…) then the sampling unit is straightforward, it is the entity.



Fish Population | Target Fish Population | Sampling Unit



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## Target Population

What do you do when your target population is a continuous media, e.g., soil, water, or air?

The sampling unit must be defined as some area, volume, or mass that may be selected from the target population.

From G4 – It may be helpful to "work backwards" and think of how you would define an individual sampling unit when trying to develop a clear definition of the target population. For example, if a 6" core is to be sent to the laboratory for analysis, the target population would be all possible 6"cores from the area under investigation.



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## Spatial and Temporal boundaries

- To determine spatial boundaries on target population:
  - Define the geographical area within which decisions apply
  - Define the media of concern
  - Divide medium into homogeneous strata
- To determine temporal boundaries on target population:
  - Determine the period of time the data should represent
    - Weather conditions, seasons, activity patterns…
  - Determine the time frame for which the decision is relevant
    - Period of 100-yrs for contaminant leaching, period of 8-yrs for average resident…



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## Constraints

Identify practical constraints such as:

- Access to the property
- Availability and operation of equipment
- Extreme heat or freezing temperatures
- Availability of trained personnel



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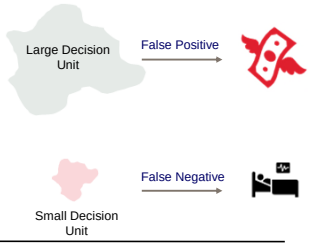
## Scale of decision

Smallest unit of area, volume, or time over which data will be collected, analyzed, aggregated, and interpreted to make a decision.

Important to consider:

- Present and future uses for decision unit
- Where the decision unit is located (remote vs densely populated)
- Requirements for potential remediation
- Consequences of a wrong decision






38

## Our incinerator's boundaries

Target population = Soil in landfill and around former incinerator

Spatial boundaries = Soils within 100' radius of former incinerator
Soils within perimeter of landfill
Soils up to 10' below land surface

Temporal boundaries = Sample results not dependent on season/weather, sampling crews available in 9 months, results will represent future resident exposure

Identify practical constraints = Freezing temperatures/snow coverage in winter

Define the scale of decision = Residential lot-sized area (1/4 acre)



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## 5. DECISION RULES

40

## 5. Develop a Decision Rule

"If…then…else…" rules.

These rules should incorporate:

- The population parameter of interest (e.g., mean, maximum, percentile)
- The scale of decision making (e.g., residential lot size, park trail [i.e., linear feet])
- The action-triggering value (e.g., screening level, regulatory cleanup limit, or project-specific risk value)
- The alternative actions



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## Developing the rule

Select the population parameter and the action level then combine with the scale of decision making (Step 4) and the alternative actions (Step 2).

Examples:

If the true mean lead concentration in the top 6" of soil across a 0.25-acre residential lot exceeds 400 ppm, then evaluate site-specific risk. If less than 400 ppm, then no further action.

If the true mean dioxin concentration in the surface 2" of soil of a decision unit (20' x 100') exceeds 1ppb, then remove a 6" layer of soil, else leave the soil intact.



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## Our Incinerator Decision Rules

If the true mean lead concentration within a residential decision unit is greater than 400 mg/kg to a depth of 2' below land surface, then the soil will be remediated.

If not, then the soil will be left *in situ*.

If the true mean lead concentration within the former incinerator area or landfill footprint is greater than the groundwater leachability concentration to a depth of 10' below land surface, then temporary monitoring wells will be installed to investigate groundwater contamination.

If not, then no further action.



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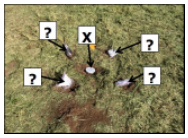
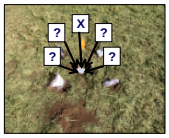
## Notes on Sampling Uncertainty

What if I moved my sample location over by a foot or two?




What if a different aliquot at a single sample location was analyzed?




Adapted from Hawaii-DU-MIS-Overview-Rbrewer-Feb-2019.pptx; https://health.hawaii.gov/heer/guidance/heer-webinars/#webinar17



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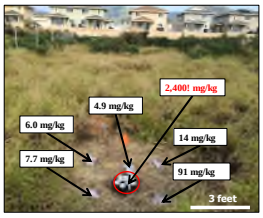
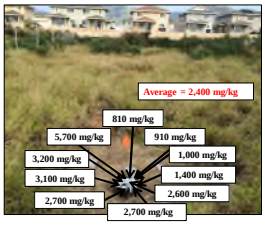
## PCBs – Waste oil Dumped Bare Soil

Variability between co-located samples
Laboratory Analysis 8082




10-g mass tested in accordance with standard method recommendations

Variability within a single sample location
Laboratory Analysis 8082




10-g mass tested in accordance with standard method recommendations



45

## Explosives-contaminated sites

**Plastic template used for consistent sampling.**
Center at sample location, sample numbers 2 and 5 oriented north-south.

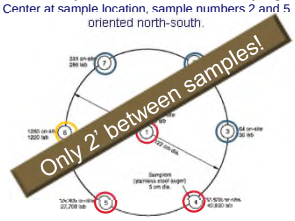



*Figure 2. Sampling scheme (TNT concentrations shown are from sampling location 1).*

**Table 1. Results from sampling location 1, Monite site.**

**a. Analytical results.**

| Sample | TNT on-site analysis (µg/g) | Laboratory analysis (µg/g) TNB | TNT | 2,4-DNT | Total |
|---|---|---|---|---|---|
| Discrete samples | | | | | |
| 1a | 42,700 | 107 | 37,500 | 70 | 37,700 |
| 1b | 36,900 | 104 | 45,000 | — | 45,100 |
| 2a | 402 | 30 | 390 | — | 420 |
| 2b | 507 | 30 | 382 | — | 412 |
| 3a | 174 | 12 | 113 | 20 | 145 |
| 3b | 154 | 11 | 116 | — | 127 |
| 4a | 28,000 | 97 | 44,400 | — | 44,500 |
| 4b | 27,600 | — | 41,200 | — | 41,200 |
| 5a | 24,400 | — | 33,000 | — | 33,000 |
| 5b | 24,400 | — | 22,400 | — | 22,400 |
| 6a | 1,240 | 42 | 1,170 | — | 1,210 |
| 6b | 1,310 | 33 | 1,200 | — | 1,230 |
| 7a | 327 | 23 | 305 | — | 328 |
| 7b | 338 | 17 | 227 | — | 244 |
| mean | 13,500 | | | | 16,300 |

Jenkins, Thomas F., et.al. *Assessment of sampling Error Associated with Collection and Analysis of Soil Samples at Explosives-Contaminated Sites*, Sept. 1996. CREEL



46

## Representativeness

Think of the size of the samples you take…

Think of the mass of the samples you take……

What are your samples representative of? 1g, 10g…

Example ¼ acre sandy front yard to 6" (277tons)



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## Notes on Measurement Uncertainty

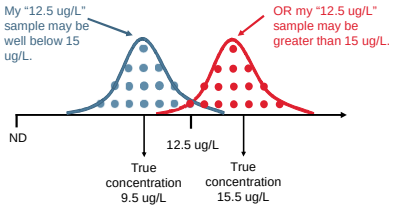
Thought experiment

You need to determine if the concentration of lead in groundwater is less than 15 ug/L.

The laboratory reports a concentration of lead in your groundwater sample of 12.5 ug/L.

Is your groundwater concentration less than 15 ug/L?

Lab reported concentrations for multiple preparations and analyses of a single sample.



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## Measurement Uncertainty

| | | |
|---|---|---|
| $12 \pm 2 < 15$ | → | Groundwater is "clean" |
| $12 \pm 5 < 15$ ? | → | Inconclusive result |

- A measurement without an estimate of its uncertainty is typically of little or no use.
- Data users need to assess total measurement uncertainty using replicate samples and statistical methods.
- Laboratory component of uncertainty is small relative to the total uncertainty.



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## 6. SPECIFY LIMITS ON DECISION ERRORS

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## 6. Specify Limits on Decision Errors

In Step 5, we imagined we knew the true mean accurately with access to perfect information and unlimited data. But we saw that in reality we will not have perfect information.

Estimates and conclusions we make from collected data may deviate from what is true within the population.

In Step 6, we need to derive performance or acceptance criteria that collected data will need to achieve to minimize the possibility of making an erroneous conclusion.



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## Intended Use of Data

The intended use of the data defines the approach:

**Decision Problems**

Addressed by performing statistical hypothesis tests on the collected data.

**Estimation Problems**

Estimate reported with a measure of uncertainty such as a standard error or confidence interval.



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## Hypothesis Tests – Decisions

With a decision, either a given situation is true or it is not true.

Definitions:

Baseline Condition = Assumed to represent the *de facto*, true condition
Alternative Condition = Other situation

- Null hypothesis, $H_0$: Baseline condition
- Alternative hypothesis, $H_1$: Potential condition

Evidence is presented to reject $H_0$ and accept $H_1$.

Uses binary logic. $H_0$ & $H_1$ are *true* or *false*. If $H_0$ is false, $H_1$ is true.



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## Hypothesis Test Examples

Demonstrate that the true mean ($\mu$) lead soil concentration is ≥ 400 mg/kg.

$H_0$: $\mu < 400$ mg/kg

$H_1$: $\mu \geq 400$ mg/kg

Demonstrate that the true mean ($\mu$) lead soil concentration is > background.

$H_0$: $\mu_{Site} \leq \mu_{BG}$
$H_1$: $\mu_{Site} > \mu_{BG}$



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## Decision Error Tolerances

| | $H_0$ True | $H_0$ False |
|---|---|---|
| Reject $H_0$ | **Type I Error ($\alpha$) or False Positive** | **Correct Decision** |
| Do not reject $H_0$ | **Correct Decision** | **Type II Error ($\beta$) Or False Negative** |



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## Decision Error Consequences

If the baseline assumption is that the program or site is in compliance, then:

Type I Error, False Positive, or False Rejection Error:
- Deciding program or site not in compliance when it is
- An overreaction to a situation
- Wasted resources, unnecessary expenditure

Type II Error, False Negative, or False Acceptance Error:
- Deciding program or site is in compliance when it is not
- A missed opportunity for correction
- Allowing a hazard to public health or the ecosystem



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## Decision Error Tolerances

| | $H_0$ True ($\mu$ < 400 mg/kg) | $H_0$ False ($\mu \geq$ 400 mg/kg) |
|---|---|---|
| Reject $H_0$ | **Acceptable rate of error $\alpha = 0.10$** (10% False Positive) | **Power $1-\beta$** (Power 95%) |
| Do not reject $H_0$ | **Confidence Level (CL) $1-\alpha$** (CL 90%) | **Acceptable rate of error $\beta = 0.05$** (5% False Negative) |



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## 7. OPTIMIZE THE SAMPLING DESIGN

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## 7. Optimize the Sampling Design

Goal – develop a resource-effective design for collecting and measuring environmental samples, either:

(a) the most resource-effective data collection process that is sufficient to fulfill study objectives,

or

(b) a data collection process that maximizes the amount of information available for synthesis and analysis within a fixed budget.

Statistical inference techniques (e.g., hypothesis tests) require a probability-based sampling design, as this type of design will allow you to properly characterize uncertainty in the outcome of the data collection process.



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## Relationships

In Step 6, we determined our tolerances on making decision errors. Now we need to determine the minimum sample size, $n_0$ needed to detect a difference between $H_1$ and the action limit in order to reject $H_0$.

$\alpha$ = false positive error
$\beta$ = false negative error
$\Delta$ = Resolution – detected change ("signal") needed to reject $H_0$
$\sigma$ = Standard deviation ("noise" / variability)

The smaller your tolerance for error and as the difference between your action limit and $H_1$ true mean decreases, the more samples you will need.



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## Statistical Software Outputs

| $n_0$ $\alpha = \beta = 0.05$ (Test to compare mean/median with decision limit) | | |
|---|---|---|
| $\Delta/\sigma$ | Normal | Distribution not assumed |
| 0.5 | 45 | 74 |
| 1 | 13 | 24 |
| 2 | 5 | 12 |

"Rule of thumb": Given no other information, let $\alpha = \beta = 0.05$ and $n_0 = 15 - 30$ grabs

Visual Sampling Plan is freeware software from the DOE Pacific Northwest National Lab http://vsp.pnnl.gov/



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## Sampling Design Considerations

Is having only 1-2 grab samples enough?

Considering previous uncertainty discussion, the answer is no.

However, it also depends on the question being asked, the CSM, and the reliability of uncertainty estimates.

Collecting 15-30 grab samples to characterize a decision unit is going to be expensive, each grab sample will need to be analyzed and the data validated. Is there another way to obtain representative data?

Have you considered Incremental Sampling Methodology (ISM)?



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## Incremental Sampling Methodology

Used when DQOs are in terms of population means for select contaminants (e.g., explosives, metals, PAHs, & high concentration VOCs)

- Normalizes distribution → Decreases *n* & simplifies calculations
- Decreases variability → Decreases uncertainty for fixed *n*
- Reduces non-detects → Simplifies calculations & reduces uncertainty



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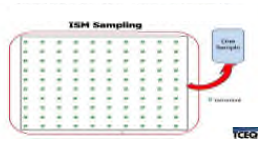
## Incremental Sampling Methodology

ISM is a composite sampling method for particulate material (e.g., soils) to estimate mean concentrations of DUs.

Many "increments" (e.g., 30 – 100 soil cores) collected *randomly* from DU (e.g., soil volume) combined to prepare each ≈1-kg ISM sample.



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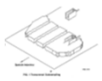
## Laboratory Processing ISM Samples

- Air drying
- Particle size selection via sieving
- Particle size reduction (grinding)
- Splitting/sub-sampling (mass reduction)
- Extraction and instrumental analyses







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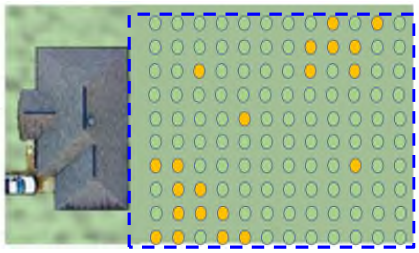
## Sampling the Decision Unit



Residential Backyard Exposure Area

Decision will be made for entire area

Site conceptual model predicts that there could be small pockets of elevated contaminants

Source: ITRC, 2020 ISM Guidance, 1.3, Figure 2.17



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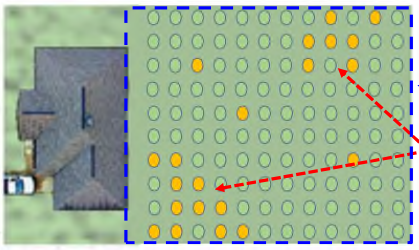
## Selecting the Decision Unit



Two Scenarios:

1. Only interested in exposure across entire area
2. Concerned about possible small areas with elevated concentrations also



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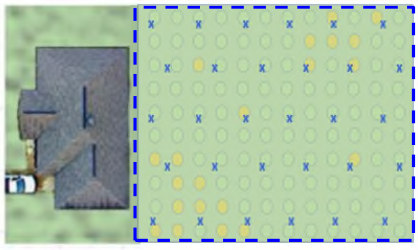
## Scenario 1



Scenario 1: DU = SU

Design decision unit to capture total exposure

- 1 decision unit
- 30 total increments x

Result: average concentration over entire area



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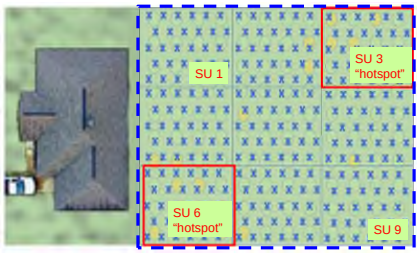
## Scenario 2




Scenario 2: DU = SU1 +…+ SU9

Design sampling units small enough to capture variability within decision unit

- 9 sampling units
- 30 increments each

Beneficial if DU mean exceeds risk action limit and hot spots are present. If DU mean is less than action limit – no benefit from increased spatial resolution.



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## Evaluating the Execution

I didn't know there was going to be a grade!



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## Defensible Data Generated When

The sampling design's primary objective is:

Collection of data that are scientifically valid and legally defensible.

Data are deemed acceptable when:

BOTH sampling and analytical uncertainties have been managed to meet accurately defined project objectives.

While the ability to document the quality of the data is significant in determining the value of a data set,

the importance of the RELATIONSHIP between **data generation** and the **intended use** of the data cannot be overstated.



71

## Data Usability Assessments…

DUA Definition: Determination of the adequacy of data, based on the results of validation and verification, for the decisions being made.

IDQTF, UFP-QAPP Manual VI, March 2005 § 5.1



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## What Should We Do Now?

Knowledge is power only when acted upon.



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## Recommendations for Next Steps

- ✓ Involve the entire project delivery team – Federal (USACE, Navy, Air Force, etc.), Contractors, Laboratories, and State Regulators, early and often throughout the Data Quality Objectives Process.
- ✓ Quantify and report measurement and total uncertainty – improves overall decision making.
- ✓ Implement representative sampling designs/methodologies.
- ✓ Perform the Data Usability Assessment as an entire project delivery team – Federal (USACE, Navy, Air Force, etc.), , Contractors, and State Regulators.



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## References


- EPA G4 Guidance on Systematic Planning Using the Data Quality Objectives Process
  https://www.epa.gov/quality/guidance-systematic-planning-using-data-quality-objectives-process-epa-qag-4
- ITRC Incremental Sampling Methodology Update
  https://ism-2.itrcweb.org/
- Visual Sampling Plan is freeware software from the DOE Pacific Northwest National Lab
  http://vsp.pnnl.gov/
- Uniform Federal Policy for Quality Assurance Project Plans - Training Materials
  https://www.epa.gov/fedfac/uniform-federal-policy-quality-assurance-project-plans-training-materials
- Uniform Federal Policy For Quality Assurance Project Plans, Munitions Response QAPP Toolkit
  https://www.epa.gov/fedfac/uniform-federal-policy-quality-assurance-project-plans-munitions-response-qapp-toolkit




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# Data Usability Assessment and its role in Decision Making

Melinda McClellan, Ph.D.
Chemist
Huntsville Environmental and Munitions Center of Expertise
Melinda.S.Mcclellan@usace.army.mil



1

## Defensible Data & Project Decisions


| | |
|---|---|
| The Primary objective for a good sampling design is | Collection of data that are scientifically valid, representative of site conditions, and legally defensible |
| Data are acceptable when | Both sampling and analytical uncertainties have been managed to meet accurately defined project objectives |
| Documented data quality is important, however | The relationship between data generation and intended use of the data cannot be overstated |

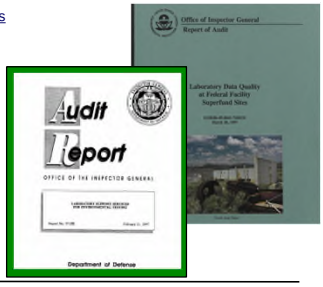


2

## Historical Perspective

1997 audits performed by EPA and DoD OIGs

- Data quality issues resulted in rework, greater cleanup costs, and time delays
- QAPPs were not well designed
- Oversight of lab data quality was lacking

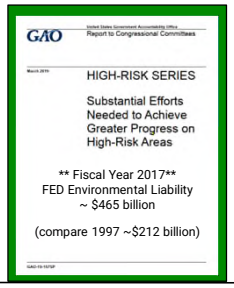





3

## Current perspective

- In 2017, GAO added the U.S. Government's Environmental Liabilities to the High-Risk List
- By 2019, only one of five criteria assessing progress was "partially met"

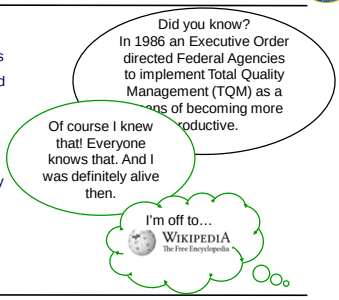





4

## What do we do?

- We need to:
  - Clearly Define our Project Objectives
  - Manage uncertainties in our data and decisions
  - Generate data that is scientifically valid and representative of site conditions
  - Generate data in a way that is legally defensible
  - Understand the quality of our data in the context of its intended use
- In other words, we need a quality management system!






5

## Down the Rabbit Hole

**Total quality management** (**TQM**) consists of organization-wide efforts to "install and make permanent climate where employees continuously improve their ability to provide on demand products and services that customers will find of particular value."

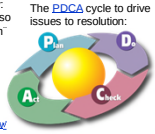
TQM enjoyed widespread attention during the late 1980s and early 1990s before being overshadowed by ISO 9000, Lean manufacturing, and Six Sigma.

In the [illegible] of 1984, an arm of the United [illegible] some of its civilian [illegible] recommendations as [illegible] Navy's operational [illegible] Navy branded the [illegible] gement" in 1985. [illegible] ad throughout the

The Navy used the following tools and techniques:

The seven basic tools of quality:
1.Cause-and-effect diagram (also known as the "fishbone diagram" or Ishikawa diagram)
2.Check sheet
3.Control chart
4.Histogram
5.Pareto chart
6.Scatter diagram
7.Stratification (alternatively, flow chart or run chart)

The PDCA cycle to drive issues to resolution:




United Sta [illegible] 88)
"Total Quali [illegible] partment of Defense is [illegible] eving performance at every leve [illegible] ility. It combines fundamental [illegible] sting improvement efforts, and spe [illegible] nder a disciplined structure focused [illegible] roving all processes. Improved performan [illegible] l satisfying such broad goals as cost, quality, schedule, and mission need and suitability. ..."

All images and images sourced from Wikipedia.



6

## PDCA by Any Other Name


**Plan**
- Develop QAPP, DQO's, MPCs

**Do**
- Follow Approved Project QAPP
- Collect and analyze samples

**Check**
- Data Verification
- Data Validation
- Data Usability Assessment (steps 1 and 2)

**Act**
- Data Usability Assessment (steps 3 and 4)
- Action based upon decision



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## Plan – Systematic Planning Process

- Starts with Systematic Planning Process and the UFP-QAPP Worksheets
  - WS #1-8, Management: Background, PDT, Communication Pathways, Training Requirements, Proof of Review, and Approval Pages
  - WS #9, Planning Sessions: Documents ALL project meetings and agreements
  - WS #10-16, Project Objectives: CSM & DQO Development, and Performance Objectives
  - WS #17-30, Design and Data Collection: Sampling and Analysis Methods, and Quality Control Requirements
  - WS #31-33, Assessment and Oversight
  - WS #34-37, Data Review



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## Do – Key Worksheets for Data Collection

- WS #12: Measurement Performance Criteria
  - Criteria that collected data must meet in order to satisfy the DQOs
  - Failures may impact end uses of the data
- WS #14/16: Project Tasks & Schedule
- WS #17: Sampling Design & Rationale
  - Process flow
  - Activities to obtain data
    - Site preparation
    - Sampling and analysis
- WS #15: PALs & Lab-Specific Limits
  - Detection & quantitation limits
- WS #18: Sampling Locations/Methods
  - Cross references sample types, locations, and methods
- WS #29: Project Documents & Records
  - QC/QA records
  - Reports
  - Field records



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## What Does the check part do?

CHECKS to see if you really DID what you PLANNED.


Data Quality is meaningful only when it relates to the intended use of the data.

Data Quality does not exist in vacuum.



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## Check – Data Review Process

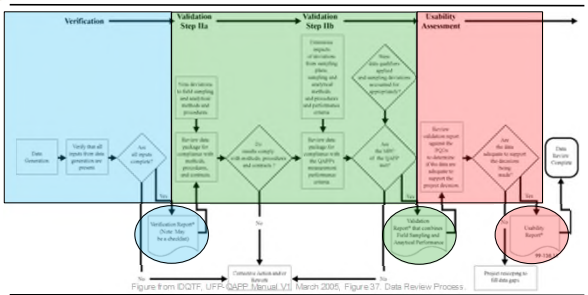


Figure from IDQTF, UFP-QAPP Manual, V1, March 2005, Figure 37. Data Review Process



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## Check – Data Review Steps Compared

| | Verification | Validation | Usability Assessment |
|---|---|---|---|
| Purpose | Ensure presence and completeness | Ensure compliance with underlying specifications (e.g., SOPs, Methods, QSM) | Adequate quality and quantity for decisions (DQOs, PARCCS) |
| Responsible Party | Varies, usually contractor | 3rd Party* Validators | Entire Project Team |
| Covers | Field records and laboratory data | Laboratory data | All project records and data |
| Timeline | Following collection | Following laboratory report issuance | End of sampling event, prior to decision making |



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## DUA Answers Four Questions

**The Four Questions**

1. Does the data quality allow a decision (or estimate) to be made with the desired level of certainty?
2. How well did the sampling design perform?
3. If the same sampling design strategy is used again for a similar study, would data be expected to support the same intended use with the desired level of certainty?
4. Were sufficient samples taken to reveal an impact if it was present?

**Why These Questions**

1. Does data provide evidence strongly in favor of one course of action over another. Yes, proceed. No, alerts decision makers to uncertainty.
2. If sampling design is very sensitive to disturbing influences, then results interpretation may be difficult.
3. As conditions vary from one location or one time to another, addresses robustness.
4. Determines if an impact may have been missed.

EPA QA/G-9, July 2000



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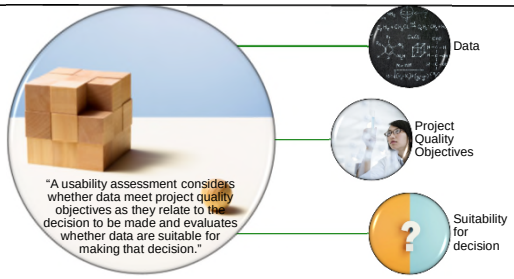
## Data Usability Assessment




IDQTF, UFP-QAPP Manual VI, March 2005 § 5.2.3



14

## DUA – a Definition

Determination of the adequacy of data, based on the results of validation and verification, for the decisions being made.

IDQTF, UFP-QAPP Manual VI, March 2005 § 5.1



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## Data & Intended Use – Compliance

**Example 1**

Wastewater effluent. Nitrate discharge permit = 10 mg/L. Sample = 0.26 mg/L.

The LCS acceptance criteria is 80 – 120%. For this batch of samples, recovery for the LCS was 78%, and the laboratory properly qualified the result with a "Q". The validator qualified the data as estimated, J-qualified.

Usability Assessment: Because the actual nitrate value was significantly lower than the permit limit, and the quality control failure was relatively minor, this result is judged to be usable.

**Example 2**

Wastewater effluent. Arsenic discharge permit = 10 ug/L. Sample = 9.5 ug/L.

LCS acceptance criteria is 75 – 125%. LCS recovery associated with this sample 65%.

Usability Assessment: These data are not usable to demonstrate compliance, due to the proximity of the sample to the action level and the low LCS recovery.

Florida DEP, Process for Assessing Data Usability, DEP-EA 001/07, March 2008 § 17



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## Data & Intended Use – In Context

**Example 1**

Four monitoring wells. Previous TCE concentrations ranged from 20-30 ug/L.

During this sampling event, TCE measured at the four wells was found to be below 1 ug/L. No laboratory QC failures were noted. However, sampling records indicated large volumes of groundwater were purged with a centrifugal pump in a short time period and dissolved oxygen levels exceeded 70%.

Usability Assessment: Because the evidence indicates that improper sampling occurred resulting in excessive aeration and degassing of volatile compounds, the data were determined to be unusable.

**Example 2**

Bombing range. Tetryl detected in soil samples above the project action limit.

No laboratory QC failures were noted. Tetryl has a lower control limit of 10% for LCS recovery as it is such a poor performer. Tetryl is only typically found at production facilities.

Usability Assessment: CSM was revisited and historical data reviewed. Laboratory was asked to investigate possibility of sample contamination. Determined samples were spiked with MS/MSD standard. PDT explained likely source of detections and that true presence of tetryl was unlikely; however, regulators requested resample.



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## Data and Intended Use – Statistics

Conceptual Site Model

Electronic Manufacturing Corporation of America operated at the site from 1965 to 1985 and sold the site to Energy Components Company in 1985. Both companies went bankrupt in 1990.

Waste oil contaminated with PCBs was sprayed on a dirt road on the site for dust suppression while the site was operational.

In 1991, chlorinated solvents were discovered in water from city wells located in a field east of the site.

Problem Statement

PCB contamination along the road may present an unacceptable risk.

Decision statement: hypotheses test

If mean concentration of PCBs in top 1" of surface soils exceeds 2 mg/kg, then take remedial action.

If mean concentration of PCBs in top 1" of surface soils does not exceed 2 mg/kg, then no action is necessary.

Null Hypothesis: True Mean $\leq$ 2 mg/Kg
vs.
Alternative Hypothesis: True Mean > 2 mg/Kg



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## Data and Intended Use – Statistics

**Results**

PCB concentration levels were measured (in mg/Kg) from 16 surface soil samples (top one inch of soil) from the dirt road. Each soil sample consists of 5 mini-samples composited together.

| | | | |
|---|---|---|---|
| 1.92 | 2.49 | 4.58 | 1.17 |
| 2.48 | 5.62 | 2.54 | 25.15 |
| 7.72 | 1.02 | 2.91 | 3.23 |
| 2.87 | 8.66 | 1.71 | 1.18 |

The mean is 4.703 mg/kg, exceeding the project action limit of 2 mg/kg.

**Usability Assessment:**

Are all these values representative of site conditions? Are there any outliers?

The result of 25.15 looks suspect, and the standard deviation of the data set is 5.900 mg/kg.

Using statistical guides, such as EPA G9, the team evaluated the summary statistics, coefficients of variation and skewness, outlier evaluation, and verification of statistical assumptions.

It was determined that statistically the mean concentration exceeded 2 mg/kg and remediation was necessary.



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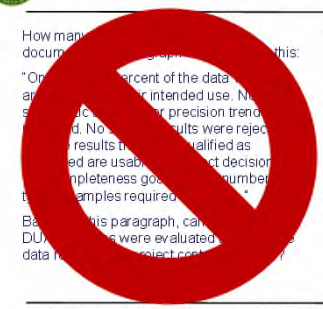
## DUA – A Semi-Current Example



Why have we accepted these paragraphs?

- True DUAs take effort
- DUAs have not been well understood
- Data validation was convoluted with data usability
- No one wants to write (or read) more about the chemistry results than they have to…

How do we do better going forward?

We need a process, with well defined steps, with well defined inputs and outputs.



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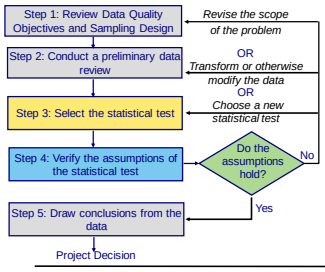
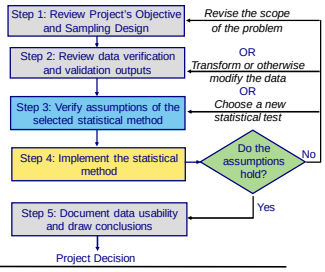
## Five Step DUA Process

EPA QA/G-9, July 2000§0.3




IDQTF, Optimized UFP-QAPP Worksheets, March 2012



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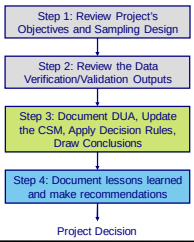
## Four Step DUA Process

IDQTF, UFP-QAPP Munitions Response Toolkit
Module 1, April 2020 WS #37



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## Let's Do A DUA

Portrait by Joseph Duplessis, 1778

"Tell me and I forget, teach me and I may remember, involve me and I learn."
-Benjamin Franklin



23

## Personnel Involved

**Entities**

- Lead Organization
- Contractor
- Subcontractors
- Stakeholders/Regulators

Each entity brings their own personnel and subject matter experts (SMEs) to the table

**Personnel**

| | |
|---|---|
| Project Managers | Risk Assessor |
| Technical Managers | Chemist |
| Resource Managers | Statisticians |
| Geologist | Analysts |
| Hydrogeologist | Field Samplers |
| Geophysicist | Safety Personnel |



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## Logistics of DUA Meeting

| In Person | Virtual – Real Time | Virtual – Asynchronous |
|---|---|---|
| • Government PM with contractor schedules, hosts, and facilitates DUA meeting<br>• Recorder documents responses to questions and distributes draft for review | • Government PM with contractor schedules, hosts, and facilitates teleconference<br>• Recorder documents responses to questions and distributes draft for review | • Designated POC may send out email request for feedback on DUA Steps/ Questions<br>• POC assembles responses and distributes draft for review |



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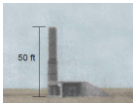
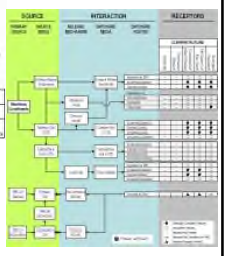
## Our Project

Recall the Incinerator and Landfill Site:

- Former incinerator used to burn household and base waste. Slag and ash dumped at landfill.
- Concerns were potential lead contamination in open recreational and potential residential site
- EPA RSL for Lead in Residential Soil: 400 mg/kg.





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## Step 1 – Review Project's Objectives and Sampling Design

- Was the problem statement correct? (DQO Step 1)
- Were study questions complete? (DQO Step 2)
- Were all necessary data inputs identified? (DQO Step 3)
- Were study boundaries (spatial and temporal) adequately defined? (DQO Step 4)
- Was the data collection approach appropriate to address the problem statement and study questions? (DQO Step 5)
- Were decision rules specified? Were alternative outcomes and their consequences clearly explained? (DQO Step 5)
- Were MPCs sufficient to address DQO Steps 1-5 and ensure the satisfaction of all data quality indicators? (DQO Step 6)
- Did the sampling design address all MPCs? Were data reduction procedures and statistical methods specified and relevant to the problem statement? Was data reduction performed in accordance with specifications? Can the data be used as intended? (DQO Step 7)

Thanks, Allyson



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## A Look at the Sampling Design

Recall our sampling design:

- ISM samples for lead in surface and subsurface soils
- 10 DUs, each of which represent residential exposure units. 1 DU = 1 SU. Each DU sampled in triplicate.
- Background DU taken in same manner at unaffected location.

Take a moment to think about assumptions:

- Soils in the surface and subsurface may be impacted and will drive the risk assessment. Soils below the subsurface will not significantly contribute to the risk assessment for residential soil or leaching to groundwater.
- DUs were appropriately established.
- Soils in area of concern are comparable to soils in background area. Background area is free of contamination.
- Results from replicate measurements will be normally distributed.
- Can you think of other assumptions?



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## A Look at the Data

CoC Information:

- Day 1:
  - Sampled 05, 10, 09, 04
- Day 2:
  - Sampled 03, 08, Bkg DU
- Day 3:
  - Sampled 02, 07, 06, 01, decon blank

Results

- Means and Standard Deviations for individual DUs shown at the right
- 4 DUs nominally exceed PAL of 400 mg/kg

| | | | | |
|---|---|---|---|---|
| DU 01<br>314 mg/kg<br>StDev:<br>132 mg/kg | DU 02<br>482 mg/kg<br>StDev:<br>121 mg/kg | DU 03<br>512 mg/kg<br>StDev:<br>179 mg/kg | DU 04<br>389 mg/kg<br>StDev:<br>74 mg/kg | DU 05<br>214 mg/kg<br>StDev:<br>45 mg/kg |
| DU 06<br>287 mg/kg<br>StDev:<br>37 mg/kg | DU 07<br>443 mg/kg<br>StDev:<br>230 mg/kg | DU 08<br>559 mg/kg<br>StDev:<br>129 mg/kg | DU 09<br>270 mg/kg<br>StDev:<br>165 mg/kg | DU 10<br>255 mg/kg<br>StDev:<br>41 mg/kg |

| |
|---|
| Bkg DU<br>250 mg/kg<br>StDev:<br>35 mg/kg |



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## Step 2 – Review Data Verification And Data Validation Outputs

Observations from the field team

- Sampling team noticed what appeared to be lead slag in the soil below the sampling depth

Observations from the DUA Team

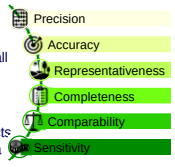
- Background concentration of 250 mg/kg is significantly higher than expected.

Observations from Laboratory and Validator

- Bottle for decon blank was broken upon receipt and could not be analyzed.
- MS and MSD performed on DU 04. Results outside lower acceptance criteria for MS recovery. Parent sample flagged J-.
- %RSD for field sampling triplicates exceeded acceptance criteria for 4 samples. Results Flagged J.

Questions to ask:

- Were MQOs achieved?
- Was the impact of all non-conformances adequately explained?
- What are the impacts on the data set as a whole?



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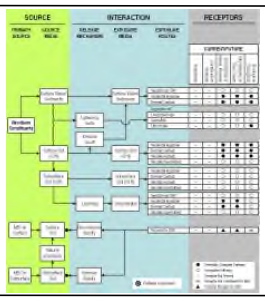
## Step 3: Document DUA, Update the CSM, Apply Decision Rules, Draw Conclusions

Document the DUA

- Follow WS 37
- Final data tables with qualifiers
- Narrative comments
- Limitations on the use of data

Update the CSM

- Remember, the CSM is a living document
- Did we learn new information – hopefully!



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## Step 3: Document DUA, Update the CSM, Apply Decision Rules, Draw Conclusions

Apply Decision Rules

Decision Rule 1

- If the true mean lead concentration within a residential decision unit is greater than 400 mg/kg to a depth of 2' below land surface, then the soil will be remediated.
- If not, then the soil will be left in situ.

Decision Rule 2

- If the true mean lead concentration within the former incinerator area or landfill footprint is greater than the groundwater leachability concentration to a depth of 10' below land surface, then temporary monitoring wells will be installed to investigate groundwater contamination.
- If not, then no further action.



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## Step 3: Document DUA, Update the CSM, Apply Decision Rules, Draw Conclusions

Draw Conclusions:

Must the soil be remediated?

What questions remain and what data do we need to solve them?

Do Not Forget to Write it Down

Draw any conclusions from the data and document how these conclusions follow from the data in a scientifically and legally defensible manner.

Consider both conclusions arising from the analytical data and those which arise from secondary data.



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## Step 4: Document lessons learned and make recommendations

Lessons Learned and Recommendations

- Document any project level lessons learned
- Document any sample-specific lessons learned
- Document any program level lessons learned
- Document any additional items which need clarification or consideration.

Prepare a Report

- This will create a record of your efforts, findings, final qualifications, and results. Along with the context needed to understand them.
- This report can be incorporated into the body of your final report deliverable or included as an appendix.



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## Just for fun… if there's time

We are going to take 1-minute for you to share examples where:

A data validator X-qualified a data point, but the PDT determines the data is usable

A data validator says the data is usable, but the PDT rejects the data as not usable



35

## What have we learned?

Data Usability Assessment is a distinct part of data review with a distinct purpose

Data Usability Assessment is a wholistic look at all available data in the context of project objectives

Data Usability Assessment involves the whole project team, not just the chemist

The Data Usability Assessment should the documented in a way that shows how the project decisions are scientifically valid and legally defensible



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## References – Process and Planning

- Process and Planning
  - IDQTF, UFP-QAPP Part 1: UFP-QAPP Manual, March 2005
  - IDQTF, UFP-QAPP Optimized UFP-QAPP Worksheets, March 2012
  - IDQTF, UFP-QAPP Munitions Response QAPP Toolkit Module 1: Remedial Investigation (RI)/ Feasibility Study (FS) Update 1, April 2020
  - IDQTF, UFP-QAPP Munitions Response QAP Toolkit Module 2: Remedial Action, March 2023
- Validation and Verification
  - EPA Guidance on Environmental Data Verification and Data Validation, EPA QA/G-8, November 2002
  - EDQW, General Data Validation Guidelines, September 16, 2019
  - EDQW, Data Validation Guidelines Module 1: Data Validation Procedure for Organic Analysis by GC/MS, May 11, 2020
  - EDQW, Data Validation Guidelines Module 2: Data Validation Procedure for Metals by ICP-OES, May 11, 2020
  - EDQW, Data Validation Guidelines Module 3: Data Validation Procedure for Per- and Polyfluoroalkyl Substances Analysis by QSM Table B-15, May 1, 2020
  - EDQW, Data Validation Guidelines Module 4: Data Validation Procedure for Organic Analysis by GC, March 9, 2021
  - EDQW, Data Validation Guidelines Module 5: Data Validation Procedure for Metals by ICP-MS, November 09, 2022
  - EDQW, Data Validation Guidelines Module 6: Data Validation Procedure for Per- and Polyfluoroalkyl Substances Analysis by QSM Table B-24, October 18, 2022
- Usability
  - EPA Guidance for Data Usability in Risk Assessment (Part A), April 1992
  - EPA QA/G-9 Guidance for Data Quality Assessment, July 2000
  - EPA QA/G-9R Data Quality Assessment: A Reviewer's Guide, February 2006
  - EPA QA/G-9S Data Quality Assessment: Statistical Methods for Practitioners, February 2006
- Additional, service specific guidance is available

# DUA "Case Studies" and Interactive Activities

William (Ed) Corl, Ph.D.
Navy Laboratory Quality and Accreditation Office

Brian Jordan
ODASD (Environment & Energy Resilience)



1

## Objectives

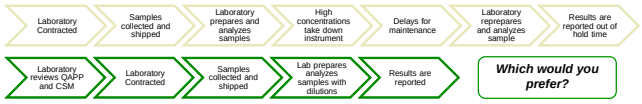
- Goals:
  - Explore workflows and communication pathways
  - Learn from one-another about how to improve
  - Take lessons into future projects
- Format:
  - Case Studies
  - Small Group Brainstorming
  - Large Room Discussion



2

## Which one sounds better?

Scenario: A project is analyzing sample from a creosote plant




***Which would you prefer?***

What are the differences in these workflows?

Take 3 minutes in your group to brainstorm as many as possible.

Think about: planning, execution, outcomes, or anything else



3

## Differences in Workflows

- Laboratory involved from beginning
- diluted before running on instrument
- no instrument went down
- Laboratory had approved documents ahead of time
- Results faster
- Can check for capacity to plan for instrument maintenance
- No qualified data for holding time
- Laboratory should have subcontracted when they knew they would be down
- Project knows what they want early



4

## Which one sounds better?

Scenario: A project is analyzing sample from a creosote plant


What are the differences?

- Communication earlier
- Communication more thorough
- Laboratory spends more time up-front
- Laboratory spends less time with instrument down for maintenance
- Results arrive sooner after sample collection
- No hold time violations



5

## Can you think of a time when…

- A laboratory ran into a problem with a sample, a batch, or a shipment of samples…
  - Could the problem have been mitigated by laboratory involvement earlier in the planning or sampling process?
- Is there information you, as a laboratory or validator, are not being provided currently that you need?
  - During Bidding?
  - Before sampling?
  - Before receipt?
  - Before preparation and analysis?
  - About sampling procedure?
  - About historical data?



6

## A Small Case Study

Scenario: A project is analyzing sample from a munitions site for metals


Where were the breakdowns in communication or planning? What could be improved?



7

## A Small Case Study

**Breakdowns**

- Laboratory not involved in QAPP planning to get proper method
- Validator did not flag – correct because of native concentration – but not narrated in validation report – DUA team does not understand why it wasn't flagged

**Improvements**

- Discuss in narrative of analytical package and validation package
- Laboratory discuss with client when the sample didn't look homogeneous – field should also note observations



8

## Path Forward

- Now that we know what we want… effective communication for good data quality
- How do we get there?
  - Take 5 minutes in your group to brainstorm as many as possible.



9

## Path Forward

- How do we get there?
  - MR-QAPP requires government to prepare Worksheet 10 [CSM] and 11 [DQOs through step 4] before RFP – could this help with all types of projects?
  - Initial communications clearly setting forth the requirements and timelines for both electronic and hard-copy deliverables
  - Laboratory given opportunity to review and comment on project QAPP
  - Validators encouraged to have open communication with project as technical experts
  - Open and transparent lines of communication on data status and potential speedbumps, not just disasters.
  - Government SMEs communicate to source selection boards the importance of quality and timeliness of services provided by primes, laboratories, and validators



10

## Call to Action

- What can we do?
  - Set clearer expectations
  - Communicate needs
  - Rethink contracting processes to prioritize quality – and pay accordingly
- What can you do?
  - Identify barriers to quality in services
  - Exercise communication pathways and establish new ones
  - Tell us what you need



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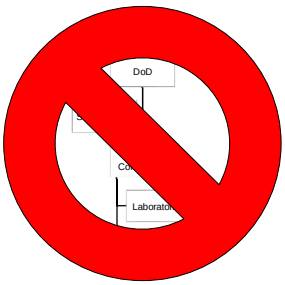
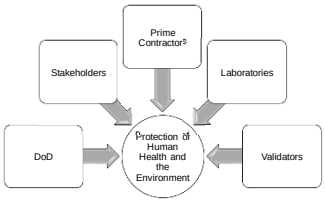
## We Want to Build Partnerships




**The New Way**






12

## We Want to Build Partnerships

If any one of the partners or stakeholders isn't a part of the discussion, we are underrepresenting the value they can bring to the process.

### The New Way