Introductions

Number of Samplers in the Room?

Background of people in the room.

Do you work with Samplers – Engineers?

What does good practices in sampling look like?

Kim Watson - RQAP-GLP, 37 Years Field & Lab Experience
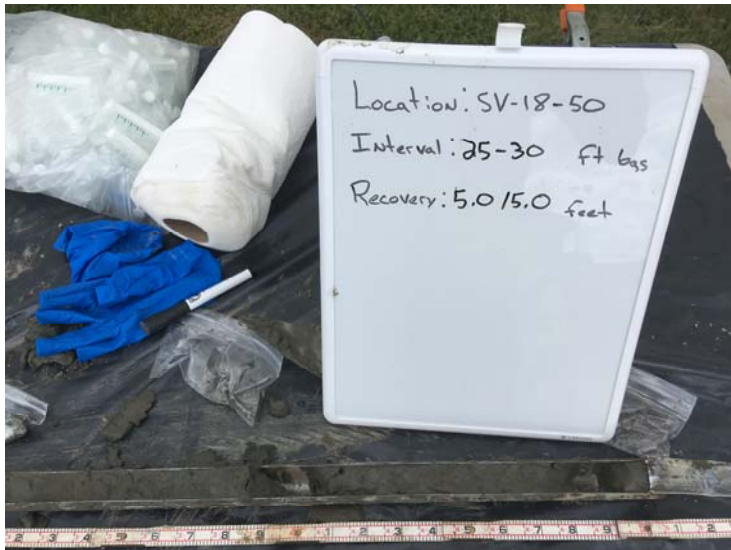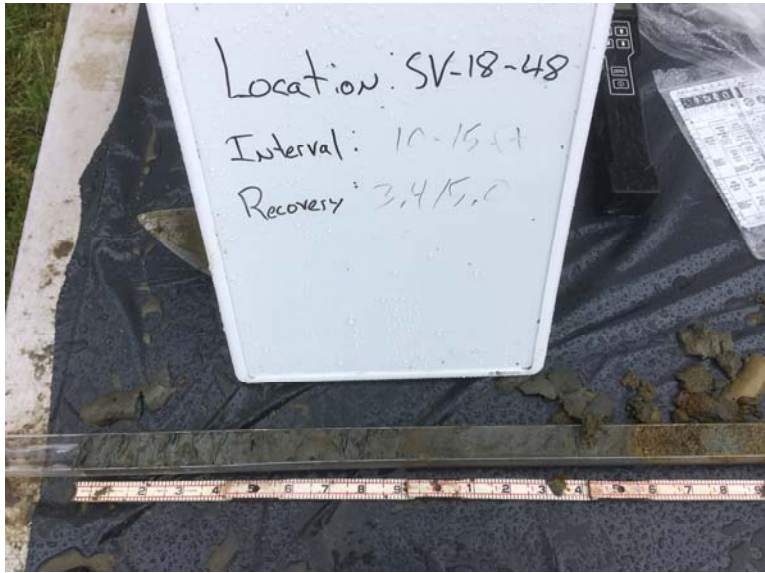
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Slide 2

KD1   Suggest Changing "Instructor" to "Presenter".
Kirstin Daigle, 7/18/2018

Objective: Steps…

Understanding a Project Life Cycle

Project Plans: Planning Process for Data Collection Activities

Project QAPP, UFP-QAPP

Sampling Plans: SOPs

Representative Sampling Concepts

Statistical Sampling Concepts

Sampling Errors

Example of Incremental Sampling – Soil, Cannabis, Food
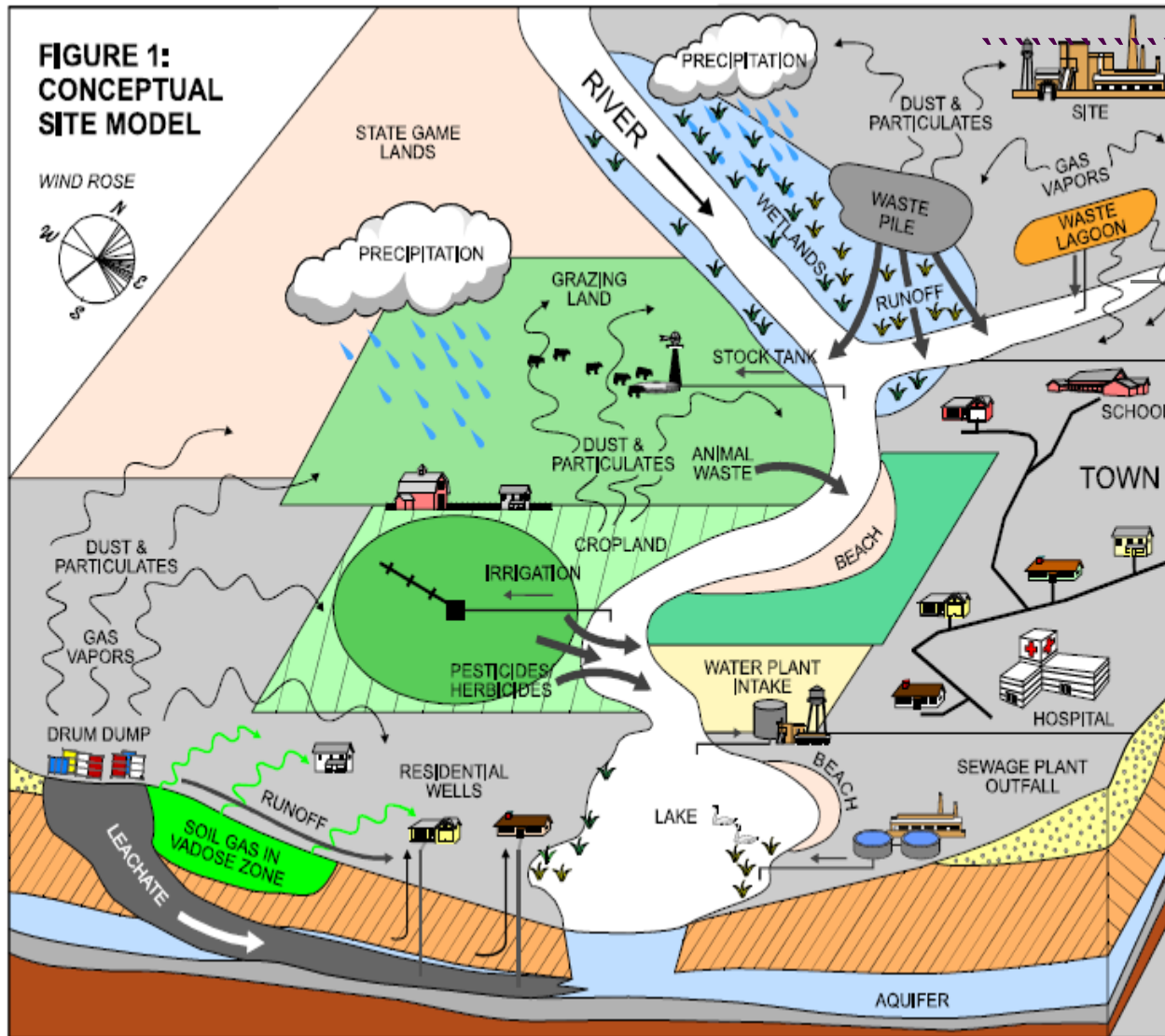
Quality systems: ISO 17025;2015, NEFAP, NELAP

Technical Requirements of Samplers

    What is competency?

SOPs

Field Audits

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Understand the Project Life Cycle

FIGURE 1: CONCEPTUAL SITE MODEL

Definition of the problem
- Project Life Cycle
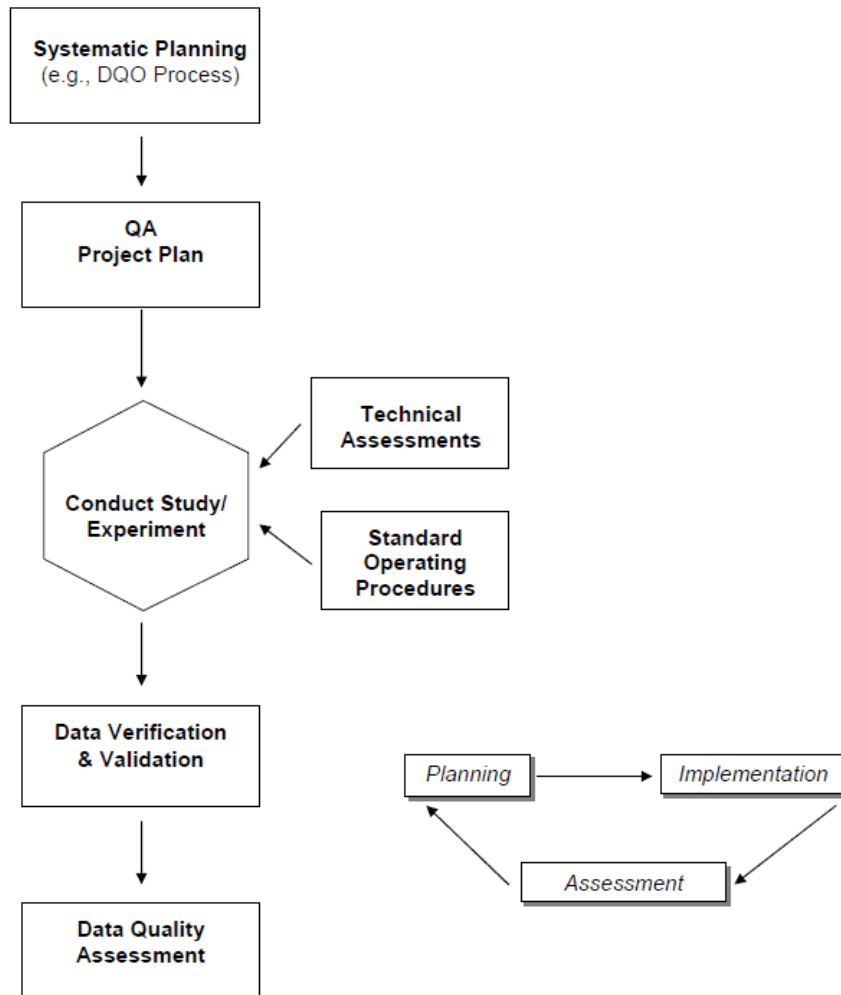- Technical and quality

Success of an environmental program or project depends on the quality of the environmental data collected and used in decision making, and Quality and adequacy of the QAPP*, & Implementation

*SAP, Work Plan, Field Sampling Plan. Blueprint, CSM

Project Quality Plans and Objectives

FIGURE 1-1

PROJECT LEVEL EPA QA SYSTEM

Systematic Planning (e.g., DQO Process)

QA Project Plan

Conduct Study/ Experiment

Technical Assessments

Standard Operating Procedures

Data Verification & Validation

Planning → Implementation → Assessment

Data Quality Assessment

SW-846 Update V   ONE - 14   Revision 2 July 2014

Project Specific QAPP

Generic QAPPs

4 basic element groups addressed in a UFP-QAPP:
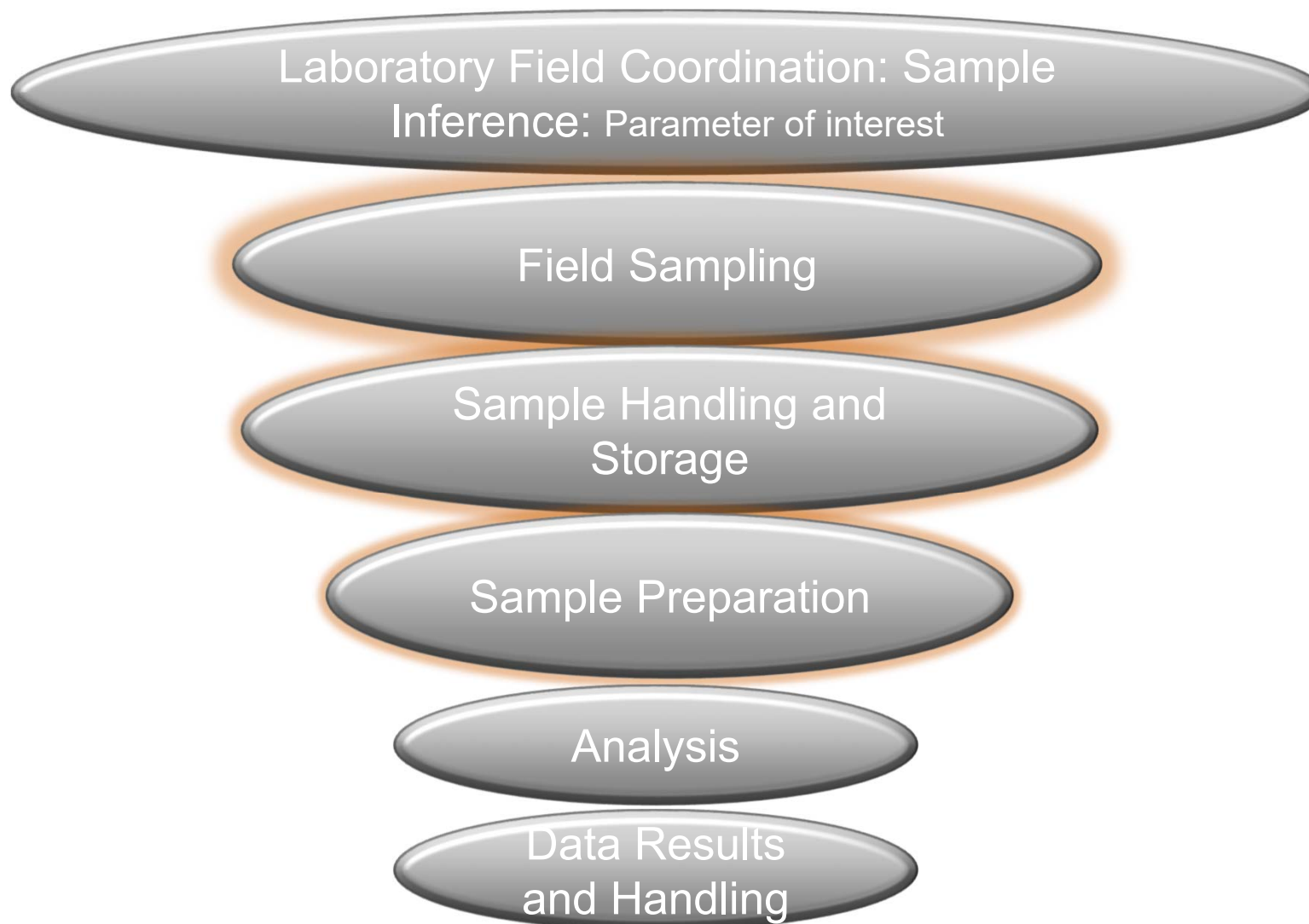
- Project Management and Objectives
- Measurement/Data Acquisition
- Assessment/Oversight
- Data Review

Puzzle

Project Management and Objectives

Scoping Se...

Assessment/ Oversight

Data Review

Measurement/ Data Acquisition

UFP QAPP 02/17/06

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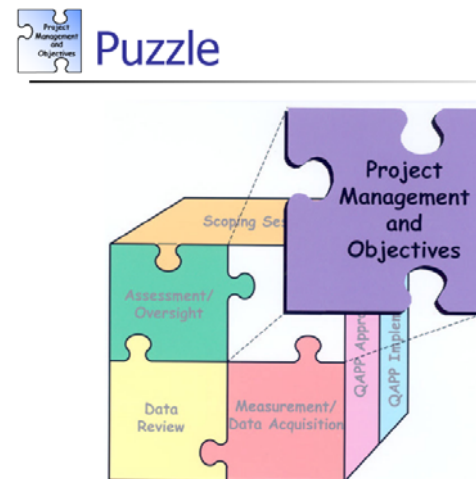
Why should Project QAPP emphasize sampling?

Because analytical data is only as good as the quality of the sample from which the data is obtained; thus decisions made from that data are highly dependent on the quality of the sample. What is the weakest link?

Laboratory Field Coordination: Sample
Inference: Parameter of interest

Field Sampling

Sample Handling and Storage

Sample Preparation

Analysis

Data Results and Handling

How do we get representative samples from the different matrices; air, water, soil, biota, food, & plants

Representative Sampling Planning

A representative sampling plan is a written document that describes the objectives and details of the sampling effort and how they will be performed.

1. Establishes the objective for sampling and analysis (i.e. parameter, DQOs).
2. Define the sample (matrix, size, type)
3. Document the statistical sampling design
4. Details the sampling procedures that will be followed
   – What SOPs will be followed?
   – What SOP modifications will be employed?
   – What tools and equipment will be used?
   – What QA/QC practices must be followed?
   – What steps must be taken for EHS & PPE?
   – Defines COC & Transport Responsibility, Storage, Handling
5. Include Site-Specific Considerations, field contaminates (PFAS; clothing)
6. What else?

Sampling Plans

Common Objectives:

- **Statistically Designed Sampling Plans**
- **Scientific and systematic approach**
- Probabilistic sampling
- Standard Sampling Methods applied with <u>consistency</u>
- Sampling Practices, including sampling tools, that are "correct" for matrix
- Control of Sampling Error
- Documentation

"The Quality of the Data Can Only be as Good as the Quality of the Sample"
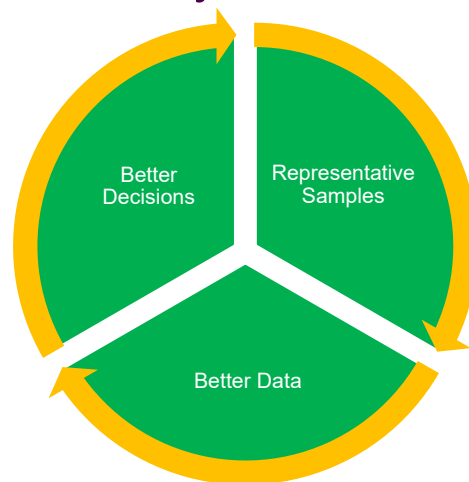
Representative & Random Sampling
Basic Concepts

Representative: *Samples that exhibit average properties of the whole*

- Accuracy: *The closeness of a sample value to its true value*
- Precision: *The closeness of repeated sample values*

Representative Sample means a sample obtained according to a sampling procedure designed to ensure that the different parts of an increment, decision unit, batch or the different properties of a batch or lot are proportionally represented.

The primary objective of sampling is to collect samples that will allow analytical measurements to be sufficiently accurate and precise. When chemical measurements are sufficiently accurate and precise, the results are considered reliable estimates of the chemical properties of the matrix.

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Statistical Concepts

Confidence Interval: A range of values so defined that there is a specified probability that the value of a parameter lies within it.

- Mean concentration
- Standard deviation of the mean

These values estimate the interval in which the true mean of the chemical concentration probably occurs under the assumption of a normal bell shaped curve.

CI is often expressed as a percent.

If the CI is 95%; if the sample is representative, the true mean is expected in about 95 out of 100 samples. (QA/QC section)

➢ *Most plans specify that the statistical design of the sampling plan must meet the CI requested by the client.*

Random Sampling

In random sampling, every unit in the population has a theoretically equal chance of being sampled and measured

Probability Sampling

- Simple Random
- Stratified Random
- Systematic Random

Sampling plans should use a random number generator to identify which location(s) to collect sample increments.

What type of probability sampling does random number generator fall under?

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Examples of Random Sampling Techniques

Just as with discrete sampling, a variety of sampling methods may be implemented with ISM sampling. One of the more common approaches in ISM is systematic random sampling (a.k.a., systematic grid sampling [Gilbert 1987]), where the DU is divided in a grid pattern, a random sampling location is identified within the first grid cell, and then samples
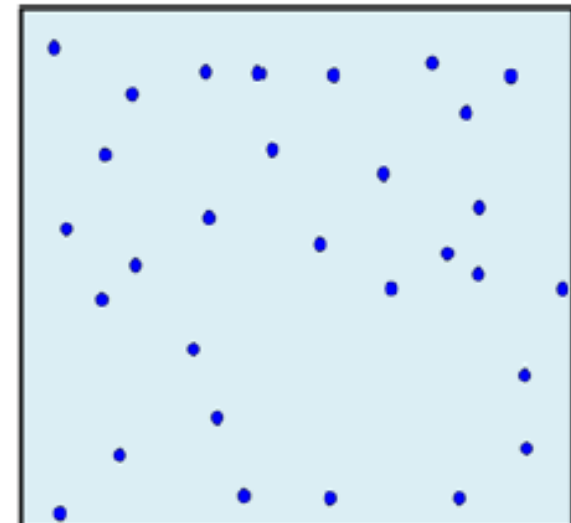
Simple random sampling, systematic random sampling, and systematic grid sampling yield unbiased estimates of the mean. The systematic sampling patterns ensure relatively even spatial distribution of samples across the site and are generally easier to implement in the field.
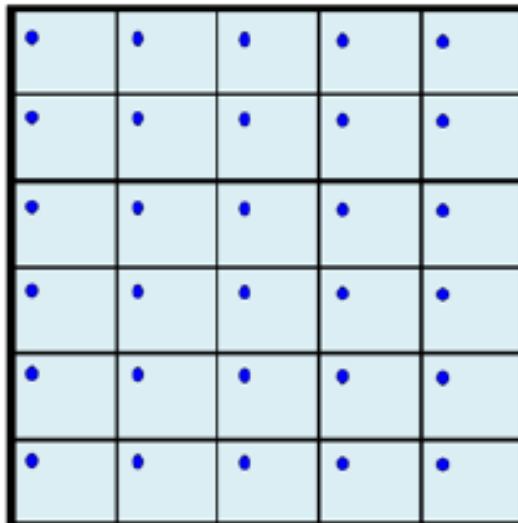
Figure 4-7. Systematic random sampling/ systematic grid sampling with a random start (Serpentine).
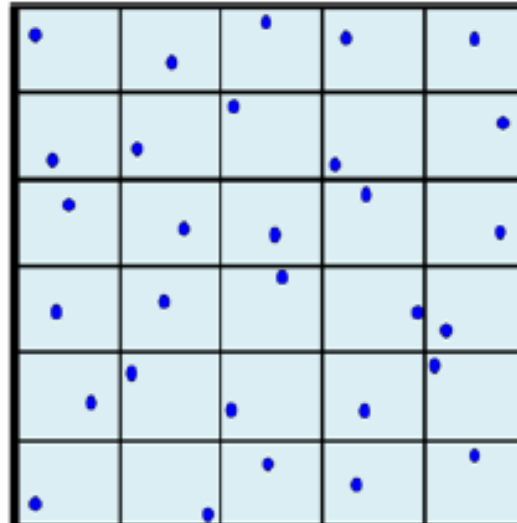
Figure 4-8. Random sampling within grids.

Figure 4-9. Simple random sampling within the entire DU.

Representative & Random Sampling Review

Accuracy is achieved by random sampling.

Precision is achieved by taking an appropriate number of samples (increments) from the population.

Random Sampling: Every unit in the population has a theoretically equal chance of being sampled and measured.

Sampling accuracy and precision may be increased by maximizing the physical size (weight or volume) of the samples that are collected and by increased the number of samples taking from a population.

Authoritative Sampling

Real World Question: A lab is presented with 30 pounds of soil from which to take 10 increments to create a composite sample within a minimum weight of 30 g. Should the sampler select only the soil from the top of the bag to make up the mass weight in order to keep harder soil clay soil intact?

What do you think? Is this type of sampling accurate and precise?

When the sampler selects the locations to sample without regard to randomization, the sampler is performing what we call authoritative sampling; authoritative sampling is not random.

Probabilistic sampling: A sampling strategy in which all material has an equiprobable chance of being collected.
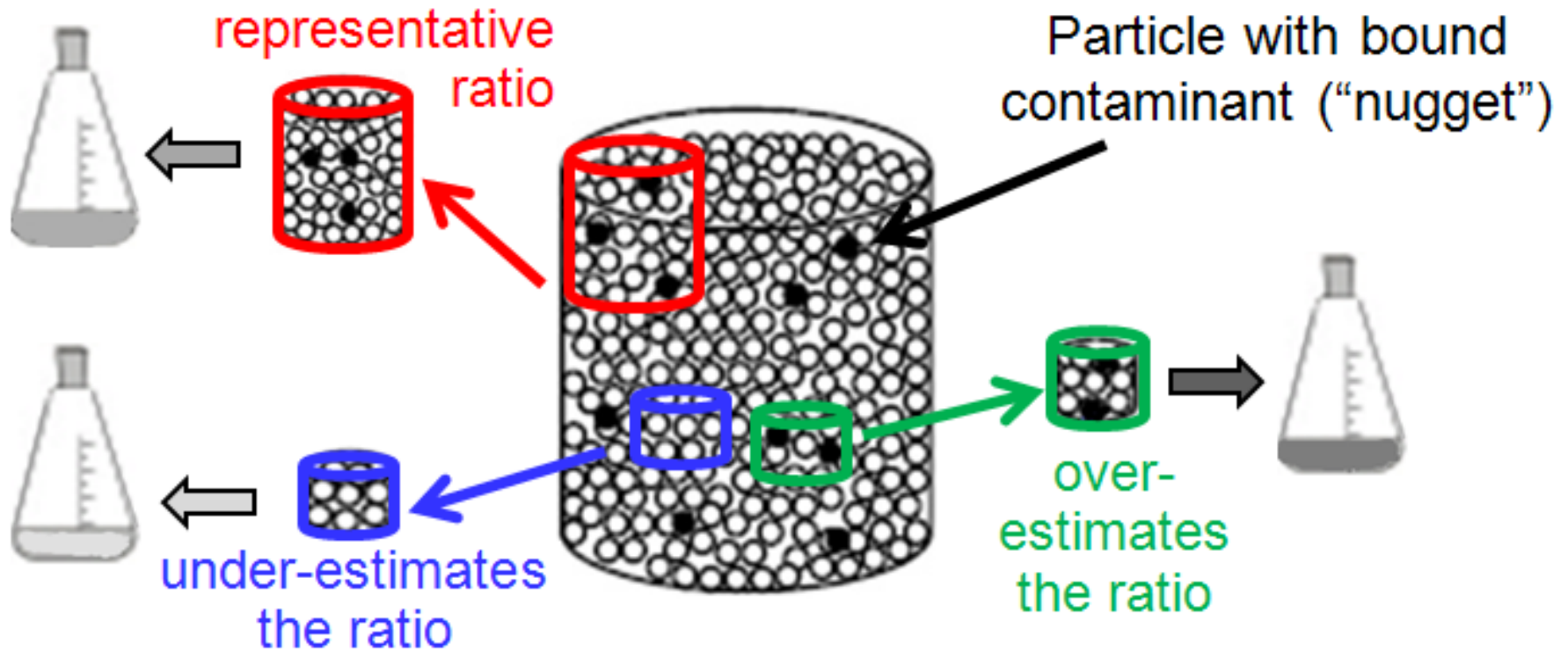
How
to?

Random Sampling

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When sampling and analyzing soil, sampling error is generally **much greater** than analytical error.

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Illustration of Sampling Error

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Best Practice: Collect Adequate Sample Mass

Minimum of 30 increments (i.e. 30 borings/cores)

May need ~100 increments to provide desired statistical significant

Increment mass: 20-60 grams (~2 ounces)

Final DU Sample: 600-2,500 grams (~1 pound)

Collect Replicate Samples

Minimum of 3 replicate samples – satisfies QC objectives (i.e. field duplicates)

Each replicate is an independent sample – not a "split" sample

Collecting the Samples

Replicate 1

Replicate 2

Replicate 3

EXAMPLE - Problem:
How to rapidly assess soil for both environmental quality and representativeness beneath the proposed grocery store, without analyzing a huge number of samples?

Incremental Soil Sampling Approach

~2.5 days with Geoprobe
Three vertically stacked "Decision Units"

      0.5 feet (foundry waste)

      2 feet

      4 feet
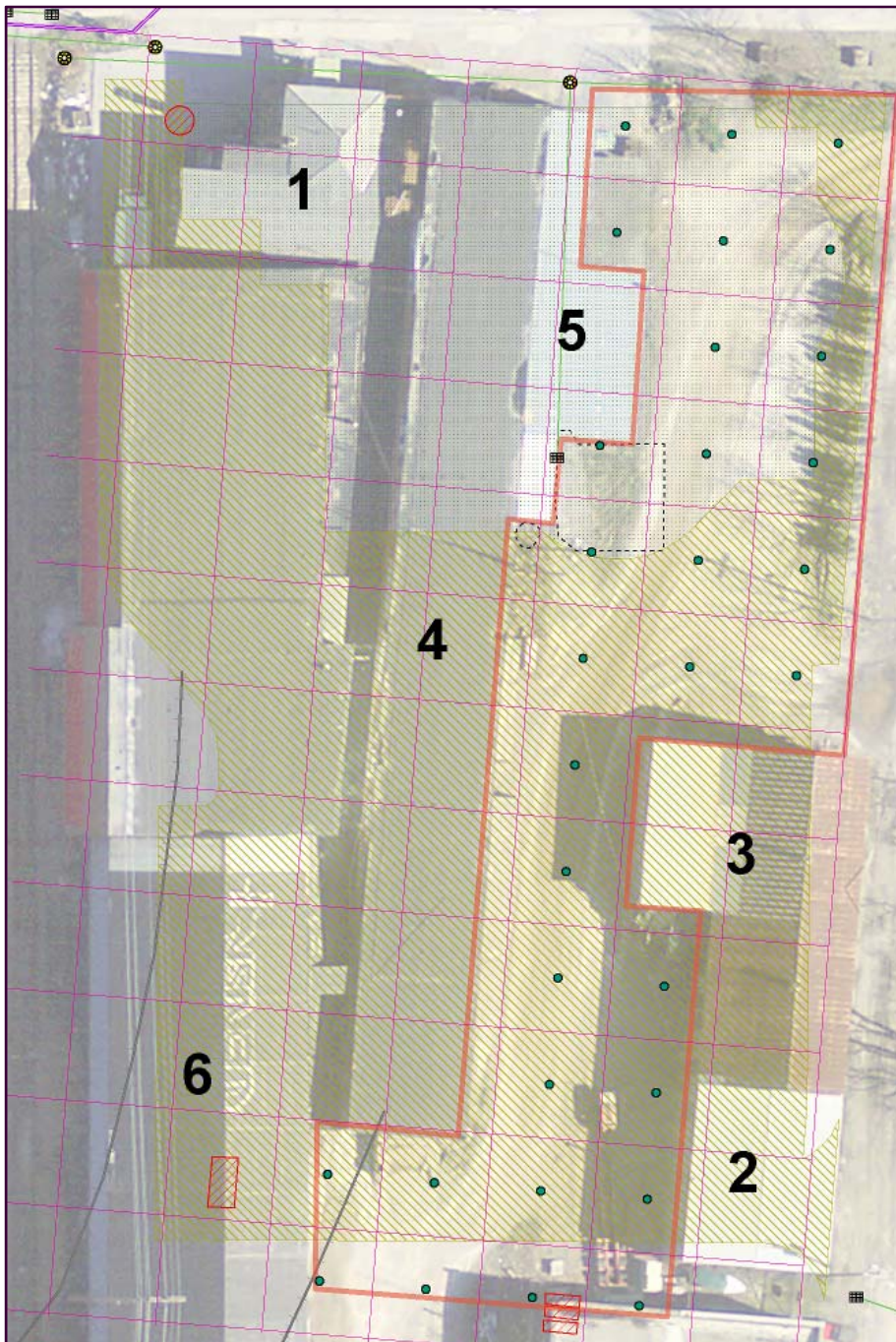
Sampled for Metals, PAHs, PCBs

Foundry fill = high spatial variability (high heterogeneity)

Goals:

(1) Identify mean concentrations

    (2) Soils accepted by a disposal facility

Implementation

← **Field Maps**
(32-ft grid; ~30 increments)
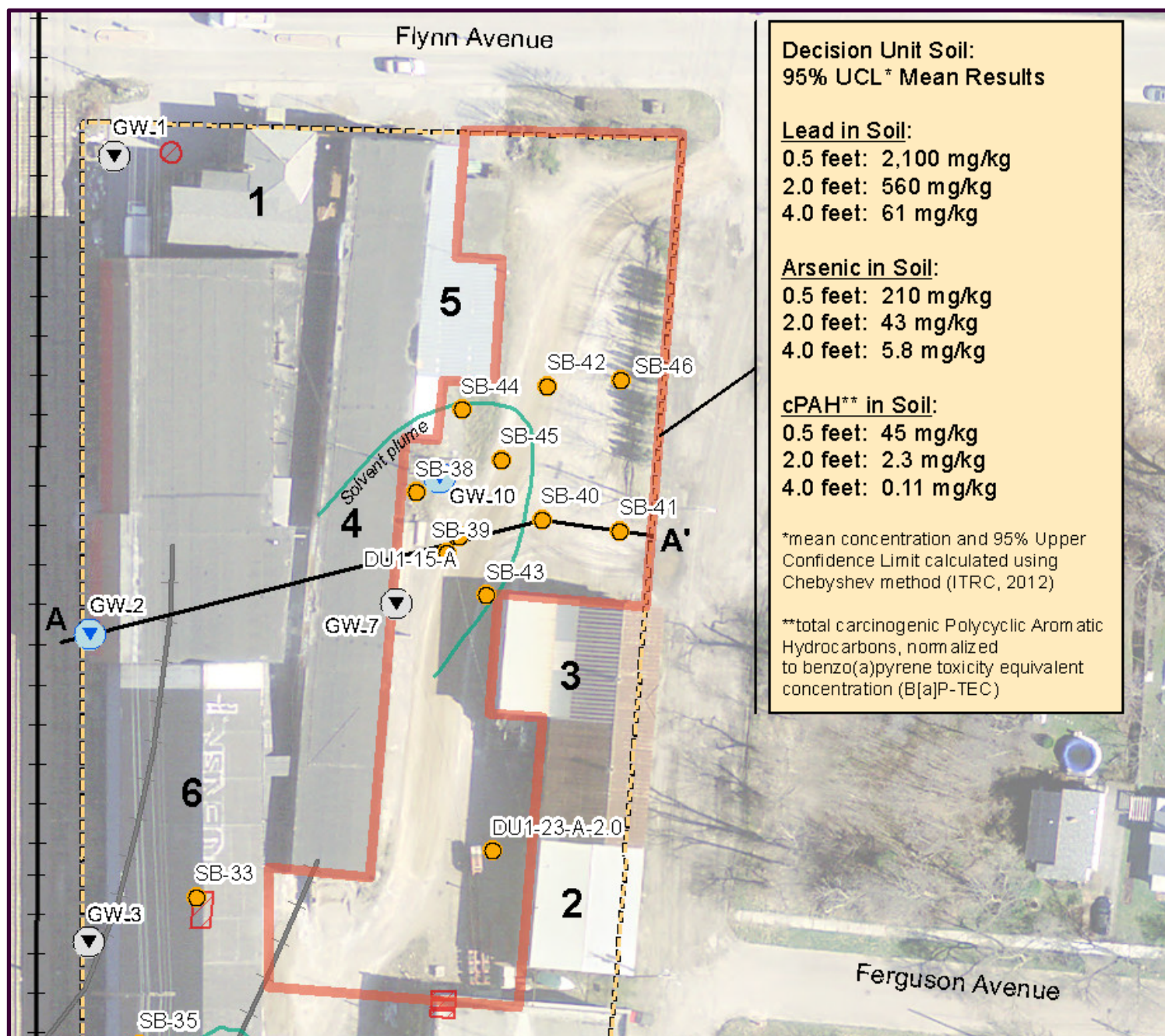
Sub-Foot GPS

Collect >2 kg of soil per DU

Sub-Sample in the Field

Submit to Lab

Calculate 95% UCL Mean

Results / Assessment

Decision Unit Soil:
95% UCL* Mean Results

Lead in Soil:
0.5 feet: 2,100 mg/kg
2.0 feet: 560 mg/kg
4.0 feet: 61 mg/kg

Arsenic in Soil:
0.5 feet: 210 mg/kg
2.0 feet: 43 mg/kg
4.0 feet: 5.8 mg/kg

cPAH** in Soil:
0.5 feet: 45 mg/kg
2.0 feet: 2.3 mg/kg
4.0 feet: 0.11 mg/kg

*mean concentration and 95% Upper
Confidence Limit calculated using
Chebyshev method (ITRC, 2012)

**total carcinogenic Polycyclic Aromatic
Hydrocarbons, normalized
to benzo(a)pyrene toxicity equivalent
concentration (B[a]P-TEC)

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Example of Representative Sampling – Cannabis..

Sampling Guidelines

1. **Check for Non-Uniformity**
   - Containers & Labeling
   - Appearance of Product
     – Color
     – Shape
     – Size
     – Treatment
2. **Ensure Access Entire Batch**
3. **Take Equal Portions from the Batch**
   - Random or Systematic Collection
   - Take minimum number of increments
4. **Use Proper Sampling Equipment**
5. **Document**
   - Sampling Form

Hierarchy of Testing Items for Food

Figure 1a. Sampling terms illustrated for tomatoes.

Graphic from GoodSamples: Guidance On Obtaining Defensible Samples

FDA, AAFCO…and Food Industry

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Control of Sampling Error

Sampling error is controlled by selecting the appropriate mass and collecting the correct number of increments to address the compositional and distributional heterogeneity of the test item; and by maintaining the sample identify within the batch.

- Compositional Heterogeneity – heterogeneity arising from differing composition among individual elements in a DU

- Distributional Heterogeneity – heterogeneity arising from the non-random spatial or temporal distribution of elements in a DU

In what ways can the "Sampler" effect sampling error?

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SUMMARY QUALITY SYSTEM STANDARDS

International Standards

- ISO 17025 - General requirements for the competence of testing and calibration laboratories -2005-05-15

OECD Organization for Economic Cooperation and Development - GLPs

- OECD Series on Principles of Good Laboratory Practice (GLP) and Compliance Monitoring

National Programs

- NELAP – Laboratory Accreditations. The purpose of this program is to establish and implement a program for the accreditation of environmental laboratories.

- NEFAP – FSMO Accreditations. The purpose of the National Environmental Field Activities Program (NEFAP) is to establish and implement an accreditation program for field sampling and measurement organizations (FSMOs). http://www.nelac-institute.org/

- EPA - GLPs – 40 CFR Part 160 Good Laboratory Practice Standards

QUALITY SYSTEM STANDARDS

Non-Governmental Programs

- NEFAP – Voluntary Accreditation Program
  - FSMO accreditation in Field Sampling and Measurements
  - Consensus Standards based on ISO 17025 quality system based
  - Accreditation Bodies – A2LA, PJLA, ANSI-ASQ National Board (ANAB-LAB)
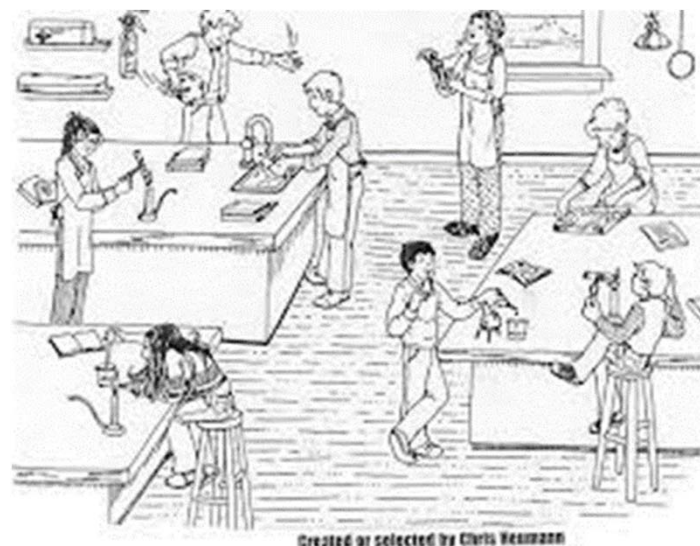- ISO 9000, 17025

Technical Requirements For Samplers

Do I have the right people to carry out the sampling?

- Personnel
  - Competence
  - Must have the necessary education, training and experience
  - Documentation of training, experience, job description
  - Sufficient number
  - Management shall formulate the GOALs with respect to training, education…
  - Technical Managers Expectations
 - Quality Assurance to establish a Field QMS

Competency
A specific range of skill, knowledge ability to do something successful being adequately or well qualified the condition of being capable of to meet demands, requirements

http://www.epa.gov/fem/pdfs/fem-lab-competency-policy.pdf

Created or selected by Chris Neumann

What should a sampler have for qualifications?

Sampler Qualifications

- Physically able to perform the duties of a sampler;
- No conflict of interest;
- Must be employed by…
- **Education / Training**
  - *Initial Classroom*
  - *Field / On the Job (OTJ)*
  - *Continuing Education*

- *Quality Systems Documentation.*
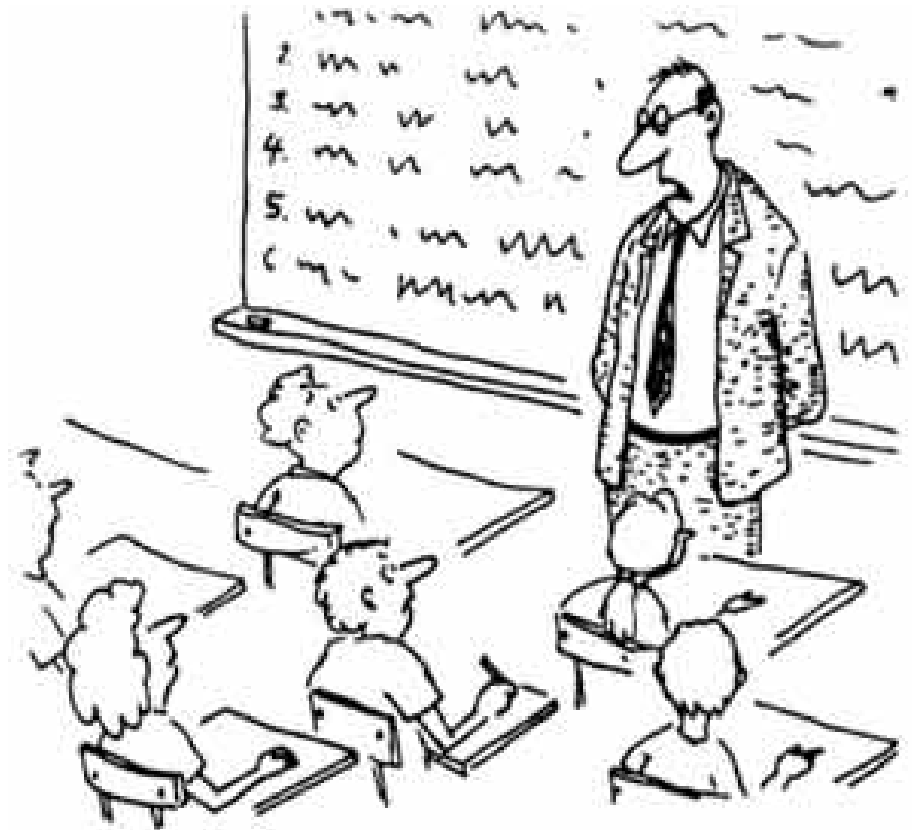
"*I expect you all to be independent, innovative, critical thinkers who will do exactly as I say!*"

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How many samplers, lab personnel, management read the QAPP.
What does the sampler need to know?

What kind of samples am I collecting?

What kind of testing is required?

How much sample is needed to perform all tests?

Where will the samples be collected?
- Is there a QAPP/ SAP

How will the sample be presented to the Laboratory?

What containers and devices should be used to collect them?

What Standard Operating Procedure(s) should be used or are needed?

Do I have the forms needed to document the sampling event?

Balance of Quality

All collected data have error.

- Nobody can afford absolute certainty.
- The Quality System seeks balance based on risk
- Environmental Data Quality Cannot Be Assured Unless There Are Quality Standards for All Steps of the Sample Collection and Analysis Processes

Increasing

Increasing

Time / Resources

QS

Uncertainty / Risk

Decreasing

Decreasing
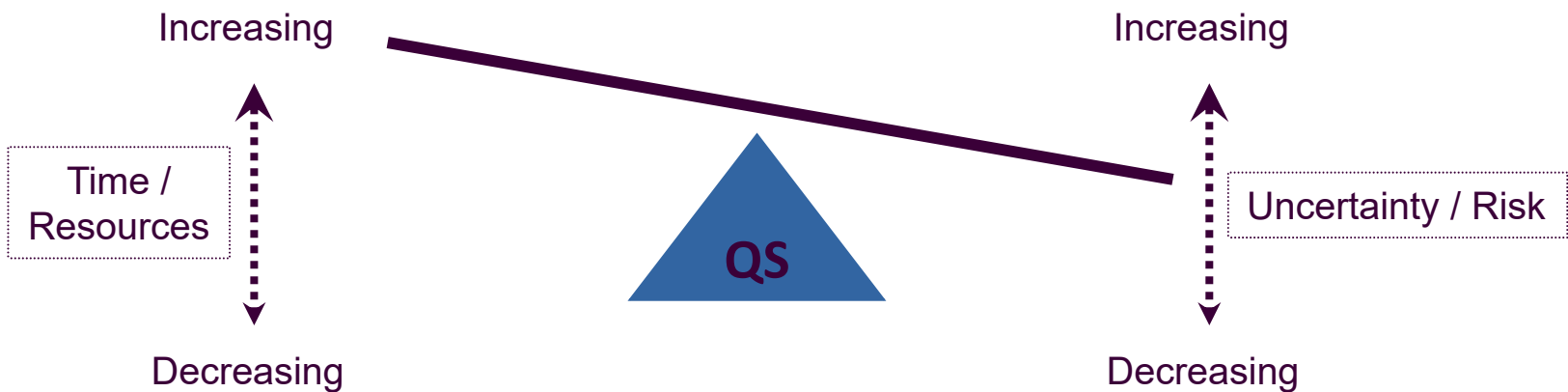
Laboratory Quality Systems: Not to be Confused with an SOP

Laboratory / FSMO Quality Systems Manual

Policy Not Procedure-Establishes policy for laboratory activities including sampling and analysis practices.

Root Cause and Correction Action

KISS

Orientation (training) of Employees

Share the Knowledge

Standard Operation Procedure - SOP

- Step wise documents that explain how to carry out a policy or procedure.

The QSM describes the laboratory and emphasizes management commitment to develop policy, procedures and resources necessary to generate reliable, defensible data.

Standard Operating Procedures

- Standard Operating Procedures (SOPs): A written document that details the method for an operation, analysis, or action, with thoroughly prescribed techniques and steps. SOPs are officially approved by laboratory management as the methods for performing certain routine or repetitive tasks.
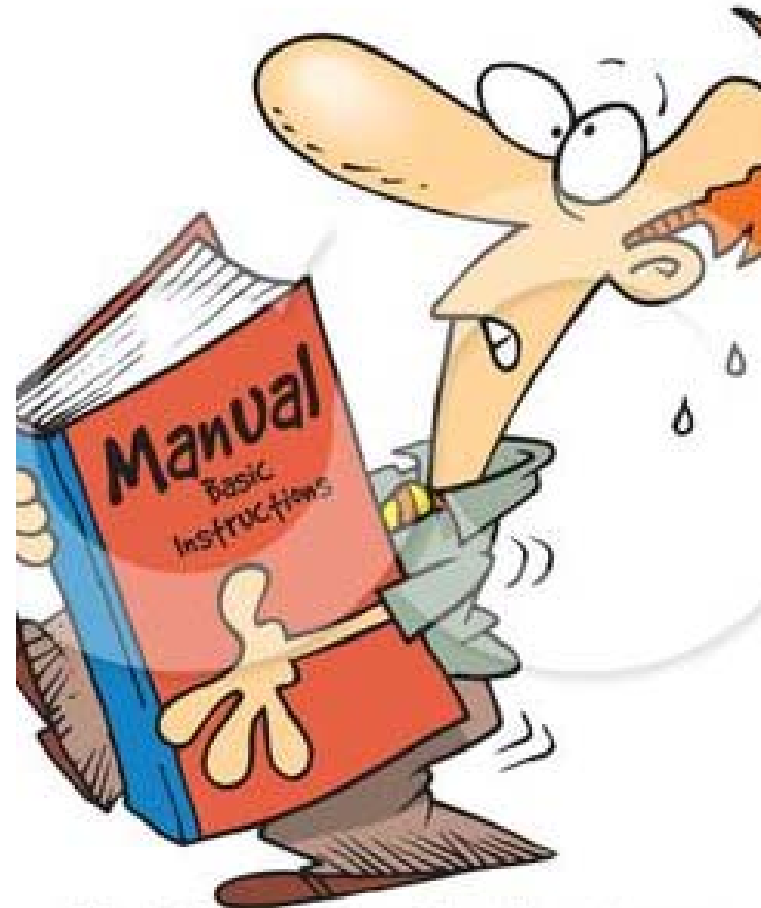
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# Field Audits - Quality Assurance System

Where?

When?

How?

Who?

What?

The laboratory or sampling facility shall adopt an ongoing system for performing audits of field activities.

The goal of the field audit is to verify that the sampling operation continues to comply with the requirements of the regulations and is being performed according to the laboratory's sampling SOP. The field audit shall address all elements of the sampling activities and shall be documented.

In Summary

- Know and understand the data objectives and plan, plan, plan
- Understand the data measurements and data acquisition methods required and the technique needed to obtain a representative sample
- Field / Lab coordination and communication
- Assessment/Oversight during the project – who is responsible for the conduct of the study
- Applicable data review and usability
- Documentation and traceability
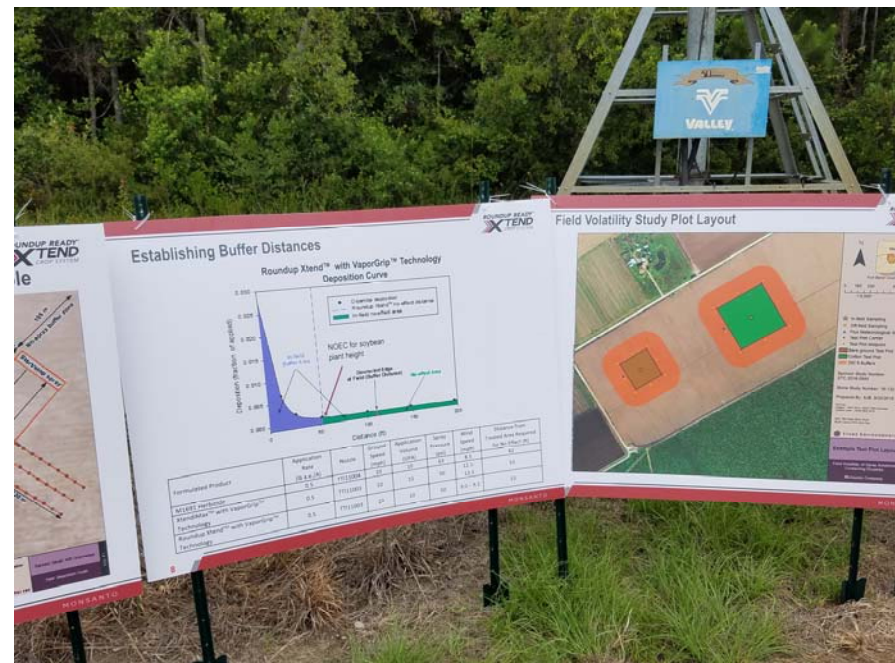
Acknowledgements/References

SW846 Chapter 9

Method 8260; Residual Solvents

www.epa.gov/epawaste/hazard/testmethods/sw846/online/index.htm

https://public.health.oregon.gov/LaboratoryServices/EnvironmentalLaboratoryAccreditation/Pages/rules.aspx

ORELAP – SOPs

- ORELAP-SOP-001R2 – Protocol for Collecting Samples for Usable Marijuana
- ORELAP-SOP-002R3 – Protocol for Collecting Samples of Cannabis Concentrates, Extracts and Products

Uniform Federal Policy for Quality Assurance Project Plans Evaluating, Assessing, and Documenting Environmental Data Collection and Use Programs

- EPA: EPA-505-B-04-900A
- DoD: DTIC ADA 427785

MA Cannabis Protocl final-revised-mdph-mmj-mips-protocol.pdf

Title: Protocol for Sampling and Analysis of Finished Medical Marijuana

Products and Marijuana-Infused Products for Massachusetts Registered

Medical Marijuana Dispensaries

ITRC (Interstate Technology & Regulatory Council). 2012. Incremental Sampling Methodology. ISM-1. Washington, D.C.: Interstate Technology & Regulatory Council, Incremental Sampling Methodology Team. www.itrcweb.org.

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Thank you.

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