STONE ENVIRONMENTAL

Representative and Random Cannabis Sampling, Sampler Quality Systems, and Demonstration of Competency in Sampler Protocols

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Division of Agrochemicals: Analytical;
Environmental & Regulatory Challenges with Legalized Cannabis.
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Topics for Discussion

Regulatory Challenges:

Regulations: Goal Obtain Defensible Samples, Protection of Public Health

Oregon Rules

Oregon Protocols

Testing Requirements

- Usable Marijuana
- Concentrates, Extracts, Products

Why the emphasis on Sampler Training

Representative & Random Sampling
Basic Concepts

Planning

Documentation

Quality Systems - Audits

Regulatory Challenges?

Oregon Administrative Rules

- OAR 333-07-010

  Marijuana Labeling, Concentration Limits and Testing

  —Usable Marijuana (333-007-0320)

  —Cannabinoid (333-007-0330 to 345)

- OAR 333-064-0100

  Marijuana Item Sampling Procedures and Testing

ORELAP Cannabis Sampling Protocols

- ORELAP-SOP-001

  Protocol for Collecting Samples of Usable Marijuana

- ORELAP-SOP-002

  Protocol for Collecting Samples of Cannabis Concentrates, Extracts and Products

Cannabis Sampling Protocols

Recommend 8 Hours of Each:

- ✓ Initial Classroom Training
- ✓ Field or On-the-Job Training
- ✓ Continuing Education / Annual Refresher

Training must cover:

- ➢ Principles of Sampling
- ➢ Procedures for Sampling
- ➢ Demonstration of competency in performing and instructing on the sampling methods

Tiered Training Approach:

- – Tier 1: Trainee understands concepts and is able to read, comprehend and carry out tasks in SOPs and Sample Analysis Plans (SAP).
- – Tier 2: On the Job Training (a) Trial –simulate (b) On-site with supervision by trainer
- – Tier 3: Demonstrated competence; able to perform tasks unsupervised; create procedure and policy.
- – Tier 4: Authorized to provide training to other staff.
- – Tier 5: Continuing Education and Refresher Training.

Cannabis Sampling Protocols

Sampler Qualifications

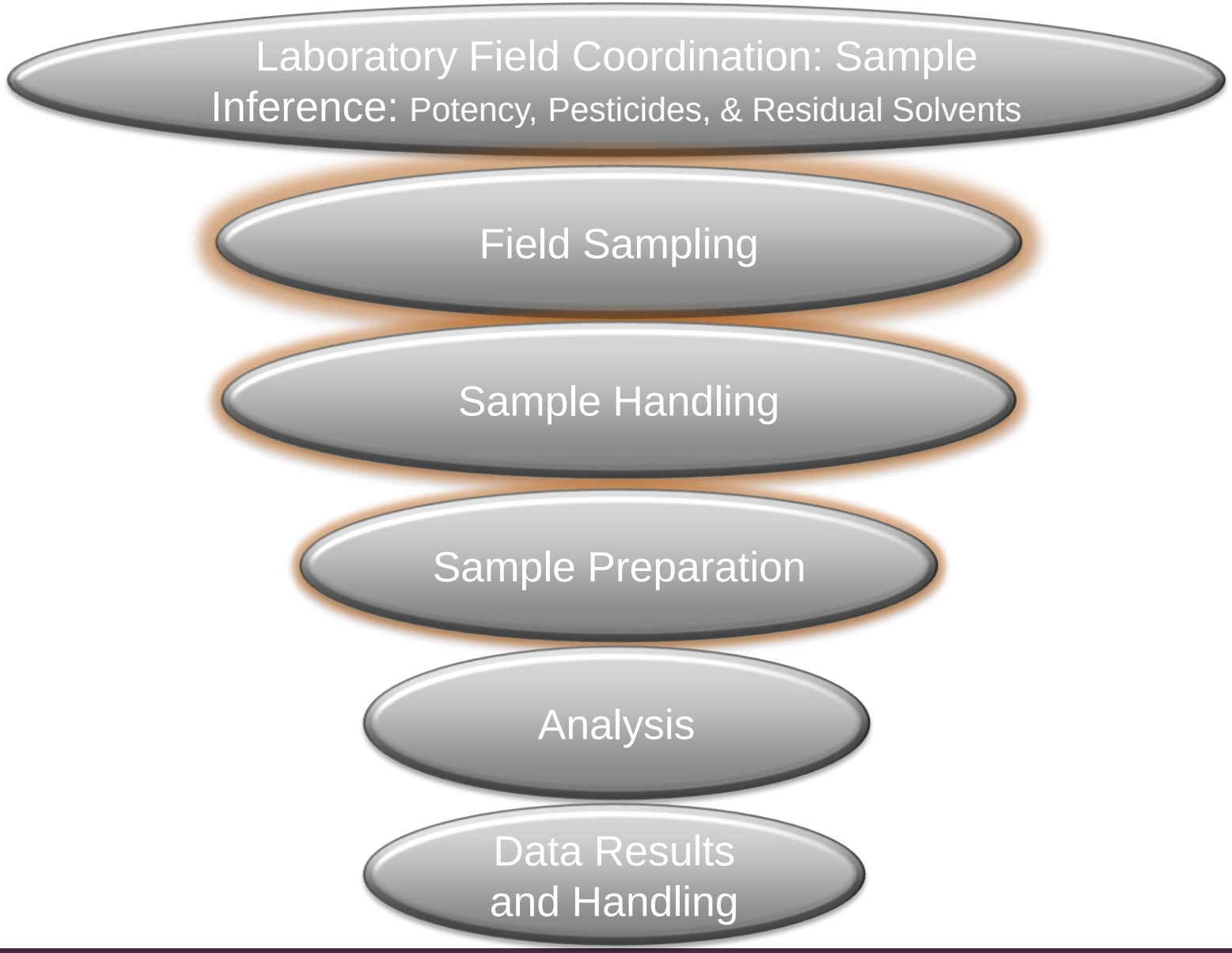
- Physically able to perform the duties of a sampler;
- No conflict of interest;
- Must be employed by an ORELAP accredited laboratory;
- Pass initial and ongoing demonstrations of capability;
- Licensed to transport the required quantity of Usable marijuana items
- **Education / Training**
  - ***Initial Classroom***
  - ***Field / On the Job (OTJ)***
  - ***Continuing Education: 8 Hr Annual Refresher***

"I expect you all to be independent, innovative, critical thinkers who will do exactly as I say!"

Why the emphasis on training?

What happens when you don't sample correctly?

What is the weakest link?



How do we get representative and random samples from the different matrices; usable, concentrates, extracts and edibles ?

edibles

-cookies

-gummies

-leathers

-hard candy

-bars

-truffles

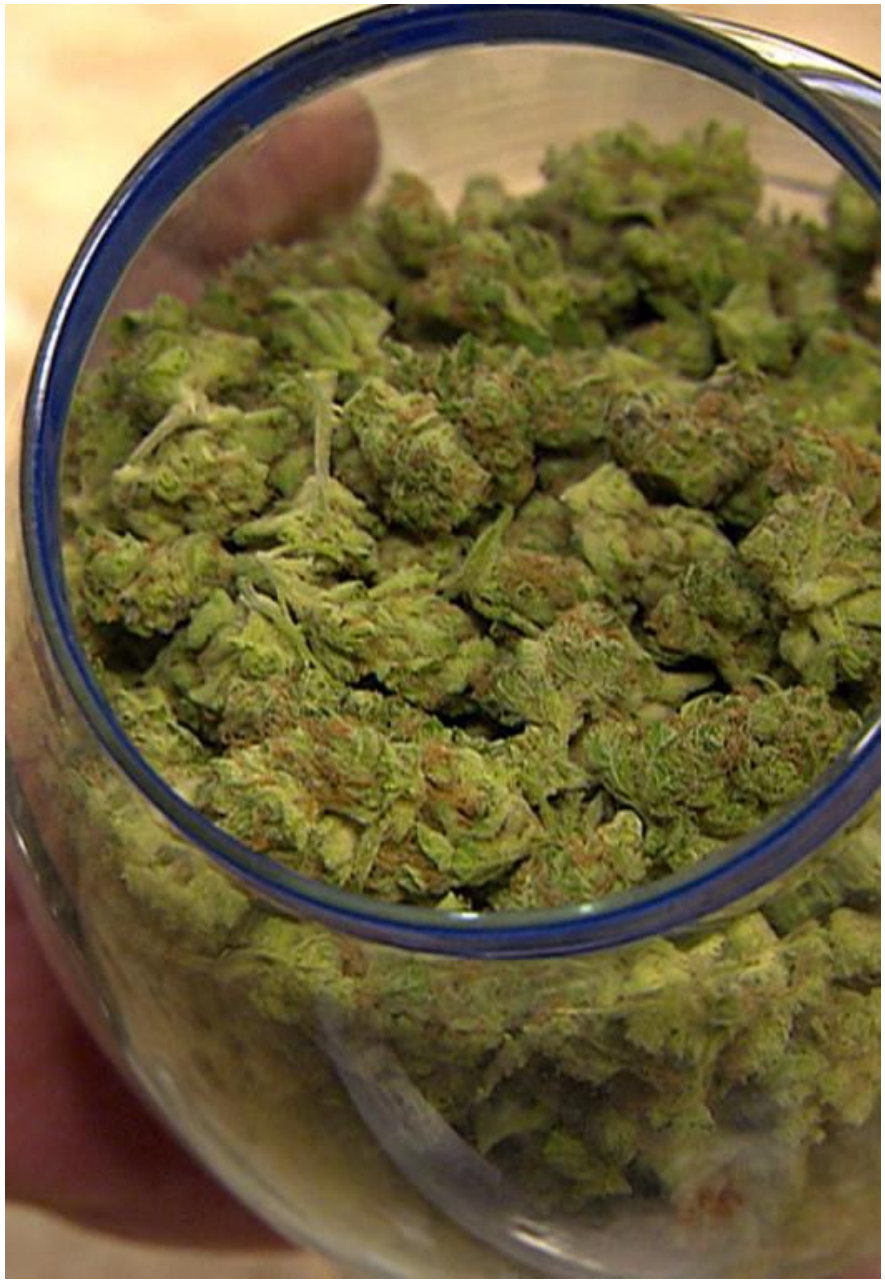
-butter

-What's next?

Representative Sampling

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



Cannabis Sampling Protocols

Common Objectives:

- **Statistically Designed Sampling Plans**
- **Scientific and systematic approach**
- Probabilistic sampling
- Standard Sampling Methods applied with consistency
- 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 – WHY??

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 a batch or lot 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.

SW846 Chapter 9

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

The ORELAP protocols specify use of random number generator to identify which location(s) to collect sample increments.

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

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.

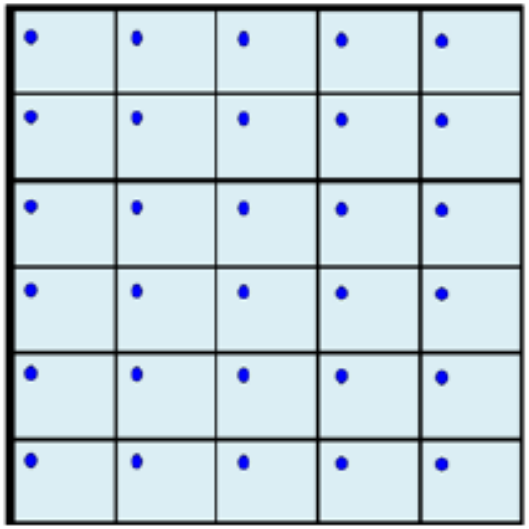


Figure 4-7. Systematic random sampling/ systematic grid sampling with a random start (Serpentine).

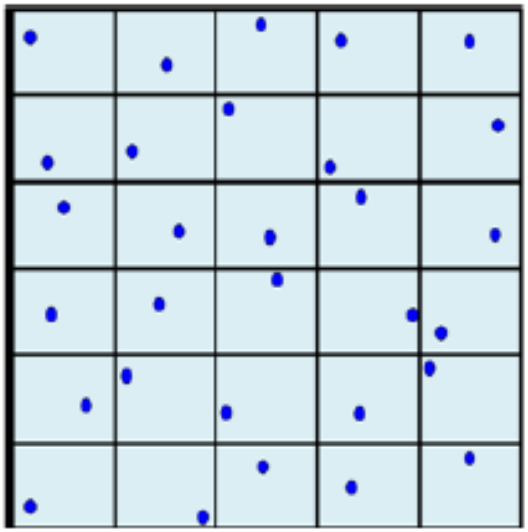


Figure 4-8. Random sampling within grids.

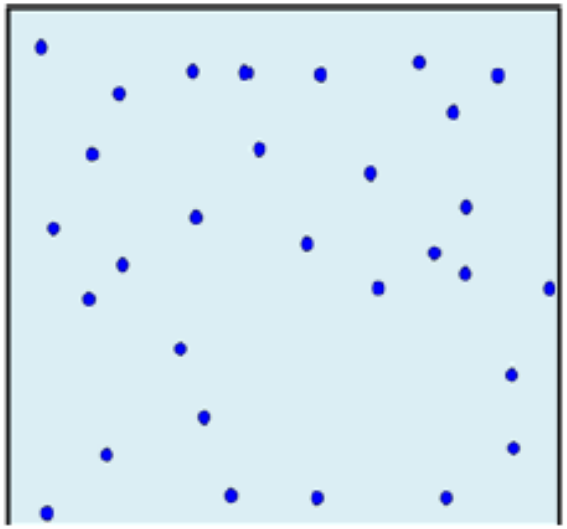


Figure 4-9. Simple random sampling within the entire DU.

Key Terms & Concepts from OAR / ORELAP

Usable Marijuana

Batch: *definite quantity of usable marijuana from a harvest lot*

Harvest Lot:

- *A specifically identified quantity of marijuana that is cultivated that is the same growing practices and harvested within a 72 hour period at the same location and cured under uniformed conditions.*

Definition changed to allow more than one strain and to clarify that marijuana harvested within a 72 hour period is part of the same harvest lot if cultivated using same growing practices and is cured under uniform conditions.

Cannabinoid

Batch: *a quantity of cannabinoid concentrate, extract or cannabinoid product from a process lot*

Process Lot:

- *Same type of concentrate or extract processed at same time using same extraction methods, SOPs and batches from the same or different harvest lots*
- *Any amount of cannabinoid product of the same type and processed at same time using the same ingredients, SOPs and batches from the same or different harvest or process lots*

Key Terms for Samples

Sample

An amount of marijuana item collected by laboratory personnel from a registrant or licensee and provided to a laboratory for testing.

Sample Increment

An amount of marijuana item collected by laboratory personnel from a registrant or licensee that may be combined for purposes of testing, or in the case of a control study, is tested individually.

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.

How Many Increments? Usable Marijuana

Sample Size

Per OAR 333-007-0360, the sample size must be sufficient to complete all analyses required, but shall in no case be less than 0.5% of the weight of the batch. Per OAR 333-007-0350, the maximum batch size is 10 lbs.

The required sample size for a given batch size based on OAR 333-007-0360 varies depending upon the size of the batch (Table 1).

Table 1 – Sample size requirements based on size of batch.

Batch size	Required sample size		
	Pounds (lbs)	Ounces (oz)	Grams (g)
≤1 lbs	0.005	0.08	2.3
1.01 ≤2 lbs	0.010	0.16	4.5
2.01 ≤3 lbs	0.015	0.24	6.8
3.01 ≤4 lbs	0.020	0.32	9.1
4.01 ≤5 lbs	0.025	0.40	11.3
5.01 ≤6 lbs	0.030	0.48	13.6
6.01 ≤7 lbs	0.035	0.56	15.9
7.01 ≤8 lbs	0.040	0.64	18.1
8.01 ≤9 lbs	0.045	0.72	20.4
9.01 ≤10 lbs	0.050	0.80	22.7

Sampling a Batch

1. When collecting a primary sample from a batch, a minimum of seven (7) increments shall be collected. Collect the increments by following different paths through the batch container or by taking the increments systematically at well-separated points along a heptagonal pattern.
2. As the batch increases in size, it is necessary to collect additional increments to make up the primary sample (Table 2). Collect seven increments plus additional increments based on 10% of the weight of batch. Do not collect more than 9 increments.

Table 2 - Number of increments for the primary sample based on batch size.

Size of batch (lbs)	≤ 2	≤ 4	≤ 6	≤ 8	≤ 10
No. of increments	7	7	8	8	9

Batch Size

Size of batch (lbs), Determines number of increments

How many increments?
Concentrate/Extract & Product (Pre-Control Study)

OAR 333-007-0360, Exhibit B

Table 5 – Sample increments per batch size of cannabinoid concentrates and extracts.

Batch Weight		Sample Increments Required (1±0.2 g)
Pounds	Kilograms	
0-0.50	0-0.23	4
0.50-1.5	0.24-0.68	8
1.51-3.00	0.69-1.36	12
3.10-6.00	1.40-2.72	16
6.10-10.00	2.77-4.54	20
10+	4.58+	32

Table 6 – Sample increments per batch size of cannabinoid products.

Units for Sale	Sample Increments
2-15	2
16-50	3
51-150	5
151-500	8
501-3,200	13
3,201 – 35,000	20

How many increments?
Concentrate/Extract & Product (Post-Control Study)

Table 7 - Sample increments per batch size of cannabinoid concentrates and extracts for the primary sample and field duplicate if concentrate or extract has certified control study.

Batch Weight		Sample Increments Required (1±0.2 g)	
Pounds	Kilograms	Primary	Field Duplicate
0-0.50	1-0.23	2	2
0.50-1.5	0.24-0.68	4	4
1.51-3.00	0.69-1.36	6	6
3.10-6.00	1.40-2.72	8	8
6.10-10.00	2.77-4.54	10	10
10+	4.58+	16	16

Primary Sample

Composite Sample: *a number of test increments that are collected and combined into a single sample for testing.*

Non-Composite Sample: *a single increment tested individually.*

Usable Marijuana

✓ Always Composite Sample

Cannabinoid

✓ Non-composite sample until a control study is complete; then composite samples are allowed.

Hierarchy of Testing Items

Graphic from GoodSample

Figure 1a. Sampling terms illustrated for tomatoes.

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

"Production error is the responsibility of the producer of the Usable Marijuana product" ORELAP SOP-001

"Manufacturing error is the responsibility of the processer of the Cannabis product" ORELAP SOP-002

In what ways can the producer or processor also effect sampling error?

Producer / Processor ⇨ Lab Communication Sampling and Analysis Test Request

A form, used by the laboratory to record ***all*** of the information necessary to create a sampling plan for a sampling event.

- This communication between the client/lab is a critical step in the sampling process.

A scientifically defective sampling plan has limited utility to the producer/processor, the sampler, the lab and the regulatory authority.

Sampling Plan

A 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.
2. Define the sample (Size, Type)
3. Documents 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
5. Includes Site-Specific Considerations
6. What else?

Sampling Report

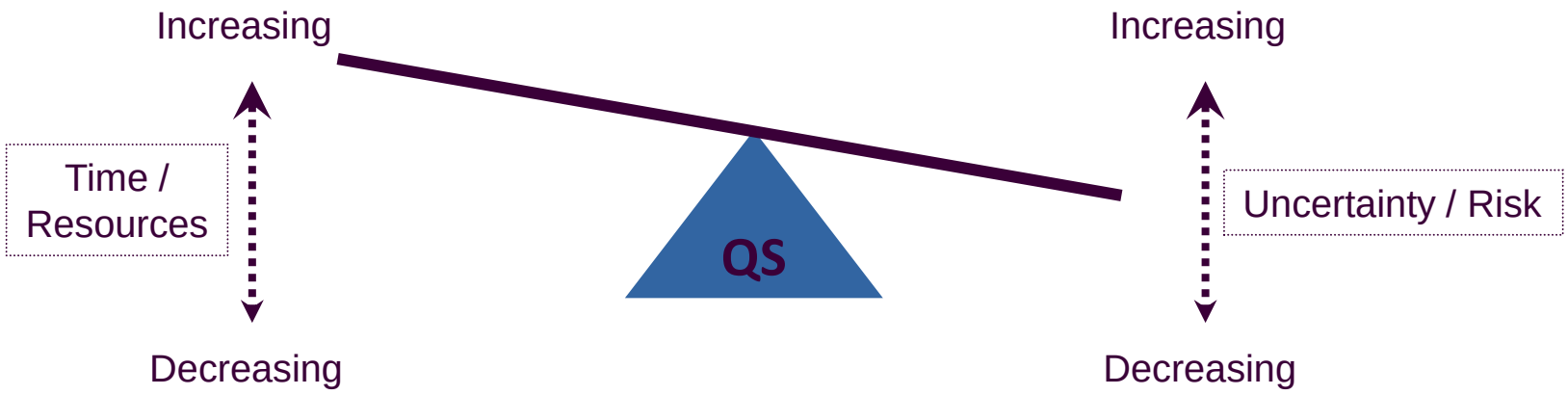
A sampling report documents the sampling activity and must include:

1. Name and Address of Producer / Processor
2. Registrant Number
3. Product Type
4. Batch Size
5. Batch ID, METRC Batch ID, and/or OHA Batch ID
6. Total Number of Batch Containers
7. Number of Containers Sampled
8. Number of Primary Samples Collected
9. Sampling Plan ID and Revision Date
10. Equipment Used for Sampling (Include Traceability, where relevant)
11. Location Samples were taken
12. Sampling Date
13. ORELAP Laboratory ID Number
14. Sampler's Name and Signature
15. Party responsible for transport information
16. Receiving Laboratory and Test Request

Balance of Quality

All collected data have error.

- Nobody can afford absolute certainty.
- The Quality System seeks balance based on risk



Laboratory and Field Quality Systems: Not to be Confused with an SOP

Laboratory Quality Systems Manual

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

Root Cause and Correction Action

KISS

Orientation 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.

Acknowledgements/References

SW846 Chapter 9
Method 8260; Residual Solvents
www.epa.gov*/epawaste/hazard/****testmethods/sw846****/online/index.htm*

Sampling and Sample Handling Working Group
FDA, AAFCO, AFDO, APHL, and Industry
October 2015
http://www.aafco.org/Publications/GOODSamples

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

MA Cannabis Protocol 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.

Thank you.

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