GOOD Test Portions

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QA / QC: A Critical Need for Laboratory Sampling

Quality Assurance
Quality Control

GOOD Test Portions: Guidance On Obtaining Defensible Test Portions

GOOD Test Portions

Laboratory Sampling Working Group
AAFCO, AFDO, and APHL
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http://www.aafco.org/Publications/GOODTestPortions

GOOD Test Portion
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GOOD Test Portions - June 2018

- Introduction
- Definitions, and Acronyms
- Expansion of GOODSamples Concepts for Laboratory Sampling
- Laboratory Sampling
- Quality Assurance and Quality Control
- Laboratory Sampling Processes
- Data Assessment and Inference
- Training
- Appendix – TOS Equations
- References

***GOODSamples* is prerequisite**

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Quality Assurance and Quality Control

- Quality Assurance
  - Validation of a Laboratory Sampling Protocol
  - Verification of Laboratory Sampling Protocol
- Quality Control Events
  - Random Error
  - Systematic Error

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Why is QA important?

- Quality management system is critical component of laboratory operations
- We want to know our methods/protocols work before we use them
- We want to know that the methods/protocols work for our sample types
- We want to know staff is properly trained before producing results
- We want to be ISO accredited

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Why do we use QC?

- Monitor and estimate random error
- Check for systematic error
- Know when a process is ”in control” or operating within predefined error tolerances
- Increase confidence

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Is there error in laboratory sampling?

- Yes!

What is the magnitude of the error?

- Generally substantially greater than analytical error.
- Is QA/QC needed. YES!

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Quality Assurance

Quality Assurance

- Validating new sampling protocols
  - Scope of application is well defined
  - Tolerable error has been established
- Validation must demonstrate that protocol meets the scope of application and tolerable error.

Validation of a Laboratory Sampling Protocol

Must meet the tolerable error

- Must confirm sample correctness (absence of systematic errors IDE and IEE)
- Must validate sufficient mass (to control FSE)
- Must validate sufficient number of increments (to control GSE)

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Two basic approaches

1. TOS Approach
   - Detailed knowledge of the material properties so that FSE equations can be utilized
   - Need in depth knowledge of TOS

2. Experimental Approach
   - Can implement tomorrow!

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Experimental approaches

- Every analyte, analyte concentration and type of material and laboratory situation is different
- So, never one size fits all!
- Three general approaches
  - Whole sample extraction
  - Materials with a know amount of analyte of interest or surrogate
  - Tracers

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Whole sample extraction option

- Compare result obtained from testing the selected portion to the result obtained from testing the entire unselected portion.
- If entire unselected portion is too large for a single extraction process, multiple extractions can be performed and proportionately combined to obtain a single test to represent all the unselected material.
- If the two results compare, the selection process is valid.

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Materials with a known amount of analyte or surrogate

- Known materials
  - Certified reference materials
  - Fortified materials
  - Incurred materials
- Materials must mimic the routine samples
- Surrogates mimic the behavior of the analyte
  - Radiolabeled compounds
  - Isotopes

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Tracers

- Tracers are unrelated to the analytes of interest but can provide useful information.
  - Sand
  - Microtracers™
- Limitation is analyte of interest may or may not behave as the tracer behaves.
  - Caution when extrapolating to routine samples

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Validation Materials

- Carefully chosen to match scope of application (materials to be routinely handled)
- Certified reference materials are not readily commercially available
- Fortified materials can be created
- Surrogates, tracers or fortified materials can be incorporated at a single point or multiple points

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Replication

- Used to estimate random error from the point of incorporation forward
- Incorporating replication a multiple selection processes allow isolation of error for a specific selection process.
- More replicates = better estimation of error

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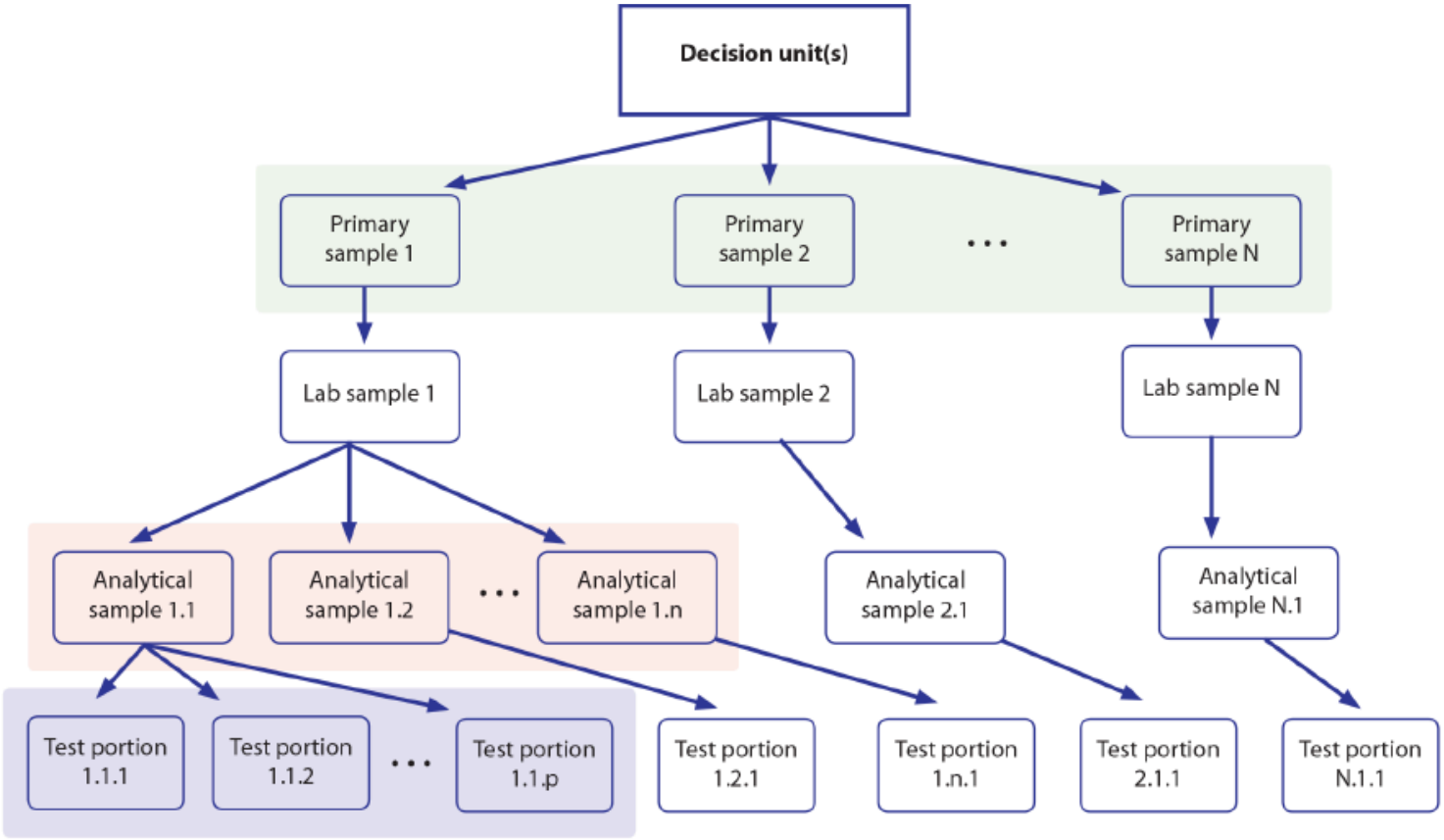
Estimating Error Contributions



N = number of primary sample replicates chosen

n = number of analytical sample replicates chosen

p = number of test portion replicates chosen

By analyzing various combinations of the test portions (see Equation 6 below), the error contributions from each mass reduction step can be estimated.

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Estimating Error Contributions

Table 2. Equations to estimate error contribution for specific selection processes

Error estimate	Relative standard deviation (RSD) calculation[1]	Error calculation[2]
Estimate of overall GEE (TSE + TAE)	RSD of 1.1.1, 2.1.1, …, N.1.1 = RSD_{ps}	$GEE = RSD_{ps}$
Estimate of overall TSE	RSD of 1.1.1, 2.1.1, …, N.1.1 = RSD_{ps}	$TSE = \sqrt{RSD_{ps}^2 - TAE^2}$
Error contribution from selection of the primary sample (ps)	RSD of 1.1.1, 2.1.1, …, N.1.1 = RSD_{ps}	$E_N = \sqrt{RSD_{ps}^2 - RSD_{as}^2}$
Error contribution from selection of the analytical sample (as)	RSD of 1.1.1, 1.2.1, …, 1.n.1 = RSD_{as}	$E_n = \sqrt{RSD_{as}^2 - RSD_{tp}^2}$
Error contribution from selection of the test portion (tp)	RSD of 1.1.1, 1.1.2, …, 1.1.p = RSD_{tp}	$E_p = \sqrt{RSD_{tp}^2 - TAE^2}$

[1]RSD is expressed as a decimal (part of 1) for all error calculations.
[2]To express calculated error as %RSD, multiply calculated GEE, TSE, E_N, E_n and E_p by 100.

Note: TAE can be determined by replicating the test on a single test portion. This is possible when the test portion is solubilized and the entire test solution is not consumed in a single test.

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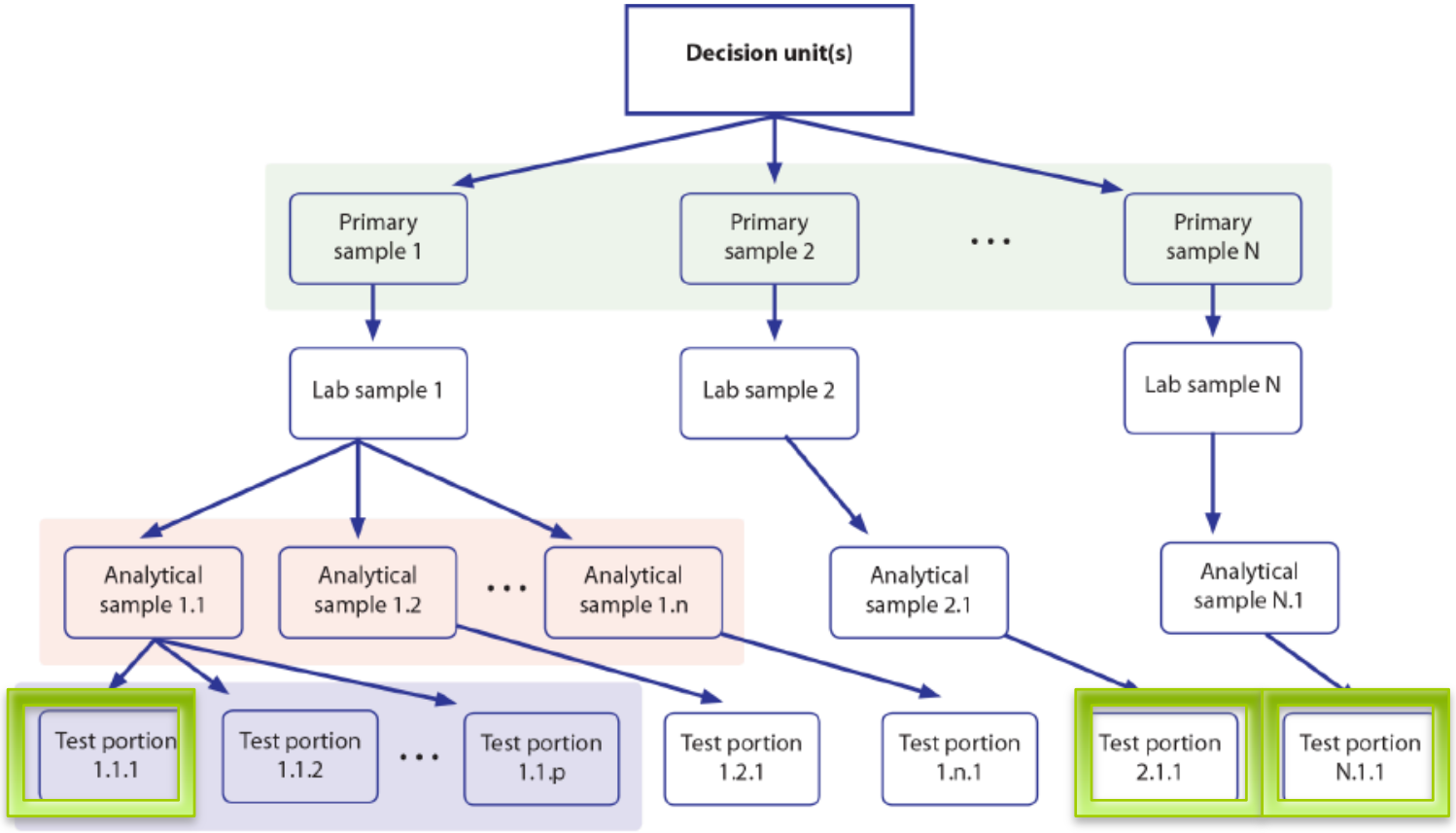
Estimating GEE



N = number of primary sample replicates chosen
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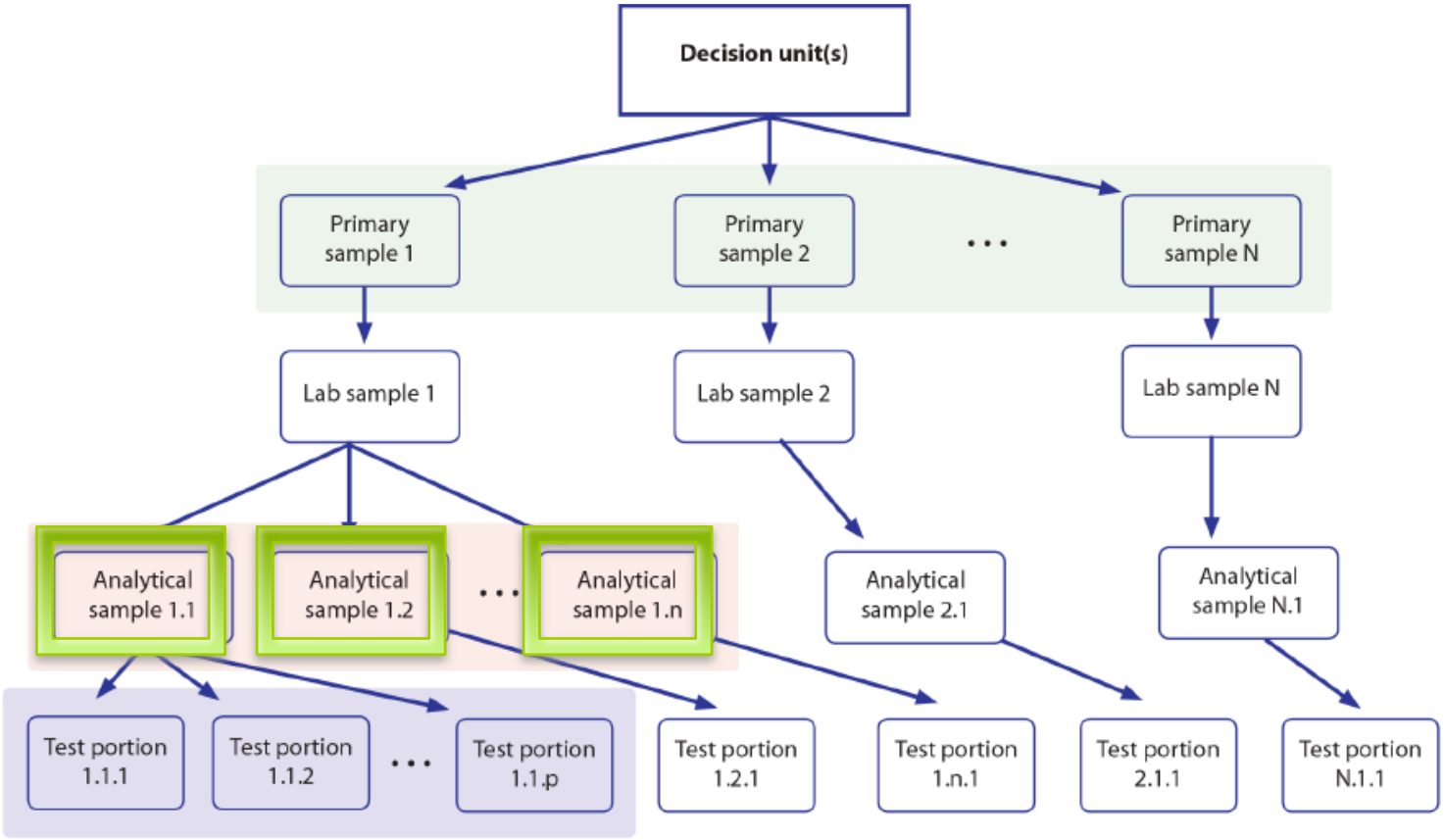
Error from Selection of Analytical Sample



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By analyzing various combinations of the test portions (see Equation 6 below), the error contributions from each mass reduction step can be estimated.

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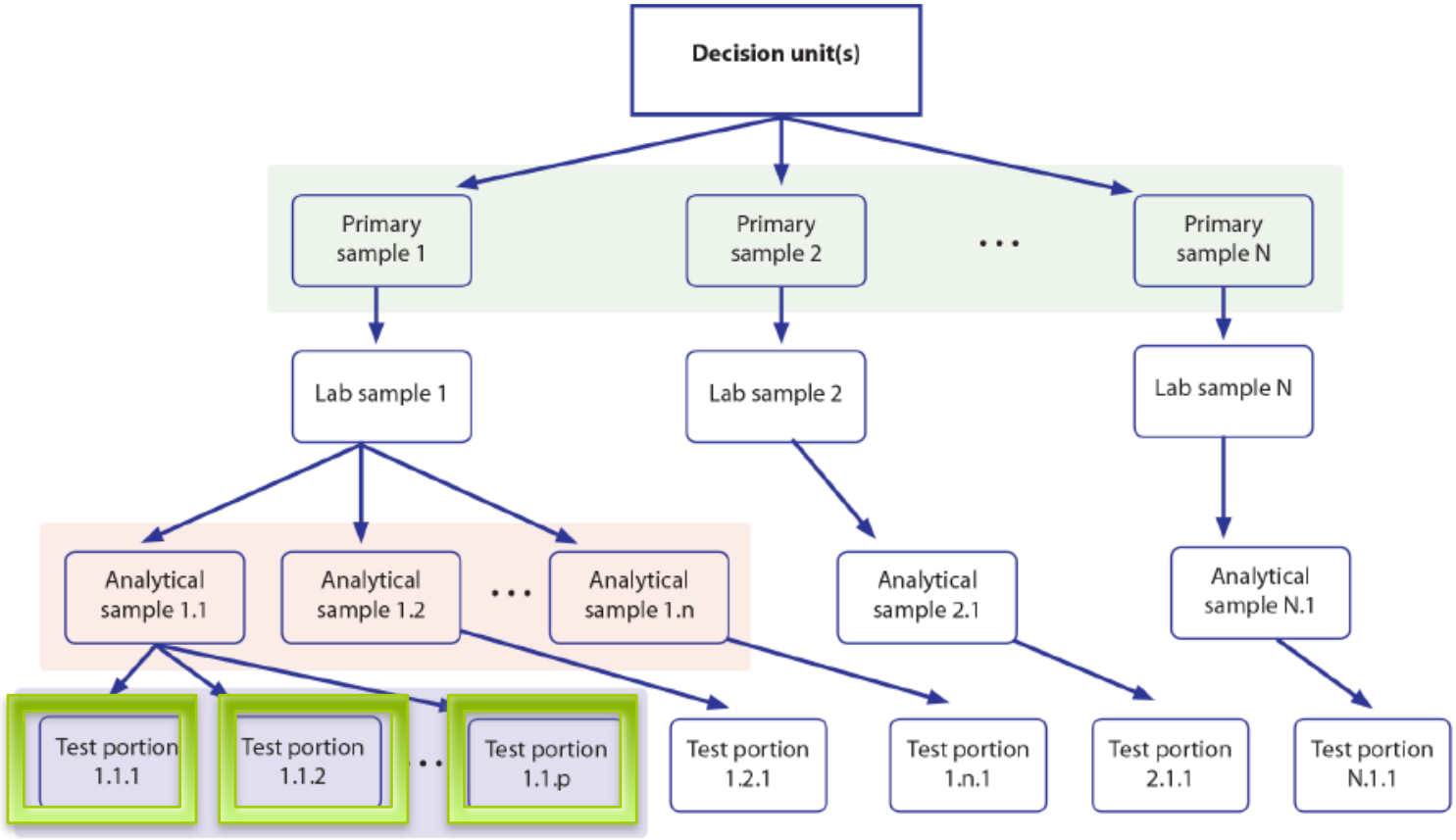
Error from Selection of Test Portion



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By analyzing various combinations of the test portions (see Equation 6 below), the error contributions from each mass reduction step can be estimated.

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Example for estimating error associated with selection of the analytical sample for As in rice

- Observing principles of TOS, multiple analytical sample prepared (1.1, 1.2, …. 1.n)
- Observing principles of TOS, replicate test portions are selected from a single analytical samples (1.1.1, 1.1.2, …. 1.1.p) and analyzed for As. The RSD is 7%.
- Observing principles of TOS, single test portions are selected from each of the single analytical samples (1.1.1, 1.2.1, …. 1.n.1) and analyzed for As. The RSD is 14%.

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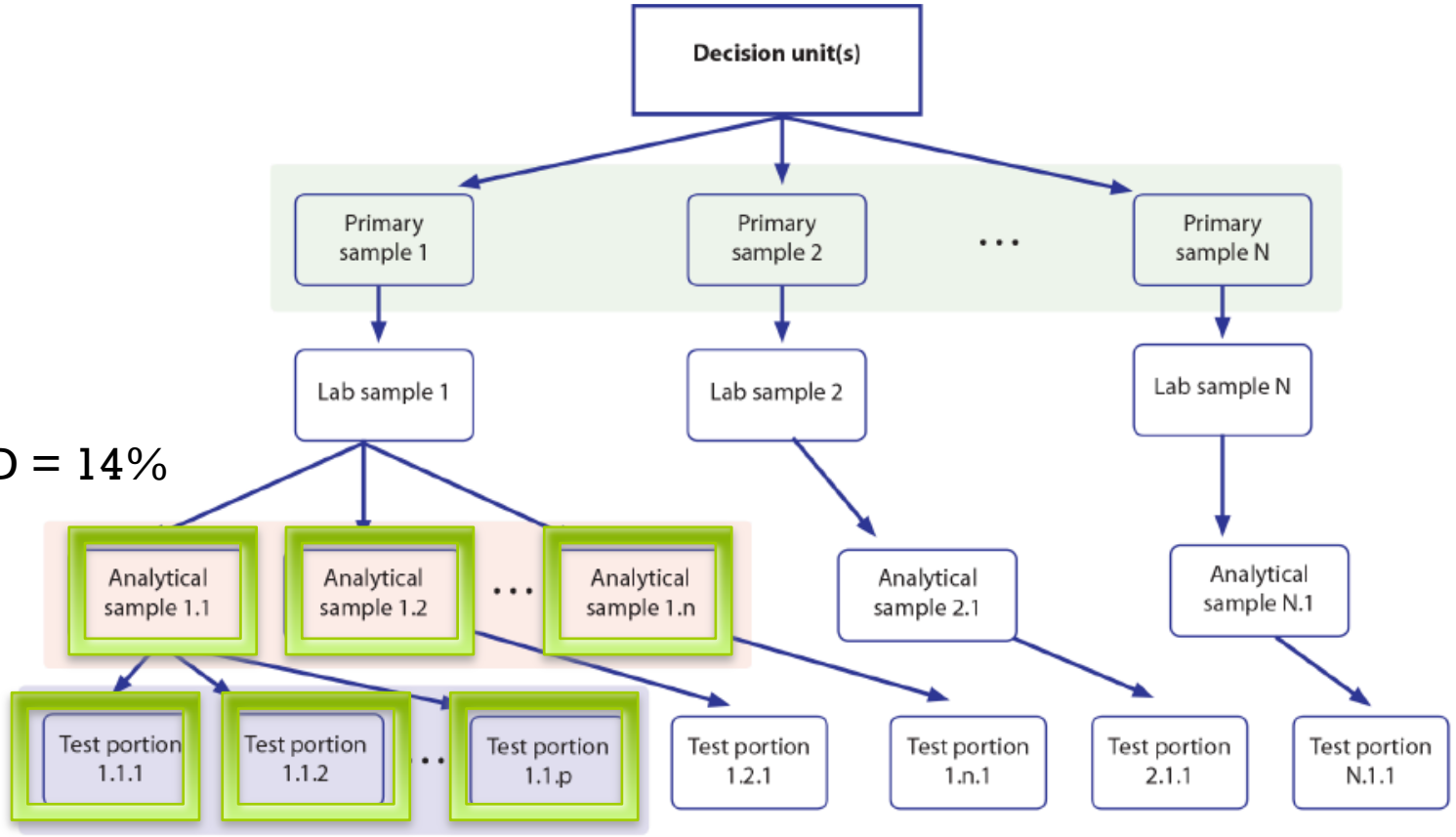
Error from Selection of Test Portion



RSD = 14%

RSD = 7%

N = number of primary sample replicates chosen

n = number of analytical sample replicates chosen

p = number of test portion replicates chosen

By analyzing various combinations of the test portions (see Equation 6 below), the error contributions from each mass reduction step can be estimated.

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Calculating Error associated with Selection of the Analytical Sample for As in Rice

- The random error contribution associated with selection of the analytical sample can be calculated:

$$\mathrm{RSD}_n, \% = \sqrt{0.14^2 - 0.07^2} \times 100 = 12\%$$

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Verifying a Laboratory Sampling Protocol

Once validated, periodic verification through performance tests

- Performance tests can be devised to verify nonselection or selection processes
  - Carryover
  - Final particle size distribution
  - Contamination
  - Cleaning procedures

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Verifying a Laboratory Sampling Protocol

Once validated, periodic verification through performance tests

- When to utilize
  - New equipment – compare old and new equipment
  - New employee
- Frequency
  - Changes in personnel, equipment, protocols.
- Need to mimic routine operations (not only best or newest equipment)

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Quality Control

Series of checks to monitor and measure error during routine nonselection and selection processes

Quality Control Checks for Random Error

- Checks for random error = replication
  - More replicates = better error estimation
  - Replicates at any process provide an estimate of error from that point forward
  - Replicating at multiple selection processes, it is possible to isolate error from an individual process
  - Replicates are collected using same tool, same number of increments but selecting increments at different random locations

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Quality Control Checks for Systematic Error

- Bias checks for analytical procedures fairly easy to incorporate. Not so easy for laboratory sampling. Each lab needs to be creative and develop fit for purpose QC.
- No QC events, so great care needs to be exercised!
  - ⃠Increment delimitation error
  - ⃠Increment extraction error
  - ⃠Increment weighting error

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Quality Control Checks for Extraneous Material Error

- Occurs when extraneous material is not removed in total or when some the decision unit is removed along with the extraneous material
- A QC measure may be to compare, either visually or by weighing, the separated extraneous material as performed by multiple analysts
- Impossible to quantify this in terms of an error; however, if the process is inconsistent, error will result

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Quality Control Checks for Contamination Introduction Error

- Contamination introduced from equipment and the environment
- Evaluate by processing material that contains no analyte and interpret as a QC blanks
- Swabs and swipes of equipment can be processed as QC blanks

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Quality Control Checks for Mass Recovery Error

- Determine by visual inspection of equipment for incomplete removal of material
- Determine by weighing material before and after processing
- While actual mass lost may be determined, error introduced by the loss of material cannot be estimated

Quality Control Checks for Analyte Integrity Error

- Cannot be quantitated, but can be detected
- Measure splits at multiple points in time and observe for trends
- Use of radiolabeled compounds or fortified materials
- Comparison with a previously validated protocol

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Discussion?

QUESTIONS?

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