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GOOD Test Portions

Guidance on Obtaining Defensible Test Portions (GOOD Test Portions)

Coming real soon….

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GOODSamples –

- Published Oct 2015 outlines a systematic approach to sampling

- Introduction
- Terms, Definitions, and Acronyms
- Management Support
- SQC Overview
- Material Properties
- Theory of Sampling

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- Sample Correctness and Tools
- Evidentiary and Analyte Integrity
- Laboratory Considerations
- Quality Control
- Inference
- Data Assessment
- Resources

GOODSamples - Oct 2015

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******GOODSamples*** is prerequisite**

Working Group Members

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- Aaron Price, Canadian Food Inspection Agency
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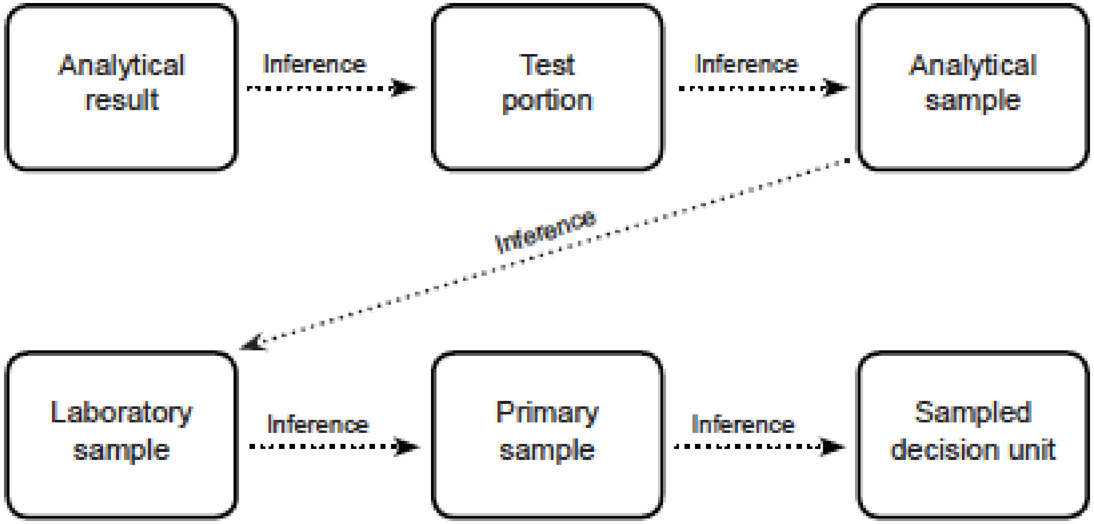
- Sharon Webb, University of KY Regulatory Services

Applying *GOODSamples* in the Laboratory

- Sampling/inference pathway



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LABORATORY SAMPLING

Laboratory Sampling consists of two processes.

- Non-selection processes: manipulation(s) to a sample taken prior to a selection process, e.g. comminution, removal of extraneous material, use of a comminution aid such as dry ice, etc.

- Selection processes: selecting a smaller mass from the larger mass

"Laboratory Sampling" term in GOOD Test Portions refers to both the non-selection and selection processes

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Sample Quality Criteria

Lab must be involved with program staff in SQC process; lab brings scientific expertise;

- What is the question?
  - What is analyte or characteristic of concern?
  - What is the concentration of concern?
  - How will inference be made?
- What is the decision unit?
- What is the desired confidence?

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Principles of Theory of Sampling

- Relationship of error to mass
- Relationship of error to increments
- Relationship of error to sample correctness

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ELABORATE ON MATERIAL PROPERTIES

- Finite vs Infinite elements. **Comminution of a finite element material results in an infinite element material.**
- Heterogeneity is the root cause of error in all sampling. Compositional and Distributional Heterogeneity (CH and DH).
- The magnitude and nature of CH and DH are unique to each material and dictate the sampling efforts. .

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EXAMPLE OF DISTRIBUTIONAL HETEROGENEITY

- Orange juice has many separate components with large distributional heterogeneity. The pulp falls quickly, the foam disperses slowly and volatiles escape rapidly.

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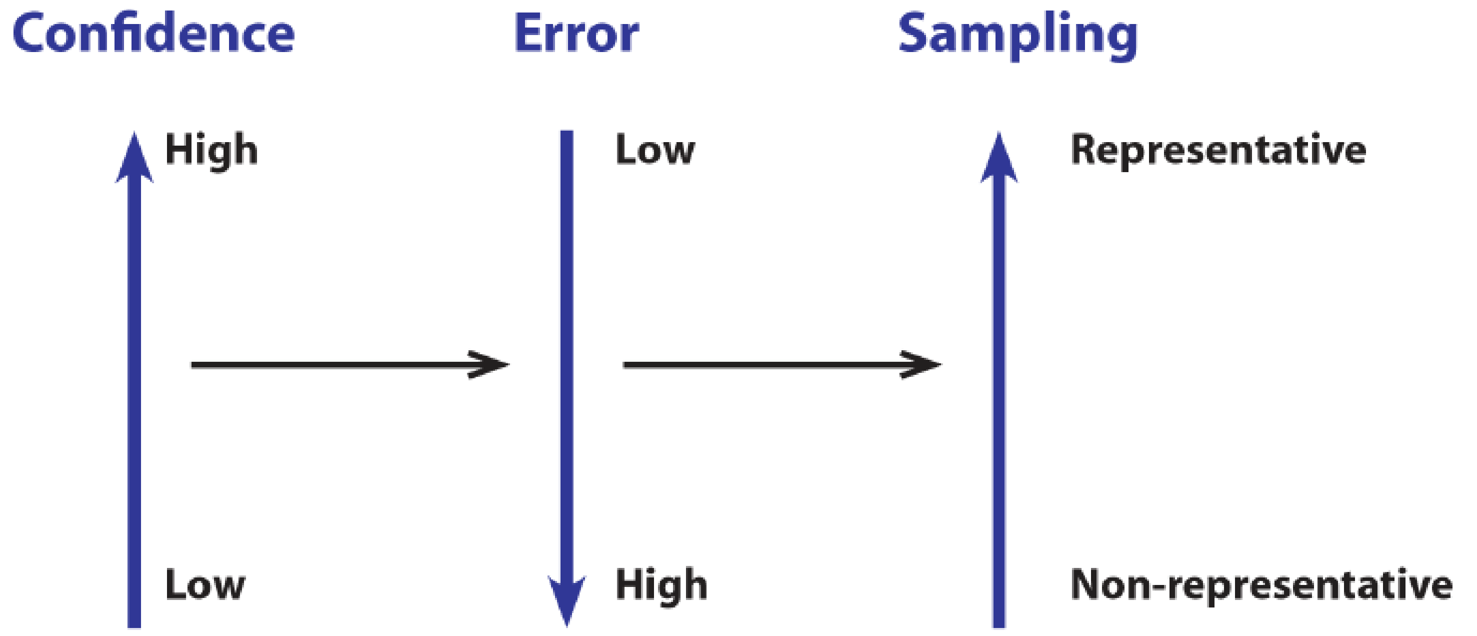
+ Total Sampling Error (TSE)



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+ Total Sampling Error (TSE)

Three types of error

- Systematic Error
- Random Error
- Blunders

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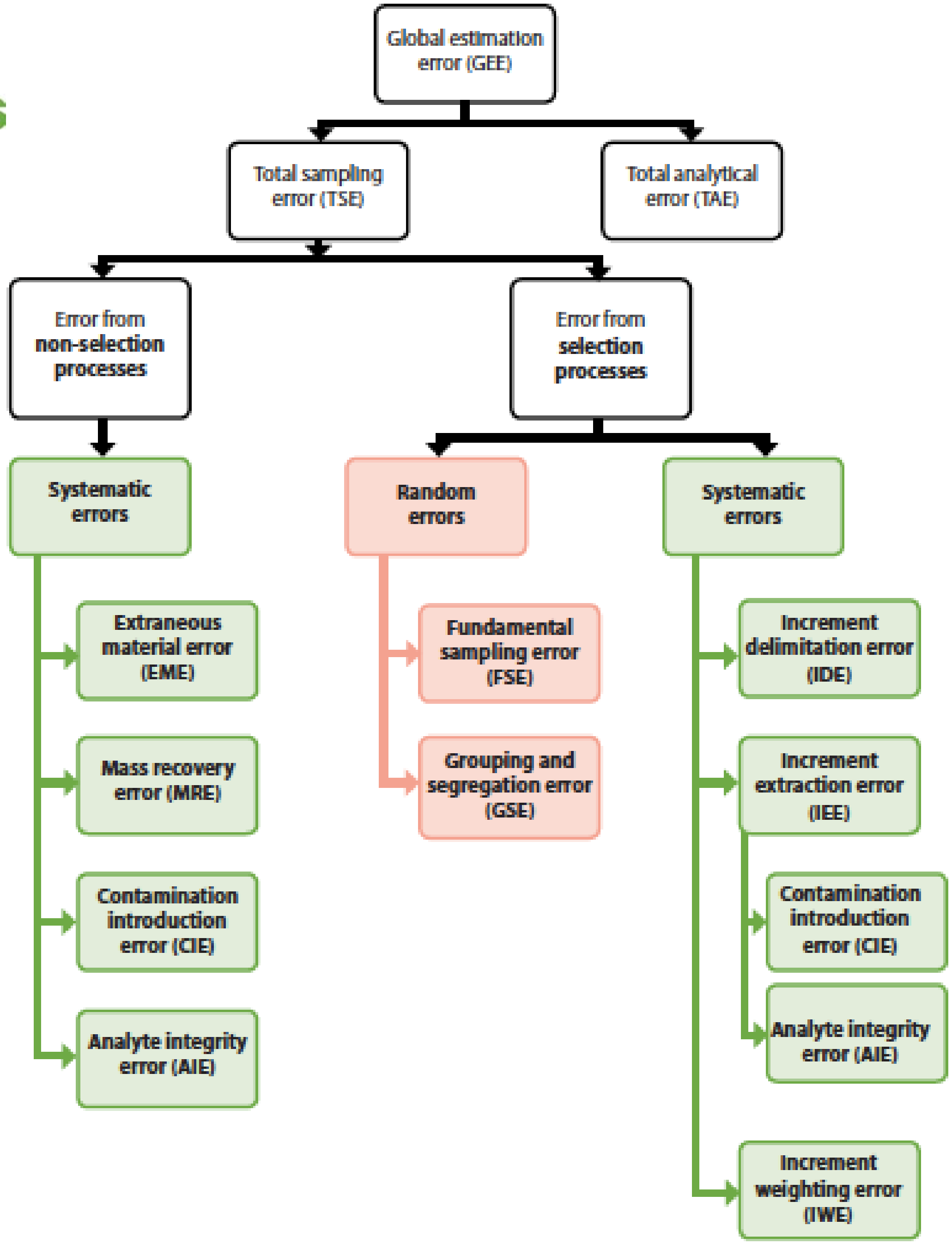
Global estimation error (GEE)
Total sampling error (TSE)
Total analytical error (TAE)
Error from non-selection processes
Error from selection processes
Systematic errors
Random errors
Systematic errors
Extraneous material error (EME)
Mass recovery error (MRE)
Contamination introduction error (CIE)
Analyte integrity error (AIE)
Fundamental sampling error (FSE)
Grouping and segregation error (GSE)
Increment delimitation error (IDE)
Increment extraction error (IEE)
Contamination introduction error (CIE)
Analyte integrity error (AIE)
Increment weighting error (IWE)

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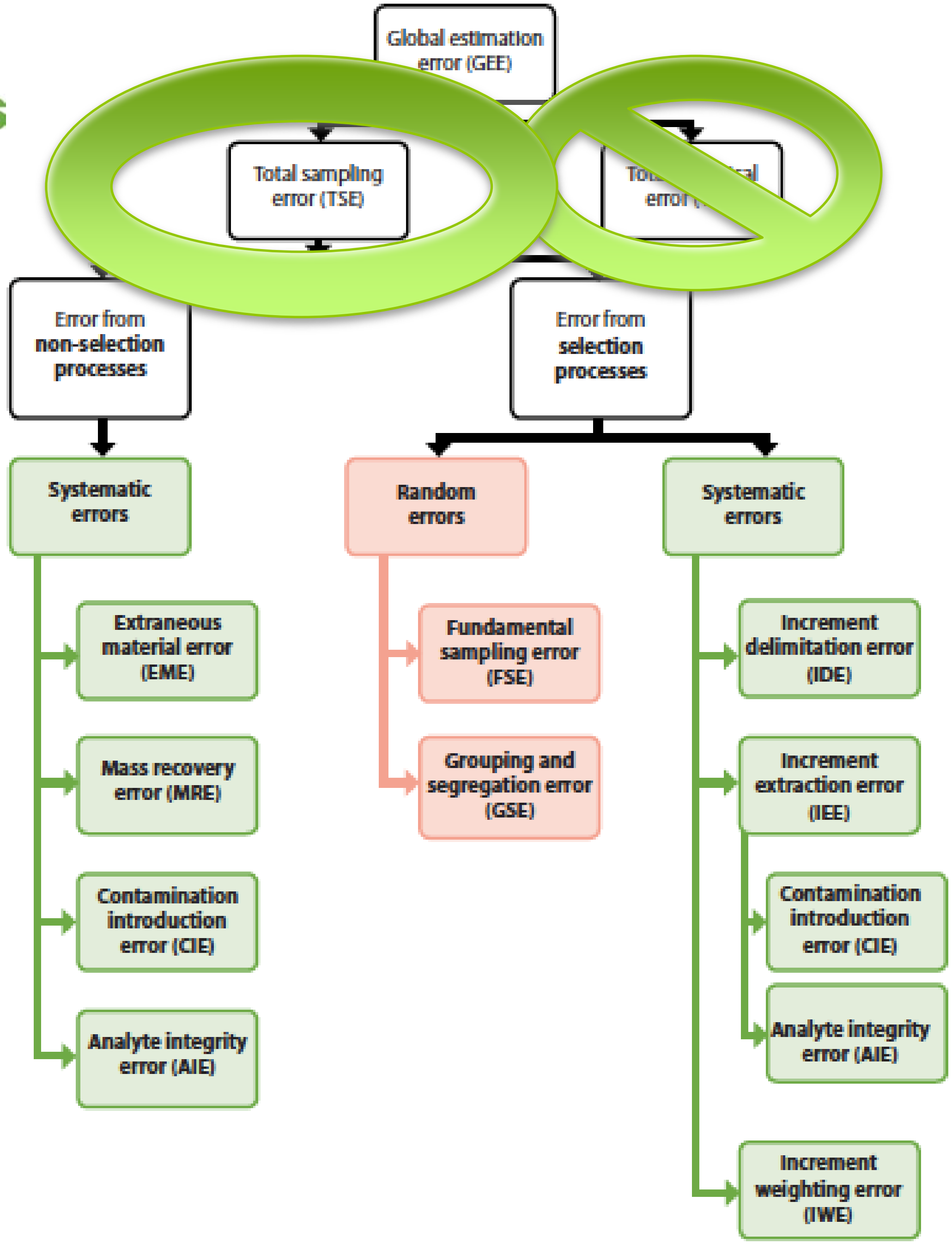

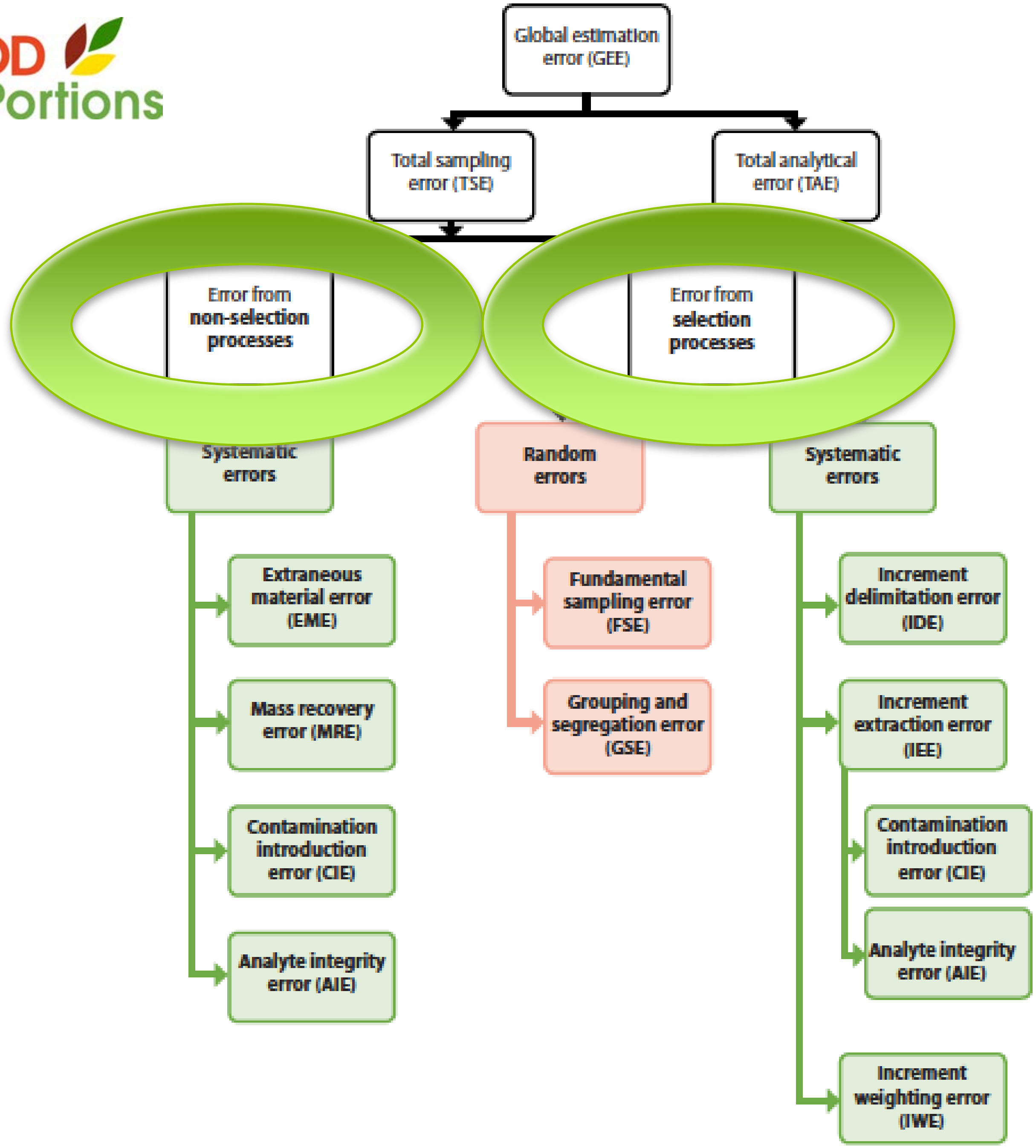
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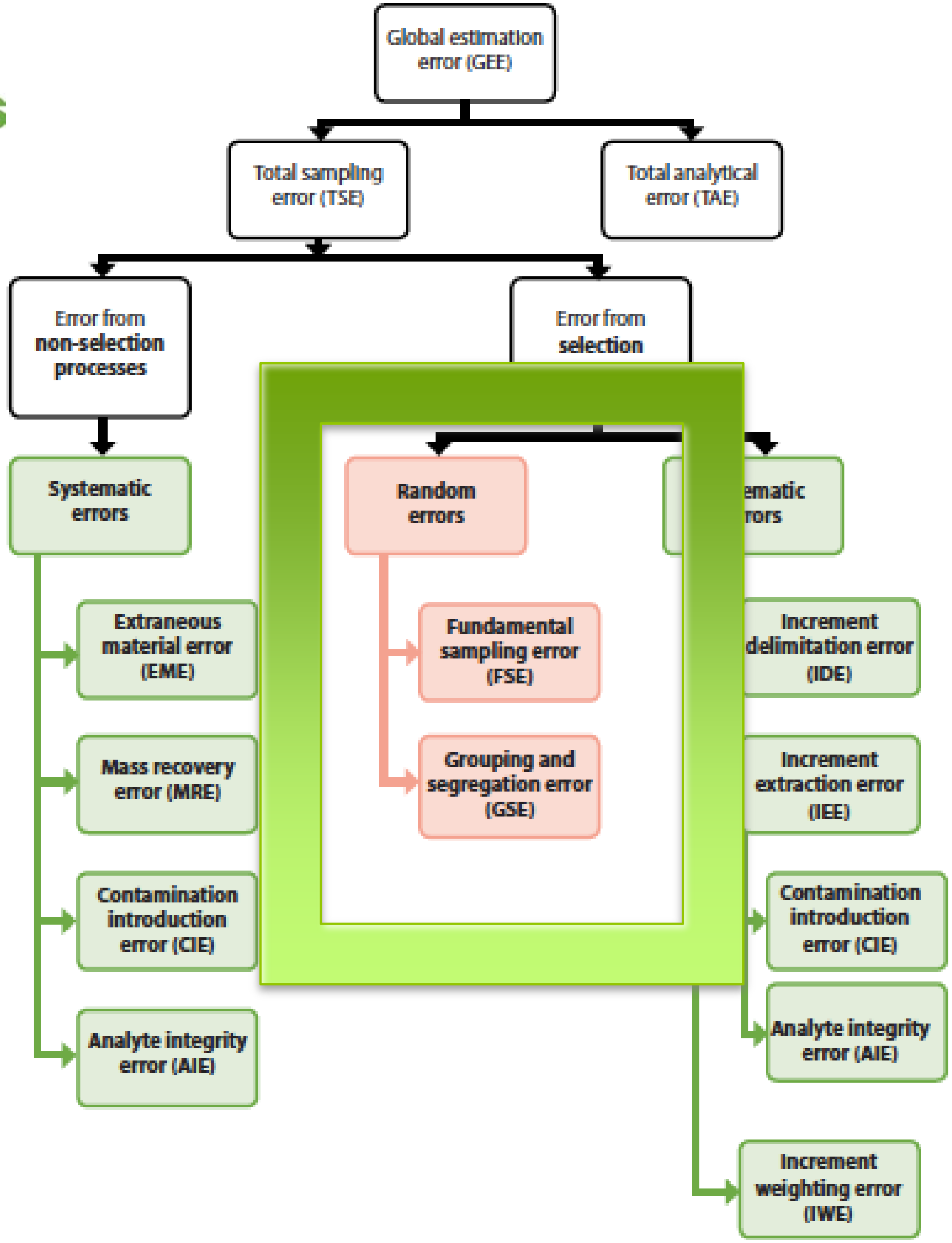

RANDOM ERRORS

- Fundamental Sampling Error (FSE)
  - Function of particle size, mass and CH
- Grouping and Segregation Error (GSE)
  - Function of number of increments and DH
- Relationships
  - Relationship of error to mass
  - Relationship of error to increments
  - Relationship of error to sample correctness

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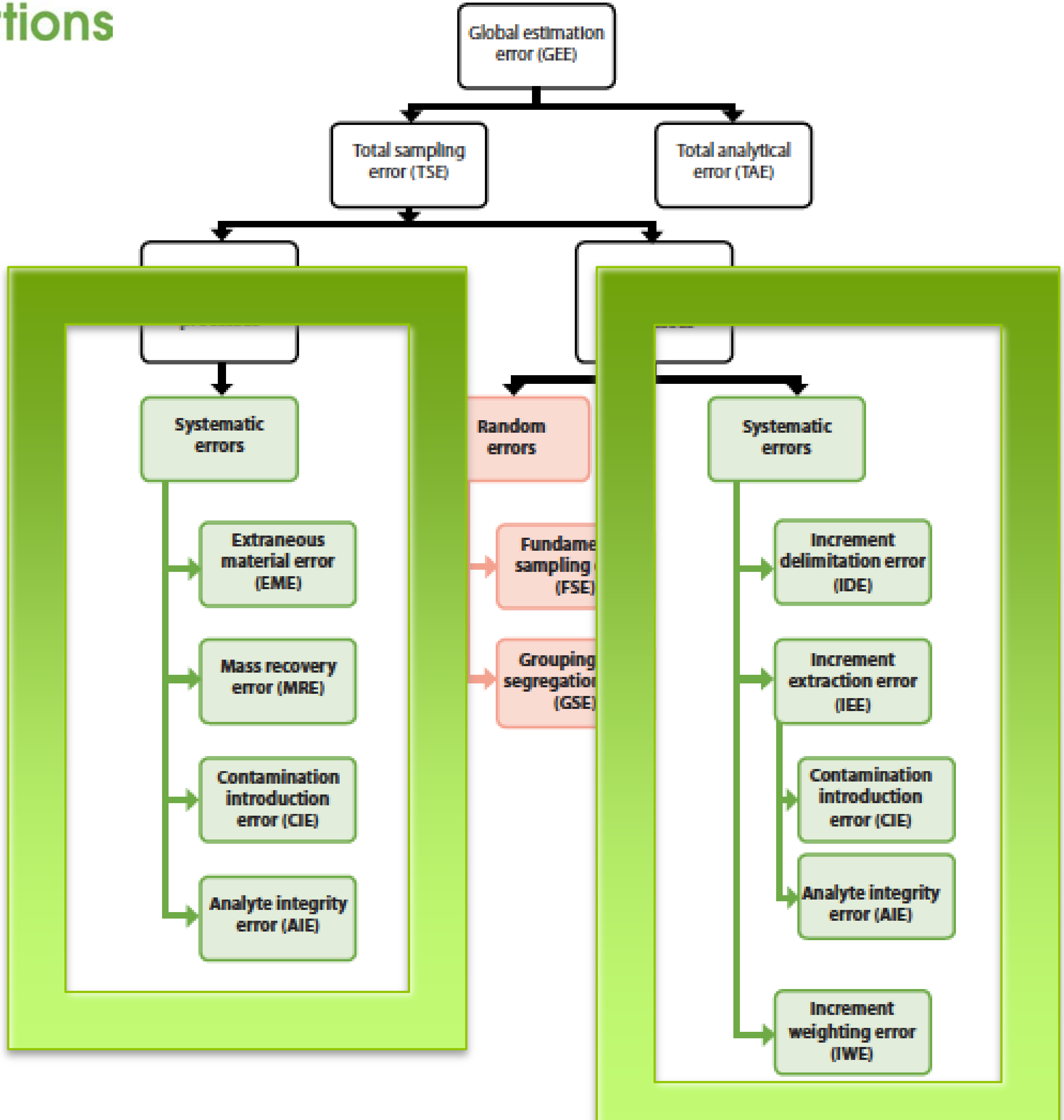
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SYSTEMATIC ERRORS (BIAS ERRORS)

- New systematic error terms introduced
- Systematic errors are impossible to estimate (unlike analytical bias errors).
- Causes of errors and practices to control errors are discussed.

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BLUNDERS

- Mistakes or accidents in the lab
- Data integrity is lost
- Blunders cannot be incorporated into a global estimation error (GSE) calculation, and must be prevented/eliminated or the procedure must be repeated

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SAMPLE CORRECTNESS

- Sample correctness is control of IDE and IEE
  - IDE occurs when all elements of a material do not have an equiprobable chance of being selected (function of tool design)
  - IEE occurs when all elements have an equal probability of being selection, but the correctly delimited elements do not become part of the increment (function of tool usage)
- Discussed for different states of materials

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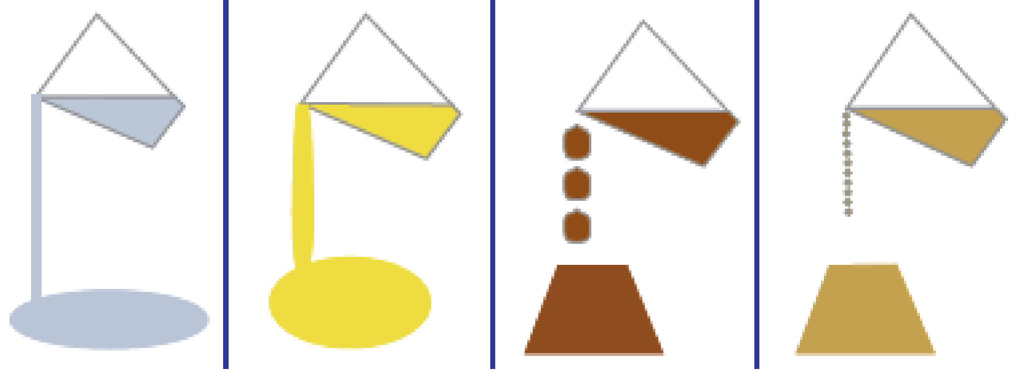
States of Materials

Liquid	Slurry	Semi-solid	Solid
• No visible particulates • Spreads	• Visible particulates • Spreads	• Moves like a liquid or solid • Stacks	• Moves like a solid • Stacks



INCREMENT DELIMITATION ERROR FOR LIQUIDS

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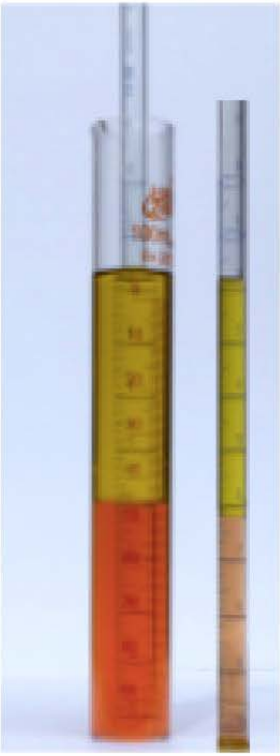
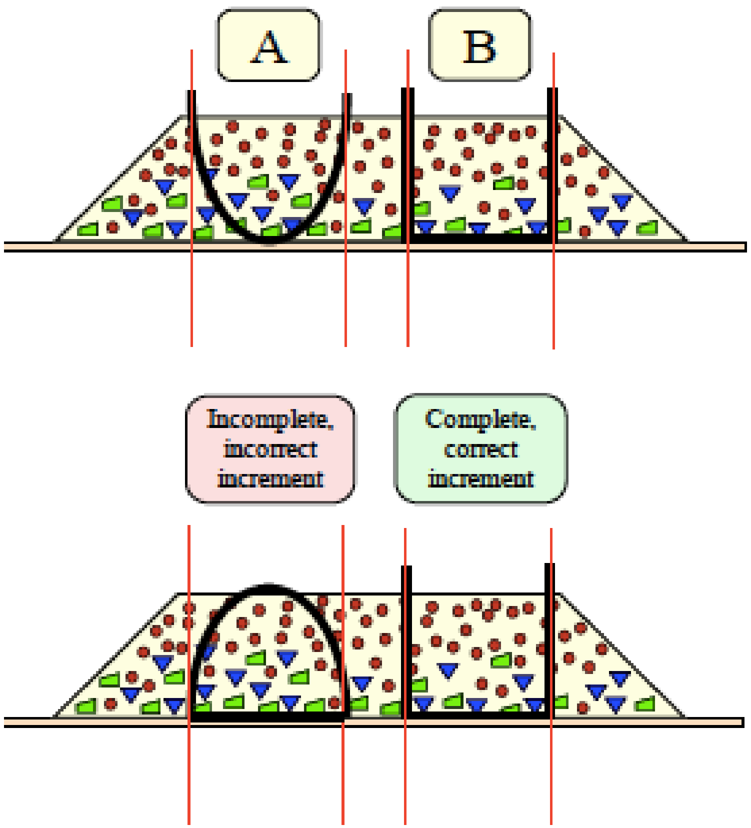
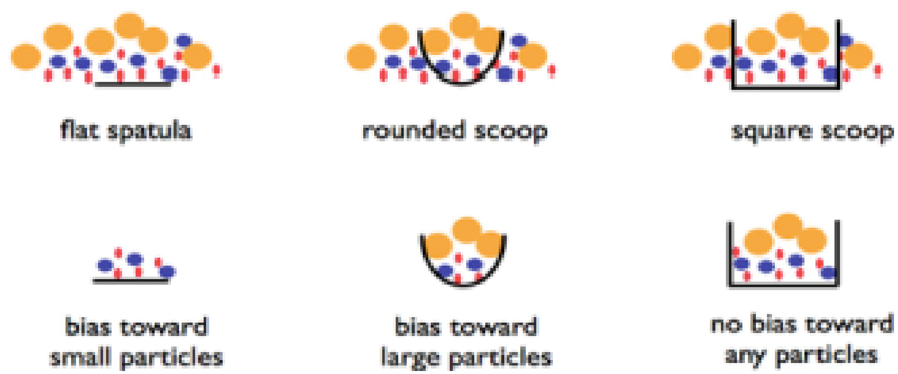
l **volumes** are represented proportionately



Equal volumes are represented disproportion

SAMPLE CORRECTNESS FOR SOLIDS





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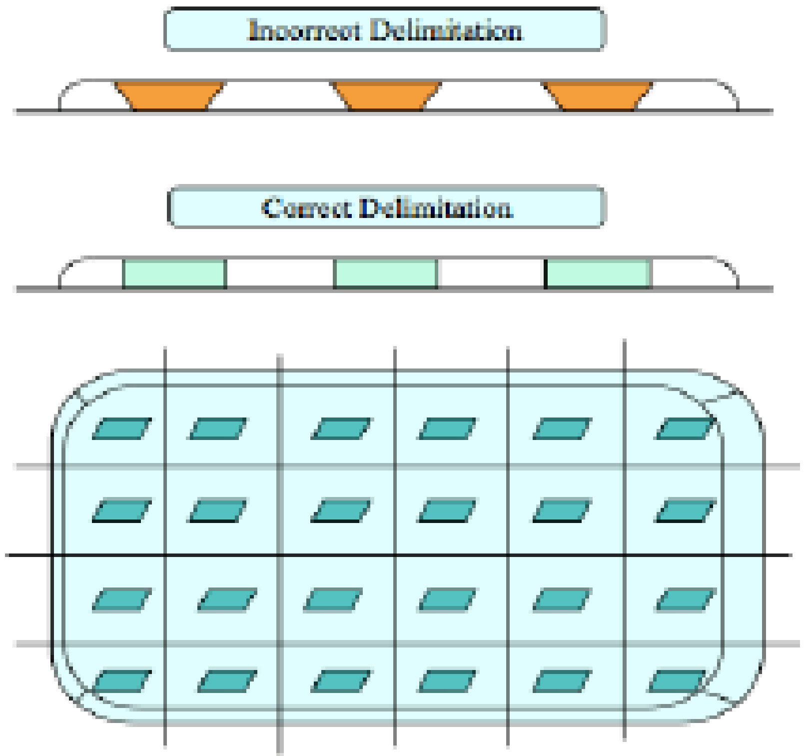
+ INCORRECT AND CORRECT DELIMITATION WITH A 2-D SLAB CAKE



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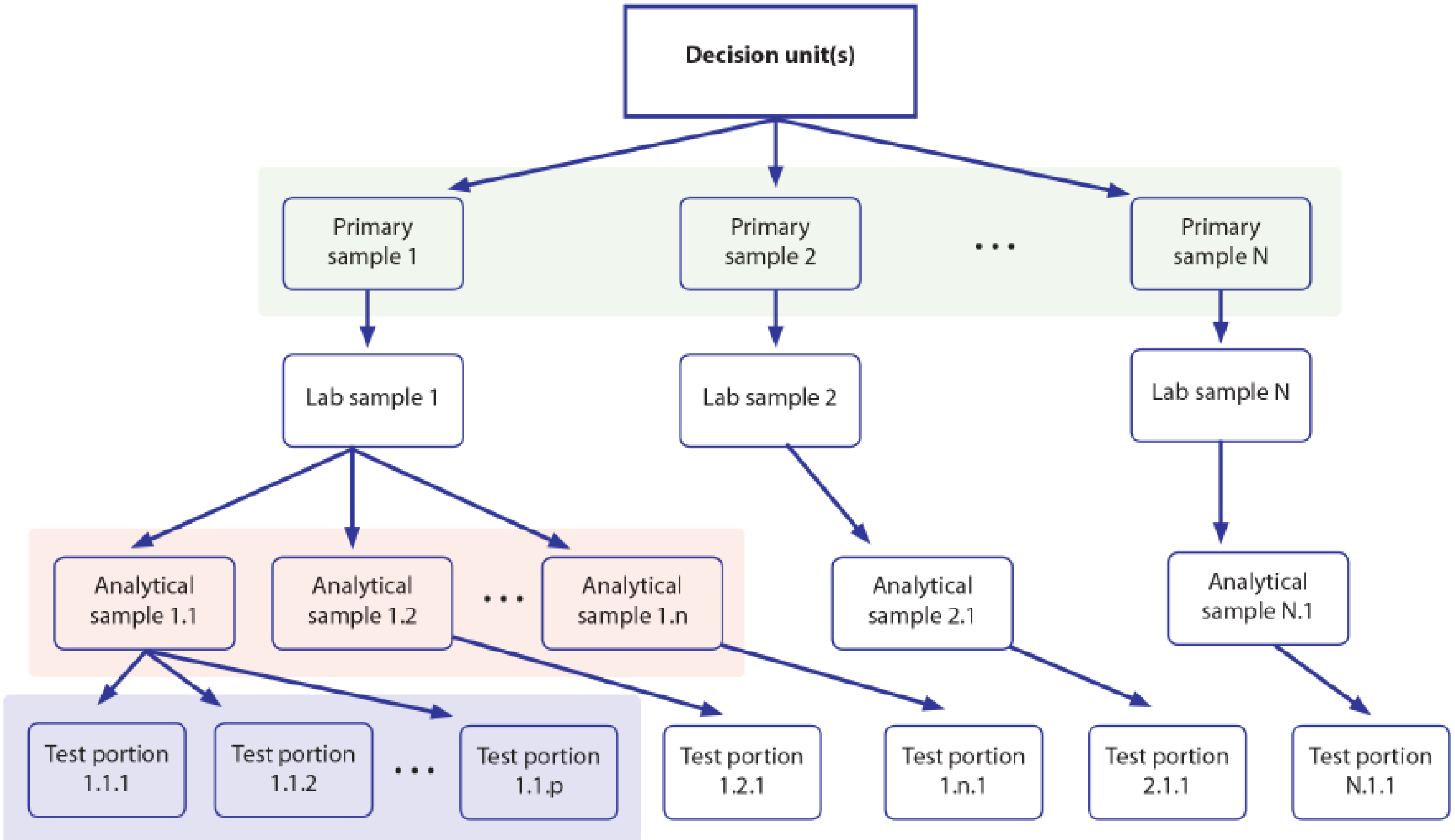
ESTIMATION OF ERROR



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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MAINTAINING INTEGRITY

- Purpose of evidentiary integrity
  - (1) trace-back information from the analytical result to receipt of the laboratory sample; and
  - (2) assurance that a sample has not been adulterated or compromised at any point from receipt through disposal.
  - (3)assurance that sampling systematic errors, random errors and blunders are sufficiently controlled to meet the SQC

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SNAPSHOTS

These are just a few snapshots of what is to come in GOOD Test Portions

Hopefully stretches your imagination in a new direction for a few minutes

Current document is ~ 70 pages of detail, so don't expect to grasp content from these snapshots

QUESTIONS?

THANK YOU!

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OR any of the WG Members