GUIDANCE ON OBTAINING DEFENSIBLE (GOOD) Test Portions

coming soon.....

Major Changes from 2000 Lab Sampling Guidelines

- Title change
- Changing the term "sample preparation" to "laboratory sampling"
- Emphasis on "Theory of Sampling" and SQC
- Introducing Selection vs Non–Selection processes
- Introducing new error terms and equations
- New options for validating laboratory sampling

Applying *GOODSamples* in the Laboratory

What are laboratories doing when testing?



Sample Quality Criteria

- Lab must be involved 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?

LAB INPUT INTO DEVELOPING SQC

- Identify a fit-for-purpose test method
- Identify and resolve analyte integrity issues
- Capacity and capability for laboratory sampling and testing
- Potential sources of contamination
- Contributions to Global Estimation Error (GEE)
- Determine the mass required for testing and QC

MATERIAL PROPERTIES

- Finite vs Infinite elements
- Comminution of a finite element material typically 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.
- > CH of a material is altered by comminution.
- DH is altered with any physical manipulation of the material (e.g., vibration causing segregation, pouring, comminution or mixing).

Example of DH



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

Total Sampling Error (TSE)

Three types of error

- Systematic Error
- Random Error
- Blunders

Total Sampling Error (TSE)



Figure 1. Relation between confidence, error, and representativeness.

RANDOM ERRORS

- Fundamental Sampling Error (FSE)
 - Function of particle size, mass and CH
 - Remains after all other errors are perfectly controlled
- Grouping and Segregation Error (GSE)
 - Function of number of increments and DH
 - DH is impacted with every handling

SYSTEMATIC ERRORS

- Many new systematic error terms will be introduced
- Systematic errors are extremely difficult, if not impossible to measure. Therefore, effort must be taken to ensure that all systematic errors are controlled to a point of being negligible through the entire laboratory sampling process.

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

MAINTAINING INTEGRITY

Traditionally the purpose of evidentiary integrity in the laboratory has been to establish (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.

MAINTAINING INTERGRITY

In additional to the traditional interpretation, evidentiary integrity assumes that the systematic errors, random errors and blunders are sufficiently controlled to meet the SQC. This includes maintaining analyte integrity and sample correctness, as described in GOODSamples; and also includes control of all other errors resulting from selection and non-selection processes.

LABORATORY SAMPLING

Laboratory Sampling consists of two major components.

- 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" within this document refers to both the non-selection and selection processes

ERRORS CONTRIBUTING TO GEE



QUESTIONS?

