



# GOOD SAMPLING DISCUSSION

## AAPFCO Administrators' Seminar

Tuesday, October 20, 2015

Nashville, TN

OFFICE OF THE TEXAS STATE CHEMIST

Texas Feed and Fertilizer Control Service • Agriculture Analytical Service

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# **GOOD*Samples*: Guidance on Obtaining Defensible Samples**

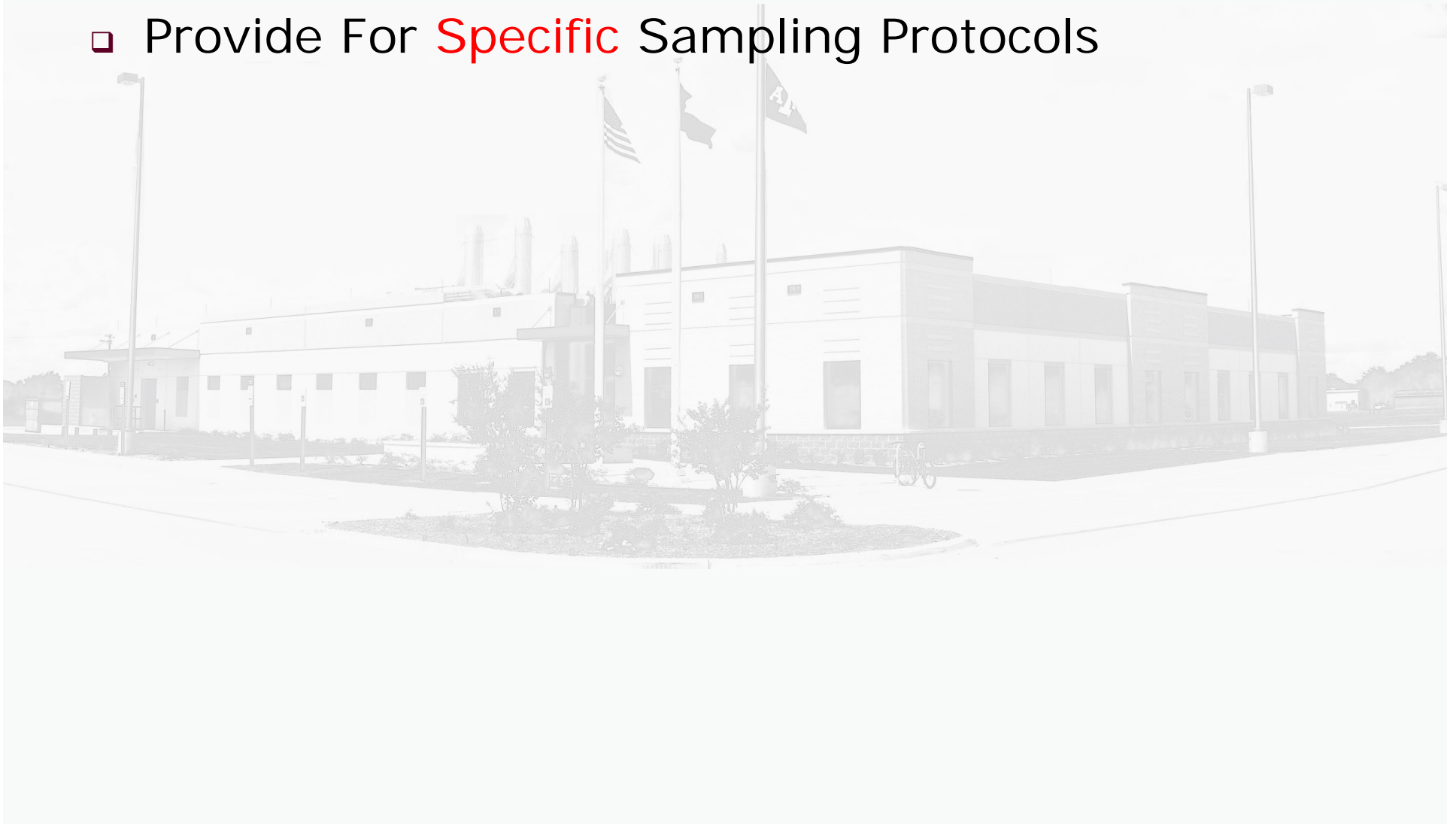
- ❑ FDA Cooperative Agreement #U18FD004710-01
- ❑ 100% Funded w/Federal Dollars from Federal Program of \$1.3 Million
- ❑ Association of Public Health Laboratories (APHL)
- ❑ Association of American Feed Control Officials (AAFCO)
- ❑ Association of Food & Drug Officials (AFDO)
- ❑ Collaborative Effort by Working Group
  - APHL, AAFCO, AFDO, and FDA

## What Does **GOODSamples** DO?

- ❑ Provides a Scientific and Systematic **Approach** to Develop or Evaluate Sampling Protocols
- ❑ Ensures the Sampling Process is Representative of the Decision Unit
- ❑ Ensures Analytical Results and Inferences/Decisions Are Defensible
- ❑ Improves Analytical Data Equivalency Among State, Federal, and Local Agencies to Enable Inter-Agency Sharing of Data Collected in Support of Food and Feed Regulatory Programs

# What Does GOOD*Samples Not Do?*

- Provide For **Specific** Sampling Protocols



# **GOOD Samples Representative Sample**

- ❑ Primary Sample, Lab Sample, Analytical Sample, and Test Portion
- ❑ Used to Provide Information About the Decision Unit (i.e. analytical result and inference)
- ❑ Information is Within the Acceptable Level of Confidence Defined by the Party
- ❑ Imprecision and Bias Are Controlled to the Acceptable Level

# Management Considerations

- ❑ Management Support is Critical for Implementation of *GOOD Samples*
- ❑ Determine Objectives for Sampling Plan
  - Purpose and Frequency for Sampling
  - Types of Commodities
  - Firms/Locations to be Sampled
  - Sampling Protocols
    - Procedures For Sampling
    - Sampling Tools
    - Sample Preparation and Shipment

## Management Considerations Cont'd

- Provide Adequate Resources and Support
  - Sufficient and Qualified Sampling and Lab Staff
  - Necessary Sampling Tools and Equipment
  - Conduct Periodic Training for Involved Staff
  - Ensure Safety and Health of Employees
  - Pre-Determine Consequences of Wrong Inference
  - Provide Adequate Facilities For Receipt, Handling, and Storage of Samples to Prevent Cross Contamination and Ensure Analyte and Evidentiary Integrity
  - Provide and Maintain Necessary Equipment for Mass Reduction, Particle Size Reduction, Sample Mixing, and Sample Storage

# Management Considerations Con't

- ❑ Coordination and Collaboration
  - Management, Sample Collector, Program, and Lab
- ❑ Training
  - The Scientific Basis For Testing
  - Theory of Sampling
  - Maintaining Sample Correctness/Analyte Integrity
  - Quality Control (QC)
  - Evidentiary Integrity
  - Selection, Use, and Maintenance of Tools and Equipment



# Sample Quality Criteria (SQC)

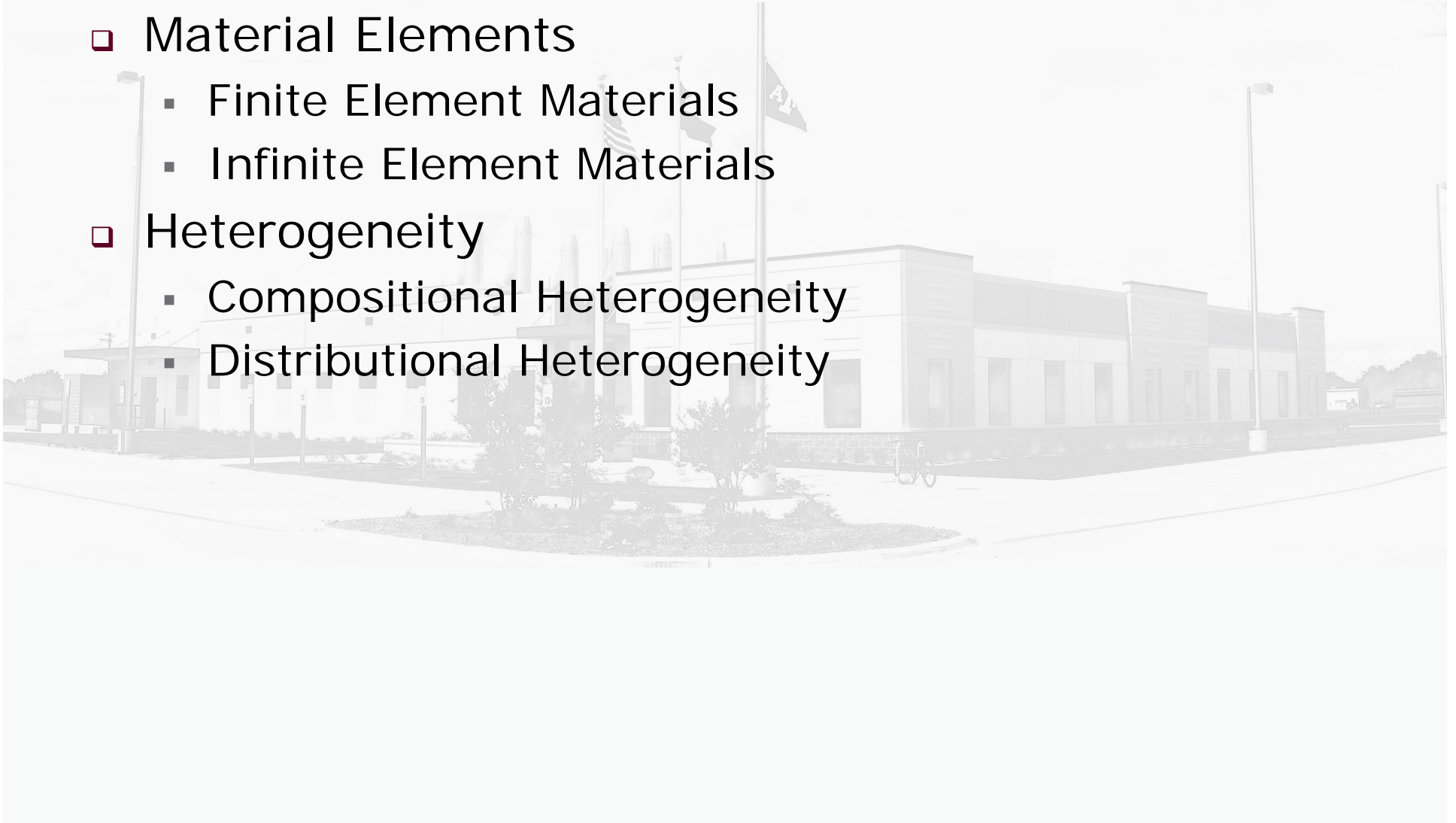
- SQC Ensures
  - All Stakeholders Agree On What Needs To Be Done
  - The Representativeness and Applicability of All Analytical Results
  - An Optimal Allocation of Resources
  - The Final Data is of Sufficient Quality to Make a Defensible Inference/Decision
  - The Equivalency of Lab Data
  - The Harmonization of Inference/Decision Making

# Sample Quality Criteria (SQC) Con't

- ❑ Determined Based on the Answers to the Following Questions
  - What is the Question to be Answered
  - What is the Decision Unit
  - What is the Desired Confidence in the Inference
- ❑ After Answering These Questions, the Sample Design Protocol Can Be Developed

# Material Properties of the Decision Unit Impact Design of Sample Protocol

- ❑ Material Elements
  - Finite Element Materials
  - Infinite Element Materials
- ❑ Heterogeneity
  - Compositional Heterogeneity
  - Distributional Heterogeneity



# Theory of Sampling

- ❑ Sampling Errors
  - Fundamental Sampling Errors
  - Grouping and Segregation Errors
  - Sample Correctness
  - Total Sampling Error
  - Summation of Errors
- ❑ Primary Sampling Issues
- ❑ Lab Subsampling Issues

# Sample Correctness, Sampling Tools, and Mass Reduction Equipment

- ❑ Sample Correctness
  - All Elements Within Decision Unit Have An Equiprobable Chance of Selection During the Sampling Process
  - The Increments Are Proportionate
- ❑ Sampling Tools
  - Depends on Decision Unit Dimension
    - Zero-Dimensional
    - One-Dimensional
    - Two-Dimensional
    - Three-Dimensional

# Sample Correctness, Sampling Tools, and Mass Reduction Equipment Cont'd

- ❑ Automated Sampling Tools
- ❑ Splitting Tools to Reduce Mass of Primary Sample
  - Rotary Splitter
  - Fractional Shoveling
  - Stationary Riffle Splitter

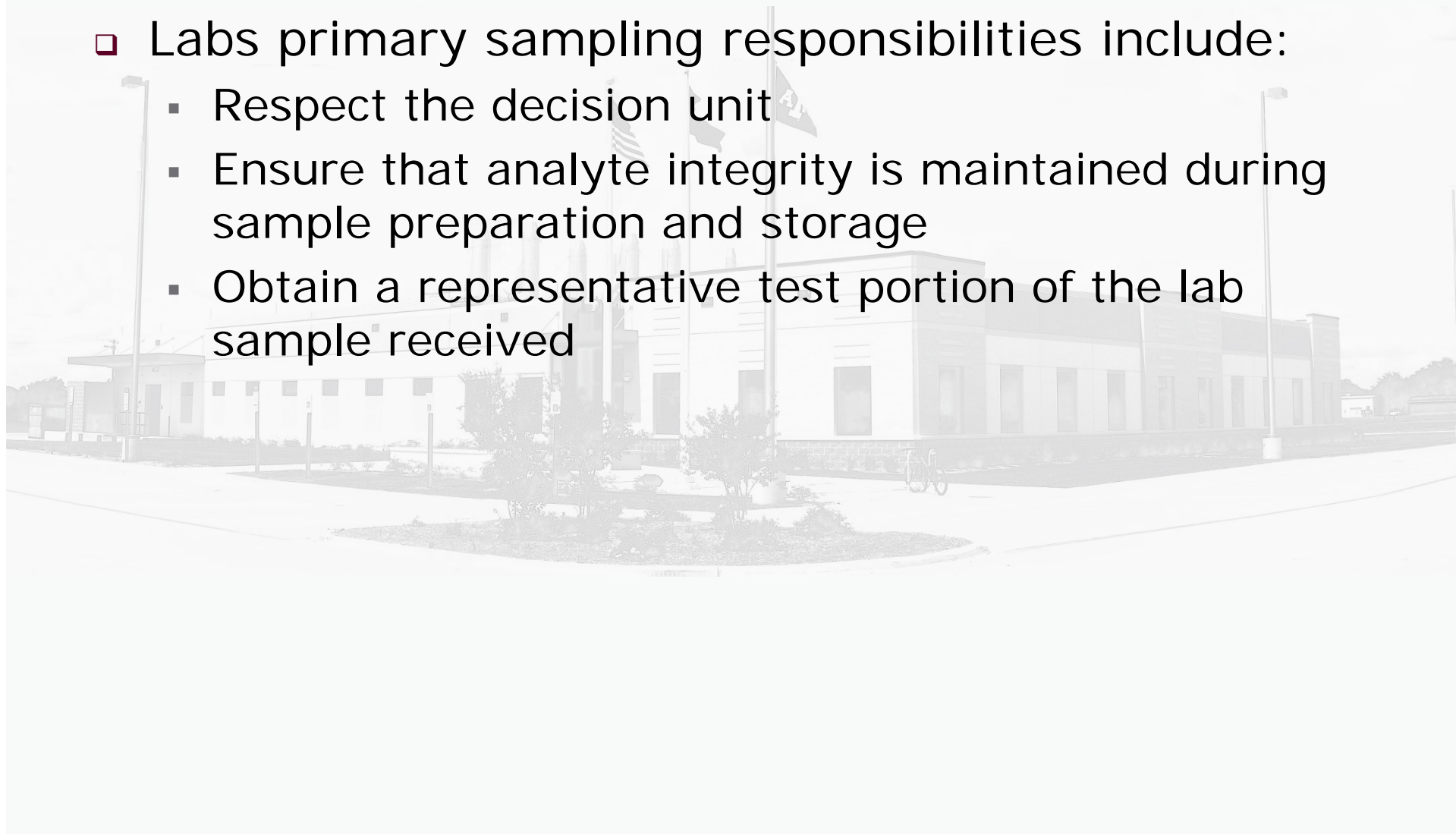


# Evidentiary and Analyte Integrity

- ❑ Evidentiary Integrity Ensures That the Evidence Has Not Been Compromised From Sample Collection Through the Generation of the Analytical Results (e.g. Chain of Custody)
- ❑ Analyte Integrity Ensures No Conditions Are Introduced That Would Impact the Analyte Concentration or Analytical Result (i.e. Oxidation, Microbial Viability, Volatilization, Degradation, Contamination, Temperature, Light, and Packaging and Shipping)

# Laboratory Sampling and Preparation

- Labs primary sampling responsibilities include:
  - Respect the decision unit
  - Ensure that analyte integrity is maintained during sample preparation and storage
  - Obtain a representative test portion of the lab sample received





# Quality Control (QC)

- ❑ Quality Control is Used To Assess
  - Data Quality
  - Monitor Process Control
  - Validate Methods
- ❑ Every Step in the Sampling Process (e.g. Collection, Splitting, Containerizing, Mass Reduction, and Preparation) Introduces Error
- ❑ Quality Control For Individual Samples is Used To
  - Estimate the Error in the Test Result For a Specific Decision Unit

## Quality Control (QC) Cont'd

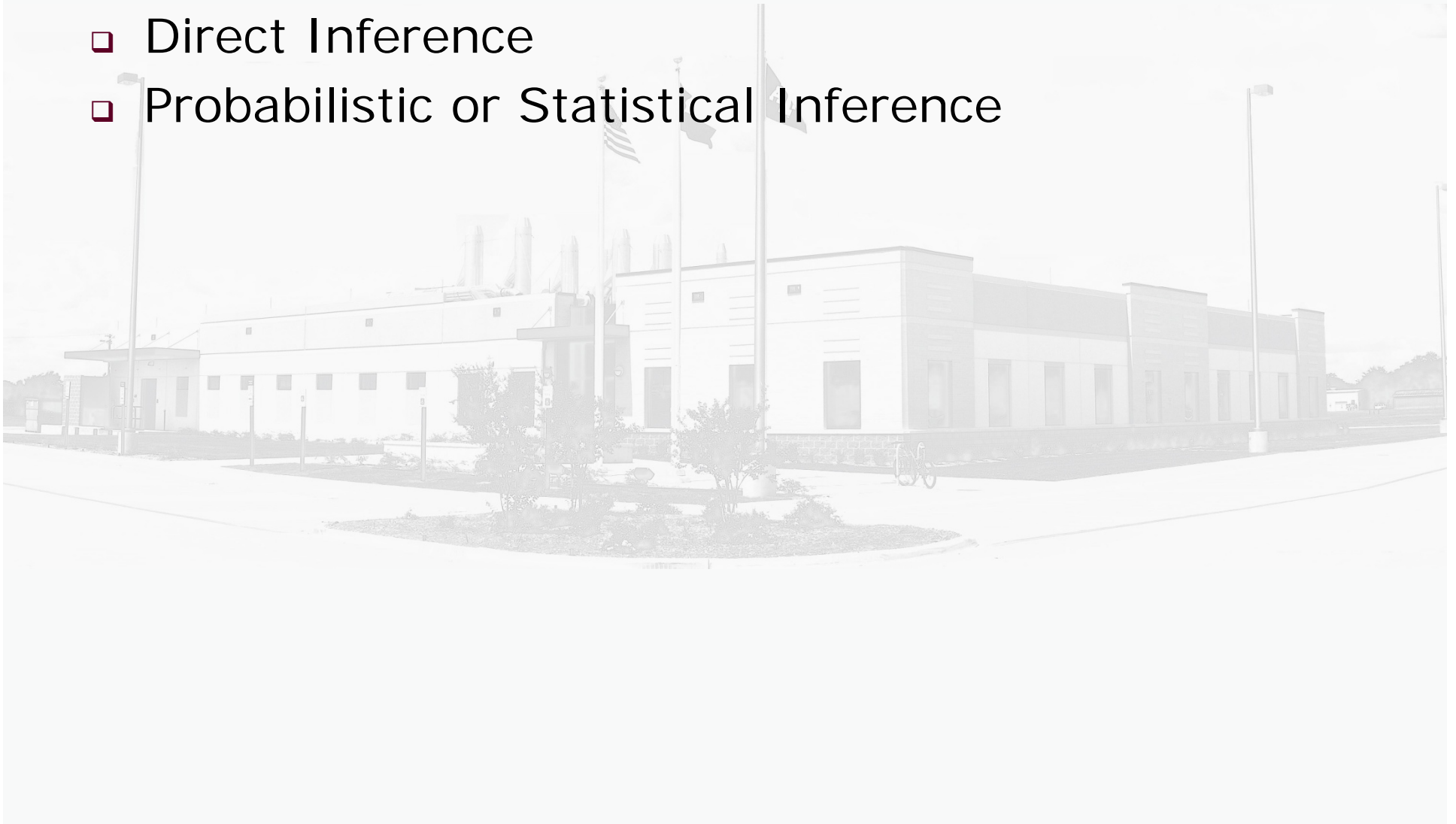
- ❑ Quality Control and Replicate Samples Can Be Used to Estimate the Imprecision of All the Mass Reduction Stages
- ❑ Replicate Samples Can Be Used to Estimate the Global Estimation Error (GEE)
- ❑ Quality Control Replicates Include
  - Primary Sample Replication
  - Analytical Sample Replication
  - Test Portion Replication

# Inference

- ❑ Process of Estimating a Concentration or Characteristic About a Decision Unit Based on the Primary Sample Collected From The Decision Unit
  - Estimation of an Average Analyte Concentration of a Decision Unit From a Sample of That Decision Unit
  - Estimation of the Percentage (Proportion) of Decision Units That Have Some Specific Concentration or Characteristic Based on Sampling Multiple Decision Units

# Inference Cont'd

- ❑ Direct Inference
- ❑ Probabilistic or Statistical Inference



# Discussion Points

- ❑ Feed vs Food vs Fertilizer
- ❑ AAFCO AVs vs AAPFCO IAs
- ❑ Macro vs Micro vs Contaminant
- ❑ **MORE MASS!**
- ❑ **MORE INCREMENTS!**

# Resources

- ❑ [www.aafco.org](http://www.aafco.org)
- ❑ Under Publications
- ❑ *GOODSamples*
- ❑ Journal of AOAC International, Volume 98, February-March

