



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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RESEARCH

GOOD*Samples*: Guidance on Obtaining Defensible Samples

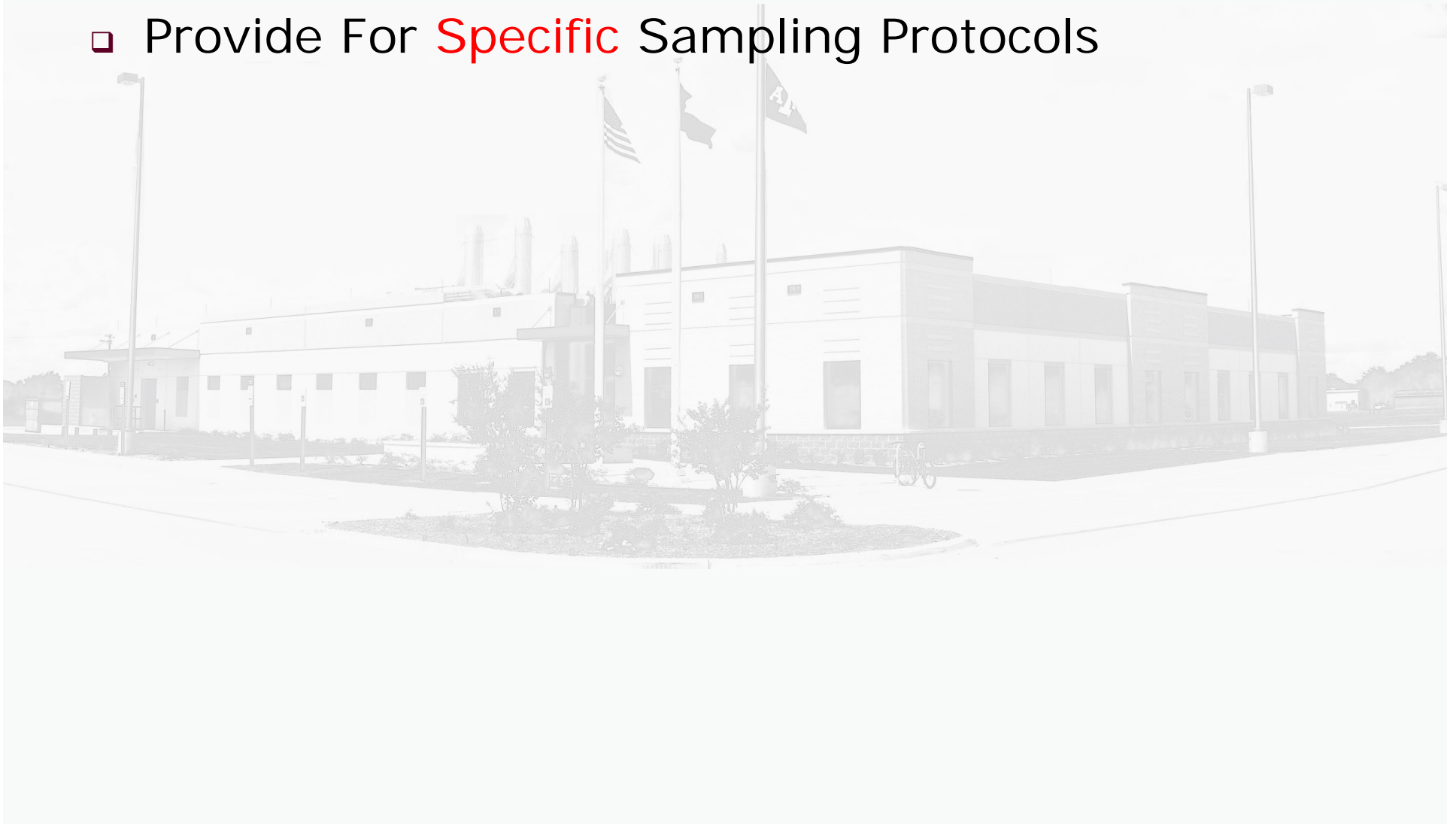
- ❑ FDA Cooperative Agreement #U18FD004710-01
- ❑ 100% Funded w/Federal Dollars from Federal Program of \$1.3 Million
- ❑ Association of Public Health Laboratories (APHL)
- ❑ Association of American Feed Control Officials (AAFCO)
- ❑ Association of Food & Drug Officials (AFDO)
- ❑ Collaborative Effort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

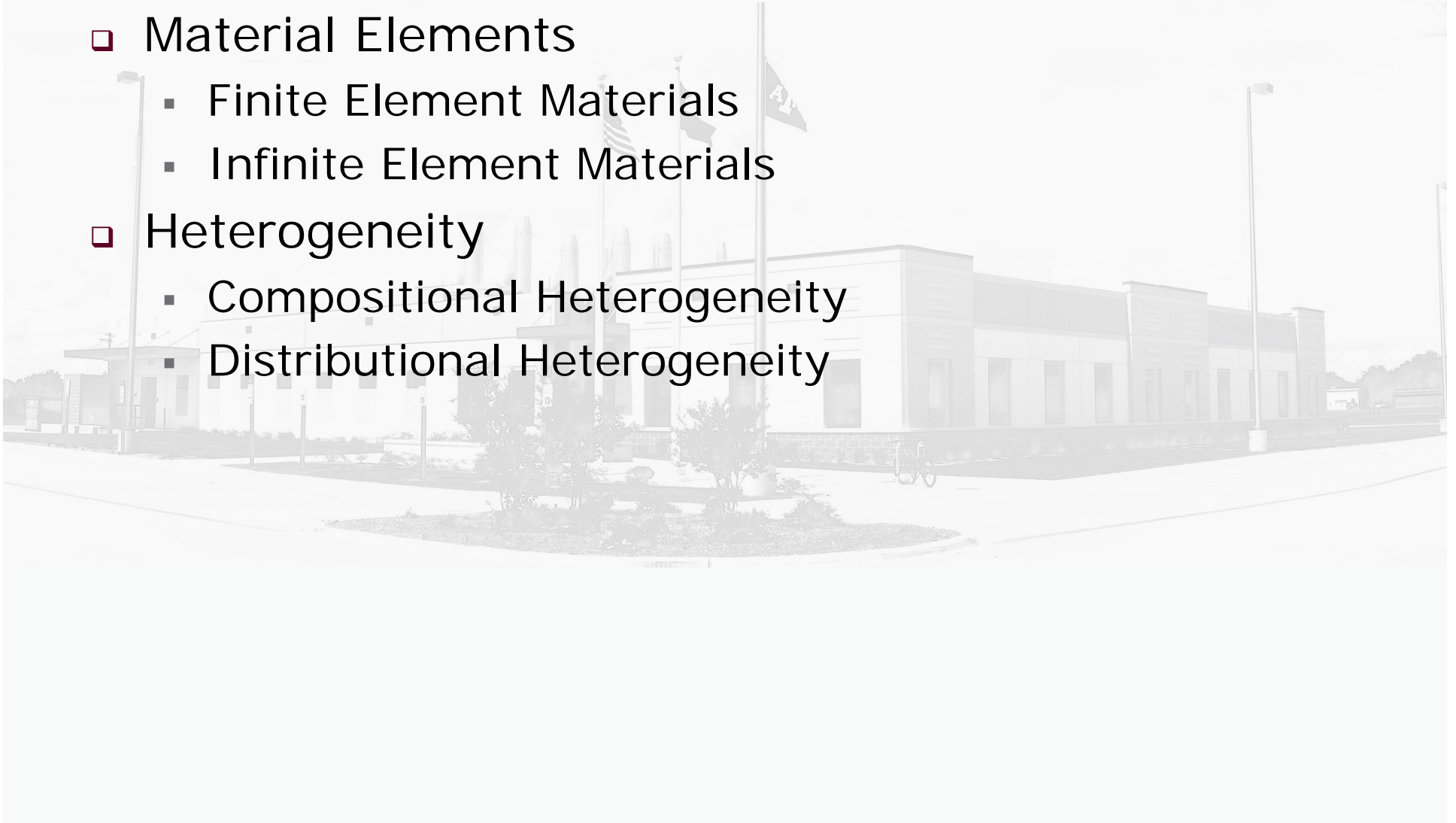
- SQC Ensures
 - All Stakeholders Agree On What Needs To Be Done
 - The Representativeness and Applicability of All Analytical Results
 - An Optimal Allocation of Resources
 - The Final Data is of Sufficient Quality to Make a Defensible Inference/Decision
 - The Equivalency of Lab Data
 - The Harmonization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

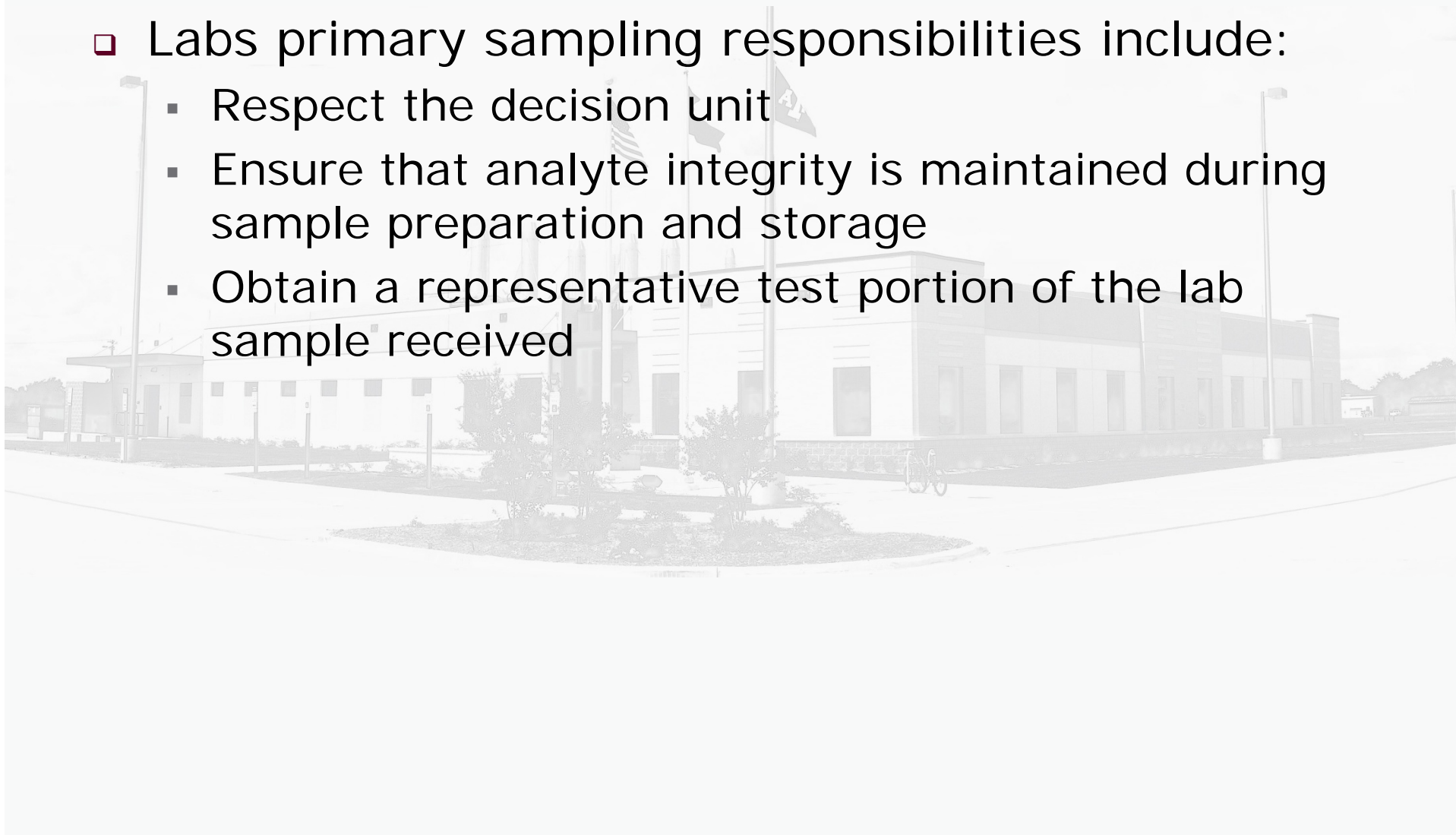


Evidentiary and Analyte Integrity

- ❑ 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

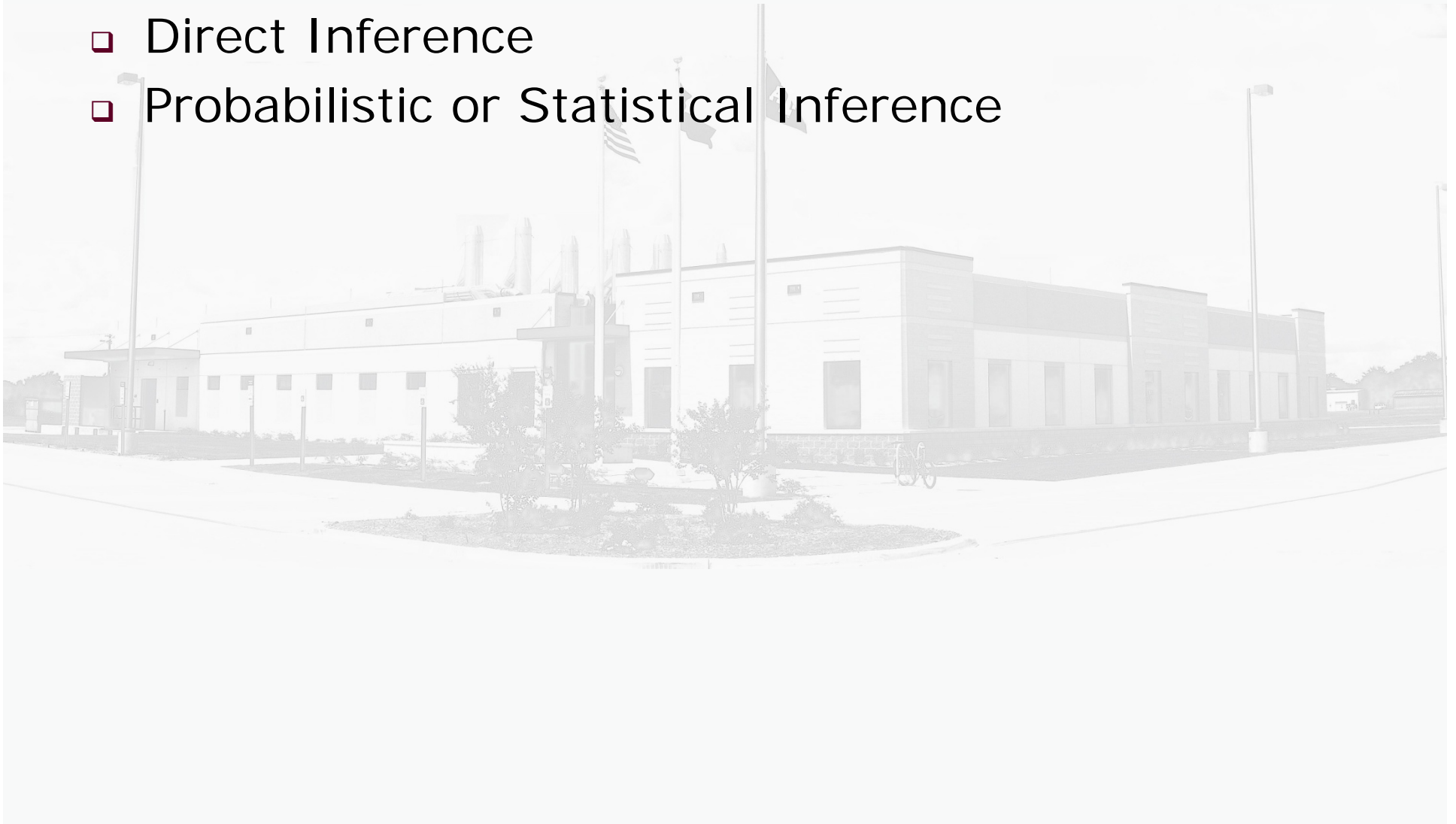
- ❑ Quality Control and Replicate Samples Can Be Used to Estimate the Imprecision of All the Mass Reduction Stages
- ❑ Replicate Samples Can Be Used to Estimate the Global Estimation Error (GEE)
- ❑ Quality Control Replicates Include
 - Primary Sample Replication
 - Analytical Sample Replication
 - Test 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
- ❑ Under Publications
- ❑ *GOODSamples*
- ❑ Journal of AOAC International, Volume 98, February-March

