WEBINAR

Critical Considerations for Laboratory Sample Submissions

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Agenda

01 Sample Quality Criteria

02 Rethinking Sample Receiving

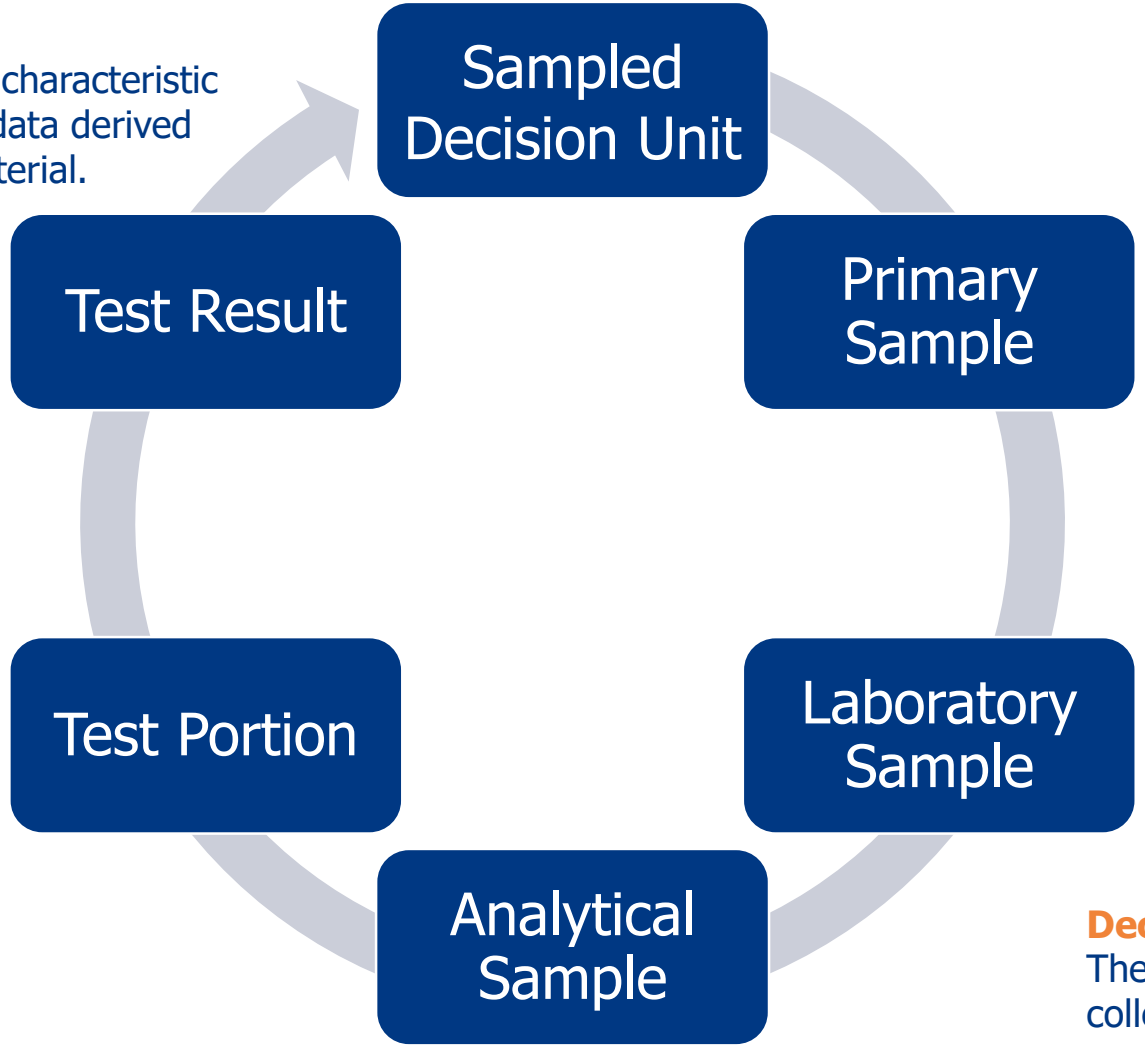
03 Rapid Response for Field Sampling

04 Selecting the Correct Method of Analysis

05 What to Do When an Applicable Test Method is Not Available

Hi, I’m calling about your test result…

Inference:
The estimation of a concentration or characteristic of a larger amount of material from data derived from testing a smaller amount of material.



Decision unit:
The material from which a primary sample is collected and to which an inference is made.

Sample Quality Criteria

A Sample is Representative if:

- It is correct
  - Correctness is maintained when biases are not introduced during the sample collection and preparation processes
- Small imprecision
  - The subsample should have the same ratios of components as the decision unit

Decision Unit

- The bulk lot of food/feed/ingredient, etc. to which inference is made
- Must be accessible
- The source from which increments are collected

Sampling Theory (SUPER brief introduction)

Minimum Mass.

- Control of fundamental sampling error (FSE) is as important for the collection of the primary sample as in the laboratory.
- FSE is especially critical in the laboratory where very small test portion masses are taken for analysis.
- Collecting random increments is generally considered the greatest challenge in the collection of the primary sample.
- The consequences of FSE are usually not considered by the laboratory when producing a result.

Minimum Number of Increments.

- Multiple increments is critical in controlling distributional heterogeneity.
- Collection of random increments is easy due to the small mass (complete accessibility) of most laboratory samples.
- A common practice of sampling is taking a single increment during mass reductions steps; however,
- A single increment is rarely acceptable for defensible sampling.

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Sample Receiving

Shipping Conditions

Distribution

Storage Conditions

Mixing

Sub-Sampling (Splitting)

Comminution (Grinding)

Sample Receiving

Shipping Conditions

Laboratory samples arrive at ambient conditions, with cold packs or dry ice, and in insulated and uninsulated packaging. Always be sure to send your samples at the conditions that will best protect their integrity, and be prepared to make an appropriate decision to proceed or hold testing if shipping time is extended.

Storage Conditions

After the sample has reached the laboratory, they are stored either at the conditions that they arrived or as otherwise specified by the sample analysis request form or other package documentation. If the laboratory retains a sample for potential retesting, be sure to specify the long-term storage conditions.

Sub-Sampling (Splitting)

It may be necessary to subsample a laboratory sample if multiple analyses are required for the same sample. If an analysis specifies an optimal amount, be sure to send enough sample to meet the "optimal" requirements, not the minimum.

Comminution (Grinding)

Depending on the analysis type, laboratory samples are often processed to produce a uniform consistency. Be sure to include information about the sample (e.g. high-moisture, heat sensitive) that may be helpful for the laboratory to complete this process so that it does not impact the analytical result.

Mixing

There are very different techniques that are used to reduce the distributional heterogeneity. Always tell the laboratory what the expected concentration of the target analyte in case a non-routine mixing technique is needed to give a representative and reproducible result

Distribution

The laboratory areas where sample testing may be separated by walls or miles. Some laboratories that perform microbiological testing may not perform any chemical analysis. Transport may affect sample integrity, so consider sending duplicate samples with you know that testing for the same sample is performed in different locations.

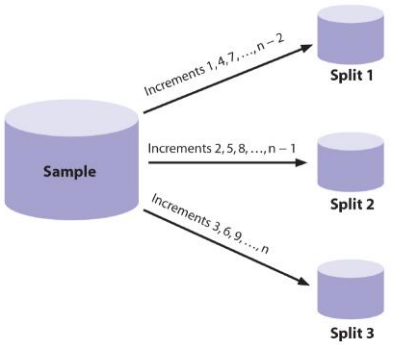
Sub-sampling (Splitting)

LIQUID SAMPLES

- Single-phase liquid without solids
  - Pour or remove an increment with any tool
- Multiphasic liquid without solids
  - Adequately mix then pour or withdraw
  - Alternatively, a cylindrical core sample can be taken from a container with vertical walls
- Liquids with suspended solids
  - Sample liquid and solids separately, or
  - Withdraw a cylindrical core
- Slurries
  - Cylindrical core with an open tube

SOLID SAMPLES

1. Stationary Splitting
2. Rotary Splitting
3. Fractional Shoveling



Mass Reduction:
The process of selecting a smaller mass from a larger mass.

Example Sample Type Classifications

Food

Cake mix, ice cream, blueberries

Food samples are either designed to be shelf stable at ambient conditions, or they will require refrigerated or frozen conditions.

Beverage

Sports drinks, tea, concentrates, soda

Depending on the processing and additives, beverages may require ambient, refrigerated or frozen conditions.

Feed

Pet food, cattle feed, liquid feeds

Dry pet food is typically designed to be shelf-stable. Some specialty diets and formulas without preservatives and fresh ingredients will likely require refrigerated or frozen conditions.

Infant Formula

Dry, Liquid, dairy-free

Most infant formulas are designed to be shelf-stable; however, depending on the region and seasonal temperatures, shipping with cold packs in insulated packaging may be recommended for some formulas.

Supplements

Powders, liquids, tablets, capsules

Supplements can have a recommended storage temperatures that range from ambient to frozen. They may also have ingredients that are vulnerable to accelerated degradation, so they should be shipped at the conditions that are recommended for long-term storage.

Dietary Ingredients

Echinacea, psyllium, whey protein

Dietary ingredients also have a wide range of recommended storage conditions. Many dietary ingredients have been stabilized for storage at ambient conditions, but fresh ingredients or those with out preservatives may require refrigerated or frozen conditions.

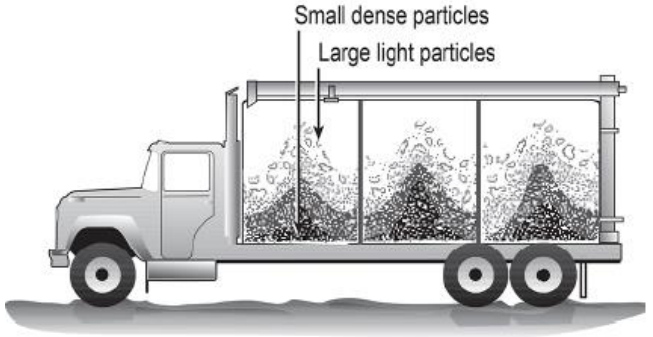
Material Properties

Compositional Heterogeneity

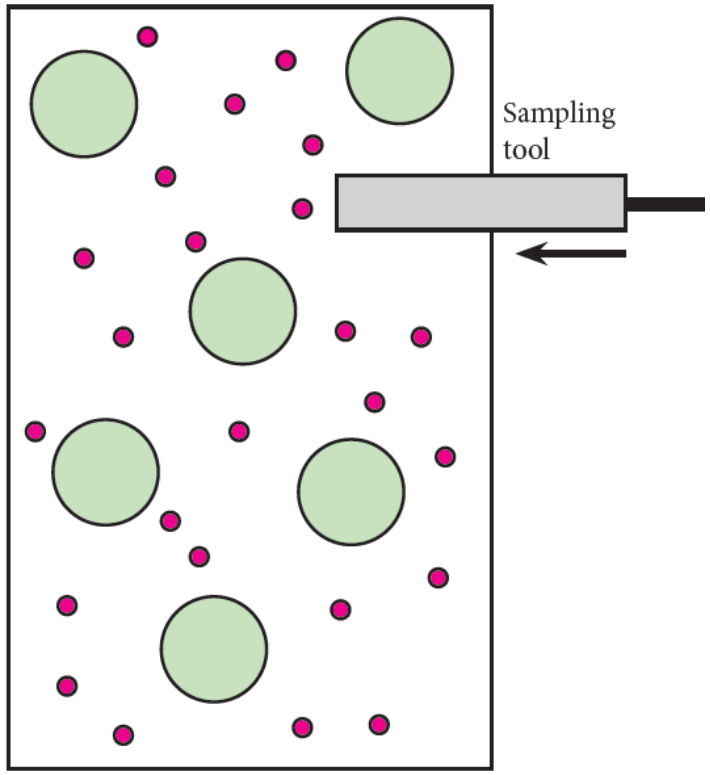
The heterogeneity arising from differing composition of the analyte of interest among individual elements (e.g., particles) in a material.

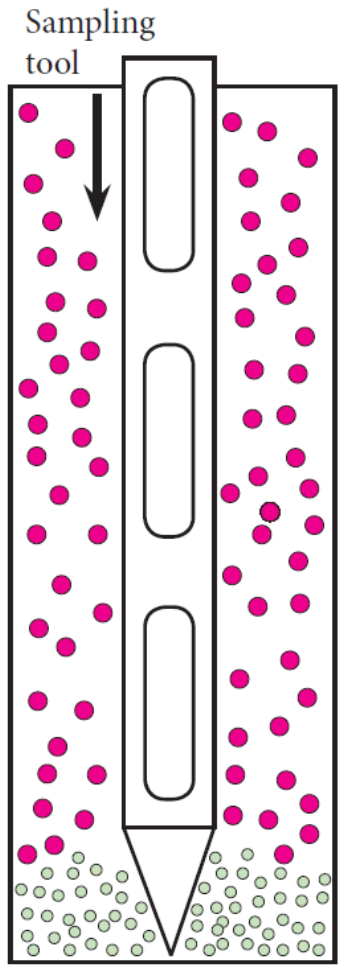
Distributional Heterogeneity

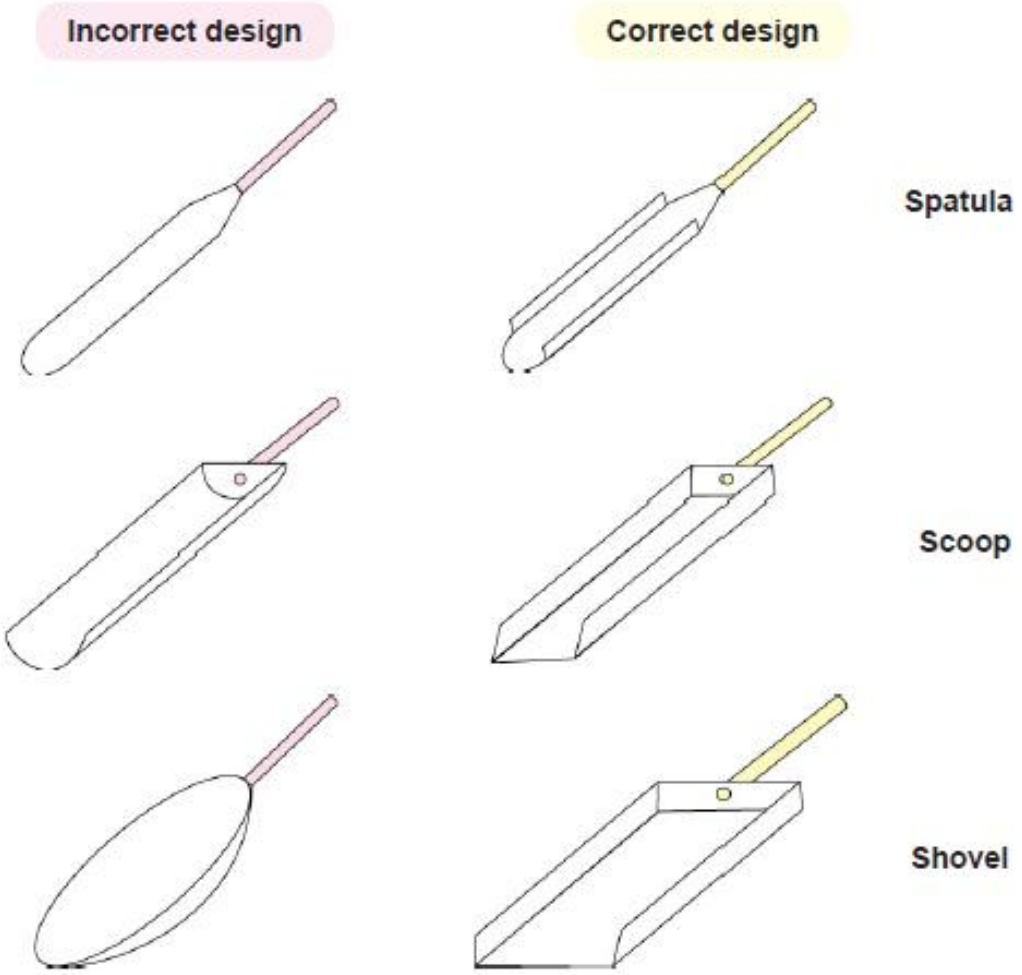
The inherent heterogeneity arising from the nonrandom spatial or temporal distribution of elements in a material; heterogeneity inherent to the manner in which elements are scattered within the material. The greater the difference in composition between elements, the greater the possible distributional heterogeneity.



Laboratory Sample Collection Best Practice Considerations







How Much Sample is Needed?

Fundamental Sampling Error (FSE):
Imprecision error due to compositional heterogeneity.

$$s^2_{FSE} = \left(\frac{1}{m_S} - \frac{1}{m_L} \right) \frac{cflgd^3}{m_S}$$

$$m_S = \frac{IH_L}{s^2_{FSE}}$$

c is the mineralogical factor (expressed in g/cm^3);
f is the particle shape factor (dimensionless);
g is the granulometric factor (dimensionless);
l is the liberation factor (dimensionless);
d is the largest particle diameter (expressed in cm;
m_S is the mass (expressed in g) of the selected portion (sample mass);
m_L is the total mass (expressed in g) from which m_S is selected;
s is the standard deviation (relative);
a_L is the proportion of the liberated material to the entire mass, and
λ is the density of the liberated material (expressed in g/cm^3).

Constant Factor of Compositional Heterogeneity (IH_L):
the grouping of all the constants $cfgd^\beta$.

Case Study
Total Mixed Ration (TMR) Cattle Feed

Problem

- High-moisture, non-homogeneous sample matrix

Approach

- Some target analytes are heat-sensitive; friction should be minimized
- Cryogrinding prevents accumulation of residue on milling equipment

Solution

- A reduction to "analytical fine" consistency

“Lab Sample”

http://www.aafco.org/Publications/**GOODTestPortions**

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Rapid Response for Field Sampling

What?

- Contamination
  - Food-borne pathogen
  - Industrial contaminants
  - Mycotoxins
  - Heavy metals
- Out of Specification
  - Adulterated products
  - Counterfeit products
  - Nutrient claims
  - Complaints

How?

- Deploy a regional *qualified* field sample collection technician.

Case Study
Soybeans

Problem

- There was a report that a product produced from a lot of soybeans was contaminated with *Listeria*.

Approach

- A regional field sample technician is deployed to collect samples from other batches of soybeans produced in the same lot that were in storage in a distribution center warehouse.

Solution

- Testing of field samples confirmed that the contamination was present in other batches of the same lot.

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Matrix Types

Food

Beverages

Feed

Infant Formula

Supplements

Ingredients & By-products

Biological Fluids

Tissue

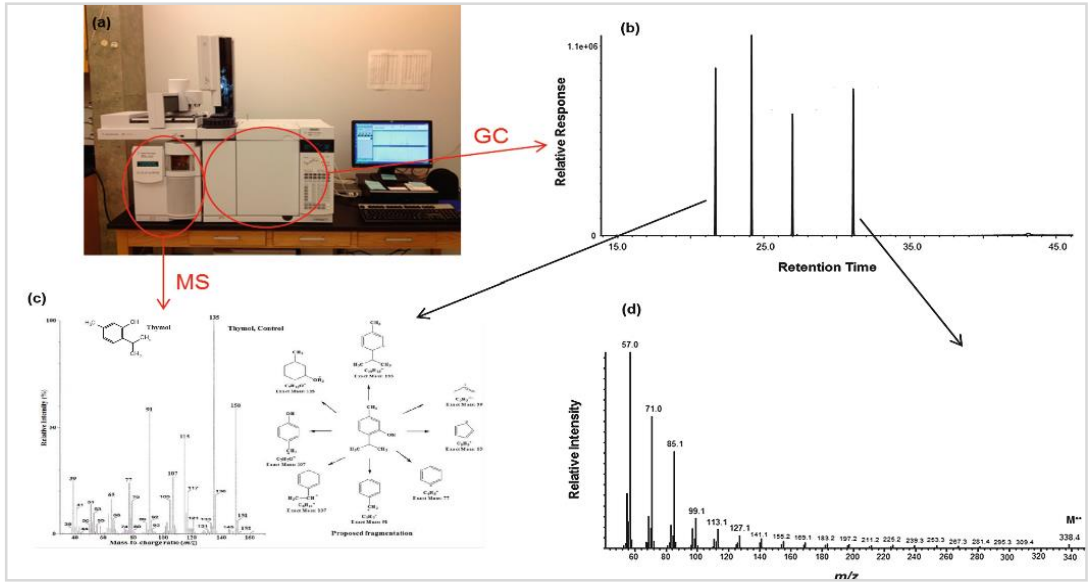
Standard or “Compendial” Methods of Analysis

U.S. Pharmacopeia

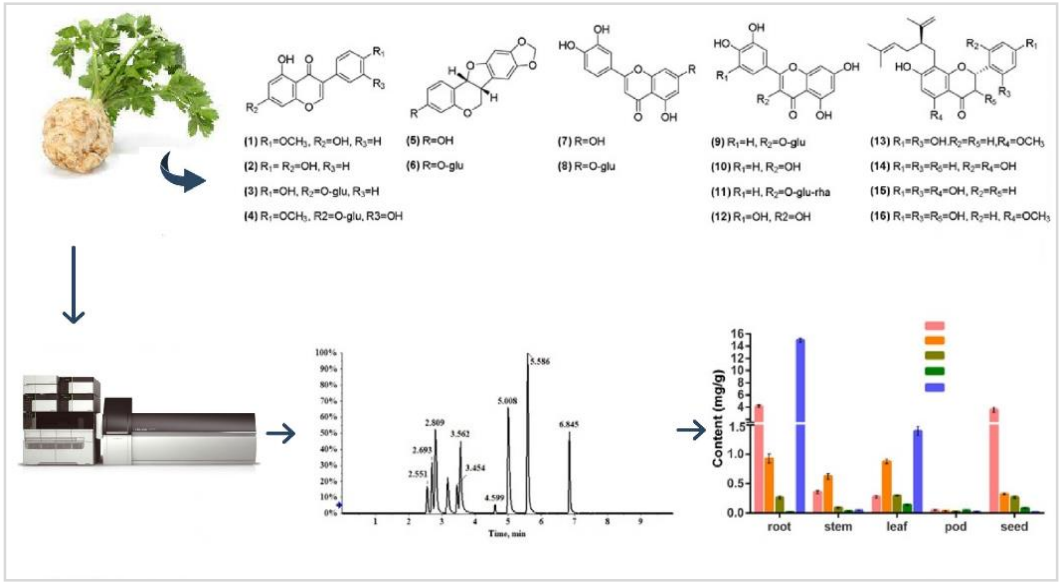
European Union Reference Laboratory

AOAC

Internally-developed or Specialty Analyses



Gas Chromatography Mass Spectrometry



Liquid Chromatography Mass Spectrometry

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Analytical Method Development

OPTION 1

Modify a Standard or "Compendial" Method

+	−
- Minimal modification - Lower cost - Less work - Less time	- Restricted to outdated technology - May require specialized equipment

OPTION 2

Develop a New Method

+	−
- Directly applicable to the sample matrix and analyte - Utilizes current technology	- Longer timeline - Complex - Higher cost

Case Study
Carvacrol, Thymol and p-Cymene in Feed

Problem

- A standard method is available for the dietary ingredient, but the method lacks specificity.

Approach

- The analytes are volatile, and the reference method utilizes a Flame ionization-type detector, which is not capable of resolving between co-eluting or very close chromatographic peaks, so a method that is less vulnerable to interferences was needed.

Solution

- The analytical procedure was modified to detect the analytes based on their molecular weight using a mass spectrometer.

Choosing the Right Partner

OPTION 1

Internal Method Development

+	−
- Lower cost	- More work
- Less external communication	- Requires significant project management
- Your team knows exactly what you need	- May not be accepted

OPTION 2

Choose a CRO or Specialty Research Laboratory

+	−
- Experienced in method development activities	- Higher cost
- Less time and troubleshooting	- Requires accurate communication

Case Study
Melamine in Infant Formula and Pet Food

Problem

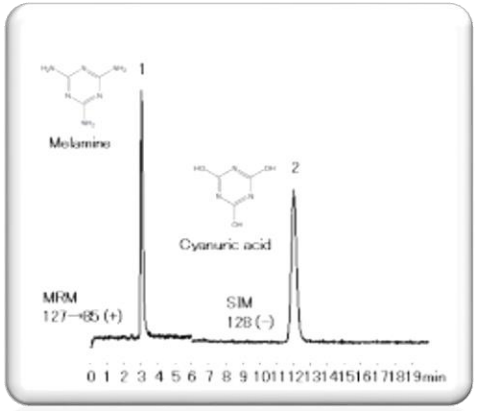
- Melamine and melamine-related compounds were found in products labeled as wheat gluten and rice protein concentrates. These products were unethically used to artificially boost quantifiable protein in infant formula and pet foods

Approach

- A method of analysis that is specific to the detection of melamine and melamine-related compounds was developed.

Solution

- A liquid chromatography triple quadrupole tandem mass spectrometry (LC-MS/MS) method developed for feed was adapted for residues of cyanuric acid and melamine developed by the FDA Center for Veterinary Medicine has been adapted for use with infant formula.



Who can I trust to develop an analytical method?

WEBINAR

Title: ANALYTICAL METHOD DEVELOPMENT

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- http://www.aafco.org/Publications/GOODTestPortions
- http://www.aafco.org/Publications/GOODSamples

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QUESTIONS?


thank you
danke
謝謝
ngiyabonga
teşekkür ederim
gracias
merci
obrigado
dziękuję
bedankt
go raibh maith agat
sukriya
kop khun krap
terima kasih
mochchakkeram
dank je
tapadh leat
grazie
arigatô
dakujem
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mauruuru
hvala
спасибо
рахмат
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