Methods and Specifications

Practical considerations for methods, specifications and applications of principles in the lab; troubleshooting.

Sue Gates
Dairy Farmers of America
May 9, 2019
Objectives

- Understand the complexities of specification agreements
- Understand the variability of methodology and testing procedures
- Guidance when misunderstandings and breakdowns occur
Background Information

• Standard Test Methods
• Product Standards
• Testing Laboratories

Standard Test Methods

• ADPI – American Dairy Products Institute
• AOAC - Association of Analytical Communities
• FDA BAM - Food and Drug Administration Bacteriological Analytical Manual
• IDF – International Dairy Federation
• ISO - International Organization of Standardization
• SMEDP - Standard Methods for the Examination of Dairy Products
Common Standards

- ADPI Standards
  - Composition
  - Typical analysis
- CODEX Standards
  - Composition
  - Typical analysis
  - Contaminant levels
- Individual Country Requirements
  - Chemical/Physical/Microbiological Properties
  - Contaminant levels

Testing Laboratories

- Central Laboratories
  - Pathogen testing
  - Specialized equipment
- Local Laboratories
  - Trained technicians
  - Split sample testing
- Commercial Certified 3rd Party Laboratories
  - Professional staff
  - Accredited by ISO or other recognized organization
Procurement

Procurement Interests:
- Source a quality product
- Negotiate acceptable terms
- Maintain adequate inventory

Vendor Specification

RAW MATERIAL SPECIFICATION

MILK POWDER

Description: Milk powder of highest quality to be used to produce a spoonable cultured milk product.

Chemical Properties

<table>
<thead>
<tr>
<th></th>
<th>LCL</th>
<th>UCL</th>
<th>LOM</th>
<th>Test Method</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>Milkfat</td>
<td>--</td>
<td>1.25%</td>
<td></td>
<td>AOAC</td>
<td></td>
</tr>
<tr>
<td>Moisture</td>
<td>--</td>
<td>5%</td>
<td></td>
<td>AOAC</td>
<td></td>
</tr>
<tr>
<td>Protein</td>
<td>--</td>
<td>30%</td>
<td></td>
<td>AOAC</td>
<td></td>
</tr>
<tr>
<td>Total Fat</td>
<td>--</td>
<td>0.15%</td>
<td></td>
<td>ADPI</td>
<td></td>
</tr>
<tr>
<td>Scorched Particles</td>
<td>--</td>
<td>7.5 mg</td>
<td></td>
<td>ADPI</td>
<td>Per 100g</td>
</tr>
<tr>
<td>Solubility Index</td>
<td>--</td>
<td>1 ml</td>
<td></td>
<td>ADPI</td>
<td></td>
</tr>
</tbody>
</table>

Microbiological Properties

<table>
<thead>
<tr>
<th></th>
<th>LCL</th>
<th>UOM</th>
<th>Test Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aerobic Plate Count</td>
<td>&lt;10 cfu/g</td>
<td>&lt;10 cfu/g</td>
<td>ADAC</td>
</tr>
<tr>
<td>Enterobacteriaceae</td>
<td>&lt;100 cfu/g</td>
<td>&lt;100 cfu/g</td>
<td>ADAC</td>
</tr>
<tr>
<td>Salmonella</td>
<td>70 cfu/g</td>
<td>70 cfu/g</td>
<td>FDA BAM</td>
</tr>
<tr>
<td>Coag Pos Staph aureus</td>
<td>&lt;10 cfu/g</td>
<td>&lt;10 cfu/g</td>
<td>FDA BAM</td>
</tr>
</tbody>
</table>

Packaging: 25kg kraft bag
Palletizing: 40 bags per pallet

2019 ADPI Dairy 360
Is Everything Represented?

- Attributes that might be important
  - Color, flavor, bulk density, flowability
- Certificates of Analysis
  - What attributes should be included?
  - What about “extra” attributes?
- How are the results expressed?
  - Dry basis?
  - Solids nonfat basis?
  - “As is” basis?

Is Everything Represented?

- Certifications
  - Religious
  - Regulatory
  - Market driven
- Contaminant Levels
  - Country requirements
  - CODEX requirements
  - Targeted consumer group
Sales

Sales Manager Interests:
- Negotiate acceptable terms
  - Specification
  - Volume
  - Delivery schedule
  - Pricing

Speciation Review

RAW MATERIAL SPECIFICATION 9999900
MILK POWDER

Description: Milk powder of highest quality to be used to produce a spoonable cultured milk product.

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<td>1.25</td>
<td>%</td>
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<tr>
<td>Moisture</td>
<td></td>
<td>5</td>
<td>%</td>
<td>AOAC</td>
<td></td>
</tr>
<tr>
<td>Protein</td>
<td>32</td>
<td>38</td>
<td>%</td>
<td>AOAC</td>
<td></td>
</tr>
<tr>
<td>Titratiable Acidity</td>
<td>0.15</td>
<td>%</td>
<td>ADPI</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Scorched Particles</td>
<td>7.5</td>
<td>mg</td>
<td>ADPI</td>
<td></td>
<td>Per 100g</td>
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<tbody>
<tr>
<td>Aerobic Plate Count</td>
<td>&lt;10,000</td>
<td>chu/g</td>
<td>AOAC</td>
</tr>
<tr>
<td>Yeast &amp; Mold</td>
<td>&lt;100</td>
<td>chu/g</td>
<td>AOAC per</td>
</tr>
<tr>
<td>Salmonella</td>
<td>Negative</td>
<td>750g</td>
<td>FDA BAM</td>
</tr>
<tr>
<td>Coag Pen Staph aureus</td>
<td>&lt;10</td>
<td>chu/g</td>
<td>FDA BAM</td>
</tr>
</tbody>
</table>

Packaging: 25kg kraft bag
Palletizing: 40 bags per pallet
Manufacturing

Producing Plant Interests:
- Appropriate food safety systems
- Supply product as agreed upon
  - Made to specification
  - Delivered on time

Producing Plant

- Secure Raw Materials
- Produce and Package Product
- Sample and Test Product
- Create Certificates of Analysis
  - Are all attributes listed?
  - Were tests conducted per specified methods?
  - Do all attributes meet requirements?
End User

End User Interests:
- Product received
  - Made to Specification
  - Delivered On Time
  - Functions in Process
  - Functions in Product

End User

- Product meets the Specification
- Do attributes need to be confirmed before receipt into plant?
- Is the Certificate of Analysis acceptable for receipt into plant?
- Product functions as expected

- What if the product does not meet expectations?
Reporting Problem

- Report problem as quickly as possible
- Define failure as specifically as possible
- Provide pictures, if possible
- Save unsatisfactory product, including packaging
- Work with Supplier
  - Determine if product will be salvaged, returned or destroyed
  - Be prepared to answer questions

Troubleshooting

Methodology Investigation
ADPI Scorched Particle Method

Basic Steps
1. Weigh out specific amount of milk powder
2. Mix milk powder into water
3. Filter milk solution through filter pad
4. Grade pad and report result

Where can this go wrong?

ADPI Scorched Particle Method

Basic Steps
1. Weigh out specific amount of milk powder
   1. Alternate sample weights may be used
      1. 50g, 64g, 100g, 250g
2. Mix milk powder into water
3. Filter milk solution through filter pad
4. Grade pad and report result
ADPI Scorched Particle Method

Basic Steps
1. Weigh out specific amount of milk powder
2. Mix milk powder into water
   1. Blenders may vary
   2. Water quality and temperature may vary
3. Filter milk solution through filter pad
4. Grade pad and report result
ADPI Scorched Particle Method

Basic Steps
1. Weigh out specific amount of milk powder
2. Mix milk powder into water
3. Filter milk solution through filter pad
   1. Vacuum source may vary
   2. Filter pad type and size may vary
4. Grade pad and report result
ADPI Scorched Particle Method

Basic Steps
1. Weigh out specific amount of milk powder
2. Mix milk powder into water
3. Filter milk solution through filter pad
4. Grade pad and report result
   1. Subjective reading and reporting
   2. Scorch or insoluble particles?
   3. Is color a concern?
Troubleshooting Summary

- Pictures, Pictures, Pictures
  - What are the lot numbers, manufacture date, package number?
  - What is the customer seeing?
- Identify and agree on a 3rd party laboratory before you need them
- Returning samples to the USA is difficult
- Can the sales manager be on site?
- Should a technical services rep visit?

Other Concerns
Microbiology methods vary

• What does “negative” mean in microbiology?
  • Negative per what?
• Is the results “absent” or <10?
  • Limit of detection for method
• What is the sample size?
• What is the dilution factor?
• What is the batch size?

Customer Specific Methods

• Specific to a product function or process
  • Heat Stability
  • Spore methods
    • Mesophilic
    • Thermophilic
    • USDEC/NIZO study recently published

Relation between spores in milk powders and predictability of spoilage of UHT milk products

https://www.dairyreporter.com/arti...
Contaminants

- Heavy Metals
  - Limit of detection
  - Unit of measure
  - Basis for limit – fluid milk or concentrated?
- Environmental Contaminants
  - Method for detection
  - Laboratory to perform testing
- Other
  - Use government monitoring results

Summary

- Specification agreements can be complex
- There is variability in methodology and testing procedures
- Good communication is important to resolve misunderstandings and breakdowns
QUESTIONS and DISCUSSION

FULL RANGE OF DAIRY OFFERINGS
DFA offers fluid milk, ingredients, consumer packaged goods and everything in-between

DAIRY MARKET EXPERTISE & INSIGHTS
• Risk management programs
• Regular updates on market dynamics

VERTICAL INTEGRATION
• From farm to consumer
• Reliable and consistent high-quality milk supply
• Sustainable and traceable supply chain
• Long-term view of partnerships
• Wide range of available milk attribute options

INNOVATION & INGENUITY
• New solutions grounded in customer insights
• Full capabilities in product customization, applications, R&D and innovation

QUALITY
Uncompromising commitment to quality, consistency, reliability and food safety