Best Practice Workgroup Update

Phosphorus

2014 Annual AAFCO Laboratory Methods & Services Committee
Mission:
- Create documents to assess methods used & to recommend methods appropriate for feed matrices
- Based on scientific justification
- Survey sent compile methods used for matrices
- Based on digestion & quantitation methods
- Matrix & phosphorus level dependent
- List of AAFCO method codes provided with survey
Survey Results

- 20 laboratories responded
- 10 government
  - 9 state & 1 federal
- 9 industry & private
- 1 anonymous (submitted via AAFCO)
Digestion Methods

* 10 Dry ashing without matrix modifier
  * 7 Industry & 3 Government
* 1 Dry ashing with matrix modifier
  * Industry
* 15 Acid digestion
  * 8 Industry & 7 Government
* 9 Microwave digestion
  * 4 Industry & 5 Government
Method References Cited

* 5 AAFCO 031.01/AOAC 965.17
  * Feed, dry ash, molybdovanadate colorimetric

* 1 AOAC 978.01
  * P in fertilizer, direct citrate extraction, autoanalyzer

* 4 AOAC 962.02
  * P > 10%, fertilizer, gravimetric quimociac

* 5 AAFCO 031.44/AOAC 985.01
  * Plants, dry ash, ICP
Method References Cited

* 2 AOAC 984.27
  * Infant formula, HClO₄ digestion, ICP
  * Modified mix of AOAC 984.27 & 985.01 (P>10%)
* 4 AAFCO 031.42
  * ICP, open vessel
* 4 AAFCO 031.43
  * ICP, microwave
* 2 AOAC 2006.03
  * Fertilizer, microwave, ICP
**Acid Digestion**

- 10 Hot plate
- 3 Block Digestor
- 1 Thomas Cain (DEENA) automated system for metal analysis

**Microwave Digestion**

**Reagents**

- 1 HNO$_3$
- 3 HNO$_3$ & HCl
- 1 HNO$_3$:HCl (8:2 v:v ratio)
- 1 HNO$_3$:HCl (3:1)
- 2 HNO$_3$, HCl, H$_2$O$_2$ (30%)
- 1 Mix of 800 mL H$_2$O, 200 mL HCl & 600 mL HNO$_3$
Digestion Summary

* 55.6% Dry Ash
* 27.8% Acid Digestion
* 16.7% Microwave Digestion

Complete Feeds (Organic based)

* 25 Lab Response
* 40% Dry Ash
* 58% Acid Digestion
* 32% Microwave Digestion
<table>
<thead>
<tr>
<th>Mineral Mix</th>
<th>Pet Foods</th>
</tr>
</thead>
<tbody>
<tr>
<td>20 Lab Response</td>
<td>8 Lab Response</td>
</tr>
<tr>
<td>5% Dry Ash</td>
<td>37.5% Dry Ash</td>
</tr>
<tr>
<td>65% Acid Digestion</td>
<td>25% Acid Digestion</td>
</tr>
<tr>
<td>30% Microwave Digestion</td>
<td>37.5% Microwave Digestion</td>
</tr>
</tbody>
</table>
Molasses Products

- 4 Lab Response
- 25% Dry Ash
- 25% Acid Digestion
- 50% Microwave Digestion
Quantitation Methods

* 28 Lab Response
* 34% Colorimetric Method
  * 5 Industry & 4 Government
* 17% Gravimetric Method
  * 3 Industry & 2 Government
* 48% ICP-OES
  * 6 Industry & 8 Government
* No labs reported using ICP-MS
<table>
<thead>
<tr>
<th>Colorimetric</th>
<th>Gravimetric</th>
</tr>
</thead>
<tbody>
<tr>
<td>* 6 Molybdovanadate Reagent</td>
<td>* All (5) use quimoiciaci method</td>
</tr>
<tr>
<td>* 2 Molybdate Reagent</td>
<td>* 1 Stated only if P&gt;10%</td>
</tr>
<tr>
<td>* 4 Spectrophotometer</td>
<td></td>
</tr>
<tr>
<td>* 4 Flow analyzer</td>
<td></td>
</tr>
<tr>
<td>* 1 Uses flow analyzer &amp;</td>
<td></td>
</tr>
<tr>
<td>spectrophotometer</td>
<td></td>
</tr>
</tbody>
</table>
**Distribution of Samples According to \( \% \text{ P} \)**

<table>
<thead>
<tr>
<th>P Level, ( % )</th>
<th>0.5</th>
<th>0.5 - 1</th>
<th>1 - 5</th>
<th>5 - 10</th>
<th>&gt;10</th>
</tr>
</thead>
<tbody>
<tr>
<td>Avg of All Labs</td>
<td>26.1%</td>
<td>50.5%</td>
<td>24.4%</td>
<td>3.8%</td>
<td>6.6%</td>
</tr>
</tbody>
</table>
Difficulties Encountered &/or Comments by Surveyed Labs

- Variability
- Liquid Feed w/High Sugar
  - Foaming during dry ashing, cloudy solution
- Colorimetric: None
- ICP: Spectral Line Overlap
  - High Cu may cause interference
  - High Ni can interfere w/Gallium internal standard
  - Wavelength works is 213.619
  - 171 & 178 drift higher over time
**Difficulties Encountered &/or Comments by Surveyed Labs**

- Certain Soy samples don’t ash well
  - Mg acetate added during ashing to help
- Repeatability with high level $\text{P}_2\text{O}_5$ by ICP
- Sample homogeneity
- Technician error, eg. Dilution
- Problems with high levels P
  - No elaboration given
- No problems with low levels
  - LN: Unsure if it is implied difficulties with high levels
- Very high levels must be diluted quite a bit
  - Adds a step & possible point of error
  - Lab uses flow analyzer
- Very few problems with ICP & included lengthy paragraph on their QC which is good
- 6 Labs
  - None or did not answer
Conclusions

- High level P samples seem to be more problematic
- Wide variety of methods or combination of methods in use
- About half of labs responding employ dry ashing, mainly on feed materials
- Mineral mixes generally undergo acid digestion
- 48% quantitate P via ICP
- 34% utilize a colorimetric method
- 17% use a gravimetric method
Questions or Comment?