

Best Practice Workgroup Update

Phosphorus

2014 Annual AAFCO Laboratory Methods &
Services Committee

Phosphorus

- * 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, HClO_4 digestion, ICP
- * 2 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

Microwave Digestion

* Reagents

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

- * 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_2O_2 (30%)
- * 1 Mix of 800 mL H_2O , 200 mL HCl & 600 mL HNO_3

Digestion Summary

Complete Feeds (Organic based)

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

- * 25 Lab Response
- * 40% Dry Ash
- * 58% Acid Digestion
- * 32% Microwave Digestion

Mineral Mix

Pet Foods

- * 20 Lab Response
- * 5% Dry Ash
- * 65% Acid Digestion
- * 30% Microwave Digestion

- * 8 Lab Response
- * 37.5% Dry Ash
- * 25% Acid Digestion
- * 37.5% Microwave Digestion

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

Colorimetric

Gravimetric

- * 6 Molybdovanadate Reagent

- * 2 Molybdate Reagent

- * 4 Spectrophotometer

- * 4 Flow analyzer

- * 1 Uses flow analyzer & spectrophotometer

- * All (5) use quimoiciac method

- * 1 Stated only if $P > 10\%$

Distribution of Samples According to % P

P Level, %	0.5	0.5 - 1	1 - 5	5 - 10	>10
Avg of All Labs	26.1%	50.5%	24.4%	3.8%	6.6%

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 Continued

- * Certain Soy samples don't ash well
 - * Mg acetate added during ashing to help
- * Repeatability with high level P_2O_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

Difficulties Encountered &/or Comments by Surveyed Labs Continued

- * 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?