LMSC---Best Practice Workgroup

Phosphorus

2016 Mid-Year AAFCO Laboratory Methods & Services Committee

History/Timeline

- Tackled & completed “Fat”
- Next Topic “Phosphorus”
- Mission:
  - Create documents to assess methods used & to recommend methods appropriate for feed matrices
  - Science based recommendations
- Survey
  - Compiled based on methodology
  - Matrix & phosphorus level dependent
  - AAFCO codes included for ease of use

Survey Results

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

Survey Results, Cont'd

- Mineral mixes generally undergo acid digestion
- 48% quantitate P via ICP
- 34% utilize a colorimetric method
- 17% use a gravimetric method

Comments about Challenges

- 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

Comments about Challenges, Cont'd

- 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

Survey Conclusions

- High level P samples seem to be more problematic
- Wide variety of methods or combination of methods in use
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Next Steps

- Phosphorus Matrix Developed

Method Code	31.01	31.02	31.03	31.04	31.06	31.07	31.08	31.41	31.42	31.43	31.44	31.51	31.52	31.53	31.99												
Method Reference	AOAC 965.17	AOAC 962.02		AOAC 953.01							AOAC 985.01					ISO 3646:1982	ISO 6491:1998	ISO 10540-	ISO 10540-	ISO 10540-	ISO 27085:2009	ISO 2962:2010	AACC 40-56.01	AACC 40-57.01	AACC 40-58.01	
Method Description	Photometric Pc16%	GGMP (AOAC 935.13 - Extraction)	Autoanalyzer	Emission Spectroscopy	Hach Method	DCP	PreWet	ICP, Dry Ash	ICP, Open Vessel	ICP, Microwave	ICP-Dry Ash	ICP-MS, Dry Ash	ICP-MS, Open Vessel	ICP-MS, Microwave	Miscellaneous	Spectrophotometric (starches & derived products)	Spectrometric (animal feeding stuffs)	Colormetric (animal & vegetable oils)	Graphite Furnace AA (animal & vegetable oils)	ICP-OES (animal & vegetable oils)	ICP-AES (animal feeding stuffs)	Molecular Absorption Spectromeric (cheese & cheese	Colometric (yeast)	Grsvimetric (feed & feed stuffs)	Volumetric (feed & feedstuffs)	
Matrix																											
Cottonseed																											
Distillers Grain, Dry																											
Distillers Grain, Wet																											
Feed, Dry																											
Feed, Premix																											
Super Phosphate																											
Triple Super Phosphate																											
Calcium metaphosphate																											
Potassium dihydrogen phosphate																											
Laying ration																											
Medicated laying ration																											
Pig finisher																											
Protein mineral concentrate																											
Vitamin mineral supplement																											
Monoammonium phosphate																											
Diammonium phosphate																											
Mixed ruminant feed																											
Livestock Mineral																											
Mineral Mix																											
Nitric phosphate																											
Sodium metaphosphate																											
Alfafa leaves																											
Citrus leaves																											
Orchard leaves																											
Pine needles																											
Tomato leaves																											
Corn leaves																											
Corn stalk																											
Peach leaves																											
Burmuds grass																											

Next Steps

- Phosphorus Matrix Developed
  - AAFCO Codes Used in CSP
  - Matrices & P levels found from original JAOC papers

A	AD	AE	AF	AG	AH	AI	AJ	AK	AL	AM	AN	AO	AP	AQ	AR	AS	AT	AU	AV	AW	AX	AY	AZ	BA	BB	BC	BD	BE	BF	BG	BH	BI
Method Code	Cottonseed	Distillers Grain, Dry	Distillers Grain, Wet	Feed, Dry	Feed, Premix	Super Phosphate	Triple Super Phosphate	Calcium Metaphosphate	Potassium Dihydrogen Phosphate	Laying Ration	Medicated Laying Ration	Pig Finisher	Protein Mineral Concentrate	Vitamin Mineral Concentrate	Monocalcium Phosphate	Diammonium Phosphate	Mixed Ruminant Feed	Livestock Mineral	Mineral Mix	Nitric Phosphate	Sodium Metaphosphate	Alfalfa Leaves	Citrus Leaves	Orchard Leaves	Pine Needles	Tomato Leaves	Corn Leaves	Corn Stalk	Peach Leaves	Burmuda Grass		
31.00 - AOAC 964.06	■					■	■	■	■						■	■																
31.01 - AOAC 965.17		■	■	■	■					■	■	■	■	■			■	■	■													
31.02 - AOAC 962.02	■					■	■	■	■						■	■				■	■											
31.03																																
31.04 - AOAC 953.01																						■	■	■	■	■						
31.06																																
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ISO 10540-2:2003																																
ISO 27085:2009																																
ISO 2962:2010																																
ISO 3946:1982																																
AACC 40-56.01																																
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JOAC (50) 937, 1975.

- William Hoover (TAMU) 50, 937, (1976)
- Technical Communication as Referee for minerals in feed
- Dry Ash
  - Low recovery of P in mineral mix feeds
- Wet Ash
- May form refractory compounds when ashed at 550°C, insoluble in following acid digestion portion of method
- Not recognized in collaborative study, sample type not included
- Recommend added statement in all dry ash method “not appropriate for mineral mix feeds”
- Statement appears in AOAC 968.08 (D)(a)

Conclusions Thus Far

- Survey based on AAFCO CSP method codes
  - High level P most challenging
- JAOAC 59, 937 (1976)
  - Dry Ash methods not appropriate method for mineral mix feed
    - Low recovery for Ca, Cu, Fe, Mn, P, Zn up to 50%
    - Mg least effected by ashing process---only up to 3%
- Matrix by Method and Scope
  - Updated
- Next Steps
  - Help with matrices for ISO methods
  - Circulate updated matrix by methods to WG
  - Write up white paper

Updates

- White Paper
- Matrix vs phosphorus
- Documents reviewed in Denver August 2015
  - No comments received
- Email sent & Documents on Foodshield
  - No comments received

- Workgroup moves that consensus has been achieved, ready for LMSC to vote to accept

Conclusions

- Best Practice Workgroup (BPW) has successfully achieved mission
- BPW moves via consensus that LMSC vote to accept documentation or reject
- BPW moves to move forward with next task
  - Moisture
  - Fiber
  - Other?