



AGRICULTURE • ENVIRONMENTAL • ANIMAL FEED • FUEL
PET FOOD • FOOD & BEVERAGE • HOME/SMALL BUSINESS

AAFCO Meeting August, 2017

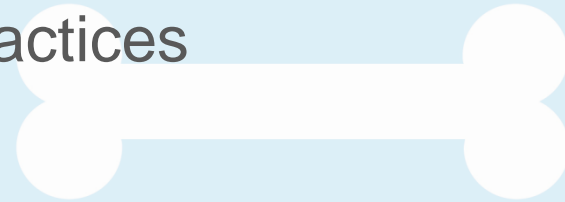
Moisture Best Practices

By

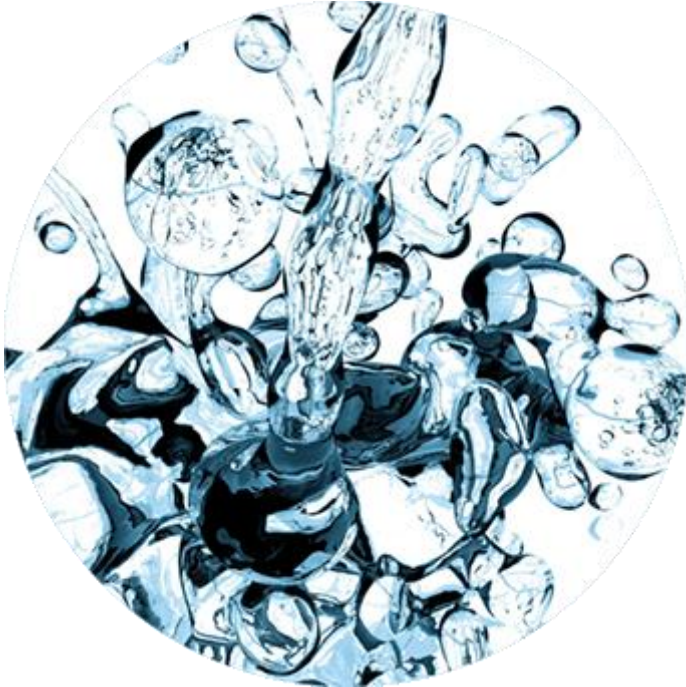
Dr. Jane Caldwell

Assistant Technical Director

Midwest Laboratories



MOISTURE DETERMINATION



Should be an “easy” analysis and everybody should get the same result

- Get sample and grind
- Weigh sample
- Dry sample
- Re-weigh residue
- Difference is “moisture”

EMPIRICAL PROCEDURE



If laboratory uses:

- Same amount of sample
- Same temperature
- Same drying time

Moisture results should be the same

FUNDAMENTAL “COMPLICATION”



Anything that evaporates or is caramelized at less than the temperature of the oven will be reported as “moisture”

Examples:

- Sugars
- Urea
- Volatile fatty acids

AAFCO PROFICIENCY PROGRAM



- Feeds – Monthly
- Pet foods
- Minerals/Premixes

AAFCO METHODS TO BE “REMOVED”

- AOAC 925.45 - Vacuum on sand
- AOAC 925.45 - Vacuum on pumice stone
- AOAC 925.04 - Moisture by Toluene Distillation

These are rarely used

MOISTURE CATEGORIES

Loss on drying – high temperature

- AOAC - 930.15 – 2 hrs. @ 135 degrees
- AOCS - Ba2a-38 - 2 hours @ 130 degrees
- AOAC – 925.10 - 1 hour @ 130 degrees (flour)

Loss on drying - low temperature

- AOAC - 934.01 - 5 hours @ 95 degrees, vacuum
- AOAC - 935.29 - 3 hour @ 104 degrees (malt)

Moisture

- AOAC - 2001.12 - KF

Moisture method spreadsheet

- 97 Identified Matrices
- 36 identified methods
- Methods have one or more materials that can be analyzed by the method (identified in scope)

AAFCO PROGRAM CHOICES

001.00	Loss on Drying	Vac 95°C 5 hr	%	AOAC: 934.01, AOCS: Ba2b-82
001.02	Loss on Drying	Vac on sand	%	AOAC: 925.45
001.03	Loss on Drying	Low temp. methods	%	ISO: 6496
001.05	Loss on Drying	LECO	%	
001.06	Loss on Drying	Vac on pumice stone	%	AOAC: 925.45
001.07	Loss on Drying	104°C 3 hr, in malt	%	AOAC: 935.29
001.08	Loss on Drying	102°C 16 hr, in meat	%	AOAC: 950.46
001.99	Loss on Drying	Miscellaneous	%	
011.01	Loss on Drying	135°C 2hr	%	AOAC: 930.15
011.02	Loss on Drying	130°C for 2 hours	%	AOCS: Ba2a-38
011.03	Loss on Drying	130°C, 1 hour, Flour	%	AOAC: 925.10
011.99	Loss on Drying	High Temp. Methods Miscellaneous	%	
010.03	Moisture	Karl-Fischer	%	AOAC: 2001.12
010.04	Moisture	Toluene Dist	%	AOAC: 925.04
010.11	Moisture	NIR	%	
010.99	Moisture	Miscellaneous	%	

MOISTURE CATEGORIES

High temperature

011.01	Loss on Drying	135°C 2hr	%	AOAC: 930.15
011.02	Loss on Drying	130°C for 2 hours	%	AOCS: Ba2a-38
011.03	Loss on Drying	130°C, 1 hour, Flour	%	AOAC: 925.10
011.99	Loss on Drying	High Temp. Methods Miscellaneous	%	

MOISTURE CATEGORIES

Low temperature

001.00	Loss on Drying	Vac 95°C 5 hr	%	AOAC: 934.01, AOCS: Ba2b-82
001.02	Loss on Drying	Vac on sand	%	AOAC: 925.45
001.03	Loss on Drying	Low temp. methods	%	ISO: 6496
001.05	Loss on Drying	LECO	%	
001.06	Loss on Drying	Vac on pumice stone	%	AOAC: 925.45
001.07	Loss on Drying	104°C 3 hr, in malt	%	AOAC: 935.29
001.08	Loss on Drying	102°C 16 hr, in meat	%	AOAC: 950.46
001.99	Loss on Drying	Miscellaneous	%	

MOISTURE CATEGORIES

Moisture

010.03	Moisture	Karl-Fischer	%	AOAC: 2001.12
010.04	Moisture	Toluene Dist	%	AOAC: 925.04
010.11	Moisture	NIR	%	
010.99	Moisture	Miscellaneous	%	

VARIATIONS

NIR – based on what of the other methods
(calibration curve)

Vacuum with different temperatures

- 70 degrees
- 100 degrees

EXAMPLE REPORT – 201723 – MILK REPLACER

						001 - LOD	010 moist	011-LOD
001 - LOD			88	3.9054		52.1	11.8	36.1
001.07	104 C 3 hr. in malt	AOAC 935.29	46	4.0218				
001.99	Low temp Misc.		20	3.8138				
010 - moisture			20	4.2896				
010.99	Misc.		16	4.4984				
011 - LOD			61	8.0582				
011.01	135 C 2 hrs.	AOAC 930.15	55	8.2736				

AAFCO SUMMARY

Comparisons:

	001 to 010	001 to 011	010 to 011
Average	101.9	121.0	118.6
Minimum	87.7	100.5	100.3
Maximum	150.7	222.0	226.0

AAFCO SUMMARY

Moisture methods referenced by category

	001 – LOD L	010 – Moisture	011 – LOD - H
Average reported	38.8	12.8	48.4
Minimum	11.6	3.4	36.1
Maximum	52.1	31.8	84.8

RECOMMENDATIONS

1. Reduce the number of reporting options for AAFCO PT samples
2. Reduce the incidences/use of “MISC” in reporting
3. Remove the high temperature LOD methods (e.g. 930.15)
4. Laboratories must have knowledge of sample matrix and use appropriate method

CONSIDERATIONS

1. How to handle “complex” feeds with multiple components – which method to use?
2. How to work with clients/industries that have historical levels and formulations based on high temperature LOD
3. How to handle/address established NIR calibrations (many based on AOAC 930.15)

METHOD VALIDATIONS - ORIGINAL

AOAC 930.15

- Used vacuum oven as the “gold standard”
- Validated with oilcake meal, fish meal, meat meal, bran cereal, shorts
- No information of previous studies nor why these materials were selected
- Current scope limitations
 - Urea containing feeds
 - High sugar contents
 - Ensiled materials
 - Milk products with > 4% sugar

METHOD VALIDATIONS - ORIGINAL

AOAC 934.01

- No mention of materials used to validate the method
- Used as reference method for AOAC 930.15

METHOD VALIDATION - ORIGINAL

AOAC 991.01 (NIR)

- Used AOAC 930.15 to create calibration equations
- Validated with alfalfa hay, grass-legume hay, Bermuda grass, barley straw, timothy/clover, sorghum silage, corn stover, orchard grass, grass hay
- Applicable since no urea or sugars

METHOD VALIDATION - ORIGINAL

AOAC 2001.12 (KF)

- Validated on soybeans, urea feed, grass hay, cereal grains, alfalfa silage, corn silage, cat food, milk replacer, alfalfa hay, oats

CURRENT VALIDATION SCOPE – the dilemma

What becomes the “gold standard” on which all method validations are based

Scope of the validation is extensive

- 97 identified matrices
- 26 identified methods
- 2522 combinations and requiring multiple samples (10 – 20), project could entail 25,000 to 50,000 samples

A rack of laboratory vials is shown on the left side of the image. The vials are arranged in a grid. The top row has three vials with brown caps. The middle row has three vials, some containing liquid. The bottom row has three vials, some containing liquid. The vials in the middle row have handwritten labels 'A1' and 'A2'. The background is a blurred laboratory setting. A white diagonal line separates the image from a solid blue background on the right.

Questions



Thank You

Dr. Jane Caldwell

402-829-9858

jcaldwell@midwestlabs.com