MOISTURE
Best Practices
Survey Results
AAFCO
August 11, 2017

Surveys were distributed via

Aglabs

AAFCO LMSC list

NFTA

Individual contacts

13 regulatory labs
7 research labs
16 private labs
36 total responses

International participation:
Tanbaya Co Japan
Down to Earth Labs Alberta
Shur Gain Quebec
Shanghai Institute of Dairy Science

please indicate how you determine moisture on the following matrices

Matrix *Temp* *Drying Time* *Oven Type* *Method Reference*

dry feeds
grains
oilseeds, unground
oilseeds, ground
pet food, dry
pet food, canned
pet food, semi-moist
distillers grains, dry
distillers grains, wet
forages, dry
forages, wet
liquid molasses feeds
molasses block
milk replacer
suet, fats, oils
other

Red font – Official method or suggested method
Blue font – regulatory lab responses
Green font – research lab responses
Purple font – private lab responses

*labs who are following official or suggested method

DRY FEEDS

AOAC 930.15 135C 2 hr or AOAC 934.01 95-100C vac 5 hr
Should be AOAC 935.29 (NFTA 2.2.2.5) 104C 3 hr

*4 labs 135C MC 2 hr
*6 labs 103-105C MC 3 hr
*2 labs 95-100C vac 5 hr

*3 labs 100-105C MC 3-4 hr
*1 lab 100C vac time not stated
1 lab 105C 16 hr

*7 labs 135C MC 2 hr
*3 labs 105C MC 3 hr
1 lab 105C MC 6 hr
1 lab 105C MC 16 hr
1 lab 80C vac 20 hr
1 lab Infrared analyzer

OILSEEDS - GROUND

AOCS Ba 2a-3B 130C MC 2 hr

*1 lab 130C MC 2 hr
1 lab 135C MC 2 hr
4 labs 104-105C MC 3-4 hr
1 lab 95-100C vac 5 hr

2 labs 105C MC 3 hr
1 lab 105C MC 16 hr
1 lab 100C vac time not stated

*2 labs 130C MC 2 hr
4 labs 135C MC 2 hr
2 labs 100 - 105C MC 3 hr
1 lab 105C MC 6 hr

PET FOOD – CANNED

none specified should be? AOAC 950.46 (meat) 102 or 125C MC 18 or 4 hr

*1 lab 125C MC 4 hr
*1 lab 101C MC 16-18 hr
4 labs 103-105C MC 3-4 hr
2 labs 135C MC 2 hr
1 lab 100C MC 24-48 hr
1 lab 85C MC 16 hr
1 lab 95-100C vac 5 hr
1 lab Karl Fischer

1 lab 100C vac time not stated

*2 labs 100-104C MC 16-18 hr
*1 lab 125C MC 4 hr
2 labs 105C MC 3 or 6 hr
1 lab 135C MC 2 hr
2 labs 65 or 60C MC 5 or 16 hr
1 lab 145C halogen moisture analyzer

DISTILLERS GRAINS - DRY

AOAC 935.29 104C MC 3 hr

*6 labs 103-104C MC 3 hr
1 lab 105C MC 4 hr
1 lab 105C MC 2 hr
1 lab 95-100C vac 5 hr
1 lab Karl Fischer AOAC 2001.12

*2 lab 105C GC 3 hr
1 lab 105C MC 16 hr
1 lab 2-step method 55C & 105C

*8 labs 105C MC 3 hr
1 lab 105C MC 6 hr
1 lab 105C MC 16 hr
2 labs 135C MC 2 hr

LIQUID MOLASSES FEEDS

AOAC 969.35 60C vac 18 hr or AOAC 966.20 Karl Fischer

*1 lab 60C vac 18 hr
1 lab 70C vac 5 hr
1 lab 100C vac 16 hr
1 lab 100C MC 24-48 hr
1 lab Karl Fischer AOAC 2001.12

1 lab 105C FD >12 hr

*2 labs 60-65 C vac 16-18 hr
1 lab 80C vac 16 hr
3 labs 65-70C vac 4-5 hr
2 labs 105C FD 3 hr
1 lab 105C MC 6 hr
1 lab 105C FD >16 hr

MILK REPLACERS

AOAC 927.05 100C vac 5 hr

*1 lab 100C vac 5 hr
1 lab 95C vac 5 hr
3 labs 103-105C MC 3 hr
1 lab 105±5C FD 4 hr±5 min
1 lab 100C MC 24-48 hr
1 lab 135C MC 2 hr

*2 labs 100C vac 5 hr
1 lab 70C vac 4 hr
3 labs 105C MC 3 hr
3 labs 100-105C FD 5-6 hr

SUET, FAT, OILS

AOCS Ca 2d-2S 20-25C vac 1 hr or AOCS Ca 2e-84 Karl Fischer

1 lab 50-55C vac 1 hr
3 labs 103-105C MC 3 hr

1 lab 80C vac 5 hr
1 lab 70C vac 4 hr
1 lab 65C MC? 5 hr
2 labs 105C MC 3 hr
1 lab 105C MC 6 hr
1 lab 130C FD 1 hr
1 lab Karl Fischer ASTME203-01

Use the 2-step method to determine total moisture on forages, wet feeds & grains

4 regulatory labs **5 research labs** **4 private labs**

1 private lab does a 1-step method by determining "as received" moisture by drying at 105C for 6 hr and assumes that lab moisture is not significant.

PARTIAL MOISTURE

NFTA 2.2.1.1 55C FD overnight or NFTA 2.2.1.2 microwave oven

***3 labs 55C FD overnight or until crisp to touch**
1 lab 97C FD until dry

***3 Labs 55C FD >12 hr or no weight loss**
2 labs 60-65C FD 12-18 hr

***5 labs 50-60C FD overnight or time determined by DM**
1 lab 65C FD time not stated
1 lab 70C MC 8 hr
1 lab 100C FD ~30 min
***1 lab microwave oven**

16 labs use the same method for all or most of their matrices

6 labs 103-105C 3 hr

1 lab 105C 4 hr

1 lab 105C 6 hr

1 lab 105C 16 hr

2 labs 135C 2 hr

2 labs 95-100C vac 5 hr

1 lab 100C vac time not specified

1 lab 55C >12 hr

How much does the oven type impact the reported moisture results under each specific method? Don't know.

For dry feeds, the matrix with the most responses, oven types used were:

Mechanical convection or forced draft – 19 labs

Gravity convection – 4 labs

Vacuum – 4 labs

Air - 1 lab

Most official methods do not specify oven type except vacuum.

TEST PORTION SIZES

Lab Moisture

1 g – 8 labs

1 – 2 g – 38 labs

2 g – 127 labs

3 g – 14 labs

5 g – 34 labs

20 – 30 g -- 22 labs (grains, oilseeds, pet foods, distillers grains, molasses feeds, milk replacer, fats)

100-200 g – 3 labs (grains, WDG)

Partial Moisture

*Entire lab sample – 2 labs

40-70 g – 2 labs

100 – 400 g – 7 labs

500g – several kg – 1 lab

MONITOR OVEN TEMPERATURE

Digital display on oven – 21 labs

Thermometer placed in oven -- 23 labs

Use both above methods – 10 labs

Viewpoint temperature monitoring system – 1 lab

Dataloggers – 1 lab

CALIBRATE THERMOMETER or DIGITAL DISPLAY

Annually – 14 labs Monthly – 4 labs

1 lab each gave 1 of the following responses:

twice a year quarterly weekly every 5 years

secondary thermometer checked each sample run

checked & recorded daily & validated every 4 months

not on a regular basis have not adopted procedure yet

as needed based on dataloggers

None or not answered – 7 labs

PARTICLE SIZE

0.75 mm – 9 labs
0.8 mm – 2 labs
1 mm – 12 labs
2 mm – 4 labs
2-4 mm – 1 lab
4 mm Wiley grind followed by 1 mm Udy grind – 1 lab
as received – 1 lab
unknown but grinder tested periodically to pass LECO combustion test -- 1 lab
unknown – use food processor on pet foods - 2 labs
not sure but pretty fine (use Perten mill) – 1 lab

MILLS USED

Retsch ZM200 – 1 lab
Wiley – 1 lab
Udy – 2 labs
food processor – 1 lab
Perten -- 1 lab

NIR

1 regulatory lab 3 research labs 10 private labs

forages – 8 labs grains – 5 labs feeds – 5 labs distillers grains – 2 labs
pet food – 1 lab soy meal – 1 lab TMR – 1 lab ingredients – 2 labs

purchased calibration equations from NIRS Consortium – 3 labs
purchased calibration equations from another source – 4 labs
in-lab developed calibration equations – 9 labs

Moisture method calibration is based upon
135C 2 hr – 2 labs 104C 3 hr – 4 labs 130C 2 hr (soy) – 2 labs
oven method - 3 labs don't know – 3 labs

Do you monitor the equation based on the same method
yes – 9 labs no – 3 labs

KARL FISCHER

2 regulatory labs	1 research lab	4 private labs
as a routine method	yes – 2 labs	no – 5 labs
only to confirm a violation	yes – 1 lab	no – 5 labs

feeds – 1 lab
distillers grains – 1 lab
canned & semi-moist pet food – 5 labs
molasses feeds – 3 labs
fats & oils – 5 labs
fuels & ethanol – 2 labs

How Often is a QC Material Analyzed

every run – 15 labs

daily – 5 labs

monthly – 2 labs

every 2 weeks – 2 labs

every half year – 1 lab

annually (moisture analyzer calibration) – 2 labs

quarterly or only as part of NFTA proficiency program – 5 labs

whenever it is a canned pet food – 1 lab

N/A – 2 labs

1 lab -- At least 1 sample will be a lab control sample and also at the beginning and end of every batch. At least 1 sample in every 10 will be duplicated within the batch as well.

How Is QC Material Stored

Sealed container – all labs

Vacuum sealed – 1 lab

In desiccator – 3 labs

Protected from light – 5 labs

Cool place or refrigerated – 5 labs

Freezer – 3 labs

Recognize that some change will occur and live with it

Typical Uncertainty Associated with your Moisture Method

don’t know or no answer – 8 labs
have not calculated – 2 labs

animal feed & dry pet food = 3.0% MU (95%CL, k=2); wet pet food = 1.0% MU (95%CL, k=2); forages = 2.0% MU (95% CL, k=2) - 1 regulatory lab

based on QC charts for each matrix - 1 lab

±3 SD – 1 lab ±2 SD – 2 labs

some labs listed percentages ranging from 0.04% - 13%

some labs provided a number within any units

Proficiency Testing Program for Moisture

AAFCO feed – 20 labs

AAFCO pet food – 15 labs

NFTA forage – 15 labs

AOCS Smalley oilseeds – 4 labs

None – 1 lab

AACC cereal grains – 2 labs

USDA meat – 1 lab

Corn Refiners Assn – 1 lab

API (American Proficiency Institute) – 2 labs

LGC for Karl Fischer – 1 lab

Masterlab (The Netherlands) yearly ring test – 1 lab

Average Number of Moisture Analyses per Year

Oven method – range from 10 to 260,000
average of regulatory labs = 370 private labs do a higher volume

Karl Fischer – range from 10 to 10,000

NIR – range from 400 – 200,000

One regulatory lab: Do not test many samples for moisture but always test AAFCO check samples. Moisture may be tested if labeled guarantees are way off to see if this might be related to moisture issues.

Private forage lab: Measure DM 2 ways on the same test sample:
Run NIR on a portion dried at 60C
Analyze separate portion dried at 105C
Compare both DM results. If there is any error, re-analyze DM.

Problems/Concerns Encountered with Moisture Analysis

I have concerns with the 2 hour 135 degree method. We see great variability in this method. I have 20+ years of moisture testing experience and feel that 135 degrees is too hot and 2 hours is too short for a moisture method. When compared to other moisture methods, this method never correlates well. It is also very sensitive to time when weighed back, even when stored in a desiccator. This method seems to be a "catch all" method acceptable across the feed industry which is the ONLY reason we use it. I don't trust the results at all compared to other methods, such as the 5 hour 100 degree vacuum oven method or KF. I know switching to a more accurate and precise method would be tough on the industry, but something should be done around this. Why use it if it isn't accurate?
from a private lab

Problems/Concerns Encountered with Moisture Analysis

Clients not aware of different methods

Different matrices need different time & temps. Labs not aware of matrix.

Believes a lot of forage labs use the 104C 3 hour method for certification and then switch to a different moisture method that works for them (meaning more convenient or faster)

Have a really hard time finding a consistent moisture QC for loss-on-drying

KF method can take a while for instrument & reagents to stabilize; reagents may need to made fresh.

Tend to lose moisture with NFTA legume QC samples. Am replacing QCs on a regular basis.

Problems/Concerns Encountered with Moisture Analysis

Used to have desiccator issues, now have desiccator work instructions.

Spillage from desiccator requiring re-analysis

Wet pet food mold even in freezer

Sample settles in post grinding jars

AAFCO PT omits their moisture results (using wrong AOAC method reference)

Maintaining consistent vacuum pressure over the drying period

Any method that uses “dry to constant weight” to determine endpoint is worthless in a production lab

Problems/Concerns Encountered with Moisture Analysis

Addition of sand to dish and then dried. Sand will support and separate the sample particles as it is drying. Described in molasses method but also useful for other matrices like slurries and process samples from food & feed industry.

We know we lose VFA from forages and count it as moisture loss.

Samples containing glycerol need to be validated for methodology as the glycerol can burn off at 105C and over estimate moisture.

Oven mapping with consistent temperature

Oven overload – moisture levels in oven can be highly variable

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