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

<table>
<thead>
<tr>
<th>Matrix</th>
<th>Temp</th>
<th>Drying Time</th>
<th>Oven Type</th>
<th>Method Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>dry feeds</td>
<td></td>
<td></td>
<td></td>
<td>forages, dry</td>
</tr>
<tr>
<td>grains</td>
<td></td>
<td></td>
<td></td>
<td>forages, wet</td>
</tr>
<tr>
<td>oilseeds, unground</td>
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<td>liquid molasses feeds</td>
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<tr>
<td>oilseeds, ground</td>
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<td>molasses block</td>
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<tr>
<td>pet food, dry</td>
<td></td>
<td></td>
<td></td>
<td>milk replacer</td>
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<tr>
<td>pet food, canned</td>
<td></td>
<td></td>
<td></td>
<td>suet, fats, oils</td>
</tr>
<tr>
<td>pet food, semi-moist</td>
<td></td>
<td></td>
<td></td>
<td>other</td>
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<tr>
<td>distillers grains, dry</td>
<td></td>
<td></td>
<td></td>
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</tr>
<tr>
<td>distillers grains, wet</td>
<td></td>
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</tr>
</tbody>
</table>
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  60°C vac 18 hr  or  AOAC 966.20 Karl Fischer

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

1 lab  105°C FD >12 hr

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

<table>
<thead>
<tr>
<th>Labs</th>
<th>Temperature</th>
<th>Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>6</td>
<td>103-105°C</td>
<td>3 hr</td>
</tr>
<tr>
<td>1</td>
<td>105°C</td>
<td>4 hr</td>
</tr>
<tr>
<td>1</td>
<td>105°C</td>
<td>6 hr</td>
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<tr>
<td>1</td>
<td>105°C</td>
<td>16 hr</td>
</tr>
<tr>
<td>2</td>
<td>135°C</td>
<td>2 hr</td>
</tr>
<tr>
<td>2</td>
<td>95-100°C</td>
<td>5 hr</td>
</tr>
<tr>
<td>1</td>
<td>100°C</td>
<td>time not specified</td>
</tr>
<tr>
<td>1</td>
<td>55°C</td>
<td>&gt;12 hr</td>
</tr>
</tbody>
</table>
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
<table>
<thead>
<tr>
<th>Name</th>
<th>Institution/Role</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lawrence Novotny,</td>
<td>South Dakota - retired from SDSU Olson Biochemistry Labs</td>
</tr>
<tr>
<td>co chair</td>
<td></td>
</tr>
<tr>
<td>Sharon Webb, co-chair</td>
<td>University of Kentucky Regulatory Services</td>
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<tr>
<td>Dan Berg</td>
<td>Covance Food Solutions (Madison WI)</td>
</tr>
<tr>
<td>Kristy Broten</td>
<td>Minnesota Dept of Agriculture - Regulatory Feed Lab</td>
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<tr>
<td>Christina V Childers</td>
<td>Mississippi State Chemical Laboratory</td>
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<tr>
<td>Andy Crawford</td>
<td>Crawford Consulting (statistian)</td>
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<tr>
<td>Claudia Galvan</td>
<td>New Mexico State Chemist Lab</td>
</tr>
<tr>
<td>Teresa M Grant</td>
<td>North Carolina Dept. of Agriculture &amp; Consumer Services - Food, Feed, Fertilizer Supervisor</td>
</tr>
<tr>
<td>Jeff Horst</td>
<td>Agri-King, Laboratory Production Manager</td>
</tr>
<tr>
<td>Jerome King</td>
<td>Midwest Labs - Omaha NE</td>
</tr>
<tr>
<td>Name</td>
<td>Organization</td>
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<tr>
<td>Jason S. Kong</td>
<td>Ohio Dept of Ag lab</td>
</tr>
<tr>
<td>Bozena D. Lusiak</td>
<td>Nestle Purina PetCare (St. Louis MO)</td>
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<tr>
<td>Kathryn Phillips</td>
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<td>Andrew Randall</td>
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<td>Bobby Sanchez</td>
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<tr>
<td>John Szpylka</td>
<td>Mérieux NutriSciences (Chicago),</td>
</tr>
<tr>
<td>Lei Tang</td>
<td>Scientific Affairs Director, Chemistry</td>
</tr>
<tr>
<td>Nancy Thiex</td>
<td>FDA Center for Veterinary Medicine,</td>
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<td></td>
<td>Division of Animal Feeds</td>
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