Recommendations and Critical Factors in Determining Moisture in Animal Feeds

AAFCO's Laboratory Methods & Services Committee
Moisture Best Practices Working Group

January 23, 2018

Terminology

Moisture

Loss on Drying

Dry Matter or Total Solids

Partial Dry Matter or Partial Moisture

Laboratory Dry Matter or Residual Moisture

Total Dry Matter or Total Moisture

DISCUSSION of METHODS

Direct Water Methods

Karl Fischer

Toluene Distillation

DISCUSSION of METHODS
Loss on Drying

AOAC 934.01 VACUUM OVEN

Used by most labs in 1920s & 1930s

Became official in 1934 by establishing temperature to be between 95-100C and time to be 5 hours

DISCUSSION of METHODS

Loss on Drying

AOAC 930.15 135C / 2 hr

Adopted tentatively as an **alternative** method to be used when a vacuum oven was not available.

Matrices evaluated were:

- Oilcake meal
- Fish meal
- Meat meal
- Bran cereal products
- (wheat) shorts

DISCUSSION of METHODS

Loss on Drying

AOAC 930.15 Limitations

1931 – cannot be used on feeds with sugar content due to caramelization

1935 – not to be used when a fat determination is to be made on the same test portion (fat oxidation will occur)

Not applicable to feeds containing urea (decomposes at 132C)

AAFCO PT data – this is the most widely used LOD method

DISCUSSION of METHODS

Loss on Drying

Thiex and Van Erem, SDSU, 1999

10 each partially dried hays, haylages, corn silage

Karl Fischer

135C 2 hr -- over estimated moisture

104C 3 hr -- most closely matched KF

104C 6 hr

DISCUSSION of METHODS

Loss on Drying

Thiex and Van Erem, SDSU, 1999

10 different feeds containing no urea

Karl Fischer

135C 2 hr -- over estimated moisture

104C 3 hr

104C 6 hr

110C 3hr -- most closely matched KF

Vacuum 95C 5 hr

DISCUSSION of METHODS

Loss on Drying

Thiex and Van Erem, SDSU, 1999

6 different feeds containing urea

Karl Fischer

135C 2 hr -- over estimated moisture

104C 3 hr -- most closely matched KF

104C 6 hr

110C 3hr

Vacuum 95C 5 hr

DISCUSSION of METHODS

Loss on Drying

Thiex and Richardson, *J Animal Sci,* 2003, evaluated various moisture studies and concluded:

Drying at 135C should be eliminated or restricted to very few materials for which it has been validated.

Drying at 104 or 105C for 3 hr most closely matched KF moisture values.

DISCUSSION of METHODS

Loss on Drying

National Forage Testing Assn Task Force in 2000

Concluded that drying at 105C for 3 hr provided the most accurate value.

NFTA Official Method 2.1.4

DISCUSSION of METHODS

Loss on Drying

Thiex SDSU 2009 study

Analyzed 30 DDGs using 4 LOD & KF methods

Drying at 105C for 3 hr most closely matched KF

Poorest performing method 135C 2 hr

DISCUSSION of METHODS

Loss on Drying

PET FOOD

No official methods

Published data includes only two pet food samples.

Dry cat food - 110°C/3 hr LOD most closely matched the KF moisture value.

Dry dog food --135°C/2 hr LOD closely matched the KF value.

Obviously more data is needed on pet foods.

DISCUSSION of METHODS

Loss on Drying

Most LOD methods are designed for low moisture (<15%) materials that can be easily comminuted to 1 mm.

The entire high moisture laboratory sample needs to be partially dried at <60C and comminuted, followed by analysis for residual moisture.

DISCUSSION of METHODS

Other Methods

NIR

Infrared moisture analyzer

Halogen moisture analyzer

RECOMMENDED METHODS

Bidwell and Sterling stated in the *JAOAC* in 1925 "*The ideal moisture method, which will probably never be found, would be applicable to all substances, would be rapid, would require little skill, would separate and determine uncombined water and nothing else, and preferably, would determine water directly and not by difference*."

RECOMMENDED METHODS

Karl Fischer: the gold standard

Use to validate all other moisture methods

AOAC 2001.12 – dry animal feeds, pet foods, grains and partially dried forages

AOAC 991.02 -- soft-moist & semi-moist pet foods

AOAC 966.20 -- liquid molasses

AOAC 984.20 -- oils and fats

RECOMMENDED METHODS

Loss on Drying

The working group is recommending the discontinuation of the commonly used AOAC 930.15 (135°C / 2 hr) method for animal feeds, forages and grains due to gross overestimation of water in most feed materials.

It is impossible to recommend one universal LOD method due to the diverse nature of animal feeds. The working group is recommending four different methods for the following matrix types.

RECOMMENDED METHODS

Loss on Drying

Dry animal feeds, grains, partially dried forages

Drying in forced draft oven at **104°C for 3 hours** (NFTA Method 2.1.4, AOAC Official Method 935.29 & 945.15)

RECOMMENDED METHODS

Loss on Drying

<u>Forages, ensiled materials, wet feeds and grains</u>

Two-step moisture method as follows:

Determine **partial dry matter** by drying the entire laboratory sample in a forced draft oven at **55°C overnight** or until dry (NFTA 2.2.1.1)

Comminute the entire laboratory sample to 1 mm particle size

Determine **laboratory dry matter** by drying a test portion in forced draft oven at **104°C for 3 hours** (NFTA Method 2.1.4)

Calculate **total dry matter:** *% total dry matter = (% partial dry matter x % laboratory dry matter) / 100*

Calculate **total moisture:** *% total moisture = 100 - % total dry matter*

RECOMMENDED METHODS

Loss on Drying

Liquid feeds

Drying in vacuum oven at **60°C for 18 hours** (AOAC Official Method 925.45D)

RECOMMENDED METHODS

Loss on Drying

Ingredients

Most ingredients used in feeds and pet foods can be analyzed for moisture using the 104C for 3 hours LOD method without introducing sources of error such as caramelization or loss of other volatiles.

In cases where a specific method has been validated or published for an ingredient by a body, and the customer requires, then the matrix specific method should be used and cited.

Oilseed meal – forced draft 130C for 2 hr (AOCS Ba2a-38)
Dried milk products – vacuum 100C 5 hr (AOAC 927.05)
Fish, seafood – forced draft 100C 1 hr (AOAC 952.08)
Meat & poultry – forced draft 125C 4 hr (AOAC 950.46B(b)

RECOMMENDED METHODS
Loss on Drying

Pet Food

The moisture working group is in the process of evaluating data on LOD methods for pet foods and will be making a recommendation in the near future.

RECOMMENDED METHODS

Reporting of Results

Moisture, loss on drying at 104°C / 3 hr
Moisture, loss on drying, vacuum 60°C / 18 hr
Moisture, Karl Fischer AOAC 2001.12
Moisture, NIRS legume hay calibration

CRITICAL FACTORS

Loss on Drying

Temperature

Must be consistent

Controlled to within ±1C

Check and record oven temp each time using a thermometer or thermocouple, not the digital display

Temperature monitoring device needs to be calibrated against NIST thermometer at least annually.

CRITICAL FACTORS

Loss on Drying

Time

Do not shorten specified time.

Can extend by 10-15 minutes

Time starts when oven returns to designated temp

CRITICAL FACTORS

Loss on Drying

Correct Method

Many laboratories **inappropriately** use the same drying temperature and time for all of their test materials. This is acceptable only if all materials tested are similar, such as in a manufacturing facility. The analyst needs to keep in mind when encountering a different matrix that it may require the use of a different method.

CRITICAL FACTORS

Loss on Drying

Oven

Forced draft or mechanical convection

Control air velocity

Shelves and containers cannot inhibit air circulation

Short recovery time

Avoid overloading

CRITICAL FACTORS

Loss on Drying

Drying Containers

Sufficiently large to provide a surface area that allows the test portion to be spread out to provide maximum exposure for drying

Should be shallow which will bring the heated oven air more closely over the surface of the test material

Aluminum is ideal since it has low specific heat resulting in rapid cooling

Covers not needed with electronic balances

CRITICAL FACTORS

Loss on Drying

Desiccation

Desiccant needs to be changed or regenerated periodically

Use desiccant with color indicator

Desiccate for short time only – until cool and not overnight

Keep desiccator closed between weighings

CRITICAL FACTORS

Loss on Drying

Balance

Verify calibration each day

Use weights that bracket the mass of the drying container & test portion

Use same balance

Calibrate annually

CRITICAL FACTORS
Karl Fischer

Maintain dry conditions for titration vessel and reagents

Standardize against known quantity of H2O

Ensure all H2O is released from test material

- special solvents – formamide or octanol
- homogenizer for solids
- thermal extraction

Replicate test portions

CRITICAL FACTORS
NIRS

Equations standardized against KF or KF validated LOD method

Update equations with recently analyzed test materials

Matrix match calibration equations to test material

CRITICAL FACTORS

All Methods

Particle and Test Portion Sizes and Comminution

Comminute to small particle size to provide maximum surface area

Small particle size ensures more representative test portion of adequate mass

Avoid heat generation during comminution

Store in air-tight container

CRITICAL FACTORS

All Methods

Quality Assurance

Proficiency testing program

QC material in each run

Control charting to monitor QC material