National Forage Testing Association Reference Method

NFTA Method 2.1.4 - Dry Matter by Oven Drying for 3 hr at 105°C

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Introduction

NFTA has done research to determine the best Dry Matter (DM) method as a reference method for forage testing. Karl Fischer dry matter determination is a direct measure of water by titration and is generally accepted as the standard against which all other procedures are compared. All oven-based determinations may differ from actual sample DM because a) some volatile compounds may be driven off at higher temperatures or with long drying periods, 2) lower temperatures or short drying periods may fail to remove all moisture from a sample. NFTA has found that the oven method described herein provided a close relationship to the Karl Fischer DM method for forage samples, and adapted as a reference method in 2000.

Scope

This procedure is applicable for the determination of dry matter on ground air-dry or partially dried (>85% dry matter) forages with low volatile acid content. Samples dried by this procedure are not appropriate for subsequent fiber, lignin, or detergent insoluble nitrogen analysis. Volatile acids and alcohols may be lost from fermented samples when using this method. This procedure is recommended for developing unfermented forage dry matter calibrations for NIR.

Basic Principle

Moisture is evaporated from the sample by oven drying. Dry matter is determined gravimetrically as the residue remaining after drying.

Equipment

Forced-air drying oven at $105^{o} \pm 3^{o}$C. Oven should be equipped with a wire rod shelf to allow the circulation of air. It should be vented and operated with vents open.

1. Analytical electronic balance, accurate to 0.1 mg
2. Aluminum dish (pan), > 50 mm diameter, <40 mm deep
3. Desiccator.
   a) Slide the desiccator lid open. Do not place the lid on the countertop with the grease side down. The grease will pick up dirt, preventing formation of a seal.
   b) Seals should be kept clean and well greased and the lid should always slide easily on or off. If the lid "grabs," it is time to remove the old grease and apply fresh lubricant.

c) If a lid can be directly lifted off the desiccator, either the desiccator was not properly sealed or, more likely, it needs fresh lubricant.

d) Rubber stoppers in the lid should always be pliable.

e) Desiccant should be checked daily and dried weekly. Replace at least twice annually. Use of desiccant with color indicator for moisture is recommended.

Reagents

None.

Safety Precautions

- Use standard precautions when working around electrical equipment or glassware.
- Make sure that all electrical equipment is properly grounded and installed and maintained by qualified electricians.

Procedure

1. Dry aluminum dish at 105°C ±3° C for at least 2 hr.
2. Remove dishes to desiccator. Sample dishes should not be packed excessively tight in a desiccator. Air movement is necessary to cool sample dishes. Dishes should not touch each other.
3. Immediately cover desiccator and allow dishes to cool to room temperature. Weigh the dishes as soon as possible after they have come to room temperature. Do not allow dishes to remain in desiccator more than 2 to 3 hr.
4. Weigh dishes (W_1) to nearest 0.1 mg, removing one at a time from desiccator and keeping desiccator closed between dish removals.
5. Add approximately 2 g ground sample to each dish. Record weight of dish and sample (W_2) to nearest 0.1 mg.
6. Shake dish gently to uniformly distribute the sample and expose the maximum area for drying.
7. Insert samples into preheated oven at 105ºC and dry for 3 hr after oven has returned to temperature. Time and temperature must be adhered to closely. Samples should be placed in drying oven so that air can circulate freely. Containers should not touch.
8. Move samples to desiccator, seal desiccator and allow to cool to room temperature. Dried forages have a great affinity for moisture and will absorb moisture from a desiccant if allowed time to do so. Weigh the samples as soon as possible after they have come to room temperature. Do not allow samples to remain in desiccator for more than 2 to 3 hr. Open a loaded desiccator very slowly after samples have cooled. A vacuum forms during cooling and abrupt opening results in turbulence, which can blow samples out of uncovered containers.
9. Weigh dish and dried sample (W_3), recording weight to nearest 0.1 mg. Desiccator lid should be slid open for the removal of each container and closed during weighing. Leaving the lid open allows samples to absorb moisture.

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Calculations

Percent Total Dry Matter (Total DM)

$$\%\ Total\ DM = \frac{W_3 - W_1}{W_2 - W_1}\ X\ 100$$

Where W_1= tare weight of dish in grams
W_2 = initial weight of sample and dish in grams
W_3 = dry weight of sample and dish in grams

Example Calculation

Weight of dish = 14.0238 grams
Weight of sample + dish = 16.0523 grams
Weight of sample + dish after drying = 15.8256 grams

$$\%\ Total\ DM = \frac{15.8256 - 14.0238}{16.0523 - 14.0238}\ X\ 100 = 88.82325\%$$

Reporting Rules

Report Dry Matter as a percent to one place past the decimal.

Example:
The above example calculation should be reported as 88.8%

Quality Control

Include at least one set of duplicates in each run if single determinations are being made.

An acceptable average standard deviation among replicated analyses for moisture or dry matter is about ±0.10 %DM, which results in a warning limit (2s) of ±0.20 %DM and a control limit (3s) of ±0.30 %DM.

Plot the results of the duplicate analyses on an R-control chart and examine the chart for trends. Results outside the warning limits warn of possible problems with the analytical system. Results outside the control limits indicate loss of control, and results of the run should be discarded. If more than five or six points in succession fall on one side or the other of the 50 percent line, it is a strong indication that something has changed and is cause for investigation.

See the NFTA's Quality Assurance/ Quality Control Guidelines document for additional guidance.

Reference

B. Shreve, N. Thiex, M. Wolf. 2006. National Forage Testing Association Reference Method: Dry Matter by Oven Drying for 3 Hours at 105° C. NFTA Reference Methods. National Forage Testing Association, Omaha, NB, www.foragetesting.org

Drying of feeds at 135°C for 2 hr. (930.15). Official Methods of Analysis. 1990. Association of Official Analytical Chemists. 15th Edition.