Direct Moisture Determination of Food and Feed Samples Comparing Automated Thermogravimetric and Air Oven loss-on-drying Techniques

Mason Marsh, Jenifer Laurin, Adam Darling | LECO Corporation, St. Joseph, MI USA

Introduction

An accurate determination of moisture content in food and feed products provides important information related to the product quality and safety (texture, taste, microbial stability), as well as a key variable used to calculate the product purity, yield, and resulting constituent analysis on a dry basis. One of the most common direct moisture determination methods is loss-on-drying using an air oven where the sample is weighed before and after exposure to an elevated temperature within an air ventilated oven. The resulting sample mass loss is calculated as moisture.

AOAC direct moisture determination methods utilizing the air oven loss-on-drying technique are widely used in the food (AOAC Method 925.10) and feed (AOAC Method 930.15) industries. A new, automated thermogravimetric moisture instrument (TGM800) is now available with the ability to meet the AOAC method sample mass, oven temperature, and air flow requirements, while providing many additional benefits including work flow efficiency, minimal operator time, and optimal analysis time due to sample batch throughput of up to 16 samples with automated end point recognition based upon sample mass constancy.

This poster presentation will cover the comparison of direct moisture loss-on-drying determination using an automated thermogravimetric moisture instrument (TGM800), and a manual air dry loss method using a 4-place balance, air oven, and desiccator. The comparison will include food matrix sample data measured with the TGM800 and air oven techniques following AOAC Method 925.10 and feed matrix sample data following AOAC Method 930.15.

Referenced Methods

- AOAC Official Method 925.10 Solids (Total) and Moisture in Flour—Air Oven Method
- AOAC Official Method 930.15 Moisture in Animal Feed—Loss-on-Drying at 135 °C for 2 hours

TGM800 Theory of Operation

The TGM800 is a thermogravimetric analyzer designed to directly determine moisture content of materials using a loss-on-drying technique. Mass loss of the sample is measured as a function of the oven temperature by controlling the atmosphere and ventilation rate. The instrument consists of a computer, an integrated four-place balance, and a multiple sample oven that allows up to 16 samples to be analyzed simultaneously.

After an analysis method has been selected, empty aluminum foil crucibles are loaded into the oven carousel. The instrument supports the use of either a 1.5 inch diameter aluminum foil crucible with a 1 gram nominal sample mass, or a 2.4 inch diameter aluminum foil crucible with a nominal sample mass of 3 grams. The analysis method controls the carousel, oven, atmosphere ventilation, and balance operation. On completion of the crucible tare, each crucible is presented to the operator for sample loading. The initial sample weight is measured and stored automatically. Once all the crucibles have been loaded, analysis begins with the oven temperature ramping to a set point, the oven atmosphere ventilation rate starting, and the sequential collection of the individual samples mass. The weight loss of each sample is monitored and the oven temperature and atmosphere ventilation rate are controlled according to the selected analysis method. Method analysis length can be programed to a fixed time or dependent on the sample mass constancy. The moisture result is calculated as a percent mass loss for each sample and reported at the end of the analysis.

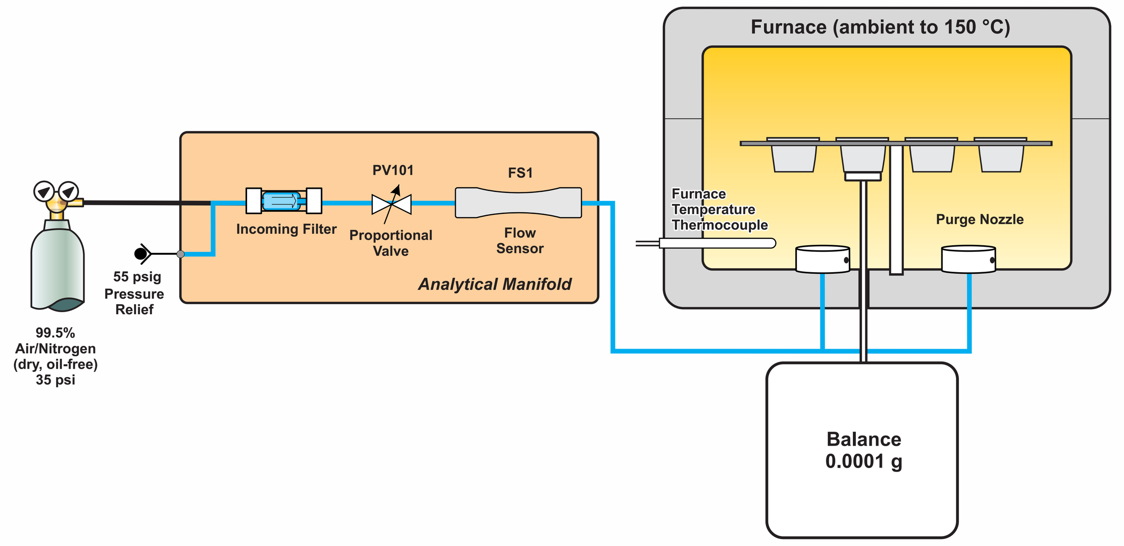
The instrument contains an intuitive touchscreen interface that enables complete access to analysis control, method settings, diagnostics, sample reporting, and more in a highly organized and immersive environment. Analysis methods can be tailored to satisfy most moisture applications with editable oven temperature, temperature ramp rate, and atmosphere and ventilation rate. The software also provides on-screen plotting of sample weight loss and temperature.

TGM800

Figure 1. 16 sample carousel using the 1.5 inch aluminum foil crucible.

Figure 2. 10 sample carousel using the 2.4 inch aluminum foil crucible.

TGM800 Flow Diagram



Methodology

AOAC Official Method 925.10—Solids (Total) and Moisture in Flour
A ~2 gram sample is weighed into an aluminum dish with a cover, dried in a ventilated oven at 130 ± 3 °C for 1 hour, with a hold time that begins when the oven attains a temperature of 130 °C. The dish is covered before removal from the oven, transported to a desiccator, and cooled to room temperature before removing the final mass.

AOAC Official Method 930.15—Moisture in Animal Feed
A ~2 gram sample is weighed into an aluminum dish with a cover, dried in an oven at 135 ± 2 °C for 2 hours. The dish is covered before removal from the oven, then transported to a desiccator, and cooled before taking the final mass.

Manual Moisture
When determining moisture using the manual method, the operator must ensure that the desiccant is new and that the desiccator is sealed properly. The oven should be verified at 130 ± 3 °C for AOAC Method 925.10 and 135 ± 2 °C for AOAC Method 930.15. The aluminum dish and lids should be cleaned and dried at 130 ± 3 °C prior to analysis when following AOAC Method 925.10.

To begin, a clean drying dish with cover is placed on a balance and the mass recorded. The sample is added and spread evenly into the dish, and the mass of the sample is recorded. Once the sample mass data for the batch of samples has been collected, the samples are placed into a preheated drying oven. The hold time starts when the oven has attained the required drying temperature of the specified method. Once the hold time limits have been reached, the oven is opened, covers are quickly placed on the trays and the samples are transferred to a desiccator. Samples are cooled in the desiccator to room temperature for 20 minutes. A final mass is recorded and the percentage of moisture is calculated.

Table 1: Manual Oven Loss-on-Drying Method Parameters

Method	AOAC 925.10	AOAC 930.15
Crucible Type	Aluminum Dish	Aluminum Dish
Nominal Mass	~2 g	~2 g
Oven Temperature	130 °C	135 °C
Hold Time	60 min	120 min

Three methods were defined based upon AOAC Method 925.10—Moisture in Flour (named Flour-Method 1, 2, and 3), and three methods were defined based upon AOAC Method 930.15—Moisture in Feeds (named Feed-Method 1, 2, and 3).

For Flour-Method 1 and Feed-Method 1, samples were weighed into a 2.4 inch diameter aluminum foil crucible at the minimum mass of 2 grams and fixed drying time required by the applicable AOAC method. These methods follow the requirements of both AOAC manual methods.

For Flour-Method 2 and Feed-Method 2, samples were weighed into a 1.5 inch diameter aluminum foil crucible at a lower mass of ~1 gram, with fixed times to follow the required drying time in the AOAC methods. These methods deviate from the AOAC method requirements by using a lower sample mass of 1 gram and a smaller diameter aluminum foil crucible.

For Flour-Method 3 and Feed-Method 3, samples were weighed into a 2.4 inch diameter aluminum foil crucible at a lower sample mass (~1 g), with the drying time determined by the sample mass constancy. These methods deviate from the AOAC methods by using a lower sample mass of ~1 gram and the automated drying time end point feature.

Table 2: TGM800 Flour Method Parameters

Method	Flour-Method 1	Flour-Method 2	Flour-Method 3
Crucible Type	2.4 in Ø Al Foil	1.5 in Ø Al Foil	2.4 in Ø Al Foil
Nominal Mass	~2 g	~1 g	~1 g
Ramp Rate	10 °C/min	10 °C/min	10 °C/min
Start Temperature	25.0 °C	25.0 °C	25.0 °C
End Temperature	130.0 °C	130.0 °C	130.0 °C
Hold Time	60 min	60 min	0 min
Ventilation Flow Rate	4.0 LPM	4.0 LPM	4.0 LPM
Final Mass	At End Of Step	At End Of Step	At Constancy

Table 3: TGM800 Feed Method Parameters

Method	Feed-Method 1	Feed-Method 2	Feed-Method 3
Crucible Type	2.4 in Ø Al Foil	1.5 in Ø Al Foil	2.4 in Ø Al Foil
Nominal Mass	~2 g	~1 g	~1 g
Ramp Rate	10 °C/min	10 °C/min	10 °C/min
Start Temperature	25.0 °C	25.0 °C	25.0 °C
End Temperature	135.0 °C	135.0 °C	135.0 °C
Hold Time	120 min	120 min	0 min
Ventilation Flow Rate	4.0 LPM	4.0 LPM	4.0 LPM
Final Mass	At End Of Step	At End Of Step	At Constancy

Sample Preparation

Four commercial flour samples were sourced representing a barley, wheat, rice, and corn matrix. Commercial flour is typically ground and passed through a 425 µm (40 mesh) screen during the production process.

The feed samples listed as hay and corn silage were ground in a cyclone mill, and passed through a 1.0 mm screen. The feed samples listed as distillers and corn grain were ground in a cyclone mill, and passed through a 0.5 mm screen.

Sample Results

A sample suite for food and feed materials was chosen to demonstrate the analytical performance and application capabilities of the method derivatives being evaluated. Four commercial flour samples were used to represent the food materials and corn, hay, and distillers grains samples were used to represent the feed materials. The average moisture results, standard deviation of the average moisture measurement, and total analysis times are listed in Table 4 and 5 below for both the food and feed materials. The total analysis calculated within the results table includes the time from the measurement of the samples initial mass to the measurement of the final mass of the last sample replicate with all of the subsequent drying, sample transfer, and cooling time (if applicable) included.

Table 4: Flour Moisture Results

Flour Samples	Replicate Number		Manual Moisture (AOAC)	Flour-Method 1 (Fixed Time) 2.40" Ø Al Crucible	Flour-Method 2 (Fixed Time) 1.51" Ø Al Crucible	Flour-Method 3 (At Constancy) 2.40" Ø Al Crucible
Barley Flour	n=7	Average Moisture (%)	7.81	7.89	7.90	7.84
		Standard Deviation	0.07	0.03	0.03	0.05
		Total Analysis Time (h:m)	1:58	1:23	1:28	0:50
		Nominal Mass	~2 grams	~2 grams	~1 gram	~1 gram
Wheat Flour	n=7	Average Moisture (%)	7.51	7.67	7.59	7.55
		Standard Deviation	0.09	0.02	0.03	0.09
		Total Analysis Time (h:m)	1:59	1:22	1:28	0:52
		Nominal Mass	~2 grams	~2 grams	~1 gram	~1 gram
Rice Flour	n=7	Average Moisture (%)	7.90	7.92	7.98	7.93
		Standard Deviation	0.04	0.02	0.03	0.09
		Total Analysis Time (h:m)	1:57	1:21	1:29	1:08
		Nominal Mass	~2 grams	~2 grams	~1 gram	~1 gram
Corn Flour	n=7	Average Moisture (%)	7.91	7.93	7.96	7.93
		Standard Deviation	0.09	0.02	0.01	0.05
		Total Analysis Time (h:m)	1:56	1:20	1:29	0:56
		Nominal Mass	~2 grams	~2 grams	~1 gram	~1 gram

Table 5: Feed Moisture Results

Feed Samples	Replicate Number		Manual Moisture (AOAC)	Feed-Method 1 (Fixed Time) 2.40" Ø Al Crucible	Feed-Method 2 (Fixed Time) 1.51" Ø Al Crucible	Feed-Method 3 (At Constancy) 2.40" Ø Al Crucible
Distillers	n=7	Average Moisture (%)	12.67	12.86	12.52	12.68
		Standard Deviation	0.33	0.04	0.01	0.19
		Total Analysis Time (h:m)	2:58	2:22	2:33	2:10
		Nominal Mass	~2 grams	~2 grams	~1 gram	~1 gram
Corn Grain	n=7	Average Moisture (%)	2.13	2.20	2.17	2.14
		Standard Deviation	0.07	0.02	0.02	0.08
		Total Analysis Time (h:m)	2:58	2:21	2:33	0:50
		Nominal Mass	~2 grams	~2 grams	~1 gram	~1 gram
Corn Silage	n=7	Average Moisture (%)	4.14	4.16	4.02	4.10
		Standard Deviation	0.27	0.03	0.03	0.11
		Total Analysis Time (h:m)	3:01	2:23	2:31	1:33
		Nominal Mass	~2 grams	~2 grams	~1 gram	~1 gram
Hay	n=7	Average Moisture (%)	3.43	3.40	3.41	3.31
		Standard Deviation	0.28	0.03	0.05	0.10
		Total Analysis Time (h:m)	2:59	2:22	2:31	1:55
		Nominal Mass	~2 grams	~2 grams	~1 gram	~1 gram

All of the moisture results obtained using the TGM800, with multiple method modifications from the AOAC requirements, were in agreement with the Manual AOAC Method results for both the food and feed materials. The precision of the TGM800 for all of the methods were similar or better than the manual AOAC Method for both the food and feed materials, with some sample matrices having as much as an order of magnitude reduction in standard deviation when measured using the TGM800. The TGM800 fixed drying time methods yielded approximately a 30 to 40 minute reduction in total analysis time versus the manual AOAC methods for both the food and feed materials due to the elimination of the requirement to transfer the samples from an oven to a desiccator for cooling prior to measurement of the final sample mass. The total analysis time from the Food- and Feed-Method 3 using the automated drying time end point detection feature of the TGM800 and the 2.4 inch diameter aluminum foil crucible with ~1 gram sample mass were reduced by an additional 20 to 60 minutes versus the fixed drying time TGM800 method and 50 to 90 minutes versus the manual AOAC methods.

Conclusion

The objective of this poster presentation was to demonstrate the analytical performance and application capabilities of the TGM800 instrument using multiple method parameters in comparison with the manual air oven methods used in the AOAC methods. The TGM800 moisture results using all of the examined method modifications were comparable to the results obtained by the manual AOAC methods. The TGM800 moisture results for most samples have significantly improved precision compared to the manual AOAC methods, with some samples having as much as an order of magnitude improvement in precision. When using fixed drying time method conditions that are compliant to the manual AOAC methods, the total analysis time of the TGM800 was 30 to 40 minutes shorter than the manual AOAC methods due to the elimination of sample transfer to a desiccator and the associated cooling time. A 50 to 90 minute time savings in total analysis time can be achieved compared to the manual AOAC method when using TGM800 method settings with the automated drying time end point detection based upon the samples mass constancy and the 2.4 inch diameter aluminum foil crucibles with ~1 gram sample mass.

The TGM800 provides the user with flexible method settings, automation, and hardware capabilities that maximize moisture determination efficiency, productivity, and analytical performance while offering the ability to meet the primary loss-on-drying method requirements for sample mass, oven temperature, and ventilation requirements

Delivering the Right Results