

Direct Moisture Determination of Various Grain/Seed Products Comparing Automated Thermogravimetric Method and the Oven Drying Methods Used by the Southern African Grain Laboratory

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Introduction

An accurate determination of moisture content in grains, grain products, and seeds provides important information related to the quality and safety, as well as being a key variable used to calculate the product purity, yield, and resulting constituent analysis on a specific moisture basis. One of the most common direct moisture determination methods is loss-on-drying using a hot air ventilation oven, where the sample is weighed off before and after exposure to an elevated temperature within an air-ventilated oven. The resulting sample mass loss is calculated as moisture.

Various methods, based on internationally accepted methods, are implemented by the Southern African Grain Laboratory for moisture determination in the various products they analyse. Some of these include AACCI, ICC, ISO, and ISTA methods.

This poster presentation will cover the comparison of direct moisture loss-on-drying determination using the automated thermogravimetric moisture instrument (TGM800), and the manual air dry loss methods using a four-place balance, air oven, and desiccator. The comparison will include sample data measured with the TGM800 and air oven techniques.

TGM800 Theory of Operation

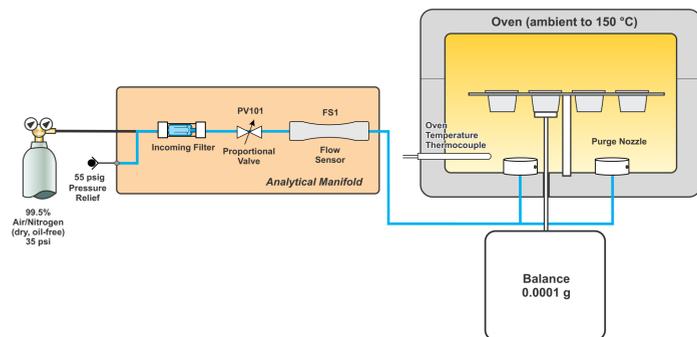
The TGM800 is a thermogravimetric analyser designed to directly determine moisture content of materials using a loss-on-drying technique. Mass loss of the sample is measured as a function of the oven temperature by controlling the atmosphere and ventilation rate. The instrument consists of a computer,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

After an analysis method has been selected, empty aluminium foil crucibles are loaded into the oven carousel. The instrument supports the use of either a 1.5 inch diameter aluminium foil crucible with a 1 gram nominal sample mass, or a 2.4 inch diameter aluminium 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 mass 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 mass 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 programmed to a fixed time, or based 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.



Flow Diagram



Methodology

AACCI 44-15.02 Moisture in Maize Meal

A ~2 g sample is weighed into a moisture dish with cover and dried in a ventilated oven at 130 °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 measuring the final mass.

ICC 110/1 Moisture in Whole Wheat Flour (2 hours)

ICC 110/1 Moisture in White Wheat Flour (1.5 hours)

A ~5 g sample is weighed into a moisture dish with lid and dried in a ventilated oven at 130 °C for 1.5 to 2 hours, 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 measuring the final mass.

ISO665-2000(E) Moisture in Whole Sunflower Seeds

A ~5 g sample is weighed into a moisture dish with lid and dried in a ventilated oven at 103 °C for 3 hours with a hold time that begins when the oven attains a temperature of 103 °C. The dish is covered before removal from the oven, transported to a desiccator, and cooled to room temperature before measuring the first mass. The sample is placed back in the oven for an additional 1 hour at 103 °C. The dish is covered before removal from the oven, transported to a desiccator, and cooled to room temperature before measuring the final mass.

Manual Moisture

When determining moisture using the manual method, the operator must ensure that the desiccant is dry, and that the desiccator is sealed properly. The oven should be verified at 130 °C ± 3 °C for the AACCI 44-15.02 and ICC 110/1 Methods, and 103 °C ± 2 °C for the ISO665-2000(E). The moisture dish and lids should be clean and dry.

To begin, a clean drying dish with lid 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, lids are quickly placed on the dishes, and the samples are transferred to a desiccator. Samples are cooled in the desiccator to room temperature. A final mass is recorded and the percentage of moisture is calculated.

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

Method	AACCI 44-15.02	CC 110/1 Whole Wheat Flour	ICC 110/1 White Wheat Flour	ISO 665-2000(E)
Nominal Mass	~2 g	~5 g	~5 g	~5 g
Oven Temperature	130 °C	130 °C	130 °C	103 °C
Hold Time	60 min	120 min	90 min	240 min

Three methods (130 °C 1.5 in, 130 °C 2.4in, and 103 °C 2.4 in) were defined based upon temperature and sample size to cover the various samples. All methods created were set to end at mass constancy to determine if there is a time saving in analysis, and therefore improvement in sample throughput.

Table 2: TGM800 Methods

Method	130 °C 1.5 in (ICC)	130 °C 2.4 in (AACCI)	103 °C 2.4 in (ISO)
Crucible	1.5 in Ø Al Foil	2.4 in Ø Al Foil	2.4 in Ø Al Foil
Nominal Mass	~1 g	~2 g	~3 g
Ramp Rate	10 °C/min	10 °C/min	10 °C/min
Start Temperature	25 °C	25 °C	25 °C
End Temperature	130 °C	130 °C	103 °C
Hold Time	0 min	0 min	0 min
Ventilation Flow	4.0 LPM	4.0 LPM	4.0 LPM
Final Mass	At Constancy	At Constancy	At Constancy

Sample Results

A sample suite was chosen to demonstrate the analytical performance and application capabilities of the method derivatives being evaluated. Six (6) whole wheat flour and white wheat flour samples, ten (10) maize meal samples, and one (1) whole seed sunflower sample were selected for analysis, as these samples were production samples being analysed by the Southern African Grain Laboratory. The TGM800 results were generated on the same day as to eliminate any variation that may occur due to moisture loss during storage. The average moisture, total analysis time, and difference between manual and TGM800 results are listed in Table 3. The total analysis time includes the time from the measurement of the sample's initial mass, to the measurement of the sample's final mass with all of the subsequent drying, transfer, and cooling times (if applicable) included.

Table 3: Sunflower Results

Samples	Manual Moisture	103 °C 2.4 in (ISO)	Difference	
1049(1)	Average Moisture (%)	6.49	6.43	-0.06
1049(2)	Average Moisture (%)	6.64	6.59	-0.05
Total Analysis Time (h:m)		6:00	2:37	-3:23

Table 4: Maize Meal Results

Samples	Manual Moisture	130 °C 2.4 in (AACCI)	Difference	
1036	Average Moisture (%)	13.48	13.40	-0.08
1037	Average Moisture (%)	13.45	13.40	-0.05
1038	Average Moisture (%)	13.19	13.10	-0.09
1040	Average Moisture (%)	12.57	12.50	-0.07
1168	Average Moisture (%)	13.87	13.80	-0.07
1169	Average Moisture (%)	13.56	13.60	0.04
1170	Average Moisture (%)	13.92	14.00	0.08
1171	Average Moisture (%)	13.56	13.60	0.04
C2291(3)	Average Moisture (%)	10.92	10.90	-0.02
C2291(4)	Average Moisture (%)	10.86	10.80	-0.06
Total Analysis Time (h:m)		2:15	1:39	-0:36

Table 5: Flour Results

Samples	Manual Moisture	130 °C 1.5 in (ICC)	Difference	
SAGL1A	Average Moisture (%)	12.65	12.60	-0.05
31	Average Moisture (%)	13.40	13.50	0.10
32	Average Moisture (%)	13.39	13.50	0.11
33	Average Moisture (%)	13.37	13.40	0.03
34	Average Moisture (%)	13.36	13.40	0.04
1156	Average Moisture (%)	13.29	13.40	0.11
White wheat Flour	Total Analysis Time (h:m)	2:45	0:41	-2:04
SAGL1B	Average Moisture (%)	9.84	9.88	0.04
Whole Wheat Flour	Total Analysis Time (h:m)	3:15	0:42	-2:33

Conclusion

The objective of this poster was to demonstrate the analytical performance and application capabilities of the TGM800 instrument using the constant mass feature to reduce analysis time in comparison with the manual air oven method used in the ICC, AACCI, and ISO methods. The TGM800 moisture results obtained using the method modifications were comparable to the results obtained by the manual methods. The analysis times of the TGM800 are significantly shorter than those of the manual method with up to a 78% reduction in analysis time for the whole wheat flour method (ICC 110/1). The TGM800 eliminates the need for sample cooldown in the desiccator, and the additional weighing step by the analyst.

The TGM800 provides the user with flexible method setting, 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.

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