The AquaLab TrueDry CV9: Moisture Content Analysis Meets Good Science

by Brady Carter

Moisture content is a measure of the quantity of water in a product reported on either a wet or dry basis. Moisture content provides valuable information about yield and purity, making it important from a financial standpoint. In addition, moisture content provides information about texture since increasing levels of moisture provide mobility and lower the glass transition temperature. In theory, moisture content determination is simply a comparison of the amount of water in a product to the mass of everything else in the product. While it is simple in theory, further investigation of moisture content demonstrates that for such a simple concept, it is an extremely complex process to actually obtain reliable results.

Moisture Content Measurement

When it comes to determining the amount of water in a product, there are many choices available. The AOAC lists 35 different methods for measuring moisture content. These are classified as either direct or indirect measurement methods. Direct moisture content methods either force water out of a sample at elevated temperatures and track the weight change or involve a chemical reaction with water and titration. The most common direct moisture methods include air-oven drying and Karl Fischer titration. Indirect methods try to predict the moisture content based on either testing under accelerated heat conditions or by correlating another measured property to the moisture content. These secondary methods require calibration to a primary or direct method. Examples of indirect measurement methods include: halogen or IR based moisture balances, NIR absorption, and dielectric capacitance. The advantage of direct methods is that they are a primary measurement typically with superior precision, but may have the disadvantage of being more labor intensive and having long analysis times. Indirect methods are typically much faster than direct methods, but are not primary measurements based on accepted standards and consequently can suffer in reliability. Due to the absence of a scientific definition of “dry”, all moisture methods suffer from the lack of a moisture standard to allow the comparison of methods or determination of accuracy. Further, any loss-on-drying method is subject to the ambient conditions under which the measurement is made. The ideal moisture method would combine high throughput testing with a primary measurement method, eliminate variability due to changing ambient conditions, and provide a scientific standard for “dry.”

AquaLab TrueDry CV-9

The AquaLab TrueDry CV9 utilizes a unique design combined with a sound scientific understanding of moisture loss to create the ideal loss-on-drying moisture analyzer. A turntable approach enables high sample throughput by analyzing up to 9 samples simultaneously using primary reference methods (Figure 1). The temperature of each sample is controlled individually using controlled contact drying and the weight loss of each sample is tracked over time. An easy to use test setup interface makes it simple to match any reference moisture method without the need to use extreme temperatures to predict the moisture content. Table 1 provides the average moisture content and Table 2 provides the precision of the TrueDry compared to a conventional oven and a moisture balance for multiple sample types. For the comparisons, 3 replicated moisture analyses were used in each of a conventional oven, moisture balance, and

TrueDry to determine the average and standard deviation for each sample type. For the TrueDry and the conventional oven, the moisture test settings were according to the Swiss Food Manual for each product type, and the settings for the moisture balance were based on test profiles pre-loaded by the manufacturer.

Table 1. Average moisture content measurements (%moisture w.b.) of a conventional oven, moisture balance, and TrueDry for 10 sample types.

Sample	Conventional Oven	Moisture Balance	TrueDry CV-9
Milk	88.68	86.95	88.16
Ketchup	67.30	64.84	66.06
Ranch Dressing	43.28	45.00	43.57
Bread	39.28	38.88	38.58
Parmesan Cheese	30.90	31.32	29.32
Sweetened Cond Milk	19.79	23.61	19.49
Flour	11.42	11.76	11.69
Whole Wheat	10.32	10.54	10.46
Coffee	5.59	5.46	5.19
Resin Pellets	0.16	0.11	0.12

Table 2. Precision of moisture content measurements (%moisture w.b.) of a conventional oven, moisture balance, and TrueDry for 10 sample types.

Sample	Conventional Oven	Moisture Balance	TrueDry CV-9
Milk	0.04	0.15	0.08
Ketchup	0.12	0.34	0.18
Ranch Dressing	0.23	0.19	0.15
Bread	0.05	0.10	0.06
Parmesan Cheese	0.43	0.42	0.33
Sweetened Cond Milk	0.47	0.40	0.38
Flour	0.09	0.05	0.04
Whole Wheat	0.05	0.04	0.02
Coffee	0.05	0.06	0.03
Resin Pellets	0.01	0.01	0.01

AquaLab TrueDry vs. Oven Loss-on-drying

A majority of loss-on-drying reference methods state that a sample should be dried at a given temperature until constant weight. However, a suggested rate of weight loss that identifies

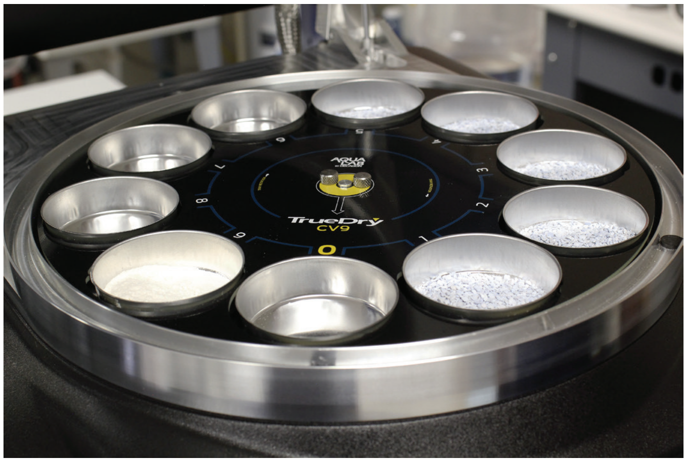

Figure 1. Turntable design of the TrueDry to enable analysis of 9 samples simultaneously.

constant weight is rarely provided. In addition, in practice, it would be very labor intensive when using a conventional oven and balance, to repeatedly check the weight of each sample while looking for constant weight. Consequently, these reference methods also include a suggested analysis time, which is what most analysts will use. These test times will be conservatively long to ensure that constant weight has been achieved. The advantage of the TrueDry is that it tracks the weight change gravimetrically and can easily compare the current weight reading to the previous reading and determine when the weight loss rate has fallen below a user controlled trigger value. By ending the moisture determination based on constant weight, the results of the TrueDry can be obtained in a shorter time period than reference methods with equivalent precision. In addition, the TrueDry eliminates multiple interruptions that are needed to move samples in and out of the oven and to take weight measurements, which also eliminates chances for mistakes that can occur each time the sample is handled. A comparison of testing performance on white bread between a conventional oven and the TrueDry indicates that while the oven required 20 minutes of work time, 110 minutes of analysis time and 3 interruptions to work flow to analyze 9 samples, the TrueDry only required 4 minutes of work time, 70 minutes of analysis time, and 1 interruption to work flow (Table 3). This works out to an average of 14 minutes total test time (this includes both preparation and test time) per sample for the oven, but only 8.7 minutes per sample in the TrueDry with an equivalent level of precision.

Table 3. Moisture content test time and precision for a conventional oven and the TrueDry on white bread.

	Conventional Oven	TrueDry CV-9
Reading Time (9 samples)	110 minutes	70 minutes
Work Time (9 samples)	20 minutes	4 minutes
Interruptions	3	1
Standard Deviation	0.05	0.06
Drying Temperature	130°C	130°C

AquaLab TrueDry vs. Moisture Balance Loss-on-drying

A moisture balance is designed to automate the weighing process of loss-on-drying, as well as speed up the test time by over-heating the sample. The test time or constant weight settings are adjusted so that results at high temperatures match the results that would be obtained using a reference method. A predetermined and unique testing program is needed for each type of sample prior to testing. Since a moisture balance is not a primary measurement, it lacks the reliability of a reference method. In addition, while the testing time for a single sample is reduced using a moisture balance, the advantage is lost when multiple samples are analyzed due to the numerous interruptions needed to record results and setup the next test. A comparison of testing performance on 9 samples of white bread between a moisture balance and the TrueDry indicates that as with the conventional oven method, the TrueDry required less time than the moisture balance. The moisture balance needed 54 minutes of work time, 197 minutes of analysis time and 10 interruptions to work flow to analyze 9 samples, which was significantly more than the TrueDry (Table 4). This works out to an average of 27.8 minutes total test time per sample for the moisture balance, significantly higher than both the oven (14 minutes) and the TrueDry (8.7 minutes).

Table 4. Moisture content test time and precision for a moisture balance and the TrueDry on white bread.

	Moisture Balance	TrueDry CV-9
Reading Time (9 samples)	197 minutes	70 minutes
Work Time (9 samples)	54 minutes	4 minutes
Interruptions	10	1
Standard Deviation	0.1	0.06
Drying Temperature	135°C	130°C

Comparison with Vacuum Oven

Many standard methods recommend using a vacuum oven at lower temperatures to determine moisture content. The idea with vacuum oven moisture is that by lowering the vapor pressure with a vacuum, an equivalent amount of water can be removed from the sample, at a lower temperature, than with a standard oven. By using lower temperatures, it is assumed that fewer volatiles other than water are being removed, presumably making a vacuum oven moisture more specific to water. In principle, this approach should work, but in practice there are several problems. It is extremely difficult to achieve sufficiently low pressures with typical laboratory vacuum pumps to match the value achieved in a conventional oven (around 1 kPa), let alone to achieve the even lower pressures needed to compensate for the lower sample temperatures. The range required is called “medium vacuum”, and pumps that work in this range are expensive and intolerant of water vapor. To remedy this, researchers often bleed desiccated air into the vacuum oven. A similar result could be obtained with much less expense by simply supplying desiccated air to a conventional oven.

A second problem comes in knowing how much to reduce the vapor pressure in the vacuum oven to make it equivalent to the conventional oven. The water activity of foods is temperature dependent, so the higher the temperature the more water is lost at a given water activity. The high temperature of an oven removes water from a sample both by increasing its vapor pressure and by loosening the grip of the matrix on the water. The vacuum oven can mimic the vapor pressure effect by lowering the surrounding pressure, but it can't loosen the water without increasing the sample temperature. If sample temperature is increased, then it is no different than a conventional oven.

Considering the potential pitfalls of vacuum oven drying, it would be desirable to replace routine vacuum oven testing with the TrueDry. This would only be possible if the TrueDry could give equivalent results to the vacuum oven for sample types that are typically recommended for vacuum oven such as high sugar systems. To test the viability of the TrueDry in measuring the moisture content of high sugar systems, the moisture content of Honey, Raspberry Syrup, and a 25% w/v sugar in water solution was determined in the TrueDry at 100 °C. These results were then compared to the results from a conventional oven at 103 °C, a moisture balance at 90 °C, and a vacuum oven at 60°C. The moisture content values from the TrueDry for all 3 samples did not significantly differ from those determined by the vacuum oven with much shorter analysis times (Table 5). Moisture values from the moisture balance differed from the vacuum oven for both honey and raspberry syrup while those from the conventional oven differed for the sugar/water solution and raspberry syrup. Based on these results, the TrueDry at higher temperatures can give equivalent results to the vacuum oven in much less time and with less labor, making it a viable alternative to vacuum oven testing.

Table 5. Average moisture content valuesa and analysis times for 3 high sugar products when tested in a moisture balance, conventional oven, vacuum oven, and the TrueDry.

Instruments	25% w/v Sugar Solution		Honey		Raspberry Syrup	
	% MC	Time (min)	% MC	Time (min)	% MC	Time (min)
Moisture Balance	73.05a	31.77	5.28a	22.96	18.04a	45.04
Conventional Oven	75.40b	180.00	13.43b	180.00	25.44b	120.00
Vacuum Oven	74.78a	1587.00	11.88b	2910.00	28.91c	2910.00
TrueDry	73.05a	68.93	11.91b	181.80	28.27	276.98

Improving Reproducibility of Loss-on-drying

It is difficult to obtain reproducible results with all loss-on-drying methods including both the moisture balance and conventional oven (de Knegt & van den Brink 1998; Reh et al. 2004). This is because relative humidity of the lab environment has an impact on moisture content results (de Knegt & van den Brink 1998). To better understand why this is the case requires a review of the drying process. The movement of water out of a sample during drying can be described by Fickian diffusion. The integrated form of the Fick equation gives the rate of water loss from a sample:

$$E = \frac{k}{x}(e_s - e_a) \qquad (1)$$

where E is the evaporation rate (g $m^{-2}s^{-1}$), k/x is the permeance (g/m^2 s kPa), and e_s and e_a are the vapor pressures of water at the sample surface and in the air (kPa). When the sample is said to be dry, E becomes zero because the sample and air vapor pressures become equal. In a well-ventilated oven the vapor pressure of the air in the oven equals the vapor pressure of the air in the laboratory in which the oven resides. The vapor pressure of air is the product of the air humidity (expressed as a fraction) and the saturation vapor pressure at air temperature. If we assume a typical laboratory relative humidity of 0.4 and a laboratory temperature of 25°C (saturated vapor pressure of 3.17 kPa), the vapor pressure of the oven air is 0.4 x 3.17 = 1.27 kPa.

The vapor pressure of the sample is the product of its water activity and the saturation vapor pressure at sample temperature. A typical standard drying oven temperature is 105°C. The saturation vapor pressure of water at that temperature is 121 kPa. Knowing this value and the vapor pressure of the oven air allows us to calculate the water activity of a dry sample. For the conditions specified water activity is $a_{wdry} = 1.27/121 = 0.01$.

This analysis should make several things clear:

1. "dry" does not mean there is no more water in the sample. There is always more water in the sample, and the amount of that water that is removable by the drying process depends only on the vapor pressure of the air around the sample and the sample temperature. Changing the analysis temperature not only changes the measurement speed, but also the final answer since at a controlled vapor pressure, different amounts of water can be removed at different temperatures. Thus increasing the temperature to speed up the test not only runs the risk of volatilizing substances other than water, it also will change the amount of water that can be removed.

> ***Sidebar: A quick note on vacuum oven moisture content***
> *Many standard methods recommend using a vacuum oven at lower temperatures to determine moisture content. The idea with vacuum oven moisture is that by lowering the vapor pressure with a vacuum, an equivalent amount of water can be removed from the sample, at a lower temperature, than with a standard oven. By using lower temperatures, it is assumed that fewer volatiles other than water are being removed, presumably making a vacuum oven moisture more specific to water. In principle, this approach should work, but in practice there are several problems. It is extremely difficult to achieve sufficiently low pressures with typical laboratory vacuum pumps to match the value achieved in a conventional oven (around 1 kPa), let alone to achieve the even lower pressures needed to compensate for the lower sample temperatures. The range required is called "medium vacuum", and pumps that work in this range are expensive and intolerant of water vapor. To remedy this, researchers often bleed desiccated air into the vacuum oven. A similar result could be obtained with much less expense by simply supplying desiccated air to a conventional oven.*
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2. Anything that alters the oven vapor pressure will alter the "dry" weight of the sample, so increased laboratory humidity, or lack of proper oven ventilation will result in increases in oven dry weight.

3. Water content measurements can never be accurate until the industry defines a "dry" water activity and requires drying methods to bring samples to that water activity.

4. The level of error introduced by varying ambient humidity will be heightened for higher moisture content samples.

The way to overcome the challenge posed by inconsistent lab humidity is to identify a dry vapor pressure and make sure every sample is dried to that vapor pressure at a chosen temperature. Then, the dry weight would be the weight of the sample when it has achieved this oven dry vapor pressure. The TrueDry is designed to maintain a constant vapor pressure in its chamber regardless of ambient conditions or oven temperature, thereby creating a scientifically "dry" condition. It does this by flowing controlled dry air into the chamber during the heating process. This design is similar to one proposed for milk powder (de Knegt & van den Brink 1998). A product tested in the TrueDry is then declared dry when its weight has stopped changing while exposed to a constant vapor pressure at the recommended temperature, thus making its weight the true dry weight.

The controlled vapor pressure technique of the TrueDry makes it possible to improve reproducibility of loss-on-drying moisture contents since it eliminates the impact of varying ambient humidities. In addition, by defining "dry" as a sample that has been equilibrated to a controlled vapor pressure, the TrueDry creates a moisture content standard for any product. The capability of the TrueDry to produce consistent results was tested by analyzing flour samples in the TrueDry and with a conventional oven while exposed to varying ambient relative humidities. Table 6 indicates that while the moisture content values determined by the TrueDry CV-9 were consistent across all humidities (reproducibility of 0.01%), the moisture content values determined using the oven decreased with increasing humidity (reproducibility of 0.32%). A decrease in the moisture contents from the oven at elevated humidities would be expected since the vapor pressure in the oven will increase as the ambient humidity increases (assuming no temperature change), causing the sample's vapor pressure to equal the oven vapor pressure earlier and less water to evaporate.

Table 6 Moisture content of flour as determined at 4 different ambient humidities in the TrueDry and a conventional oven.

Ambient Relative Humidity	Conventional Oven	TrueDry CV-9
30	11.97a	11.79a
40	11.79b	11.79a
60	11.44c	11.81a
70	11.26d	11.80a

Conclusion

The TrueDry CV-9 improves on all currently used loss-on-drying moisture methods including a moisture balance and a conventional oven. It makes possible the simultaneous analysis of multiple samples using accepted reference methods and its per sample analysis time is less than other loss-on drying methods. It also controls the vapor pressure inside the oven during the analysis, thereby eliminating the impact of varying humidity, increasing reproducibility, and establishing a "dry" standard.

Literature Cited

de Knegt, R. J., and H. van den Brink, 1998 Improvement of the Drying Oven Method for the Determination of the Moisture Content of Milk Powder. International Dairy Journal 8: 733-738.

Reh, C., S. N. Bhat, and S. Berrut, 2004 Determination of water content in powdered milk. Food Chemistry 86: 457-464.