

MBSERIES

Moisture Analyzers Guide

A Guide to Moisture Content Analysis



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Introduction to Moisture Content and Overview of Measuring Techniques

Definition of Moisture Content

Water is essential to life; it plays a critical role in the physical and chemical functions of our bodies, the food we eat, and the materials that surround us. In many industries it is important—if not critical—to measure the water content of substances in order to assess quality, adjust manufacturing processes, and ensure that products meet regulations and guidelines. The amount of available water dictates the shelf life and stability of many systems; for example, the presence of water in food greatly impacts its susceptibility to chemical, enzymatic, and microbial activity.

Water content is also important for the processing and handling of:

- Cosmetics
- Pharmaceuticals
- Food
- Personal care products
- Pulp and paper products
- Specialty chemicals

Measuring the amount of water contained in certain materials can be very difficult due to the complexity of the water molecule and its strong intermolecular bonding capabilities. In most cases, measurement of water is better defined as the measurement of moisture content, defined as the mass of water per unit mass of dry material.

The MB Series of moisture analyzers (or moisture balances) from OHAUS measure moisture thermogravimetrically. Thermogravimetric moisture analysis defines moisture as the loss of mass observed when the sample is heated and is based, in theory, on the vaporization of water during the drying process; this measurement does not distinguish weight loss of water from loss of volatile components or sample decomposition. For this reason, moisture content as measured by thermogravimetric techniques includes all substances which vaporize when heating a sample and are measured as weight loss during the heating process. Therefore, we use the term "moisture content" rather than "water content" when using a thermogravimetric device.

This is especially important to note when testing materials with components having a higher vapor pressure than water. For example, high volatility components contained within the sample such as alcohols will evaporate along with the water.

What Is Thermogravimetric Moisture Analysis?

Moisture content influences the weight, density, viscosity, refractive index, and electrical conductivity of a material. Methods for testing moisture content tend to exploit one or more of these physical or chemical properties. Direct measurements address the presence of water itself, either through its removal or through chemical interaction. The use of a thermogravimetric moisture analyzer is a way to directly measure the moisture content of a sample by using the loss on drying (LOD) technique. LOD measures the weight of a sample before and after a drying procedure and uses the weight delta to determine the percentage of moisture as the weight removed by the drying process in comparison to the initial weight of the sample.

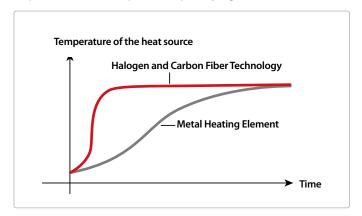
Typically this process is done in a drying oven with a balance to determine the initial and final weight of the sample and using a simple mathematical calculation to determine the moisture content ([initial weight – end weight]/initial weight). This process typically takes several hours to complete and is vulnerable to user error. A moisture analyzer works on the same principle, but is an automated system that employs a microprocessor-controlled heating element and a precision balance all in one device; by using such a device, the moisture content of a sample can be measured in minutes rather than hours.

Carbon-Fiber, Halogen & Metal Heating

Thermogravimetric moisture analyzers efficiently dry a sample by transferring energy by both radiation (the transmission of electromagnetic energy through a medium—in this case, the sample) and convection (heat transfer by air motion). In comparison, a conventional drying oven uses mostly convection to dry a sample. OHAUS MB Series Moisture Analyzer models are equipped with Carbon-Fiber, halogen & metal heating elements that all radiate energy in the infrared spectrum.

Infrared (IR) radiation is part of the electromagnetic spectrum, falling between microwave energy and visible light. Infrared waves include thermal radiation and have the wavelength frequency range from 0.75 micrometers (long wavelength limit of visible red light) to 1.5 micrometers (borders on microwaves). Infrared energy is not visible to the human eye. The red light often associated with infrared heating is actually reflected red light from the visible spectrum.

Some moisture analyzers utilize a metal heating element, which is simply a low-resistance piece of metal which converts electricity into heat. Such heaters are ideal for an environment (such as food processing) where the presence of glass components are prohibited due to regulatory or safety concerns. Metal heaters are not ideal as they have a very large thermal mass and take significantly longer to heat up than halogen heaters, and are thereby harder to control and do not provide optimum repeatability in a moisture analyzer. Halogen radiators have a tungsten heating element contained in a compact glass tube filled with halogen gas to preserve the tungsten element. The halogen radiator emits infrared radiation in the short wavelength range of 0.75–1.5 micrometers. The compact nature of the halogen radiator improves the heating/cooling response time, shortening the time for the heating unit to reach full heating power and ultimately shortening time requirements to complete sample drying; it also allows finer control during the heating process.



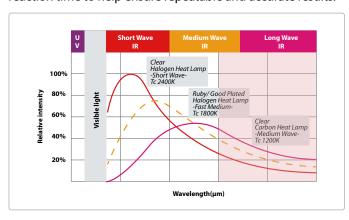
Fast heating to minimize testing time.

Infrared halogen heating technology begins drying each sample in seconds and performs up to 40% faster than traditional infrared methods, for higher throughput and a more efficient work day.

The carbon fiber (CF) heater is constructed using individual carbon fiber strands contained in a glass tube. There are similarities to a halogen heater, but CF provides several significant benefits over the halogen source.

CF heating has a higher thermal efficiency than halogen heating. The carbon fiber element generates heat using mostly medium and long wave infrared energy, which is electromagnetic energy further away from the visible spectrum. This enables the input energy to the carbon fiber heating element (voltage and current) to be converted into more heat and less visible light. CF heating also emits little to no UV radiation and no harmful gases.

Carbon fiber heating lamps have a longer service life and are more energy-efficient. Carbon fiber heating has inherited all the advantages of halogen heating, but with a much more optimized design. This results in higher thermal efficiency and fast reaction time to help ensure repeatable and accurate results.



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2. Installation and Initial Setup

It is imperative that the moisture analyzer unit is correctly installed in order to ensure high repeatability and the best possible results. Because environmental changes can adversely affect the result of a moisture measurement, the moisture analyzer should typically be placed in a location where environmental factors (temperature, humidity, vibration, etc.) are as stable as possible. It is not only important to ensure that the temperature is stable during a measurement, but also that the moisture analyzer is located in an environment similar to that when the device was calibrated. If the operating conditions change, the device must be recalibrated using a temperature adjustment kit to obtain the best possible results. Refer to your operating manual for instructions on how to perform a temperature adjustment and a 50g calibration weight on your MB Series moisture analyzer.

In the next section, we'll explore the factors that should be considered when placing your moisture analyzer.

Selecting a Location

Physical Location

As with any balance, a moisture analyzer should be placed on a solid table or workbench that is free of vibration. The surface should be stable enough that no vibrations are registered as a change in weight when it is pushed on, or when walking around the area. Please note that the unit contains a fan that circulates ambient air through a chamber within the device to help ensure a constant temperature at the weighing cell when heating. To ensure that this fan functions properly, the moisture analyzer should be placed in an open area and not directly against a wall.

For extremely sensitive samples or for cases where sensitivity in reading is critical, consider placing the analyzer in an environmental chamber where temperature and humidity are tightly controlled.

Temperature

Changing temperatures and cold-start conditions (the starting state of the moisture analyzer after it has not been used for a period of time) may adversely affect your results. Ideally, an operating temperature of 20°C should be maintained. Once the device is installed, the temperature should remain constant and the device should not be placed in close proximity to other objects (including windows) that emit heat. The operating temperature should never exceed the limits specified in the operating manual or data sheet.

Humidity

It is also important to avoid rapid changes in humidity, which can affect the weighing cell and cause it to drift, potentially leading to an inaccurate measurement result. The relative humidity should be approximately 50% and the ambient relative humidity should never exceed the limits specified in the operating manual or data sheet. Once the device is installed, the ambient relative humidity should remain constant.

Air Currents

As with any balance, the moisture analyzer should be placed in a location free of air currents. This includes drafts caused by open windows or HVAC systems.

Initial Setup

Turning On

Once the moisture analyzer has been set up for use, it is best practice to keep the device plugged in. While the display may be turned off to conserve power, the weighing cell should be powered constantly to ensure that it is always ready for measurement. When the analyzer has been plugged in for the first time, allow it to warm up for 3 hours before initial use.

Leveling

It is important to level the moisture analyzer to ensure best weighing accuracy. To level the analyzer, rotate the leveling feet until the level bubble is in the correct position. Consult your device's operating manual for additional information about how to properly level your moisture analyzer.

Adjusting

The OHAUS MB Series of moisture analyzers arrive adjusted and ready to use in optimum environments. It is recommended to readjust the analyzer by performing a weight and temperature adjustment before it is first used and if any changes are made to the ambient conditions (including moving it to a new location) in order to compensate for changes that may affect how much energy is delivered to the sample surface. It is also recommended to periodically adjust the moisture analyzer based on use to compensate for any buildup of substances on interior surfaces (see the cleaning and maintenance section of this document for additional details).

3. Moisture Determination Using an MB Series Moisture Analyzer



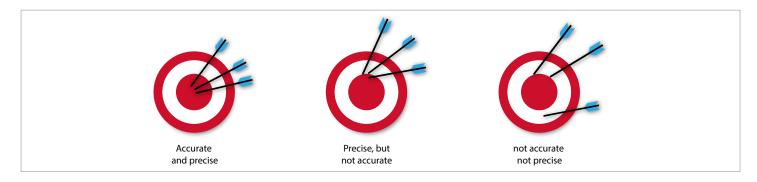
Please note: Although this section refers specifically to the OHAUS MB120 moisture analyzer (and some features may not be available on other models), the principles described here can be applied to any thermogravimetric moisture analyzer.

What Is a Method?

A method is a set of parameters that defines how a sample is dried. A method consists of a drying program, temperature(s), a shut-off criterion, and other parameters that define how the results are displayed (e.g., units of measure). The user can determine the best method to dry a sample to produce the required result.

Accuracy vs. Precision

Accuracy is how close a measured value is to an actual or true value. Precision is how close measured values are to each other, commonly measured by the standard deviation of a set of values. It is important to note that the goal of a moisture analyzer is to be precise—so that multiple samples measured under the same conditions produce a set of results with very little variance. The user must select a method and prepare the sample in a way to ensure accuracy.



In order to illustrate this concept, consider the example of baking cookies. If the dough is prepared in the same way and applied in homogeneous and consistent chunks on a baking pan, it is possible that the oven may undercook (if the temperature is too low or baking time is too short) or burn (if the temperature is too high or baking time is too long) the dough. However, if the dough is prepared consistently and baked using the optimum temperature and time, the cookies should come out exactly the same each time. Likewise, if the user of a thermogravimetric instrument provides the correct inputs, the results should be of very low variance.

Method Parameters

Drying Temperature

The MB Series of moisture analyzers (depending on model) provides a wide range of drying temperatures. While temperatures from $40^{\circ}\text{C}-230^{\circ}\text{C}$ can be achieved in a drying application, most samples are measured in the range of $100^{\circ}\text{C}-140^{\circ}\text{C}$.

Drying Profiles

Moisture content (MC) is greatly influenced by the drying temperature used to drive off the moisture. Excessive heating may result in a high percentage MC reading due to sample decomposition or changes in chemical structure. Besides giving artificially high readings, results are very difficult to reproduce in tests when the drying profile is too harsh. Conversely, lower heating levels may preserve sample integrity but prolong the drying process, making the test unrealistic for process use.

The MB Series of moisture analyzers offers a series of drying programs that allows users to customize the sample drying profile. By customizing the drying program, moisture measurements can be optimized to enhance drying conditions and shorten runtime while minimizing sample decomposition or change in chemical structure, ultimately improving testing accuracy and reproducibility. The four basic temperature profiles—standard, fast, step, and ramp—can be customized by specifying a target temperature(s).

Standard

The standard drying profile is the most common and is sufficient for most samples. In this drying profile the target temperature is reached and sustained until the end of the measurement.

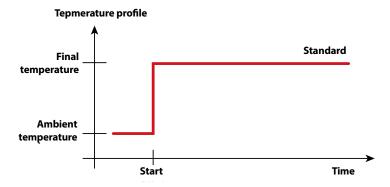


Figure 1. Standard drying profile

Fast

The fast drying profile is suitable for samples with higher moisture content, as it relies on available moisture to prevent charring of the sample. In this drying profile, the target temperature is exceeded by 40% for the first 3 minutes, then reverts to the target temperature which is sustained until the end of the measurement.

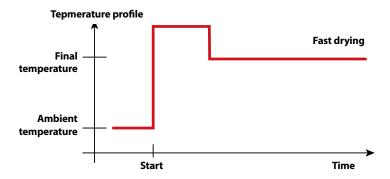


Figure 2. Fast drying profile

Step

The step drying profile allows for multiple temperatures to be sustained for defined periods of time, allowing for tighter control of the drying temperature. This profile can be useful for samples in which a lower temperature is first used to dry and measure surface moisture and a higher temperature to release and measure bound moisture. Alternatively, a higher temperature may be used first to burn off volatile solvents, then a lower temperature(s) can be used to measure water content.

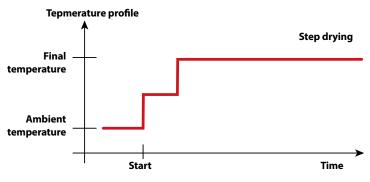


Figure 3. Step drying profile

Ramp

The gentle ramp drying profile allows the user to ramp up the temperature slowly over a period of time. This can be useful to dry a substance with a high sugar content, where a slow temperature ramp will increasingly allow bound water to be evaporated before a caramelized layer is formed, trapping bound water underneath.

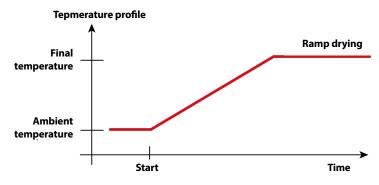


Figure 4. Ramp drying profile

Shut-Off Criteria

A shut-off criterion defines when the moisture analyzer stops heating and considers a measurement complete. This can be done either manually or via several built-in options to ensure accurate, reproducible results. When and how the instrument terminates a heating profile can be programmed according to runtime or according to steady-state weight conditions. The available switch-off criteria for the MB Series are defined as:

- 1. Manual The user defines end of run and shuts off instrument manually.
- 2. **Timed** Instrument shuts off automatically at a preset time during analysis (e.g., 10 minutes).
- 3. **Auto** The instrument automatically shuts off based on weight loss per unit time. For the built-in automatic shutoff criteria, the end of a measurement is reached when the overall change in weight observed is less than 1 milligram per period of time. The available preprogrammed choices are:

A10 = < 1 mg. weight loss in 10 seconds (quick-drying samples/fast measurements or reference)

A30 = < 1 mg. weight loss in 30 seconds (quick-drying samples/fast measurements)

A60 = < 1 mg. weight loss in 60 seconds (most sample types)

A90 = < 1 mg. weight loss in 90 seconds (slow-drying samples)

A150 = < 1 mg. weight loss in 150 seconds (extreme slow-drying samples)

4. **AFREE** – Auto-free switch-off; allows the user to define shut-off criteria according to weight loss per unit time or weight loss percentage per unit time.

Sample Preparation

The amount of sample chosen can impact the moisture reading; it is crucial that an appropriate amount be used to obtain a meaningful reading. Typically, a sample size of 5–10 grams is recommended; the minimum allowed weight is 0.5 grams. Small sample sizes should only be used when material is difficult to obtain or expensive.

It is important that the sample be evenly distributed on the sample pan and that the physical state of the material allows the absorption of IR and dissipation of moisture. Although some samples can be directly added to the sample pan, at times the sample requires some alteration to its physical state (e.g., pulverizing or grinding) in order to enhance the drying process. It is important that the sample does not gain or lose moisture during this process. With a little care and planning, change in moisture content during sample preparation can easily be avoided.

It is best to test the sample immediately after preparation. Storing the sample in a hermetically sealed container will also help prevent moisture migration before and between analyses.

Sample Quantity and Distribution

Sample size is dictated by distribution needs in the sampling pan and moisture content of the sample. Depending on the moisture content, optimization of drying conditions and reproducibility may be influenced by the amount of sample being evaluated. An example of this can be found in the Appendix for the evaluation of molasses (Case Study #2).

Distribution of the sample in the holding pan will also affect moisture reading and reproducibility. The sample should ideally be distributed in a thin, even layer across the surface of the pan. The sample may burn where it is spread too thin, and may retain moisture where it is piled too thick—and both will affect the accuracy and reproducibility of the final moisture reading.

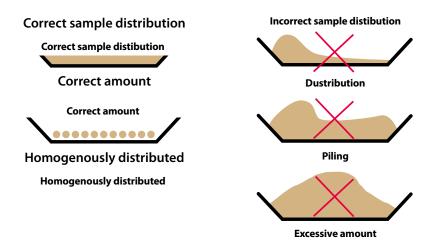


Figure 5. Examples of good and bad sample distribution in drying pan

Use of Glass Fiber Pads

Glass fiber pads are useful media for applying liquid samples, providing an inert, porous support. Dispersing the liquid into the fiber pad decreases sample surface tension and increases overall surface area, shortening analysis time. For very sensitive measurements or research purposes, the pads can be retained in a desiccator to avoid affecting the moisture reading. (This is not essential for routine analysis.)

Glass pads can also be used for material that is sensitive to heat or has skin-forming properties during the drying process. Using the pad as a top layer or sandwiching the material between two pads protects the sample from IR radiation. The sample can be dried by conventional heat rather than directly from IR radiation.

4. Getting Started: Sample Consideration and Method Development

The OHAUS MB Series of moisture analyzers can be used to analyze a wide variety of materials. The MB Series provides a wide range of options (e.g., temperature, drying programs, shut-off criteria) you can use to build the best method to analyze a given substance. However, creating an optimal method can be challenging; therefore, we recommend taking sufficient time to experiment with method development.

When designing and optimizing the testing protocol, it is important to understand your material. Consider the following three factors before getting started:

Approximation of moisture content	 Based on information in the literature Calculated from starting ingredients Estimated by comparison to related materials
Sensitivity to heat	 Presence of volatile constituents besides water Presence of flammable constituents Combustion properties of sample
Physical state of sample	 Surface properties, enhanced IR absorption Even sample distribution to heat Enhanced thermal conductivity Ability to dissipate heat and moisture from surface

The most common way to develop a method for a particular substance is to obtain a reference value, then build a method to reproduce the target value with the shortest possible drying time. To obtain a reference value, use the conventional LOD procedure and an analyzer and drying oven. Alternatively, you may use a desiccator, Karl Fisher titrator, or other methods.

Once you have obtained a reference value, you can begin to develop an appropriate method on a moisture analyzer. We recommend preparing a sample and drying it using a best-guess temperature, then analyzing the resulting drying curve. For example:

- Standard drying @ 120°C
- Shut-off criterion A60

Understanding the drying curve generated during sample drying will help to define appropriate test conditions for your sample. Asymptotic drying curves are indicative of samples which reach a constant moisture value during the drying process. Optimizing the temperature profile and shut-off criterion is generally simple, resulting in repeatable data.

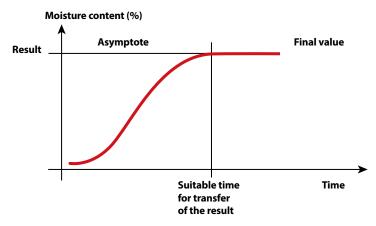


Figure 6. Representation of asymptotic curve, steady-state weight

In the example shown here (Figure 6), the sample has reached a steady state and achieved the automatic shut-off criterion. If the final value achieved is lower than the expected value, this indicates that the temperature is not high enough to release all the bound moisture; increase the temperature and repeat the measurement until an optimal temperature is achieved. Conversely, if the resulting value is too high, this could indicate that the sample has changed composition (burned); decrease the temperature and repeat the measurement. It is important to visually inspect the sample to determine if burning or charring has taken place.

In other cases, samples may never reach a constant weight throughout the drying process, resulting in a drying profile similar to that illustrated in Figure 7. This type of curve is indicative of a sample undergoing thermal decomposition or continual vaporization of volatile components. Optimization in this case may require lowering the temperature profile used for drying. Timed switch-off and consistent initial sample weight also help improve repeatability.

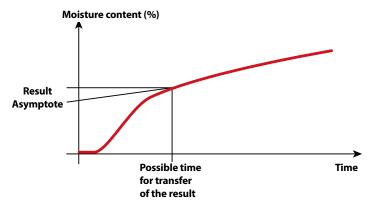


Figure 7. Representation of drying curve which does not reach constant weight

In some cases, it may be necessary to determine the moisture content only using the moisture analyzer (i.e., no reference value is given or can be measured). In these cases, method development should be done as defined above, with the goal of achieving the fastest drying time and most repeatable results. Several measurements should be made to identify and validate a sound method. Please note that the result may not indicate the actual moisture content, but instead serve an analog or proxy to the moisture value (i.e., while the final value is not the full and true moisture content, deviations in the actual moisture content of a sample can be detected).

Product Tip: SmartGuide™

Configuring a moisture analyzer to accurately measure a new sample can be a daunting, frustrating, and time consuming task. OHAUS has simplified the process and eliminated the steep learning curve associated with method development with SmartGuide™; an innovative and revolutionary new software feature now available on the MB120 Moisture Analyzer. SmartGuide™ analyzes and determines a heating method with limited user input. Manual method development is a thing of the past; allowing you to start measuring samples quickly and efficiently in a fraction of the time.

GLP/ISO Compliance of Your Moisture Analyzer

To maintain competitiveness, quality control is critical for most companies—and a moisture analyzer is an important part of your quality control system. The OHAUS MB120 is designed to be either easily integrated into a general quality system such as GLP/GMP, or implemented into your organization as a standard such as ISO9000. GLP requirements and ISO standards require traceable documentation of all adjustments/calibration procedures and tests that are performed on a measuring instrument. The weighing component of any OHAUS moisture analyzer can be adjusted following the written procedure in the manual and using a certified weight.

The heating or temperature measurement component of the analyzer can also be adjusted using a procedure unique for moisture analyzers. The heating element can be adjusted following the procedure outlined in the instruction manual. This procedure can be done using a calibrated thermometer to ensure that the moisture content is determined under identical conditions regardless of the location.

These adjustments can be documented utilizing the product software and an attached printer.

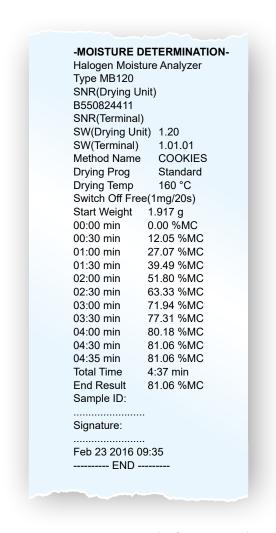


Figure 8. A printout example after test completion



5. Cleaning and Maintenance

Keeping your moisture analyzer clean and calibrated will help to ensure that the unit operates optimally and results are consistent as possible. It is important to check that all surfaces of the heating chamber are free of dust and debris from previous measurements. Keep the area around the sample pan clean by removing the wind ring and emptying it when needed. A mild cleaning agent is recommended to wipe down all surfaces. The MB120 analyzers allow the protective glass ring below the heating element to be removed for easy cleaning (no tools needed), so that a consistent amount of energy is transferred uniformly to the sample.

Troubleshooting

The table below provides possible solutions for issues that may arise while using the MB120.

Problem	Possible Solutions
Sample burning during analysis	 Lower temperature Try step or ramp program to control temperature Shorten runtime/exposure to heat Protect sample by covering with glass fiber pad
Analysis time takes too long	 Increase drying temperature Use rapid or step program Decrease sample size Increase surface area by using glass fiber pad
Results are not accurate	 Increase sample weight (low % MC) Decrease sample weight (high % MC) Try automatic shut-off criterion Review drying profile for constant weight Ensure homogeneous sampling
Results are not reproducible	 Ensure sample preparation is consistent and does not influence sample Try automatic shut-off criterion Assess drying profile, sample burning or not drying sufficiently
Sample loses weight during weighing	 Allow time for instrument to cool between measurements Add sample to drying pan outside of the drying unit
Sample does not reach constant weight during drying	Use timed shut-off criterionLower drying temperature
Sample melts during heating	Use glass fiber pad
Sample has low moisture content	Increase sample size
Sample contains flammable material	Follow safety directions in instruction manual

Additional Tips for Using a Moisture Analyzer

OHAUS MB Series moisture analyzers are excellent tools for routine analysis in the process environment. The instruments are rugged, simple to operate, and provide rapid, reliable data. The devices may also be used in the research laboratory as an investigative tool for basic scientific studies. Certain precautions should be taken to ensure that measurements made with the devices are reproducible and as accurate as possible. The following are some suggestions for controlling operating variables for moisture analyzers:

- Allow adequate cooling between sample runs. If the machine is still very hot from the previous run, it may affect the initial weight reading of the sample and cause inaccuracy in final percentage MC.
- Keep starting weight for test material consistent. Since the final reading (weight) is a factor of the drying process, consistent starting
 weight will minimize differences due to physical parameters of sample introduction and drying profile.
- When possible, try to control the laboratory environment. For extremely sensitive samples or for cases where sensitivity in reading is critical, consider working in an environmental chamber where temperature and humidity are tightly controlled. In general, it is best to set up the instrument in an area free of windows to minimize exposure to temperature extremes, drafts, and other environmental conditions.

Case Study # 1: Sample Homogeneity

The following case study illustrates the need for sample homogeneity. Here, a sample of yellow cake was evaluated for % moisture. Portions of the cake were indiscriminately broken off the cake, then further broken apart into small crumbs and distributed on the weighing pan. The samples were dried using a step profile (140°C 3 min., 110°C until dry). Completion of run was determined by automatic switch-off (A30).

Results from the first three analyses:

% Moisture content = 35.03, 36.05, 32.95 Mean value = 34.68 Standard deviation = 1.58

The test results showed considerable scatter, with a very high standard deviation. Evaluation of the drying curve and end product after drying showed sufficient drying without sample decomposition. This suggested that the temperature profile was appropriate for the cake and that the problem may be due to inconsistency within the sample itself. A new sample was prepared by taking a representative cross section of the cake, breaking it into a fine crumb, and mixing well. A second set of analyses was conducted on the more homogenous blend of the cake sample.

The following are results from the second set of analyses using a homogeneous cake sample:

% Moisture content = 33.60, 33.83, 33.42 Mean value = 33.62 Standard deviation = 0.2

The data obtained from these samples are shown to be more repeatable with standard deviation within the working range of the instrument.

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OHAUS Corporation

Headquartered in Parsippany, NJ, OHAUS Corporation manufactures an extensive line of weighing scales, lab equipment and lab instruments that meet the weighing, sample processing and measurement needs of multiple industries. We are a global leader in the laboratory, industrial and education markets, as well as a host of specialty markets, including the food preparation, pharmacy and jewelry industries. An ISO 9001:2015 manufacturer, OHAUS lab balances, industrial scales, lab equipment and lab instruments are precise, reliable and affordable, and backed by industry-leading customer support.

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The management system governing the manufacture of this product is ISO 9001:2015 certified.

