

moisture analyzer

Cook Book



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1. Introduction to Moisture Content and Overview of Measuring Techniques

1.1 Definition of Moisture Content

We can all appreciate the importance of water. Water is essential to life itself, playing a critical role in the physical and chemical functions of our bodies, the food we eat and the materials that surround us. 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.

Moisture content is defined as *the mass of water per unit mass of dry material.*

The MB45 Moisture Analyzer from Ohaus measures 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 those substances which vaporize on heating a sample, that are measured as weight loss during the heating process.*

1.2 Who Measures Moisture and Why

Understanding the water content of material is a common interest and concern to many diverse industries. Moisture content is important for the processing and handling of cosmetics, pharmaceuticals, food, personal care products, pulp and paper products and specialty chemicals, to name just a few. The amount of available water will also dictate 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.

Here are just some industrial examples, supporting the need to monitor moisture content:

- **Microbial stability**
- **Shelf life stability to enzymatic reaction**
- **Functionality of ingredients and final products**
- **Processing functionality**
- **Packaging requirements**
- **Textural properties**
- **Flow properties/viscosity**
- **Concentration**
- **Standard of identity**

1.3 Methods for Measuring Moisture Content

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. Indirect methods monitor the presence of water by measuring other attributes that are affected by its presence. The method that is most appropriate for your material will depend on a variety of factors including, sample type and size, measurement speed and ease, and testing environment.

Methods used for moisture measurement fall into one of the following four categories: **thermogravimetric**, **chemical**, **spectroscopic**, and **other**, which includes a handful of techniques that fall outside of these descriptors. Each category exploits a different trait that water exhibits on the testing material, offering some advantages and limitations. These categories are outlined on the following pages.

1.3.1. Thermogravimetric Method

Principle of method:

Moisture content is defined as the weight loss of mass that occurs as the material is heated. The sample weight is taken prior to heating and again after reaching a steady-state mass after drying. Various methods can be used for drying as outlined in Section 1.4.

Advantages of method:

Testing is simple, and generally does not require a high capital cost. Depending on the drying technology employed, analysis time can be very short. Methods are well established and recognized throughout the industry for moisture determination.

Limitation of method: Thermogravimetric methods do not distinguish weight loss due to vaporization of water from other volatile compounds that may be driven off from the heating process. Oils, alcohol, organic solvents and other volatile constituents will be registered as % moisture using thermogravimetric analysis. If drying temperature is too high, it may result in sample decomposition.

Application of method: Thermogravimetric analysis can be applied to most systems with a moisture content of $>0.1\%$. Consideration should be made to the composition of the material, including presence of other volatile compounds and potentially flammable components.

1.3.2. Chemical Method

Principle of method:

Water is chemically converted into a reaction product that can be quantitatively measured. Karl Fisher Titration is the most recognized chemical method for moisture analysis and is considered the industry standard for many systems.

Advantages of method:

Chemical methods give a high degree of accuracy and can be applied to materials with very low moisture content and hygroscopic materials.

Limitations of method:

Wet chemistry, requiring the handling and disposal of solvents and potentially hazardous materials. Titration techniques can be subjective, often requiring trained staff to run the analysis. Analysis must be conducted in a laboratory setting.

Application of method:

Used for samples with low moisture content of $>0.1\%$. Originally developed for moisture analysis of non-aqueous liquids but can also be applied to solids providing they can be solubilized into the working solutions.

1.3.3. Spectroscopic Method

Principle of method:

Spectroscopic measurement of moisture is based on the absorption and scatter of radiant energy when applied to a system containing water.

Advantages of method:

Rapid test method allows for real time analysis, may be applied to in line processing.

Limitations of method:

Requires purchase of capital. Other factors besides water may influence readings such as density, temperature and other properties, which are indirectly affected by their moisture content. IR spectroscopy has low penetration depth and can only be used to describe surface moisture.

Application of method:

Useful for line process evaluation of moisture content. NMR spectroscopy is a useful research tool for studying the presence of moisture and moisture migration.

1.3.4. Other Methods for Moisture Measurement

Other methods for moisture analysis includes an assortment of tests for monitoring moisture content that fall outside the three previously described categories. Generally, these methods are indirect moisture measurements, determining moisture content through a secondary parameter. Some test methods included in this category are electrical conductivity (dielectric constant), refractometry and density determination. Generally, these methods have very specific applications, for example, the dielectric probe used by a painter to approximate the moisture in wallboard prior to painting.

1.4 Summary of Methods Used for Moisture Measurement

Table 1 summarizes moisture methodology, grouped according to Thermogravimetric, Chemical, Spectroscopic and Other-.

TABLE 1. METHODS OF MOISTURE MEASUREMENT.

METHOD	PRINCIPLE OF ANALYSIS	MOISTURE RANGE	ACCURACY	WATER SELECTIVITY
THERMOGRAVIMETRIC				
Oven drying	Heating by convection , difference in mass before and after drying.	0.5-100%	0.1-0.5%	no
Infrared drying	Heating by absorption of IR radiation, mass loss continuously measured during drying.	0.5-99%	0.1-0.5%	no
Halogen drying	Heating by IR radiation with halogen radiator, continuous determination of mass during drying.	0.5-99%	0.1-0.5%	no
Microwave drying	Heating by absorption of microwave, mass determination before and after drying.	2-99%	0.1-0.5%	no
CHEMICAL				
Karl Fisher titration	Chemical conversion of water to measurable reaction product.	0-100%	ppm.	yes
Ca Carbide method	Chemical conversion of water to measurable reaction product.	1-100%	0.1-0.5%	yes
Distillation	Water measured as condensed distillate from organic solvent.	3-70%	1%	yes
SPECTROSCOPIC				
Infrared Spectroscopy	Water measured by selective absorption/reflection in the infrared range.	1-80%	0.3-1%	yes
Microwave Spectroscopy	Water measured by selective absorption/reflection in the microwave range.	2-70%	0.1-0.5%	yes
NMR Spectroscopy	Measurement of nuclear magnetic resonance.	0-15%	0.10%	yes
OTHERS METHODS				
Conductivity	Measurement of electrical conductivity.	>3%	0.5-1%	no
Dielectric Capacity	Measures the dielectric constant.			no
Refractometry	Measurement of the refractive index.	0-50%	0.5-1%	no
Sound Ranging	Acoustic, radio frequency penetration/absorption in particles.	40-100%	0.1-2.0%	no

1.5 Statistical Requirements- for “Robust” Test Method

Robust test methods are ideal for the processing environment, when reliable data must be generated in a reasonable time and with a high degree of accuracy. Ideally, the testing protocol should be simple to perform and adaptable to many conditions. The MB45 moisture analyzer meets these requirements, making it a useful tool for moisture measurement in a variety of conditions and for a variety of samples. Yet, even robust test procedures have an underlying need for scientific rigor and sound statistical backing. Statistical requirements for a robust test method is best defined as repeatability and reproducibility. Figure 1 below illustrates a data distribution curve, showing mean (center of distribution) and standard deviation (concentration of data about the mean).

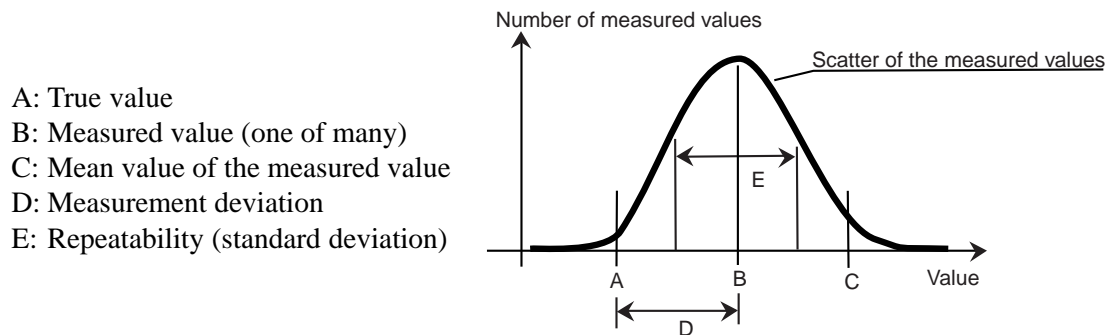


Figure 1. Data Distribution Curve.

Repeatability describes the degree of scatter from repeated data collection of the same sample under the same environmental conditions. This is defined according to the standard deviation of the collected data. As with other statistical analysis, the lower the number, the more reliable the test.

Reproducibility is defined as the degree of scatter (also measured as the standard deviation) from the same type of samples tested at different locations and different environmental conditions. Again, lower values indicate better reproducibility.

2. Infrared Drying Technology

2.1 Infrared Radiation

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 1000 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, on which it borders. Table 2 gives the approximate wavelengths, frequencies and energies for selected regions of the electromagnetic spectrum.

TABLE 2. SPECTRUM OF ELECTROMAGNETIC RADIATION.

REGION	WAVELENGTH (ANGSTROMS)	WAVELENGTH (CENTIMETERS)	FREQUENCY (Hz)	ENERGY (eV)
Radio	$>10^9$	>10	$<3 \times 10^9$	$<10^{-5}$
Microwave	10^9-10^6	10-0.01	$3 \times 10^9-3 \times 10^{12}$	$10^{-5}-0.01$
Infrared	10 ⁶ -7000	0.01-7x10 ⁻⁵	$3 \times 10^{12}-4.3 \times 10^{14}$	0.01-2
Visible	7000-4000	$7 \times 10^{-5}-4 \times 10^{-5}$	$4.3 \times 10^{14}-7.5 \times 10^{14}$	2-3
Ultraviolet	4000-10	$4 \times 10^{-5}-10^{-7}$	$7.5 \times 10^{14}-3 \times 10^{17}$	3-10 ³
X-Rays	10-0.1	$10^{-7}-10^{-9}$	$3 \times 10^{17}-3 \times 10^{19}$	10 ³ -10 ⁵
Gamma Rays	<0.1	$<10^{-9}$	$>3 \times 10^{19}$	$>10^5$

The notation “eV” stands for electron-volts, a common unit of energy measure in atomic physics. A graphical representation of the electromagnetic spectrum is shown in Figure 2.

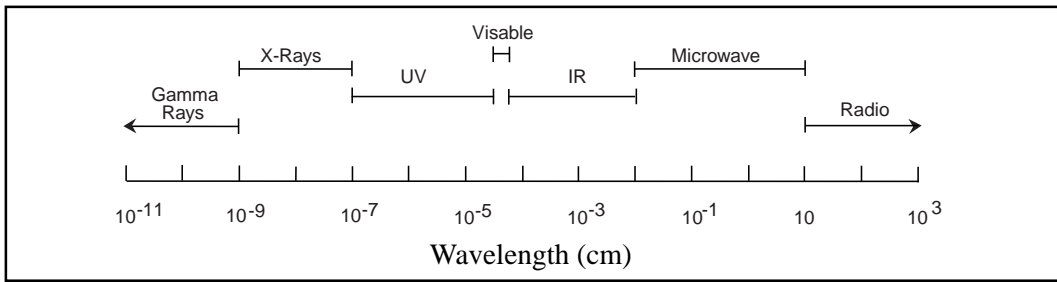


Figure 2. The Electromagnetic Spectrum, including Infrared (IR) Range.

Travelling at the speed of light, IR radiation does not convert to heat until it strikes and is absorbed by a material. Upon absorption, the rays cause molecular vibration at the surface of the material. The friction created by this intermolecular vibration is what generates heat, which is ultimately passed throughout the material by thermal conductivity. The rate of sample heating is highly dependant on the material and it's absorbent properties at the surface of the sample.

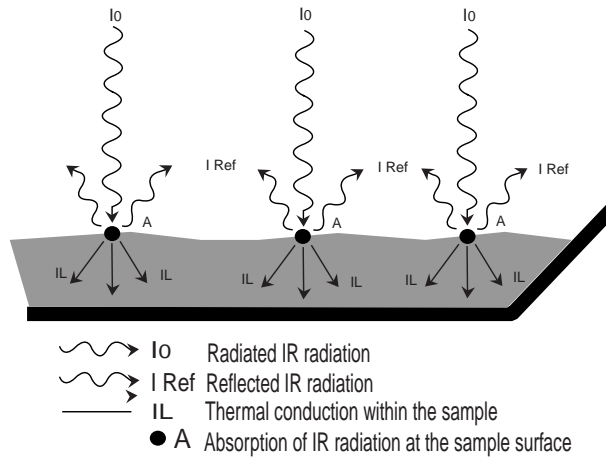


Figure 3. Infrared Radiation Striking Surface of Sample During Drying.

2.1 Halogen Radiator

The halogen radiator is based on advanced infrared heating technology. The radiator is comprised of 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 micrometer. The compact nature of the halogen radiator improves the heating/cooling response time, shortening the time to the heating unit to reach full heating power and ultimately shortening time requirements to complete sample drying.

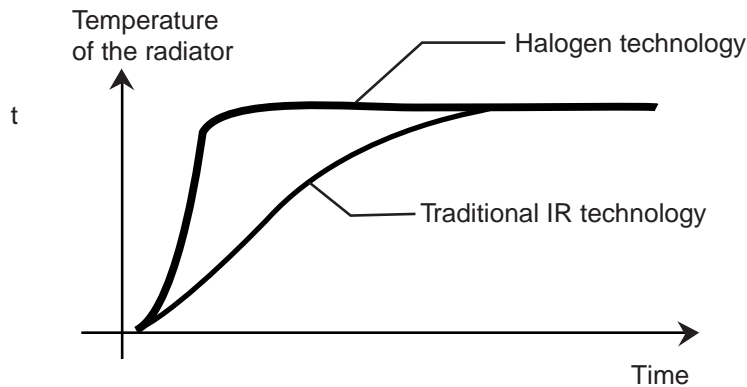


Figure 4. Comparison of Halogen Radiator and Traditional IR Radiation.

3. MB45 Moisture Analyzer

3.1 Introduction to MB45 Moisture Analyzer

The MB45 Moisture Analyzer is a Thermogravimetric moisture analyzer based on the halogen radiator technology. The instrument can be used to measure the moisture content of a wide array of materials. As with other thermogravimetric tools, moisture content is represented as the weight loss of the sample after drying. The MB45 moisture analyzer is equipped with a halogen radiator which is more responsive to the heating and cooling requirements than traditional IR heaters, thus shortening run time. To ensure even heat distribution, a gold-plated reflective shield lines the inside lid of the analyzer. The shield is gold-plated specifically for its reflective properties in the IR wavelength and its ability to dissipate latent heat. A highly sensitive precision balance is built into the unit that allows for detection of weight changes of 1.0 mg and 0.01% moisture loss. The balance continuously records and displays weight change throughout the drying process. A final reading is determined once the balance has reached a steady state weight.

Overview of the MB45 Moisture Analyzer

The following list describes some of the added features of the MB45 Moisture Analyzer, which aid in its ease of operation and usefulness to industry.

- Up to twice as fast as traditional IR Moisture Analyzers
- Fully programmable with front panel controls
- Easy to follow menus for simplified operation
- Ability to customize heating profile to accommodate unique sample types
- Results displayed as % moisture content, % solids, weight or % regain
- Built-in library can store up to 50 profiles complete with setup parameters
- Durable and chemically resistant construction
- Easy loading disposable sample pans, will hold up to 45g material
- Choice of five languages (English, French, Spanish, German, Italian) for operation
- Continuously records and displays test data during drying process
- Built-in RS232 communication interface

3.2 Moisture Determination using MB45

3.2.1. Getting started- sample consideration and method development:

The MB45 Moisture Analyzer can be used to analyze a wide variety of materials. The instrument offers four options for customizing the drying profile, which will help improve accuracy and speed of sample testing. The first step in designing a testing protocol for your sample is to consider your needs and ways to best capitalize on the strengths of the unit. The greatest strength of the MB45 is its responsive heating capability that provides accurate, reproducible moisture measurements while minimizing analysis time. Efficient/rapid heating will shorten the run time providing your sample is not highly sensitive to heat.

When designing and optimizing the testing protocol, it is important to have an understanding of your material. The following three areas are important considerations 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

3.2.2. Sampling and Sample Preparation

Representative sampling

Sampling and sample preparation will have a great influence on moisture readings and reproducibility. Sample collection may mean obtaining samples from the processing line at given time intervals or on a batch to batch basis for any given day. Whatever the case, some thought should be put into when and how you obtain your samples and how many samples are required to provide statistically-sound, representative data.

For reproducible results, it is important that the test sample be a representative, homogenous mix of the material in question. In many systems, it is common for moisture content to vary throughout the material. For example, it is common for the surface and edges to contain less moisture than interior portions. In order to get a representative sample, the material, as a whole should be homogeneously mixed and portions of this mix used for later testing. (See Appendix, Case Study #1)

Sample size reduction

Sample size and need for sample reduction can also impact the moisture reading. 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 infrared radiation and dissipation of moisture. Some samples can be added to the sample pan as is but there are times when the sample will require some alteration to its physical state to enhance the drying process. It may be necessary to pulverize, grind or somehow alter sample size and shape prior to moisture analysis. 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.

Sample size reduction can be accomplished using a mortar and pestle to crush the material or grinder for harder materials such as grains. It is important not to overdo the grinding and overheat the sample. After grinding, the sample has more exposed surface area and may absorb or lose moisture depending on the environmental conditions. For this reason, it is best to test the sample immediately after grinding. 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. Uneven distribution may result in sample burning where spread too thin and moisture retention where the sample is piled too thick. Either case will affect the accuracy and reproducibility of the final moisture reading.

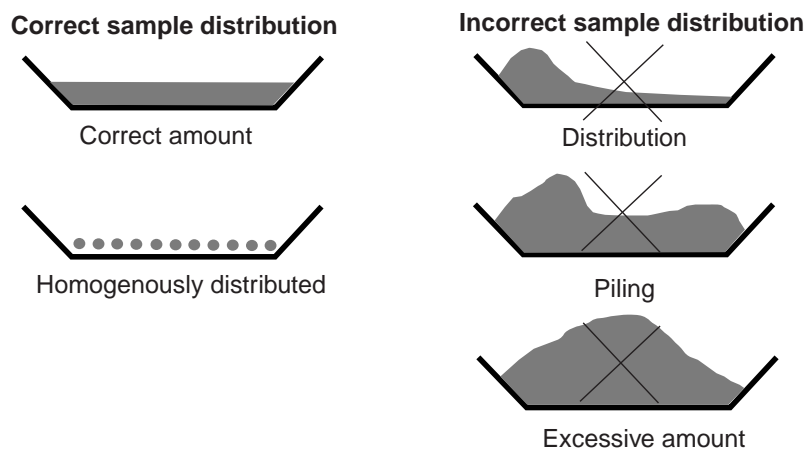


Figure 5. Examples of Good and Bad Sample Distribution in Drying Pan

Use of glass fiber pads

Glass fiber pads provide a useful medium for applying liquid samples. Dispersing the liquid into the fiber pad decreases sample surface tension and increases overall surface area which aids in shortening analysis time. The glass fiber pads are a useful medium for this purpose, providing an inert, porous support. For very sensitive measurements, or research purposes, the pads can be retained in a desiccator so as not to affect the moisture reading. This is not essential for routine analysis.

Glass pads are also 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 the IR radiation. The sample can be dried by conventional heat rather than directly from the IR radiation.

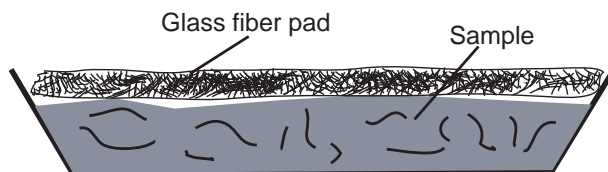


Figure 6. Protection of Sample to IR Radiation Through Use of Glass Fiber Pad.

3.2.3 Customizing the Drying Program

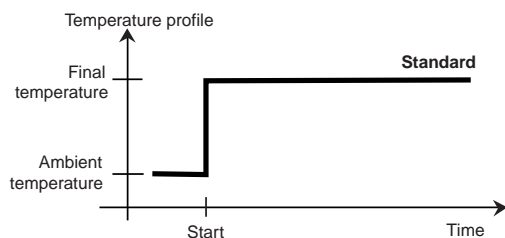
Moisture content is greatly influenced by the drying temperature used to drive off the moisture. Excessive heating may result in high % MC reading due to sample decomposition or changes in chemical structure. Besides giving artificially high readings, results are very difficult to reproduce in tests when the drying profile is too harsh. Conversely, gentle programs may preserve sample integrity but can prolong the drying process, making the test unrealistic for process use. The MB45 allows the user to customize the sample drying profile through its built-in series of drying programs. By customizing the drying program, moisture measurements can be optimized to enhance drying conditions and shorten run time while minimizing sample decomposition or change in chemical structure. Ultimately, this improves testing accuracy and reproducibility.

Choosing a drying profile

There are four basic temperature profiles available for the MB45, all of which can be customized for temperature, ranging between 50°C and 200°C. The four temperature profiles include standard, fast, step and ramp.

Temperature Profiles

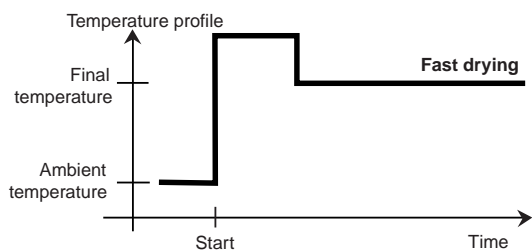
1. Standard



Guideline for Application

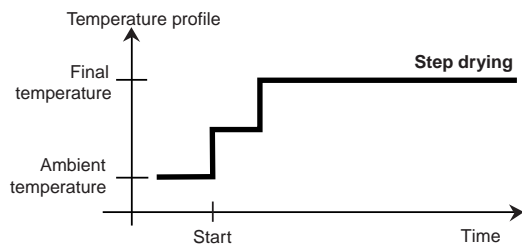
Useful for moisture measurement of most samples.
May find other programs will enhance drying/shorten run time.

2. Fast



Most suitable for samples with 5-15% moisture content.
Halogen radiator exceeds drying temperature in first minute,
Relies on available moisture to prevent sample charring at onset.
Excessive heat overrides endothermic heat of vaporization.

3. Step

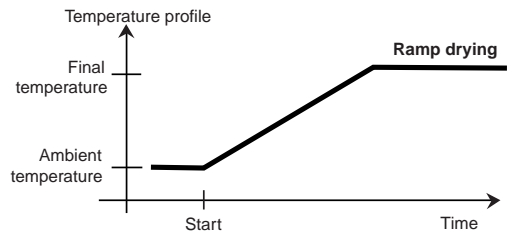


Useful for sample with moisture content >15 %.

Similar applications to fast program but step process allows for tighter control of the drying temperature.

Step process also allows for two stage drying, measurement of surface water, followed by higher temperatures and measurement of bound water.

4. Ramp



Most applicable to heat sensitive samples.

Gentle ramping of temperature protects the sample to the full power of the halogen heater.

Switch-off Criterion

Switch-off criterion for the MB45 defines when the instrument should end a run. This can be done manually but several built-in options relieve the user of this responsibility and helps to ensure accurate, reproducible results. When and how the instrument terminates a heating profile can be programmed according to run time or according to steady-state weight conditions. Switch-off criterion for the MB45 are outlined as follows.

1. **Manual** Operator defines end of run, shut off instrument manually.

2. **Timed** Instrument shuts off automatically at a preset time during analysis.

3. **Auto** Instrument automatically shuts off, based on weight loss per unit time.
A30 = < 1mg. 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)

4. **AFREE** Auto free switch-off, allows the user to define shut off criteria according to weight loss per unit time, rather than use the preset programs listed above

3.2.4. Understanding the Drying Curve

Understanding the drying curve generated during sample drying will help in defining 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 switch-off criterion is generally simple, resulting in repeatable data.

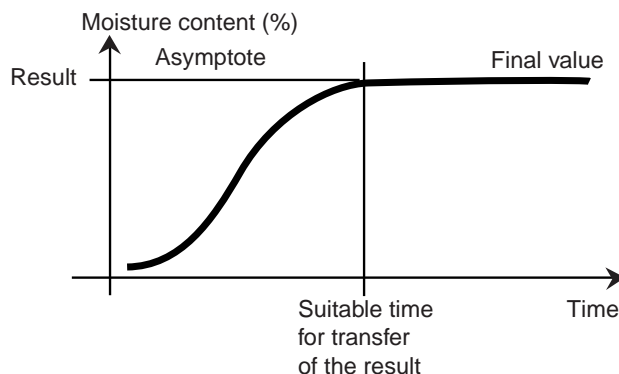


Figure 7. Representation of Asymptotic Curve, Steady-State Weight.

3.2.4. Understanding the Drying Curve (Cont.)

In other cases, samples may never reach a constant weight throughout the drying process, resulting in a drying profile similar to that illustrated below. 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 helps improve repeatability.

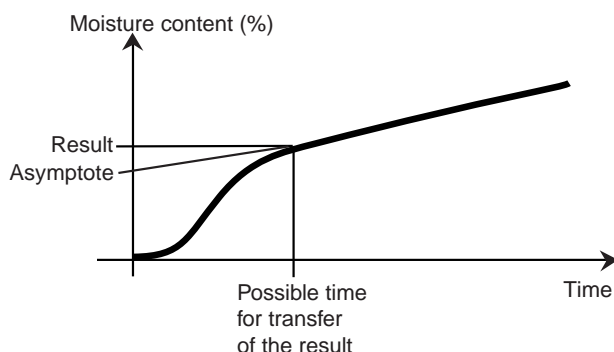


Figure 8. Representation of Drying Curve Which Does Not Reach Constant Weight.

3.3 GLP/ISO Compliance of your Moisture Analyzer

Quality management has become an important instrument for most companies in order to maintain competitiveness.

The MB45, as any analyzer, is an important part of the quality control system. The functions of the Ohaus MB45 have been designed to be easily integrated into either a general quality system such as GLP/GMP or be implemented into your organization as a standard, such as ISO 9000.

Regular calibration, cleaning and maintenance are required for all test equipment for good quality control, and all such procedures must be documented.

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 the MB45 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.

3.4 Applications Table-MB45 Moisture Analyzer

Table 3 was compiled using the MB45 Moisture Analyzer. A representative mix of samples from across the industry were evaluated for % moisture content. Sample preparation, heating programs, switch-off criterion and resulting moisture content (as % MC) are defined. This table may serve as a starting reference while defining working programs for your sample. It is best to optimize the program for your specific needs.

TABLE 3. APPLICATION EXAMPLES.

<u>Sample</u>	<u>Target Weight</u>	<u>Prep. Method</u>	<u>Heating Profile</u>	<u>Shut-off Criteria</u>	<u>Time</u>	<u>%MC</u>	<u>p-value (%)</u>
<u>Dry Food Ingredients</u>							
flour	3 gms.	as is, well mixed	fast, 130C	A/60	4 min.	13.4	0.11
corn meal	3 gms.	as is, well mixed	fast, 130C	A/60	4 min.	12.5	0.15
cocoa powder	3 gms.	as is, well mixed	standard 160C	timed	5 min.	7.4	0.12
pancake mix	3 gms.	as is, well mixed	standard, 160C	timed	5 min.	12.4	0.07
cake mix	3 gms.	as is, well mixed	standard, 140C	manual	4 min.	5.2	0.09
powdered milk	3 gms.	as is, well mixed	fast, 80C	manual	4 min.	3.7	0.2
instant coffee	3 gms.	as is, well mixed	standard, 95C	A/30	5 min.	14.5	0.06
<u>Sugar Syrups</u>							
honey	1 gm.	sandwich in pads	step 130C-5 min., 110C 3 min.	timed	8 min.	16.1	0.07
molasses	1 gm.	sandwich in pads	step 130C-5 min., 110C 3 min.	timed	8 min.	21.7	0.48
corn syrup	1 gm.	sandwich in pads	step 140C, 3 min., 110C 6 min.	A/30	9 min.	21.7	0.1
<u>Finished Baked/Fried Foods</u>							
cake	3 gms.	uniform mix of crumb	step, 140C, 3 min., 110C 4 min.	A/30	7 min.	33.6	0.2
cracker	3 gms.	grind uniform crumb	fast 80C	A/60	4 min.	3.7	0.04
sugar cookie	5 gms.	grind uniform crumb	fast 95C	timed	4 min.	5.3	0.03
potato chip	3 gms.	small pieces	standard 95C	A/30	2 min.	0.78	0.08
roasted peanuts	3 gms.	grind. 15 sec..	standard 95C	timed	5 min.	1.3	0.04
<u>Misc. Foods</u>							
carrot	3 gms.	shredded	step, 180C, 3min, 120C, 3 min.	A/30	18 min.	89.4	0.08
dehydrated vegetables	2 gms	as is	rapid, 80C	A/30	3 min.	2.4	0.01
dried herbs	1 gm.	as is	standard 110C	A/30	2 min.	9.8	0.04
snack pudding	2 gms	sandwich between pads	step, 180C, 3min, 120C, 3 min.	A/30	15 min.	71	0.1
creamy salad dressing	3 gms	spread on glass pad	step, 180C, 3min, 120C, 3 min.	manual	4 min.	34.9	0.95
lowfat salad dressing	2 gms	sandwich between pads	rapid, 130C	A/30	10 min.	72.6	0.14
<u>Animal Feed/Grains</u>							
dry dog food	5 gms.	grind., 30 sec.	fast, 80C	timed	4 min.	5.8	0.08
pelleted pet food	5 gms.	grind., 30 sec.	fast, 110C	timed	4 min.	11.3	0.13
cracked corn	5 gms.	grind., 30 sec.	fast, 110C	timed	4 min.	10.2	0.04
rye seed	5 gms.	grind., 45 sec.	fast, 110C	timed	4 min.	10.6	0.21
<u>Personal Care</u>							
liquid hand soap	1 gms.	spread thin on pad	step 180C, 3 min., 120C 1 min.	A/30	4 min.	82	0.09
bar soap	2 gms.	shaved thin into dish	standard 110C	A/30	7 min.	9.74	0.21
toothpaste	1 gms.	spread thin on pad	fast 130C	A/30	3 min.	34.7	0.03
skin cream	1 gms.	spread thin on pad	step 180C, 3 min., 120C 8 min.	A/30	11 min.	87.7	0.35
stick deodorant	2 gms.	shaved thin into dish	std. 110C	A/30	9 min.	36.7	0.4
powdered detergent	3 gms.	as is	fast, 110C	A/30	3 min.	6.2	0.22
<u>Misc.</u>							
latex paint	1 gm.	sandwich between pads	fast, 170C	A/30	5 min.	58.3	0.26
wood glue	1 gm.	spread thin on pad	standard 135C	A/30	7 min.	52.9	0.42
mortar mix	3 gms.	as is	fast, 200C	A/30	1 min.	1.73	0.04
potting soil mix	3 gms.	as is	fast, 200C	manual	5 min.	68.9	0.66

Note: No measures were taken to control the environmental conditions during storage and testing of all materials listed above. Moisture measurements of food samples are comparable to representative values in the USDA Nutrient Database for Standard Reference.

3.5. Troubleshooting

Table 4 provides possible solutions for issues that may arise while using the MB45.

TABLE 4. TROUBLESHOOTING.

Problem	Possible Solutions
Sample burning during analysis	Lower temperature Try step or ramp program to control temperature Shorten run time/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 switch-off criterion Review drying profile for constant weight
Results are not reproducible	Ensure homogeneous sampling Try automatic switch-off criterion Assess drying profile, sample burning or not drying sufficiently
Sample loses weight during weighing	Allow time for instrument to cool between runs Add sample to drying pan outside of the drying unit
Sample does not reach constant weight during drying	Use timed switch-off criterion Lower drying temperature
Sample melts during heating	Use glass fiber pad
Sample has low moisture content	Increase sample size
Sample has moisture > 15%	Use step drying profile Decrease sample size
Sample contains flammable material	Follow safety directions in instruction manual

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Appendix

1. Guidelines for Using the MB45 as a Research Tool

The MB45 is an excellent tool for routine analysis in the process environment. The instrument is rugged, simple to operate and provides rapid, reliable data. The MB45 may also be used in the research laboratory as an investigative tool for basic scientific studies. Certain precautions should be taken to enhance the reproducibility of data for use as a research tool.

As with other research studies, it is important to minimize variables that could influence the results. Some suggestions on how to control operating variables for the MB45 are listed as follows:

- 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 % MC.
- Try to 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, you may want to work 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.

2. 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 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. Results from these tests are described as follows.

Results from second set of analyses using homogeneous cake sample

% Moisture = 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.

3. Case Study # 2 - Sample Size Reduction- Molasses

The following example illustrates a case where sample quantity had to be reduced in order to obtain reproducible data. Here, molasses was evaluated for % moisture. The sample was sandwiched between glass fiber pads and evaluated by a step drying profile (130°C, 5 min., 110°C, 5 min. Timed switch-off at 10 minutes).

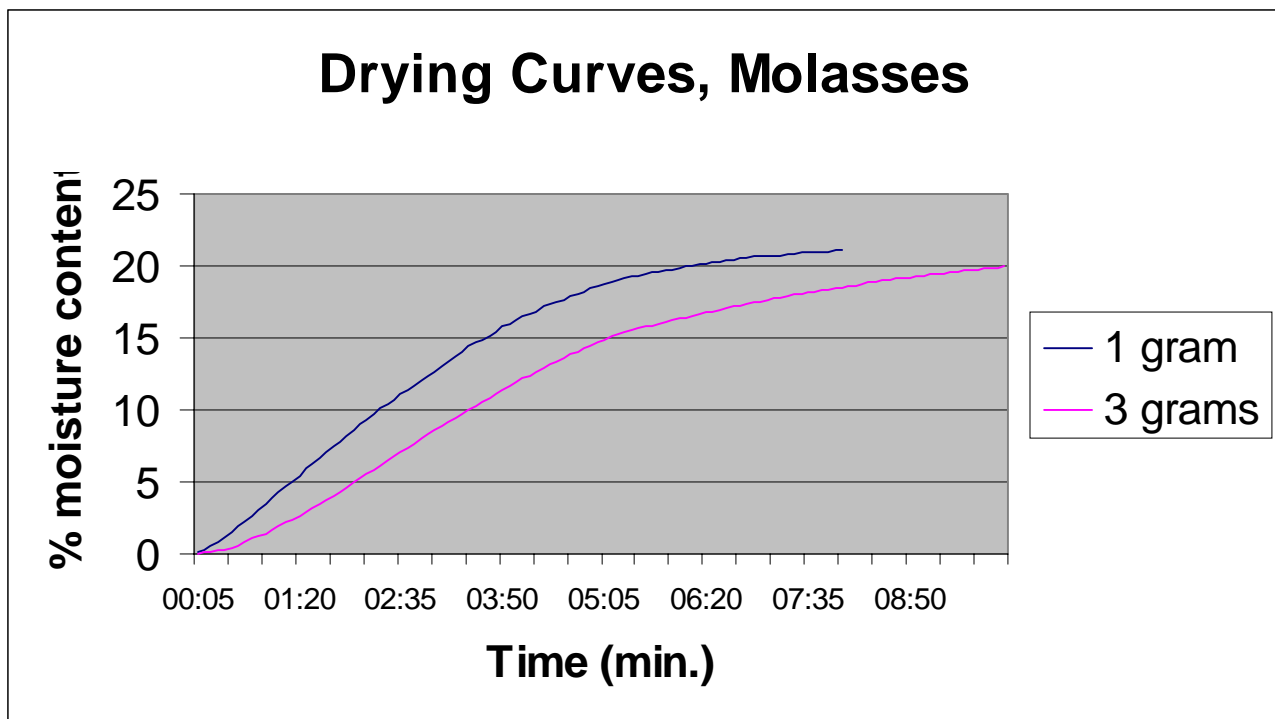
The first set of analyses was conducted using 3gms of molasses.

% Moisture content = 16.96, 19.11, 19.99
Mean value = 18.68
Standard deviation = 1.56

The samples were rerun using the identical temperature profile but reducing the sample size to one gram.

% Moisture content = 21.13, 22.03, 21.88
Mean value = 21.68
Standard deviation = 0.48

Comparison of the drying profiles confirms the improvements in sample drying by reducing sample size.





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