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Application manual

Moisture analyzer

KERN DIS

Type TDIS-A
Version 1.0
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1 General information

The moisture content of a sample is not just the amount of water in the material. "Material moisture" refers to all volatile substances that escape during heating and cause a sample to lose weight. These include

- ⇒ Water
- ⇒ Fats
- ⇒ Oils
- ⇒ Alcohols
- ⇒ Organic solvents
- ⇒ Flavorings
- ⇒ Volatile components
- ⇒ Possible decomposition substances (if heated too much)

There are many methods for determining the moisture content of a sample. The methods can be divided into two categories:

With the absolute methods, the moisture content of a sample is determined directly (e.g. weight loss through drying). These methods include drying in a drying cabinet, infrared drying and microwave drying. All three methods work thermogravimetrically.

The derived methods are used for indirect determination. A physical property is measured that is related to the moisture (e.g. absorption of electromagnetic radiation). These methods include Karl Fischer titration, infrared spectroscopy, microwave spectroscopy, etc.

1.1 Drying methods

Halogen moisture analyzer
Drying cabinet method
Microwave dryer
Karl Fischer method

Why do different methods determine different material moisture contents?

- ⇒ In addition to the water, the drying oven also vaporizes volatile components. The sample is often not completely dried due to the weak heating by convection drying. The measured value is above the water content but below the total moisture content.
- ⇒ In addition to water, the infrared or halogen dryer also vaporizes volatile and non-volatile components. The total moisture content of the sample is determined through intensive heating by means of absorption drying. The measured value is usually higher than the reference drying cabinet method. (Problems: e.g. pore closure, surface burns)
- ⇒ In addition to water, a microwave dryer only evaporates small quantities of highly volatile components. Due to the dipole-oriented absorption drying, the measured value is very close to the water content and therefore below the drying cabinet value.
- ⇒ Karl Fischer titration uses a chemical reaction to determine the number of water molecules. The measured value corresponds very precisely to the water content.

Two reference methods are internationally recognized:

- ⇒ The drying oven method for determining material moisture
- ⇒ Karl Fischer titration for determining the water content
- ⇒ All other methods must - if necessary - be adjusted to one of these two methods!

1.1.1 Drying cabinet method

In the traditional drying oven method, a hot air stream heats the sample from the outside in, against the flow of rising moisture and the evaporative cooling that occurs on the surface. Long drying times are often required.

A drying cabinet is a device for dehumidifying an object, usually by dehumidifying the air, using hygroscopic materials (sorbents). Drying ovens are available for temperature ranges from room temperature to around 250°C.

Procedure (approx.):

- Weighing the sample bowl
- Weigh sample
- Dry sample for 1 hour
- Cool the sample in the desiccator (see below) for 20 min.
- Weigh back sample
- Manual calculation of results

- Dry the sample for 30 minutes
- Cool the sample in the desiccator for 20 min.
- Weigh back sample
- Recalculation of results
- Repeat steps until the sample is constant in weight

What is a desiccator?

A desiccator (also: exsiccator, from the Latin exsiccare: to dry out) is a chemical laboratory device that is mainly used for drying solid chemical substances in preparative chemistry.

A desiccator is a thick-walled container, usually made of glass (or more rarely plastic), which is sealed airtight with a ground-glass lid. To ensure this seal, the ground joint is usually coated with ground joint grease. The lower part of the desiccator is filled with a drying agent and the substance to be dried is placed on a plastic or ceramic insert above the drying agent.

The desiccant removes the evaporated solvent released by the substance to be dried from the air inside the desiccator. This allows the substance to lose further adsorbed solvent; this process continues until the

A certain residual solvent content is reached which, due to the intensity of the drying medium and the adsorptive properties of the substance, cannot be further reduced or the drying agent (its capacity) is exhausted. If water is to be removed, the primary property of the drying agent is referred to as hygroscopicity. Commonly used are calcium chloride, phosphorus pentoxide, sulphuric acid or silica gel; a moisture indicator can be added to the latter (Blue gel). In addition to water, residues of other solvents can also be absorbed, depending on the choice of drying agent.

1.1.2 Karl Fischer method

This method is used to determine the quantity of water content. The specific reaction of water to a Karl Fischer reagent containing iodine, sulphur dioxide and pyridine in the presence of methanol is used. This method can be used either as coulometric titration or volumetric titration. In coulometric titration, the sample is added to the Karl Fischer reagent and subjected to electrolytic oxidation to generate iodine. Since the iodine is generated in proportion to the amount of electricity according to Faraday's law, the amount of water can be determined immediately according to the amount of electricity required for the electrolytic oxidation. In volumetric titration, the sample is added to a suitable dehydrated solvent in a titration flask that has been made anhydrous with a titrant. The titration is then carried out using a titrant with a previously standardized titre (mg H₂O/ml). The moisture content of the sample is determined from the titration volume (ml). Automatic volumetric titrators based on these methods are commercially available.

1.1.3 Thermogravimetric measurement with halogen moisture analyzer

The halogen moisture analyzers from KERN provide fast and reliable measurement results. The instruments measure according to the principle of thermogravimetry.

The sample is weighed and heated with a halogen lamp (infrared radiation). The weight loss is recorded continuously and drying is stopped after a defined criterion. The moisture content is automatically calculated from the weight difference.

Drying with a halogen lamp is a further development of the infrared drying method.

During the drying process in a halogen moisture analyzer, the sample absorbs the infrared radiation from a halogen lamp. Most of the radiation penetrates the sample and is only converted into thermal energy there. This causes the sample to heat up very quickly.

A small part of the radiation is reflected or transmitted by the sample. The amount of reflected radiation depends mainly on whether it is a light or dark sample

You know this effect from everyday life:

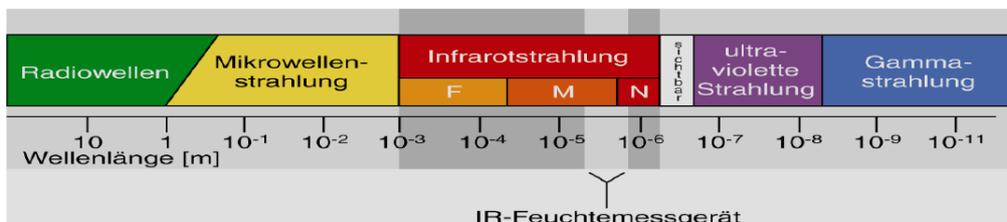
If you are standing in the sun wearing black clothing, you will be much hotter than if you are wearing a white shirt.

For a darker substance, a slightly lower drying temperature should always be selected than for a light-colored sample.

The penetration depth of the IR radiation depends on the permeability of the sample. If the permeability is low, the IR radiation only penetrates into the upper layers. The thermal conductivity of the substance is therefore decisive for the further transport of heat into the deeper layers. The higher the thermal conductivity, the faster and more homogeneously the sample heats up.

For this reason, the substance must be evenly and thinly distributed on the sample pan, see section 2.1.1.

1.1.3.1 Wave spectrum



Infrared radiation is a sub-range of the electromagnetic wave spectrum.

This non-visible thermal radiation occurs on the long-wave side of the optical spectrum, i.e. after the red light. Infrared radiation is subject to the laws of optics and can be focused using a concave mirror, for example.

2 Halogen moisture analyzer operation

The quality of the measurement results stands and falls with the optimal preparation of the sample and the correct selection of the most important measurement parameters such as

- ⇒ Sample size
- ⇒ Drying temperature
- ⇒ Switch-off criterion
- ⇒ Drying time

The optimum drying temperature and drying time depend on the type and size of the sample and the desired accuracy of the measurement result. They can only be determined experimentally.

2.1 Sample size

As a general rule, the more inhomogeneous the sample, the larger the sample quantity required to achieve a repeatable result.

Experience has shown that a practical sample quantity is approx. 5 to 15 g (2 to 5 mm height). Failure to do so may result in incomplete drying, prolonged measuring time, incrustations, burns and non-reproducible measuring results.

2.2 Sample preparation

Only ever prepare one sample for the measurement. This prevents the sample from exchanging moisture with the environment. If several samples have to be taken at the same time, they should be packed in airtight containers so that they do not change during storage.

Spread the sample evenly and thinly on the sample pan to obtain reproducible results. Uneven application results in an inhomogeneous heat distribution in the sample to be dried, which leads to incomplete drying or an extension of the measuring time. If the sample is piled up, the upper layers are heated more strongly, resulting in burns or incrustations. The high layer thickness or possible incrustation makes it impossible for the moisture to escape from the sample. This residual moisture means that measurement results obtained in this way are not comprehensible and reproducible.

The optionally available glass fiber filters should be used for liquid, pasty, greasy, melting and highly reflective samples. This also applies to samples that form a skin on the surface when exposed to heat. The glass fiber filter ensures even and rapid heat distribution and prevents the formation of an impermeable skin on the sample surface.

Sample preparation for solids:



- Distribute powdery and granular samples evenly on the sample tray
- Reduce coarse-grained samples with a mortar or grinder. Avoid applying any heat when crushing the sample, as this leads to moisture loss.

Sample preparation with high moisture content:

To measure ketchup, mayonnaise or toothpaste, press the required amount of sample onto the sample tray and spread it with a spatula. Work quickly, as evaporation can start as soon as the sample is spread if it has a high content of volatile components.

Sample preparation for liquids:



Pour the liquid into the tray to distribute it over the entire surface. However, a very viscous sample will not spread easily. In this case, spread the sample evenly in the tray using a spatula.

For liquids, pastes or melting samples, it is recommended to use a glass fiber filter. The glass fiber filter has the following advantages:

- Even distribution due to capillary effect
- No drip formation
- Rapid evaporation due to larger surface area

2.3 Define measurement parameters

The following measurement parameters are available to adapt the moisture analyzer to the sample to be measured:

- ⇒ Drying mode
- ⇒ Switch-off criterion
- ⇒ Result display

2.3.1 Drying modes

The selectable drying modes vary from appliance to appliance. Detailed information can be found in the operating instructions supplied with the respective appliance

As a rule, you can select the settings for temperature control according to the properties of the sample from the modes described below.

Examples of settings:



Standard drying

Standard drying is suitable for most sample types.

1. Automatic end mode: AUTO

The sample is heated to the set temperature at normal power and is then kept at this temperature. The sample mass then decreases over time. The measurement is stopped automatically when the set weight loss (ΔM) is reached.

By setting the automatic switch-off condition to a low value, a measured moisture content value is obtained that is closer to the actual value, but the measuring time is extended as a result. By setting a higher value, the measurement can be ended quickly, but in some cases the measurements may stop before the water has evaporated sufficiently. The automatic switch-off condition must be set so that it corresponds to the properties of the sample.

2. Time-controlled end: TIME

The sample is heated to the set temperature at normal power and is then held at this temperature. The measurement is ended when the set time has elapsed.

This method is suitable for samples where a small loss of mass can persist indefinitely.



3. Rapid drying

Rapid drying can be used for samples with a moisture content of approx. 5% - 15% (e.g. liquids) and heat-resistant samples with high decomposition temperatures. Sodium tartrate dihydrate and fine flour are examples of such measurements.

During rapid drying, a preheating stage is switched on, i.e. the temperature is increased very quickly and exceeds the set drying temperature until it falls below the set target value (e.g. weight loss/30 sec).

The temperature is then reduced to the set value. Depending on the setting, drying ends when the set time has elapsed or the set weight loss (ΔM) has been reached



4. Gentle drying

Gentle drying is suitable for substances that do not tolerate rapid heating by the radiators. There are also substances that form a skin when heated quickly. This skin then influences the evaporation of the trapped moisture. This soft type of heating is also suitable for these substances.

With gentle drying, the temperature is increased to the set value more slowly than with standard drying.

Depending on the setting, the measurement ends when the set time has elapsed or the set weight loss (ΔM) has been reached.

Gentle drying is suitable for samples that cannot tolerate rapid heating by the radiators. It is also suitable for samples that form a skin when heated quickly. This skin subsequently influences the evaporation of the trapped moisture.



5. Step drying

Step drying is suitable for measuring each component when the evaporation temperature of water and the volatile components contained in the sample are different

As the sample temperature rises, the water evaporates and the change in moisture content normally decreases steadily. In some cases, however, the moisture content increases again above a certain temperature. This is probably because the water evaporates initially, but then the less volatile substances begin to evaporate or the sample begins to decompose. Such measurements are not reliable as an accurate measurement of the moisture content is impossible. This phenomenon occurs, for example, when soybeans are measured. In this case, step drying can be useful to evaporate the water at low temperature before increasing the temperature to determine the moisture content of the high boiling point ingredients. However, it is difficult to separate ingredients that have a boiling point close to that of water, or that have similarly high boiling points.

The individual stages can be freely selected in terms of duration and heating step.

Depending on the setting, the measurement ends at level 2 or 3 when the set time has elapsed or the set weight loss (ΔM) has been reached

2.3.2 Switch-off criterion

The switch-off criterion determines when the measurement is ended and the result is displayed. The moisture analyzers usually offer two different switch-off criteria.

The time-controlled switch-off (TIME) or the weight loss per time unit (AUTO). The integrated scale continuously determines the weight loss of the sample during drying. If the weight loss (ΔM) falls below a certain time (Δt), drying is stopped and the result is displayed

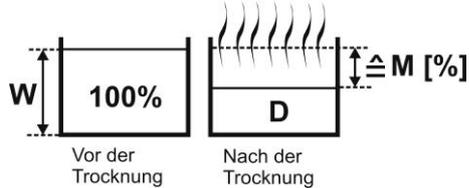
2.3.3 Result display

Various types of result display are available.

g: Residual weight in grams

The weight of the sample is displayed in grams.

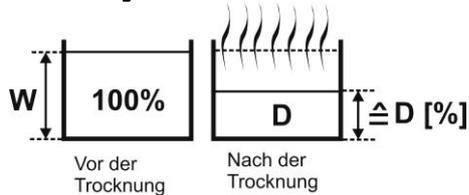
% M: Moisture content



The moisture content of the sample is displayed as a percentage of the wet weight
(W = wet weight = starting weight = 100%)

$$M [0...-100\%] = \frac{\text{Wet weight } W - \text{Dry weight } D}{\text{Wet weight } W} \times 100\%$$

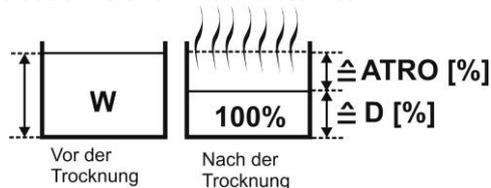
% D: Dry content



The dry content of the sample is displayed as a percentage of the wet weight
(W = wet weight = starting weight = 100%)

$$D [100...0\%] = \frac{\text{Dry weight } D}{\text{Wet weight } W} \times 100\%$$

ATRO %M: ATRO moisture content



The moisture content of the sample is displayed as a percentage of the dry weight
(D = dry weight = Final weight = 100%)

$$ATRO [0...-999\%] = \frac{\text{Wet weight } W - \text{Dry weight } D}{\text{Dry weight } D} \times 100\%$$



ATRO is a unit that is used exclusively in the wood industry

3 Example measurements with halogen moisture analyzer

3.1 General information

In principle, the sample-specific parameters can usually only be determined experimentally,

or

They are based on existing standards, internal company directives or subsequent recommendations.

Note:

The drying temperature has a significant influence on the measurement duration. It should be selected so that the sample neither decomposes nor changes its chemical structure. A drying temperature that is too low unnecessarily extends the drying time. It should also be noted that certain samples can release different amounts of moisture at different drying temperatures. This is the case with substances in which the moisture is bound to varying degrees or those that tend to decompose. Minimal deviations can be adjusted to the moisture content values of the reference method by changing the drying temperature.

We recommend the following procedure for selecting the temperature:

- Estimate the moisture content of the sample.
- Determine the decomposition temperature of the sample by testing
- Compare the measurement results with the reference method, if this exists.

If too much moisture was removed, the drying temperature must be reduced. If the measurement results are too low, the drying temperature may be too low or the drying time may have been too short.

3.2 General sample instructions

- i**
- For pasty samples such as ketchup, it is recommended to use a glass fiber filter (optional). This reduces the drying time and increases reproducibility.
 - For liquids, it is recommended to use a glass fiber filter (optional). This reduces the drying time and increases reproducibility.
 - For dark-colored samples, set the drying temperature as low as possible.

3.3 Assistance

Malfunction

Sample burns during analysis

Possible solution

- Set a lower temperature
- Use step or ramp program for temperature control
- Shorten runtime/heat exposure.
- Protect the sample by covering it with a glass fiber carpet pad

Analysis takes too long

- Increase drying temperature
- Use fast or step program
- Reduce sample quantity
- Increase surface area by using a glass fiber carpet pad

Results are inaccurate

- Increase sample weight (for low moisture content)
- Reduce sample weight (for high moisture content)
- Use automatic switch-off criterion
- Check drying profile for constant weight
- Ensure that the sample is homogeneous

- | | |
|---|--|
| Results are not reproducible | <ul style="list-style-type: none"> • Ensure that the sample preparation is uniform and does not affect the sample • Use automatic switch-off criterion • Evaluate drying profile, check whether the sample burns or does not dry sufficiently |
| Sample loses weight during weighing | <ul style="list-style-type: none"> • Allow the device to cool down sufficiently between measurements • Place the sample outside the drying unit in the drying pan |
| Sample does not reach a constant weight during drying | <ul style="list-style-type: none"> • Use time-controlled switch-off criterion • Lower the drying temperature |
| Sample melts during heating | <ul style="list-style-type: none"> • Use fiberglass carpet pad |
| Sample has a low moisture content | <ul style="list-style-type: none"> • Increase sample quantity |
| Sample contains flammable material | <ul style="list-style-type: none"> • Follow the safety instructions in the operating instructions |

3.4 Food - Dry food

<i>Food/ Product</i>	<i>Sample weight</i>	<i>Sample preparation</i>	<i>Setting</i>	<i>Switch-off criterion</i>	<i>Time</i>	<i>% MC</i>	<i>p-value (%)</i>
Dry food							
<i>Cake mix</i>	3 g	Unchanged	Standard 140°C	Manual	4 min	5,2	0,09
<i>Cocoa powder</i>	3 g	Unchanged	Standard 160°C	TIME	5 min	7,4	0,12
<i>Corn flour</i>	3 g	Unchanged	Rapid drying 130°C	A/60	4 min	12,5	0,15
<i>Flour</i>	3 g	Unchanged	Rapid drying 130°C	A/60	4 min	13,4	0,11
<i>Milk powder</i>	3 g	Unchanged	Rapid drying 80°C	Manual	4 min	3,7	0,2
<i>Pancake mix</i>	3 g	Unchanged	Standard 160°C	TIME	5 min	12,4	0,07
<i>Instant coffee</i>	3 g	Unchanged	Standard 95°C	A/30	5 min	14,5	0,06

3.5 Food - Sugar syrups

<i>Food/ Product</i>	<i>Sample weight</i>	<i>Sample preparation</i>	<i>Setting</i>	<i>Switch-off criterion</i>	<i>Time</i>	<i>% MC</i>	<i>p-value (%)</i>
Sugar syrups							
<i>Honey</i>	1 g	Between 2 glass fiber filters	Step drying 130°C + 110°C	TIME	5 min + 3 min = 8 min	16,1	0,07
<i>Corn syrup</i>	1 g	Between 2 glass fiber filters	Step drying 140°C + 110°C	TIME	3 min + 6 min = 9 min	21,7	0,1
<i>Molasses</i>	1 g	Between 2 glass fiber filters	Step drying 130°C + 110°C	TIME	5 min + 3 min = 8 min	21,7	0,48

3.6 Food - Baked/dried foods

<i>Food/ Product</i>	<i>Sample weight</i>	<i>Sample preparation</i>	<i>Setting</i>	<i>Switch-off criterion</i>	<i>Time</i>	<i>% MC</i>	<i>p-value (%)</i>
<i>Baked/dried foods</i>							
<i>Chips</i>	3 g	Crushed	Standard 95°C	A/30	2 min	0,78	0,08
<i>Cracker</i>	3 g	Grind evenly	Rapid drying 80°C	A/60	4 min	3,7	0,04
<i>Roasted peanuts</i>	3 g	Grind, 15 s	Standard 95°C	TIME	5 min	1,3	0,04
<i>Cake</i>	3 g	Grind evenly	Step drying 140°C + 110°C	A/30	3 min + 4 min = 7 min	33,6	0,2
<i>Sugar cookie</i>	5 g	Grind evenly	Rapid drying 95°C	TIME	4 min	5,3	0,03

3.7 Food - Miscellaneous

<i>Food/ Product</i>	<i>Sample weight</i>	<i>Sample preparation</i>	<i>Setting</i>	<i>Switch-off criterion</i>	<i>Time</i>	<i>% MC</i>	<i>p-value (%)</i>
<i>Miscellaneous</i>							
<i>Creamy salad dressing</i>	3 g	Distribute glass fiber filters evenly	Step drying 180°C + 120°C	Manual	3 min + 3 min = 6 min	34,9	0,95
<i>Low-fat salad dressing</i>	2 g	Between 2 glass fiber filters	Rapid drying 130°C	A/30	10 min	72,6	0,14
<i>Dried vegetables</i>	2 g	Unchanged	Rapid drying 80°C	A/30	3 min	2,4	0,01
<i>Dried herbs</i>	1 g	Unchanged	Standard 110°C	A/30	2 min	9,8	0,04
<i>Carrot</i>	3 g	Crushed	Step drying 180°C + 120°C	A/30	3 min + 3 min = 6 min	89,4	0,08
<i>Pudding</i>	2 g	Between 2 glass fiber filters	Step drying 180°C + 120°C	A/30	3 min + 3 min = 6 min	71	0,1

3.8 Pet food / Cereals

<i>Food/ Product</i>	<i>Sample weight</i>	<i>Sample preparation</i>	<i>Setting</i>	<i>Switch-off criterion</i>	<i>Time</i>	<i>% MC</i>	<i>p-value (%)</i>
<i>Pet food / Cereals</i>							
<i>Broken corn</i>	5 g	Grinding, 30 s	Rapid drying 110°C	TIME	4 min	10,2	0,04
<i>Pelleted pet food</i>	5 g	Grinding, 30 s	Rapid drying 110°C	TIME	4 min	11,3	0,13
<i>Rye seed</i>	5 g	Grinding, 45 s	Rapid drying 110°C	TIME	4 min	10,6	0,21
<i>Dry dog food</i>	5 g	Grinding, 30 s	Rapid drying 80°C	TIME	4 min	5,8	0,08

3.9 Industrial products - Personal care

<i>Food/ Product</i>	<i>Sample weight</i>	<i>Sample preparation</i>	<i>Setting</i>	<i>Switch-off criterion</i>	<i>Time</i>	<i>% MC</i>	<i>p-value (%)</i>
<i>Body care</i>							
<i>Deodorant</i>	2 g	thinly sliced in a bowl	Standard 110°C	A/30	9 min	36,7	0,4
<i>Liquid hand soap</i>	1 g	Distribute glass fiber filters evenly	Step drying 180°C + 120°C	A/30	3 min + 1 min = 4 min	82	0,09
<i>Skin cream</i>	1 g	Distribute glass fiber filters evenly	Step drying 180°C + 120°C	A/30	3 min + 8 min = 11 min	87,7	0,35
<i>Soap</i>	2 g	thinly sliced in a bowl	Standard 110°C	A/30	7 min	9,74	0,21
<i>Powder detergent</i>	3 g	Unchanged	Rapid drying 110°C	A/30	3 min	6,2	0,22
<i>Toothpaste</i>	1 g	Distribute glass fiber filters evenly	Rapid drying 130°C	A/30	3 min	34,7	0,03

3.10 Industrial products - Miscellaneous

<i>Food/ Product</i>	<i>Sample weight</i>	<i>Sample preparation</i>	<i>Setting</i>	<i>Switch-off criterion</i>	<i>Time</i>	<i>% MC</i>	<i>p-value (%)</i>
<i>Pet food / Cereals</i>							
<i>Potting soil</i>	3 g	Unchanged	Rapid drying 200°C	Manual	5 min	68,9	0,66
<i>Wood glue</i>	1 g	Distribute glass fiber filters evenly	Standard 135°C	A/30	7 min	52,9	0,42
<i>Latex paint</i>	1 g	Between 2 glass fiber filters	Rapid drying 170°C	A/30	5 min	58,3	0,26
<i>Mortar mix</i>	3 g	Unchanged	Rapid drying 200°C	A/30	1 min	1,73	0,04