

# KERN & Sohn GmbH

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# **Application notes Moisture analyzer**

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#### 1 General

The moisture of a sample is not only the water content in the material. "Material moisture" is understood to be all volatile substances which escape during heating and result in a weight loss of a sample. These include:

- Water
- Fats
- Oils
- Alcohols
- Organic solvents
- Flavorings
- Volatile constituents
- Where necessary, Decomposing substance (if heated too strongly)

A variety of methods exist to determine the moisture content of a sample. The methods can be divided in two categories:

The absolute method is used to directly determine the moisture content of a sample (e.g. weight loss by drying). These methods include drying in the drying cabinet, infrared drying and micro-wave drying. All three methods work thermogravimetrically.

Indirect determination is carried out by means of the derived methods. A physical property is measured which is in connection with the moisture (e.g. absorption of electromagnetic radiation). These methods include the Karl-Fischer titration, the infrared spectroscopy, the micro-wave spectroscopy, etc.

#### 1.1 Drying methods

Halogen moisture analyzer
Drying cabinet method
Micro-wave dryer
Karl-Fischer method

Why do different methods determine different material moistures?

- The drying cabinet evaporates beside water also slightly volatile constituents. Often the sample is not dried completely due to the weak heating by means of convection heat. The measuring value is above the water content but below the total moisture content.
- The infrared or halogen dryer evaporates beside water also highly or hardly volatile constituents. The total moisture of the sample is determined by intensive heating using the absorption drying method. In most cases, the measuring value is above the reference method of the drying cabinet.
   (Problems: e.g. pore sealing, surface burns)
- A micro-wave dryer evaporates beside water only low amounts of highly volatile constituents. Due to absorption drying – orientated to dipoles – the measuring value is very close at the water content and thus below the value of the drying cabinet.
- The Karl-Fischer titration method determines the number of water molecules by a chemical reaction. The measuring value corresponds to the water content with highest accuracy.

Two reference methods are recognized internationally:

- The drying cabinet method to determine the material moisture
- The Karl-Fischer titration to determine the water content
- All other methods have to be adapted to one of the two methods, if necessary!

#### 1.1.1 Drying cabinet method

For the traditional drying cabinet method, a hot air flow heats the sample from outside to inside against the flow of the rising moisture and the evaporation coldness at the surface. Often long drying periods are required.

A drying cabinet is an apparatus to de-moisture an item mostly by de-moistening the air and using hygroscopic materials (sorbents). Drying cabinets are offered for temperature ranges from room temperature up to approx. 250 °C.

#### Procedure (approx.):

- ⇒ Weigh sample tray
- ⇒ Weigh in sample
- ⇒ Dry sample for 1 hour
- ⇒ Cool down sample in desiccator (see below) for 20 min
- ⇒ Weigh back sample
- ⇒ Calculate result manually
- ⇒ Re-dry sample for 30 min
- ⇒ Cool down sample in desiccator for 20 min
- ⇒ Weigh back sample
- ⇒ Calculate the result again
- ⇒ Repeat the steps until the sample has a constant weight

#### What is a desiccator?

A desiccator (from Latin exsiccare – drying out) is a chemical laboratory apparatus which is mostly used to dry solid chemical substances in preparative chemistry. A desiccator is in most cases a thick-wall vessel of glass (more seldom of plastic material) which is sealed hermetically by a plane ground lid. To ensure this sealing, typically grinding grease is applied to the ground surface. The bottom part of the desiccator is filled with a desiccant and the substance to be dried is placed onto an insert of plastic or ceramic material on top of the desiccant.

The desiccant absorbs the evaporated solvent emitted by the substance to be dried from the air inside the desiccator. Thus the substance may lose further adsorbed solvent: this process is maintained until a

certain residual contents of solvent is obtained which – due to the intensity of the drying medium and the adsorptive properties of the substance – cannot be reduced further or the desiccant (its capacity) is exhausted. If water shall be extracted, the overriding property of the desiccant is called hygroscopy. Calcium chloride, phosphor pentoxide, sulphuric acid or silica gel are used commonly, a moisture indicator (blue gel) may be added to the latter. In addition to water also residues of other solvents can be absorbed depending on the desiccant selected.

#### 1.1.2 Karl-Fischer method

This method is used to determine the amount of water content. Here, the specific reaction of water to a Karl Fischer reagent is used which contains iodine, sulphur dioxide and pyridine in the presence of methanol. This method can be applied either as coulometric titration or volumetric titration. For the coulometric titration, the sample is added to the Karl Fischer reagent and subjected to electrolytic oxidation in order to produce iodine. As the iodine is generated according to Faraday's law related to the amount of electricity, the amount of water can be determined immediately according to the amount of electricity which is required for electrolytic oxidation. For the volumetric titration, the sample is added to an appropriate dehydrated solvent in a titration flask which had been dehydrated by means of a titrant. Titration is then carried out by means of a titrant with previously standardized titer (mg H2O/ml). The moisture content of the sample is determined from the titration volume (ml). Automatic volumetric titrators on the basis of these methods are commercially available.

# 1.1.3 Thermo-gravimetric measurement by means of halogen moisture analyzer

The KERN halogen moisture analyzer supplies quick and reliable measuring results. The instruments measure according to the principle of thermo-gravimetry.

The sample is weighed and heated by a halogen lamp (infrared radiation). The weight loss is recorded continuously and drying is finished according to a defined criterion. The moisture content is calculated automatically from the weight difference. Drying by means of halogen spotlights is a further development of the infrared drying method.

During the drying process in a halogen moisture analyzer, the sample absorbs the infrared radiation of a halogen lamp. Radiation penetrates most part of the sample and is converted there in heat energy. In this way, the sample is heated very quickly. A low part of the radiation is reflected by the sample or passes the latter. The amount of reflected radiation depends to a large part on the fact whether it is a light or a dark sample.

You know this effect from everyday life:

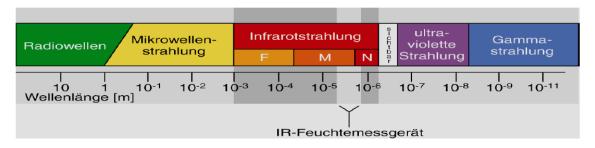
If you wear dark clothes and stay in the sun you feel much warmer than when wearing a white shirt.

In any case, a slightly lower drying temperature should be selected for a darker substance than for a lighter sample.

The depth of penetration of the IR radiation depends on the permeability of the sample. At a lower permeability, the IR radiation penetrates only the top layers. Thus the heat conductivity of the substance is decisive for the further transport of heat into the deeper layers. The higher the heat conductivity the more quickly and homogenously the sample is heated.

This is the reason why the substance must be distributed evenly and in a thin layer at the sample tray, refer to chapter 2.1.1.

#### 1.1.3.1 Wave spectrum



Infrared radiation is part of the electro-magnetic wave spectrum.

This invisible heat radiation occurs at the long-wave end of the optical spectrum, i.e. following the red light, infrared radiation is subject to the laws of optics and can be bundled, for instance by means of a concave mirror.

#### 2 Operation of the halogen moisture analyzer

The quality of the measuring results depends highly on the optimum preparation of the sample and the correct selection of the major measuring parameters such as

- ⇒ Sample size
- ⇒ Drying temperature
- ⇒ Shutoff criterion
- ⇒ Drying period

The optimum drying temperature and drying period depend on the type and size of the sample and the required accuracy of the measuring result. They can be determined only by experiments.

#### 2.1 Sample size

General rules: The more inhomogeneous the sample the larger the sample amount which is required to obtain a reproducible result

A practical sample amount is typically approx. 5 to 15 g
(2 to 5 mm height). Otherwise, incomplete drying, longer measuring time, incrustation, burns and non-reproducible measuring results may occur.

#### 2.2 Preparing a sample

Prepare one sample at a time for measuring. This prevents the sample from exchanging moisture with its surroundings. If several samples have to be taken at the same time, they should be packed in airtight boxes so that they do not undergo changes during storage.

To receive reproducible results, spread the sample thinly and evenly on a sample dish.

Patchy spreads will produce inhomogeneous heat distribution in the sample to be dried resulting in incomplete drying and increased measuring time. Sample clusters generate increased heating of the upper layers resulting in combustion or incrustation. The high layer thickness or possibly arising incrustation makes it impossible for the moisture to escape from the sample. Due to this residual moisture, measured results calculated in this way will not be comprehensible or reproducible. The optionally available fiber glass filters should be used for liquid, pasty, fatcontaining, melting and highly reflecting samples. This is also applicable to samples at which a skin develops at the surface at heat exposure. The fiber glass filter ensures even and quick heat distribution and prevents the development of an impermeable skin at the sample surface.

# Preparing a sample from solids:



- Spread powdery or grainy samples evenly on the sample dish.
- Grind coarse samples using a mortar or a shredder. When grinding the sample avoid any heat supply as this may cause loss of humidity.

#### Preparation of samples with high moisture content:

To measure ketchup, mayonnaise or tooth paste press the required sample amount into the sample tray and spread it by means of a spatula. Work quickly because evaporation may start already when spreading the sample if it has a high content of highly volatile constituents.

#### Preparing a sample from liquids:



Pour the liquid into the tray in order to distribute it across the whole area. However, it is not so easy to distribute a highly viscous sample. In this case distribute the sample evenly in the tray by means of a spatula.

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

- Even distribution thanks to capillary attraction
- no formation of droplets
- fast evaporation due to a greater surface

#### 2.3 Definition of measuring parameters

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

- ⇒ Drying mode
- ⇒ Shutoff criterion
- ⇒ Result display

#### 2.3.1 Drying modes

The drying modes which can be selected differ from apparatus to apparatus. For detailed information please refer to the operating manual supplied with each apparatus.

From the modes described in the following, normally you can select the settings for temperature control according to the properties of the sample.

# **Setting examples:**



#### Standard drying

Standard drying is suitable for most types of sample.

#### 1. Automatic end mode: AUTO

The sample is heated to the set temperature at normal output and is then kept at this temperature. The sample mass is reduced in the course of time. Measurement is finished automatically if the set weight loss ( $\Delta M$ ) is reached. When setting the automatic switch-off conditions to a low value, you will obtain a measured moisture content value which comes closer to the actual value, but the measuring time is increased. When setting a high value, measurement can be finished quickly, but in some cases the measurements may be aborted before the water is 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 output and is then kept at this temperature. Measurement is finished when the set time elapsed.

This method is suitable for samples where a low measuring loss may last for an indefinite period of time.



#### **Quick drying**

Quick drying can be applied for sample with moisture contents from approx. 5 % to 15 % (e.g. liquids) and heat-resistant samples with high decomposition temperatures.

Sodium tartrate dehydrate and fine-grain flour are examples for such measurements.

A preheating stage is switched on for Rapid drying, i.e. the temperature will increase very quickly and will exceed the preset drying temperature until it falls below the preset reference value (e.g. weight loss/30 sec).

Then the temperature is controlled down to the set value. Drying is finished depending the setting when the set time elapsed or the set weight loss ( $\Delta M$ ) is reached.



#### **Gentle drying**

Gently drying is suitable for substances which do not tolerate quick heating by the spotlights. There are also substances which develop a skin during quick heating. This skin will then affect the evaporation of the trapped moisture. For such substances, the soft mode of warming is equally suitable.

The Slow drying temperature is increased more slowly to the preset value than for Standard drying.

Measurement is finished depending on the setting when the set time elapsed of the set weight loss ( $\Delta M$ ) is reached.

Slow drying is suitable for samples that cannot tolerate rapid heating by the heaters. The same applies to samples that form a skin during rapid heating. This skin will then affect the evaporation of the trapped moisture.



#### **Drying in stages**

Drying in stages is suitable for measuring each constituent if the evaporation temperature of water and the volatile constituents which are contained in the sample are different.

With rising sample temperature, the water evaporates and the change of the moisture content will constantly decrease in normal cases. In some cases, however, the moisture content increases again above a certain temperature. Presumably because the water evaporates at the beginning, but then the hardly volatile substances start evaporating or the sample starts decomposing. Such measurements are not reliable because exact measurement of the moisture content is not possible. This phenomenon occurs for example when measuring soy beans. In this case, drying in stages may be useful in order to evaporate the water at low temperature before increasing the temperature in order to determine the moisture content of the constituents with high boiling point. However, it is difficult to separate constituents the boiling point of which is close to that of water or constituents with similarly high boiling points.

The individual steps are freely selectable regards duration and temperature rising step.

Measurement is finished depending on the setting of phase 2 or 3 when the set time elapsed or the set weight loss ( $\Delta M$ ) is reached.

#### 2.3.2 Shutoff criterion

The switch-off criterion determines when the measurement is finished and the result is indicated. Typically, the moisture analyzers offer two different switch-off criterions. Time-controlled switch-off (TIME) or weight reduction per unit of time (AUTO). The integrated scale continuously determines the weight loss of the sample during drying. If the weight loss ( $\Delta$ M) is less than specified after a certain period of time ( $\Delta$ t) drying is finished and the result is indicated

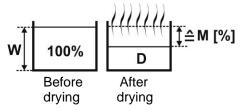
#### 2.3.3 Result display

The result display enables the selection of a display in % moisture, % dry mass, ATRO\* and residual weight in grams.

#### g: Weight in grams

The weight of th3 sample is displayed in grams.

#### % M: Moisture content

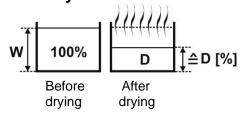


The moisture content of the sample is displayed as a percentage of the wet weight

(W = wet weight = initial weight = 100%)

M 10 4000/1	Wet weight <b>W</b> – Dry weight <b>D</b>	4000/
<b>M</b> [0100%]	Wet weight <b>W</b>	——— x 100%

#### % D: Dry content

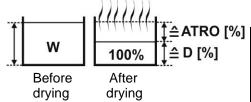


The dry content of the sample is displayed as a percentage of the wet weight

(= W = wet weight = initial weight = 100%)

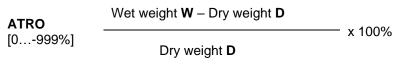
<b>D</b> [400 00/1	Dry weight <b>D</b>	4000/
<b>D</b> [1000%]	Wet weight <b>W</b>	– x 100%

# 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%)





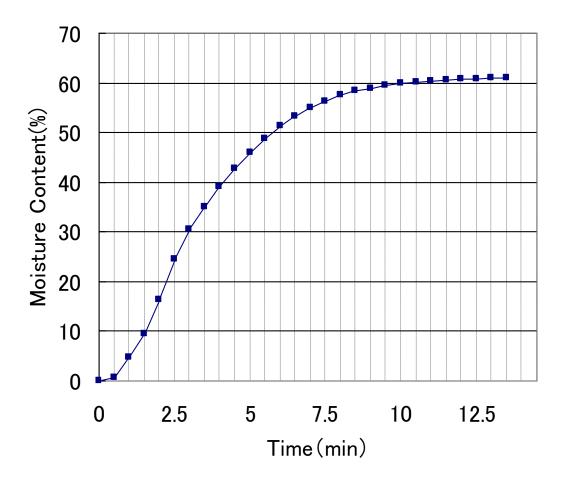
ATRO is a unit which is exclusively used in the timber industry

#### 2.4 Data transfer to a printer or PC

The following conditions must be met to provide successful communication between the moisture analyser and the printer.

- Disconnect moisture analyser from the power supply and connect to the printer interface with a suitable cable. Faultless operation requires an adequate KERN interface cable.
- Communication parameters (baud rate, bits and parity) of moisture analyser and printer must match.

We recommend our transfer software 'Balance Connection KERN SCD 4.0' for the import of data to a PC program. Software, e.g. Excel, can be used to draw graphics.



# 3 Exemplar measurements with Halogene Moisture Analyzer (KERN DBS 60-3)

#### 3.1 General hints

Generally in most cases it is only possible to develop sample-specific parameters by trial and error.

or

Guidance is available in form of existing standards, in-house directives or following recommendations.

#### Note:

The drying temperature influences considerably the measuring time. It must be selected in a way that the sample neither does decay nor changes its chemical structure. A too deep drying temperature lengthens innecessarily the drying time. Also observe that certain samples at different drying temperatures emit different quantities of moisture. This is the case in substances, where moisture is bound at different degrees or which tend to decomposition. Minimum divergencies may be adapted by changing the drying temperature to the moisture contents value of the reference procedure.

For selecting the temperature we recommend the following procedure:

- > Evaluate the moisture content of the sample.
- Define the decomposition temperature of the sample by trials
- Comparison of the measuring results with the reference procedure, if it exists.

If too much moisture has been separated, the drying temperature must be reduced. In case of too low measurement results, the drying temperature is possibly too deep or the drying period was too short.

#### 3.2 Foodstuffs

#### 3.2.1 Table salt (NaCl)

Settings: Standard drying 200°C/ TIME 10 min

	Sample weight (g)	Humidity (%)
4	E 022 ~	0.00
1.	5.032 g	0.08
2.	5.021 g	0.09
3.	5.052 g	0.08
MW		0.083

# Sample before measurement



Distributed evenly on the tray

#### Sample after measurement



Nearly no change

Since this salt was extracted using an ion exchange resin, it contained almost no impurities. The packaging was opened just prior to the measurement so that the salt did not absorb moisture. The moisture content was extremely low.

#### 3.2.2 Dried basil

Settings: Standard drying 90°C/ AUTO 0.05 %( $\Delta M$ )

	Sample weight (g)	Drying time (min)	Humidity (%)
1.	1.327 g	7:16	8.59
2.	1.310 g	6:07	8.40
3.	1.385 g	6:20	8.28
MW			8.42

# Sample before measurement



Distributed evenly on the tray



Nearly no change

# 3.2.3 Paprika powder

Settings: Standard drying 12°C/ AUTO 0.05 %( $\Delta M$ )

	Sample weight (g)	Drying time (min)	Humidity (%)
1.	2.802 g	6:40	7.35
2.	2.775 g	5:31	7.60
3.	2.757 g	5:15	7.36
MW			7.43

# Sample before measurement



Distributed evenly on the tray



no change

# 3.2.4 Cumin

Settings: Standard drying 120°C/ AUTO 0.05 %( $\Delta M$ )

	Sample weight	Drying time	Humidity
	(g)	(min)	(%)
1.	2.815 g	9:26	8.53
2.	2.798 g	7:43	8.51
3.	2.818 g	9:08	8.69
MW			8.57

# Sample before measurement



Distributed evenly on the tray



Nearly no change

# 3.2.5 Oregano

Settings: Standard drying 90°C/ AUTO 0.05 %( $\Delta M$ )

	Sample weight	Drying time	Humidity
	(g)	(min)	(%)
1.	1.135 g	5:03	7.76
2.	1.116 g	5:18	7.17
3.	1.129 g	5:16	7.35
MW			7.42

# Sample before measurement



Distributed evenly on the tray



Nearly no change

# 3.2.6 Crystal sugar

Settings: Standard drying 160°C/TIME 5 min

	Sample weight (g)	Humidity (%)
1.	5.003 g	0.14
2.	5.007 g	0.12
3.	5.043 g	0.14
MW		0.13

# Sample before measurement



Distributed evenly on the tray

# Sample after measurement



No discoloration, but surface slightly solidified.

! At a high drying temperature, crystalline sugar may caramelize (refer to the following photo), exact measurement is prevented in this way.



# 3.2.7 **Butter**

Settings: Standard drying 120°C/ AUTO 0.05 %( $\Delta M$ )

	Sample weight	Drying time	Humidity
	(g)	(min)	(%)
1.	2.327 g	8:54	13.88
2.	2.325 g	6:43	14.62
3.	2.316 g	6:55	14.81
MW			14.43

# Sample before measurement



Distributed evenly on the tray



Noticeable change

#### 3.2.8 Gouda cheese

Settings: Standard drying 120°C/ AUTO 0.05 %( $\Delta M$ )

	Sample weight (g)	Drying time (min)	Humidity (%)
1.	1.544 g	28:05	39.05
2.	1.515 g	29:31	38.42
3.	1.530 g	27:06	37.98
MW		•	38.48

# Sample before measurement



Distributed evenly on the tray



Almost no change in colour

# 3.2.9 Processed cheese (Cheddar)

Settings: Standard drying 120°C/ AUTO 0.05 %( $\Delta M$ )

	Sample weight (g)	Drying time (min)	Humidity (%)
1	1.317 g	57:45	29.23
2.	1.321 g	56:49	29.67
3.	1.335 g	58:57	29.21
MW			29.37

# Sample before measurement



Distributed evenly on the tray



Noticeable colour change

#### 3.2.10 Cream cheese

Settings: Standard drying 100°C/ AUTO 0.10 %( $\Delta M$ )

	Sample weight (g)	Drying time (min)	Humidity (%)
1.	7.924 g	1:00:09	69.96
2.	7.904 g	56:02	70.46
3.	7.932 g	56:18	70.49
MW			70.30

# Sample before measurement



Distributed evenly on the tray



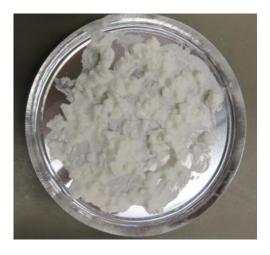
Almost no change in colour

# 3.2.11 Cottage cheese

Settings: Standard drying 120°C/ AUTO 0.05 %( $\Delta M$ )

	Sample weight	Drying time	Humidity
	(g)	(min)	(%)
1.	7.900 g	59:57	75.00
2.	7.965 g	54:58	75.25
3.	7.963 g	59:54	75.70
MW			75.31

# Sample before measurement



Distributed evenly on the tray



Almost no change in colour

#### 3.2.12 Milk

- For liquids we recommend using a fiber glass filter (option) Thus the drying time is reduced and reproducibility is increased.
- Settings: Standard drying 140°C/ TIME 10 min

	Sample weight (g)	Humidity (%)
1.	1.081 g	87.70
2.	1.025 g	87.61
3.	1.031 g	87.68
MW		87.66

Settings: Standard drying 140°C/ AUTO 0.05 %(ΔM)

	Sample weight (g)	Drying time (min)	Humidity (%)
1.	1.036 g	7:10	87.47
2.	1.168 g	8:01	87.52
MW			87.50

#### Sample before measurement



1 g milk distributed evenly via a fiber glass filter

i

#### Sample after measurement



After the extraction of moisture, a thin yellow grease stain remains

The measurements were finished time-controlled once (TIME) and once automatically (AUTO). Both switch-off criteria indicate nearly the same results for samples which contain plenty of water and the main constituents of which have a relatively high boiling point.

# 3.2.13 Milk powder

Settings: Standard drying 105°C/ AUTO 0.05 %( $\Delta M$ )

	Sample weight (g)	Drying time (min)	Humidity (%)
1.	1.819 g	5:18	5.99
2.	1.808 g	6:01	5.86
3.	1.855 g	5:31	5.82
MW			5.89

# Sample before measurement



Distributed evenly on the tray



Nearly no change

# 3.2.14 Tomato ketchup



For pasty samples such as ketchup we recommend using a fiber glass filter (option). Thus the drying time is reduced and reproducibility is increased.

Settings: Standard drying 140°C/ AUTO 0.1 %(ΔM)

	Sample weight	Drying time	Humidity
	(g)	(min)	(%)
1.	2.544 g	19:50	69.32
2.	2.450 g	19:30	69.36
3.	2.619 g	20:00	69.53
MW			69.40

# Sample before measurement



Distributed evenly at a fiber glass filter



Dark discoloration

#### 3.2.15 Mayonnaise

- For pasty samples, such as mayonnaise, we recommend using a fiber glass filter (option). Thus the drying time is reduced and reproducibility is increased.
- Settings: Standard drying 160°C/TIME 10 min

	Sample weight (g)	Humidity (%)
1.	1.078 g	20.52
2.	0.964 g	20.71
3.	1.097 g	20.60
MW		20.61

Settings: Standard drying 160°C/ AUTO 0.05 %(ΔM)

	Sample weight (g)	Drying time (min)	Humidity (%)
1.	0.952 g	6:38	20.38
2.	1.319 g	6:21	20.42
MW			20.40

#### Sample before measurement



Sample distributed evenly by a spatula on a fiber glass filter

#### Sample after measurement



Water and oils evaporate, minor discoloration

The measurements were finished time-controlled once (TIME) and once automatically (AUTO). Both switch-off criteria indicate nearly the same results for samples which contain plenty of water and the main constituents of which have a relatively high boiling point.

i

#### 3.2.16 Instant coffee

- For samples with a dark color set the drying temperature as low as possible.
- Settings: Standard drying 120°C/ TIME 10 min

	Sample weight	Humidity
	(g)	(%)
1.	0.994 g	7.33
2.	1.079 g	7.50
3.	0.980 g	7.45
MW		7.43

Settings: Standard drying 120°C/ AUTO 0.05 %(ΔM)

	Sample weight (g)	Drying time (min)	Humidity (%)
1.	1.033 g	7:42	6.97
2.	0.749 g	7:06	7.06
MW			7.02

#### Sample before measurement



Sample after measurement



Distributed evenly on the tray

Nearly no discoloration

- The measurements were finished time-controlled once (TIME) and once automatically (AUTO). Both switch-off criteria indicate approximately the same results.
  - ! If a high drying temperature (e.g. 200 °C) is set for dark-colored samples, the sample may carbonize (decompose) (refer to the following photo) which prevents exact measurement.



#### 3.2.17 Ground coffee

- For samples with a dark color set the drying temperature as low as possible.
- Settings: Standard drying 120°C/ AUTO 0.05 %(ΔM)

	Sample weight	Drying time	Humidity
	(g)	(min)	(%)
1.	4,983 g	5:30	5,40
2.	4,980 g	5:30	5,35
3.	4,972 g	5:37	5,45
MW			5,40

# Sample before measurement



Distributed evenly on the tray



No discoloration

#### 3.2.18 Coffee beans

The raw (unroasted) coffee beans were sticky due to their moisture content and it was difficult to crush them in the coffee mill. When distributing in the sample tray, 2-3 mm lumps developed.

The roasted coffee beans were finely milled as usual (uniform particle size). It took about 17 minutes to dry the unroasted coffee which was due to the difficulties when drying the lumps.

The drying time of roasted coffee was shorter due to the fine particles

#### Unroasted coffee beans

Settings: Standard drying 140°C/ AUTO 0.05 %(ΔM)

	Sample weight	Drying time	Humidity
	(g)	(min)	(%)
1.	5.162 g	17:00	9.40
2.	5.140 g	16:34	9.42
3.	5.021 g	17:35	9.14
MW			9.32

#### Roasted coffee beans

Settings: Standard drying 140°C/ AUTO 0.05 %(ΔM)

	Sample weight	Drying time	Humidity
	(g)	(min)	(%)
1.	3.028 g	5:57	2.58
2.	3.020 g	7:10	2.68
	3.057 g	8:10	2.78
MW			2.68

#### Sample before measurement



Distributed evenly on the tray, some lumps of raw beans visible.



Parts of surface burnt, despite this high reproducibility achieved.

#### **3.2.19 Green tea**

Settings: Standard drying 120°C/ AUTO 0.05 %( $\Delta M$ )

	Sample weight (g)	Drying time (min)	Humidity (%)
1.	5.056 g	9:15	3.76
2.	5.099 g	9:00	3.75
3.	5.022 g	9:00	3.78
MW			3.76

# Sample before measurement



Distributed evenly on the tray



Nearly no change only the bright green color seems more subdued

# 3.2.20 Barley flakes

Settings: Standard drying 200°C/ AUTO 0.05 %( $\Delta M$ )

	Sample weight	Drying time	Humidity
	(g)	(min)	(%)
1.	5.758 g	9:50	12.64
2.	5.748 g	10:27	12.67
3.	5.710 g	9:58	12.64
MW			12.65

# Sample before measurement



Distributed evenly on the tray



Nearly no change, slight yellow coloration only

#### 3.2.21 Wheat bran

Settings: Standard drying 90°C/ AUTO 0.05 %( $\Delta M$ )

	Sample weight	Drying time	Humidity
	(g)	(min)	(%)
1.	2.633 g	10:08	11.32
2.	2.660 g	11:03	11.01
3.	2.616 g	10:22	10.89
MW			11,07

# Sample before measurement



Distributed evenly on the tray



Nearly no change

### 3.2.22 Coarse oat flakes

Settings: Standard drying 90°C/ AUTO 0.05 %( $\Delta M$ )

	Sample weight	Drying time	Humidity
	(g)	(min)	(%)
1.	2.612 g	12:03	8.61
2.	2.604 g	13:28	8.87
3.	2.651 g	12:03	8.77
MW			8.75

# Sample before measurement



Distributed evenly on the tray



Nearly no change

#### 3.2.23 Milled hazel nuts

Settings: Standard drying 130°C/ AUTO 0.05 %( $\Delta M$ )

	Sample weight	Drying time	Humidity
	(g)	(min)	(%)
1.	5.058 g	6:50	4,33
2.	5.148 g	6:27	4,28
3.	5.010 g	6:58	4,37
MW			4,32

## Sample before measurement



Distributed evenly on the tray

## Sample after measurement



Nearly no change

! If a high drying temperature (e.g. 190 °C) is set, the sample may carbonize (decompose) (refer to the following photo) which prevents exact measurement.



#### 3.2.24 Rice

Settings: Standard drying 200°C/ AUTO 0.05 %( $\Delta M$ )

	Sample weight	Drying time (min)	Humidity (%)
	(g)		
1.	5.938 g	14:19	14.55
2.	5.942 g	13:40	14.47
3.	5.979 g	13:45	14.43
MW			14.48

# Sample before measurement



Distributed evenly on the tray



Yellow coloration

#### 3.2.25 Lemon ice-cream

For pasty samples such as ice-cream we recommend using a fiber glass filter (option). Thus the drying time is reduced and reproducibility is increased.

Settings: Standard drying 140°C/TIME 12 min

	Sample weight	Humidity
	(g)	(%)
1.	2.544 g	84.47
2.	2.450 g	84.73
3.	2.619 g	84.38
MW		84.53

## Sample before measurement



Distributed evenly on a fiber glass filter at room temperature



Yellow coloration

### 3.2.26 Dried mango

Settings: Standard drying 120°C/ TIME 30 min

	Sample weight	Humidity
	(g)	(%)
1.	3.301 g	8.79
2.	3.748 g	9.04
3.	4.474 g	8.38
MW		8.74

### Sample before measurement



2-3mm dices distributed evenly on the tray

### Sample after measurement



Brown coloration, external layer hard, internal core remained soft.

At a measurement at 120°C for 12 h, a moisture content of 23.88 % was measured. The sample was carbonized after the measurement, probably due to the sugar content.



# 3.2.27 Dried apples

Settings: Standard drying 90°C/ AUTO 0.02 %( $\Delta M$ )

	Sample weight	Drying time	Humidity
	(g)	(min)	(%)
1.	1.419 g	5:20	3.52
2.	1.405 g	4:09	3.13
3.	1.413 g	7:28	3.61
MW			3.42

# Sample before measurement



Distributed evenly on the tray



Nearly no change

### 3.2.28 Dried cranberries

Settings: Standard drying 120°C/ AUTO 0.05 %( $\Delta M$ )

	Sample weight	Drying time	Humidity
	(g)	(min)	(%)
1.	2.609 g	18:25	12.30
2.	2.638 g	14:19	11.68
3.	2.947 g	20:13	12.08
MW			12.02

# Sample before measurement



Distributed evenly on the tray



Nearly no change

# 3.2.29 Dried plums

Settings: Standard drying 110°C/ AUTO 0.05 %( $\Delta M$ )

	Sample weight (g)	Drying time (min)	Humidity (%)
1.	6.258 g	48:46	13.69
2.	5.398 g	47:05	13.26
3.	5.289 g	43:14	14.14
MW			13.69

# Sample before measurement



Distributed evenly on the tray



Nearly no change

### 3.2.30 Roasted cashew nuts

Settings: Standard drying 130°C/TIME 15 min

	Sample weight	Drying time	Humidity
	(g)	(min)	(%)
1.	3.704 g	15:00	3.78
2.	3.742 g	15:00	3.63
3.	3.851 g	15:00	3.71
MW			3.70

# Sample before measurement



Distributed evenly on the tray



Nearly no change

### 3.2.31 Corn starch

Settings: Standard drying 180°C/ AUTO 0.02 %( $\Delta M$ )

	Sample weight (g)	Drying time (min)	Humidity (%)
1	5.133 g	9:49	12.27
1.			
2.	4.910 g	9:14	12.10
3.	5.097 g	9:12	12.14
MW			12.17

# Sample before measurement



Distributed evenly on the tray



Nearly no change

#### 3.2.32 Palm oil



For pasty samples such as palm oil we recommend using a fiber glass filter (option). Thus the drying time is reduced and reproducibility is increased.

Settings: Standard drying 120°C/TIME 5 min

	Sample weight	Humidity
	(g)	(%)
1.	2.504 g	0.40
2.	2.660 g	0.41
3.	2.537 g	0.43
MW		0.41

## Sample before measurement



Distributed evenly on a fiber glass filter



Nearly no change, distributed completely

## 3.2.33 Potato chips

Settings: Standard drying 105°C/ AUTO 0.01 %( $\Delta M$ )

	Sample weight (g)	Drying time (min)	Humidity (%)
1.	4.140 g	20:08	2.63
2.	4.111 g	16:21	2.51
3.	4.157 g	16:13	2.57
MW			2.57

# Sample before measurement



Distributed evenly on the tray



Nearly no change

# 3.3 Industrial products, plastic materials, sludge, etc.

### 3.3.1 Sodium tartrate dihydrate

Sodium tartrate dihydrate (NaOOCCH(OH)CH(OH)COONa· $2H_2O$ ) is a stable nontoxic substance. Due to its relatively good agreement with the theoretical moisture value which can be calculated from its molecular formula [36.03 (two water molecules) / 230.08 (total molecular weight) = 15.66 %] it is used in general as test substance for moisture analyzers.

The measured moisture content of 15.80 % deviates slightly from the theoretical value (15.66 %). The cause is, for instance, the absorption of air moisture during storage.

Settings: Standard drying 160°C/TIME 15 min

	Sample weight (g)	Humidity (%)
1.	5.103 g	15.79
2.	5.064 g	15.80
3.	5.021 g	15.80
MW		15.80

### Sample before measurement



Distributed evenly on the tray



Nearly no change

#### 3.3.2 Plastic granulate

Besides the polymer material, plastic granulate also contains other volatile components that escape during drying. As the moisture at the surface of the granulate had to be measured, the drying temperature was set to 100°C. Drying was difficult due to the low drying temperature. In drying mode TIME, the moisture content increases obviously with the time which is due to the following evaporation from inside the granulate.

The measuring time is reduced in drying mode AUTO.

If beside the moisture also other constituents escape during the drying process, the halogene moisture analyzer is not suitable for measuring the absolute moisture content.

Settings: Standard drying 100°C/ TIME 25 min

	Sample weight	Humidity
	(g)	(%)
1.	10.080 g	0.12
2.	10.016 g	0.13
3.	10.290 g	0.13
MW		0.13

Settings: Standard drying 100°C/ AUTO 0.05 %(ΔM)

	Sample weight	Drying time	Humidity
	(g)	(min)	(%)
1.	10.67 g	1:59	0.07
2.	10.56 g	1:59	0.07
MW			0.07

Sample before measurement



Distributed evenly on the tray

Sample after measurement



Nearly no change

# 3.3.3 Sephacryl

Settings: Standard drying 140°C/ AUTO 0.05 %( $\Delta M$ )

	Sample weight	Drying time	Humidity
	(g)	(min)	(%)
1.	0.983 g	9:20	90.13
2.	0.992 g	10:07	90.22
3.	0.991 g	10:39	90.31
MW			90.22

Sample before measurement



Distributed evenly on the tray

Sample after measurement



No change

# 3.3.4 Metal powder

Settings: Standard drying 120°C/ AUTO 0.05 %( $\Delta M$ )

	Sample weight	Drying time	Humidity
	(g)	(min)	(%)
1.	10.279 g	00:53	0.15
2.	10.022 g	00:59	0.15
3.	9.942 g	00:57	0.17
MW			0.1567

## Sample before measurement



Distributed evenly on the tray



No change

### 3.3.5 Washing powder

The measurements were carried out with commercial washing powder which contains blue particles of a bleaching agent.

The drying temperature was set to low 120°C so that the appearance of the sample remained virtually the same after drying.

Evaporation continued after the comparable long drying time of 13 minutes. It is understood that moisture or a volatile constituent evaporate from inside the cleaning particles, but it is not clear which of the constituents evaporated.

The switch-off condition is 0.05 %. If this setting is reduced, the measuring time is increased.

Increasing the drying temperature will probably decompose the sample. Samples with such a gradual evaporation are difficult to measure because the relationship between the drying type and sample decomposition must be taken into account when selecting the measuring conditions

Settings: Standard drying 160°C/ AUTO 0.05 %(∆M)

	Sample weight	Drying time	Humidity
	(g)	(min)	(%)
1.	5.086 g	13:15	9.58
2.	5.035 g	12:50	9.75
3.	5.043 g	13:20	9.89
MW			9.79

### Sample before measurement



Distributed evenly on the tray



Nearly no change

# 3.3.6 Liquid detergent

Settings: Standard drying 120°C/ AUTO 0.05 %( $\Delta M$ )

	Sample weight (g)	Drying time (min)	Humidity (%)
1.	1.949 g	20:30	57.26
2.	1.933 g	22:17	57.99
3.	1.943 g	22:00	57.23
MW			57.49

# Sample before measurement



Distributed evenly on the tray



No change

## 3.3.7 Hand soap

Settings: Standard drying 200°C/TIME 16 min

	Sample weight	Humidity
	(g)	(%)
1.	2.373 g	9.14
2.	2.683 g	9.21
3.	2.712 g	8.92
MW		9.09

# Sample before measurement



Chips distributed evenly on the tray



Brown coloration and small bubbles at the surface

# 3.3.8 Liquid soap

Settings: Standard drying 120°C/ AUTO 0.05 %( $\Delta M$ )

	Sample weight	Drying time	Humidity
	(g)	(min)	(%)
1.	1.506 g	11:20	89.11
2.	1.518 g	11:19	89.26
3.	1.508 g	11:14	89.46
MW			89.27

# Sample before measurement



Distributed evenly on the tray



No change

# 3.3.9 Dishwashing liquid

Settings: Standard drying 120°C/ AUTO 0.05 %( $\Delta M$ )

	Sample weight	Drying time	Humidity
	(g)	(min)	(%)
1.	1.509 g	13:21	71.44
2.	1.559 g	12:26	71.90
3.	1.572 g	13:17	71.63
MW			71.65

# Sample before measurement



Distributed evenly on the tray



No change

### 3.3.10 Water-based paint

In addition to water, the paint contains quite a number of volatile constituents, thus drying takes a long time. Even at a drying temperature of 200°C, drying was not finished completely.

As the moisture content is above 50 %, the measuring values will not change considerably when changing the switch-off condition.

Settings: Standard drying 160°C/ AUTO 0.05 %(∆M)

	Sample weight	Drying time	Humidity
	(g)	(min)	(%)
1.	1.113 g	8:53	52.83
2.	1.171 g	10:12	52.09
3.	1.025 g	9:15	52.24
MW			52.39

### Sample before measurement



Distributed evenly on the tray



Nearly no change

#### 3.3.11 Sludge cake

Sludge cake is the dried residue of water treatment plants, and then it is incinerated. Measurement of the moisture content is important because high water content will require additional energy for incineration.

A sludge cake sample which contains moisture and fibers was placed at a sample tray and crumbled into approx. 10 mm pieces. Due to the bad smell, little time only was spent for crumbling the sample.

The results indicate a moisture content of 81 % and are well reproducible. This good reproducibility was apparently achieved because the sample contains – beside water – only a few volatile constituents

Settings: Standard drying 200°C/ AUTO 0.05 %(ΔM)

	Sample weight (g)	Drying time (min)	Humidity (%)
1.	2.170 g	21:03	81.84
2.	2.074 g	21:34	81.20
3.	2.231 g	21:57	81.62
MW			81.55

#### Sample before measurement



Crumbled sample distributed on the tray



Volume reduced due to drying

### 3.3.12 Potting soil

Potting soil contains, for example, compost, chicken manure and earth. This sample also contained wood chips which may have contributed to a slightly increased reproducibility of the moisture content. Due to the low drying temperature of 120°C, nearly no change of the appearance could be detected after drying Settings: Standard drying 120°C/ AUTO 0.05 %( $\Delta$ M)

	Sample weight	Drying time	Humidity
	(g)	(min)	(%)
1.	4.973 g	15:40	33.62
2.	5.065 g	15:50	33.98
3.	5.032 g	15:00	33.59
MW			33.40

#### Sample before measurement



Distributed evenly on the tray



Nearly no change

#### 3.3.13 Sawdust

The drying temperature was set to 160°C because such samples may ignite at drying temperatures of 200°C. 4 g sawdust were distributed evenly in a slightly thicker layer on the tray.

Despite the thick layer, effective evaporation was achieved due to the high number of intermediate spaces. This resulted in short drying times and good reproducibility. Settings: Standard drying 160°C/ AUTO 0.02  $\%(\Delta M)$ 

	Sample weight (g)	Drying time (min)	Humidity (%)
1.	4.176 g	8:50	34.05
2.	4.032 g	8:30	34.67
3.	4.054 g	8:00	34.41
MW			34.38

### Sample before measurement



Distributed evenly in an approx. 4 mm layer on a tray



Surface burnt slightly, brownish color, despite this good reproducibility

#### 3.3.14 Small animal litter

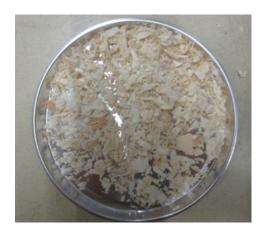
Settings: Standard drying 105°C/ AUTO 0.05 %( $\Delta M$ )

	Sample weight	Drying time	Humidity
	(g)	(min)	(%)
1.	1.006 g	3:38	9.94
2.	1.001 g	3:35	10.19
3.	1.013 g	3:28	10.27
MW			10.13

# Sample before measurement



Distributed evenly on the tray



No change

## 3.3.15 Toner

Settings: Gentle drying 100°C/ TIME 2 min

	Sample weight (g)	Humidity (%)	Drying time (Heating-up time 5 min + 2 min at 100°C)
1.	3.100 g	0.77	7:00
2.	3.102 g	0.71	7:00
3.	3.051 g	0.66	7:00
MW		0.71	

# Sample before measurement



Distributed evenly on the tray



Melted with encrusted surface

### **3.3.16 Lip stick**

In addition to water, lip stick contains volatile constituents such as alcohols. The sample melts during heating, after cooling down to room temperature, however, the initial viscosity and texture are maintained.

Settings: Standard drying 100°C/ TIME 3 min

	Sample weight	Humidity
	(g)	(%)
1.	0.666 g	0.75
2.	0.768 g	0.78
3.	0.923 g	0.65
MW		0.73

### Sample before measurement



3 mm pieces distributed on the tray



The lip stick melted after 1 min.

### 3.3.17 Corn silage

The measurements are carried out with commercial corn silage which is used in biogas plants or as animal food.

Measuring the moisture content is important because the quality or the silage depends on the dry content.

As the corn silage contains fibres of different size and whole corn grains, carry out sample extraction and sample preparation as follows.

#### Sample extraction

Influence of the sample extraction on reproducibility:

- Extract as many samples as possible from various spots and mix them up thoroughly
- During sample collection avoid absorption or moisture exhalation
- Keep it in an air-tight container that the sample does not change during storage.

### Preparing a sample

Crushing the sample into small pieces causes a faster and more effective moisture loss.

- Crush the sample with a powerful electric crusher (e.g. 1000 Watt) .
- Work without delay, that the sample does not heat up during crushing, as this would cause loss of moisture already during preparation.
- To achieve high reproducibility, always use the same sample quantity throughput, such as 5g.
- Spread the sample evenly on the sample dish.

Sample before crushing



Sample after crushing



Due to the low drying temperature of 120°C, nearly no change of the appearance could be detected after drying

Settings: Standard drying 120°C/ AUTO 0.1 %(ΔM)

	Sample weight (g)	Drying time (min)	Dry material content (%)
1.	5.007 g	22:52	32,59
2.	5.009 g	22:31	32,88
3.	5.038 g	21:07	32,65
MW			32.74

## Sample before measurement



Distributed evenly on the tray



Nearly no change

# **3.3.18 Gypsum**

Settings: Standard drying 105°C/ AUTO 0.01 %( $\Delta M$ )

	Sample weight	Drying time	Humidity
	(g)	(min)	(%)
1.	5.346 g	10:15	0,77
2.	5.074 g	10:23	0,75
3.	5.072 g	10:32	0,75
MW			0,76

# Sample before measurement



Distributed evenly on the tray



No change

### 3.3.19 Glue for wood

Settings: Standard drying 120°C/ AUTO 0.05 %( $\Delta M$ )

	Sample weight	Drying time	Humidity
	(g)	(min)	(%)
1.	1.333 g	6:21	48.69
2.	1.312 g	13:05	49.24
3.	1.325 g	10:51	48.00
MW			48,64

# Sample before measurement



Distributed evenly on the tray



No change

### 3.3.20 Tobacco

Settings: Standard drying 95°C/ TIME 20min

	Sample weight	Drying time	Humidity
	(g)	(min)	(%)
1.	2.603 g	20:00	9.95
2.	2.612 g	20:00	10.15
3.	2.612 g	20:00	9.92
MW			10.00

# Sample before measurement



Distributed evenly on the tray



No change