Moisture Analysis

Correct Installation
Flawless Operation
Quick Measurement
Precise Results

Procedures
Basic Principles

Guide to Moisture Analysis

Fundamentals and Applications

METTLER TOLEDO

Contents

1. Introduction

Moisture affects the processibility, shelf life, usability and quality of many products such as pharmaceutical substances, plastics and foods. Information about and monitoring of moisture content is therefore very important. Most substances have an optimum moisture content for obtaining the best possible processing results and therefore attaining maximum quality. Furthermore, moisture content impacts on price and there are statutory rules for some products governing the maximum permissible moisture content (e.g. as defined by national food regulations).

This means that trade and industry need to determine moisture content levels. These moisture determinations need to be carried out reliably and at speed so that any interventions in the production process can be made quickly to avoid interruptions. One quick and accurate way of determining moisture is thermogravimetric measurement using a Halogen Moisture Analyzer: The sample is weighed and heated with an infrared radiator (halogen lamp). The loss in weight is continuously recorded and drying ends once a defined criterion is reached. The moisture content is automatically calculated from the difference in weight (see 3.1 "Measurement principle").

During thermogravimetric measurement, the loss in mass cannot just be selectively assigned to a loss of water because substances other than water may evaporate when heated. This is why we speak of moisture content when using thermogravimetric procedures (see 5. "Technical terms").

2. Structure and content of this brochure

This guide should assist you in determining moisture with the Halogen Moisture Analyzer. It provides information about the key points which are important for working with this instrument and helps you to quickly, reliably and effortlessly determine moisture content.

As well as providing information about installation, location, and the care and handling of samples, the brochure illustrates how you can find the optimum settings for your sample (see 3.3.1"Method development"). You will be able to quickly and easily use the Halogen Moisture Analyzer to trace the moisture values determined with a reference procedure (e.g. oven). You will also find useful tips on how you can obtain outstanding results with special samples, such as liquids or substances which form a skin.

This guide is supplemented by information on method validation, several application examples and a brief comparison of different technologies which can be used to determine the moisture of substances.

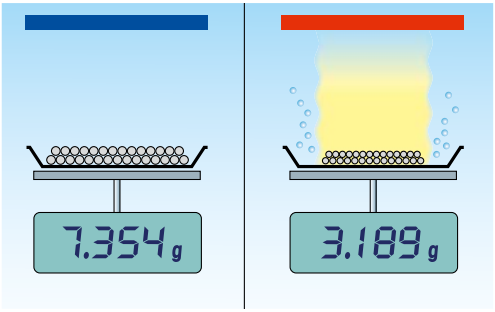
3. Moisture determination with the Halogen Moisture Analyzer

3.1. Measuring principle

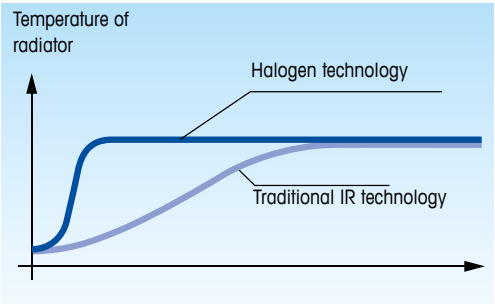
The way in which the moisture is determined using the Halogen Moisture Analyzer is explained here. This includes the method of drying (heating the sample using thermal radiation) and the principle of the switch-off criterion.

The Halogen Moisture Analyzer

The Halogen Moisture Analyzer works along the lines of the thermogravimetric principle, i.e. the sample's start weight is recorded, then a halogen radiator dries it while an integrated balance continually records the sample weight. The total loss in weight is interpreted as the moisture content.

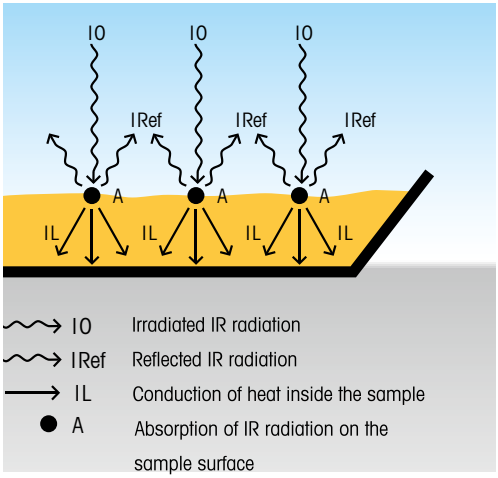


Drying with the halogen radiator is a further development of the infrared drying method. The heating element consists of a glass pipe filled with halogen gas. As the mass of the halogen radiator is very low compared with that of a conventional infrared radiator, the maximum heating output can be reached quickly and outstanding controllability is achieved. In combination with the gold-plated reflector, this ensures an optimum, even distribution of the thermal radiation over the entire sample surface. This is indispensable to achieve repeatable results.

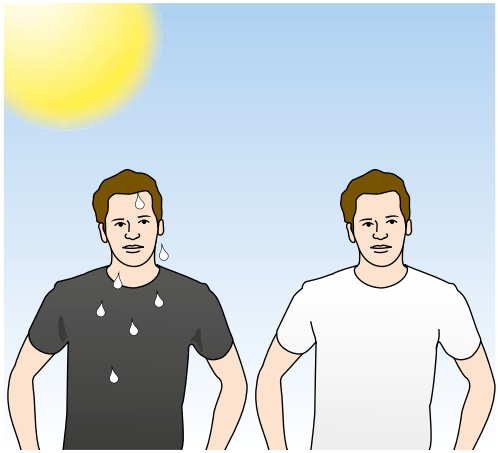


The drying process

In contrast to the traditional oven in which the sample is heated using convection and dried over a long period of time, the sample in the Halogen Moisture Analyzer absorbs the infrared radiation (thermal radiation) from the halogen lamp and, as a result, heats up very quickly.



Different substances have different absorption characteristics. These depend primarily on the color and the material. You should therefore ensure that the sample is homogeneous and of even granulation. Smooth and light surfaces usually reflect infrared radiation more, so less energy is absorbed and the sample warms up less. This means that the absorption characteristics of a sample influence the effective sample temperature.

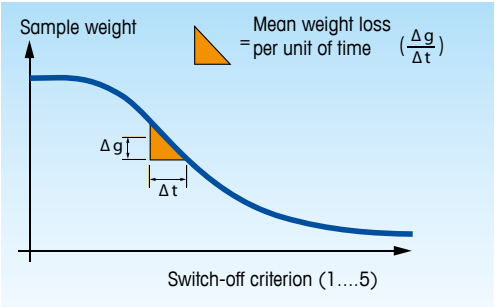


You will be familiar with this absorption effect: If you stand in the sun wearing a black shirt, you will get much hotter than if you were wearing a white one. You should therefore select a slightly lower measurement temperature for a dark sample than for a light sample.

The switch-off criterion

The switch-off criterion (AK) determines the point at which measurement with the Halogen Moisture Analyzer is automatically ended and the result displayed. The Halogen Moisture Analyzer offers two different kinds of switch-off criteria: A time-controlled form of switching off or the decrease in weight per time unit. The integrated balance continually determines the loss in weight of the sample during drying. If the loss in weight (Δ g) falls below the prescribed figure over a certain time (Δ t), the drying process is terminated at this level of dryness and the result displayed. You can select from 5 pre-defined switch-off criteria.

AK 1 (1 mg/10 s): Suited to quick trend measurements
AK 2 (1 mg/20 s): Intermediate level
AK 3 (1 mg/50 s): Standard setting, suited to most types of sample
AK 4 (1 mg/90 s): Intermediate level
AK 5 (1 mg/140 s): Suited to samples that dry slowly and have a low moisture content (e.g. plastics)



The switch-off criterion selected influences the measurement period and measurement accuracy. The drying process will be completed in the shortest time if AK 1 is selected, but the drying is then often not fully finished and repeatability is reduced. By selecting the switch-off criterion you optimize the duration of the measurement period against the required accuracy of the measurement result.

3.2. Installation

3.2.1. Location of the Halogen Moisture Analyzer

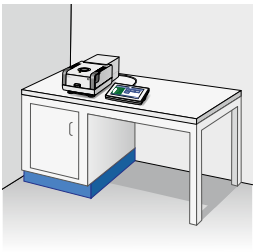
Since moisture measurement using the Moisture Analyzer is based on a high-precision weighing procedure, accuracy and repeatability are closely linked to the instrument's location. To ensure that your Moisture Analyzer works under the best conditions, please observe the following guidelines:

Weighing bench

- Stable (lab bench, stone bench)
  Your weighing bench should not sag when work is carried out on it and should transfer as few vibrations as possible.
- Antimagnetic (no steel plate)
- Wall or floor installation
  The bench should either stand on the floor or be secured to the wall, but not both at the same time otherwise vibrations from both sides will be transferred.

The weighing bench must be stable enough so that the weight display does not change when you lean on the table or step up to the workstation.

Work room



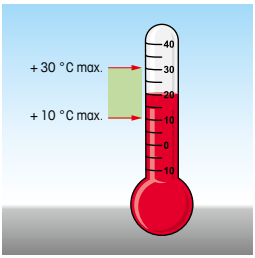
- Vibration-free
- Free from drafts

Place the bench in a corner of the room. These are the most vibration-free areas of a building.

Free space

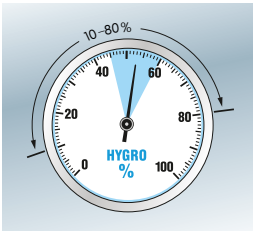
- Ensure sufficient free space around the instrument to prevent a build-up of heat and overheating (approx. 1 m of free space above the Moisture Analyzer).
- Ensure sufficient distance from combustible materials.
- Ensure sufficient space from other sensitive measuring devices.

Temperature



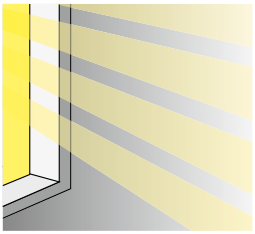
- Keep the room temperature as constant as possible.
  Weighing results depend on temperature!
- Do not place the Moisture Analyzer near heaters or windows (thermal radiation).
- The room temperature should be between 10 °C and 30 °C.

Atmospheric humidity



- Ideally, the relative atmospheric humidity (% RH) should be between 45 and 60%. Balances should never be operated above or below the measuring range of 10 to 80% RH.

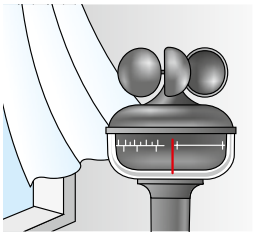
Some samples are very hygroscopic, i.e. they take moisture out of the surrounding air. You should therefore try to keep the relative atmospheric humidity as constant as possible to ensure good repeatability of results.



Light

- If possible, place the Moisture Analyzer on a window-free wall.
  Direct sunlight (= heat) will influence the weighing result.

Note: Active cooling protects the weighing cell from the heat produced by the halogen radiator. However, the weighing cell is not protected from thermal radiation at the sides and direct sunlight can therefore influence the weighing result.



Air flow

- Do not place the Moisture Analyzer in the air flow of air conditioners or devices with ventilators, such as computers or large laboratory devices.
- Place the Moisture Analyzer at a sufficient distance from radiators.
  As well as the effects of temperature radiation, these also have strong and interfering air flows.
- Do not place the instrument next to a door.
- Avoid places with high traffic (drafts).
- If possible, keep the windows closed to prevent drafts.

3.2.2. Commissioning

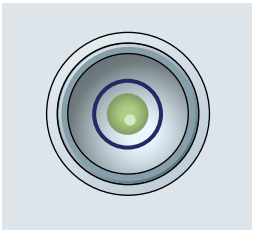
Halogen Moisture Analyzers are high-precision measuring instruments.
If you follow the tips provided below you will establish a good basis for reliable results.

Switching on

- Do not disconnect the Moisture Analyzer from the power supply to allow a thermal balance to be reached inside the instrument.
- If you switch the instrument off, use the On/Off button to do so. The Moisture Analyzer then goes into Standby Mode.

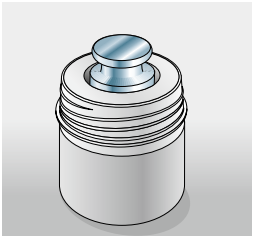
Tip: When first connected to the power supply, we recommend an acclimatization period of at least 30 minutes.

Leveling



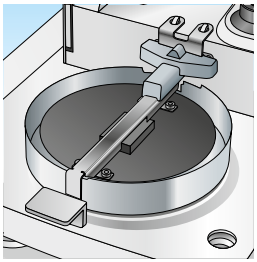
- Align the Moisture Analyzer.
  The instrument has feet screws and a leveling check (level indicator) for accurate alignment. When the level indicator is in the center, the device is horizontal.
- Check that the device is stable.

Adjustment



- Adjust the balance and heating module regularly, especially

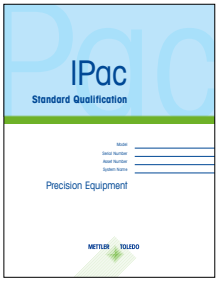
  - when you operate the Moisture Analyzer for the first time,
  - after changing location,
  - after major changes to room temperature,
  - after leveling (balance only).



- Adjust the device under operating conditions.
- The frequency of adjustments depends on your quality requirements and safety risks.

Note: Only adjust the Moisture Analyzer after the acclimatization period and when cold (both the device and the temperature adjustment kit), e.g. in the morning before the first measurement. In this way you ensure that all adjustments are made with the equipment in the same state.

Tip: Use weights and thermometers with a calibration certificate. This is the only way to ensure the traceability of your test equipment.



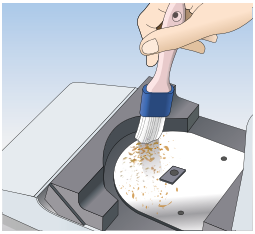
Equipment qualification

Service engineers from METTLER TOLEDO will assist you with installation, qualification and perfect configuration of your instrument. This includes a calibration certificate, user instructions and a definition for routine tests and therefore guarantees immediate use of the Moisture Analyzer and reliable routine operation.

3.2.3. Routine operation

To ensure precise measurement results the following information about care, calibration intervals and maintenance should be observed:

Caring for the Moisture Analyzer



- Keep the sample pan area clean (e.g. using a brush).
- Clean dirt off the temperature sensor and protective glass on the heating module (for details, see operating instructions).
- Use a mild cleaning agent (e.g. glass cleaner) to clean the instrument and sample pan area.
- Replace the halogen radiator reflector if the reflector layer is damaged.

Calibration and maintenance intervals

- By regularly calibrating (testing) and if necessary adjusting the heating module, you will ensure consistent and reproducible heat output for the entire lifetime of your instrument. We therefore recommend that you define test intervals for testing the weighing unit and heating module (depending on risk).
- To ensure quality moisture results at all times, METTLER TOLEDO offers SmartCal. This unique temperature sensitive substance with a known moisture content is used in one single test to quickly and easily verify the instrument's overall functionality. The SmartCal test is based on a regular measurement with a moisture analyzer. Refer to the SmartCal User Guide for more information.
  www.mt.com/smartcal-userguide
- Annual maintenance performed by the METTLER TOLEDO service team will guarantee the quality, measurement accuracy and value retention of your Halogen Moisture Analyzer.

www.mt.com/smartcal

Tip: The Halogen Moisture Analyzer HX204 allows you to freely select the temperature of the adjustment at between 80 °C and 200 °C and the test at between 50 °C and 200 °C. This allows you to test the heating output at your specific drying temperature (e.g. 130 °C).

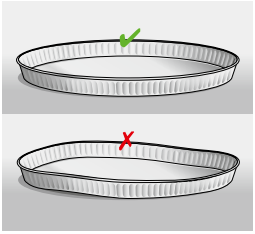
3.2.4. Sample Handling

Safety information

- Some samples require particular care as they can harm people or damage objects. These include combustible, explosive, toxic or corrosive substances and/or samples which release these substances when dried/heated.
- Never dry combustible or explosive substances.
  If in any doubt, use small sample volumes (max. 1 gram) and low temperatures.
- Carry out a risk analysis (e.g. with regard to risk of explosion, combustibility, toxicity and corrosiveness of sample and the vapors released when heated).
- If necessary, work in a fume cupboard (adjustment should then be undertaken there).

Warning: The surface temperature of the radiator is higher than the measurement temperature and hence may ignite combustible vapours as they pass by.

Note: Please note that the user bears all responsibility and liability for damage resulting from the use of the aforementioned types of samples.



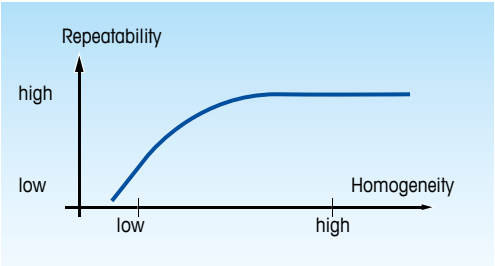
Sample pans

- Only use clean sample pans for moisture determination.
- Do not use deformed sample pans.

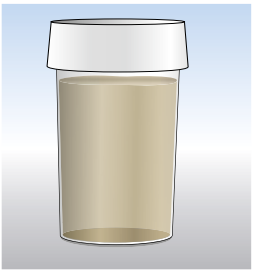
Tip: Using single-use aluminum sample pans guarantees reliable measurements free from the influence of residue remaining from previous samples or cleaning agents. These aluminum sample pans are also available in a reinforced version. These are suitable for samples which contract when drying and may deform the pan.
Please dispose of the used pans properly.

Sampling

The way in which samples are taken has a major impact on the reproducibility of the measurement results:

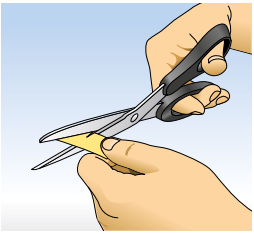


- Representative of total volume
- Ensure homogeneity (mixed well), e.g. mix and stir the total volume first etc.
- Sufficient sampling
- No addition or removal of moisture when taking samples (work as quickly as possible)
- If measurements are not being taken straight away: Store in airtight container without an air cushion (fully filled)



Sample preparation

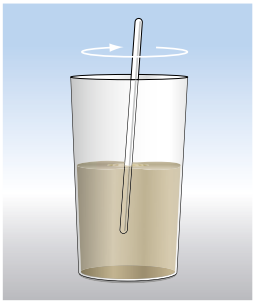
The correct preparation of samples once they have been taken is also key to repeatable and reliable results.



- Ensure even granulation (particle size).
- If necessary increase the sample surface area by breaking up the sample. This will ensure a better and faster release of moisture during drying (faster diffusion of moisture to the surface).
- The sample should not be heated at this stage as this would cause moisture to be lost during preparations.

Mechanical crushing can be carried out e.g. using a mortar, grinder (water-cooled) or simply by cutting.

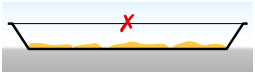
Tip: You can increase the surface area and therefore speed up the drying of liquids by using a glass-fiber filter.

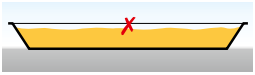


Sample application

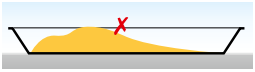
An evenly spread sample results in a homogeneous distribution of heat throughout the product being measured and the moisture can diffuse evenly out of the sample. This generates results which are easier to reproduce.

- Carefully mix your sample before adding to the sample pan.
- Always use the same volume of sample to achieve good repeatability



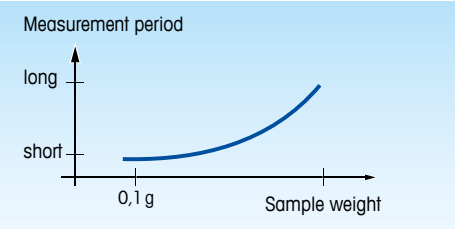


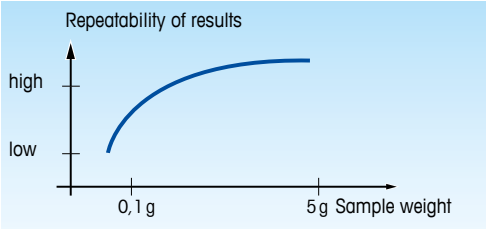
- Use the right sample volume. The pan should be thinly and evenly covered with the sample over the total surface area.



- Spread the sample evenly over the pan (do not build up piles).

Influence of sample weight on repeatability and length of measurement:





A higher sample weight will mean that more water is evaporated and moisture determination will take longer. In addition, too large a sample volume can result in an uneven distribution of heat and thus to less precise results. On the other hand, repeatability decreases (higher standard deviation) as the sample weight decreases:

Standard deviation for 2 g sample[1]: max. 0.05%
Standard deviation for 10 g sample[1]: max. 0.01%

[1]Assuming an ideal sample where all of the moisture can always be removed without causing decomposition (e.g. moist sand). Deviations result from the substance-dependent uncertainty and the repeatability guaranteed by the instrument (in this case: HX204). In reality, differences in measurements occurring in one series of measurements (not ideal samples) may be greater than the values shown in the table.

3.3. Methods and special samples

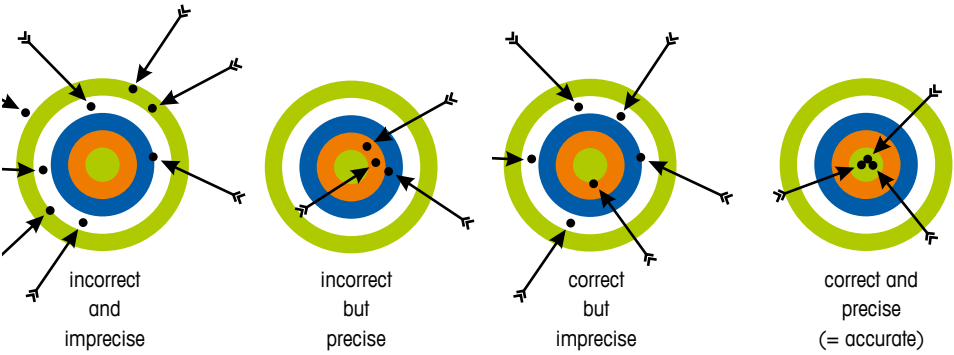
The Halogen Moisture Analyzer is a user-friendly measuring device which allows the moisture content of samples to be quickly and easily determined. There are often statutory requirements, standards customarily used in the trade or internal corporate instructions for substances which define the method of moisture content determination. The oven method or Karl Fischer titration is usually used as the reference procedure.

In such cases, the aim is to obtain the same results with the Halogen Moisture Analyzer as with the reference procedure (or for the deviation from the reference value to be known and reproducible). In order to achieve this, adjustments must be made to the setting parameters such as drying temperature, drying program (see 3.1 "Measurement Principle") and sample weight as well as the handling of the sample. This is known as method development where the aforementioned parameters describe a method. The same method may be used for different substances.

The following chapter describes the basics of how development of a method can be carried out. After this you will find information about how you can work with special samples to achieve accurate measurement results.

It may however be the case that you are not using a reference procedure and therefore have no reference value. The objective of method development in this case is to find parameters with which you gain repeatable (precise) measurement results which you can use to assess the quality of your samples.

You can optimize your measurements in three respects: correctness, precision (repeatability) and speed. The diagram here explains the terms correctness, accuracy and precision.



3.3.1. Method development

- Note the basic requirements stated in chapter "3.2 Installation".
- We would also recommend running a method development over a short period of time. This ensures that the sample doesn't change in the meantime and thereby impact on the measurement result.
- It is best to develop the method under the operating conditions at the place where the Moisture Analyzer is installed.
- Take and prepare your samples in the same way as for the reference procedure (typically drying oven).
- Note how to handle samples (see 3.2.4 "Sample handling").

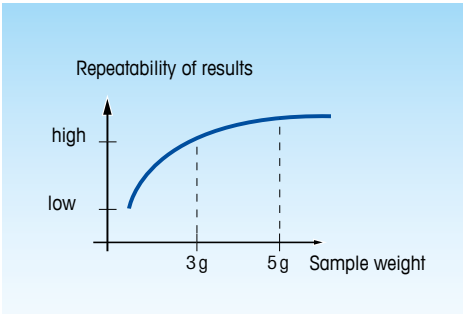
1. First measurement: Reaching the reference value

Look for existing methods which have been specially developed for Moisture Analyzers from METTLER TOLEDO. These will give you an idea of possible suitable settings for an initial measurement.

- Perhaps you already have a method for a similar sample?
- Refer to METTLER TOLEDO's application database, this contains lots of existing methods.

www.mt.com/moisture-methods

If you find a similar sample, use the parameters of this method for the initial measurement with your substance. If you don't find any similar methods, use the following basic settings:

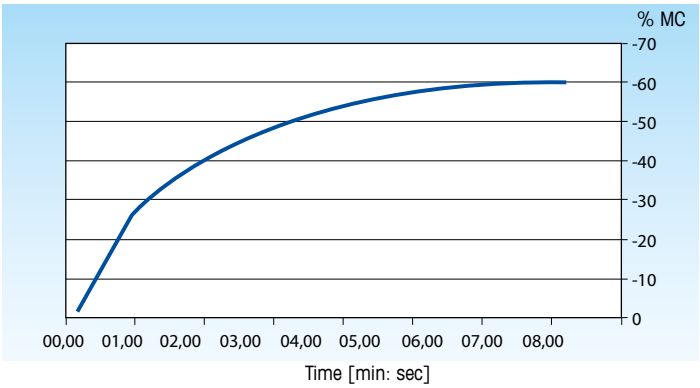


- Standard drying
- Temperature:
  1) Temperature of oven method
  2) If there are no oven methods available:
     Organic (T-sensitive) sample: 105 °C,
     Inorganic (not T-sensitive) sample: 150 °C
- Switch-off criterion 3 (1 mg/50 s)
- 3–5 g sample (spread evenly over the sample pan)

Take an initial measurement and record the measurement trend.

Tip: Consult the notes for special samples such as liquid samples provided under 3.3.2 "Special samples" and the safety declarations under 3.2.4 "Sample handling".

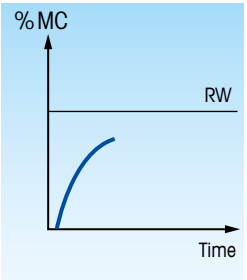
Tip: Displaying data and drying curves is very easy with HX204, HS153 and HC103. By using the comfortable function of exporting results as Excel compatible CSV files.



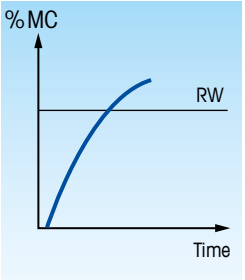
2. Analysis of drying characteristics

- Look at the sample and evaluate it: Severe discoloration or melting indicate that you have selected too high a temperature.
- Read off the result and look at the drying curve to evaluate the drying behavior.

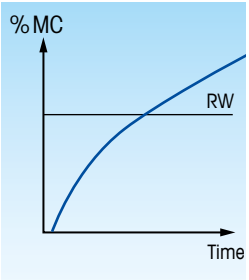
RW = reference value



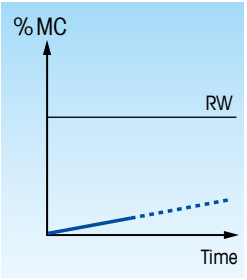
Value falls below reference value: Increase the temperature.



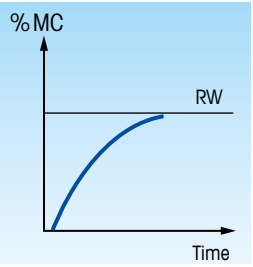
Reference value is exceeded: Reduce the temperature.



The Moisture Analyzer doesn't switch off. The switch-off criterion is not fulfilled because decomposition reactions are taking place: Reduce the temperature.



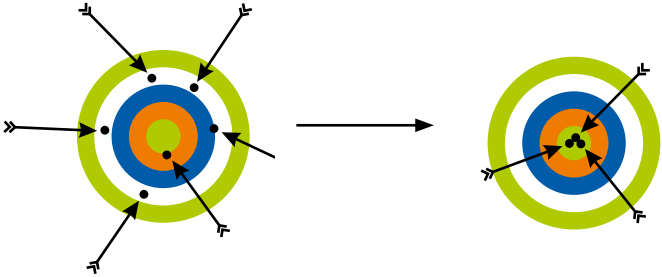
The switch-off criterion is not fulfilled. The measurement is stopped by the user because either no moisture is released or is escaping very slowly: Increase the temperature.



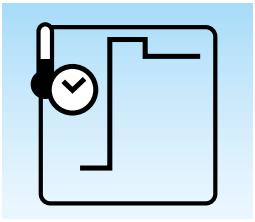
Adjust the temperature until you reach the reference value (correctness).

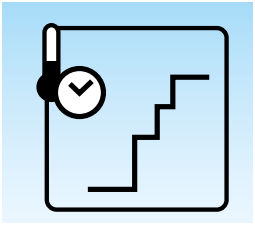
Now check repeatability by, for example, taking three measurements and calculating the mean value and standard deviation.

3. Optimizing precision



- Increase the volume, especially for samples with low moisture levels.
- Optimize the taking and preparation of samples. In particular, ensure a good homogeneity and even distribution of the sample.
- Use the more accurate switch-off criterion 4 (1 mg/90 s) or 5 (1 mg/140 s)





4. Optimizing speed

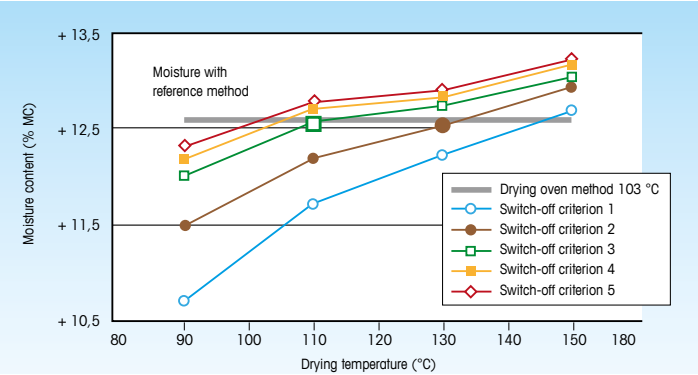
The following information will help you gain your measurement results faster. However the factors stated may also have an impact on the precision of your results:

- Reduce the sample weight.
- Increase the surface area of your sample.
- Use the rapid drying program if the moisture content is higher than 30%.
- Step drying with the HX204 can be used in a similar way to rapid drying. The benefit of this process is that you can select the temperature and period of increase as you want.

Test measurement

The top models HX204 and HS153 offer the test measurement function. This auxiliary function helps you find a suitable switch-off criterion. Select a temperature and sample volume for drying and run a test measurement.

You can see directly on the displayed drying curve when each switch-off criterion was reached and the measurement result at that time. If you take this test measurement at different temperatures, you can establish a suitable switch-off criterion for a particular temperature at which you reach the reference value (see graph).



In this instance test measurements were taken with fine flour at 4 different temperatures and the results of the switch-off criteria compared against the drying temperature. The aim is to reach the reference value from the determination with the oven at 103 °C. You can clearly see that selecting 90 °C would be too low a temperature and 150° too high a temperature. The points at which the five curves intersect with the reference lines show possible settings for the temperature and switch-off criterion combination. Possible settings are e.g. 110 °C with switch-off criterion 3 or 130 °C with switch-off criterion 2. Switch-off criteria 3 and 4 usually produce results which are easy to repeat. Switch-off criterion 2 should only be used if the time factor is more important than repeatability. As mentioned before, best practice is to perform multiple measurements with the selected method to confirm satisfying repeatability.

3.3.2. Special samples

Certain samples need a special procedure for quickly and correctly determining moisture. This section contains information about how you can work with such samples to optimize moisture determination.

Paste-like, greasy and melting samples

- Use the glass-fiber filter to increase the surface area of the samples.
- Tare the filter with the sample pan and then place the sample on it.

The liquid contained in the substance is evenly spread over the whole area of the filter in the filter's capillaries. This increases the sample's surface area and the moisture can then evaporate quickly, easily and fully.

Liquid and very moist samples

- Use the glass-fiber filter.
- Tare the filter with the sample pan and then place the sample on it.
- Rapid drying is suited to samples with a very high moisture content (> 30%). In this process, the target temperature is exceeded by 40% for 3 minutes to accelerate the measuring process.
- Step drying (HX204 only) can be used as an alternative to rapid drying. Here the duration of the temperature increase and the temperature are freely selectable.

Because of surface tension, liquid samples often form drops on the sample pan. This prevents a fast drying process because evaporation takes place over a limited liquid surface area. Using the glass-fiber filter spreads the sample over a large area. This often more than halves the duration of the measurement and better repeatability is also achieved.

Samples with very low moisture content

MC<1%

- Use a sufficiently high sample weight (e.g. 20–30 g).
- If the moisture only escapes very slowly, use switch-off criterion 5 (1 mg/140 s).
- Use a high resolution (0.1 mg) (HX204 only).
- Use standby temperature (HX204 only).
- Preheat sample pan for 1 minute at standby temperature and then tare. This improves the repeatability of the results.

Substances which form a skin and are sensitive to temperature

- Select the gentle drying program
- Use the glass-fiber filter (tare the filter along with the sample pan and then cover the sample with the filter from above).

This means that the sample is covered by the glass-fiber filter and therefore shielded from IR radiation to prevent burning. This results in the sample being warmed more gently. Using gentle drying (slow heating to target temperature) increases this effect. We would also recommend this process for substances that form skins or crusts because the skin or crust impairs moisture evaporation. The formation of crusts is prevented by covering the sample with the glass-fiber filter and gentle drying.

Samples containing sugar

- Select moderate temperature. Samples containing high levels of sugar will caramelize on the surface (above around 110 °C) and thereby prevent moisture from escaping.
- Use gentle drying.
- Apply thin coat of sample.

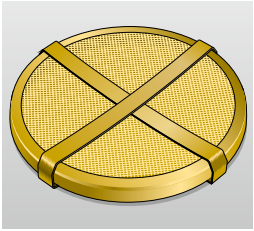
Samples with highly volatile components

- Work with manual start.
- Use gentle drying if necessary.
- If the vapors are toxic, run a risk analysis and work under a fume cupboard.
- If samples or vapors are highly flammable, run a risk analysis and, if necessary, do not use the Halogen Moisture Analyzer for drying.
- Standardize the processing of the samples (the weighing-in period before the start of the measurement should always be the same) to improve repeatability.

Highly volatile samples which contain solvents (note Safety information, see 3.2.4 "Sample handling") may lose weight before you start the drying process. This will distort the result. The sample should therefore always be processed in the same way (e.g. speed) so that any deviation is as low as possible.

Rapid heating and therefore rapid evaporation of the sample may also result in condensation forming under the sample pan. Using the manual start or gentle drying may reduce condensation levels as well as prevent a high concentration of highly volatile vapors.

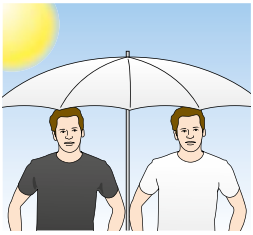
Bulky or intumescent samples



- Use the sample pan for bulky samples (HA-CAGE)

Intumescent samples or those which are bulky, such as textiles, may produce incorrect measurement results as they but up against surrounding parts of the Halogen Moisture Analyzer. We recommend that you use the HA-CAGE to dry such samples.

Unevenly colored samples



- Cover the sample with the glass-fiber filter.

Because of the different absorption characteristics, the sample heats up to different levels in different areas. The glass-fiber filter ensures even warming.

Plastic granules

- High resolution (0.1 mg, HX204 only)
- Standby temperature (100 °C)
- SOC delay: 5 min
- Switch-off criterion 5
- 30 g sample weight
- Heat the aluminum sample pan for 1 minute at standby temperature, and then tare it.

Plastic granules usually require a very low moisture content (e.g. 0.1%) for processing (e.g. injection molding). A high sample weight of 30 g is therefore needed to obtain good repeatability. Step drying is used because plastic loses moisture very slowly. The sample is heated for the 5 minutes of the first step without the switch-off criterion becoming active. The second step is not needed and is therefore set to 0 minutes. The switch-off criterion is only active once these first two steps have been completed. Using step drying therefore prevents the measurement being aborted prematurely.

3.4. Validating a method

If you need to demonstrate that the Halogen Moisture Analyzer produces the same results as the reference method, the following information will be of use to you. One possible procedure is presented using the example of ethyl cellulose. Depending on the industry in question, the validation requirements may differ.

1.) You perform a method development and determine the parameters with which you can measure the correct moisture content of your sample. In other words, you obtain both correct and precise results when compared to the reference method (in this case: drying oven).

	Drying oven method according to USP/Ph. Eur	Halogen Moisture Analyzer HX204
Sample weight	1 g	1 g
Temperature	105 °C	105 °C
Drying program	–	Standard
Switch-off criterion	–	5
Moisture content		
(mean value, n=6)	1.68%	1.68%
Standard deviation	0.01%	0.03%

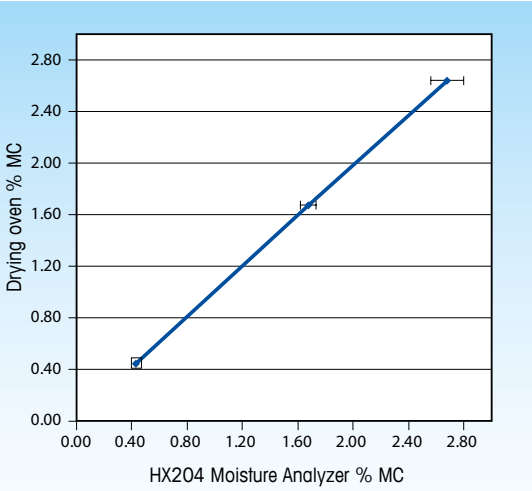
2.) Now check the linearity for a particular range.

Here, you want to demonstrate that the Halogen Moisture Analyzer produces the same values as the reference procedure even if the substance has a different moisture content to the one used for method development.

To do this, condition, for example, two further moisture contents for your sample. In this example dry the ethyl cellulose to 0.4% moisture content and moisten it to 2.6% moisture content.

Now measure the moisture content of the two conditioned samples both with the reference method and the method developed for the Halogen Moisture Analyzer. Calculate the mean values and standard deviations of the results of both procedures and evaluate whether the results are within your defined tolerances. You can draw a diagram like the one below.

The validation graph clearly shows that the results of the moisture measurement recorded with the HX204 match those values obtained using an oven. However, the results are obtained up to 10 times faster.



3.5. Application examples

The application experts of METTLER TOLEDO have prepared many ready-made moisture methods for the use with METTLER TOLEDO moisture analyzers for a variety of samples tested in food, pharmaceutical, plastics and other industries. These methods are referenced, thus matching the result from the drying oven (or Karl Fischer titrator). The online application database allows you to search and find the method that suits best to your needs:

Note: for each method in the database, you receive:

1. The method datasheet as a PDF which details the settings to use. Page 2 of the datasheet shows the drying curve and the comparison of the results with those of the drying oven reference method.
2. An XML file for fast, easy and error-free upload of the method parameters to your moisture analyzer via USB. (HX204, HS153 and HC103 moisture analyzers)

www.mt.com/moisture-methods

Method Datasheet

Moisture Determination

Coffee, ground

Sample Description	
Analysis	Moisture Content
Sample	Coffee, ground
Sample Characterization	Powder of ground coffee beans

Method Parameters		
	Halogen Moisture Analyzer	**Drying Oven**
Instrument model	HB43-S	Convection Oven
Sample weight	3 g	5 g
Temperature	120 °C	103 °C
Drying time	SOC (~5 min)	360 min
Drying program	Standard	Not applicable
Switch-off criterion (SOC)	3 (1 mg/50 s)	Not applicable
Sample preparation and procedure	Stir sample, use spatula to distribute evenly on the sample pan.	Dry weighing container with cover in oven (103°C, 1h), allow to cool in desiccator, weigh. Stir sample, add to container, weigh. Dry in oven (5 h), allow to cool down to room temperature in desiccator, weigh. Dry sample again in oven (1 h), cool in desiccator, weigh.
Comments		

METTLER TOLEDO

4. Overview of different technologies for moisture determination

Various measurement processes have been developed for determining moisture content. The table below shows a selection of typical measurement technologies and describes the advantages and disadvantages of these procedures.

	Drying oven*	Infrared drying	Halogen drying *	Microwave drying
Procedure	Thermogravimetry	Thermogravimetry	Thermogravimetry	Thermogravimetry
Measuring method	Heating of sample by convection. Determination of mass before and after drying.	Heating through absorption of IR radiation from a metal radiator. Continual determination of mass during drying process.	Heating through absorption of IR radiation from a halogen radiator. Continual determination of mass during drying process.	Heating through absorption of microwaves. Determination of mass before and after drying.
Advantages	• Often reference procedure • Even distribution of heat over sample • Several samples can be determined at the same time • Large sample volumes possible	• Short measurement period (typically 5–15 min.) • Large sample volumes possible • Simple handling • Compact instrument	• Good temperature control • Even distribution of heat over sample • Excellent cold/warm starting characteristics • Quick measurement (typically 3–10 min.) • Large sample volumes possible • Simple handling • Compact instrument	• Very quick (typically 2–5 min.) • Large sample volumes possible
Disadvantages	• Very long determination period (hours) • Substances other than water may evaporate • Prone to errors because of the high level of handling involved	• Substances other than water may evaporate • Difficult to control	• Substances other than water may evaporate	• Not suited to substances with a low moisture content • Moderate temperature control • Substances other than water may evaporate

Karl Fischer titration*	**Calcium hydride**	**Microwave spectroscopy**	**Infrared spectroscopy**	**Refractometry***
Chemical	Chemical	Spectroscopy	Spectroscopy	Optical
Iodine is titrated and consumed in the presence of water. This consumption of iodine is proportional to the water content of the sample.	Hydrogen is released in the presence of water. The volume of hydrogen is proportional to the volume of water and can be measured as a change in pressure or using a hydrogen electrode.	Measurement of absorption/reflection of microwave radiation	Measurement of absorption/reflection of IR radiation	Measurement of refractive index
• Reference procedure • Water-selective • Suited to extremely low moisture levels	• Water-selective • Moderately fast (15–30 min.)	• Very short measuring time (less than 1 min.) • Online measurement possible	• Very short measuring time (less than 1 min.) • Online measurement possible	• Rapid procedure • Little effort • Mobile
• Reagents and lab needed • Wet-chemical procedure (trained staff needed)	• Prone to incorrect working • Reagent is needed	• Substance-specific calibration required • Influenced by: Bulk density and grain size	• Substance-specific calibration required • Measurement of surface moisture only • Depends on temperature and grain size	• Only suited to a few substances (e.g. sugar solutions)

* METTLER TOLEDO instruments available.

5. Technical terms

Moisture (moisture content): In thermogravimetric processes the moisture of a material includes all substances which volatilize during warming and therefore contribute to the material's loss of mass. Alongside water this may also include alcohol or decomposition products. When using thermogravimetric measurement methods (drying using infrared, halogen, microwaves or ovens) no distinction is made between water and highly volatile components.

Dry content: Solid proportion of a mixture made up of solid and liquid substances in relation to the mixture's total mass.

Thermogravimetry / thermogravimetric moisture determination:
Thermogravimetric processes are weighing-drying methods in which the samples are dried until a constant mass (or defined time) is reached.
The change in mass is interpreted as released moisture.

Note: If the substances contain other volatile components in addition to water, the measurement result must not be described as water content. However, if you know the water content of such a sample (e.g. using water-selective Karl Fischer titration), you can determine this value using a thermogravimetric process (e.g. halogen drying) by selecting appropriate drying parameters.

Reference procedure: Measurement process for determining the moisture content which allows for traceability to (statutory) standards. Different components (water, other volatile substances) can be measured depending on the reference procedure used.

Drying oven procedure: Thermogravimetric method for determining the moisture content of a sample. This sample is dried in the oven for a defined period of time at constant temperature. The moisture content percentage is determined from the difference in weight before and after drying. For historical reasons this procedure often forms part of legislation (Food regulations, USP[1)] etc.)

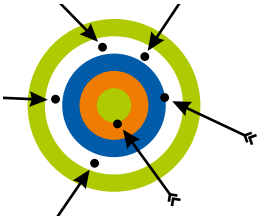
Level of dryness: A sample's level of dryness is the defined decrease in weight (Δ g) during a defined time unit (switch-off criteria) assuming the start weight is always the same.

[1)] USP United States Pharmacopeia: Loss on drying [USP<731>]

Method: A method describes how the correct result is achieved. This includes all the steps required, such as device settings, selection of measurement parameters, preparation and processing of samples.

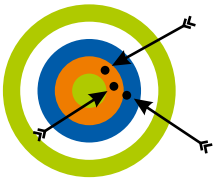
Infrared (radiation): Infrared rays are electromagnetic waves (780 nm to 1 mm) which come after visible light (380–780 nm) in the electromagnetic spectrum. People cannot see these rays but they are perceived as warmth.

Correctness: Qualitative term, describing a judgement of the systematic deviation of measurements. The extent to which the expected value (mean value) of a series of measured values matches the true value of the object being measured ([ISO[2)] 5725] 3.7).



Note: The correctness can be evaluated only when there are several measured values, as well as a recognized correct reference value.

Precision: Qualitative term, describing a judgement of the mean variation of measurements. The extend to which independent measured values obtained under stipulated coniditions match one another ([ISO[2)] 5725] 3.1.2). Precision depends only on the distribution of random deviations and does not relate to the true value of the measurement variable (accuracy).



Example: The ability of a measuring instrument to supply measured values that seldom deviate.

Note: Precision can be evaluated only when there are several measured values.

Repeatability: Extent to which results from a series of measurements of the same measured quantity, carried out under the same measurement conditions, match one another.

The series of measurements must be carried out by the same operator, using the same method, in the same position on the support (sample pan), in the same installation location, under constant ambient conditions and without interruption. The standard deviation of the measurement series is a suitable measurement for

[2)] ISO International Standards Organization

expressing the value of the repeatability. The degree of repeatability is not only a characteristic determined by the Moisture Analyzer. Repeatability is also affected by the ambient conditions (drafts, temperature fluctuations, vibrations), by the sample and by the consistent preparation of the samples.

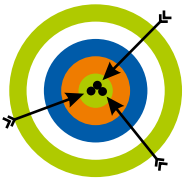
Mean value:

$$\overline{x} = \frac{1}{n}\sum_{i=1}^{n} x_i$$

x_i = i–th result of the series
n: number of measurements, usually 10

The standard deviation s is used as a measure of the repeatability.

$$s_x = \sqrt{\frac{1}{n-1}\sum_{i=1}^{n}\left(x_i - \bar{x}\right)^2}$$



Accuracy: Qualitative name for the degree to which test results approximate to the reference value, which can be the correct or expected value, depending on the definition or agreement [DIN[3)] 55350-13].
Accuracy in repeated measurements requires correctness and precision. This does not necessarily apply to an individual measurement.

Reproducibility: The degree of approximation between the measured values of the same measured variable, even though the individual measurements are carried out under different conditions (which are specified) with regard to

- the measurement process
- the observer
- the measuring device
- the measuring location
- the conditions of use
- the time

[3)] DIN German Institute for Standardization

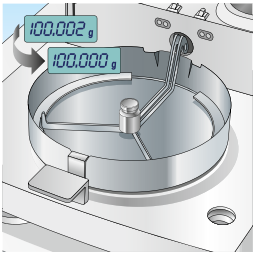
Qualification (Equipment Qualification): Check and documentation of whether the equipment and the technology used are suitable for the intended task.
The following stages are combined under Equipment Qualification (EQ): Design Qualification (DQ), Installation Qualification (IQ), Operational Qualification (OQ), Performance Qualification (PQ) and Maintenance Qualification (MQ).

DQ: Definition of the requirements of equipment specifications and documentation of the decision-making process.
IQ: Assurance and documentation that the equipment supplied corresponds to the ordered specifications and that the equipment is installed correctly and the surroundings are suitable for operation.
OQ: Documentation of the equipment's functionality according to the defined specifications.
PQ: Documentation that the equipment satisfies the requirements and specifications of routine operation.
MQ: Description and documentation of all the measures needed for the planned maintenance, periodic calibration, care of the instrument and user training.

Validation: The provision of evidence and its documentation to show that a device (or a method) delivers the result expected.

Calibration (testing): Determining the deviation between the measured value and the true value of the measurement variable under specified measuring conditions without making any changes (adjustment).

Adjustment: Adjustment of the measuring device such that the measurement is correct: First the deviation is noted between the measured value and true value (calibration), then the corresponding correction is undertaken.



6. Index

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8. Accessories

HX204/HS153/HC103/HE73/HE53	Part No.	Quantity
Sample handling		
Aluminum sample pan	13865	80
Prof. aluminum sample pan (extra strong)	11113863	80
Textile cage for bulky samples (HA-CAGE)	214695	1
Glass fibre filters	214464	100
Printer		
P-56RUE printer	30094673	
RS-P25 printer with RS232 interface	11124300	
Printer paper (for P25)	72456	5 rolls
Printer paper (self-adhesive)	11600388	3 rolls
Printer ribbon, black	65975	2
Quality management		
HX/HS/HC certified temperature adjustment set	30020851	
HE certified temperature adjustment set	30134141	
cSmartCal Reference substance (certified, set of 24)	30005791	
cSmartCal Reference substance (certified, set of 12)	30005793	
SmartCal Reference substance (set of 24)	30005790	
SmartCal Reference substance (set of 12)	30005792	
StarterPac cSmartCal (all accessories, protocols and 12 tests)	30005918	
Certified adjustment weight 100 g (HX/HS/HC)	11119531	
Certified adjustment weight 50 g (HE)	11119460	
Accessories		
HX/HS protective cover for terminal	30003957	1
HX/HS stand for terminal	30018474	1
HX/HS dust filter	30020838	50
HX/HS printer support	30066692	1
HC dust filter housing	30216118	
HC dust filter	11113883	50

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