Cooperation Centre for Scientific Research Relative to Tobacco

CORESTA Guide N° 6

A User Guideline for the Use of Balances for Cigarettes and Cigarette Related Products

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Physical Test Methods Sub-Group

CORESTA TECHNICAL GUIDE N° 6

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A User Guideline for the use of balances for cigarettes and cigarette related products

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0. GLOSSARY AND ABBREVIATIONS

°C	Degrees Celsius
HTP	Heated Tobacco Product
RH	Relative humidity
ISO	International Organization for Standardization
CORESTA	Cooperation Centre for Scientific Research Relative to Tobacco
CE	Conformité Européene
Artificial/Dummy Cigarette	Test piece with the similar physical properties to a cigarette but constructed so that its primary physical parameters (i.e. weight, length, circumference, pressure drop and ventilation) are stable and repeatable.
Shewhart Chart	A statistical tool in the form of a control chart intended to assess the nature of variation in a process

1. INTRODUCTION

1.1 Purpose

The amount of tobacco in cigarettes and of filtertow in filter rods have a direct effect on the smoking performance of the assembled cigarette and are a principal measure for the quality control of cigarette manufacture. The same applies to the use of heated tobacco products (HTPs). This guideline is intended as a reference for manufacturers of cigarettes and cigarette filter rods as to the best practice for weighing, so as to obtain consistently the most accurate and most reliable measurements despite the many external influences that can affect the measured value.

1.2 Outline

The following information is based on the work done in the CORESTA Monitoring and Maintenance of Physical Test Methods Sub-Group, since 2011 called the Physical Test Methods (PTM) Sub-Group. In order to maintain the validity of weighing, attention needs to be given to all of the factors that influence accuracy. This can only be done by understanding the nature and magnitude of such influences and how they might be minimised and controlled.

This revision includes some minor editorial and technical updates and adds reference to the new generation of heated tobacco products.

2. THE MEASUREMENT PROCESS

2.1 Introduction

Physical measurements on cigarettes and filter rods bring together several factors, including samples, instruments, calibration standards and various procedures and techniques. Each of these factors must be optimised if it is not to disturb or corrupt the measurement process. Hence, it is important to understand the effect that external influences might have on each of these factors and the influence of each factor on the overall process. Where optimisation is not practical or possible, allowance needs to be made for potential errors incurred.

Though it is the mass of cigarettes and filters, expressed in units of milligram, gram or kilogram, that is measured with the balance, however, the noun weight is often used in this context. Weight is the gravitational force that is acting on the body which mass is measured. It is expressed in units of Newton and should not be mixed up with the mass in units of kilogram.

This guide is intended as a supplement to the manuals provided by the supplier of the balance or the measurement device.

2.2 Examples of use of balances

Examples of use of balances in the tobacco industry for cigarettes or cigarette related products include:

- Weighing of cigarette / HTP
- Weighing of filter rods
- Weighing of filter tips and filter segments
- Analytical (laboratory) weighing
- Weighing of tobacco bales

- Weighing of tobacco for moisture determination
- Weighing of tobacco samples for density determination
- Weighing of filter rods for plasticiser determination

2.3 Factors affecting the measurement

The measured value is affected by several factors:

- ambient conditions at the time of measurement (e.g. temperature, humidity, vibrations, air flow, ...)
- moisture content at the time of measurement, which is likely to depend on the time and ambient conditions during conditioning, and sample temperature
- potentially the rate of re-equilibration if the measurement conditions (particularly humidity and temperature) are different from those used for conditioning
- temperature of standards and ambient conditions at the time of calibration checks
- temperature of standards and ambient conditions at the time of instrument adjustment

In addition, any errors within the instrument that affect the measurement and any errors in the marked value of the calibration standard (e.g. damage or contamination) as well as operational errors can have an effect on the measured value.

Finally, when expressing or interpreting measurement results, it is necessary to understand the capability and uncertainty of the measurement process. This will ensure that valid inferences are drawn from the results. To maintain the validity of measurements, the full measurement process should take account of all of these influences.

These factors are discussed in the following chapters.

2.3.1 Instrument related factors

- Measurement range
- Accuracy / Sensitivity
- Temperature coefficient
- Linearity
- Sensitivity to loading position
- Drift
- Display of result
- Similarity in value of calibration standard and expected measurement result
- Use of gloves or tweezers for small calibration weights
- Location
- Vibrations
- Levelling
- Temperature (warming by sunlight)
- Air velocity (e.g. by climatisation)
- Gravity
- Electromagnetic disturbances

Definitions:

The sensitivity is the change in a displayed value divided by the change in the load signal generated by the mass on the pan. If a balance or scale with a digital display has been correctly adjusted, the sensitivity must always be exactly 1. The equation for the sensitivity is given by the number of scale intervals that correspond to the change in load.

A sensitivity error is caused by using inappropriate calibration weights to adjust a balance or scale. The sensitivity error is always indicated as a relative number.

The temperature coefficient characterises the effect of changes in the zero point or sensitivity of the balance due to changes in temperature. It is expressed in weight deviation per Kelvin.

The linearity error (usually referred to as linearity) indicates how much a balance or a scale deviates from the theoretically linear slope of the characteristic calibration curve.

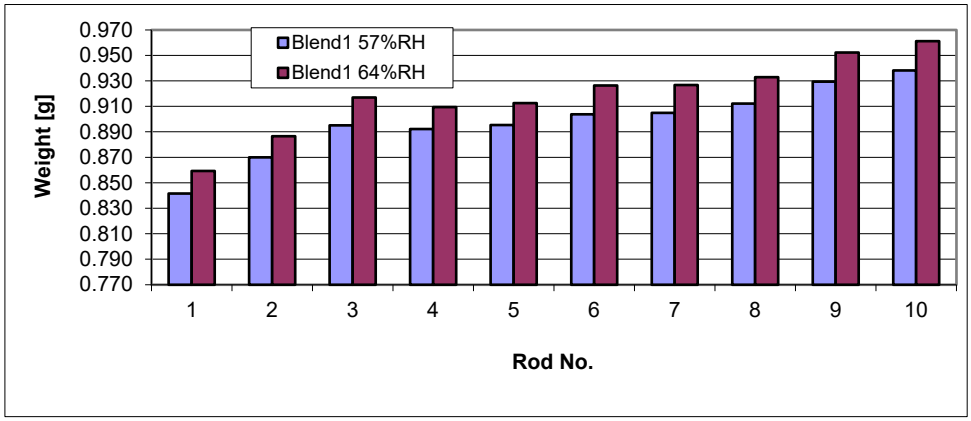
The off-center load error, also called "corner load error" or "eccentric loading error", refers to the change in readout when the same load is placed in various positions on the weighing pan or load plate. This value can be negative or positive. Therefore, especially when balances with high resolution are used, the sample to be weighed should always be placed exactly in the middle of the weighing pan or load plate.

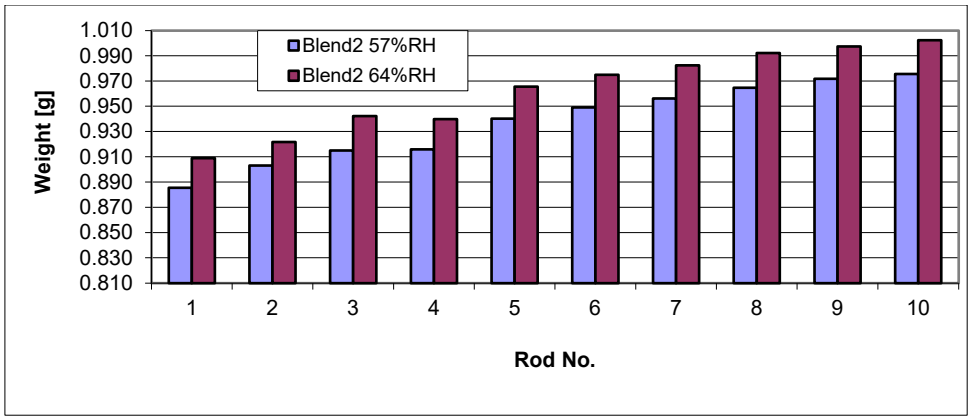
The zero-point-drift is the drift of the zero point of the balance over the time caused by several factors.

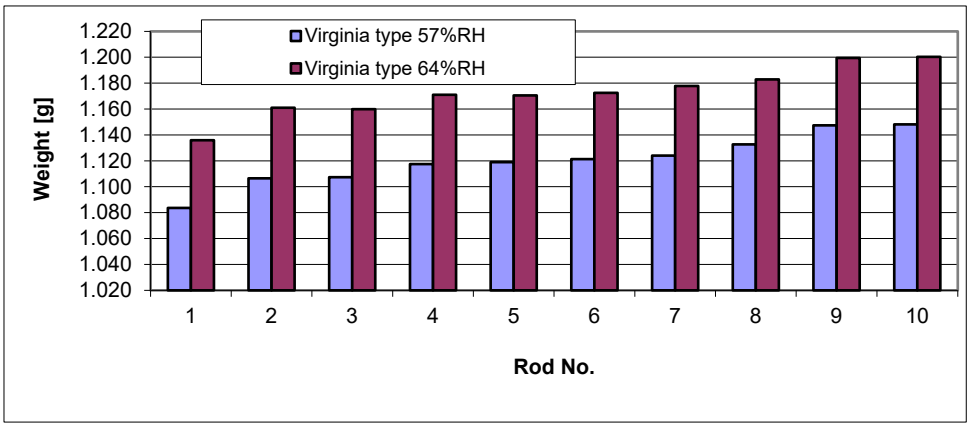
2.3.2 Product related factors

- Damaged product (typically lost tobacco)
- Product moisture
- Electrostatic charge
- Sample temperature (floating effects)

The influence of tobacco moisture to the weight of tobacco rods is displayed in Figures 1-3. The data are taken from a study published 1967 [3]. The Figures show that although the samples are conditioned in an environment within the required limits of ISO 3402, i.e. (60±3) % RH, different weighing results are possible. Here a difference of ±(3-4) % RH is generating a difference in weight results of 20 mg or more per rod.







Figures 1-3: Influence of relative humidity on weighing results of tobacco rods stored for 24 hours at 57 % or 64 % RH.

It should also be noted that the new generation of HTPs, which contain relatively large amounts of glycerol, are being seen to exhibit large humidity-related changes. Early indications are that conditioning and measurement of these products will be undertaken at significantly lower levels of humidity than the traditional 60 % RH adopted for combusted products. If this remains the case then it will pose a challenge for laboratories equipped only with a laboratory held under ISO 3402 conditions.

More widely, hygroscopic samples, such as some laboratory chemicals used in analysis of tobacco products, cannot be precisely analyzed because they absorb moisture which causes a constant increase in weight. If appropriate steps cannot be taken to control the humidity at the weighing location, it is recommended that the sample be weighed in an enclosed container that is suitable for its size (even if this is generally not a practical option for individual products measured using industry-standard tobacco instrumentation).

The influence of sample temperature is often underestimated. During very precise weighing procedures, it is imperative that the sample be adapted to the ambient temperature. Otherwise, convection currents on the surface of the sample may lead to lower results in measurement. For example, if beakers with a large surface area are used during weighing, temperature differences of a few Kelvin can cause the readout to differ in the gram [g] range.

2.3.3 Operation related factors

- Check levelling
- Zero-check
- Central positoning of the sample
- Free placement of the sample
- Changing of moisture due to handling or different ambient conditions
- Time between sampling and measurement
- Time between applying and reading of result
- Loss of tobacco particles
- Correct counting of individual samples for group measurements

For further information see section 6, Making measurements

2.4 Selection of useful balance

Possible selection criteria:

- Type of product
- Requested range
- Product geometry
- Requested accuracy (resolution of selected balance should be $1/10^{th}$ of requested accuracy)
- Place of installation
- Electromagnetic compatibility (EMC) approved

Equipment qualification provides documented evidence that an instrument is appropriate for its intended use to ensure that it will operate on demand, under specified service conditions, to meet system performance and accuracy requirements.

Equipment qualification is subdivided into 4 sections:

1. Design qualification
2. Installation qualification
3. Operational qualification
4. Performance qualification

2.4.1 Design qualification

In design qualification, the user defines the requirements of the test or measuring equipment. Parameters such as accuracy, method of measurement, and requirements of the supplier that relate to design validation or services, must be defined before purchasing. The purpose of design qualification is to ensure that the weighing system is suitable for the particular application.

2.4.2 Installation qualification

Installation qualification describes startup and the detailed sequence of setting up the measuring equipment. Special attention must be paid to the completeness and correct installation of the equipment supplied. To operate high-resolution analytical and microbalances, the use of specially designed anti-vibration balance tables should be considered. In addition, the environmental conditions (particularly the temperature) should be kept as constant as possible.

2.4.3 Operational qualification

Operational qualification describes the metrological testing of a weighing instrument at the place of installation. Adequately trained personnel must test weighing instruments using the corresponding auxiliary equipment and weights that have the appropriate accuracy. In addition, the test results must be documented in a calibration certificate or test report of the weighing instrument. This testing must be performed at established intervals (known as "intervals of confirmation").

2.4.4 Performance qualification

All manufacturers' specifications refer to nearly ideal measurement conditions as recommended in the installation and operating instructions. In practice, however, operators frequently operate weighing instruments under conditions that differ from these. Therefore a performance qualification is required to verify that the measuring equipment functions as intended in its normal operating environment (e.g. weighing a sample under a laboratory fume hood).

3. CONFIRMING CORRECT INSTRUMENT FUNCTION

3.1 Introduction

Checks for instrument function are intended to assure that all instrument components and systems are set up and operating correctly. They are separate from any measures of instrument performance and are a necessary pre-condition for all of the following checks and validations.

3.2 Methods of checking and assuring instrument function

The first step in assuring correct function should be to create a programme of regular preventative maintenance. Instrument manufacturers provide information and guidance on instrument maintenance and offer programmes of planned maintenance. These should include attention on a regular basis.

Checks on instrument:

- Levelling
- Zero-check
- Check for visible damage
- Checks for build up of contaminants within the instrument:
- Remove all tobacco fallout and clean
- Carry out a calibration check to confirm correct operation following any maintenance or adjustment

4. CALIBRATION CHECK AND ADJUSTMENT

4.1 Introduction

The previous sections covered a series of factors that can adversely affect the accuracy of test and measuring equipment in a variety of ways. The influence of these factors can be minimized by calibration checks and adjustments (if required) on a regular basis.

4.2 Definitions

4.2.1 Calibration check identifies the deviation between the readout on the balance and a reference. Calibration check is the most important source of information for checking an instrument's uncertainty of measurement under actual installation and operating conditions. Therefore it plays a central role in controlling the accuracy of inspection, measuring and test equipment.

4.2.2 **Adjustment** always entails corrective intervention in the balance or scale to eliminate the existing error, the so called systematic error, as far as possible. During adjustment, the readout is compared to the nominal value of the calibration standard (whose value is indicated on an accompanying certificate), and the resulting correction factor is stored in the balance's or scale's processor.

How frequently a balance or scale needs to be adjusted depends mainly on the following parameters:

- The frequency of weighing procedures
- The ambient conditions
- The effects of an incorrect result

A variety of instruments and methods exist for performing both of these procedures. In general, a distinction is made between internal and external calibration check and adjustment.

4.2.3 **External calibration check and adjustment** is a procedure used mainly on older-model balances and scales or those with high throughputs. Comparison and correction are accomplished using one or more weights whose value and uncertainty must be known and documented. National testing laboratories, calibration laboratories and qualified manufacturers provide appropriately certificated weights or calibration services to support this purpose.

4.2.4 **Internal calibration check and adjustment** is a procedure that uses a reference weight built into the balance or scale. The exact value of this weight was previously determined and stored as a fixed value in the instrument's processor. This partly automated calibration feature, which is operated at the touch of a button, has become the standard.

The most advanced balances and scales are equipped with a fully automatic calibration check and adjustment device that initiates calibration checks after a user-defined amount of time has elapsed or a temperature off-set has been determined.

The internal weights are protected from dirt and damage and are always at the same temperature as the balance. Internal weights must also be checked and recertified at certain intervals to ensure that they are within tolerance limits

4.3 Essential properties of calibration standards

- They have repeatable values with a low uncertainty of the calibrated value
- All calibration standards should be provided with a certificate that:
- Records the value(s) and unique identification of the standard
- Gives information about the conditions of calibration and the uncertainty of the assigned value
- Defines the method and validity of the calibration, quoting any limits or restrictions to the use of the standard

4.4 Recommendations for the management of standards

The validity of measurements can be improved if a suitable regime is used for the management of standards. In particular:

- Store free of dust, dry and at laboratory temperature and handle standards carefully using cotton gloves or tweezers.
- Carry out a visual check on a regular basis. In particular check for:
  - Damage (blemishes, chips, cracks)
  - Contamination
- Introduce a documented programme of checking for calibration standards against the reference set, including:
  - Any standard suspected of damage or contamination
  - Any new standard before it is released for use
  - All standards at regular, fixed intervals
- Recalibrate standards regularly. The interval chosen should reflect the type of duty to which the standards are put - heavily used standards will require recalibration more frequently than lightly used ones. Set a maximum interval for recalibration.
- Maintain the reference standard set within its validity period – recalibrate at least every 2 years.
- Any standard found to be damaged, contaminated or out of value should be withdrawn from service until it can be replaced or recalibrated.

5. CHECKING THE VALIDITY OF MEASUREMENTS

This process comprises the following stages:

- The calibration process
  - Calibration checking
  - Adjustment
  - Calibration check following adjustment
- Checking Instrument performance.

5.1 The calibration process

5.1.1 The purpose of calibration checking

Calibration checking is an essential part of the overall calibration process and is made to confirm whether the instrument is measuring within the required limits of accuracy. A calibration check should be carried out at regular intervals and frequently enough to detect changes in instrument calibration due to the known influences.

These influences include:

- The effects of measurement system drift
- The effects of changes in ambient conditions

As a minimum, it is recommended that a calibration check is carried out every manufacturing period or shift and at least once per day if the weighing period lasts several days.

If a calibration check shows that an instrument is measuring outside the required limits then it needs to be adjusted.

5.1.2 Precautions when checking calibration

A calibration check of an instrument can be made by measuring a calibration standard in the instrument. The measured value of the standard will indicate how closely the instrument is measuring to the true value. A decision can then be made as to whether to adjust the instrument or not.

When carrying out calibration checking it is necessary to ensure that the measured value is not corrupted and so avoid unnecessary adjustment. In particular:

- The standard should be in thermal equilibrium with the measurement environment.
- Ensure that the instrument is stable before checking

See also section 4.2.4: internal calibration checks and adjustment

5.1.3 A recommended process for checking calibration

To carry out a calibration check, proceed as follows:

- Referring to the manufacturer's instructions, place the instrument into the correct mode to check the calibration
- Make sure that the calibration standard is in thermal equilibrium with the instrument
- Place the calibration standard in the center of the scale pan and close the lid (if installed)
- Observe the measured value until it becomes stable and then note the value
- Compare the measured value with the assigned value of the standard – if the measured value differs from the assigned value by more than the allowable limit, then the instrument should be adjusted.

Note: The allowable limits should be set by reference to the uncertainty of the measurement and the capability of the process. This is most easily determined by regular use of a Shewhart Chart (control chart) to plot the measured values for the calibration standards [4, 5]. For additional information on setting limits of uncertainty, please see section 5.2

If the measured value is within the allowable limit then the instrument does not require adjustment (recalibration).

5.1.4 Adjustment

The process for adjustment should be made with reference to the instrument manufacturer's instructions. The essential points for adjustment are summarised here for convenience:

- Only adjust if there is a good cause, e.g.:

  The calibration check is out of limits

  After major work or disturbance to the instrument

- Refer to Shewhart chart. Unnecessary adjustment can lead to a worsening of the measurement variability and hence of the process that is being controlled
- Always ensure that the value of the calibration standard is slightly larger than the largest value of the sample weight that is to be measured.
- Observe the same precautions against bias and environmental effects as for calibration checking

- The easiest way to achieve this is to make the adjustment immediately following a calibration check – this will use the standard in its previously equilibrated state and will result in the minimum amount of handling of the standard.
- Carry out a calibration check after adjustment to confirm that no error has been made and that the instrument is now measuring within the allowable limits.
- Always check with at least two standards of different values to ensure correct adjustment.

5.2 Checking instrument performance

This is a means of assessing the dynamic measurement performance of a measuring instrument. It should be carried out periodically, particularly after any significant service work or repairs to the instrument.

The purpose of this check is to confirm:

- That the instrument is functioning and operating correctly over full scale
- The instrument is adjusted correctly for the product being measured and is capable of measuring the product correctly
- The measurement capability of the instrument, in particular it may be used to assess the measurement:
- Repeatability
- Instrument-to-Instrument variability

These statistics allow an assessment of the allowable limits for calibration checking in reference to ISO 7870 and ISO 7873. An instrument cannot be more accurate or precise than its uncertainty of measurement.

5.2.1 Measuring repeatability

Repeatability is defined as: “The maximum difference to be expected between repeat measurements of a sample, made on one instrument, by one operator at the same location and within a short period of time”.

Repeatability is normally tested by measuring a series of samples twice in quick succession in one instrument without any intermediate adjustment of the instrument.

The samples used should be stable and should closely represent the actual samples normally measured on the instrument. Depending upon the type of instrument being tested, suitable samples are:

- Cured filter rods
- Artificial cigarettes (dummy cigarettes)
- Actual cigarettes / tobacco containing products

Precautions should be taken to avoid sample degradation. Dependent upon the type of sample, these include:

- Minimising the amount of handling of the samples
- Providing a cushion in the sample collection bin to minimise damage as the samples are dropped through the instrument (test stations)
- Avoiding significant moisture loss and/or tobacco loss from cigarettes

5.2.2 Measuring instrument-to-instrument variability

Tests of instrument-to-instrument variability are a means of assessing the maximum difference to be expected between the measured values of a sample when measured on two different instruments. It can be influenced by many different factors, including the instrument repeatability, differences between the calibration standards used to calibrate each instrument and local differences in operator procedure and technique.

Instrument-to-instrument variability testing is carried out in a similar way to repeatability testing, except that the measurements are made on different instruments and may be carried out over an extended period of time. In addition, there may be several measurement runs, each on a different instrument. This allows testing across a population of instruments within an organisation and allows comparison between any one instrument and the mean of all the instruments. This can be used to determine any biases between the instruments.

Samples for instrument-to-instrument variability testing can be the same as those for repeatability testing. However cigarettes are not normally sufficiently robust to withstand transport between sites, so samples are generally confined to filter rods or artificial/dummy cigarettes.

6. MAKING MEASUREMENTS

If possible, samples should be conditioned with regard to the corresponding ISO standards (ISO 3402 for tobacco products or ISO 187 for paper products). The ambient conditions (relative humidity, temperature) should be documented with the results.

Read the supplied user manual of the balance carefully.

- Check levelling
  - Only correct levelling will deliver accurate weighing results.
- Zero-check
  - Over time a zero point drift will appear. This can be caused by particles left on the weighing pan and/or electronic drift or other factors. To ensure a correct weighing result it is mandatory to control if the display shows zero and if not to press the tare button before starting a measurement.

    In test stations the offset for the zero check will be normally done automatically.
- If necessary do a calibration check.
- Check correct counting of individual samples for group measurements
  - If the counting of the individuals is wrong, the group measurement will not give a true result.
  - In many cases the group measurement is used to calculate the result for an individual sample (e.g. single rod), therefore the group measurement result divided by the "planned" number of individuals must also give a wrong calculated result.
- Prevent changing of moisture content due to handling or different ambient conditions

  The weighing result is dependent on the moisture content of the sample. Ambient conditions (e.g. relative humidity, temperature, air velocity, e.g. due to acclimatisation) may change this moisture content and so the weighing result - even touching the sample with the hands may change the moisture content of the sample.

- Minimise or define time between sampling and measurement
  - To obtain reproducible results the time between sampling and measurement should be defined.

    See also: Changing of moisture due to handling or different ambient conditions
- Place the sample on the weighing pan
  - Ensure central position of the sample

    Placing the sample on the edges of the weighing pan may give lower weighing results.
  - Ensure free placement of the sample

    During a measurement it has to be observed that the sample is placed free on the weighing pan. If the sample touches any other part of the scale (except the weighing pan) the result may be too low.
  - Prevent loss of tobacco particles

    Loss of tobacco particles will decrease the weighing result.
  - Prevent avoidable vibrations.
  - Prevent any airflow.
- Take reading
  - Minimise or define time between placing the sample and reading of result
  - It must be ensured that the displayed value is stable before a measurement is taken.

  See also: Changing of moisture content due to handling or different ambient conditions

  This is also important for samples which have a significantly different temperature than room temperature or which have to be cooled down before the measurement. In this case keep possible moisture change in mind.

  Product depending, or depending on the aim of the measurement, it is recommended to repeat the weighing process and take the average as the result.

7. REFERENCES

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Note:

[1] and [2] are used in general terms for generating this document. Their use is not marked in the text particularly.