



## Asia-Pacific Legal Metrology Forum

### APLMF Guide Document on Rice Moisture Measurement

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provided by Working Group Quality Measurement on Agricultural Products

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#### Foreword

The Asia-Pacific Legal Metrology Forum (APLMF) is composed member economies of APEC and observer economies with a mission to develop legal metrology infrastructure in accordance with each member economy's needs. At the same time APLMF has common interest in promoting economic activities by: harmonizing its development with the mutually acceptable infrastructure; following the model regulations proposed by OIML (International Organization in Legal Metrology); and adopting informative documents to its metrological services.

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## **1 Introduction**

Grain moisture is an important field of measurement in legal metrology that is closely related to reliability in the international/domestic trade and quality of life. Grains are usually required to be dried after harvest before sale or transaction because raw and wet grains are not suitable for long-term storage as they deteriorate rapidly. Dried grains are also convenient for efficient transportation because of their decreased weight. In other words, a transaction of wet grain implies that unnecessary moisture is transported and is sold to the customers. On the other hand, drying procedure of grain requires additional cost and time to the producers and/or traders. This is the primary reason why the price of dried grain per weight is usually higher than that of wet grain. Moisture content therefore is considered as an important characteristic of the product in domestic and international trades due to its strong influence on the price.

Due to such circumstances, measurement instruments for grain moisture are under a legal metrological control in many economies to ensure fair trade among producers, traders, exporters, importers and customers. In connection with the importance of this field of measurement, high-level knowledge and operative skills are required in calibrating and verifying grain moisture meters supported by a sound traceability system. This requirement arose from a basic nature of the measurements in this field. The measurement of grain moisture inherently involves a fundamental difficulty in defining the ‘measurand’ (object to be measured), and a measurement result therefore strongly depends on the characteristics of the sample.

In order to provide knowledge and skills and to look for a possibility to establish a sound traceability system in this field, the APLMF Working Group Quality Measurement on Agricultural Products has conducted training courses/workshops on traceability in rice moisture measurement since 2001. The texts and materials provided in these training programs are utilized for the present guide document.

## **2 Scope and objectives**

This guide document is dedicated to the officers and field inspectors responsible for metrological control on grain moisture meters in the APLMF region. It may be also utilized as a text book in the domestic/international training programs conducted in the region. This document provides guides to: the basic understandings of grain moisture; moisture measurement; reference method; traceability system; consideration of uncertainty; practical measuring instruments used in the field; and practical procedures for calibrating, testing and verifying grain moisture meters.

The target instruments of this document are inferential and electric moisture meters used in real fields of production or transaction of agricultural products. Among such instruments, small-sized resistance and capacitance type moisture meters are emphasized on in this document because such instruments are used widely in the Asian economies. Rice is selected as the primary target sample because it is widely produced in the economies and Working Group has sufficient knowledge on this product. Some parts of this document though, can be applied to any kinds of grain.

A reference method represented by the drying method is not directly included in the scope. It is mentioned, however, with practical procedures as an important method necessary to establish a

sound traceability system.

This document is a complementary guide supporting the OIML Recommendations such as R 59 (*Moisture meters for cereal grains and oilseeds: 2016*)<sup>1)</sup> and other international/regional standards including ISO 665<sup>2)</sup>, ISO 711<sup>3)</sup>, ISO 712<sup>4)</sup>, ISO 6540<sup>5)</sup>, ISO 7700<sup>6)7)</sup> and ISO 24557<sup>8)</sup>. These external documents are referred to in this document.

### **3 Terminology**

#### **3.1 Terms in VIM and VIML**

Terms closely related to this guide document, which are defined in the International Vocabulary of Metrology (VIM)<sup>9)</sup> and International Vocabulary of Terms in Legal Metrology (VIML)<sup>10)</sup>, are cited in this clause in alphabetical order with the clause numbers in the original documents.

##### **3.1.1 Calibration (VIM 2.39)**

Operation that, under specified conditions, in a first step, establishes a relation between the quantity values with measurement uncertainties provided by measurement standards and corresponding indications with associated measurement uncertainties and, in a second step, uses this information to establish a relation for obtaining a measurement result from an indication

NOTE 1: A calibration may be expressed by a statement, calibration function, calibration diagram, calibration curve, or calibration table. In some cases, it may consist of an additive or multiplicative correction of the indication with associated measurement uncertainty.

NOTE 2: Calibration should not be confused with adjustment of a measuring system, often mistakenly called “self-calibration”, nor with verification of calibration.

##### **3.1.2 Calibration curve (VIM 4.31)**

Expression of the relation between indication and corresponding measured quantity value

NOTE: A calibration curve expresses a one-to-one relation that does not supply a measurement result as it bears no information about the measurement uncertainty.

##### **3.1.3 Maximum permissible error (VIM 4.26/VIML 0.05)**

Extreme value of measurement error, with respect to a known reference quantity value, permitted by specifications or regulations for a given measurement, measuring instrument, or measuring system

NOTE 1: Usually the term “maximum permissible errors” or “limits of error” are used, where there are two extreme values.

NOTE 2: The term “tolerance” should not be used to designate ‘maximum permissible error’.

[VIML] NOTE 3: Usually the term “maximum permissible error” is abbreviated to “MPE”, or “mpe”.

### **3.1.4 Measurand (VIM 2.3)**

Quantity intended to be measured.

NOTE 1: The specification of a measurand requires knowledge of the kind of quantity, description of the state of the phenomenon, body, or substance carrying the quantity, including any relevant component, and the chemical entities involved.

NOTE 2: In the second edition of the VIM and in IEC 60050-300:2001, the measurand is defined as the “particular quantity subject to measurement”.

NOTE 3: The measurement, including the measuring system and the conditions under which the measurement is carried out, might change the phenomenon, body, or substance such that the quantity being measured may differ from the measurand as defined. In this case, adequate correction is necessary.

EXAMPLE 1: The potential difference between the terminals of a battery may decrease when using a voltmeter with a significant internal conductance to perform the measurement. The open-circuit potential difference can be calculated from the internal resistances of the battery and the voltmeter.

EXAMPLE 2: The length of a steel rod in equilibrium with the ambient Celsius temperature of 23 °C will be different from the length at the specified temperature of 20 °C, which is the measurand. In this case, a correction is necessary.

NOTE 4: In chemistry, “analyte”, or the name of a substance or compound, are terms sometimes used for ‘measurand’. This usage is erroneous because these terms do not refer to quantities.

### **3.1.5 Measurement uncertainty, uncertainty of measurement (VIM 2.26)**

Non-negative parameter characterizing the dispersion of the quantity values being attributed to a measurand, based on the information used

NOTE 1: Measurement uncertainty includes components arising from systematic effects, such as components associated with corrections and the assigned quantity values of measurement standards, as well as the definitional uncertainty. Sometimes estimated systematic effects are not corrected for but, instead, associated measurement uncertainty components are incorporated.

NOTE 2: The parameter may be, for example, a standard deviation called standard measurement uncertainty (or a specified multiple of it), or the half-width of an interval, having a stated coverage probability.

NOTE 3: Measurement uncertainty comprises, in general, many components. Some of these may be evaluated by Type A evaluation of measurement uncertainty from the statistical distribution of the quantity values from series of measurements and can be characterized by standard

deviations. The other components, which may be evaluated by Type B evaluation of measurement uncertainty, can also be characterized by standard deviations, evaluated from probability density functions based on experience or other information.

NOTE 4: In general, for a given set of information, it is understood that the measurement uncertainty is associated with a stated quantity value attributed to the measurand. A modification of this value results in a modification of the associated uncertainty.

### **3.1.6 Metrological traceability (VIM 2.41)**

Property of a measurement result whereby the result can be related to a reference through a documented unbroken chain of calibrations, each contributing to the measurement uncertainty

NOTE 1: For this definition, a ‘reference’ can be a definition of a measurement unit through its practical realization, or a measurement procedure including the measurement unit for a non-ordinal quantity, or a measurement standard.

NOTE 2: Metrological traceability requires an established calibration hierarchy. NOTE 3: Specification of the reference must include the time at which this reference was used in establishing the calibration hierarchy, along with any other relevant metrological information about the reference, such as when the first calibration in the calibration hierarchy was performed.

NOTE 4: For measurements with more than one input quantity in the measurement model, each of the input quantity values should itself be metrologically traceable and the calibration hierarchy involved may form a branched structure or a network. The effort involved in establishing metrological traceability for each input quantity value should be commensurate with its relative contribution to the measurement result.

### **3.1.7 Type approval (VIML 2.05)**

Decision of legal relevance, based on the review of the type evaluation report, that the type of a measuring instrument complies with the relevant statutory requirements and results in the issuance of the type approval certificate

### **3.1.8 Verification of a measuring instrument (VIML 2.09)**

Conformity assessment procedure (other than type evaluation) which results in the affixing of a verification mark and/or issuing of a verification certificate

## **3.2 Other terms used in this guide document**

### **3.2.1 Adjustment**

Control of the moisture content of a grain sample by changing the value artificially using a drying or moistening procedure

NOTE: This term is used in this document in a different meaning from ‘adjustment of a measuring system’ defined in in Clause 3.11 in VIM.

### **3.2.2 Calibrate, calibration, calibration equation, calibration parameter**

These terms are used based on the definition in Clause 2.39 of VIM (see 3.1.1). However, there are minor differences in practical meaning when they are used in the context of grain moisture measurement as explained in the following notes.

NOTE 1: ‘Calibrate’ or ‘calibration’ sometimes includes a redefinition of the calibration equation (or changes in the calibration parameters) as a result of a calibration. This is a usage defined in NOTE 1 in Clause 2.39 of VIM. The calibration equation defines a calibration curb between the moisture content as a true value obtained with a reference method (or a standard meter) and a reading of a moisture meter to be calibrated.

NOTE 2: ‘Calibration’ is sometimes used as an abbreviation of ‘calibration equation’ or ‘calibration parameter(s)’.

### **3.2.3 Form (of product)**

A form of agricultural product in which the product is measured, traded and consumed. In the case of rice, there are three forms; paddy rice, brown rice and polished rice.

### **3.2.4 Grain**

This term indicates several categories of agricultural product. It includes small, hard, dry seeds with or without hulls or fruit layers, which are harvested for human or animal consumption<sup>20</sup>). In this document, grain means a part of agricultural products used as a sample to be measured with a moisture meter or a reference method. Rice, wheat, barley, corn, oil seeds and pulses are typical examples of grain.

### **3.2.5 Inferential measurement method (meter)**

A measurement method (meter) for moisture content of grain which is unable to conduct a direct and absolute measurement and needs a calibration with a reference method in advance

NOTE: This method is also referred as an ‘indirect method’ or a ‘comparison method’.

### **3.2.6 Meter-to-meter comparison**

A practical method to calibrate a moisture meter in a lower level of traceability using a comparison between two moisture meters intermediated by a grain sample

NOTE: In this method, a pair of small samples taken from a homogeneous lump of grain is measured both with (A) a standard meter and with (B) the meter to be calibrated. The meter (B) is calibrated against the measurement result obtained by the meter (A). Reference value of moisture content of the sample (or lump) is not necessary because it is used merely as an

intermediate material.

### **3.2.7 Moisture content**

The moisture content (*MC*) in a solid or liquid material which is equivalent to a ratio of the mass of water and aqueous substances to the total mass of the sample. In this guide document, it is defined by **Equation 3** in 6.2.6.

NOTE: Moisture content is usually expressed in percent (%).

### **3.2.8 Moisture meter**

An instrument that measures a physical (electrical or optical) parameter of a grain sample, and infers its moisture content based on a calibration equation with indication of the result on a display unit

### **3.2.9 Product, agricultural product**

One of agricultural products identified as a botanical nomenclature 'species'. Rice, wheat, corn, potato, tomato, banana are typical examples of products. A product in a species is usually divided into different varieties.

### **3.2.10 Reference method (for moisture content)**

A measurement method for grain moisture which enables an absolute determination of moisture content and used as the primary standard for traceability

NOTE: This method is usually specified by a national or a regional authority. A drying method using an oven is frequently employed as a reference method.

### **3.2.11 Sample, reference sample**

A lump (100 g – 1 kg typically) of grain with homogeneous characteristics (moisture content, variety, grade, place and year of harvest) to be measured with a moisture meter or a reference method

NOTE 1: 'Reference sample' is a sample, to which a representative value of moisture content has been given using a reference method. A reference sample is used as a measurement standard to calibrate a moisture meter.

NOTE 2: A sample is usually contained in a sealed plastic container and stored in a refrigerator to prevent a change in characteristics. A small portion selected randomly from the lump of sample is also referred to as a 'sample'.

### **3.2.12 Standard, primary standard, secondary standard, working standard**

Measurement standard (5.1 in VIM) used to maintain the traceability system for grain moisture

measurement. The standard usually constitutes a hierarchy scheme composed of moisture meters; (1) primary standard, (2) secondary standard and (3) working standard.

NOTE 1: A moisture meter, which has been calibrated against the reference samples with an absolute value of moisture content, is used as the primary standard.

NOTE 2: For the primary, secondary and working standards, a moisture meter with a high and stable accuracy is used.

NOTE 3: The hierarchy scheme depends on the metrological system in each economy/region. In some regions, the secondary standard is omitted, and a working standard is calibrated directly by the primary standard.

### **3.2.13 Variety**

A botanical nomenclature which identifies a biological variation under a species (agricultural product). The following are examples of varieties<sup>20)</sup> for rice; Japonica (including Akitakomachi, Koshihikari and Sasanishiki), Indica (including Jasmine), Javanica and so on.

NOTE: One variety may have different physical and/or biological characteristics. They also depend on the year and place of harvest.

## **3.3 Abbreviations and acronyms**

BIPM: International Bureau of Weights and Measures

IEC: International Electrotechnical Committee

ISO: International Organization for Standardization

MC: moisture content

MPE: maximum permissible error

NIR: near infrared

OIML: International Organization of Legal Metrology

USDA: United States Department of Agriculture

## **4 Basic understanding of moisture contained in grains**

### **4.1 What is the water<sup>13)</sup>**

The earth is a unique planet in the solar system with a huge amount of water (H<sub>2</sub>O) on the surface. It enables many creatures (animals and plants) to exist on the planet. Therefore, it is needless to say that water is the most important substance for all kinds of creatures. For example, water accounts for 80 % of a baby body, and 60 % of an adult one. Water has extraordinarily high melting point (0 °C) and boiling (100 °C) point compared to those of other hydrogen compounds (CH<sub>4</sub>, SiH<sub>4</sub>, H<sub>2</sub>S...). This means that water has a strong intermolecular force due to its unique molecular structure.

Due to the same reason, water requires a huge amount of energy (specific heat and latent heat) when it is heated, melted and evaporated. Such a characteristic of water greatly helps to keep our

climate far more moderate compared to the climates in other planets. The volume of water of a unit weight expands when it freezes. It is an unusual phenomenon observed only for bismuth, gallium, germanium, silicon and water. Other materials shrink when they freeze. This phenomenon enables an iceberg to float on the sea (and an ice in a glass too). It arises from a unique structure of ice that has an extra space in the molecule.

Water has 18 kinds of isotopes with slightly different characteristics and it easily dissolves almost all kinds of elements and substances. This characteristic produced infinite kinds of water and water solutions on the earth.

## 4.2 Moisture in microscopic structure of grains

Water shows unique characteristics when it is contained in an extremely small space. As an example, a thin (< 0.1 mm) layer of water between a pair of glass plates has very high viscosity and low freezing point of less than -20 °C. This guide targets the water absorbed or contained in microscopic structures of grains as a biological material. Water contained in such materials shows unique behaviors similar to the case between glass plates. In the study of moisture contained in biological materials, it is considered that water is contained in one of the three forms shown below.

- (1) Water of crystallization: water molecules that compose a part of the molecule of base material. The water is strongly necessary to maintain the structure of the base molecule. A typical example of such materials is chalcantite ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ).
- (2) Bound water: water molecules bound to the surface of the base molecule (protein, etc.). They play an important biological role to protect the base molecule.
- (3) Free water: bulk water contained in a rather macroscopic structure. This form of water may cause deterioration by bacteria.

Water in the forms (1) or (2) does not freeze below 0°C, and it is difficult to remove them by heating without modifying (or destroying) the base material. In grain moisture measurements, 'dry sample' means a condition without (3) free water.

The cereal grains contain mainly of (i) carbohydrates, (ii) proteins, (iii) fats and (iv) water. These compounds have a hydrophilic (water friendly) part in the molecules, therefore microscopic structure changes with absorption of water. In a dry grain, molecules are connected tightly with strong bonds. In a wet grain, the bonds become weak and the structure inflates as a result of absorption of water.

## 4.3 A phenomenon caused by free water

The density of brown rice has a peak at lower moisture content of near 7 %. This phenomenon is considered as a result of moistening procedure. In this procedure, free water filled the space between microscopic structures composed of grain molecules which have a density higher than that of water (Figure 1).

Free water freezes at 0 °C and 1 atm. A peak of specific heat capacity of grain is therefore observed as a result of the phase change at a temperature near 0 °C. The moisture content of grains

in a condition of constant temperature and humidity becomes closer asymptotically to a stable state called as 'equilibrium' (Figure 2). As a result of this phenomenon, moisture content of grain in equilibrium with the atmosphere has a close relationship between the environmental conditions as shown in Figure 3. However, the moisture content also has a 'hysteresis' in which different values are observed between moistening and drying processes. A typical hysteresis curve of grain is given by Figure 4.

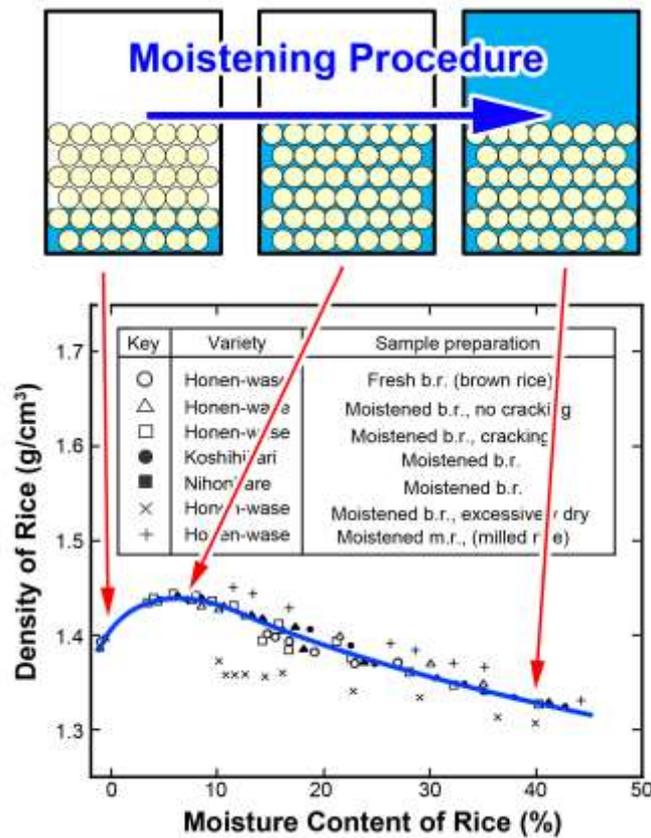


Figure 1: moistening procedure of rice

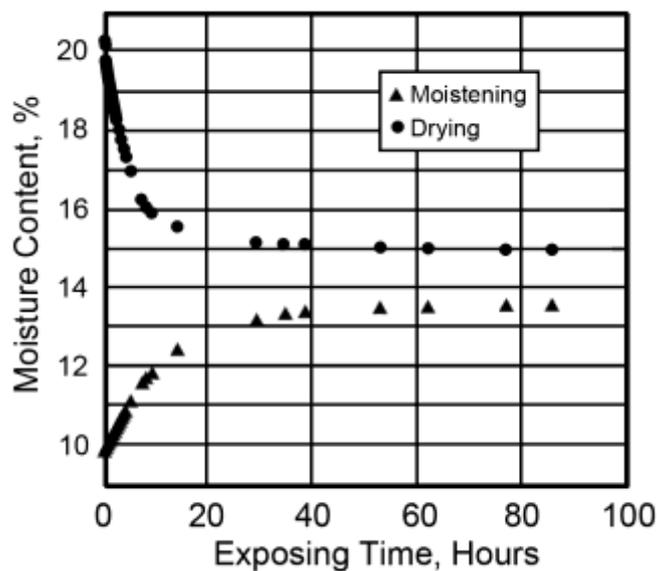
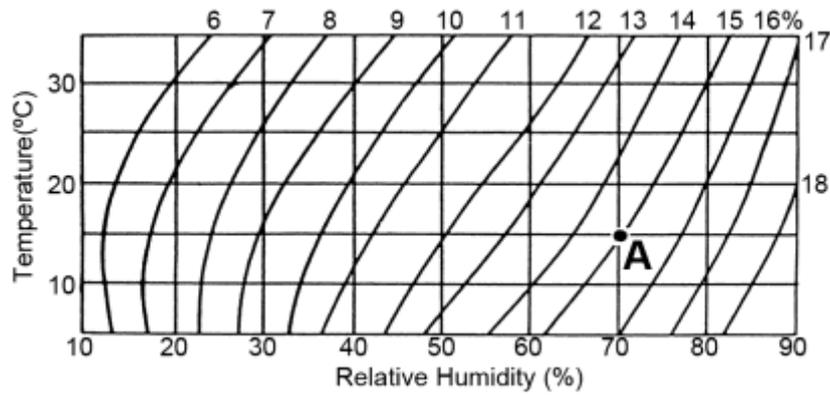
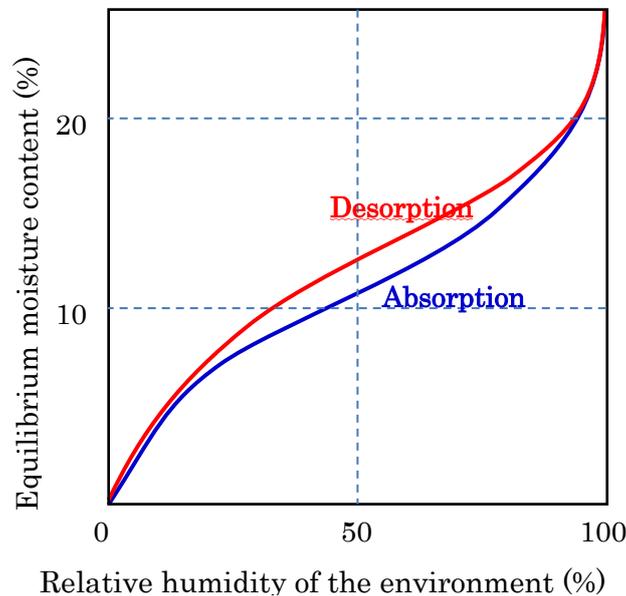


Figure 2: Moistening and drying process of rice with a hysteresis.



The moisture content of paddy rice exposed to the environmental condition of 15 °C and 70% RH (indicated as 'A') gradually gets closer to 15 %

**Figure 3: Moisture content of paddy rice in equilibrium with the environment.**



**Figure 4: Hysteresis in moisture content of corn in equilibrium with the environment**

#### 4.4 Quality of agricultural products and moisture content

As mentioned in Chapter 1, quality (moisture content, protein content, sugar content, etc.) of agricultural products (rice, wheat, maize, beans, etc.) is closely connected to fair trade because prices or acceptance criteria of a product are usually determined based on it. Among the physical quantities representing quality, moisture content is closely related to the stability of the product in long-term storage. Moisture content is also important for trade because moist grains must be dried artificially using energy and cost before transaction. This process usually boosts up its unit price.

#### 4.5 Typical measurement methods for grain moisture

Based on the backgrounds mentioned in 4.4, many measurement methods for moisture content have been developed in scientific metrology and industrial /agricultural measurements as listed below.

- (1) Drying method at the atmospheric pressure (or reduced pressure)\*
- (2) Chemical method (Karl Fischer titration method in analytical chemistry)
- (3) Distillation method (chemical method using toluene for distillation)
- (4) Practical methods
- (5) Electrical resistance method\*\*
- (6) Electrical capacitance method\*\*
- (7) Near infrared absorption method\*\*
- (8) Microwave absorption method
- (9) Infrared-heated moisture balance (simplified dry oven method)

\* An absolute and direct measurement method used to provide reference data/samples in many economies.

\*\* Practical methods which are widely used in the real fields.

## **5 Traceability for moisture measurement of grains**

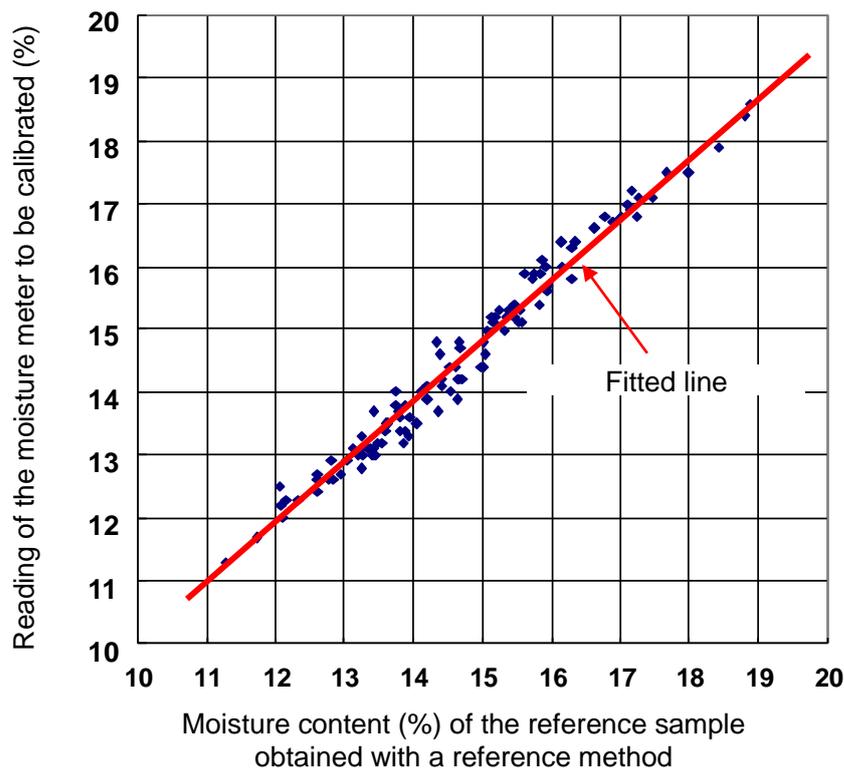
### **5.1 Basic understanding of traceability**

There have been strong needs for traceability in grain moisture measurement from a practical field in scientific and legal metrology. The needs are categorized into several fields such as: international and domestic reliability in fair trade; regional primary traceability system; requirements for a practical system; harmonization with existing international standards; need for a system applicable to in-service meters; and low cost and easiness in adoption and operation.

Regardless of such practical needs, the real situation of traceability is far from the establishment of the sole and internationally-accepted system. It is because of the inherent nature of grain moisture that strongly depends on measurand, measurement methods and procedures. A reference sample of grain is usually provided with a reference method represented by a drying method (Chapter 6), which is considered as the primary standard for traceability in grain moisture measurement. Meanwhile, the drying method is destructive since a sample is dried up after a measurement and its characteristics have been modified significantly. Such a nature of the drying method makes it difficult to establish traceability because it is impossible to use the same sample in both a reference method and a moisture meter to be calibrated.

Most of the moisture measurements in the field of production are conducted using electrical measurement methods (Chapter 7). These methods are appropriate for uses in the field because they enable a rapid measurement with a small instrument in a light weight. These methods are also called as 'inferential' measurement method because they are unable to obtain the absolute value by itself, and they must be calibrated in advance against the reference sample with known moisture content.

An electrical moisture meter used in the field is calibrated against the reference method using the reference sample. In this process, a set of reference samples with different moisture content (10-30 levels) are provided using the reference method. Because the drying method is destructive, a lump of grain sample with a sufficient amount (100 g - 1 kg) with homogeneous characteristics is prepared. Only a small portion (10 g) of the lump is used for the drying method and its moisture content is determined absolutely. The rest of the lump is used as a reference sample assuming that it has the same moisture content (and other characteristics) with that of the small portion. In order to assure this important assumption in the drying method, the lump of sample must be homogenized and stabilized well before use.



**Figure 5: A reading of a resistance moisture meter against the moisture content measured with a reference method (brown rice, 113 samples/30 varieties)**

Using a moisture meter to be calibrated, the moisture contents of a set of reference samples are measured. **Figure 5** shows a typical result of such measurement for calibration. The red line in this figure represents a fitted line as a result of a least-square method applied to the entire results. A set of procedures for calibration is concluded by obtaining this fitted line. This line is equivalent to a calibration equation which is the most important information stored in a moisture meter and defines a relationship between a physical quantity (i.e., electrical resistivity, capacitance) and moisture content.

The above procedure is usually used to establish traceability between the reference method (drying method) and primary/secondary standard meters (inferential-type). For the procedures to set up traceability in the lower levels (third standard or less), a simplified calibration method may be employed, in which a meter-to-meter comparison is conducted through a mediation of a sample

without a moisture value. In this method, the secondary standard meter is used as a master meter for calibration.

## 5.2 Current situation in traceability

Establishment of the traceability system for moisture measurement means an idealistic situation in which all moisture meters in any economy (or region, or the world) are traceable to unique primary standard (reference method) with a chain of comparisons. It is, however, unrealistic due to two main reasons.

One is the fact that there is no unique reference sample of grain with the constant and universal characteristics. Such a situation is far from an ideal state represented by the mass standard for which the unique global prototype is maintained in BIPM in Paris. Actually, a calibration curve of a moisture meter varies depending on the variety of grain, and there are thousands of varieties in the world even for one species of grain. In addition, a calibration curve may differ even for the same variety depending on the year and the place of harvest. Furthermore, it may change even for the same sample during long-term storage. Due to such uncertain and unpredictable factors, it is impossible to define measurand precisely in moisture measurement.

The other reason is a lack of consistency and reproducibility in the reference method (drying method). It is expected ideally that only aqueous materials are removed during the drying process. The actual decrease of sample weight during the drying process, however, depends on the drying condition (temperature and time length). It means that obtained value of moisture content of the same sample differs significantly depending on a drying condition. At present, there are several different international standards for the drying method using different drying conditions (see Chapter 6).

As far as the moisture meters are used only in a small region, where the same variety of grain is produced and the same reference method (drying method) is employed, unique calibration curve may be applied to all meters. However, when moisture meters are used in a wider region for different varieties, measurement uncertainty due to the difference in sample characteristics is not negligible. Such a problem becomes significant when a meter is exported to another economy or region where a different variety is produced and a different reference method is employed.

## 5.3 Proposed practical traceability system

Because of the strong dependency of measurement results of moisture content on the measurand and measurement method, it is difficult to establish a global tractability system in grain moisture. Therefore, this guide document proposes an alternative and practical solution in which only one traceability system would be set up in a smaller region where one agricultural product is cultivated and one reference method (drying method) is employed. **Figure 6** shows a diagram of the proposed traceability system. Each procedure of the system is explained in details below.

- (1) The national (or regional) authority should employ one reference method for moisture content. A drying method based on international (or regional) standard such as ISO 712 is usually adopted as the reference method. This drying method is used to provide reference samples for

calibrating the primary standard (meter) for the entire traceability system which is to be prepared in Step (4).

- (2) Collect samples of a species of product (rice, wheat, corn etc.) including different varieties which are widely produced/traded in the region. In this procedure, varieties in the same category of product should be selected. For example in the case of rice, varieties belong to japonica rice may be selected but varieties belong to different subcategories (japonica, indica and javanica) should not be mixed. Provide at least a set of 30 reference samples with a wide range of moisture contents (10-30 % for rice). The amount of each sample should be sufficient for the use in further repetitions of calibration. The samples must be homogenized and stabilized before use. See Chapter 9 for the instructions to prepare the samples.
- (3) Measure moisture contents of the 30 reference samples using the reference method, and determine their reference values of moisture content.
- (4) Select a moisture meter (inferential-type) in high and stable quality which is used as the primary standard for the entire traceability system. Due to significant inefficiency in the reference method (drying method), this meter is used as the practical primary standard. In preparation for loss or damage of the primary standard meter, provide two additional spare meters of the same model.
- (5) Calibrate the primary standard against the 30 reference samples prepared in Step (3), and redefine its calibration curb. Two spare meters should be also calibrated against the same reference samples. The procedures (1)-(5) are usually performed at a national (or regional) authority in legal metrology or at the National Metrology Institute (NMI).
- (6) Select 5 reference samples from the 30 reference sample provided in Step (3) within the entire range of moisture content. Moisture contents of the 5 samples shall distribute evenly in the target range.
- (7) Calibrate the working standard meters against the primary standard using the 5 reference samples. Even if the samples have reference values, a meter-to-meter calibration method is recommended because moisture contents of the samples may change after the determination of their reference values.
- (8) Calibrate moisture meters in service in the field with a meter-to-meter calibration method using the working standard as a master meter. The 5 reference samples or a sample without a value of moisture content may be used for the comparison. The procedures (6)-(8) are usually performed at a local office in legal metrology, and working standard meters are maintained at the same office.
- (9) Repeat the entire procedure periodically (once a year is recommended) to maintain the traceability system because characteristics of grains, reference samples and standard/working meters vary gradually in the course of time.

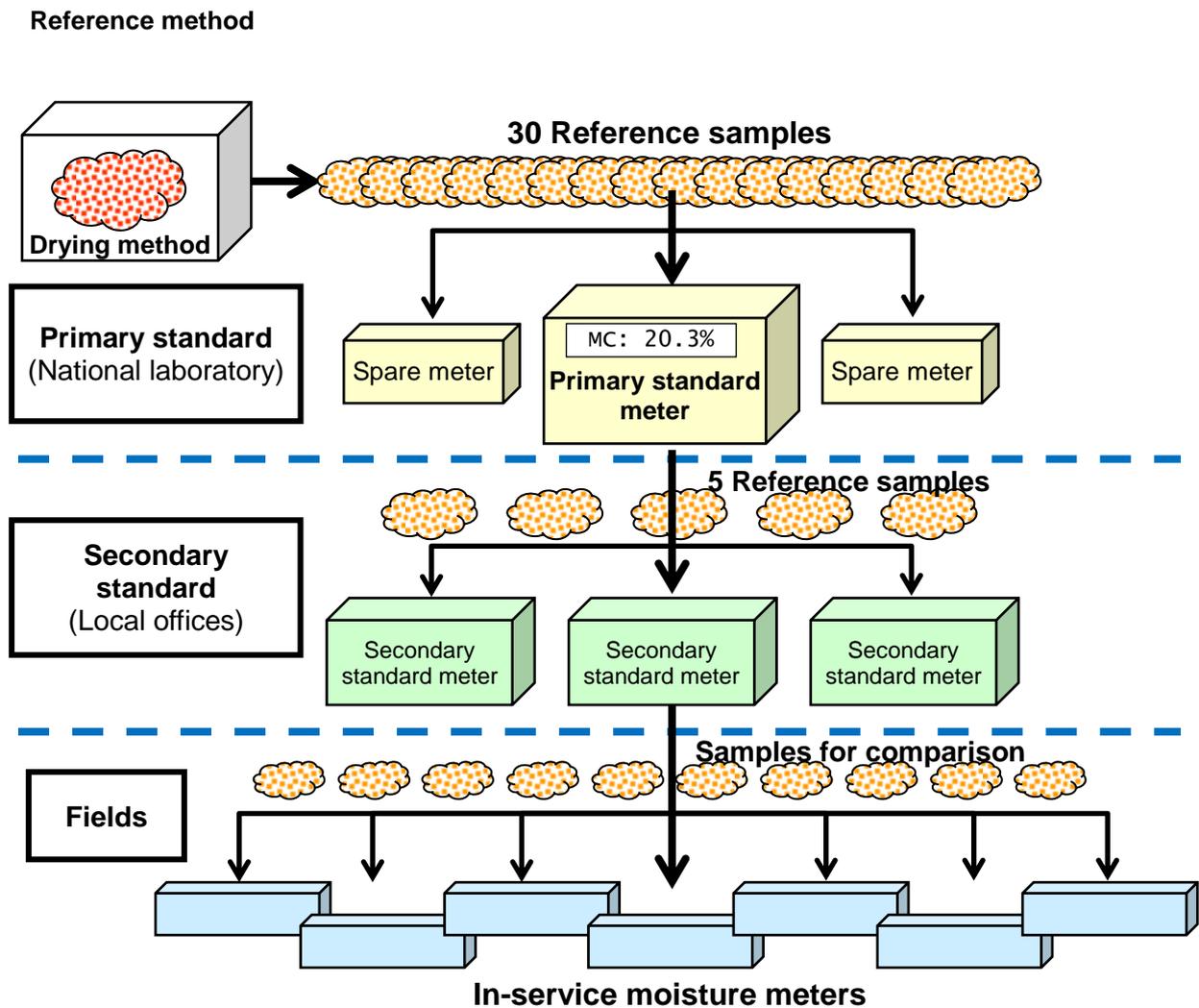


Figure 6: Proposed practical traceability system for moisture content of grains

The traceability system mentioned above may be employed in the framework of legal metrology based on a measurement law in each economy. For example, Thailand has already employed this system and has established the original initial/periodical verification system for rice moisture meters.

#### 5.4 Uncertainty in moisture measurement

Procedure for evaluating measurement uncertainty is an essential part in establishing traceability system. In the measurement of moisture content of grains however, generally it is difficult to evaluate uncertainty due to the inherent nature existing in this field (see 5.2). This clause explains basic understanding of uncertainty and elaborates current situation about the consideration on uncertainty in the measurement of moisture contents.

### 5.4.1 Basic concept of uncertainty

The term ‘measurement error’ has been frequently used as a traditional keyword in metrology. This keyword is understood to represent a value (measurement quantity value minus true value) which is based on the assumption that the true value (or reference value) is already known. A measurement error is considered to be composed of systematic error (or bias) and random error.

In the first edition of GUM (*Guide to the Expression of Uncertainty in Measurement*) published in 1993<sup>11)</sup>, a new keyword ‘measurement uncertainty’ was introduced. This term corresponds to a doubt (or validity) of a measurement result expressed with a positive quantity. In the concept of uncertainty, it is considered impossible to know a ‘true value’.

The concept of ‘uncertainty’, however, is still ambiguous. The followings are several definitions of uncertainty in the documents which are commonly referred to in metrology. They give a general picture of what ‘uncertainty’ implies.

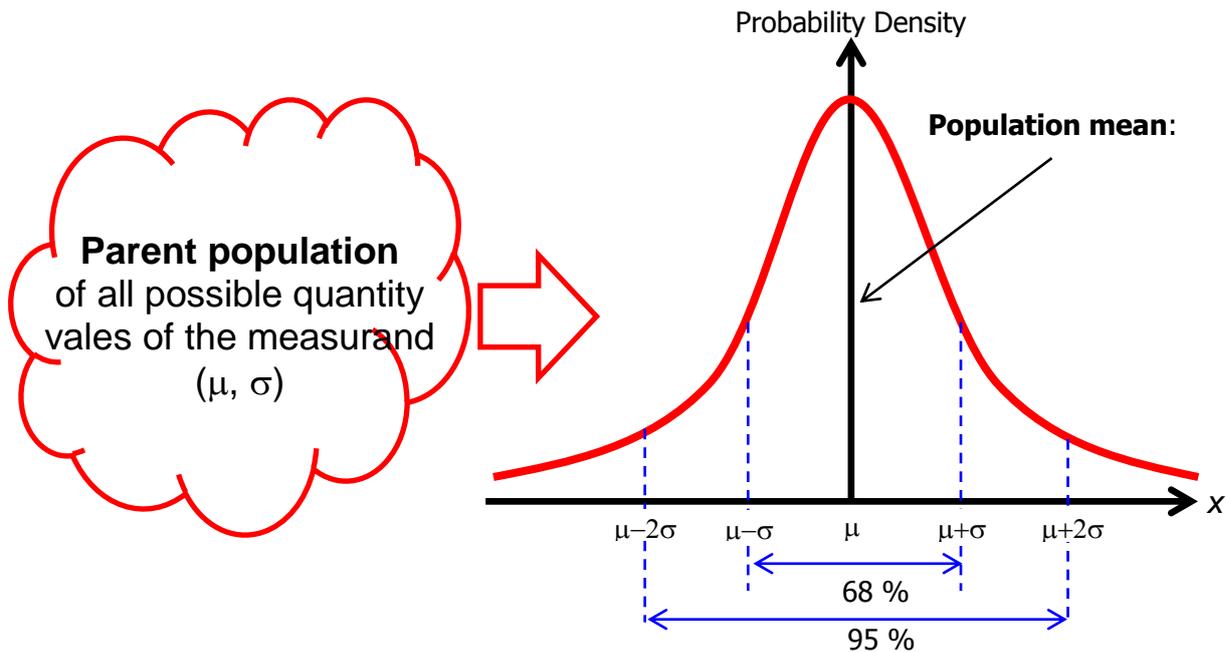
- VIM2 (1993) cited in GUM (2008)<sup>11)</sup>: *Parameter, associated with the result of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the measurand.*
- VIM3 (2008)<sup>9)</sup>: *Non-negative parameter characterizing the dispersion of the quantity values being attributed to a measurand, based on the information used.*
- GUM (2008)<sup>11)</sup>: *Parameter, associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand.*

Because a true value is considered as unknowable, the concept of uncertainty strongly depends on statistical approach. The following are basic assumptions to be applied when we consider uncertainty.

- (1) A parent population is always assumed as an ideal and virtual group which is composed of infinite numbers of quantity values representing a measurand (an object to be measured). A measurement result is considered as one sample taken randomly from the parent population.
- (2) A statistical approach is employed assuming that the quantity values belong to the parent population follow a normal distribution.
- (3) It is impossible to know the true value of the measurand unless it is defined independently. Instead, the best estimated value of the measurand accompanied with a value of uncertainty (as a parameter associated with the dispersion of quantity values) is evaluated. A result of measurement is considered to be complete only when it is accompanied with a statement of uncertainty.
- (4) Dispersion means unpredictable and random errors which remain even after every effort has been made to correct all recognizable sources of uncertainty. If there is an unknown systematic error, the actual value of uncertainty may become much larger than that we evaluated.

### 5.4.2 Statistical background based on a normal distribution

In the concept of uncertainty, quantity values of a measurand are assumed to follow a normal distribution when an infinitely large number of measurements are conducted on the measurand. This assumption is based on the Central Limit Theorem, which states that a combination of arbitrary distributions with different shapes approaches to a normal distribution as the total number of distributions increases. This virtual group of an infinite number of quantity values is considered as the parent population. In this concept, population mean ( $\mu$ ) is considered as the best estimate of the measurand, and the quantity values have dispersion with a standard deviation ( $\sigma$ ) as illustrated in **Figure 7**.



**Figure 7: Parent population and distribution of quantity values**

Based on this assumption, a measurement is considered as a procedure to choose one sample randomly from the parent population. This sample is equivalent to one measurement result representing the measurand. The scatter around the mean evaluated for a small group of samples is an important parameter closely related to the measurement uncertainty. The standard deviation of the scatter is called ‘standard uncertainty’. Because it is fundamentally impossible however to obtain the mean ( $\mu$ ) and standard deviation ( $\sigma$ ) of the parent population, the mean ( $m$ ) and standard deviation ( $s$ ) of the small group are evaluated with a statistical or an empirical method.

In addition, another uncertainty with a wider range ( $k\sigma$ ) is called as ‘expanded uncertainty’. The ratio  $k$  is called as ‘coverage factor’ and a value of two is commonly employed for  $k$ . Within the range of expanded uncertainty with  $k = 2$  (both positive and negative), nearly 95 % of quantity values are included. Therefore, the factor of  $k = 2$  is sometimes referred as ‘range of 95 % confidence’.

The mean ( $\mu$ ) of the parent population is sometimes deemed as the true value regardless the difficulty to obtain it. Another fact should be noted, however, that the mean is merely ‘believed’ as the true value. If there is an unknown error factor, the true value may exist far from the mean. Nobody can guarantee that the mean value is always the true value.

### 5.4.3 Practical methods to evaluate uncertainty

In uncertainty analysis, a standard (or expanded) uncertainty of the measurement result is evaluated using either of the two methods:

**Type A:** Evaluation by applying a statistical method on the measured quantity values (measurement data). In this method, standard uncertainty is evaluated as the standard deviation which is obtained numerically.

**Type B:** Evaluation by an analytical method based on other information. In this method, a standard uncertainty is obtained analytically from the knowledge about the quantity values which has been obtained in advance. Specifications and characteristics of the measuring instrument and/or the sample to be measured are used commonly as the knowledge base.

There may be cases where it is impossible to obtain the value of uncertainty of target measurand directly, and the value must be evaluated using other values that compose the measurement result. In such cases, it is allowed to evaluate the uncertainty indirectly. Assuming that an arbitrary quantity  $y$  is determined with an equation  $y = f(x_1, x_2, x_3, \dots, x_n)$  by several independent parameters ( $x_i$ ), the combined uncertainty ( $u_c$ ) is given by the equation:

$$u_c^2(y) = \sum_{i=1}^n \left[ \frac{\partial y}{\partial x_i} \right]^2 u^2(x_i) \quad (1)$$

In a simple case ( $y = x_1 x_2 x_3$ ), the relative combined uncertainty [ $u_c(y)/y$ ] is expressed as:

$$[u_c(y)/y]^2 = [u(x_1)/x_1]^2 + [u(x_2)/x_2]^2 + [u(x_3)/x_3]^2 \quad (2)$$

In other words, relative combined uncertainty is equivalent to the square root of the sum of squares of relative uncertainty of all associated parameters only if  $x_i$  is independent and there are no correlations in each other. From the structure of **Equations 1 and 2**, it is understood easily that small error sources are negligible in the uncertainty analysis.

Because the target uncertainty is usually composed of several independent factors for uncertainty, this method for obtaining a combined uncertainty is used frequently based on the method either Type A or B. An uncertainty analysis should begin with a consideration with analysis on the independent factors which contribute the target measurement uncertainty.

### 5.4.4 Consideration of uncertainty in grain moisture measurement

In practical measurements on grain samples, it is frequently required to evaluate the uncertainty involved in the measurement result obtained by a specific moisture meter in service. There are, however, a great amount of unpredictable factors in the procedures for evaluating the uncertainty in grain moisture measurement. Major sources of uncertainty could be: (1) non-uniformity/instability of sample, (2) difference in (or a lack of) sample characteristic due to its variety or year/place of harvest (and so on), (3) sampling method, (4) sample treatment and (5) a measurement standard (or reference method) used to calibrate the moisture meter.

When characteristics of the sample are not (or poorly) specified, the factors (1) and (2) become significant, and accordingly, an evaluation of uncertainty becomes difficult or the overall uncertainty

becomes significantly large. In such a case, we cannot distinguish errors due to the sample and the measurement procedure including the measuring instrument. In particular, evaluation of measurement uncertainty for fresh grain with high moisture content (over 20 % for rice) is difficult because it is unstable in characteristics and easy to be deteriorated.

Due to above reasons, it is impossible to evaluate unique value of uncertainty applicable to all grains and to all types of meters. A realistic evaluation could be achieved only if (1) a specified sample with homogeneous characteristic, (2) specified set of moisture meters and (3) a specified reference method would be employed. Experimental analyses on measurement uncertainty under such a specified condition are already reported in ISO 712<sup>4)</sup> and in the article by Tanaka, et al<sup>14)</sup>.

It is easy to provide an artificial sample with a constant property. However, it merely behaves as a standard resistor/capacitor to check the electric performance or a standard filter to check the optical measuring instrument. Uncertainty due to electrical or optical measurement is generally very small compared to those arise from the grain sample and measurement methods/conditions. Even if an artificial material with an ability to absorb/exhale moisture is provided, its characteristics would be different from those of real grain.

A universal evaluation method for uncertainty is requested in the member economies, but it is difficult to achieve due to above reasons. Employment of only one kind of meter with single calibration curve for all varieties might be an ultimate and easy solution. With this scheme, the only traceability system in one economy could be realized virtually. A measurement result by such a meter, however, is deemed as an unrealistic index for moisture content which may deviate from the true physical value. Such a scheme might be acceptable in one economy, but it may cause troubles in international trades. As it is mentioned in clause 5.3, this guide document proposes to employ a regional traceability system for moisture content with one agricultural product and one reference method.

The specifications prepared by the manufacturer of a moisture meter usually provide a kind of measurement uncertainty (or maximum limit of errors) when the meter has been manufactured and calibrated at the factory. The evaluation of uncertainty of a meter in service however strongly depends on the sample, environmental conditions, history/period of use, and procedures of measurement. Responsibility to evaluate the uncertainty in a practical use therefore belongs to the users, a testing/verification institute, or a national/regional authority.

## **6 Reference method for moisture content (drying method)**

### **6.1 Basic understanding of drying method**

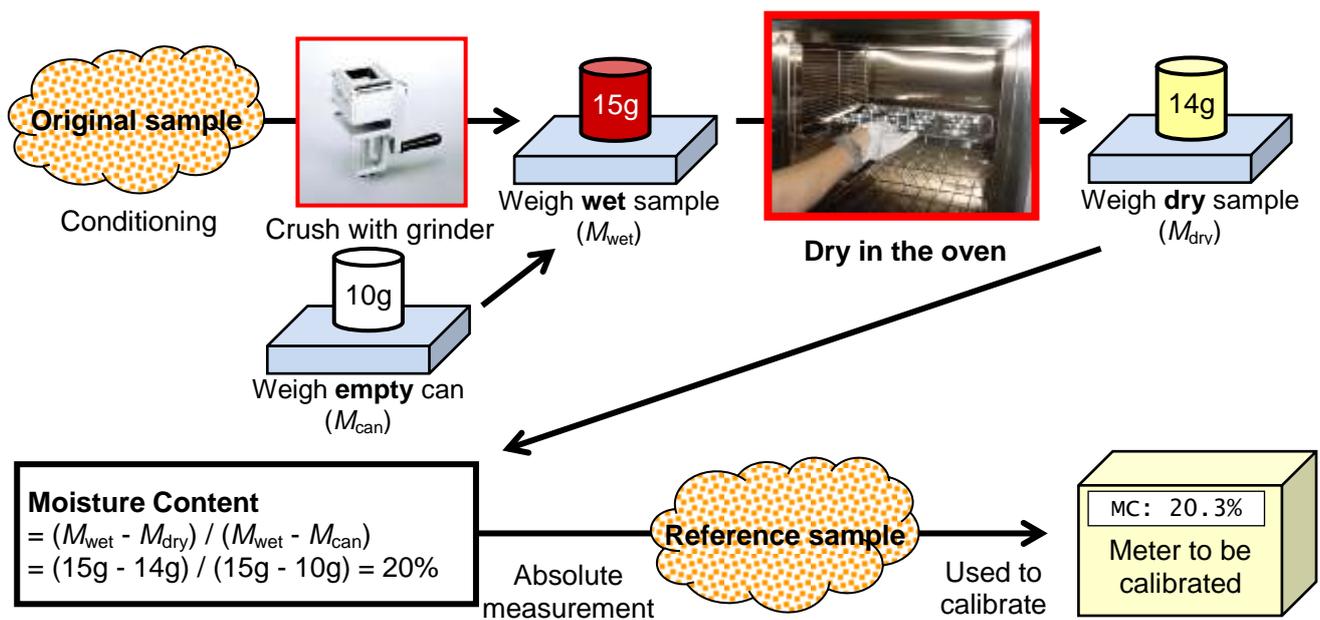
A drying method using an oven is capable of giving accurate and absolute measurement results of moisture content. This method is therefore widely employed as a reference method for traceability system for moisture contents in grains and other agricultural products.

In this method, a small portion of the finely ground sample is heated and dried in an air oven at a constant and uniform temperature. An absolute value of moisture content is calculated from the weight loss before and after drying based on an assumption that only aqueous substances are

evaporated off during this process. There is, however, an inherent problem that it is impossible to separate aqueous and non-aqueous substances in the grain clearly. Therefore, this method is empirical in nature. It means that the measurement results vary depending on drying conditions such as grain size, drying time, heating temperature and environmental humidity.

**Table 1: Internationally or regionally-recognized air oven methods for grain**

Organization/Products	ISO 712	USDA (see 6.4)	Japan/Rep. Korea (see 6.5)
Cereal grain	130 °C, 2 hrs	130 °C, 1 hr	106.5 °C, 5 hrs
Beans	130 °C, 2 hrs	103 °C, 72 hrs	106.5 °C, 5 hrs
Peas and lentils	130 °C, 2 hrs	130 °C, 1 hr	106.5 °C, 5 hrs
Maize [Food]	130-133 °C, 4 hrs	103 °C, 72 hrs	106.5 °C, 5 hrs
			[Feed]
Grain sorghum [Food]	130 °C, 2 hrs	130 °C, 1 hr	106.5 °C, 5 hrs
			[Feed]
Soybeans	103 °C, 3 hrs + extra	130 °C, 1 hr	106.5 °C, 5 hrs



**Figure 8: Schematic diagram of the procedure in the dry oven method**

Due to such a nature, different technical standards for drying method have been developed in various economies, regions and international organizations. Typical internationally-recognized drying methods are listed in **Table 1**. In general, as drying temperature becomes higher and drying time is extended longer, an apparent value of moisture content measured with a drying method becomes higher even for the same sample. It is due to a fact that aqueous substances cannot be separated clearly from others in grain, and some residual substances still evaporate gradually in a long time at a high temperature. A flow chart illustrating the procedures in a typical drying method

is given by **Figure 8**.

## 6.2 A drying method based on ISO 712

ISO 712: *Cereals and cereal products - Determination of moisture content - Reference method*<sup>4)</sup> is one of the air oven drying methods which is used world-wide for determining the moisture content in agricultural products. This international standard was revised in 2009 as fourth edition, and specifies a routine method for determination of the moisture content of cereals and cereal products.

### 6.2.1 Application

This method applies to wheat, rice (paddy, husked and milled), barley, millet, rye, oats, triticale, sorghum in the form of grains, milled grains and semolina or flour. ISO 712 is not applicable to maize and pulses. Instead, the method for maize is specified in ISO 6540<sup>5)</sup> and the method for pulses is specified in ISO 24557<sup>8)</sup>.

### 6.2.2 Definition of moisture

In ISO 712, moisture content is defined as “loss in mass, expressed as a percentage, undergone by the product under the conditions specified in this International standard”.

### 6.2.3 Apparatus and equipment needed in ISO 712

- (1) Analytical balance capable of weighing within an accuracy of  $\pm 1$ mg
- (2) Grinding mill with the following characteristics:
  - i) It is made of material which does not absorb moisture, easy to clean, has as little dead space as possible, enables grinding to be carried out rapidly/uniformly, does not generate heat, and does not allow the sample to contact the atmosphere for a long time.
  - ii) The mill is adjustable so as to obtain the particles complying with the criteria in **Table 2**. This table indicates the size and proportion of particles of the sample for which an additional grinding is not required. If the original particles are small enough, grinding is not needed.

**Table 2: Criteria to size and proportion of grain particles for which a grinding process is not required before drying**

Particle size (mm)	Proportion of the particles contained in the sample
$\leq 1.7$ (1.8)	100 %
$> 1.0$ (1.0)	$< 10$ %
$< 0.5$ (0.56)	$> 50$ %

( ) Mesh size of the sieve used to screen the particles.

- (3) Sample can (dish): A metal can which has suitable dimensions, non-corrodible under the test conditions, with a sufficiently tight-fitting lid (**Figure 9**). It should provide a sufficient surface

area for the contained sample with an areal density of sample less than 0.3 g/cm<sup>2</sup>.

- (4) Oven: The oven is electrically heated at a constant temperature between 130 and 133 °C (left in **Figure 10**). It shall have a sufficient heating capacity such that the original set temperature (e.g. 131 °C) would be regained within 30 min even after an insertion of the maximum number of test samples. The effective ventilation inside the oven is also one of important factors. To assess its effectiveness, apparent moisture content of durum wheat semolina is measured twice after drying for 2 hours and then after drying for 3 hours. (It takes 5 hours in total for drying.) Then, the effectiveness is assured if difference between the two apparent moisture contents is within 0.15 %. Otherwise, the amount of ventilated air must be adjusted.
- (5) Desiccator: it shall contain effective amount of desiccant, for which silica-gel is used frequently (right in **Figure 10**).



**Figure 9: An empty can (left) and cans containing rice samples (right)**



**Figure 10: A drying oven (left) and a desiccator (right)**

#### **6.2.4 Preparation of the test sample and preconditioning**

Refer Chapter 9 for the procedures of preparation of reference samples. Before grinding the samples, assessments are needed on particle size and moisture content of the original sample. If particle sizes of the sample conform to the criteria in Table 2, the sample shall not be ground. Otherwise, the moisture content of the sample shall be measured. If the measured moisture content is in the range between 9 % and 15 %, the sample shall be ground immediately.

If the moisture content is higher than 15 %, the sample shall go through a preconditioning process because a wet sample cannot be ground properly. In this process, the sample is dried until its moisture content decreases less than 15 %. The practical procedures are as follows.

- (1) Put an amount of slightly more than 5 g of a test sample into a metal can without a lid.
- (2) Weigh the total weight of the can including the test sample with a resolution of 1 mg.
- (3) Place the can in a drying oven at 130-133 °C for 7 -10 min for preconditioning in order to bring the moisture content to a value between 9 % and 15 %. The preconditioning time is different depending on the kind of sample and its original moisture content. The higher the original moisture content is, the longer the drying time is, and vice-versa.
- (4) Take the can out of the oven and cool down to the room temperature with a waiting time for 2 hours. If we put it on a desk made of stone or steel, the waiting time can be decreased.
- (5) Weigh the can including the test sample with a resolution of 1mg.
- (6) Grind the sample. Then, a preconditioned sample with an amount of 5 g is prepared.

### 6.2.5 Procedures for drying method

ISO 712 specifies to measure a pair of sample cans containing the same sample with an identical amount. The specified procedures are as follows:

- (1) Heat a drying oven up to the temperature  $130\text{ °C} \pm 3\text{ °C}$ .
- (2) Put test sample of an amount of  $5\text{ g} \pm 1\text{ g}$  to each of the two sample cans and close the lids.
- (3) Weigh each sample can with the lid with a resolution of 1 mg.
- (4) Put the sample cans into the drying oven with the lids open. The lid shall be placed under each can. Wait two hours from the moment when the oven resumes the original temperature of  $130\text{ °C} \pm 3\text{ °C}$ .
- (5) Open the oven, close the lid of each can, and take out the cans with the lids. Put them into a desiccator on a desk with the lids closed. Wait until the cans are cooled down to the room temperature (20-40 min).
- (6) Weigh each sample can with the lid containing the sample with a resolution of 1 mg.

### 6.2.6 Calculation of results and assessment

The moisture content ( $MC$ ) is given by the following equation in a case without a preconditioning.

$$MC(\%) = (1 - w_1/w_0) \times 100 \quad (3)$$

where  $w_0$  and  $w_1$  are the mass of the test portion and the dried test portion, respectively. In the case using a preconditioning, the moisture content is expressed by the following equation.

$$MC(\%) = \{1 - (w_1 - w_3)/(w_0 - w_2)\} \times 100 \quad (4)$$

where  $w_2$  and  $w_3$  are the mass of before and after preconditioned, respectively.

The calculated moisture content in percent should be rounded to the second decimal place. ISO 712 requires comparing the measurement results of moisture content for the pair of samples. If the absolute difference between the two results exceeds a criterion of 0.12 %, repeat the entire procedure in 6.2.5 until the difference becomes equal or less than the criterion.

### 6.2.7 Test report

The test report is recommended to include the following information:

- (1) Sample identification,
- (2) Sampling method,
- (3) Reference to international standard,
- (4) Test results,
- (5) Repeatability, and
- (6) Operating details that might influence the test results.

### 6.3 A drying method based on ISO 6540 for maize

ISO 6540<sup>5)</sup> specifies a drying method applicable to maize. There are two methods in this international standard: (1) a reference method for ground samples and (2) a routine method for whole grains are provided. Because this standard is based on the procedures specified in ISO 712, there are many descriptions in common between the two standards. A preconditioning with drying of a wet sample is also required before grinding it. A fundamental difference from ISO 712 exists in the drying method which has been improved to accommodate maize with a high moisture content that sometimes exceeds 40 %. Differences of ISO 6540 (reference method) from the requirements of ISO 712 are listed below.

- When a preconditioning is not required, a sample (8 g) is taken from a lump of ground grain (30 g) and dried at 130 °C for 4 hours.
- When a preconditioning is required, a lump of whole grain (100 g) is dried at 60-80 °C in advance. A portion (30 g) is taken from the dried grain and it is ground. Then, a sample (8 g) is taken from the ground grain.
- In the routine method (2), a sample of whole grain (20-40 g) is dried without grinding at 130 °C for 38 hours.
- Allowable maximum differences of the measurement results between a pair of samples are 0.15 % for the reference method (1) and 0.2% for the routine method (2).

## 6.4 A drying method employed by USDA in the United States

In the United States, a reference (drying) method<sup>15)</sup> is specified under a supervision of USDA (United States Department of Agriculture). This method is based on a reference method (44-15a) provided by the American Association of Cereal Chemists (AACC), and is practically implemented by GIPSA (Grain Inspection, Packers and Stockyards Administration) that belongs to USDA and aims to facilitate trade of agricultural products in the USA.

This method is composed of eight sub-methods which are tailored to accommodate a wide variety of products in type/size of grain and moisture level. The sub-methods employ a drying temperature of 130 °C and drying time of one hour in most cases as summarized in **Table 1** in 6.1.

This method of GIPSA/USDA is specified as a reference method for grain moisture meters in the handbook 44<sup>16)</sup> of NIST (National Institute of Standards and Technology), which specifies model technical specifications for measuring instruments controlled under the legal metrological system in each state. This handbook is also used as the basis for the type approval tests under NTEP (National Type Evaluation Program) in NCWM (National Conference on Weights and Measures).

## 6.5 A drying method employed in Japan and Republic of Korea

There is an original standard for drying method in Japan specified in an Agricultural Products Inspection Act provided by Ministry of Agriculture, Forestry and Fisheries (MAFF). This standard is applied in Japan as well as in Republic of Korea to the products: paddy, brown rice, white rice, wheat, barley, naked barley, buck wheat, soybean and beans in form of grain. Basic concepts and structure of the standard are similar to those of ISO 712. Significant differences from ISO 712 exist in the drying temperature ( $106.5 \pm 1.0$  °C) and the drying time (5 hours).

# 7 Electrical and inferential measurement methods for moisture content

## 7.1 Introduction of electrical moisture meters

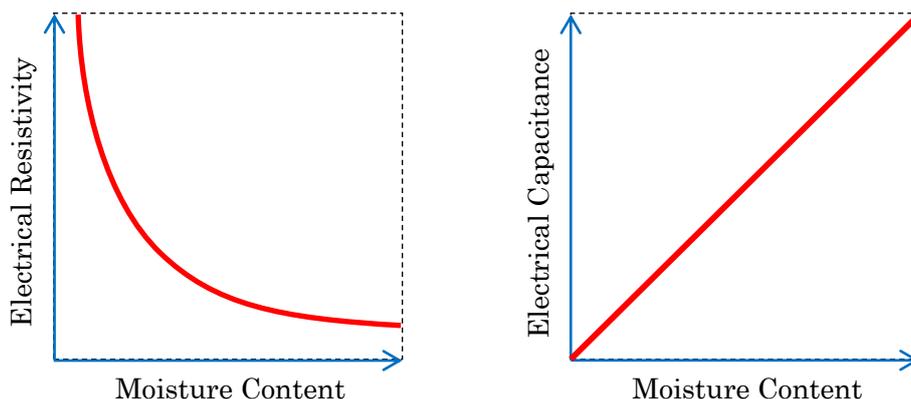
Electrical moisture meters enable a quick measurement of moisture content in grain. Therefore, electrical moisture meters are widely used in the inspection and the quality control of rice and other grains. Electrical moisture meters are classified into three types:

- (1) Electrical resistance (conductance) type,
- (2) Electrical capacitance (dielectric constant) type, and
- (3) Near infrared (NIR) type.

These moisture meters are utilized to measure electric or optical properties of grains, which generally have dependence on the moisture content, and to estimate the content empirically using a calibration equation. Therefore, it is impossible to measure moisture content in a sample directly. For example, the Electrical resistance of grain decreases according to an increase of its moisture content and the capacitance increases nearly proportional to the moisture content (**Figure 11**). In the case of NIR type, the meter detects an optical absorption ratio of infrared light when it passes

through the sample. The absorption ratio generally increases with moisture content. Changes in electric/optical properties attributed to the moisture content are therefore utilized in the measurements.

A disadvantage as well as the most critical requirement to the inferential moisture meters is the fact that they have to be calibrated in advance against a sufficient number of reference samples with a known value of moisture content. The calibration procedure includes a determination of a calibration equation as an approximation of measurement results on the reference samples.

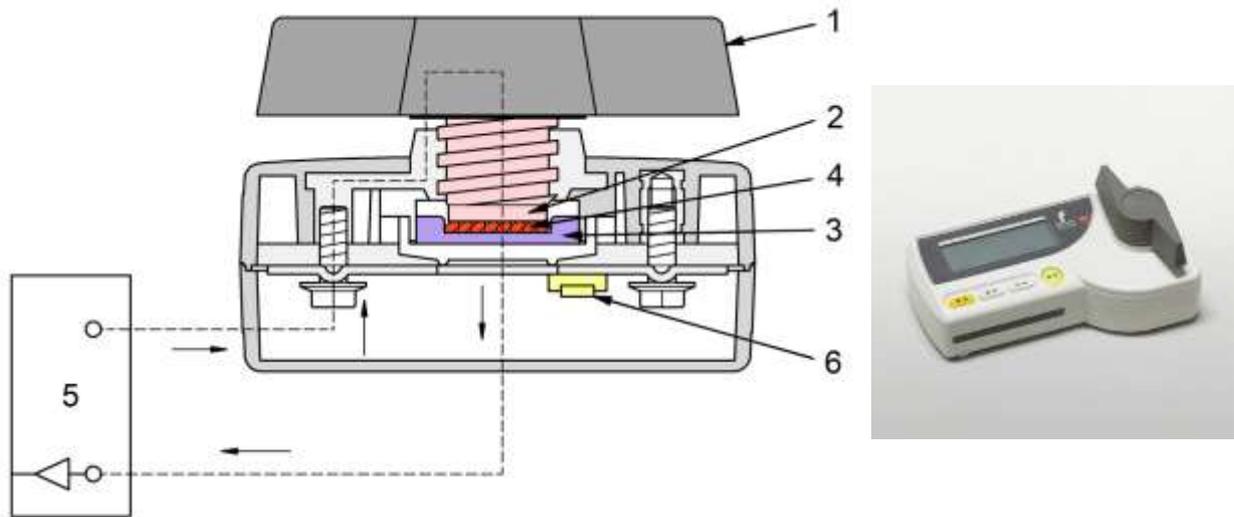


**Figure 11: Typical relationships between moisture content versus electrical resistivity and electrical capacitance of grains**

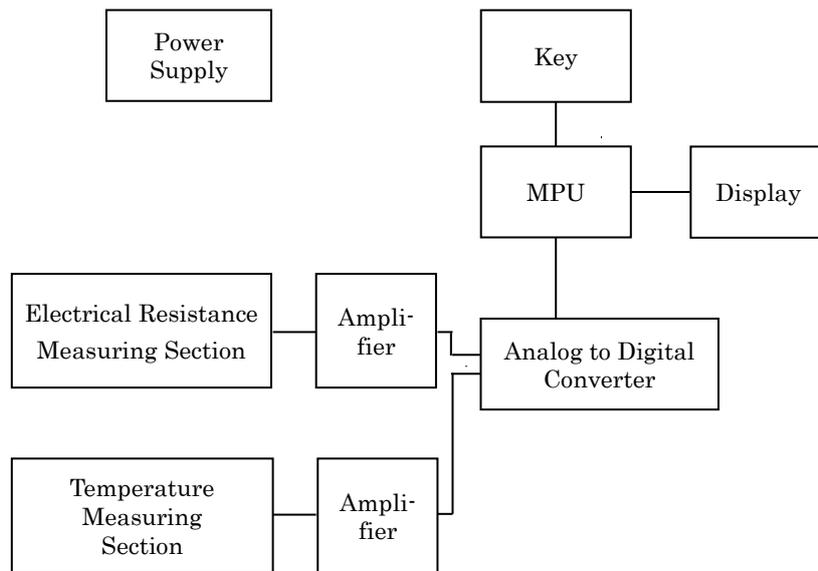
## 7.2 Electrical resistance type moisture meters

Electrical resistance (i.e., conductive) type moisture meters measure the electrical resistance of sample, and they have a typical structure and components as shown in **Figures 12 and 13**. For measurement of the moisture content, a small amount of sample (1 g typically) is crushed and sandwiched between two electrodes and electric current flows through the sample from one electrode to another. The magnitude of current, measured by the electronic circuit, is converted into moisture content and is indicated on the display unit. This type of meter has advantages such as, small size, light weight, easy operation/maintenance, rapid measurement, and long-time stability in metrological characteristics.

In **Figure 12**, a grain sample is set on a sample tray in the testing chamber (4) between upper electrode (2) and lower electrode (3), and crushed by the handle (1). A/D converter (5) detects the electric current passing through the sample, and converts it to digital data in order to compute moisture content. Temperature sensor (6) measures the temperature of the moisture meter simultaneously.



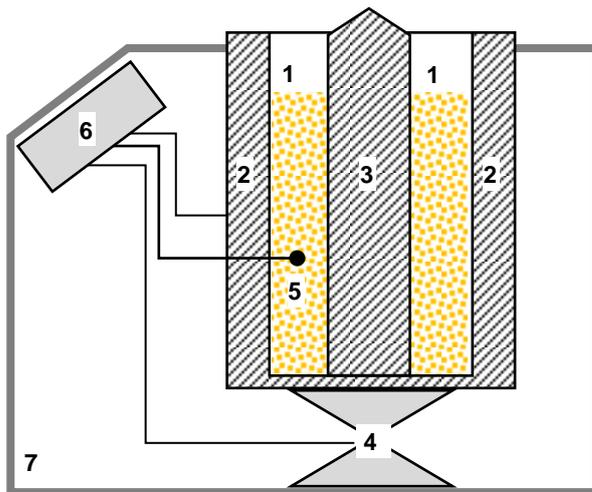
**Figure 12: A cross-sectional view of an electrical resistance type grain moisture meter and its outside view**



**Figure 13: A block diagram of an electrical resistance grain moisture meter**

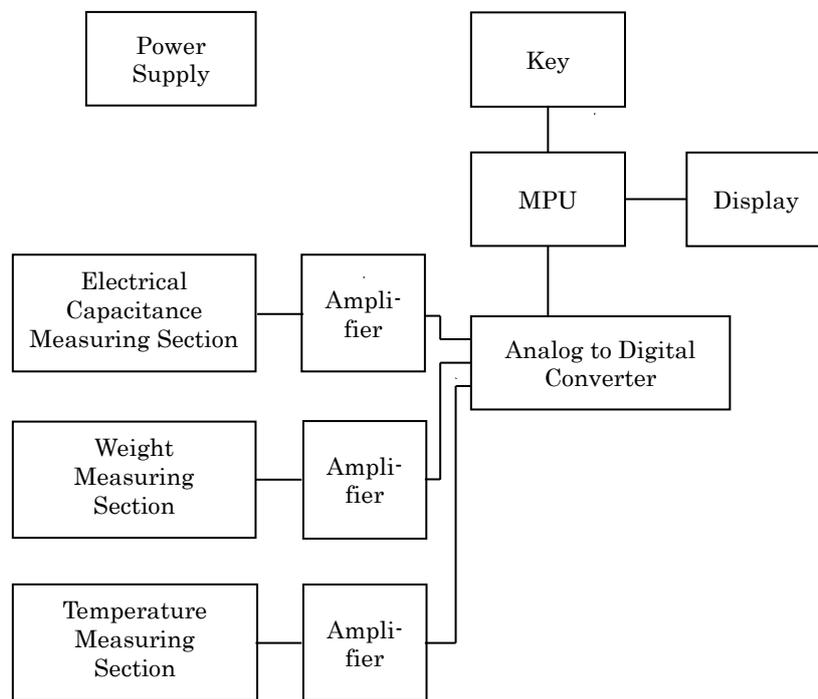
### 7.3 Electrical capacitance type moisture meter

Electrical capacitance type moisture meter utilizes electromagnetic wave (20-150 MHz) applied to a sample between a pair of electrodes. The electromagnetic wave is partly absorbed by molecular movements of the sample. The absorption ratio of the wave has a close relationship with the Electrical capacitance of the sample which is proportional to its dielectric constant (relative permittivity). The Electrical capacitance is almost in proportion to the moisture content as shown in **Figure 11**.



1: sample chamber (annular shape), 2: outer electrode, 3: center electrode, 4: weighing instrument (load cell), 5: temperature sensor, 6: electric circuit (including a processing unit and a display unit), and 7: housing

**Figure 14: A schematic diagram of an electrical capacitance type moisture meter and its outside view**



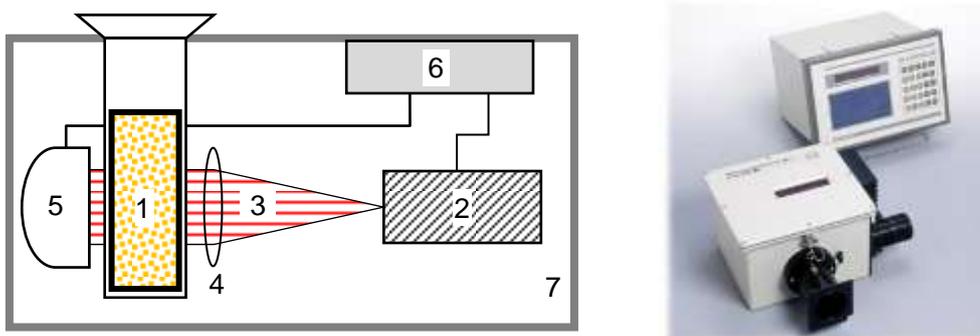
**Figure 15: A block diagram of a capacitance (dielectric) grain moisture meter**

Thus, the absorption ratio of the electromagnetic wave is related to the moisture content with an aid of a calibration equation. Weight of the sample must be measured simultaneously because the absorption ratio is also related to the amount of sample. Electrical capacitance type meters have a typical structure and components as shown in **Figures 14 and 15**.

For measurement of the moisture content, a lump of sample (100-500 g typically) is put into a cylindrical container with a pair of electrodes and electromagnetic wave passing through the sample. The magnitude of absorption of the electromagnetic wave is measured by the electronic measuring unit, converted into moisture content and indicated on the display unit. This type of meter has advantages such as, being non-destructive without a need for crushing the sample, measurement of the mean value of a lump of sample, easiness in operation/maintenance, rapid measurement, and a long-time stability of metrological characteristics.

### 7.4 Near infrared (NIR) type moisture meter

Near infrared type moisture meter irradiates infrared light (800-2400 nm in wavelength) to a sample. As the light passes through the sample, it is partly absorbed by molecular movements of grain. An absorption ratio of the infrared light is related to the moisture content by a calibration equation. This type of meter has similar advantages with those of the capacitance type (7.3). In addition, it enables a rapid and remote measurement even on a sample in motion during a transportation process in a factory. Such meters have a typical structure and components as shown in Figures 16 and 17.



1: sample holder (transparent and rectangular shape), 2: light source (laser or monochromator), 3: infrared light beam, 4: lens, 5: photo detector, 6: electric circuit (amplifier, control unit, processing unit and display unit) and 7: housing

Figure 16: A schematic diagram of a near infrared (NIR) type moisture meter and its outside view

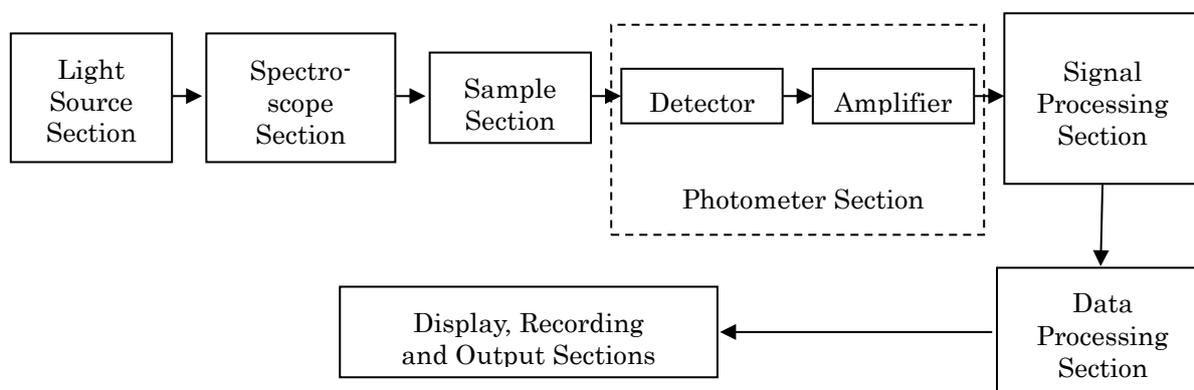


Figure 17: A block diagram of a near infrared (NIR) type grain moisture meter  
Example of a dispersive near infrared spectrophotometer

## 8 Checking procedure for moisture meters based on ISO 7700

### 8.1 Introduction of ISO 7700

This chapter briefly introduces ISO 7700: 2008 “Checking the performance of moisture meters in use - Part 1 and 2” <sup>67)</sup>. This international standard specifies the method for checking the performance of moisture meters in service, and it is employed in many economies/regions. The primary purpose is to decide whether replacement or repair is required for a moisture meter. This standard, however, is not intended for use in legal metrology, such as type approval or verification of moisture meters (see Chapter 10 about OIML R 59 for legal metrology). This standard introduces a comparison between the measurement results of moisture content of reference samples obtained by (1) a moisture meter to be tested and (2) the reference method (drying method). Procedures specified in ISO 7700 are illustrated in **Figure 18**.

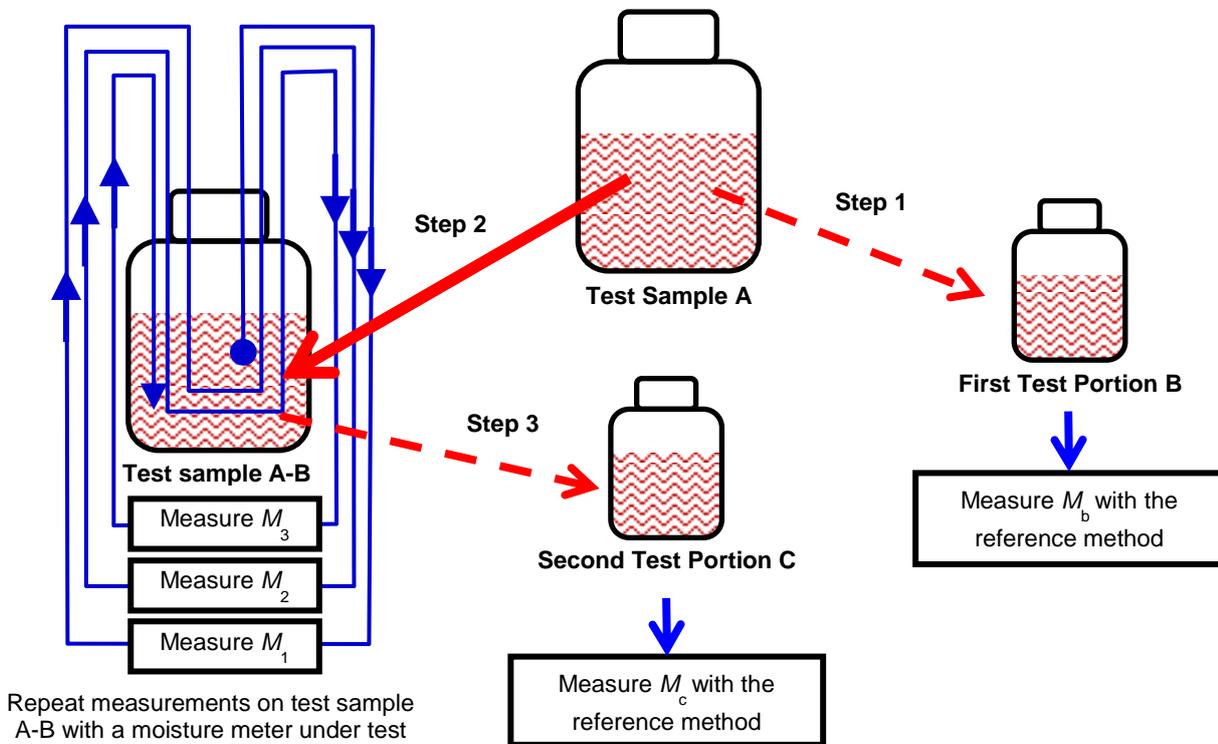


Figure 18: A schematic diagram of calibration procedures based on ISO 7700

### 8.2 Preparation of the samples

Select varieties from those most prevalent in the region. Clean the samples by removing impurities and undersize grains. Select at least two cereal species (or two different grain types for maize or rice). From each species, select two samples at least with different moisture contents within the measurement range, and store them in bottles (2/3 filled each). These bottles are called as samples “A”. These samples shall have natural moisture without conditioning.

### 8.3 Procedures for checking

#### (1) Determination of the initial moisture content

Quickly take a test portion (B) from each of the samples (A). Put the test portion (B) into other bottles (2/3 filled) by taking care not to modify moisture content. Determine the moisture content of the test portion (B) using a reference method (oven method) in accordance with ISO 712 or ISO 6540. The samples left in the bottles of sample (A) are referred to as samples “A-B” in the following procedures.

#### (2) Stabilization of temperature

Stabilize the temperatures of test samples (A minus B) and moisture meter until equilibrium is established. Because a temperature difference between the meter and the samples may influence the measurement results, measure and record the room temperature with a thermometer. Recommended room temperature is in the range of 15 °C to 25 °C.

#### (3) Check of the moisture meter

Homogenize the test sample (A-B) by shaking the bottle. Open the bottle and make sure there is no odor of corruption or fermentation. Take a small portion from the test sample (A-B). Repeat at least three successive measurements of moisture content on this portion with the moisture meter to be tested. After each measurement, return the portion to the bottle of test sample (A-B), and re-mix the bottle before next measurement.

#### (4) Determination of the final moisture content

After checking the moisture meters, take quickly a second reference test portion “C” from the test sample (A-B). Put the test portion (C) into another bottle (2/3 filled) by taking care not to modify moisture content. Determine the moisture content of the test portion (C) by the reference method.

### 8.4 Treatment of the measurement results

For each of the test samples (A) or (A-B), the following values shall be obtained:

- $MC_b$  : Moisture content of the first test portion B (before checking)
- $MC_c$  : Moisture content of the second test portion C (after checking)

If the difference between  $MC_b$  and  $MC_c$  exceeds 0.3 %, repeat the entire procedures for checking. The mean of the two values is assumed as the true value ( $MC_{true} = [MC_b + MC_c]/2$ ) of the moisture content of the sample (A). Then, calculate errors ( $E_i$ ) from the true value ( $E_i = MC_i - MC_{true}$ ) for the three (or more) measurement results ( $MC_i$ ) on the sample (A-B).

## 8.5 Assessment of the measurement results with MPEs

In order that a moisture meters passes the checking, the errors ( $E_i$ ) at a  $MC$  (moisture content) shall be less than the MPEs shown below:

- Cereal grains (other than maize, rice and sorghum):  
MPE = 0.7 % (constant value) at  $MC \leq 10$  %  
MPE = 3 % (relative value) + 0.4 % (constant value) at  $MC > 10$  %
- Maize, rice and sorghum:  
MPE = 0.8 % (constant value) at  $MC \leq 10$  %  
MPE = 4 % (relative value) + 0.4 % (constant value) at  $MC > 10$  %

In the above expressions, 'relative value' means a relative fraction of  $MC$  expressed in percent (%). For example: in a case of cereal at  $MC = 20$  %,  $MPE = 0.4 + 20 \times 0.03 = 1.0$  %, and in a case of maize at  $MC = 30$  %,  $MPE = 0.4 + 30 \times 0.04 = 1.6$  %.

## 9 Preparation of reference samples

This chapter describes recommended methods and procedures to prepare/condition reference samples and adjust their moisture contents. Reference samples with a stable and homogeneous quality are necessary to maintain the traceability system, and used practically for the reference method (drying method) and calibration of moisture meters using a meter-to-meter comparison. Rice is considered as the model grain used for the present methods and procedures.

### 9.1 Collection and preconditioning of grain reference samples

As mentioned in Chapter 5, selection of reference samples is a critical issue to establish the traceability system proposed by this guide document (see **Figure 6** in 5.3). From a view point of setting up the sole traceability in the region, the reference sample shall represent the target varieties of the grain product (i.e., rice, wheat, barley) which are widely produced and are traded in the region. In order to comply with this fundamental requirement, a large number of grain samples of the product, consisting of as many varieties as possible, should ideally be collected from various places in the region. In reality, however, due to the limitations to time and labor cost, the collection is conducted as efficiently as possible.

There is no essential difference among the samples used for each step necessary to maintain the traceability system. The step may correspond to the reference methods (oven method) or calibrations of primary/secondary standard meters. It is therefore recommended that a national/regional authority/laboratory provide a set of reference samples with a sufficient amount and to utilize them for all purposes. Usually, the original collected sample is conditioned and is separated into several samples with different levels of moisture content. Practical procedures for preparation and conditioning the samples are described below.

#### 9.1.1 Collection of grain for reference samples

Before collecting grain samples, the kind and form of the target grain shall be decided by the

national/regional authority. The 'form' means the most common form of product in which the grains are traded and are distributed. In the case of rice, it indicates one or more forms among paddy rice, brown rice and polished rice. The following are the basic policies to be maintained when reference samples are collected.

- (1) Collect samples in order that they include more than 70 % of the varieties of the target grain produced and circulated in the economy/region.
- (2) Collect samples from more than 70 % of the places where the target grain is produced. Where, the percentage may be evaluated based on the number of administrative districts or total area of cultivation.
- (3) Record information of the collected samples by specifying kind/variety of grain, places of production/sampling and year of harvest. Other information may be specified as necessary.
- (4) Use fresh grains that were harvested within one year. Aged grains more than one year should be considered as informative. If aged grains, however, are customarily distributed and traded in the region, collect approximately the same number of samples produced in each year (current year, one year ago, two years ago and so on).
- (5) Make a realistic collection plan by taking actual situation of production and/or circulation of grains (amount, variety, places and so on) into consideration. If the sample collection plan disregards real situation, collected samples would not be considered as representatives of the target varieties. Thus, the sample collection plan is important to ensure the generality and representativeness of the reference samples.
- (6) The total amount of samples should be decided based on the plans in which the samples are conditioned and utilized. The minimum amount (A) for one kind of sample at one level of moisture content is decided as the total amount necessary for the reference method and meter-to-meter comparisons. In the case of calibrating a resistance-type moisture meter, a typical value of the minimum amount (A) is 50 g. It is recommended to prepare twice of the minimum amount ( $2 \times A$ ) considering a reduction by removing foreign materials. If one sample is divided into two levels of moisture content, the minimum amount of sample to be collected should be  $2 \times 2 \times A$ .
- (7) When the locations of sampling and the laboratory for sample conditioning are different, the sample should be immediately moved to the laboratory. It is necessary to control storage conditions (temperature and humidity) for the sample in order to prevent a transformation during temporal storage or transportation.

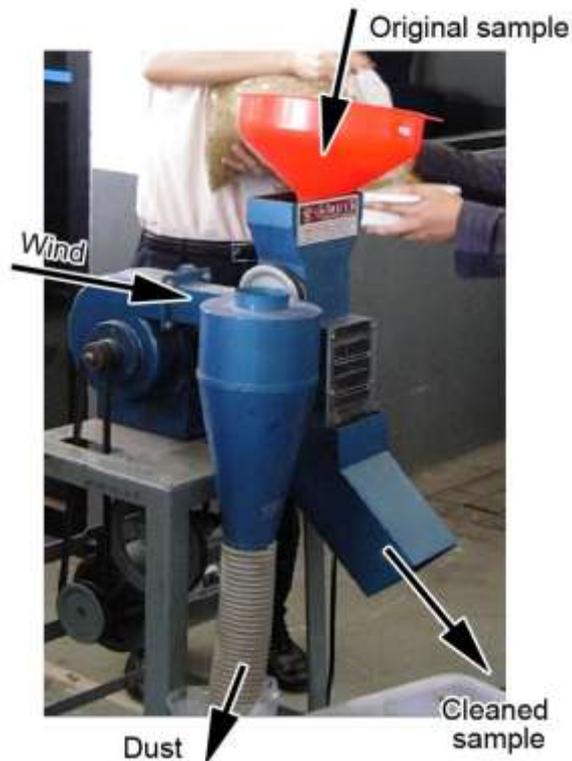
### 9.1.2 Preconditioning of sample

Collected samples sometimes contain foreign materials, such as small pieces of stones, straw, dust, and so on. As these foreign materials may cause errors in moisture content or may damage the instruments, they should be removed using the following procedures.

- (1) When a roller-type electric husker is available, it should be used as a wind selector (**Figure 19**). Separate the two rollers to the maximum distance and maintain sufficient air flow to prevent

the husker from being stuck by the sample. If a husker is not available, use a combination of two sieves A and B (**Figure 20**). Sieve A has rectangular holes with the size 1.7 mm × 10.5 mm and Sieve B has holes with the size 2.3 mm × 10.5 mm. [Hereafter, procedures using the sieves are shown in parentheses.]

- (2) Adjust the wind control damper of the wind selector (husker) and remove light foreign materials such as straw pieces. [For sieves: Put a sample of approximately 500 g onto Sieve A and shake it. Then, remove foreign materials that rose to the surface or dropped under the sieve as shown in **Figure 20**.]
- (3) Foreign materials with a high density (small pieces of stones, clods, and so on.) cannot be removed by wind. Place the sample on a bat or sieve and shake a little. Pick out foreign materials by hand and remove them. [For sieves: Put the sample left at the procedure (2) onto Sieve B and shake it. Then, remove any foreign materials.]
- (4) After conditioning by removing foreign materials, measure the moisture content of the sample three to five times using a calibrated moisture meter. The average of the measured values should be recorded as the original moisture content of the sample.
- (5) The sample should be mixed well and then should be divided to make reference samples at different levels of moisture contents.



**Figure 19: Preconditioning of grain using an electric husker**



**Figure 20: Cleaning procedure of grain with a sieve**

## 9.2 Adjustment of moisture content

A procedure for calibrating moisture meters by defining a calibration curve requires many reference samples in a wide range of moisture content. There are two requirements in arranging samples at different moisture contents. Firstly, the moisture contents of the samples should cover both the maximum and minimum moisture contents within the target range of calibration. Secondly, distribution of the discrete moisture contents should be uniform over the entire range of calibration. A typical example of distribution is given in **Figure 5** in 5.1.

For these purposes, it is realistic and convenient to select one sample, which has original moisture content close to the target value, from many collected samples with different moisture contents in a wide range. Then, adjust moisture content of the selected sample to the target value by measuring its moisture content repeatedly. The adjustment is conducted either by drying the sample or by drying after moistening. The former method is used when the target value is lower than the original moisture content and the latter is used otherwise. A moistening procedure requires a longer time and it may change the original characteristics of grain however. It is therefore strongly recommended to use a drying method. Regarding artificial moistening of the sample, see 9.2.2.

In reality, a lump of fresh sample with the same characteristics (variety, year/place of harvest) is normally provided in a bag or a container. Then, it is divided into two lumps of sample, and moisture content of each sample is adjusted independently with a drying procedure down to two different levels. For example, a fresh sample with the moisture content of 30 % may be adjusted to two target moisture levels, 12 % and 15 %, respectively. Repeating this procedure for various samples by varying the target moisture levels, many reference samples with different moisture levels in a wide range should be provided.

### 9.2.1 Procedures for adjustment by drying

- (1) Prepare a mesh tray (or a sheet of newspaper) as a sample holder.
- (2) Measure the temperature and relative humidity in the room.
- (3) Find the equilibrium moisture content  $MC_e(t, h)$  of paddy from the room temperature ( $t$  °C) and relative humidity ( $h$  %) using the graph in **Figure 3** in 4.3. If a sample other than paddy is used, **Figure 3** should be considered as reference data.

- (4) Measure the tare weight of the mesh tray (or newspaper) by a balance with a resolution of 1 g.
- (5) Put a sample with original moisture content ( $MC_0$  in %) on the mesh tray (or a newspaper) and level it evenly (**Figure 21**). Measure the weight ( $W_0$ ) of the sample with the balance used above.
- (6) Use a drying method when the target moisture content ( $MC_x$  in %) is lower than the original moisture content ( $MC_0$ ). The weight of the sample when its moisture content reaches  $MC_x$  is calculated as  $W_x = W_0 \times (100 - MC_0)/(100 - MC_x)$ .
- (7) When [equilibrium content ( $MC_e$ ) < target content ( $MC_x$ )], spread the sample thinly on a sheet of newspaper and blow air using an electric fan (**Figure 21**). Measure the mass of the sample at appropriate intervals. When it reaches  $W_x$ , store the sample into a polyethylene bag.
- (8) When [equilibrium content ( $MC_e$ ) > target content ( $MC_x$ ), or  $MC_x \approx MC_e$ ], place the sample together with the mesh tray into a dryer and dry the sample at 30°C. Measure the mass of the sample at an appropriate interval. When it reaches  $W_x$ , store the sample into a polyethylene bag. While drying, set the air damper at 50 % open.
- (9) If the moisture content of the sample does not decrease to the target value due to low ambient temperature and/or high relative humidity, reconsider (or change) the lower limit of the target moisture content. If a sample with high moisture content is exposed to a temperature higher than the room temperature for a long time, the sample may be transformed due to the effects of breathing and bacteria. The target moisture content should be set within the range of the safe storage period of the sample by considering the original moisture content. Recommended maximum periods of safe storage are given in **Table 3**.

**Table 3 Maximum safe storage period of paddy rice (informative)**

Moisture content of sample (%)	Ambient temperature		
	20 °C	10 °C	5 °C
	Maximum safe storage period (days)		
16	60	120	170
22	7	14	20
26	2	4	5

- (10) Samples stored in polyethylene bags should be thoroughly mixed (**Figure 22**). Record sample information on a label and attach to the bags (**Figure 22**), then homogenize the sample as described in Clause 9.3.1.



Figure 21: Drying on a tray (left) and using an electric fan (right)



Figure 22: Samples mixed and stored in bags (left) and an example of sample label (right)

### 9.2.2 Procedures for adjustment by moistening (informative)

It is well-known that electrical or optical characteristics of two samples with the same moisture content may slightly be different depending on their hysteresis whether the sample has been dried or moistened before reaching the moisture level. In the measurements of moisture content, a sample after a drying process is generally considered as appropriate to be measured. It is because most of the grain samples are prepared using a drying process, and they are usually sold in a dry condition at the market.

An artificial moistening process, however, may be requested in preparing reference samples in a wide range of moisture level. Although such a moistening procedure is not recommended generally, the following precautions should be observed when such a procedure is inevitable.

- (1) A sample should be moistened in an atmospheric environment with a high humidity (70-90 %) near the room temperature. Procedures shown in **Figure 23**, which uses a closed tray with a mesh to separate the sample from the water, are recommended.



From left to right; (1) provide a tray with a mesh and a cover, (2) pour water on the bottom of the tray, (3) put the mesh and place the sample flatly on it, then close the cover and (4) wait until the sample is moistened in the tray.

**Figure 23: Procedures for moistening sample**

- (2) Forced moistening methods such as, dipping in water or spraying with water, shall not be used. If such methods are used, transformation in physical and chemical characteristics of the grain may occur.
- (3) The target moisture content ( $MC_x$ ) should always be reached after a drying process. It means that the sample should be over-moistened (target level +2%), and then dried slightly.
- (4) It is not recommended to moisten the sample by a large amount, or moisten up to a level higher than 21 %. When a sample with high moisture content is needed, another natural sample that originally has high moisture content should be sought. Before employing a moistening method, find a natural sample which has moisture content close to the target level ( $MC_x$ ).
- (5) After the moistening process, the sample should be stored in a polyethylene bag attached with a suitable label specifying necessary information. Then the sample should be subjected to the following homogenization and storage processes.
- (6) A special care should be taken because the sample deteriorates easily under the moistening process with high humidity. See **Table 3** for the maximum storage periods.

### 9.3 Homogenization of samples and storage at low temperature

A sample reached at the target moisture content should be homogenized in moisture content and should be stored at low temperature (5 °C).

#### 9.3.1 Homogenization

Even after the mean moisture content of a sample reached the target value, an inhomogeneity of moisture content among grain kernels is still unavoidable. In general, there is a tendency that the inhomogeneity becomes larger after the mean moisture content is artificially changed over a large amount in a short time. In general, internal and surface diffusion of moisture among grain kernels become larger as the storage temperature rises. At the same time, the grains breathe more actively and bacteria on them are also active at a high temperature, and accordingly these effects accelerate deterioration and/or transformation of the sample. Therefore, the condition for better homogeneity and that for long-term storage are under a trade-off relationship, and both of them are closely related to the temperature. It is therefore necessary to take these contradictory effects into consideration to

decide the best period for homogenization. Typical examples of the period are shown in **Table 4**.

**Table 4: Examples of the period for homogenization (informative)**

When a sample has been adjusted to the moisture content of 22 %, a recommended homogenization period would be 5 days at 20°C and 10 days at 10°C.

Moisture content of the sample (%)	Ambient temperature	
	20 °C	10 °C
	Period for homogenization (days)	
16	20	40
22	5	10
26	2	4

### 9.3.2 Low temperature storage

Storage of grain samples at low temperature suppresses self-digestion by breathing as well as deterioration by bacteria. It is not possible, however, to maintain the quality of the samples forever even if they are stored at 5 °C. Large temperature distribution within the sample also accelerates its deterioration.

For better storage, it is necessary to seal the bag containing a sample. When the surface area of the sealed bag is large, temperature distribution in the sample becomes smaller and becomes close to that of the environment. A sample with moisture content higher than 20 % should be used within 30 days even if it is stored at low temperature.

### 9.3.3 Precautions to use the sample stored at a low temperature

When a sample, which is stored at a low temperature for a long time, is used for a measurement at the room temperature, the container should not be opened immediately after taking it out of the refrigerator. When a cold sample is exposed to the atmosphere, vapor condensates on the surface and it changes the moisture content. In addition, when an electric moisture meter is used, a measurement error may be caused if a large temperature difference exists between the instrument and the sample. Therefore, after a sample is stored at low temperature, leave the sample bag at the room temperature without opening the bag for at least 4 hours before measurement.

Even if measurements on the sample with high moisture content cannot be completed within one day, the sample should not be exposed to the room temperature for a long time. Therefore, in order to prevent transformation of the sample, it is recommended to take a small but sufficient amount of sample from the bag.

## 10 Outline of OIML R 59

### 10.1 Introduction of R 59

*[Note by the WG: This clause will be updated after a new R 59 is published in 2016 or later.]*

OIML R 59 (1984) *Moisture Meters for Cereal Grains and Oilseeds*<sup>1)</sup> is one of the OIML International Recommendations which have been provided as model technical regulations of measuring instruments to be introduced by the member states for type approvals and/or verifications. Although OIML is not an enforcement organization, the member states are morally recommended to implement the requirements of OIML Recommendations (as well as other decisions by OIML) in their national legislation systems in legal metrology.

R 59 practically aims at moisture meters based on near infrared, electrical capacitance and electrical resistance types. R 59 consists of (1) technical requirements, (2) test procedures for conformity to the requirements, and (3) test report format. This basic structure is common for most of the OIML Recommendations.

R 59 is provided and is maintained by OIML TC 17/SC 1 (Humidity) chaired jointly by the PR China and the USA. A process to revise R 59 (1984) started in 2001. In this process, APLMF Working Group Quality Measurement on Agricultural Products has submitted comments from a viewpoint of Asian economies.

## 10.2 Outline of R 59

*[Note by the WG: This clause will be updated after a new R 59 is published in 2016 or later.]*

R 59 specifies general technical requirements on construction, indication device, conversion tables, and security requirements to measurement. The scope mentions that this recommendation is applied to measurement of moisture contents of grains and oilseeds with a direct or indirect (with a conversion table) method. This recommendation is applicable only to static samples (do not move during measurement) in two categories of meter A and B explained below.

- **Category A** (automatic): Entirely automatic and single complete instrument with a printing device. Security to measurement results is required.
- **Category B** (non-automatic): Moisture meters which do not belong to the category A. The completeness of the instrument and security are also required.

In definitions of terms, ‘moisture content’ is defined as “a loss of mass under test conditions in accordance with ISO 712”. Maximum permissible errors (MPE) are defined for: (1) purposes of type approval, initial verification and verifications in-service; (2) categories of the instruments of classes I & II; and (3) species of sample: maize, rice, sorghum, sunflower, and so forth. For example, MPEs in a type approval for the instrument in ‘class I’ used for rice are 0.4 % in mass fraction ( $\leq 10$  % MC) and 4 % in moisture content ( $> 10$  % MC).

To ensure fairness of the measurement operations, the sellers are required to guarantee the following items: installation condition (instrument should be visible by all parties), preparation of sample and condition for use. For the metrological controls, attachment of a stamping/verification mark is required based on the national/regional requirements. Regarding seals for protection, parts containing mechanical and/or electrical measuring functions shall be sealed from unauthorized access. Inscriptions on the instrument should identify information on; manufacturer, model, serial number, class, range, calibration, type approval, measuring range, temperature range of use, and so

forth (if applicable).

In addition to the main text, R 59 is supplemented with three appendixes shown below.

*Appendix I: Practical reference methods for the verification of moisture meters used for the determination of moisture content of cereal grains.* This appendix introduces a reference method for cereal grains with a reference to ISO 712 (1979).

*Appendix II: Routine reference method for the verification of moisture meters used for the determination of moisture and volatile matter content of oilseeds.* This appendix introduces a routine reference method for oilseeds with a reference to ISO 665<sup>2)</sup> (1977).

*Appendix III: Metrological controls.* This annex introduces model procedures for type approval and initial/periodical verifications. ISO 7700 part 1 and 2 (1984) are referred as the reference method for checking the moisture meters.

## **11 Software and sealing for the moisture meters**

There has been an increasing concern about sealing of metrological parameters in the moisture meters to prevent a fraud or an unauthorized use of the instrument. Because all of the latest moisture meters are an electrical-type with digital processing, security on the software installed in the instrument has also become important.

To meet those requirements on software-controlled measuring instruments, OIML TC 5/SC 2 (software) developed D 31 “General requirements for software controlled measuring instruments” in 2008<sup>17)</sup>. This document is a kind of directory that includes all requirements on software applicable to all digital measuring instruments under legal metrology. Because of its wide range of application, these requirements are provided in two levels; (I) normal severity level and (II) raised severity level. D 31 also specifies two validation procedures for software in Table 2; (A) normal examination level and (B) extended examination level. The severity level and validation procedure are closely linked.

When the requirements in D 31 are applied, it should be noted that all of them would not be applied automatically to all measuring instrument. It is rather recommended that a respective OIML TC/SC or national authority should choose the best severity level as well as validation procedure to be applied to the instrument under test/verification.

Method for sealing has changed due to technological development. A traditional method was a hard sealing using stickers, wires and sealing metals. As software-controlled instruments are used widely, a soft sealing method of the instrument with a password (and other methods) has become more common. Moreover, many moisture meters are connected recently to the Internet. For these meters, a protection from all unauthorized access through the network has become important.

A new version OIML R 59 is expected to be published in 2016 or later. This version will include many practical requirements on software and sealing based on D 31 compared to those mentioned in R 59 (1984). This guide proposes to follow the new R 59 for requirements on software applicable to moisture meters for type approval or verification in each economy/region.

## 12 References

### 12.1 Publications

- 1) OIML R 59: 2016 “Moisture meters for cereal grains and oilseeds” (to be published)
- 2) ISO 665: 2000 “Oilseeds - Determination of moisture and volatile matter content”
- 3) ISO 711: 1985 “Determination of moisture content - basic reference method” (withdrawn)
- 4) ISO 712: 2009 (fourth edition) “Cereals and cereal products - Determination of moisture content - Reference method”
- 5) ISO 6540:1980 “Maize - Determination of moisture content (on milled grains and on whole grains)”/ Section one : “Reference method” / Section two : “Routine method on whole grains”
- 6) ISO 7700-1: 2008 “Checking the performance of moisture meters in use - Part 1: moisture meters for serials”
- 7) ISO 7700-2: 2011 "Checking the performance of moisture meters in use - Part 2: moisture meters for oilseeds"
- 8) ISO 24557: 2009 “Pulses - Determination of moisture content - Air-oven method”
- 9) JCGM 200:2012 “International Vocabulary of Metrology – Basic and General Concepts and Associated Terms (VIM 3rd edition)”
- 10) OIML V1: 2013 “International vocabulary of terms in legal metrology (VIML)”
- 11) ISO/IEC Guide 98:1993 “Guide to the expression of uncertainty in measurement (GUM)”
- 12) JCGM 100:2008 “Evaluation of measurement data - Guide to the expression of Uncertainty in Measurement (GUM)”
- 13) Hisashi Uedaira, “What is the water - as seen in the micro behavior” (in Japanese), Blue Backs (B-1646), Kodansha Ltd. (2009)
- 14) Hideyuki Tanaka, Toshimichi Watanabe and Mihoko Yabe, “Evaluation of uncertainty of the rice moisture measurement by the drying method”, Proc. XVIII IMEKO World Congress, Rio de Janeiro, Brazil (2006)
- 15) G. Dian Lee, “Grain Moisture Air-Oven Reference Methods in the United States”, Weights and Measures Connection Newsletter of NIST (National Institute of Standards and Technology), Vol. 3-6 (November, 2012)
- 16) Section 5.56 (a) of NIST Handbook 44 “Specifications, Tolerances, and Other Technical Requirements for Weighing and Measuring Devices” (2015 Edition)
- 17) OIML D 31: 2008 “General requirements for software controlled measuring instruments”

### 12.2 Web links

- 18) <http://www.aplmf.org/>
- 19) <http://oiml.org/>
- 20) <http://en.wikipedia.org/>

## **13 Annex: Instruction manual for checking grain moisture meters (informative example)**

### **13.1 Introduction**

This chapter provides an informative annex as an example of instruction manual how to check the moisture meters in service. Resistance-type and capacitance-type meters of Kett Electric Laboratory Corporation are selected as model instruments. This manual is also applicable to other moisture meters used in the member economies which have similar functions and capabilities.

Accuracy check of moisture meter is most important to keep the meters in a normal condition. Annual check is recommended, and a shorter interval is preferred.

Normally, an electrical moisture meter is composed of two main parts: electric circuits and a mechanical part. A part with electric circuits can be easily checked with an electrical checker. It is not easy to check the mechanical part, however. Therefore, a reference grain sample, whose moisture is already known, is used for this purpose instead of a complicated checking procedure. If the electric circuit is operating normally and the meter indicated the same moisture value with that obtained with a primary/working standard meter, the moisture meter is regarded functioning properly.

In this procedure, it is important that both a moisture meter and a reference sample shall be at the same and stable temperature. When the measurement results of two or more meters are compared, all meters shall be also at the same temperature. If there is a difference of 1 °C between two moisture meters, it is estimated that there will be a difference of 0.1 % in measured values of moisture content. Therefore, leave both a moisture meter and a reference sample at the same place under the same environmental condition for two hours at least. Avoid leaving them in a place of sunlight or wind of an air conditioner.

Check the temperature of moisture meters and the room temperature from time to time. The temperature difference between them shall be within 3 degrees. Replace or repair the moisture meter(s) if some of them seem to have a wrong indication in moisture content or internal temperature.

### **13.2 Directions to use a checker kit (RfC) for a resistance-type**

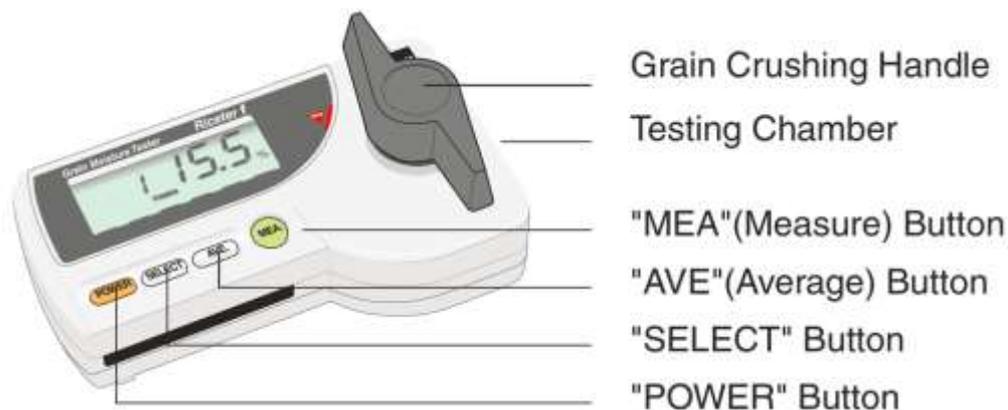
#### **13.2.1 Tools and samples**

The following tools and reference sample are needed (**Annex figures 1 and 2**).

- A resistance type moisture meter (model Rf)
- A checker kit (model RfC) for the moisture meter (model Rf)
- Reference samples
- A standard thermometer

Notes: Sample tray, handle and inside the main unit need to be cleaned. Checking points using the checker kit (RfC) correspond to 13.0 % and 18.0 % in moisture content. Reference samples must be

homogenized and have uniform moisture content. The temperature of moisture meters and reference samples should be at the same condition.



**Annex figure 1: Resistance type moisture meter (model Rf)**



**Annex figure 2: Reference sample with RfC (left) and standard thermometer (right)**

### **13.2.2 Check of display**

Press “POWER” button and confirm if all segments should be displayed. If not, change or repair the moisture meter and check it again. To turn off, press “POWER” button again after finishing the check.

### **13.2.3 Check of the internal temperature**

Keep pressing “AVE” and press “POWER”. Internal temperature of the meter should be displayed, and it shall agree with the room temperature within  $\pm 3$  °C. If one or two of meters seem to indicate wrong temperature, wait for a while or change the meter(s), and check it again.

### **13.2.4 Check of battery voltage**

Press “SELECT”, then current battery voltage should be displayed. If it is under 5.0 V, replace all batteries. Press “POWER” after checking.

### **13.2.5 Cleaning**

Clean up crushing handle, sample tray and measuring chamber (**Annex figure 3**).



Annex figure 3: Cleaning of the moisture meter

### 13.2.6 Setting up of the meter and checker

Insert the checker (RfC) into the moisture meter completely (Annex figure 4). For the old-type checker kit (RfC) shown in the Annex figure 4 on the left, the checking point is 13.1 % or 17.7 % and it is selected with the red lever (black lever is not used). Although this manual follows the old-type checker kit, the latest kit is shown on the right with the checking points of 13.0 % and 18.0 %.



Annex figure 4: Conventional type checker (left) and the latest type checker kit (right)

### 13.2.7 Check of the 13.1 % point using the checker kit (RfC)

Set the red lever at upper position (Annex figure 5). Turn the crushing handle completely until it stops. Keep pressing “SELECT” and Press “POWER”. A value of 13.1 (13.0 for the latest type)  $\pm$  0.1 % should be displayed. Otherwise, change or repair the meter and check it again.



Annex figure 5: Check for the 13.1 % point using RfC

### 13.2.8 Check of the 17.7 % point by the checker (RfC)

Set the red lever at lower position (Annex figure 6). Press “AVE”. A value 17.7 (18.0 for the latest type)  $\pm$  0.1 % should be displayed. Otherwise, change or replace the meter and check it again.



**Annex figure 6: Check for the 17.7 % point by RfC**

Note 1: If a value “4.7” is displayed, check the lever position of RfC. The position may be at neutral or RfC may have something in trouble.

Note 2: Calibration check procedure should be completed within 25 sec. If no operation is performed for approximately 25 seconds after power ON, the power is automatically turned OFF. If the power is OFF, conduct the procedure in 13.2.6 again.

### **13.2.9 Actual moisture measurement with the moisture meter (Rf)**

### **13.2.10 Cleaning**

Clean up the testing chamber, crushing handle and sample tray (**Annex figure 3**).

### **13.2.11 Check of the sample tray**

If the sample tray has damages as follows, it should be replaced with a new one:

- i) crack or removal of electro-plating is found (it may cause inaccurate measurement),
- ii) dimples on the metal surface are wore off,
- iii) a crack on the sample tray is found, and
- iv) change in color of the metal part.

### **13.2.12 Preparation of reference samples**

Mix well the reference samples which are used for measurement (**Annex figure 7**).



**Annex figure 7: An example of mixing procedure; with a plastic bag (left) and with a specialized machine using homogenizing plastic bins (right)**

### **13.2.13 Setting of the sample**

Take one layer of reference sample on the sample tray and remove any foreign grains or materials (**Annex figure 8**).



**Annex figure 8: Sample set in the tray in one layer. Good (left), too little (middle) and too much (right)**

### **13.2.14 Mounting of the sample tray**

Insert the sample tray into the testing chamber completely and fully rotate crushing handle until the stop position. After each measurement, clean the end of crushing handle, testing chamber and sample tray to avoid trouble. Use a new reference sample every time of measurement. Do not measure the same sample that was measured previously.

### **13.2.15 Measurement**

Make measurement five times for each reference sample using the primary standard (moisture meter) and record them to obtain an average moisture content of reference samples. Compare the measurement results between the primary standard and working standard (moisture meter) with the reference sample. Then, evaluate in-service moisture meters using the working standard with the same reference sample.

### 13.3 Directions to use a checker kit (PM-45C) for a capacitance-type

#### 13.3.1 Tools and samples

The following tools and reference sample are needed (**Annex figure 9**).

- A capacitance-type moisture meter (PM-450)
- A checker kit (model PM-45C) for the moisture meter (model PM-450)
- Reference samples
- A standard thermometer

Notes: The sample cup and the main unit need to be cleaned. Calibration check points are 4.0 pF, 9.6 pF, 16.7 pF and 28.0 pF (PM-45C). Actual reference sample must be homogenized and have a uniform moisture content. The temperatures of moisture meters and a reference sample should be the same.



**Annex figure 9: Model PM-450 (left), checker kit PM-45C (middle) and standard thermometer (right)**

#### 13.3.2 Check of display

Clean the testing chamber, otherwise it may cause a measurement error. Press “POWER”. Confirm if all segments should be displayed. If not, change or repair the meter and check it again. After checking, press “POWER” to turn the power off.

#### 13.3.3 Check of temperature

Keep pressing buttons “MEA” and “AVE”, and press “POWER” (**Annex figure 10**). Select “M3 TEMP” with pressing “SELECT (increasing)” or “AVE (decreasing)”. Press “MEA”, then sample temperature is displayed (M3 STMP). Press “SELECT”, then unit temperature is displayed (M3 UTMP). Temperatures of the sample and unit shall agree within  $\pm 3$  °C. If some of the meters seem to have wrong temperature indications, wait more time or replace the meter(s).

#### 13.3.4 Check of battery voltage

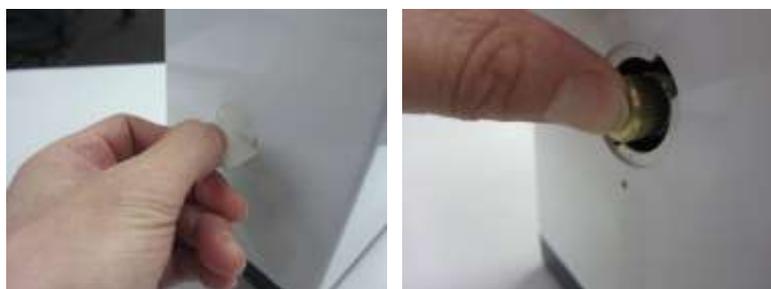
Press “SELECT” again, then battery voltage is displayed (M3 BATT) (**Annex figure 10**). The voltage should be 5.5 V or more. Otherwise, replace all batteries.



Annex figure 10: Display during checking temperature and battery voltage

### 13.3.5 Check of capacitance

Check capacitances at the four points using standard checkers (standard capacitors of 28.0, 16.7, 9.6 and 4.0 pF) with the following procedures. Remove a rear lid of meter by lid remover (**Annex figure 11**). Insert a standard checker. Keep pressing buttons “MEA” and “AVE”, and press “POWER”. Select “M1 CCHK” with pressing “SELECT” for increasing and “AVE” for decreasing. Press “MEA” and check if “0.0 pF ( $\pm 0.02$ )” is displayed. Otherwise, re-calibrate or change the meter. Check points of capacitance are given in **Table 5**.



Annex figure 11: Use of a standard checker (standard capacitor)

Table 5: Check points of capacitance using the standard checkers, and requirements to their accuracy

Capacitance at the check points	Accuracy of the standard checkers
28.0 pF	$28.00 \pm 0.20$ pF
16.7 pF	$16.70 \pm 0.15$ pF
9.6 pF	$9.60 \pm 0.15$ pF
4.0 pF	$4.00 \pm 0.15$ pF

### 13.3.6 Check of the built-in scale

Check of the built-in scale (weighing instrument using a load cell) should be performed using a standard weight (200 g) with the following procedures (**Annex figure 12**). Put the moisture meter on a horizontal surface. Put a sample cup on the electrode upside down. Keep pressing “MEA” and “AVE”, and press “POWER”. Select “M2 WCHK” with pressing “SELECT” for increasing or “AVE” for decreasing. Press “MEA” and check if a weight is displayed as “0.00 g ( $\pm 0.5$  g)”. Put the standard weight of 200 g on the electrode. Check if the measured weight is displayed within a range “200.0  $\pm$  1.0 g”. Otherwise, check the standard weight again, calibrate the meter, or replace the scale assembly (load cell).



**Annex figure 12: Check of the built-in scale**

## **13.4 Actual measurement using a moisture meter: PM-450**

### **13.4.1 Preparation**

Check the size of sample cup (240 ml) because a proper sample cup should be used. If the sample cup is incorrect in size or others, a measurement result would be inaccurate. Mix the reference samples well to be used for the measurement.

### **13.4.2 Pouring of the sample**

Place the moisture meter on a horizontal surface using a built-in level scale. Press “POWER” and select the product by pressing “Select”. Pour the reference sample into the sample cup correctly (left in **Annex figure 13**). Do not take a sample directly from the bag with the sample cup (right in **Annex figure 13**). Remove other kernels and foreign materials (if any). Remove the funnel of the cup to eliminate excess sample and level the top of sample. Pour the sample into the testing chamber during the “sample” icon on the display blinks. See **Annex figures 14-16** for these procedures.

Note: Pour the reference sample into the center of the testing chamber at a constant speed for entire sample to be loaded within 5 to 6 seconds. Pour the sample evenly and flatly.



Good (left) and no good (right).

**Annex figure 13: Taking out sample from the bag**



Good (left), too little (middle) and too much (right)

**Annex figure 14: Fill levels of the sample in the cup**



**Annex figure 15: Removal of excess sample**



Poring procedure (left), good (middle) and no good (right)

**Annex figure 16: Pouring sample into a testing chamber**

### 13.4.3 Measurement

Read moisture content (%) directly. If it indicates “Lo”, moisture content is below the measuring range. If it indicates “Hi”, moisture content is above the measuring range. For other error messages, refer to the operation manual. Press “AVE” for average measurement results.