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Traceability in Grain Moisture Measurement

and

Related Issues in Metrology

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at

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1. Basic understanding of grain moisture measurement

What is WATER (1)?

•**The earth** is a unique planet in the solar system which has huge amount of **water (H_2O)** on the surface. It enables many creatures **(animals and plants)** exist on the planet.

•Therefore, it is needless to say that **water** is the **most important substance** for all kinds of creatures. For example, 80 % of the body of a **baby** and 60 % of that of an **adult** consists of **water**.

•**Water** has extraordinarily high **meting (0°C)** & **boiling (100°C)** temperatures compared to those of other hydrogen compounds (CH_4, SiH_4, H_2S...). It means that **water** has a **strong intermolecular force** due to its unique molecular structure.

1. Basic understanding of grain moisture measurement

What is WATER (2)?

•Due to the same reason, water requires **huge amount of energy** (specific heat & latent heat) when it is heated, melted and evaporated. It greatly helps to keep our climate much more **moderate** compared to those in other planets.

•The volume of **water** of a unit weight **expands** when it **freezes.** It is unusual because most of other materials shrink. It enables an iceberg to float on the sea (and an ice in a glass too). It arises from an unique structure of ice that has an **inner 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.

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1. Basic understanding of grain moisture measurement

Microscopic behaviors of water in a small space:

•Water shows **unique and different 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** less than -20 °C.

•Main target of the present course is water absorbed or contained in microscopic structures of **grains** as a biological material. **Water** contained in such materials shows unique behaviors in similar to the case between glass plates.

•In the study of moisture contained in biological materials including grains, it is considered that **water** is contained in one of the three forms either (1) water of crystallization, (2) bound water and (3) free water.

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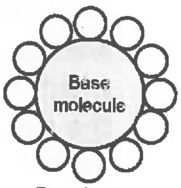
Characteristics of water in grains

Water contained in grains is in the three forms:

(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 mole An example is chalcanthite ($CuSO_4 \cdot 5H_2O$).

Chalcanthite

(2)**Bound water:** water molecules bound tightly to the surface of the base molecule (protein, etc.). They play an important biological role to protect the base molecule.



Bound water

(3)**Free water:** bulk water in the a rather **macroscopic** structure. This form of water may cause a **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**.

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Structure of cereal grains

- The cereal grains are mainly consisted of (1) carbon hydrates, (2) proteins, (3) fats and (4) **water**.
- These compounds have a **hydrophilic** (water friendly) part in the molecules, therefore **microscopic structure** changes with absorption of water as shown by the two figures.

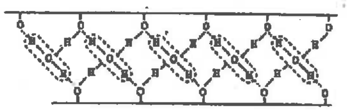
Figure (a): Dry grain

- Molecules are connected tightly with **strong bonds**.

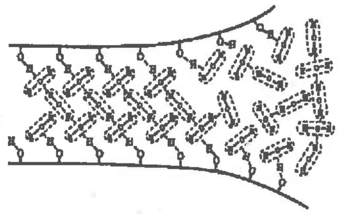
Figure (b): Wet grain

- The bonds become **weak** and the structure **inflates** as a result of absorption of **water**.

(a)



(b)



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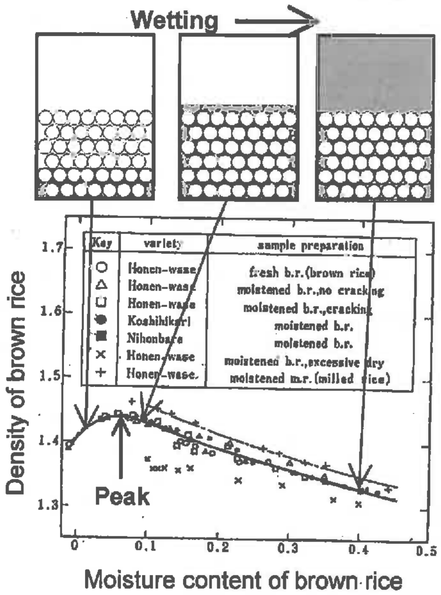
A phenomenon due to the free water (1)

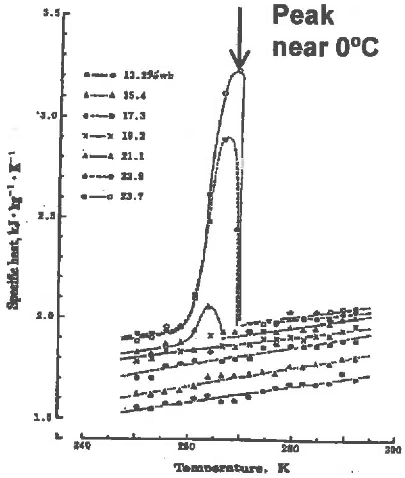
- ✓ The **density** of brown rice have a **peak** at lower MC near 7 %.
- ✓ This phenomenon is considered as a result of **wetting procedure** in which **free water filled the space** between microscopic structure composed of grain molecules which has a density higher than that of water.



A phenomenon due to the free water (2)

- ✓ **Free water** (excess moisture contained in grains) **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** near 0 °C.

Peak near 0°C



Specific heat capacity of brown rice near 0 °C

Equilibrium and hysteresis of moisture content (MC)

- The MC of grains in a condition of constant temperature and humidity, becomes closer asymptotically to an stable state called as **equilibrium** (Fig. A).
- However, the equilibrium MC is different between wetting and drying processes (Fig. A) due to **hysteresis**.
- A typical hysteresis curve of grains with an abscissa of humidity is given by Fig. B.

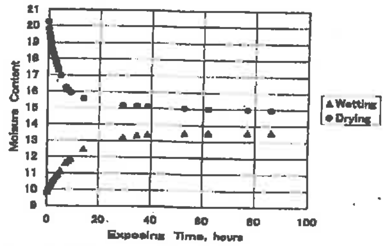


Fig. A. MC of brown rice under wetting and drying processes (20°C, 70%RH).

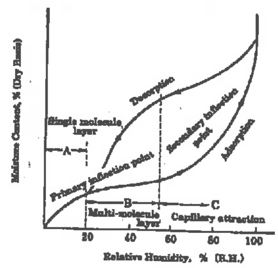


Fig. B. A hysteresis curve of MC of grain.

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1. Basic understanding of grain moisture measurement

Quality of agricultural products and moisture content

- Quality (moisture content, protein content, sugar content, etc.) of **agricultural products** (rice, wheat, maize, beans, etc.) is an important characteristic for **safe and healthy life**.
- The quality is also connected to **fair trade** because prices (or acceptance criteria) of the product are usually determined based on its quality.
- Because of the importance, **a category of instruments** for such qualities is **controlled** under legal metrology authorities in particular in the economies producing/exporting cereal grains.
- Among the qualities, **Moisture content** (MC) is closely related to the **stability of the product** in long-term storage. Because **moist grains deteriorates** quickly, they must be **dried artificially** (with energy & cost) before transactions. This process usually makes the **unit price** of a dry grain more expensive. Therefore, moisture content is considered as an important property in many Asian countries.

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1. Basic understanding of grain moisture measurement

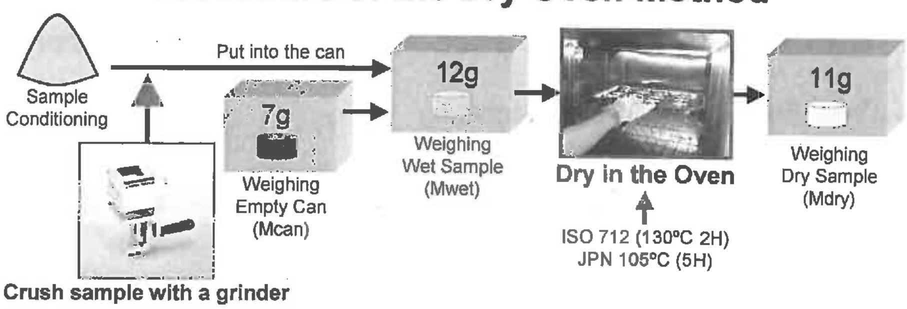
Typical measurement methods for grain moisture

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

Electric resistance method**
Electric capacitance method**
Near infrared absorption method**
Microwave absorption method
Infrared-heated moisture balance (simplified dry oven method)

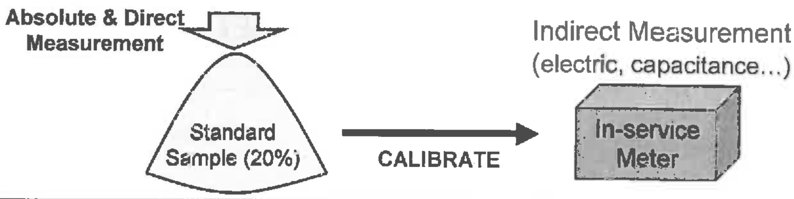
* An **absolute & direct** measurement method used to provide reference data/samples in many countries.
** Practical methods which are widely used in the real field.

Procedure of the Dry Oven Method



Moisture Content = (Mwet - Mdry) / (Mwet - Mcan)

= (12g - 11g) / (12g - 7g) = 0.2



Important note to dry oven method (1)
Lack of harmonization in drying conditions that affects measurement results

Organization /country \ Products	ISO 712 (widely used in EU & Asia)	Japan (original)	USDA US. Department of Agriculture	AOAC Assoc. of Official Anal. Chemists	ASAE American Soc. of Agricultural Engineers
Cereal grain	130°C 2h	105°C 5h	130°C 1h	135°C 2h	103°C 20h (barley) 19h (wheat)
Beans	130°C 2h	105°C 5h	130°C 1h	135°C 2h	103°C 72h
Peas and lentils	130°C 2h	105°C 5h	130°C 1hr	135°C 2h	
Maize (Food) Maize (Feed)	130-133°C 4h	105°C 5h 135°C	103°C 72h		103°C 72h
Grain Sorghum (Food/Feed)	130°C 2h	105°C 5h 135°C 2h	130°C 1hr	135°C 2h	103°C 18h
Soybeans	130°C 2h	105°C 5h	130°C 1hr	135°C 2h	103°C 72h

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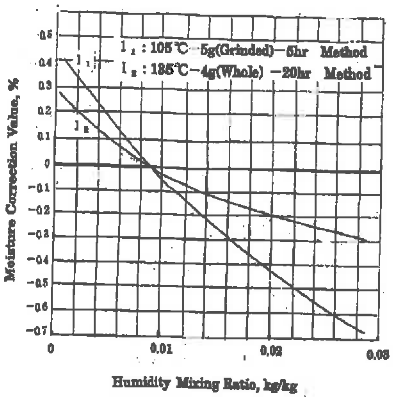
Important note to dry oven method (2) Need for correction based on ambient condition

- The **drying process** is affected by **ambient humidity** and **temperature** where a dry oven is operated.
- The right figure shows the **effect of ambient humidity** to the measured moisture content in regard to the reference condition.
- The typical reference conditions are **20°C & 60%** RH* (Japan) and **20°C & 40-70%** RH* (ISO 712).

*RH: Relative Humidity



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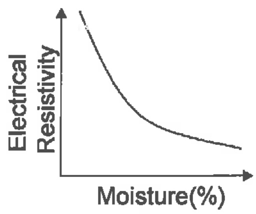
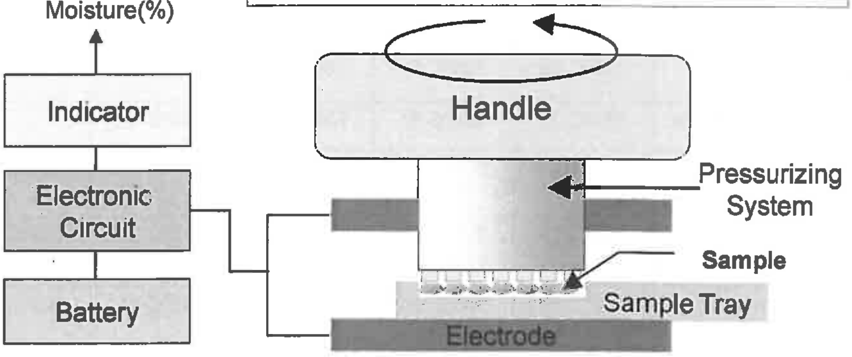
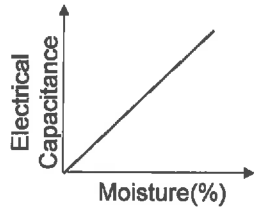
Practical Method (1) Electric Resistance Grain Moisture Meter

- Crush sample by sandwiching with the electrodes.
- Measure volumetric electrical resistivity of sample (has a negative slope to moisture) and covert it into moisture content.
- Small sample size & handheld.
- Widely used (about 70% market) in Asian countries.

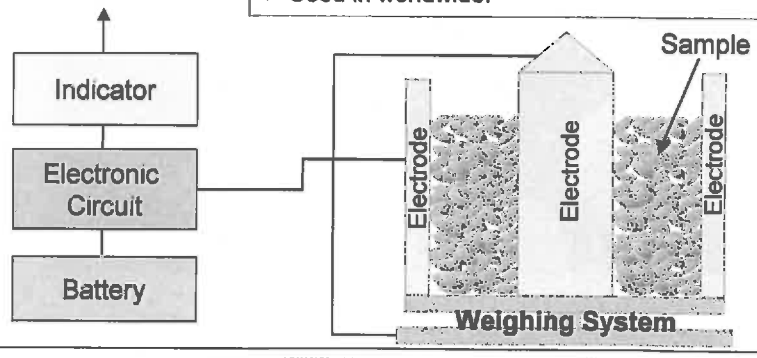


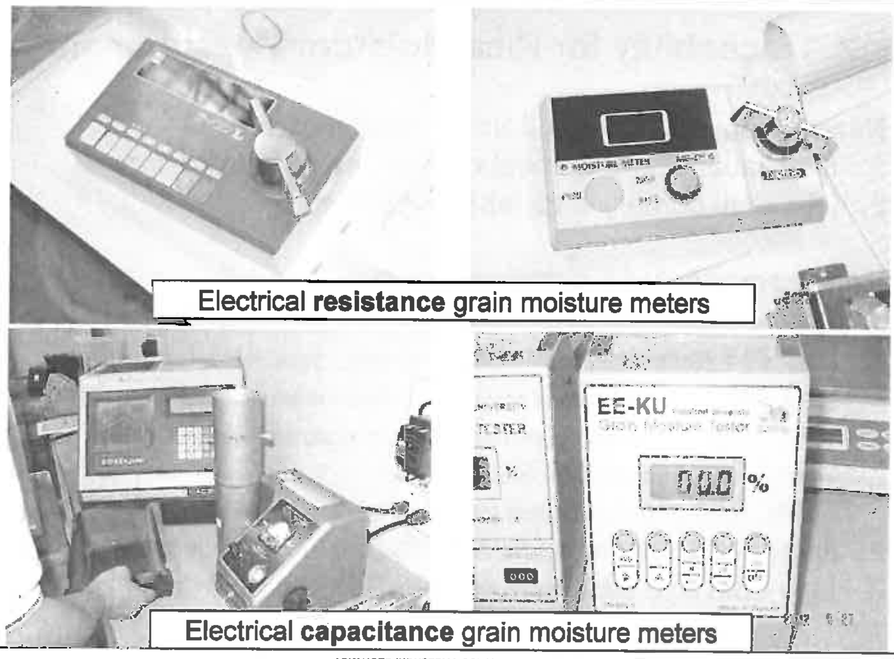


Practical Method (2)
Electric Capacitance Grain Moisture Meter



- Pour sample into a **cylindrical container** with a pair of electrodes.
- Measure **electrical capacitance** (almost proportional to moisture) and **mass** of the sample. Then, covert into moisture content.
- Larger sample size but **no need for crushing**.
- Used in worldwide.





Electrical **resistance** grain moisture meters

Electrical **capacitance** grain moisture meters

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Rice inspection at Yamagata Food Office (Japan)

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2. Traceability for Rice Moisture Measurement

Needs from a practical field in legal metrology

1. International and domestic **confidence** in **fair trade**
2. Regional primary **traceability system**

Requirements for a practical system

1. Harmonization with existing international standards:
   ISO 711 (Determination of moisture content - basic reference method),
   ISO 712 (Determination of moisture content - reference method),
   ISO 7700 (Checking the performance of moisture meters in use - Part 2: moisture meters for oilseeds) and
   OIML R59 (Moisture Meters for Cereal Grain and Oilseeds)
2. Need for a system applicable to **in-service meters.**
3. Low **cost** and **easiness** in adoption and operation.

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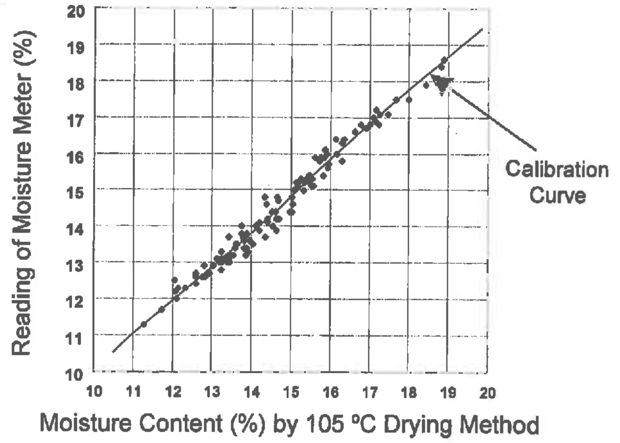
An Example of Practical Calibration

*A meter usually calibrated by **defining a curve** based on the reference data obtained with the dry oven method.*



A calibration curve of a resistance moisture meter
(105 °C drying method, 2001FY brown rice, 113 samples / 30 varieties)

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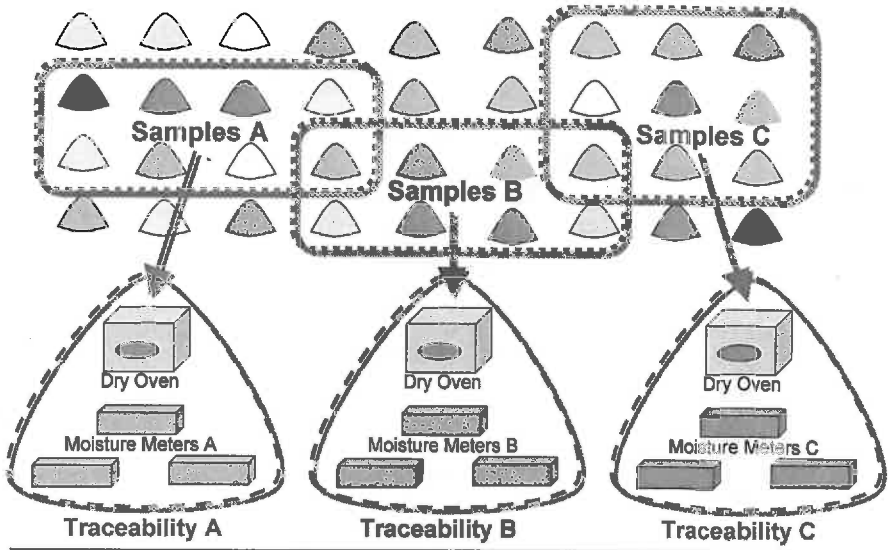
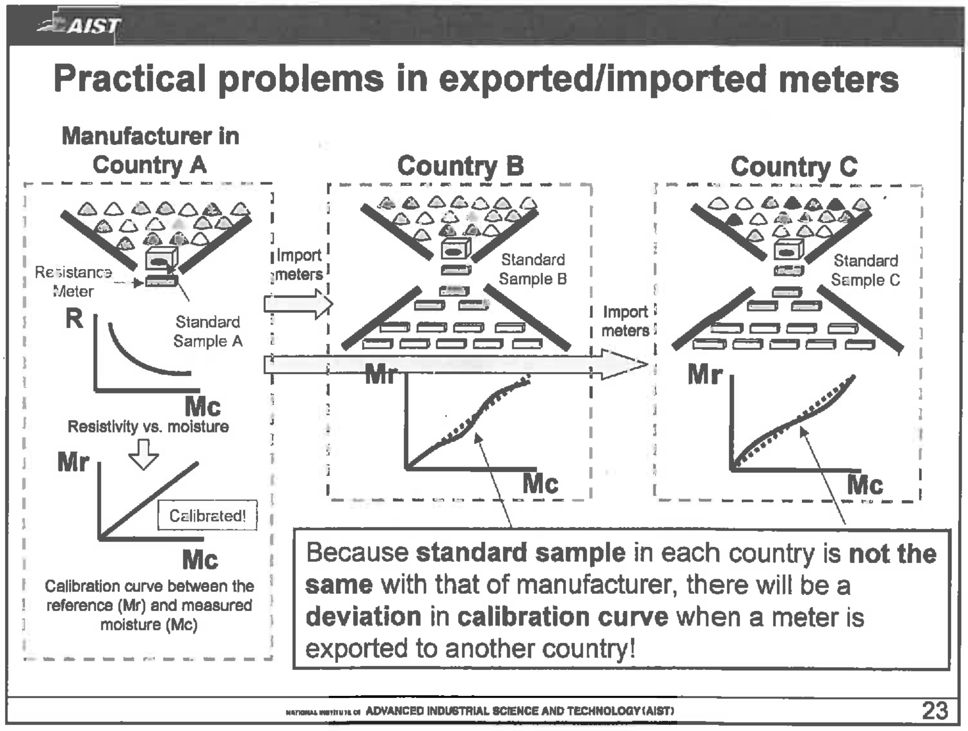
Measurement system without traceability



Lack of Representativeness of Sample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Practical problems in exported/imported meters
Manufacturer in Country A
Country B
Country C
Resistance Meter
R
Standard Sample A
Mc
Resistivity vs. moisture
Mr
Calibrated!
Mc
Calibration curve between the reference (Mr) and measured moisture (Mc)
Import meters
Standard Sample B
Import meters
Standard Sample C
Mr
Mc
Mr
Mc
Because standard sample in each country is not the same with that of manufacturer, there will be a deviation in calibration curve when a meter is exported to another country!
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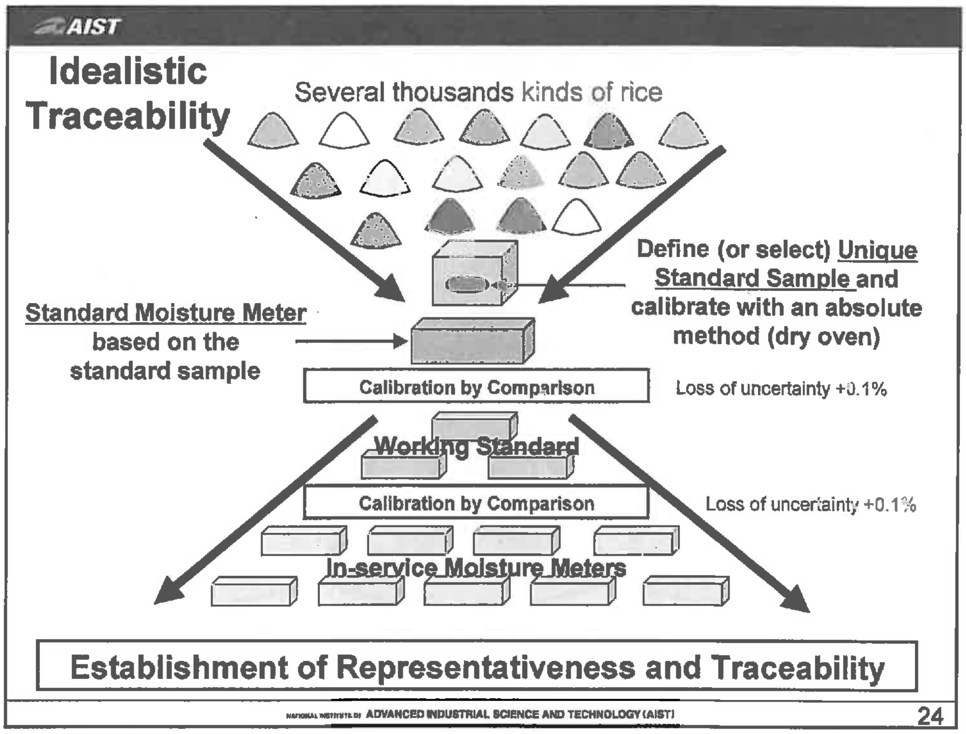
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Idealistic Traceability
Several thousands kinds of rice
Define (or select) Unique Standard Sample and calibrate with an absolute method (dry oven)
Standard Moisture Meter based on the standard sample
Calibration by Comparison
Loss of uncertainty +0.1%
Working Standard
Calibration by Comparison
Loss of uncertainty +0.1%
In-service Moisture Meters
Establishment of Representativeness and Traceability
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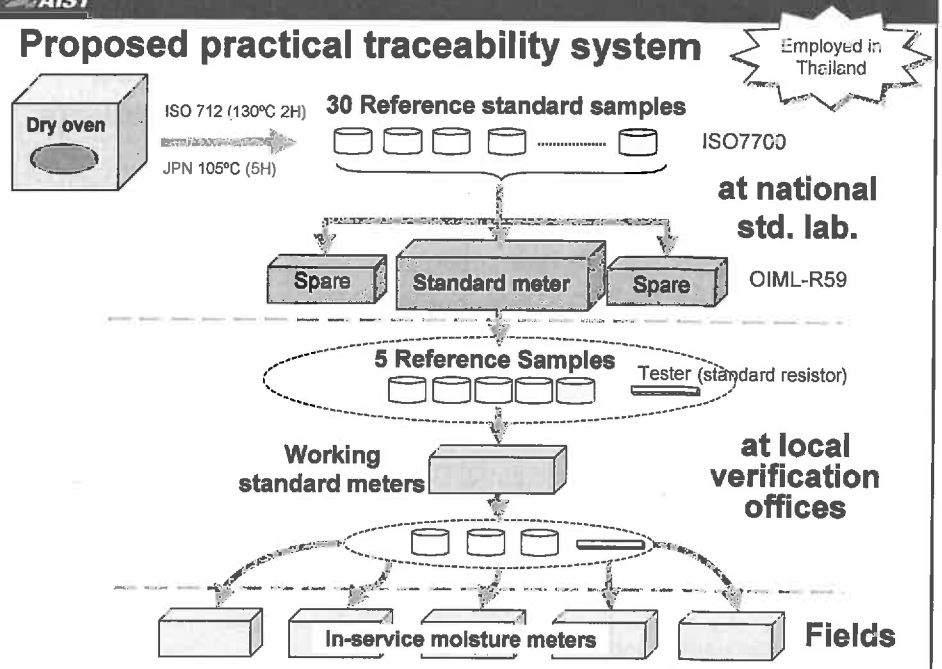
Proposed practical traceability system



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2. Traceability for Rice Moisture Measurement Summary

- ✓ A **global and unique traceability system** is important but it is difficult to realize it in the present situation.
- ✓ The difficulty is primarily due to a **lack of reproducibility** which arises both from measurement method and sample (measurand).
- ✓ We should seek a **realistic solution** by specifying a **reference method** (oven method with a specified condition) and **standard sample** to be used in each economy/region.
- ✓ There is a strong need for a **practical solution** by legal metrological authorities in respond to a **fraud** in transactions.

(Personal comment) Employment of one kind of meter with unique calibration curve for all varieties might be an ultimate solution. With this scheme, a unique traceability system in an economy could be realized virtually. However, a measurement result becomes an unrealistic index which may deviate from the true physical value. It might be acceptable in one economy, but it makes troubles in the international trade.

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3. Activities of APLMF Working Group on Quality Measurements of Agricultural Products (Brief history)

1. This WG was set up in **2001** as **'WG on Rice Moisture Meters'** with a chairperson, Mr. Issei Akamatsu (NMIJ).
2. In 2001-2005, the WG **conducted five training courses** on rice moisture meters with support by APLMF, APEC, Japanese Gov. and Kett Lab. The courses contributed to **capacity building** in verification and proposed a new **traceability system**.
3. In 2005, the WG was **renamed** as 'WG on Measurement of Moisture Content of Agricultural Commodities' and the chairperson was taken over by Mr. Hiroshi Kitano (NMIJ).
4. In 2007, the WG was **renamed again** as present and the chairperson was taken over by Matsumoto. **Three workshops** with support by APLMF/APEC, and **one training course** in a self-fund scheme were conducted.

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List of APLMF Training Courses/Workshops on Rice Moisture Measurement and Quality of Agricultural Products

Course Title	Date (d/m/y)	Place (Host)	Trainers / Speakers	Trainees
Study Tour for Rice Moisture Measurement*	30/09-5/10/2001	Several places in Japan (NMIJ)	I. Akamatsu (NMIJ) and others (JPN)	9 from 7 econ.
Training on Calibration of Rice Moisture Meters*	19-30/08/2002	Khon Kaen, Thailand (CBWM)	I. Akamatsu, H. Tanaka (NMIJ), T. Watanabe, N.Yoshida (Kett Co.)	23 from 7 econ.
Training on Calibration of Rice Moisture Meters*	30/08-10/09/2004	Bien-hoa, Vietnam (STAMEQ)	I. Akamatsu (NMIJ), T. Watanabe, N. Yoshida, T. Suzuki (Kett Co.)	About 20
Training on Calibration of Rice Moisture Meters*	11-29/11/2004	Chiang Mai, Thailand (CBWM)	I. Akamatsu, H. Tanaka (NMIJ), T. Watanabe, N. Yoshida, M. Yabe (Kett)	About 23 from ASEAN
Training on Calibration of Rice Moisture Meters*	15-26/08/2005	Manila, Philippines (ITDI)	I. Akamatsu, H. Tanaka (NMIJ), T. Watanabe, N. Yoshida (Kett)	From ASEAN
Workshop on Metrology of Agricultural Products and Foods	7-9/02/2007	Chiang Mai, Thailand (CBWM)	24 from 11 econ	About 80 incl speakers
Workshop on Metrology in Food Safety, Agricultural Products and Products Safety	4-6/06/2008	Hangzhou, PR China (AQSIQ)	24 from 14 econ	About 70 including speakers
Workshops on Metrology in Food Safety and Agricultural Products	23-25/09/2009	Ho Chi Minh City, Vietnam (STAMEQ)	18 from 10 econ	20 from 11 econ
Training Course on Traceability in Rice Moisture Measurement	28/05-01/06/2012	Bandung, Indonesia (DoM)	T. Matsumoto and Kett Elec. Co.	36 from 3 economies
Training Course on Traceability in Rice Moisture Measurement	25-29/11/2013	Chiang Mai, Thailand (CBWM)	T. Matsumoto and Kett Elec. Co.	33 from 8 economies

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Workshops on Metrology in Food Safety and Agricultural Products
(Ho Chi Minh, Vietnam in 2009)

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Outcomes of the APLMF Workshops on Metrology on Agricultural Products

Comments and requests in the three workshops in 2007-2009:

1. Need for metrological infrastructure and primary methods.
2. Need for a unique traceability system with uncertainty.
3. Need for primary methods in biological measurement / food safety.
4. Need for regionally/internationally certified CRMs with database.
5. Need for international/regional proficiency tests and inter-comparisons.
6. Issues on accreditation schemes and legal aspects.
7. Issues on prepackages and labeling of products/foods.
8. Cooperation between metrology and other fields (biology, food safety ...).
9. Cooperation between APLMF (legal) and APMP (scientific) in metrology.
10. Need for other products (coffee, tea, starch, milk, water, etc.).
11. Need for training programs and knowledge bases.

Discussions in APLMF after the workshops:

1. Too many needs but they were far **out of the scope** of APLMF.
2. There is a continuous need for **rice moisture measurement**.
3. APLMF should concentrate on the **original background** by considering the above outcomes

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Training Course on Traceability in Rice Moisture Measurement
(Bandung, Indonesia in 2012)

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Outcomes of Training Course on Traceability in Rice Moisture Measurement in 2012

1. A **tour** to a local testing center => OK
2. A lecture to **control** sample moisture => OK
3. A practice to **calibrate/adjust** meters => OK
4. Utilize time **effectively** => OK (we will try)
5. An **uncertainty** analysis in practical use => Partly OK
6. A need for a method to prevent **a fraud** => OK
7. A standard sample dedicated for **each measurement method** => Partly OK
8. A need for **other kind** of grains => OK for corns
9. A practical drying method with **sunshine** => Not yet
10. A fully **artificial** standard sample => Difficult and to be explained.
11. A **sound traceability** system in a region/world. =>Not yet (important)
12. A **harmonization** among international regulations => Not yet (important)
13. A need for an **inter-comparison** =>Not yet (a long-term objective)
14. A **guide document** on grain moisture => Not yet but still working
15. Proposal from the APLMF to **OIML TC17/SC1** => In progress (important)
16. **Continue** the training program on grain moisture measurement => OK
17. Training activities in **each economy** => Partly OK (we guess so)
18. A need for **facility** => Depends on each economy (important)

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4. Uncertainty Analysis of Rice Moisture Measurement

4.1 Basic understanding of uncertainty

4.2 Current situation of BIPM and OIML regarding uncertainty

4.3 Practical considerations of uncertainty for rice moisture measurement

4.4 Experimental studies on uncertainty in grain moisture measurement

4.5 Summary and suggestions regarding uncertainty

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4.1 Basic understanding of uncertainty (1) Two important but different concepts

✓ **Measurement Error (*traditional keyword*):**

A concept equivalent to the value: [<u>**measurement quantity value - true value**</u>] which has been used widely in metrology based on an assumption that the <u>**true value**</u> (or reference value) is known. A measurement error is usually composed of (1) <u>systematic error</u> and (2) <u>random error</u>.

✓ **Uncertainty / Measurement Uncertainty (*new keyword*):**

An expression of a <u>**doubt**</u> (or validity) of a measurement result which is usually expressed with a <u>**positive quantity**</u> associated with uncertainty. However, it is considered as impossible to know the <u>**true value**</u>.

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4.1 Basic understanding of uncertainty (2)
Definitions of 'uncertainty'

- ✓ ***VIM2** (1993)*: 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)*: Non-negative parameter characterizing the dispersion of the quantity values being attributed to a measurand, based on the information used.*
- ✓ ***GUM** (2008)**: parameter, associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand*

* **VIM: International Vocabulary** in **Metrology** 2nd edition (1993) and 3rd edition (2008).

** **GUM:** Evaluation of measurement data - **Guide** to the expression of **Uncertainty** in **Measurement** (JCGM 100:2008)

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4.1 Basic understanding of uncertainty (3)
Other Important Keywords in GUM (JCGM 100:2008)

- ✓ (Measurable) **quantity**: attribute of a phenomenon, body or substance that may be distinguished qualitatively and determined quantitatively.
- ✓ **Value** (of a quantity): magnitude of a particular quantity generally expressed as a unit of measurement multiplied by a number. *For example, a length of the copper bar at 273 K was 101.23 mm.*
- ✓ **Measurand**: particular quantity subject to measurement. *This is a virtual concept without a value. For example, the length of a copper bar at 273 K and 0.1 MPa.*
- ✓ **Measurement**: set of operations having the object of determining a value of a quantity.
- ✓ **Result of measurement**: value attributed to a measurand, obtained by measurement.
- ✓ **Repeatability** (of results of measurements): closeness of the agreement between the results of **successive measurements** of the same measurand carried out under the **same conditions** of measurement.
- ✓ **Reproducibility** (of results of measurements): closeness of the agreement between the results of measurements of the same measurand carried out under **changed conditions** of measurement

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4.1 Basic understanding of uncertainty (4)
Important concepts of uncertainty

- ✓ A **parent population** is assumed. It is composed of an infinite number of quantity values representing the **measurand**. A **measurement result** is considered as **one** sample taken from the parent population.
- ✓ **Statistical approach** is employed assuming that the quantity values follow a **normal distribution**.
- ✓ It is considered as **impossible** to know the **true value** of the measurand (unless it is defined independently). Instead, (1) the **best estimated value** of the measurand and (2) a **value of uncertainty** (associated with the dispersion of quantity values) are evaluated.
- ✓ **A result** of measurement becomes **complete** only when it is accompanied by a **statement of uncertainty**.
- ✓ **'Dispersion'** means unknown errors that remains even **after every effort has been made** to correct for all recognizable sources of uncertainty.

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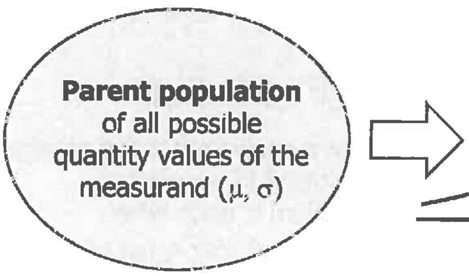
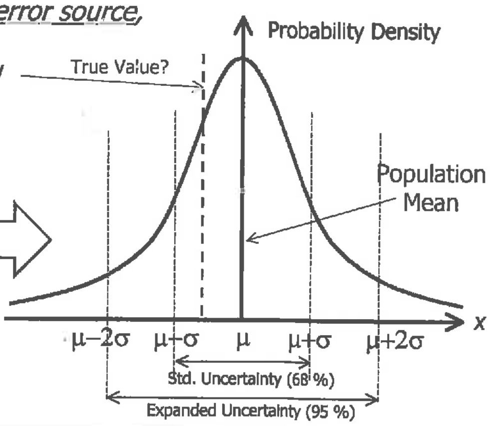
4.1 Basic understanding of uncertainty (5)

Quantity values are assumed to follow a **normal distribution** based on the *Central Limit Theorem*. **Population mean** (μ) is considered as the **best estimate** of the measurand, and the values have a dispersion with a **standard deviation** (σ).

However, if there is an unknown error source, the true value may exist far from the mean. => But nobody knows!





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4.1 Basic understanding of uncertainty (6)

In an uncertainty analysis, a standard deviation (σ) of the quantity values is estimated using either of the two methods:

✓***Type A**: Evaluation by applying a **numerical statistical method** on the measured quantity values.*

✓***Type B**: Evaluation by an **analytical method** based on other information.*

The estimated standard deviation (σ) is also called as "**standard uncertainty**" (≅ 68% confidence) and another uncertainty with a wider range ($k\,\sigma$) is called as **"expanded uncertainty"**. The ratio k is called as coverage factor, and $k = 2$ (≅ 95% confidence) is commonly used.

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4.1 Basic understanding of uncertainty (7)

When y is determined with a function $y = f(x_1, x_2, x_3, , x_n)$ by several independent parameters (x_i), the **combined uncertainty** (U_c) is given by the equation:

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

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

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

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 are independent (no correlation) in each other.

This method is frequently used to evaluate u_c. It means that small error sources are negligible in the uncertainty analysis.

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4.2 Current situation of BIPM and OIML regarding uncertainty (1)

BIPM and JCGM (Joint Committee for Guides in Metrology) have been drafting and publishing the two important documents regarding uncertainty.

✓*VIM: International Vocabulary of Metrology – Basic and General Concepts and Associated Terms, JCGM 200:2012 (JCGM 200:2008 with minor corrections)*

✓*GUM: Evaluation of measurement data – Guide to the expression of uncertainty in measurement, JCGM 100:2008 (GUM 1995 with minor corrections)*

** **JCGM** has also published **supporting documents** for VIM and GUM.*

** **OIML** has published these documents as **V2-200** (VIM) and **G1-100** (GUM).*

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4.2 Current situation of BIPM and OIML regarding uncertainty (2)

- ✓ **In OIML**, TC3/SC5 (Conformity Assessment) chaired by NIST in USA has been drafting a **new guide document** "*The role of measurement uncertainty in conformity assessment decisions in legal metrology*".
- ✓ The first committee draft (CD1) of this document was circulated in 2009. The next CD has not been prepared.
- ✓ This draft document introduced the **concept of uncertainty** into legal metrology by adding new key words "***guard band***" and "***shared risk***". The **former** is usually applied when measurement uncertainty is relatively **large** and the **latter** is applied when it is **small** enough.
- ✓ *(Personal comment) There is an inherent difficulty because of a disagreement between the concept of uncertainty and the traditional conformity assessment procedure in legal metrology. The former employs a probabilistic approach while the latter requires a clear determination (pass/fail) in type approvals and verifications.*

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4.3 Practical considerations of uncertainty for rice moisture measurement (1)

- The results of grain moisture measurement are strongly affected by the selection of **sample**, measurement **procedure** and **equipment** including measurement instruments.
- On the contrary, uncertainty due to **electrical measurement** (resistivity or capacitance) is very small.
- **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, (3) sampling method, (4) sample treatment and (5) a standard used to calibrate the measurement instrument.

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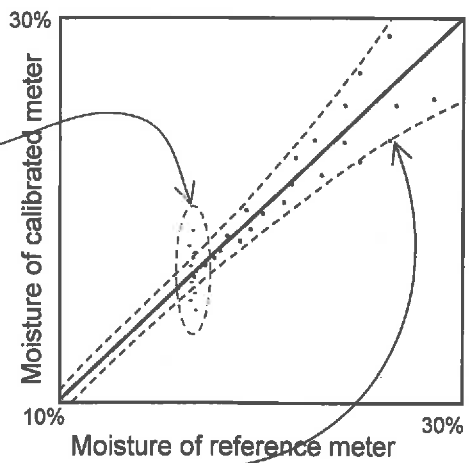
4.3 Practical considerations of uncertainty for rice moisture measurement (2)

✓ An experimental analysis on **a specific lot**, which has almost constant & uniform characteristics, is possible (already done).

✓ An analysis on a set of samples of the same variety with **different moisture levels** may be possible. *However, the uncertainty in high level could become large.*



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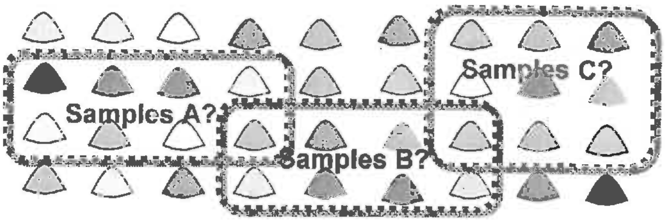
4.3 Practical considerations of uncertainty for rice moisture measurement (3)

- ✓ When **characteristics** of the sample is **not** (or poorly) specified, an evaluation of uncertainty becomes **difficult**. In this case, we **can not distinguish** errors due to sample, measurement procedure and instrument. In other words, it means that "***measurand***" is not defined clearly.
- ✓ Uncertainty can not be evaluated without specifying **sample characteristics**.



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4.4 Experimental studies on uncertainty in grain moisture measurement (1)

A Study by NMIJ and Kett Elec. Co. in 2004

- ✓ An **experimental study** to evaluate **uncertainty** in the **dry oven** method on rice samples was performed by Dr. Hideyuki Tanaka (NMIJ) and Kett Electric Co. in 2004.
- ✓ Test measurements were performed by placing **25 sample cans** in a dry oven at the same time. One cycle of measurement was repeated **twice.**
- ✓ **ANOVA** (Analysis of variance by Sir Ronald Aylmer Fisher) was employed in statistical analysis.

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Sources of Uncertainty Considered in the Evaluation

No.	Sources of uncertainty	Methods for evaluation
1	Non-uniform distribution of **temperature** in the dry oven	Evaluated from the deviation between the sample cans placed at different regions in the oven.
2	Repeatability and deviation between **different sample** cans	Calculated simultaneously from the above result (No. 1).
3	Reproducibility in the crushing process of sample using **different grinders**	Compared measurement results using different grinders with the same type.
4	Uncertainty in **mass measurement**	Evaluated uncertainties for weighing can, samples and calibration of the weighing instrument.

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Result of Evaluation of Uncertainties

Symbol	Source	Standard Uncertainty	Sensitivity Coefficient	Standard Uncertainty (%)
u_T	Uncertainty caused by the distribution of temperature in the dryer	0.01239 (%)	1	0.01239
u_R	Uncertainties caused by the repeatability and the deviation between samples	0.01825 (%)	1	0.01825
u_G	Uncertainty caused by the reproducibility of the grinders	0.06350 (%)	1	0.06350
u_{m0}	Uncertainty caused by the mass of a sample before drying + the mass of a weighing can	0.0001109 (g)	17.1715 (%/g)	0.001904
u_S	Uncertainty in the calibration of a weighing machine	0.000025 (g)		
u_{mr}	Uncertainty of the weighing can and samples	0.0001080 (g)		
u_{m1}	Uncertainty caused by the mass of a sample after drying + the mass of a weighing can	0.0001109 (g)	-19.9605 (%/g)	0.002214
u_S	Uncertainty in the calibration of a weighing machine	0.000025 (g)		
u_{mr}	Uncertainty of the weighing can and samples	0.0001080 (g)		
u_{mc}	uncertainty of the weighing can	0.0000629 (g)	2.78894 (%/g)	0.0001754
u_S	Uncertainty in the calibration of a weighing machine	0.000025 (g)		
u_{CAN}	Uncertainty of the weighing can	0.0000577 (g)		
		Combined Standard Uncertainty(%)		0.06729
		Expanded Uncertainty(%) (k=2)		0.13

✓Total evaluated uncertainty was **0.13% (k=2).**

✓**Grinder** was the major source (0.06%).

(except uncertainty due to selection of the standard sample)

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4.4 Experimental studies on uncertainty in grain moisture measurement (2)

A report in the Annex A of ISO 712 :2009

✓ Repeatability, reproducibility and critical difference of the methods were evaluated by an inter laboratory test participated by **19 laboratories**. The test were conducted on **7 kinds of products** of semolina, wheat (4 kinds), barley and rice.

✓ The test reported that the **repeatability was 0.04 %** and **reproducibility was 0.16 %** which were expressed in a standard deviation of MC averaged for all kinds of product.

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4.5 Summary and suggestions regarding uncertainty

✓ Too many and too unpredictable **uncertainty factors**.

✓ It is **impossible** to evaluate **unique parameter of uncertainty** applicable to **all grains**. What we can do is an assessment in a **specified condition** (variety, instrument type, ref. method…).

✓ Uncertainty in the **high moisture range** (over 20%) becomes **large** due to the difficulty in measurement on moist samples in a stable and constant condition.

✓ It is easy to provide an **artificial sample** with a constant property. However, it merely behaves as a **standard resistor/capacitor** to check electrical performance. Even if an artificial material with an ability to **absorb/exhale moisture** is provided, its characteristics will be different from those of a real grain.

This is the end of an uncertain story!
Please be reminded 'Uncertainty is Uncertain'.

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5. Conclusions

- ✓ There are increasing concerns in quality of agricultural products, in particular in **moisture content**.
- ✓ There is a need for a **practical solution** for a legal metrological authority in respond to a **fraud** in transactions.
- ✓ Establishment of an internationally accepted **reference method** and **standard sample** for moisture content is an important and long-term objectives. We should continue progress in a **practical level** within a limited region/economy.
- ✓ There is a further need for **uncertainty analysis** on grain moisture.
- ✓ Further **cooperation** is needed between the international/regional organizations: OIML, BIPM, ISO, CODEX, APLMF, APMP and so on.

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A part of this material was provided by the Kett Electric Laboratory and Dr. Hideyuki Tanaka of NMIJ.

A literature, "What is Water" by Dr. Hisashi Uedaira, Bluebacks, Kodansha Ltd. (2009) was referred to provide the contents about structure of water.

Thank you for your kind attention!