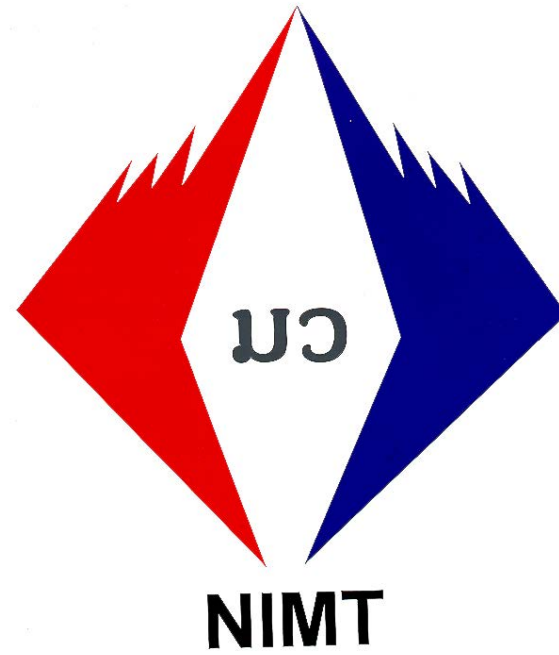


Innovative Approaches to Moisture Content Measurement in Rice/Paddy Grains: Thailand's Perspective



Abstract

The certified reference materials (CRMs) for moisture content (MC) determination in rice grains were meticulously developed by the National Institute of Metrology, Thailand (NIMT). These CRMs are critical in ensuring the precision and accuracy of moisture measurements in rice grains. They serve a dual purpose: verifying the performance of moisture meters/analyzers and confirming the credibility of the produced CRMs through comparative measurements with 11 domestic laboratories. The development was carried out in three phases:

Phase 1: The preparation of these CRMs involved incubating the rice grains in a controlled environment with specific temperature and humidity settings, allowing the grains to either absorb or desorb moisture to achieve the desired levels (5% to 25%). The candidate CRMs were stabilized in amber glass or polypropylene bottles for long-term stability. Their homogeneity was evaluated, and trend analysis was used to assess instability, in accordance with ISO Guide 35. The moisture content values were determined using the loss-on-drying method (ISO 712), with measurement uncertainties ranging from 0.15% to 0.30% over three months.

Phase 2: A study was conducted to verify the properties and consistency of the CRMs by comparing measurement results and evaluating the measurement capabilities of laboratories using the CRMs produced by NIMT. This study also aimed to assess the competency of various laboratories by comparing the results of moisture measurements in rice grains using the drying method specified in ISO 712. The rice samples, including paddy and milled rice with constant moisture content, were prepared using the same method as the CRM production process, ensuring consistency and reliability at moisture content levels ranging from 12% to 18%. The study involved 11 laboratories, and the standard deviation results from all participants were analyzed to calculate Z'-Scores, revealing that 90% of the laboratories met the "satisfactory" criteria. However, this program did not include analysis of measurement uncertainty results.

Phase 3: These CRMs have been successfully developed and can be used for quality assurance of grain moisture measurement, particularly in Thailand. The next step is to assess the quality system for the production of CRMs to enhance confidence among domestic laboratories, in line with NIMT's responsibilities.

Phase 1: Preparation of CRMs

Raw material was collected during the harvest season from a research paddy field in Pathumthani at least 5 kg for a level of moisture content. Then the raw material was clean with two sieves: one having rectangular holes with 1.7 mm x 10.5 mm and another with 2.3 mm x 10.5 mm in order to pick out foreign substances. Next the raw material was put in a large bin and mixed in a shaker for almost one week. After that, the raw sample was filled in large plastic bags and stored in the refrigerator at the temperature of 2 °C to 8 °C waiting for moisture adjustment.



Pic. 1 Raw material was collected.



Pic. 2 Adjust moisture sample by drying.

When the raw material has a higher moisture content (greater than 20%), the moisture level can be adjusted by drying the material under appropriate conditions in an oven. Once the desired moisture content is reached, the sample is placed in a test tray inside a chamber, and water is poured at the bottom of the tray. The sample will then absorb moisture from water vapor (pure water to be saturated) until it reaches the target weight, which corresponds to the desired moisture content. This process allows the sample to equilibrate to the target moisture level specified for the test.

Assigned value (X_{crm})

According to ISO 712: 2009 [2], a small portion (5 g) of a finely ground sample is heated and dried in an air oven at 130 °C for 2 hours. Based on an assumption that only water is evaporated during the drying process, the percent moisture content is calculated from the weight before and after drying as following on eq. (1)

$$MC = \frac{m_0 - m_1}{m_0} \times 100 \quad (1)$$

Where

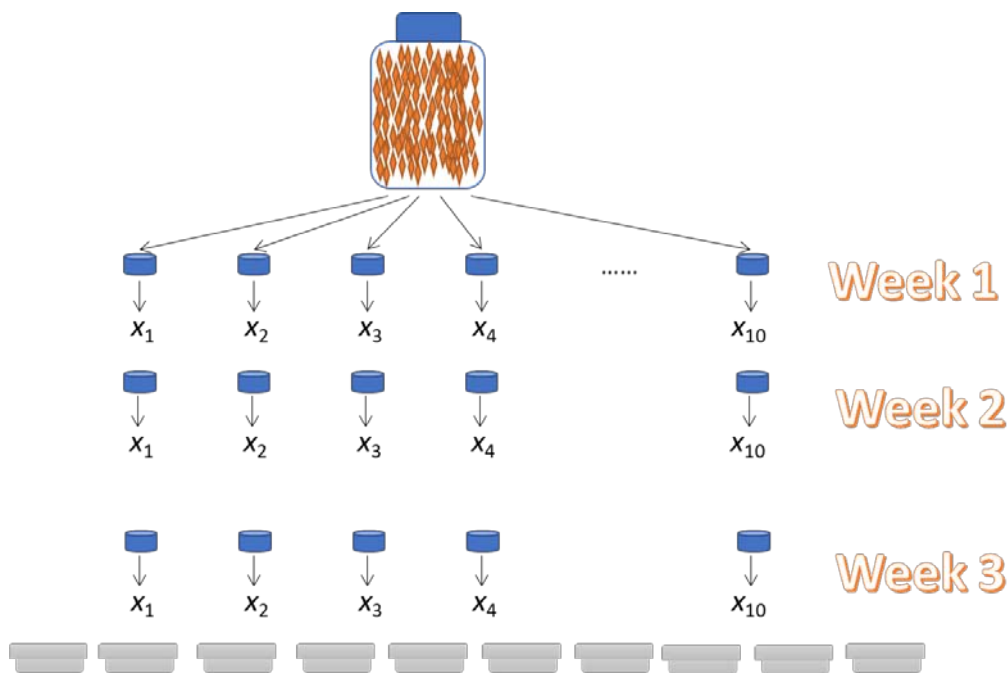
MC moisture content, %

m₀ mass of sample before drying, g

m₁ mass of sample after drying, g.

Uncertainty of CRMs (U_{crm})

The homogeneity of each CRM was evaluated according to the guidelines outlined in ISO Guide 35. The assessment involved taking 10 subsamples, each with three replicates, from a single bottle. This approach allowed for the examination of the uniform distribution of properties within the material. The instability of the CRM refers to its ability to maintain consistent properties over time. To assess the long-term instability, the linear regression method recommended in the guidelines was utilized. This approach allowed for the evaluation of the drift rate of the moisture content and the estimation of the material's shelf life. The measurement uncertainties ranging from 0.15% to 0.30% over three months.



Pic. 3 A scheme for evaluating the homogeneity and stability of CRMs.

Phase 2: Verify and consistency of the CRMs

Preparation of sample for moisture content in rice/paddy

Rice/paddy samples were prepared using the CRMs method. Four samples rang (12 to 18) %MC passed the homogeneity test, including preliminary stability studies. Each 20 g/sample was vacuum-sealed in plastic bags with labeled sample codes (P01, P02 for paddy and R03, R04 for milled rice), without moisture level shown. The samples were distributed to 11 participant laboratories.



Pic. 4 Samples of rice/paddy are packaged in a vacuum sealed plastic bag at 20 g/bag/moisture level.

Study of the homogeneity and uncertainty of rice moisture samples

Samples of rice moisture content were randomized three times to study homogeneity according to ISO Guide 35, using one-way ANOVA at a 95% confidence level. The F statistic was compared with the F critical value. If the F statistic is less than the F critical value, it indicates that the prepared sample is sufficiently homogeneous.

Number of sample	F statistic	F critical	Conclusion	u' _{bb}
P01	0.779	2.393	Sufficiently homogeneous	0.050 %MC
P02	0.563	2.393	Sufficiently homogeneous	0.025 %MC
R03	0.122	2.393	Sufficiently homogeneous	0.023 %MC
R04	0.202	2.393	Sufficiently homogeneous	0.019 %MC

Table 1 The study result for homogeneity of samples by one-way ANOVA

Stability study of rice moisture samples

Short-term stability tests used an isochronous scheme under transport conditions for up to 3 days. Samples were placed in an oven at 40°C (estimated shipping temperature) and analyzed after 4 days under repeatability conditions. Some were stored at (6 ± 2)°C for long-term stability studies. Results showed stability at 40°C for 4 days (t-calculate < t-critical at 95% confidence) as summarized in Table 2.

Number of sample	t-calculate	t-critical	Conclusion
P01	1.903	2.571	Stable
P02	0.573	2.447	Stable
R01	0.663	2.571	Stable
R02	0.057	2.447	Stable

Table 2 The study result for stability of rice moisture samples at 40 °C

Table 3 shows the comparison of moisture measurement results in paddy and milled rice, based on the CRM preparation method. All 11 laboratories used measurement standards to determine values from the moisture content of rice references according to ISO 712 (Determination of Moisture Content - Reference Method). However, the proficiency testing program did not include an analysis of measurement uncertainty. Instead, the standard deviation results from all 11 laboratories were analyzed to calculate Z'-Scores, revealing that 90% of the laboratories met the 'satisfactory' criteria for measurement results.

Laboratory's Code	Z'-Scores			
	P01	P02	R01	R02
PR2022.1	0.78	3.06	-0.68	-1.27
PR2022.2		-1.34		-0.74
PR2022.3	-0.09	-2.25	-0.10	-0.88
PR2022.4	-0.73	-0.37	2.40	-0.54
PR2022.5	0.66	-0.07	-0.03	0.15
PR2022.6	0.20	-0.17	0.55	0.07
PR2022.7	-1.88	3.01	-1.01	1.80
PR2022.8	-0.77		-0.83	
PR2022.9	0.84	0.49	0.26	0.97
PR2022.10	-0.46	-0.07	0.48	0.15
NIMT	0.98	0.47	0.19	0.61

Table 3 The result of Z'-Scores are shown for all participants.

Phase 3: Used for quality assurance

These CRMs have been successfully developed and can be used for quality assurance in grain moisture measurement, particularly in Thailand. They have been launched to clients and optimized to achieve an accepted uncertainty range of 0.15% to 0.30% over a shelf life of three months. Our CRMs support a wide range of moisture content, from 5% to 25%, and are compatible with various types of grains or flours, depending on the required characteristics of the equipment used for moisture measurement. This innovation was awarded 3rd place in the Industrial Category at the Rice Innovation Award 2024, organized by Thai Rice Foundation under Royal Patronage in collaboration with the National Innovation Agency.

The next step is to assess the quality system for the production of CRMs to enhance confidence among domestic laboratories, in line with NIMT's responsibilities.



Pic. 4 CRMs moisture in rice/paddy



Pic. 5 3rd of Rice Innovation Award 2024

Reference

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- 2.ISO 712:2009 “Cereals and cereal products – Determination of moisture content - Reference method
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Contact the Author



Mrs. Thasorn Sinhaneti
National Institute of Metrology (Thailand),
Pathumthani, Thailand
E-mail: thasorn@nimt.or.th

Ms.Photjanat Phimnom
National Institute of Metrology (Thailand),
Pathumthani, Thailand
E-mail: photjanatp@nimt.or.th

