Evaluation of Moisture Content Consistency Using Standard Loss-on-Drying Methods.

Abstract :

Accurate moisture content determination is essential for quality control and material characterization in industrial and laboratory applications. Several techniques are commonly used for moisture determination; however, their consistency and agreement must be validated against traceable reference methods. This study aims to evaluate the agreement, consistency, and measurement uncertainty between two standard moisture determination techniques developed at the National Institute for Standards (NIS): the oven-drying (loss-on-drying) method and the moisture balance method. Moisture content measurements were performed on solid samples covering a wide range of moisture levels, with the oven-drying method serving as the reference technique. Statistical analyses, including correlation analysis, linear regression, bias evaluation, and Bland–Altman analysis, were applied. Measurement uncertainty was evaluated in accordance with the Guide to the Expression of Uncertainty in Measurement (GUM). The results showed a strong linear association between the two methods, with a correlation coefficient of 0.89. A systematic negative bias of −0.42% was observed for the moisture balance. However, all observed differences were within the expanded pair uncertainty (±0.68%), confirming consistency between the two methods within the stated uncertainty limits. These findings support the suitability of the moisture balance for routine moisture determination when appropriate calibration and uncertainty evaluation are applied.

Key words:-
Moisture content, Oven-drying method, Moisture balance, Agreement analysis, Measurement uncertainty

1.Introduction

Moisture content determination is widely recognized as a critical parameter in material characterization and quality control across industrial and laboratory applications [1,2]. Severalstandardized techniques have been established for moisture determination, each with specific advantages and limitations depending on material type and moisture range [3,4].

Among the available methods, the oven-drying (loss-on-drying) technique is commonly accepted as a reference method due to its traceability and standardized operating conditions [5]. Moisture balance instruments are increasingly used for routine measurements because of their rapid response and operational simplicity; however, their performance must be validated against reference methods [6].

Despite the widespread use of moisture balance instruments for routine moisture determination, many previous studies have focused primarily on correlation or repeatability analysis, with limited attention to metrological agreement and uncertainty-based consistency with traceable reference methods. Comprehensive evaluations combining bias estimation, Bland–Altman agreement analysis, and GUM-compliant uncertainty assessment remain insufficiently addressed.

Therefore, the objective of this study is to compare an accredited oven-drying (loss-on-drying) reference method with a moisture balance method developed at the National Institute for Standards (NIS), evaluate their agreement using statistical and metrological tools, and assess their consistency through uncertainty-based analysis. The significance of this work lies in providing a traceable and uncertainty-supported validation framework that supports the reliable use of moisture balance instruments for routine moisture content determination and process monitoring.

2. Methodology

Two commonly used techniques for moisture determination were investigated: the oven-drying (Dry Method) and the moisture balance method. Both methods are based on the principle of mass loss due to moisture evaporation; however, they differ in procedure, measurement time, and operational characteristics.

2.1 Oven-Drying Method (Dry Method)

The oven-drying method is based on measuring the mass loss of a sample after exposure to a controlled temperature for a specified period. Samples were dried in a laboratory oven at temperatures ranging from 105 to 110 °C until a constant mass was achieved as shown in Figure 1. Moisture content was calculated from the difference between the initial and final sample masses.

The main apparatus used in this method included:

- Laboratory oven for controlled drying,
- High-precision balance (AT 201) with a resolution of ±0.001 g,
- Sample containers,
- Desiccator to cool samples and prevent moisture reabsorption,
- Heat-resistant gloves for safe handling.

Although the oven-drying method is time-consuming, it provides high accuracy and reliability, making it suitable for solid and bulk materials requiring precise moisture determination.

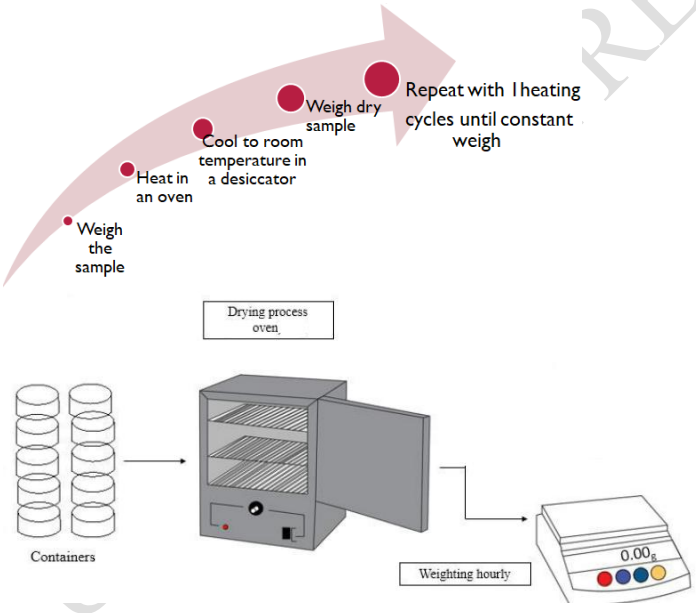

Fig. 1. Schematic diagram of the oven-drying method

2.2 Moisture Balance Method

Moisture balance instruments integrate precision weighing with controlled heating to enable real-time moisture determination [7]. In this study, a KERN DBS60-3 moisture balance was used. The sample was placed on the weighing pan, and the instrument recorded the initial mass before applying controlled heating to evaporate moisture. The mass was continuously monitored until stabilization, and the moisture content was calculated automatically as a percentage of mass loss.

This method provides rapid results and ease of operation with minimal sample handling, making it suitable for routine measurements. However, the instrument requires calibration to correct for systematic deviations and ensure accurate quantitative results.

The findings of this study are limited to the tested solid samples and the investigated moisture range. Extrapolation of the results to other material types or significantly different moisture contents should be performed with caution.

3. Results

Moisture content measurements were performed on samples covering a wide range of moisture levels using both methods. For each sample, five repeated measurements were conducted to assess repeatability and consistency [8-10]. The oven-drying method was considered the reference technique due to its traceability and accredited status.

3.1 Comparison of Moisture Content Results

Table 1 presents the moisture content values obtained using both methods. The resultsin Figure 2 show close agreement across the moisture range investigated. Slightly lower values were generally observed for the moisture balance, particularly at higher moisture levels.

Table 1. Moisture content results obtained using the oven-drying method and moisture balance

Samples	Oven dry method%	Moisture balance%
A	0.21	0.26
B	2.63	1.89
C	12.46	12.04
D	**17.85**	**17.3**3
E	**20.63**	19.95

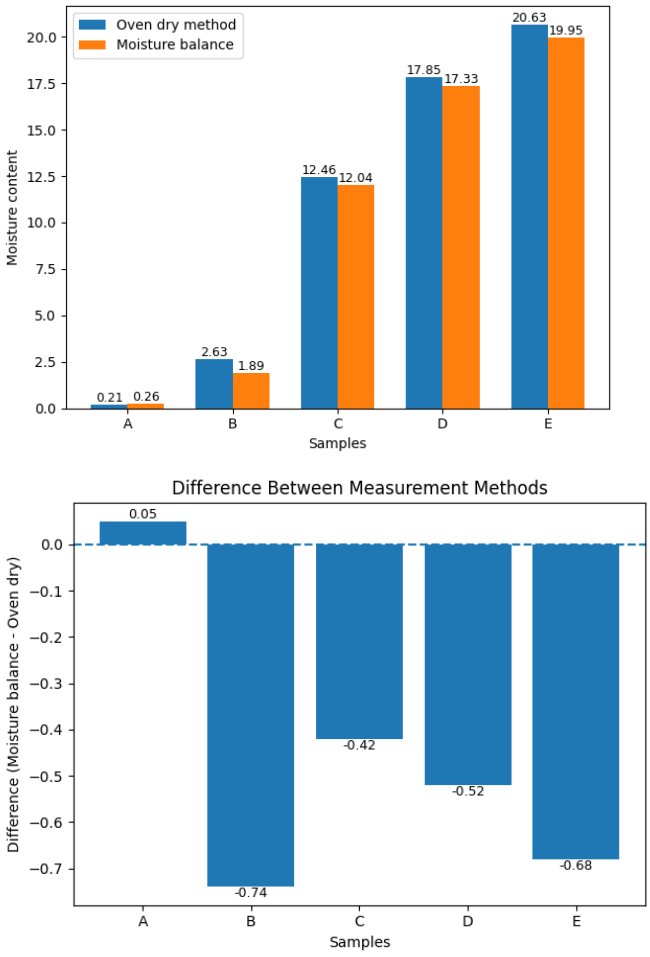

Fig. 2. Comparison between two methods

3.2 Correlation Analysis

The linear association between the two measurement techniques was evaluated using the Pearson correlation coefficient, calculated according to Eq. (1). The resulting correlation coefficient was r = 0.99, indicating a strong positive correlation. This confirms that both methods consistently track variations in moisture content across the investigated range.

$$r = \frac{\sum(x_i - \bar{x})(y_i - \bar{y})}{\sqrt{\sum(x_i - \bar{x})^2 \sum(y_i - \bar{y})^2}} \quad (1)$$

Correlation analysis was employed to evaluate the linear association between the two methods; Although a high correlation coefficient indicates a strong linear association, correlation alone does not imply agreement between measurement methods [11]. Therefore, correlation analysis was complemented by regression analysis, bias evaluation, Bland–Altman agreement analysis, and uncertainty-based consistency assessment.

3.3 Linear Regression Analysis

Linear regression analysis is commonly applied to identify proportional and constant bias between measurement methods and to support calibration model development [12,13]

The regression relationship is expressed by Eq. (2):

$$\boldsymbol{Balance_{Method} = -1.06 + 1.08\, x\, Dry_{Method}} \quad (2)$$

The regression slope (1.08) is close to unity, indicating acceptable proportional agreement. However, the non-zero intercept (−1.06) reveals the presence of a systematic offset between the two methods. This offset suggests that calibration of the moisture balance is required for accurate quantitative measurements as shown in Figure 3.

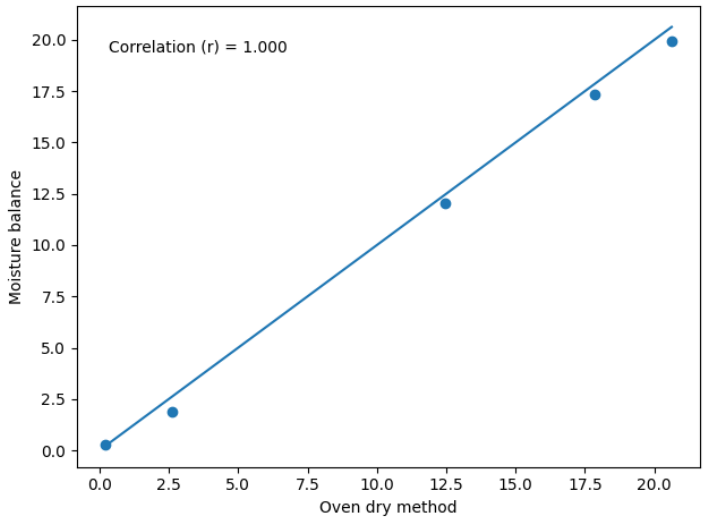

Fig. 3. Linear regression for consistency check between two methods

3.4 Bias (Mean difference) Evaluation

The systematic difference between the oven-drying method and the moisture balance was quantified by calculating the mean bias using Eq. (3). For each measurement run, the difference was defined as di $= (m_{balance\ ,i-} - m_{dry\ ,i})$.

$$\boldsymbol{Bias = \frac{1}{n}\sum_{i=1}^{n}(m_{balance,i-}m_{dry,i})} \quad (3)$$

Based on five repeated measurements performed at a representative moisture level as shown in Table 2, the calculated mean bias was −0.42%, indicating a consistent underestimation as shown in Figure 4 of moisture content by the moisture balance relative to the oven-drying reference method.

Table 2. Repeated measurements used for bias evaluation

Run	Oven dry method (%)	Moisture balance (%)	Difference (%)
1	12.5	12.3	0.2
2	12.6	12.1	0.5
3	12.3	11.8	0.5
4	12.7	12.3	0.4
5	12.2	11.7	0.5

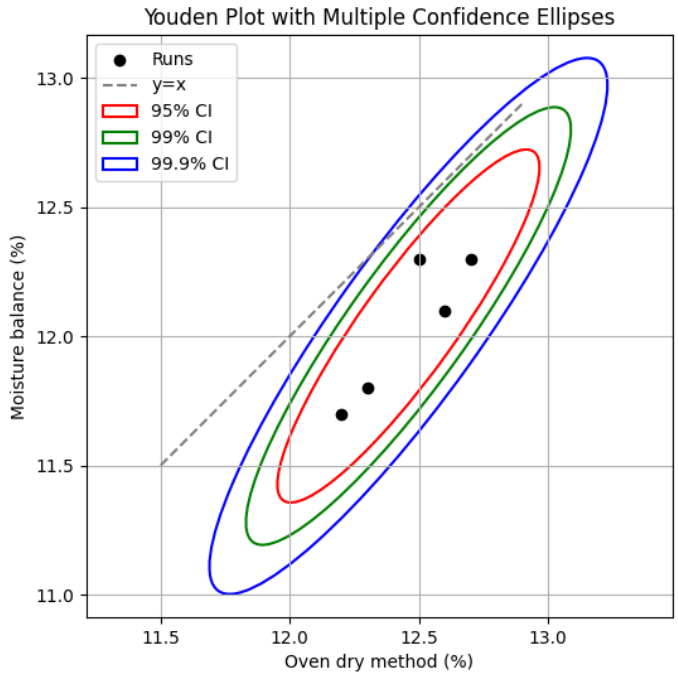

Fig. 4. Underestimated bias against the equivalence between two methods

3.5 Agreement Analysis (Bland–Altman Method)

Agreement between the two methods was further assessed using Bland–Altman analysis [14]. The standard deviation of the differences between paired measurements was calculated using Eq. (4) and found to be 0.13%.

$$S_d = \frac{1}{n-1}\sqrt{\sum_{i=1}^{n}[(m_{balance,i} - m_{dry,i}) - Bias]^2} \quad (4)$$

The 95% limits of agreement were determined using Eq. (5) and ranged from −0.67% to −0.17%. All observed differences were found to lie within these limits, demonstrating acceptable

agreement between the two methods within the investigated moisture range, despite the presence of a systematic negative bias.

$$\mathbf{LoA} = \mathbf{Bias} \pm \mathbf{1.96xSd} \quad (5)$$

3.6.Uncertainty Evaluation

Measurement uncertainty was assessed in accordance with the principles of the Guide to the Expression of Uncertainty in Measurement (GUM) which provides a harmonized framework for combining statistical and non-statistical uncertainty components [15–17]. The evaluation considered both statistical contributions derived from repeated measurements (Type A) and non-statistical contributions associated with instrument performance and experimental conditions (Type B).

For the oven-drying method, the combined standard uncertainty (u_1) was determined by accounting for repeatability, balance resolution, oven temperature stability, and sample handling effects. The resulting combined standard uncertainty ranged from 0.12% to 0.22%.

Similarly, the combined standard uncertainty for the moisture balance method (u_2) was evaluated by considering measurement repeatability, instrument resolution, heating uniformity, and calibration-related contributions. The obtained combined standard uncertainty ranged from 0.16% to 0.26%.

For the oven-drying method, repeatability and balance resolution were identified as the dominant contributors to the combined uncertainty, particularly at low moisture levels. For the moisture balance, repeatability and heating uniformity were the main contributors, while calibration-related effects became more significant at higher moisture contents.

The combined standard uncertainty was calculated using the root-sum-square approach, while the expanded uncertainty was obtained by applying a coverage factor of k = 2, corresponding to a confidence level of approximately 95% [18-21], resulting in U_1 = 0.24% to 0.44% and U_2 = 0.32% to 0.52%, respectively.

The combined standard uncertainty was calculated as $u_C = \sqrt{\sum_{i=1}^{n} u_i^2}$, and the expanded uncertainty was obtained as $U = k\,x\,u_C$, where k is the coverage factor.

The Bland–Altman analysis assumes that the differences between paired measurements are approximately normally distributed and independent, which was verified through exploratory data analysis. The GUM-based uncertainty evaluation assumes linearity and independence of uncertainty components, which is considered valid for the applied measurement models.

3.7 Degree of Equivalence and Consistency Assessment

The degree of equivalence approach is widely used in metrology to assess consistency between measurement results obtained using different methods or laboratories [22,23]

The expanded pair uncertainty associated with the difference between the two measurement methods was calculated according to $U_P = k\ x\ \sqrt{(u_1^2 + u_2^2)}$ by combining the individual standard uncertainties u_1 and u_2. The resulting expanded pair uncertainty was U_p = 0.68%. The degree of equivalence was then expressed as $D = m1 - m2 \pm Up$

In the present study, the observed mean difference between the two methods was −0.42%, which lies within the expanded pair uncertainty of ±0.68%. This confirms that the oven-drying method and the calibrated moisture balance are consistent within the stated uncertainty limits.

From a calibration and metrological perspective, the oven-drying method serves as a reliable reference standard due to its traceability and validated performance. Although the moisture balance exhibited a systematic negative bias, its magnitude remains small and can be effectively corrected using the derived regression model. When supported by appropriate calibration and comprehensive uncertainty evaluation, the moisture balance method is suitable for routine moisture content determination and process monitoring.

Potential influencing factors such as sample heterogeneity, operator handling, ambient laboratory conditions, and measurement duration were considered during the experimental design. All measurements were performed under controlled laboratory conditions by the same trained operator to minimize operator-induced variability. While the oven-drying method requires significantly longer measurement time, the moisture balance offers rapid results, which is advantageous for routine applications.

4. Conclusions

This study presented a comparative evaluation of two standard moisture determination techniques: the oven-drying (loss-on-drying) method and the moisture balance method. The oven-drying method, serving as the reference technique due to its traceability and accredited performance, was used to assess the consistency and agreement of the moisture balance measurements. The results demonstrated a strong linear correlation between the two methods (r = 0.89), indicating that both techniques reliably track changes in moisture content over the investigated range. Although a systematic negative bias of −0.42% was observed for the moisture balance method, Bland–Altman analysis confirmed that all measurement differences fell within the calculated 95% limits of agreement. Measurement uncertainty evaluation, performed in accordance with the Guide to the Expression of Uncertainty in Measurement (GUM), showed that the observed differences between the two methods were smaller than the expanded pair uncertainty. This confirms the consistency of the two methods within the stated uncertainty limits. In conclusion, the moisture balance method demonstrates acceptable agreement with the oven-drying reference method and c can be reliably used for routine moisture determination within the investigated moisture range, provided that regular calibration is applied and measurements at extreme moisture levels are verified against a reference method, provided that appropriate calibration and traceable reference measurements are applied. Future work will focus on inter-laboratory comparisons, evaluation of additional material types, and extending the investigation to wider moisture ranges to further support the metrological validation of moisture balance instruments.

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275

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