NIST Internal Report
NIST IR 8574

Cannabis Laboratory Quality Assurance Program: Exercise 3 Moisture Final Report

Andrea J. Yarberry
Charles A. Barber
Walter B. Wilson
Laura J. Wood

This publication is available free of charge from:
https://doi.org/10.6028/NIST.IR.8574

NIST NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY
U.S. DEPARTMENT OF COMMERCE

NIST Internal Report
NIST IR 8574

Cannabis Laboratory Quality Assurance Program: Exercise 3 Moisture Final Report

Andrea J. Yarberry
Charles A. Barber*
Walter B. Wilson
Laura J. Wood †
Chemical Sciences Division
Material Measurement Laboratory

**Former NIST employee; all work for this publication was done while at NIST.*
† Retired NIST Author

This publication is available free of charge from:
https://doi.org/10.6028/NIST.IR.8574

April 2025

U.S. Department of Commerce
Howard Lutnick, Secretary

National Institute of Standards and Technology
Craig Burkhardt, Acting Under Secretary of Commerce for Standards and Technology and Acting NIST Director

NIST IR 8574
April 2025

Certain equipment, instruments, software, or materials, commercial or non-commercial, are identified in this paper in order to specify the experimental procedure adequately. Such identification does not imply recommendation or endorsement of any product or service by NIST, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

NIST Technical Series Policies
Copyright, Use, and Licensing Statements
NIST Technical Series Publication Identifier Syntax

Publication History
Approved by the NIST Editorial Review Board on 2025-04-25

How to Cite this NIST Technical Series Publication
Yarberry AJ, Barber CA, Wilson WB, Wood LJ (2025) Cannabis Laboratory Quality Assurance Program: Exercise 3 Moisture Final Report. (National Institute of Standards and Technology, Gaithersburg, MD), NIST Internal Report (IR) NIST IR 8574. https://doi.org/10.6028/NIST.IR.8574

Author ORCID iDs
Andrea J. Yarberry: 0000-0001-6899-3000
Charles A. Barber: 0000-0002-4968-7486
Walter B. Wilson: 0000-0003-1763-781X
Wood LJ: 0000-0002-4294-7636

Contact Information
cannabis@nist.gov

Abstract

NIST launched a Cannabis Laboratory Quality Assurance Program (CannaQAP) in 2020 to assist laboratories in demonstrating and improving cannabis (hemp and marijuana) measurement comparability and competence. CannaQAP provided tools that allowed analysts and laboratories to assess how their in-house methods perform relative to the community and to an accepted value. This study in Exercise 3 of CannaQAP focused on the determination of moisture in one hemp material provided by NIST. This report provides a detailed description of the results of this study. The wide range of moisture content reported by participating laboratories using several different drying methods indicates the need for consistent hemp drying method(s) for accurate and precise measurements.

Keywords

CannaQAP; Cannabis; hemp; moisture.

Table of Contents

List of Tables

List of Figures

Acknowledgments

Melissa M. Phillips is acknowledged for her work in preparing the data submission website and assisting Shaun Kotoski and Carolyn Burdette in preparing data tables for the preliminary reports provided to the participants.

Catherine A. Rimmer, Johanna Camara, Marcela Najarro, and Colleen Bryan Sallee of the NIST Material Measurement Laboratory are acknowledged for their careful review of this report.

Executive Summary

One hundred and seventy-six laboratories signed up to participate in Exercise 3 of the Cannabis Laboratory Quality Assurance Program (CannaQAP) to measure cannabinoids, toxic elements, and/or moisture in five cannabis (hemp and marijuana) plant samples and two cannabis oil samples. One hundred and sixteen laboratories were selected to receive Plant Sample 5, with 89 submitting results. Plant Sample 5 was grown in the United States, prepared at NIST following normal reference material procedures, and analyzed by NIST to ensure the total Δ^9-THC mass fraction was less than 0.3 %. The target mass fraction value for moisture was provided for Plant Sample 5 by NIST and determined using a loss-on-drying method in a desiccator for 36 days.

The consensus mean from the 89 participants was approximately 2 % greater than the target mean provided by NIST using a variety of analytical methods: desiccator, freeze dryer, Karl Fisher titration, forced air oven drying, vacuum oven, loss on drying via oven/balance, and thermogravimetric analysis (TA). Only 20 % of the participants reported a mean ± standard deviation within the NIST target range. The majority of laboratories with mean moisture values within the target range used either TA or selected "other" as their method option. There are no official guidelines available for within-laboratory (repeatability, RSD_r) variabilities, but participants did perform similarly to other published studies.

In general, systematic and/or random errors were the cause of the majority of inaccurate or less precise results. These errors are most likely the result of inappropriate sample storage and/or weighing inconsistencies. Because the initial conditions impact how moisture is removed from the sample, participants should also ensure that the initial wet sample mass and depth in the crucible are similar among sample replicates. When an oven method is used, the type of oven will affect the temperature variation, with convection ovens having higher variability than forced air ovens. When using an oven drying method, participants should run preliminary studies to determine if crucible configuration affects the precision of the measurements, as hot spots in an oven can lead to high variability across samples.

1. Introduction

The Cannabis Laboratory Quality Assurance Program (CannaQAP) was formed in 2020 to assist laboratories in demonstrating and improving measurement comparability and competence in cannabis plant and cannabis-derived matrices. CannaQAP offers the opportunity for participating laboratories to assess their in-house methods for measuring cannabinoids, moisture content, and toxic elements. Reports and certificates of participation are provided to laboratories that may be used to validate their analytical methods, demonstrate compliance with cannabis good manufacturing practices (cGMPs), and fulfill proficiency testing (PT) requirements established by related accreditation bodies when PTs are not available.

In the third exercise of CannaQAP, two oil samples and five plant samples were shipped to participants for analysis of cannabinoids, moisture, and toxic elements. This report summarizes the results of the moisture measurements made on Plant Sample 5 and provides detailed discussions for potential measurement biases.

1.1. Overview of Data Treatment and Representation

In addition to this report, individualized data tables and certificates were provided to the participants who submitted data for this study. Examples of the data tables using NIST data are included in this report. Community tables and figures are provided to participants using randomized laboratory codes, with identities known only to NIST and the individual laboratories. The statistical approaches are outlined below for each type of data representation.

1.1.1. Statistics

Data tables and figures throughout this report contain information about the performance of each laboratory relative to that of the other participants in this study and, if available, relative to a target around the expected result. All calculations are performed in PROLab Plus (QuoData GmbH, Dresden, Germany). The consensus means and standard deviations are calculated according to the robust Q/Hampel method outlined in ISO 13528:2022, Annex C [1].

1.1.2. Individualized Data Table

The individualized data table contains data that is specific to each participating laboratory. The purpose of the table is to allow participants to directly compare their data to the summary statistics (consensus or community data as well as NIST certified, non-certified, or estimated values, when available). Participating laboratories received uniquely coded individualized data tables in a separate distribution that included a randomized laboratory code, located in the upper left section of the data table. Example individualized data tables are included in this report with the section allocated for individual laboratory data (Section 1. Your Results) shaded as illustrated in **Table 1-1**.

Table 1-1. Example of an individualized data table.

(Lab Name)

Exercise 3 – Moisture in Hemp Plant Samples

	Lab Code:	**(Code)**	**Section 1. Your Results**				**Section 2. Community Results**			**Section 3. Target**	
	Sample[a]	Units[b]	x_i	s_i	Z'_{comm}	Z_{NIST}	N	x*	s*	x_{NIST}	*u*
c_1[c]	a_1	b_1	*Individual laboratory results will appear in this section; laboratory-specific results were provided to each participant separately from this report.*				N_1	x^*_1	s^*_1	x_{NIST1}	u_1
…	…	…					…	…	…	…	…
…	…	…					…	…	…	…	…
c_n	a_n	b_n					N_n	x^*_n	s^*_n	x_{NISTn}	u_n

x_i	Mean of reported values	N	Number of quantitative values reported	x_{NIST}	NIST-assessed value
s_i	Standard deviation of reported values			*u*	standard uncertainty about the NIST-assessed value
Z'_{comm}	Z'-score with respect to community consensus	x*	Robust mean of reported values		
Z_{NIST}	Z-score with respect to NIST value	s*	Standard deviation for proficiency testing		

[a] Samples used in the study.
[b] Units used to describe the measured values.
[c] Analytes measured in the study.

Section 1 of the data table (Your Results) contains the laboratory results as reported, including the mean and standard deviation when multiple values were reported. A blank indicates that NIST does not have data on file for that laboratory for the corresponding analyte. An empty box for standard deviation indicates that the participant reported a single value or a limit of quantification (LOQ).

Also included in Section 1 are two Z-scores. The first Z-score, Z'_{comm}, is calculated with respect to the community consensus value, taking into consideration bias that may result from the uncertainty in the assigned consensus value, using the consensus mean (x^*), robust estimate of the standard deviation of the reported values (s^*), and standard deviation for proficiency assessment (σ_{PT}) determined from the Q/Hampel estimator Eq. (1).

$$Z'_{\mathrm{comm}} = \frac{x_i - x^*}{\sqrt{\sigma_{\mathrm{PT}}^2 + s^{*2}}} \quad (1)$$

The second Z-score, Z_{NIST}, is calculated with respect to the target value (x_{NIST}, NIST certified, non-certified, or estimated value, when available), and either U_{95}, where U_{95} is the expanded uncertainty on an assigned value), Eq (2), or U_{NIST} (where U_{NIST} is an estimated expanded uncertainty of NIST and/or other measurements), Eq (3).

$$\boldsymbol{Z_{\mathrm{NIST}}} = \frac{\boldsymbol{x_i - x_{\mathrm{NIST}}}}{\boldsymbol{2 * U_{95}}} \quad (2)$$

$$Z_{\mathrm{NIST}} = \frac{x_i - x_{\mathrm{NIST}}}{2 * U_{\mathrm{NIST}}} \quad (3)$$

The significance of the *Z*-score and *Z'*-score is as follows [1]:

- $|Z|$ < 2 indicates that the laboratory result is considered to be within the community consensus range (for Z'_{comm}) or NIST target range (for Z_{NIST}).
- 2 < $|Z|$ < 3 indicates that the laboratory result is considered to be marginally different from the community consensus value (for Z'_{comm}) or NIST target value (for Z_{NIST}).
- $|Z|$ > 3 indicates that the laboratory result is considered to be significantly different from the community consensus value (for Z'_{comm}) or NIST target value (for Z_{NIST}).

Section 2 of the data table (Community Results) contains the consensus results, including the number of laboratories reporting quantitative values for each analyte (*n*), the mean value determined for each analyte (x^*), and a robust estimate of the standard deviation of the reported values (s^*) [1]. Additional information on the calculation of the consensus mean and standard deviation can be found in Section 1.1.1.

Section 3 of the data table (Target) contains the NIST target values for each analyte (x_{NIST}), when available. When possible, the target value is a certified value, a non-certified value, or a value determined at NIST. A NIST certified value is a value for which NIST has the highest confidence in its accuracy and that all known or suspected sources of bias and variability have been considered [2]. For samples in which a NIST certified or non-certified value is not available, a target value may be determined at NIST using an established method, or data from a collaborating laboratory. The target value represents the mean of at least three replicates. For materials acquired from and/or evaluated as a part of another interlaboratory study or proficiency testing program, the consensus value and uncertainty from the completed round are used as the target range.

In this study, a target value for the moisture content in Plant Sample 5 was determined at NIST using the desiccator drying method outlined in Section 2.1. The target value represents the mean of a minimum of three independent measurements, which permitted NIST to provide an expanded uncertainty (U_{NIST}) to encompass variability due to inhomogeneity among packaged units. A unique feature of NIST QAPs is the accuracy-based component provided by comparing participant results to a NIST-measured value.

1.1.3. Summary Data Table

This data table includes a summary of all reported data for a particular analyte in a particular study. Participants can compare the raw data from their laboratory to data reported by the other participating laboratories and to the consensus data. A blank indicates that the laboratory signed up and received samples for that analyte and matrix, but NIST does not have data on file for that laboratory. The standard deviation (SD) for the target value in this table is the uncertainty (U_{NIST} *or* U_{95}) around the target value. Data highlighted in red have been flagged as a data entry of zero or results that include text (e.g., "< LOQ" or "present"). Data highlighted in blue have been identified as outside the consensus tolerance limits and would be estimated to yield $|Z'_{comm}|$ > 2. The summary data tables are presented in the format shown in **Table 1-2**.

Table 1-2. Example data summary table.

		Analyte									
		Sample 1 (units)					Sample 2 (units)				
		A	B	C	Avg[a]	SD[b]	A	B	C	Avg	SD
Individual Results	Target				c_1	d_1				c_2	d_2
	e_1	x_{A1-1}	x_{B1-1}	x_{C1-1}	$\bar{x}_{1-1}$	s_{1-1}	x_{A2-1}	x_{B2-1}	x_{C2-1}	$\bar{x}_{1-2}$	s_{1-2}
	…	…	…	…	…	…	…	…	…	…	…
	e_n	x_{A1-n}	x_{B1-n}	x_{C1-n}	$\bar{x}_{n-1}$	s_{n-1}	x_{A2-n}	x_{B2-n}	x_{C2-n}	$\bar{x}_{n-2}$	s_{n-2}
Community Results		Consensus Mean			f_1		Consensus Mean			f_2	
		Consensus Standard Deviation			g_1		Consensus Standard Deviation			g_2	
		Maximum			h_1		Maximum			h_2	
		Minimum			i_1		Minimum			i_2	
		N			j_1		N			j_2	

[a] Arithmetic average of sample replicates.
[b] Standard deviation of sample replicates.
[c] Target value for the sample.
[d] Standard deviation of the target value for the sample.
[e] Laboratory identifier for the participant.
[f] Robust mean of reported results.
[g] Robust standard deviation of reported results.
[h] Maximum of reported average results.
[i] Minimum of reported average results.
[j] Number of quantitative values reported.

1.1.4. Figures

1.1.4.1. Data Summary View (Method Comparison Data Summary View)

In this view (**Fig. 1-1**), individual laboratory data (diamonds) and the individual laboratory SD (rectangle) are plotted. Laboratories reporting values below the LOQ are shown in this view as downward triangles beginning at the LOQ, reported as Quantification Limit (QL) on the figures. Laboratories reporting values below LOQ can still be successful in the study if the target value is also below the laboratory LOQ. The solid blue line represents the consensus mean and the green shaded area represents the 95 % confidence interval for the consensus mean, which is based on the standard uncertainty of the consensus mean (u_{mean}). The uncertainty in the consensus mean is calculated using the repeatability standard deviation (s_{r}), the reproducibility standard deviation (s_{R}), the number of participants reporting data ($n_{\text{particpants}}$), and the average number of replicates reported by each participant ($n_{\text{Average Number of Replicates per Participant}}$) (Eq. 4). The uncertainty about the consensus mean is independent of the range of tolerance (solid red lines). Where appropriate, two consensus means may be calculated for the same sample if bimodality is identified in the data. In this case, two consensus means and ranges will be displayed in the data summary view.

$$u_{mean} = \sqrt{\frac{s_{\text{R}}^2 - s_{\text{r}}^2}{n_{\text{particpants}}} + \frac{s_{\text{R}}^2}{n_{\text{participants}} \times n_{\text{Average Number of Replicates per Participant}}}} \qquad (4)$$

The red-shaded region in the data summary figure represents the NIST target range for "acceptable" performance, which encompasses the NIST target value (solid green line) bounded

by twice its uncertainty (U_{95} or U_{NIST}). The solid red lines represent the range of tolerance (values that result in an acceptable Z'-score, $|Z'| \leq 2$). If the lower limit is below zero, the lower limit is set to zero. In the data summary view, the relative locations of individual laboratory data and consensus ranges with respect to the NIST target range can be compared easily. In most cases, the NIST target range, and the consensus range overlap, which is the expected result. Major program goals include both reducing the size of the consensus range and centering the consensus range about the NIST target value. Analysis of an appropriate reference material as part of a quality control scheme can help to identify sources of bias for laboratories reporting results that are significantly different from the target range. In the case in which a method comparison is relevant, different colored data points may be used to identify laboratories that used a specific approach for sample preparation, analysis, or quantitation.

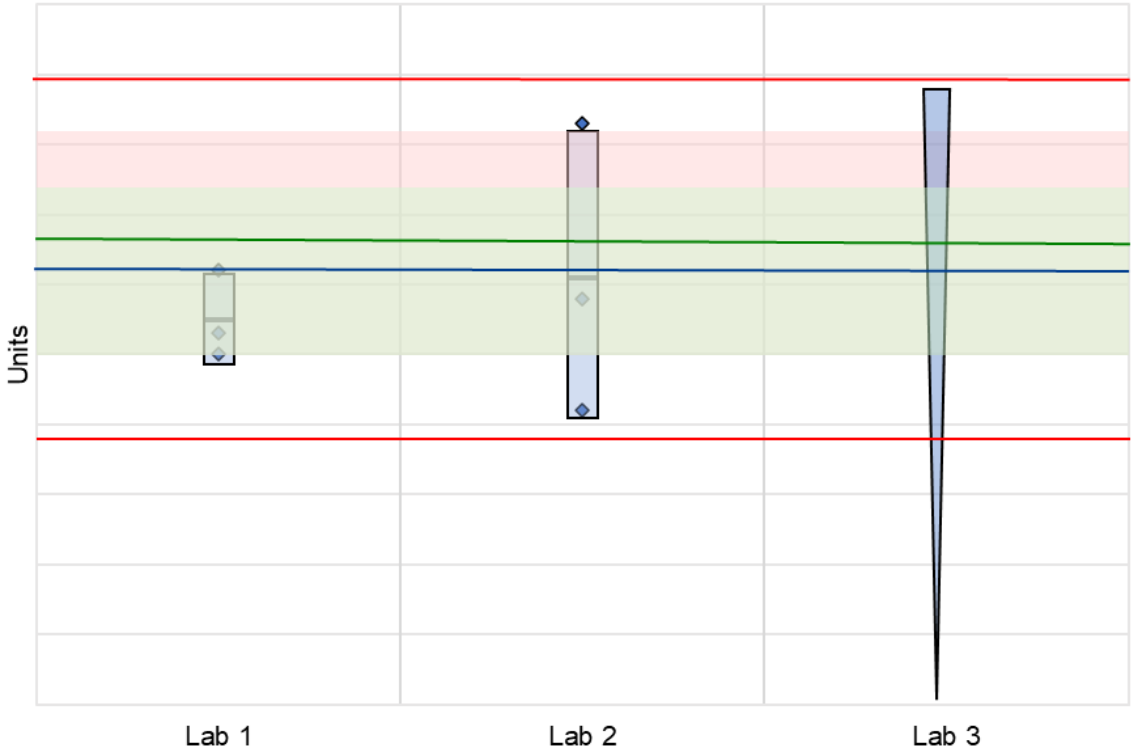

Fig. 1-1. Example data sample summary view.

1.1.4.2. Box Plots

The box plot comparison of the analytical methods used to determine moisture content by participants in this study was performed using R Statistical Software version 4.3.3 [3]. The ggplot2 package and the geom_boxplot() function were used for visualization [4]. The geom_boxplot() function uses the inter quartile range (IQR) for the box, which extends from the 25th percentile to the 75th percentile of the data. The line in the box is the 50th percentile of the data or the median. The whiskers in the plot extend from the edge of the box to the furthest data point, which is within 1.5 times the IQR. Any points outside the whiskers, are considered

outliers and are represented as dots on the figure. Each box plot in the figure represents the data submitted for each analytical method.

1.2. Study Material Acquisition and Preparation

Hemp grown and harvested in the United States was used to prepare Plant Sample 5. The bulk hemp buds, leaves, and stems were ground using a blender and then sieved to a particle size range of 250 µm to 710 µm. This hemp material was weighed into polyethylene packets (5 g), and then the packets were sealed and placed into aluminized mylar bags, each with a silica desiccant pouch. The packaged Plant Sample 5 was stored at -20 °C until being shipped to participants for analysis.

2. NIST Method for Hemp Plant Moisture Analysis

2.1. Desiccator Drying Method

The desiccator drying method was used for assigning NIST target and uncertainty values for moisture in Plant Sample 5. A Mettler AT261 Delta Range balance was used to weigh samples. The balance calibration was verified prior to each use using standard masses ranging from 0.5 g to 20 g traceable to the SI through the standard mass set maintained by the Inorganic Chemical Metrology Group at NIST.

In brief, ten packets of Plant Sample 5 were randomly selected for moisture analysis and the packets were rotated to mix prior to sampling. Hemp from each packet was placed into a pre-weighed, glass weighing vessel (m_b) to a depth of approximately one centimeter. The vessels containing hemp (m_w) were then weighed and placed into a desiccator over magnesium perchlorate [$Mg(ClO_4)_2$]. The weight of the samples in glass vessels was recorded on days (5, 7, 14, 21, 28, and 36). The moisture (%) content was then calculated based on the final vessel with hemp weight (m_d) on Day 36 using Eq. (5). This approach assumed that all mass lost was due to moisture alone.

$$\boldsymbol{moisture} = \frac{(\boldsymbol{m_w - m_d})}{(\boldsymbol{m_w - m_b})} \text{ x } 100\ \% \tag{5}$$

2.2. Uncertainty Calculations

The uncertainty associated with desiccator drying is calculated from the repeatability (SD) of the set of 10 sample means (u_a) and the balance-specific standard uncertainty of each weighing (m_b, m_w, and m_d), estimated to be ± 0.01 mg and normalized by $\sqrt{3}$ before being entered into the uncertainty equation (Eq. (7)). The standard uncertainty of the balance for each weighing was converted to moisture content by dividing the uncertainty by the respective mean sample mass (u_b, Eq. (6)).

$$standard\ balance\ uncertainty\ (u_{b,x}) = \frac{\left(\frac{0.01}{\sqrt{3}}\right)}{m_x} \tag{6}$$

The expanded uncertainty value, U (Eq. (7)), is expressed at an approximate confidence level of 95 % by choosing the expansion factor, $k = 2.26$, calculated based on degrees of freedom.

$$U = k\sqrt{\left(u_a^2 + u_{b,b}^2 + u_{b,w}^2 + u_{b,d}^2\right)} \tag{7}$$

3. Moisture Determination

3.1. Executive Summary

3.2. Study Overview

Moisture determination for cannabis is necessary for reporting cannabinoid mass fractions on a dry-mass basis, as required by the *Agriculture Improvement Act of 2018* (Farm Bill) [5]. Inaccurate moisture values can result in misidentification of cannabis as hemp or marijuana by biasing the values above or below the identification threshold. Through participation in this study, laboratories can better understand the performance of their in-house methods relative to those being used by others in the community.

3.3. Sample Information

Participants were provided with one packet of Plant Sample 5. Each packet contained 5 g of cannabis plant material (summarized in Section 1.2). Participants were asked to store the materials at –20 °C in the original unopened packets, prepare three sub-samples for analysis, and report three moisture values from the single packet provided. Participants were instructed to allow the contents of the packet to equilibrate at room temperature for 1 h and then mix thoroughly prior to subsampling for moisture analysis. The approximate moisture content was not reported to participants prior to the study. The target value for moisture in Plant Sample 5 was determined using the desiccator method described in Section 2.2.1 and the uncertainty was calculated as described in Section 2.2.2. The target values and uncertainties for moisture are provided in **Table 3-1**.

Table 3-1. Individualized data summary table for moisture in Plant Sample 5.

(Lab Name)

Exercise 3 – Moisture in Hemp Plant Material

	Lab Code:	**(Code)**	**1. Your Results**				**2. Community Results**			**3. Target**	
	Sample	Units	x_i	s_i	Z'_{comm}	Z_{NIST}	N	x*	s*	x_{NIST}	u_{NIST}
Moisture	Plant Sample 5	%	*Individual laboratory results will appear in this section; laboratory-specific results were provided to each participant separately from this report.*				89	9.04	1.90	7.02	0.19

x_i	Mean of reported values	N	Number of quantitative values reported	x_{NIST}	NIST-assessed value
s_i	Standard deviation of reported values			u_{NIST}	standard uncertainty about the NIST-assessed value
Z'_{comm}	Z'-score with respect to community consensus	x*	Robust mean of reported values		
Z_{NIST}	Z-score with respect to NIST value	s*	Standard deviation for proficiency assessment		

3.4. Study Results and Discussion

Laboratories reported the use of seven analytical methods to determine moisture content: desiccator (1 %, DES), freeze dryer (2 %, FD), Karl Fisher titration (7 %, KF), forced air oven (15 %, FAO), vacuum oven (10 %, VO), loss on drying via oven/balance (1 %, LOD), and thermogravimetric analysis (31 %, TA). Approximately one-third of the participants reported "other" as the analytical method used for moisture determination (**Fig. 3-1**).

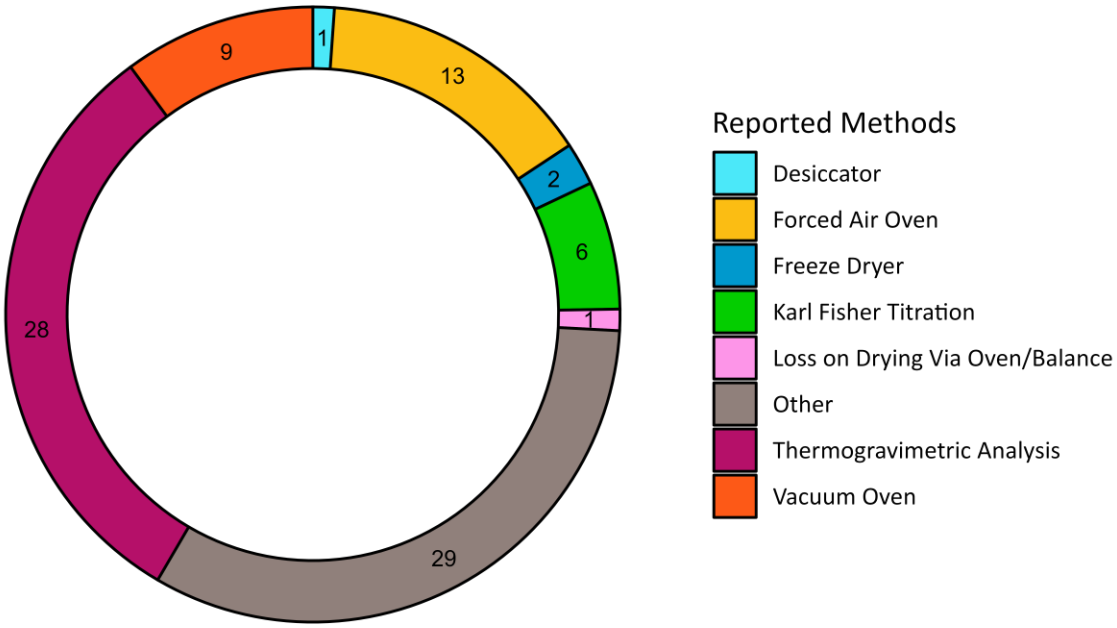

Fig. 3-1. Moisture determination methods reported by participants returning results.

Results reported by each participating laboratory are included in **Table 3-2**. A total of 116 laboratories signed up to participate in the moisture study of CannaQAP Exercise 3. Moisture results were reported for Plant Sample 5 by 89 laboratories (77 %). Eighty-three laboratories (72 %) reported at least duplicate measurements.

Table 3-2. Data summary table for moisture in Plant Sample 5. Data highlighted in blue have been identified as outside the consensus tolerance limits and resulted in an unacceptable Z'_{comm} score, $|Z'_{comm}| > 2$. Note: This table spans four pages; the NIST values and consensus values are included on all pages for convenience. The value of N represents laboratories that reported at least duplicate results.

		Moisture				
		Plant Sample 5 (%)				
	Lab	A	B	C	Avg	SD
Individual Results	Target				7.02	0.19
	C001	10.30	10.20	10.10	10.20	0.10
	C004	10.29	10.26	10.78	10.44	0.29
	C005	10.00	10.50	9.20	9.90	0.66
	C008	7.94	7.91	7.76	7.87	0.10
	C009	8.16	8.66	8.40	8.41	0.25
	C010	11.37	11.28	11.23	11.29	0.07
	C012	6.77	6.79	6.38	6.65	0.23
	C014	9.80	9.90	9.84	9.85	0.05
	C015	7.30	6.60	5.30	6.40	1.01
	C018					
	C022	10.26	10.20	10.18	10.21	0.04
	C023	8.44	8.78	8.59	8.60	0.17
	C026	6.55	6.58	6.69	6.61	0.07
	C027	10.20	10.30	10.50	10.33	0.15
	C028	6.90	6.82	6.91	6.88	0.05
	C030	10.89	10.98	10.49	10.79	0.26
	C033	7.42	6.71	6.47	6.87	0.49
	C035	8.11	8.04	8.23	8.13	0.10
	C036	9.23	7.98	9.04	8.75	0.67
	C039	10.20	10.20	10.20	10.20	0.00
	C041					
	C042					
	C043	9.93	9.74	8.35	9.34	0.86
	C045	8.55	7.86	8.22	8.21	0.35
	C046					
	C047	9.32	8.62	9.90	9.28	0.64
	C049	9.33	9.48	9.22	9.34	0.13
	C050	8.40	12.00		10.20	2.55
Community Results		Consensus Mean			9.04	
		Consensus Standard Deviation			1.90	
		Maximum			13.23	
		Minimum			4.93	
		N			83	

Table 3-2 continued. Data summary table for moisture in Plant Sample 5.

		Moisture				
		Plant Sample 5 (%)				
	Lab	A	B	C	Avg	SD
Individual Results	Target				7.02	0.19
	C053	9.50	9.40	9.40	9.43	0.06
	C057	8.58	8.72	8.31	8.54	0.21
	C058	10.12	10.59	10.67	10.46	0.30
	C060					
	C061	13.26	13.24	13.18	13.23	0.04
	C064	8.96	8.93	9.00	8.96	0.04
	C065	7.15	7.11	7.14	7.13	0.02
	C067	8.33	9.99	8.01	8.78	1.06
	C068	10.13			10.13	
	C069	7.40	7.46	7.32	7.39	0.07
	C070					
	C071					
	C072					
	C074					
	C077	11.60	11.71	10.95	11.42	0.41
	C078	11.70	11.66	11.66	11.67	0.02
	C079	5.60	4.40	4.80	4.93	0.61
	C080	10.93	10.74	11.08	10.92	0.17
	C083					
	C085					
	C088	6.17	6.94	6.69	6.60	0.39
	C089	6.27	6.29	6.27	6.28	0.01
	C090	7.90	7.80	7.80	7.83	0.06
	C091	8.45	7.36	7.41	7.74	0.62
	C093	9.89	9.01	8.78	9.23	0.59
	C094	7.26	7.28	7.11	7.22	0.09
	C096	7.21	7.67	7.69	7.52	0.27
	C098	12.63	12.98	13.04	12.88	0.22
	C099	10.59	10.65	10.62	10.62	0.03
	C100					
	C101	9.47	9.56	9.45	9.49	0.06
Community Results		Consensus Mean			9.04	
		Consensus Standard Deviation			1.90	
		Maximum			13.23	
		Minimum			4.93	
		N			83	

Table 3-2 continued. Data summary table for moisture in Plant Sample 5.

		Moisture				
		Plant Sample 5 (%)				
	Lab	A	B	C	Avg	SD
Individual Results	Target				7.02	0.19
	C106	10.60			10.60	
	C108	6.20			6.20	
	C111	8.70	8.89	8.91	8.83	0.12
	C114	11.31	11.15	11.15	11.20	0.09
	C115	8.91	9.46	8.15	8.84	0.66
	C118	9.70	9.33	9.16	9.40	0.28
	C119	8.93	8.65	8.53	8.70	0.21
	C120	8.20	7.11	7.60	7.64	0.55
	C121	8.11	8.74	8.48	8.44	0.32
	C122	9.07	9.34	9.89	9.43	0.42
	C124	10.41	10.03	9.99	10.14	0.23
	C125	10.83	10.43	9.98	10.41	0.43
	C130					
	C133	10.70	10.60	10.60	10.63	0.06
	C135					
	C138	6.02	6.41	6.23	6.22	0.20
	C139	7.07	7.14	7.36	7.19	0.15
	C140					
	C141					
	C142	10.16	10.33	9.84	10.11	0.25
	C143					
	C148	11.33	10.38	11.01	10.91	0.48
	C151	7.10	7.30	8.00	7.47	0.47
	C152	10.85	10.34		10.60	0.36
	C153					
	C154	12.18	12.32	13.07	12.52	0.48
	C156	10.83	10.57	11.02	10.81	0.23
	C158					
	C160	5.81	5.87	5.81	5.83	0.03
	C161	6.76	6.77	6.68	6.74	0.05
	C168	7.25	7.13	7.16	7.18	0.06
Community Results		Consensus Mean			9.04	
		Consensus Standard Deviation			1.90	
		Maximum			13.23	
		Minimum			4.93	
		N			83	

Table 3-2 continued. Data summary table for moisture in Plant Sample 5.

		Moisture				
		Plant Sample 5 (%)				
	Lab	A	B	C	Avg	SD
Individual Results	Target				7.02	0.19
	C170	6.49	6.54	6.68	6.57	0.10
	C171	11.59	11.84	10.60	11.34	0.66
	C173	8.18			8.18	
	C177	10.20	10.10	9.90	10.07	0.15
	C178					
	C179					
	C181	10.12	10.15	10.20	10.16	0.04
	C182	6.34	6.83	7.61	6.93	0.64
	C183	10.48			10.48	
	C184	9.66	9.51	9.54	9.57	0.08
	C185	8.32	8.45	8.32	8.36	0.08
	C186					
	C188	9.86	9.90	9.97	9.91	0.06
	C189	10.60	10.90	10.40	10.63	0.25
	C190	5.48	4.99	5.05	5.17	0.27
	C191	9.14	8.89	9.32	9.12	0.22
	C192	12.15	11.88	11.84	11.96	0.17
	C194					
	C196	12.78	12.19	11.41	12.13	0.69
	C197	7.53	7.38	7.48	7.46	0.08
	C198					
	C199	8.58	8.52	8.56	8.55	0.03
	C201	9.77			9.77	
Community Results		Consensus Mean			9.04	
		Consensus Standard Deviation			1.90	
		Maximum			13.23	
		Minimum			4.93	
		N			83	

The consensus mean of reported moisture content (9.04 %) was 28 % greater than the NIST target value (7.02 %), with no overlap between the consensus (green) and target (red) ranges (**Fig. 3-2**). There were 18 participants (20 % of participants) that reported a mean ± SD within the NIST target range. A total of 10 participants (11 % of participants) reported mean moisture values within the target range. The majority of laboratories with mean moisture values within the target range used either the TA or “other” methods. General considerations for gravimetric methods include sample size for analysis, the surface area and depth of the sample in the

weighing dish, and whether a lid was used. For larger sample masses, the moisture analysis will take longer and could result in uneven drying or decomposition of other components in the sample due to extensive drying times. The size and shape of the weighing dishes used will impact the surface area of the sample, with a smaller surface area and larger sample depth resulting in less consistent drying. A lid or cover should be used while moving the weighing dish between the oven, desiccator, and balance to prevent spillage of the sample and moisture wicking back into the sample. The weighing dish should also be dried in an oven or desiccator prior to use and stored in a functioning desiccator until use. Otherwise, the accumulated moisture on the weighing dish can contribute to the moisture content measurement of the sample. Regardless of the moisture method being used, environmental exposure to the open atmosphere can result in moisture loss or gain depending on the relative humidity and temperature in the laboratory. The analyst should try to minimize the amount of time a sample is left uncovered to reduce the effects of environmental exposure.

Fig. 3-2. Moisture in Plant Sample 5 (data summary view - analytical method).

In this view, individual laboratory data (diamonds) are plotted with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green-shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the boundaries of the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable $\boldsymbol{Z'_{comm}}$ score, $|\boldsymbol{Z'_{comm}}| \leq \mathbf{2}$. The red-shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ($\boldsymbol{U_{NIST}}$) and represents the range that results in an acceptable $\boldsymbol{Z_{NIST}}$ score, $|\boldsymbol{Z_{NIST}}| \leq \mathbf{2}$.

Figure 3-3 shows the reported moisture values organized by analytical method. The mean moisture values (dots in **Fig. 3-3**) from all of the reported analytical methods were 8 % to 56 % greater than the NIST target value (**Table 3-3**). The mean moisture value reported from laboratories using the KF method was the closest to the target value (8 % greater), followed by the single laboratory reporting use of the desiccator method (**Table 3-3**).

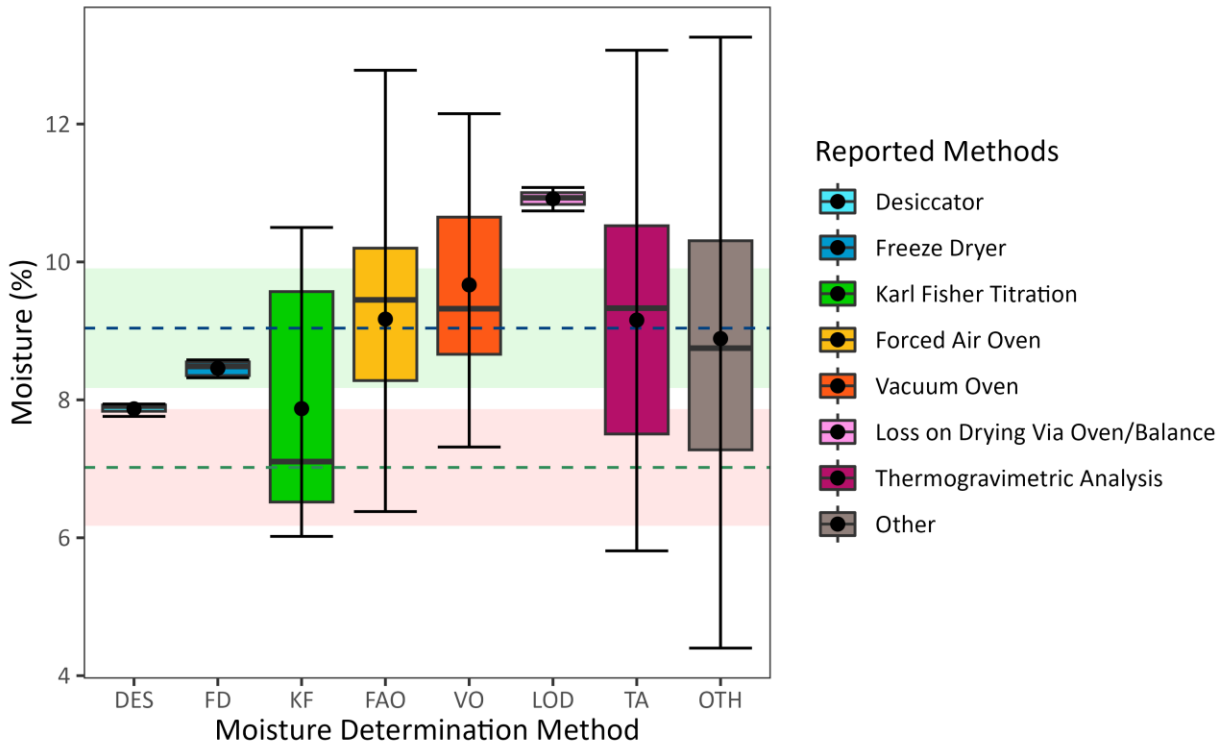

Fig. 3-3. Box and whisker plot of moisture data organized by analytical method.

The top and bottom sides of the boxes represent the upper and lower quartiles, respectively. The box covers the interquartile interval, where 50 % of the data is found. The whiskers extend from the minimum of the lower quartile to the minimum reported value and the maximum of the upper quartile to the maximum reported value. The solid blue line represents the consensus mean, and the green-shaded region represents the 95 % confidence interval for the consensus mean. The red-shaded region represents the NIST range of tolerance, which encompasses the target value (green line) bounded by twice its uncertainty ($\boldsymbol{U_{\mathrm{NIST}}}$) and represents the range that results in an acceptable $\boldsymbol{Z_{\mathrm{NIST}}}$ score, $|\boldsymbol{Z_{\mathrm{NIST}}}| \leq \mathbf{2}$.

Table 3-3. Mean moisture values determined from the analytical methods reported in this study.

Method	Number of Labs	Mean Moisture (%)	Bias[a] (%)
Thermogravimetric Analysis	28	9.25	32
Oven Drying, Forced-Air Oven (AOAC 986.25 & 990.20)	13	9.15	30
Karl Fisher Titration	6	7.59	8
Other	29	8.89	27
Oven Drying, Vacuum Oven	9	9.67	38
Freeze Dryer	2	8.46	21
Desiccator	1	7.87	12
Loss on Drying Via Oven/Balance	1	10.92	56

[a] **Bias was calculated by:** $\frac{(\boldsymbol{Mean\ moisture - NIST\ Target})}{\boldsymbol{NIST\ Target}} * \mathbf{100}$

Mean values can be misleading when data from the reported methods are distributed bimodally. The kernel density estimation of the four analytical methods that were used by more than two participants are shown in **Fig. 3-4**. The distribution of data reported by participants using each of the four methods is bimodal, with the first peak for TA, KF, and FAO methods approximately within the NIST target range (**Fig. 3-4**: A, C, and D, respectively). Data from laboratories using the VO method were skewed greater than data from the other methods, with the first peak being in line with the consensus range. When data are distributed bimodally, it indicates that the data come from two distinct clusters, which could be a result of varying method parameters for a given analytical method.

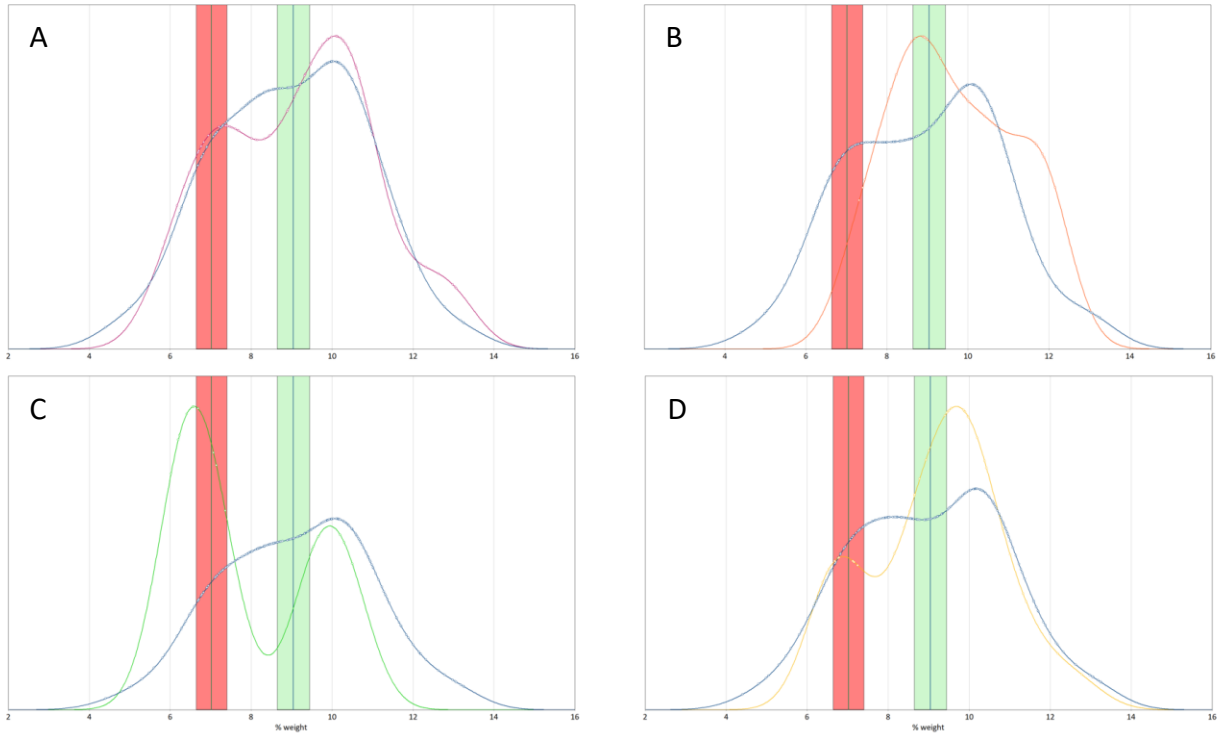

Fig. 3-4. Kernel density plots for individual analytical methods compared to the kernel density estimation of the data from the remaining analytical methods.
The analytical methods presented are A) thermogravimetric analysis, B) oven drying, vacuum oven, C) Karl Fischer titration, and C) oven drying, forced air oven.

The bimodal distribution for the oven-based methods (TA, FAO, and VO) utilized by participants is likely due to differences in oven temperature and drying time. It has been noted previously that increased time in an oven can result in an increased moisture content measurement [6] [7]. Studies have also compared the effects of oven temperature on moisture content and found that an increase in oven temperature generally corresponds to an increase in moisture content results [6] [8]. Differences in moisture content measurements at different durations in an oven and increased oven temperatures could be due to a difference in the adsorption energy of the water molecules within the material [8], loss of water due to side-reactions during heating [6] [7], or an increased loss of volatile organic compounds (VOCs) [6] [8]; all

resulting in inflated moisture content values. The emission rate of VOCs, such as terpenes in cannabis, increases at higher temperatures, resulting in an overestimation of moisture content. In the case of vacuum ovens, it has been suggested that when determining the moisture content of a sample with a high concentration of volatiles, a correction factor should be used to compensate for the loss of volatiles [9]. Because cannabis has a high percentage of terpenes, the greater overall moisture content measured by vacuum oven drying may be due to terpene loss. In general, participants using lower oven temperatures and shorter drying times likely reported lower moisture content values than participants using higher temperatures and longer drying durations, resulting in the bimodal distributions.

Unlike the oven-based methods, KF is a volumetric titration that relies on the reduction of iodine by sulfur dioxide in the presence of water [9] [10]. Typically, the reagent used for this titration contains iodine, sulfur dioxide, pyridine, and methanol; however, some reagents use other amines instead of pyridine. If the water in the sample is inaccessible, then the water in the sample is extracted by a solvent first, and the extract is titrated with a KF reagent. At a minimum, the water equivalence of the reagent needs to be standardized against a known quantity of water before each run, before KF can be used on a sample. The preferred standard to use for this process is sodium tartrate dihydrate because it is stable. Other standards include pure water, which is challenging to use because of the inaccuracy in measuring the small amounts required, and water-in-methanol is not stable over more extended periods [9]. The bimodal distribution of moisture content values measured using KF may be due to a mixture of participants relying on old standardization data and participants doing the reagent standardization directly before or after the sample analysis. It is also possible that the higher moisture content measurements are the result of moisture infiltration during the titration. It is important to note that titration vessels and solvent reservoirs must have air-tight seals and be fitted with drying tubes to prevent atmospheric moisture from interfering with the results [10]. The reagents and solvents must also be free of residual moisture [9].

When compared to the NIST target value, the one lab using the desiccator method reported data with a mean moisture value 12 % greater than the NIST value (**Table 3-3**). Moisture determination using the desiccator method is highly dependent upon the depth of the cannabis in the crucible, the duration of time the cannabis remains in the desiccator, and the amount of moisture the desiccant has taken on over time. When evaluating the moisture content of Plant Sample 5 at NIST, it was determined that 36 days in the desiccator was sufficient and that prior to 36 days, the rate of moisture loss was too high to assume all moisture had been removed from the sample. Mass loss from the sample in the desiccator is considered to be moisture; however, volatile compounds can also be lost from the sample after all of the moisture has been lost. There is no standardized desiccation duration for these plant materials and laboratories using this method should do preliminary experiments to determine the appropriate duration based on the plant material.

Regardless of the method, it is good practice to use a control material that is analyzed in tandem with the unknown sample. Method blanks are also useful in determining whether environmental conditions impacted measurements.

The within-laboratory variabilities (RSD_r), regardless of method, ranged from 0 % to 25 % for data reported by laboratories that made more than one moisture measurement, with a consensus RSD_r of 2.2 % (**Fig. 3-2**). The consensus RSD_r fell within the robust mean RSD_rs reported previously by the University of Kentucky Hemp Proficiency Testing program measuring over 20 study samples, which ranged from 2.29 % to 5.42 % [11].

There are no official guidelines available that specify RSD_r requirements for moisture measurements in cannabis plant material; however, the unofficial draft Association of Official Analytical Collaboration (AOAC) Standard Method Performance Requirements (SMPR) for the determination of moisture in hemp and cannabis plant matter [12] suggested that the RSD_r for cannabis with moisture content less than 15 % should be ≤ 2 %. The AOAC SMPR for quantitation of cannabinoids in plant materials of hemp specifies that the RSD_r for cannabinoids in hemp should be ≤ 5 % on a dry mass basis [13], which indicates that the within-laboratory variability of moisture in hemp should not be greater than 5 %. For laboratories reporting at least duplicate measurements, 21 % of the RSD_rs were greater than 5 %. Laboratories reporting use of the "other" analytical method and TA accounted for 53 % and 26 % of the laboratories with higher RSD_rs, respectively. **Table 3-4** shows average within-laboratory variabilities for moisture measurements from analytical methods that were used by more than two laboratories. While the average RSD_r for these methods is below 5 %, the RSD_r for moisture measurements by laboratories using TA is nearly double that of the other three methods. The higher within-laboratory variability for TA could be due to participants using instruments that can only analyze one sample at a time rather than having all replicates in an oven simultaneously. Any instrumental drift that occurs during TA using single sample instruments will not be spread over the batch, resulting in higher within-laboratory variability. Participants reporting the use of the DES, FD, and LOD methods did not report any moisture values within the target range. The same laboratories had within-laboratory variabilities < 2 %, indicating that these methods may not be accurate but are repeatable.

In general, high within-laboratory variabilities can be caused by systematic and/or random errors. The errors most likely to occur when taking moisture measurements result from sample storage and weighing inconsistencies. A sample and crucible can take on moisture after leaving the oven, desiccator, or freeze dryer. It is essential to store the crucibles containing the samples in a desiccator after they are removed from the drying apparatus. It is equally important to start with a crucible that has been adequately dried and stored in a desiccator prior to use. The crucible and sample should be weighed when the sample and crucible are at ambient temperature. Otherwise, buoyancy errors will result in an incorrect final sample mass. If the samples were not all treated identically with respect to pre-drying and/or post-drying storage conditions, the resulting RSD_r would be elevated. It is also important to make sure that the initial wet sample mass and depth in the crucible are similar because the initial conditions impact how moisture is removed from the sample. When an oven method is used, the type of oven will affect the temperature variation, with convection ovens having higher variability than forced air ovens. Temperature differential in vacuum ovens is dictated by the position of the inlet and discharge manifolds and the amount of glass on the oven door. When using an oven drying method, participants should run preliminary studies to determine if crucible

configuration affects the precision of the measurements, as hot spots in an oven can lead to high variability across samples.

The between-laboratory variability (RSD_R) for moisture measurements in this study was 21 %, which was within the range of overall trueness % RSDs reported by the University of Kentucky Hemp Proficiency Testing program measuring 20 study samples (13.6 % to 31.9 %) [11]. While there is no official AOAC requirement for between-laboratory variability of moisture measurements, the draft AOAC SMPR [12] suggested an RSD_R of ≤ 6 %. The SMPR for quantitation of cannabinoids in plant materials of hemp specifies that the RSD_R for cannabinoids in hemp should be ≤ 10 % on a dry weight basis [13], which indicates that the between-laboratory variability of moisture in hemp should not be greater than 10 %. However, the AOAC SMPRs are meant to be applied to the use of a single analytical method, not multiple methods as is the case for this study. For comparison to the two AOAC SMPRs, the RSD_Rs of the individual reported methods were assessed (**Table 3-4**). Interestingly, the between-laboratory variabilities of the individual methods were similar to the overall RSD_R and well above the AOAC recommendations. Environmental conditions among laboratories, variance in oven temperatures, crucible cool-down protocol, and depth of sample in the crucible are the most likely reasons for the high between-laboratory variabilities.

Table 3-4. Average within- and between laboratory variabilities for individual methods used by laboratories in this study.

Analytical Method	n	RSD_R (%)	RSD_r (%)
Thermogravimetric Analysis	28	19.6	4.3
Vacuum Oven	9	22.3	1.1
Forced Air	13	19.6	2.4
Karl Fisher	6	18.8	2.1

4. References

[1] I. O. f. Standardization, "Statistical methods for use in proficiency testing by interlaboratory comparisons (ISO Standard No. 13528:2022)," 2022.

[2] C. R. Beauchamp, J. E. Camara, J. Carney, S. J. Choquette, K. D. Cole, P. C. DeRose, D. L. Duewer, M. S. Epstein, M. C. Kline, K. A. Lippa, E. Lucon, J. Malloy, M. A. Nelson, K. W. Phinney, M. Polakoski, A. Possolo, L. C. Sander, J. E. Schiel, K. E. Sharpless, B. Toman, M. R. Winchester and D. Windover, "Metrological tools for the reference materials and reference instruments of the NIST Material Measurement Laboratory (US Department of Commerce, Washington, DC)," *NIST Special Publication (SP) 260-136,* 2021.

[3] R. C. Team, *_R: A language and environment for statistical computing_,* Vienna: R Foundation for Statistical Computing, 2024.

[4] H. Wickham, *ggplot2: elegant graphics for data analysis,* New York: Springer-Verlag, 2016.

[5] *Agriculture Improvement Act of 2018 (Pub. L. No. 115-334, 2018),* 2018.

[6] J. Y. Ahn, D. Y. Kil, C. Kong and B. G. Kim, "2014," *Asian Australas. J. Anim. Sci.,* vol. 27, no. 11, pp. 1615-1622, 2014.

[7] D. F. Quirino, N. S. A. Lima, M. N. N. Palma, M. O. Franco and E. Detmann, "Evaluation of heating times for loss on drying at 105 C for estimation of laboratory dry matter in animal feeds," *Journal of AOAC International,* vol. 106, no. 2, pp. 261-266, 2023.

[8] R. Samuelsson, J. Burvall and R. Jirjis, "Comparison of different methods for the determination of moisture content in biomass," *Biomass and Bioenergy,* vol. 30, pp. 939-934, 2006.

[9] R. L. Bradley, "Moisture and total solids analysis," in *Food Analysis*, S. S. Nielsen, Ed., West Lafayette, IN: Purdue University, 2010, pp. 85-104.

[10] A. M. B. P. W. Group, *Recommendations and critical factors in determining moisture in animal feeds,* Association of American Feed Control Officials, 2018.

[11] F. a. E. College of Agriculture, "Division of Regulatory Services: Hemp Proficiency Testing Summary Reports," 2018-2024. [Online]. Available: https://www.rs.uky.edu/regulatory/hpt/reports.php. [Accessed 18 October 2023].

[12] "AOAC SMPR 2020.xxx: Determination of moisture in hemp and cannabis plant matter," *Journal of AOAC International,* 2020.

[13] "AOAC SMPR 2019.003: Standard method performance requirements (SMPRs) for quantitation of cannabinoids in plant material of hemp (low THC varieties cannabis sp.)," *Journal of AOAC International,* 2019.