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Charles A. Barber*
Colleen E. Bryan Sallee
Carolyn Q. Burdette
Shaun P. Kotoski*
Melissa M. Phillips
Walter B. Wilson
Laura J. Wood (retired)*
Chemical Sciences Division
Material Measurement Laboratory

**Former NIST employee; all work for this publication was done while at NIST*

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NIST Author ORCID iDs

Charles A. Barber: 0000-0002-4968-7486
Colleen E. Bryan Sallee: 0000-0002-2334-3925
Carolyn Q. Burdette: 0000-0002-0843-9224
Melissa M. Phillips: 0000-0003-0477-7637
Walter B. Wilson: 0000-0003-1763-781X
Laura J. Wood: 0000-0002-4294-7636

Abstract

In 2020, NIST launched Cannabis Laboratory Quality Assurance Program (CannaQAP) to improve the comparability of the analytical measurements of cannabis and cannabis-derived products in forensic and cannabis (hemp and marijuana) testing laboratories. CannaQAP is an interlaboratory study mechanism that is similar to a proficiency testing scheme; however, the focus is towards education without assigning pass/fail grades to the anonymized participants. CannaQAP helps inform NIST about the current measurement capabilities of, and challenges faced by the analytical cannabis community. This in turn assists NIST in the design and characterization of cannabis reference materials (RMs). This study of Exercise 2 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 loss reported by participating laboratories using several different drying methods indicates the need for consistent hemp drying method(s) for accurate and precise measurements.

Keywords

Cannabis; Cannabis Laboratory Quality Assurance Program (CannaQAP); hemp; moisture.

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List of Acronyms

cGMP	current Good Manufacturing Practice
CannaQAP	Cannabis Laboratory Quality Assurance Program
DSQAP	Dietary Supplements Laboratory Quality Assurance Program
HAMQAP	Health Assessment Measurements Quality Assurance Program
LOQ	Limit of Quantification
NIST	National Institute of Standards and Technology
QAP	Quality Assurance Program
QL	Quantification Limit
RM	Reference Material
SD	Standard Deviation
SDPA	Standard Deviation for Proficiency Assessment
TGA	Thermogravimetric Analysis

Introduction

Cannabis Laboratory Quality Assurance Program (CannaQAP) offers the opportunity for laboratories to assess their in-house measurements of cannabinoids, other desirable components (e.g., moisture), and contaminants (e.g., toxic elements) in samples distributed by NIST. Reports and certificates of participation are provided to participants and may be used as part of their laboratory's validation process, to demonstrate compliance with cGMPs, and to potentially fulfill proficiency requirements established by related accreditation bodies. In addition, CannaQAP is designed to support the development and dissemination of analytical methods and reference materials. In the future, results from CannaQAP exercises could be used by NIST to identify problematic matrices and analytes for which consensus-based methods of analysis would benefit the stakeholders in numerous cannabis communities.

NIST has decades of experience in the administration of QAPs, and CannaQAP builds on the approach taken by DSQAP and HAMQAP by emphasizing emerging and challenging measurements in various cannabis and cannabis-derived matrices. NIST QAPs can be viewed as a perpetual interlaboratory study mechanism that is akin to a proficiency testing scheme but without the pass/fail grade. Instead, the goal is centered on improving measurement comparability and/or competence for the participant and NIST results. These improvements focus around identifying biases among the different sample preparation methods, analytical methods, and/or calibration approaches. In areas where few standard methods have been recognized, CannaQAP offers a unique tool for assessment of the quality of measurements and provides feedback about performance that can assist participants in improving laboratory operations.

This report summarizes the results from the second exercise of CannaQAP, specifically the determination of moisture in a hemp plant sample provided by NIST. One hundred thirty-five laboratories responded to the call for participants in the moisture study of the exercise distributed in January 2021. Samples were shipped to participants in April 2021 and results were returned to NIST by May 2021. This report contains the final data and information that was disseminated to the participants in June 2021. The results of the study are summarized below in a series of text, tables, and figures.

Overview of Data Treatment and Representation

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

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 relative to a target around the expected result, if available. All calculations are performed in PROLab Plus (QuoData GmbH, Dresden, Germany).[1] The consensus means and standard deviations are calculated according to the robust Q/Hampel method outlined in ISO 13528:2015, Annex C.[2]

Individualized Data Table

The data in this table is individualized to each participating laboratory and is provided 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). The upper left of the data table includes the randomized laboratory code. An example individualized data table is included in this report using sample NIST data; participating laboratories received uniquely coded individualized data tables in a separate distribution.

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 or matrix. An empty box for standard deviation indicates that the participant reported a single value and therefore that value was not included in the calculation of the consensus data [1]. An example individualized data table is included in this report using NIST data in Section 1 to protect the identity and performance of participants.

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*), consensus standard deviation (s*), and standard deviation for proficiency assessment (SDPA, σ_{PT}^2) determined from the Q/Hampel estimator:

$$Z'_{\text{comm}} = \frac{x_i - x*}{\sqrt{\sigma_{PT}^2 + s^{*2}}}$$

The second Z-score, Z_{NIST}, is calculated with respect to the target value determined at NIST, using x_{NIST} and $2*U_{95}$ (the expanded uncertainty on the certified or reference value, U_{95}, or twice the standard deviation of NIST or other measurements):

$$Z_{\text{NIST}} = \frac{x_i - x_{\text{NIST}}}{2 * U_{95}}$$

or

$$Z_{\text{NIST}} = \frac{x_i - x_{\text{NIST}}}{2 * U_{\text{NIST}}}$$

The significance of the *Z*-score and *Z′*-score is as follows:

[1] Certain commercial equipment, instruments, or materials are identified in this certificate to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

- $|Z| < 2$ 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 more than a single quantitative value for moisture, the mean value determined for moisture, and a robust estimate of the standard deviation of the reported values[1]. Consensus means and standard deviations are calculated using the laboratory means; if a laboratory reported a single value, the reported value is not included in determination of the consensus values[1]. Additional information on calculation of the consensus mean and standard deviation can be found in the previous section.

Section 3 of the data table (*Target*) contains the target value for moisture. When possible, the target value is a certified value, a non-certified value, or a value determined at NIST. In this study, target values for the hemp sample were determined at NIST using a desiccator method summarized in the Study Material Preparation and Characterization Section below. The target value for Hemp Sample 7 represents the mean of ten tested samples dried in a desiccator over fresh magnesium perchlorate ($Mg(ClO_4)_2$) for 21 days. These measurements allowed for NIST to provide either a SD or an expanded uncertainty (U_{95}) to encompass variability due to inhomogeneity between packaged units.

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 for 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. Data highlighted in blue have been identified as outside the consensus tolerance limits and would be estimated to yield $|Z'_{comm}| > 2$.

Figures

Data Summary View (Method Comparison Data Summary View)

In this view, individual laboratory data (diamonds) are plotted with the individual laboratory standard deviation (rectangle). The assigned value, relative SDPA, relative repeatability SD, and range of tolerance for the analyte were calculated according to Q/Hampel are provided in each descriptive caption. The consensus mean for the analyte is the assigned value; the relative SDPA is calculated by dividing the SDPA by the consensus mean times 100; the relative repeatability SD is calculated by dividing the repeatability SD by the consensus mean times 100; and the upper and lower values for the consensus mean's range of tolerance are listed. The blue solid line represents the consensus mean, and the green shaded area represents the 95 % confidence interval for the consensus mean, based on the standard error of the consensus mean. The uncertainty in the consensus mean (u_{mean}) is calculated using the equation below, based on the repeatability standard deviation (s_r), the reproducibility standard deviation (s_R), the number of participants reporting data ($n_{participants}$), and the average number of replicates reported by each participant ($n_{Average\ Number\ of\ Replicates\ per\ Participant}$). The uncertainty about the consensus mean is independent of the range of tolerance.

$$u_{mean} = \sqrt{\frac{s_R^2 - s_r^2}{n_{particpants}} + \frac{s_R^2}{n_{participants} \times n_{Average\ Number\ of\ Replicates\ per\ Participant}}}$$

The red shaded region represents the target zone for "acceptable" performance, which encompasses the NIST target value 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, $|Z' \text{ score}| \leq 2$). If the lower limit is below zero, the lower limit has been set to zero. In this view, the relative locations of individual laboratory data and consensus zones with respect to the target zone can be compared easily. In most cases, the target zone and the consensus zone overlap, which is the expected result. Major program goals include both reducing the size of the consensus zone and centering the consensus zone about the 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 zone. 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 to sample preparation, analysis, or quantitation.

1. Study Material Preparation and Characterization

1.1 NIST Method for Material Characterization for Moisture

1.1.1. Desiccator Drying Method

Ten randomly selected packets of Plant Sample 7 were selected for desiccator drying at NIST. Participants will be able to compare and assess their in-house moisture measurements to the target value determined at NIST. A Mettler AT261 Delta Range analytical balance was used for weighing samples. The balance is serviced and calibrated annually by Mettler. Prior to use, calibration is verified using standard masses ranging from 0.5 g to 20 g that are traceable to the SI through the standard mass set maintained by the Inorganic Measurement Science Group at NIST.

Samples were prepared by taking a 1-cm aliquot (1.2 g) from each packet of Plant Sample 7. The packets were rotated to mix prior to sampling. Samples from each packet were taken and placed in pre-weighed, glass weighing vessels (m_b). The vessels were again weighed with sample (m_w) and placed in a desiccator over fresh magnesium perchlorate ($Mg(ClO_4)_2$). The samples were removed after five days and the weights were recorded (m_d). Samples were placed back in the desiccator and weighed with the weights, (m_d), recorded on the following days: day 7, day 14, day 22, day 28, day 35, and day 41.

1.1.2. Calculations

The percent results were calculated using this equation:

$$\text{moisture} = \frac{(m_w - m_d)}{(m_w - m_b)} \times 100\ \%$$

This approach is based on the assumption that all mass losses were due to loss of moisture alone.

The uncertainty associated with each value for moisture content is calculated from the repeatability of the set of 10 sample means and the uncertainty associated with the use of the analytical balance using this equation:

$$U = k\sqrt{({u_a}^2 + {u_{b1}}^2 + {u_{b2}}^2 + {u_{b3}}^2)}$$

where u_a is the standard deviation of moisture results for 10 samples. The value for each u_b is the standard uncertainty of each weighing (m_b, m_w, and m_d) estimated to be ± 0.01 mg and normalized by dividing by √3 before entry into the uncertainty equation. For each u_b this number is converted to moisture content by division of the mean sample mass value to which it pertains. The expanded uncertainty values, U, are expressed at an approximate confidence level of 95 % by choosing the expansion factor, $k = 2.26$, calculated based on degrees of freedom (df).

1.2. Participant Instructions

1.2.1. Plant Sample 7

Participants were provided with one packet containing approximately 5 g of dried plant material. This plant material was prepared at NIST from commercial hemp biomass. Before use, the contents of the packet should be allowed to equilibrate at room temperature for 1 h and mixed thoroughly prior to subsampling for analysis. The sample size should be appropriate to the moisture method used. NIST has measured the tetrahydrocannabinol (THC) content of this sample to ensure that the total THC mass fraction is less than or equal to 0.3 %.

Participants were instructed that samples should be stored at controlled freezer conditions, ≈ –20 °C, in the original, unopened packet until ready for use.

Participants were instructed to prepare three samples and report three moisture values from the single packet provided in units of mass percent (%).

2. Moisture Determination

2.1. Study Overview

The medicinal and recreational use of cannabis (hemp and marijuana) and cannabis-derived products continues to increase across the United States. Moisture content is an important component of cannabis both from a quality standpoint and from a safety concern. Effective drying practices ensure a high-quality cannabis product. Over-drying damages trichomes and affects yield. Under-drying violates regulations and increases the chances of contamination from mold, fungi, or microorganisms. Moisture can affect the flavor and the potency (THC levels) of cannabis. At the time of this report, some states are requiring moisture testing of cannabis including CA, DC, HI, WA, and AK, so it is important to be able to accurately analyze the moisture content in cannabis plant material.

2.2. Reporting Statistics

2.2.1. Moisture

The enrollment and reporting statistics for moisture are described in the table below.

Analyte	Number of Participants	Percent Reporting Results
Moisture	135	79 %

Most laboratories reported using thermogravimetric analysis (TGA) for determination of moisture in the Plant Sample 7 (see table below). Additional sample analysis details are summarized at the end of the report in the appendix.

Reported Sample Analysis Method	Percent Reporting Method
Desiccator	2 %
Freezer-Dryer	1 %
Karl Fisher Titration	3 %
Oven Drying, Forced Air Oven	19 %
Oven Drying, Vacuum Oven	11 %
Thermogravimetric Analysis	27 %
Weight Loss after Ignition in Muffle Furnace	2 %
Other	35 %

The between-laboratory variability for the determination of moisture in the hemp sample was 2.5 %. The range of the variability of individual laboratory means for determination of moisture in the hemp sample was between 0.1 % and 39 %.

2.3. Study Results

2.3.1. Moisture

- The mass fraction (%) of moisture in the hemp plant sample was determined by NIST using a desiccator method as described in Section 1 and summarized in **Table 2-1** and **Table 2-2**.
- Figures are provided summarizing the reported results. Data from participants submitting only one measurement were included in **Table 2-2** but were not included in the calculation of consensus statistics.
- The consensus range for moisture in Plant Sample 7 was completely above the NIST range of tolerance.
- Laboratories reporting outlying results with respect to the NIST range of tolerance and the consensus range of tolerance ($|Z'_{\mathrm{comm}}| > 2$) are summarized in the table below.

Samples	Number (%) of Laboratory Means Outside NIST Range of Tolerance	Number (%) of Laboratory Means Outside Consensus Range of Tolerance
Plant Sample 7	85 (86 %)	6 (6 %)

2.4. Study Discussion and Technical Recommendations

The following recommendations are based on results obtained from the participants in this study.

- Sixteen laboratories out of 106 laboratories that submitted results reported values which overlapped the NIST range of tolerance, **Figure 2-1**. Their drying methods included desiccator drying, vacuum oven drying, forced air oven drying, and TGA indicating that no one method was better at drying the hemp plant sample then another.
- The three methods reported to be used most, forced air oven drying, vacuum oven drying, and TGA, can be seen in **Figure 2-2** through **Figure 2-4** respectively.
  - In **Figure 2-2**, the forced air oven drying moisture loss ranged from 3.2 % to 21 %. This wide range would vary depending on time samples are held in the oven and temperature of the oven.
  - The same is true of vacuum oven drying, **Figure 2-3**. The moisture loss for this technique ranged from 4.4 % to 12 %. Often vacuum oven drying is performed without heat and time in the oven should be specified.
  - The TGA method, shown in **Figure 2-4**, has moisture losses ranging from 0.9 % to 11 %. With TGA it is important to observe the point where the moisture loss becomes constant and does not go beyond this point.
- From the different methods used, and the wide range of moisture loss, the need for a consistent drying method or methods is needed. This should include established temperatures and times held in ovens, etc. These methods should also establish that only water and residual solvents are being lost when determining moisture and other samples contents such as carbon are not being lost.

Table 2-1. Individualized data summary table (NIST) for moisture in Plant Sample 7.

National Institute of Standards and Technology

CannaQAP Exercise 2 - Spring 2021

	Lab Code:	**NIST**	**1. Your Results**				**2. Community Results**			**3. Target**	
Analyte	Sample	Units	x_i	s_i	Z'_{comm}	Z_{NIST}	N	x*	s*	x_{NIST}	U
Moisture	Plant Sample 7	%	5.20	0.20			99	7.06	0.18	5.20	0.20

x_i Mean of reported values
s_i Standard deviation of reported values
Z'_{comm} Z'-score with respect to community consensus
Z_{NIST} Z-score with respect to NIST value

N Number of quantitative values reported
x* Robust mean of reported values
s* Robust standard deviation

x_{NIST} NIST-assessed value
U expanded uncertainty about the NIST-assessed value

Table 2-2. Data summary table for moisture in Plant Sample 7.

Data highlighted in blue have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| > 2$. Note: This table spans four pages; the NIST target value and community results are included on all four pages for convenience.

		Moisture				
		Plant Sample 7 (%)				
	Lab	A	B	C	Avg	SD
Individual Results	Target				5.20	0.20
	B001	8.4	8.6	8.5	8.50	0.10
	B003	7.31	7.52	7.31	7.38	0.12
	B004	7.8	7.3	8	7.70	0.36
	B006	11.78	9.91	9.43	10.37	1.24
	B007	6.63	6.27	6.59	6.50	0.20
	B008	0.14	0.19	0.24	0.19	0.05
	B009					
	B012	5.78	5.17	5.22	5.39	0.34
	B013					
	B014	5.85	6.36	6.6	6.27	0.38
	B016	8.14	8.76	8.56	8.49	0.32
	B018	5.17	5.53	5.15	5.28	0.21
	B022	7.8	6.65	7.2	7.22	0.58
	B023	4.12	4.27	4.14	4.18	0.08
	B024	6.99	6.67	7.2	6.95	0.27
	B027	5.31	5.84	6.39	5.85	0.54
	B028					
	B029	5.41	5.49	5.96	5.62	0.30
	B030	7.54	7.56	7.58	7.56	0.02
	B031					
	B033	7.4	8.9	8.2	8.17	0.75
	B035	7.9	7.8	7.9	7.87	0.06
	B037					
	B038	7.55			7.55	
	B041	5.344	5.065	5.018	5.14	0.18
	B043	5.575			5.58	
	B044	4.99	5.13	5.1	5.07	0.07
	B047	7.02	6.69	6.08	6.60	0.48
	B048					
	B052	8.75	8.54	8.6	8.63	0.11
Community Results		Consensus Mean			7.06	
		Consensus Standard Deviation			0.18	
		Maximum			20.67	
		Minimum			0.19	
		N			99	

	Lab	Moisture Plant Sample 7 (%) A	B	C	Avg	SD
Individual Results	Target				5.20	0.20
	B053	9.36	10.07	9.83	9.75	0.36
	B054	5.35	5.41	6.15	5.64	0.45
	B055	6.14446	6.05049	5.99334	6.06	0.08
	B056	6.61	5.9	6.4	6.30	0.36
	B058	7.73	7.64	7.46	7.61	0.14
	B060	7.92	7.93	7.92	7.92	0.01
	B061	7.05	7.26	7.55	7.29	0.25
	B062	6.15	6.1		6.13	0.04
	B063					
	B064	5			5.00	
	B065	8.76	8.28	7.42	8.15	0.68
	B066	8.74	6.84	7.61	7.73	0.96
	B068					
	B069	7.09	6.65	6.9	6.88	0.22
	B070	6.92	8.75	7.39	7.69	0.95
	B072	3.23	3.15	3.18	3.19	0.04
	B073	7.4			7.40	
	B077	6.71	7.06	6.65	6.81	0.22
	B078					
	B079					
	B082	6.92	7.28	7.75	7.32	0.42
	B084	7.899	7.317	6.084	7.10	0.93
	B086	8.788	8.728	8.713	8.74	0.04
	B088	8.51	8.52	9.98	9.00	0.85
	B089	9.6885	9.6185		9.65	0.05
	B090	3.87	3.84	3.73	3.81	0.07
	B094	8.87	8.84	8.73	8.81	0.07
	B095	9.5	9.9	9.9	9.77	0.23
	B096	6.41	6.43	6.25	6.36	0.10
	B097	4.93	4.82	4.9	4.88	0.06
	B098	0.9	0.89	0.92	0.90	0.02
	B099					
	B100	6.8			6.80	
	B102	8.54	8.32	8.29	8.38	0.14
	B106	8.2	8.36	8.46	8.34	0.13
	B107	8.75	8.89	8.41	8.68	0.25
Community Results		Consensus Mean			7.06	
		Consensus Standard Deviation			0.18	
		Maximum			20.67	
		Minimum			0.19	
		N			99	

	Lab	Moisture Plant Sample 7 (%) A	B	C	Avg	SD
Individual Results	Target				5.20	0.20
	B108					
	B109	8	7.84	6.5	7.45	0.82
	B110	10.4	10.2	9.99	10.20	0.21
	B111					
	B113	6.47	8.29	8.09	7.62	1.00
	B115					
	B116	12.98	11.43	11.18	11.86	0.98
	B117	6.34	6.397	5.986	6.24	0.22
	B120					
	B122					
	B125	4.6	4.65	4.64	4.63	0.03
	B126	8.69	8.39	8.54	8.54	0.15
	B127	8.538	8.539	8.409	8.50	0.07
	B129					
	B131	4.3	4.75	4.64	4.56	0.23
	B132					
	B136	8.911	6.92	7.067	7.63	1.11
	B137	7.71	7.38	7.8	7.63	0.22
	B142	4.12	3.94	3.8	3.95	0.16
	B144	6	4.37	3.93	4.77	1.09
	B146					
	B147	4.89	5.28	4.8	4.99	0.26
	B148					
	B149	7.82	7.2	6.48	7.17	0.67
	B150	4.69	4.34	4.81	4.61	0.24
	B152					
	B153	6.97	7.27	8.03	7.42	0.55
	B158					
	B159	8.9	9	9.1	9.00	0.10
	B160	6.16	6.26	6.38	6.27	0.11
	B161	4.51	4.45	4.32	4.43	0.10
	B164	8.17			8.17	
	B168	8.6	7.14	7.14	7.63	0.84
	B172					
Community Results		Consensus Mean			7.06	
		Consensus Standard Deviation			0.18	
		Maximum			20.67	
		Minimum			0.19	
		N			99	

	Lab	Moisture Plant Sample 7 (%) A	B	C	Avg	SD
Individual Results	Target				5.20	0.20
	B174	7.53	8.85	8.33	8.24	0.66
	B176	5.85	5.89	5.86	5.87	0.02
	B178	8.22	9.2	8.82	8.75	0.49
	B181	7.64	6.91	7.47	7.34	0.38
	B182	7.5	7.73	7.73	7.65	0.13
	B183	8.31	8.31	8.21	8.28	0.06
	B184	10.1	7.34	10.1	9.18	1.59
	B186	0.99	0.43	0.88	0.77	0.30
	B188					
	B189	8.32	8.64	8.12	8.36	0.26
	B190	4.69	4.8	4.72	4.74	0.06
	B192	7.54	7.18	7.09	7.27	0.24
	B193	6.15	5.95	5.9	6.00	0.13
	B195	9.6	9.4	9	9.33	0.31
	B200	6.87	6.73	6.72	6.77	0.08
	B201	4.65	4.76	5.13	4.85	0.25
	B202	7.76	7.91	7.67	7.78	0.12
	B204	6.05	6.05	6.16	6.09	0.06
	B205	20	22	20	20.67	1.15
	B206	6.15	6.14	7.27	6.52	0.65
	B208					
	B210					
	B212	8.76	8.51	7.77	8.35	0.51
	B213					
	B215	4.37			4.37	
	B216	8.1	7.9	8.4	8.13	0.25
	B217	7.2	7.16	6.19	6.85	0.57
	B219	8.44	8.97	8.62	8.68	0.27
	B220					
	B221	8.29	8.39	8.34	8.34	0.05
	B222	7.1	7.05	7.08	7.08	0.03
	B223	12.76	10.9	8.66	10.77	2.05
	B224	5.65	5.8	5.85	5.77	0.10
	B228	8.38	8.2	8.25	8.28	0.09
	B230					
Community Results		Consensus Mean			7.06	
		Consensus Standard Deviation			0.18	
		Maximum			20.67	
		Minimum			0.19	
		N			99	

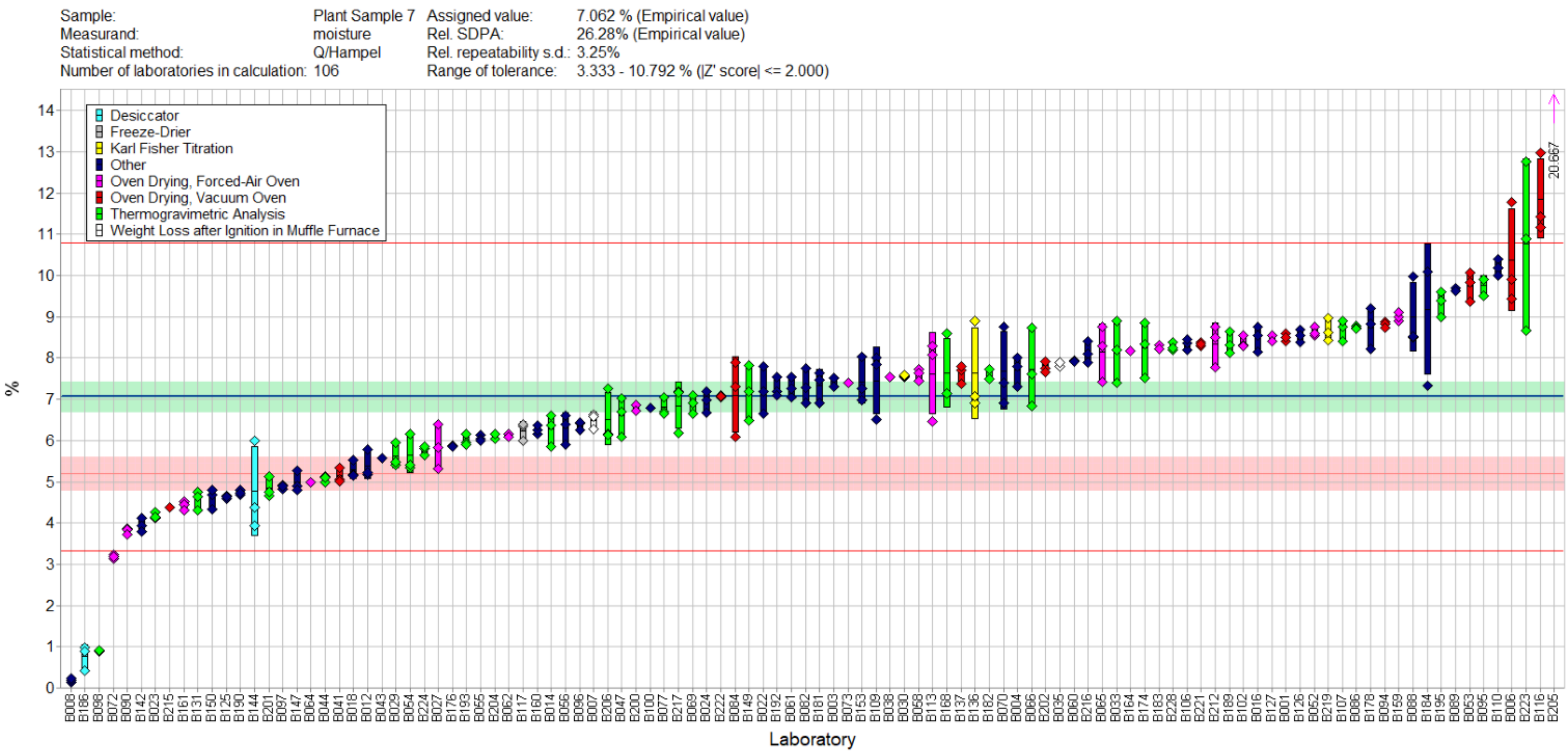


Fig. 2-1. Moisture in Plant Sample 7 (data summary view – analysis method).In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analysis method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$.

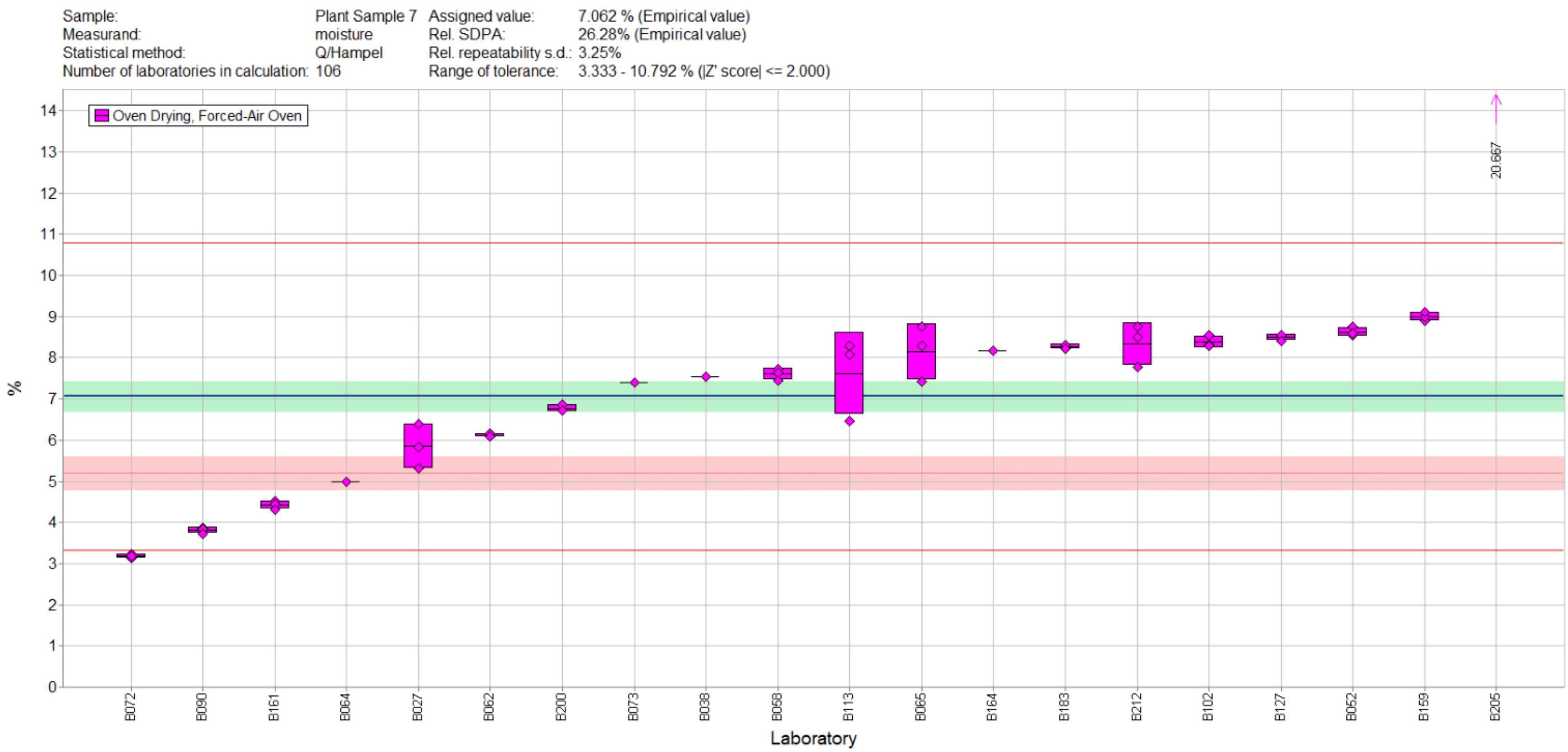


Fig. 2-2. Moisture in Plant Sample 7 (data summary view – analysis method, forced air oven drying).In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

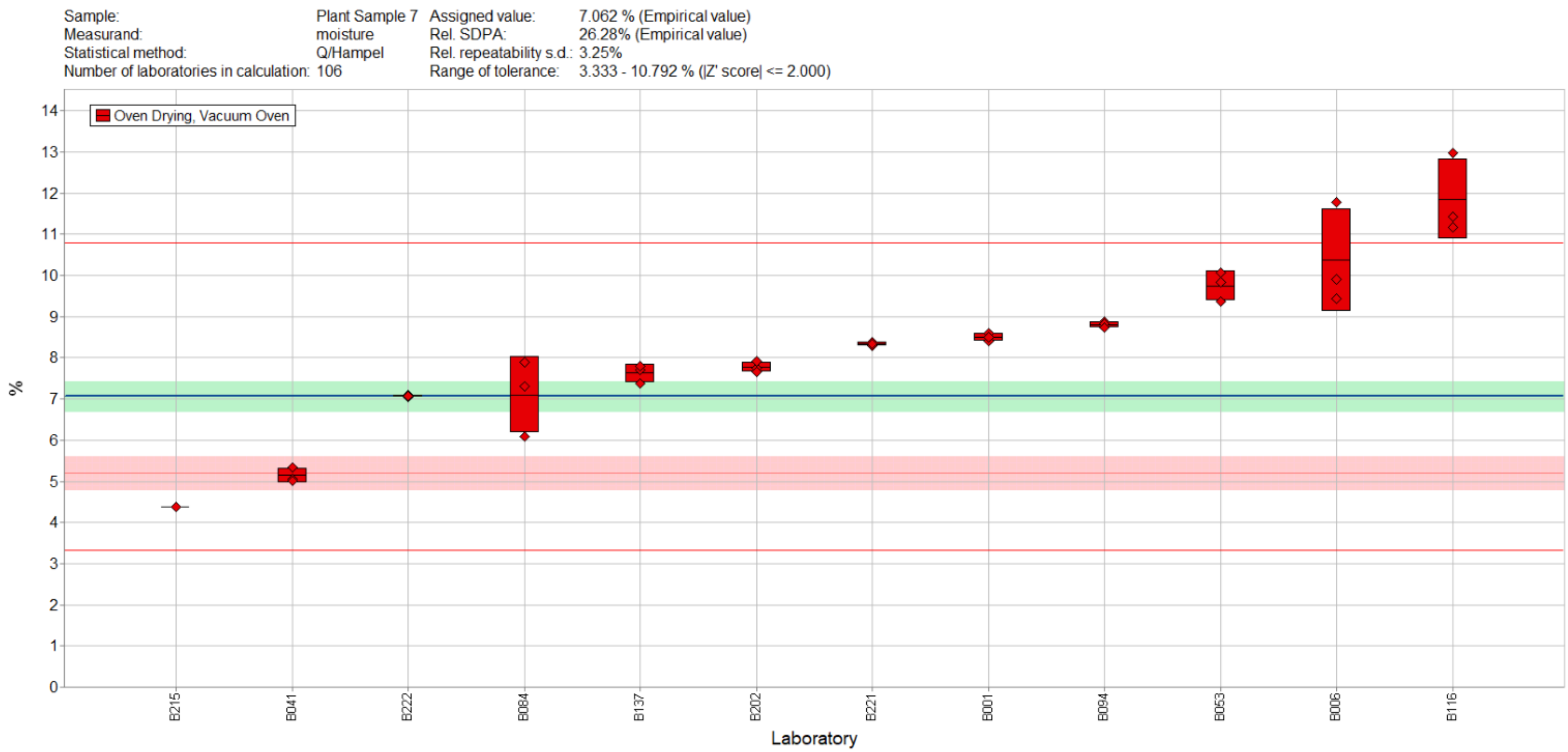


Fig. 2-3. Moisture in Plant Sample 7 (data summary view – analytical method, vacuum oven drying). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

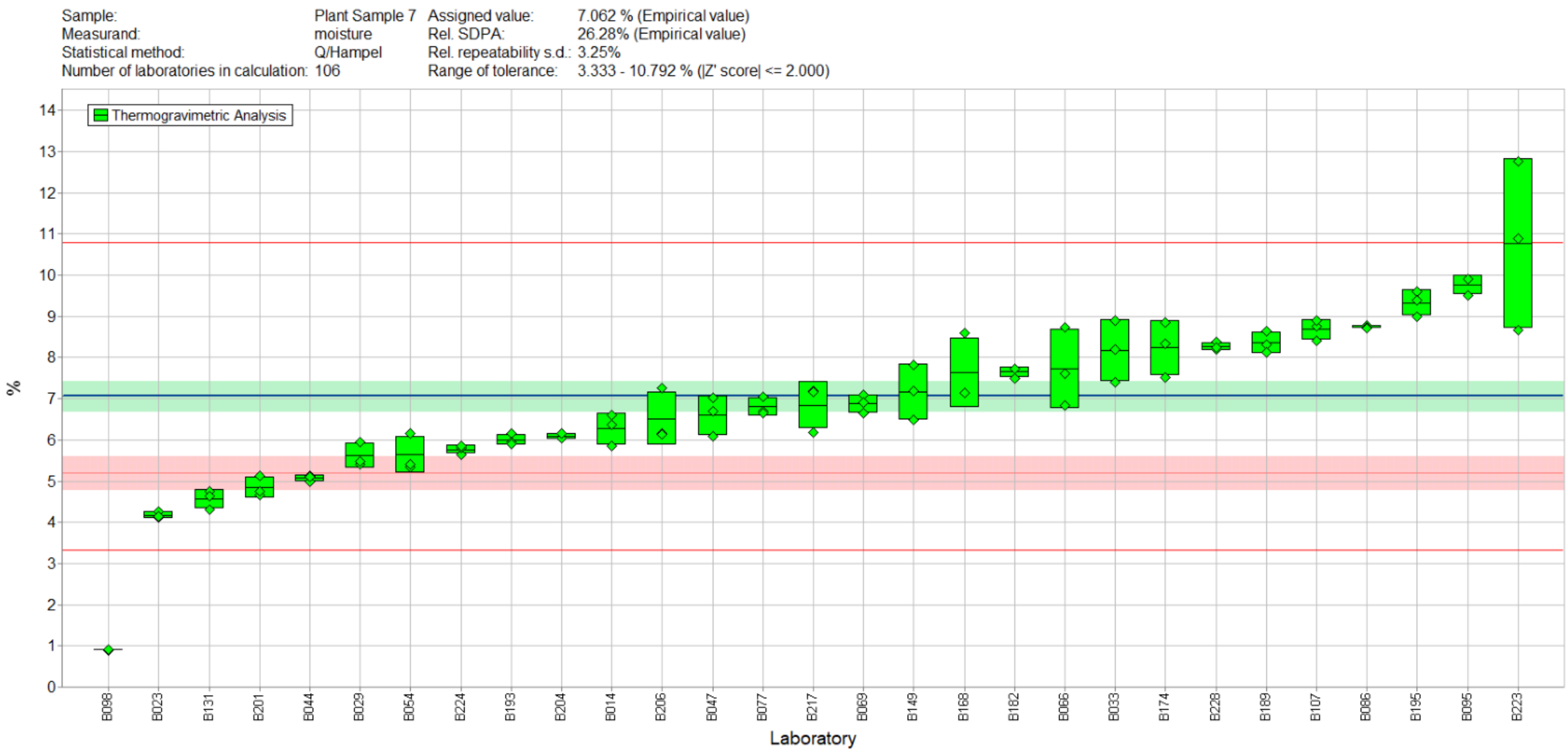


Fig. 2-4. Moisture in Plant Sample 7 (data summary view – analytical method, thermogravimetric analysis). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

References

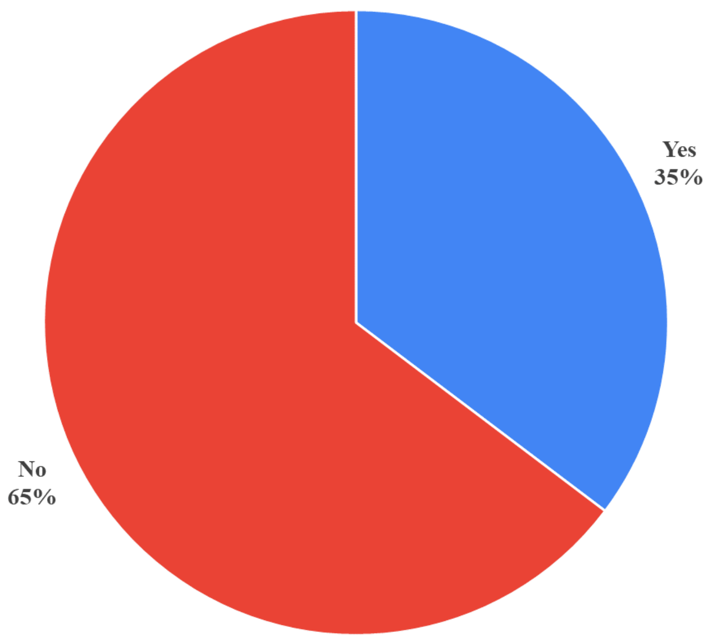
[1] ISO (2022)– *ISO 13528:2022 Statistical methods for use in proficiency testing by interlaboratory comparison*).

Appendix A. Method Questionnaire Responses

Seventeen laboratories completed the method questionnaire out of 106 labs that participated, including NIST.

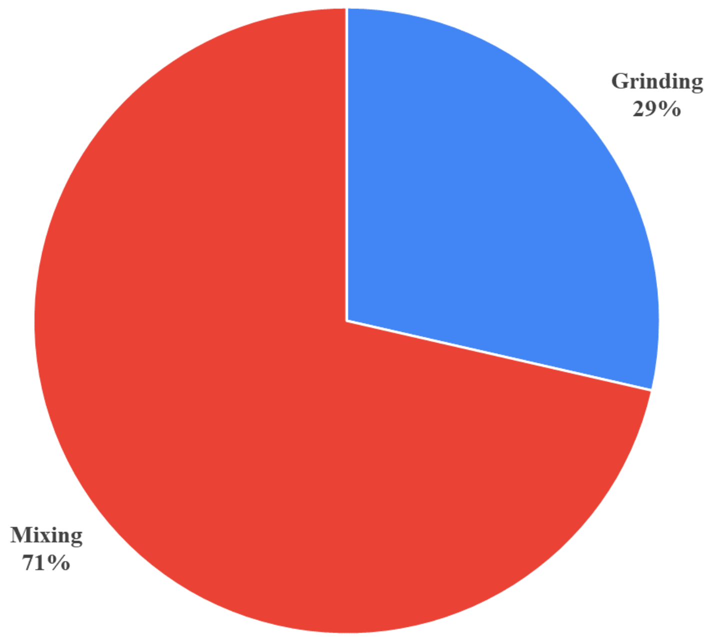
Laboratory Code		
B007	B056	B168
B008	B072	B219
B022	B073	B228
B024	B117	
B033	B126	
B053	B144	
B054	B164	

Homogenization



Response	Laboratory									
Yes	B033	B054	B056	B126	B144	B228				
No	B007	B008	B022	B024	B053	B072	B073	B117	B164	B168
	B219									

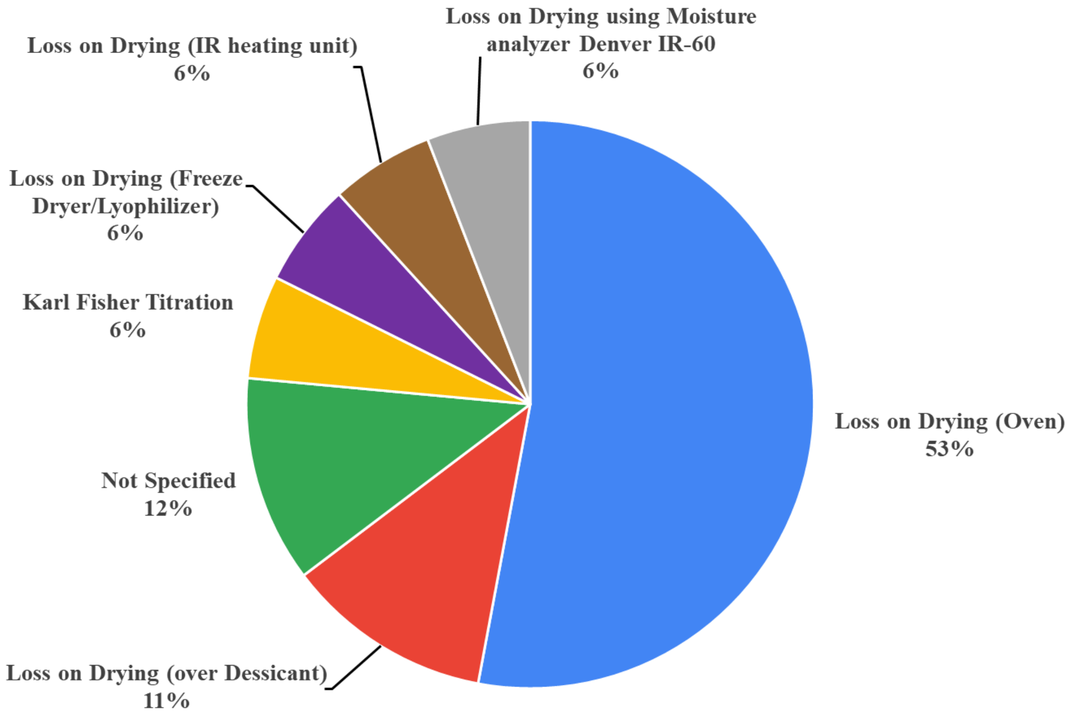
Homogenization Method



Homogenization Method	Laboratory				
Mixing	B054	B126	B144	B228	B219*
Grinding	B033	B056			

*This laboratory reported that they did not homogenize the material, but reported mixing the samples prior to a removal of a test portion for analysis

Analytical Method



Analytical Method	Laboratory								
Loss on Drying (Oven)	B007	B008	B024	B053	B054	B072	B073	B126	B164
Loss on Drying (over Desiccant)	B022	B144							
Karl Fisher Titration	B219								
Loss on Drying (Freeze Dryer/Lyophilizer)	B117								
Loss on Drying (IR heating unit)	B168								
Loss on Drying using Moisture analyzer Denver IR-60	B056								
Not Specified	B033	B228							