

NIST Internal Report NIST IR 8452

Cannabis Laboratory Quality Assurance Program: Exercise 2 Toxic Elements Final Report

Charles A. Barber Colleen E. Bryan Sallee Carolyn Q. Burdette Shaun P. Kotoski Melissa M. Phillips Walter B. Wilson Laura J. Wood

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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 Materials 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 a 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 CannaQAP Exercise 2 focused on the determination of toxic elements in two hemp materials and a control material provided by NIST. Arsenic, cadmium, lead, mercury, beryllium, cobalt, chromium, manganese, molybdenum, nickel, selenium, uranium, and vanadium were the toxic elements chosen based on interest expressed by the cannabis community for safety and regulations. This report provides a detailed description of the results of this study.

Keywords

Arsenic; cadmium; cannabis; Cannabis Laboratory Quality Assurance Program (CannaQAP); hemp; lead; mercury; toxic elements.

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List of Acronyms

AAS	Atomic Absorption Spectroscopy		
cGMP	current Good Manufacturing Practice		
COA	Certificate of Analysis		
CRM	Certified Reference Material		
CannaQAP	Cannabis Quality Assurance Program		
CV-AAS	Cold Vapor Atomic Absorption Spectroscopy		
ID-CV-ICP-MS	Isotope Dilution Cold Vapor Inductively Coupled Plasma Mass		
	Spectrometry		
DC AAS	Direct Combustion Atomic Absorption Spectrometry		
DSQAP	Dietary Supplements Laboratory Quality Assurance Program		
HAMQAP	Health Assessment Measurements Quality Assurance Program		
ICP-MS	Inductively Coupled Plasma Mass Spectrometry		
ICP-OES	Inductively Coupled Plasma Optical Emission Spectrometry		
ID ICP-MS	Isotope Dilution Inductively Coupled Plasma Mass Spectrometry		
INAA	Instrumental Neutron Activation Analysis		
IS	Internal Standard		
LOQ	Limit of Quantification		
NAA	Neutron Activation Analysis		
NIST	National Institute of Standards and Technology		
NRC	National Research Council		
QAP	Quality Assurance Program		
QL	Quantification Limit		
QQQ-ICP-MS	Triple Quadrupole Inductively Coupled Plasma Mass Spectrometry		
RM	Reference Material		
RNAA	Radiochemical Neutron Activation Analysis		
RSD	Relative Standard Deviation		
SD	Standard Deviation		
SDPA	Standard Deviation for Proficiency Assessment		
SPE	Solid Phase Extraction		
SRM	Standard Reference Material		

Introduction

NIST 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 toxic elements in two hemp samples and a control material provided by NIST. One hundred twenty-five laboratories requested samples for the toxic elements study of the exercise following the call for participants in January 2021. Samples were shipped to participants in April 2021 and results were submitted to NIST by May 2021 from 93 laboratories. 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 for the 13 toxic elements.

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 these 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 13528:2022, Annex C [1].

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, reference, or estimated values, when available). The upper left of the data table includes the randomized laboratory code. Example individualized data tables are 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. An empty box for standard deviation indicates that the participant reported a single value or a value below the LOQ and therefore that value was not included in the calculation of the consensus data [1]. Example individualized data tables are 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:

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

The second Z-score, Z_{NIST} , is calculated with respect to the target value (NIST certified, non-certified, reference, or estimated value) when available, 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):

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

or

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

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 each analyte, the mean value determined from reported values for each analyte, 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 values for each analyte, when available. When possible, the target value is a certified, non-certified, or reference value, or a value determined at NIST. In this study, target values for the hemp samples were determined at NIST through a validated ICP-MS, ICP-OES, DC AAS, or QQQ-ICP-MS method summarized in the Study Material Preparation and Characterization Section below. The target values for Hemp Sample 1 and Plant Sample 4 represent the mean of at least three tested samples with single preparations from three packages. These measurements allowed for NIST to provide either a standard deviation (SD) or an expanded uncertainty (U_{95}) to encompass variability due to inhomogeneity within and between packaged units. The target values for the control material, SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*), are from the Certificate of Analysis (COA) [2], adjusted for moisture and reported as an as-received value.

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 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 $|Z'_{comm}| > 2$.

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). Numerical values for the assigned value (consensus mean), relative SDPA, relative repeatability SD, and range of tolerance for the analyte were calculated according to Q/Hampel are provided in each descriptive caption [1]. Laboratories reporting values below LOQ are shown in this view as downward triangles beginning at the LOQ, reported as QL on the figures.

Laboratories reporting values below LOQ can still align with the target value when the target value is also below the laboratory LOQ. 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 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 $(n_{particpants})$, 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.

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

The red shaded region represents the target zone that 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 the target zone |Z'| 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 consensus zone overlaps with the target zone. However, 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 deviate from the target value. 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.

Sample/Sample Comparison View

In this view, the individual laboratory results for one sample (e.g., NIST SRM or RM with a certified, non-certified, or NIST-determined value; a less challenging matrix) are compared to the results for another sample (e.g., NIST SRM or RM with a more challenging matrix; a commercial sample). The solid red box represents the target zone for the first sample (x-axis) and the second sample (y-axis), if available. The dotted blue box represents the consensus zone for the first sample (x-axis) and the second sample (y-axis). The axes of this graph are centered about the consensus mean values for each sample or control, to a limit of twice the range of tolerance (values that result in the target zone |Z' score $| \le 2$). Depending on the variability in the data, the axes may be scaled proportionally to better display the individual data points for each laboratory. These views emphasize trends in the data that may indicate potential calibration issues or method biases. One program goal is to identify such calibration or method biases and assist participants in improving analytical measurement capabilities. In some cases, when two equally challenging materials are provided, the same view (sample/sample comparison) can be helpful in identifying commonalities or differences in the analysis of the two materials.

1. Study Material Preparation and Characterization

1.1. Study Materials Preparation

NRC HEMP-1 (Plant Sample 1)

NRC HEMP-1 was prepared by NRC Canada as their first Hemp Certified Reference Material (CRM) released in November 2021 [3]. NRC HEMP-1 was originally packaged into amber bottles (≈ 15 g) and stored at -20 °C. Upon arrival at NIST, 108 amber bottles were stored at -80 °C until sample packaging for this study. The material was removed from original packaging and mixed for 15 min. During sample packaging, a portion of the bulk material was removed and packaged in small quantities (≈ 3 g) into plastic bags which were heat sealed, placed in mylar bags with desiccant silica pouches, and stored at -20 °C until shipment to participating laboratories.

Plant Sample 4

Plant Sample 4 was prepared at NIST through the grinding of a bulk hemp plant material freshly harvested in the United States, which included the plant buds, leaves, and stems. The ground material was sieved to ensure a particle size between 250 μ m to 710 μ m and mixed for 30 min. The bulk material was immediately stored in the dark at -80 °C. During sample packaging, a portion of the bulk material was removed and packaged in small quantities (\approx 3 g) into plastic bags which were heat sealed, placed in mylar bags with desiccant silica pouches and stored at -20 °C until shipment to participating laboratories.

SRM 1575a Trace Elements in Pine Needles (Pinus taeda)

SRM 1575a was prepared by NIST primarily for use in the evaluation of techniques employed in the analysis of pine needles and materials of a similar matrix [2]. A unit of SRM 1575a consists of approximately 50 g of dried pine needles previously jet-milled, radiation sterilized, blended, and stored at room temperature. In this study, the material was removed from 14 units and mixed for 15 min. During sample packaging, a portion of the bulk material was removed and packaged in small quantities (\approx 3 g) into plastic bags which were heat sealed, placed in mylar bags with desiccant silica pouches and stored at -20 °C until shipment to participating laboratories.

Certified values for Cd and Hg in SRM 1575a were assigned using CV ID-ICP-MS, ICP-MS, and RNAA. Reference values for As, Co, Pb, Mn, Ni, and Se in SRM 1575a were assigned using INAA and ICP-MS. An information value for Cr in SRM 1575a was provided using INAA ICP-MS, and ICP-OES. Values were not assigned in SRM 1575a for Be, Mo, or U in the COA.

1.2. NIST Methods for Material Characterization for Toxic Elements

ICP-MS Analysis of As, Cd, Co, Cr, Mo, Ni, Pb, Se, and U

Three packets each of NRC HEMP-1 (Plant Sample 1) and Plant Sample 4, and two bottles of the control material, SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*), were characterized for As, Cd, Co, Cr, Mo, Ni, Pb, Se, and U content by ICP-MS at NIST.

Sample Preparation

Nominal 1 g aliquots were taken from each packet of NRC HEMP-1 (Plant Sample 1) and Plant Sample 4, and single or duplicate 1 g aliquots were taken from each bottle of SRM 1575a. Samples were placed into Teflon microwave vessels along with 8 mL of Optima grade HNO₃, 2 mL of

Optima grade HF, and internal standard solutions of Sb (8 ng/g) and Rh (1 ng/g). The samples were digested using a microwave sample preparation system (CEM MARSXpress, Matthews, NC, USA) at 800 W (100 % power) for 15 min ramp time at 195 °C (20 min hold time) followed by 1600 W (85 % power) for 20 min ramp time at 205 °C (15 min hold time). Samples were then transferred to polyethylene bottles and diluted to 60 g using 18 M Ω ·cm water. Samples were diluted so that As, Cd, Co, Cr, Mo, Ni, Pb, Se, and U were present at approximate mass fractions of 0.001 mg/kg to 0.004 mg/kg for analysis by ICP-MS.

<u>Analysis</u>

Digested samples were analyzed using an Agilent 7500cs ICP-MS equipped with a Peltier-cooled, inert sample introduction system. Two bottles of an additional control material, SRM 1573a Tomato Leaves, were also analyzed for all elements of interest and as a control material for Mo and U, which do not have mass fraction values assigned in SRM 1575a. The analytes in the prepared solutions were measured according to the parameters below using H₂ as a collision gas to minimize polyatomic interferences for As, Cd, Cr, Mo, Ni, Pb, Se, and U and using He as a collision gas to minimize polyatomic interferences for Co.

	ICP-MS Pa	arameter	<u>Setting</u>	
	Argon flow		15 L/min	
	Auxiliary 1	flow	0.8 L/min	
	Nebulizer	flow	1 L/min	
	Radiofrequ	uency (RF) power	1500 W	
	Mass	Internal	Integration	Read
Element	<u>(amu)</u>	<u>standard (mass)</u>	time/point (s)	<u>time/mass (s)</u>
As	75	Rh (103)	0.1	3
Cd	114	Sb (123)	0.1	3
Co	59	Sb (123)	0.1	3
Cr	52	Rh (103)	0.1	3
Mo	95	Rh (103)	0.1	3
Ni	60	Rh (103)	0.1	3
Pb	207	Rh (103)	0.1	3
Se	78	Sb (123)	0.1	3
U	238	Sb (123)	0.1	3

Quantitation

Analyte mass fractions were quantified by the method of standard additions. To increase the precision of the instrumental measurements, Sb and Rh were added as internal standards. Ten instrumental measurements were averaged for each sample aliquot and each spiked aliquot. All results have been corrected for the mean blank values from their corresponding runs by subtracting the mean total micrograms of a given analyte found in the blanks from the total micrograms of that analyte found in each individual sample.

ICP-OES Analysis of Mn

Three packets each of NRC HEMP-1 (Plant Sample 1) and Plant Sample 4, and two bottles of the control material, SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*), were characterized for Mn content by ICP-OES at NIST.

Sample Preparation

Nominal 1 g aliquots were taken from each packet of NRC HEMP-1 (Plant Sample 1) and Plant Sample 4, and single or duplicate 1 g aliquots were taken from each bottle of SRM 1575a. Samples were placed into Teflon microwave vessels along with 8 mL of Optima grade HNO₃ and 2 mL of Optima grade HF. The samples were digested using a microwave sample preparation system (CEM MARSXpress, Matthews, NC, USA) at 800 W (100 % power) for 15 min ramp time at 195 °C (20 min hold time) followed by 1600 W (85 % power) for 20 min ramp time at 205 °C (15 min hold time). Samples were then transferred to polyethylene bottles and diluted to 60 g using 18 MΩ·cm water. Samples were diluted so that Mn was present at an approximate mass fraction of 0.6 mg/kg for analysis by ICP-OES.

<u>Analysis</u>

Digested samples were analyzed using a Perkin-Elmer Optima 8300 Dual View ICP-OES according to the parameters in table below.

ICP-OES Parameter	Setting
Argon flow	12 L/min
Auxiliary flow	0.2 L/min
Nebulizer flow	0.7 L/min
Radiofrequency (RF) power	1500 W

	Wavelength		Integration	
Element	<u>(nm)</u>	Plasma View	<u>time (s)</u>	Read time (s)
Mn	257.610	Axial	0.10	1.000

Quantitation

Mn mass fractions were quantified by using a four-point calibration curve with values ranging from 0.3 mg/kg to 1.2 mg/kg. Four instrumental measurements were averaged for each sample aliquot. All results for Mn have been corrected for the mean blank values from their corresponding runs by subtracting the mean total micrograms of Mn found in the blanks from the total micrograms of Mn found in each individual sample.

QQQ-ICP-MS Analysis of Be and V

Three packets each of NRC HEMP-1 (Plant Sample 1) and Plant Sample 4 were characterized for Be and V content by QQQ-ICP-MS at NIST. SRM 1547 Peach Leaves was used as a control material for V and Be. The COA for SRM 1547 includes a certified value for V, and the target value for Be was based on previous in-house analysis of this material.

Sample Preparation

Nominal 0.5 g aliquots were taken from each packet of NRC HEMP-1 (Plant Sample 1) and Plant Sample 4, and four 0.5 g aliquots were taken from one bottle of SRM 1547. Samples were placed into quartz microwave vessels. Five milliliters of Optima grade HNO₃ and 1 mL Optima grade HCl were added to each vessel along with an internal standards solution of Sc and Y (each approximately 0.88 mg/kg). The samples were digested using an Anton Paar Multiwave 3000 microwave sample preparation system (Ashland, VA) at 600 W, 10 min ramp time and 15 min hold time followed by 1400 W, 10 min ramp time and 20 min hold time. Samples were then transferred to polypropylene centrifuge tubes and diluted to 50 g using 18 M Ω ·cm water. Half of

each sample solution was then transferred into another centrifuge tube; one tube was spiked with Be and V solution; and both tubes were diluted back to 50 g with 18 M Ω ·cm water. Samples were diluted so that Be and V were present at appropriate mass fractions for analysis by QQQ-ICP-MS.

<u>Analysis</u>

Digested samples were analyzed using an Agilent 8800 QQQ-ICP-MS system (Agilent, Santa Clara, CA) according to the parameters below. The analytes in the prepared solutions were monitored in both the no gas and He gas mode. No gas mode was utilized for the analysis of Be with additional forward Ar gas added (0.2 L/min) and the He collision mode (4.8 mL/min) was utilized for measuring V.

	Parameter	Se	tting
	Argon carrier	gas flow 1.06	L/min
	Nebulizer pum	np 0.1	l rps
	Radiofrequenc	xy (RF) power 15.	50 W
	Mass	Internal standard	Integration time/mass
Element	<u>(amu)</u>	<u>(mass)</u>	<u>(s)</u>
Be	9	Sc (45)	0.1
V	51	Y (89)	0.1

Quantitation

Analyte mass fractions were quantified by the method of single-point standard additions with use of internal standards (Sc and Y). Single-point standard additions methods mitigate matrix effects by splitting a single sample and spiking one of the sample splits with the elements being measured using NIST SRM 3100 series single-element standard solutions to prepare the spikes. Procedural blanks have been analyzed for Be and V concurrently with samples. The mass fractions of the analytes in hemp and control material samples were procedural blank corrected by subtracting the mean of the procedural blank measurements (μ g/kg).

DC AAS Analysis of Hg

Four packets each of NRC HEMP-1 (Plant Sample 1) and Plant Sample 4 were characterized for Hg content by DC AAS at NIST. SRM 1547 Peach Leaves was used as a control material for Hg, as the COA for SRM 1547 includes a certified value for Hg.

Sample Preparation

Nominal 0.1 g aliquots were taken from each packet of NRC HEMP-1 (Plant Sample 1) and Plant Sample 4, and six 0.1 g aliquots were taken from one bottle of SRM 1547. Samples were placed into nickel weigh boats. Procedural blanks and control material samples were bracketed between hemp samples to verify instrument calibration and monitor instrumental drift.

<u>Analysis</u>

Samples were analyzed using a direct Hg analyzer DMA-80 (Milestone Scientific, Shelton, CT) according to the parameters below.

	Method Parameters	
Ramp Time (s)	Temperature (°C)	Hold Time (s)
90	200	30
90	650	180
90	200	30
60	300	60
60	450	30
60	650	240
	<u>Ramp Time (s)</u> 90 90 90 60 60 60 60	Ramp Time (s) Method Parameters 90 200 90 650 90 200 90 300 60 450 60 650

Quantitation

Analyte mass fractions were quantified by external calibration using gravimetrically aliquoted masses of aqueous dilutions of SRM 3133 Mercury Standard Solution, ranging from between 0.0206 g and 0.9793 g, to prepare calibration curves. Procedural blanks were analyzed for Hg concurrently with samples. The mass fractions of the analytes in hemp and control material samples were procedural blank corrected by subtracting the mean of the procedural blank measurements (μ g/kg).

1.3 Participant Instructions

Hemp Plant Samples

Participants were provided with one packet of NRC HEMP-1 (Plant Sample 1) and one packet of Plant Sample 4 each containing approximately 3 g of dried hemp plant material. Participants were asked to store the sample under controlled freezer conditions (\approx -20 °C) in the original unopened packet. Before use, participants were instructed to allow the contents of the packet to equilibrate at room temperature for at least 1 h before mixing thoroughly. A sample size of 0.5 g was recommend based on homogeneity measurements at NIST to help minimize variability caused by sampling. Participants were asked to prepare three samples and report three mass fraction (mg/kg) values from the single packets provided on an as-received basis.

Control Sample: SRM 1575a Trace Elements in Pine Needles (Pinus taeda)

Participants were provided with one packet of SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) containing approximately 3 g of material. Participants were asked to store the sample at room temperature (20 °C to 25 °C) in the original unopened packet. Participants were instructed to thoroughly mix the contents of the packet prior to subsampling. A sample size of 0.25 g was recommend based on homogeneity measurements at NIST to help minimize variability caused by sampling. Participants were asked to prepare three samples and report three mass fraction (mg/kg) values from the single packet provided on an as-received basis.

2. Arsenic, Cadmium, Mercury, and Lead

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. While consumers may not be fully aware of potential safety concerns with product use, stakeholders in the cannabis community are concerned about toxic element contaminants in cannabis products. To fully understand the impact of these contaminants on consumers, analytical methods must accurately determine the levels of toxic elements in a variety of product types. Hemp is a known hyperaccumulator and historically has been used as a phytoremediator to remove toxic elements from soil. As a result, a significant potential exists for human exposure to toxic elements in hemp and marijuana raw materials and finished products [4, 5]. All states have regulations with mandated maximum levels for arsenic, cadmium, lead, and mercury and some states require testing for additional elements. This section of the report will cover results reported for arsenic, cadmium, lead, and mercury in the two hemp samples as well as the control sample. Results for additional elements included in this study will be covered in the subsequent section.

2.2. Reporting Statistics

The enrollment and reporting statistics for As, Cd, Hg, and Pb are described in the table below for each analyte. Reported values may include non-quantitative results (zero or below LOQ).

		Percent Reporting Results			
Analytes	Number of Participants	NRC HEMP-1	Plant Sample 4	<u>SRM 1575a</u>	
As	124	75 %	73 %	73 %	
Cd	124	75 %	75 %	74 %	
Hg	120	73 %	73 %	73 %	
Pb	122	75 %	75 %	74 %	

Most laboratories reported using microwave digestion for determination of As, Cd, Hg, and Pb in the two hemp samples and SRM 1575a (see table below). Additional sample preparation details are summarized at the end of the report in the appendix.

	Percent Reporting Results					
<u>Analytes</u>	Microwave Digestion	Hot Block Digestion	Open Beaker Digestion	<u>None/Not</u> Specified		
As	91 %	4 %	2 %	2 %		
Cd	91 %	4 %	2 %	2 %		
Hg	92 %	5 %	2 %	1 %		
Pb	91 %	5 %	2 %	2 %		

Most laboratories reported using ICP-MS for the determination of As, Cd, Hg, and Pb in the two hemp samples and SRM 1575a (see table below). Additional method details are summarized at the end of the report in the appendix.

	Percent Reporting Results					
<u>Analytes</u>	ICP-MS	ICP-OES	ID ICP-MS	<u>NAA</u>	<u>CV AAS</u>	<u>Other/None</u> <u>Selected</u>
As	88 %	5 %	2 %	1 %	-	3 %
Cd	85 %	5 %	2 %	1 %	-	3 %
Hg	85 %	6 %	3 %	1 %	1 %	3 %
Pb	88 %	7 %	2 %	-	-	4 %

The between-laboratory variabilities for determination of As, Cd, Hg, and Pb in the three samples are shown in the table below.

	Between	<u>ı-Laboratory Variability (</u>	<u>% RSD)</u>
Analytes	NRC HEMP-1	Plant Sample 4	<u>SRM 1575a</u>
As	3 %	3 %	3 %
Cd	1 %	1 %	1 %
Hg	5 %	8 %	3 %
Pb	2 %	2 %	1 %

The range of the variability of individual laboratory means for determination of As, Cd, Hg, and Pb in the three samples are shown in the table below.

<u>Analytes</u>	Within	-Laboratory Variability (%	<u>% RSD)</u>
	NRC HEMP-1	Plant Sample 4	<u>SRM 1575a</u>
As	0.5 % to 29 %	2 % to 57 %	0.5 % to 26 %
Cd	0.1 % to 77 %	0.5 % to 25 %	0.2 % to 22 %
Hg	0.4 % to 92 %	1 % to 59 %	0.9 % to 35 %
Pb	0.3 % to 33 %	0.4 % to > 100 %	0.4 % to 48 %

2.3. Study Results

Arsenic (As)

• The mass fractions (mg/kg) of As in the two hemp samples were determined by NIST using ICP-MS as described in the Section 1. These NIST-determined values and the COA reference value for As in SRM 1575a were used as the target values in this study as summarized in **Table 2-1** and **Table 2-2**.

- Figure 2-1 to Fig. 2-9 summarize the reported results for As in the control and two hemp samples. Data from participants submitting only one measurement were included in these figures as well as Table 2-2, but were not included in the calculation of consensus statistics.
- The consensus ranges for As in NRC HEMP-1, Plant Sample 4, and SRM 1575a were completely within the NIST ranges of tolerance. Quantitative and qualitative results reported by participating laboratories are summarized in the table below.

		Number of	Number of
	Total Number of	Laboratories	Laboratories
	Laboratories	<u>Reporting</u>	<u>Reporting</u>
<u>Samples</u>	Reporting Results	<u>Qualitative Results</u>	Quantitative Results
NRC HEMP-1	93	2	91
Plant Sample 4	91	34	57
SRM 1575a	90	31	59

• Laboratories reporting outlying results with respect to the NIST range of tolerance and consensus range of tolerance ($|Z'_{comm}| > 2$) are summarized in the table below.

	Number (%) of	Number (%) of	
	<u>Laboratory Means</u> Outside NIST Range	Laboratory Means	<u>Number (%) of</u> Laboratories
Samples	of Tolerance	Range of Tolerance	Reporting LOQs
NRC HEMP-1	6 (6 %)	10 (11 %)	2 (1 %)
Plant Sample 4	20 (21 %)	3 (3 %)	34 (37 %)
SRM 1575a	27 (30 %)	4 (4 %)	31 (34 %)

• The number of laboratories reporting results for As outside consensus ranges of tolerance for multiple samples are summarized in the table below.

	<u>Number of Laboratory</u> Mean Values Outside	Percentage of Laboratory Mean Values Outside both
Samples	both Consensus Ranges	Consensus Ranges
NRC HEMP-1 / Plant Sample 4	9 of 61	15 %
NRC HEMP-1 / SRM 1575a	8 of 59	14 %
Plant Sample 4 / SRM 1575a	6 of 55	11 %

Cadmium (Cd)

- The mass fractions (mg/kg) of Cd in the two hemp samples were determined by NIST using ICP-MS as described in Section 1. These NIST-determined values and the certified value for Cd in SRM 1575a were used as the target values in this study as summarized in **Table 2-1** and **Table 2-3**.
- Figure 2-10 to Fig. 2-18 summarizes the reported results for Cd in the control and two hemp samples. Data from participants submitting only one measurement were included in these figures as well as Table 2-3 but were not included in the calculation of consensus statistics.
- The consensus ranges for Cd in NRC HEMP-1, Plant Sample 4, and SRM 1575a were completely within the NIST range of tolerance. Quantitative and qualitative results reported by participating laboratories are summarized in the table below.

		Number of	Number of
	Total Number of	Laboratories	<u>Laboratories</u>
	Laboratories	<u>Reporting</u>	<u>Reporting</u>
Samples	Reporting Results	Qualitative Results	Quantitative Results
NRC HEMP-1	93	10	83
Plant Sample 4	93	15	78
SRM 1575a	92	5	87

• Laboratories reporting outlying results with respect to the NIST range of tolerance and consensus range of tolerance ($|Z'_{comm}| > 2$) are summarized in the table below.

	Number (%) of	Number (%) of	Number (%) of
	Laboratory Means	Laboratory Means	Laboratories
	Outside NIST	Outside Consensus	<u>Reporting</u>
Samples	Range of Tolerance	Range of Tolerance	LOQs
NRC HEMP-1	4 (4 %)	15 (16 %)	10 (11 %)
Plant Sample 4	0 (0 %)	5 (5 %)	15 (16 %)
SRM 1575a	48 (52 %)	7 (8 %)	5 (5 %)

• The number of laboratories reporting results for Cd outside consensus ranges of tolerance for multiple samples are summarized in the table below.

	Number of Laboratory	Percentage of
	Mean Values Outside	Laboratory Mean
	both Consensus	Values Outside both
Samples	Ranges	Consensus Ranges
Hemp Sample 1 / Plant Sample 4	5 out of 77	6 %
Hemp Sample 1 / SRM 1575a	17 out of 82	21 %
Plant Sample 4 / SRM 1575a	8 out of 78	10 %

Mercury (Hg)

- The mass fractions (mg/kg) of Hg in the two hemp samples were determined by NIST using DC AAS as described in Section 1. The NIST-determined values and the certified value for SRM 1575a were used as target values as summarized in **Table 2-1** and **Table 2-4**.
- Figure 2-19 to Fig. 2-27 summarizes the reported results for Hg in the control and two hemp samples. Data from participants submitting only one measurement were included in these figures as well as Table 2-4 but were not included in the calculation of consensus statistics.
- The consensus range for Hg in NRC HEMP-1 was completely within the NIST target range for these samples. The consensus ranges for Hg in Plant Sample 4 and SRM 1575a overlapped the upper portions of the NIST target ranges. Quantitative and qualitative results reported by participating laboratories are summarized in the table below.

		Number of	Number of
	Total Number of	Laboratories	Laboratories
	Laboratories	Reporting	Reporting
<u>Samples</u>	Reporting Results	Qualitative Results	Quantitative Results
NRC HEMP-1	88	33	55
Plant Sample 4	88	45	43
SRM 1575a	88	22	66

• Laboratories reporting outlying results with respect to the NIST range of tolerance and consensus range of tolerance ($|Z'_{comm}| > 2$) are summarized in the table below.

	<u>Number (%) of</u> <u>Laboratory Means</u> <u>Outside NIST</u>	<u>Number (%) of</u> <u>Laboratory Means</u> <u>Outside Consensus</u>	<u>Number (%) of</u> <u>Laboratories</u> <u>Reporting</u>
Samples	Range of Tolerance	Range of Tolerance	LOQs
NRC HEMP-1	29 (33 %)	9 (10 %)	33 (38 %)
Plant Sample 4	24 (28 %)	7 (8 %)	45 (51 %)
SRM 1575a	44 (50 %)	6 (7 %)	22 (25 %)

• The number of laboratories reporting results for Hg outside consensus ranges of tolerance for multiple samples are summarized in the table below.

	Number of Laboratory	Percentage of
	Mean Values Outside	Laboratory Mean
	both Consensus	Values Outside both
Samples	Ranges	Consensus Ranges
Hemp Sample 1 / Plant Sample 4	10 out of 42	24 %
Hemp Sample 1 / SRM 1575a	9 out of 55	16 %
Plant Sample 4 / SRM 1575a	9 out of 41	22 %

Lead (Pb)

- The mass fractions (mg/kg) of Pb in the two hemp samples were determined by NIST using ICP-MS as described in Section 1. The NIST-determined values and the reference value for SRM 1575a were used as the target values in this study as summarized in **Table 2-1** and **Table 2-5**.
- Figure 2-28 to Fig. 2-36 summarizes the reported results for Pb in the control and two hemp samples. Data from participants submitting only one measurement were included in these figures as well as Table 2-5 but were not included in the calculation of consensus statistics.
- The consensus ranges for Pb in Plant Sample 4 and SRM 1575a were completely within the NIST target ranges. The consensus range for Pb in NRC HEMP-1 was completely below the NIST target range. Quantitative and qualitative results reported by participating laboratories are summarized in the table below.

		Number of	Number of
	Total Number of	Laboratories	Laboratories
	Laboratories	Reporting	<u>Reporting</u>
Samples 1	Reporting Results	Qualitative Results	Quantitative Results
NRC HEMP-1	92	2	90
Plant Sample 4	91	7	84
SRM 1575a	90	10	80

• Laboratories reporting outlying results with respect to the NIST range of tolerance and consensus range of tolerance ($|Z'_{comm}| > 2$) are summarized in the table below.

	<u>Number (%) of</u> <u>Laboratory Means</u> Outside NIST	<u>Number (%) of</u> <u>Laboratory Means</u> Outside Consensus	<u>Number (%) of</u> <u>Laboratories</u> Reporting
<u>Samples</u>	Range of Tolerance	Range of Tolerance	LOQs
NRC HEMP-1	73 (79 %)	14 (15 %)	2 (2 %)
Plant Sample 4	8 (9 %)	8 (9 %)	7 (8 %)
SRM 1575a	11 (12 %)	13 (14 %)	10 (11 %)

• The number of laboratories reporting results for Hg outside consensus ranges of tolerance for multiple samples are summarized in the table below.

	<u>Number of Laboratory</u>	Percentage of
	Mean Values Outside	Laboratory Mean
	both Consensus	Values Outside both
Samples	Ranges	Consensus Ranges
NRC HEMP-1 / Plant Sample 4	14 out of 83	6 %
NRC HEMP-1 / SRM 1575a	14 out of 80	18 %
Plant Sample 4 / SRM 1575a	14 out of 78	18 %

2.4. Study Discussion and Technical Recommendations

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

- Sample preparation methods should be well established before analyzing unknown samples. Established quality control materials (SRM, CRM, RM and in-house materials when not commercially available) and established methods of analysis should be used whenever possible.
- The very low levels of toxic elements are challenging, and laboratories must balance many factors when deciding on the best methods to use.
 - Detection of the analytes in the sample may be improved by limiting the number of dilutions performed, however matrix effects may become more significant with fewer dilutions.
 - The method of standard additions may improve LOQs, accuracy, and precision, but is time consuming.
 - Analysis of an appropriate number of procedural blanks is critical in the determination of LOQ or when trying to reduce within-laboratory variability. Analysis of numerous blanks (usually the number of blanks equal the number of samples, or 10 when determining LOQ) can provide information about whether the source of variability is from the sample or from the sample preparation method.
- Calibration curves should be linear when used for quantitation.
 - Standards must include the lowest and highest values expected to be measured in the sample solutions. Several standards in between the highest and lowest standards should also be included to ensure linearity.
 - Accurate measurements can be achieved by making sure the sample concentrations fall within the middle of the calibration curve.
 - The calibration curve must be checked for linearity at the point of the expected sample concentrations.
- All results should be reported accurately.
 - Zero is not a quantity that can be measured. If values are below LOQ, results should be reported as such. A more appropriate result would be to report that a value is below the LOQ (e.g., "< 0.02 mg/kg").
 - Laboratories reporting results flagged as outliers should check for calculation errors when preliminary data tables are sent for inspection. One example is to confirm that factors for all dilutions have been properly tabulated or that results are reported in the requested units.

Arsenic (As)

- Most laboratories reported using microwave digestion as their sample preparation method and ICP-MS as their analytical method. With so few other techniques reported, no significant trend was observed showing that one technique performed better than another.
- To ensure complete digestion of the materials prior to analysis, high temperatures in a closed system are required.
 - Arsenic is volatile and can be lost during sample preparation. A vigorous microwave digestion should convert all volatile organoarsenic species to arsenic acid (AsV), after which point subsequent heating will not result in loss of arsenic.
 - Closed-vessel digestions should be opened with care ensuring that no arsenic is lost because of inadvertent venting.

- Open beaker digestion may lead to low results due to loss of volatile arsenic species.
- Difficulty in the digestion of samples can cause bias and/or increased variability between samples. Higher temperatures or the use of a small amount of HF in addition to oxidizing reagents may be required for complete digestion of hemp materials prior to analysis.
- Where laboratories reported results closer to the target value for one material than for a second material, the differences in the two matrices (hemp versus pine needles) or in the concentration levels (NRC HEMP-1 versus Plant Sample 4) may have resulted in difficulties in preparation and/or analysis.
 - An appropriate number of procedural blanks should be prepared along with the samples, especially when measured values are close to detection or quantitation limits.
 - Failure to completely digest the organic constituents may produce interferences that cause signal enhancement or suppression, introducing measurement bias in one of the matrices. Collision cell technology can be used to minimize the molecular ion interferences that may be found when analyzing arsenic in these materials.
- Measurement methods should be reported correctly and completely. For example, some laboratories reported using ID ICP-MS as the analytical method, which is not practicable for arsenic measurement because arsenic is monoisotopic.

Cadmium (Cd)

- Most laboratories reported using microwave digestion as their sample preparation method and ICP-MS as their analytical method. With so few other techniques reported, no significant trend was observed showing one technique performed better than another. As shown in the sample/sample plots, Fig. 2-16 through Fig. 2-18, most laboratories were able to measure Cd accurately in Plant Sample 4.
- Several laboratories reported values below LOQ for Cd in NRC HEMP-1 and Plant Sample 4 while far fewer laboratories reported values below LOQ for SRM 1575a. Because the level of Cd in SRM 1575a is much lower than the level in NRC HEMP-1, incomplete digestion of the hemp samples or the difference in the sample matrices may have led to inability of laboratories to measure or report Cd values above the LOQ.
- The boiling point of Cd is high and volatile loss of Cd should not be a concern, so high temperatures are recommended to ensure a complete digestion. Incomplete digestions can cause matrix produced interferences.
- Spectral/isobaric interferences can make Cd difficult to measure accurately by ICP-MS. High concentrations of certain elements (e.g., Mo, Sn, or Zr) are known to cause interferences in the analysis of Cd by ICP-MS. Isobaric spectral interferences such as ⁹⁵Mo¹⁶O⁺ and ⁹⁷Mo¹⁶O⁺ can affect the accuracy of Cd determination at 111 u and 113 u by ICP-MS. Most ICP-MS instruments allow an elemental survey of the sample prior to the measurement of analytes of interest without the need for calibration standards. Such a scan of the sample before analysis will help to identify any potential interferences in the sample that will need to be addressed.
 - Anion exchange separation of matrix elements prior to ICP-MS can reduce interferences; however, this option can be more time consuming.
 - Collision cell technology can be used to minimize molecular interferences that may be found in these three materials.
 - The use of ID ICP-MS is a good choice for analytical measurements of Cd.
 - There are eight different stable isotopes for Cd.
 - Can be used with SPE to decrease the uncertainty due to interferences, especially Mo.

Mercury (Hg)

- **Figures 2-25** through **2-27** show that many laboratories reported Hg results outside of the NIST range of tolerance or that were below the laboratory LOQ. The very low levels of Hg in the samples, especially Plant Sample 4, may have resulted in inaccuracies or inability to detect Hg.
- Mercury is volatile so care must be taken to not lose Hg during sample preparation. Microwave digestion is the recommended digestion technique for mercury analysis by ICP since high temperatures from a microwave will ensure a complete digestion and the closed vessels will prevent Hg loss from volatility.
- A sufficient number of procedural blanks should be used to determine an accurate LOQ for Hg and accommodate for high levels in blanks and backgrounds that may lead to high detection limits and make determination of low-level samples difficult.
- Low concentrations of mercury are not stable in solution over time.
 - Samples should be prepared as near as possible to the time of analysis.
 - Samples containing low concentrations of Hg may be more stable by adding some HCl (3 to 5 %) to diluted HNO₃ sample digests.
 - Acidification of sample solutions will help prevent loss of Hg by adsorption.
 - Addition of dichromate to sample solutions will help prevent loss of Hg through volatilization.
- The sensitivity of ICP-MS for Hg is low and requires a long washout time but may be improved by using cold vapor Hg generation.
- Mercury carryover between samples is common and can lead to erratic results. Adequate washout time is needed after each measurement by ICP-MS. The use of dilute HCl in the rinse solution may decrease the length of the washout time needed.
- Use of DC AAS or direct mercury analyzers for Hg analytical methods allows low detection limits and does not require sample preparation, which reduces sample throughput time.
- In some cases, laboratories reporting measured values at or above the upper limit of the range of tolerance also reported larger within-laboratory variability indicating a potential calibration issue.

Lead (Pb)

- **Figures 2-28** and **2-29** show that most laboratories were below the NIST range of tolerance for Pb in NRC HEMP-1.
- Lead is easily digested, and volatile loss of lead is not a concern. However, digestion with HCl may form insoluble PbCl₂ precipitate so digestion with HNO₃ is recommended. Because the level of lead in NRC HEMP-1 is approximately 20 times greater than in Plant Sample 4 and SRM 1575a, PbCl₂ precipitation may have resulted in low results being reported in NRC HEMP-1 if the sample digestion was not conducted consistently between materials. If HCl is used in digestion, then repeated washings of the side of the beaker with dilute acid may redissolve the PbCl₂.
- Since no linear trend was observed in **Fig. 2-34** through **Fig. 2-36** between the reported results for lead in the different materials, the sample preparation or analysis of NRC HEMP-1 may have been more difficult compared to the sample preparations of either Plant Sample 4 or SRM 1575a.
- Some laboratories reported high within-laboratory variability in one or more materials, especially noticeable in Plant Sample 4.

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• The low lead levels, difficulties in sample preparation, incomplete sample digestion, or calibration curves which do not encompass all sample solutions measured could result in high within-laboratory variability. Sample solutions which fall above the upper limit of the calibration curve may give an erroneous value.

Table 2-1. Individualized data summary table (NIST) for As, Cd, Hg, and Pb in hemp and control samples.

	Lab Code:	NIST	1. Your Results				2. Community Res			ults 3. Target	
Analyte	Sample	Units	x _i	s _i	Z' _{comm}	Z _{NIST}	N	x*	s*	X _{NIST}	U
Arsenic (As)	SRM 1575a Trace Elements in Pine Needles (Pinus taeda)	mg/kg	0.0379	0.0039			51	0.0420	0.0011	0.0379	0.0039
Arsenic (As)	NRC HEMP-1 (Plant Sample 1)	mg/kg	2.153	0.868			83	1.847	0.049	2.153	0.868
Arsenic (As)	Plant Sample 4	mg/kg	0.0340	0.0080			50	0.0405	0.0013	0.0340	0.0080
Cadmium (Cd)	SRM 1575a Trace Elements in Pine Needles (Pinus taeda)	mg/kg	0.2262	0.0078			78	0.2164	0.0027	0.2262	0.0078
Cadmium (Cd)	NRC HEMP-1 (Plant Sample 1)	mg/kg	0.1890	0.0540			76	0.1569	0.0023	0.1890	0.0540
Cadmium (Cd)	Plant Sample 4	mg/kg	0.0825	0.0592			71	0.0766	0.0011	0.0825	0.0592
Mercury (Hg)	SRM 1575a Trace Elements in Pine Needles (Pinus taeda)	mg/kg	0.0387	0.0014			56	0.0394	0.0012	0.0387	0.0014
Mercury (Hg)	NRC HEMP-1 (Plant Sample 1)	mg/kg	0.0156	0.0020			50	0.01721	0.00089	0.0156	0.0020
Mercury (Hg)	Plant Sample 4	mg/kg	0.00732	0.00088			35	0.00829	0.00070	0.00732	0.00088
Lead (Pb)	SRM 1575a Trace Elements in Pine Needles (Pinus taeda)	mg/kg	0.1622	0.0291			72	0.1491	0.0021	0.1622	0.0291
Lead (Pb)	NRC HEMP-1 (Plant Sample 1)	mg/kg	3.562	0.456			83	2.413	0.046	3.562	0.456
Lead (Pb)	Plant Sample 4	mg/kg	0.1860	0.0520			78	0.2029	0.0032	0.1860	0.0520
			x _i Mean of reported values			N Number	of quantitativ	e x	NIST-as	sessed value	
			s _i Standard	deviation o	f reported v	alues	values re	eported		U expanded	uncertainty
		Z' _{co}	mm Z'-score	with respec	t to commu	nity	x* Robust r	nean of repor	ted	about the	NIST-assessed valu
			consensu	6			values				

National Institute of Standards and Technology

 $Z_{\text{NIST}}\,$ Z-score with respect to NIST value $$s^{*}$$ Robust standard deviation

Table 2-2. Data summary table for arsenic (As) in hemp and control samples. **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 result in an unacceptable Z'_{comm} score, $|Z'_{comm}| > 2$. *Note*: This table spans three pages; the NIST target values and consensus values are included on all three pages for convenience.

		Arsenic (As)														
		SRM 15	75a Traco (Pinus	e Element s <i>taeda)</i> (1	ts in Pine mg/kg)	Needles	NRC HEMP-1 (Plant Sample 1) (mg/kg)				Plant Sample 4 (mg/kg)					
	Lab	Α	В	С	Avg	SD	Α	В	С	Avg	SD	Α	В	С	Avg	SD
	Target				0.0379	0.0039				2.153	0.868				0.0340	0.0080
	B001	0.042	0.0321	0.0368	0.0370	0.0050	2.07	1.67	1.62	1.787	0.247	0.0272	0.0248	0.0275	0.0265	0.0015
	B003	1.71579	1.69561	1.80187	1.7378	0.0564	0.02282	0.02423	0.02085	0.023	0.002	0.015845	0.02188	0.01509	0.0176	0.0037
	B004	<1	<1	<1			1.71	1.67	1.73	1.703	0.031	<1	<1	<1		
	B006															
	B009															
	B012	0.041	0.043	0.042	0.0420	0.0010	2.06218	1.98047	2.00654	2.016	0.042	0.0356	0.0369	0.0339	0.0355	0.0015
	B013															
	B015						0.1	0.102	0.103	0.102	0.002					
	B016	< 0.15	< 0.15	< 0.15			2.27	2.19	2.15	2.203	0.061	< 0.15	< 0.15	< 0.15		
	B017	0.020	0.027	0.020	0.0255	0.0000	1.402	1 505	1.400	1.465	0.051	0.022	0.024	0.02	0.0220	0.0000
	B018	0.038	0.037	0.038	0.0377	0.0006	1.482	1.505	1.408	1.465	0.051	0.032	0.034	0.03	0.0320	0.0020
	B020	0.0449	0.0455	0.0407	0.04(7	0.0027	1 0 1 0 2	1 0027	1 0002	1 002	0.097	0.0420	0.0422	0.04(2	0.0429	0.0021
	B023	0.0448	0.0455	0.0497	0.0467	0.0027	1.8183	1.9027	1.9895	1.903	0.080	0.0429	0.0423	0.0462	0.0438	0.0021
	D027	<0.05	<0.05	<0.05			2.62	2.72	2.31	2.085	0.138	<0.03	<0.05	0.0304	0.0304	
ults	B028	0.0375	0.0347	0.038	0.0367	0.0018	0.465	0.467	0.47	0.467	0.003	0.048	0.051	0.046	0.0483	0.0025
dividual Res	B029	<0.0373	<0.0347	<0.038	0.0307	0.0018	2 21	2 3 5 6	2 106	2 224	0.126	<0.043	<0.031	0.040	0.0690	0.0025
	B031	~0.045	~0.045	~0.045			2.21	2.550	2.100	2.224	0.120	~0.045	~0.050	0.007	0.0070	
	B035	< 0.05	< 0.05	< 0.05			1.672	1.517	1.729	1.639	0.110	0.032	0.032	0.034	0.0327	0.0012
	B037															
l	B040															
	B041	0.041	0.036	0.037	0.0380	0.0026	1.587	1.582	1.474	1.548	0.064	0.035	0.038	0.028	0.0337	0.0051
	B043	0.05			0.0500		2.68			2.680		0.06			0.0600	
	B044	0.04	0.04	0.04	0.0400	0.0000	2.04	1.57	1.85	1.820	0.236	0.03	0.04	0.04	0.0367	0.0058
	B049						2.44	2.55	2.39	2.460	0.082					
	B052	0.029	0.03	0.032	0.0303	0.0015	2.11	1.96	1.68	1.917	0.218	< 0.20	< 0.20	< 0.20		
	B057	0.048			0.0480		2.06			2.060		0.05			0.0500	
	B058	0.0466	0.0428		0.0447	0.0027	1.92	1.85	2.08	1.950	0.118	0.0468	0.0271	0.0871	0.0537	0.0306
	B060	0.0275	0.03	0.0284	0.0286	0.0013	1.629	1.73	1.654	1.671	0.053	0.035	0.036	0.033	0.0347	0.0015
	B061	0.0503	0.0606	0.0548	0.0552	0.0052	0.3308	0.3203	0.3253	0.325	0.005	0.0456	0.0514	0.0479	0.0483	0.0029
	B062	< 0.05	< 0.05	< 0.05			1.85951	1.95931	1.8209	1.880	0.071	< 0.05	< 0.05	< 0.05		
	B063															
	B064	1.302			1.3020		1.302			1.302		< 0.00049				
	B066	< 0.05	< 0.05	< 0.05			1.727	1.696	1.811	1.745	0.060	< 0.05	< 0.05	< 0.05		
	B069	0.04	0.039	0.042	0.0403	0.0015	2.058	2.057	1.991	2.035	0.038	0.036	0.044	0.04	0.0400	0.0040
	B070	0.042	0.044	0.042	0.0427	0.0012	1.766	1.667	1.929	1.787	0.132	0.038	0.041	0.036	0.0383	0.0025
uity .		Consensu	is Mean	1 During	0.0420		Consense	us Mean	1 Denier'	1.847		Consensus	Mean	Desisti	0.0405	
nur ults		Consensu	is Standar	a Deviatio	0.0011		Consensu	us Standar	a Deviatio	0.049		Consensus	Standard	Deviation	0.0013	
Res		Minimun	n		1./3/8		Minimum	n		2.792		Minimum			0.0690	
°C C		N	L		0.0000		N	1		0.023		N			0.0000	
		IN			51		IN			83		IN			50	

	Table	2-2.	continued.
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		Arsenic (As)														
		SRM 15	75a Traco (Pinus	e Element s <i>taeda)</i> (1	ts in Pine mg/kg)	Needles	NRC	HEMP-1	(Plant Sa	mple 1) (1	ng/kg)	Plant Sample 4 (mg/kg)				
	Lab	Α	В	С	Avg	SD	Α	В	С	Avg	SD	Α	В	С	Avg	SD
	Target				0.0379	0.0039				2.153	0.868				0.0340	0.0080
	B077	0.0444	0.0372	0.0373	0.0396	0.0041	1.6538	1.7913	1.5176	1.654	0.137	0.0348	0.0446	0.032	0.0371	0.0066
	B078															
	B079															
	B081	0.044	0.047	0.053	0.0480	0.0046	1.851	1.593	1.902	1.782	0.166	0.045	0.043	0.043	0.0437	0.0012
	B082	0.044	0.041	0.044	0.0430	0.0017	1.595	1.655	1.298	1.516	0.191	0.035	0.034	0.032	0.0337	0.0015
	B084	0.00	0.00	0.00	0.0000	0.0000	2.4	2.44	2.42	2.420	0.020	0.00	0.00	0.00	0.0000	0.0000
	B088	<1	<1	<1			2.9152	1.696	2.006	2.206	0.634	<1	<1	<1		
	B090	< 0.099	< 0.100	< 0.098			2.001	1.978	1.958	1.979	0.022	< 0.097	< 0.097	< 0.094		
	B091															
	B094	< 0.09	< 0.09	< 0.09			2.44	2.59	2.43	2.487	0.090	< 0.05	< 0.05	< 0.05		
	B095	0.05	0.039	0.048	0.0457	0.0059	1.882	1.767	1.904	1.851	0.074	0.034	0.04	0.063	0.0457	0.0153
	B097	0.044	0.036	0.045	0.0417	0.0049	1.115	0.984	1.031	1.043	0.066	0.0174	0.0203	0.0122	0.0166	0.0041
	B100	< 0.1					2.26			2.260		< 0.1				
	B102	0.02731	0.03567	0.03824	0.0337	0.0057	0.64198	0.54552	0.55833	0.582	0.052	0.02322	0.03599	0.02944	0.0296	0.0064
	B104	0.03	0.029	0.03	0.0297	0.0006	1.679	1.635	1.64	1.651	0.024	0.032	0.031	0.03	0.0310	0.0010
	B106	< 0.100	< 0.100	< 0.100			2.33	2.22	2.34	2.297	0.067	< 0.100	< 0.100	< 0.100		
	B107	0.0381	0.0435	0.0332	0.0383	0.0052	1.8007	1.7356	1.9518	1.829	0.111	0.0322	0.0328	0.0404	0.0351	0.0046
ts	B108															
sul	B109	< 0.05	< 0.05	< 0.05			1.3	1.22	1.29	1.270	0.044	< 0.05	0.05	< 0.05	0.0500	
Re	B110	0.0356	0.0357	0.0409	0.0374	0.0030	2.37	2.27	2.37	2.337	0.058	0.0433	0.0353	0.0417	0.0401	0.0042
ual	B111															
vid	B112	0.042	0.043	0.042	0.0423	0.0006	1.966	1.856	2.052	1.958	0.098	0.037	0.038	0.04	0.0383	0.0015
ibu	B113	0	0	0	0.0000		0.984	0.978	0.96	0.974	0.012	0	0	0	0.0000	
-	B116	0.055	0.053	0.054	0.0540	0.0010	2.618	3.136	2.622	2.792	0.298	0.052	0.047	0.045	0.0480	0.0036
	B119															
	B120															
	B122															
	B125	0.0482	0.0515	0.0456	0.0484	0.0030	0.509	0.489	0.467	0.488	0.021	0.0394	0.0355	0.0398	0.0382	0.0024
	B129															
	B130	0.05	0.05	0.05	0.0500		1.62	1.63	1.59	1.613	0.021	0.05	0.05	0.05	0.0500	
	B137	< 0.00017	< 0.00017	< 0.00017			1.7662	1.9754	1.9208	1.887	0.109	< 0.00017	< 0.00017	< 0.00017		
	B139	0.041	0.041	0.04	0.0407	0.0006	1.52	1.37	1.47	1.453	0.076	0.032	0.038	0.034	0.0347	0.0031
	B141	0.047	0.044	0.046	0.0457	0.0015	1.941	2.072	1.93	1.981	0.079	0.054	0.037	0.033	0.0413	0.0112
	B142	0.027	0.027	0.024	0.0260	0.0017	2.705	2.665	2.456	2.609	0.134	0.048	0.047	0.045	0.0467	0.0015
	B146	< 0.100	< 0.100	< 0.100			1.69	1.67	1.7	1.687	0.015	< 0.100	< 0.100	< 0.100		
	B147	< 0.05	< 0.05	< 0.05			2.28	2.09	2.02	2.130	0.135	< 0.05	< 0.05	< 0.05		
	B148															
	B149	< 0.050	< 0.050	< 0.050			1.569	1.39	1.39	1.450	0.103	< 0.050	< 0.050	< 0.050		
	B152															
	B153	< 0.001	< 0.001	< 0.001			< 0.001	< 0.001	< 0.001			< 0.001	< 0.001	< 0.001		
	B155	< 0.1	< 0.1				2.2	1.8	1.89	1.963	0.210	< 0.1	< 0.1	< 0.1		
	B159	0.044	0.04	0.037	0.0403	0.0035	1.779	1.874	1.983	1.879	0.102	0.054	0.046	0.044	0.0480	0.0053
ţ		Consensu	ls Mean		0.0420		Consense	us Mean		1.847		Consensus	Mean		0.0405	
uni lts		Consensu	us Standar	d Deviatio	0.0011		Consensu	us Standar	d Deviatio	0.049		Consensus	s Standard	Deviation	0.0013	
nm		Maximun	n		1.7378		Maximun	n		2.792		Maximum			0.0690	
R. O		Minimum	ı		0.0000		Minimum	ı		0.023		Minimum			0.0000	
C		Ν			51		Ν			83		Ν			50	

Table 2-2. continued.

		Arsenic (As)														
		SRM 1575a Trace Elements in Pine Needles (Pinus taeda) (mg/kg)					NRC HEMP-1 (Plant Sample 1) (mg/kg)					Plant Sample 4 (mg/kg)				
	Lab	Α	В	С	Avg	SD	Α	В	С	Avg	SD	Α	В	С	Avg	SD
Individual Results	Target				0.0379	0.0039				2.153	0.868				0.0340	0.0080
	B160	0.044	0.065	0.042	0.0503	0.0127	1.981	1.943	1.911	1.945	0.035	0.034	0.041	0.039	0.0380	0.0036
	B161															
	B163	0.0468	0.0483	0.046	0.0470	0.0012	2.4	2.34	2.53	2.423	0.097	0.0538	0.0439	0.0385	0.0454	0.0078
	B172	.0.00	.0.00	.0.06			1 505	1.504	1 515	1 720	0.015	0.053	0.051	0.024	0.0460	0.0104
	B174	< 0.06	<0.06	< 0.06	0.0202	0.0041	1.707	1.736	1.717	1.720	0.015	0.053	0.051	0.034	0.0460	0.0104
	B1/6	0.0346	0.03/3	0.0427	0.0382	0.0041	1.2701	1.284/	1.2985	1.284	0.014	0.0625	0.0617	0.0546	0.0596	0.0043
	B170 B179	0.0341	0.0396	0.0337	0.0332	0.0003	1.20	2.017	1.10	1.207	0.004	0.0288	0.0312	0.0333	0.0312	0.0024
	B180	< 0.05	< 0.05	< 0.05	0.0397	0.0005	1.984	1.857	2 054	1.998	0.109	< 0.0559	< 0.05	< 0.05	0.0548	0.0011
	B181	0.0658	0.00	0.00	0.0658		2.334	11007	21001	2.334	0110)	0.0567	0.00	0100	0.0567	
	B182	< 0.1	< 0.1	< 0.1			1.86	2	1.95	1.937	0.071	< 0.1	< 0.1	< 0.1		
	B183	0.0422			0.0422		1.9273			1.927		0.036			0.0360	
	B184	< 0.088	< 0.088	< 0.088			1.81	1.52	1.67	1.667	0.145	< 0.088	< 0.088	< 0.088		
	B186	< 0.059	< 0.0593	< 0.0594			2.3	2.13	2.27	2.233	0.091	< 0.059	< 0.059	0.0684	0.0684	
	B188															
	B189	< 0.1	< 0.1	< 0.1			0.301	0.306	0.295	0.301	0.006	< 0.1	< 0.1	< 0.1		
	B190	0.0484	0.0485	0.048	0.0483	0.0003	1.7483	1.6993	1.7722	1.740	0.037	0.0666	0.0671	0.0693	0.0677	0.0014
	B192	0.0485	< 0.0482	< 0.0482	0.0485		2.13	2.02	2.02	2.057	0.064	< 0.0482	< 0.0482	< 0.0482		
	B193	0.0457	0.0426	0.0488	0.0457	0.0031	1.67	1.7	1.73	1.700	0.030	0.0342	0.0428	0.0358	0.0376	0.0046
	B195	< 0.05	< 0.05	< 0.05			1.78	1.78	1.78	1.780		< 0.05	< 0.05	<0.05		
	B198	0.026	0.044	0.042	0.0407	0.0042	2.04	1.05	2.14	2.042	0.005	0.0246	0.0229	0.0226	0.0227	0.0010
	B200	0.036	0.044	0.042	0.0407	0.0042	2.04	2.42	2.14	2.043	0.095	0.0346	0.0338	0.0320	0.0337	0.0010
	B202	0.0432	0.0460	0.0439	0.0400	0.0018	1.81	1.82	1.20	1.843	0.070	0.0409	0.0439	0.0496	0.0409	0.0030
	B203	0.039	0.050	0.042	0.0390	0.0042	2.328	2 515	2.249	2.364	0.137	0.030	0.041	0.033	0.0420	0.0010
	B201	0.051	0.048	0.043	0.0473	0.0040	1.663	1.672	1.87	1.735	0.117	0.041	0.034	0.032	0.0357	0.0047
	B206	0.0355	0.0369	0.0362	0.0362	0.0007	0.096	0.093	0.0949	0.095	0.002	0.032	0.027	0.026	0.0283	0.0032
	B208															
	B211	< 0.16					< 0.16					< 0.16				
	B212	< 0.04	< 0.04	< 0.04			2.245	2.272	2.291	2.269	0.023	< 0.04	< 0.04	< 0.04		
	B213						2.4	2.3	2.2	2.300	0.100	< 0.2	< 0.2	< 0.2		
	B214						0.007			0.007						
	B215	< 0.0990	0.044	0.044	0.0417	0.0040	0.886	1.550	1.40	0.886	0.0(2	<0.09/1	<0.026	<0.026		
	D210	0.037	0.044	0.044	0.0417	0.0040	1.455	2.052	1.49	1.494	0.005	<0.030	0.035	<0.030	0.0200	0.0025
	B217	0.040	0.039	0.039	0.0420	0.0032	1.975	2.032	1.750	1.920	0.152	0.041	0.055	0.041	0.0390	0.0035
	B220	0.04	0.04	0.03	0.0367	0.0058	1.88	1.79	1.74	1.803	0.071	0.03	0.03	0.04	0.0333	0.0058
	B222	0.032	0.028	0.028	0.0293	0.0023	1.596	1.596	1.664	1.619	0.039	0.032	0.033	0.031	0.0320	0.0010
	B223	< 0.2	< 0.2	< 0.2			1.27	1.39	1.39	1.350	0.069	< 0.2	< 0.2	< 0.2		
	B224	0.05	0.03	0.04	0.0400	0.0100	1.97	1.87	1.86	1.900	0.061	0.04	0.04	0.04	0.0400	
	B226	< 0.1010 < 0.1010 < 0.0998					2.069	2.06036	2.00901	2.046	0.032	< 0.0992	< 0.0996	< 0.0984		
	B228															
	B230															
	B231									1						0.004.6
	B232	0.04008	0.03798	0.03525	0.0378	0.0024	1.78582	1.844//4	1.85981	1.830	0.039	0.0354/1	0.03414	0.03736	0.0357	0.0016
	B235	0.0563	0.0544	0.0533	0.0547	0.0015	2.1	2.87	2.65	2.540	0.397	0.0494	0.0451	0.0505	0.0483	0.0029
Community Results		Consensus Iviean			0.0420		Consensus Mean			0.049		Consensus Mean			0.0405	
		Maximum 1 73			1 7378		Maximum	is stanuar n		2 792		Maximum			0.0690	
		Minimum	Minimum				Minimum	 1		0.023		Minimum	Minimum			
		N			51		N			83		N			50	

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Fig. 2-1. Total arsenic in NRC HEMP-1 (Plant Sample 1) (data summary view – sample preparation method). **In** this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus the overlapping of the 95 % confidence interval for the consensus the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).


Fig. 2-2. Total arsenic in NRC HEMP-1 (Plant Sample 1) (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed as indicated in the figure key. 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, $|Z'_{comm}| \leq 2$. 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, $|Z_{NIST}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 2-3. Total arsenic in Plant Sample 4 (data summary view – sample preparation method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 2-4. Total arsenic in Plant Sample 4 (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean that results the overlapping of the 95 % confidence interval for the consensus mean that results in an acceptable Z_{NIST} score, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig 2-5. Total arsenic in SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (data summary view – sample preparation method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed as indicated in the figure key. 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, $|Z'_{comm}| \leq 2$. 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, $|Z_{NIST}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean that results the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 2-6. Total arsenic in SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Exercise: CannaQAP Exercise 2, Measurand: total arsenic

Fig. 2-7. Laboratory means for total arsenic in NRC HEMP-1 (Plant Sample 1) and Plant Sample 4 (sample/sample comparison view). In this view, the individual laboratory mean for one sample (NRC HEMP-1) is compared to the individual laboratory mean for a second sample (Plant Sample 4). The solid red box represents the NIST range of tolerance for the two samples, Plant Sample 4 (x-axis) and NRC HEMP-1 (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 1$ 2. The dotted blue box represents the consensus range of tolerance for Plant Sample 4 (x-axis) and NRC HEMP-1 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{comm}| \leq 2$.



Exercise: CannaQAP Exercise 2, Measurand: total arsenic

Fig. 2-8. Laboratory means for total arsenic in NRC HEMP-1 (Plant Sample 1) and SRM 1575a Trace Elements in Pine Needles (Pinus taeda) (sample/sample comparison view). In this view, the individual laboratory mean for one sample (NRC HEMP-1) is compared to the individual laboratory mean for a second sample (SRM 1575a). The solid red box represents the NIST range of tolerance for the two samples, SRM 1575a (x-axis) and NRC HEMP-1 (y-axis), which encompasses the target values bounded by their uncertainties (UNIST) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \le 2$. The dotted blue box represents the consensus range of tolerance for SRM 1575a (x-axis) and NRC HEMP-1 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{comm}| \le 2$.



Exercise: CannaQAP Exercise 2, Measurand: total arsenic

Fig. 2-9. Laboratory means for total arsenic in Plant Sample 4 and SRM 1575a Trace Elements in Pine Needles (Pinus taeda) (sample/sample comparison view). In this view, the individual laboratory mean for one sample (Plant Sample 4) is compared to the individual laboratory mean for a second sample (SRM 1575a). The solid red box represents the NIST range of tolerance for the two samples, SRM 1575a (x-axis) and Plant Sample 4 (y-axis), which encompasses the target values bounded by their uncertainties (UNIST) and represents the range that results in an acceptable ZNIST score, $|Z_{\text{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 1575a (x-axis) and Plant Sample 4 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{comm}| \le 2$.

Table 2-3. Data summary table for cadmium (Cd) in the hemp and control samples. 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, $|Z'_{\text{comm}}| > 2$. *Note*: This table spans three pages; the NIST values and consensus values are included on all three pages for convenience.

								(Cadmium	(Cd)						
		SRM 15	75a Traco <i>(Pinus</i>)	e Element <i>taeda)</i> (1	ts in Pine mg/kg)	Needles	NRC	HEMP-1	(Plant Sa	mple 1) (r	ng/kg)		Plant S	Sample 4 (mg/kg)	
	Lab	Α	В	С	Avg	SD	Α	В	С	Avg	SD	Α	В	С	Avg	SD
	Target				0.2262	0.0078				0.1890	0.0540				0.0825	0.0592
	B001	0.241	0.223	0.21	0.2247	0.0156	0.174	0.162	0.159	0.1650	0.0079	0.0727	0.0732	0.0736	0.0732	0.0005
	B003	0.16111	0.16663	0.17115	0.1663	0.0050	0.22066	0.22571	0.21826	0.2215	0.0038	0.084463	0.08172	0.07935	0.0818	0.0026
	B004	<1	<1	<1			<1	<1	<1			<1	<1	<1		
	B006															
	B009	0.000	0.000	0.00	0.0050	0.006	0.1(202	0.150.40	0.16650	0.1667	0.0025	0.0000	0.05065	0.07550	0.0704	0.0007
	B012	0.233	0.229	0.22	0.2273	0.0067	0.16303	0.17048	0.16652	0.1667	0.0037	0.0809	0.07865	0.07559	0.0784	0.0027
	B013	0.000	0.005	0.005	0.00(0)	0.0015	0.151	0.1.(1	0.155	0.1(00	0.0001	0.007	0.055	0.004	0.0055	0.0007
	B015	0.238	0.235	0.235	0.2360	0.0017	0.171	0.161	0.155	0.1623	0.0081	0.096	0.077	0.084	0.0857	0.0096
	B016	0.19	0.19	0.2	0.1933	0.0058	0.15	< 0.15	0.16	0.1550	0.00/1	< 0.15	< 0.15	< 0.15		
	B017	0.200	0.202	0.200	0.20(0	0.0020	0.15	0.152	0.147	0.1500	0.0020	0.076	0.077	0.072	0.0750	0.0020
	B018	0.209	0.203	0.206	0.2060	0.0030	0.15	0.155	0.14/	0.1500	0.0030	0.076	0.077	0.072	0.0750	0.0026
	B020	0.2449	0.2452	0.2271	0.2424	0.0046	0.1750	0 1622	0 1672	0 1695	0.0060	0.005	0 1022	0.005	0.0074	0.0042
	B023 B027	0.2440	0.2455	0.2371	0.2424	0.0040	0.1759	0.1025	0.1075	0.1005	0.0009	0.095	0.1022	0.095	0.09/4	0.0042
	B027	0.244	0.231	0.240	0.2470	0.0030	0.190	0.195	0.180	0.1923	0.0033	0.0940	0.0915	0.0903	0.0941	0.0024
	B020	0.209	0.212	0.223	0 2147	0.0074	0.14	0.151	0.148	0 1463	0.0057	0.089	0.078	0.075	0.0807	0.0074
ults	B030	0.20)	0.182	0.225	0.2147	0.0310	0.174	0.151	0.140	0.1740	0.0070	0.077	0.077	0.075	0.0740	0.0052
Res	B031	0.21	0.102	0.25	0.2175	0.0510	0.171	0.101	0.107	0.1710	0.0070	0.077	0.077	0.000	0.0710	0.0052
all	B035	0 207	0.208	0.211	0 2087	0.0021	0 149	0 1 5 3	0.156	0 1 5 2 7	0.0035	0.07	0.075	0.075	0.0733	0.0029
idu	B037															
vibr	B040															
In	B041	0.206	0.219	0.205	0.2100	0.0078	0.155	0.156	0.144	0.1517	0.0067	0.073	0.076	0.07	0.0730	0.0030
	B043	0.238			0.2380		0.173			0.1730		0.084			0.0840	
	B044	0.21	0.21	0.21	0.2100	0.0000	0.17	0.13	0.16	0.1533	0.0208	0.07	0.08	0.08	0.0767	0.0058
	B049	0.23	0.234	0.23	0.2313	0.0023	0.169	0.173	0.167	0.1697	0.0031	0.082	0.08	0.082	0.0813	0.0012
	B052	0.24	0.23	0.22	0.2300	0.0100	0.15	0.16	0.16	0.1567	0.0058	0.075	0.076	0.082	0.0777	0.0038
	B057	0.26			0.2600		0.18			0.1800		0.083			0.0830	
	B058	0.265	0.242	0.226	0.2443	0.0196	0.166	0.136	0.165	0.1557	0.0170	0.0791	0.0654	0.0794	0.0746	0.0080
	B060	0.1853	0.1932	0.1788	0.1858	0.0072	0.117	0.118	0.114	0.1163	0.0021	0.063	0.066	0.067	0.0653	0.0021
	B061	0.1831	0.1927	0.1824	0.1861	0.0058	0.0218	0.0249	0.0238	0.0235	0.0016	0.0765	0.0761	0.081	0.0779	0.0027
	B062	0.2203	0.21636	0.2069	0.2145	0.0069	0.1444	0.15089	0.13862	0.1446	0.0061	0.07658	0.07991	0.07778	0.0781	0.0017
	B063															
	B064	0.16			0.1600		0.16			0.1600		< 0.00020				
	B066	0.225	0.228	0.232	0.2283	0.0035	0.169	0.172	0.176	0.1723	0.0035	0.083	0.079	0.084	0.0820	0.0026
	B069	0.207	0.206	0.21	0.2077	0.0021	0.148	0.151	0.153	0.1507	0.0025	0.074	0.078	0.073	0.0750	0.0026
	B070	0.249	0.236	0.227	0.2373	0.0111	0.17	0.144	0.166	0.1600	0.0140	0.085	0.079	0.075	0.0797	0.0050
	B077 B078	0.1999	0.2072	0.2061	0.2044	0.0039	0.1503	0.1513	0.1537	0.1518	0.0017	0.0798	0.08/8	0.0723	0.0800	0.0078
ζ.		Consense	us Mean		0.2164		Consens	us Mean		0.1569		Consensu	s Mean		0.0766	
lts		Consense	us Standar	d Deviatio	0.0027		Consens	us Standar	d Deviatio	0.0023		Consensu	s Standard	Deviation	0.0011	
imn		Maximur	n		0.3761		Maximur	n		0.2587		Maximum			0.1500	
R		Minimum	1		0.1447		Minimun	1		0.0051		Minimum			0.0485	
<u> </u>		Ν			78		Ν			76		Ν			71	

Table	2-3.	continued.
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								Ca	dmium (Cd)							
		SRM 1575	a Trace Ele <i>taea</i>	ments in Pin <i>la)</i> (mg/kg)	e Needle	s (Pinus	NRC	С НЕМР-1 (Plant Sampl	e 1) (mg/l	kg)		Plant S	ample 4 (m	g/kg)	
	Lab	Α	В	С	Avg	SD	Α	В	С	Avg	SD	Α	В	С	Avg	SD
	Target				0.2262	0.0078				0.1890	0.0540				0.0825	0.0592
	B079															
	B081	0.223	0.234	0.268	0.2417	0.0235	0.141	0.041	0.042	0.0747	0.0574	0.07	0.074	0.071	0.0717	0.0021
	B082	0.244	0.228	0.267	0.2463	0.0196	0.188	0.176	0.137	0.1670	0.0267	0.074	0.08	0.08	0.0780	0.0035
	B084	0.209	0.21	0.2095	0.2095	0.0005	0.154	0.149	0.151	0.1513	0.0025	0.0728	0.0696	0.0712	0.0712	0.0016
	B088	<1	<1	<1			<1	<1	<1			<1	<1	<1		
	B090	0.218	0.216	0.221	0.2183	0.0025	0.156	0.157	0.159	0.1573	0.0015	< 0.097	< 0.097	< 0.094		
	B091															
	B094	0.18	0.18	0.18	0.1800		< 1.22	< 1.22	< 1.22			0.07	0.06	0.06	0.0633	0.0058
	B095	0.22	0.221	0.226	0.2223	0.0032	0.167	0.147	0.155	0.1563	0.0101	0.082	0.071	0.08	0.0777	0.0059
	B097	0.243	0.258	0.249	0.2500	0.0075	0.0873	0.093	0.0856	0.0886	0.0039	0.0951	0.0921	0.0911	0.0928	0.0021
	B100	0.21			0.2100		0.16			0.1600		0.15			0.1500	
	B102	0.18569	0.20101	0.19533	0.1940	0.0077	0.1338	0.14393	0.15155	0.1431	0.0089	0.07661	0.07382	0.067	0.0725	0.0049
	B104	0.216	0.229	0.225	0.2233	0.0067	0.158	0.152	0.153	0.1543	0.0032	0.077	0.078	0.077	0.0773	0.0006
	B106	0.288	0.241	0.282	0.2703	0.0256	0.18	0.16	0.18	0.1733	0.0115	0.105	< 0.100	0.116	0.1105	0.0078
	B107	0.2039	0.2033	0.2019	0.2030	0.0010	0.1478	0.1413	0.1611	0.1501	0.0101	0.0/16	0.0793	0.0/4/	0.0752	0.0039
	B108	0.00	0.00	0.00	0.0000		0.15	0.10	0.14	0.15/5	0.0200	0.00	0.07	0.00	0.05/5	0.0050
	B109	0.22	0.22	0.22	0.2200		0.15	0.18	0.14	0.1567	0.0208	0.08	0.07	0.08	0.0767	0.0058
ults	B110	0.203	0.196	0.187	0.1953	0.0080	0.156	0.156	0.144	0.1520	0.0069	0.0732	0.0693	0.0634	0.0686	0.0049
esu	BIII	0.000	0.24	0.000	0.0005	0.0010	0.146	0.125	0.16	0.1450	0.0105	0.070	0.070	0.000	0.0000	0.002(
II R	BI12	0.238	0.24	0.238	0.2387	0.0012	0.146	0.135	0.16	0.1470	0.0125	0.079	0.078	0.083	0.0800	0.0026
mp	BI15 D116	0.201	0.179	0.178	0.1800	0.0130	0.102	0.17(0.103	0.1050	0.0044	0.059	0.000	0.052	0.0557	0.0035
livi	B110	0.233	0.230	0.233	0.2340	0.0017	0.167	0.170	0.101	0.1680	0.0075	0.088	0.092	0.088	0.0893	0.0023
Inc	D119 D120															
	D120															
	D122 D125	0.22	0.210	0.222	0 2202	0.0015	0.0227	0.0218	0.0221	0.0220	0.0010	0.0615	0.0672	0.0683	0.0657	0.0027
	B129	0.22	0.217	0.222	0.2205	0.0015	0.0557	0.0510	0.0551	0.0327	0.0010	0.0015	0.0072	0.0005	0.0057	0.0037
	B130	0.22	0.22	0.23	0 2233	0.0058	0.14	0.13	0.11	0.1267	0.0153	0.07	0.07	0.07	0.0700	
	B130	0.368	0 4044	0.356	0.3761	0.0252	0 2072	0 2276	0.227	0.2206	0.0116	0.1124	0.1078	0.1227	0.1143	0.0076
	B139	0.21	0.206	0.211	0.2090	0.0026	0.16	0.16	0.168	0.1627	0.0046	0.074	0.075	0.073	0.0740	0.0010
	B141	0.22	0.217	0.214	0.2170	0.0030	0.158	0.16	0.146	0.1547	0.0076	0.081	0.079	0.079	0.0797	0.0012
	B142	0.17	0.178	0.169	0.1723	0.0049	0.229	0.235	0.225	0.2297	0.0050	0.109	0.111	0.114	0.1113	0.0025
	B146	0.218	0.212	0.215	0.2150	0.0030	0.165	0.167	0.161	0.1643	0.0031	< 0.100	< 0.100	< 0.100		
	B147	0.21	0.2	0.22	0.2100	0.0100	0.15	0.15	0.14	0.1467	0.0058	0.07	0.07	0.07	0.0700	
	B148															
	B149	0.3352	0.235	0.244	0.2714	0.0554	< 0.050	< 0.050	< 0.050			0.05693	< 0.050	< 0.050	0.0569	
	B152															
	B153	< 0.00025	< 0.00025	< 0.00025			< 0.00025	< 0.00025	< 0.00025			< 0.00025	< 0.00025	< 0.00025		
	B155	<2	<2				<5	<5	<5			<5	<5	<5		
	B159	0.249	0.222	0.252	0.2410	0.0165	0.148	0.156	0.164	0.1560	0.0080	0.074	0.082	0.069	0.0750	0.0066
	B160	0.216	0.305	0.212	0.2443	0.0526	0.157	0.15	0.154	0.1537	0.0035	0.07	0.078	0.079	0.0757	0.0049
	B161															
ity		Consensus	Mean		0.2164		Consensus 1	Mean		0.1569		Consensus	Mean		0.0766	
uni		Consensus	Standard Dev	viation	0.0027		Consensus	Standard Dev	viation	0.0023		Consensus	Standard De	viation	0.0011	
mm		Maximum			0.3761		Maximum			0.2587		Maximum			0.1500	
C oi		Minimum			0.1447		Minimum			0.0051		Minimum			0.0485	
-		N			78		N			76		N			71	

								Ca	dmium (Cd)							
		SRM 1575	a Trace Ele <i>taed</i>	ments in Pin la) (mg/kg)	e Needle	s (Pinus	NRO	C HEMP-1 (Plant Sampl	e 1) (mg/l	kg)		Plant S	ample 4 (m	g/kg)	
	Lab	Α	В	С	Avg	SD	Α	В	С	Avg	SD	Α	В	С	Avg	SD
	Target				0.2262	0.0078				0.1890	0.0540				0.0825	0.0592
	B163	0.287	0.285	0.248	0.2733	0.0220	0.151	0.155	0.158	0.1547	0.0035	0.0704	0.0707	0.0631	0.0681	0.0043
	B172															
	B174	0.211	0.207	0.211	0.2097	0.0023	0.152	0.155	0.141	0.1493	0.0074	0.078	0.076	0.073	0.0757	0.0025
	B176	0.2151	0.2326	0.2104	0.2194	0.0117	0.0914	0.0961	0.0979	0.0951	0.0034	0.0787	0.0791	0.0796	0.0791	0.0005
	B1/8	0.176	0.161	0.161	0.1660	0.0087	0.0757	0.077	0.0789	0.0772	0.0016	0.0563	0.0548	0.0569	0.0560	0.0011
	B1/9 D180	0.2350	0.2333	0.2576	0.2355	0.0022	0.1907	0.1957	0.2255	0.2055	0.0158	0.07927	0.08403	0.08016	0.0812	0.0025
	B180 B181	0.210	0.235	0.232	0.2337	0.0180	0.105	0.104	0.107	0.1650	0.0021	0.077	0.075	0.075	0.0750	0.0020
	B182	0.220	0.22	0.21	0.2200	0.0153	0.105	0.15	0.15	0.1500		<0.1	< 0.1	< 0.1	0.0000	
	B183	0.2671	0.22	0.21	0.2671	0.0155	0.1798	0.15	0.15	0.1798		0.0832	-0.1	-0.1	0.0832	
	B184	0.2	0.191	0.18	0.1903	0.0100	0.145	0.135	0.137	0.1390	0.0053	< 0.063	0.065	0.069	0.0670	0.0028
	B186	0.214	0.209	0.216	0.2130	0.0036	0.155	0.154	0.158	0.1557	0.0021	0.0877	0.0821	0.0832	0.0843	0.0030
	B188	-														
	B189	0.17	0.163	0.169	0.1673	0.0038	< 0.1	< 0.1	< 0.1			< 0.1	< 0.1	< 0.1		
	B190	0.1846	0.1852	0.1884	0.1861	0.0020	0.1109	0.1078	0.1126	0.1104	0.0024	0.0679	0.0674	0.0603	0.0652	0.0043
	B192	0.143	0.14	0.151	0.1447	0.0057	0.102	0.113	0.114	0.1097	0.0067	0.0487	0.0453	0.0516	0.0485	0.0032
	B193	0.206	0.208	0.235	0.2163	0.0162	0.147	0.147	0.152	0.1487	0.0029	0.068	0.0647	0.0799	0.0709	0.0080
	B195	0.214	0.204	0.211	0.2097	0.0051	0.154	0.149	0.154	0.1523	0.0029	0.0738	0.0738	0.0788	0.0755	0.0029
Its	B198															
esu	B200	0.219	0.22	0.227	0.2220	0.0044	0.171	0.175	0.179	0.1750	0.0040	0.089	0.084	0.084	0.0857	0.0029
I R	B202	0.209	0.218	0.213	0.2133	0.0045	0.148	0.149	0.151	0.1493	0.0015	0.0794	0.0753	0.073	0.0759	0.0032
qua	B203	0.229	0.216	0.236	0.2270	0.0101	0.163	0.163	0.163	0.1630	0.0000	0.07	0.074	0.077	0.0737	0.0035
livi	B204	0.23	0.228	0.226	0.2280	0.0020	0.166	0.166	0.165	0.1657	0.0006	0.085	0.081	0.089	0.0850	0.0040
Inc	B205	0.185	0.194	0.199	0.1927	0.0071	0.273	0.254	0.249	0.2587	0.0127	0.077	0.101	0.08	0.0860	0.0131
	B200 D208	0.191	0.180	0.191	0.1895	0.0029	0.0052	0.005	0.0051	0.0051	0.0001	0.054	0.0561	0.0577	0.0559	0.0019
	B208	<0.002					<0.002					<0.002				
	B212	0.2122	0.2285	0.226	0.2222	0.0088	0.1694	0.1694	0.1698	0.1695	0.0002	0.0822	0.0822	0.0813	0.0819	0.0005
	B212	0.2122	0.2200	0.220	0.2222	0.0000	0.18	0.17	0.16	0.1700	0.0100	0.09	0.07	0.09	0.0833	0.0115
	B214															
	B215	0.198			0.1980		< 0.0990					< 0.0971				
	B216	0.181	0.176	0.185	0.1807	0.0045	0.125	0.13	0.126	0.1270	0.0026	0.063	0.066	0.062	0.0637	0.0021
	B217	0.21	0.188	0.186	0.1947	0.0133	0.14	0.148	0.15	0.1460	0.0053	0.064	0.073	0.066	0.0677	0.0047
	B220															
	B221	0.196	0.193	0.186	0.1917	0.0051	0.162	0.146	0.15	0.1527	0.0083	0.07	0.069	0.065	0.0680	0.0026
	B222	0.21	0.24	0.22	0.2233	0.0153	0.159	0.163	0.163	0.1617	0.0023	0.081	0.076	0.082	0.0797	0.0032
	B223	0.202	0.201	0.205	0.2027	0.0021	< 0.2	< 0.2	< 0.2			< 0.2	< 0.2	< 0.2		
	B224	0.21	0.23	0.23	0.2233	0.0115	0.16	0.19	0.17	0.1733	0.0153	0.08	0.08	0.07	0.0767	0.0058
	B226	0.2246465	0.2232794	0.2171657	0.2217	0.0040	0.1660931	0.1640244	0.1655378	0.1652	0.0011	< 0.1004	< 0.0992	< 0.0996		
	B228															
	B230															
	B231	0 2222721	0 2277051	0 220976	0.2200	0.0022	0 1652425	0 1606672	0 1740008	0 1606	0.0044	0.006007	0.082405	0.006721	0.0800	0.0060
	B232 B235	0.2323721	0.2277931	0.229870	0.2300	0.0023	0.1032433	0.1690072	0.1740098	0.1090	0.0044	0.0618	0.085495	0.090721	0.0890	0.0009
~	11233	Consensus	Mean	0.477	0.2164	0.0241	Consensus	Mean	0.101	0.1520	0.0173	Consensus	Mean	0.0727	0.0766	0.0100
ts t		Consensus	Standard Dev	viation	0.0027		Consensus	Standard Dev	viation	0.0023		Consensus	Standard De	viation	0.0011	
umu		Maximum			0.3761		Maximum			0.2587		Maximum			0.1500	
lom Re		Minimum			0.1447		Minimum			0.0051		Minimum			0.0485	
C		Ν			78		Ν			76		Ν			71	



Fig. 2-10. Cadmium in NRC HEMP-1 (Plant Sample 1) (data summary view – sample preparation method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus the overlapping of the 95 % confidence interval for the consensus the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 2-11. Cadmium in NRC HEMP-1 (Plant Sample 1) (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 2-12. Cadmium in Plant Sample 4 (data summary view – sample preparation method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 2-13. Cadmium in Plant Sample 4 (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. The red shaded region represents the Interval for the consensus mean that result is uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 2-14. Cadmium in SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (data summary view – sample preparation method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed as indicated in the figure key. 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, $|Z'_{comm}| \leq 2$. 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, $|Z_{NIST}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

Fig. 2-15. Cadmium in SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

Exercise: CannaQAP Exercise 2, Measurand: cadmium

Fig. 2-16. Laboratory means for cadmium in NRC HEMP-1 (Plant Sample 1) and Plant Sample 4 (sample/sample comparison view). In this view, the individual laboratory mean for one sample (NRC HEMP-1) is compared to the individual laboratory mean for a second sample (Plant Sample 4). The solid red box (only the left limit is shown due to the scale of the figure) represents the NIST range of tolerance for the two samples, Plant Sample 4 (x-axis) and NRC HEMP-1 (y-axis), which encompasses the target values bounded by their uncertainties (UNIST) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \le 2$. The dotted blue box represents the consensus range of tolerance for Plant Sample 4 (x-axis) and NRC HEMP-1 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{comm}| \le 2$.

Exercise: CannaQAP Exercise 2, Measurand: cadmium No. of laboratories: 82

Fig. 2-17. Laboratory means for cadmium in NRC HEMP-1 (Plant Sample 1) and SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (sample/sample comparison view). In this view, the individual laboratory mean for one sample (NRC HEMP-1) is compared to the individual laboratory mean for a second sample (SRM 1575a). The solid red box (the top limit is not shown due to the scale of the figure)represents the NIST range of tolerance for the two samples, SRM 1575a (x-axis) and NRC HEMP-1 (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 1575a (x-axis) and NRC HEMP-1 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

Fig. 2-18. Laboratory means for cadmium in Plant Sample 4 and SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (sample/sample comparison view). In this view, the individual laboratory mean for one sample (Plant Sample 4) is compared to the individual laboratory mean for a second sample (SRM 1575a). The solid red box (only the left and right limits are shown due to the scale of the figure) represents the NIST range of tolerance for the two samples, SRM 1575a (x-axis) and Plant Sample 4 (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 1575a (x-axis) and Plant Sample 4 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

Table 2-4. Data summary table for mercury (Hg) in the hemp and control samples. 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 result in an unacceptable Z'_{comm} score, $|Z'_{comm}| > 2$. *Note*: This table spans three pages; the NIST values and consensus values are included on all three pages for convenience.

SRM 1575a Trace Elements in Pine Needles (Pinus taeda) (mg/kg) NRC HEMP-1 (Plant Sample 1) (mg/kg) Plant Sa Lab A B C Avg SD A B C Avg Avg D Avg D D D D D D D D D D D D D D D	mple 4 (mg C 0.006 0.00217 <1 0.0074	Avg 0.00732 0.00733 0.00229	SD 0.00088 0.00257 0.00016
Lab A B C Avg SD A B C Avg SD A B Target 0.0387 0.0014 0.01559 0.0200 0.01559 0.00200 0.01657 0.01013 0.0103 0.0057	C 0.006 0.00217 <1 0.0074	Avg 0.00732 0.00733 0.00229	SD 0.00088 0.00257 0.00016
Target 0.0387 0.0014 0.01559 0.00200 B001 0.0427 0.0368 0.0465 0.0420 0.0049 0.0176 0.017 0.0151 0.01657 0.0103 0.0057	0.006 0.00217 <1 0.0074	0.00732 0.00733 0.00229	0.00088 0.00257 0.00016
B001 0.0427 0.0368 0.0465 0.0420 0.0049 0.0176 0.017 0.0151 0.01657 0.00131 0.0103 0.0057	0.006 0.00217 <1	0.00733 0.00229	0.00257 0.00016
	0.00217 <1 0.0074	0.00229	0.00016
B003 0.00902 0.00876 0.00986 0.0092 0.0006 0.034216 0.034084 0.031774 0.03336 0.00137 0.00223 0.002478	<1		
B004 <1 <1 <1 <1 <1 <1 <1 <1	0.0074		
8006	0.0074		
8009	0.0074		
B012 0.039 0.047 0.056 0.0473 0.0085 0.010992 0.011311 0.01119 0.01116 0.00016 0.0073 0.0078	0.001.	0.00750	0.00026
8013			
B015 0.045 0.048 0.048 0.0470 0.0017			
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	< 0.10		
B018 0.037 0.04 0.04 0.0390 0.0017 <0.027 <0.027 <0.027 <0.027 <0.025 <0.025	< 0.025		
	0.0004	0.01152	0.00202
B023 0.0605 0.0578 0.0543 0.0575 0.0031 0.0181 0.0187 0.0143 0.01705 0.00239 0.0139 0.0123	0.0084	0.01153	0.00283
B027 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05	<0.05		
	<0.012		
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	<0.012		
	<0.044		
	0.006	0.00633	0.00058
	0.000	0.00055	0.00050
	< 0.01		
8043 <0.01 <0.02 >0.02			
B044 0.04 0.04 0.04 0.040 0.0000 0.03 0.02 0.03 0.02667 0.00577 0.02 0.01	0.01	0.01333	0.00577
B049			
$\begin{array}{ c c c c c c c c c c c c c c c c c c c$	< 0.10		
B057 0.045 0.0450 0.017 0.01700 <0.014			
B058 0.0385 0.0319 0.0326 0.0343 0.0036 0.0134 0.0114 0.0132 0.01267 0.00110 <0.00977 <0.00953	< 0.00959		
B060 0.1 0.102 0.11 0.1040 0.0053 0.079 0.071 0.065 0.07167 0.00702 0.02 0.019	0.02	0.01967	0.00058
B061 0.0139 0.0145 0.0112 0.0132 0.0018 <0.00005	< 0.00005		
B062 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 <	< 0.05		
B063			
B064 0.0002 0.0002 <0.0002 <0.0002 <0.0002			
B066 <0.05	< 0.05		
B069 0.031 0.029 0.031 0.0303 0.0012 0.01 0.01 0.011 0.01033 0.00058 0.004 0.005	0.006	0.00500	0.00100
B070 0.0408 0.0371 0.0351 0.0377 0.0029 0.0163 0.0129 0.0198 0.01633 0.00345 0.0061 0.0061	0.0054	0.00587	0.00040
B0// 0.0389 0.0383 0.0378 0.0378 0.0383 0.0006 0.0146 0.0148 0.0151 0.01483 0.00025 0.0067 0.0077	0.007	0.00713	0.00051
B0/8 0.01721 C 14		0.00920	
Consensus vican 0.0394 Consensus Mean 0.01/21 Consensus Mean Consensus Avadead Davisition 0.001/21 Consensus Studied Davistion 0.00092	viction	0.00829	
a Consensus standard Deviation 0.0012 Consensus standard Deviation 0.00099 Consensus standard Deviation Maximum 0.1589 Maximum 0.2590 Maximum	viduOfi	0.000/0	
иалпаніі 0.1000 Махіпалі 0.2000 Махіпалі		0.11300	
O Manuality O.0000 Manuality O.00000 Manuality N 56 N 50 N		35	

	Table	2-4.	continued.
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									Mercury (I	Hg)						
		SRM 15	75a Trac <i>(Pinu</i> :	e Elemen s <i>taeda)</i> (i	ts in Pine mg/kg)	Needles	NRC	C HEMP-1 (Plant Samp	le 1) (mg/	kg)		Plant Sa	mple 4 (mg	/kg)	
	Lab	Α	В	С	Avg	SD	Α	В	С	Avg	SD	Α	В	С	Avg	SD
	Target				0.0387	0.0014				0.01559	0.00200				0.00732	0.00088
	B079															
	B081	0.034	0.042	0.046	0.0407	0.0061	< 0.015	< 0.015	< 0.015			< 0.015	< 0.015	< 0.015		
	B082	0.041	0.039	0.044	0.0413	0.0025	0.008	0.01	0.004	0.00733	0.00306	0.006	0.005	0.006	0.00567	0.00058
	B084	0.0552	0.0582	0.0504	0.0546	0.0039	0.0242	0.0246	0.0202	0.02300	0.00243	0.00	0.00	0.00	0.00000	0.00000
	B088	<1	<1	<1			<1	<1	<1			<1	<1	<1		
	B090	< 0.050	< 0.050	< 0.050			< 0.247	< 0.234	< 0.244			<0.49	<0.48	<0.47		
	B091	. 0. 00	. 0. 00	. 0. 00			. 1. 00	. 1. 22	. 1. 2.2			.0.05	.0.05	.0.05		
	B094	< 0.09	< 0.09	< 0.09	0.0422	0.0015	< 1.22	< 1.22	< 1.22	0.02000	0.002(1	< 0.05	< 0.05	< 0.05	0.012(7	0.00(42
	B095	0.042	0.045	0.043	0.0433	0.0015	0.023	0.016	0.021	0.02000	0.00361	0.02	0.01	0.008	0.01267	0.00643
	B097	0.049	0.048	0.048	0.0485	0.0006	0.0178	0.0179	0.0177	0.01780	0.00010	<0.014	0.012	0.011	0.01255	0.00133
	B100	0.034	0.02786	0.03004	0.0340	0.0013	0.014	0.008	0.00010	0.01400	0.00065	~0.01	0.00621	0.00388	0.00505	0.00117
	B102	0.02777	0.02780	0.03004	0.0280	0.0013	0.00810	0.008	0.00919	0.00843	0.00005	<0.01	<0.0021	<0.01	0.00303	0.00117
	B104 B106	<0.037	<0.030	<0.035	0.0500	0.0010	<0.013	0.012	0.012	0.01233	0.00058	<0.01	<0.01	<0.01		
	B107	0.0304	0.0309	0.0312	0.0308	0.0004	0.0215	0.0118	0.0119	0.01507	0.00557	0.0058	0.0054	0.0056	0.00560	0.00020
	B107	0.0504	0.0507	0.0512	0.0508	0.0004	0.0215	0.0110	0.0117	0.01507	0.00557	0.0050	0.0054	0.0050	0.00500	0.00020
	B100	0.04	0.04	0.04	0.0400		< 0.02	< 0.02	< 0.02			< 0.02	< 0.02	< 0.02		
s	B110	0.0438	0.0371	0.0338	0.0382	0.0051	0.0184	0.0153	0.0147	0.01613	0.00199	0.0105	0.0092	0.0081	0.00927	0.00120
sult	B111				0.000											
Re	B112	0.04	0.041	0.04	0.0403	0.0006	0.013	0.018	0.016	0.01567	0.00252	0.0058	0.0063	0.0072	0.00643	0.00071
ual	B113	0	0	0	0.0000		0	0	0	0.00000		0	0	0	0.00000	
vidı	B116	0.043	0.042	0.044	0.0430	0.0010	0.016	0.017	0.017	0.01667	0.00058	0.008	0.009	0.008	0.00833	0.00058
ndř	B119															
-	B120															
	B122															
	B125	0.0409	0.027	0.0236	0.0305	0.0092	0.0729	0.0526	0.048	0.05783	0.01325	0.0507	0.0295	0.0141	0.03143	0.01838
	B129															
	B130	0.037	0.037	0.036	0.0367	0.0006	0.014	0.012	0.013	0.01300	0.00100	0.006	0.006	0.006	0.00600	
	B137	0.064	0.0643	0.062	0.0634	0.0013	0.02	0.0221	0.0216	0.02123	0.00110	0.0097	0.0108	0.0099	0.01013	0.00059
	B139	0.044	0.045	0.045	0.0447	0.0006	0.018	0.016	0.016	0.01667	0.00115	< 0.010	< 0.010	<0.010	0.00.000	
	BI41	0.044	0.039	0.038	0.0403	0.0032	0.013	0.013	0.012	0.01267	0.00058	0.005	0.005	0.005	0.00500	
	B142	0.037	0.037	0.035	0.0363	0.0012	0.023	0.025	0.024	0.02400	0.00100	< 0.015	< 0.015	< 0.015		
	B146	< 0.0537	< 0.0537	<0.0537	0.0200		< 0.0537	< 0.0537	< 0.0537	0.01000		< 0.0537	< 0.0537	< 0.0537		
	D147	0.03	0.03	0.03	0.0300		0.01	0.01	0.01	0.01000		<0.01	<0.01	<0.01		
	D140	0.00625	0.101	0.180	0.1599	0.0541	0.1104	0.152	0.162	0.14180	0.02756	0.05628	0.126	0.126	0.10043	0.04603
	B149 B152	0.09025	0.191	0.169	0.1366	0.0341	0.1104	0.133	0.102	0.14180	0.02750	0.03028	0.150	0.150	0.10945	0.04005
	B152	<0.00102	<0.00102	2<0.00102			<0.00102	<0.00102	<0.00102			<0.00102	<0.00102	<0.00102		
	B155	<1	<1	5100102			<2	<2	<2			<1	<1	<1		_
	B159	0.04	0.038	0.05	0.0427	0.0064	< 0.020	0.032	0.021	0.02650	0.00778	< 0.020	< 0.020	< 0.020		
	B160	0.031	0.045	0.034	0.0367	0.0074	0.017	0.016	0.016	0.01633	0.00058	0.007	0.008	0.007	0.00733	0.00058
	B161															
ţ,		Consense	ıs Mean		0.0394		Consensus	Mean		0.01721		Consensus	Mean		0.00829	
uni Its		Consense	ıs Standar	d Deviatio	0.0012		Consensus	Standard De	viation	0.00089		Consensus	Standard De	viation	0.00070	
nm		Maximun	n		0.1588		Maximum			0.26500		Maximum			0.11500	
B A		Minimum	ı		0.0000		Minimum			0.00000		Minimum			0.00000	
Ŭ		Ν			56		Ν			50		Ν			35	

Table 2-4. cd	ontinued.
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									Mercury (I	Ig)						
		SRM 15	75a Traco (Pinus	e Element s <i>taeda)</i> (1	ts in Pine mg/kg)	Needles	NRC	HEMP-1 (Plant Samp	le 1) (mg/	kg)		Plant Sa	mple 4 (mg	/kg)	
	Lab	Α	В	С	Avg	SD	Α	В	С	Avg	SD	Α	В	С	Avg	SD
	Target				0.0387	0.0014				0.01559	0.00200				0.00732	0.00088
	B172															
	B174	< 0.06	< 0.06	< 0.06			< 0.06	< 0.06	< 0.06			0.007	0.007	0.003	0.00567	0.00231
	B176	0.0388	0.0404	0.04	0.0397	0.0008	0.0247	0.0274	0.022	0.02470	0.00270	0.0086	0.0095	0.0076	0.00857	0.00095
	B178	0.0657	0.0525	0.0512	0.0565	0.0080	0.0281	0.0283	0.0283	0.02823	0.00012	0.0182	0.0176	0.0182	0.01800	0.00035
	B179	0.0456	0.0434	0.0421	0.0437	0.0018	0.017	0.0167	0.0198	0.01783	0.00171	0.009335	0.008443	0.007797	0.00853	0.00077
	B180	0.058	0.035	0.051	0.0480	0.0118	0.012	0.013	0.023	0.01600	0.00608	< 0.01	< 0.01	< 0.01		
	B181	0.0388			0.0388		0.016			0.01600		0.008			0.00800	
	B182	< 0.1	< 0.1	< 0.1			< 0.1	< 0.1	< 0.1			< 0.1	< 0.1	< 0.1		
	B183	0.0417			0.0417		0.0159			0.01590		0.0083			0.00830	
	B184	< 0.095	< 0.095	< 0.095			0.265	< 0.095	< 0.095	0.26500		0.115	< 0.095	< 0.095	0.11500	
	B186	0.0327	0.0335	0.0343	0.0335	0.0008	0.0131	0.0136	0.0133	0.01333	0.00025	0.00569	0.00648	0.00593	0.00603	0.00041
	B188															
	B189	< 0.1	< 0.1	< 0.1			< 0.1	< 0.1	< 0.1			< 0.25	< 0.25	< 0.25		
	B190	0.036	0.035	0.0351	0.0354	0.0006	0.0567	0.0266	0.0205	0.03460	0.01938	0.0132	0.0103	0.0098	0.01110	0.00184
	B192	0.0345	0.0333	0.0338	0.0339	0.0006	0.0141	0.0137	0.0136	0.01380	0.00026	< 0.00722	< 0.00722	< 0.00722		
	B193	0.0182	0.022	0.0231	0.0211	0.0026	0.102	0.0333	0.0152	0.05017	0.04579	< 0.00461	< 0.00451	< 0.00506		
ts	B195	0.0354	0.0358	0.0386	0.0366	0.0017	0.0161	0.0152	0.0169	0.01607	0.00085	< 0.01	< 0.01	< 0.01		
Inse	B202	0.0361	0.0371	0.0372	0.0368	0.0006	0.0142	0.0145	0.014	0.01423	0.00025	0.00634	0.00607	0.0058	0.00607	0.00027
Ř	B203	0.037	0.038	0.044	0.0397	0.0038	0.022	0.024	0.016	0.02067	0.00416	0.009	0.013	0.016	0.01267	0.00351
na	B204	0.041	0.041	0.041	0.0410		0.017	0.016	0.016	0.01633	0.00058	0.009	0.008	0.008	0.00833	0.00058
ivid	B205	0.03	0.03	0.031	0.0303	0.0006	0.01	0.01	0.011	0.01033	0.00058	0.003	0.003	0.003	0.00300	
ipu	B206	0.0448	0.0448	0.0441	0.0446	0.0004	< 0.02	< 0.02	< 0.02			< 0.02	< 0.02	< 0.02		
-	B208															
	B211	<1					<1					<1				
	B212	0.0414	0.04	0.04	0.0405	0.0008	< 0.04	< 0.04	< 0.04			< 0.04	< 0.04	< 0.04		
	B213						0.02	0.02	0.02	0.02000	0.00000	< 0.01	< 0.01	< 0.01		
	B214															
	B215	< 0.0990					< 0.0990					< 0.0971				
	B216	0.029	0.028	0.03	0.0290	0.0010	< 0.015	< 0.015	< 0.015			< 0.015	< 0.015	< 0.015		
	B217	0.032	0.031	0.029	0.0307	0.0015	< 0.036	< 0.028	< 0.011			< 0.006	< 0.006	< 0.006		
	B220															
	B221	0.06	0.06	0.06	0.0600		0.04	0.04	0.02	0.03333	0.01155	0.02	0.02	0.02	0.02000	
	B222	0.057	0.054	0.048	0.0530	0.0046	0.016	0.014	0.052	0.02733	0.02139	0.046	0.03	0.021	0.03233	0.01266
	B223	< 0.1	< 0.1	< 0.1			< 0.1	< 0.1	< 0.1			< 0.1	< 0.1	< 0.1		
	B224	0.06	0.05	0.05	0.0533	0.0058	0.01	0.03	0.02	0.02000	0.01000	0.01	0.01	0.02	0.01333	0.00577
	B226	< 0.1010	< 0.1012	< 0.0998			< 0.1012	< 0.1016	< 0.0994			< 0.1004	< 0.0996	< 0.09843		
	B228															
	B230															
	B231															
	B232	0.05063	0.04514	0.0423	0.0460	0.0042	0.033837	0.02992	0.02648	0.03008	0.00368	0.0165174	0.0152433	0.0143356	0.01537	0.00110
ity		Consensu	is Mean		0.0394		Consensus	Mean		0.01721		Consensus	Mean		0.00829	
un		Consensu	ıs Standar	d Deviatio	0.0012		Consensus	Standard De	viation	0.00089		Consensus	Standard De	viation	0.00070	
mn		Maximun	n		0.1588		Maximum			0.26500		Maximum			0.11500	
[™] Co		Minimum	L		0.0000		Minimum			0.00000		Minimum			0.00000	
-		Ν			56		Ν			50		N			35	

Fig. 2-19. Total mercury in NRC HEMP-1 (Plant Sample 1) (data summary view – sample preparation method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus the overlapping of the 95 % confidence interval for the consensus the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

Fig. 2-20. Total mercury in NRC HEMP-1 (Plant Sample 1) (data summary view – analytical method). In this view, individual analytical method data points are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus the overlapping of the 95 % confidence interval for the consensus the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

Fig. 2-21. Total mercury in Plant Sample 4 (data summary view – sample preparation method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

Fig. 2-22. Total mercury in Plant Sample 4 (data summary view – analytical method). In this view, individual analytical method data points are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean the overlapping of the 95 % confidence interval for the consensus mean for the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

Fig. 2-23. Total mercury in SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (data summary view – sample preparation method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

Fig. 2-24. Total mercury in SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (data summary view – analytical method). In this view, individual analytical method data points are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

Fig. 2-25. Laboratory means for total mercury in NRC HEMP-1 (Plant Sample 1) and Plant Sample 4 (sample/sample comparison view). In this view, the individual laboratory mean for one sample (NRC HEMP-1) is compared to the individual laboratory mean for a second sample (Plant Sample 4). The solid red box represents the NIST range of tolerance for the two samples, Plant Sample 4 (x-axis) and NRC HEMP-1 (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The dotted blue box (the bottom limit is not shown due to the scale of the figure) represents the consensus range of tolerance for Plant Sample 4 (x-axis) and NRC HEMP-1 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

Fig. 2-26. Laboratory means for total mercury in NRC HEMP-1 (Plant Sample 1) and SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (sample/sample comparison view). In this view, the individual laboratory mean for one sample (NRC HEMP-1) is compared to the individual laboratory mean for a second sample (SRM 1575a). The solid red box represents the NIST range of tolerance for the two samples, SRM 1575a (x-axis) and NRC HEMP-1 (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{NIST}| \le 2$. The dotted blue box represents the consensus range of tolerance for SRM 1575a (x-axis) and NRC HEMP-1 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{comm}| \le 2$.

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Fig. 2-27. Laboratory means for total mercury in Plant Sample 4 and SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (sample/sample comparison view). In this view, the individual laboratory mean for one sample (Plant Sample 4) is compared to the individual laboratory mean for a second sample (SRM 1575a). The solid red box represents the NIST range of tolerance for the two samples, SRM 1575a (x-axis) and Plant Sample 4 (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{NIST}| \le 2$. The dotted blue box (the bottom limit is not shown due to the scale of the figure) represents the consensus range of tolerance for SRM 1575a (x-axis) and Plant Sample 4 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{comm}| \le 2$.

Table 2-5. Data summary table for lead (Pb) in the hemp and control samples. 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, $|Z'_{\text{comm}}| > 2$. *Note*: This table spans three pages; the NIST values and consensus values are included on all three pages for convenience.

									Lead (Pb)							
		SRM 15	575a Trac <i>(Pinu</i> s	e Element s <i>taeda)</i> (1	s in Pine I ng/kg)	Needles	NRC	HEMP-1	(Plant Sar	nple 1) (n	ng/kg)		Plant S	Sample 4 (mg/kg)	
	Lab	Α	В	С	Avg	SD	Α	В	С	Avg	SD	Α	В	С	Avg	SD
	Target				0.1622	0.0291				3.562	0.456				0.1860	0.0520
	B001	0.158	0.15	0.144	0.1507	0.0070	2.7	2.65	2.53	2.627	0.087	0.172	0.178	0.226	0.1920	0.0296
	B003	2.38844	2.31929	2.47074	2.3928	0.0758	0.15602	0.15033	0.14606	0.151	0.005	0.19326	0.19347	0.17309	0.1866	0.0117
	B004	<1	1.31	<1	1.3100		2.54	2.59	2.78	2.637	0.127	<1	<1	<1		
	B006															
	B009															
	B012	0.148	0.15	0.142	0.1467	0.0042	2.2873	2.24812	2.21272	2.249	0.037	0.1916	0.2378	0.1738	0.2011	0.0330
	B013															
	B015	0.14	0.144	0.138	0.1407	0.0031	2.437	2.361	2.33	2.376	0.055	0.212	0.176	0.227	0.2050	0.0262
	B016	0.13	0.12	0.13	0.1267	0.0058	2.05	2.09	2.06	2.067	0.021	0.16	0.17	0.17	0.1667	0.0058
	B017	0.144	0.144	0.145	0 1 4 4 2	0.0007	2.242	2.244	2 2 2 1	2 2 2 0	0.007	0.004	0.017	0.0(0)	0.05(0	0.0250
	B018	0.144	0.144	0.145	0.1443	0.0006	2.242	2.244	2.231	2.239	0.007	0.284	0.21/	0.268	0.2563	0.0350
	B020	0.1520	0 1576	0 1594	0.1566	0.0024	2 2 2 0 1	2 5000	2 4675	2 470	0.006	0 1952	0.1094	0.1702	0 1976	0.0008
	D023	0.1339	0.1370	0.1384	0.1300	0.0024	2.3691	2.3808	2.4073	2.479	0.090	0.1855	0.1984	0.1/92	0.1870	0.0098
	B027	0.144	0.140	0.158	0.1455	0.0050	2.70	2.70	2.30	2.027	0.231	0.2	0.178	0.5	0.2921	0.1799
	B020	0.13	0.13	0.139	0.1330	0.0052	1.84	1.97	2.12	1 977	0.140	0.153	0.275	0.178	0 2020	0.0644
	B030	0.146	0.111	0.133	0.1300	0.0177	2.725	2.756	2.532	2.671	0.121	0.155	0.196	0.196	0.1887	0.0127
	B031	01110	01111	01100	0.1200	0.0177	21720	2.750	21002	2.071	0.1121	0.17.1	0.1770	0.1.70	011007	0.0127
sult	B035	0.14	0.139	0.142	0.1403	0.0015	2.561	2.525	2.616	2.567	0.046	0.23	0.263	0.177	0.2233	0.0434
Res	B037															
lai	B040															
vidı	B041	0.132	0.135	0.13	0.1323	0.0025	2.278	2.266	2.103	2.216	0.098	0.157	0.17	0.146	0.1577	0.0120
ibr	B043	0.145			0.1450		2.486			2.486		0.202			0.2020	
Ч	B044	0.14	0.14	0.14	0.1400	0.0000	2.79	2.15	2.35	2.430	0.327	0.17	0.17	0.17	0.1700	0.0000
	B049	0.154	0.152	0.151	0.1523	0.0015	2.68	2.8	2.5	2.660	0.151	0.202	0.181	0.247	0.2100	0.0337
	B052	0.15	0.14	0.14	0.1433	0.0058	2.31	2.53	2.18	2.340	0.177	0.17	0.21	0.18	0.1867	0.0208
	B057	0.17			0.1700		2.65			2.650		0.22			0.2200	
	B058	0.17	0.159	0.147	0.1587	0.0115	2.22	2.19	2.43	2.280	0.131	0.207	0.149	0.19	0.1820	0.0298
	B060	0.1489	0.15	0.1452	0.1480	0.0025	2.165	2.151	2.149	2.155	0.009	0.159	0.168	0.155	0.1607	0.0067
	B061	0.124	0.1377	0.1603	0.1407	0.0183	1.693	1.743	1.783	1.740	0.045	0.1981	0.1801	0.1809	0.1864	0.0102
	B062	0.16206	0.14703	0.12719	0.1454	0.0175	2.17553	2.28968	2.06035	2.175	0.115	0.23759	0.19408	0.18456	0.2054	0.0283
	B063															
	B064	2.554			2.5540		2.554			2.554		0.19			0.1900	
	B066	0.167	0.171	0.174	0.1707	0.0035	2.842	2.762	2.926	2.843	0.082	0.2	0.296	0.213	0.2363	0.0521
	B069	0.129	0.128	0.127	0.1280	0.0010	2.069	2.085	2.01	2.055	0.040	0.198	0.172	0.279	0.2163	0.0558
	B070	0.153	0.145	0.139	0.1457	0.00/0	1.897	1.796	2.215	1.969	0.219	0.269	0.215	0.1/4	0.2193	0.0476
	D079	0.1005	0.1550	0.14/3	0.1559	0.0005	2.4424	2.3085	2.2099	2.407	0.182	0.2432	0.1805	0.19/4	0.2090	0.0515
	D070															
	B079 B081	0.145	0.154	0.171	0.1567	0.0132	2 370	2.02	2 364	2 254	0.203	0.243	0.185	0.180	0.2057	0.0324
	B082	0.143	0.154	0.171	0.1563	0.0132	2.579	2.02	1 925	2.2.54	0.205	0.167	0.164	0.189	0.2037	0.0324
	B084	0.132	0.131	0.100	0.1287	0.0058	2.44	2.73	2.54	2.505	0.147	0.167	0.171	0.158	0.1653	0.0067
v		Consensu	ıs Mean		0.1491		Consensi	is Mean		2.413		Consensu	ıs Mean		0.2029	
ts		Consensu	is Standard	1 Deviation	0.0021		Consensu	is Standard	l Deviation	0.046		Consensu	is Standard	l Deviation	0.0032	
Ins		Maximun	1		3.7959		Maximun	n		6.840		Maximun	n		1.5581	
Our		Minimum			0.1027		Minimum	L		0.151		Minimum			0.1297	
0		Ν			72		Ν			83		Ν			78	

Table 2-5. continued

		Lead (Pb)														
		SRM 15	75a Trac (Pinus	e Element s <i>taeda)</i> (r	s in Pine I ng/kg)	Needles	NRC HEMP-1 (Plant Sample 1) (mg/kg)					Plant Sample 4 (mg/kg)				
	Lab	Α	В	С	Avg	SD	Α	В	С	Avg	SD	Α	В	С	Avg	SD
	Target				0.1622	0.0291				3.562	0.456				0.1860	0.0520
	B088	<1	3.9128	3.6789	3.7959	0.1654	4.5805	2.7228	3.3067	3.537	0.950	1.062	2.0613	1.5511	1.5581	0.4997
	B090	< 0.248	< 0.249	< 0.246			2.74	2.658	2.639	2.679	0.054	0.19	0.24	0.236	0.2220	0.0278
	B091															
	B094	< 0.35	< 0.35	< 0.35			< 4.87	< 4.87	< 4.87			0.2	0.2	0.2	0.2000	0.0000
	B095	0.142	0.141	0.147	0.1433	0.0032	2.671	2.419	2.714	2.601	0.159	0.207	1.011	0.181	0.4663	0.4719
	B097	0.192	0.195	0.202	0.1963	0.0051	2.65	2.68	2.69	2.673	0.021	0.215	0.23	0.204	0.2163	0.0131
	B100	0.13			0.1300		2.1			2.100		< 0.1				
	B102	0.13216	0.13539	0.13519	0.1342	0.0018	1.94532	2.07234	2.2278	2.082	0.141	0.16567	0.17527	0.23437	0.1918	0.0372
	B104	0.146	0.153	0.152	0.1503	0.0038	2.447	2.367	2.357	2.390	0.049	0.193	0.195	0.196	0.1947	0.0015
	B106	< 0.250	< 0.250	< 0.250			2.9	2.76	2.69	2.783	0.107	< 0.250	< 0.250	< 0.250		
	B107	0.1399	0.1415	0.1409	0.1408	0.0008	2.26	2.2112	2.238	2.236	0.024	0.1719	0.3155	0.1798	0.2224	0.0807
	B108															
	B109	0.14	0.14	0.14	0.1400		2.38	3.83	2.18	2.797	0.900	0.18	0.19	0.2	0.1900	0.0100
	B110	0.137	0.138	0.148	0.1410	0.0061	2.59	2.5	2.51	2.533	0.049	0.177	0.169	0.2	0.1820	0.0161
	B111															
lts	B112	0.166	0.167	0.164	0.1657	0.0015	1.835	1.94	2.142	1.972	0.156	0.222	0.224	0.221	0.2223	0.0015
esul	B113	0.116	0.098	0.094	0.1027	0.0117	1.852	1.803	1.759	1.805	0.047	0.125	0.13	0.134	0.1297	0.0045
I K	B116	0.16	0.155	0.157	0.1573	0.0025	2.904	3.052	2.977	2.978	0.074	0.197	0.268	0.204	0.2230	0.0391
ual	B119															
ivid	B120					_		_								_
pu	B122															
-	B125	0.347	0.332	0.385	0.3547	0.0273	6.66	6.8	7.06	6.840	0.203	0.512	0.512	0.54	0.5213	0.0162
	B129															
	B130	0.16	0.16	0.15	0.1567	0.0058	2.52	2.39	2.4	2.437	0.072	0.2	0.2	0.18	0.1933	0.0115
	B137	0.2262	0.248	0.2148	0.2297	0.0169	3.488	3.928	3.784	3.733	0.224	0.2248	0.2252	0.2574	0.2358	0.0187
	B139	0.155	0.155	0.164	0.1580	0.0052	2.78	2.69	2.81	2.760	0.062	0.198	0.394	0.205	0.2657	0.1112
	B141	0.137	0.138	0.13	0.1350	0.0044	2.37	2.38	2.234	2.328	0.082	0.211	0.167	0.177	0.1850	0.0231
	B142	0.134	0.142	0.136	0.1373	0.0042	4.618	5.09	4.317	4.675	0.390	0.33	0.398	0.309	0.3457	0.0465
	B146	0.212	0.156	0.152	0.1733	0.0335	2.53	2.57	2.54	2.547	0.021	0.216	0.214	0.54	0.3233	0.1876
	B147	0.14	0.13	0.13	0.1333	0.0058	1.96	2.01	1.87	1.947	0.071	0.23	0.16	0.16	0.1833	0.0404
	B148															
	B149	0.2656	0.248	0.248	0.2539	0.0102	5.337	5.42	5.2	5.319	0.111	0.3777	0.819	0.312	0.5029	0.2757
	B152															
	B153	<0.00145<0.00145<0.00145			0.1400 0.0151		3.9469	3.21	3.7833	3.647	0.387	< 0.00145	< 0.00145	< 0.00145		
	B159	0.144	0.123	0.153	0.1400	0.0154	2.081	2.237	2.372	2.230	0.146	0.188	0.192	0.17	0.1833	0.0117
	B160	0.19	0.281	0.188	0.2197	0.0531	2.801	2.741	2.694	2.745	0.054	0.22	0.362	0.243	0.2750	0.0762
	B161						1.75	1.6	1.8	1.717	0.104					
	B163	0.203	0.203	0.184	0.1967	0.0110	3.62	3.6	3.84	3.687	0.133	0.248	0.252	0.231	0.2437	0.0112
ity		Consensus Mean			0.1491		Consensus Mean			2.413		Consensu	is Mean		0.2029	
uut ults		Consensus Standard Deviation			0.0021		Consensus Standard Deviation			0.046		Consensus Standard Deviation			0.0032	
mm tesu		Maximum			3.7959		Maximun	1				Maximun	Maximum		1.5581	
Col R		Minimum			0.1027		Minimum			0.151		Minimum	Minimum			
		N			72		N			83		N 78				

Table 2-5. continu	ued.
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		Lead (Pb)																
		SRM 15	75a Trac <i>(Pinu</i> s)	e Element s <i>taeda)</i> (1	s in Pine I ng/kg)	Needles	NRC HEMP-1 (Plant Sample 1) (mg/kg)					Plant Sample 4 (mg/kg)						
	Lab	Α	В	С	Avg	SD	Α	В	С	Avg	SD	Α	В	С	Avg	SD		
	Target				0.1622	0.0291				3.562	0.456				0.1860	0.0520		
	B172																	
	B174	0.146	0.135	0.142	0.1410	0.0056	2.033	2.084	2.037	2.051	0.028	0.196	0.184	0.193	0.1910	0.0062		
	B176	0.1299	0.1315	0.1318	0.1311	0.0010	2.3967	2.6607	2.6409	2.566	0.147	0.1939	0.1905	0.1942	0.1929	0.0021		
	B178	0.174	0.162	0.156	0.1640	0.0092	2.65	2.55	2.69	2.630	0.072	0.184	0.172	0.207	0.1877	0.0178		
	B179	0.1528	0.1536	0.1549	0.1538	0.0011	2.714	2.625	2.71	2.683	0.050	0.1861	0.2263	0.1784	0.1969	0.0257		
	B180	< 0.1	< 0.1	< 0.1			2.222	2.275	2.371	2.289	0.076	0.252	0.23	0.217	0.2330	0.0177		
	B181	0.165			0.1650		2.722			2.722		0.199			0.1990			
	B183	0.164			0.1640		2.513			2.513		0.199			0.1990			
	B184	0.15	0.186	0.141	0.1590	0.0238	2.21	1.98	2.04	2.077	0.119	0.192	0.174	0.216	0.1940	0.0211		
	B186	0.141	0.136	0.145	0.1407	0.0045	2.12	2.01	2.07	2.067	0.055	0.171	0.175	0.191	0.1790	0.0106		
	B188																	
	B189	< 0.25	< 0.25	< 0.25			1.78	1.91	1.81	1.833	0.068	< 0.1	< 0.1	< 0.1				
	B190	0.1301	0.1306	0.2766	0.1791	0.0844	1.3929	1.4583	1.4907	1.447	0.050	0.4542	0.1277	0.1274	0.2364	0.1886		
	B192	0.142	0.143	0.144	0.1430	0.0010	2.56	2.49	2.51	2.520	0.036	0.199	0.178	0.198	0.1917	0.0118		
	B193	0.278	0.237	0.267	0.2607	0.0212	1.195	1.18	1.2	1.192	0.010	0.248	0.295	0.248	0.2637	0.0271		
	B195	0.159	0.155	0.142	0.1520	0.0089	2.59	2.49	2.47	2.517	0.064	0.188	0.199	0.19	0.1923	0.0059		
	B198																	
lts	B200	0.142	0.152	0.159	0.1510	0.0085	3.48	3.54	3.57	3.530	0.046	0.186	0.177	0.205	0.1893	0.0143		
ssul	B202	0.156	0.157	0.159	0.1573	0.0015	2.65	2.71	2.6	2.653	0.055	0.172	0.185	0.196	0.1843	0.0120		
ndividual Re	B203	0.164	0.157	0.16	0.1603	0.0035	2.65	2.81	2.77	2.743	0.083	0.195	0.246	0.195	0.2120	0.0294		
	B204	0.145	0.145	0.149	0.1463	0.0023	2.407	2.469	2.324	2.400	0.073	0.185	0.209	0.22	0.2047	0.0179		
	B205	0.141	0.144	0.143	0.1427	0.0015	2.168	2.319	2.574	2.354	0.205	0.178	0.162	0.194	0.1780	0.0160		
	B206	0.156	0.155	0.154	0.1550	0.0010	1.25	1.32	1.3	1.290	0.036	0.207	0.149	0.184	0.1800	0.0292		
	B208																	
	B211	< 0.07					< 0.07					< 0.07						
	B212	0.139	0.139	0.1445	0.1408	0.0032	2.655	2.455	2.753	2.621	0.152	0.1707	0.1843	0.2073	0.1874	0.0185		
	B213						2.9	2.7	2.6	2.733	0.153	0.18	0.19	0.24	0.2033	0.0321		
	B214																	
	B215	< 0.0990					1.2			1.200		0.144			0.1440			
	B216	<0.167	< 0.167	< 0.167			2.185	2.465	2.312	2.321	0.140	0.18	0.432	0.291	0.3010	0.1263		
	B217	0.15	0.141	0.139	0.1433	0.0059	2.585	2.714	2.541	2.613	0.090	0.181	0.172	0.177	0.1767	0.0045		
	B220															0.0404		
	B221	0.31	0.23	0.23	0.2567	0.0462	2.63	2.5	2.45	2.527	0.093	0.29	0.28	0.21	0.2600	0.0436		
	B222	0.15	0.16	0.15	0.1533	0.0058	2.175	2.194	2.307	2.225	0.071	0.018	0.19	0.4	0.2027	0.1913		
	B223	<0.5	<0.5	<0.5	0.1 (22	0.0050	2.3	2.52	2.38	2.400	0.111	<0.5	<0.5	<0.5	0.0000	0.01.52		
	B224	0.17	0.16	0.16	0.1633	0.0058	2.9	2.92	2.76	2.860	0.087	0.22	0.2	0.19	0.2033	0.0153		
	B226	0.14352	0.13583	0.13277	0.1374	0.0055	1.78846	1.81148	1.73625	1.779	0.039	0.1741	0.1756	0.17461	0.1748	0.0008		
	B228																	
	B230																	
	B231	0.1 (201	0.15055	0.1(107	0.1.610	0.0000	0.55007	0.50000	2 5002	2 (10	0.115	0.01005	0.10016	0.10056	0.0000	0.0077		
	B232	0.16301	0.15855	0.16137	0.1610	0.0023	2.57996	2.58329	2.7803	2.648	0.115	0.21085	0.19916	0.19956	0.2032	0.0066		
	B235	0.241	0.218	0.205	0.2213	0.0182	3.98	4.32	3.96	4.087	0.202	0.234	0.292	0.261	0.2623	0.0290		
iity		Consensu	Consensus Mean 0.1491					is Mean		2.413	5 Consensus M			n 0.2029				
aun		Consensu	Consensus Standard Deviation 0.0021				Consensus Standard Deviation 0.046					Consensus Standard Deviation 0.0032						
Comm Resu		Maximum	Maximum 3.7959				Maximum 6.840					Maximum 1.5581						
		Minimum 0.1027					Minimum			0.151		Minimum			0.1297			
		N 72					N 83						N 78					

Fig. 2-28. Lead in NRC HEMP-1 (Plant Sample 1) (data summary view – sample preparation method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed as indicated in the figure key. 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, $|Z'_{comm}| \leq 2$. 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, $|Z_{NIST}| \leq 2$.


Fig. 2-29. Lead in NRC HEMP-1 (Plant Sample 1) (data summary view – analytical method). In this view, individual analytical method data points are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$.



Fig. 2-30. Lead in Plant Sample 4 (data summary view – sample preparation method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 2-31. Lead in Plant Sample 4 (data summary view – analytical method). In this view, individual analytical method data points are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean that results in an acceptable Z_{NIST} score, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 2-32. Lead in SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (data summary view – sample preparation method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed as indicated in the figure key. 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, $|Z'_{comm}| \leq 2$. 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, $|Z_{NIST}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 2-33. Lead in SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (data summary view – analytical method). In this view, individual analytical method data points are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed as indicated in the figure key. 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, $|Z'_{comm}| \leq 2$. 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, $|Z_{NIST}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 2-34. Laboratory means for lead in NRC HEMP-1 (Plant Sample 1) and Plant Sample 4 (sample/sample comparison view). In this view, the individual laboratory mean for one sample (NRC HEMP-1) is compared to the individual laboratory mean for a second sample (Plant Sample 4). The solid red box (the right limit is not shown due to the scale of the figure) represents the NIST range of tolerance for the two samples, Plant Sample 4 (x-axis) and NRC HEMP-1 (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{NIST}| \le 2$. The dotted blue box represents the consensus range of tolerance for Plant Sample 4 (x-axis) and NRC HEMP-1 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{comm}| \le 2$.



Fig. 2-35. Laboratory means for lead in NRC HEMP-1 (Plant Sample 1) and SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (sample/sample comparison view). In this view, the individual laboratory mean for one sample (NRC HEMP-1) is compared to the individual laboratory mean for a second sample (SRM 1575a). The solid red box (the top limit is not shown due to the scale of the figure) represents the NIST range of tolerance for the two samples, SRM 1575a (x-axis) and NRC HEMP-1 (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The dotted blue box represents the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.



Exercise: CannaQAP Exercise 2, Measurand: lead

Fig. 2-36. Laboratory means for lead in Plant Sample 4 and SRM 1575a Trace Elements in Pine Needles (Pinus taeda) (sample/sample comparison view). In this view, the individual laboratory mean for one sample (Plant Sample 4) is compared to the individual laboratory mean for a second sample (SRM 1575a). The solid red box represents the NIST range of tolerance for the two samples, SRM 1575a (x-axis) and Plant Sample 4 (yaxis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 1575a (x-axis) and Plant Sample 4 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{comm}| \le 2$.

3. Beryllium, Cobalt, Chromium, Molybdenum, Manganese, Nickel, Selenium, Uranium, and Vanadium

3.1. Study Overview

The medicinal and recreational use of cannabis (hemp and marijuana) and cannabis-derived products continues to increase across the United States. While consumers may not be fully aware of potential safety concerns with product use, stakeholders in the cannabis community are concerned about toxic element contaminants in cannabis products. To fully understand the impact of these contaminants on consumers, analytical methods must accurately determine the levels of toxic elements in a variety of product types. Hemp is a known hyperaccumulator and historically has been used as a phytoremediator to remove toxic elements from soil. As a result, a significant potential exists for human exposure to toxic elements following hemp consumption. In addition to arsenic, cadmium, lead, and mercury described in the previous section, lesser-known toxic elements are also important to measure accurately as more states are requiring testing in finished cannabis products and raw ingredients. Several of the elements offered in this study are known carcinogens, including Cr, Se, and Be, with Be being one of the most toxic. Beryllium, Mn, and Ni are known to affect the lungs, while Co, Mo, Ni, and U are known to affect the heart, kidneys, thyroid, or joints [6]. Results for these elements in this study will be covered in the subsequent section.

3.2. Reporting Statistics

The enrollment and reporting statistics for the additional toxic elements are described in the table below for each analyte. Reported values may include non-quantitative results (zero or below LOQ).

		Percent Reporting Results			
<u>Analytes</u>	Number of Participants	NRC HEMP-1	Plant Sample 4	<u>SRM 1575a</u>	
Be	51	53 %	51 %	51 %	
Co	57	56 %	54 %	53 %	
Cr	75	64 %	61 %	61 %	
Mo	55	53 %	53 %	49 %	
Mn	58	59 %	59 %	57 %	
Ni	66	61 %	59 %	59 %	
Se	59	56 %	56 %	54 %	
U	45	47 %	42 %	40 %	
V	52	50 %	48 %	46 %	

Most laboratories reported using microwave digestion for determination of Be, Co, Cr, Mo, Mn, Ni, Se, U, and V in the two hemp samples and SRM 1575a (see tables below). Additional sample preparation details are summarized at the end of the report in the appendix.

Analytes	Microwave Digestion	<u>Hot Block</u> Digestion	Open Beaker Digestion	Solvent Extraction	<u>None/Not</u> Specified
Be	89 %		7 %		4 %
Co	84 %	3 %	6 %		6 %
Cr	85 %	6 %	4 %		4 %
Mo	83 %	3 %	7 %		7 %
Mn	79 %	6 %	9 %		6 %
Ni	82 %	5 %	5 %	3 %	5 %
Se	85 %	3 %	6 %		6 %
U	81 %	5 %	5 %		9 %
V	80 %	4 %	8 %		8 %

Percent Reporting Results

Most laboratories reported using ICP-MS for the determination of Be, Co, Cr, Mo, Mn, Ni, Se, U, and V in the two hemp samples and SRM 1575a (see tables below). Additional method details are summarized at the end of the report in the appendix.

Percent Reporting Results

Analytes	ICP-MS	ICP-OES	ID ICP-MS	<u>NAA</u>	<u>Other/Not</u> Specified
Be	89 %	7 %	4 %		
Со	81 %	13 %	3 %	3 %	%
Cr	83 %	8 %	4 %	2 %	2 %
Mo	86 %	7 %	3 %	3 %	%
Mn	79 %	15 %	3 %	3 %	%
Ni	79 %	10 %	5 %	3 %	3 %
Se	88 %	6 %	3 %	3 %	%
U	81 %	9 %	5 %	5 %	%
V	80 %	12 %	4 %	4 %	%

The between-laboratory variabilities for determination of Be, Co, Cr, Mo, Mn, Ni, Se, U, and V in the three samples are shown in the table below.

	Between-Laboratory Variability (% RSD)			
<u>Analytes</u>	NRC HEMP-1	Plant Sample 4	<u>SRM 1575a</u>	
Be	5 %	19 %	8 %	
Co	5 %	3 %	3 %	
Cr	4 %	3 %	3 %	
Mo	4 %	2 %	8 %	
Mn	3 %	2 %	2 %	
Ni	4 %	2 %	2 %	
Se	11 %	7 %	6 %	
U	7 %	10 %	6 %	
V	5 %	7 %	5 %	

The range of the variability of individual laboratory means for determination of Be, Co, Cr, Mo, Mn, Ni, Se, U, and V in the three samples are shown in the table below.

	Within-	Laboratory Variability (<u>% RSD)</u>
<u>Analytes</u>	NRC HEMP-1	Plant Sample 4	<u>SRM 1575a</u>
Be	1 % to 81 %	6 % to >100 %	3 % to 39 %
Co	1 % to 34 %	1 % to 23 %	0.9 % to 24 %
Cr	0.3 % to 37 %	1 % to 47 %	0.5 % to 47 %
Mo	0.9 % to 29 %	1 % to 21 %	2 % to 29 %
Mn	0.1 % to 75 %	1 % to 23 %	0.2 % to 11 %
Ni	0.5 % to 9 %	0.5 % to 24 %	0.6 % to >100 %
Se	1 % to 73 %	1 % to 75 %	0.5 % to 67 %
U	0.4 % to 17 %	5 % to 42 %	3 % to 20 %
V	0.4 % to 27 %	1 % to 27 %	0.9 % to 29 %

3.3. Study Results

The mass fractions (mg/kg) for Be, Co, Cr, Mn, Ni, Se, U, and V in the two hemp samples were determined by NIST, are described in Section 1. The NIST determined values and the reference values for SRM 1575a, where available, were used as the target values as summarized in **Table 3-1**.

Beryllium (Be)

• Figure 3-1 to Fig. 3-9 summarizes the reported results for Be in the two hemp samples. Data from participants submitting only one measurement were included in these figures as well as in Table 3-2 but were not included in the calculation of consensus statistics. The figures are summarized in the table below.

• The consensus range for Be in Plant Sample 4 was completely within the NIST range of tolerance. The consensus range for Be in NRC HEMP-1 overlapped the lower portion of the NIST range of tolerance. Quantitative and qualitative results reported by participating laboratories are summarized in the table below. No target value was provided for Be on the COA for SRM 1575a.

	Total Number of	Number of	Number of
~ 1	<u>Laboratories</u>	Laboratories Reporting	Laboratories Reporting
Samples	Reporting Results	Qualitative Results	Quantitative Results
NRC HEMP-1	27	3	24
Plant Sample 4	26	13	13
SRM 1575a	26	10	16

• Laboratories reporting outlying results with respect to the NIST range of tolerance and consensus range of tolerance ($|Z'_{comm}| > 2$) are summarized in the table below.

	Number (%) of	Number (%) of	Number (%) of
	Laboratory Means	Laboratory Means	Laboratories
	Outside NIST Range	Outside Consensus	<u>Reporting</u>
Samples	of Tolerance	Range of Tolerance	LOQs
NRC HEMP-1	13 (48 %)	4 (15 %)	3 (11 %)
Plant Sample 4	6 (23 %)	0 (0 %)	13 (50 %)
SRM 1575a	N/A	3 (12 %)	9 (35 %)

• The number of laboratories reporting results for Be outside consensus ranges of tolerance for multiple samples are summarized in the table below.

	<u>Number of Laboratory</u> Mean Values Outside	Percentage of Laboratory Mean Values Outside
<u>Samples</u>	both Consensus Ranges	both Consensus Ranges
NRC HEMP-1 / Plant Sample 4	3 out of 13	23 %
NRC HEMP-1 / SRM 1575a	5 out of 16	31 %
Plant Sample 4 / SRM 1575a	2 out of 13	15 %

Cobalt (Co)

• Figure 3-10 to Fig. 3-18 summarizes the reported results for Co in the two hemp samples. Data from participants submitting only one measurement were included in these figures as well as in Table 3-3 but were not included in the calculation of consensus statistics. The figures are summarized in the table below.

• The consensus ranges for Co in all three samples were completely within the NIST range of tolerance. Quantitative and qualitative results reported by participating laboratories are summarized in the table below.

	Total Number of	Number of	Number of
	Laboratories	Laboratories Reporting	Laboratories Reporting
<u>Samples</u>	Reporting Results	Qualitative Results	Quantitative Results
NRC HEMP-1	32	1	31
Plant Sample 4	31	4	27
SRM 1575a	30	6	24

Laboratories reporting outlying results with respect to the NIST range of tolerance and consensus range of tolerance ($|Z'_{comm}| > 2$) are summarized in the table below.

	Number (%) of Laboratory Means	<u>Number (%) of</u> <u>Laboratory Means</u>	Number (%) of Laboratories
Samples	Range of Tolerance	Range of Tolerance	<u>LOQs</u>
NRC HEMP-1	5 (16 %)	4 (13 %)	1 (3 %)
Plant Sample 4	2 (6 %)	3 (10 %)	4 (13 %)
SRM 1575a	6 (20 %)	5 (17 %)	6 (20 %)

• The number of laboratories reporting results for Co outside consensus ranges of tolerance for multiple samples are summarized in the table below.

<u>Samples</u>	<u>Number of Laboratory</u> <u>Mean Values Outside</u> <u>both Consensus Ranges</u>	Percentage of Laboratory Mean Values Outside both Consensus Ranges
NRC HEMP-1 / Plant Sample 4	5 out of 27	19 %
NRC HEMP-1 / SRM 1575a	6 out of 24	25 %
Plant Sample 4 / SRM 1575a	5 out of 24	21 %

Chromium (Cr)

- Figure 3-19 to Fig. 3-27 summarizes the reported results for Cr in the two hemp samples. Data from participants submitting only one measurement were included in these figures as well as in Table 3-4 but were not included in the calculation of consensus statistics. The figures are summarized in the table below.
- The consensus ranges for Cr in both hemp samples was completely within the NIST range of tolerance. Quantitative and qualitative results reported by participating laboratories are

summarized in the table below. The target value provided for Cr on the COA for SRM 1575a did not provide an uncertainty.

	Total Number of	Number of	Number of
Samples	Laboratories Reporting Results	<u>Laboratories Reporting</u> <u>Qualitative Results</u>	<u>Laboratories Reporting</u> <u>Quantitative Results</u>
NRC HEMP-1	48	0	48
Plant Sample 4	46	3	43
SRM 1575a	46	5	41

• Laboratories reporting outlying results with respect to the NIST range of tolerance and consensus range of tolerance ($|Z'_{comm}| > 2$) are summarized in the table below.

<u>Number (%) of</u> <u>Laboratory Means</u> Outside NIST	<u>Number (%) of</u> <u>Laboratory Means</u> Outside Consensus	Number (%) of Laboratories Reporting
Range of Tolerance	Range of Tolerance	LOQs
3 (6 %)	4 (8 %)	0 (0 %)
5 (11 %)	6 (13 %)	3 (7 %)
N/A	5 (11 %)	5 (11 %)
	<u>Number (%) of</u> <u>Laboratory Means</u> <u>Outside NIST</u> <u>Range of Tolerance</u> 3 (6 %) 5 (11 %) N/A	Number (%) of Laboratory Means Outside NIST Range of ToleranceNumber (%) of Laboratory Means Outside Consensus Range of Tolerance3 (6 %)4 (8 %)5 (11 %)6 (13 %)N/A5 (11 %)

• The number of laboratories reporting results for Cr outside consensus ranges of tolerance for multiple samples are summarized in the table below.

	Number of Laboratory	Percentage of Laboratory
	Mean Values Outside	Mean Values Outside
<u>Samples</u>	both Consensus Ranges	both Consensus Ranges
NRC HEMP-1 / Plant Sample 4	5 out of 43	12 %
NRC HEMP-1 / SRM 1575a	5 out of 41	12 %
Plant Sample 4 / SRM 1575a	5 out of 40	13 %

Molybdenum (Mo)

- Figure 3-28 to Fig. 3-36 summarizes the reported results for Mo in the two hemp samples. Data from participants submitting only one measurement were included in these figures as well as in Table 3-5 but were not included in the calculation of consensus statistics. The figures are summarized in the table below.
- The consensus ranges for Mo in both hemp samples was completely within the NIST range of tolerance. Quantitative and qualitative results reported by participating laboratories are summarized in the table below. No target value was provided for Mo on the COA for SRM 1575a.

	Total Number of	Number of	Number of
	Laboratories	Laboratories Reporting	Laboratories Reporting
Samples	Reporting Results	Qualitative Results	Quantitative Results
NRC HEMP-1	29	4	25
Plant Sample 4	29	5	24
SRM 1575a	27	11	16

	Number (%) of	Number (%) of	Number (%) of
	Laboratory Means	Laboratory Means	Laboratories
	Outside NIST	Outside Consensus	Reporting
Samples	Range of Tolerance	Range of Tolerance	LOQs
NRC HEMP-1	3 (10 %)	3 (10 %)	4 (14 %)
Plant Sample 4	12 (41 %)	2 (7 %)	5 (17 %)
SRM 1575a	N/A	2 (7 %)	11 (41 %)

• The number of laboratories reporting results for Mo outside consensus ranges of tolerance for multiple samples are summarized in the table below.

	Number of Laboratory	Percentage of Laboratory
<u>Samples</u>	Mean Values Outside both Consensus Ranges	Mean Values Outside both Consensus Ranges
NRC HEMP-1 / Plant Sample 4	3 out of 24	13 %
NRC HEMP-1 / SRM 1575a	2 out of 16	13 %
Plant Sample 4 / SRM 1575a	2 out of 16	13 %

Manganese (Mn)

- Figure 3-37 to Fig. 3-45 summarizes the reported results for Mn in the two hemp samples. Data from participants submitting only one measurement were included in these figures as well as in Table 3-6 but were not included in the calculation of consensus statistics. The figures are summarized in the table below.
- The consensus ranges for Mn in all three samples were completely within the NIST range of tolerances. Quantitative and qualitative results reported by participating laboratories are summarized in the table below.

	Total Number of	Number of	Number of
	Laboratories	Laboratories Reporting	Laboratories Reporting
<u>Samples</u>	Reporting Results	Qualitative Results	Quantitative Results
NRC HEMP-1	34	0	34
Plant Sample 4	34	0	34
SRM 1575a	33	0	33

	Number (%) of	Number (%) of	Number (%) of
	Laboratory Means	Laboratory Means	Laboratories
	Outside NIST	Outside Consensus	<u>Reporting</u>
Samples No. 1	Range of Tolerance	Range of Tolerance	LOQs
NRC HEMP-1	17 (50 %)	4 (12 %)	0 (0 %)
Plant Sample 4	10 (29 %)	1 (3 %)	0 (0 %)
SRM 1575a	11 (33 %)	4 (12 %)	0 (0 %)

• The number of laboratories reporting results for Mn outside consensus ranges of tolerance for multiple samples are summarized in the table below.

	Number of Laboratory	Percentage of Laboratory
	Mean Values Outside	Mean Values Outside
Samples	both Consensus Ranges	both Consensus Ranges
NRC HEMP-1 / Plant Sample 4	7 out of 34	21 %
NRC HEMP-1 / SRM 1575a	7 out of 33	21 %
Plant Sample 4 / SRM 1575a	4 out of 33	12 %

Nickel (Ni)

- Figure 3-46 to Fig. 3-54 summarizes the reported results for Ni in the two hemp samples. Data from participants submitting only one measurement were included in these figures as well as in Table 3-7 but were not included in the calculation of consensus statistics. The figures are summarized in the table below.
- The consensus ranges for Ni in all three samples were completely within the NIST range of tolerances. Quantitative and qualitative results reported by participating laboratories are summarized in the table below.

	Total Number of	Number of	Number of
	Laboratories	Laboratories Reporting	Laboratories Reporting
Samples	Reporting Results	Qualitative Results	Quantitative Results
NRC HEMP-1	40	3	37
Plant Sample 4	39	2	37
SRM 1575a	39	3	36

	Number (%) of	Number (%) of	Number (%) of
	Laboratory Means	Laboratory Means	Laboratories
	Outside NIST	Outside Consensus	Reporting
Samples 1997	Range of Tolerance	Range of Tolerance	LOQs
NRC HEMP-1	4 (10 %)	7 (18 %)	3 (8 %)
Plant Sample 4	2 (5 %)	4 (10 %)	2 (5 %)
SRM 1575a	8 (21 %)	7 (18 %)	3 (8 %)

• The number of laboratories reporting results for Ni outside consensus ranges of tolerance for multiple samples are summarized in the table below.

	<u>Number of Laboratory</u> Mean Values Outside	Percentage of Laboratory Mean Values Outside
Samples	both Consensus Ranges	both Consensus Ranges
NRC HEMP-1 / Plant Sample 4	7 out of 36	19 %
NRC HEMP-1 / SRM 1575a	10 out of 36	28 %
Plant Sample 4 / SRM 1575a	6 out of 35	17 %

Selenium (Se)

- Figure 3-55 to Fig. 3-63 summarizes the reported results for Se in the two hemp samples. Data from participants submitting only one measurement were included in these figures as well as in Table 3-8 but were not included in the calculation of consensus statistics. The figures are summarized in the table below.
- The consensus range for Se in all three samples overlapped the upper portions of the NIST range of tolerances. Quantitative and qualitative results reported by participating laboratories are summarized in the table below.

	Total Number of	Number of	Number of
	Laboratories	Laboratories Reporting	Laboratories Reporting
<u>Samples</u>	Reporting Results	Qualitative Results	Quantitative Results
NRC HEMP-1	33	8	25
Plant Sample 4	33	10	23
SRM 1575a	32	9	23

	Number (%) of	Number (%) of	Number (%) of
	Laboratory Means	Laboratory Means	Laboratories
	Outside NIST	Outside Consensus	Reporting
<u>Samples</u>	Range of Tolerance	Range of Tolerance	<u>LOQs</u>
NRC HEMP-1	15 (45 %)	1 (3 %)	8 (24 %)
Plant Sample 4	13 (39 %)	3 (9 %)	10 (30 %)
SRM 1575a	17 (53 %)	5 (16 %)	9 (28 %)

• The number of laboratories reporting results for Se outside consensus ranges of tolerance for multiple samples are summarized in the table below.

	<u>Number of Laboratory</u> <u>Mean Values Outside</u>	Percentage of Laboratory Mean Values Outside
Samples	both Consensus Ranges	both Consensus Ranges
NRC HEMP-1 / Plant Sample 4	4 of 22	18 %
NRC HEMP-1 / SRM 1575a	5 of 21	24 %
Plant Sample 4 / SRM 1575a	5 of 21	24 %

Uranium (U)

- Figure 3-64 to Fig. 3-72 summarizes the reported results for U in the two hemp samples. Data from participants submitting only one measurement were included in these figures as well as in Table 3-9 but were not included in the calculation of consensus statistics. The figures are summarized in the table below.
- The consensus ranges for U in both hemp samples was completely within the NIST range of tolerances. Quantitative and qualitative results reported by participating laboratories are summarized in the table below. No target value was provided for U on the COA for SRM 1575a.

	Total Number of	Number of	Number of
	Laboratories	Laboratories Reporting	Laboratories Reporting
Samples	Reporting Results	Qualitative Results	Quantitative Results
NRC HEMP-1	21	2	19
Plant Sample 4	19	7	12
SRM 1575a	18	6	12

	Number (%) of	<u>Number (%) of</u>	Number (%) of
	Laboratory Means	Laboratory Means	Laboratories
	Outside NIST	Outside Consensus	Reporting
Samples 199	Range of Tolerance	Range of Tolerance	LOQs
NRC HEMP-1	5 (24 %)	3 (14 %)	2 (10 %)
Plant Sample 4	0 (0 %)	0 (0 %)	7 (37 %)
SRM 1575a	N/A	0 (0 %)	6 (33 %)

• The number of laboratories reporting results for U outside consensus ranges of tolerance for multiple samples are summarized in the table below.

	<u>Number of Laboratory</u> Mean Values Outside	Percentage of Laboratory Mean Values Outside
Samples	both Consensus Ranges	both Consensus Ranges
NRC HEMP-1 / Plant Sample 4	0 out of 12	0 %
NRC HEMP-1 / SRM 1575a	1 out of 12	8 %
Plant Sample 4 / SRM 1575a	0 out of 12	0 %

Vanadium (V)

- Figure 3-73 to Fig. 3-81 summarizes the reported results for V in the two hemp samples. Data from participants submitting only one measurement were included in these figures as well as in Table 3-10 but were not included in the calculation of consensus statistics. The figures are summarized in the table below.
- The consensus range for V in Plant Sample 1 was completely within the NIST range of tolerances. The consensus range for V in NRC HEMP-1 overlapped the lower portion of the NIST range of tolerance. Quantitative and qualitative results reported by participating laboratories are summarized in the table below. No target value was provided for V on the COA for SRM 1575a.

	Total Number of	Number of	Number of
	Laboratories	Laboratories Reporting	Laboratories Reporting
Samples	Reporting Results	Qualitative Results	Quantitative Results
NRC HEMP-1	26	1	25
Plant Sample 4	25	5	20
SRM 1575a	24	6	18

	Number (%) of	Number (%) of	Number (%) of
	Laboratory Means	Laboratory Means	Laboratories
	Outside NIST	Outside Consensus	<u>Reporting</u>
Samples	Range of Tolerance	Range of Tolerance	LOQs
NRC HEMP-1	12 (46 %)	3 (12 %)	1 (4 %)
Plant Sample 4	8 (32 %)	2 (8 %)	5 (20 %)
SRM 1575a	N/A	2 (8 %)	6 (25 %)

• The number of laboratories reporting results for V outside consensus ranges of tolerance for multiple samples are summarized in the table below.

	Number of Laboratory Mean Values Outside	Percentage of Laboratory Mean Values Outside both
Samples	both Consensus Ranges	<u>Consensus Ranges</u>
NRC HEMP-1 / Plant Sample 4	2 out of 19	11 %
NRC HEMP-1 / SRM 1575a	3 out of 18	17 %
Plant Sample 4 / SRM 1575a	2 out of 18	11 %

3.4. Study Discussion and Technical Recommendations

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

- Most laboratories used microwave digestion as their sample preparation method and ICP-MS as their analytical method. With so few other techniques reported, no clear bias was observed showing one technique performed better than another.
 - ICP-MS is a good technique for these elements with most ionizing very efficiently, > 90 %, in the Ar plasma [7]. Ionization efficiency can be calculated using the Saha-Eggert Equation [8].
 - ICP-OES can also be used effectively, when elemental mass fractions are in the parts per million (ppm) range (Mn, V).
 - In some cases, open beaker digestion or hot block digestion approaches did not perform as well as microwave digestion (Be, Co, Se).

- In some cases, where ICP-OES was employed, the sample-to-sample variability was great (Cr, Mn in NRC HEMP-1).
- Some elements (Be, Co, Mn, V) are monoisotopic, so reporting ID-ICP-MS as the analytical method is not a practicable for these elements. Vanadium has a radioactive isotope (V⁵⁰) which could be used in laboratories with appropriate facilities.
- Sample preparation methods should be well established before analyzing unknown samples. Use established quality control materials (SRM, CRM, RM and in-house materials when not commercially available) and established methods of analysis should be used whenever possible.
- The very low levels of toxic elements are challenging and laboratories must balance many factors when deciding on the best methods to use.
 - Detection of the analytes in the sample may be improved by limiting the number of dilutions performed, however matrix effects may become more significant with fewer dilutions.
 - The method of standard additions may improve LOQs, accuracy, and precision, but is time consuming.
 - Analysis of an appropriate number of procedural blanks is critical in the determination of LOQ or when trying to reduce within-laboratory variability. Analysis of many blanks (usually the number of blanks equal the number of samples, or 10 when determining LOQ) can provide information about whether the source of variability is from the sample or from the sample preparation method.
- Calibration curves must be linear when used for quantitation.
 - Standards must include the lowest and highest values expected to be measured in the sample solutions. Several standards in between the highest and lowest standards should also be included to ensure linearity.
 - Accurate measurements can be achieved by making sure the sample concentrations fall within the middle of the calibration curve.
 - The calibration curve must be checked for linearity at the point of the expected sample concentrations.
- All results should be reported accurately.
 - Zero is not a quantity that can be measured. If values are below LOQ, results should be reported as such. A more appropriate result would be to report that a value is below the LOQ (e.g., "< 0.02 %").
 - Laboratories reporting results flagged as outliers should check for calculation errors when preliminary data tables are sent for inspection. One example is to confirm that factors for all dilutions have been properly tabulated or that results are reported in the requested units.

Beryllium (Be)

- Beryllium concentrations were very low in Plant Sample 4 and SRM 1575a. In both materials, a greater percentage of laboratories reported results below LOQ than for NRC HEMP-1.
- Laboratories that reported using open beaker digestion ICP-OES were unable to report a quantitative value for Be.
- Beryllium dissolves in HNO₃, and once dissolved it should be stored in dilute HNO₃ in plastic bottles for better stability.
 - Low concentrations of Be (< 100 μ g/g) are not stable for very long in solution.
 - Beryllium is only about 75 % ionized in the Ar plasma.

Cobalt (Co)

- Most laboratories were able to measure Co in both hemp samples well, but several laboratories had problems measuring Co in SRM 1575a. Pine needles may have been harder to digest, and the level of Co in the SRM was 40 times lower than in NRC HEMP-1 and four times lower than in Plant Sample 4.
- The high temperatures of a microwave digestion system should ensure complete digestion of the materials prior to analysis.
 - Difficulty in the digestion of samples can cause bias and/or increased variability between samples. Use of higher temperatures, a small amount of HF, or the addition of oxidizing reagents (peroxide (H₂O₂), perchloric acid (HClO₄)) will ensure complete digestion of the materials prior to analysis.
 - Cobalt is stable in a dilute HNO₃ solution after samples have been completely digested.
- Where laboratories reported results closer to the NIST target range for one material than for a second material, the differences in the two matrices or in the concentration levels may have resulted from difficulties in preparation and analysis.
- Cobalt is relatively immune from interferences when using ICP-MS. Collision cell technology using He can be used to minimize any interferences that may be found.

Chromium (Cr)

- The consensus ranges for Cr were very small for all three materials.
- Several laboratories reporting use of open beaker digestion or ICP-OES had either large within laboratory variability or reported values below their LOQ, indicating that these approaches were not successful for Cr in these materials.
- Incomplete digestion of the hemp samples may result in large within laboratory variability.
- Chromium is soluble in most acids but is most stable in dilute HNO₃.
- Spectral/isobaric interferences can make Cr difficult to measure accurately by ICP-MS.
 - High concentrations of certain elements (Ar, C, Cl, S) may cause interferences, including Cl⁻ or S⁻ containing compounds. A scan of the sample before analysis will indicate any potential interferences in the sample that will need to be addressed.
 - Collision cell technology can be used to minimize molecular interferences that may be found in these three materials.

Molybdenum (Mo)

- Most laboratories were able to measure Mo well.
- Addition of a trace amount of HF to sample solutions will increase stability of Mo. If HF is used, sample solutions must be stored in plastic rather than glass containers.
- Molydenum oxide (MoO) isotopic patterns can be confused with Cd isotopes.
- Some Mo isotopes may be subject to interference from zirconium hydride (ZrH). This may cause problems if hydrogen (H₂) is the choice of gas used for collision cell.

Manganese (Mn)

• For Mn determination, most laboratories used ICP-MS. ICP-OES is also a good choice for the analytical method since these samples had high concentrations of Mn. Two of the laboratories that used ICP-OES (with different sample preparation methods) for NRC HEMP-1 had a larger sample-to-sample variability.

- Manganese is easily digested, and volatile loss of Mn is not a concern. However, the purity of the acid should be checked for trace work analysis.
- Mn is stable in dilute HNO₃.
- A linear trend was observed in Fig. 3-43 for the comparison of NRC HEMP-1 and Plant Sample 4.
 - A linear trend may indicate a problem with the calibration curve.
 - There may have been an error with the calibration curve towards the lower concentration levels since there are many data points located outside of the NIST range of tolerance for both materials. If the calibration curve is nonlinear at the lower section of the curve, it will lead to a bias in value reporting.
- Some laboratories reported high within laboratory variability in either one or more materials, especially noticeable in Plant Sample 4 where laboratories reported high concentration values and in the ICP-OES reported values for NRC HEMP-1.
- For samples analyzed by ICP-MS, collision cell technology can be used to minimize molecular interferences that may be found in these three materials. Materials with high K levels can form KO which interferes with Mn determination.

Nickel (Ni)

- For Ni determination, most laboratories used ICP-MS. The Ni levels were high in these samples, so ICP-OES would also be an appropriate analytical method.
- Most laboratories did well measuring Ni in the samples with the majority of the laboratories overlapping, or coming close, to the consensus mean.
- Nickel is not easily soluble in concentrated HNO₃ but once the sample material is in solution Ni is stable in dilute HNO₃.
- A linear trend was observed in Fig. 3-52 for the comparison of NRC HEMP-1 and Plant Sample 4.
 - There may have been a problem with the calibration curve, especially at the lower concentrations because a lot of laboratories appear to be below the NIST range of tolerance in this figure.
- Greater within laboratory variability was seen where laboratories reported greater concentration levels for SRM 1575a.
 - There may have been more difficulty in either the sample preparation or the analytical measurements of SRM 1575a. Samples may not have been completely dissolved creating greater variability in the measurements.
- Ni⁶⁰ is the preferred isotope for Ni determination, as Ni⁵⁸ suffers interferences from iron (Fe) and argon oxide (ArO). Additionally, materials high in calcium (Ca) can form calcium oxide (CaO) interferences. Collision cell technology can be used to minimize interferences that may be found in these three materials.

Selenium (Se)

- Many laboratories reported values above both the consensus mean and the NIST target range for NRC HEMP-1 and SRM 1575a. About half of the laboratories reported values above the consensus mean and the NIST target range for Plant Sample 4.
 - High values may be from signal enhancement biases due to interferences.
- Selenium is highly volatile and may be lost during open beaker digestion.

- Closed-vessel digestions should be opened with care ensuring that no Se is lost as a result of inadvertent venting.
- Once in solution, Se is stable in dilute HNO₃.
- A linear trend was observed in Fig. 3-63 for the comparison of Plant Sample 4 and SRM 1575a.
 - This may be caused by a calibration issue.
- A trend was not apparent in either Fig. 3-61 or Fig. 3-62.
 - Selenium is only 35 % ionized in the Ar plasma, leading to poor sensitivity.
 - Most Se isotopes suffer isobaric overlap (Se⁷⁴, Se⁷⁶, Se⁷⁸, Se⁸⁰, Se⁸²) or from polyatomic interferences, mainly from Ar² (Se⁷⁶, Se⁷⁸, Se⁸⁰) causing signal suppression or enhancement leading to bias of the measurements. One of the most useful isotopes for quantitation is Se⁷⁷, but it suffers from argon chloride (ArCl) interference in high Cl matrices.
 - Collision cell technology can be used to minimize interferences that may be found in these three materials.

Uranium (U)

- For Plant Sample 4 and SRM 1575a, ICP-MS were the only analytical methods reported that were able to provide quantitative results.
- Uranium is stable in dilute HNO₃.
- Uranium concentrations were very low in Plant Sample 4 and SRM 1575a. Laboratories reported a greater percentage of results that were below LOQ in these two samples.
- A linear trend was observed in Fig. 3-71 for the comparison of NRC HEMP-1 and SRM 1575a.
 - A linear trend may indicate a problem with the calibration curve.
 - The large difference in U concentrations of these two samples may also have been the cause of this trend. If sample solutions are not diluted to the same approximate concentrations for analysis, samples values may be calculated on a nonlinear portion of the calibration curve leading to a bias in value reporting.
- A trend was not apparent in either Fig. 3-70 or Fig. 3-72. However, many laboratories reported high values for U in NRC HEMP-1.

Vanadium (V)

- For the greater concentration of V in NRC HEMP-1, ICP-OES may also be a practical analytical method since V is very sensitive by ICP-OES.
- Once in solution, V is stable in dilute acids.
- A linear trend was observed in **Fig. 3-79** for the comparison of NRC HEMP-1 and Plant Sample 4 and in **Fig. 3-81** for the comparison of SRM 1575a and Plant Sample 4.
 - A linear trend may indicate a problem with the calibration curve.
 - The large difference in V concentrations of these two samples may also have been the cause of this trend.
- If V is measured by ICP-MS, it should be analyzed using the V⁵¹ isotope. Vanadium is considered monoisotopic, as V⁵⁰ is radioactive. in addition, V⁵⁰ has low abundance and has known interferences.

Table 3-1. Individualized data summary table (NIST) for Be, Co, Cr, Mo, Mn, Ni, Se, U, and V in the hemp and control samples.

	Lab Code:	NIST		1. Your	Results		2. (Community l	Results	3. T	arget
Analyte	Sample	Units	x _i	\mathbf{s}_{i}	Z' _{comm}	Z _{NIST}	Ν	x*	s*	X _{NIST}	U
Beryllium (Be)	SRM 1575a Trace Elements in Pine Needles (Pinus taeda)	mg/kg					15	0.0070	0.0006		
Beryllium (Be)	mg/kg	0.242	0.023			24	0.205	0.010	0.242	0.023	
Beryllium (Be)	Plant Sample 4	mg/kg	0.002	0.001			11	0.00274	0.00052	0.002	0.001
Cobalt (Co)	SRM 1575a Trace Elements in Pine Needles (Pinus taeda)	mg/kg	0.0592	0.0039			23	0.0589	0.0015	0.0592	0.0039
Cobalt (Co)	NRC HEMP-1 (Plant Sample 1)	mg/kg	1.854	0.340			30	1.720	0.081	1.854	0.340
Cobalt (Co)	Plant Sample 4	mg/kg	0.2396	0.1172			26	0.1722	0.0043	0.2396	0.1172
Chromium (Cr)	SRM 1575a Trace Elements in Pine Needles (Pinus taeda)	mg/kg	0.388				36	0.344	0.010	0.388	
Chromium (Cr)	NRC HEMP-1 (Plant Sample 1)	mg/kg	12.72	4.49			45	8.68	0.33	12.72	4.49
Chromium (Cr)	Plant Sample 4	mg/kg	0.307	0.220			39	0.472	0.014	0.307	0.220
Manganese (Mn)	SRM 1575a Trace Elements in Pine Needles (Pinus taeda)	mg/kg	473.8	23.3			29	458.0	8.3	473.8	23.3
Manganese (Mn)	NRC HEMP-1 (Plant Sample 1)	mg/kg	401.0	26.4			32	366.9	9.4	401.0	26.4
Manganese (Mn)	Plant Sample 4	mg/kg	142.0	7.6			32	135.4	2.4	142.0	7.6
Molybdenum (Mo)	SRM 1575a Trace Elements in Pine Needles (Pinus taeda)	mg/kg					15	0.0184	0.0015		
Molybdenum (Mo)	NRC HEMP-1 (Plant Sample 1)	mg/kg	0.661	0.096			24	0.612	0.022	0.661	0.096
Molybdenum (Mo)	Plant Sample 4	mg/kg	0.3180	0.0120			24	0.3244	0.0073	0.3180	0.0120
Nickel (Ni)	SRM 1575a Trace Elements in Pine Needles (Pinus taeda)	mg/kg	1.427	0.194			35	1.332	0.029	1.427	0.194
Nickel (Ni)	NRC HEMP-1 (Plant Sample 1)	mg/kg	7.11	4.36			36	5.67	0.21	7.11	4.36
Nickel (Ni)	Plant Sample 4	mg/kg	3.670	1.320			35	3.262	0.062	3.670	1.320
Selenium (Se)	SRM 1575a Trace Elements in Pine Needles (Pinus taeda)	mg/kg	0.0961	0.0078			23	0.1191	0.0069	0.0961	0.0078
Selenium (Se)	NRC HEMP-1 (Plant Sample 1)	mg/kg	0.305	0.060			25	0.462	0.051	0.305	0.060
Selenium (Se)	Plant Sample 4	mg/kg	0.8100	0.0120			23	0.1058	0.0077	0.8100	0.0120
Uranium (U)	SRM 1575a Trace Elements in Pine Needles (Pinus taeda)	mg/kg					12	0.00525	0.00034		
Uranium (U)	NRC HEMP-1 (Plant Sample 1)	mg/kg	0.454	0.120			19	0.268	0.020	0.454	0.120
Uranium (U)	Plant Sample 4	mg/kg	0.00426	0.00120			10	0.00326	0.00031	0.00426	0.00120
Vanadium (V)	SRM 1575a Trace Elements in Pine Needles (Pinus taeda)	mg/kg					17	0.1041	0.0050		
Vanadium (V)	NRC HEMP-1 (Plant Sample 1)	mg/kg	16.70	1.70			24	13.83	0.70	16.70	1.70
Vanadium (V)	Plant Sample 4	mg/kg	0.233	0.033			20	0.208	0.015	0.233	0.033
	*		x _i Mean of	reported va	alues	N	Number	of quantitativ	re X _N	VIST NIST-as	sessed va
			- Ct11	4	£	- 1	,	- 1		17 1 1	, .

National Institute of Standards and Technology

x* Robust mean of reported

about the NIST-assessed value

consensus

 Z'_{comm} Z'-score with respect to community Z_{NIST} Z-score with respect to NIST value

values

s* Robust standard deviation

Table 3-2. Data summary table for beryllium (Be) in the hemp and control samples. 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 result in an unacceptable Z'_{comm} score, $|Z'_{comm}| > 2$. *Note*: This table spans two pages; the NIST values and consensus values are included on both pages for convenience.

		Beryllium (Be)														
		SRM 15	75a Trac <i>(Pinus</i>	e Element s <i>taeda)</i> (1	s in Pine ng/kg)	Needles	NRC HEMP-1 (Plant Sample 1) (mg/kg)				Plant Sample 4 (mg/kg)					
	Lab	Α	В	С	Avg	SD	Α	В	С	Avg	SD	Α	В	С	Avg	SD
	Target									0.242	0.023				0.002	0.001
	B001	0.0069	0.0062	0.0055	0.0062	0.0007	0.165	0.188	0.199	0.184	0.017	0.0021	0.0028	0.0021	0.00233	0.00040
	B003	0.12905	0.16962	0.16547	0.1547	0.0223	0.00549	0.00364	0.00041	0.003	0.003	0.0002	0.00203		0.00111	0.00130
	B004	<1	<1	<1			<1	<1	<1			<1	<1	<1		
	B006															
	B017															
	B018	< 0.055	< 0.055	< 0.055			0.181	0.19	0.19	0.187	0.005	< 0.064	< 0.064	< 0.064		
	B020															
	B028															
	B031															
ts	B035	< 0.1	< 0.1	< 0.1			0.192	0.204	0.193	0.196	0.007	< 0.1	< 0.1	< 0.1		
sult	B037															
idividual Re	B049						0.243	0.266	0.225	0.245	0.021					
	B058	< 0.0784	< 0.0783	< 0.0764			0.174	0.172	0.185	0.177	0.007	< 0.0391	< 0.0381	< 0.0384		
	B061															
	B066	0.005	0.006	0.006	0.0057	0.0006	0.206	0.204	0.199	0.203	0.004	0.002	0.003	0.003	0.00267	0.00058
Ţ	B078															
	B079															
	B084															
	B088	<1	<1	<1			<1	<1	<1			<1	<1	<1		
	B095	0.005	0.004	0.006	0.0050	0.0010	0.158	0.138	0.152	0.149	0.010	0.001	0.002	0.001	0.00133	0.00058
	B097	< 0.05	< 0.05	< 0.05			0.205	0.546	0.282	0.344	0.179	< 0.05	< 0.05	< 0.05		
	B102															
	B111															
	B112	0.005	0.005	0.006	0.0053	0.0006	0.168	0.168	0.15	0.162	0.010	< 0.003	< 0.003	< 0.003		
	B113	0	0	0	0.0000											
	B119															
Ϋ́.		Consensu	ıs Mean		0.0070		Consensu	ıs Mean		0.205		Consense	us Mean		0.00274	
uni Its		Consensu	ıs Standar	d Deviatio	0.0006		Consensu	ıs Standar	d Deviation	0.010		Consense	us Standar	d Deviatio	0.00052	
Im		Maximun	1		0.1547		Maximun	1		0.344		Maximur	n		0.00683	
Re		Minimum	L		0.0000		Minimum			0.003		Minimum	Minimum		0.00000	
Ŭ		Ν			15		Ν			24 N		Ν	N		11	

	Table	3-2.	continued.
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		SRM 1575a Trace Elements in Pine Needles <i>(Pinus taeda)</i> (mg/kg)						NRC HEMP-1 (Plant Sample 1) (mg/kg)				Plant Sample 4 (mg/kg)				
	Lab	Α	В	С	Avg	SD	Α	В	С	Avg	SD	Α	В	С	Avg	SD
	Target									0.242	0.023				0.002	0.001
	B122															
	B125	0.0125	0.0103	0.0054	0.0094	0.0036	0.0396	0.0339	0.042	0.039	0.004	0.00639	0.00685	0.00724	0.00683	0.00043
	B130	0.01	0.01	0.01	0.0100		0.2	0.19	0.2	0.197	0.006	0	0	0	0.00000	
	B139	< 0.007	< 0.007	< 0.007			0.244	0.275	0.243	0.254	0.018	< 0.007	< 0.007	< 0.007		
	B141	0.006	0.005	0.007	0.0060	0.0010	0.231	0.223	0.222	0.225	0.005	0.006	0.001	0.002	0.00300	0.00265
	B142															
	B148															
	B161															
	B163	0.0074	0.00699	0.00696	0.0071	0.0002	0.257	0.253	0.271	0.260	0.009	0.00347	0.00285	0.00255	0.00296	0.00047
ults	B179	0.0065	0.0065	0.006	0.0063	0.0003	0.207	0.198	0.208	0.204	0.006	0.00596	0.00594	0.00596	0.00595	
Res	B180	0.015	0.012	0.012	0.0130	0.0017	0.171	0.168	0.177	0.172	0.005	< 0.01	< 0.01	< 0.01		
all	B200	0.0059	0.0075	0.0068	0.0067	0.0008	0.2892	0.3128	0.3396	0.314	0.025	0.0041	0.0035	0.0036	0.00373	0.00032
Individu	B202															
	B203	0.005	0.008	0.005	0.0060	0.0017	0.206	0.216	0.211	0.211	0.005	0.002	0.001	0.002	0.00167	0.00058
	B208															
	B211	< 0.136					< 0.136					< 0.136				
	B213						0.22	0.22	0.19	0.210	0.017	< 0.02	< 0.02	< 0.02		
	B214															
	B221	< 0.01	< 0.01	< 0.01			0.158	0.15	0.149	0.152	0.005	< 0.01	< 0.01	< 0.01		
	B222	< 0.01	0.019	0.012	0.0155	0.0049	0.159	0.172	0.196	0.176	0.019	< 0.01	< 0.01	< 0.01		
	B228															
	B230															
	B231															
	B232	0.00683	0.00504	0.00667	0.0062	0.0010	0.18047	0.19395	0.2012	0.192	0.011	0.00353	0.00261	0.00167	0.00261	0.00093
	B235	0.00831	0.00681	0.00693	0.0074	0.0008	0.218	0.297	0.286	0.267	0.043	0.00252	0.00246	0.00342	0.00280	0.00054
ity		Consense	us Mean		0.0070		Consens	us Mean		0.205		Consens	us Mean		0.00274	
nun ults		Consense	us Standar	d Deviation	0.0006		Consens	us Standar	d Deviatio	0.010		Consens	us Standar	d Deviatio	0.00052	
lmn Resi		Maximur	n		0.1547		Maximur	n		0.344		Maximur	n		0.00683	
Co H		Minimum	1		0.0000		Minimum	1		0.003		Minimum	1		0.00000	
		N			15		N			24		N			11	



Fig. 3-1. Beryllium in NRC HEMP-1 (Plant Sample 1) (data summary view – sample preparation method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus the overlapping of the 95 % confidence interval for the consensus the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 3-2. Beryllium in NRC HEMP-1 (Plant Sample 1) (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean that results in an acceptable Z_{NIST} score, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 3-3. Beryllium in Plant Sample 4 (data summary view – sample preparation method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 3-4. Beryllium in Plant Sample 4 (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. The red shaded region represents the Interval for the consensus mean that result is uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 3-5. Beryllium in SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (data summary view – sample preparation method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed as indicated in the figure key. 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, $|Z'_{comm}| \leq 2$. A NIST value has not been determined in this material.



Fig. 3-6. Beryllium in SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. A NIST value has not been determined in this material.



Exercise: CannaQAP Exercise 2, Measurand: beryllium

Fig. 3-7. Laboratory means for beryllium in NRC HEMP-1 (Plant Sample 1) and Plant Sample 4 (sample/sample comparison view). In this view, the individual laboratory mean for one sample (NRC HEMP-1) is compared to the individual laboratory mean for a second sample (Plant Sample 4). The solid red box represents the NIST range of tolerance for the two samples, Plant Sample 4 (x-axis) and NRC HEMP-1 (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The dotted blue box (the bottom limit is not shown due to the scale of the figure) represents the consensus range of tolerance for Plant Sample 4 (x-axis) and NRC HEMP-1 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{comm}| \leq 2$.



Exercise: CannaQAP Exercise 2, Measurand: beryllium

Fig. 3-8. Laboratory means for beryllium in NRC HEMP-1 (Plant Sample 1) and SRM 1575a Trace Elements in Pine Needles (Pinus taeda) (sample/sample comparison view). In this view, the individual laboratory mean for one sample (NRC HEMP-1) is compared to the individual laboratory mean for a second sample (SRM 1575a). The dotted blue box represents the consensus range of tolerance for SRM 1575a (x-axis) and NRC HEMP-1 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{comm}| \le 2$.



Fig. 3-9. Laboratory means for beryllium in Plant Sample 4 and SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (sample/sample comparison view). In this view, the individual laboratory mean for one sample (Plant Sample 4) is compared to the individual laboratory mean for a second sample (SRM 1575a). The dotted blue box represents the consensus range of tolerance for SRM 1575a (x-axis) and Plant Sample 4 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{comm}| \le 2$.
Table 3-3. Data summary table for cobalt (Co) in the hemp and control samples. 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, $|Z'_{comm}| > 2$. *Note*: This table spans two pages; the NIST values and consensus values are included on both pages for convenience.

								(Cobalt (Co)							
		SRM 15	75a Traco (Pinus	e Element s <i>taeda)</i> (r	s in Pine I mg/kg)	Needles	NRC	HEMP-1	(Plant Sar	mple 1) (n	ng/kg)		Plant §	Sample 4 (mg/kg)		
	Lab	Α	В	С	Avg	SD	Α	В	С	Avg	SD	Α	В	С	Avg	SD	
	Target				0.0592	0.0039				1.854	0.340				0.2396	0.1172	
	B001	0.0582	0.0577	0.0521	0.0560	0.0034	1.42	1.98	1.13	1.510	0.432	0.15	0.154	0.152	0.1520	0.0020	
	B003	1.61688	1.60915	1.71398	1.6467	0.0584	0.05694	0.06036	0.05637	0.058	0.002	0.17482	0.16784	0.16227	0.1683	0.0063	
	B004	<1	<1	<1			1.44	1.38	1.45	1.423	0.038	<1	<1	<1			
	B006				í												
	B017																
	B018	0.057	0.056	0.056	0.0563	0.0006	1.375	1.395	1.29	1.353	0.056	0.176	0.16	0.16	0.1653	0.0092	
	B020																
	B028				í												
	B031																
	B035	0.055	0.055	0.056	0.0553	0.0006	1.674	1.659	1.747	1.693	0.047	0.148	0.154	0.154	0.1520	0.0035	
	B037																
ts	B040				1												
Ins	B049	0.063	0.059	0.06	0.0607	0.0021	1.78	1.85	1.7	1.777	0.075	0.167	0.165	0.173	0.1683	0.0042	
Re	B058	< 0.0784	< 0.0783	< 0.0764	1		1.65	1.63	1.92	1.733	0.162	0.174	0.149	0.188	0.1703	0.0198	
I ndividual I	B061																
	B066	0.057	0.058	0.058	0.0577	0.0006	1.598	1.59	1.627	1.605	0.019	0.17	0.164	0.169	0.1677	0.0032	
	B078																
-	B079				1												
	B084																
	B088	<1	<1	<1	1		3.3573	1.7823	2.1424	2.427	0.825	<1	<1	<1			
	B095	0.099	0.069	0.065	0.0777	0.0186	1.678	1.615	1.717	1.670	0.051	0.189	0.171	0.16	0.1733	0.0146	
	B097	0.045	0.058	0.054	0.0523	0.0067	2.43	2.78	2.68	2.630	0.180	0.37	0.26	0.25	0.2933	0.0666	
	B100	< 0.017					1.496			1.496		0.146			0.1460		
	B102				1												
	B110	0.0563	0.0562	0.0542	0.0556	0.0012	1.94	1.89	1.87	1.900	0.036	0.196	0.172	0.175	0.1810	0.0131	
	B111				1					1							
	B112	0.058	0.062	0.066	0.0620	0.0040	1.62	1.71	1.72	1.683	0.055	0.183	0.191	0.191	0.1883	0.0046	
	B113	0.06	0.055	0.052	0.0557	0.0040	1.328	1.321	1.276	1.308	0.028	0.15	0.151	0.146	0.1490	0.0026	
	B119																
	B122																
ţ		Consensu	ıs Mean		0.0589		Consenst	us Mean		1.720		Consensu	ls Mean		0.1722		
uni Its		Consensu	ıs Standarc	1 Deviation	0.0015	1	Consenst	us Standar	d Deviation	0.081		Consensu	us Standar	d Deviation	0.0043		
Imr		Maximum	1		1.6467	1	Maximun	n		5.603		Maximun	a		1.2167		
R.		Minimum			0.0503	ļ	Minimum	1		0.058		Minimum	i	0.1460			
U		Ν			24	1	Ν			30		Ν			26		

lable 3-3. continued	able 3-3. co	ntinued.	
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								(Cobalt (Co)							
		SRM 15	575a Trac <i>(Pinu</i> :	e Element s <i>taeda)</i> (1	s in Pine I ng/kg)	Needles	NRC	HEMP-1	(Plant Sa	mple 1) (n	ıg/kg)	Plant Sample 4 (mg/kg)					
	Lab	Α	В	С	Avg	SD	Α	В	С	Avg	SD	Α	В	С	Avg	SD	
	Target				0.0592	0.0039				1.854	0.340				0.2396	0.1172	
	B125	0.0485	0.0536	0.0489	0.0503	0.0028	0.412	0.394	0.357	0.388	0.028	0.137	0.147	0.155	0.1463	0.0090	
	B130	0.06	0.06	0.06	0.0600		1.45	1.41	1.39	1.417	0.031	0.16	0.17	0.16	0.1633	0.0058	
	B139	0.064	0.062	0.064	0.0633	0.0012	2.18	2.13	2.16	2.157	0.025	0.19	0.218	0.209	0.2057	0.0143	
	B141	0.06	0.056	0.054	0.0567	0.0031	1.661	1.864	1.609	1.711	0.135	0.175	0.161	0.157	0.1643	0.0095	
	B142																
	B148																
	B155	< 0.1	< 0.1				2.1	2.3	2.2	2.200	0.100	< 0.2	< 0.2	< 0.2			
	B161						1.32	1.27	1.29	1.293	0.025						
	B163	0.219	0.218	0.19	0.2090	0.0165	4.85	4.7	5.08	4.877	0.191	1.09	1.12	1.01	1.0733	0.0569	
	B172																
ults	B176																
kes	B179	0.066	0.0638	0.0652	0.0650	0.0011	1.716	1.682	1.735	1.711	0.027	0.1786	0.1729	0.1672	0.1729	0.0057	
al F	B180	0.159	0.141	0.146	0.1487	0.0093	1.816	1.866	1.915	1.866	0.050	0.184	0.214	0.193	0.1970	0.0154	
npi	B200	0.059	0.058	0.059	0.0587	0.0006	2.11	2.06	2.02	2.063	0.045	0.178	0.196	0.177	0.1837	0.0107	
divi	B202																
Ч	B203	0.061	0.059	0.062	0.0607	0.0015	1.87	1.93	2.1	1.967	0.119	0.169	0.174	0.172	0.1717	0.0025	
	B208																
	B211	< 0.003					< 0.003					< 0.003					
	B213						2.2	2.1	2	2.100	0.100	0.2	0.19	0.24	0.2100	0.0265	
	B214																
	B221	0.056	0.056	0.052	0.0547	0.0023	1.76	1.63	1.64	1.677	0.072	0.185	0.18	0.186	0.1837	0.0032	
	B222	0.063	0.061	0.058	0.0607	0.0025	1.734	1.731	1.694	1.720	0.022	0.17	0.18	0.18	0.1767	0.0058	
	B228																
	B230																
	B231																
	B232	0.06813	0.06302	0.06759	0.0662	0.0028	1.62717	1.61653	1.70011	1.648	0.045	0.17219	0.18234	0.18149	0.1787	0.0056	
	B235	0.289	0.25	0.237	0.2587	0.0271	5.19	6.01	5.61	5.603	0.410	1.14	1.18	1.33	1.2167	0.1002	
ity		Consensu	is Mean		0.0589		Consensu	is Mean		1.720		Consensu	is Mean		0.1722		
un		Consensu	is Standard	d Deviation	0.0015		Consensu	is Standard	l Deviation	0.081		Consensu	is Standard	1 Deviation	0.0043		
mm		Maximun	1		1.6467		Maximum	1		5.603		Maximun	n		1.2167		
Col R		Minimum			0.0503		Minimum			0.058		Minimum			0.1460		
-		Ν			24		Ν			30		Ν			26		



Fig. 3-10. Cobalt in NRC HEMP-1 (Plant Sample 1) (data summary view – sample preparation method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus the overlapping of the 95 % confidence interval for the consensus the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).





Fig. 3-11. Cobalt in NRC HEMP-1 (Plant Sample 1) (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Figure 3-12. Cobalt in Plant Sample 4 (data summary view – sample preparation method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 3-13. Cobalt in Plant Sample 4 (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 3-14. Cobalt in SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (data summary view – sample preparation method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed as indicated in the figure key. 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, $|Z'_{comm}| \leq 2$. 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, $|Z_{NIST}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 3-15. Cobalt in SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 3-16. Laboratory means for cobalt in NRC HEMP-1 (Plant Sample 1) and Plant Sample 4 (sample/sample comparison view). In this view, the individual laboratory mean for one sample (NRC HEMP-1) is compared to the individual laboratory mean for a second sample (Plant Sample 4). The solid red box (the top and bottom limits are not shown due to the scale of the figure) represents the NIST range of tolerance for the two samples, Plant Sample 4 (x-axis) and NRC HEMP-1 (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for Plant Sample 4 (x-axis) and NRC HEMP-1 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.



Exercise: CannaQAP Exercise 2, Measurand: cobalt

Fig. 3-17. Laboratory means for cobalt in NRC HEMP-1 (Plant Sample 1) and SRM 1575a Trace Elements in Pine Needles (Pinus taeda) (sample/sample comparison view). In this view, the individual laboratory mean for one sample (NRC HEMP-1) is compared to the individual laboratory mean for a second sample (SRM 1575a). The solid red box represents the NIST range of tolerance for the two samples, SRM 1575a (x-axis) and NRC HEMP-1 (y-axis), which encompasses the target values bounded by their uncertainties (UNIST) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \le 2$. The dotted blue box represents the consensus range of tolerance for SRM 1575a (x-axis) and NRC HEMP-1 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{comm}| \le 2$.



Exercise: CannaQAP Exercise 2, Measurand: cobalt No. of laboratories: 24

Fig. 3-18. Laboratory means for cobalt in Plant Sample 4 and SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (sample/sample comparison view). In this view, the individual laboratory mean for one sample (Plant Sample 4) is compared to the individual laboratory mean for a second sample (SRM 1575a). The solid red box (the top and bottom limits are not shown due to the scale of the figure) represents the NIST range of tolerance for the two samples, SRM 1575a (x-axis) and Plant Sample 4 (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The dotted blue box represents the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

Table 3-4. Data summary table for chromium (Cr) in the hemp and control samples. 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, $|Z'_{comm}| > 2$. *Note*: This table spans two pages; the NIST values and consensus values are included on both pages for convenience.

						Chromium (Cr)											
		SRM 15'	75a Traco <i>(Pinus</i>	e Elements s <i>taeda)</i> (n	s in Pine ng/kg)	Needles	NRC	HEMP-1	(Plant Sai	nple 1) (r	ng/kg)	Plant Sample 4 (mg/kg)					
	Lab	Α	В	С	Avg	SD	Α	В	С	Avg	SD	Α	В	С	Avg	SD	
	Target				0.388					12.72	4.49				0.307	0.220	
	B001	0.355	0.363	0.286	0.335	0.042	4.41	5.12	5.07	4.87	0.40	0.367	0.392	0.368	0.376	0.014	
	B003	10.6259	9.97441	11.9114	10.837	0.986	0.62134	0.57275	0.40954	0.53	0.11	1.11422	0.65697	0.5437	0.772	0.302	
	B004	<1	<1	<1			5.93	5.95	5.77	5.88	0.10	<1	<1	<1			
	B006										_						
	B017							- 403				0.460					
	B018	0.309	0.3	0.315	0.308	0.008	7.183	7.183	7.427	7.26	0.14	0.468	0.454	0.405	0.442	0.033	
	B020																
	B028																
	B031	0.295	0.201	0.204	0.207	0.004	0.005	0.124	0.020	0.00	0.49	0.200	0.41	0.205	0.207	0.012	
	B035	0.285	0.291	0.284	0.287	0.004	8.905	8.134	9.029	8.69	0.48	0.386	0.41	0.395	0.397	0.012	
	B037																
	B040	0.404	0.410	0.410	0.414	0.000	0.37	10	0.53	0.63	0.33	0.512	0 407	0.505	0.505	0.008	
	B058	0.253	0.205	0.419	0.714	0.005	7.6	7 47	7.11	7 39	0.35	0.312	0.39	0.359	0.369	0.008	
	B061	1 1 2 5	1 2402	1 2647	1 210	0.025	12.9	13.4	12.35	12.88	0.23	2 6085	3 3214	2 5784	2.836	0.018	
	B066	0.483	0.442	0.669	0.531	0.121	10.847	10 435	10.992	10.76	0.33	0.592	0.635	0.572	0.600	0.032	
ult	B070	0.318	0.301	0.348	0.322	0.024	5 768	6 869	7 825	6.82	1.03	0.446	0.832	0.466	0.581	0.217	
Res	B077	0.312	0.323	0.3275	0.321	0.008	8.9121	9.4748	8.2248	8.87	0.63	0.4748	0.4726	0.4126	0.453	0.035	
Ial	B078																
vidı	B079																
ndi	B081	0.304	0.34	0.374	0.339	0.035	8.303	6.823	8.203	7.78	0.83	0.402	0.513	0.432	0.449	0.057	
II	B084	0.279	0.276	0.272	0.276	0.004	7.81	8.61	8.06	8.16	0.41	0.375	0.371	0.367	0.371	0.004	
	B088	<1	<1	<1			15.4501	7.7397	9.5576	10.92	4.03	<1	2.0946	1.0628	1.579	0.730	
	B090	0.31	< 0.299	< 0.296	0.310		9.841	9.812	9.324	9.66	0.29	0.495	0.576	0.416	0.496	0.080	
	B091																
	B094	0.3	0.3	0.3	0.300		9.69	9.5	9.45	9.55	0.13	0.42	0.43	0.43	0.427	0.006	
	B095	0.387	0.391	0.38	0.386	0.006	8.742	5.795	9.235	7.92	1.86	0.487	0.537	0.335	0.453	0.105	
	B097	0.386	0.391	0.332	0.370	0.033	10.6	11.9	12.9	11.80	1.15	1.2	0.761	0.907	0.956	0.224	
	B100	0.33			0.330		7.63			7.63		0.375			0.375		
	B102																
	B107	0.3486	0.3343	0.326	0.336	0.011	11.8265	10.4189	10.7513	11.00	0.74	0.5284	0.4677	0.6372	0.544	0.086	
	B109	< 0.5	< 0.5	< 0.5			3.02	2.87	2.55	2.81	0.24	< 0.5	1.12	< 0.5	1.120		
	BIII	0.2.42	0.060	0.265	0.057	0.012	5.045	6 720	6 101	6.05	0.45	0.201	0.425	0.466	0.421	0.020	
	BI12	0.342	0.363	0.365	0.357	0.013	5.845	6.738	6.181	6.25	0.45	0.391	0.435	0.466	0.431	0.038	
	BII3	0.365	0.349	0.234	0.316	0.071	6.364	6.404	6.363	6.38	0.02	0.276	0.587	0.304	0.389	0.172	
	B110	0.338	0.315	0.372	0.342	0.029	8.419	8.84	8.675	8.64	0.21	0.431	0.465	0.428	0.441	0.021	
	B119 D122																
>	D122	Consense	ıs Mean		0 344		Consensi	is Mean		8 68		Consensi	ıs Mean		0.472		
nity s		Consense	is Standar	d Deviatio	0.010		Consensi	is Standan	d Deviatio	0.33		Consensi	is Standan	d Deviatio	0.014		
mu		Maximum		a De fano!	10 837		Maximum		a De natio	13.67		Maximum		a Devano	2,836		
om Re		Minimum			0.189		Minimum			0.53		Minimum			0.367		
С		N			36		N			45		N			39		

SRM 1575a Trace Elements in Pine Needles (Pinus taeda) (mg/kg) NRC HEMP-1 (Plant Sample 1) (mg/kg) Lab A B C Avg SD A B C Avg SD A Target 0.388 0.388 12.72 4.49 12.72 4.49 B125 1000000000000000000000000000000000000	Plant Sample 4 B C 0.46 0.48 2 0.5214 0.4996 7 0.483 0.498 5 0.489 0.484	Avg SD 0.307 0.220 0.477 0.015 0.505 0.014 0.479 0.021
Lab A B C Avg SD A B C Avg SD A Target 0.388 0.388 12.72 4.49 12.72 4.49 12.72 <t< th=""><th>B C 0.46 0.48 2 0.5214 0.4996 7 0.483 0.498 5 0.489 0.484</th><th>Avg SD 0.307 0.220 0.477 0.015 0.505 0.014 0.479 0.021</th></t<>	B C 0.46 0.48 2 0.5214 0.4996 7 0.483 0.498 5 0.489 0.484	Avg SD 0.307 0.220 0.477 0.015 0.505 0.014 0.479 0.021
Target 0.388 12.72 4.49 B125 B130 0.34 0.36 0.32 0.340 0.020 8.7 8.66 8 8.45 0.39 0.49	0.46 0.48 2 0.5214 0.4996 7 0.483 0.498 5 0.489 0.484	0.307 0.220 0.477 0.015 0.505 0.014 0.479 0.021
B125 B130 0.34 0.36 0.32 0.340 0.020 8.7 8.66 8 8.45 0.39 0.49	0.46 0.48 2 0.5214 0.4996 7 0.483 0.498 5 0.489 0.484	0.477 0.015 0.505 0.014 0.479 0.021
B130 0.34 0.36 0.32 0.340 0.020 8.7 8.66 8 8.45 0.39 0.49	0.46 0.48 2 0.5214 0.4996 7 0.483 0.498 5 0.489 0.484	0.477 0.015 0.505 0.014 0.479 0.021
	2 0.5214 0.4996 7 0.483 0.498 5 0.489 0.484	0.505 0.014
B137 0.4154 0.4456 0.398 0.420 0.024 8.99 9.674 9.514 9.39 0.36 0.495	7 0.483 0.498 5 0.489 0.484	0.479 0.021
B139 0.341 0.335 0.325 0.334 0.008 7.92 7.19 7.73 7.61 0.38 0.457	5 0.489 0.484	0.479 0.021
B141 0.435 0.437 0.44 0.437 0.003 9.397 9.086 8.951 9.14 0.23 0.523		0.499 0.022
B142		
B148		
	0.0 0.0	0.700 0.100
B155 <0.5 <0.5 <15 15 15 15 15 0.7 0.7	0.6 0.8	0.700 0.100
BI01 2.95 2.94 2.98 2.90 0.02 B162 0.556 0.442 0.264 0.454 0.007 12.22 11.72 12.5 12.40 0.01 0.60	1 0.527 0.406	0.546 0.055
B105 0.330 0.442 0.304 0.434 0.097 12.23 11.75 13.3 12.49 0.91 0.004	+ 0.337 0.490	0.340 0.033
B174 0.200 0.258 0.286 0.281 0.021 7.086 7.002 7.262 7.12 0.13 0.43	5 0.408 0.454	0.433 0.023
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	0.408 0.434	0.455 0.025
B179 0.3651 0.3882 0.796 0.516 0.242 10.054 9.27 9.851 9.73 0.41 0.485	4 0 5026 0 4606	0.483 0.021
	5 0.386 0.404	0.395 0.009
	<1 <1	01009
$\frac{2}{2}$ B183 0.3691 0.369 9.1174 9.12 0.43		0.430
B192 0.343 0.358 0.401 0.367 0.030 10 8.11 8.93 9.01 0.95 0.447	3 0.516 0.541	0.502 0.048
B198		
H B200 0.315 0.344 0.347 0.335 0.018 10.3 11.5 10.9 10.90 0.60 0.47	0.495 0.517	0.494 0.024
B202		
B203 0.422 0.356 0.378 0.385 0.034 10.3 10.7 10.4 10.47 0.21 0.430	5 0.494 0.486	0.472 0.031
B204 0.319 0.339 0.319 0.326 0.012 10.06 10.264 9.324 9.88 0.49 0.488	8 0.462 0.515	0.488 0.027
B208		
B211 0.3 0.300 8.6 8.60 0.6		0.600
B213 9 9 7 8.33 1.15 <1	<1 <1	
B214		
B220		
B221 0.48 0.3 0.27 0.350 0.114 6.01 5.71 5.84 5.85 0.15 0.37	0.36 0.37	0.367 0.006
B222 0.34 0.33 0.39 0.353 0.032 9.555 9.274 9.027 9.29 0.26 0.41	0.43 0.49	0.443 0.042
B226 0.32434 0.30901 0.3003 0.311 0.012 10.7971 11.4785 9.04374 10.44 1.26 0.455	32 0.47341 0.44301	0.457 0.015
8228		
B230 D221		
B231 D222 0.2(787 0.2(252 0.2507 0.2(2 0.004 0.7215(0.5075 0.12555 0.0.0 0.4(1	LC 0 40995 0 46927	0.476 0.020
B222 0.50/8/ 0.50253 0.53720 0.505 0.004 6.75120 6.53903 9.15353 6.60 0.30 0.400 D225 0.518 0.441 0.422 0.464 0.047 123 13 6.12 12 07 0.68 0.511	10 0.49885 0.40827	0.476 0.020
Consensus Mean 0.344 Consensus Mean 8.68 Conse	nsus Mean	0.337 0.018
Consensus Standard Deviation 0.010 Consensus Standard Deviation 0.33 Conse	nsus Standard Deviatio	1 0 014
Maximum 10.837 Maximum 13.67 Maxim	um	2.836
$\begin{bmatrix} \mathbf{B} & \mathbf{Z} \end{bmatrix}$ Minimum 0.189 Minimum 0.53 Minim	um	0.367
0 N 36 N 45 N		39



Fig. 3-19. Chromium in NRC HEMP-1 (Plant Sample 1) (data summary view – sample preparation 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 sample preparation method employed as indicated in the figure key. 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, $|Z'_{comm}| \leq 2$. 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, $|Z_{NIST}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 3-20. Chromium in NRC HEMP-1 (Plant Sample 1) (data summary view – analytical 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 analytical method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 3-21. Chromium in Plant Sample 4 (data summary view – sample preparation method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 3-22. Chromium in Plant Sample 4 (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. The red shaded region represents the Interval for the consensus mean that result is uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 3-23. Chromium in SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (data summary view – sample preparation method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed as indicated in the figure key. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid, bold 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, $|Z'_{comm}| \leq 2$. The red line represents the non-certified value determined in this material.



Fig. 3-24. Chromium in SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed as indicated in the figure key. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid, bold 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, $|Z'_{comm}| \leq 2$. The red line represents the non-certified value determined in this material.



Exercise: CannaQAP Exercise 2, Measurand: chromium

Fig. 3-25. Laboratory means for chromium in NRC HEMP-1 (Plant Sample 1) and Plant Sample 4 (sample/sample comparison view). In this view, the individual laboratory mean for one sample (NRC HEMP-1) is compared to the individual laboratory mean for a second sample (Plant Sample 4). The solid red box (the right and bottom limits are not shown due to the scale of the figure) represents the NIST range of tolerance for the two samples, Plant Sample 4 (x-axis) and NRC HEMP-1 (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for Plant Sample 4 (x-axis) and NRC HEMP-1 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z_{comm} score, $|Z_{\text{comm}}| \leq 2$.



Exercise: CannaQAP Exercise 2, Measurand: chromium No. of laboratories: 41

Fig. 3-26. Laboratory means for chromium in NRC HEMP-1 (Plant Sample 1) and SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (sample/sample comparison view). In this view, the individual laboratory mean for one sample (NRC HEMP-1) is compared to the individual laboratory mean for a second sample (SRM 1575a). The dotted blue box represents the consensus range of tolerance for SRM 1575a (x-axis) and NRC HEMP-1 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{comm}| \le 2$.



Fig. 3-27. Laboratory means for chromium in Plant Sample 4 and SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (sample/sample comparison view). In this view, the individual laboratory mean for one sample (Plant Sample 4) is compared to the individual laboratory mean for a second sample (SRM 1575a). The dotted blue box represents the consensus range of tolerance for SRM 1575a (x-axis) and Plant Sample 4 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{comm}| \le 2$.

Table 3-5. Data summary table for molybdenum (Mo) in the hemp and control samples. 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, $|Z'_{comm}| > 2$. *Note*: This table spans two pages; the NIST values and consensus values are included on both pages for convenience.

								Mol	ybde num ((Mo)							
		SRM 15	75a Trac <i>(Pinus</i>	e Element s <i>taeda)</i> (1	ts in Pine mg/kg)	Needles	NRCI	HEMP-1	(Plant Sa	mple 1) (r	ng/kg)	Plant Sample 4 (mg/kg)					
	Lab	Α	В	С	Avg	SD	Α	В	С	Avg	SD	Α	В	С	Avg	SD	
	Target									0.661	0.096				0.3180	0.0120	
	B001	0.0194	0.0269	0.0153	0.0205	0.0059	0.546	0.594	0.593	0.578	0.027	0.295	0.311	0.323	0.3097	0.0140	
	B003	0.63628	0.6226	0.65783	0.6389	0.0178	0.02602	0.0354	0.04634	0.036	0.010	0.36125	0.33382	0.34126	0.3454	0.0142	
	B004	<1	<1	<1			<1	<1	<1			<1	<1	<1			
	B006																
	B017																
	B018	< 0.028	< 0.028	< 0.028			0.549	0.559	0.557	0.555	0.005	0.299	0.284	0.286	0.2897	0.0081	
	B020																
	B028														l		
	B031																
	B035	0.012	0.012	0.013	0.0123	0.0006	0.618	0.559	0.576	0.584	0.030	0.316	0.304	0.312	0.3107	0.0061	
	B037																
ts	B040																
Inse	B049						0.609	0.62	0.554	0.594	0.035	0.32	0.297	0.301	0.3060	0.0123	
I R(B058	< 0.0784	< 0.0783	< 0.0764			0.588	0.554	0.673	0.605	0.061	0.33	0.246	0.355	0.3103	0.0571	
ual	B061																
vid	B066	0.021	0.02	0.024	0.0217	0.0021	0.514	0.456	0.449	0.473	0.036	0.296	0.303	0.295	0.2980	0.0044	
ibu	B078																
Г	B079														l		
	B084																
	B088	<1	<1	<1			1.3496	<1	<1	1.350		<1	<1	<1			
	B095	0.013	0.018	0.016	0.0157	0.0025	0.566	0.472	0.545	0.528	0.049	0.332	0.313	0.288	0.3110	0.0221	
	B097	< 0.05	< 0.05	< 0.05			0.0574	0.0582	0.0568	0.057	0.001	0.0421	0.0314	0.0289	0.0341	0.0070	
	B100	< 0.263					< 0.878					< 0.878					
	B102																
	B107	0.0129	0.0137	0.0141	0.0136	0.0006	0.6922	0.6332	0.648	0.658	0.031	0.3809	0.3289	0.3511	0.3536	0.0261	
	B111																
	B112	0.018	0.023	0.024	0.0217	0.0032	0.565	0.535	0.558	0.553	0.016	0.268	0.275	0.284	0.2757	0.0080	
	B113																
	B119																
	B122																
ţ		Consensu	ıs Mean		0.0184		Consensu	ıs Mean		0.612		Consense	us Mean		0.3244	_	
uni lts		Consensu	ıs Standar	d Deviation	0.0015		Consensus Standard Deviati			0.022		Consense	us Standar	d Deviatio	0.0073		
nm		Maximun	n		0.6389		Maximun	1		1.350		Maximum			0.4029		
PO R		Minimum	ι .		0.0123		Minimum	1		0.036 Minimum				0.0341			
9		Ν			15		Ν			24		Ν			24		

	r			-		-		Mol	ybde num ((Mo)							
		SRM 157	75a Trace (Pinus	e Element s taeda) (1	ts in Pine mg/kg)	Needles	NRC	HEMP-1	(Plant Sa	mple 1) (r	ng/kg)		Plant S	ample 4 (mg/kg)		
	Lab	Α	В	С	Avg	SD	Α	В	С	Avg	SD	Α	В	С	Avg	SD	
	Target									0.661	0.096				0.3180	0.0120	
	B139	0.027	< 0.020	< 0.020	0.0270		0.71	0.735	0.728	0.724	0.013	0.333	0.364	0.351	0.3493	0.0156	
	B141	0.014	0.013	0.012	0.0130	0.0010	0.583	0.588	0.566	0.579	0.012	0.326	0.296	0.301	0.3077	0.0161	
	B142																
	B148									1							
	B155	<1	<1				<1	<1	<1			<1	<1	<1			
	B161									1							
	B163	0.0177	0.019	0.0157	0.0175	0.0017	0.669	0.66	0.723	0.684	0.034	0.343	0.345	0.318	0.3353	0.0150	
	B176	< 0.003	< 0.003	< 0.003			0.5253	0.4928	0.4923	0.503	0.019	0.4299	0.4153	0.3634	0.4029	0.0350	
	B179	0.0149	0.0146	0.0175	0.0157	0.0016	0.696	0.715	0.667	0.693	0.024	0.3482	0.33	0.3448	0.3410	0.0097	
ults	B180	< 0.1	< 0.1	< 0.1			0.584	0.574	0.574	0.577	0.006	0.276	0.282	0.293	0.2837	0.0086	
Xes	B198																
al F	B202									I							
idu	B203	0.022	0.019	0.019	0.0200	0.0017	0.654	0.633	0.627	0.638	0.014	0.305	0.344	0.326	0.3250	0.0195	
div	B208									I							
ĥ	B211	< 0.038					< 0.038					< 0.038					
	B212	< 0.25	< 0.25	< 0.25			0.6153	0.5933	0.6223	0.610	0.015	0.2805	0.3017	0.2846	0.2889	0.0112	
	B213						0.8	0.7	0.7	0.733	0.058	0.4	0.3	0.4	0.3667	0.0577	
	B214			!		!	[I							
	B221	0.021	0.021	0.018	0.0200	0.0017	0.599	0.575	0.578	0.584	0.013	0.358	0.348	0.362	0.3560	0.0072	
	B222	0.044	0.044	0.033	0.0403	0.0064	0.556	0.51	0.524	0.530	0.024	0.37	0.35	0.34	0.3533	0.0153	
	B228																
	B230			!		!				I							
	B231																
	B232	0.01577	0.016	0.0127	0.0148	0.0018	0.72981	0.66995	0.68514	0.695	0.031	0.32257	0.34695	0.3091	0.3262	0.0192	
	B235	0.0198	0.0175	0.0161	0.0178	0.0019	0.747	0.82	0.756	0.774	0.040	0.331	0.342	0.336	0.3363	0.0055	
Â,	ī '	Consensu	ls Mean		0.0184	!	Consense	us Mean		0.612		Consensu	us Mean		0.3244	_	
uni Its	1 '	Consensu	us Standar	d Deviatio	0.0015	I	Consensu	us Standar	d Deviatio	0.022	I	Consensu	us Standaro	d Deviatio	0.0073		
nm esu	1 '	Maximum	.1		0.6389	1	Maximur	n		1.350	1	Maximun	n		0.4029		
B R	1 '	Minimum	1		0.0123	1	Minimum	1		0.036	I	Minimum	1		0.0341		
U U	1 '	Minimum N			15		N			24	1	Ν		24	ļ		



Fig. 3-28. Molybdenum in NRC HEMP-1 (Plant Sample 1) (data summary view – sample preparation method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus the overlapping of the 95 % confidence interval for the consensus the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 3-29. Molybdenum in NRC HEMP-1 (Plant Sample 1) (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 3-30. Molybdenum in Plant Sample 4 (data summary view – sample preparation method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 3-31. Molybdenum in Plant Sample 4 (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean the results in an acceptable Z_{NIST} score, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 3-32. Molybdenum in SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (data summary view – sample preparation method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. A NIST value has not been determined in this material.



Fig. 3-33. Molybdenum in SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed as indicated in the figure key. 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, $|Z'_{comm}| \leq 2$. NIST value has not been determined in this material.



Exercise: CannaQAP Exercise 2, Measurand: molybdenum

Fig. 3-34. Laboratory means for molybdenum in NRC HEMP-1 (Plant Sample 1) and Plant Sample 4 (sample/sample comparison view). In this view, the individual laboratory mean for one sample (NRC HEMP-1) is compared to the individual laboratory mean for a second sample (Plant Sample 4). The solid red box represents the NIST range of tolerance for the two samples, Plant Sample 4 (x-axis) and NRC HEMP-1 (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 1$ 2. The dotted blue box represents the consensus range of tolerance for Plant Sample 4 (x-axis) and NRC HEMP-1 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{comm}| \leq 2$.



Exercise: CannaQAP Exercise 2, Measurand: molybdenum

Fig. 3-35. Laboratory means for molybdenum in NRC HEMP-1 (Plant Sample 1) and SRM 1575a Trace Elements in Pine Needles (Pinus taeda) (sample/sample comparison view). In this view, the individual laboratory mean for one sample (NRC HEMP-1) is compared to the individual laboratory mean for a second sample (SRM 1575a). The dotted blue box represents the consensus range of tolerance for SRM 1575a (x-axis) and NRC HEMP-1 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{comm}| \le 2$.



Fig. 3-36. Laboratory means for molybdenum in Plant Sample 4 and SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (sample/sample comparison view). In this view, the individual laboratory mean for one sample (Plant Sample 4) is compared to the individual laboratory mean for a second sample (SRM 1575a). The dotted blue box represents the consensus range of tolerance for SRM 1575a (x-axis) and Plant Sample 4 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{comm}| \le 2$.

Table 3-6. Data summary table for manganese (Mn) in the hemp and control samples. 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, $|Z'_{comm}| > 2$. Note: This table spans two pages; the NIST values and consensus values are included on both pages for convenience.

								Mar	iganese (N	Mn)						
		SRM 15	75a Trac (Pinu)	e Element s <i>taeda)</i> (1	ts in Pine I mg/kg)	Needles	NRC	HEMP-1	(Plant Sa	mple 1) (1	Plant Sample 4 (mg/kg)					
	Lab	Α	В	С	Avg	SD	Α	В	С	Avg	SD	Α	В	С	Avg	SD
	Target				473.8	23.3				401.0	26.4				142.0	7.6
	B001	486	452	423	453.7	31.5	373	368	382	374.3	7.1	139	129	121	129.7	9.0
	B003	397.265	394.672	395.319	395.8	1.4	469.696	551.852	456.925	492.8	51.5	140.032	159.016	167.723	155.6	14.2
	B004	390	393	390	391.0	1.7	279	271	266	272.0	6.6	109	110	106	108.3	2.1
	B006															
	B017															
	B018	447.2	450.5	450.5	449.4	1.9	303.7	315.7	321.9	313.8	9.3	126.2	136.8	137.4	133.5	6.3
	B020															
	B028															
	B031															
	B035	444.6	450.3	447.9	447.6	2.9	380.5	363.1	382.3	375.3	10.6	137.8	136.1	138.9	137.6	1.4
	B037															
Its	B040															
esu	B049	468	456	417	447.0	26.7	395	393	385	391.0	5.3	141	137	138	138.7	2.1
ual Ro	B058	476	427	450	451.0	24.5	327	253	355	311.7	52.7	105	108	134	115.7	15.9
lua	B061															
ivic	B066	456.385	432.828	436.783	442.0	12.6	364.914	364.65	350.931	360.2	8.0	129.074	125.089	123.806	126.0	2.7
pu	B078															
_	B079															
	B084															
	B088	489.149	501.361	477.594	489.4	11.9	681.891	350.293	440.519	490.9	171.4	152.794	141.499	163.91	152.7	11.2
	B095	442.885	443.889	447.63	444.8	2.5	339.151	362.853	348.405	350.1	11.9	137.48	133.022	128.576	133.0	4.5
	B097	621	618	620	619.7	1.5	421	434	468	441.0	24.3	234	167	159	186.7	41.2
	B100	445.63			445.6		358.7			358.7		133.79			133.8	
	B102															
	B107	807.908	816.151	815.586	813.2	4.6	669.268	626.776	642.385	646.1	21.5	151.147	146.123	143.854	147.0	3.7
	B111															
	B112	491.8	446.7	496	478.2	27.3	328.7	332.5	358.2	339.8	16.0	117.9	124.4	125.8	122.7	4.2
	B113	523.917	486.405	463.342	491.2	30.6	287.891	287.662	28.433	201.3	149.7	123.624	116.707	116.854	119.1	4.0
	B119															
	B122															
ity		Consensu	is Mean		458.0		Consense	us Mean		366.9		Consense	is Mean		135.4	
un ilts		Consensu	ıs Standar	d Deviatio	8.3		Consense	us Standar	d Deviatio	9.4		Consense	us Standar	d Deviatio	2.4	
mm test		Maximun	n		813.2		Maximur	n		646.1		Maximur	n		186.7	
^B Co		Minimum	L		376.0		Minimum	1		201.3		Minimum	1		108.3	
-		Ν			29		Ν			32		Ν			32	

								Man	iganese (N	(In)							
		SRM 15	75a Trac <i>(Pinu</i>)	e Element s <i>taeda)</i> (1	ts in Pine 1 mg/kg)	Needles	NRC	HEMP-1	(Plant Sai	mple 1) (r	ng/kg)	Plant Sample 4 (mg/kg)					
	Lab	Α	В	С	Avg	SD	Α	В	С	Avg	SD	Α	В	С	Avg	SD	
	Target				473.8	23.3				401.0	26.4				142.0	7.6	
	B125	376	376	376	376.0		401	366	335	367.3	33.0	135	142	135	137.3	4.0	
	B139	485	486	484	485.0	1.0	390	379	379	382.7	6.4	125	137	134	132.0	6.2	
	B141	430.374	421.405	411.146	421.0	9.6	341.221	333.434	324.377	333.0	8.4	133.935	127.642	129.599	130.4	3.2	
	B142																
	B148																
	B155	475			475.0		389	374		381.5	10.6	147	152		149.5	3.5	
	B161	386	384	387	385.7	1.5	314	296	308	306.0	9.2	112	116	114	114.0	2.0	
	B163	573	560	473	535.3	54.4	457	442	472	457.0	15.0	149	153	137	146.3	8.3	
	B176	432.742	436.956	438.929	436.2	3.2	361.026	360.821	361.764	361.2	0.5	128.953	134.027	128.957	130.6	2.9	
	B179	497.236	501.396	502.676	500.4	2.8	381.49	379.991	384.048	381.8	2.1	146.8	145.6	140.4	144.3	3.4	
ts	B180	442.7	443.9	439.4	442.0	2.3	334.9	348.9	363.4	349.1	14.3	131.6	128.6	128.8	129.7	1.7	
Ins	B192	580	578	610	589.3	17.9	462	420	421	434.3	24.0	160	181	187	176.0	14.2	
Re	B200	452	475	481	469.3	15.3	409	399	405	404.3	5.0	147	154	150	150.3	3.5	
ual	B202																
vid	B203	488	470	480	479.3	9.0	389	402	393	394.7	6.7	132	145	139	138.7	6.5	
ipu	B208																
I	B211	428.8			428.8		314.9			314.9		123.1			123.1		
	B212	476.48	469.67	482.16	476.1	6.3	343.64	331.86	340.31	338.6	6.1	128.95	145.71	140.32	138.3	8.6	
	B213						380	370	370	373.3	5.8	130	130	140	133.3	5.8	
	B214																
	B220																
	B221	429	445	386	420.0	30.5	351	332	330	337.7	11.6	122	119	120	120.3	1.5	
	B222	447	479	442	456.0	20.1	303.463	320.4	330.84	318.2	13.8	129	137	133	133.0	4.0	
	B228																
	B230																
	B231																
	B232	459.751	467.357	467.258	464.8	4.4	336.152	342.922	349.819	343.0	6.8	142.032	148.65	140.503	143.7	4.3	
	B235	658	577	544	593.0	58.7	465	535	491	497.0	35.4	141	148	142	143.7	3.8	
ty		Consensu	ıs Mean		458.0		Consense	ıs Mean		366.9		Consense	ıs Mean		135.4		
uni lts		Consensu	ıs Standar	d Deviatio	8.3		Consense	us Standar	d Deviatio	9.4		Consense	us Standar	d Deviatio	2.4		
nmi		Maximun	n		813.2		Maximur	n		646.1		Maximur	n		186.7		
R.		Minimum	L		376.0		Minimum	1		201.3		Minimum	1		108.3		
0		Ν			29		Ν			32		Ν			32		



Fig. 3-37. Manganese in NRC HEMP-1 (Plant Sample 1) (data summary view – sample preparation 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 sample preparation method employed as indicated in the figure key. 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, $|Z'_{comm}| \leq 2$. 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, $|Z_{NIST}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).


Fig. 3-38. Manganese in NRC HEMP-1 (Plant Sample 1) (data summary view – analytical 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 analytical method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 3-39. Manganese in Plant Sample 4 (data summary view – sample preparation 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 sample preparation method employed as indicated in the figure key. 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, $|Z'_{comm}| \leq 2$. 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, $|Z_{NIST}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 3-40. Manganese in Plant Sample 4 (data summary view – analytical 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 analytical method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 3-41. Manganese in SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (data summary view – sample preparation 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 sample preparation method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 3-42. Manganese in SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (data summary view – analytical 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 analytical method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Exercise: CannaQAP Exercise 2, Measurand: manganese No. of laboratories: 34

Fig. 3-43. Laboratory means for manganese in NRC HEMP-1 (Plant Sample 1) and Plant Sample 4 (sample/sample comparison view). In this view, the individual laboratory mean for one sample (NRC HEMP-1) is compared to the individual laboratory mean for a second sample (Plant Sample 4). The solid red box represents the NIST range of tolerance for the two samples, Plant Sample 4 (x-axis) and NRC HEMP-1 (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for Plant Sample 4 (x-axis) and NRC HEMP-1 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.



Fig. 3-44. Laboratory means for manganese in NRC HEMP-1 (Plant Sample 1) and SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (sample/sample comparison view). In this view, the individual laboratory mean for one sample (NRC HEMP-1) is compared to the individual laboratory mean for a second sample (SRM 1575a). The solid red box represents the NIST range of tolerance for the two samples, SRM 1575a (x-axis) and NRC HEMP-1 (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{NIST}| \le 2$. The dotted blue box represents the consensus range of tolerance for SRM 1575a (x-axis) and NRC HEMP-1 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{comm}| \le 2$.



Exercise: CannaQAP Exercise 2, Measurand: manganese No. of laboratories: 33

Fig. 3-45. Laboratory means for manganese in Plant Sample 4 and SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (sample/sample comparison view). In this view, the individual laboratory mean for one sample (Plant Sample 4) is compared to the individual laboratory mean for a second sample (SRM 1575a). The solid red box represents the NIST range of tolerance for the two samples, SRM 1575a (x-axis) and Plant Sample 4 (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{NIST}| \le 2$. The dotted blue box represents the consensus range of tolerance for SRM 1575a (x-axis) and Plant Sample 4 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{comm}| \le 2$.

Table 3-7. Data summary table for nickel (Ni) in the hemp and control samples. 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, $|Z'_{comm}| > 2$. *Note*: This table spans two pages; the NIST values and consensus values are included on both pages for convenience.

		Nickel (Ni)															
		SRM 15	75a Trac <i>(Pinu</i> s	e Elements s <i>taeda)</i> (n	s in Pine I ng/kg)	Needles	NRC	HEMP-1	(Plant Sar	nple 1) (n	ıg/kg)	Plant Sample 4 (mg/kg)					
	Lab	Α	В	С	Avg	SD	Α	В	С	Avg	SD	Α	В	С	Avg	SD	
	Target				1.427	0.194				7.11	4.36				3.670	1.320	
	B001	1.49	1.39	1.31	1.397	0.090	6.58	6.52	6.3	6.47	0.15	3.24	3.27	3.36	3.290	0.062	
	B003	6.79149	9.97441	11.9114	9.559	2.585	1.29971	1.42833	1.22236	1.32	0.10	4.02959	3.70498	3.51212	3.749	0.262	
	B004	1.36	1.36	1.36	1.360	0.000	5.18	5.1	5.17	5.15	0.04	2.94	2.94	3.02	2.967	0.046	
	B006																
	B017																
	B018	1.3	1.313	1.308	1.307	0.007	4.69	4.865	4.273	4.61	0.30	3.33	3.173	3.054	3.186	0.138	
	B020																
	B028										_						
	B031																
	B035	1.295	1.277	1.294	1.289	0.010	6.061	5.485	5.976	5.84	0.31	2.987	3.099	3.027	3.038	0.057	
	B037																
	B040	1.42		1.20	1 407	0.021	6.1.5	6.46	5.05	6.10	0.05	2.41	2.22	2.45	2 202	0.067	
	B049	1.43	1.4	1.39	1.407	0.021	6.15	6.46	5.97	6.19	0.25	3.41	3.32	3.45	3.393	0.067	
ults	B058	1.55	1.39	1.39	1.443	0.092	5.54	5.46	6.2	5.73	0.41	3.26	2.84	3.41	3.170	0.295	
dual Resu	B061	0.9593	1.0238	0.965	0.983	0.036	0.5943	0.6293	0.6193	0.61	0.02	2.6361	2.65/4	2.7297	2.6/4	0.049	
	B066	1.211	1.18/	1.235	1.211	0.024	5.354	5.378	5.293	5.34	0.04	3.22	3.085	3.129	3.145	0.069	
	B078 D070																
livi	D0/9	1 201	1 410	1 5 5 1	1 424	0.125	5 226	1562	5 2 2	5.04	0.42	2 245	2 2 2 2	2 262	2 200	0.046	
Inc	D081	1.301	1.419	1.551	1.424	0.123	5.250	4.302	5.55	5.04	0.42	3.243	5.555	5.205	3.280	0.040	
	B084	<1	<1	<1			<1	<1	<1			<1	<1	<1			
	B000	1 249	1 233	1 242	1 241	0.008	5 75	5.82	5 802	5 79	0.04	3 095	2 984	3 214	3.098	0.115	
	B091	1.247	1.235	1.272	1.241	0.000	5.15	5.62	5.002	5.17	0.04	5.075	2.704	5.214	5.070	0.115	
	B095	1 368	8 39	1 394	3 717	4 047	5 852	5 508	5.97	5 78	0.24	3 571	3 376	3 175	3 374	0.198	
	B097	1.500	1.92	2.01	1 967	0.045	19.1	19.9	21.3	20.10	1.11	6.6	4 55	4 43	5 193	1 220	
	B100	<1 349	1.72	2.01	1.907	0.015	<0.404	17.7	21.5	20.10	1.111	1.9	1.55	1.15	1 900	1.220	
	B102	11017					01101								11900		
	B107	1.4541	1.4644	1.4529	1.457	0.006	6.4509	6.2862	6.3526	6.36	0.08	3.4766	3.5845	3.464	3.508	0.066	
	B109	1.25	1.31	1.23	1.263	0.042	4.44	4.13	4.11	4.23	0.19	2.82	3.18	3	3.000	0.180	
	B111																
	B112	1.284	1.311	1.346	1.314	0.031	5.769	5.752	5.339	5.62	0.24	2.826	3.12	2.843	2.930	0.165	
	B113	1.395	1.187	1.166	1.249	0.127	29.377	28.877	28.433	28.90	0.47	3.622	3.761	3.441	3.608	0.160	
	B119																
	B122																
ty		Consensu	s Mean		1.332		Consensu	is Mean		5.67		Consensu	is Mean		3.262		
uni Its		Consensu	s Standard	l Deviation	0.029		Consensu	is Standard	l Deviation	0.21		Consensu	is Standard	l Deviation	0.062		
nm		Maximum	ı		9.559		Maximum	ı		54.97		Maximum			20.133		
Com		Minimum			0.800		Minimum			0.61		Minimum			1.900		
<u> </u>		Ν			35		Ν			36		Ν			35		

Table 3-7.	continued.
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								I	Nickel (Ni)					Nickel (Ni)													
	_	SRM 15	75a Traco (Pinus)	e Elements s <i>taeda)</i> (n	s in Pine N ng/kg)	Needles	NRC	HEMP-1	(Plant Sar	nple 1) (n	ıg/kg)		Plant S	ample 4 (1	mg/kg)													
	Lab	Α	В	С	Avg	SD	Α	В	С	Avg	SD	Α	В	С	Avg	SD												
	Target				1.427	0.194				7.11	4.36				3.670	1.320												
	B125	1.048	1.198	1.088	1.111	0.078	1.188	1.138	1.028	1.12	0.08	2.458	2.608	2.818	2.628	0.181												
	B130	1.38	1.36	1.32	1.353	0.031	4.78	4.69	4.59	4.69	0.10	3.11	3.02	2.95	3.027	0.080												
	B139	1.42	1.38	1.41	1.403	0.021	7.29	7.1	7.03	7.14	0.13	3.52	3.8	3.82	3.713	0.168												
	B141	1.314	1.277	1.242	1.278	0.036	5.709	6.007	5.541	5.75	0.24	3.165	3.194	3.121	3.160	0.037												
	B142																											
	B148																											
	B155	<10	<10				<10	<10	<10			<10	<10	<10														
	B161	0.8	0.75	0.85	0.800	0.050	4.25	3.85	4.15	4.08	0.21																	
	B163	5.3	5.24	4.58	5.040	0.399	48.7	47.9	50.6	49.07	1.39	20.2	21	19.2	20.133	0.902												
	B172																											
	B174	1.377	1.217	1.436	1.343	0.113	5.471	5.184	5.302	5.32	0.14	3.48	3.156	3.415	3.350	0.171												
	B179	1.459	1.49	1.59	1.513	0.068	6.519	6.417	6.473	6.47	0.05	3.561	3.717	3.375	3.551	0.171												
2	B180	1.24	1.56	3.907	2.236	1.456	6.413	6.502	6.854	6.59	0.23	3.38	3.537	3.481	3.466	0.080												
lus	B182	1.47	1.49	1.28	1.413	0.116	5.76	5.84	5.44	5.68	0.21	3.14	3.47	3.44	3.350	0.182												
ual Re	B192	1.28	1.28	1.36	1.307	0.046	6.13	5.95	5.62	5.90	0.26	2.69	3.04	3.23	2.987	0.274												
	B198																											
vid	B200	1.36	1.41	1.39	1.387	0.025	6.75	6.74	6.68	6.72	0.04	3.58	3.66	3.42	3.553	0.122												
ibu	B202																											
-	B203	1.44	1.41	1.42	1.423	0.015	6.33	6.65	6.63	6.54	0.18	3.2	3.48	3.42	3.367	0.147												
	B204	1.148	1.139	1.132	1.140	0.008	5.832	5.877	5.899	5.87	0.03	3.369	3.109	3.696	3.391	0.294												
	B208																											
	B211	1.49			1.490		5.6			5.60		3.1			3.100													
	B213						7.6	7.2	6.5	7.10	0.56	4	3.4	3.8	3.733	0.306												
	B214																											
	B220																											
	B221	1.31	1.29	1.19	1.263	0.064	6.22	5.87	5.91	6.00	0.19	3.23	3.14	3.34	3.237	0.100												
	B222	1.43	1.46	1.41	1.433	0.025	5.277	5.036	5.438	5.25	0.20	3.37	3.63	3.6	3.533	0.142												
	B228																											
	B230																											
	B231																											
	B232	1.43333	1.4515	1.44134	1.442	0.009	6.24358	6.22493	6.44572	6.30	0.12	3.42726	3.64896	3.43354	3.503	0.126												
	B235	6.25	5.51	5.14	5.633	0.565	59.3	54.3	51.3	54.97	4.04	19.8	20	19.9	19.900	0.100												
<u>x</u>		Consensu	s Mean		1.332		Consensu	ıs Mean		5.67		Consensu	ıs Mean		3.262													
ts init		Consensu	s Standard	d Deviation	0.029		Consensu	ıs Standard	d Deviation	0.21		Consensu	ıs Standard	Deviation	0.062													
		Maximum	i.		9.559		Maximum	1		54.97		Maximum	1		20.133													
Re		Minimum			0.800		Minimum			0.61		Minimum			1.900													
0		Ν			35		Ν			36		Ν			35													



Fig. 3-46. Nickel in NRC HEMP-1 (Plant Sample 1) (data summary view – sample preparation method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 3-47. Nickel in NRC HEMP-1 (Plant Sample 1) (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 3-48. Nickel in Plant Sample 4 (data summary view – sample preparation method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean that results in an acceptable Z_{NIST} score, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 3-49. Nickel in Plant Sample 4 (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. The red shaded region represents the Interval for the consensus mean that result is uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 3-50. Nickel in SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (data summary view – sample preparation method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed as indicated in the figure key. 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, $|Z'_{comm}| \leq 2$. 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, $|Z_{NIST}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 3-51. Nickel in SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 3-52. Laboratory means for nickel in NRC HEMP-1 (Plant Sample 1) and Plant Sample 4 (sample/sample comparison view). In this view, the individual laboratory mean for one sample (NRC HEMP-1) is compared to the individual laboratory mean for a second sample (Plant Sample 4). The solid red box represents the NIST range of tolerance for the two samples, Plant Sample 4 (x-axis) and NRC HEMP-1 (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for Plant Sample 4 (x-axis) and NRC HEMP-1 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.



Fig. 3-53. Laboratory means for nickel in NRC HEMP-1 (Plant Sample 1) and SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (sample/sample comparison view). In this view, the individual laboratory mean for one sample (NRC HEMP-1) is compared to the individual laboratory mean for a second sample (SRM 1575a). The solid red box represents the NIST range of tolerance for the two samples, SRM 1575a (x-axis) and NRC HEMP-1 (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{NIST}| \le 2$. The dotted blue box represents the consensus range of tolerance for SRM 1575a (x-axis) and NRC HEMP-1 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{comm}| \le 2$.



Fig. 3-54. Laboratory means for nickel in Plant Sample 4 and SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (sample/sample comparison view). In this view, the individual laboratory mean for one sample (Plant Sample 4) is compared to the individual laboratory mean for a second sample (SRM 1575a). The solid red box represents the NIST range of tolerance for the two samples, SRM 1575a (x-axis) and Plant Sample 4 (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{NIST}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 1575a (x-axis) and Plant Sample 4 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{comm}| \leq 2$.

Table 3-8. Data summary table for selenium (Se) in the hemp and control samples. 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, $|Z'_{comm}| > 2$. *Note*: This table spans two pages; the NIST values and consensus values are included on both pages for convenience.

	ļ							Sr	elenium (S	Selenium (Se)										
		SRM 15	75a Traco (Pinus	e Element s taeda) (1	ts in Pine mg/kg)	Needles	NRC	HEMP-1	(Plant Sai	mple 1) (r	ng/kg)		Plant Sample 4 (mg/kg)							
	Lab	Α	В	С	Avg	SD	Α	В	С	Avg	SD	Α	В	С	Avg	SD				
	Target				0.0961	0.0078				0.305	0.060				0.8100	0.0120				
	B001	0.131	0.0819	0.115	0.1093	0.0250	0.33	0.28	0.227	0.279	0.052	0.0618	0.0812	0.076	0.0730	0.0100				
	B003	0.56863	0.70599	0.66687	0.6472	0.0708	0.02151	0.18229	0.13926	0.114	0.083	0.10719	0.15077	0.1677	0.1419	0.0312				
	B004	<1	<1	<1			<1	<1	<1			<1	<1	<1						
	B006									í				1						
	B017																			
	B018	0.112	0.114	0.097	0.1077	0.0093	0.54	0.622	0.555	0.572	0.044	< 0.182	< 0.182	< 0.182						
	B020																			
i I	B028				[i				!						
i I	B031																			
i I	B035	< 0.25	< 0.25	< 0.25			0.503	0.529	0.51	0.514	0.013	< 0.25	< 0.25	< 0.25						
	B037																			
ts	B040									·										
Ins	B049	0.109	0.12	0.114	0.1143	0.0055	0.431	0.43	0.446	0.436	0.009	0.099	0.099	0.092	0.0967	0.0040				
Re	B058	< 0.196	< 0.196	< 0.191			0.407	0.417	0.384	0.403	0.017	0.147	0.107	0.117	0.1237	0.0208				
ual	B061	0.1402	0.1615	0.1434	0.1484	0.0115	0.1037	0.0937	0.0882	0.095	0.008	0.104	0.0951	0.1046	0.1012	0.0053				
vid	B066	0.03	0.082	0.027	0.0463	0.0309	0.245	0.245	0.221	0.237	0.014	0.042	0.009	0.018	0.0230	0.0171				
ibu	B078																			
-	B079									1										
	B084																			
	B088	1.3747	1.5598	1.2385	1.3910	0.1613	<1	<1	<1	1		<1	<1	<1						
	B095	0.107	0.187	0.215	0.1697	0.0560	0.625	0.531	0.669	0.608	0.070	0.142	0.257	0.056	0.1517	0.1008				
	B100	< 0.139					< 0.464			1		< 0.464								
	B102																			
	B107	0.114	0.108	0.1042	0.1087	0.0049	< 0.006	< 0.006	< 0.006	1		0.169	0.1604	0.1509	0.1601	0.0091				
	B111																			
	B112	0.123	0.126	0.125	0.1247	0.0015	0.365	0.361	0.33	0.352	0.019	0.11	0.107	0.099	0.1053	0.0057				
	B113																			
	B119			- I						1				l l						
	B122																			
	B125	0.148	0.157	0.126	0.1437	0.0159	0.1048	0.0974	0.0904	0.098	0.007	0.103	0.111	0.116	0.1100	0.0066				
È		Consensu	ıs Mean		0.1191	I	Consens	us Mean		0.462		Consens	us Mean		0.1058					
uni Its		Consensu	ıs Standar	d Deviatio	0.0069	ŀ	Consens	us Standar	d Deviatio	0.051	I	Consens	us Standar	d Deviatio	1 0.0077					
nm		Maximum	а		1.3910	ŀ	Maximur	m		1.223	,	Maximur	n		0.5299					
R	1	Minimum	ı		0.0463	I	Minimur	n		0.095	,	Minimum	1		0.0230					
0		Ν			23	I	Ν			25	,	Ν			23					

		Selenium (Se)															
		SRM 15	75a Trac <i>(Pinus</i>	e Element s <i>taeda)</i> (1	ts in Pine mg/kg)	Needles	NRC	HEMP-1	(Plant Sa	mple 1) (1	ng/kg)	Plant Sample 4 (mg/kg)					
	Lab	Α	В	С	Avg	SD	Α	В	С	Avg	SD	Α	В	С	Avg	SD	
	Target				0.0961	0.0078				0.305	0.060				0.8100	0.0120	
	B130	0.12	0.13	0.12	0.1233	0.0058	0.81	0.76	0.85	0.807	0.045	0.1	0.11	0.1	0.1033	0.0058	
	B137	0.4316	0.497	0.4626	0.4637	0.0327	0.4562	0.8566	0.7328	0.682	0.205	0.4922	0.499	0.5986	0.5299	0.0596	
	B139	< 0.151	< 0.151	< 0.151			< 0.151	< 0.151	< 0.151			< 0.151	< 0.151	< 0.151			
	B141	0.138	0.112	0.129	0.1263	0.0132	0.324	0.321	0.317	0.321	0.004	0.108	0.083	0.091	0.0940	0.0128	
	B142																
	B148																
	B152																
	B155	<1	<1				<1	<1	<1			<1	<1	<1			
	B161																
	B163	0.12	0.116	0.102	0.1127	0.0095	0.744	0.711	0.76	0.738	0.025	0.115	0.111	0.102	0.1093	0.0067	
	B176	< 0.006	< 0.006	< 0.006			0.5595	0.7277	0.5632	0.617	0.096	< 0.006	< 0.006	< 0.006			
ults	B179	0.0969	0.0995	0.0994	0.0986	0.0015	0.651	0.639	0.673	0.654	0.017	0.08173	0.09054	0.08255	0.0849	0.0049	
tesu	B180	0.206	< 0.1	0.527	0.3665	0.2270	0.397	0.361	0.346	0.368	0.026	0.168	0.139	0.102	0.1363	0.0331	
al F	B192	0.131	0.128	0.138	0.1323	0.0051	1.29	1.16	1.22	1.223	0.065	0.114	0.117	0.129	0.1200	0.0079	
qui	B202																
livi	B203	0.091	0.103	0.105	0.0997	0.0076	0.35	0.336	0.328	0.338	0.011	0.067	0.068	0.065	0.0667	0.0015	
ľ	B208																
	B211	< 0.275					< 0.275					< 0.275					
	B213						0.3	0.29	0.29	0.293	0.006	0.08	0.07	0.08	0.0767	0.0058	
	B214																
	B220																
	B221	< 0.1	< 0.1	< 0.1			< 0.1	< 0.1	< 0.1			< 0.1	< 0.1	< 0.1			
	B222	0.092	0.1	0.09	0.0940	0.0053	0.347	0.416	0.442	0.402	0.049	0.07	0.067	0.081	0.0727	0.0074	
	B226	0.12257	0.12186	0.12112	0.1218	0.0007	0.57388	0.53944	0.55895	0.557	0.017	0.09871	0.09562	0.09656	0.0970	0.0016	
	B228																
	B230																
	B231																
	B232	0.13687	0.14892	0.13834	0.1414	0.0066	0.37434	0.40382	0.42516	0.401	0.026	0.11351	0.12629	0.12924	0.1230	0.0084	
	B235	0.127	0.115	0.116	0.1193	0.0067	0.801	0.849	0.804	0.818	0.027	0.234	0.256	0.261	0.2503	0.0144	
Į,		Consensu	us Mean		0.1191		Consensu	ıs Mean		0.462		Consens	us Mean		0.1058		
ini ts		Consensu	us Standar	d Deviatio	0.0069		Consense	us Standar	d Deviatio	0.051	0.051 Consensus Standard			d Deviatio	Deviatio 0.0077		
Im		Maximum	n		1.3910		Maximum	n		1.223		Maximur	n		0.5299		
Om		Minimum	ı		0.0463		Minimum	ı		0.095		Minimun	1		0.0230		
0		Ν			23		Ν			25		Ν			23		



Fig. 3-55. Selenium in NRC HEMP-1 (Plant Sample 1) (data summary view – sample preparation method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 3-56. Selenium in NRC HEMP-1 (Plant Sample 1) (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean that results in an acceptable Z_{NIST} score, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

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Fig. 3-57. Selenium in Plant Sample 4 (data summary view – sample preparation method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 3-58. Selenium in Plant Sample 4 (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. The red shaded region represents the Interval for the consensus mean that result is uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 3-59. Selenium in SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (data summary view – sample preparation method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed as indicated in the figure key. 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, $|Z'_{comm}| \leq 2$. 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, $|Z_{NIST}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).





Fig. 3-60. Selenium in SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Exercise: CannaQAP Exercise 2, Measurand: selenium No. of laboratories: 22

Fig. 3-61. Laboratory means for selenium in NRC HEMP-1 (Plant Sample 1) and Plant Sample 4 (sample/sample comparison view). In this view, the individual laboratory mean for one sample (NRC HEMP-1) is compared to the individual laboratory mean for a second sample (Plant Sample 4). The solid red box represents the NIST range of tolerance for the two samples, Plant Sample 4 (x-axis) and NRC HEMP-1 (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The dotted blue box (the left limit is not shown due to the scale of the figure) represents the consensus range of tolerance for Plant Sample 4 (x-axis) and NRC HEMP-1 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.



Fig. 3-62. Laboratory means for selenium in NRC HEMP-1 (Plant Sample 1) and SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (sample/sample comparison view). In this view, the individual laboratory mean for one sample (NRC HEMP-1) is compared to the individual laboratory mean for a second sample (SRM 1575a). The solid red box represents the NIST range of tolerance for the two samples, SRM 1575a (x-axis) and NRC HEMP-1 (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{NIST}| \le 2$. The dotted blue box (the bottom limit is not shown due to the scale of the figure) represents the consensus range of tolerance for SRM 1575a (x-axis) and NRC HEMP-1 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{comm}| \le 2$.



Exercise: CannaQAP Exercise 2, Measurand: selenium

Fig. 3-63. Laboratory means for selenium in Plant Sample 4 and SRM 1575a Trace Elements in Pine Needles (Pinus taeda) (sample/sample comparison view). In this view, the individual laboratory mean for one sample (Plant Sample 4) is compared to the individual laboratory mean for a second sample (SRM 1575a). The solid red box represents the NIST range of tolerance for the two samples, SRM 1575a (x-axis) and Plant Sample 4 (y-axis), which encompasses the target values bounded by their uncertainties (UNIST) and represents the range that results in an acceptable ZNIST score, $|Z_{\text{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 1575a (x-axis) and Plant Sample 4 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{comm}| \le 2$.

Table 3-9. Data summary table for uranium (U) in the hemp and control samples. 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, $|Z'_{\text{comm}}| > 2$.

		Uranium (U)														
		SRM 15	75a Trac (Pinu:	e Elemen s <i>taeda)</i> (ts in Pine mg/kg)	Needles	NRC	C HEMP-	1 (Plant S	ample 1) (n	ng/kg)		Plant S	Sample 4	(mg/kg)	
	Lab	Α	В	С	Avg	SD	Α	В	С	Avg	SD	Α	В	С	Avg	SD
	Target									0.454	0.120				0.00426	0.00120
	B001	0.004	0.005	0.0037	0.00423	0.00068	0.193	0.214	0.215	0.207	0.012	0.0021	0.0019	0.002	0.00200	0.00010
	B003															
	B004	<1	<1	<1			<1	<1	<1			<1	<1	<1		
	B006															
	B017															
	B020															
	B028															
	B031															
	B035	< 0.01	< 0.01	< 0.01			0.243	0.25	0.247	0.247	0.004	< 0.01	< 0.01	< 0.01		
	B037															
	B049						0.268	0.283	0.248	0.266	0.018					
	B061															
	B066	0.005	0.006	0.006	0.00567	0.00058	0.32	0.29	0.318	0.309	0.017	0.004	0.004	0.004	0.00400	0.00000
	B078															
	B079															
	B084															
~	B102															
	B111															
	B112	0.0065	0.007	0.008	0.00717	0.00076	0.145	0.139	0.186	0.157	0.026	0.0041	0.0045	0.004	0.00420	0.00026
sult	B113			_												
Re	B119															
ıal	B122															
idu	B125	0.0033	0.0029	0.0037	0.00330	0.00040	0.126	0.163	0.171	0.153	0.024	0.002	0.002	0.002	0.00200	
vibi	B139	0.004	0.004	0.005	0.00433	0.00058	0.242	0.234	0.26	0.245	0.013	0.003	0.003	0.003	0.00300	
H	B141	0.004	0.006	0.005	0.00500	0.00100	0.252	0.25	0.231	0.244	0.012	0.006	0.004	0.003	0.00433	0.00153
	B142															
	B148	.0.1	.0.1				0.53	0.54	0.52	0.522	0.007	-0.1	-0.1	-0.1		
	BI55	<0.1	<0.1				0.53	0.54	0.53	0.533	0.006	<0.1	<0.1	<0.1		
	BIGI	0.00/05	0.00607	0.00565	0.00600	0.00020	11.8	11.8	12.2	11.933	0.231	0.00450	0.00056	0.0000	0.00000	0.00070
	B163	0.00625	0.00607	0.00567	0.00600	0.00030	0.399	0.384	0.385	0.389	0.008	0.00478	0.00356	0.0033	0.00388	0.00079
	B1/9	0.00468	0.00444	0.00433	0.00448	0.00018	0.235	0.23	0.239	0.235	0.005	0.00268	0.00246	0.00236	0.00250	0.00016
	B200	0.0052	0.0056	0.0055	0.00543	0.00021	0.4/4	0.506	0.509	0.496	0.019	0.0044	0.0039	0.0043	0.00420	0.00026
	B202	0.005	0.000	0.005	0.00522	0.00059	0.270	0.277	0.270	0.279	0.001	0.004	0.005	0.002	0.002(7	0.00152
	B203	0.005	0.006	0.005	0.00533	0.00058	0.279	0.277	0.279	0.278	0.001	0.004	0.005	0.002	0.00367	0.00155
	B208	<1					<1					<1				
	D211 D212	<1					0.27	0.25	0.21	0.242	0.021	< 0.02	< 0.02	< 0.02		
	D213						0.27	0.23	0.21	0.243	0.031	< 0.02	< 0.02	< 0.02		
	D214	<0.01	<0.01	<0.01			0.197	0.179	0.160	0.179	0.000	<0.01	<0.01	<0.01		
	B221 B222	<0.01	<0.01	<0.01			0.187	0.178	0.109	0.178	0.009	<0.01	<0.01	<0.01		
	B222	<0.01	<0.01	<0.01			0.229	0.229	0.230	0.231	0.004	<0.01	<0.01	<0.01		
	B220 B230															
	B230															
	B232	0.007	0.00529	0.00485	0.00572	0.00113	0 24526	0 23871	0.25673	0 247	0.009	0.00251	0.00287	0.00342	0.00293	0.00045
	B235	0.00727	0.00605	0.00584	0.00639	0.00077	0.397	0.417	0.403	0.406	0.009	0.00255	0.00244	0.0023	0.00243	0.00013
x	2200	Consensi	is Mean		0.00525		Consensi	ıs Mean	005	0.268	0.013	Consens	us Mean	5.0025	0.00326	0.00015
ts :		Consensi	is Standar	d Deviatio	0.00034		Consensi	is Standar	d Deviation	0.020		Consens	us Standar	d Deviatio	0.00031	
mu sult		Maximum	1		0.00717		Maximum	an		11.933		Maximur	n		0.00433	
om Re		Minimum	L		0.00330		Minimum	1		0.153		Minimum	1		0.00200	
C		N			12		N			19		N			10	



Fig. 3-64. Uranium in NRC HEMP-1 (Plant Sample 1) (data summary view – sample preparation method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



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Fig. 3-65. Uranium in NRC HEMP-1 (Plant Sample 1) (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean that results in an acceptable Z_{NIST} score, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

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Fig. 3-66. Uranium in Plant Sample 4 (data summary view – sample preparation method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 3-67. Uranium in Plant Sample 4 (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. The red shaded region represents the range that results in an acceptable Z_{NIST} score, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (red region) and the NIST range of tolerance (red region).



Fig. 3-68. Uranium in SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (data summary view – sample preparation method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed as indicated in the figure key. 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, $|Z'_{comm}| \leq 2$. A NIST value has not been determined in this material.


Fig. 3-69. Uranium in SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$.



Fig. 3-70. Laboratory means for uranium in NRC HEMP-1 (Plant Sample 1) and Plant Sample 4 (sample/sample comparison view). In this view, the individual laboratory mean for one sample (NRC HEMP-1) is compared to the individual laboratory mean for a second sample (Plant Sample 4). The solid red box (the right limit is not shown due to the scale of the figure) represents the NIST range of tolerance for the two samples, Plant Sample 4 (x-axis) and NRC HEMP-1 (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{NIST}| \le 2$. The dotted blue box represents the consensus range of tolerance for Plant Sample 4 (x-axis) and NRC HEMP-1 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{comm}| \le 2$.



Exercise: CannaQAP Exercise 2, Measurand: uranium No. of laboratories: 12

Fig. 3-71. Laboratory means for uranium in NRC HEMP-1 (Plant Sample 1) and SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (sample/sample comparison view). In this view, the individual laboratory mean for one sample (NRC HEMP-1) is compared to the individual laboratory mean for a second sample (SRM 1575a). The dotted blue box represents the consensus range of tolerance for SRM 1575a (x-axis) and NRC HEMP-1 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{comm}| \le 2$.



Fig. 3-72. Laboratory means for uranium in Plant Sample 4 and SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (sample/sample comparison view). In this view, the individual laboratory mean for one sample (Plant Sample 4) is compared to the individual laboratory mean for a second sample (SRM 1575a). The dotted blue box represents the consensus range of tolerance for SRM 1575a (x-axis) and Plant Sample 4 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{comm}| \le 2$.

Table 3-10. Data summary table for vanadium (V) in the hemp and control samples. 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, $|Z'_{comm}| > 2$. *Note*: This table spans two pages; the NIST values and consensus values are included on both pages for convenience.

		Vanadium (V)														
		SRM 15	75a Traco <i>(Pinus</i>)	e Element s <i>taeda)</i> (1	ts in Pine mg/kg)	Needles	NRC	HEMP-1	(Plant Sa	mple 1) (n	ng/kg)		Plant S	ample 4 (mg/kg)	
	Lab	Α	В	С	Avg	SD	Α	В	С	Avg	SD	Α	В	С	Avg	SD
	Target									16.70	1.70				0.233	0.033
	B001	0.0789	0.0844	0.0879	0.0837	0.0045	6.39	8.36	8.28	7.68	1.12	0.165	0.142	0.137	0.148	0.015
	B003															
	B004	<1	<1	<1			10.14	9.72	9.31	9.72	0.42	<1	<1	<1		
	B006															
	B017															
	B018	0.094	0.096	0.094	0.0947	0.0012	12.183	12.051	12.413	12.22	0.18	0.216	0.191	0.189	0.199	0.015
	B020															
	B028															
	B031															
	B035	0.089	0.087	0.087	0.0877	0.0012	15.491	14.03	15.249	14.92	0.78	0.186	0.201	0.181	0.189	0.010
lts	B037															
dual Resul	B040															
	B049	0.096			0.0960		13.4	14.4	8.9	12.23	2.93	0.212	0.139	0.138	0.163	0.042
	B058	0.105	0.0904	0.0936	0.0963	0.0077	14.1	13.5	13.1	13.57	0.50	0.174	0.131	0.219	0.175	0.044
livi	B061															
Ind	B066	0.112	0.106	0.113	0.1103	0.0038	15.805	15.129	15.18	15.37	0.38	0.214	0.232	0.183	0.210	0.025
	B078															
	B079															
	B084	.1	.1	. 4			.1	.1	.1			10 (100	0.000	10.540	10.144	0.750
	B088	<1	<1	<1	0.1227	0.0264	<1	<1	<1	10.57	2.22	10.6199	9.2692	10.542	10.144	0.758
	B095	0.164	0.116	0.121	0.1337	0.0264	14.305	8.727	14.674	12.57	3.33	0.238	0.242	0.282	0.254	0.024
	B102															
	BIII D112	0.102	0.106	0.080	0.0000	0.0080	10.22	11.74	0.72	10.50	1.04	0.149	0.17	0.147	0.155	0.012
	D112	0.102	0.100	0.089	0.0990	0.0089	10.52	11./4	9.72	10.39	1.04	0.148	0.17	0.147	0.155	0.015
	D115															
	D119															
	B122 B125	0.0763	0.0892	0.0767	0.0807	0.0073	2 75	2 75	2.56	2 69	0.11	0.157	0 149	0.15	0.152	0.004
~	D125	Consensi	Is Mean	0.0707	0.1041	0.0075	Consensi	is Mean	2.50	13.83	0.11	Consensi	is Mean	0.15	0.208	0.004
nit.		Consensi	is Standar	d Deviatio	0.0050		Consensus Standard Deviatio: 0.70				Consensus Standard Deviation 0.015					
mu sult		Maximun	1		0.3623		Maximun	n		25.87		Maximum	1		10.144	
om Re		Minimum	-		0.0807		Minimum	1		2.69		Minimum	-		0.141	
Co F		N			17		N			24		N 20				

								anadium (n (V)							
		SRM 15	75a Trace <i>(Pinus</i>	Element <i>taeda)</i> (1	ts in Pine mg/kg)	Needles	NRC	HEMP-1	(Plant Sa	mple 1) (n	ng/kg)		Plant S	ample 4 (1	mg/kg)	
	Lab	Α	В	С	Avg	SD	Α	В	С	Avg	SD	Α	В	С	Avg	SD
	Target									16.70	1.70				0.233	0.033
	B130	0.13	0.13	0.12	0.1267	0.0058	14.53	14.18	14.03	14.25	0.26	0.25	0.26	0.24	0.250	0.010
	B139	0.111	0.111	0.105	0.1090	0.0035	15.3	15.5	16.2	15.67	0.47	0.2	0.235	0.238	0.224	0.021
	B141	0.106	0.104	0.106	0.1053	0.0012	16.125	16.083	15.302	15.84	0.46	0.301	0.192	0.225	0.239	0.056
	B142															
	B148															
	B155	<2					21.4	19.1		20.25	1.63	<1	<1			
	B161						5.6	5	5.6	5.40	0.35					
	B163	0.203	0.207	0.22	0.2100	0.0089	25.6	24.9	27.1	25.87	1.12	0.44	0.353	0.346	0.380	0.052
s	B176															
sult	B179	0.1144	0.1194	0.118	0.1173	0.0026	16.23	15.81	16.91	16.32	0.56	0.2266	0.2712	0.2123	0.237	0.031
idual Res	B180	< 0.1	< 0.1	< 0.1			15.724	15.672	16.572	15.99	0.51	0.139	0.129	0.154	0.141	0.013
	B202															
	B203	0.321	0.371	0.395	0.3623	0.0378	16.7	17.5	16.9	17.03	0.42	0.261	0.281	0.297	0.280	0.018
vibr	B208															
I	B211	< 0.002					14.7			14.70		< 0.002				
	B213						16	15	13	14.67	1.53	< 0.5	< 0.5	< 0.5		
	B214															
	B221	0.14	0.09	0.085	0.1050	0.0304	8.7	8.3	8.2	8.40	0.26	0.151	0.147	0.146	0.148	0.003
	B222	< 0.01	< 0.01	< 0.01			15.114	15.116	14.846	15.03	0.16	< 0.01	< 0.01	< 0.01		
	B228															
	B230															
	B231															
	B232	0.10915	0.10873	0.1107	0.1095	0.0010	14.2907	14.0678	15.0788	14.48	0.53	0.19337	0.23767	0.24844	0.226	0.029
	B235	0.117	0.112	0.104	0.1110	0.0066	20.5	20.6	20.4	20.50	0.10	0.294	0.231	0.25	0.258	0.032
Ś		Consensu	ıs Mean		0.1041		Consensu	ıs Mean		13.83		Consense	ıs Mean		0.208	
ts ts		Consensu	is Standard	d Deviatio	0.0050		Consensu	ıs Standar	d Deviatio	0.70		Consense	ıs Standar	d Deviatio	0.015	
and Inc.		Maximun	n		0.3623		Maximun	n		25.87		Maximur	n		10.144	
Re		Minimum	L		0.0807		Minimum 2.69			Minimum 0.141						
0	N 17			N 24				N 20								



Fig. 3-73. Vanadium in NRC HEMP-1 (Plant Sample 1) (data summary view – sample preparation method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 3-74. Vanadium in NRC HEMP-1 (Plant Sample 1) (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

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Fig. 3-75. Vanadium in Plant Sample 4 (data summary view – sample preparation method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. 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, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 3-76. Vanadium in Plant Sample 4 (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. The red shaded region represents the Interval for the consensus mean that result is uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{NIST}| \le 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).



Fig. 3-77. Vanadium in SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (data summary view – sample preparation method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed as indicated in the figure key. 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, $|Z'_{comm}| \leq 2$. A NIST value has not been determined in this material.



Fig. 3-78. Vanadium in SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed as indicated in the figure key. 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, $|Z'_{comm}| \le 2$. A NIST value has not been determined in this material.



Fig. 3-79. Laboratory means for vanadium in NRC HEMP-1 (Plant Sample 1) and Plant Sample 4 (sample/sample comparison view). In this view, the individual laboratory mean for one sample (NRC HEMP-1) is compared to the individual laboratory mean for a second sample (Plant Sample 4). The solid red box represents the NIST range of tolerance for the two samples, Plant Sample 4 (x-axis) and NRC HEMP-1 (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for Plant Sample 4 (x-axis) and NRC HEMP-1 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.



Exercise: CannaQAP Exercise 2, Measurand: vanadium No. of laboratories: 18

Fig. 3-80. Laboratory means for vanadium in NRC HEMP-1 (Plant Sample 1) and SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (sample/sample comparison view). In this view, the individual laboratory mean for one sample (NRC HEMP-1) is compared to the individual laboratory mean for a second sample (SRM 1575a). The dotted blue box represents the consensus range of tolerance for SRM 1575a (x-axis) and NRC HEMP-1 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{comm}| \le 2$.



Fig. 3-81. Laboratory means for vanadium in Plant Sample 4 and SRM 1575a Trace Elements in Pine Needles (*Pinus taeda*) (sample/sample comparison view). In this view, the individual laboratory mean for one sample (Plant Sample 4) is compared to the individual laboratory mean for a second sample (SRM 1575a). The dotted blue box represents the consensus range of tolerance for SRM 1575a (x-axis) and Plant Sample 4 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{comm}| \le 2$.

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Appendix A. Method Questionnaire Responses

Forty-nine laboratories completed the method questionnaire out of the 93 labs that reported results.

Lab Identification Code

Lab Identification Code											
B003	B035	B082	B142	B172	B193	B212					
B012	B041	B088	B146	B178	B195	B213					
B015	B060	B100	B147	B181	B202	B216					
B016	B061	B102	B153	B182	B203	B217					
B027	B064	B104	B154	B186	B204	B222					
B029	B066	B109	B155	B190	B205	B223					
B030	B077	B113	B159	B192	B206	B224					

Sample Preparation

Homogenization



Response					Labo	oratory				
Yes	B012	B015	B027	B029	B030	B035	B064	B066	B077	B088
	B100	B104	B109	B146	B159	B186	B190	B195	B202	B204
	B205	B206	B212	B213	B224					
No	B003	B016	B041	B060	B061	B082	B102	B113	B142	B147
	B153	B154	B155	B172	B178	B181	B182	B192	B193	B203
	B216	B217	B222	B223						

Homogenization Method



Homogenization Method		Laboratory												
Mixed	B012	B029	B030	B035	B064	B077	B088	B100	B109	B146				
	B159	B186	B190	B212	B213	B016*	B060*	B142*	B178*					
Grinding	B066	B202	B205	B224										
Cryogrinding	B027	B206												
Cut with Scissors	B015													
Not Specified	B104	B195	B204											

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

Sample Size



Sample Size					Labo	ratory				
0.125 g	B178									
0.15 g	B077									
0.2 g	B193	B205	B224							
0.25 g	B102	B186								
0.3 g	B142	B153								
0.5 g	B012	B015	B016	B035	B041	B060	B082	B088	B100	B104
	B113	B146	B147	B154	B155	B159	B172	B181	B182	B190
	B192	B204	B206	B223						
Not Specified	B003	B027	B029	B030	B061	B064	B066	B109	B195	B202
	B203	B212	B213	B216	B217	B222				

Sample Preparation Method Type



Preparation Method		Laboratory											
Microwave Digestion	B003	B012	B015	B016	B027	B029	B030	B035	B041	B060			
	B061	B064	B066	B077	B082	B100	B102	B104	B113	B142			
	B146	B147	B153	B154	B159	B172	B178	B181	B182	B186			
	B190	B192	B193	B195	B202	B204	B206	B212	B213	B216			
	B217	B222	B223	B224									
Hot Block Digestion	B109	B205											
Open Breaker Digestion	B088												
Samples were not digested	B155												
Not Specified	B203												

Acids Used for Digestion



Digestion Acids		Laboratory										
HCl, HNO ₃	B003	B027	B029	B041	B060	B061	B064	B066	B077	B088		
	B102	B104	B109	B142	B146	B172	B181	B186	B192	B193		
	B195	B204	B206	B212	B213	B216	B224					
HNO ₃	B015	B035	B082	B113	B153	B159	B178	B190	B202	B217		
	B222	B223										
H ₂ O ₂ , HNO ₃	B012	B016	B100	B147	B154							
HCl, H ₂ O ₂ , HNO ₃	B030	B182										
H ₂ O ₂ , HNO ₃ , H ₂ SO ₄	B205											
Samples were not digested	B155											
Not Specified	B203											

Maximum Microwave Temperature



*Laboratories that did not specify their microwave procedures were not included on the figure

Maximum Temperature		Laboratory												
180 °C	B012	B064	B113	B154	B159	B178								
185 °C	B027													
190 °C	B182	B222												
195 ° С	B153													
200 °C	B016	B041	B082	B104	B172	B192								
210 °C	B003	B146												
225 °C	B142													
230 °C	B060	B077												
240 °C	B181	B193	B224											
Not Specified	B015	B029	B030	B035	B061	B066	B100	B102	B147	B186				
	B190	B195	B202	B204	B206	B212	B213	B216	B217	B223				

Length of Microwave Digestion Method



*Laboratories that did not specify their microwave procedures were not included on the figure

Digestion Time					Labo	ratory				
20 min	B012	B146	B222	B224						
30 min	B041	B060	B104							
35 min	B172	B192								
40 min	B077	B159	B182							
45 min	B142									
50 min	B153	B178								
55 min	B027	B193								
60 min	B003	B064	B181							
65 min	B016									
70 min	B204									
73 min	B082									
75 min	B113									
90 min	B154									
Not Specified	B015	B029	B030	B035	B061	B066	B100	B102	B147	B186
	B190	B195	B202	B206	B212	B213	B216	B217	B223	

General Analytical Methods

Analytical Method



Analytical Method	Laboratory									
ICP-MS	B003	B012	B015	B016	B027	B029	B030	B035	B041	B060
	B061	B064	B066	B077	B082	B100	B102	B104	B109	B113
	B142	B146	B147	B154	B159	B172	B178	B181	B182	B186
	B190	B192	B193	B195	B202	B204	B206	B212	B216	B217
	B222	B223	B224							
AAS	B153									
ICP-MS, CV-AAS	B205									
ICP-OES	B088									
ICP-OES, ICP-MS	B213									
NAA	B155									
Not Specified	B203									

General Calibration Approach



Calibration Approach	Laboratory								
Calibration Curve	B015	B029	B030	B035	B060	B061	B064	B082	
	B113	B142	B147	B153	B159	B178	B181	B182	
	B190	B193	B205	B212	B213	B216	B217	B222	
Calibration Curve with Standards	B003	B012	B016	B041	B077	B088	B100	B102	
	B104	B109	B146	B154	B172	B186	B192	B195	
	B202	B204	B206	B223	B224				
Standards Only	B027	B066	B155						
Not Specified	B203								

Calibration Approach



Calibration Approach	Laboratory							
Calibration Curve	B015	B029	B030	B035	B060	B061	B064	B082
	B113	B142	B147	B153	B159	B178	B181	B182
	B190	B193	B205	B212	B213	B216	B217	B222
Calibration Curve with Internal	B003	B012	B016	B041	B077	B100	B102	B104
Standard	B109	B146	B154	B172	B186	B192	B195	B202
	B206	B223	B224					
Calibration Curve with External Standards	B088							
Calibration Curve with Internal and	B204							
External Standards								
Internal Standard	B027							
External Standards	B155							
Internal and External Standards	B066							
Not Specified	B203							