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

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

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## Publication History

Approved by the NIST Editorial Review Board on 2022-12-12

## How to Cite this NIST Technical Series Publication

Barber CA, Bryan Sallee CE, Burdette CQ, Kotoski SP, Phillips MM, Wilson WB, Wood LJ (2022) Cannabis Quality Assurance Program: Exercise 2 Toxic Elements Final Report. (National Institute of Standards and Technology, Gaithersburg, MD), NIST Interagency/Internal Report (IR) NIST IR 8452.
https://doi.org/10.6028/NIST.IR. 8452

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#### 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, $Z^{\prime}$ 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 ( $\mathrm{x}^{*}$ ), consensus standard deviation ( $\mathrm{s}^{*}$ ), and standard deviation for proficiency assessment (SDPA, $\sigma_{P T}^{2}$ ) determined from the $\mathrm{Q} /$ Hampel estimator:

$$
Z^{\prime}{ }_{\mathrm{comm}}=\frac{x_{i}-x *}{\sqrt{\sigma_{P T}^{2}+s^{* 2}}}
$$

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

$$
Z_{\mathrm{NIST}}=\frac{x_{i}-x_{\mathrm{NIST}}}{2 * U_{95}}
$$

or

$$
Z_{\mathrm{NIST}}=\frac{x_{i}-x_{\mathrm{NIST}}}{2 * U_{\mathrm{NIST}}}
$$

The significance of the $Z$-score and $Z^{\prime}$-score is as follows:

[^0]- $|Z|<2$ indicates that the laboratory result is considered to be within the community consensus range (for $Z^{\prime}{ }_{\text {comm }}$ ) or NIST target range (for $Z_{\text {NIST }}$ ).
- $2<|Z|<3$ indicates that the laboratory result is considered to be marginally different from the community consensus value (for $Z_{\text {comm }}^{\prime}$ ) or NIST target value (for $Z_{\text {NIST }}$ ).
- $|Z|>3$ indicates that the laboratory result is considered to be significantly different from the community consensus value (for $Z^{\prime}$ comm) or NIST target value (for $Z_{\text {NIST }}$ ).

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 $\left|Z^{\prime}{ }_{\text {comm }}\right|>2$.

## 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 $\mathrm{Q} /$ 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 $\left(s_{r}\right)$, the reproducibility standard deviation $\left(s_{\mathrm{R}}\right)$, the number of participants reporting data ( $n_{\text {particpants }}$ ), and the average number of replicates reported by each participant ( $n_{\text {Average Number of Replicates per Participant }}$ ). The uncertainty about the consensus mean is independent of the range of tolerance.

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

The red shaded region represents the target zone that encompasses the NIST target value bounded by twice its uncertainty ( $U_{95}$ or $U_{\text {NIST }}$ ). The solid red lines represent the range of tolerance (values that result in the target zone $\mid Z^{\prime}$ score $\mid \leq 2$ ). 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 $\mid Z^{\prime}$ score $\mid \leq 2$ ). 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 ( $\approx 15 \mathrm{~g}$ ) and stored at $-20^{\circ} \mathrm{C}$. Upon arrival at NIST, 108 amber bottles were stored at $-80^{\circ} \mathrm{C}$ 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 $(\approx 3 \mathrm{~g})$ into plastic bags which were heat sealed, placed in mylar bags with desiccant silica pouches, and stored at $-20^{\circ} \mathrm{C}$ 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 $250 \mu \mathrm{~m}$ to $710 \mu \mathrm{~m}$ and mixed for 30 min . The bulk material was immediately stored in the dark at $-80^{\circ} \mathrm{C}$. During sample packaging, a portion of the bulk material was removed and packaged in small quantities ( $\approx 3 \mathrm{~g}$ ) into plastic bags which were heat sealed, placed in mylar bags with desiccant silica pouches and stored at $-20^{\circ} \mathrm{C}$ 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 ( $\approx 3 \mathrm{~g}$ ) into plastic bags which were heat sealed, placed in mylar bags with desiccant silica pouches and stored at $-20^{\circ} \mathrm{C}$ 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 $\mathrm{As}, \mathrm{Co}, \mathrm{Pb}, \mathrm{Mn}, \mathrm{Ni}$, 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, $\mathrm{Cd}, \mathrm{Co}, \mathrm{Cr}, \mathrm{Mo}, \mathrm{Ni}, \mathrm{Pb}, \mathrm{Se}$, 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 $\mathrm{HNO}_{3}, 2 \mathrm{~mL}$ of

Optima grade HF, and internal standard solutions of $\mathrm{Sb}(8 \mathrm{ng} / \mathrm{g})$ and $\mathrm{Rh}(1 \mathrm{ng} / \mathrm{g})$. 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 $195{ }^{\circ} \mathrm{C}$ ( 20 min hold time) followed by 1600 W ( $85 \%$ power) for 20 min ramp time at $205{ }^{\circ} \mathrm{C}$ ( 15 min hold time). Samples were then transferred to polyethylene bottles and diluted to 60 g using $18 \mathrm{M} \Omega \cdot \mathrm{cm}$ water. Samples were diluted so that $\mathrm{As}, \mathrm{Cd}, \mathrm{Co}, \mathrm{Cr}, \mathrm{Mo}, \mathrm{Ni}, \mathrm{Pb}, \mathrm{Se}$, and U were present at approximate mass fractions of $0.001 \mathrm{mg} / \mathrm{kg}$ to $0.004 \mathrm{mg} / \mathrm{kg}$ for analysis by ICP-MS.

## Analysis

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 $\mathrm{H}_{2}$ as a collision gas to minimize polyatomic interferences for $\mathrm{As}, \mathrm{Cd}, \mathrm{Cr}, \mathrm{Mo}, \mathrm{Ni}, \mathrm{Pb}, \mathrm{Se}$, and U and using He as a collision gas to minimize polyatomic interferences for Co.

| ICP-MS Parameter | $\underline{\text { Setting }}$ |
| :--- | :---: |
| Argon flow | $15 \mathrm{~L} / \mathrm{min}$ |
| Auxiliary flow | $0.8 \mathrm{~L} / \mathrm{min}$ |
| Nebulizer flow | $1 \mathrm{~L} / \mathrm{min}$ |
| Radiofrequency (RF) power | 1500 W |


| Element | $\frac{\text { Mass }}{\text { (amu) }}$ | Internal standard (mass) | Integration time/point (s) | Read time/mass (s) |
| :---: | :---: | :---: | :---: | :---: |
| 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 $\mathrm{HNO}_{3}$ and 2 mL of Optima grade HF. The samples were digested using a microwave sample preparation system (CEM MARSXpress, Matthews, NC, USA) at $800 \mathrm{~W}\left(100 \%\right.$ power ) for 15 min ramp time at $195^{\circ} \mathrm{C}(20$ min hold time) followed by $1600 \mathrm{~W}\left(85 \%\right.$ power) for 20 min ramp time at $205^{\circ} \mathrm{C}(15 \mathrm{~min}$ hold time). Samples were then transferred to polyethylene bottles and diluted to 60 g using $18 \mathrm{M} \Omega \cdot \mathrm{cm}$ water. Samples were diluted so that Mn was present at an approximate mass fraction of $0.6 \mathrm{mg} / \mathrm{kg}$ for analysis by ICP-OES.

## Analysis

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

| ICP-OES Parameter | $\underline{\text { Setting }}$ |
| :--- | :---: |
| Argon flow | $12 \mathrm{~L} / \mathrm{min}$ |
| Auxiliary flow | $0.2 \mathrm{~L} / \mathrm{min}$ |
| Nebulizer flow | $0.7 \mathrm{~L} / \mathrm{min}$ |
| Radiofrequency (RF) power | 1500 W |


| Element | $\frac{\text { Wavelength }}{}$ |  |  |
| :--- | :--- | :--- | :--- | :--- |
|  | $\frac{(\mathrm{nm})}{\text { Integration }}$ |  |  |
| 257.610 | $\frac{\text { Plasma View }}{\text { Axial }}$ | $\frac{\text { time }(\mathrm{s})}{0.10}$ | $\frac{\text { Read time }(\mathrm{s})}{1.000}$ |

## Quantitation

Mn mass fractions were quantified by using a four-point calibration curve with values ranging from $0.3 \mathrm{mg} / \mathrm{kg}$ to $1.2 \mathrm{mg} / \mathrm{kg}$. 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 $\mathrm{HNO}_{3}$ and 1 mL Optima grade HCl were added to each vessel along with an internal standards solution of Sc and Y (each approximately $0.88 \mathrm{mg} / \mathrm{kg}$ ). The samples were digested using an Anton Paar Multiwave 3000 microwave sample preparation system (Ashland, VA) at $600 \mathrm{~W}, 10 \mathrm{~min}$ ramp time and 15 min hold time followed by $1400 \mathrm{~W}, 10 \mathrm{~min}$ ramp time and 20 min hold time. Samples were then transferred to polypropylene centrifuge tubes and diluted to 50 g using $18 \mathrm{M} \Omega \cdot \mathrm{cm}$ 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 $18 \mathrm{M} \Omega \cdot \mathrm{cm}$ water. Samples were diluted so that Be and V were present at appropriate mass fractions for analysis by QQQ-ICP-MS.

## Analysis

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 $(0.2 \mathrm{~L} / \mathrm{min})$ and the He collision mode ( $4.8 \mathrm{~mL} / \mathrm{min}$ ) was utilized for measuring V .

| Parameter | $\underline{\text { Setting }}$ |
| :--- | :---: |
| Argon carrier gas flow | $1.06 \mathrm{~L} / \mathrm{min}$ |
| Nebulizer pump | 0.1 rps |
| Radiofrequency (RF) power | 1550 W |


|  | Mass | Internal standard | Integration time/mass |
| :---: | :---: | :---: | :---: |
| Element | (amu) | (mass) | (s) |
| 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 ( $\mu \mathrm{g} / \mathrm{kg}$ ).

## 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.

## Analysis

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

| Sample Type | Ramp Time (s) | Method Parameters <br> Temperature $\left({ }^{\circ} \mathrm{C}\right)$ | Hold Time (s) |
| :---: | :---: | :---: | :---: |
| Aqueous Solution (calibration curve) | 90 | 200 | 30 |
|  | 90 | 650 | 180 |
| Plant Materials | 90 | 200 | 30 |
|  | 60 | 300 | 60 |
|  | 60 | 450 | 30 |
|  | 60 | 650 | 240 |

## 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 ( $\mu \mathrm{g} / \mathrm{kg}$ ).

### 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 ( $\approx-20^{\circ} \mathrm{C}$ ) 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 ( $\mathrm{mg} / \mathrm{kg}$ ) 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 ( $20^{\circ} \mathrm{C}$ to $25^{\circ} \mathrm{C}$ ) 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 ( $\mathrm{mg} / \mathrm{kg}$ ) 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 following hemp consumption. Currently each state sets allowable limits for specific 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 $\mathrm{As}, \mathrm{Cd}, \mathrm{Hg}$, 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 | SRM 1575a |
| :---: | :---: | :---: | :---: | :---: |
| 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 $\mathrm{As}, \mathrm{Cd}, \mathrm{Hg}$, 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

|  | Microwave Digestion | Hot Block Digestion | Open Beaker Digestion | None/Not |
| :---: | :---: | :---: | :---: | :---: |
| Analytes |  |  |  | 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 $\mathrm{As}, \mathrm{Cd}, \mathrm{Hg}$, 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

| Analytes | ICP-MS | ICP-OES | ID ICP-MS | NAA | CV AAS | Other/None |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  |  |  |  |  | Selected |
| 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 $\mathrm{As}, \mathrm{Cd}, \mathrm{Hg}$, and Pb in the three samples are shown in the table below.

Between-Laboratory Variability (\% RSD)

| Analytes | NRC HEMP-1 |  | Plant Sample 4 |
| :---: | :---: | :---: | :---: |
|  | $3 \%$ | $3 \%$ |  |
| As | $1 \%$ | $1 \%$ | $3 \%$ |
| Cd | $5 \%$ | $8 \%$ | $1 \%$ |
| Hg | $2 \%$ | $2 \%$ | $3 \%$ |
| Pb |  |  | $1 \%$ |

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

|  | Within-Laboratory Variability (\% RSD) |  |  |  |
| :---: | :---: | :---: | :---: | :---: |
| Analytes | $\underline{\text { NRC HEMP-1 }}$ |  | Plant Sample 4 | SRM 1575a |
| 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 ( $\mathrm{mg} / \mathrm{kg}$ ) 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.

| Samples | Total Number of <br> Laboratories <br> Reporting Results | Number of <br> Laboratories Reporting Qualitative Results | Number of <br> Laboratories Reporting <br> 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 $\left(\left|Z_{\text {comm }}^{\prime}\right|>2\right)$ are summarized in the table below.

|  |  |  |  |
| :---: | :---: | :---: | :---: |
|  | Number (\%) of Laboratory Means |  |  |
|  | Outside NIST Range | Outside Consensus | Laboratories |
| Samples | of Tolerance | Range of Tolerance | Reporting LOQ |
| 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.

Samples
NRC HEMP-1 / Plant Sample 4
NRC HEMP-1 / SRM 1575a
Plant Sample 4 / SRM 1575a

| $\frac{\text { Number of Laboratory }}{\frac{\text { Mean Values Outside }}{\text { both Consensus Ranges }}}$ | $\frac{\text { Percentage of Laboratory }}{\text { Mean Values Outside both }}$ |
| :---: | :---: |
| 9 of 61 | $\frac{\text { Consensus Ranges }}{15 \%}$ |
| 8 of 59 | $14 \%$ |
| 6 of 55 | $11 \%$ |

## Cadmium (Cd)

- The mass fractions ( $\mathrm{mg} / \mathrm{kg}$ ) 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.

- Laboratories reporting outlying results with respect to the NIST range of tolerance and consensus range of tolerance $\left(\left|Z_{\text {comm }}^{\prime}\right|>2\right)$ are summarized in the table below.

|  | 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 | 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 Mean Values Outside both Consensus | Percentage of Laboratory Mean 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 ( $\mathrm{mg} / \mathrm{kg}$ ) 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.

| Samples | Total Number of Laboratories Reporting Results | Number of Laboratories Reporting Qualitative Results | Number of <br> Laboratories Reporting 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 $\left(\left|Z_{\text {comm }}^{\prime}\right|>2\right)$ are summarized in the table below.

|  | 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 | 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.



## Lead (Pb)

- The mass fractions ( $\mathrm{mg} / \mathrm{kg}$ ) 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.

| Samples | Total Number of <br> Laboratories <br> Reporting Results | Number of <br> Laboratories Reporting Qualitative Results | Number of <br> Laboratories Reporting 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 $\left(\left|Z_{\text {comm }}^{\prime}\right|>2\right)$ are summarized in the table below.

|  | 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 | 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.

Samples
NRC HEMP-1 / Plant Sample 4
NRC HEMP-1 / SRM 1575a
Plant Sample 4 / SRM 1575a

Number of Laboratory
Mean Values Outside both Consensus Ranges
14 out of 83
14 out of 80
14 out of 78

Percentage of
Laboratory Mean
Values Outside both
Consensus Ranges
$6 \%$
18 \%
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., " $<0.02 \mathrm{mg} / \mathrm{kg} "$ ).
- 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., $\mathrm{Mo}, \mathrm{Sn}$, or Zr ) are known to cause interferences in the analysis of Cd by ICP-MS. Isobaric spectral interferences such as ${ }^{95} \mathrm{Mo}^{16} \mathrm{O}^{+}$and ${ }^{97} \mathrm{Mo}^{16} \mathrm{O}^{+}$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 $\mathrm{HCl}(3$ to $5 \%$ ) to diluted $\mathrm{HNO}_{3}$ 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 $\mathrm{PbCl}_{2}$ precipitate so digestion with $\mathrm{HNO}_{3}$ is recommended. Because the level of lead in NRC HEMP-1 is approximately 20 times greater than in Plant Sample 4 and SRM $1575 \mathrm{a}, \mathrm{PbCl}_{2}$ 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 $\mathrm{PbCl}_{2}$.
- 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.
- 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 $\mathrm{As}, \mathrm{Cd}, \mathrm{Hg}$, and Pb in hemp and control samples.

## National Institute of Standards and Technology

| CannaQAP Exercise 2 - Spring 2021 |  |  |  |  |  |  | 2. Community Results |  |  | 3. Target |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  | Lab Code: | NIST | 1. Your Results |  |  |  |  |  |  |  |  |
| Analyte | Sample | Units | $\mathrm{x}_{\mathrm{i}}$ | $\mathrm{s}_{\mathrm{i}}$ | $\mathrm{Z}_{\text {comm }}^{\prime}$ | $\mathrm{Z}_{\text {NST }}$ | N | $\mathrm{x}^{*}$ | s* | ${ }^{\mathrm{x}_{\text {NIST }}} \quad U$ |  |
| Arsenic (As) | SRM 1575a Trace Elements in Pine Needles (Pinus taeda) | mgkg | 0.0379 | 0.0039 |  |  | 51 | 0.0420 | 0.0011 | 0.0379 | 0.0039 |
| Arsenic (As) | NRC HEMP-1 (Plant Sample 1) | mgkg | 2.153 | 0.868 |  |  | 83 | 1.847 | 0.049 | 2.153 | 0.868 |
| Arsenic (As) | Plant Sample 4 | mgkg | 0.0340 | 0.0080 |  |  | 50 | 0.0405 | 0.0013 | 0.0340 | 0.0080 |
| Cadmium (Cd) | SRM 1575a Trace Elements in Pine Needles (Pinus taeda) | mgkg | 0.2262 | 0.0078 |  |  | 78 | 0.2164 | 0.0027 | 0.2262 | 0.0078 |
| Cadmium (Cd) | NRC HEMP-1 (Plant Sample 1) | mgkg | 0.1890 | 0.0540 |  |  | 76 | 0.1569 | 0.0023 | 0.1890 | 0.0540 |
| Cadmium (Cd) | Plant Sample 4 | mgkg | 0.0825 | 0.0592 |  |  | 71 | 0.0766 | 0.0011 | 0.0825 | 0.0592 |
| Mercury ( Hg ) | SRM 1575a Trace Elements in Pine Needles (Pinus taeda) | mgkg | 0.0387 | 0.0014 |  |  | 56 | 0.0394 | 0.0012 | 0.0387 | 0.0014 |
| Mercury (Hg) | NRC HEMP-1 (Plant Sample 1) | mgkg | 0.0156 | 0.0020 |  |  | 50 | 0.01721 | 0.00089 | 0.0156 | 0.0020 |
| Mercury (Hg) | Plant Sample 4 | mgkg | 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) | mgkg | 3.562 | 0.456 |  |  | 83 | 2.413 | 0.046 | 3.562 | 0.456 |
| Lead (Pb) | Plant Sample 4 | mgkg | 0.1860 | 0.0520 |  |  | 78 | 0.2029 | 0.0032 | 0.1860 | 0.0520 |
|  |  |  | Mean of Standard Z'-score consensu Z-score | reported deviation of with respe ith respec | eported o comn <br> NIST |  | Number values Robus values Robus | quantita <br> orted <br> an of repo <br> ndard dev |  | NIST-as expanded about the | essed value <br> uncertainty <br> NIST-assessed value |

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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right|>2$. Note: This table spans three pages; the NIST target values and consensus values are included on all three pages for convenience.

|  |  | Arsenic (As) |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  | SRM 1575a Trace Elements in Pine Needles (Pinus taeda) (mg/kg) |  |  |  |  | NRC HEMP-1 (Plant Sample 1) (mg/kg) |  |  |  |  | Plant Sample 4 (mg/kg) |  |  |  |  |
|  | Lab | A | B | C | Avg | SD | A | B | C | Avg | SD | A | B | C | 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 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B023 | 0.0448 | 0.0455 | 0.0497 | 0.0467 | 0.0027 | 1.8183 | 1.9027 | 1.9893 | 1.903 | 0.086 | 0.0429 | 0.0423 | 0.0462 | 0.0438 | 0.0021 |
|  | B027 | <0.05 | <0.05 | <0.05 |  |  | 2.82 | 2.72 | 2.51 | 2.683 | 0.158 | <0.05 | <0.05 | 0.0504 | 0.0504 |  |
|  | B028 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B029 | 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 |
|  | B030 | $<0.043$ | <0.045 | <0.045 |  |  | 2.21 | 2.356 | 2.106 | 2.224 | 0.126 | $<0.043$ | $<0.038$ | 0.069 | 0.0690 |  |
|  | B031 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 |
| 为 |  | Consensus Mean <br> Consensus Standard Deviatiol <br> Maximum <br> Minimum <br> N |  |  | $\begin{gathered} \hline 0.0420 \\ 0.0011 \\ 1.7378 \\ 0.0000 \\ 51 \end{gathered}$ |  | Consensus Consensus Maximum Minimum N | us Mean <br> us Standard <br> n | Deviatio | $\begin{gathered} \hline 1.847 \\ 0.049 \\ 2.792 \\ 0.023 \\ 83 \\ \hline \end{gathered}$ |  | Consensus <br> Consensus <br> Maximum <br> Minimum <br> N | Mean Standard D | Deviation | $\begin{gathered} \hline 0.0405 \\ 0.0013 \\ 0.0690 \\ 0.0000 \\ 50 \\ \hline \end{gathered}$ |  |

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 | A | B | C | Avg | SD | A | B | C | Avg | SD | A | B | C | 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 | 0.035670 | 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 |
|  | B108 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 |  |
|  | 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 |
|  | B111 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 |
|  | 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<0.0. | <0.00017<0.00. | <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 | $<0.001$ | $<0.001<0$. | <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 |
| $\begin{aligned} & 0 \\ & 0 \end{aligned}$ |  | Consensus Mean <br> Consensus Standard Deviatio <br> Maximum <br> Minimum <br> N |  |  | $\begin{gathered} \hline 0.0420 \\ 0.0011 \\ 1.7378 \\ 0.0000 \\ 51 \\ \hline \end{gathered}$ |  | Consensus Mean <br> Consensus Standard Deviatio <br> Maximum <br> Minimum <br> N |  |  | $\begin{gathered} \hline 1.847 \\ 0.049 \\ 2.792 \\ 0.023 \\ 83 \\ \hline \end{gathered}$ |  | $\begin{array}{\|l} \hline \text { Consensus } \\ \text { Consensus } \\ \text { Maximum } \\ \text { Minimum } \\ \mathrm{N} \\ \hline \end{array}$ | Mean <br> Standard | Deviation | $\begin{gathered} \hline 0.0405 \\ 0.0013 \\ 0.0690 \\ 0.0000 \\ 50 \\ \hline \end{gathered}$ |  |

Table 2-2. continued.



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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq$ 2. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (UNIST) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 2-2. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 2-4. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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 2-5. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (UNIST) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 2-6. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq$ 2. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIIT }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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).

Exercise: CannaQAP Exercise 2, Measurand: total arsenic
No. of laboratories: 61


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 ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq$ 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$.

Exercise: CannaQAP Exercise 2, Measurand: total arsenic
No. of laboratories: 59


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 $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$.

Exercise: CannaQAP Exercise 2, Measurand: total arsenic
No. of laboratories: 55


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 ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$.

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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right|>2$. Note: This table spans three pages; the NIST values and consensus values are included on all three pages for convenience.


Table 2-3. continued.

|  |  | Cadmium (Cd) |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  | SRM 1575a Trace Elements in Pine Needles (Pinus taeda) ( $\mathrm{mg} / \mathrm{kg}$ ) |  |  |  |  | NRC HEMP-1 (Plant Sample 1) (mg/kg) |  |  |  |  | Plant Sample 4 (mg/kg) |  |  |  |  |
|  | Lab | A | B | C | Avg | SD | A | B | C | Avg | SD | A | B | C | 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 | 0.0716 | 0.0793 | 0.0747 | 0.0752 | 0.0039 |
|  | B108 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 |
|  | 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 |
|  | B111 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B112 | 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 |
|  | B113 | 0.201 | 0.179 | 0.178 | 0.1860 | 0.0130 | 0.102 | 0.11 | 0.103 | 0.1050 | 0.0044 | 0.059 | 0.056 | 0.052 | 0.0557 | 0.0035 |
|  | B116 | 0.233 | 0.236 | 0.233 | 0.2340 | 0.0017 | 0.167 | 0.176 | 0.161 | 0.1680 | 0.0075 | 0.088 | 0.092 | 0.088 | 0.0893 | 0.0023 |
|  | B119 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B120 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B122 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B125 | 0.22 | 0.219 | 0.222 | 0.2203 | 0.0015 | 0.0337 | 0.0318 | 0.0331 | 0.0329 | 0.0010 | 0.0615 | 0.0672 | 0.0683 | 0.0657 | 0.0037 |
|  | B129 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 |  |
|  | B137 | 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 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  |  | Consensus <br> Consensus <br> Maximum <br> Minimum <br> N | Mean Standard Dev | jation | $\begin{gathered} \hline 0.2164 \\ 0.0027 \\ 0.3761 \\ 0.1447 \\ 78 \\ \hline \end{gathered}$ |  | Consensus <br> Consensus <br> Maximum <br> Minimum <br> N | Mean Standard Dev | iation | $\begin{gathered} \hline 0.1569 \\ 0.0023 \\ 0.2587 \\ 0.0051 \\ 76 \\ \hline \end{gathered}$ |  | Consensus Consensus Maximum Minimum N | Mean <br> Standard Dev | viation | $\begin{gathered} \hline 0.0766 \\ 0.0011 \\ 0.1500 \\ 0.0485 \\ 71 \\ \hline \end{gathered}$ |  |

Table 2-3. continued.



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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq$ 2. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (UNIST) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 2-11. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 2-12. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 2-13. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 2-14. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (UNIST) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq$ 2. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (UNIST) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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).

Exercise: CannaQAP Exercise 2, Measurand: cadmium
No. of laboratories: 77


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 $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$.

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 ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$.

Exercise: CannaQAP Exercise 2, Measurand: cadmium
No. of laboratories: 78


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 ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$.

Table 2-4. Data summary table for mercury $(\mathrm{Hg})$ 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right|>2$. Note: This table spans three pages; the NIST values and consensus values are included on all three pages for convenience.


Table 2-4. continued.


Table 2-4. continued.



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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq$ 2. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (UNIST) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 2-20. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq$ 2. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 2-21. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 2-22. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 2-23. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 2-24. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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).

Exercise: CannaQAP Exercise 2, Measurand: total mercury
No. of laboratories: 42


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 ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq$ 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$.


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 (UNIST) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$.

Exercise: CannaQAP Exercise 2, Measurand: total mercury
No. of laboratories: 41


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 ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. The dotted blue box (the bottom limit is not shown due to the scale of the figure) represents the consensus range of tolerance for 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$.

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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right|>2$. Note: This table spans three pages; the NIST values and consensus values are included on all three pages for convenience.

|  |  | Lead (Pb) |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  | 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 | A | B | C | Avg | SD | A | B | C | Avg | SD | A | B | C | 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 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B018 | 0.144 | 0.144 | 0.145 | 0.1443 | 0.0006 | 2.242 | 2.244 | 2.231 | 2.239 | 0.007 | 0.284 | 0.217 | 0.268 | 0.2563 | 0.0350 |
|  | B020 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B023 | 0.1539 | 0.1576 | 0.1584 | 0.1566 | 0.0024 | 2.3891 | 2.5808 | 2.4675 | 2.479 | 0.096 | 0.1853 | 0.1984 | 0.1792 | 0.1876 | 0.0098 |
|  | B027 | 0.144 | 0.148 | 0.138 | 0.1433 | 0.0050 | 2.76 | 2.76 | 2.36 | 2.627 | 0.231 | 0.2 | 0.178 | 0.5 | 0.2927 | 0.1799 |
|  | B028 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B029 | 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 | 0.174 | 0.196 | 0.196 | 0.1887 | 0.0127 |
|  | B031 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 |
|  | B037 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B040 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 |
|  | B043 | 0.145 |  |  | 0.1450 |  | 2.486 |  |  | 2.486 |  | 0.202 |  |  | 0.2020 |  |
|  | 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 | 0.0070 | 1.897 | 1.796 | 2.215 | 1.969 | 0.219 | 0.269 | 0.215 | 0.174 | 0.2193 | 0.0476 |
|  | B077 | 0.1605 | 0.1536 | 0.1475 | 0.1539 | 0.0065 | 2.4424 | 2.5683 | 2.2099 | 2.407 | 0.182 | 0.2452 | 0.1863 | 0.1974 | 0.2096 | 0.0313 |
|  | B078 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B079 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B081 | 0.145 | 0.154 | 0.171 | 0.1567 | 0.0132 | 2.379 | 2.02 | 2.364 | 2.254 | 0.203 | 0.243 | 0.185 | 0.189 | 0.2057 | 0.0324 |
|  | B082 | 0.152 | 0.151 | 0.166 | 0.1563 | 0.0084 | 2.692 | 2.479 | 1.925 | 2.365 | 0.396 | 0.167 | 0.164 | 0.202 | 0.1777 | 0.0211 |
|  | B084 | 0.132 | 0.132 | 0.122 | 0.1287 | 0.0058 | 2.44 | 2.73 | 2.54 | 2.570 | 0.147 | 0.167 | 0.171 | 0.158 | 0.1653 | 0.0067 |
| $\begin{aligned} & 0 \\ & 0 \\ & 0 \end{aligned}$ |  | Consensus Mean <br> Consensus Standard Deviation <br> Maximum <br> Minimum <br> N |  |  | $\begin{gathered} \hline 0.1491 \\ 0.0021 \\ 3.7959 \\ 0.1027 \\ 72 \\ \hline \end{gathered}$ |  | Consensus Mean <br> Consensus Standard Deviation <br> Maximum <br> Minimum <br> N |  |  | $\begin{gathered} \hline 2.413 \\ 0.046 \\ 6.840 \\ 0.151 \\ 83 \\ \hline \end{gathered}$ |  | Consensu Consensu Maximum Minimum N | s Mean Standard D | Deviation | 0.2029 0.0032 1.5581 0.1297 78 |  |

Table 2-5. continued.


Table 2-5. continued.

|  |  | Lead (Pb) |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  | 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 | A | B | C | Avg | SD | A | B | C | Avg | SD | A | B | C | 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 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 |
|  | 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 |
|  | 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 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 |  |  | 2.3 | 2.52 | 2.38 | 2.400 | 0.111 | <0.5 | <0.5 | <0.5 |  |  |
|  | 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 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 |
| $\begin{aligned} & 0 \\ & 0 \end{aligned}$ |  | Consensus <br> Consensus <br> Maximum <br> Minimum <br> N | s Mean Standard | Deviation | $\begin{gathered} \hline 0.1491 \\ 0.0021 \\ 3.7959 \\ 0.1027 \\ 72 \\ \hline \end{gathered}$ |  | Consensus <br> Consensus <br> Maximum <br> Minimum <br> N | Mean Standard D | Deviation | $\begin{gathered} \hline 2.413 \\ 0.046 \\ 6.840 \\ 0.151 \\ 83 \\ \hline \end{gathered}$ |  | Consensu Consensu Maximum Minimum N | s Mean Standard | Deviation | $\begin{gathered} 0.2029 \\ 0.0032 \\ 1.5581 \\ 0.1297 \\ 78 \\ \hline \end{gathered}$ |  |



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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq$ 2. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (UNIST) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$.


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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\mathrm{NIST}}$ score, $\left|Z_{\mathrm{NIST}}\right| \leq 2$.


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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 2-31. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 2-32. 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 $Z_{\mathrm{comm}}^{\prime}$ score, $\left|Z_{\mathrm{comm}}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (UNIST) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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).

Exercise: CannaQAP Exercise 2, Measurand: lead
No. of laboratories: 83


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 ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$.

Exercise: CannaQAP Exercise 2, Measurand: lead
No. of laboratories: 80


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 ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$.

Exercise: CannaQAP Exercise 2, Measurand: lead
No. of laboratories: 78


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 ( $y$ axis), which encompasses the target values bounded by their uncertainties ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$.

## 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 $\mathrm{Cr}, \mathrm{Se}$, and Be , with Be being one of the most toxic. Beryllium, Mn , and Ni are known to affect the lungs, while $\mathrm{Co}, \mathrm{Mo}, \mathrm{Ni}$, 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

| Analytes | Number of Participants | NRC HEMP-1 | Plant Sample 4 | SRM 1575a |
| :---: | :---: | :---: | :---: | :---: |
| 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 $\mathrm{Be}, \mathrm{Co}, \mathrm{Cr}, \mathrm{Mo}, \mathrm{Mn}$, $\mathrm{Ni}, \mathrm{Se}, \mathrm{U}$, 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 | Percent Reporting Results |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: |
|  | Microwave Digestion | Hot Block Digestion | Open Beaker Digestion | Solvent Extraction | None/Not <br> 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 \% |

Most laboratories reported using ICP-MS for the determination of $\mathrm{Be}, \mathrm{Co}, \mathrm{Cr}, \mathrm{Mo}, \mathrm{Mn}, \mathrm{Ni}, \mathrm{Se}, \mathrm{U}$, 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 | NAA | $\frac{\text { Other/Not }}{\text { Specified }}$ |
| :---: | :---: | :---: | :---: | :---: | :---: |
| Be | 89 \% | $7 \%$ | 4 \% | -- | -- |
| Co | 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 $\mathrm{Be}, \mathrm{Co}, \mathrm{Cr}, \mathrm{Mo}, \mathrm{Mn}, \mathrm{Ni}, \mathrm{Se}, \mathrm{U}$, and V in the three samples are shown in the table below.

## Between-Laboratory Variability (\% RSD)

| Analytes | NRC HEMP-1 | Plant Sample 4 | SRM 1575a |
| :---: | :---: | :---: | :---: |
| 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 $\mathrm{Be}, \mathrm{Co}, \mathrm{Cr}, \mathrm{Mo}$, $\mathrm{Mn}, \mathrm{Ni}, \mathrm{Se}, \mathrm{U}$, and V in the three samples are shown in the table below.

| Analytes | Within-Laboratory Variability (\% RSD) |  |  |
| :---: | :---: | :---: | :---: |
|  | NRC HEMP-1 | Plant Sample 4 | SRM 1575a |
| 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 ( $\mathrm{mg} / \mathrm{kg}$ ) for $\mathrm{Be}, \mathrm{Co}, \mathrm{Cr}, \mathrm{Mn}, \mathrm{Ni}, \mathrm{Se}, \mathrm{U}$, 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 31.

## 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.
\(\left.$$
\begin{array}{ccccc} & \begin{array}{c}\frac{\text { Total Number of }}{\text { Laboratories }} \\
\text { Seporting Results }\end{array} & \begin{array}{c}\frac{\text { Number of }}{\text { Laboratories Reporting }}\end{array} & \begin{array}{c}\frac{\text { Number of }}{\text { Laboratories Reporting }}\end{array}
$$ <br>

Sualitative Results\end{array}\right) ~\)| $\underline{\text { Quantitative Results }}$ |
| :---: | :---: | :---: |

- Laboratories reporting outlying results with respect to the NIST range of tolerance and consensus range of tolerance $\left(\left|Z_{\text {comm }}^{\prime}\right|>2\right)$ are summarized in the table below.

|  | Number (\%) of | Number (\%) of | Number (\%) of |
| :---: | :---: | :---: | :---: |
|  | Laboratory Means | Laboratory Means | Laboratories |
|  | Outside NIST Range | Outside Consensus | Reporting |
| 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.


## Samples

NRC HEMP-1 / Plant Sample 4
NRC HEMP-1 / SRM 1575a
Plant Sample 4 / SRM 1575a

| $\frac{\text { Number of Laboratory }}{\frac{\text { Mean Values Outside }}{\text { both Consensus Ranges }}}$ |  |
| :---: | :---: | | $\frac{\text { Percentage of Laboratory }}{\frac{\text { Mean Values Outside }}{\text { both Consensus Ranges }}}$ |
| :---: |

## 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.

|  | $\frac{\text { Total Number of }}{\text { Laboratories }}$ | Number of <br> Samples | Laboratories Reporting <br> Reporting Results | Number of <br> Laboratories Reporting <br> NRC HEMP-1$\quad 32$ |
| :---: | :---: | :---: | :---: | :---: |

Laboratories reporting outlying results with respect to the NIST range of tolerance and consensus range of tolerance $\left(\left|Z_{\text {comm }}^{\prime}\right|>2\right)$ are summarized in the table below.

|  | 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 | 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.

|  | N | Percentage of Laboratory |
| :---: | :---: | :---: |
|  | Mean Values Outside | Mean Values Outside both |
| Samples | both Consensus Ranges | 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.

|  | $\frac{\text { Total Number of }}{\text { Laboratories }}$ <br> Reaorting Results | Number of <br> Laboratories Reporting | Number of <br> Laboratories Reporting <br> Samples | Qualitative Results |
| :---: | :---: | :---: | :---: | :---: |
| 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 $\left(\left|Z_{\text {comm }}^{\prime}\right|>2\right)$ are summarized in the table below.

|  | 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 (6\%) | 4 (8\%) | 0 (0\%) |
| Plant Sample 4 | 5 (11\%) | 6 (13 \%) | 3 (7\%) |
| SRM 1575a | N/A | 5 (11\%) | 5 (11\%) |

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

| Samples | $\frac{\text { Number of Laboratory }}{\text { Mean Values Outside }}$ <br> $\underline{\text { both Consensus Ranges }}$ | $\frac{\text { Percentage of Laboratory }}{\text { Mean Values Outside }}$ <br> $\underline{\text { both Consensus Ranges }}$ |
| :---: | :---: | :---: |
| 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.

|  | $\frac{\text { Total Number of }}{\text { Laboratories }}$ <br> Samples | Number of <br> Leporting Results | Number of <br> Labories Reporting <br> Sualitative Results | Laboratories Reporting |
| :---: | :---: | :---: | :---: | :---: |
| NRC HEMP-1 | 29 | 4 | 25 |  |
| Plant Sample 4 | 29 | 5 | 24 |  |
| SRM 1575a | 27 | 11 | 16 |  |

- Laboratories reporting outlying results with respect to the NIST range of tolerance and consensus range of tolerance $\left(\left|Z_{\text {comm }}^{\prime}\right|>2\right)$ are summarized in the table below.

|  | 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.

| Samples | $\frac{\text { Number of Laboratory }}{\frac{\text { Mean Values Outside }}{}}$ | $\frac{\text { Percentage of Laboratory }}{\underline{\text { Mean Values Outside }}}$ <br> NRC HEMP-1 / Plant Sample 4 |
| :---: | :---: | :---: |
| NRC HEMP-1 / SRM 1575a | 3 out of 24 |  |
| Plant Sample 4 / SRM 1575a | 2 out of 16 | $13 \%$ |
| both Consensus Ranges |  |  |

## 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.

|  | $\frac{\text { Total Number of }}{\text { Laboratories }}$ <br> Samples | Number of <br> Leporting Results | Number of <br> Laboratories Reporting <br> Nualitative Results | Laboratories Reporting <br> Quantitative Results |
| :---: | :---: | :---: | :---: | :---: |
| PRC HEMP-1 | 34 | 0 | 34 |  |
| Plant Sample 4 | 34 | 0 | 34 |  |
| SRM 1575a | 33 | 0 | 33 |  |

- Laboratories reporting outlying results with respect to the NIST range of tolerance and consensus range of tolerance $\left(\left|Z_{\text {comm }}^{\prime}\right|>2\right)$ are summarized in the table below.

|  | Number (\%) | Number (\%) of | Number |
| :---: | :---: | :---: | :---: |
|  | Laboratory Means | Laboratory Means | Laboratories |
|  | Outside NIST | Outside Consensus | Reporting |
| Samples | 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.

$\frac{\text { Number of Laboratory }}{\frac{\text { Mean Values Outside }}{\text { both Consensus Ranges }}}$$\quad$| $\frac{\text { Percentage of Laboratory }}{\frac{\text { Mean Values Outside }}{\text { both Consensus Ranges }}}$ |
| :---: |
| 7 out of 34 |

## 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.

|  | $\frac{\text { Total Number of }}{\text { Laboratories }}$ <br> Samples | Number of <br> Laboratories Reporting Results | Number of <br> Laboratories Reporting <br> Sualitative Results | Quantitative Results |
| :---: | :---: | :---: | :---: | :---: |
| NRC HEMP-1 | 40 | 3 | 37 |  |
| Plant Sample 4 | 39 | 2 | 37 |  |
| SRM 1575a | 39 | 3 | 36 |  |

- Laboratories reporting outlying results with respect to the NIST range of tolerance and consensus range of tolerance $\left(\left|Z_{\text {comm }}^{\prime}\right|>2\right)$ are summarized in the table below.

|  | 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 | 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.

| Samples | Number of Laboratory Mean Values Outside both Consensus Ranges | Percentage of Laboratory Mean Values Outside 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.

|  | $\frac{\text { Total Number of }}{\text { Laboratories }}$ <br> Samples | Number of <br> Laborting Results | Number of <br> Qualitative Reporting | Laboratories Reporting |
| :---: | :---: | :---: | :---: | :---: |
| Quantitative Results |  |  |  |  |

- Laboratories reporting outlying results with respect to the NIST range of tolerance and consensus range of tolerance $\left(\left|Z_{\text {comm }}^{\prime}\right|>2\right)$ are summarized in the table below.

|  | 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 | 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.

|  | Number of Laboratory | Percentage of Lab |
| :---: | :---: | :---: |
|  | Mean Values Outside | 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.

|  | $\frac{\text { Total Number of }}{\text { Laboratories }}$ <br> Samples | Number of <br> Leporting Results | Number of <br> Laboratories Reporting | Laboratories Reporting |
| :---: | :---: | :---: | :---: | :---: |
| NRC HEMP-1 | 21 | 2 | 19 |  |
| Plant Sample 4 | 19 | 7 | 12 |  |
| SRM 1575a | 18 | 6 | 12 |  |

- Laboratories reporting outlying results with respect to the NIST range of tolerance and consensus range of tolerance $\left(\left|Z_{\text {comm }}^{\prime}\right|>2\right)$ are summarized in the table below.

|  | 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 | 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.

Samples
NRC HEMP-1 / Plant Sample 4
NRC HEMP-1 / SRM 1575a
Plant Sample 4 / SRM 1575a

$\frac{$|  Number of Laboratory  |
| :--- |
|  Mean Values Outside  |}{both Consensus Ranges} $\quad$| $\frac{\text { Percentage of Laboratory }}{\frac{\text { Mean Values Outside }}{\text { both Consensus Ranges }}}$ |
| :---: |

## 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.

|  | $\frac{\text { Total Number of }}{\text { Laboratories }}$ <br> Samples | $\underline{\text { Number of }}$ <br> Laborting Results | $\underline{\text { Number of }}$ <br> Qualitative Reporting | $\underline{\text { Laboratories Reporting }}$ |
| :---: | :---: | :---: | :---: | :---: |
| NRC HEMP-1 | 26 | 1 | 25 |  |
| Plantitative Results |  |  |  |  |

- Laboratories reporting outlying results with respect to the NIST range of tolerance and consensus range of tolerance $\left(\left|Z_{\text {comm }}^{\prime}\right|>2\right)$ are summarized in the table below.

|  | 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 | 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 | Percentage of Laboratory |
|  | Mean Values Outside | Mean Values Outside both |
| Samples | both Consensus Ranges | Consensus Ranges |
| 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 ( $\mathrm{Mn}, \mathrm{V}$ ).
- In some cases, open beaker digestion or hot block digestion approaches did not perform as well as microwave digestion ( $\mathrm{Be}, \mathrm{Co}, \mathrm{Se}$ ).
- In some cases, where ICP-OES was employed, the sample-to-sample variability was great ( $\mathrm{Cr}, \mathrm{Mn}$ in NRC HEMP-1).
- Some elements ( $\mathrm{Be}, \mathrm{Co}, \mathrm{Mn}, \mathrm{V}$ ) are monoisotopic, so reporting ID-ICP-MS as the analytical method is not a practicable for these elements. Vanadium has a radioactive isotope $\left(\mathrm{V}^{50}\right)$ 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 $\mathrm{HNO}_{3}$, and once dissolved it should be stored in dilute $\mathrm{HNO}_{3}$ in plastic bottles for better stability.
- Low concentrations of $\mathrm{Be}(<100 \mu \mathrm{~g} / \mathrm{g})$ 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 $\left(\mathrm{H}_{2} \mathrm{O}_{2}\right)$, perchloric acid $\left(\mathrm{HClO}_{4}\right)$ ) will ensure complete digestion of the materials prior to analysis.
- Cobalt is stable in a dilute $\mathrm{HNO}_{3}$ 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 $\mathrm{HNO}_{3}$.
- Spectral/isobaric interferences can make Cr difficult to measure accurately by ICP-MS.
- High concentrations of certain elements ( $\mathrm{Ar}, \mathrm{C}, \mathrm{Cl}, \mathrm{S}$ ) may cause interferences, including $\mathrm{Cl}^{-}$or $\mathrm{S}^{-}$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 $(\mathrm{ZrH})$. This may cause problems if hydrogen $\left(\mathrm{H}_{2}\right)$ 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 $\mathrm{HNO}_{3}$.
- 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 $\mathrm{HNO}_{3}$ but once the sample material is in solution Ni is stable in dilute $\mathrm{HNO}_{3}$.
- 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.
- $\mathrm{Ni}^{60}$ is the preferred isotope for Ni determination, as $\mathrm{Ni}^{58}$ suffers interferences from iron ( Fe ) and argon oxide (ArO). Additionally, materials high in calcium (Ca) can form calcium oxide $(\mathrm{CaO})$ 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 $\mathrm{HNO}_{3}$.
- 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 $\left(\mathrm{Se}^{74}, \mathrm{Se}^{76}, \mathrm{Se}^{78}, \mathrm{Se}^{80}, \mathrm{Se}^{82}\right)$ or from polyatomic interferences, mainly from $\mathrm{Ar}^{2}\left(\mathrm{Se}^{76}, \mathrm{Se}^{78}, \mathrm{Se}^{80}\right)$ causing signal suppression or enhancement leading to bias of the measurements. One of the most useful isotopes for quantitation is $\mathrm{Se}^{77}$, 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 $\mathrm{HNO}_{3}$.
- 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 $\mathrm{V}^{51}$ isotope. Vanadium is considered monoisotopic, as $\mathrm{V}^{50}$ is radioactive. in addition, $\mathrm{V}^{50}$ has low abundance and has known interferences.

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

## National Institute of Standards and Technology

| CannaQAP Exercise 2 - Spring 2021 |  |  |  |  |  |  | 2. Community Results |  |  | 3. Target |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| Analyte | Lab Code: | NIST | 1. Your Results |  |  |  |  |  |  |  |  |
|  | Sample | Units | $\mathrm{x}_{\mathrm{i}}$ | $\mathrm{S}_{\mathrm{i}}$ | $\mathrm{Z}_{\text {comm }}$ | $\mathrm{Z}_{\text {NIST }}$ | N | $\mathrm{x}^{*}$ | s* | $\mathrm{x}_{\text {NIST }}$ | $U$ |
| Beryllium (Be) | SRM 1575a Trace Elements in Pine Needles (Pinus taeda) | $\mathrm{mg} / \mathrm{kg}$ |  |  |  |  | 15 | 0.0070 | 0.0006 |  |  |
| Beryllium (Be) | NRC HEMP-1 (Plant Sample 1) | $\mathrm{mg} / \mathrm{kg}$ | 0.242 | 0.023 |  |  | 24 | 0.205 | 0.010 | 0.242 | 0.023 |
| Beryllium (Be) | Plant Sample 4 | $\mathrm{mg} / \mathrm{kg}$ | 0.002 | 0.001 |  |  | 11 | 0.00274 | 0.00052 | 0.002 | 0.001 |
| Cobalt (Co) | SRM 1575a Trace Elements in Pine Needles (Pinus taeda) | $\mathrm{mg} / \mathrm{kg}$ | 0.0592 | 0.0039 |  |  | 23 | 0.0589 | 0.0015 | 0.0592 | 0.0039 |
| Cobalt (Co) | NRC HEMP-1 (Plant Sample 1) | $\mathrm{mg} / \mathrm{kg}$ | 1.854 | 0.340 |  |  | 30 | 1.720 | 0.081 | 1.854 | 0.340 |
| Cobalt (Co) | Plant Sample 4 | $\mathrm{mg} / \mathrm{kg}$ | 0.2396 | 0.1172 |  |  | 26 | 0.1722 | 0.0043 | 0.2396 | 0.1172 |
| Chromium ( Cr ) | SRM 1575a Trace Elements in Pine Needles (Pinus taeda) | $\mathrm{mg} / \mathrm{kg}$ | 0.388 |  |  |  | 36 | 0.344 | 0.010 | 0.388 |  |
| Chromium (Cr) | NRC HEMP-1 (Plant Sample 1) | $\mathrm{mg} / \mathrm{kg}$ | 12.72 | 4.49 |  |  | 45 | 8.68 | 0.33 | 12.72 | 4.49 |
| Chromium (Cr) | Plant Sample 4 | $\mathrm{mg} / \mathrm{kg}$ | 0.307 | 0.220 |  |  | 39 | 0.472 | 0.014 | 0.307 | 0.220 |
| Manganese (Mn) | SRM 1575a Trace Elements in Pine Needles (Pinus taeda) | $\mathrm{mg} / \mathrm{kg}$ | 473.8 | 23.3 |  |  | 29 | 458.0 | 8.3 | 473.8 | 23.3 |
| Manganese (Mn) | NRC HEMP-1 (Plant Sample 1) | $\mathrm{mg} / \mathrm{kg}$ | 401.0 | 26.4 |  |  | 32 | 366.9 | 9.4 | 401.0 | 26.4 |
| Manganese (Mn) | Plant Sample 4 | $\mathrm{mg} / \mathrm{kg}$ | 142.0 | 7.6 |  |  | 32 | 135.4 | 2.4 | 142.0 | 7.6 |
| Molybdenum (Mo) | SRM 1575a Trace Elements in Pine Needles (Pinus taeda) | $\mathrm{mg} / \mathrm{kg}$ |  |  |  |  | 15 | 0.0184 | 0.0015 |  |  |
| Molybdenum (Mo) | NRC HEMP-1 (Plant Sample 1) | $\mathrm{mg} / \mathrm{kg}$ | 0.661 | 0.096 |  |  | 24 | 0.612 | 0.022 | 0.661 | 0.096 |
| Molybdenum (Mo) | Plant Sample 4 | $\mathrm{mg} / \mathrm{kg}$ | 0.3180 | 0.0120 |  |  | 24 | 0.3244 | 0.0073 | 0.3180 | 0.0120 |
| Nickel ( Ni ) | SRM 1575a Trace Elements in Pine Needles (Pinus taeda) | $\mathrm{mg} / \mathrm{kg}$ | 1.427 | 0.194 |  |  | 35 | 1.332 | 0.029 | 1.427 | 0.194 |
| Nickel ( Ni ) | NRC HEMP-1 (Plant Sample 1) | $\mathrm{mg} / \mathrm{kg}$ | 7.11 | 4.36 |  |  | 36 | 5.67 | 0.21 | 7.11 | 4.36 |
| Nickel ( Ni ) | Plant Sample 4 | $\mathrm{mg} / \mathrm{kg}$ | 3.670 | 1.320 |  |  | 35 | 3.262 | 0.062 | 3.670 | 1.320 |
| Selenium (Se) | SRM 1575a Trace Elements in Pine Needles (Pinus taeda) | $\mathrm{mg} / \mathrm{kg}$ | 0.0961 | 0.0078 |  |  | 23 | 0.1191 | 0.0069 | 0.0961 | 0.0078 |
| Selenium (Se) | NRC HEMP-1 (Plant Sample 1) | $\mathrm{mg} / \mathrm{kg}$ | 0.305 | 0.060 |  |  | 25 | 0.462 | 0.051 | 0.305 | 0.060 |
| Selenium (Se) | Plant Sample 4 | $\mathrm{mg} / \mathrm{kg}$ | 0.8100 | 0.0120 |  |  | 23 | 0.1058 | 0.0077 | 0.8100 | 0.0120 |
| Uranium (U) | SRM 1575a Trace Elements in Pine Needles (Pinus taeda) | $\mathrm{mg} / \mathrm{kg}$ |  |  |  |  | 12 | 0.00525 | 0.00034 |  |  |
| Uranium (U) | NRC HEMP-1 (Plant Sample 1) | $\mathrm{mg} / \mathrm{kg}$ | 0.454 | 0.120 |  |  | 19 | 0.268 | 0.020 | 0.454 | 0.120 |
| Uranium (U) | Plant Sample 4 | $\mathrm{mg} / \mathrm{kg}$ | 0.00426 | 0.00120 |  |  | 10 | 0.00326 | 0.00031 | 0.00426 | 0.00120 |
| Vanadium (V) | SRM 1575a Trace Elements in Pine Needles (Pinus taeda) | $\mathrm{mg} / \mathrm{kg}$ |  |  |  |  | 17 | 0.1041 | 0.0050 |  |  |
| Vanadium (V) | NRC HEMP-1 (Plant Sample 1) | $\mathrm{mg} / \mathrm{kg}$ | 16.70 | 1.70 |  |  | 24 | 13.83 | 0.70 | 16.70 | 1.70 |
| Vanadium (V) | Plant Sample 4 | $\mathrm{mg} / \mathrm{kg}$ | 0.233 | 0.033 |  |  | 20 | 0.208 | 0.015 | 0.233 | 0.033 |
|  |  |  | Mean of <br> Standard <br> Z'-score <br> consensus | reported deviation with respe | es <br> reported <br> o comn |  | Numb <br> values <br> Robus <br> values | quantita <br> orted <br> an of rep |  |  | essed value uncertainty NIST-assessed value |
|  |  | $\mathrm{Z}_{\text {NIST }}$ | Z-score with respect to NIST value |  |  |  | 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right|>2$. Note: This table spans two pages; the NIST values and consensus values are included on both pages for convenience.

|  |  | Beryllium (Be) |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  | 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 | A | B | C | Avg | SD | A | B | C | Avg | SD | A | B | C | Avg | SD |
| 泉 | 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 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B035 | <0.1 | <0.1 | $<0.1$ |  |  | 0.192 | 0.204 | 0.193 | 0.196 | 0.007 | $<0.1$ | $<0.1$ | $<0.1$ |  |  |
|  | B037 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 | $<0.0391<0.0$ | <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 |
|  | 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 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| $\begin{gathered} \text { con } \\ 0 \\ 0 \end{gathered}$ |  | Consensus <br> Consensus <br> Maximum <br> Minimum <br> N | us Mean us Standard | Deviatios | $\begin{gathered} \hline 0.0070 \\ 0.0006 \\ 0.1547 \\ 0.0000 \\ 15 \end{gathered}$ |  | Consensus Consensus Maximum Minimum N | s Mean standard | d Deviatio | $\begin{gathered} \hline 0.205 \\ 0.010 \\ 0.344 \\ 0.003 \\ 24 \end{gathered}$ |  | Consensus <br> Consensus <br> Maximum <br> Minimum <br> N | s Mean Standard | Deviatio | $\begin{gathered} 0.00274 \\ 0.00052 \\ 0.00683 \\ 0.00000 \\ 11 \end{gathered}$ |  |

Table 3-2. continued.



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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq$ 2. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIIT }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 3-2. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 3-3. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 3-4. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 3-5. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq$ 2. A NIST value has not been determined in this material.


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 ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$.

Exercise: CannaQAP Exercise 2, Measurand: beryllium
No. of laboratories: 16


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 $Z_{\text {comm }}^{\prime}$ Score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$.

Exercise: CannaQAP Exercise 2, Measurand: beryllium
No. of laboratories: 13


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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$.

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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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right|>2$. Note: This table spans two pages; the NIST values and consensus values are included on both pages for convenience.

|  |  | Cobalt (Co) |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  | 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 | A | B | C | Avg | SD | A | B | C | Avg | SD | A | B | C | 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 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B040 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 |
|  | B058 | $<0.0784<$ | $<0.0783<$ | <0.0764 |  |  | 1.65 | 1.63 | 1.92 | 1.733 | 0.162 | 0.174 | 0.149 | 0.188 | 0.1703 | 0.0198 |
|  | 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 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B084 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B088 | <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 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| $\begin{gathered} \text { n } \\ 0 \\ 0 \end{gathered}$ |  | Consensus Mean <br> Consensus Standard Deviation <br> Maximum <br> Minimum <br> N |  |  | $\begin{gathered} 0.0589 \\ 0.0015 \\ 1.6467 \\ 0.0503 \\ 24 \end{gathered}$ |  | Consensus Mean <br> Consensus Standard Deviation <br> Maximum <br> Minimum <br> N |  |  | 1.720 |  | Consensus Mean 0.1722 |  |  |  |  |
|  |  |  |  |  | 0.081 |  |  |  |  | Consensus | Standard | Deviation | 0.0043 |  |
|  |  |  |  |  | 5.603 |  |  |  |  | Maximum |  |  | 1.2167 |  |
|  |  |  |  |  | 0.058 |  |  |  |  | Minimum |  |  | 0.1460 |  |
|  |  |  |  |  | 30 |  |  |  |  | N |  |  | 26 |  |

Table 3-3. continued.

|  |  | Cobalt (Co) |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  | 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 | A | B | C | Avg | SD | A | B | C | Avg | SD | A | B | C | 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 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B176 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 |
|  | 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 |
|  | 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 |
|  | B202 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 |
|  |  | Consensus Mean <br> Consensus Standard Deviation <br> Maximum <br> Minimum <br> N |  |  | 0.0589 |  | Consensus Mean Consensus Standard Deviation |  |  | 1.720 |  | Consensus Mean <br> Consensus Standard Deviation |  |  | 0.1722 |  |
|  |  |  |  |  | 0.0015 |  |  |  |  | 0.081 |  |  |  |  | 0.0043 |  |
|  |  |  |  |  | 1.6467 |  | Maximum |  |  | 5.603 |  | Maximum |  |  | 1.2167 |  |
|  |  |  |  |  | 0.0503 |  | Minimum |  |  | 0.058 |  | Minimum |  |  | 0.1460 |  |
|  |  |  |  |  | 24 |  |  |  |  | 30 |  | N |  |  | 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq$ 2. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIIT }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 3-11. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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).


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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 3-13. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 3-14. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (UNIST) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq$ 2. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIIT }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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).

Exercise: CannaQAP Exercise 2, Measurand: cobalt
No. of laboratories: 27


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 ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. The dotted blue box represents the consensus range of tolerance for Plant Sample 4 (xaxis) and NRC HEMP-1 (y-axis), calculated as the values above and below the consensus means that result in an acceptable $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$.

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


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 $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$.

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 ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ Score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$.

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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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right|>2$. Note: This table spans two pages; the NIST values and consensus values are included on both pages for convenience.


Table 3-4. continued.



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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 3-21. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 3-22. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 3-23. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red line represents the non-certified value determined in this material.

Exercise: CannaQAP Exercise 2, Measurand: chromium
No. of laboratories: 43


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 (UNIST) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$.

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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$.

Exercise: CannaQAP Exercise 2, Measurand: chromium
No. of laboratories: 40


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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$.

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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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right|>2$. Note: This table spans two pages; the NIST values and consensus values are included on both pages for convenience.

|  |  | Molybdenum (Mo) |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  | 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 | A | B | C | Avg | SD | A | B | C | Avg | SD | A | B | C | 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 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B040 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B049 |  |  |  |  |  | 0.609 | 0.62 | 0.554 | 0.594 | 0.035 | 0.32 | 0.297 | 0.301 | 0.3060 | 0.0123 |
|  | 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 |
|  | B061 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 |
|  | B078 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B079 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| $\begin{gathered} \text { sen } \\ 0 \\ 0 \end{gathered}$ |  | Consensus Mean <br> Consensus Standard Deviatiol <br> Maximum <br> Minimum <br> N |  |  | 0.0184 |  | Consensus Mean |  |  | 0.612 |  | Consensus Mean |  |  | 0.3244 |  |
|  |  |  |  |  | 0.0015 |  | Consensus Standard Deviatio |  |  | 0.022 |  | Consensus Standard Deviatio |  |  | 0.0073 |  |
|  |  |  |  |  | 0.6389 |  | Maximum |  |  | 1.350 |  | Maximum |  |  | 0.4029 |  |
|  |  |  |  |  | 0.0123 |  | Minimum |  |  | 0.036 |  | Minimum |  |  | 0.0341 |  |
|  |  |  |  |  | 15 |  |  |  |  | 24 |  |  |  |  | 24 |  |

Table 3-5. continued.

|  |  | Molybdenum (Mo) |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  | 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 | A | B | C | Avg | SD | A | B | C | Avg | SD | A | B | C | 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 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B155 | $<1$ | $<1$ |  |  |  | $<1$ | $<1$ | $<1$ |  |  | $<1$ | $<1$ | $<1$ |  |  |
|  | B161 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 |
|  | 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 |
|  | B198 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B202 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 |
|  | B208 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 |
| cos |  | Consensus Mean <br> Consensus Standard Deviatiol <br> Maximum <br> Minimum <br> N |  |  | 0.0184 |  | Consensus Mean <br> Consensus Standard Deviatio <br> Maximum <br> Minimum <br> N |  |  | $\begin{gathered} \hline 0.612 \\ 0.022 \\ 1.350 \\ 0.036 \\ 24 \end{gathered}$ |  | Consensus Mean <br> Consensus Standard Deviatio <br> Maximum <br> Minimum <br> N |  |  | 0.3244 |  |
|  |  |  |  |  | 0.0015 |  |  |  |  | 0.0073 |  |  |  |  |
|  |  |  |  |  | 0.6389 |  |  |  |  | 0.4029 |  |  |  |  |
|  |  |  |  |  | 0.0123 |  |  |  |  | 0.0341 |  |  |  |  |
|  |  |  |  |  | 15 |  |  |  |  | 24 |  |  |  |  |



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 $Z_{\text {comm }}^{\prime} s c o r e,\left|Z_{\text {comm }}^{\prime}\right| \leq$ 2. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (UNIST) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 3-29. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 3-30. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 3-31. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 3-32. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. NIST value has not been determined in this material.

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


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 ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq$ 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$.

Exercise: CannaQAP Exercise 2, Measurand: molybdenum
No. of laboratories: 16


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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$.

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Exercise: CannaQAP Exercise 2, Measurand: molybdenum
No. of laboratories: 16


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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$.

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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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right|>2$. Note: This table spans two pages; the NIST values and consensus values are included on both pages for convenience.


Table 3-6. continued.

|  |  | Manganese (Mn) |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  | 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 | A | B | C | Avg | SD | A | B | C | Avg | SD | A | B | C | 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 |
|  | 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 |
|  | B192 | 580 | 578 | 610 | 589.3 | 17.9 | 462 | 420 | 421 | 434.3 | 24.0 | 160 | 181 | 187 | 176.0 | 14.2 |
|  | B200 | 452 | 475 | 481 | 469.3 | 15.3 | 409 | 399 | 405 | 404.3 | 5.0 | 147 | 154 | 150 | 150.3 | 3.5 |
|  | B202 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B203 | 488 | 470 | 480 | 479.3 | 9.0 | 389 | 402 | 393 | 394.7 | 6.7 | 132 | 145 | 139 | 138.7 | 6.5 |
|  | B208 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 |
| 为 |  | Consensus <br> Consensus <br> Maximum <br> Minimum <br> N | as Mean s Standard | Deviatio | $\begin{gathered} \hline 458.0 \\ 8.3 \\ 813.2 \\ 376.0 \\ 29 \end{gathered}$ |  | Consensus Consensus Maximum Minimum N | Mean Standard | Deviatio | $\begin{gathered} 366.9 \\ 9.4 \\ 646.1 \\ 201.3 \\ 32 \end{gathered}$ |  | Consensus <br> Consensus <br> Maximum <br> Minimum <br> N | s Mean s Standard | Deviatio | $\begin{gathered} \hline 135.4 \\ 2.4 \\ 186.7 \\ 108.3 \\ 32 \\ \hline \end{gathered}$ |  |



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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 3-39. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 3-41. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 3-42. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIITT }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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).

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 ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq$ 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$.

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


SRM 1575a Trace Elements in Pine Needles (Pinus taeda) [mg/kg]
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 (UNIST) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$.

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


SRM 1575a Trace Elements in Pine Needles (Pinus taeda) [mg/kg]
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 ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$.

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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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right|>2$. Note: This table spans two pages; the NIST values and consensus values are included on both pages for convenience.

|  |  | Nickel ( Ni ) |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  | 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 | A | B | C | Avg | SD | A | B | C | Avg | SD | A | B | C | 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 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 |
|  | 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 |
|  | B061 | 0.9593 | 1.0238 | 0.965 | 0.983 | 0.036 | 0.5943 | 0.6293 | 0.6193 | 0.61 | 0.02 | 2.6361 | 2.6574 | 2.7297 | 2.674 | 0.049 |
|  | B066 | 1.211 | 1.187 | 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 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B079 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B081 | 1.301 | 1.419 | 1.551 | 1.424 | 0.125 | 5.236 | 4.562 | 5.33 | 5.04 | 0.42 | 3.245 | 3.333 | 3.263 | 3.280 | 0.046 |
|  | B084 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B088 | $<1$ | $<1$ | $<1$ |  |  | $<1$ | $<1$ | $<1$ |  |  | $<1$ | <1 | $<1$ |  |  |
|  | B090 | 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 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 | 1.97 | 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 | $<1.349$ |  |  |  |  | $<0.404$ |  |  |  |  | 1.9 |  |  | 1.900 |  |
|  | B102 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| $\begin{aligned} & \text { 感 } \\ & 0 \\ & 0 \end{aligned}$ |  | Consensus <br> Consensus <br> Maximum <br> Minimum <br> N | Mean Standard | Deviation | $\begin{gathered} \hline 1.332 \\ 0.029 \\ 9.559 \\ 0.800 \\ 35 \\ \hline \end{gathered}$ |  | Consensus <br> Consensus <br> Maximum <br> Minimum <br> N | is Mean <br> s Standard $\qquad$ | Deviation | $\begin{gathered} \hline 5.67 \\ 0.21 \\ 54.97 \\ 0.61 \\ 36 \\ \hline \end{gathered}$ |  | Consensus <br> Consensus <br> Maximum <br> Minimum <br> N | s Mean <br> Standard | Deviation | $\begin{gathered} 3.262 \\ 0.062 \\ 20.133 \\ 1.900 \\ 35 \\ \hline \end{gathered}$ |  |

Table 3-7. continued.

|  |  | Nickel (Ni) |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  | 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 | A | B | C | Avg | SD | A | B | C | Avg | SD | A | B | C | 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 |
|  | 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 |
|  | 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 |
|  | 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 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 |
|  | 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 |
| $\begin{aligned} & \text { 为 } \\ & 0 \end{aligned}$ |  |   <br> Consensus Mean 1.332 <br> Consensus Standard Deviation 0.029 <br> Maximum 9.559 <br> Minimum 0.800 <br> N 35 |  |  |  |  | Consensus Mean <br> Consensus Standard Deviation <br> Maximum <br> Minimum <br> N |  |  | $\begin{gathered} 5.67 \\ 0.21 \\ 54.97 \\ 0.61 \\ 36 \\ \hline \end{gathered}$ |  | Consensus <br> Consensus <br> Maximum <br> Minimum <br> N | s Mean standard | Deviation | $\begin{gathered} \hline 3.262 \\ 0.062 \\ 20.133 \\ 1.900 \\ 35 \\ \hline \end{gathered}$ |  |



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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq$ 2. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (UNIST) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 3-47. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 3-48. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 3-49. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIIT }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 3-50. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (UNIST) and represents the range that results in an acceptable $Z_{\text {NIIT }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq$ 2. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIIT }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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).

Exercise: CannaQAP Exercise 2, Measurand: nickel
No. of laboratories: 36


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 ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$.

Exercise: CannaQAP Exercise 2, Measurand: nickel
No. of laboratories: 36


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 (UNIST) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$.

Exercise: CannaQAP Exercise 2, Measurand: nickel
No. of laboratories: 35


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 ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$.

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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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right|>2$. Note: This table spans two pages; the NIST values and consensus values are included on both pages for convenience.

|  |  | Selenium (Se) |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  | 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 | A | B | C | Avg | SD | A | B | C | Avg | SD | A | B | C | 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 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B028 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B031 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B035 | $<0.25$ | <0.25 | <0.25 |  |  | 0.503 | 0.529 | 0.51 | 0.514 | 0.013 | $<0.25$ | $<0.25$ | <0.25 |  |  |
|  | B037 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B040 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 |
|  | 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 |
|  | 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 |
|  | 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 |
|  | B078 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B079 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B084 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B088 | 1.3747 | 1.5598 | 1.2385 | 1.3910 | 0.1613 | <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 | $<0.139$ |  |  |  |  | $<0.464$ |  |  |  |  | $<0.464$ |  |  |  |  |
|  | B102 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B107 | 0.114 | 0.108 | 0.1042 | 0.1087 | 0.0049 | <0.006 | <0.006 | <0.006 |  |  | 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 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 |
| con |  | Consensus Mean <br> Consensus Standard Deviatiol <br> Maximum <br> Minimum <br> N |  |  | 0.1191 |  | Consensus Mean <br> Consensus Standard Deviatio <br> Maximum <br> Minimum <br> N |  |  | 0.462 |  | Consensu | S Mean |  | 0.1058 |  |
|  |  |  |  |  | 0.0069 |  |  |  |  | 0.051 |  | Consensu | S Standard | Deviatio | 0.0077 |  |
|  |  |  |  |  | 1.3910 |  |  |  |  | 1.223 |  | Maximum |  |  | 0.5299 |  |
|  |  |  |  |  | 0.0463 |  |  |  |  | 0.095 |  | Minimum |  |  | 0.0230 |  |
|  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |

Table 3-8. continued.

|  |  | Selenium (Se) |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  | 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 | A | B | C | Avg | SD | A | B | C | Avg | SD | A | B | C | 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 |  |  |
|  | 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 |
|  | 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 |
|  | 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 |
|  | B202 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 |
|  | 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 |
| $\begin{gathered} \text { cos } \\ 0 \\ 0 \end{gathered}$ |  | Consensus Consensus Maximum Minimum N | s Mean <br> standard | Deviatios | $\begin{gathered} 0.1191 \\ 0.0069 \\ 1.3910 \\ 0.0463 \\ 23 \end{gathered}$ |  | Consensus Consensus Maximum Minimum N | is Mean <br> standard | Deviatio | $\begin{gathered} \hline 0.462 \\ 0.051 \\ 1.223 \\ 0.095 \\ 25 \end{gathered}$ |  | Consensus Consensus Maximum Minimum N | s Mean <br> Standard | Deviatio | $\begin{gathered} 0.1058 \\ 0.0077 \\ 0.5299 \\ 0.0230 \\ 23 \end{gathered}$ |  |



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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq$ 2. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (UNIST) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 3-56. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 3-57. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 3-58. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 3-59. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (UNIST) and represents the range that results in an acceptable $Z_{\text {NIIT }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq$ 2. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIIT }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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).

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 ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq$ 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$.


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 (UNIST) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. The dotted blue box (the bottom limit is not shown due to the scale of the figure) represents the consensus range of tolerance for 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$.

Exercise: CannaQAP Exercise 2, Measurand: selenium
No. of laboratories: 21


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 ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$.

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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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right|>2$.

|  |  | Uranium (U) |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  | 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 | A | B | C | Avg | SD | A | B | C | Avg | SD | A | B | C | 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 |
|  | B113 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B119 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B122 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 |  |
|  | 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 |  |
|  | 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 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B155 | <0.1 | <0.1 |  |  |  | 0.53 | 0.54 | 0.53 | 0.533 | 0.006 | $<0.1$ | <0.1 | $<0.1$ |  |  |
|  | B161 |  |  |  |  |  | 11.8 | 11.8 | 12.2 | 11.933 | 0.231 |  |  |  |  |  |
|  | B163 | 0.00625 | 0.006070 .0 | 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 |
|  | B179 | 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 | 0.474 | 0.506 | 0.509 | 0.496 | 0.019 | 0.0044 | 0.0039 | 0.0043 | 0.00420 | 0.00026 |
|  | B202 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 | 0.00153 |
|  | B208 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B211 | <1 |  |  |  |  | <1 |  |  |  |  | $<1$ |  |  |  |  |
|  | B213 |  |  |  |  |  | 0.27 | 0.25 | 0.21 | 0.243 | 0.031 | $<0.02$ | $<0.02$ | <0.02 |  |  |
|  | B214 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B221 | $<0.01$ | $<0.01$ | $<0.01$ |  |  | 0.187 | 0.178 | 0.169 | 0.178 | 0.009 | $<0.01$ | $<0.01$ | $<0.01$ |  |  |
|  | B222 | $<0.01$ | <0.01 | <0.01 |  |  | 0.229 | 0.229 | 0.236 | 0.231 | 0.004 | $<0.01$ | <0.01 | <0.01 |  |  |
|  | B228 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B230 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B231 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B232 | 0.007 | 0.005290 | 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 | 0.010 | 0.00255 | 0.00244 | 0.0023 | 0.00243 | 0.00013 |
| 会 |  | Consensu <br> Consensu <br> Maximum <br> Minimum <br> N | us Mean us Standard n $\qquad$ | Deviatio: | $\begin{gathered} \hline 0.00525 \\ 0.00034 \\ 0.00717 \\ 0.00330 \\ 12 \\ \hline \end{gathered}$ |  | Consensus <br> Consensus <br> Maximum <br> Minimum <br> N | as Mean us Standard <br> n | Deviatio | $\begin{gathered} \hline 0.268 \\ 0.020 \\ 11.933 \\ 0.153 \\ 19 \\ \hline \end{gathered}$ |  | Consensus <br> Consensus <br> Maximum <br> Minimum <br> N | Mean Standard | Deviatio: | 0.00326 0.00031 0.00433 0.00200 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq$ 2. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIIT }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 3-65. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 3-66. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 3-67. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 3-68. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq$ 2.

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


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 ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$.

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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$.

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


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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$.

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Table 3-10. Data summary table for vanadium $(\mathrm{V})$ 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right|>2$. Note: This table spans two pages; the NIST values and consensus values are included on both pages for convenience.

|  |  | Vanadium (V) |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  | 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 | A | B | C | Avg | SD | A | B | C | Avg | SD | A | B | C | 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 | $<1$ | $<1$ |  |  |  | 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 |
|  | B037 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 |
|  | B061 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B088 | <1 | <1 | <1 |  |  | <1 | $<1$ | <1 |  |  | 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 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B111 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B112 | 0.102 | 0.106 | 0.089 | 0.0990 | 0.0089 | 10.32 | 11.74 | 9.72 | 10.59 | 1.04 | 0.148 | 0.17 | 0.147 | 0.155 | 0.013 |
|  | B113 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | B119 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 |
| $\begin{gathered} 0 \\ 0 \end{gathered}$ |  | Consensus Mean <br> Consensus Standard Deviatiol <br> Maximum <br> Minimum <br> N |  |  | 0.1041 |  | Consensus Mean <br> Consensus Standard Deviatio <br> Maximum <br> Minimum <br> N |  |  |  |  | Consensus Mean Consensus Standard Deviatio |  |  | 0.208 |  |
|  |  |  |  |  | 0.0050 |  |  |  |  | 13.830.70 |  |  |  |  | 0.015 |  |
|  |  |  |  |  | 0.3623 |  |  |  |  | 25.87 |  | Maximum |  |  | 10.144 |  |
|  |  |  |  |  | 0.0807 |  |  |  |  | 2.69 |  | Minimum |  |  | 0.141 |  |
|  |  |  |  |  | 17 |  |  |  |  | 24 |  | N |  |  | 20 |  |

Table 3-10. continued.

|  |  | Vanadium (V) |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  | 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 | A | B | C | Avg | SD | A | B | C | Avg | SD | A | B | C | 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 |
|  | B176 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 |
|  | 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 |
|  | B208 |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 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 |
| $\begin{gathered} \text { on } \\ 0 \\ 0 \end{gathered}$ |  | Consensus <br> Consensus <br> Maximum <br> Minimum <br> N | s Mean standard |  | $\begin{gathered} \hline 0.1041 \\ 0.0050 \\ 0.3623 \\ 0.0807 \\ 17 \\ \hline \end{gathered}$ |  | Consensus <br> Consensus <br> Maximum <br> Minimum <br> N | is Mean standard | Deviatio | $\begin{gathered} 13.83 \\ 0.70 \\ 25.87 \\ 2.69 \\ 24 \end{gathered}$ |  | Consensus <br> Consensus <br> Maximum <br> Minimum <br> N | s Mean <br> Standard | Deviatio | $\begin{gathered} \hline 0.208 \\ 0.015 \\ 10.144 \\ 0.141 \\ 20 \end{gathered}$ |  |



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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq$ 2. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 3-74. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 3-75. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 3-76. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq 2$. 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. 3-77. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$. 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq$ 2. A NIST value has not been determined in this material.

Exercise: CannaQAP Exercise 2, Measurand: vanadium
No. of laboratories: 19


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 ( $U_{\text {NIST }}$ ) and represents the range that results in an acceptable $Z_{\text {NIST }}$ score, $\left|Z_{\text {NIST }}\right| \leq$ 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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$.

## 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 $Z_{\text {comm }}^{\prime}$ Score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$.

## Exercise: CannaQAP Exercise 2, Measurand: vanadium

No. of laboratories: 18


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 $Z_{\text {comm }}^{\prime}$ score, $\left|Z_{\text {comm }}^{\prime}\right| \leq 2$.

## References

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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 |  |  |  |  |  |  |
| :--- | :--- | :--- | :--- | :--- | :--- | :--- |
| 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 |

December 2022

## Sample Preparation

## Homogenization



| Response | Laboratory |  |  |  |  |  |  |  |  |  |
| :--- | :--- | :--- | :--- | :--- | :--- | :--- | :--- | :--- | :--- | :--- |
| 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 |  |  |  |  |  |  |

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Homogenization Method


| Homogenization <br> Method | Laboratory |  |  |  |  |  |  |  |  |  |
| :--- | :--- | :--- | :--- | :--- | :--- | :--- | :--- | :--- | :--- | :---: |
| Mixed | B012 | B029 | B030 | B035 | B064 | B077 | B088 | B100 | B109 |  |
|  | 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 | Laboratory |  |  |  |  |  |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| 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 | $\begin{aligned} & \text { B012 } \\ & \text { B113 } \\ & \text { B192 } \end{aligned}$ | B015 | B016 | B035 | B041 | B060 | B082 | B088 | B100 | B104 |
|  |  | B146 | B147 | B154 | B155 | B159 | B172 | B181 | B182 | B190 |
|  |  | B204 | B206 | B223 |  |  |  |  |  |  |
| Not Specified | $\begin{aligned} & \text { B003 } \\ & \text { B203 } \end{aligned}$ | B027 | B029 | B030 | B061 | B064 | B066 | B109 | B195 | B202 |
|  |  | B212 | B213 | B216 | B217 | B222 |  |  |  |  |

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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 |  |  |  |  |  |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| $\mathrm{HCl}, \mathrm{HNO}_{3}$ | 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 |  |  |  |
| $\mathrm{HNO}_{3}$ | B015 | B035 | B082 | B113 | B153 | B159 | B178 | B190 | B202 | B217 |
|  | B222 | B223 |  |  |  |  |  |  |  |  |
| $\mathrm{H}_{2} \mathrm{O}_{2}, \mathrm{HNO}_{3}$ | B012 | B016 | B100 | B147 | B154 |  |  |  |  |  |
|  | B030 | B182 |  |  |  |  |  |  |  |  |
| $\mathrm{H}_{2} \mathrm{O}_{2}, \mathrm{HNO}_{3}, \mathrm{H}_{2} \mathrm{SO}_{4}$ | B205 |  |  |  |  |  |  |  |  |  |
| Samples were not digested | B155 |  |  |  |  |  |  |  |  |  |
| Not Specified | B203 |  |  |  |  |  |  |  |  |  |

December 2022
Maximum Microwave Temperature

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

| Maximum Temperature | Laboratory |  |  |  |  |  |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| $180{ }^{\circ} \mathrm{C}$ | B012 | B064 | B113 | B154 | B159 | B178 |  |  |  |  |
| $185{ }^{\circ} \mathrm{C}$ | B027 |  |  |  |  |  |  |  |  |  |
| $190{ }^{\circ} \mathrm{C}$ | B182 | B222 |  |  |  |  |  |  |  |  |
| $195{ }^{\circ} \mathrm{C}$ | B153 |  |  |  |  |  |  |  |  |  |
| $200{ }^{\circ} \mathrm{C}$ | B016 | B041 | B082 | B104 | B172 | B192 |  |  |  |  |
| $210{ }^{\circ} \mathrm{C}$ | B003 | B146 |  |  |  |  |  |  |  |  |
| $225{ }^{\circ} \mathrm{C}$ | B142 |  |  |  |  |  |  |  |  |  |
| $230{ }^{\circ} \mathrm{C}$ | B060 | B077 |  |  |  |  |  |  |  |  |
| $240{ }^{\circ} \mathrm{C}$ | 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 | Laboratory |  |  |  |  |  |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| 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 |  |  |  |  |  |  |  |  |  |

December 2022
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 |  |  |  |  |  |  |  |

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December 2022
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 Standard | B003 | B012 | B016 | B041 | B077 | B100 | B102 | B104 |
|  | B109 | B146 | B154 | B172 | B186 | B192 | B195 | B202 |
|  | B206 | B223 | B224 |  |  |  |  |  |
| Calibration Curve with External Standards | B088 |  |  |  |  |  |  |  |
| Calibration Curve with Internal and External Standards | B204 |  |  |  |  |  |  |  |
| Internal Standard | B027 |  |  |  |  |  |  |  |
| External Standards | B155 |  |  |  |  |  |  |  |
| Internal and External Standards | B066 |  |  |  |  |  |  |  |
| Not Specified | B203 |  |  |  |  |  |  |  |


[^0]:    ${ }^{1}$ 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.

