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Abstract

The Health Assessment Measurements Quality Assurance Program (HAMQAP) was launched in collaboration with the NIH ODS in 2017. HAMQAP was established to enable laboratories to improve the accuracy of measurements in samples that represent human intake (e.g., foods, dietary supplements, tobacco) and samples that represent human metabolism (e.g., blood, serum, plasma, urine) for demonstration of measurement proficiency and/or compliance with various regulations. Analytes are paired, where possible, to represent the full spectrum of health assessment. Exercise 7 of this program offered the opportunity for laboratories to assess their in-house measurements of nutritional elements (calcium, magnesium, and zinc), toxic elements (arsenic, cadmium, lead, and mercury), water-soluble vitamins (vitamins B_2 and B_6 and homocysteine), fat-soluble vitamins (vitamin K), phytochemicals (gingerols), and protein source identification (pea, rice, soy, and milk) in foods and dietary supplements, as well as corresponding biomarkers/metabolites in clinical specimens (human blood, and human and animal serum).

Keywords

Clinical Measurements; Dietary Supplements; Food Safety; Metabolites; Nutritional; Quality Assurance; Reference Materials.

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List of Acronyms

AAS	Atomic Absorption Spectroscopy
AMRM	Analytical Methods and Reference Materials
CDC	US Centers for Disease Control and Prevention
CGMP	Current Good Manufacturing Practice
COA	Certificate of Analysis
CRM	Certified Reference Material
DSQAP	Dietary Supplements Laboratory Quality Assurance Program
FAQAP	Fatty Acids in Human Serum and Plasma Quality Assurance Program
FDA	US Food and Drug Administration
HAMQAP	Health Assessment Measurements Quality Assurance Program
HPTLC	High-Performance Thin-Layer Chromatography
IC	Ion Chromatography
IC-MS	Ion Chromatography Mass Spectrometry
ICP-MS	Inductively Coupled Plasma Mass Spectrometry
KED	Kinetic Energy Discrimination
ICP-OES	Inductively Coupled Plasma Optical Emission Spectrometry
ID ICP-MS	Isotope Dilution Inductively Coupled Plasma Mass Spectrometry
JCTLM	Joint Committee for Traceability in Laboratory Medicine
LC-Abs	Liquid Chromatography with Absorbance Detection
LC-FLD	Liquid Chromatography with Fluorescence Detection
LC-MS	Liquid Chromatography Mass Spectrometry
LC-MS/MS	Liquid Chromatography with Tandem Mass Spectrometry
LOQ	Limit of Quantification
MMQAP	Micronutrients Measurement Quality Assurance Program
NIST	National Institute of Standards and Technology
NIH	National Institutes of Health
ODS	Office of Dietary Supplements
PDA	Photodiode-Array Detection
QAP	Quality Assurance Program
QL	Quantification Limit
RM	Reference Material
RSD	Relative Standard Deviation
RMP	Reference Measurement Procedure
SD	Standard Deviation
SODF	Solid Oral Dosage Form
SRM	Standard Reference Material
VitDQAP	Vitamin D Metabolites Quality Assurance Program

Introduction

The NIST HAMQAP was formed in 2017, in part as a collaboration with the National Institutes of Health Office of Dietary Supplements (NIH ODS) and represents ongoing efforts at NIST that were supported previously via historical QAPs, including the Dietary Supplements Laboratory Quality Assurance Programs (DSQAP), Fatty Acids in Human Serum and Plasma Quality Assurance Program (FAQAP), Micronutrients Measurement Quality Assurance Program (MMQAP), and Vitamin D Metabolites Quality Assurance Program (VitDQAP).

NIST has decades of experience in the administration of QAPs and HAMQAP builds upon the approach taken by DSQAP by providing a wide range of matrices and analytes. The HAMQAP design combines activities of DSQAP, FAQAP, MMQAP, and VitDQAP, and emphasizes emerging and challenging measurements in the dietary supplement, food, and clinical matrix categories. Samples that represent human intake (e.g., food, dietary supplements, natural products) are paired with samples that represent human metabolism (e.g., blood, serum, plasma, urine) where possible, to represent the full spectrum of intake and metabolism for health assessment, including but not limited to measurements of nutritional and toxic elements, water- and fat-soluble vitamins, fatty acids, active and/or marker compounds, and contaminants.

HAMQAP offers the opportunity for laboratories to evaluate in-house methods on a wide variety of challenging, real-world matrices and to demonstrate that their performance is comparable to that of the community and that their methods provide accurate results. In areas where few standard methods have been recognized, HAMQAP offers a unique tool for assessment of the quality of measurements and provides feedback about performance that can assist participants in improving laboratory operations. Reports and certificates of participation are provided and may be used to demonstrate compliance with the Current Good Manufacturing Practice regulations (CGMPs) or to demonstrate proficiency to accreditation bodies when a formal proficiency testing program is not available. In addition, NIST and HAMQAP assist the NIH ODS Analytical Methods and Reference Materials (AMRM) Program in supporting the development and dissemination of analytical tools and reference materials. Results from HAMQAP exercises can be used by NIH ODS and NIST to identify problematic matrices and analytes for which consensus-based methods of analysis would benefit the dietary supplements and clinical communities.

This report summarizes the results from the seventh exercise of HAMQAP. Forty-seven laboratories responded to the dietary intake portion and six laboratories responded to the human metabolites portion of the call for participants distributed in March 2021 (see table below). Samples were shipped to participants in June 2021 and results were returned to NIST by September 2021. This report contains the final data and information that was disseminated to the participants in December 2022.

Study Group	Dietary Intake Study	Human Metabolites Study
Nutritional Elements	Zn, Mg, and Ca Nutritionally Fortified Water	Zn, Mg, and Ca Human and Animal Serum
Toxic Elements	As, Cd, Pb, and Hg Black Cohosh Extract, Ashwagandha Extract	As, Cd, Pb, and Hg Human Blood, Animal Serum
Water-Soluble Vitamins	Vitamins B2 and B6 Multivitamin, Protein Powder	Vitamins B2 and B6, Homocysteine Human Serum
Fat-Soluble Vitamins	Vitamin K Multivitamin, Kelp	Vitamin K Human Serum
Botanicals	Gingerols Ginger Rhizome and Extract, Ginger-Containing Dietary Supplements	Not Offered
Protein Source Identification	Protein Source Protein Powder Supplements	Not Offered

Each study group is summarized in a series of tables, figures, and text, and reported by section. Within the section, each study is summarized individually, and then conclusions are drawn for the entire study group when possible.

Overview of Data Treatment and Representation

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

Statistics

Data tables and figures throughout this report contain information about the performance of each laboratory relative to that of the other participants in this study and relative to a target around the expected result, if available. All calculations are performed in PROLab Plus (QuoData GmbH, Dresden, Germany).[1] The consensus means and standard deviations are calculated according to the robust Q/Hampel method outlined in ISO 13528:2015, Annex C. [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, or estimated values, when available). Participating

[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.

laboratories receive uniquely coded individualized data tables in a separate distribution, with the randomized laboratory code in the upper left of the data table ("NIST" for the examples in this report).

Section 1 of the data table (*Your Results*) contains the laboratory results as reported, including the mean and standard deviation when multiple values were reported. A blank indicates that NIST does not have data on file for that laboratory for the corresponding analyte or matrix. An empty box for standard deviation indicates that the participant reported a single value or a value below the Limit of Quantification (LOQ) and therefore that value was not included in the calculation of the consensus data.**Error! Bookmark not defined.** Example individualized data tables are included in S ection 1 of this report using NIST data to protect the identity and performance of participants.

Also included in Section 1 are two Z-scores. The first Z-score, Z'_{comm}, is calculated with respect to the community consensus value, taking into consideration bias that may result from the uncertainty in the assigned consensus value, using the consensus mean (x*), consensus standard deviation (s*), and standard deviation for proficiency assessment (SDPA, σ_{PT}^2) determined from the Q/Hampel estimator:

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

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

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

or

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

Significance of the *Z*-scores:

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

Section 2 of the data table (*Community Results*) contains the consensus results, including the number of laboratories reporting more than a single quantitative value for each analyte, the mean value determined for each analyte, and a robust estimate of the standard deviation of the reported values.**Error! Bookmark not defined.** Consensus means and standard deviations are calculated using the l aboratory means; if a laboratory reported a single value, the reported value is not included in determination of the consensus values.[3] 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 a NIST Standard Reference Material (SRM) or Reference Material (RM) is used as a sample in the study, the NIST certified or non-certified values and their associated uncertainties (U_{95}) are used as target values. The criteria used by NIST to assign certified and non-certified values is described elsewhere. [2] Target values for other study samples may be determined at NIST or by a collaborating laboratory as the mean of at least three replicates. Target values may also be determined from another interlaboratory study or proficiency testing program, where the consensus value and uncertainty from the completed round is used as the target range, or based on information provided by the material manufacturer. The exact methods for determination of the study target values are outlined in detail within each section of this report.

Summary Data Table

This data table includes a summary of all reported data for a particular analyte in a particular study. Participants can compare the raw data for their laboratory to data reported by the other participating laboratories and to the consensus data. A blank indicates that the laboratory signed up and received samples for that analyte and matrix, but NIST does not have data on file for that laboratory. Data highlighted in red have been flagged as a data entry of zero or results that include text (e.g., "< LOQ" or "present"). Data highlighted in blue have been identified as outside the consensus tolerance limits and would be estimated to yield $|Z'_{\text{comm}}| > 2$ by the NIST software package.

Figures

Data Summary View (Method Comparison Data Summary View)

In this view, individual laboratory data (circles) are plotted with the individual laboratory standard deviation (rectangle). Laboratories reporting values below the LOQ are shown in this view as downward triangles beginning at the LOQ, reported as QL on the figures. Laboratories reporting values as "below LOQ" can still be successful in the study if the target value is also below the laboratory LOQ. The blue solid line represents the consensus mean, and the green shaded area represents the 95 % confidence interval for the consensus mean, based on the standard error of the consensus mean. The uncertainty in the consensus mean is calculated using the equation below, based on the repeatability standard deviation (s_{r}), the reproducibility standard deviation (s_{R}), the number of participants reporting data, and the average number of replicates reported by each participant. The uncertainty about the consensus mean is independent of the range of tolerance.

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

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

Sample/Sample Comparison View

In this view, the individual laboratory results for one sample (e.g., NIST material with a certified target value, a less challenging matrix) are compared to the results for another sample (e.g., NIST material with a more challenging matrix, a commercial sample). The solid red box represents the target range for the first sample (x-axis) and the second sample (y-axis), if available. The dotted blue box represents the consensus range 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, to a limit of twice the range of tolerance (values that result in an acceptable Z'_{comm} score, $|Z'_{comm}| \leq 2$). Depending on the variability in the data, the axes may be scaled proportionally to better display the individual data points for each laboratory. In some cases, when the consensus and target ranges have limited overlap, the solid red box may only appear partially on the graph. If the variability in the data is high (greater than 100 % RSD), the dotted blue box may also only appear partially on the graph. 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.

Overall Technical Recommendations

The following general technical recommendations are important to consider for achieving accurate and precise measurements. For specific recommendations focused on a particular sample matrix or analyte type, see the individual study technical recommendation sections.

- Analysis of quality assurance materials (commercially available reference materials or appropriately characterized in-house materials) helps to establish that sample preparation methods and analytical methods are appropriate and performing as expected.
- Analysis of blanks can provide information about sources of analytical variability, such as from the sample preparation procedure or the material itself. Analysis of an appropriate number of procedural blanks (e.g., equal to the number of samples) is important, especially when determining an LOQ or when trying to reduce sample-to-sample variability.
- Calibration is critical to successful measurements.
    - When using a calibration curve, linearity must be ensured at the concentrations of the sample solutions being measured and the range of calibrant concentrations should encompass the sample mass fractions. No sample mass fractions should be outside of the linear range.
    - Calibrants should be evaluated for purity and presence of residual solvents prior to use. The measured purity should be used to correct the gravimetric or volumetric concentrations of the solutions used for calibration.
    - Individually matched calibrants should be used for quantitation whenever possible to avoid potential biases that may arise during sample preparation or from differences in chromatographic retention time or detector sensitivity.
    - The addition of an internal standard is recommended to help improve the precision of the instrumental measurements. Selecting the appropriate internal standard will help to correct measurement variability between the calibration standards and the samples.
- Calculations and reporting units must be verified prior to submission of results. Laboratories often report results in the wrong units or forget a dilution factor during the calculation of the final results, resulting in poor performance on the study. Laboratories reporting results which have been flagged as outside of consensus tolerance limits when sent preliminary data sheets should check for these types of errors and provide corrected results.
- Results should be recorded appropriately in the online data entry system.
    - Zero is not a quantity that can be measured.
    - If values are below LOQ, results should be reported as such (e.g., “< 0.02 %”).
    - Blank data entry fields are only appropriate when no measurements were made.

1. Nutritional Elements (Calcium, Magnesium, Zinc)

1.1. Study Overview

Consumers expect labeling information to be accurate on the food and dietary supplement products they purchase in order to make informed choices. In the U.S., accurate measurements of nutrients on the levels claimed on Nutrition Facts and Supplement Facts labels are needed to ensure compliance with the FDA regulations. Appropriate levels of trace minerals are essential for the body to function properly, and deficiencies or excess consumption can lead to potential health risks. Certain foods are often fortified with trace minerals, and these nutrients are also increasingly found in nutritionally fortified waters. Testing of these minerals in nutritionally supplemented water can help ensure accurate product labeling.

In this study, participants were provided with samples of SRM 1643f Trace Elements in Water (Water A) and a nutritionally enhanced water sample (Water B) for dietary intake. Participants were asked to use in-house analytical methods to determine the mass fractions (mg/kg) of calcium (Ca), magnesium (Mg), and zinc (Zn) in the dietary intake samples.

1.2. Sample Information

Water A. Participants were provided with one bottle containing 250 mL of SRM 1643f Trace Elements in Water. Participants were asked to store the material at controlled room temperature, 20 °C to 25 °C, in the original unopened bottle sealed inside the original aluminized plastic bag to maintain stability, to prepare three samples, and to report three values from the one bottle provided. Before use, participants were instructed to thoroughly mix the contents of the bottle prior to removal of a test portion for analysis, and to use a sample size of at least 0.5 g for the determination of Ca, Mg, and Zn. Approximate analyte levels were not disclosed to participants prior to the study. The target values for Ca, Mg, and Zn in SRM 1643f were determined at NIST using ICP-MS or ICP-OES. The certified values and uncertainties from the COA at the time of this report are provided in the table below.

Analyte	Target Mass Fractions in SRM 1643f (mg/kg)
Calcium (Ca)	29.140 ± 0.32
Magnesium (Mg)	7.38 ± 0.058
Zinc (Zn)	0.0737 ± 0.0017

Water B. Participants were provided with one bottle containing 500 mL of nutritionally enhanced water. Participants were asked to store the material at controlled room temperature, 20 °C to 25 °C, in the original unopened bottle, to prepare three samples, and to report three values from the single bottle provided. Before use, participants were instructed to thoroughly mix the contents of the bottle prior to removal of a test portion for analysis, and to use a sample size of at least 0.5 g for the determination of Ca, Mg, and Zn. Approximate analyte levels were not disclosed to participants prior to the study. The target values and standard deviations for Ca, Mg, and Zn were determined at NIST using ICP-OES and are provided in the table below.

Analyte	Target Mass Fractions in Water B (mg/kg)
Calcium (Ca)	283.3 ± 36.4
Magnesium (Mg)	91.0 ± 8.8
Zinc (Zn)	5.12 ± 1.72

1.3. Study Results

The enrollment and reporting statistics for the dietary intake study is described in the table below. The table below lists the participation statistics for each analyte. Reported values may include non-quantitative results (zero or below LOQ) but are included in the participation statistics.

Analyte	Number of Laboratories Requesting Samples	Number of Laboratories Reporting Results (Percent Participation)	
		SRM 1643f	Water B
Calcium (Ca)	33	22 (67 %)	22 (67 %)
Magnesium (Mg)	33	22 (67 %)	22 (67 %)
Zinc (Zn)	34	22 (65 %)	23 (68 %)

The between-laboratory variabilities were below 15 % for most analytes in both materials. The between-laboratory variability was 43 % for Zn in SRM 1643f.

Analyte	Between-Laboratory Variability (% RSD)	
	SRM 1643f	Water B
Calcium (Ca)	14 %	8 %
Magnesium (Mg)	11 %	8 %
Zinc (Zn)	43 %	12 %

The within-laboratory variabilities ranged from 0.1 % to 33 % for all analytes in Water B. In SRM 1643f, the within-laboratory variabilities ranged from 0.2 % to >100 % for all analytes (see table below).

Analyte	Within-Laboratory Variability (% RSD)	
	SRM 1643f	Water B
Calcium (Ca)	0.2 % to 19 %	0.2 % to 10 %
Magnesium (Mg)	0.2 % to > 100 %	0.1 % to 33 %
Zinc (Zn)	0.4 % to 74 %	0.1 % to 20 %

About half of laboratories reported using microwave digestion for sample preparation prior to determination of Ca, Mg, and Zn. Other reported sample preparation methods included hot block digestion, dilution, and solvent or solid phase extraction.

Reported Sample Preparation Method	Percent Reporting (Averaged for both sample types) Ca	Mg	Zn
Microwave Digestion	48 %	48 %	51 %
Hot Block Digestion	27 %	27 %	27 %
Dilution	9 %	9 %	7 %
Solvent Extraction and Solid Phase Extraction	5 %	5 %	4 %
Other/None Reported	11 %	11 %	11 %

About half of the laboratories reported using ICP-OES for the determination of Ca, Mg, and Zn. Other reported analytical methods included ICP-MS, ID ICP-MS, and AAS.

Reported Analytical Method	Percent Reporting (Averaged for both sample types) Ca	Mg	Zn
ICP-OES	50 %	50 %	47 %
ICP-MS	27 %	27 %	31 %
ID ICP-MS	5 %	5 %	4 %
AAS	5 %	5 %	4 %
Other/None Reported	14 %	14 %	13 %

The accuracy of results varied by element and by sample, as described in the table below. Only 11 % to 20 % of laboratories were within the NIST range of tolerance for the three elements measured in SRM 1643f.

	Relative to NIST Range of Tolerance for SRM 1643f			Water B		
Position of	Ca	Mg	Zn	Ca	Mg	Zn
Consensus Mean	Slightly above	Above	Slightly Above	Within	Within	Within
Consensus Range	Overlapping upper edge	Above	Overlapping upper edge	Centered	Centered	Centered
Corresponding Figures	1-1, 1-2	1-6, 1-7	1-11, 1-12	1-3, 1-4	1-8, 1-9	1-13, 1-14

1.4. Nutritional Elements Technical Recommendations

The following recommendations are based on results obtained from the participants in this study. Additional overall technical recommendations can be found on page 6.

- No trends were observed based on the sample preparation method or analytical method used for any element.
- SRM 1643f is an elementally enhanced water containing dilute nitric acid. Water B is a commercially available nutritionally enhanced water, with some sugars added. Both water samples were thought to be ideal matrices for straightforward digestion protocols/programs, as they can be diluted and analyzed directly, eliminating the sample digestion step and any possible errors that may be associated with the digestion.
    - For Zn, the between-laboratory variability was 43 % in SRM 1643f and 12 % for Water B. Zn is at a significantly lower level in SRM 1643f compared to Water B.
- The most likely source of error in this study is related to calibration.
    - Calibration curves should include the lowest and highest expected sample solution concentrations, plus one or two intermediate concentration points in the calibration curve. Sample solution concentrations should not go beyond the linear range of the calibration curve. This can result in extrapolation of calibration curves and leading to false values.
    - Calibration curves must be linear at the point of the expected sample solution concentrations.
    - Sample solutions may require dilution fall into the linear range of the calibration curve.
    - The method of standard additions can be used to overcome effects caused by the sample matrix. If used, the highest concentration of the calibration curve will need to be extended based on the total concentration of the analyte in the spiked solution which equals the analyte spike plus the analyte in the unspiked solution.
- When using ICP-MS, be sure to make proper use of the instrumental features:
    - Many ICP-MS instruments operate in pulse counting mode, which is more sensitive than analog mode. Instruments typically switch between pulse counting and analog modes automatically depending on the dynamic range in use, and therefore the instrument must be calibrated for both modes. To ensure that the calibration curve is linear in the pulse counting mode, consider using a narrower range of calibration points and ensure all solutions are diluted to fall within this lower range.
    - The biggest interference for Ca measurement by ICP-MS is $^{40}Ar^{+}$. To mitigate this interference, KED mode can be used when available. If using ^{44}Ca for Ca measurement, He must be used as the collision gas. If using ^{40}Ca, H_2 should be used as the collision gas.
    - Quantification of Mg can be affected by $^{12}C_2^{+}$ interferences, which can be minimized by using He gas with KED mode. Washout between samples is typically not a problem with Mg determinations.
    - KED mode can reduce PO_2^{+} and SO_2^{+} interferences on Zn determination.

- When using ICP-OES, monitoring more than one wavelength for each analyte in conjunction with the use of a reference material helps not only to identify interferences or background shifts due to matrix effects at a given wavelength, but also to identify and prevent bias.
- Addition of internal standards is recommended to help improve the precision of the instrumental measurements. Selecting the appropriate internal standard will help to eliminate noise sources by simultaneous measuring the internal standard and the analyte of interest. [3]

Table 1-1. Individualized data table (NIST) for nutritional elements in water.

Exercise 7 - Nutritional Elements

		Lab Code:	**NIST**	**1. Your Results**				**2. Community Results**			**3. Target**	
Analyte	Sample		Units	x_i	s_i	Z'_{comm}	Z_{NIST}	N	x*	s*	x_{NIST}	*U*
Calcium	SRM 1643f Trace Elements in Water		mg/kg	29.14	0.32			22	30.7	4.2	29.14	0.32
Calcium	Water B		mg/kg	283	36			22	274.6	23.2	283	36
Magnesium	SRM 1643f Trace Elements in Water		mg/kg	7.38	0.06			22	8.04	0.86	7.38	0.06
Magnesium	Water B		mg/kg	91.0	8.8			22	88.3	7.3	91.0	8.8
Zinc	SRM 1643f Trace Elements in Water		mg/kg	0.074	0.002			22	0.090	0.039	0.074	0.002
Zinc	Water B		mg/kg	5.12	1.72			23	5.53	0.64	5.12	1.72

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

Table 1-2. Data summary table for calcium in water. Data highlighted in blue have been identified as outside the consensus range of tolerance and resulted in an unacceptable Z'_{comm} score, $|Z'_{comm}| > 2$.

		Calcium									
		SRM 1643f Trace Elements in Water (mg/kg)					Water B (mg/kg)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				29.14	0.32				283.3	36.4
	G001	35.3	34.5	35.1	34.97	0.42	299.5	302.7	306.9	303.0	3.7
	G002										
	G005	30	30	30	30	0	270	270	280	273.3	5.8
	G007	28.3	28.2	27.7	28.07	0.32	281.6	280.9	282.4	281.6	0.8
	G008	43.24	36.2	32.63	37.36	5.40	327.4	335.3	338.9	333.9	5.9
	G009										
	G014	28.4	29.6	30.1	29.37	0.87	262	260	257	259.7	2.5
	G015										
	G016										
	G017	33.7	34.95	34.67	34.44	0.66	270.7	275.8	270.8	272.4	2.9
	G018	30.8	31.3	31.3	31.13	0.29	267.3	264.6	269.1	267.0	2.3
	G019	39.96	39.1	34.12	37.73	3.15	335.01	320.5	317.84	324.5	9.2
	G020										
	G021	29.23	29.42		29.33	0.13	270	271		270.5	0.7
	G025	28.4	30.7	30.6	29.90	1.30	230	228	252	236.7	13.3
	G026	8.627	8.243	8.526	8.47	0.20	77.605	76.488	78.14	77.4	0.8
	G027	28.075	28.525	28.868	28.49	0.40	275.097	272.833	272.951	273.6	1.3
	G028	23.5	23.1	22.7	23.10	0.40	232	230	226	229.3	3.1
	G029	45.1	40.8	45.5	43.80	2.61	229.7	265.1	280.6	258.5	26.1
	G030	30.63	30.99	30.91	30.84	0.19	272.5	268.3	270	270.3	2.1
	G031	30.2	27.9	28.7	28.93	1.17	275	286	277	279.3	5.9
	G032	32	32	32	32	0	262	268	273	267.7	5.5
	G033	27.6	26.7	27.1	27.13	0.45	258	258	257	257.7	0.6
	G034	27.8	27.94	28.16	27.97	0.18	328.06	320.47	308.79	319.1	9.7
	G036										
	G037	28.124	28.817	29.315	28.75	0.60	259.109	265.178	263.784	262.7	3.2
	G038	48.1	60.9	42.1	50.37	9.60	290	295	306	297.0	8.2
	G039										
	G045										
	G046	27.5	27.6	27.5	27.53	0.06	261	279	255	265.0	12.5
	G047										
	G048										
Community Results		Consensus Mean			30.67		Consensus Mean			274.6	
		Consensus Standard Deviation			4.17		Consensus Standard Deviation			23.2	
		Maximum			50.37		Maximum			333.9	
		Minimum			8.47		Minimum			77.4	
		N			22		N			22	

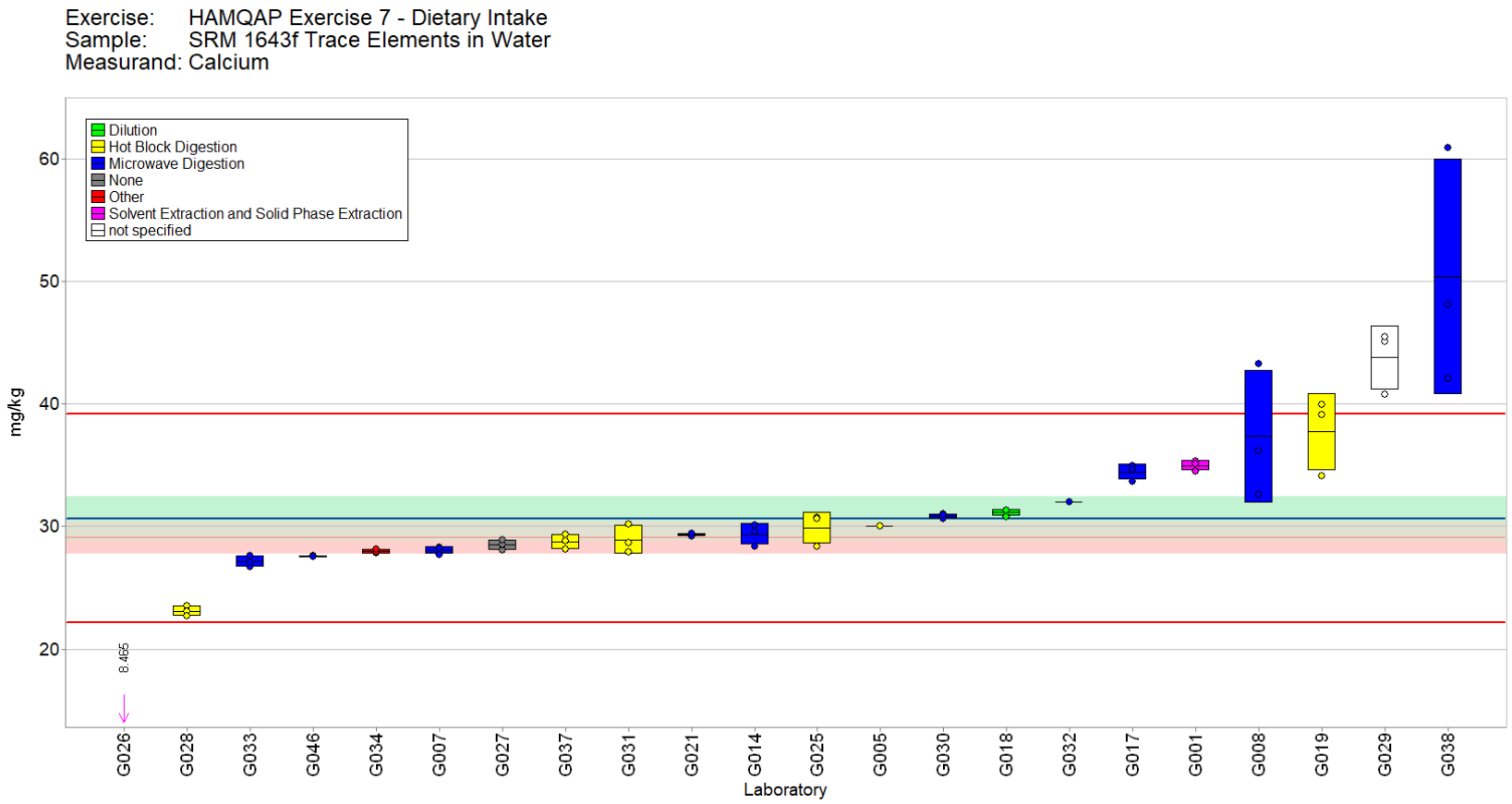

Fig. 1-1. Calcium in SRM 1643f Trace Elements in Water (data summary view – sample preparation method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the sample preparation method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The beige shaded region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

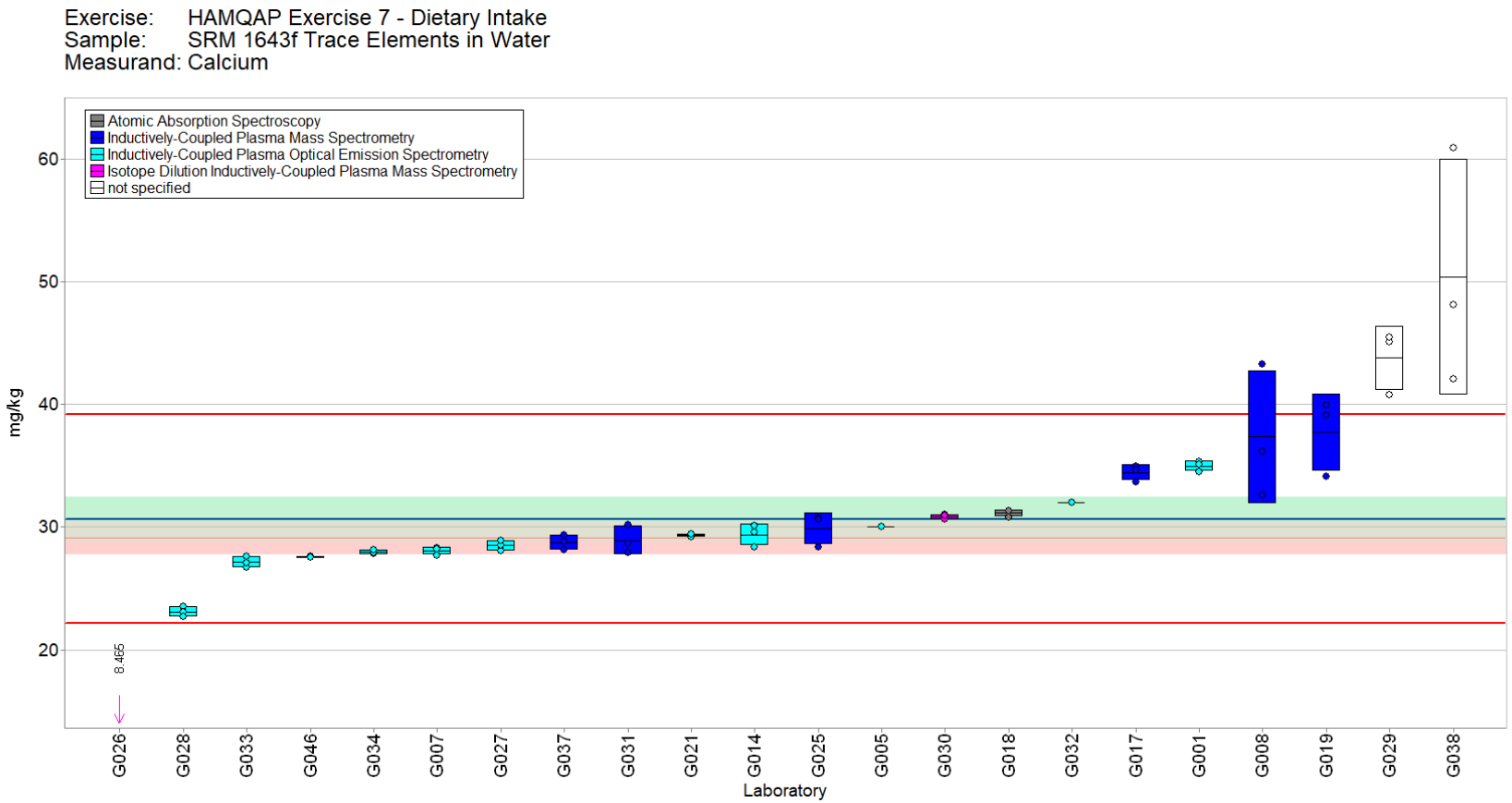

Fig. 1-2. Calcium in SRM 1643f Trace Elements in Water (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The beige shaded region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

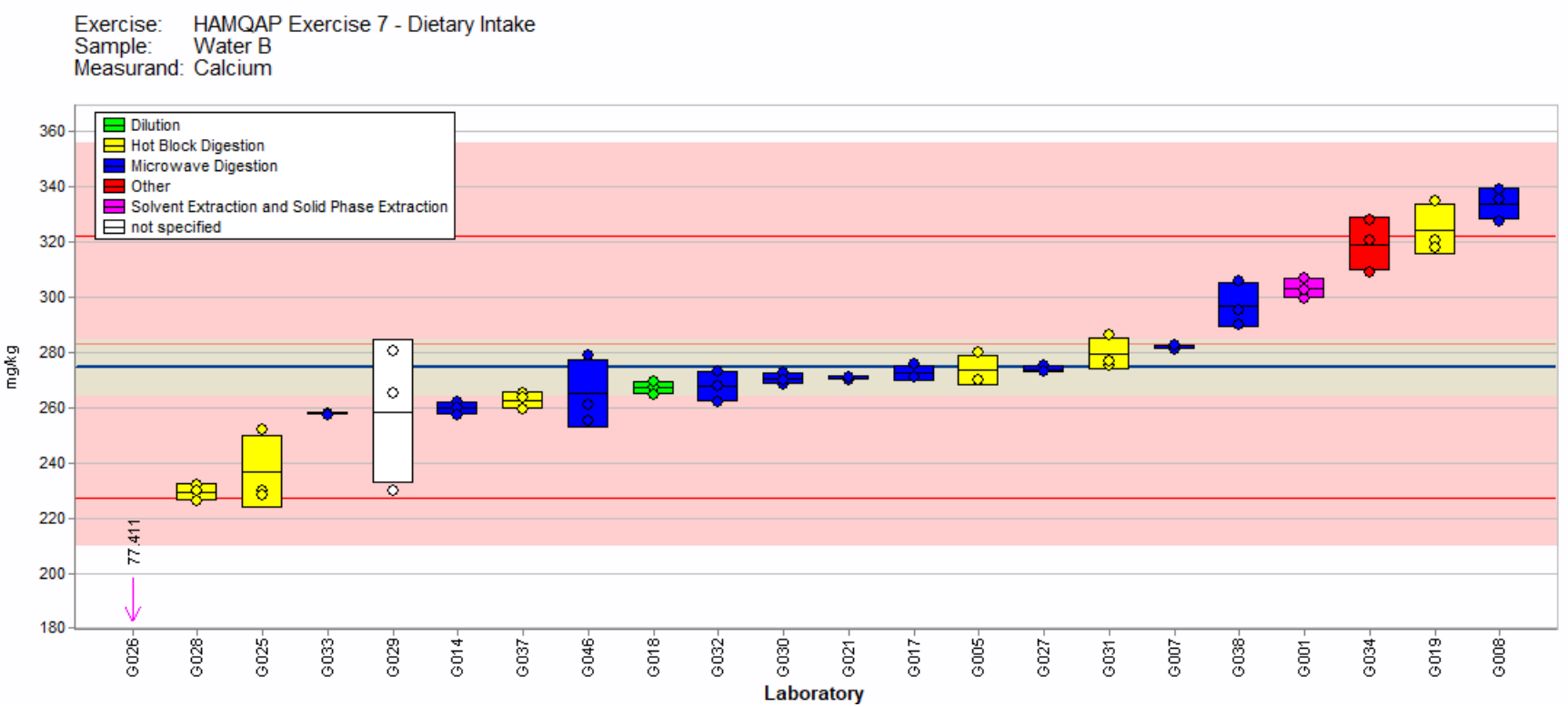

Fig. 1-3. Calcium in Water B (data summary view – sample preparation method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the sample preparation method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The beige shaded region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

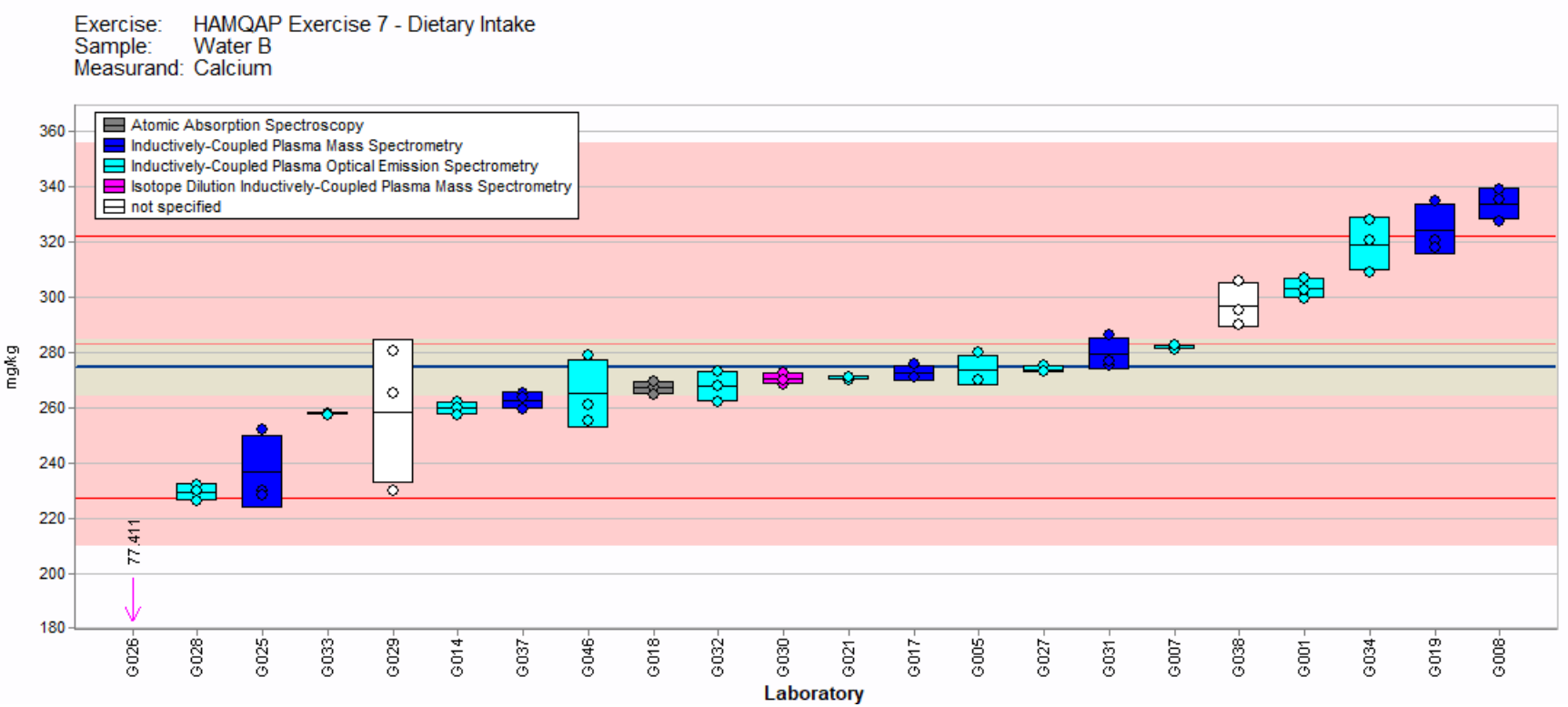

Fig. 1-4. Calcium in Water B (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The beige shaded region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

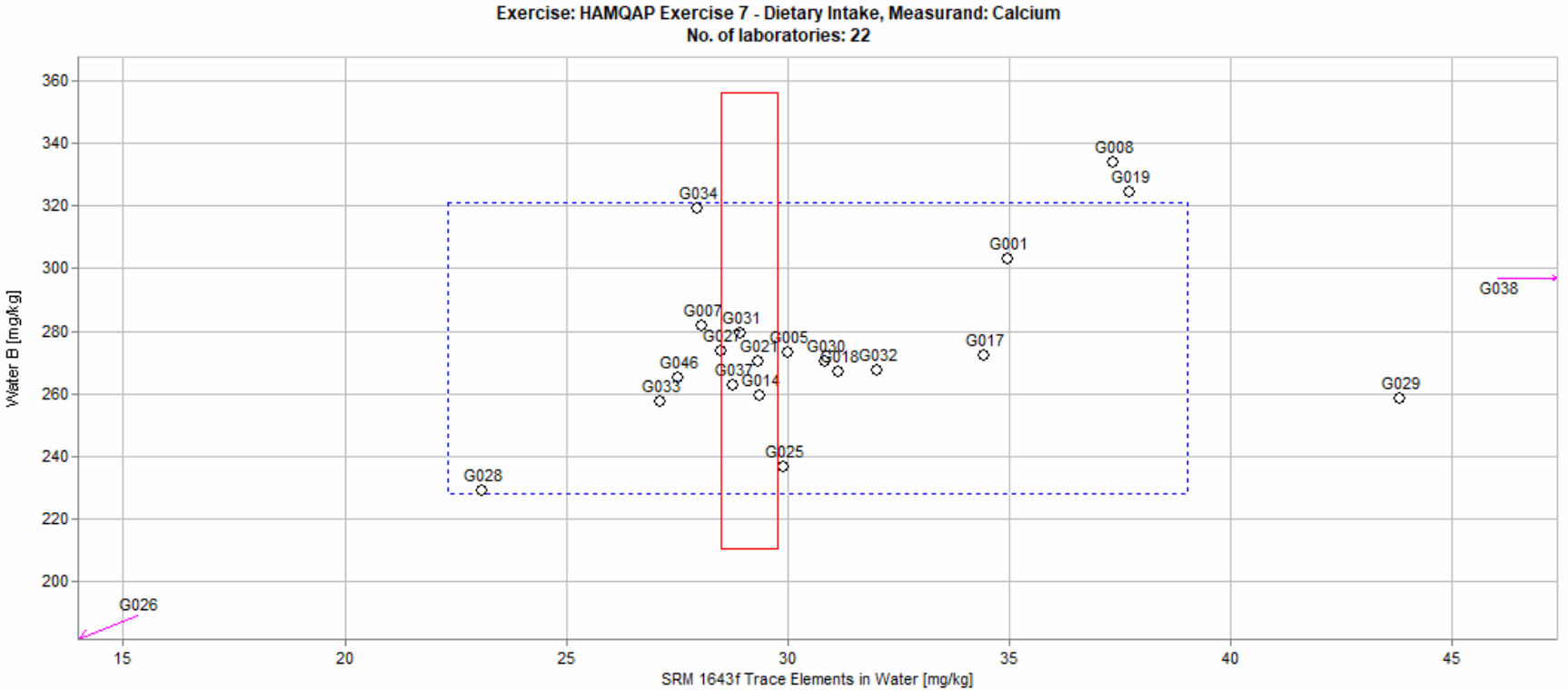

Fig. 1-5. Laboratory means for calcium in SRM 1643f Trace Elements in Water and Water B (sample/sample comparison view). In this view, the individual laboratory mean for one sample (SRM 1643f) is compared to the individual laboratory mean for a second sample (Water B). The solid red box represents the NIST range of tolerance for the two samples, SRM 1643f (x-axis) and Water B (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 1643f (x-axis) and Water B (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

Table 1-3. Data summary table for magnesium in water samples. Data highlighted in blue have been identified as outside the consensus range of tolerance resulted in an unacceptable Z'_{comm} score, $|Z'_{comm}| > 2$.

		Magnesium									
		SRM 1643f Trace Elements in Water (mg/kg)					Water B (mg/kg)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				7.38	0.06				91.0	8.8
	G001	8.1	8.1	8.1	8.1	0	87.2	87	88.1	87.4	0.6
	G002										
	G005	< 10	< 10	< 10			87	89	88	88.0	1.0
	G007	7.6	7.3	7.4	7.43	0.15	91.4	90.8	91.8	91.3	0.5
	G008	9.448	9.503	9.046	9.33	0.25	106.9	101.7	102.9	103.8	2.7
	G009										
	G014	7.59	7.43	7.6	7.54	0.10	91.3	89	88.1	89.5	1.7
	G015										
	G016										
	G017	9.28	9.55	9.5	9.44	0.14	93.5	95.3	93.5	94.1	1.0
	G018	9.4	9.5	9.3	9.40	0.10	52.9	54	53.8	53.6	0.6
	G019	10.27	10.29	8.72	9.76	0.90	615.95	594.12	585.7	598.6	15.6
	G020										
	G021	7.59	7.573		7.58	0.01	88.2	88.3		88.3	0.1
	G025	8.47	7.91	7.75	8.04	0.38	83.8	84.5	84.9	84.4	0.6
	G026	7.632	6.979	7.91	7.51	0.48	88.24	78.568	90.938	85.9	6.5
	G027	7.465	7.554	7.635	7.55	0.09	86.219	91.477	93.499	90.4	3.8
	G028	6.68	6.76	6.48	6.64	0.14	78.8	79.1	79.2	79.0	0.2
	G029	11.2	10.3	12.1	11.20	0.90	76.2	86.7	137.9	100.3	33.0
	G030	7.86	7.91	7.94	7.90	0.04	92	90.7	91.3	91.3	0.7
	G031	8.37	8.12	8.47	8.32	0.18	88.1	88.6	88.7	88.5	0.3
	G032	< 10	< 10	< 10			78	79	79	78.7	0.6
	G033	8.12	7.73	7.94	7.93	0.20	85.2	85.2	84.6	85.0	0.3
	G034	7.86	7.93	7.89	7.89	0.04	133.73	136.19	134.12	134.7	1.3
	G036										
	G037	7.221	7.411	7.502	7.38	0.14	86.425	89.177	88.655	88.1	1.5
	G038	50.9	8.71	7.36	22.32	24.76	82.9	89.1	88.6	86.9	3.4
	G039										
	G045										
	G046	7.5	5.48	7.25	6.74	1.10	82.5	85.1	82	83.2	1.7
	G047										
	G048										
Community Results		Consensus Mean			8.04		Consensus Mean			88.3	
		Consensus Standard Deviation			0.86		Consensus Standard Deviation			7.3	
		Maximum			22.32		Maximum			598.6	
		Minimum			6.64		Minimum			53.6	
		N			20		N			22	

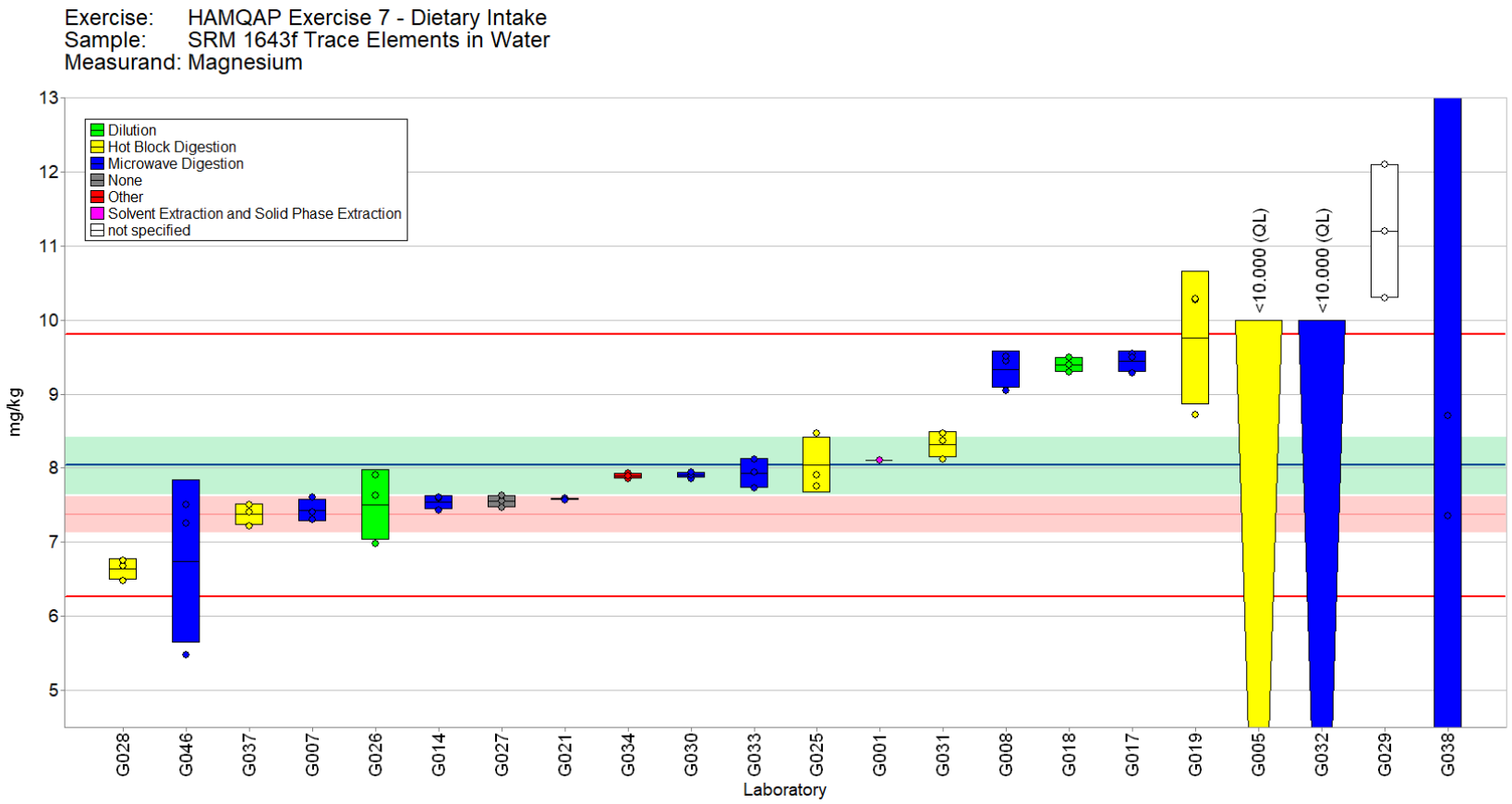

Fig. 1-6. Magnesium in SRM 1643f Trace Elements in Water (data summary view – sample preparation method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$.

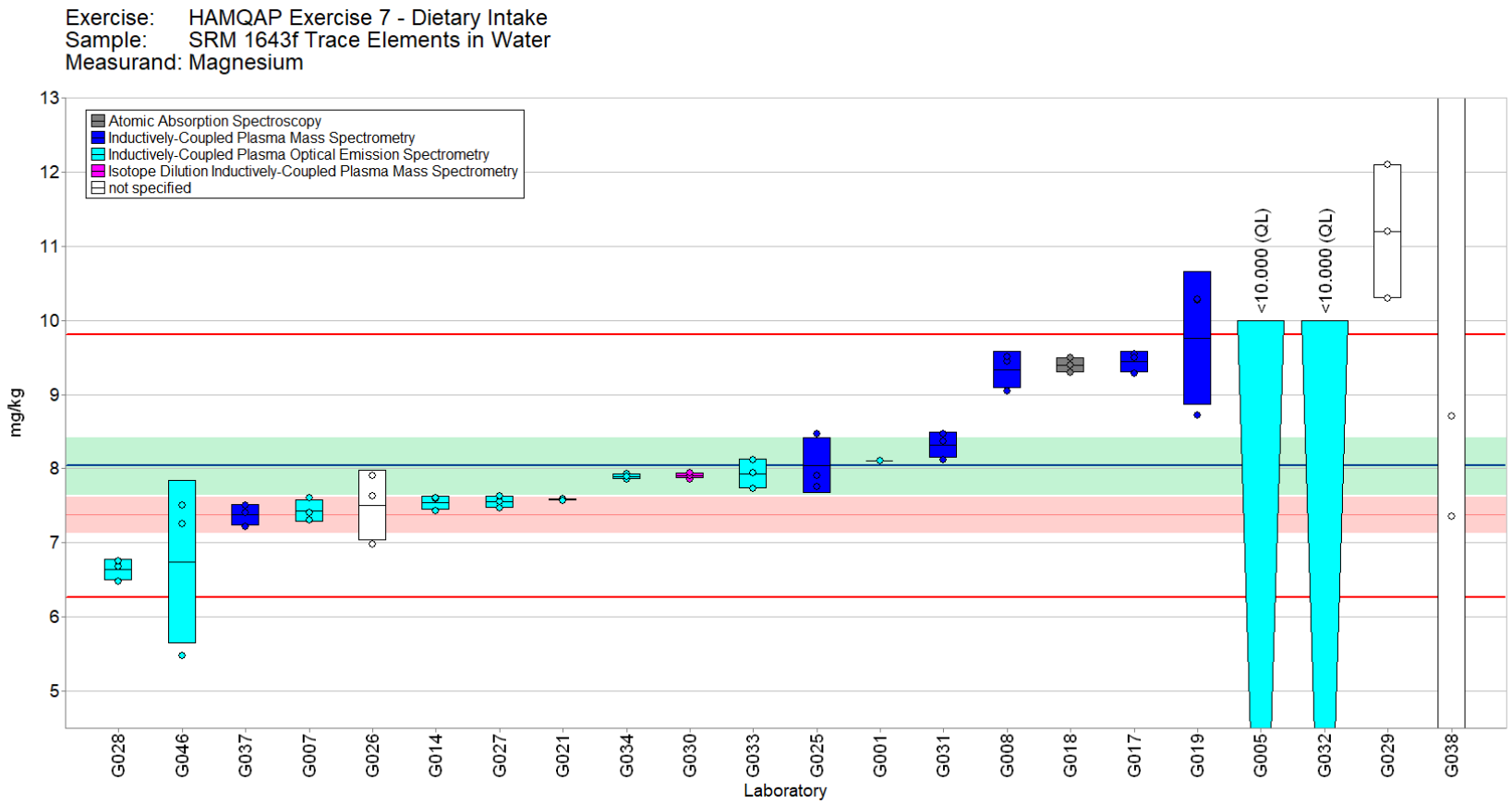

Fig. 1-7. Magnesium in SRM 1643f Trace Elements in Water (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$.

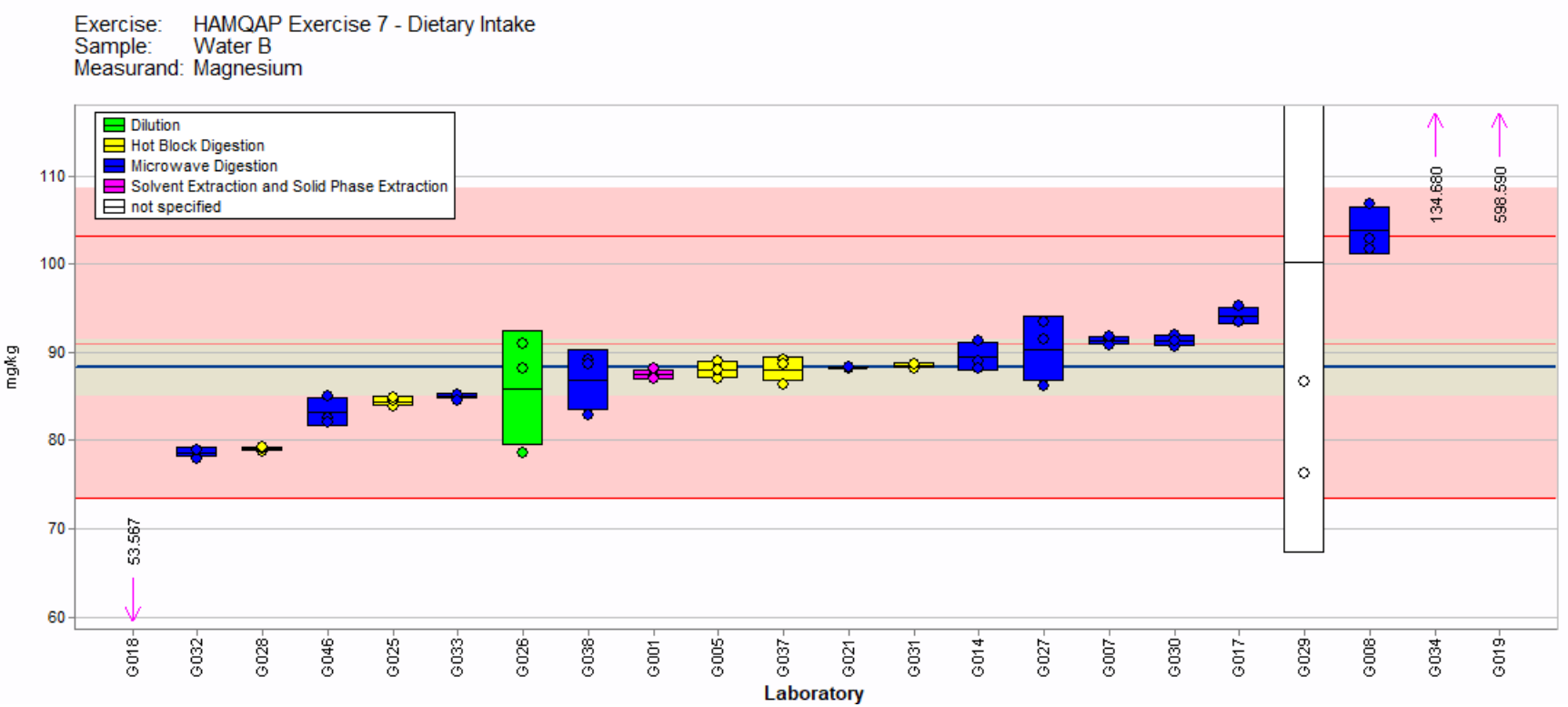

Fig. 1-8. Magnesium in Water B (data summary view – sample preparation method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the sample preparation method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The beige shaded region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

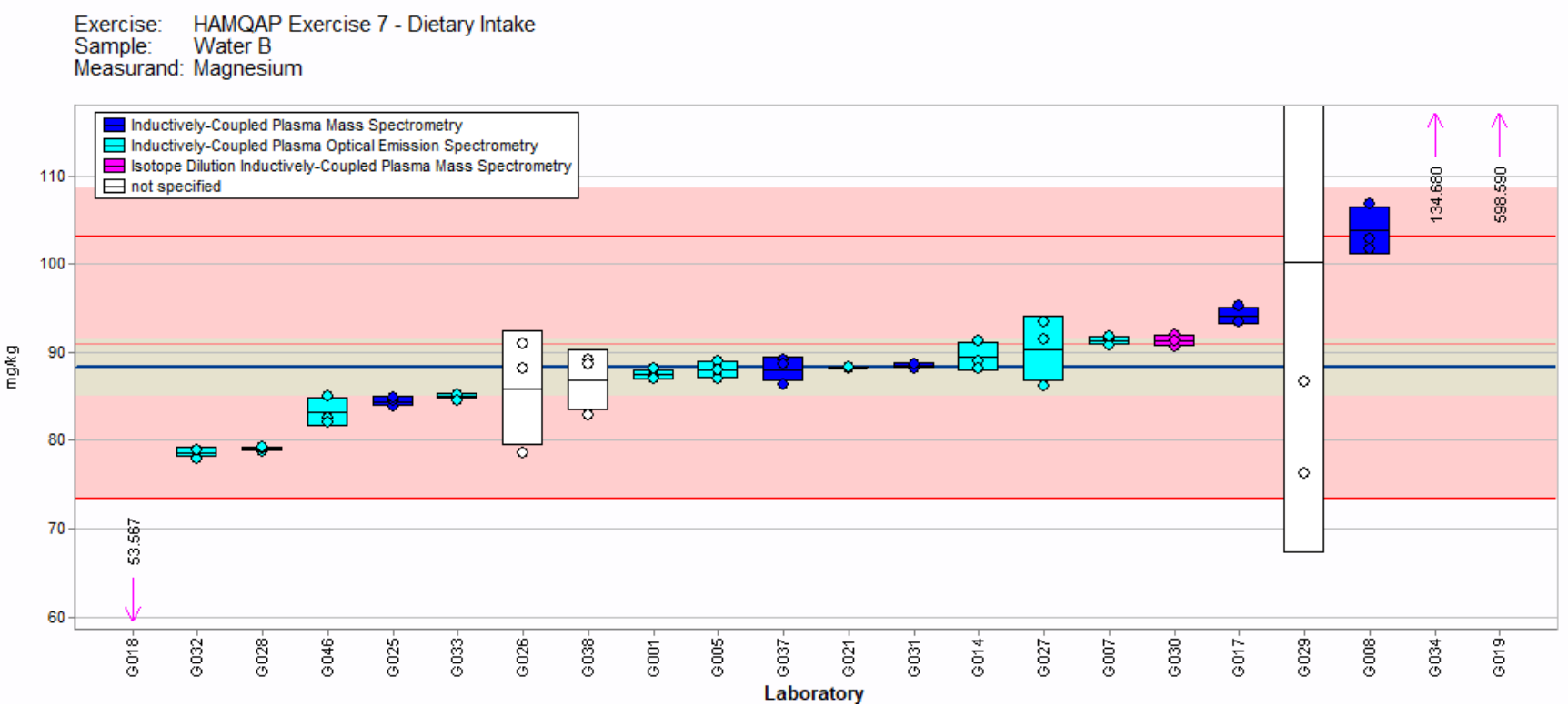

Fig. 1-9. Magnesium in Water B (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The beige shaded region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

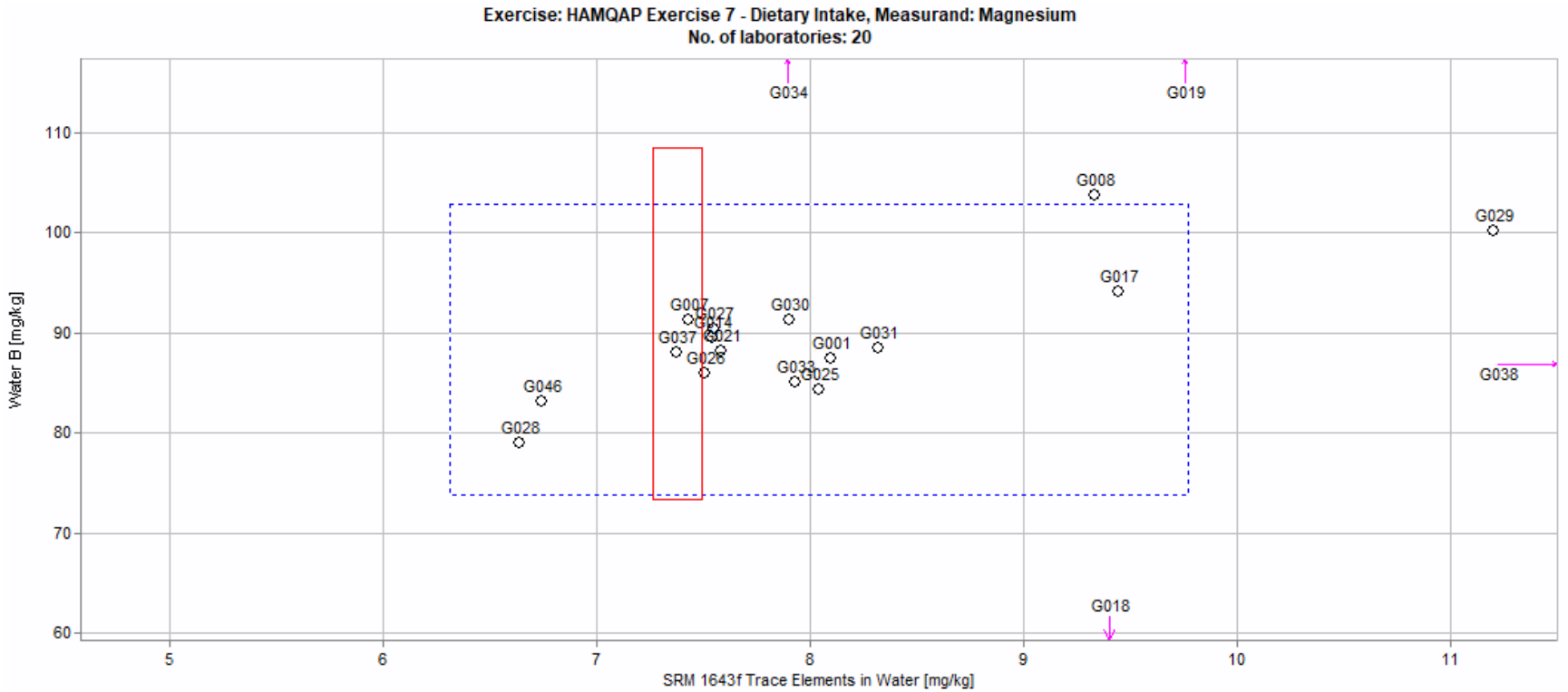

Fig. 1-10. Laboratory means for magnesium in SRM 1643f Trace Elements in Water and Water B (sample/sample comparison view). In this view, the individual laboratory mean for one sample (SRM 1643f) is compared to the individual laboratory mean for a second sample (Water B). The solid red box represents the NIST range of tolerance for the two samples, SRM 1643f (x-axis) and Water B (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 1643f (x-axis) and Water B (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

Table 1-4. Data summary table for zinc in water samples. Data highlighted in blue have been identified as outside the consensus tolerance limits and resulted in an unacceptable Z'_{comm} score, $|Z'_{comm}| > 2$.

		Zinc									
		SRM 1643f Trace Elements in Water (mg/kg)					Water B (mg/kg)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				0.074	0.002				5.12	1.72
	G001	0.2	0.2	0.2	0.2	0	5.6	5.7	7.8	6.37	1.24
	G002										
	G005	< 0.50	< 0.50	< 0.50			5.6	5.6	5.7	5.63	0.06
	G007	< 0.18	< 0.18	< 0.18			5.22	5.18	5.24	5.21	0.03
	G008	0.4172	1.563	0.5508	0.844	0.627	4.512	4.368	5.467	4.78	0.60
	G009										
	G010	0.334			0.334		4.47			4.47	
	G014	0.0995	0.0767	0.0768	0.084	0.013	5.55	5.58	5.53	5.55	0.03
	G015										
	G016										
	G017	0.09	0.09	0.09	0.09	0	5.83	6.1	5.96	5.96	0.14
	G018	0.064	0.0636	0.064	0.064	0.0002	5.8	5.9	5.9	5.87	0.06
	G019	0.59	0.15	0.51	0.417	0.234	6.66	6.33	6.44	6.48	0.17
	G020										
	G021	0.072	0.071		0.072	0.001	5.81	5.82		5.82	0.01
	G025	0.076	0.077	0.076	0.076	0.001	4.92	4.82	5.63	5.12	0.44
	G026	0.068	0.074	0.063	0.068	0.006	5.262	5.507	5.218	5.33	0.16
	G027	0.078	0.082	0.079	0.080	0.002	5.769	5.713	5.729	5.74	0.03
	G028	1.43	1.33	1.45	1.403	0.064	5.71	5.85	5.87	5.81	0.09
	G029	0.12	0.1	0.1	0.107	0.012	3.3	3.6	3.8	3.57	0.25
	G030	0.069	0.07	0.07	0.070	0.001	7.42	5.42	5.56	6.13	1.12
	G031	0.079	0.076	0.079	0.078	0.002	5.11	5.2	5.17	5.16	0.05
	G032	0.4	0.5	0.5	0.467	0.058	5.7	5.8	5.9	5.80	0.10
	G033	< 2.49	< 2.49	< 2.49			5.56	5.55	5.54	5.55	0.01
	G034						3.1	3.2	3.21	3.17	0.06
	G036										
	G037	0.088	0.088	0.088	0.088	0	5.688	5.762	5.837	5.76	0.07
	G038	0.17	0.16	0.12	0.150	0.026	3.4	3.92	3.95	3.76	0.31
	G039										
	G045										
	G046	< 5	< 5	< 5			< 5	< 5	6.34	6.34	
	G047										
	G048										
Community Results		Consensus Mean			0.090		Consensus Mean			5.53	
		Consensus Standard Deviation			0.039		Consensus Standard Deviation			0.64	
		Maximum			1.403		Maximum			6.48	
		Minimum			0.064		Minimum			3.17	
		N			17		N			21	

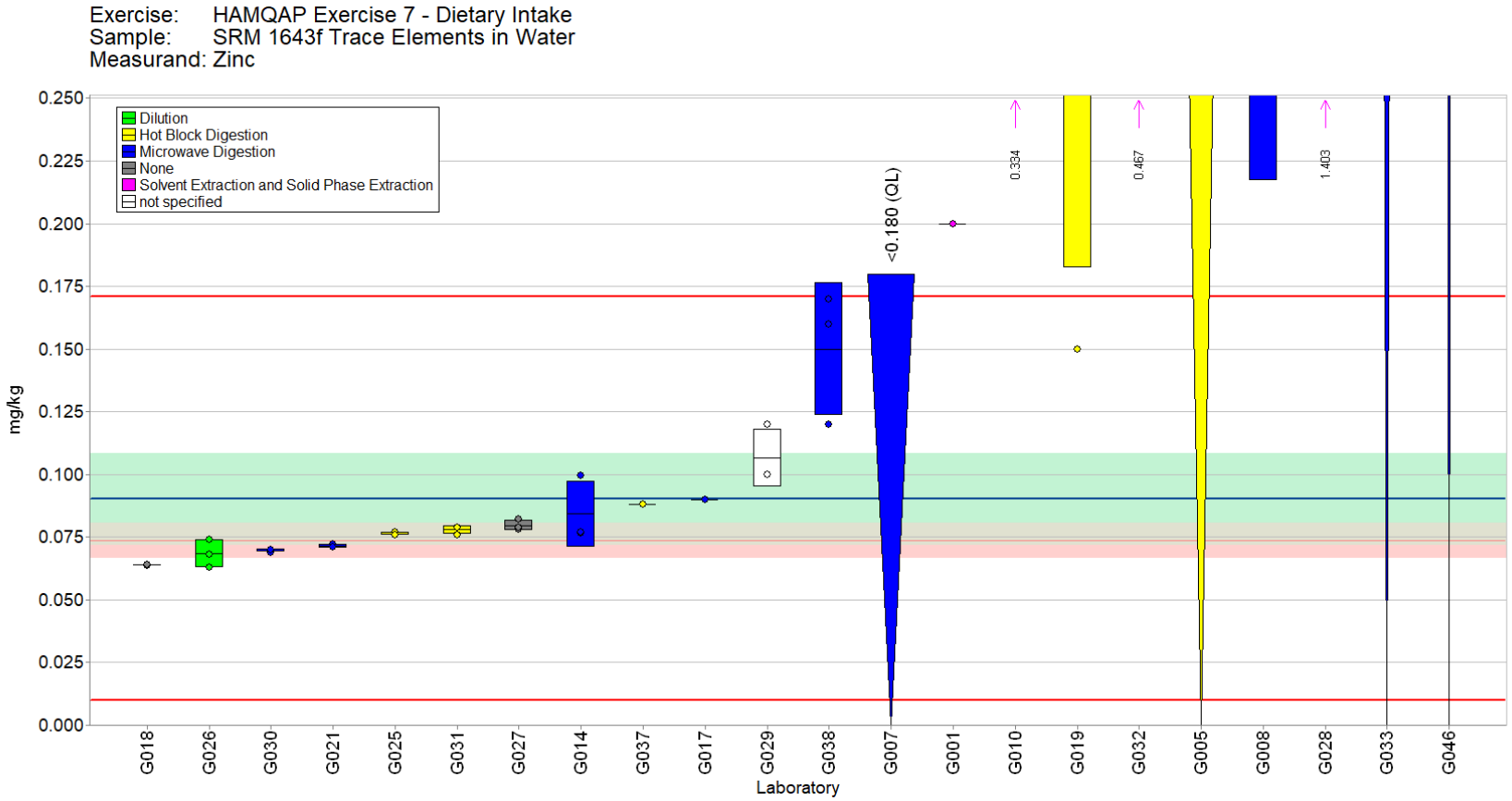

Fig. 1-11. Zinc in SRM 1643f Trace Elements in Water (data summary view – sample preparation method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The beige shaded region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

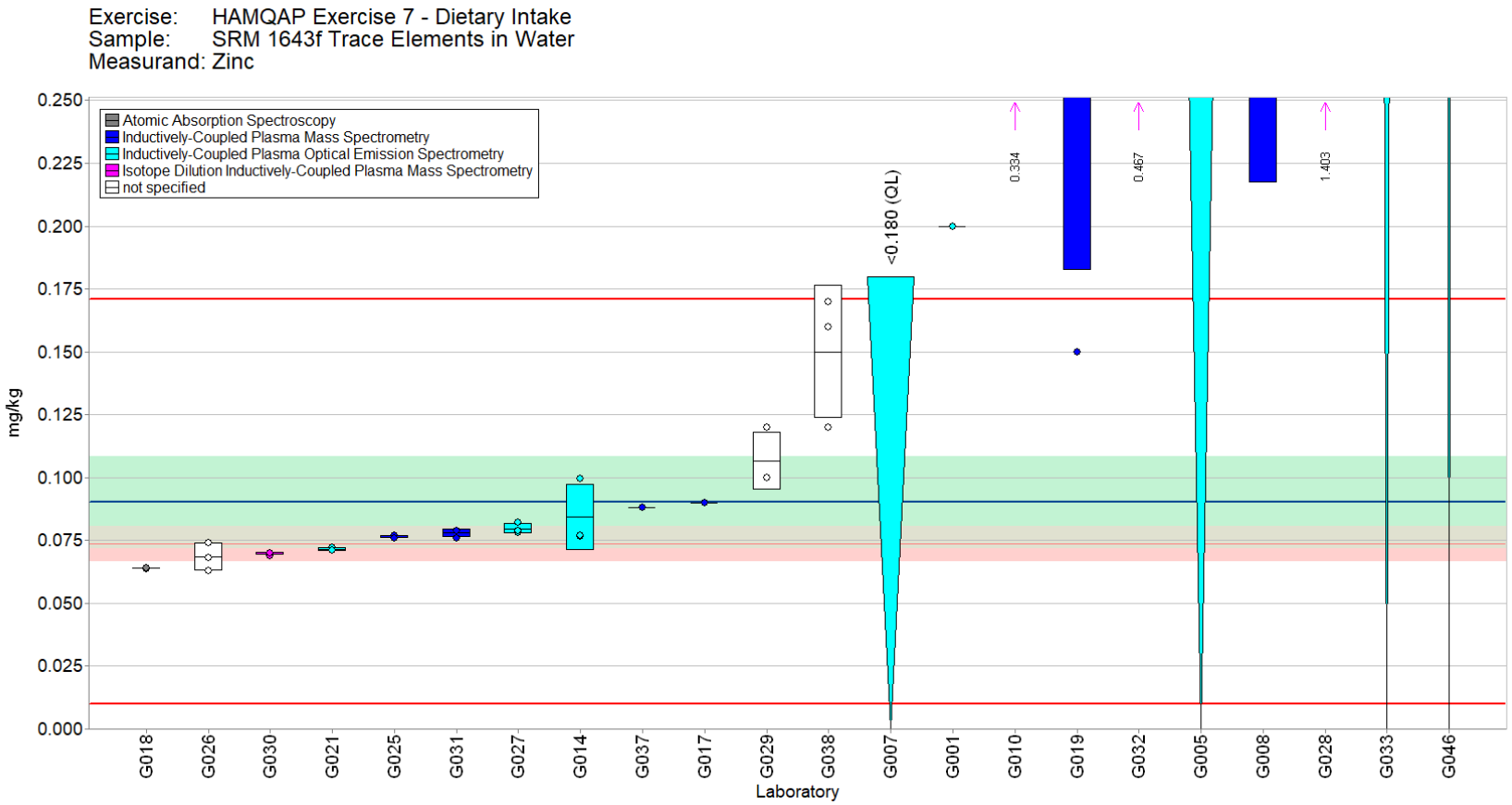

Fig. 1-12. Zinc in SRM 1643f Trace Elements in Water (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The beige shaded region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

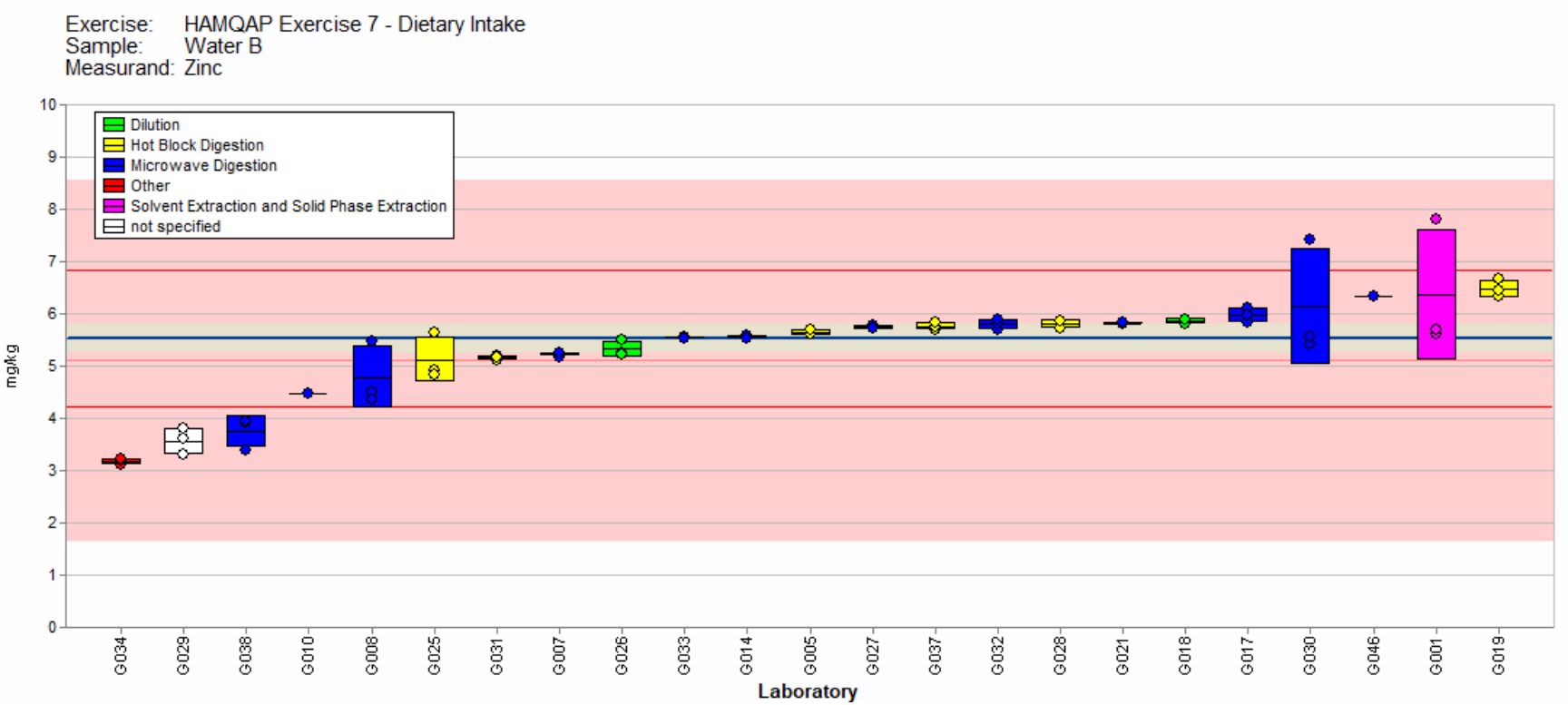

Fig. 1-13. Zinc in Water B (data summary view – sample preparation method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the sample preparation method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The beige shaded region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

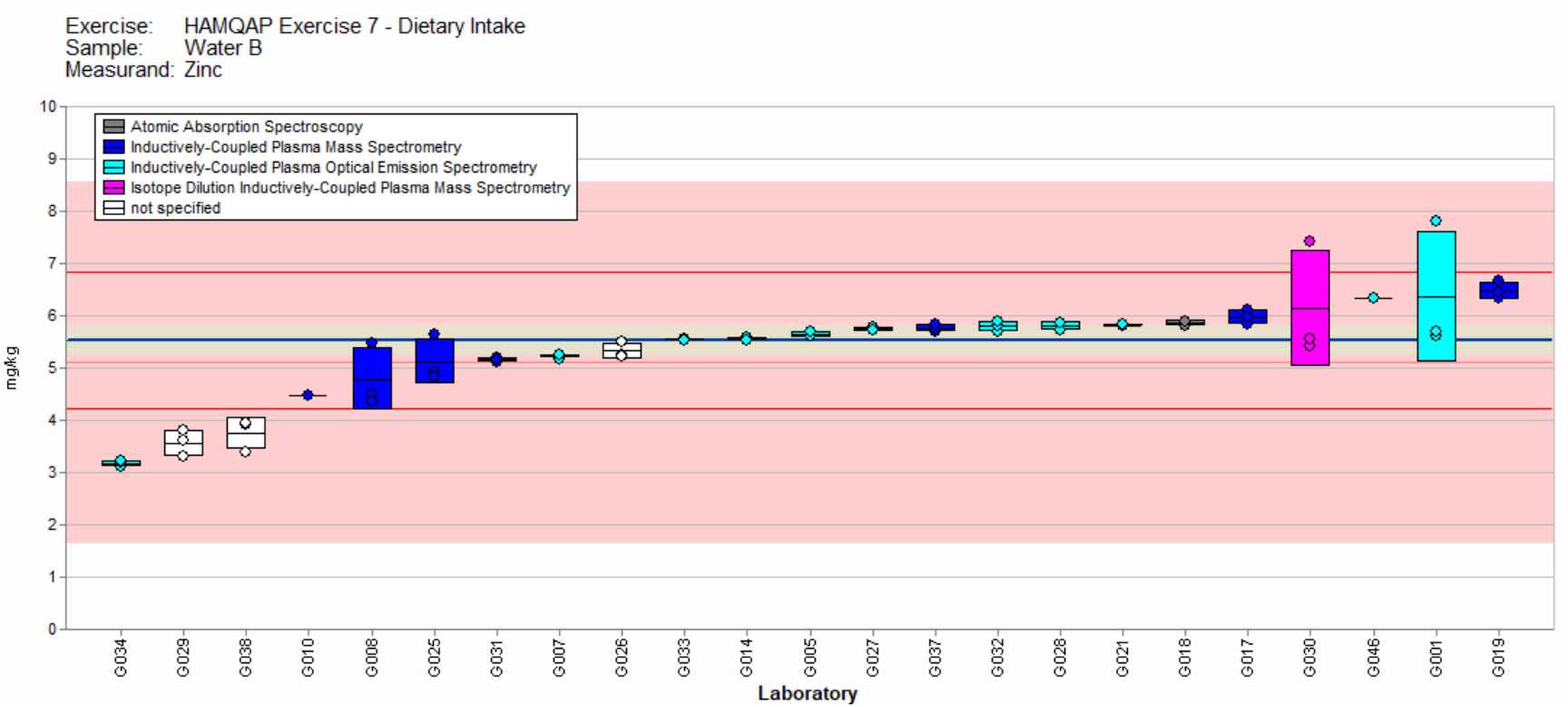

Fig. 1-14. Zinc in Water B (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The beige shaded region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

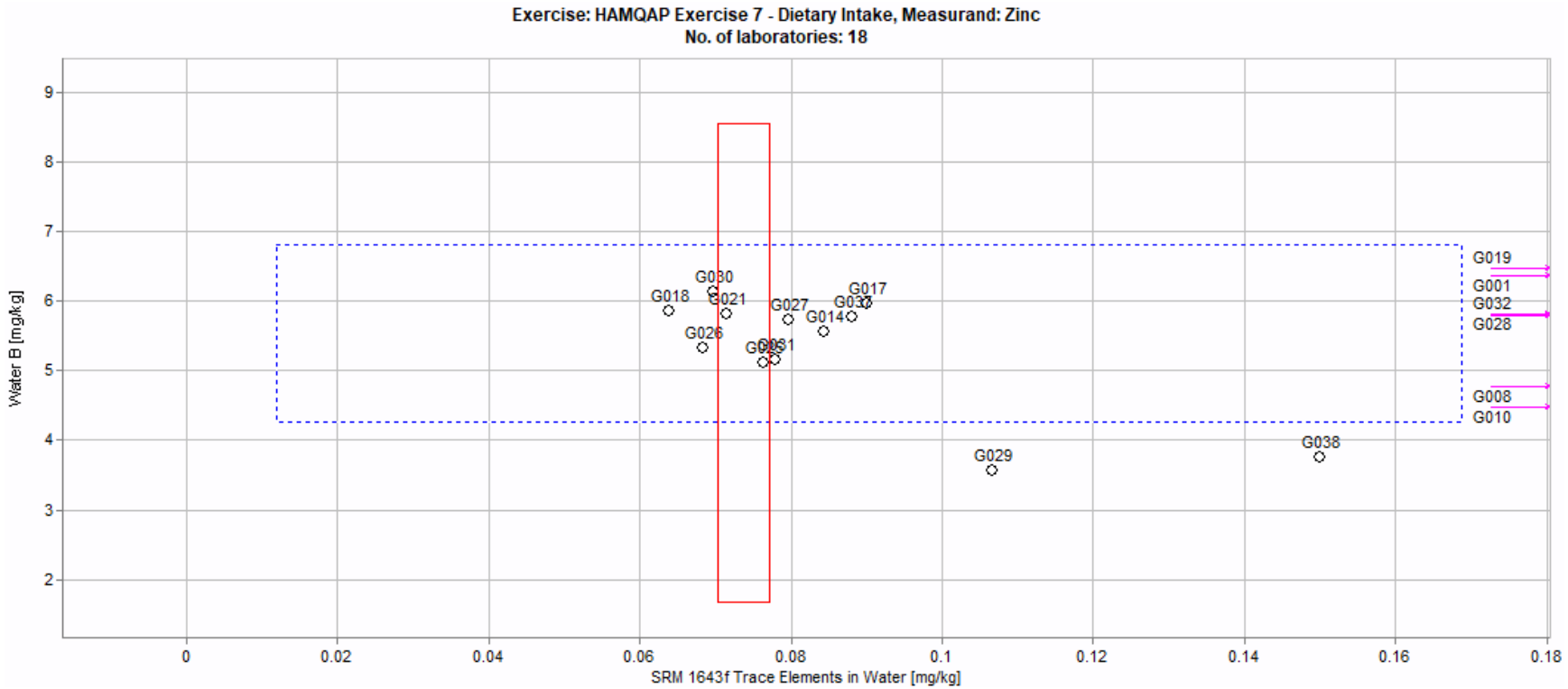

Fig. 1-15. Laboratory means for zinc in SRM 1643f Trace Elements in Water and Water B (sample/sample comparison view). In this view, the individual laboratory mean for one sample (SRM 1643f) is compared to the individual laboratory mean for a second sample (Water B). The solid red box represents the NIST range of tolerance for the two samples, SRM 1643f (x-axis) and Water B (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 1643f (x-axis) and Water B (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

2. Toxic Elements (Arsenic, Cadmium, Lead, and Mercury)

2.1. Study Overview

Plant uptake of toxic elements from the air, water, or soil may result in contamination of certain foods and dietary supplements. [4] Furthermore, the processing of plant materials may also increase the mass fractions of these toxic elements in consumer products. Consumption of such contaminated foods can cause illness, impairment or, at high doses and exposures, death. Testing of these environmental toxins in foods and supplements can help ensure product safety while testing biological samples such as serum can assess exposure and risk.

In this study, participants were provided with samples of black cohosh (*Actaea racemose*) extract and ashwagandha (*Withania somnifera*) extract as representations of dietary intake samples. Participants were asked to use in-house analytical methods to determine the mass fractions (ng/g) of arsenic (As), cadmium (Cd), lead (Pb), and mercury (Hg) in the dietary intake samples on an as-received basis (i.e., not moisture corrected).

2.2. Sample Information

Black Cohosh Extract. Participants were provided with three packets, each containing approximately 1 g of powdered black cohosh extract. Participants were asked to store the material at controlled room temperature, 20 °C to 25 °C, in the original unopened packets, to prepare one sample, and report one value from each packet provided. Before use, participants were instructed to mix the contents of each packet thoroughly, allow contents to settle for one minute prior to opening to minimize the loss of fine particles, and to use a sample size of at least 0.5 g. Approximate analyte levels were not disclosed to participants prior to the study and target values for As, Cd, Pb, and Hg in this material were not available at the time of this report.

Ashwagandha Extract. Participants were provided with three packets, each containing approximately 1.5 g of powdered ashwagandha extract. Participants were asked to store the material at controlled room temperature, 20 °C to 25 °C, in the original unopened packets, to prepare one sample, and report one value from each packet provided. Before use, participants were instructed to mix the contents of each packet thoroughly, allow contents to settle for one minute prior to opening to minimize the loss of fine particles, and to use a sample size of at least 0.5 g. Approximate analyte levels were not disclosed to participants prior to the study. The target values for As, Cd, and Pb in ashwagandha extract were determined at NIST using ICP-MS. The values and standard deviations are provided in the table below on an as-received basis. A target value for Hg was not available in this material at the time of the report.

Analyte	Target Mass Fractions in Ashwagandha Extract (ng/g)		
Arsenic (As)	32.07	±	4.33
Cadmium (Cd)	7.46	±	0.49
Lead (Pb)	9.61	±	0.38

2.3. Study Results

The enrollment and reporting statistics for the toxic element studies are described in the table below. Reported values may include non-quantitative results (zero or below LOQ) that are only included in the participation statistics.

Analyte	Number of Laboratories Requesting Samples	Number of Laboratories Reporting Results (Percent Participation)	
		Black Cohosh Extract	Ashwagandha Extract
Arsenic (As)	38	27 (71 %)	27 (71 %)
Cadmium (Cd)	38	26 (68 %)	27 (71 %)
Lead (Pb)	38	27 (71 %)	27 (71 %)
Mercury (Hg)	37	25 (68 %)	25 (68 %)

The between-laboratory variabilities ranged from 22 % to 58 % for As, Cd, and Pb in both materials. The variabilities were higher ≥ 70 % for Hg in both materials.

Analyte	Between Laboratory Variability (% RSD)	
	Black Cohosh Extract	Ashwagandha Extract
Arsenic (As)	22 %	47 %
Cadmium (Cd)	26 %	24 %
Lead (Pb)	23 %	58 %
Mercury (Hg)	> 100 %	70 %

The within-laboratory variabilities ranged from 0.2 % to 35 % for As and Cd in both materials. The variabilities were 2 % to > 100 % for Pb and Hg in both materials.

Analyte	Within-Laboratory Variability Ranges (% RSD)	
	Black Cohosh Extract	Ashwagandha Extract
Arsenic (As)	0.8 % to 19 %	1 % to 17 %
Cadmium (Cd)	0.6 % to 35 %	0.2 % to 33 %
Lead (Pb)	0.4 % to 82 %	1 % to 67 %
Mercury (Hg)	2 % to > 100 %	20 % to > 100 %

Most laboratories reported using microwave digestion as their sample preparation method for both ashwagandha extract and the black cohosh extract. Other reported sample preparation methods included hot block digestion, solvent or solid phase extraction, and thermal decomposition.

	Percentage of Laboratories Reporting (Averaged for both sample types)			
Sample Preparation Method	As	Cd	Pb	Hg
Microwave Digestion	63 %	58 %	61 %	56 %
Hot Block Digestion	22 %	23 %	22 %	20 %
Solvent Extraction and Solid Phase Extraction	4 %	4 %	4 %	4 %
Thermal Decomposition	-	-	-	4 %
Other/None Reported	11 %	15 %	13 %	16 %

Most laboratories reported using ICP-MS as the analytical method for both ashwagandha extract and the black cohosh extract. Other reported analytical methods included ID ICP-MS, ICP-OES, IC-MS, AAS, and LC-MS/MS.

	Percentage of Laboratories Reporting (Averaged for both sample types)			
Analytical Method	As	Cd	Pb	Hg
ICP-MS	65 %	66 %	67 %	68 %
ICP-MS (KED Mode)	-	4 %	4 %	4 %
ID ICP-MS	11 %	8 %	9 %	8 %
ICP-OES	7 %	8 %	7 %	4 %
IC-MS	4 %	-	-	-
AAS	4 %	8 %	6 %	8 %
LC-MS/MS	2 %	-	-	-
Other/None Reported	7 %	8 %	7 %	8 %

The accuracy of results varied by element in the ashwagandha extract as described in the table below. NIST ranges were not available for Hg in the ashwagandha extract or for any element in the black cohosh extract.

	Relative to NIST Range of Tolerance for Ashwagandha Extract		
Position of	As	Cd	Pb
Consensus Mean	Within	Below	Above
Consensus Range	Centered	Below	Overlapping upper edge
Corresponding Figures	2-1, 2-2	2-6, 2-7	2-11, 2-12

2.4. Toxic Elements Technical Recommendations

The following observations and recommendations are based on results obtained from the participants in this study. Additional overall technical recommendations can be found on page 6.

Arsenic

- Most laboratories reported using microwave digestion as their sample preparation method prior to determination of As. The high temperatures of a microwave digestion system should ensure complete digestion of the materials prior to analysis.
- Arsenic is volatile and can be lost during sample preparation.
  - A vigorous microwave digestion should convert all volatile organoarsenic species in solution to arsenic acid (AsV). At this point, subsequent heating of the solution will not result in loss of arsenic.
  - Microwave digestion vessels should be opened slowly and carefully to ensure that no arsenic is lost due to inadvertent venting.
  - Open vessel digestions should be performed slowly and carefully to ensure that no arsenic is lost. Arsenic is easily lost during open beaker digestions.
- **Figure 2-5** shows a slight upward trend in the data, which may indicate sample preparation issues or calibration issues. Failure to eliminate the organic constituents due to incomplete sample digestion may produce interferences that cause signal enhancement or suppression, thereby introducing measurement bias in the sample matrix. An incomplete sample digestion can cause increased within-laboratory variability.
- Most laboratories reported using ICP-MS as their analytical method for determination of As in these samples.
  - Collision cell technology with He and/or H_2 can be used to minimize $^{40}Ar^{35}Cl^{+}$ isobaric interference at arsenic mass 75 u. Reaction gas O_2 can also be used to shift the analytical mass to 91 u by measuring arsenic analyte as $^{75}As^{16}O^{+}$ thereby avoiding the $^{40}Ar^{35}Cl^{+}$ isobaric interference at 75 u.
  - Some laboratories erroneously reported using ID ICP-MS as the analytical method. ID ICP-MS cannot be used for monoisotopic elements such as As.

Cadmium

- Most laboratories used microwave digestion as their sample preparation method prior to determination of Cd.
  - The boiling point of Cd is high, therefore volatile loss of Cd should not be a concern during sample preparation.
  - Most laboratories reported values below the target for Cd in the ashwagandha material or below their LOQ. Difficulty with extraction of Cd from the ashwagandha matrix may be one cause of low results.

- Most laboratories reported using ICP-MS as their analytical method for determination of Cd in these samples.
    - Isobaric spectral interferences such as $^{95}Mo^{16}O^{+}$ and $^{97}Mo^{16}O^{+}$ can affect the accuracy of Cd determination at 111 u and 113 u by ICP-MS.
        - High concentrations of certain elements (e.g., Mo, Sn, Zr) are known to cause interferences in the analysis of Cd 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 analytes of interest from potential interferences prior to ICP-MS can improve accuracy, albeit time-consuming.
        - Using collision cell technology with He and/or H_2 can minimize molecular ion interferences.
- Most laboratories reported values below the target for Cd in the ashwagandha material or below their LOQ. The low mass fraction of Cd present in the material may be one cause of measurement challenges.

Lead

- The overall data shows good performance for Pb, without trends indicating overall matrix or calibration challenges.
- Several laboratories were outside the consensus range of tolerance for one or both materials and may have had calibration problems or difficulty with the sample matrices.
    - Lead is easily digested using routine methods, and volatile loss of lead is not a concern.
    - Digestion of samples with HCl may form insoluble $PbCl_2$ precipitates.
    - Precipitation would be more problematic for the 10-fold greater level of Pb in the black cohosh extract than the ashwaganda extract. Precipitation of $PbCl_2$ may have caused a low bias in the black cohosh results if the sample digestion was not conducted consistently between materials.
    - For Pb analysis, digestion with high purity HNO_3 is recommended

Mercury

- Only 25 % of the reporting laboratories in the Hg study provided quantitative results.
- Mercury is volatile and can be lost during sample preparation. Use of microwave digestion is recommended to ensure a complete digestion at high temperature with closed vessels to prevent loss of volatile Hg.
- Blank and background levels for Hg measurements may be large, limiting low level detection and quantitation. An appropriate number of procedural blanks (e.g., equal to the number of samples) should be analyzed to determine an accurate LOQ.

- Low mass fractions of Hg are not stable in solution over time.
  - Samples should be prepared as near as possible to the time of analysis.
  - Addition of HCl (3 % to 5 %) to dilute HNO_3 may increase stability.
  - Acidification of sample solutions will help prevent loss of Hg by adsorption.
  - Addition of dichromate will help prevent loss of Hg through volatilization.
- Methods for determination of Hg using ICP-MS often have low sensitivity and retention of Hg within the sample introduction system requires long washout times. Using cold vapor Hg generation increases the sensitivity of ICP-MS and allows lower levels of Hg to be measured through more efficient transfer of the sample to the ICP.
- Carryover of Hg between samples is common and can lead high variability. Adequate washout time is needed between each sample measurement, and the use of dilute HCl or gold in the rinse solution may decrease the length of the washout time needed.
- Use of direct combustion AAS or direct mercury analyzers for determination of Hg allows low detection limits and does not require sample preparation, which increases sample throughput.

Table 2-1. Individualized data table (NIST) for toxic elements in black cohosh and ashwagandha extracts.

Exercise 7 - Toxic Elements

		Lab Code:	**NIST**	**1. Your Results**				**2. Community Results**			**3. Target**	
Analyte	Sample	Units		x_i	s_i	Z'_{comm}	Z_{NIST}	N	x*	s*	x_{NIST}	*U*
Arsenic	Black Cohosh Extract	ng/g						27	96.9	21.3		
Arsenic	Ashwagandha Extract	ng/g		32.1	4.3			27	31.6	14.7	32.1	4.3
Cadmium	Black Cohosh Extract	ng/g						26	14.0	3.6		
Cadmium	Ashwagandha Extract	ng/g		7.46	0.49			27	5.0	1.2	7.46	0.49
Lead	Black Cohosh Extract	ng/g						27	278	64		
Lead	Ashwagandha Extract	ng/g		9.61	0.38			27	11.7	6.7	9.61	0.38
Mercury	Black Cohosh Extract	ng/g						25	4.2	4.5		
Mercury	Ashwagandha Extract	ng/g						25	5.9	4.1		

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

Table 2-2. Data summary table for arsenic in black cohosh and ashwagandha extracts. Data highlighted in blue have been identified as outside the consensus tolerance limits and resulted in an unacceptable Z'_{comm} score, $|Z'_{comm}| > 2$.

		Arsenic									
		Black Cohosh Extract (ng/g)					Ashwagandha Extract (ng/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target									32.07	4.33
	G001	110	120	110	113.3	5.8	< 70	< 70	< 70		
	G002										
	G005	99.9	95.1	90.6	95.2	4.7	30.2	31	30.7	30.63	0.40
	G008	164.5	236.3	233.7	211.5	40.7	112.2	136.7	159.8	136.2	23.8
	G009										
	G010	78			78		32			32	
	G011	44.701	45.779	44.294	44.9	0.8	< 6.945	< 6.945	< 6.945		
	G012	100	100	100	100	0	30	30	30	30	0
	G013	87.9	96.8	93.6	92.8	4.5	31.5	30.1	29.4	30.33	1.07
	G014	82	86	88	85.3	3.1	26	30	31	29.00	2.65
	G015										
	G016	124.55	123.39	140.26	129.4	9.4	48.12	52.92	57.14	52.73	4.51
	G017	100	90	90	93.3	5.8	< 50	< 50	< 50		
	G019	126.96	123.59	124.18	124.9	1.8	122.22	126.62	120.54	123.1	3.1
	G020	90	98	94	94.0	4.0	30	35	42	35.67	6.03
	G021	93.6	92.5	92.3	92.8	0.7	29.2	30.6	30.8	30.20	0.87
	G023										
	G024										
	G025	105	105	105	105	0	36	37	35	36	1.0
	G026										
	G027	80	83	82	81.7	1.5	30	31	29	30	1.0
	G028	93	90	84	89.0	4.6	117	138	145	133.3	14.6
	G029	142	130	120	130.7	11.0	60	60	52	57.3	4.6
	G030	51	62	75	62.7	12.0	< 4	11	< 4	11.00	
	G031	94.4	93.9	90.6	93.0	2.1	30.3	31.2	31.6	31.03	0.67
	G032	100	100	100	100	0	< 50	< 50	< 50		
	G033	90	90	90	90	0	< 40	50	< 40	50	
	G034	104.2	102.5	104.4	103.7	1.0	32.8	34.1	31.2	32.70	1.45
	G036										
	G037	104	102	103	103.0	1.0	< 100	< 100	< 100		
	G038	0.078	0.084	0.08	0.081	0.003	0.024	0.024	0.021	0.02	0.002
	G039	0.088	0.088	0.093	0.090	0.003	0.033	0.03	0.027	0.03	0.003
	G043	112	108	108	109.3	2.3	42	42	43	42.3	0.6
	G045										
	G046	99.2	92.8	88.4	93.5	5.4	23.8	25.9	23.1	24.27	1.46
	G047										
	G048										
Community Results		Consensus Mean			96.89		Consensus Mean			31.61	
		Consensus Standard Deviation			21.30		Consensus Standard Deviation			14.74	
		Maximum			211.50		Maximum			136.23	
		Minimum			0.08		Minimum			0.02	
		N			26		N			19	

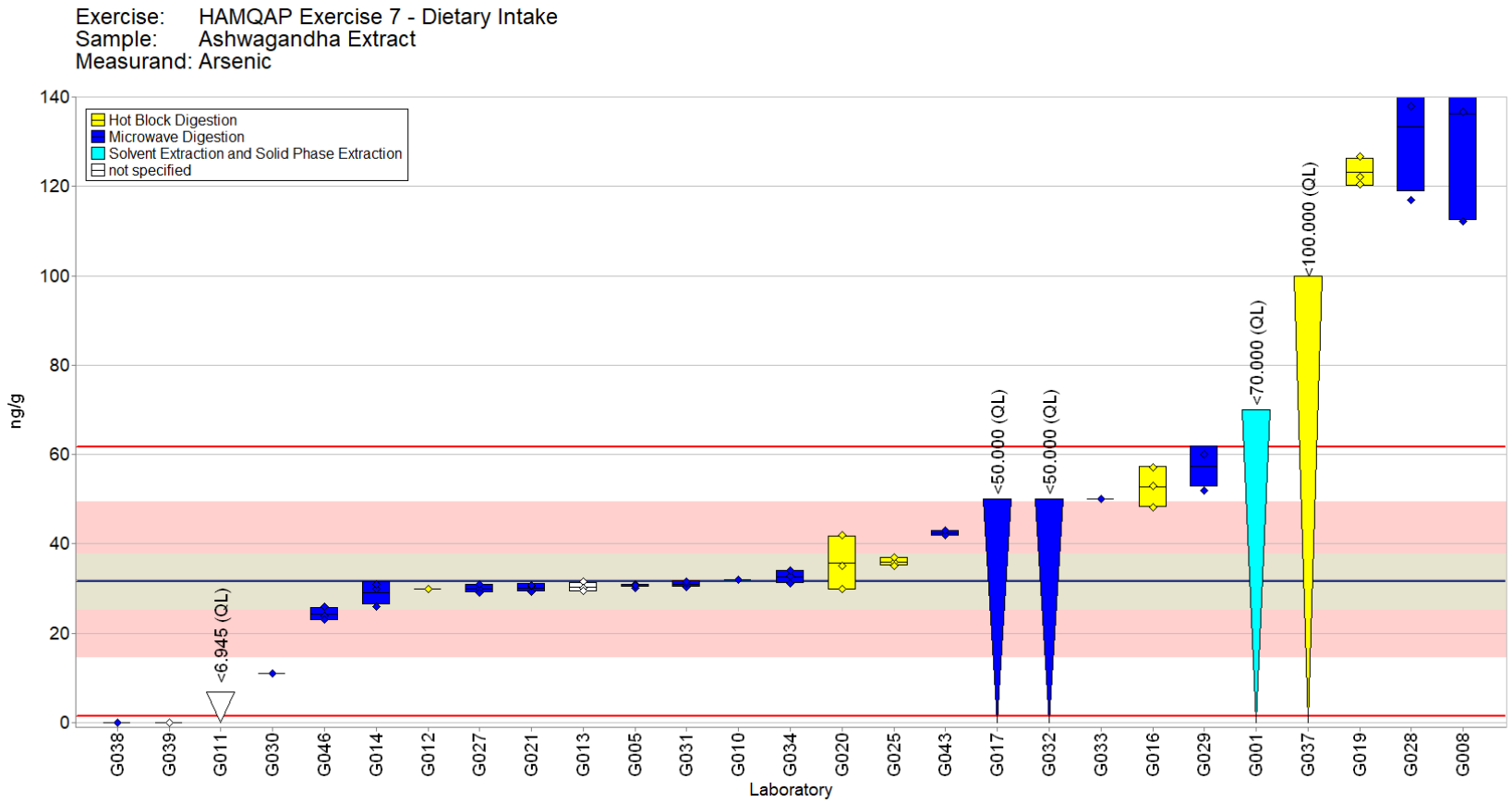

Fig. 2-1. Arsenic in ashwagandha extract (data summary view – sample preparation method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The beige shaded region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

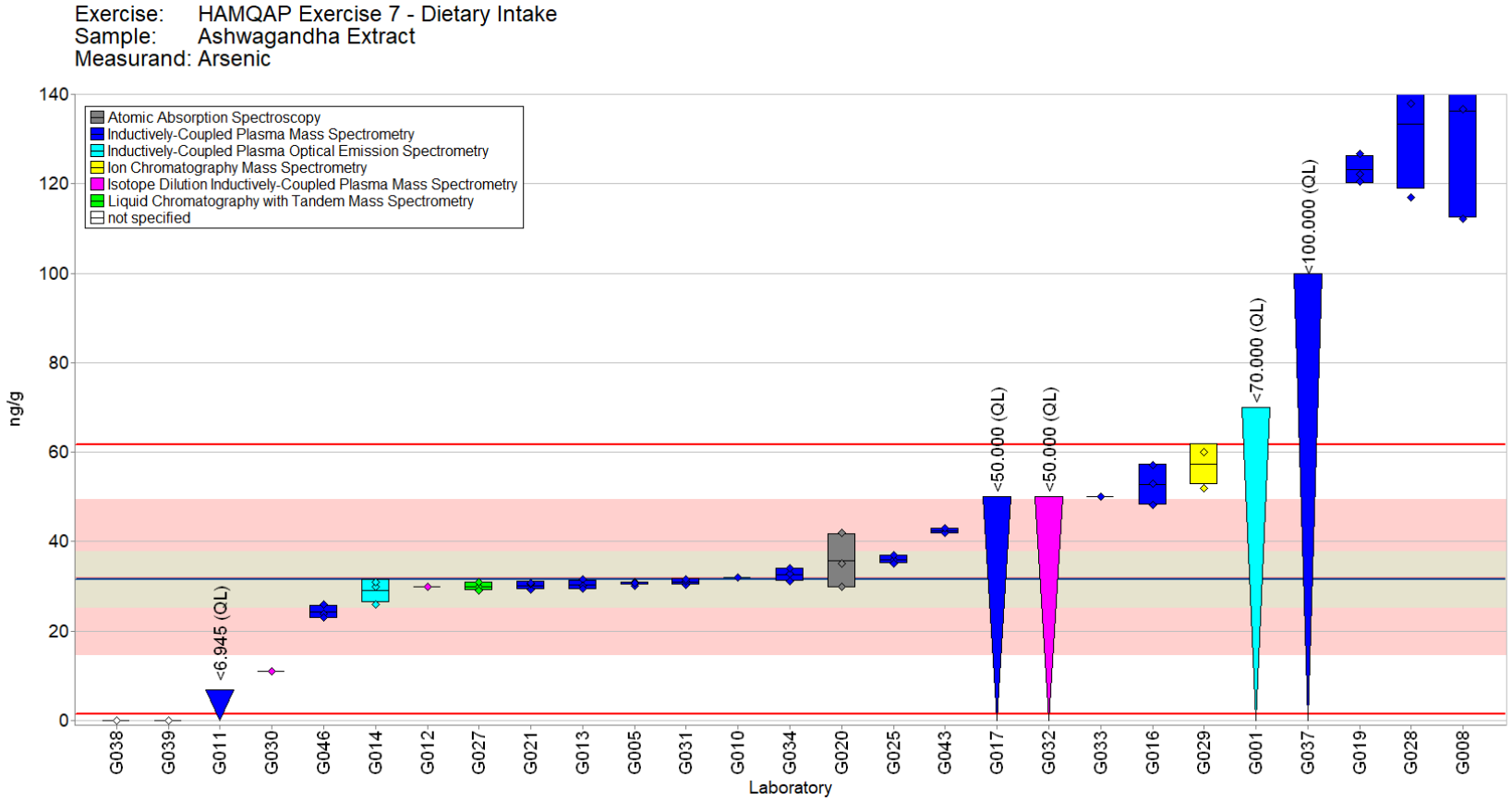

Fig. 2-2. Arsenic in ashwagandha extract (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents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

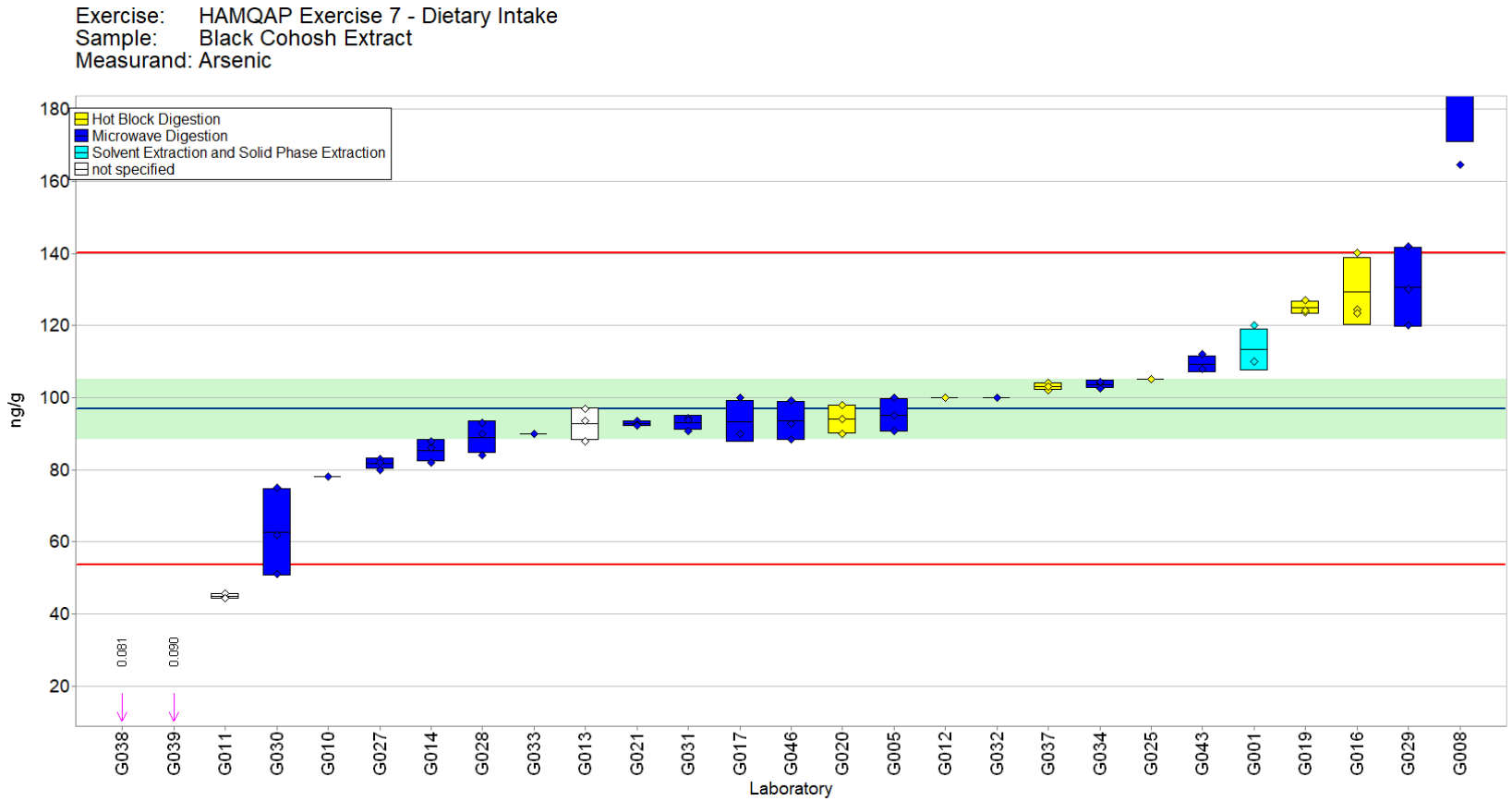

Fig. 2-3. Arsenic in black cohosh extract (data summary view – sample preparation method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the sample preparation method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. A target value has not been determined in this material.

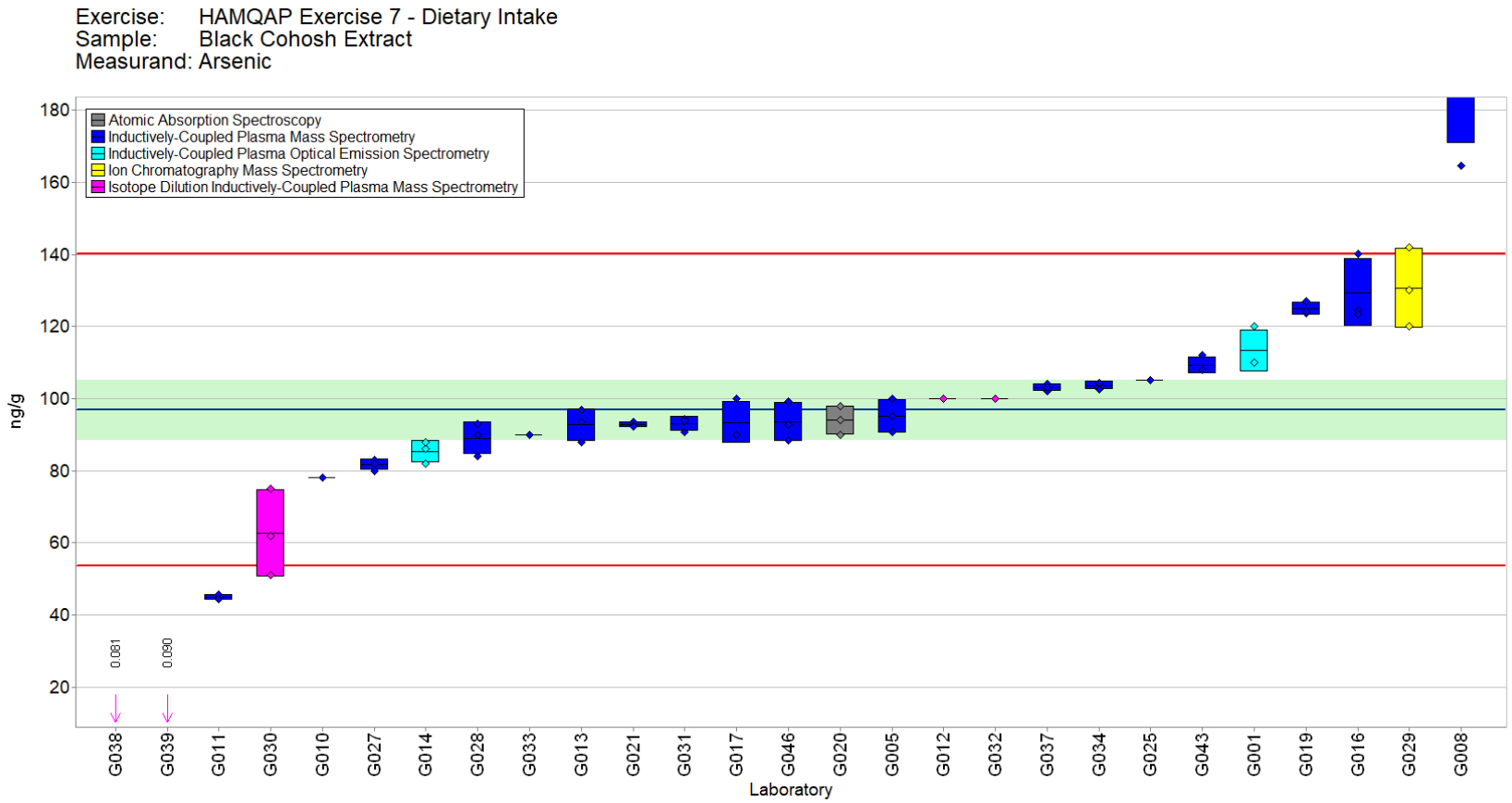

Fig. 2-4. Arsenic in black cohosh extract (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. A target value has not been determined in this material.

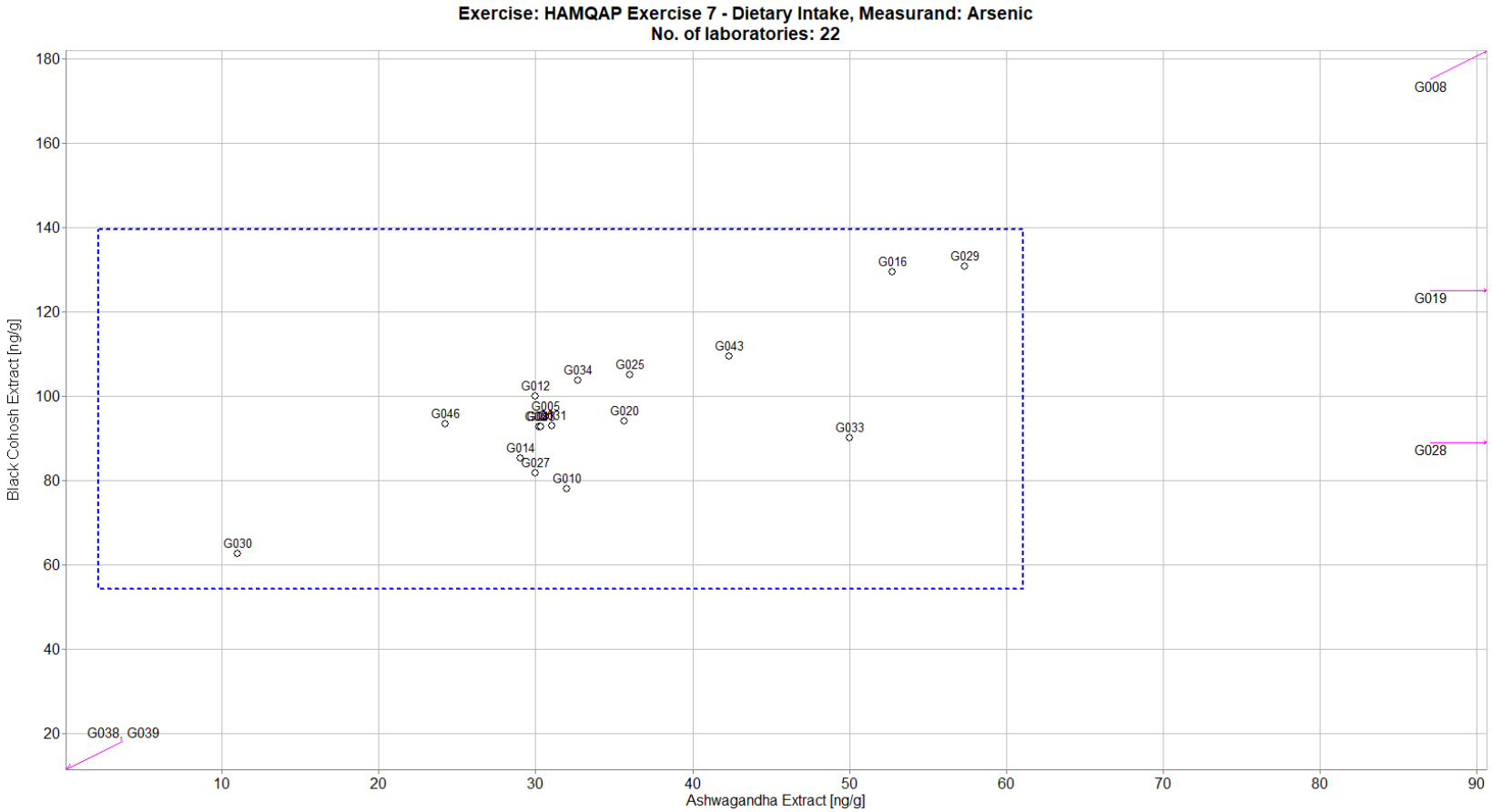

Fig. 2-5. Laboratory means for arsenic in ashwagandha extract and black cohosh extract (sample/sample comparison view). In this view, the individual laboratory mean for one sample (ashwagandha extract) is compared to the mean for a second sample (black cohosh extract). The dotted blue box represents the consensus range of tolerance for ashwagandha extract (x-axis) and black cohosh extract (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

Table 2-3. Data summary table for cadmium in black cohosh and ashwagandha extracts. Data highlighted in blue have been identified as outside the consensus tolerance limits and resulted in an unacceptable Z'_{comm} score, $|Z'_{comm}| > 2$.

		Cadmium									
		Black Cohosh Extract (ng/g)					Ashwagandha Extract (ng/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target									7.46	0.49
	G001	20	10	20	16.67	5.77	< 10	< 10	< 10		
	G002										
	G005						< 10	< 10	< 10		
	G008	17.84	15.75	17.67	17.09	1.16	5.903	5.943	5.917	5.92	0.02
	G009										
	G010	12			12		6			6	
	G011	14.291	15.808	11.206	13.77	2.35	< 4.224	4.332	4.74	4.54	0.29
	G012	13	12	12	12.33	0.58	4	4	5	4.33	0.58
	G013	12.9	14.2	14.1	13.73	0.72	5.5	< 5	5.35	5.43	0.11
	G014	11	19	11	13.67	4.62	< 8	< 8	< 8		
	G015										
	G016	< 0.5	< 0.5	< 0.5			< 0.5	< 0.5	< 0.5		
	G017	14	20	12	15.33	4.16	< 10	< 10	< 10		
	G019	12.7	12.31	12.59	12.53	0.20	5.25	5.23	5.24	5.24	0.01
	G020	10	10	12	10.67	1.15	< 9	< 9	< 9		
	G021	17.6	17.7	17.8	17.70	0.10	< 10	< 10	< 10		
	G023										
	G024										
	G025	15	13	15	14.33	1.15	6	6	5	5.67	0.58
	G026										
	G027	11	12	12	11.67	0.58	5	5	4	4.67	0.58
	G028	16	18	18	17.33	1.15	5	5	6	5.33	0.58
	G029	< 4	< 4	< 4			< 4	< 4	< 4		
	G030	13	14	17	14.67	2.08	3	4	2	3	1
	G031	13.7	13.2	10.7	12.53	1.61	4.8	4	5	4.60	0.53
	G032	< 1	< 1	< 1			< 10	< 10	< 10		
	G033	20	20	10	16.67	5.77	10	10	10	10	0
	G034	15.1	14.8	14.3	14.73	0.40	5.9	5.2	4.9	5.33	0.51
	G036										
	G037	13.1	12.9	12.6	12.87	0.25	5.4	5.1	4.8	5.10	0.30
	G038	0.014	0.015	0.012	0.014	0.002	0.005	0.006	0.005	0.005	0.001
	G039	0.013	0.016	0.013	0.014	0.002	< 0.01	< 0.01	< 0.01		
	G043	< 20	< 20	< 20			< 10	< 10	< 10		
	G045										
	G046	14	14	13.4	13.80	0.35	< 10	< 10	< 10		
	G047										
	G048										
Community Results		Consensus Mean			14.00		Consensus Mean			5.04	
		Consensus Standard Deviation			3.56		Consensus Standard Deviation			1.19	
		Maximum			17.70		Maximum			10.00	
		Minimum			0.014		Minimum			0.005	
		N			21		N			14	

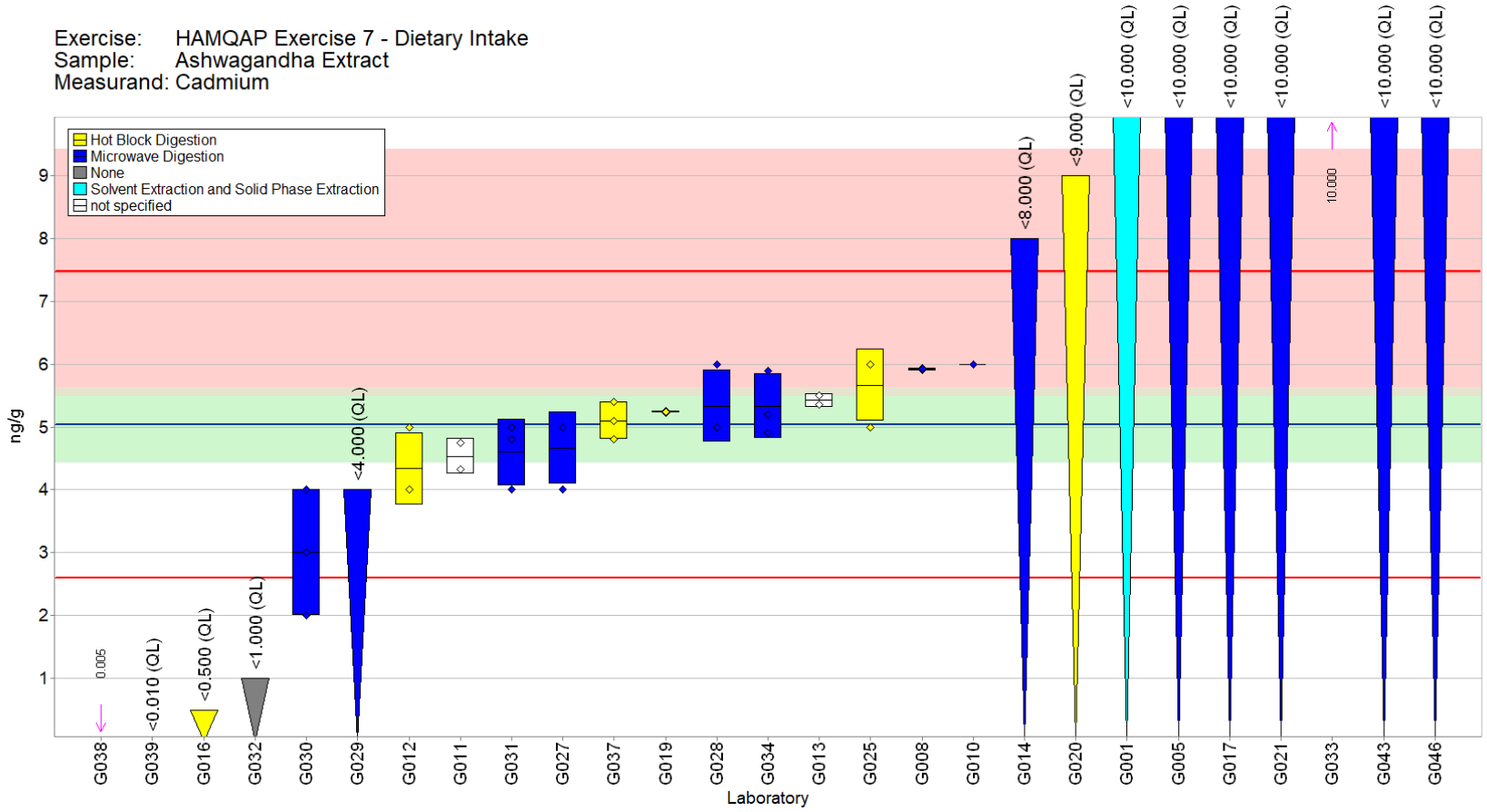

Fig. 2-6. Cadmium in ashwagandha extract (data summary view – sample preparation method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

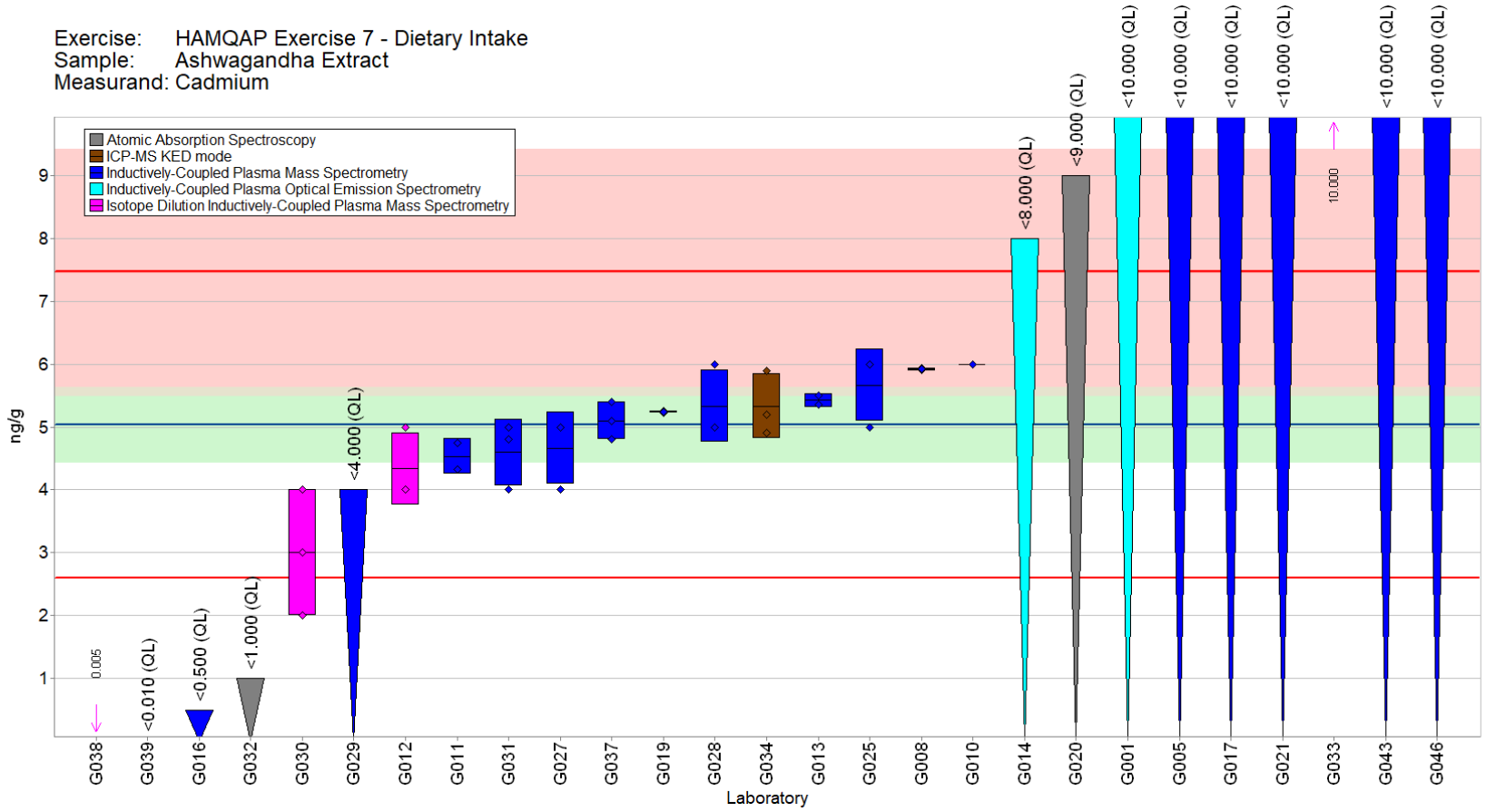

Fig. 2-7. Cadmium in ashwagandha extract (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents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

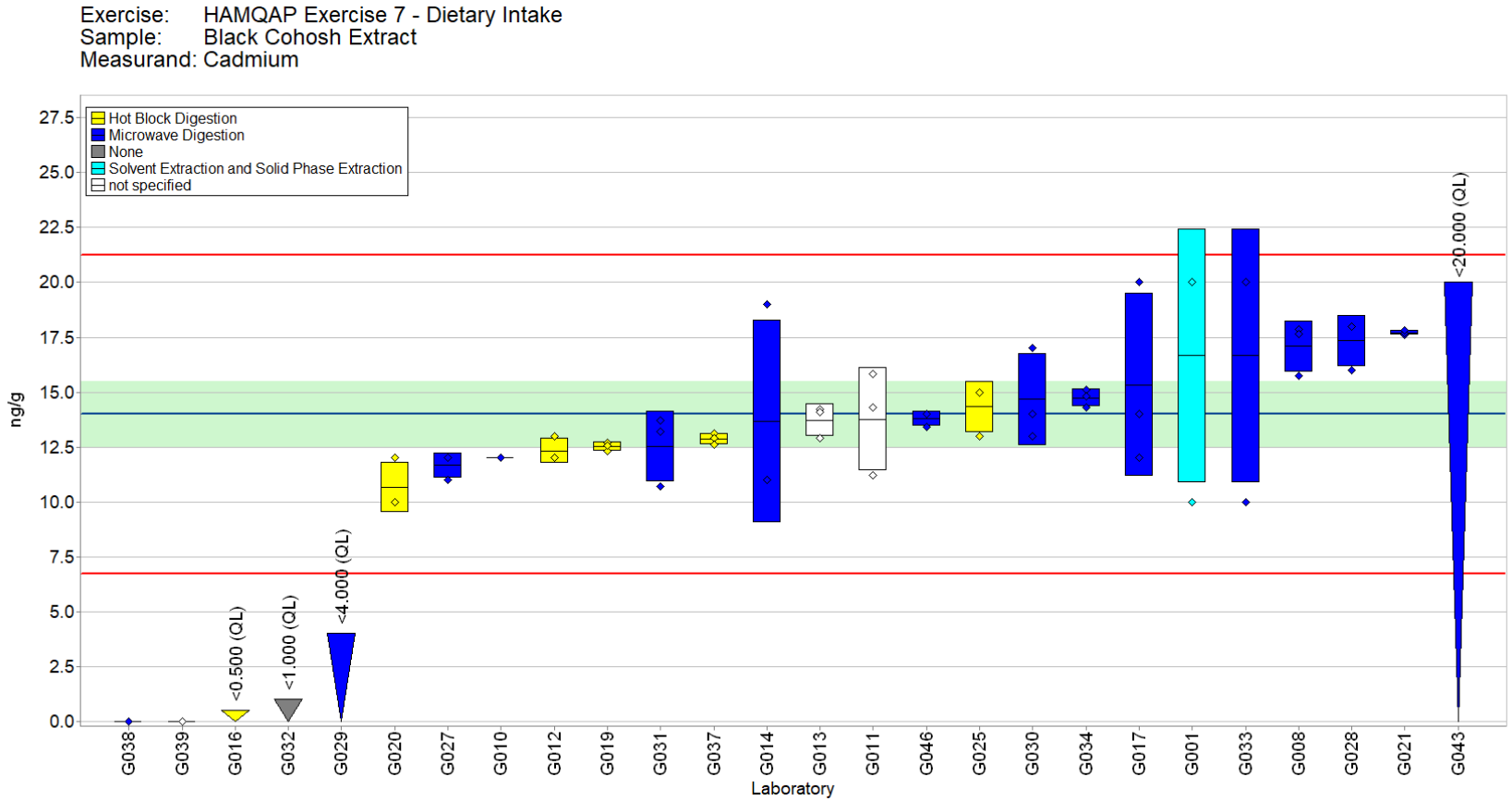

Fig. 2-8. Cadmium in black cohosh extract (data summary view – sample preparation method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. A target value has not been determined in this material.

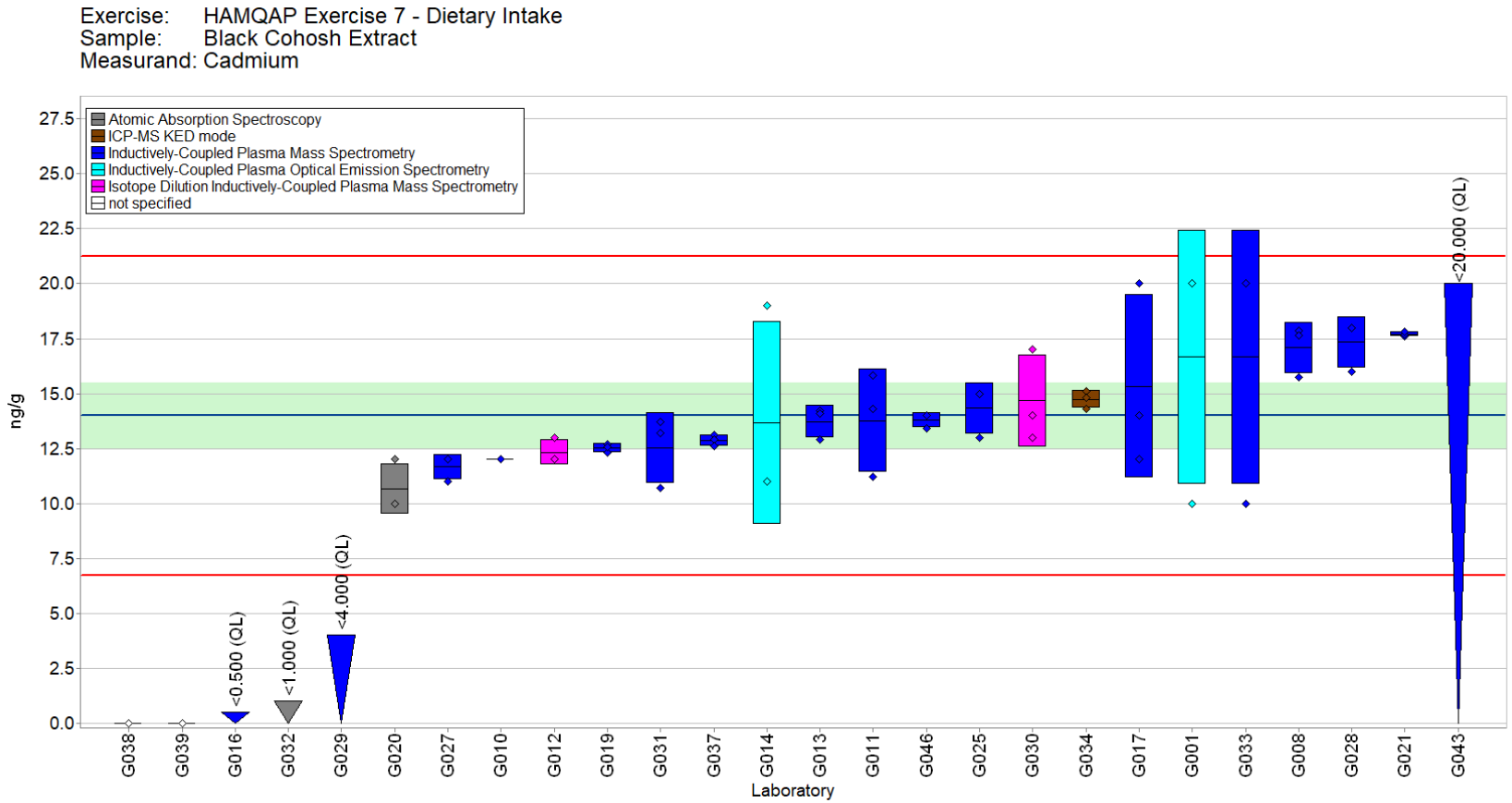

Fig. 2-9. Cadmium in black cohosh extract (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. A target value has not been determined in this material.

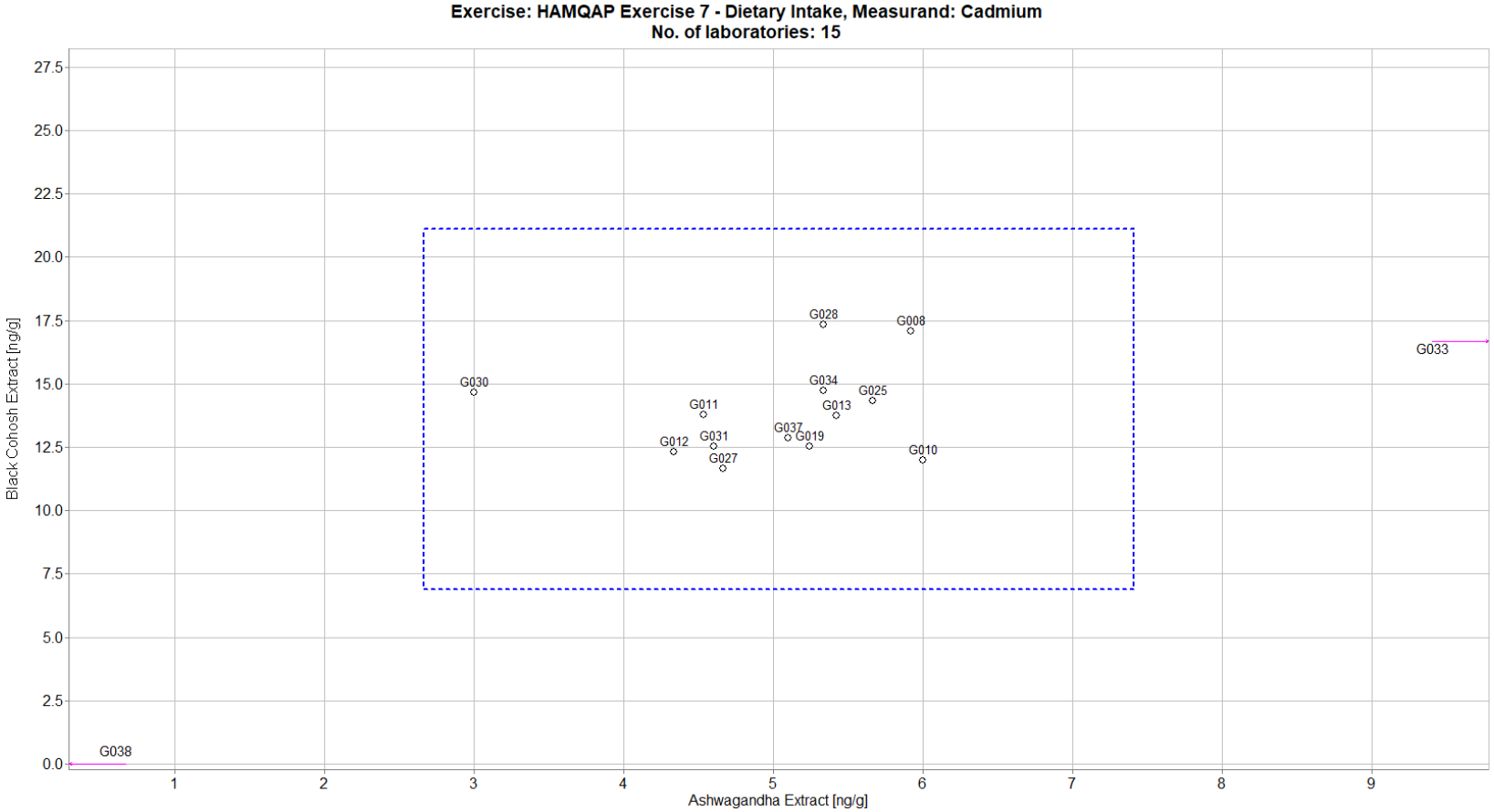

Fig. 2-10. Laboratory means for cadmium in ashwagandha extract and black cohosh extract (sample/sample comparison view). In this view, the individual laboratory mean for one sample (ashwagandha extract) is compared to the mean for a second sample (black cohosh extract). The dotted blue box represents the consensus range of tolerance for ashwagandha extract (x-axis) and black cohosh extract (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

Table 2-4. Data summary table for lead in black cohosh and ashwagandha extracts. Data highlighted in blue have been identified as outside the consensus tolerance limits and resulted in an unacceptable Z'_{comm} score, $|Z'_{comm}| > 2$.

		Lead									
		Black Cohosh Extract (ng/g)					Ashwagandha Extract (ng/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target									9.61	0.38
	G001	< 30	< 30	< 30			< 20	< 20	< 20		
	G002										
	G005	289	277	283	283.0	6.0	11	11	10	10.67	0.58
	G008	336.9	346.6	432.1	371.9	52.4	23.61	23.77	25.64	24.34	1.13
	G009										
	G010	725			725		13			13	
	G011	302.929	291.185	291.344	295.2	6.7	11.883	11.528	15.048	12.82	1.94
	G012	270	240	260	256.7	15.3	10	10	10	10	0
	G013	279	287	278	281.3	4.9	9.82	10.7	13.9	11.47	2.15
	G014	263	274	257	264.7	8.6	< 9	10	< 9	10	
	G015										
	G016	312.74	292.12	293.37	299.4	11.6	9.42	10.37	12.6	10.80	1.63
	G017	460	400	380	413.3	41.6	60	140	40	80.00	52.92
	G019	5.52	4.61	4.36	4.83	0.61	69.7	23.3	24.96	39.32	26.32
	G020	260	258	264	260.7	3.1	10	15	10	11.67	2.89
	G021	273	274	272	273.0	1.0	18	18.1	18.5	18.20	0.26
	G023										
	G024										
	G025	238	231	230	233.0	4.4	9	9	9	9	0
	G026										
	G027	234	215	216	221.7	10.7	26	16	16	19.33	5.77
	G028	299	298	296	297.7	1.5	18	18	19	18.33	0.58
	G029	334	1268	348	650.0	535.2	< 4	< 4	< 4		
	G030	275	224	335	278.0	55.6	< 1	4	< 1	4	
	G031	267	227	225	239.7	23.7	9.2	8.6	7.8	8.53	0.70
	G032	191	175	188	184.7	8.5	< 50	< 50	< 50		
	G033	280	280	330	296.7	28.9	< 40	< 40	< 40		
	G034	332.3	287.5	284.4	301.4	26.8	10.9	13.1	10.1	11.37	1.55
	G036										
	G037	254	251	279	261.3	15.4	< 15	< 15	< 15		
	G038	0.281	0.323	0.29	0.30	0.02	0.013	< 0.01	0.011	0.012	0.001
	G039	0.29	0.32	0.3	0.30	0.02	0.049	0.046	0.05	0.048	0.002
	G043	294	286	276	285.3	9.0	< 20	< 20	< 20		
	G045										
	G046	307	316	325	316.0	9.0	14.9	15.3	15.3	15.17	0.23
	G047										
	G048										
Community Results		Consensus Mean			278.1		Consensus Mean			11.68	
		Consensus Standard Deviation			64.0		Consensus Standard Deviation			6.72	
		Maximum			725.0		Maximum			80.00	
		Minimum			0.30		Minimum			0.012	
		N			25		N			18	

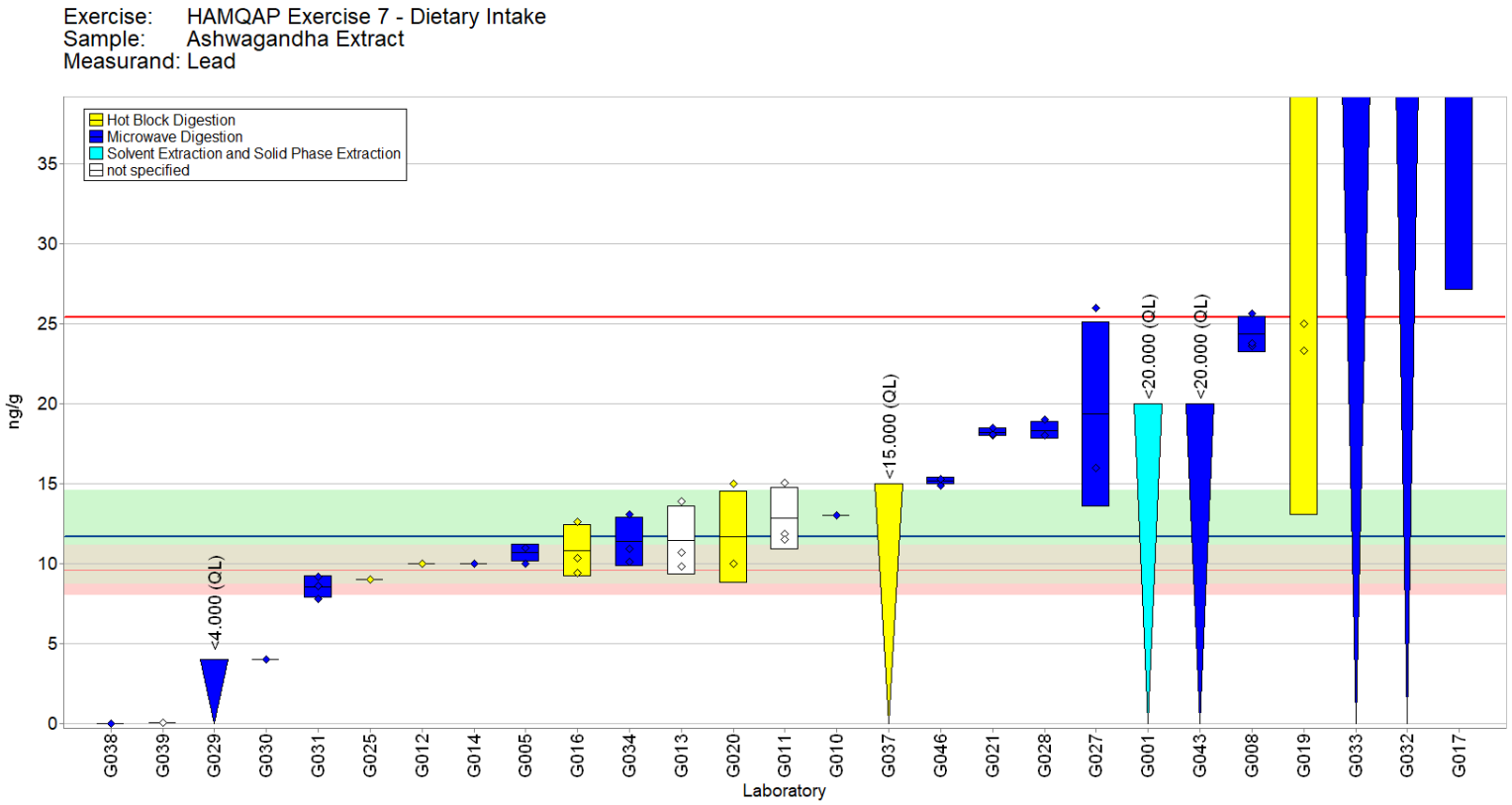

Fig. 2-11. Lead in ashwagandha extract (data summary view – sample preparation method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$, with the lower range set at zero. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The beige shaded region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

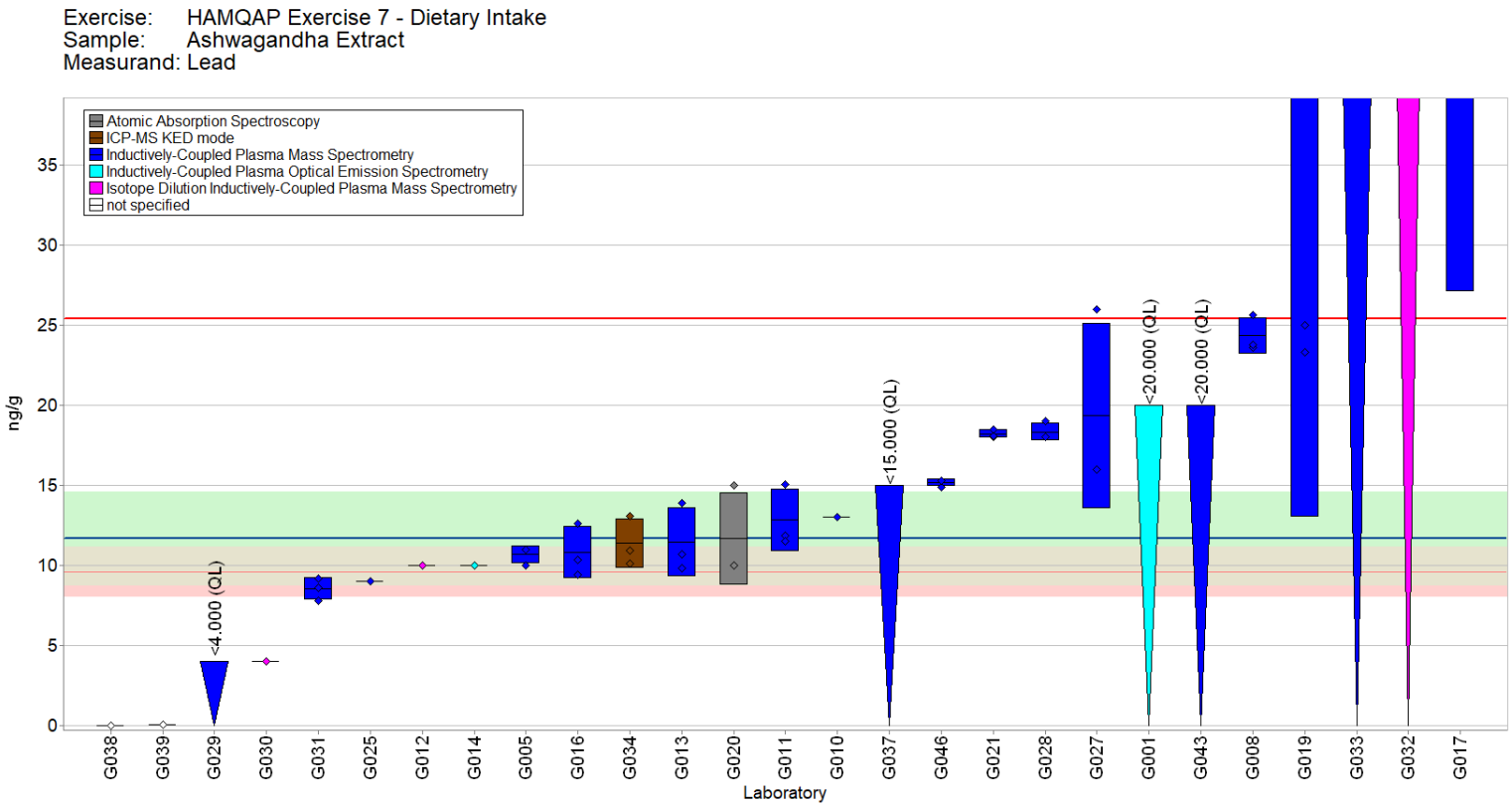

Fig. 2-12. Lead in ashwagandha extract (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$, with the lower range set at zero. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The beige shaded region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

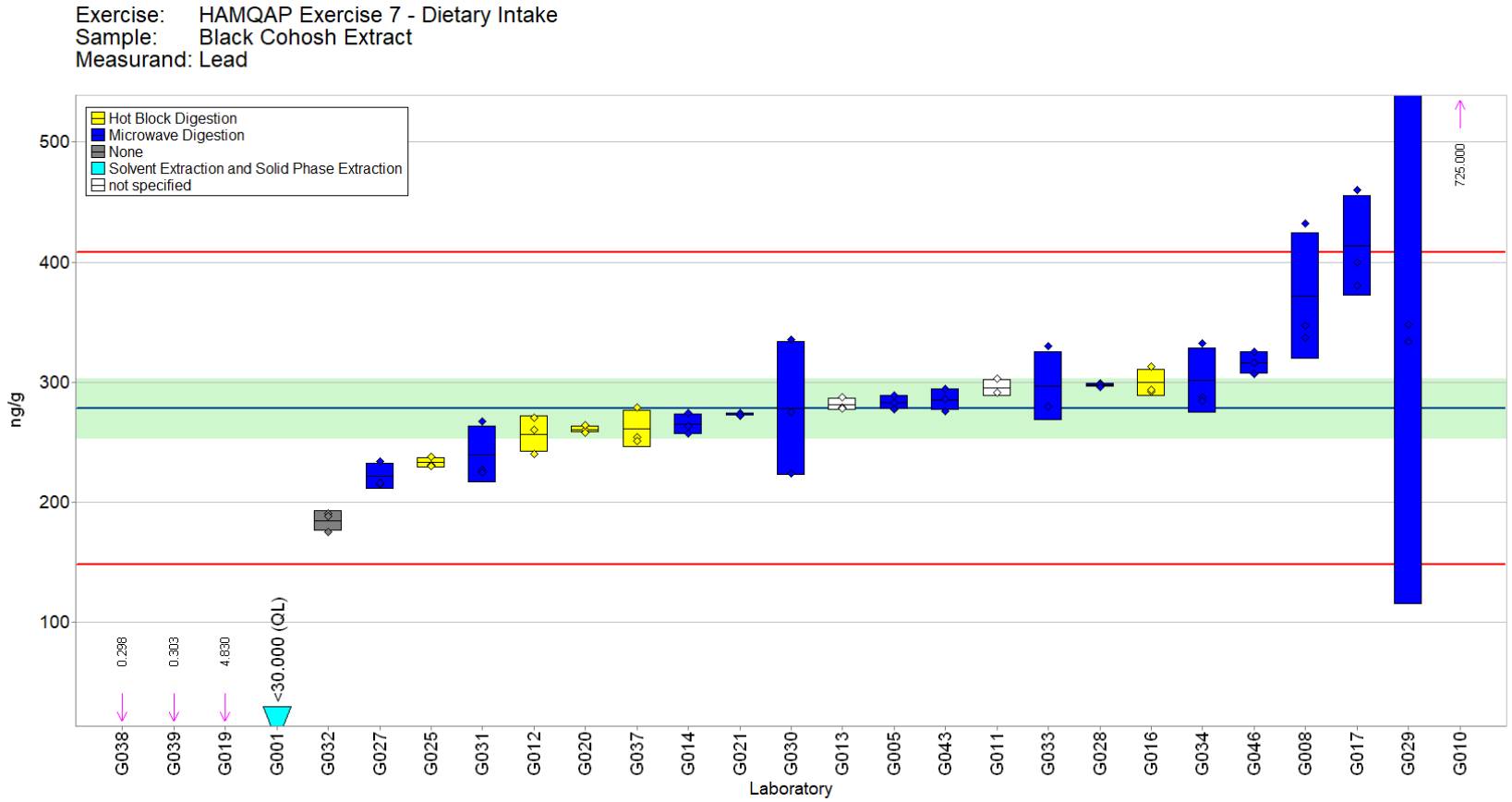

Fig. 2-13. Lead in black cohosh extract (data summary view – sample preparation method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the sample preparation method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. A target value has not been determined in this material.

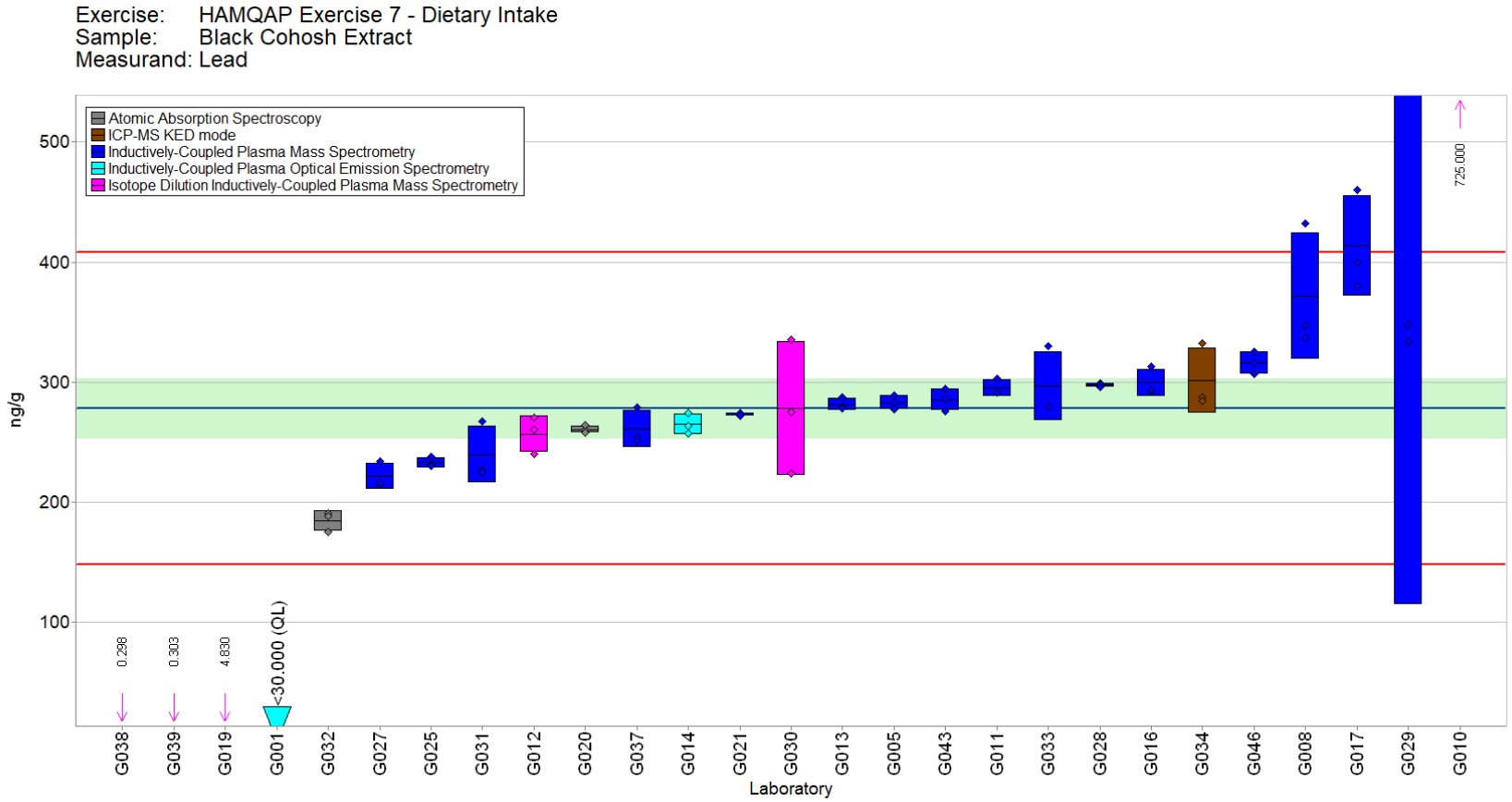

Fig. 2-14. Lead in black cohosh extract (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. A target value has not been determined in this material.

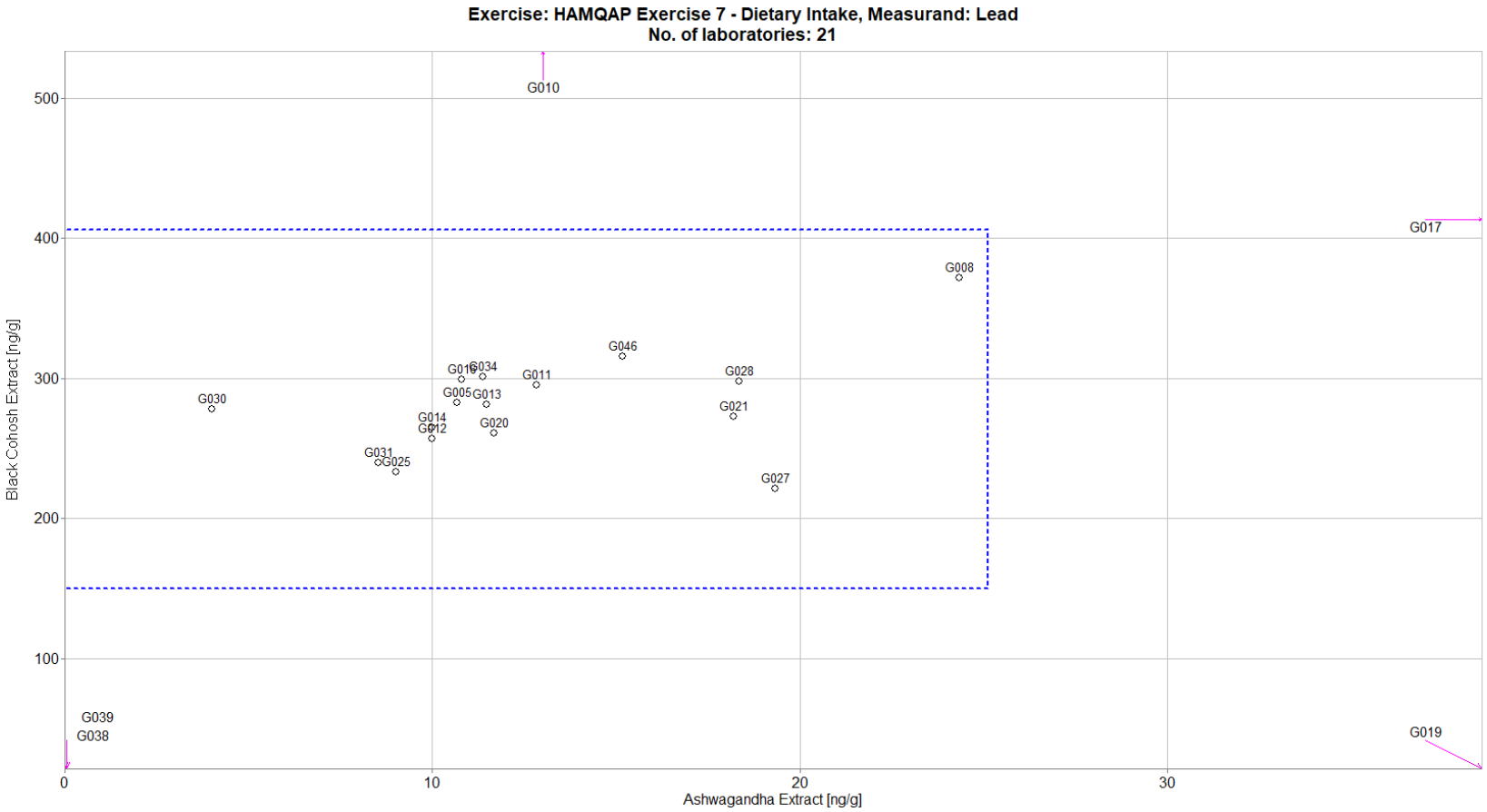

Fig. 2-15. Laboratory means for lead in ashwagandha extract and black cohosh extract (sample/sample comparison view). In this view, the individual laboratory mean for one sample (ashwagandha extract) is compared to the mean for a second sample (black cohosh extract). The dotted blue box represents the consensus range of tolerance for ashwagandha extract (x-axis) and black cohosh extract (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

Table 2-5. Data summary table for mercury in black cohosh and ashwagandha extracts. Data highlighted in blue have been identified as outside the consensus tolerance limits and resulted in an unacceptable Z'_{comm} score, $|Z'_{comm}| > 2$. Data points highlighted in red have a zero or a non-numeric data point.

		Mercury									
		Black Cohosh Extract (ng/g)					Ashwagandha Extract (ng/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target										
	G001	280	290	290	286.7	5.8	10	10	10	10	0
	G002										
	G005	< 10	< 10	< 10			< 10	< 10	< 10		
	G008	0	1.969	0	0.66	1.14	17.71	3.962	1.972	7.88	8.57
	G009										
	G010	8			8		5			5	
	G011	< 1.652	< 1.652	< 1.652			< 1.652	< 1.652	< 1.652		
	G012	< 5	< 5	< 5			< 5	< 5	< 5		
	G013	< 5	< 5	< 5			< 5	< 5	< 5		
	G014	2.4	2	1.9	2.10	0.26	1.5	1.1	2.4	1.67	0.67
	G015										
	G016	< 0.1	< 0.1	< 0.1			< 0.1	< 0.1	< 0.1		
	G017	< 50	< 50	< 50			< 50	< 50	< 50		
	G019	249.72	263.42	267.42	260.2	9.3	6.96	5.74	4.34	5.68	1.31
	G021										
	G023										
	G024										
	G025	6	6	3	5.00	1.73	4	2	3	3.0	1.0
	G026										
	G027	5	5	6	5.33	0.58	7	5	5	5.67	1.15
	G028	< 1	< 1	< 1			< 1	< 1	< 1		
	G029	< 4	< 4	< 4			< 4	< 4	< 4		
	G030	< 3	< 3	< 3			< 3	< 3	< 3		
	G031	4	< 2.1	< 2.1	4		< 1.8	< 1.8	< 1.8		
	G032	< 1	< 1	< 1			< 1	< 1	< 1		
	G033	< 10	< 10	< 10			< 10	< 10	< 10		
	G034	5.4	4.2	3.8	4.47	0.83	9.8	7.2	7.4	8.13	1.45
	G036										
	G037	< 7	< 7	< 7			< 7	< 7	< 7		
	G038	< 0.005	< 0.005	< 0.005			< 0.005	< 0.005	< 0.005		
	G039	< 0.01	< 0.01	< 0.01			< 0.01	< 0.01	< 0.01		
	G043	< 10	< 10	< 10			< 10	< 10	< 10		
	G045										
	G046	< 10	< 10	< 10			< 10	< 10	< 10		
	G047										
	G048										
Community Results		Consensus Mean			4.22		Consensus Mean			5.88	
		Consensus Standard Deviation			4.47		Consensus Standard Deviation			4.12	
		Maximum			286.7		Maximum			10.0	
		Minimum			0.66		Minimum			1.67	
		N			7		N			7	

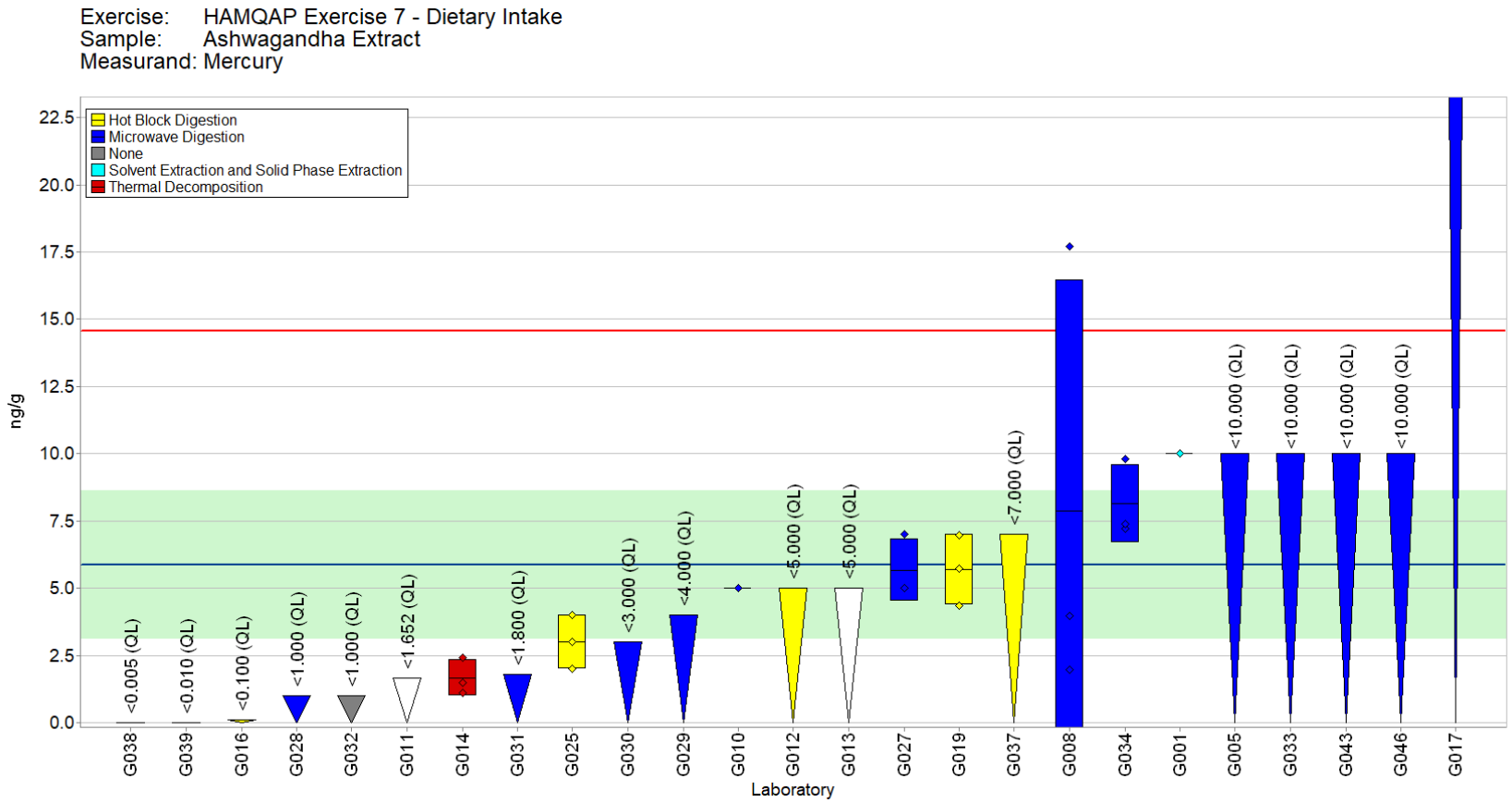

Fig. 2-16. Mercury in ashwagandha extract (data summary view – sample preparation method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$, with the lower range set at zero. A target value has not been determined in this material.

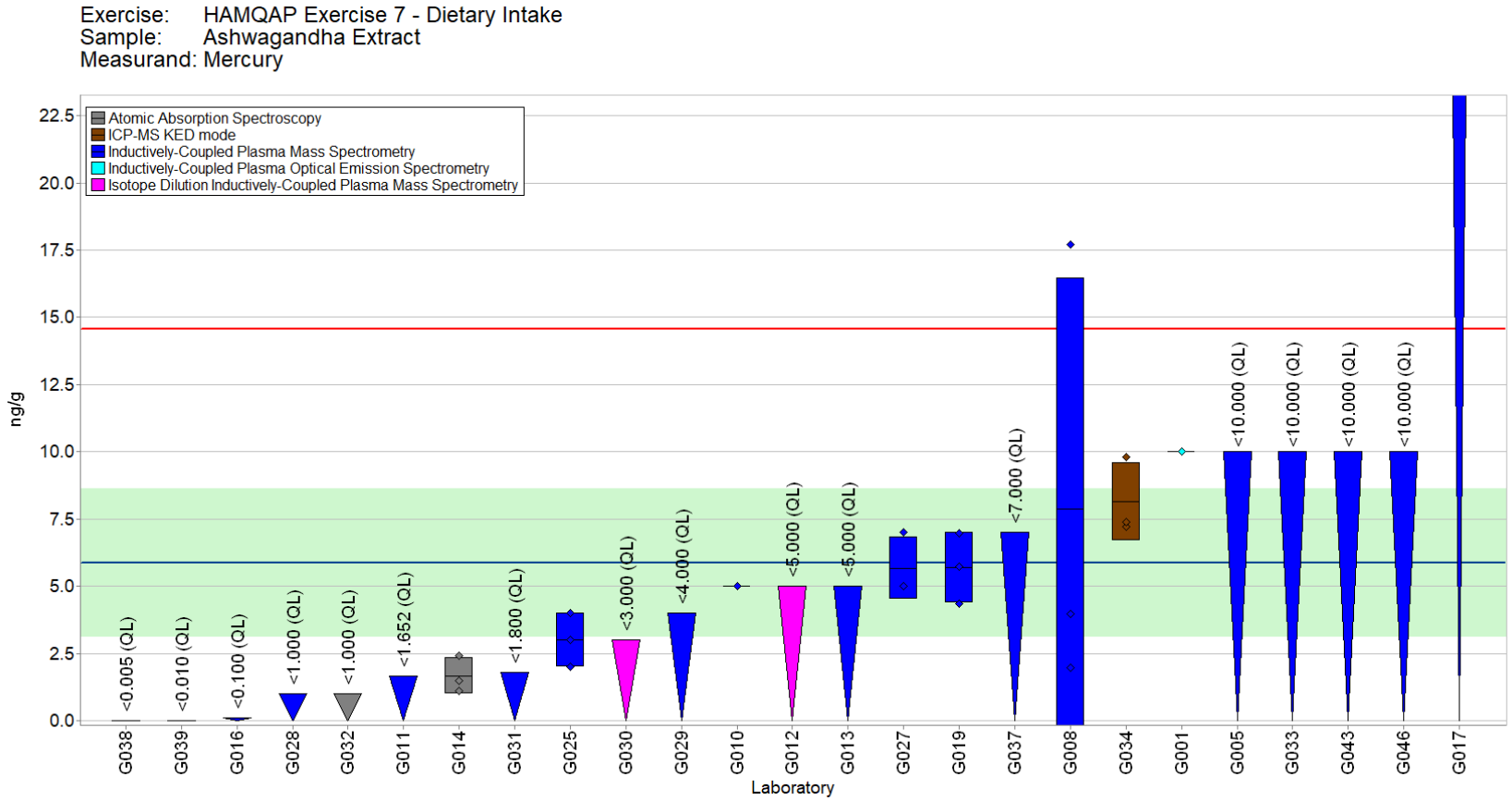

Fig. 2-17. Mercury in ashwagandha extract (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$, with the lower range set at zero. A target value has not been determined in this material.

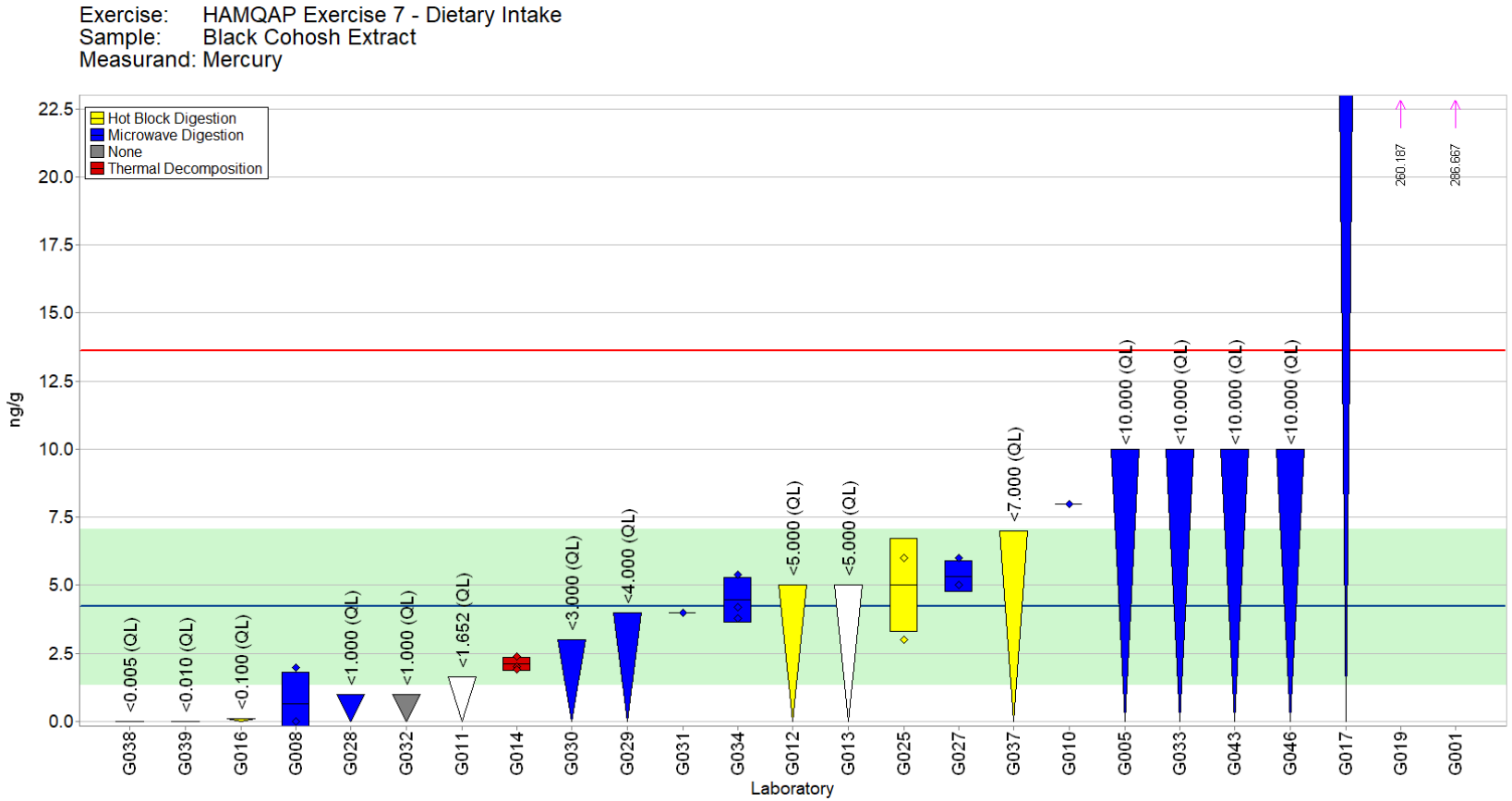

Fig. 2-18. Mercury in black cohosh extract (data summary view – sample preparation method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the sample preparation method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$, with the lower range set at zero. A target value has not been determined in this material.

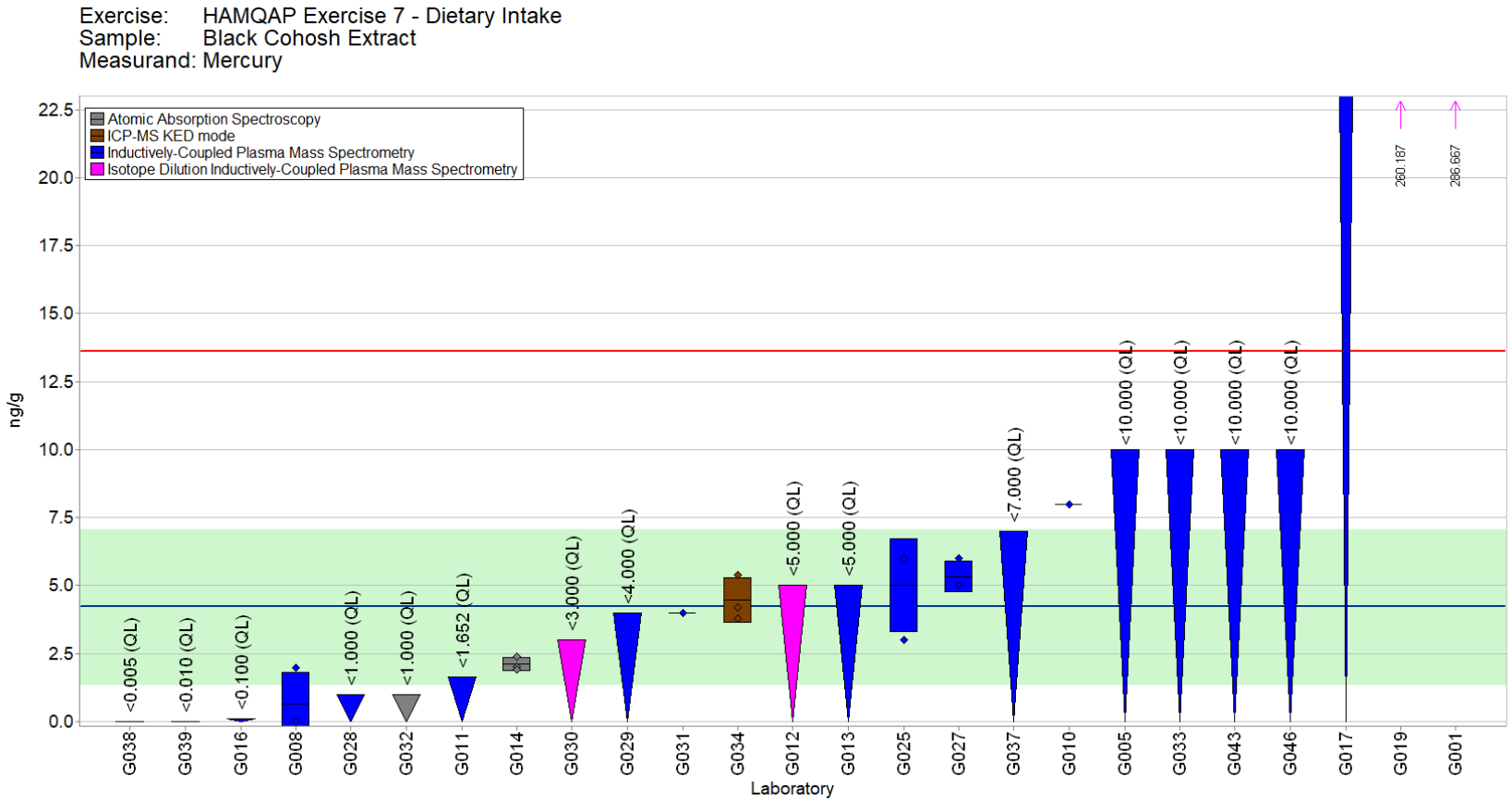

Fig. 2-19. Mercury in black cohosh extract (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$, with the lower range set at zero. A target value has not been determined in this material.

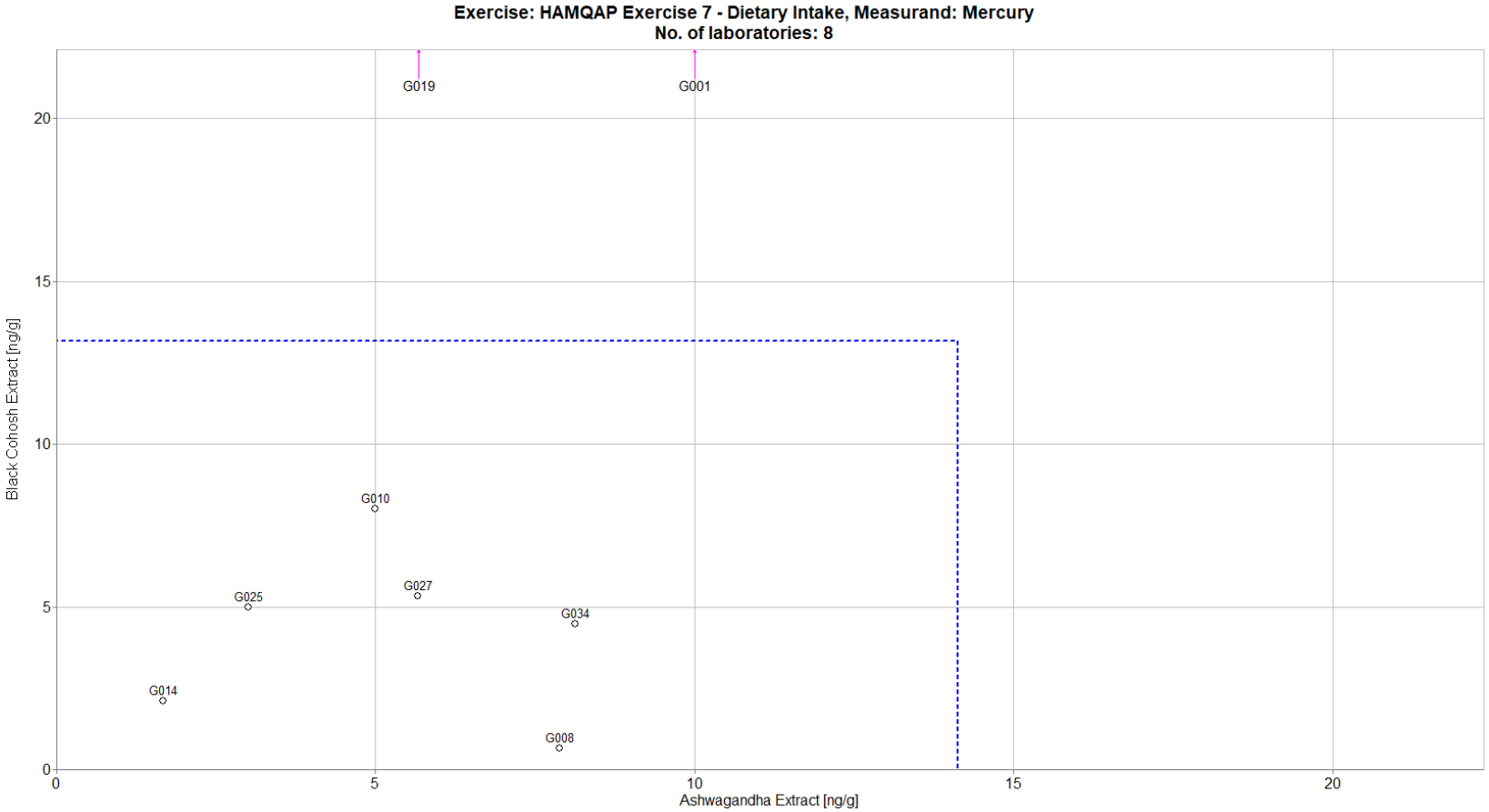

Fig. 2-20. Laboratory means for mercury in ashwagandha extract and black cohosh extract (sample/sample comparison view). In this view, the individual laboratory mean for one sample (ashwagandha extract) is compared to the mean for a second sample (black cohosh extract). The dotted blue box represents the consensus range of tolerance for ashwagandha extract (x-axis) and black cohosh extract (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

3. Water-Soluble Vitamins (Vitamins B_2 and B_6)

3.1. Study Overview

Vitamin B_2 (riboflavin) and vitamin B_6 (pyridoxine) are water-soluble vitamins present in some foods both naturally and through fortification and are available as dietary supplements. Vitamin B_2 has roles in energy production, cellular function, growth, and development as well as in metabolism of fats, drugs, and steroids. [5] Vitamin B_6 is important for a wide variety of functions in the body, particularly in protein and amino acid metabolism. Vitamin B_6 vitamers are also involved in the biosynthesis of neurotransmitters, in maintaining normal levels of homocysteine in the blood, in gluconeogenesis and glycogenolysis, in immune functions, and in hemoglobin formation. [6] Testing of these vitamins in foods and supplements can help ensure accurate dietary intake estimates and product labeling.

In this study, participants were provided with samples of multivitamin tablets and protein powder as representative dietary intake samples. Participants were asked to use in-house analytical methods to determine the mass fractions (mg/kg) of vitamin B_2 as riboflavin and vitamin B_6 as pyridoxine in the dietary intake samples on an as-received basis (i.e., not moisture corrected).

3.2. Sample Information

Multivitamin A. Participants were provided with three bottles, each containing 30 multivitamin tablets. Participants were asked to store the material at controlled room temperature, 20 °C to 25 °C, in the original unopened bottles, and to prepare one sample, and report one value from each bottle provided. Before use, participants were instructed to grind all 30 tablets and mix the resulting powder thoroughly prior to removal of a test portion for analysis, and to use a sample size of at least 0.2 g. After grinding, participants were instructed to store the resulting powder at –20 °C or colder and analyze the material within two days for analytes in this study. Approximate analyte levels were not disclosed to participants prior to the study. The target values for riboflavin and pyridoxine in the multivitamin sample were determined using data from the manufacturer of the material and results from a previous HAMQAP exercise. [7] The values and standard deviations for vitamin B_2 (riboflavin) and vitamin B_6 (pyridoxine) are provided in the table below on an as-received basis.

Analyte	Target Mass Fraction in Multivitamin A (mg/kg)
Vitamin B_2 (Riboflavin)	1311 ± 93
Vitamin B_6 (Pyridoxine)	1360 ± 36

Protein Sample D. Participants were provided with one packet containing 10 g of protein powder. Participants were asked to store the material at controlled room temperature, 20 °C to 25 °C, in the original unopened packet, to prepare three samples, and report three values from the single packet provided. Before use, participants were instructed to mix the contents of each packet thoroughly, allow contents to settle for one minute prior to opening to minimize the loss of fine particles, and to use a sample size appropriate for their usual in-house method of analysis. Approximate analyte levels were not disclosed to participants prior to the study. The target values for riboflavin and pyridoxine in the protein sample were determined from the product Nutrition Facts label. The values and uncertainties (20 % of target value) for vitamin B_2 (riboflavin) and vitamin B_6 (pyridoxine) are provided in the table below on an as-received basis.

Analyte	Target Mass Fraction in Protein Sample D (mg/kg)
Vitamin B_2 (Riboflavin)	50 ± 10
Vitamin B_6 (Pyridoxine)	60 ± 12

3.3. Study Results

The participation/enrollment and reporting statistics for each analyte in the dietary intake study are described in the table below. Reported values may include non-quantitative results (zero or below LOQ) that are only included in the participation statistics.

Analyte	Number of Laboratories Requesting Samples	Number of Laboratories Reporting Results (Percent Participation)	
		Multivitamin A	Protein Sample D
Vitamin B_2 (Riboflavin)	34	21 (62 %)	16 (47 %)
Vitamin B_6 (Pyridoxine)	35	21 (60 %)	16 (46 %)

The between-laboratory variabilities were less than 31 % for riboflavin and pyridoxine in both samples.

Analyte	Between-Laboratory Variability (% RSD)	
	Multivitamin A	Protein Sample D
Vitamin B_2 (Riboflavin)	11 %	20 %
Vitamin B_6 (Pyridoxine)	17 %	31 %

Most laboratories who reported sample preparation methods indicated using some form of solvent extraction for determination of vitamins B_2 and B_6 in both samples.

	Percentage of Laboratories Reporting			
	Multivitamin A		Protein Sample D	
Sample Preparation Method	Vitamin B_2 (Riboflavin)	Vitamin B_6 (Pyridoxine)	Vitamin B_2 (Riboflavin)	Vitamin B_6 (Pyridoxine)
Solvent Extraction	47 %	47 %	36 %	36 %
Dilution	16 %	16 %	21 %	29 %
Acid Hydrolysis	5 %	-	14 %	-
Solvent Extraction & Solid Phase Extraction	5 %	5 %	7 %	7 %
Base Hydrolysis	5 %	-	7 %	-
Other/None Reported	21 %	31 %	14 %	28 %

Most laboratories reported using LC-Abs as their analytical method for determination of vitamins B_2 and B_6 in both samples.

	Percentage of Laboratories Reporting			
	Multivitamin A		Protein Sample D	
Analytical Method	Vitamin B_2 (Riboflavin)	Vitamin B_6 (Pyridoxine)	Vitamin B_2 (Riboflavin)	Vitamin B_6 (Pyridoxine)
LC-Abs	58 %	47 %	43 %	47%
LC-MS	5 %	5 %	14 %	7 %
LC-MS/MS	16 %	21 %	14 %	20 %
LC-FLD	16 %	21 %	21 %	26 %
Other/None Reported	5%	5 %	7%	-

The consensus and target ranges were mostly in agreement for both vitamins in both samples, as described in the table below.

	Relative to NIST Range of Tolerance for			
	Multivitamin A		Protein Sample D	
Position of	Vitamin B_2 (Riboflavin)	Vitamin B_6 (Pyridoxine)	Vitamin B_2 (Riboflavin)	Vitamin B_6 (Pyridoxine)
Consensus Mean	Within	Within	Within	Within
Consensus Range	Centered	Within but high	Centered	Centered
Corresponding Figures	3-1, 3-2	3-6, 3-7	3-3, 3-4	3-8, 3-9

3.4. Water-Soluble Vitamins Technical Recommendations

The following recommendations are based on results obtained from the participants in this study. Additional overall technical recommendations can be found on page 6.

- Overall performance in this study was excellent. The consensus means and ranges were consistent with the target ranges for three of the four analyte/sample pairs. No evidence of method bias was observed.
  - Extraction of these fortified nutrients from these matrices should be relatively straightforward.
  - The slight high bias in the consensus mean and range for pyridoxine in the multivitamin sample could indicate a potential issue with chromatographic interferences.
  - The between-laboratory variabilities were slightly higher for the protein powder than for the multivitamin, and the number of reporting laboratories was lower for the protein powder. The sample complexity and the lower analyte mass factions in Protein Powder D may have been a challenge for some laboratories.
- Both riboflavin and pyridoxine may decompose in light. Samples and standards should be prepared under amber or attenuated lighting and protected from light during storage.

Table 3-1. Individualized data table (NIST) for vitamin B_2 (riboflavin) and vitamin B_6 (pyridoxine) in multivitamin tablets and protein powder.

Exercise 7 - Water-Soluble Vitamins

		Lab Code: NIST	**1. Your Results**				**2. Community Results**			**3. Target**	
Analyte	Sample	Units	x_i	s_i	Z'_{comm}	Z_{NIST}	N	x*	s*	x_{NIST}	*U*
Vitamin B2 (Riboflavin)	Multivitamin A	mg/kg	1312	187			21	1332	141	1312	187
Vitamin B2 (Riboflavin)	Protein Sample D	mg/kg	50	10			16	48.1	9.4	50	10
Vitamin B6 (Pyridoxine)	Multivitamin A	mg/kg	1360	73			21	1432	236	1360	73
Vitamin B6 (Pyridoxine)	Protein Sample D	mg/kg	60	12			16	57.2	17.9	60	12

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

Table 3-2. Data summary table for vitamin B_2 (riboflavin) in multivitamin tablets and protein powder. Data highlighted in blue have been identified as outside the consensus tolerance limits and resulted in an unacceptable Z'_{comm} score, $|Z'_{comm}| > 2$.

		Vitamin B2 (Riboflavin)									
		Multivitamin A (mg/kg)					Protein Sample D (mg/kg)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				1312	187				50.0	10.0
	G001	1391.37	1460.91	1456.26	1436	39	48.14	50.98	48.74	49.3	1.5
	G002										
	G003	1120	1170	1170	1153	29	42.9	40	39.6	40.8	1.8
	G005	1230	1330	1300	1287	51	43.2	47.1	45.1	45.1	2.0
	G006										
	G008	1246	1239	1274	1253	19	45.52	45.53	46.32	45.79	0.46
	G010										
	G012	1410	1360	1250	1340	82					
	G013	1430	1600	1470	1500	89	46.5			46.5	
	G014	1550	1570	1540	1553	15	65.2	75.4	74.4	71.7	5.6
	G015										
	G016										
	G018	1348.2	1330.6	1318.5	1332	15	49	49.1	45.1	47.7	2.3
	G019	1517.95	1497.16	1472.75	1496	23	42626.19	44103.57	42680.88	43137	838
	G020	1230	1200	1240	1223	21					
	G021	1368	1368	1385	1374	10	59.6	58.6	58.8	59.00	0.53
	G023										
	G024										
	G026										
	G027	1400	1371.83	1308.63	1360	47					
	G028										
	G030	1368.5	1383.8	1456.7	1403	47	53.8	48.6	49.2	50.5	2.8
	G032	1233.81	1233.66	1219.3	1228.9	8.3	45.25	47.08	47.4	46.6	1.2
	G033	1270	1290	1270	1277	12	52.4	52.1	53.5	52.67	0.74
	G034	1124	1169	1071	1121	49	42	41	42	41.67	0.58
	G036										
	G038	946	1100	1070	1039	82	10	17.4	23.5	17.0	6.8
	G039	1389	1418	1368	1392	25	46.38	47.22	50.42	48.0	2.1
	G041	1350	1400	1510	1420	82					
	G044	1310	1330	1310	1317	12					
	G045										
	G046	1394	1386	1393	1391.0	4.4	92	89	88	89.7	2.1
	G048										
Community Results		Consensus Mean			1332		Consensus Mean			48.13	
		Consensus Standard Deviation			141		Consensus Standard Deviation			9.45	
		Maximum			1553		Maximum			43137	
		Minimum			1039		Minimum			16.97	
		N			21		N			15	

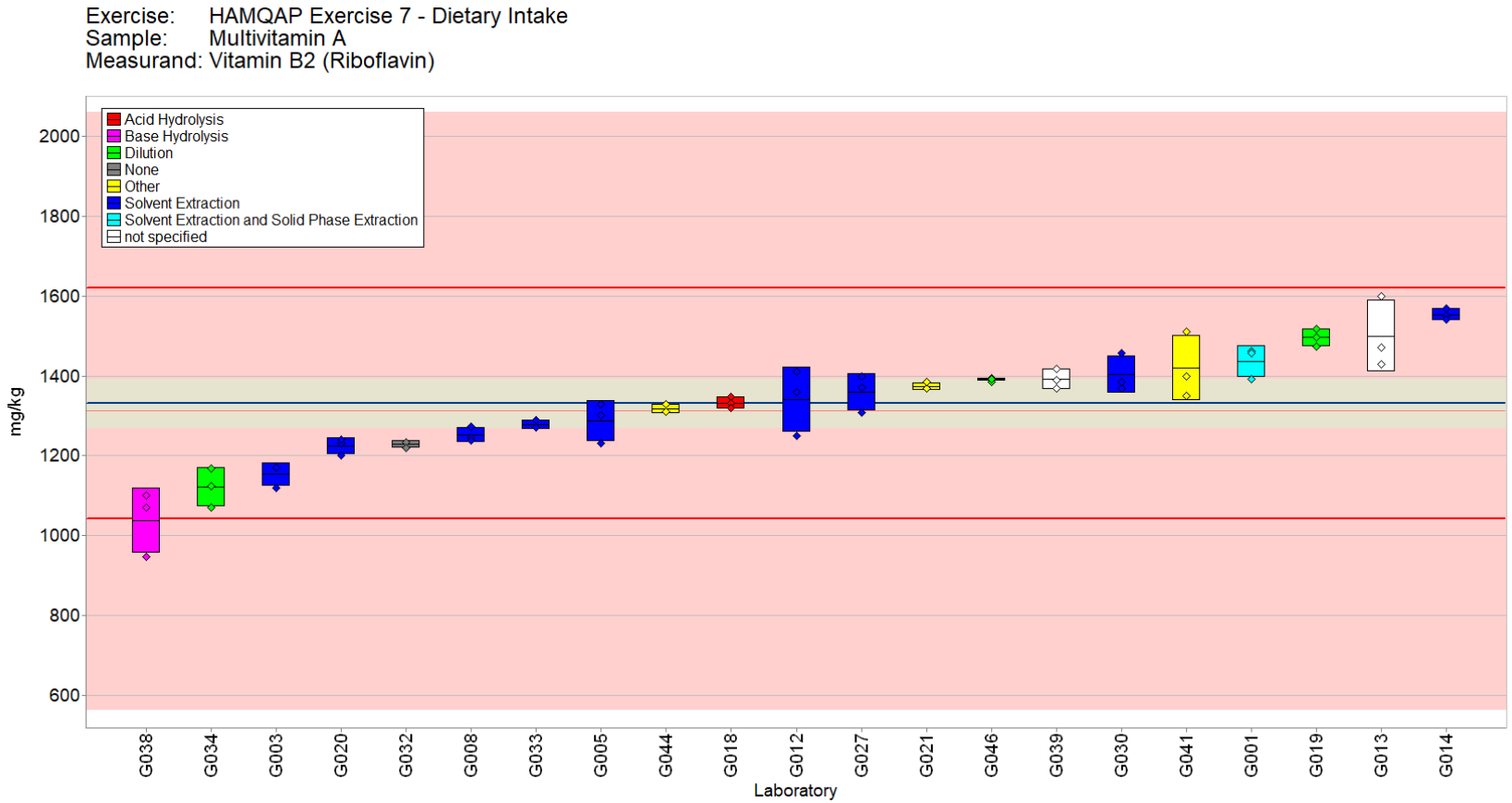

Fig. 3-1. Vitamin B_2 (riboflavin) in Multivitamin A (data summary view – sample preparation method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the sample preparation method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The beige shaded region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

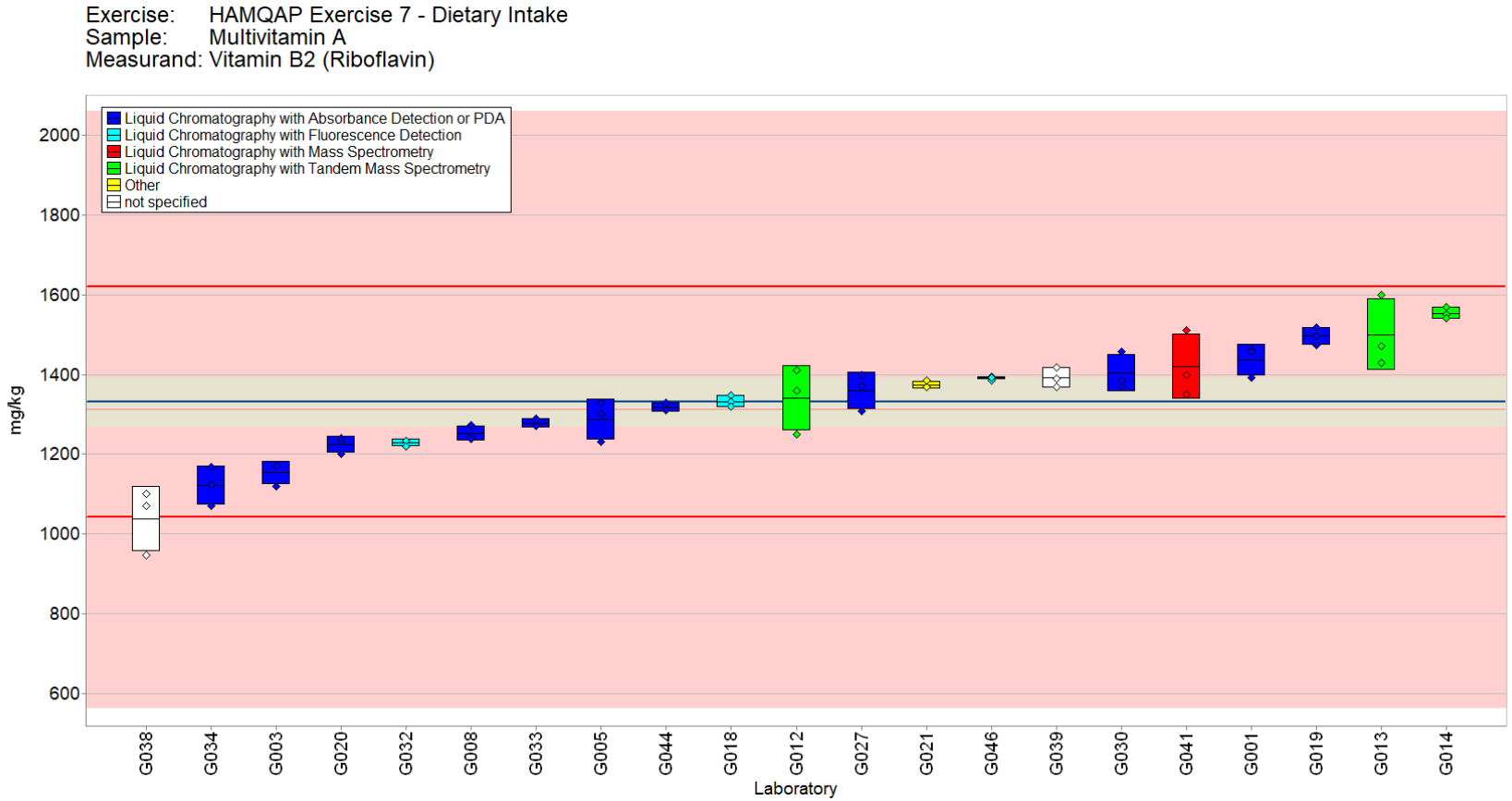

Fig. 3-2. Vitamin B_2 (riboflavin) in Multivitamin A (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The beige shaded region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

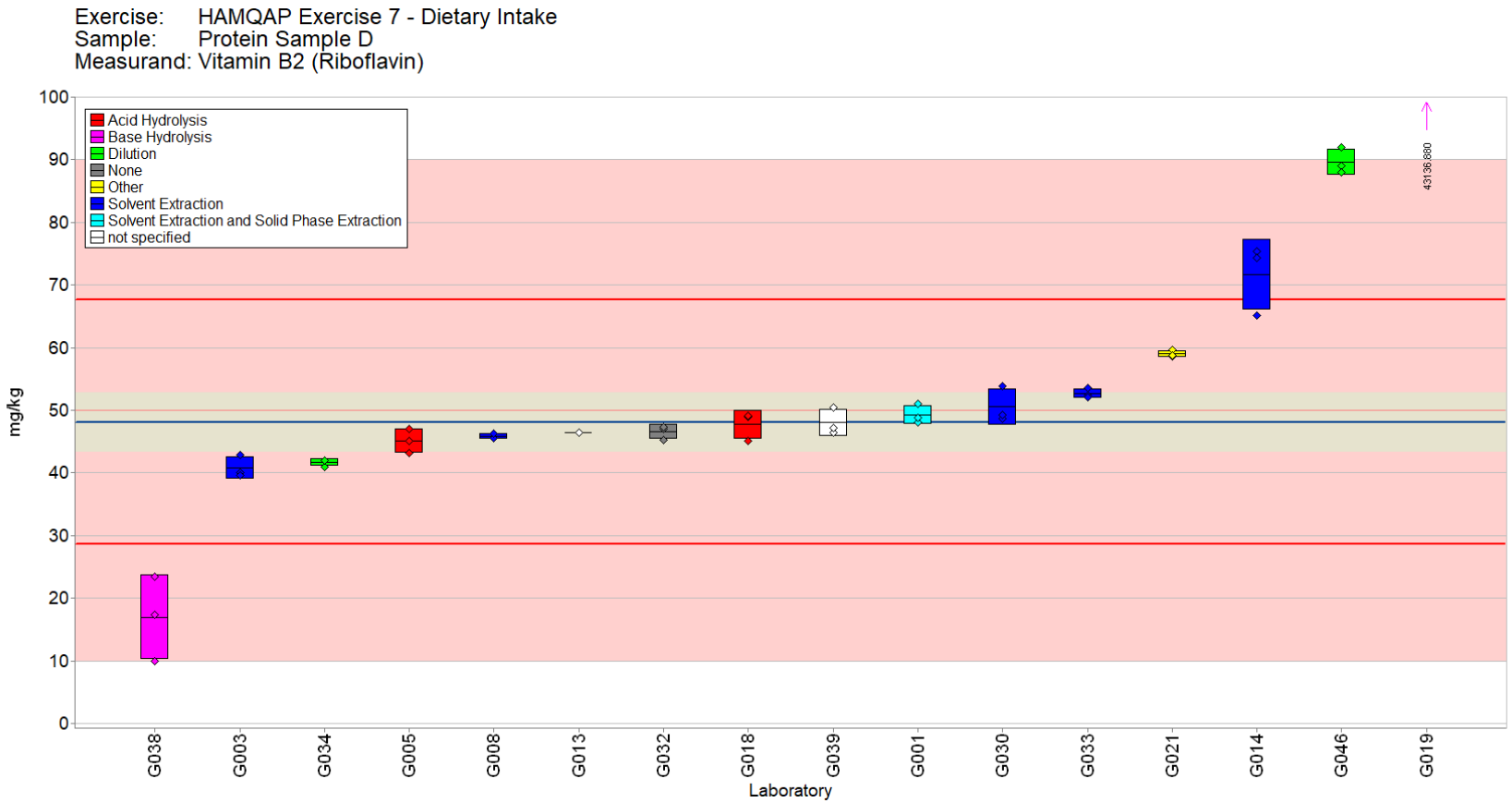

Fig. 3-3. Vitamin B_2 (riboflavin) in Protein Sample D (data summary view – sample preparation method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the sample preparation method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The beige shaded region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

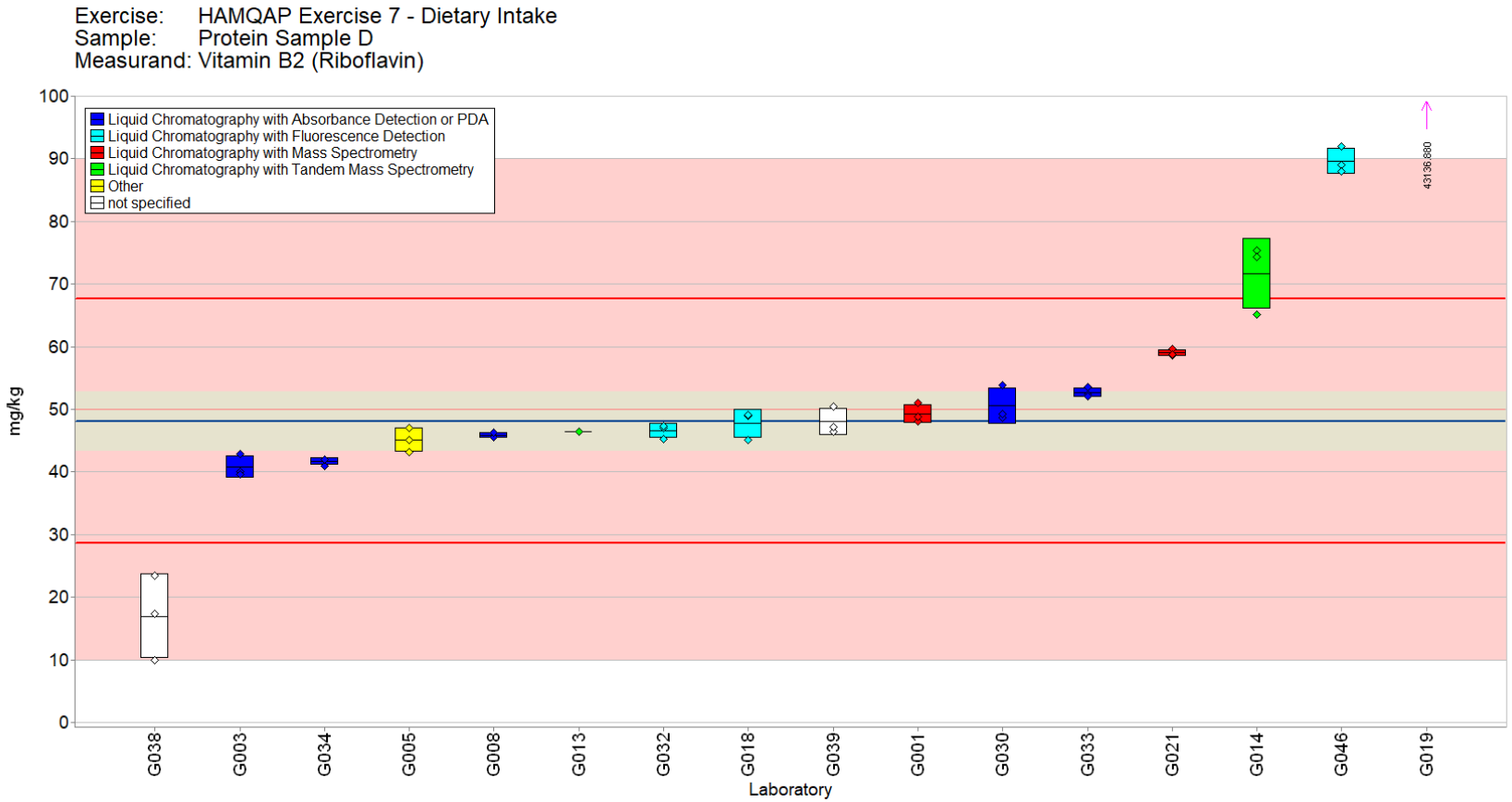

Fig. 3-4. Vitamin B_2 (riboflavin) in Protein Sample D (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The beige shaded region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

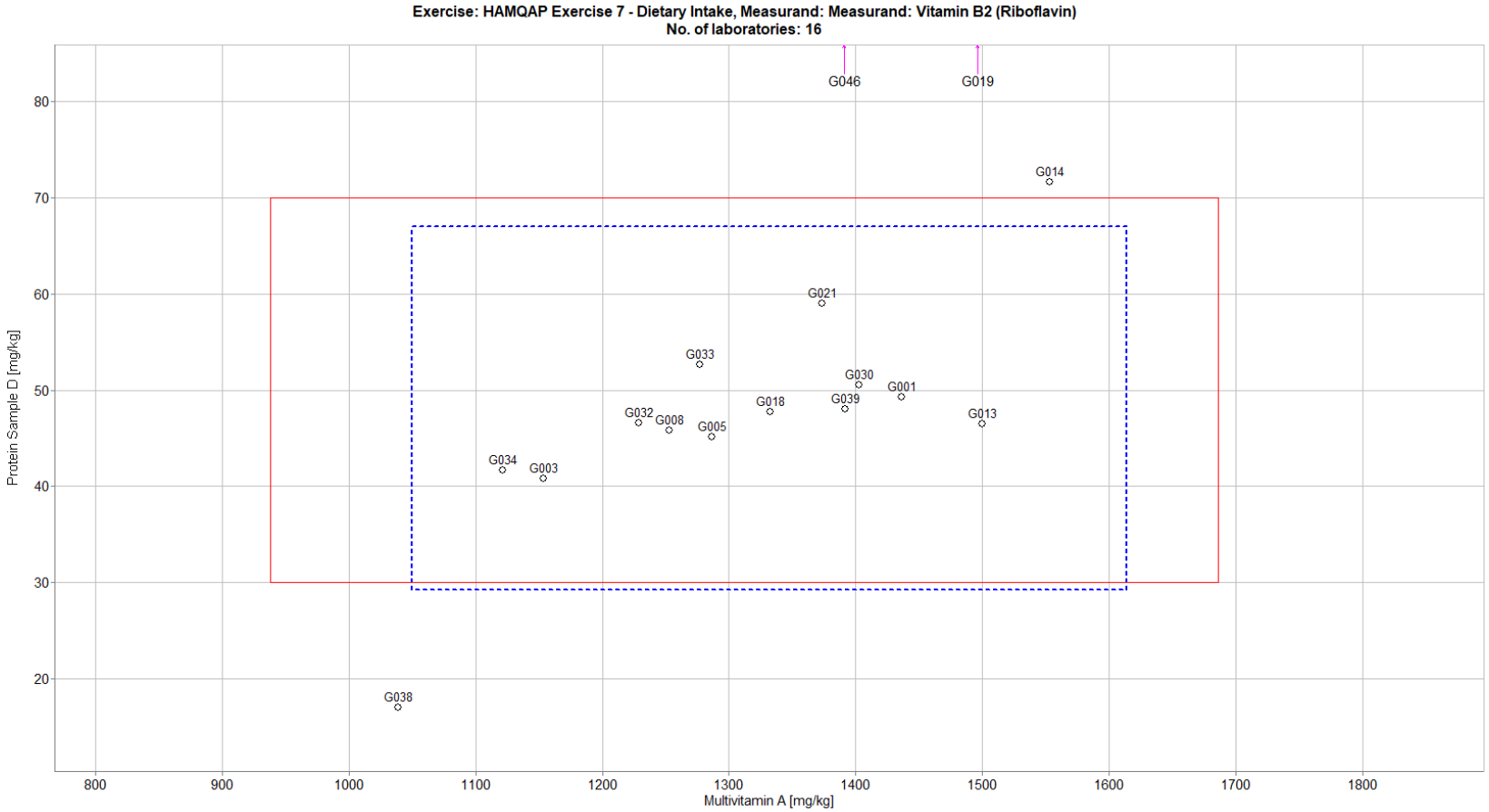

Fig. 3-5. Laboratory means for vitamin B_2 (riboflavin) in Multivitamin A and Protein Sample D (sample/sample comparison view). In this view, the individual laboratory mean for one sample (Multivitamin A) is compared to the mean for a second sample (Protein Sample D). The solid red box represents the NIST range of tolerance for the two samples, Multivitamin A (x-axis) and Protein Sample D (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for Multivitamin A (x-axis) and Protein Sample D (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

Table 3-3. Data summary table for vitamin B_6 (pyridoxine) in multivitamin tablets and protein powder. Data highlighted in blue have been identified as outside the consensus tolerance limits and resulted in an unacceptable Z'_{comm} score, $|Z'_{comm}| > 2$.

		Vitamin B6 (Pyridoxine)									
		Multivitamin A (mg/kg)					Protein Sample D (mg/kg)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				1360	73				60.0	12.0
	G001	1550.38	1546.82	1536.05	1544.4	7.5	92.8	102.66	98.36	97.9	4.9
	G002										
	G003	1710	1720	1620	1683	55	56	51.5	61.6	56.4	5.1
	G005	1430	1450	1480	1453	25	62.2	63	61.1	62.1	1.0
	G006										
	G008	1292	1283	1297	1290.7	7.1	61.77	59.6	72.57	64.6	6.9
	G010										
	G012	1260	1290	1220	1257	35					
	G013	1370	1360	1400	1377	21	53.8			53.8	
	G014	1370	1490	1420	1427	60	59.9	35.7	45.5	47.0	12.2
	G015										
	G016										
	G018	256.9	250.8	256.3	254.7	3.4	107.7	108.5	111.2	109.1	1.8
	G019	1835.68	1829.07	1849.69	1838	11	71.75	64.46	62.61	66.3	4.8
	G020	1490	1466	1499	1485	17					
	G021	1346	1326	1318	1330	14	45.9	46.2	45.8	45.97	0.21
	G023										
	G024										
	G025	1900	1950	1950	1933	29	50	52	54	52.0	2.0
	G026										
	G027	1251.69	1254.45	1193.19	1233	35					
	G028										
	G030	1548	1547.2	1569.1	1555	12	57.7	68.5	47.6	57.9	10.5
	G032	1103.69	1093.14	1164.48	1120	39	48.87	36.33	51	45.4	7.9
	G033	1360	1340	1330	1343	15					
	G034						54	52	64	56.7	6.4
	G036										
	G038	1360	1215	1510	1362	148	11.5	24	19	18.2	6.3
	G039	1224	1220	1194	1213	16	36.84	37.76	39.5	38.0	1.4
	G041	1330	1360	1350	1347	15					
	G044	1270	1300	1250	1273	25					
	G045										
	G046	1800	1770	1743	1771	29	68	57	85	70.0	14.1
	G048										
Community Results		Consensus Mean			1432		Consensus Mean			57.16	
		Consensus Standard Deviation			236		Consensus Standard Deviation			17.87	
		Maximum			1933		Maximum			109.13	
		Minimum			255		Minimum			18.17	
		N			21		N			15	

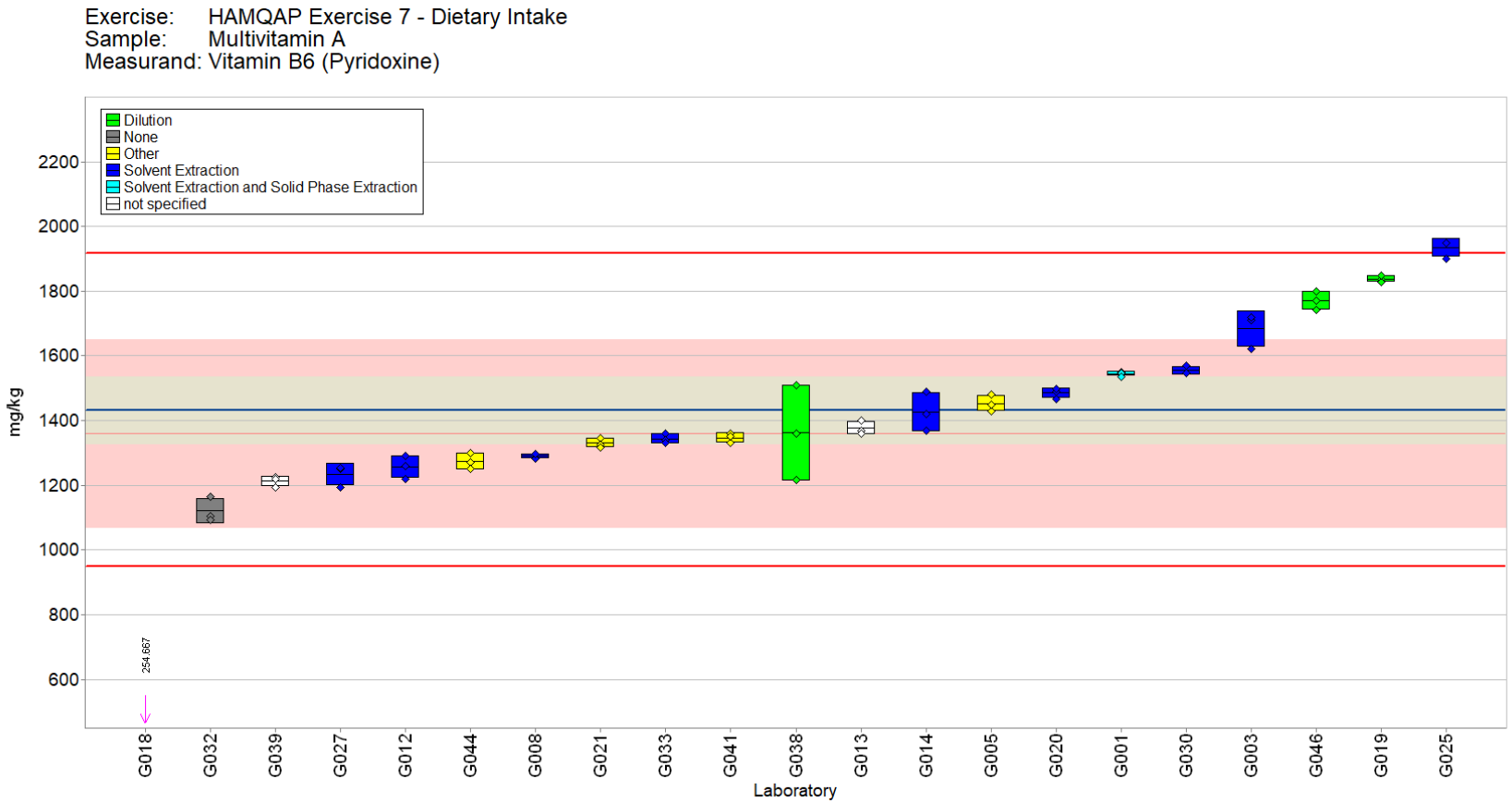

Fig. 3-6. Vitamin B_6 (pyridoxine) in Multivitamin A (data summary view – sample preparation method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the sample preparation method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The beige shaded region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

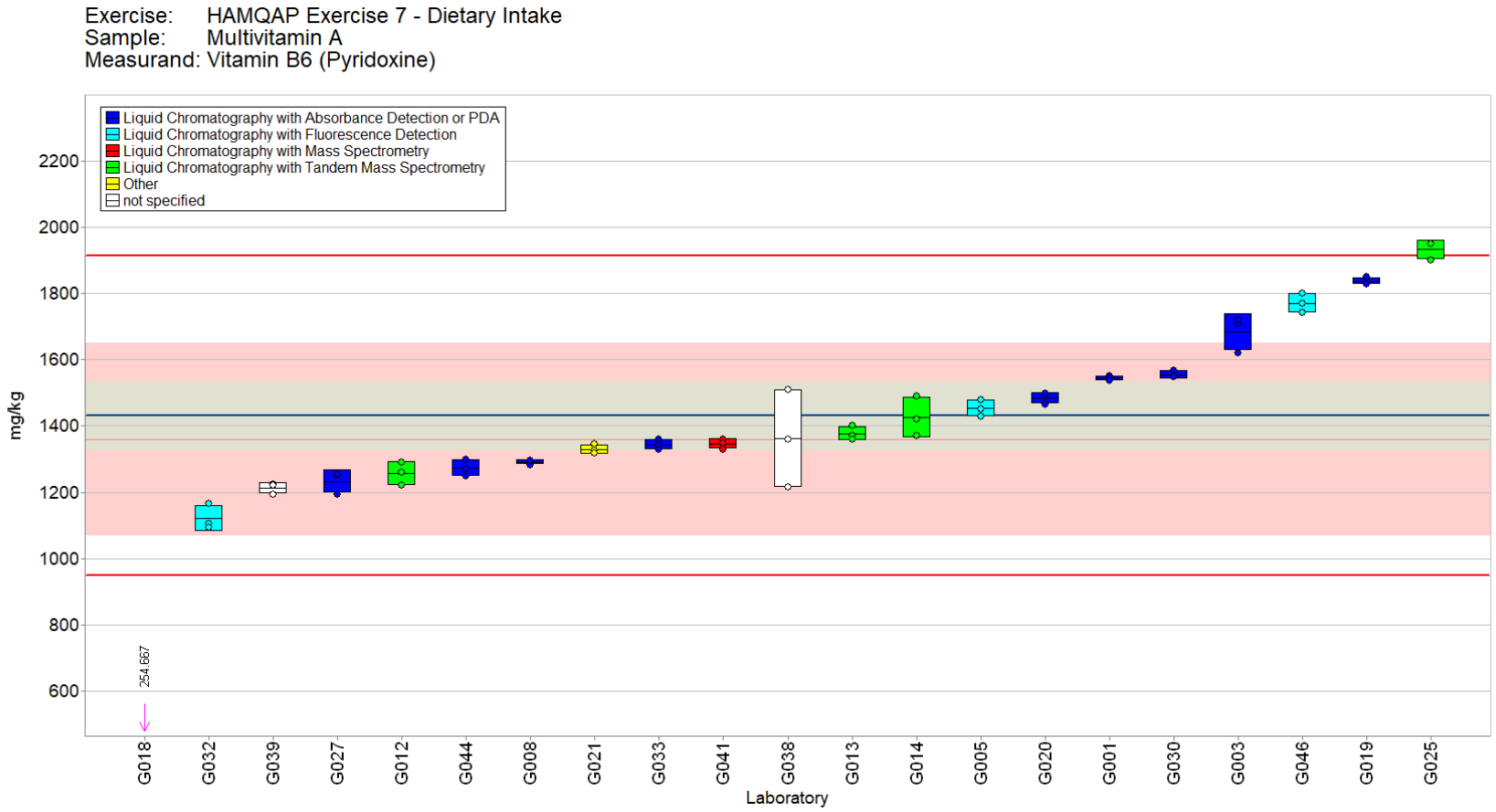

Fig. 3-7. Vitamin B_6 (pyridoxine) in Multivitamin A (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The beige shaded region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

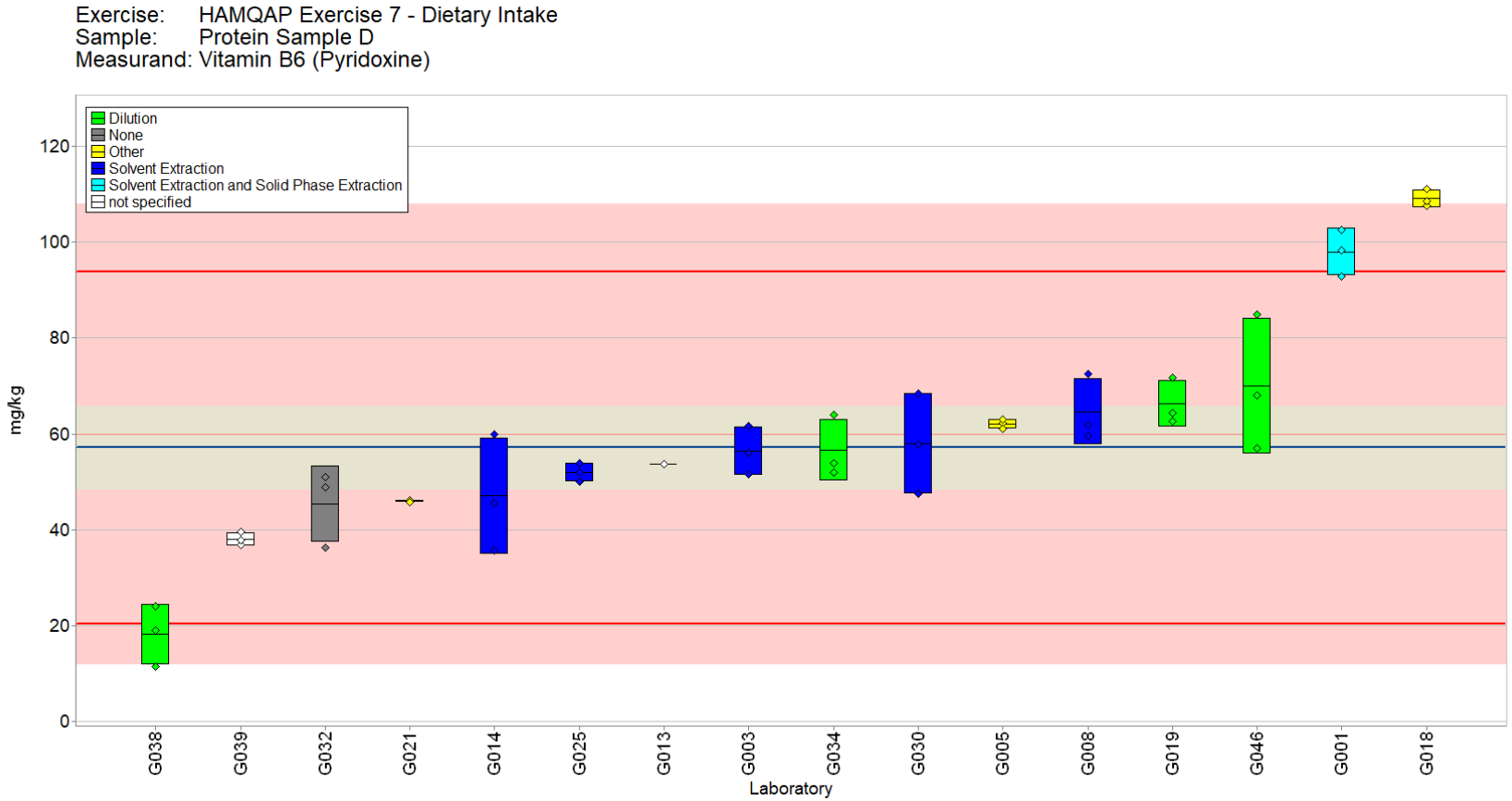

Fig. 3-8. Vitamin B_6 (pyridoxine) in Protein Sample D (data summary view – sample preparation method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the sample preparation method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The beige shaded region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

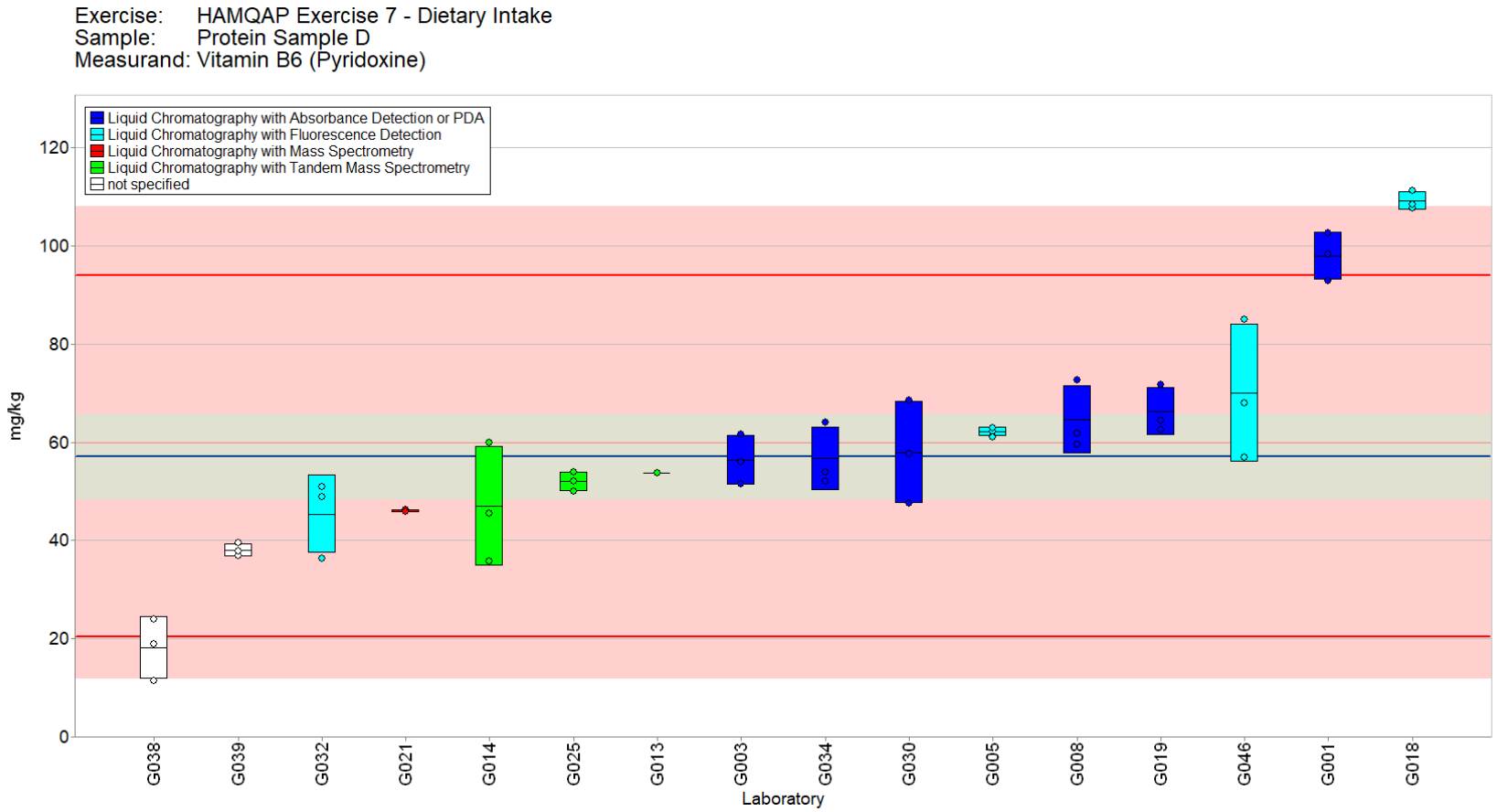

Fig. 3-9. Vitamin B_6 (pyridoxine) in Protein Sample D (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The beige shaded region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

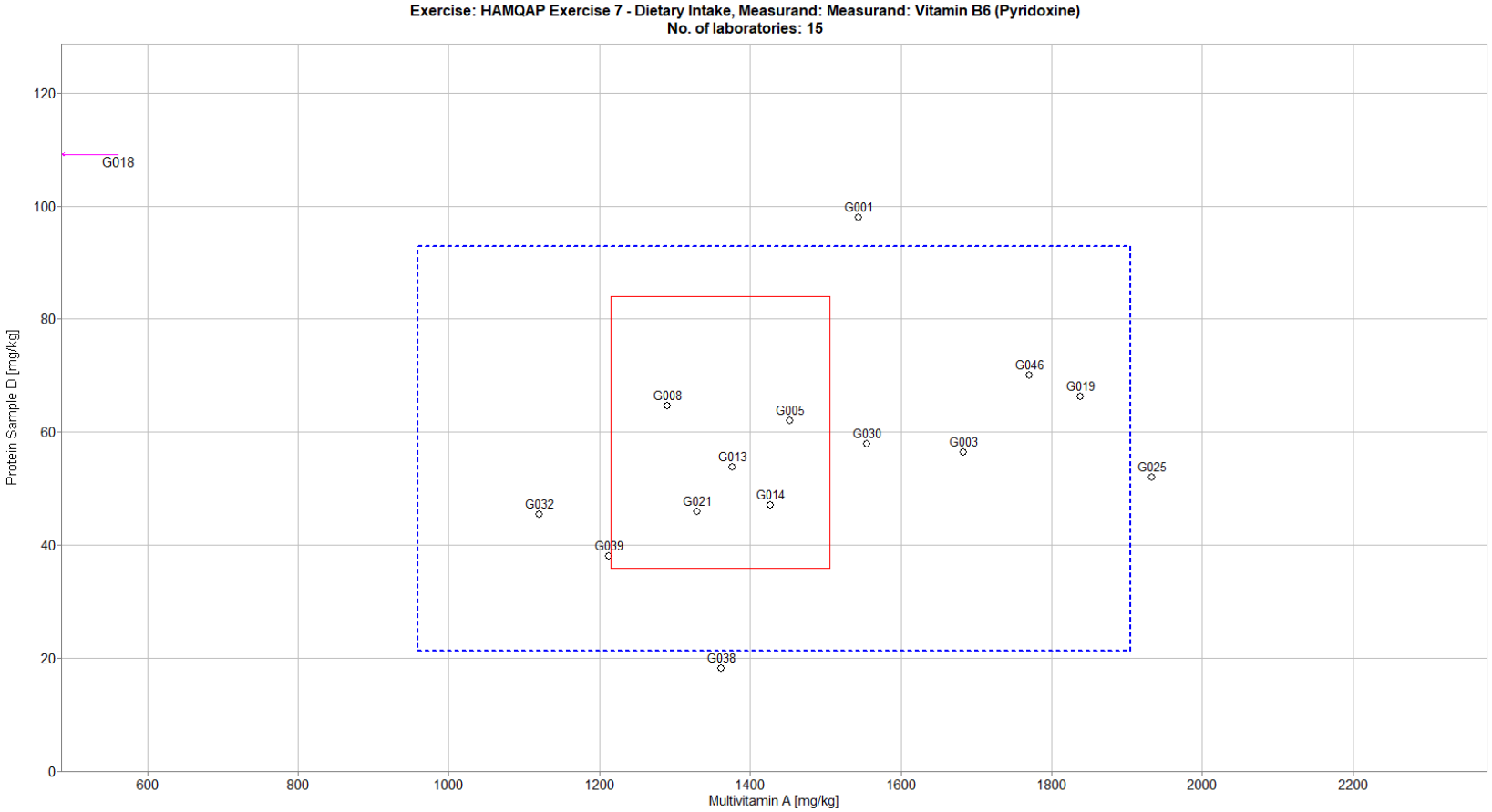

Fig. 3-10. Laboratory means for vitamin B_6 (pyridoxine) in Multivitamin A and Protein Sample D (sample/sample comparison view). In this view, the individual laboratory mean for one sample (Multivitamin A) is compared to the mean for a second sample (Protein Sample D). The solid red box represents the NIST range of tolerance for the two samples, Multivitamin A (x-axis) and Protein Sample D (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for Multivitamin A (x-axis) and Protein Sample D (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

4. Fat-Soluble Vitamins (Vitamin K)

4.1. Study Overview

Vitamin K is a family of fat-soluble vitamins found in some foods and available as a dietary supplement. Vitamin K has important functions in homeostasis and bone metabolism. The naturally occurring compounds include phylloquinone (vitamin K_1) and menaquinones (vitamin K_2), each having multiple forms. Food sources of phylloquinone include vegetables, especially green leafy vegetables, vegetable oils, and some fruits. Meat, dairy foods, and eggs contain low levels of phylloquinone but modest amounts of menaquinones. Fermented foods, such as natto, cheeses, and sauerkraut, can contain high amounts of menaquinones, varying in levels depending on the bacteria present and the fermentation conditions. Vitamin K deficiency can impair blood clotting and has been linked to osteoporosis and coronary heart disease. The population groups most likely to have inadequate vitamin K are newborns not treated with vitamin K at birth and people with malabsorption disorders. Adverse effects of excessive vitamin K intake have not been identified, though the effectiveness of anticoagulant medications that antagonize vitamin K activity (notably Warfarin (Coumadin®)) can be reduced with high vitamin K intake, and certain other medications can reduce vitamin K levels (e.g., antibiotics, bile acid sequestrants). [8] Testing laboratories must use fit-for-purpose methods and standards that can support reliable and accurate measurements for product labeling to prevent adverse outcomes.

In this study, participants were provided with samples of kelp and multivitamin tablets as representative dietary intake samples. Participants were asked to use in-house analytical methods to determine the mass fractions (mg/kg) of vitamin K in several forms in the dietary intake samples on an as-received basis (i.e., not moisture corrected).

4.2. Sample Information

Kelp. Participants were provided with three packets, each containing approximately 5 g of powdered kelp. Participants were asked to store the material at controlled room temperature, 20 °C to 25 °C, to prepare a single sample, and to report a single value from each packet provided. Before use, participants were instructed to mix the contents of each packet thoroughly, allow contents to settle for one minute prior to opening to minimize the loss of fine particles, and to use a sample size appropriate for their usual in-house method of analysis for the determination vitamin K. Approximate analyte levels were not disclosed to participants prior to the study. The target value for total vitamin K_1 (phylloquinone) was determined by results from a previous interlaboratory comparison. [9] The value and uncertainty for total vitamin K_1 provided in the table below on an as-received basis. Target values for cis-vitamin K_1, trans-vitamin K_1, total vitamin K_2, vitamin K_2 MK-4, vitamin K_2 MK-7, and vitamin K_2 MK-9 in the kelp were not available at the time of this report.

Analyte	Target Mass Fraction in Kelp (mg/kg)
Total Vitamin K_1 (phylloquinone)	2.1 ± 1.3

Multivitamin A. Participants were provided with three bottles, each containing 30 multivitamin tablets. Participants were asked to store the material, in the original unopened bottles, at controlled room temperature, 20 °C to 25 °C. Before use, participants were instructed to grind all 30 tablets and mix the resulting powder thoroughly prior to removal of a test portion for analysis, and to use a sample size of at least 2 g for the determination of vitamin K_1. After grinding, participants were instructed to store the resulting powder at –20 °C or colder and analyze the material within two days for analytes in this study. Approximate analyte levels were not disclosed to participants prior to the study. The target value for total vitamin K_1 (phylloquinone) in the multivitamin sample was determined by the manufacturer of the material (n = 10 using LC-FLD). The value and standard deviation for total vitamin K_1 are provided in the table below on an as-received basis. Target values for *cis*-vitamin K_1, *trans*-vitamin K_1, total vitamin K_2, vitamin K_2 MK-4, vitamin K_2 MK-7, and vitamin K_2 MK-9 in the multivitamin were not available at the time of this report. It is also worth noting that vitamin K_2 was not expected in the material based on the production formulation information.

Analyte	Target Mass Fraction in Multivitamin A (mg/kg)
Total Vitamin K_1 (phylloquinone)	16.52 ± 0.34

4.3. Study Results

The participation/enrollment and reporting statistics for each analyte in the dietary intake study is described in the table below. Reported values may include non-quantitative results (zero or below LOQ) that are only included in the participation statistics.

Analyte	Number of Laboratories Requesting Samples	Number of Laboratories Reporting Results (Percent Participation)	
		Kelp	Multivitamin A
Total Vitamin K_1 (phylloquinone)	24	10 (42 %)	14 (58 %)
cis-vitamin K_1	24	1 (4 %)	2 (8 %)
trans-vitamin K_1	22	1 (5 %)	2 (9 %)
Total Vitamin K_2	23	3 (13 %)	4 (17 %)
Vitamin K_2 MK-4	23	5 (22 %)	7 (30 %)
Vitamin K_2 MK-7	25	5 (20 %)	8 (32 %)
Vitamin K_2 MK-9	21	1 (5 %)	2 (10 %)

About half of the laboratories returned results for total vitamin K_1 (phylloquinone), with between-laboratory variabilities of 53 % and 32 % for the kelp and multivitamin, respectively.

Most laboratories that provided sample preparation information reported using solvent extraction. Dilution and solvent extraction with solid phase extraction preparation techniques were also reported. The reported sample preparation methods are listed below.

Reported Sample Preparation Method	Percent Reporting % (Averaged for both sample types) Total Vitamin K_1 (phylloquinone)	Vitamin K_2 MK-4
Solvent Extraction	55 %	46 %
Dilution	10 %	18 %
Solvent Extraction and Solid Phase Extraction	10 %	-
Other/None	25 %	36 %

An even distribution of analytical methods was reported for the determination of vitamin K, with 50 % to 55 % reporting LC with spectrophotometric detection (Abs or FLD), and 35 % to 42 % reporting LC with mass spectrometric detection (MS or MS/MS). The remaining participants did not report analytical method information.

Reported Analytical Method	Percent Reporting % (Averaged for both sample types) Total Vitamin K_1 (phylloquinone)	Vitamin K_2 MK-4
LC-Abs	30 %	33 %
LC-MS	15%	25 %
LC-MS/MS	20 %	17 %
LC-FLD	25 %	17 %
Other/None	10 %	8 %

For the determination of total vitamin K_1 (phylloquinone) in both kelp and multivitamin, all but one laboratory was within the NIST range of tolerance, and the consensus ranges were also within the NIST ranges of tolerance.

4.4. Fat-Soluble Vitamins Technical Recommendations

The following recommendations are based on results obtained from the participants in this study. Additional overall technical recommendations can be found on page 6. Due to the low response for other measurands, only figures for total vitamin K_1 (phylloquinone) are provided.

- Most participants can measure total vitamin K_1 in kelp and multivitamin materials. Based on sample preparation techniques and analytical methods reported, no method bias was observed.
- Other than total vitamin K_1, the participation rates were low and, as a result, meaningful observations could not be made for these measurands. The low participation may be due to the low levels present in the materials or the lack of established methods for measuring isomers of vitamin K_1 and vitamin K_2. The multivitamin material was also not expected to contain vitamin K_2.
- For fat-soluble vitamins, especially those with multiple unique chemical forms, the analytes being measured and reported must be understood. Pure standards of different forms (i.e., isomers) can be difficult to obtain. Access to high quality and well-characterized calibrants can reduce measurement biases and misinterpretation of results.
- Vitamin K_1 may be reported as a total, or as the cis- and trans-isomers. Some analytical methods partially or completely separate the isomers, and components can be measured both individually and as a sum to determine total vitamin K_1. Other methods in which the isomers coelute can only be used for reporting total vitamin K_1. For understanding and assessment of vitamin bioactivity, methods must be able to separate and quantify individual forms (including isomers).
- While sample preparation techniques must be able to fully extract the analytes from the sample matrix, analysts must also be mindful of analyte degradation and/or conversion. The use of reduced lighting/yellow lighting and storage of materials in the dark (or in amber colored vials) can significantly reduce UV-induced analyte degradation.

Table 4-1. Individualized data table (NIST) for vitamin K in kelp and multivitamin tablets.

Exercise 7 - Fat-Soluble Vitamins

		Lab Code:	**NIST**	**1. Your Results**				**2. Community Results**			**3. Target**	
Analyte	Sample	Units		x_i	s_i	Z'_{comm}	Z_{NIST}	N	x*	s*	x_{NIST}	*U*
Total Vitamin K1 (phylloquinone)	Kelp	mg/kg		2.1	1.3			10	1.9	1.0	2.1	1.3
Total Vitamin K1 (phylloquinone)	Multivitamin A	mg/kg		16.52	0.34			14	14.3	4.6	16.52	0.34
cis-vitamin K1	Kelp	mg/kg						1				
cis-vitamin K1	Multivitamin A	mg/kg						2				
trans-vitamin K1	Kelp	mg/kg						1				
trans-vitamin K1	Multivitamin A	mg/kg						2				
Total Vitamin K2	Kelp	mg/kg						3				
Total Vitamin K2	Multivitamin A	mg/kg						4				
Vitamin K2 MK-4	Kelp	mg/kg						5	1.7	3.8		
Vitamin K2 MK-4	Multivitamin A	mg/kg						7	0.42	0.59		
Vitamin K2 MK-7	Kelp	mg/kg						5				
Vitamin K2 MK-7	Multivitamin A	mg/kg						8				
Vitamin K2 MK-9	Kelp	mg/kg						1				
Vitamin K2 MK-9	Multivitamin A	mg/kg						2				

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

Table 4-2. Data summary table for total vitamin K_1 (phylloquinone) in kelp and multivitamin tablets. Data points highlighted in blue have been identified as outside the consensus tolerance limits and resulted in an unacceptable Z'_{comm} score, $|Z'_{\text{comm}}| \geq 2$.

		Total Vitamin K1 (phylloquinone)									
		Kelp (mg/kg)					Multivitamin A (mg/kg)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				2.1	1.3				16.52	0.34
	G003						11	9.75	9.63	10.13	0.76
	G006										
	G008										
	G010										
	G012	1.66	1.83	1.72	1.74	0.09	9.88	10.2	10.2	10.09	0.18
	G013	1.96	2.19	1.94	2.03	0.14	14.3	15.2	15.1	14.87	0.49
	G014	2.47	2.62	2.47	2.520	0.087	12.4	12.2	11.8	12.13	0.31
	G016										
	G019	3.16	2.3	2.6	2.69	0.44	19.69	17.7	17.85	18.4	1.1
	G021	0.66	0.61	0.65	0.640	0.026	10.1	10.2	10.3	10.20	0.10
	G024										
	G027	2.1491	1.8031	2.0496	2.00	0.18	14.7658	14.9077	14.6816	14.79	0.11
	G028										
	G030	2.21	2.46	2.71	2.46	0.25	13.11	12.8	12.31	12.74	0.40
	G032						16.544	15.23	15.208	15.66	0.77
	G034	30.6	30.9	31.5	31.00	0.46	1086.4	1428.8	643.6	1053	394
	G036										
	G038	2.42	2.64	2.15	2.40	0.25	15.7	14.7	15.4	15.27	0.51
	G041						14.3	14.8	14.9	14.67	0.32
	G042	0.07	0.08	0.08	0.077	0.006	23.6	29.5	21.7	24.9	4.1
	G044						15.7	17.4	15.7	16.27	0.98
	G046										
	G048										
Community Results		Consensus Mean			1.9		Consensus Mean			14.34	
		Consensus Standard Deviation			1.0		Consensus Standard Deviation			4.59	
		Maximum			31.00		Maximum			1053	
		Minimum			0.08		Minimum			10.09	
		N			10		N			14	

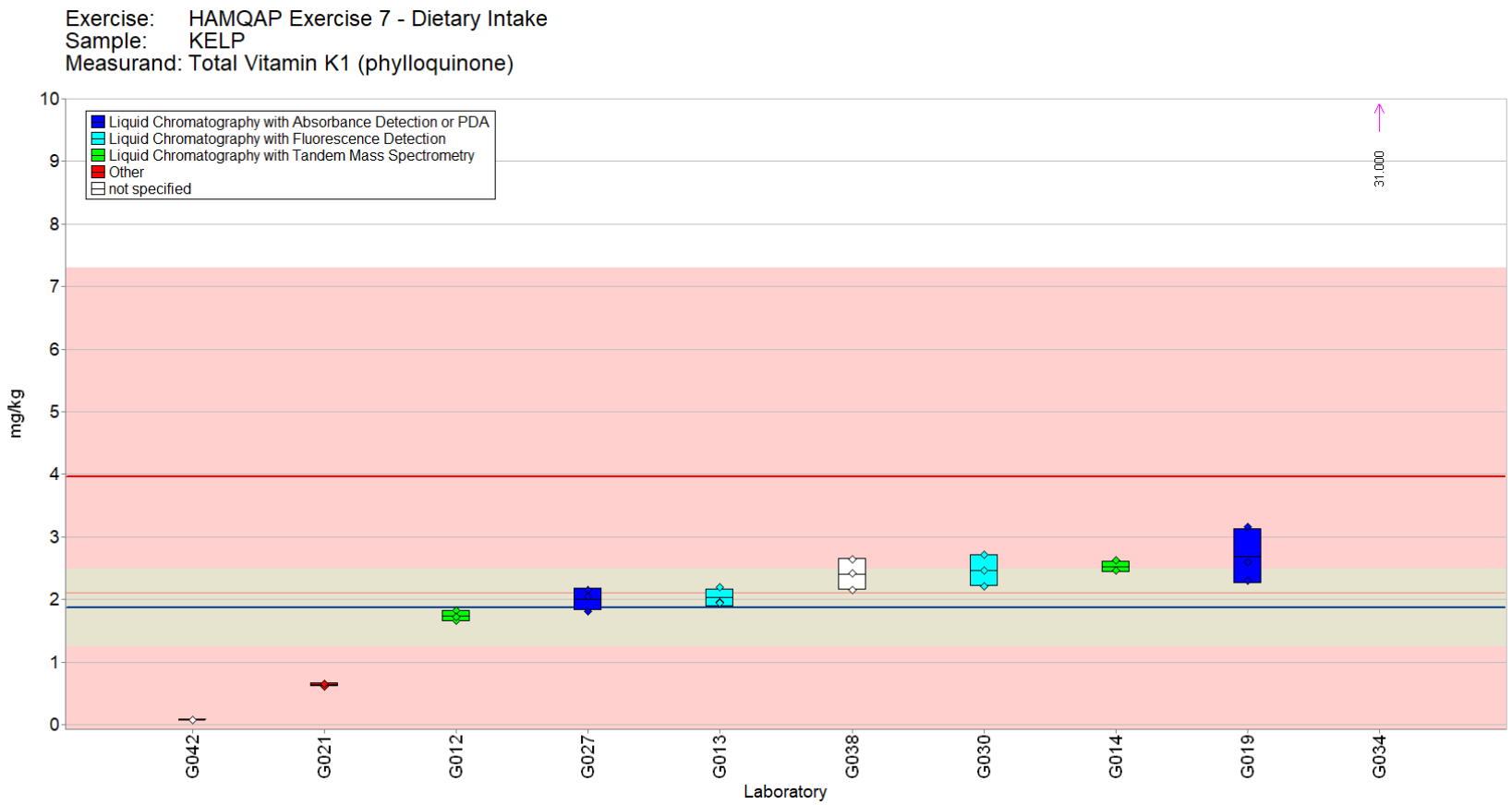

Fig. 4-1. Total Vitamin K_1 (Phylloquinone) in Kelp (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$, with the lower range set at zero. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The beige shaded region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

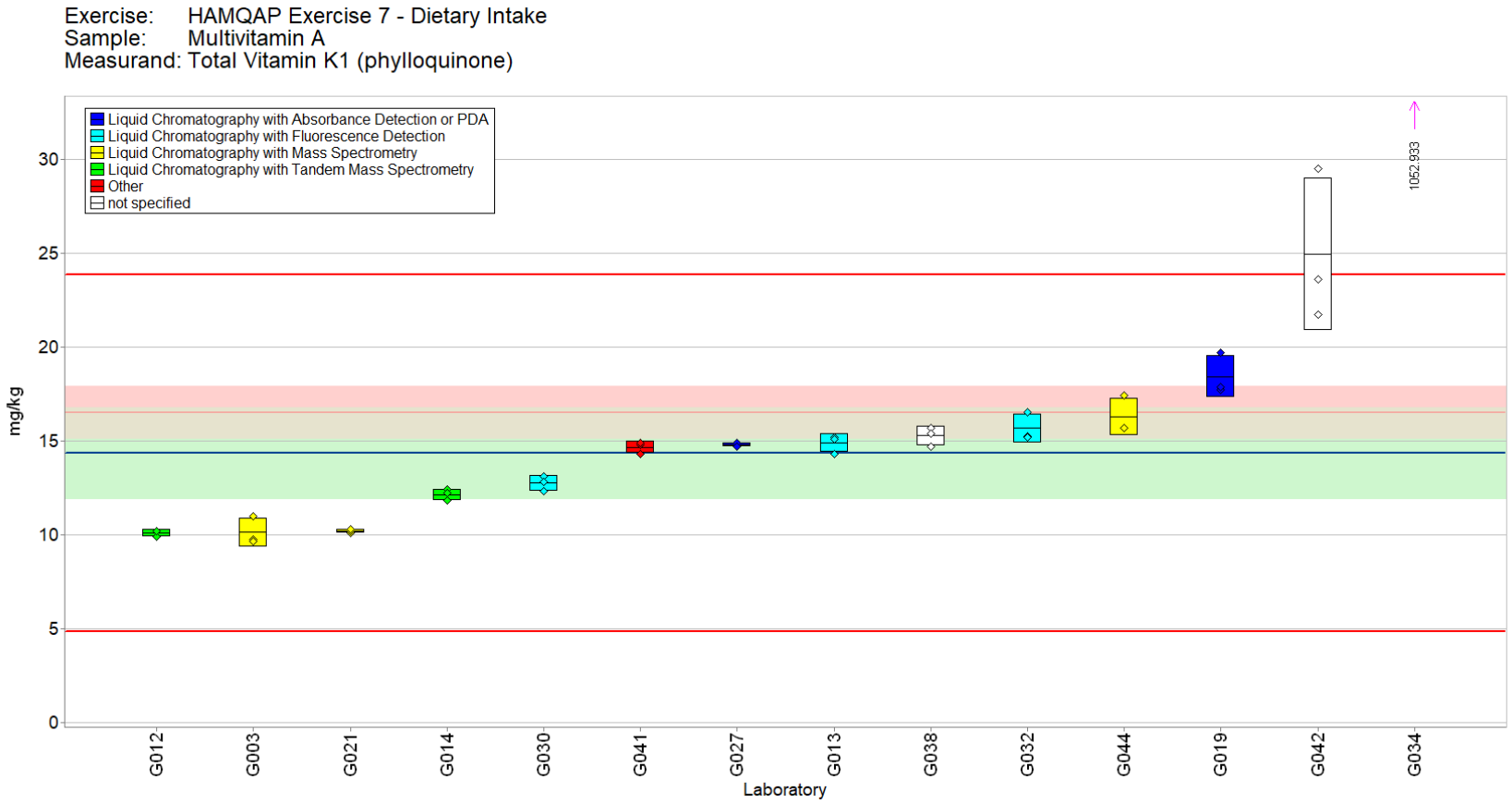

Fig. 4-2. Total Vitamin K_1 (Phylloquinone) in Multivitamin A (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The beige shaded region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

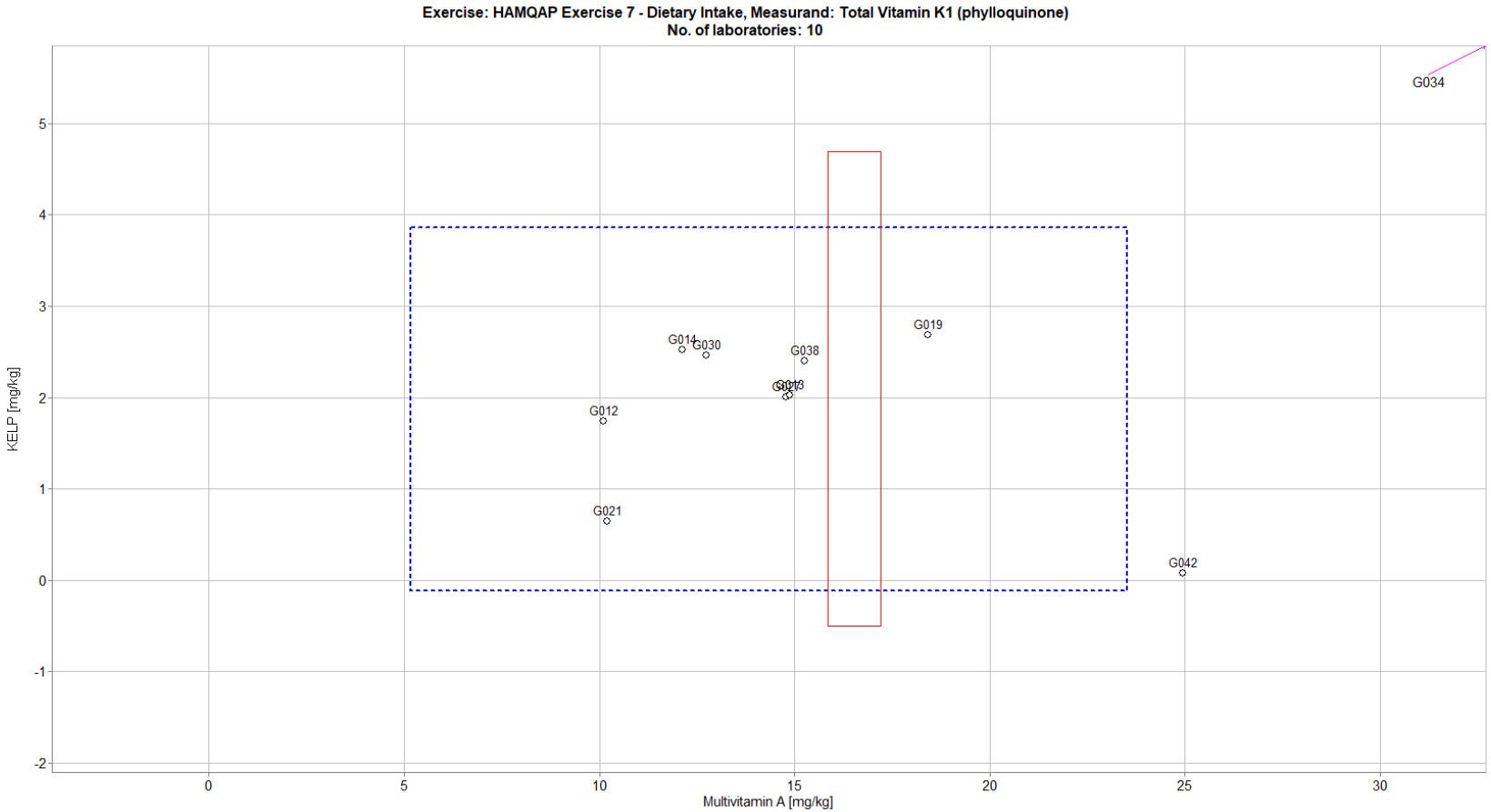

Fig. 4-3. Laboratory means for Total Vitamin K_1 (Phylloquinone) in Kelp and Multivitamin A (sample/sample comparison view). In this view, the individual laboratory mean for one sample (multivitamin) is compared to the individual laboratory mean for a second sample (kelp). The solid red box represents the NIST range of tolerance for the two samples, multivitamin (x-axis) and kelp (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for multivitamin (x-axis) and kelp (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

Table 4-3. Data summary table for total vitamin K_2 in kelp and multivitamin tablets.

		Total Vitamin K2									
		Kelp (mg/kg)					Multivitamin A (mg/kg)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target										
	G003										
	G006										
	G008										
	G010										
	G013	< 0.149	< 0.149	< 0.149							
	G014										
	G016										
	G019										
	G020						14.5	14.56	14.61	14.56	0.06
	G021										
	G027	0.8289	0.7237	0.8283	0.794	0.061					
	G028										
	G030										
	G032										
	G034										
	G036										
	G038										
	G041						< 3.16	< 3.16	< 3.16		
	G042	< 0.05	< 0.05	< 0.05			< 0.05	< 0.05	< 0.05		
	G044						< 100	< 100	< 100		
	G046										
	G048										
Community Results		Consensus Mean					Consensus Mean				
		Consensus Standard Deviation					Consensus Standard Deviation				
		Maximum			0.794		Maximum			14.56	
		Minimum			0.794		Minimum			14.56	
		N			1		N			1	

Table 4-4. Data summary table for vitamin K_2 MK-4 in kelp and multivitamin tablets. Data points highlighted in red have a zero or a non-numeric data point.

		Vitamin K2 MK-4									
		Kelp (mg/kg)					Multivitamin A (mg/kg)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target										
	G003						< 1.25	< 1.25	< 1.25		
	G006										
	G008										
	G010										
	G014	< 1	< 1	< 1			< 1	< 1	< 1		
	G016										
	G019	2.54	2.46	2.84	2.61	0.20	0.53	0.59	0.57	0.563	0.031
	G020										
	G021						0.29	0.28	0.26	0.277	0.015
	G024										
	G027	0.8289	0.7237	0.8283	0.794	0.061					
	G028										
	G030	<	<	<							
	G032										
	G034										
	G036										
	G038										
	G041						< 3.16	< 3.16	< 3.16		
	G042	< 0.05	< 0.05	< 0.05			< 0.05	< 0.05	< 0.05		
	G044						< 50	< 50	< 50		
	G046										
	G048										
Community Results		Consensus Mean			1.70		Consensus Mean			0.420	
		Consensus Standard Deviation			3.75		Consensus Standard Deviation			0.588	
		Maximum			2.61		Maximum			0.563	
		Minimum			0.794		Minimum			0.277	
		N			2		N			2	

5. Botanicals (Gingerols)

5.1. Study Overview

Ginger (*Zingiber officinale*) is a leafy plant native to Asia and the rhizome has been used for medicinal and culinary purposes for thousands of years. As a dietary supplement, ginger has been widely studied for the relief and prevention of nausea and vomiting. [10, 11] Gingerols, the major phytochemical constituents of ginger, have been investigated for anticancer, anti-inflammatory, anti-fungal, antioxidant, neuroprotective, and gastroprotective properties. [12] Raw ginger contains high levels of gingerols, which are thermally labile compounds that form shogaols, paradols, and zingerone upon heating or drying of ginger. Accurate determination of these compounds in foods or supplements is important to ensure product quality and to facilitate standardization for clinical investigations of health effects.

In this study, participants were provided with samples of SRM 3398 Ginger (*Zingiber officinale*) Rhizome and RM 8666 Ginger (*Zingiber officinale*) Extract. Participants were asked to use either their in-house analytical methods or AOAC First Action *Official Method* 2018.04 to determine the mass percent (% w/w) of select gingerols and shogaols, and "Total Ginger Constituents" as a sum of the determined measurands. In addition to these two samples, participants that indicated intent to follow the AOAC 2018.04 also received a ginger constituent mixture (USP Catalog # 1291446), powdered ginger (USP Catalog # 1291504), and four commercial ginger-containing supplements. Laboratories that indicated intent to use AOAC 2018.04 were also provided a copy of the method and offered the opportunity to request and receive method consumables (LC column and guard column from Phenomenex, analyte standards from ChromaDex). The data collected from participants using AOAC 2018.04 will be used to evaluate method reproducibility and assist in the multi-laboratory validation of the method.

5.2. Sample Information

Ginger Rhizome. Participants were provided with three packets, each containing 1.6 g of SRM 3398 Ginger (*Zingiber officinale*) Rhizome. Participants were asked to store the material at controlled room temperature, 20 °C to 25 °C, to prepare one sample, and report one value from each packet provided. Before use, participants were instructed to mix the contents of the packet thoroughly, allow contents to settle for one minute prior to opening to minimize the loss of fine particles, and to use a sample size at least 0.5 g to determine the mass percent (% w/w) of select gingerols and shogaols. Participants indicating the intent to use AOAC 2018.04 were asked to refer to the method instructions for recommended sample sizes. The approximate analyte levels were not disclosed to participants prior to the study. The target values for 6-gingerol, 8-gingerol, 10-gingerol, 6-shogaol, 8-shogaol, and 10-shogaol in SRM 3398 were determined at NIST using liquid chromatography with UV absorbance detection (LC-Abs). Total ginger constituents is determined as the sum of the measured gingerols and shogaols. The values and uncertainties are provided in the table below, in % w/w on an as-received basis accounting for the moisture content of the material (7.05 %) and in mg/g on a dry-mass basis from the COA at the time of this report.

Gingerols and Shogaols in SRM 3398

Analyte	Target Value Mass Percent (% w/w)	COA Value Mass Fraction (mg/g)
Total Ginger Constituents	0.9392 ± 0.0076	10.104 ± 0.082
6-Gingerol	0.3643 ± 0.0055	3.919 ± 0.059
8-Gingerol	0.0574 ± 0.0012	0.618 ± 0.013
10-Gingerol	0.0831 ± 0.0017	0.894 ± 0.018
6-Shogaol	0.2515 ± 0.0040	2.706 ± 0.043
8-Shogaol	0.0682 ± 0.0020	0.734 ± 0.021
10-Shogaol	0.1146 ± 0.0020	1.233 ± 0.021

Ginger Extract. Participants were provided with three packets, each containing 3 g of RM 8666 Ginger (*Zingiber officinale*) Extract. Participants were asked to store the material at controlled room temperature, 20 °C to 25 °C, to prepare one sample, and to report one value from each packet provided. Before use, participants were instructed to mix the contents of the packet thoroughly, allow contents to settle for one minute prior to opening to minimize the loss of fine particles, and to use a sample size at least 0.25 g to determine the mass percent (% w/w) of select gingerols and shogaols. Participants indicating the intent to use AOAC 2018.04 were asked to refer to the method instructions for recommended sample sizes. The approximate analyte levels were not disclosed to participants prior to the study. The target for 6-gingerol, 8-gingerol, 10-gingerol, 6-shogaol, 8-shogaol, and 10-shogaol in RM 8666 were determined at NIST using LC-Abs. Total ginger constituents is determined as the sum of the measured gingerols and shogaols. The values and uncertainties are provided in the table below in % w/w on an as-received basis accounting for the moisture content of the material (6.71 %) and in mg/g on a dry-mass basis from the COA at the time of this report.

Gingerols and Shogaols in RM 8666

Analyte	Target Value Mass Percent (% w/w)	COA Value Mass Fraction (mg/g)
Total Ginger Constituents	3.791 ± 0.038	40.64 ± 0.41
6-Gingerol	2.230 ± 0.036	23.90 ± 0.39
8-Gingerol	0.3551 ± 0.0076	3.806 ± 0.082
10-Gingerol	0.4432 ± 0.0052	4.751 ± 0.056
6-Shogaol	0.5181 ± 0.0070	5.554 ± 0.075
8-Shogaol	0.0914 ± 0.0031	0.980 ± 0.033
10-Shogaol	0.1535 ± 0.0035	1.645 ± 0.038

Participants intending to follow AOAC First Action *Official Method* 2018.04 were provided six additional samples and asked to refer to AOAC 2018.04 method instructions for recommended sample sizes for each of the materials.

Ginger Mixture. Participants provided with one bottle containing 0.6 mg of ginger constituent mixture (USP Catalog # 1291446 [13]). Participants were asked to store the material at controlled room temperature, 20 °C to 25 °C, to prepare three samples, and to report three values from the single bottle provided. Before use, participants were instructed to mix the contents of the bottle thoroughly, allow contents to settle for one minute prior to opening to minimize the loss of fine particles, and to refer to AOAC 2018.04 method instructions for recommended sample sizes. The approximate analyte levels were not disclosed to participants prior to the study. The target values and uncertainties (10 % of target value) for 6-gingerol and 6-shogaol in the ginger mixture were determined by USP [14] and are provided in the table below.

Gingerols and Shogaols in USP Ginger Mixture

Analyte	Target Value Mass Percent (% w/w)		
6-Gingerol	8.70	±	0.87
6-Shogaol	12.3	±	1.2

Ginger Powder. Participants were provided with one bottle containing approximately 500 mg of powdered ginger (USP Catalog # 1291504 [15]). Participants were asked to store the material at controlled room temperature, 20 °C to 25 °C, and to prepare three samples and report three values from the single bottle provided. Before use, participants were instructed to mix the contents of the bottle thoroughly, allow contents to settle for one minute prior to opening to minimize the loss of fine particles, and to refer to AOAC 2018.04 method instructions for recommended sample sizes. The approximate analyte levels were not disclosed to participants prior to the study. The target values for 6-gingerol, 8-gingerol, 10-gingerol, 6-shogaol, 8-shogaol, 10-shogaol, and 6-paradol in the ginger powder were determined by a collaborating laboratory using AOAC 2018.04. The target values and uncertainties, determined using the reported intermediate precision of the method, are provided in the table below.

Gingerols and Shogaols in USP Ginger Powder

Analyte	Target Value Mass Percent (% w/w)		
Total Ginger Constituents	1.074	±	0.027
6-Gingerol	0.5541	±	0.0077
8-Gingerol	0.1397	±	0.0095
10-Gingerol	0.1764	±	0.0069
6-Shogaol	0.1161	±	0.0035
8-Shogaol	0.0250	±	0.0019
10-Shogaol	0.0482	±	0.0019
6-Paradol	0.0146	±	0.0016

Supplement A. Participants were provided with three packets, each containing 10 ginger tablets. The pressed tablets each contained approximately 150 mg of ginger root extract, as well as inactive ingredients including croscarmellose sodium and lactose monohydrate. Participants were asked to store the material at controlled room temperature, 20 °C to 25 °C, to prepare one sample, and to

report one value from each packet provided. Before use, participants were instructed to grind all 10 tablets and mix the resulting powder thoroughly prior to removal of a test portion for analysis. The approximate analyte levels were not disclosed to participants prior to the study, and target values were not available for these materials at the time of this report.

Supplement B. Participants were provided with three packets, each containing 10 ginger capsules. These plant-derived capsules each contained approximately 0.55 g of ground ginger root. Participants were asked to store the material at controlled room temperature, 20 °C to 25 °C, to prepare one sample, and to report one value from each packet provided. Before use, participants were instructed to combine the contents of all 10 capsules (remove capsules shells) and mix the resulting powder thoroughly prior to removal of a test portion for analysis. The approximate analyte levels were not disclosed to participants prior to the study, and target values were not available for these materials at the time of this report.

Supplement C. Participants were provided with three packets, each containing 10 ginger softgel capsules. These softgel capsules each contained approximately 250 mg of an extract blend composed of ginger oil (gingerols and shogaols) and turmeric oil (turmerones). Participants were asked to store the material at controlled room temperature, 20 °C to 25 °C, to prepare one sample, and to report one value from each packet provided. Before use, participants were instructed to mix and blend all 10 softgel capsules thoroughly and then use an appropriate tool to transfer resulting liquid prior to removal of a test portion for analysis. The approximate analyte levels were not disclosed to participants prior to the study, and target values were not available for these materials at the time of this report.

Supplement D. Participants were provided with one bottle containing 30 mL of an ethanolic ginger tincture. This tincture contained approximately 800 mg of ginger rhizome extract per 1 mL of tincture. Participants were asked to store the material at controlled room temperature, 20 °C to 25 °C, in the original unopened bottle, to prepare three samples, and report to three values from the single bottle provided. Before use, participants were instructed to thoroughly mix the contents of the bottle prior to removal of a test portion for analysis. The approximate analyte levels were not disclosed to participants prior to the study, and target values were not available for these materials at the time of this report.

5.3. Study Results

Twenty-one laboratories enrolled in the gingerols study and received SRM 3398 Ginger Rhizome and RM 8666 Ginger Extract. Eleven of these laboratories indicated intent to use AOAC 2018.04 and received 6 additional ginger-containing samples. The enrollment and reporting statistics for the botanicals study are described in the tables below. One laboratory was unable to receive samples due to import customs issues and was therefore not included in the participation statistics. Some of the reported values were non-quantitative (zero or below LOQ) and are only included in the participation and reporting statistics.

The participation of the 21 laboratories for the analytes in SRM 3398 Ginger Rhizome and RM 8666 Ginger Extract was good for gingerols and shogaols (62 % to 71 % of participants returned results) and fair for 6-paradol and zingerone (38 % to 48 % of participants returned results).

	Number of Laboratories Reporting Results (Percent Participation)	
Analyte	SRM 3398	RM 8666
Total Ginger Constituents	11 (52 %)	11 (52 %)
6-Gingerol	15 (71 %)	15 (71 %)
8-Gingerol	15 (71 %)	15 (71 %)
10-Gingerol	15 (71 %)	15 (71 %)
6-Shogaol	14 (67 %)	14 (67 %)
8-Shogaol	14 (67 %)	13 (62 %)
10-Shogaol	14 (67 %)	14 (67 %)
6-Paradol	8 (38 %)	8 (38 %)
Zingerone	8 (38 %)	10 (48 %)

The participation of the 11 laboratories for all analytes in ginger powder and ginger containing supplements was good, with 64 % to 91 % return of results. Fewer laboratories returned results for the ginger mixture (27 % to 55 %).

	Number of Laboratories Reporting Results (Percent Participation)		
Analyte	Ginger Mixture	Ginger Powder	Supplements A, B, C, & D
Total Ginger Constituents	6 (55 %)	10 (91 %)	10 (91 %)
6-Gingerol	6 (55 %)	10 (91 %)	10 (91 %)
8-Gingerol	4 (36 %)	10 (91 %)	10 (91 %)
10-Gingerol	4 (36 %)	10 (91 %)	10 (91 %)
6-Shogaol	6 (55 %)	10 (91 %)	10 (91 %)
8-Shogaol	4 (36 %)	10 (91 %)	9 to 10 (82 % to 91 %)
10-Shogaol	4 (36 %)	10 (91 %)	9 (82 %)
6-Paradol	3 (27 %)	7 (64 %)	7 to 8 (64 % to 73 %)
Zingerone	4 (36 %)	7 (64 %)	7 to 8 (64 % to 73 %)

The between-laboratory variabilities were < 30 % for most analytes in SRM 3398 Ginger Rhizome and RM 8666 Ginger Extract. Higher between-laboratory variabilities were observed for 8-gingerol, 6-paradol, and zingerone in both materials and for 10-gingerol in RM 8666 Ginger Extract.

	Between-Laboratory Variability (% RSD)	
Analyte	SRM 3398	RM 8666
Total Ginger Constituents	27 %	18 %
6-Gingerol	39 %	22 %
8-Gingerol	50 %	59 %
10-Gingerol	26 %	42 %
6-Shogaol	20 %	29 %
8-Shogaol	26 %	26 %
10-Shogaol	21 %	23 %
6-Paradol	59 %	58 %
Zingerone	45 %	61 %

The between-laboratory variabilities were < 30 % for most analytes in the 6 additional ginger containing materials.

- Higher between-laboratory variabilities were observed for 6-paradol and zingerone in all samples, for 10-gingerol and 10-shogaol in Supplement C, and for 8-shogaol in Supplement D.
- Extremely high between-laboratory variabilities were observed for all compounds in the ginger mixture, which only contained 6-gingerol and 6-shogoal.

			Between-Laboratory Variability (% RSD)			
Analyte	Ginger Mixture	Ginger Powder	Supplement A	Supplement B	Supplement C	Supplement D
Total Ginger Constituents	75 %	12 %	12 %	15 %	19 %	19 %
6-Gingerol	67 %	17 %	19 %	15 %	12 %	19 %
8-Gingerol	> 100 %	23 %	33 %	15 %	18 %	36 %
10-Gingerol	> 100 %	18 %	36 %	27 %	47 %	20 %
6-Shogaol	93 %	13 %	22 %	20 %	18 %	20 %
8-Shogaol	> 100 %	24 %	24 %	23 %	33 %	43 %
10-Shogaol	-	28 %	24 %	37 %	52 %	33 %
6-Paradol	-	61 %	56 %	62 %	> 100 %	100 %
Zingerone	> 100 %	> 100 %	63 %	67 %	30 %	100 %

The within-laboratory variabilities were < 5 % for most analytes in SRM 3398 Ginger Rhizome and RM 8666 Ginger Extract.

Analyte	Within-Laboratory Variability (Median % RSD) SRM 3398	RM 8666
Total Ginger Constituents	2.1 %	1.7 %
6-Gingerol	3.0 %	1.4 %
8-Gingerol	6.7 %	2.0 %
10-Gingerol	5.2 %	2.8 %
6-Shogaol	2.0 %	1.5 %
8-Shogaol	2.7 %	5.1 %
10-Shogaol	2.1 %	1.4 %
6-Paradol	9.1 %	7.6 %
Zingerone	26 %	5.7 %

The within-laboratory variabilities were very good for most analytes in the 6 additional ginger containing materials. The Ginger Mixture material only contained 6-gingerol and 6-shogaol, and the within-laboratory variabilities were good for these analytes. The % RSDs for the analytes not present in the Ginger Mixture are shown in grey in the table below.

Analyte	Within-Laboratory Variability (Median % RSD) Ginger Mixture	Ginger Powder
Total Ginger Constituents	4.5 %	1.9 %
6-Gingerol	4.3 %	1.8 %
8-Gingerol	38 %	2.7 %
10-Gingerol	17 %	2.5 %
6-Shogaol	4.5 %	1.8 %
8-Shogaol	29 %	10 %
10-Shogaol	-	4.8 %
6-Paradol	-	14.8 %
Zingerone	47 %	4.4 %

Analyte	Within-Laboratory Variability (Median % RSD) Supplement A	Supplement B	Supplement C	Supplement D
Total Ginger Constituents	2.4 %	1.0 %	1.3 %	3.1 %
6-Gingerol	1.7 %	1.0 %	0.7 %	4.8 %
8-Gingerol	3.2 %	1.9 %	1.5 %	13 %
10-Gingerol	11 %	1.7 %	1.1 %	4.2 %
6-Shogaol	2.4 %	0.9 %	3.6 %	3.0 %
8-Shogaol	8.6 %	8.2 %	1.9 %	14 %
10-Shogaol	3.5 %	3.8 %	3.4 %	11 %
6-Paradol	17 %	10.1 %	4.5 %	27 %
Zingerone	10.8 %	8.0 %	6.7 %	-

Most laboratories reported using either solvent extraction or dilution as the sample preparation method for the determination of gingerols. The percentages in the table below are based only on laboratories that returned results. AOAC 2018.04 uses dilution with acidified water and methanol, which could be interpreted by laboratories as either solvent extraction *or* dilution.

Reported Sample Preparation Method	Average Percent Reporting SRM 3398 & RM 8666	Ginger Mixture	Ginger Powder, Supplements A, B, C, & D
Solvent Extraction	49 %	58 %	51 %
Dilution	18 %	29 %	20 %
Other	18 %	-	9 %
None Reported	15 %	13 %	19 %

Most laboratories reported using LC-Abs as the analytical method for the determination of gingerols. The percentages in the table below are based only on laboratories that returned results. AOAC 2018.04 uses LC-Abs.

Reported Analytical Method	Average Percent Reporting SRM 3398 & RM 8666	Ginger Mixture	Ginger Powder, Supplements A, B, C, & D
AOAC 2018.04	15 %	15 %	20 %
LC-Abs	56 %	71 %	49 %
LC-FLD	1 %	-	1 %
Other	12 %	-	9 %
None Reported	16 %	14 %	21 %

5.4. Botanicals Technical Recommendations

The following recommendations and observations are based on results obtained from the participants in this study. Additional overall technical recommendations can be found on page 6.

- Consensus means were in better agreement to the NIST target range of tolerance the ginger extract (RM 8666) than for the ginger rhizome (SRM 3398), which may indicate challenges with sample preparation. Laboratories reporting results below the target values or reporting a large sample-to-sample variability for the rhizome material should examine their sample preparation procedure.
    - The gingerols in the extract have already been processed from a ginger plant matrix and are likely to be more freely soluble in the extraction solvent than the gingerols in the rhizome.
    - Complete extraction of gingerols from plant matrices may require use of less common solvents or multiple extraction cycles. Sample preparation techniques should be optimized to yield the most exhaustive extraction of the analyte from the matrix. Parameters to consider may include but are not limited to solvent volume relative to sample mass, solvent composition, number of extraction cycles, extraction time, and physical technique (e.g., ultrasonic bath, shaker, rotary mixer).
- In general, 6-paradol and zingerone had higher between-laboratory variabilities in all materials, likely due to the low mass fractions present in the materials.

- The data collected from this study was intended to help evaluate reproducibility of AOAC 2018.04. Additional rounds of this study will be needed to gather enough data to evaluate reproducibility of the AOAC method.
    - Of the ten laboratories that indicated intent to follow AOAC 2018.04, only four confirmed use and one reported that they did not follow the method.
    - For additional studies, there will be an effort to ensure better return of method information.
- As stated in the method performance requirements of AOAC SMPR 2017.02, the RSD_r should be ≤ 5 %, and the RSD_R should be ≤ 8 %. The AOAC 2018.04 method already established acceptable RSDr values. Additionally, the results of this study also show very good promise as the within-laboratory variabilities (RSD_r) were ≤ 5 %, for most of the gingerols and shogaols in most of the test samples.
- As stated in the method performance requirements of AOAC SMPR 2017.02, the RSD_R should be ≤ 8 %. The results of this study show RSD_Rs higher than 8 %, though it is not conclusive, as there were not enough labs that confirmed use of AOAC 2018.04. However, the results did show promise for the method validation.
    - If outliers are removed from the results for laboratories indicating intent to use AOAC 2018.04, the RSD_Rs ranged from 6 % to 64 %. Total Ginger Constituents in Ginger Supplement A was the only measurand-sample type combination that had an RSD_R of ≤ 8 %. When looking across measurands, the average RSD_R for the Total Ginger Constituents was best at 13 %. When looking across sample types, the average RSD_R for the Gingerol Supplement A was best at 20 %.

Table 5-1. Individual data table (NIST) for gingerols in ginger rhizome and ginger extract.

Exercise 7 - Botanicals - Gingerols

		Lab Code:	**NIST**	**1. Your Results**				**2. Community Results**			**3. Target**	
Analyte	Sample		Units	x_i	s_i	Z'_{comm}	Z_{NIST}	N	x*	s*	x_{NIST}	*U*
Total Ginger Constituents	SRM 3398 Ginger Rhizome		% w/w	0.939	0.008			11	0.63	0.17	0.939	0.008
Total Ginger Constituents	RM 8666 Ginger Extract		% w/w	3.791	0.038			11	3.23	0.57	3.791	0.038
6-gingerol	SRM 3398 Ginger Rhizome		% w/w	0.364	0.005			15	0.186	0.072	0.364	0.005
6-gingerol	RM 8666 Ginger Extract		% w/w	2.23	0.036			15	1.90	0.42	2.23	0.036
8-gingerol	SRM 3398 Ginger Rhizome		% w/w	0.057	0.001			15	0.034	0.017	0.057	0.001
8-gingerol	RM 8666 Ginger Extract		% w/w	0.355	0.008			15	0.30	0.18	0.355	0.008
10-gingerol	SRM 3398 Ginger Rhizome		% w/w	0.083	0.002			15	0.058	0.015	0.083	0.002
10-gingerol	RM 8666 Ginger Extract		% w/w	0.443	0.005			15	0.41	0.17	0.443	0.005
6-shogaol	SRM 3398 Ginger Rhizome		% w/w	0.252	0.004			14	0.220	0.044	0.252	0.004
6-shogaol	RM 8666 Ginger Extract		% w/w	0.518	0.007			14	0.48	0.14	0.518	0.007
8-shogaol	SRM 3398 Ginger Rhizome		% w/w	0.068	0.002			14	0.054	0.014	0.068	0.002
8-shogaol	RM 8666 Ginger Extract		% w/w	0.091	0.003			13	0.092	0.024	0.091	0.003
10-shogaol	SRM 3398 Ginger Rhizome		% w/w	0.115	0.002			14	0.106	0.022	0.115	0.002
10-shogaol	RM 8666 Ginger Extract		% w/w	0.153	0.004			14	0.145	0.033	0.153	0.004
6-paradol	SRM 3398 Ginger Rhizome		% w/w					8	0.017	0.010		
6-paradol	RM 8666 Ginger Extract		% w/w					8	0.091	0.053		
zingerone	SRM 3398 Ginger Rhizome		% w/w					8	0.011	0.005		
zingerone	RM 8666 Ginger Extract		% w/w					10	0.041	0.025		

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

Table 5-2. Individual data table (NIST) for gingerols in ginger mixture and ginger powder.

Exercise 7 - Botanicals - Gingerols

		Lab Code:	**NIST**	**1. Your Results**				**2. Community Results**			**3. Target**	
Analyte	Sample		Units	x_i	s_i	Z'_{comm}	Z_{NIST}	N	x*	s*	x_{NIST}	*U*
Total Ginger Constituents	Ginger Mixture (USP Cat # 1291446)		% w/w					6	20	17		
Total Ginger Constituents	Powdered Ginger (USP Cat # 1291504)		% w/w	1.074	0.054			10	1.06	0.12	1.074	0.054
6-gingerol	Ginger Mixture (USP Cat # 1291446)		% w/w	8.7	1.7			6	7.8	5.2	8.7	1.7
6-gingerol	Powdered Ginger (USP Cat # 1291504)		% w/w	0.554	0.015			10	0.436	0.074	0.554	0.015
8-gingerol	Ginger Mixture (USP Cat # 1291446)		% w/w					4	1.7	5.5		
8-gingerol	Powdered Ginger (USP Cat # 1291504)		% w/w	0.140	0.019			10	0.116	0.027	0.140	0.019
10-gingerol	Ginger Mixture (USP Cat # 1291446)		% w/w					4	1.3	4.9		
10-gingerol	Powdered Ginger (USP Cat # 1291504)		% w/w	0.176	0.014			10	0.237	0.042	0.176	0.014
6-shogaol	Ginger Mixture (USP Cat # 1291446)		% w/w	12.3	2.5			6	8.1	7.5	12.3	2.5
6-shogaol	Powdered Ginger (USP Cat # 1291504)		% w/w	0.116	0.007			10	0.16	0.021	0.116	0.007
8-shogaol	Ginger Mixture (USP Cat # 1291446)		% w/w					4	0.23	0.83		
8-shogaol	Powdered Ginger (USP Cat # 1291504)		% w/w	0.025	0.004			10	0.034	0.008	0.025	0.004
10-shogaol	Ginger Mixture (USP Cat # 1291446)		% w/w					4	0.004	0.008		
10-shogaol	Powdered Ginger (USP Cat # 1291504)		% w/w	0.048	0.004			10	0.064	0.018	0.048	0.004
6-paradol	Ginger Mixture (USP Cat # 1291446)		% w/w					3				
6-paradol	Powdered Ginger (USP Cat # 1291504)		% w/w	0.015	0.003			7	0.018	0.011	0.015	0.003
zingerone	Ginger Mixture (USP Cat # 1291446)		% w/w					4	3.5	5.2		
zingerone	Powdered Ginger (USP Cat # 1291504)		% w/w					7	0.001	0.005		

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

Table 5-3. Individual data table (NIST) for gingerols in ginger supplements.

Exercise 7 - Botanicals - Gingerols

	Lab Code:	NIST	1. Your Results				2. Community Results			3. Target	
Analyte	Sample	Units	x_i	s_i	Z'_{comm}	Z_{NIST}	N	x*	s*	x_{NIST}	*U*
Total Ginger Constituents	Ginger Supplement A	% w/w					10	0.434	0.051		
Total Ginger Constituents	Ginger Supplement B	% w/w					10	1.53	0.22		
Total Ginger Constituents	Ginger Supplement C	% w/w					10	9	1.7		
Total Ginger Constituents	Ginger Supplement D	% w/w					10	0.227	0.044		
6-gingerol	Ginger Supplement A	% w/w					10	0.191	0.037		
6-gingerol	Ginger Supplement B	% w/w					10	0.78	0.12		
6-gingerol	Ginger Supplement C	% w/w					10	5.31	0.66		
6-gingerol	Ginger Supplement D	% w/w					10	0.103	0.019		
8-gingerol	Ginger Supplement A	% w/w					10	0.03	0.01		
8-gingerol	Ginger Supplement B	% w/w					10	0.148	0.022		
8-gingerol	Ginger Supplement C	% w/w					10	0.98	0.18		
8-gingerol	Ginger Supplement D	% w/w					10	0.025	0.009		
10-gingerol	Ginger Supplement A	% w/w					10	0.045	0.016		
10-gingerol	Ginger Supplement B	% w/w					10	0.271	0.072		
10-gingerol	Ginger Supplement C	% w/w					10	1.4	0.66		
10-gingerol	Ginger Supplement D	% w/w					10	0.05	0.01		
6-shogaol	Ginger Supplement A	% w/w					10	0.101	0.022		
6-shogaol	Ginger Supplement B	% w/w					10	0.211	0.041		
6-shogaol	Ginger Supplement C	% w/w					10	0.68	0.12		
6-shogaol	Ginger Supplement D	% w/w					10	0.03	0.006		
8-shogaol	Ginger Supplement A	% w/w					9	0.021	0.005		
8-shogaol	Ginger Supplement B	% w/w					10	0.04	0.009		
8-shogaol	Ginger Supplement C	% w/w					10	0.21	0.07		
8-shogaol	Ginger Supplement D	% w/w					9	0.007	0.003		
10-shogaol	Ginger Supplement A	% w/w					9	0.033	0.008		
10-shogaol	Ginger Supplement B	% w/w					9	0.07	0.026		
10-shogaol	Ginger Supplement C	% w/w					9	0.23	0.12		
10-shogaol	Ginger Supplement D	% w/w					9	0.012	0.004		
6-paradol	Ginger Supplement A	% w/w					7	0.009	0.005		
6-paradol	Ginger Supplement B	% w/w					8	0.021	0.013		
6-paradol	Ginger Supplement C	% w/w					8	0.16	0.17		
6-paradol	Ginger Supplement D	% w/w					7	0.004	0.004		
zingerone	Ginger Supplement A	% w/w					7	0.008	0.005		
zingerone	Ginger Supplement B	% w/w					7	0.009	0.006		
zingerone	Ginger Supplement C	% w/w					8	0.1	0.03		
zingerone	Ginger Supplement D	% w/w					7	0.001	0.001		

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

Table 5-4. Data summary table for total ginger constituents in ginger rhizome and ginger extract. Data points highlighted in blue have been identified as outside the consensus tolerance limits and resulted in an unacceptable Z'_{comm} score, $|Z'_{\text{comm}}| \geq 2$.

		Total Ginger Constituents									
		SRM 3398 Ginger Rhizome (% w/w)					RM 8666 Ginger Extract (% w/w)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				0.939	0.008				3.791	0.038
	G001										
	G003										
	G004	0.5821	0.5442	0.5819	0.569	0.022	3.5495	3.559	3.6191	3.576	0.038
	G008										
	G009										
	G019	0.57	0.59	0.63	0.597	0.031	3.09	3.24	3.21	3.180	0.079
	G020										
	G021	0.553	0.6	0.578	0.577	0.024	3.263	3.22	3.262	3.248	0.025
	G023	1.113	1.089	1.1645	1.122	0.039	4.8886	4.8889	4.9533	4.910	0.037
	G026										
	G027	0.65259	0.65443	0.65547	0.654	0.001	2.16379	2.23784	2.24583	2.216	0.045
	G029	0.86	0.84	0.85	0.850	0.010	3.71	3.66	3.63	3.667	0.040
	G030	0.609	0.616	0.619	0.615	0.005	1.93	2.15	1.9	1.993	0.137
	G033										
	G034										
	G036										
	G037	0.457	0.463	0.477	0.466	0.010	3.57	3.57	3.6	3.580	0.017
	G039	0.737	0.735	0.733	0.735	0.002	3.559	3.655	3.675	3.630	0.062
	G041										
	G042										
	G044	0.559	0.538	0.561	0.553	0.013	3.5	3.4	3.4	3.433	0.058
	G046	0.3925	0.4005	0.4019	0.398	0.005	1.8902	2.023	2.1883	2.034	0.149
Community Results		Consensus Mean			0.627		Consensus Mean			3.228	
		Consensus Standard Deviation			0.171		Consensus Standard Deviation			0.573	
		Maximum			1.122		Maximum			4.910	
		Minimum			0.398		Minimum			1.993	
		N			11		N			11	

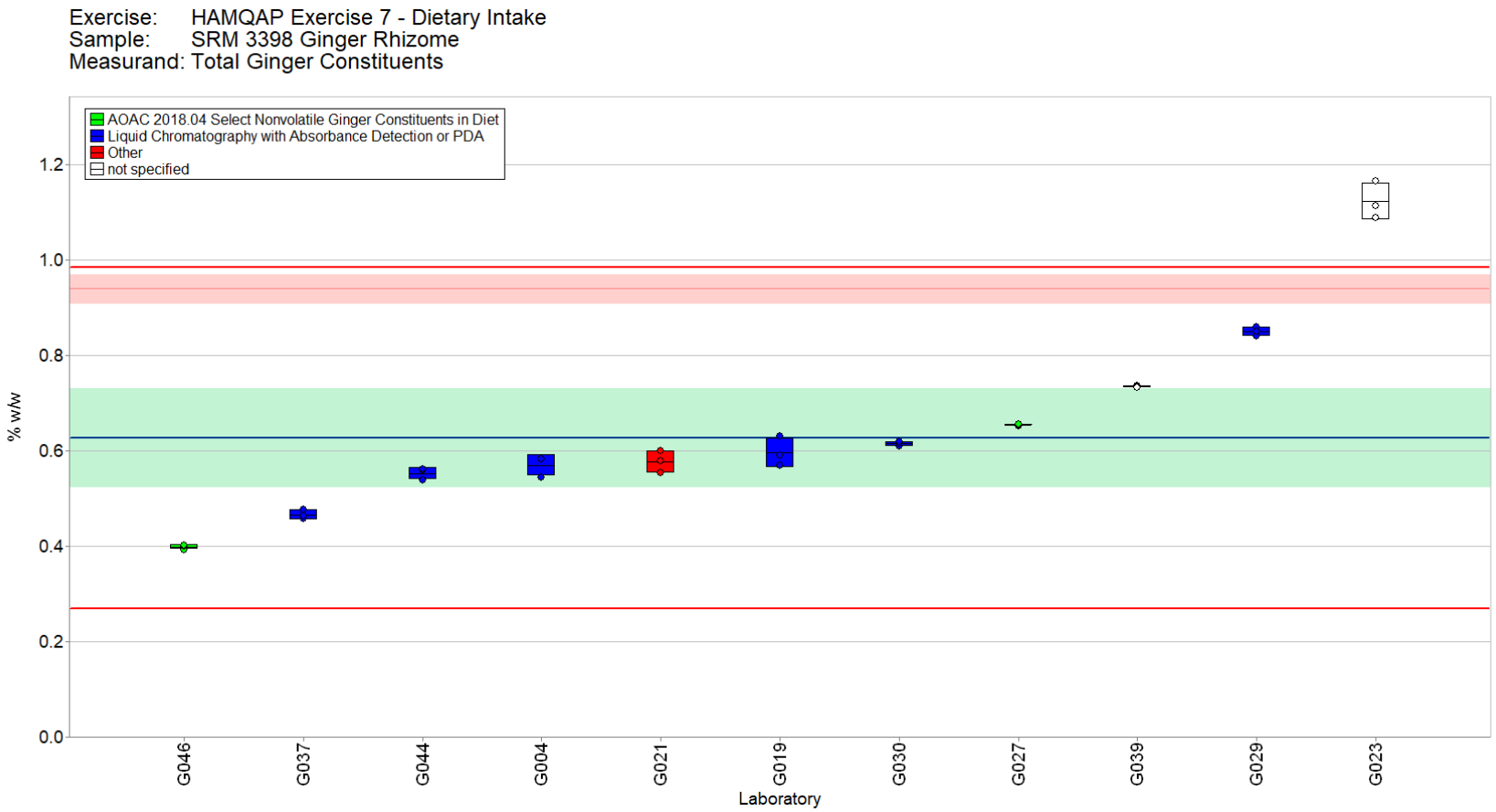

Fig. 5-1. Total ginger constituents in SRM 3398 Ginger (*Zingiber officinale*) Rhizome (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

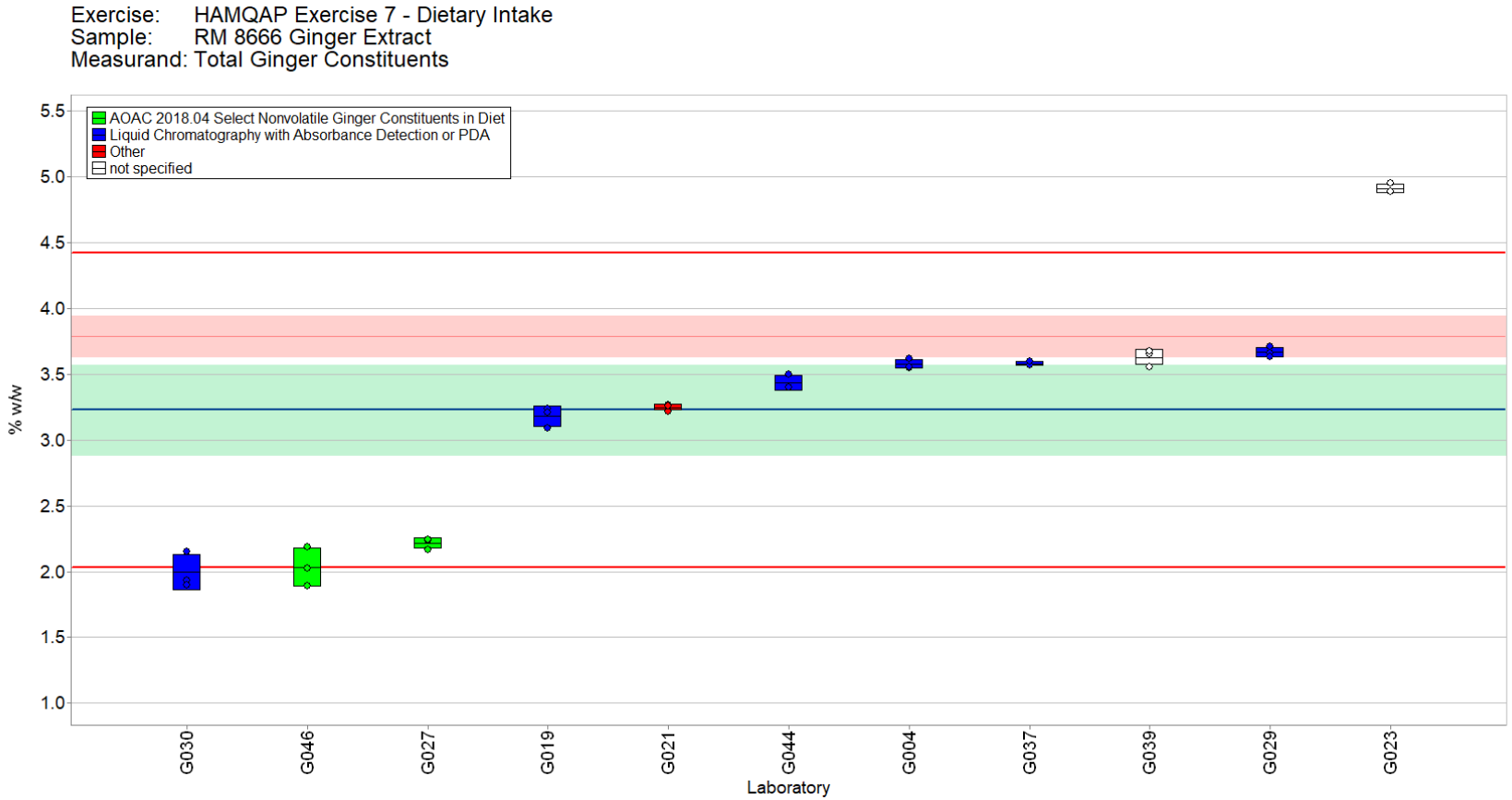

Fig. 5-2. Total ginger constituents in in RM 8666 Ginger (*Zingiber officinale*) Extract (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$.

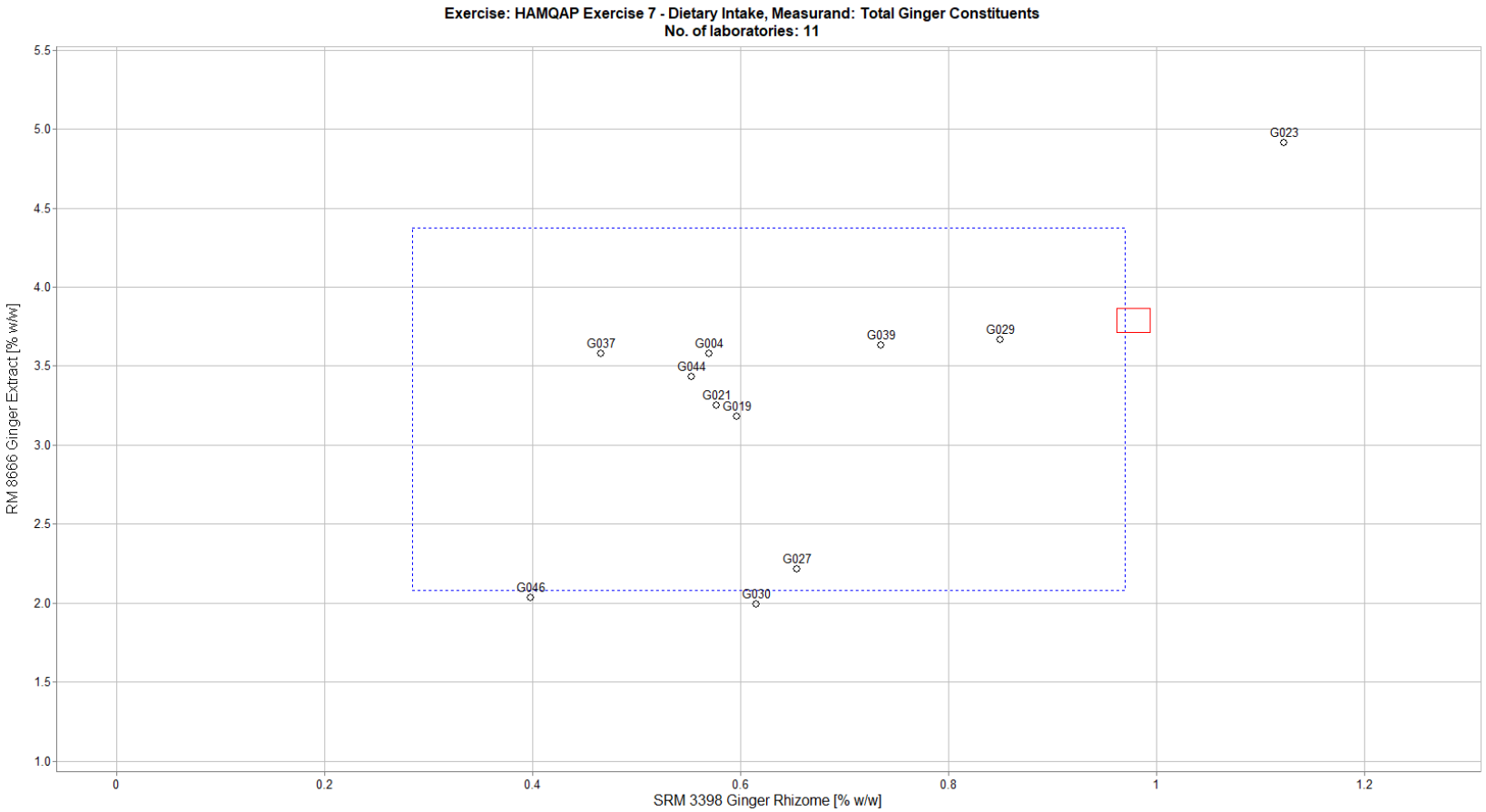

Fig. 5-3. Laboratory means for total ginger constituents in SRM 3398 Ginger (*Zingiber officinale*) Rhizome and RM 8666 Ginger (*Zingiber officinale*) Extract (sample/sample comparison view). In this view, the individual laboratory mean for one sample (SRM 3398) is compared to the individual laboratory mean for a second sample (RM 8666). The solid red box represents the NIST range of tolerance for the two samples, SRM 3398 (x-axis) and RM 8666 (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3398 (x-axis) and RM 8666 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

Table 5-5. Data summary table for 6-gingerol in ginger rhizome and ginger extract. Data points highlighted in blue have been identified as outside the consensus tolerance limits and resulted in an unacceptable Z'_{comm} score, $|Z'_{\text{comm}}| \geq 2$.

		6-gingerol									
		SRM 3398 Ginger Rhizome (% w/w)					RM 8666 Ginger Extract (% w/w)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				0.364	0.005				2.230	0.036
	G001										
	G003	0.261	0.275	0.274	0.270	0.008	2.11	2.02	2.05	2.060	0.046
	G004	0.121	0.111	0.1258	0.119	0.008	1.8686	1.8775	1.906	1.884	0.020
	G008										
	G009										
	G019	0.15	0.14	0.15	0.147	0.006	1.75	1.85	1.8	1.800	0.050
	G020										
	G021	0.132	0.138	0.143	0.138	0.006	1.931	1.916	1.924	1.924	0.008
	G023	0.2944	0.2854	0.3054	0.295	0.010	2.5753	2.583	2.5936	2.584	0.009
	G026										
	G027	0.16896	0.16871	0.16736	0.168	0.001	0.93698	0.98131	0.98892	0.969	0.028
	G029	0.31	0.3	0.3	0.303	0.006	2.12	2.08	2.08	2.093	0.023
	G030	0.121	0.121	0.119	0.120	0.001	0.817	0.965	0.814	0.865	0.086
	G033										
	G034	0.15	0.24	0.24	0.210	0.052	2.57	2.55	2.52	2.547	0.025
	G036										
	G037	0.0555	0.058	0.0576	0.057	0.001	2.05	2.04	2.06	2.050	0.010
	G039	0.228	0.231	0.233	0.231	0.003	2.021	2.072	2.065	2.053	0.028
	G041	0.269	0.291	0.269	0.276	0.013	2.24	2.21	2.2	2.217	0.021
	G042	0.16	0.162	0.163	0.162	0.002	1.013	1.088	1.133	1.078	0.061
	G044	0.163	0.16	0.161	0.161	0.002	2.26	2.19	2.2	2.217	0.038
	G046	0.1221	0.1136	0.1166	0.117	0.004	1.2889	1.3635	1.4437	1.365	0.077
Community Results		Consensus Mean			0.186		Consensus Mean			1.90	
		Consensus Standard Deviation			0.072		Consensus Standard Deviation			0.42	
		Maximum			0.303		Maximum			2.58	
		Minimum			0.057		Minimum			0.87	
		N			15		N			15	

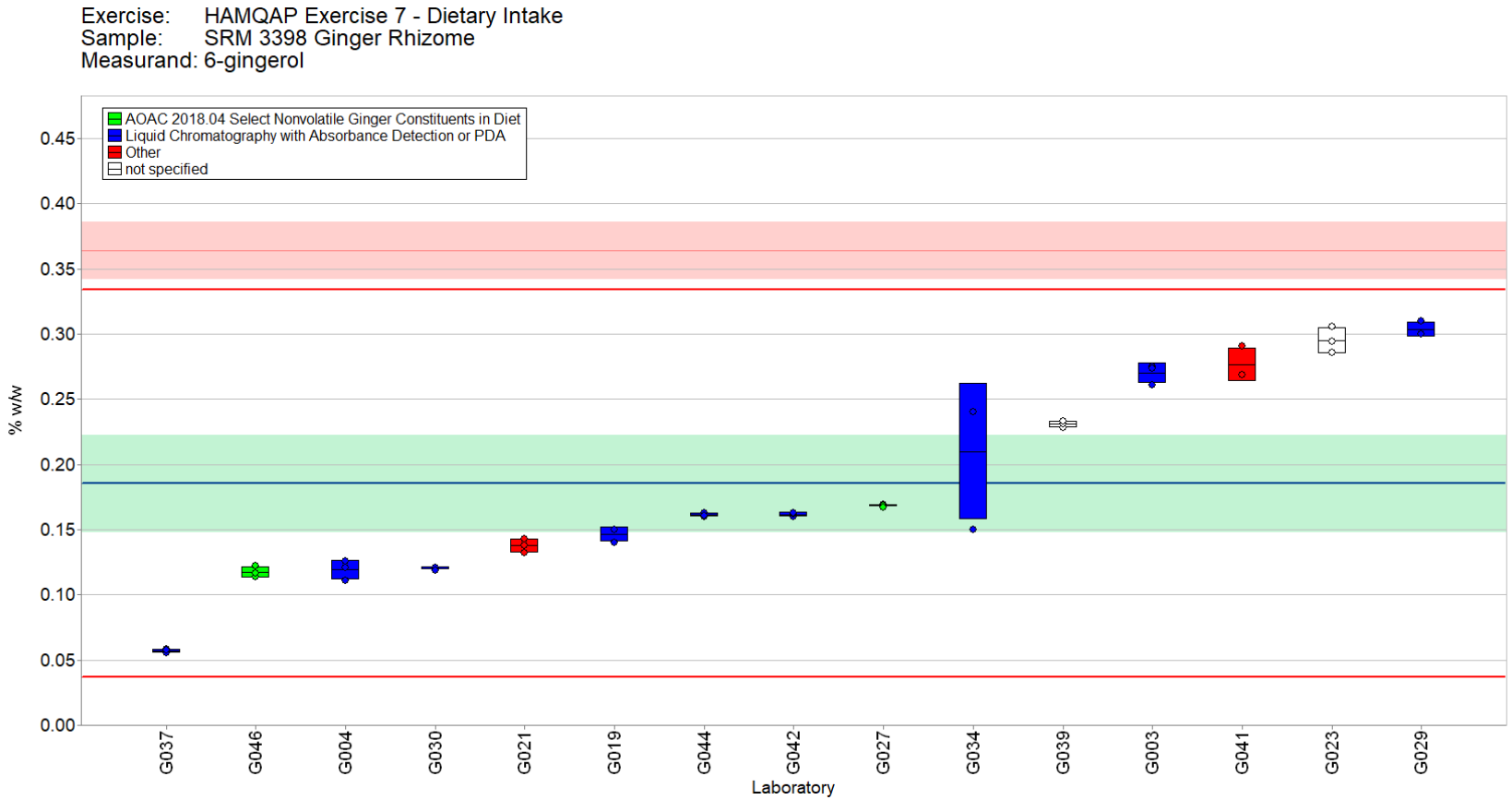

Fig. 5-4. 6-gingerol in SRM 3398 Ginger (*Zingiber officinale*) Rhizome (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

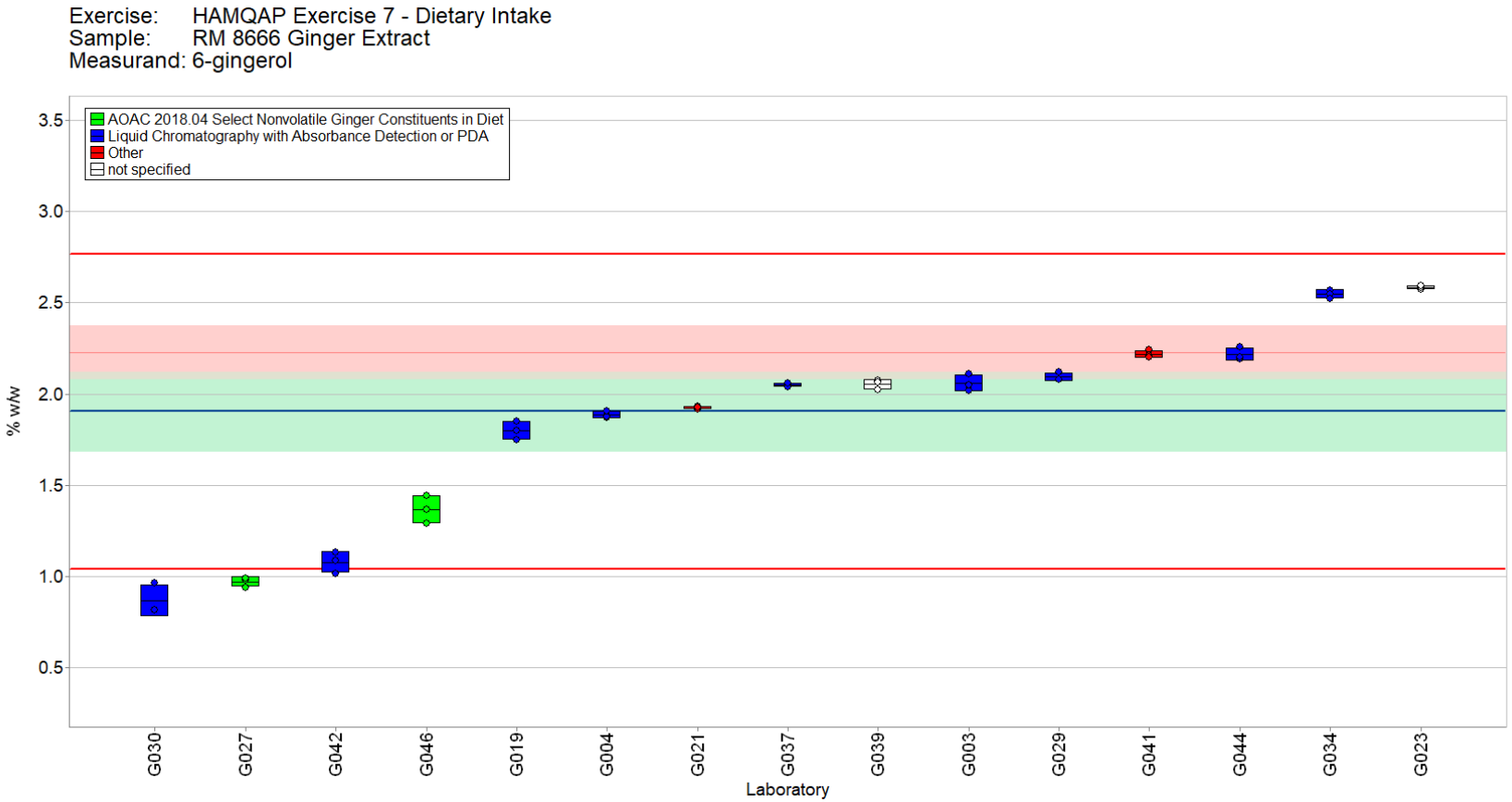

Fig. 5-5. 6-gingerol in RM 8666 Ginger (*Zingiber officinale*) Extract (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

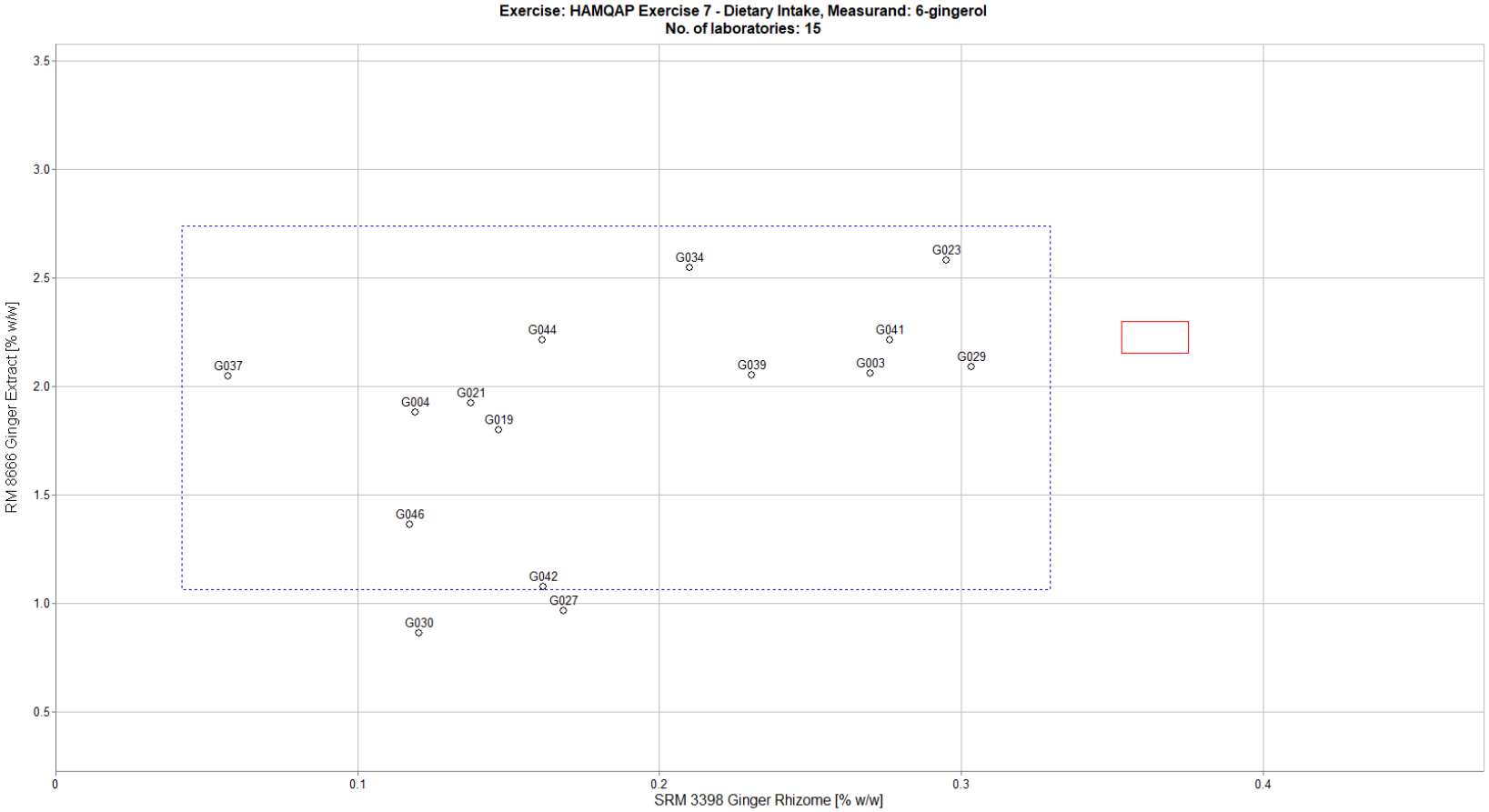

Fig. 5-6. Laboratory means for 6-gingerol in SRM 3398 Ginger (*Zingiber officinale*) Rhizome and RM 8666 Ginger (*Zingiber officinale*) Extract (sample/sample comparison view). In this view, the individual laboratory mean for one sample (SRM 3398) is compared to the individual laboratory mean for a second sample (RM 8666). The solid red box represents the NIST range of tolerance for the two samples, SRM 3398 (x-axis) and RM 8666 (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3398 (x-axis) and RM 8666 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

Table 5-6. Data summary table for 8-gingerol in ginger rhizome and ginger extract. Data points highlighted in blue have been identified as outside the consensus tolerance limits and resulted in an unacceptable Z'_{comm} score, $|Z'_{\text{comm}}| \geq 2$.

		8-gingerol									
		SRM 3398 Ginger Rhizome (% w/w)					RM 8666 Ginger Extract (% w/w)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				0.057	0.001				0.355	0.008
	G001										
	G003	0.0507	0.05	0.0512	0.051	0.001	0.35	0.336	0.338	0.341	0.008
	G004	0.0508	0.0438	0.046	0.047	0.004	0.3903	0.3908	0.401	0.394	0.006
	G008										
	G009										
	G019	0.02	0.03	0.03	0.027	0.006	0.26	0.26	0.27	0.263	0.006
	G020										
	G021	0.04	0.046	0.045	0.044	0.003	0.287	0.279	0.28	0.282	0.004
	G023	0.2247	0.2413	0.2474	0.238	0.012	0.7163	0.7436	0.7291	0.730	0.014
	G026										
	G027	0.03027	0.0303	0.03374	0.031	0.002	0.16261	0.16834	0.17056	0.167	0.004
	G029	0.03	0.03	0.03	0.03	0	0.29	0.29	0.29	0.29	0
	G030	0.0263	0.0259	0.026	0.0261	0.0002	0.145	0.167	0.134	0.149	0.017
	G033										
	G034	0.24	0.26	0.28	0.260	0.020	1.01	0.99	0.96	0.987	0.025
	G036										
	G037	0.0295	0.0287	0.0287	0.0290	0.0005	0.283	0.281	0.283	0.282	0.001
	G039	0.023	0.024	0.026	0.024	0.002	0.244	0.252	0.248	0.248	0.004
	G041	0.0508	0.053	0.0537	0.053	0.002	0.367	0.361	0.36	0.363	0.004
	G042	0.0305	0.0341	0.0336	0.033	0.002	0.137	0.139	0.138	0.138	0.001
	G044	0.0412	0.0471	0.0461	0.045	0.003	0.48	0.468	0.461	0.470	0.010
	G046		0.0094	0.0086	0.009	0.001	0.1189	0.1311	0.1476	0.133	0.014
Community Results		Consensus Mean			0.034		Consensus Mean			0.301	
		Consensus Standard Deviation			0.017		Consensus Standard Deviation			0.179	
		Maximum			0.260		Maximum			0.987	
		Minimum			0.009		Minimum			0.133	
		N			15		N			15	

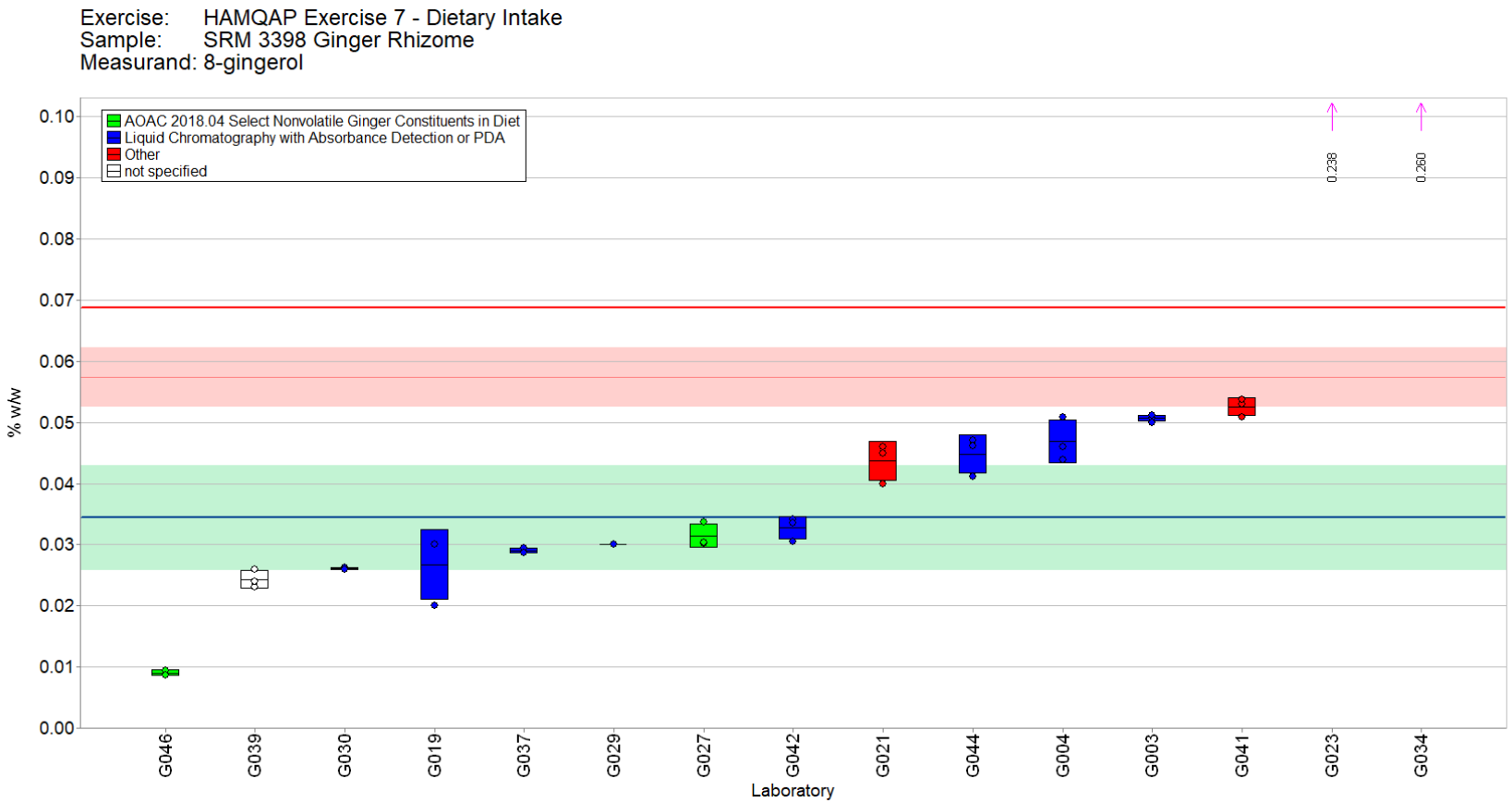

Fig. 5-7. 8-gingerol in SRM 3398 Ginger (*Zingiber officinale*) Rhizome (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

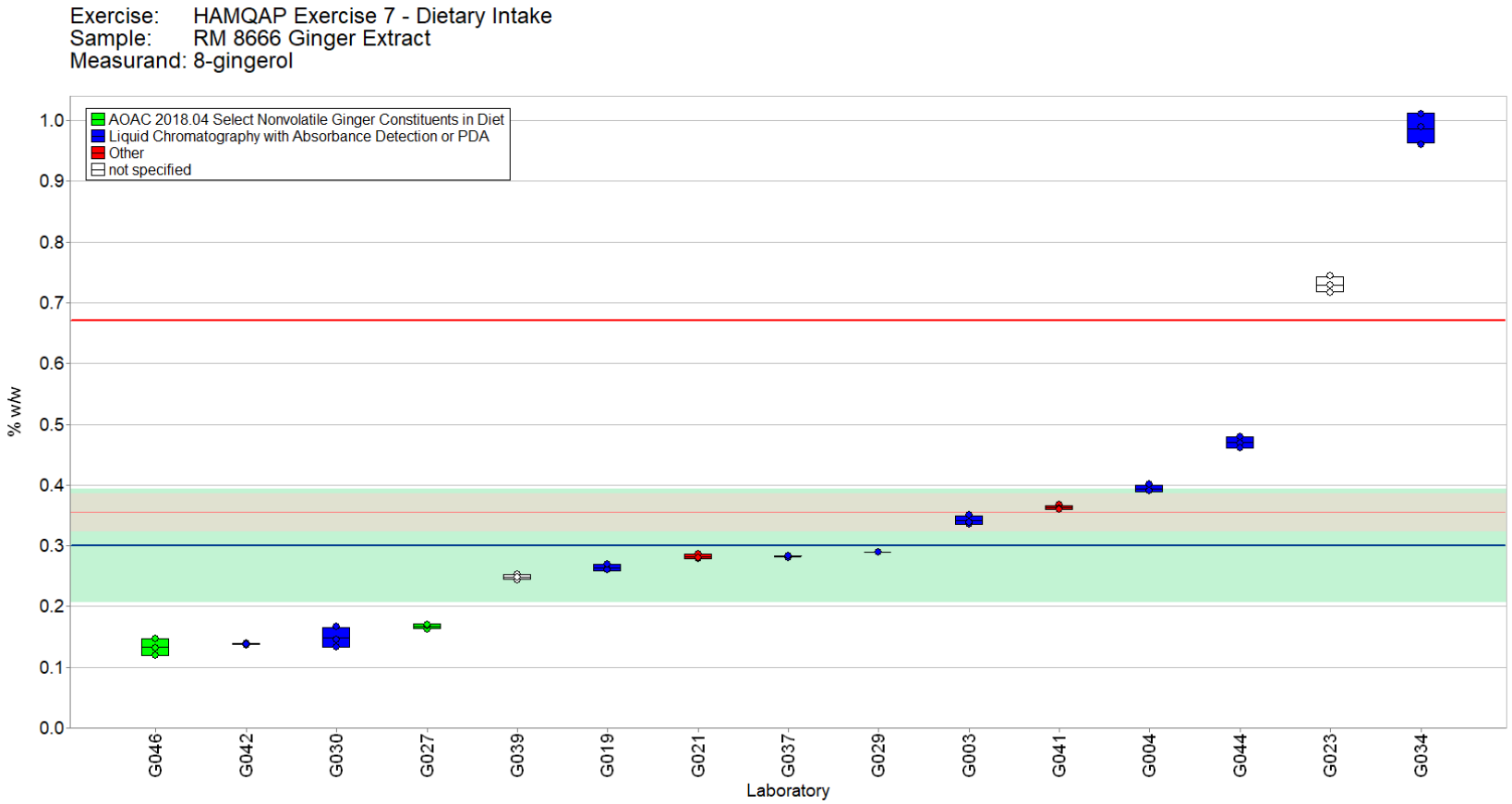

Fig. 5-8. 8-gingerol in RM 8666 Ginger (Zingiber officinale) Extract (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$, with the lower range set at zero. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The beige shaded region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

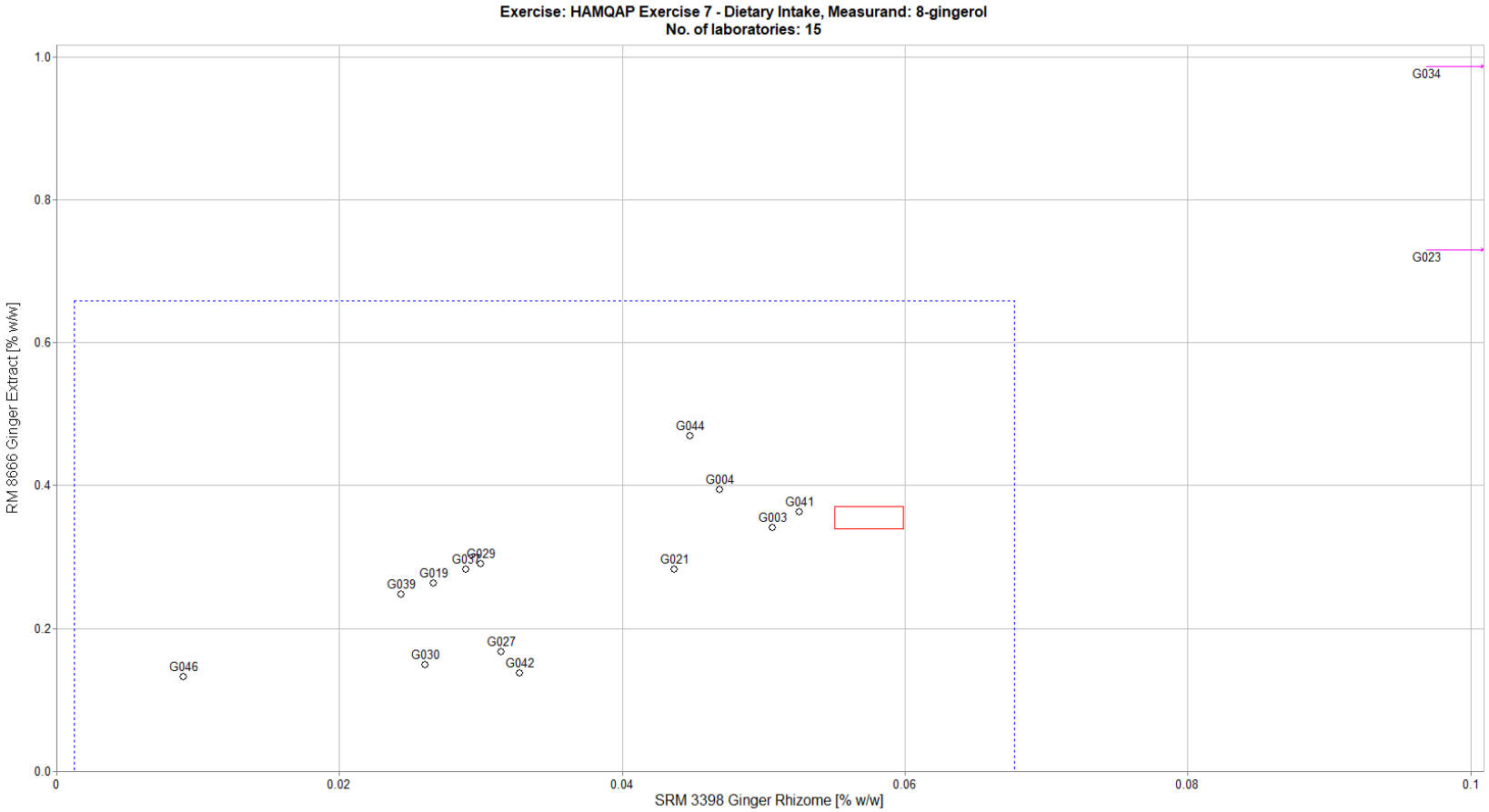

Fig. 5-9. Laboratory means for 8-gingerol in SRM 3398 Ginger (*Zingiber officinale*) Rhizome and RM 8666 Ginger (*Zingiber officinale*) Extract (sample/sample comparison view). In this view, the individual laboratory mean for one sample (SRM 3398) is compared to the individual laboratory mean for a second sample (RM 8666). The solid red box represents the NIST range of tolerance for the two samples, SRM 3398 (x-axis) and RM 8666 (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3398 (x-axis) and RM 8666 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

Table 5-7. Data summary table for 10-gingerol in ginger rhizome and ginger extract. Data points highlighted in blue have been identified as outside the consensus tolerance limits and resulted in an unacceptable Z'_{comm} score, $|Z'_{\text{comm}}| \geq 2$.

		10-gingerol									
		SRM 3398 Ginger Rhizome (% w/w)					RM 8666 Ginger Extract (% w/w)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				0.083	0.002				0.443	0.005
	G001										
	G003	0.0569	0.0563	0.0554	0.056	0.001	0.519	0.498	0.509	0.509	0.011
	G004	0.0578	0.0561	0.0613	0.058	0.003	0.5457	0.5483	0.5421	0.545	0.003
	G008										
	G009										
	G019	0.04	0.04	0.04	0.04	0	0.28	0.29	0.27	0.280	0.010
	G020										
	G021	0.09	0.103	0.101	0.098	0.007	0.446	0.426	0.452	0.441	0.014
	G023	0.0996	0.0752	0.1054	0.093	0.016	0.6162	0.5849	0.6124	0.605	0.017
	G026										
	G027	0.05046	0.0517	0.05245	0.052	0.001	0.30246	0.30656	0.31184	0.307	0.005
	G029	0.06	0.06	0.06	0.06	0	0.42	0.41	0.41	0.413	0.006
	G030	0.0525	0.0595	0.0597	0.057	0.004	0.306	0.334	0.28	0.307	0.027
	G033										
	G034	0.52	0.47	0.39	0.460	0.066	0.91	0.96	0.93	0.933	0.025
	G036										
	G037	0.0401	0.0367	0.0479	0.042	0.006	0.449	0.474	0.474	0.466	0.014
	G039	0.055	0.055	0.053	0.054	0.001	0.416	0.439	0.437	0.431	0.013
	G041	0.0629	0.0618	0.0659	0.064	0.002	0.448	0.443	0.438	0.443	0.005
	G042	0.0567	0.0568	0.0559	0.056	0.000	0.234	0.236	0.253	0.241	0.010
	G044	0.0704	0.0663	0.0696	0.069	0.002	0.114	0.112	0.112	0.113	0.001
	G046	0.0439	0.0443	0.0448	0.0443	0.0005	0.3303	0.3485	0.373	0.351	0.021
Community Results		Consensus Mean			0.058		Consensus Mean			0.410	
		Consensus Standard Deviation			0.015		Consensus Standard Deviation			0.172	
		Maximum			0.460		Maximum			0.933	
		Minimum			0.04		Minimum			0.113	
		N			15		N			15	

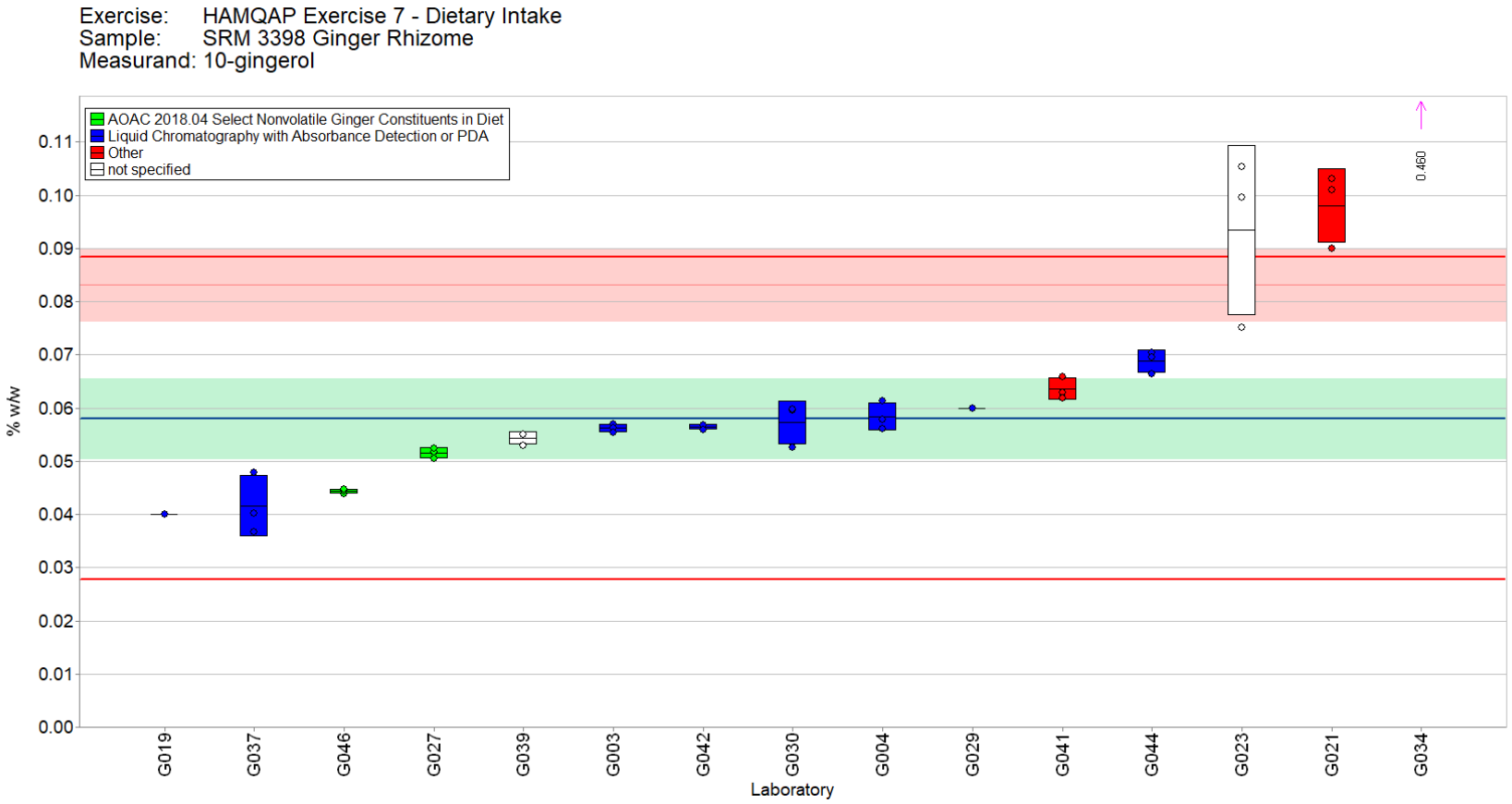

Fig. 5-10. 10-gingerol in SRM 3398 Ginger (*Zingiber officinale*) Rhizome (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

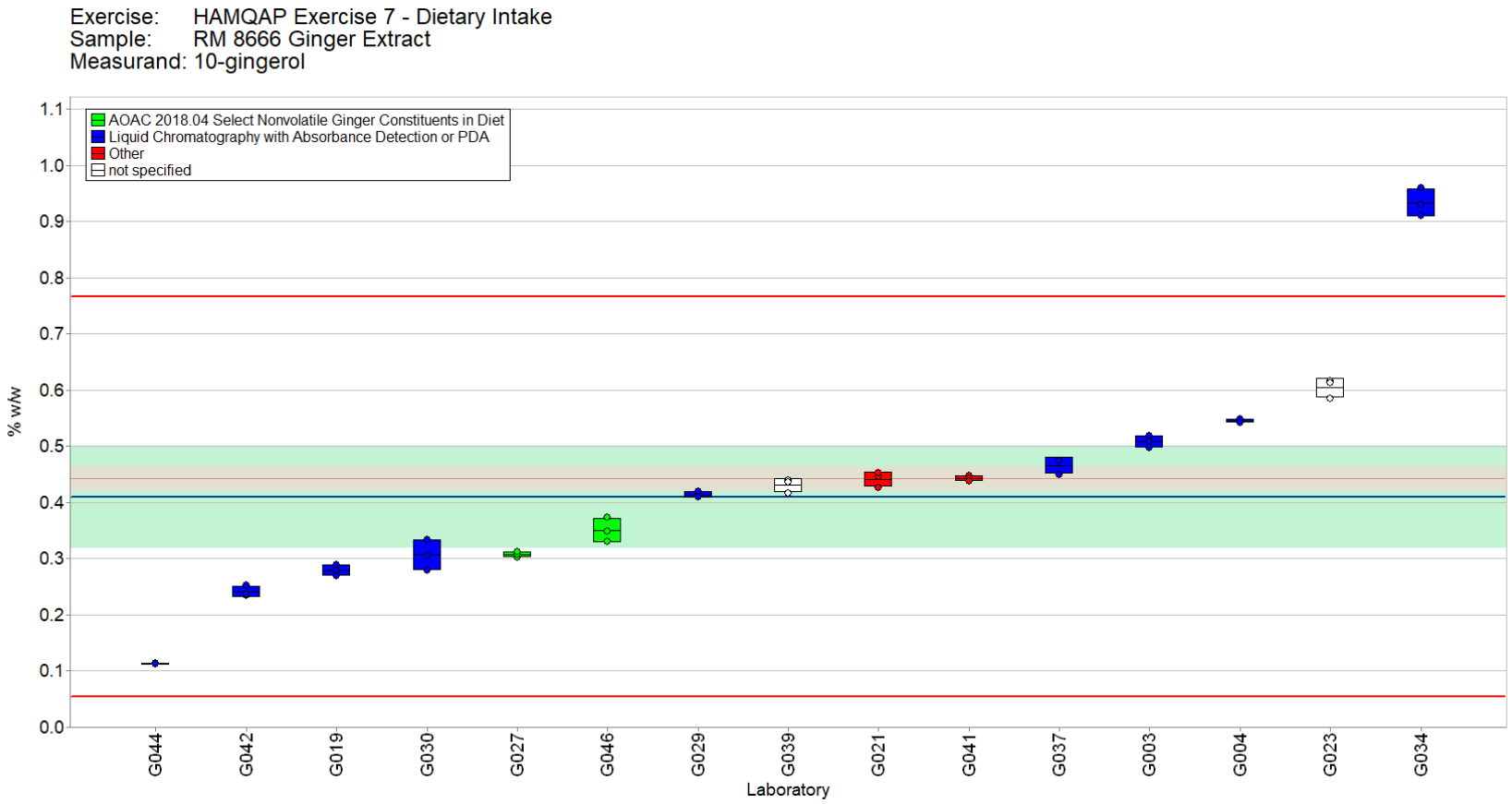

Fig. 5-11. 10-gingerol in RM 8666 Ginger (*Zingiber officinale*) Extract (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The beige shaded region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

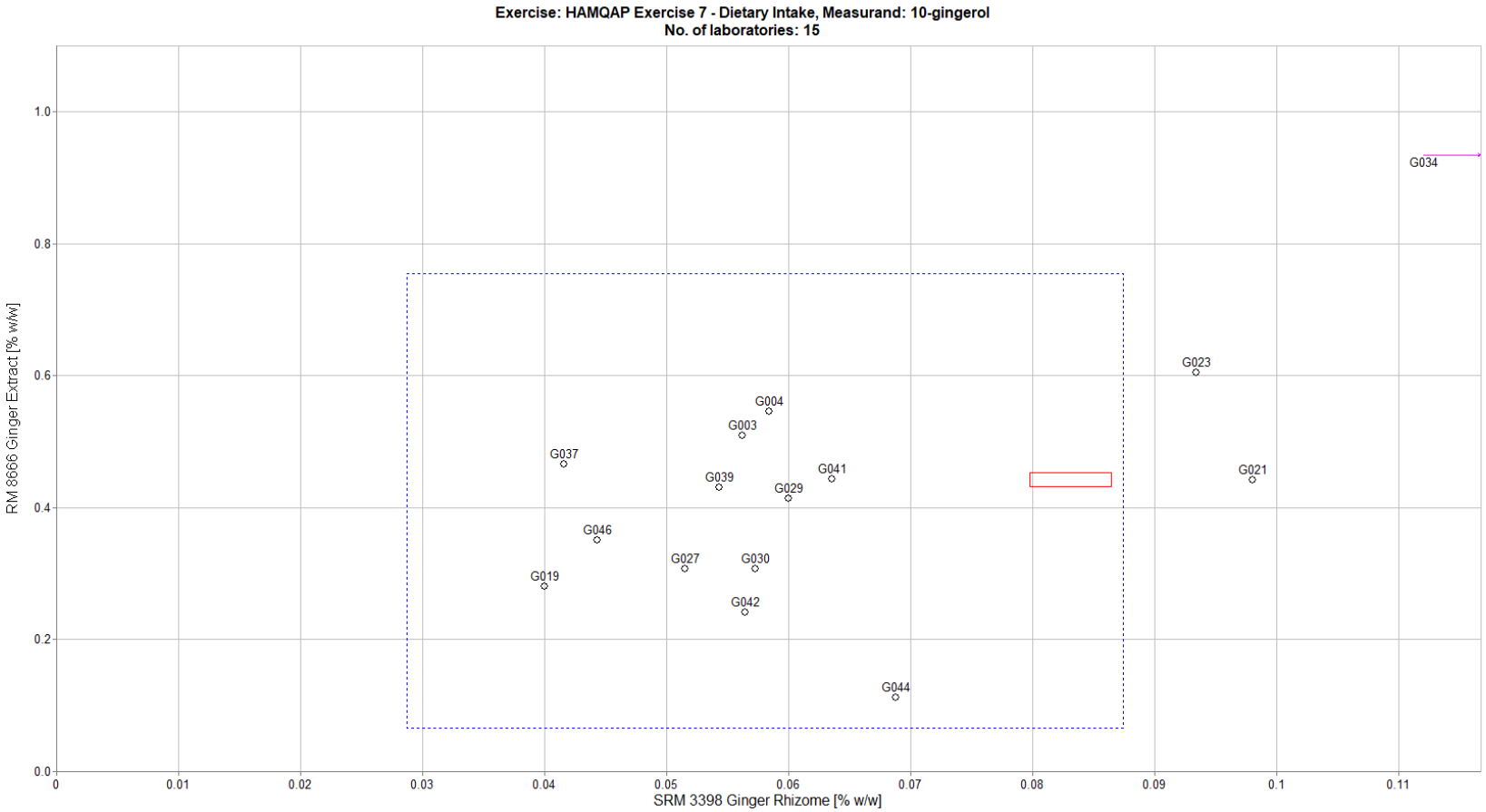

Fig. 5-12. Laboratory means for 10-gingerol in SRM 3398 Ginger (*Zingiber officinale*) Rhizome and RM 8666 Ginger (*Zingiber officinale*) Extract (sample/sample comparison view). In this view, the individual laboratory mean for one sample (SRM 3398) is compared to the individual laboratory mean for a second sample (RM 8666). The solid red box represents the NIST range of tolerance for the two samples, SRM 3398 (x-axis) and RM 8666 (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3398 (x-axis) and RM 8666 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

Table 5-8. Data summary table for 6-shogaol in ginger rhizome and ginger extract. Data points highlighted in blue have been identified as outside the consensus tolerance limits and resulted in an unacceptable Z'_{comm} score, $|Z'_{\text{comm}}| \geq 2$.

		6-shogaol									
		SRM 3398 Ginger Rhizome (% w/w)					RM 8666 Ginger Extract (% w/w)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				0.252	0.004				0.518	0.007
	G001										
	G003	0.252	0.255	0.262	0.256	0.005	0.627	0.603	0.617	0.616	0.012
	G004	0.191	0.1825	0.1892	0.188	0.004	0.464	0.4621	0.4801	0.469	0.010
	G008										
	G009										
	G019	0.16	0.16	0.17	0.163	0.006	0.35	0.36	0.37	0.360	0.010
	G020										
	G021	0.173	0.187	0.171	0.177	0.009	0.377	0.369	0.377	0.374	0.005
	G023	0.2386	0.2409	0.2442	0.241	0.003	0.5693	0.5712	0.5852	0.575	0.009
	G026										
	G027	0.21581	0.22358	0.22887	0.223	0.007	0.42683	0.4427	0.3195	0.396	0.067
	G029	0.25	0.25	0.25	0.25	0	0.54	0.54	0.53	0.537	0.006
	G030	0.223	0.226	0.229	0.226	0.003	0.409	0.429	0.408	0.415	0.012
	G033										
	G034										
	G036										
	G037	0.219	0.221	0.225	0.222	0.003	0.541	0.534	0.543	0.539	0.005
	G039	0.209	0.204	0.202	0.205	0.004	0.463	0.47	0.47	0.468	0.004
	G041	0.254	0.255	0.246	0.252	0.005	0.644	0.644	0.637	0.642	0.004
	G042	0.268	0.276	0.272	0.272	0.004	0.468	0.474	0.474	0.472	0.003
	G044	0.257	0.238	0.257	0.251	0.011	0.61	0.592	0.599	0.600	0.009
	G046	0.1054	0.1097	0.1089	0.108	0.002	0.1125	0.1159	0.1393	0.123	0.015
Community Results		Consensus Mean			0.220		Consensus Mean			0.481	
		Consensus Standard Deviation			0.044		Consensus Standard Deviation			0.138	
		Maximum			0.272		Maximum			0.642	
		Minimum			0.108		Minimum			0.123	
		N			14		N			14	

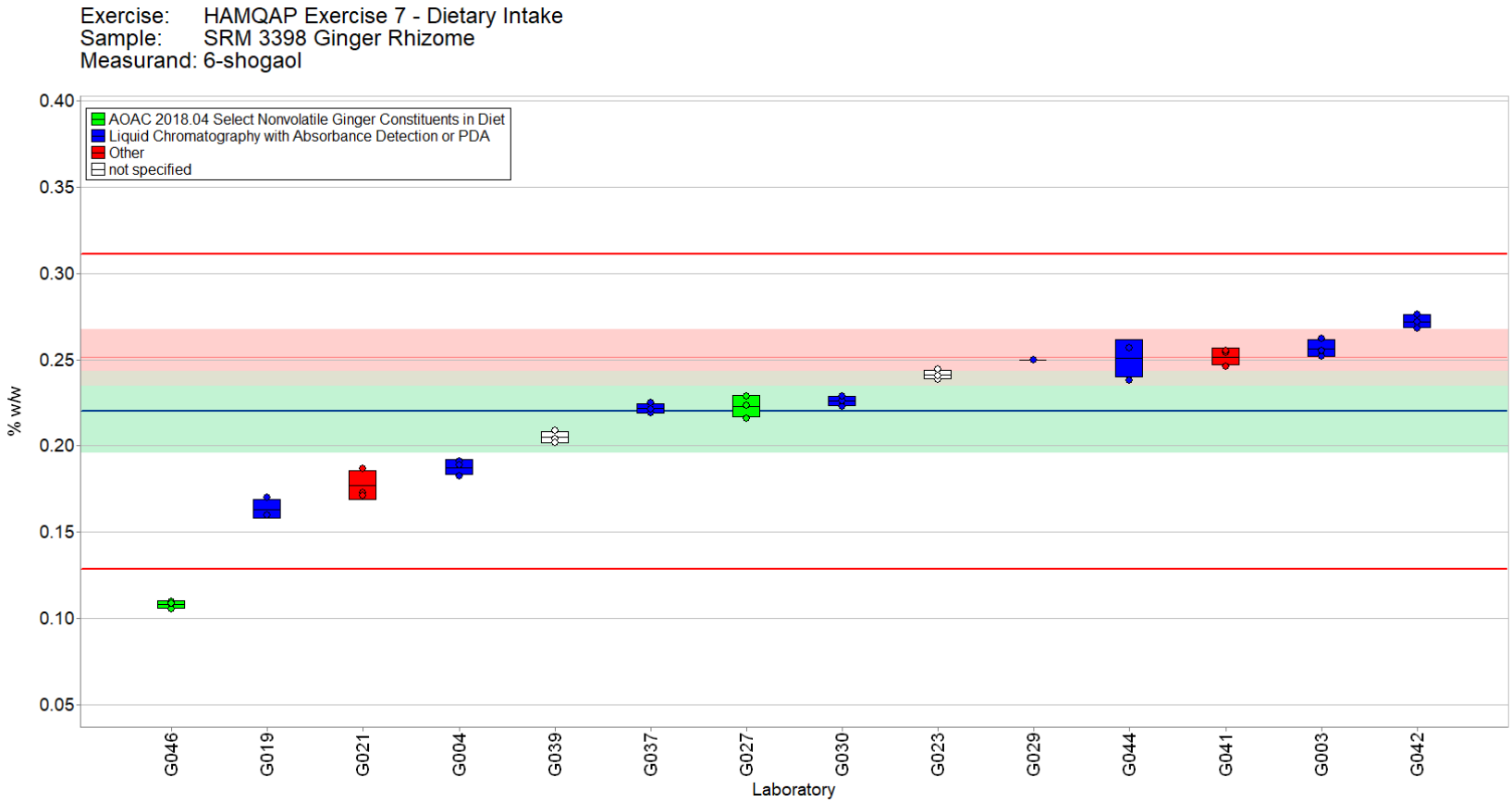

Fig. 5-13. 6-shogaol in SRM 3398 Ginger (*Zingiber officinale*) Rhizome (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

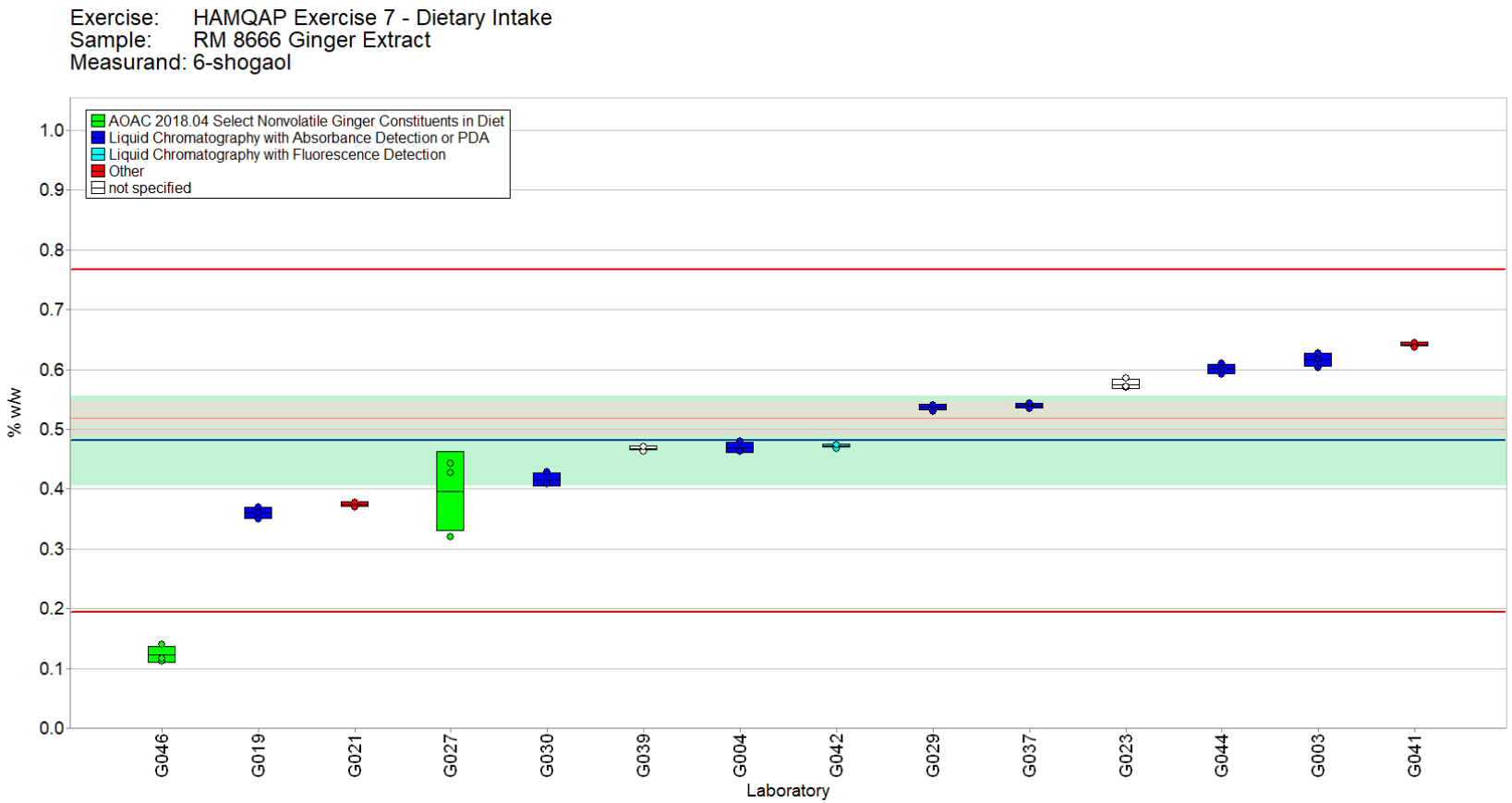

Fig. 5-14. 6-shogaol in RM 8666 Ginger (*Zingiber officinale*) Extract (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The beige shaded region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

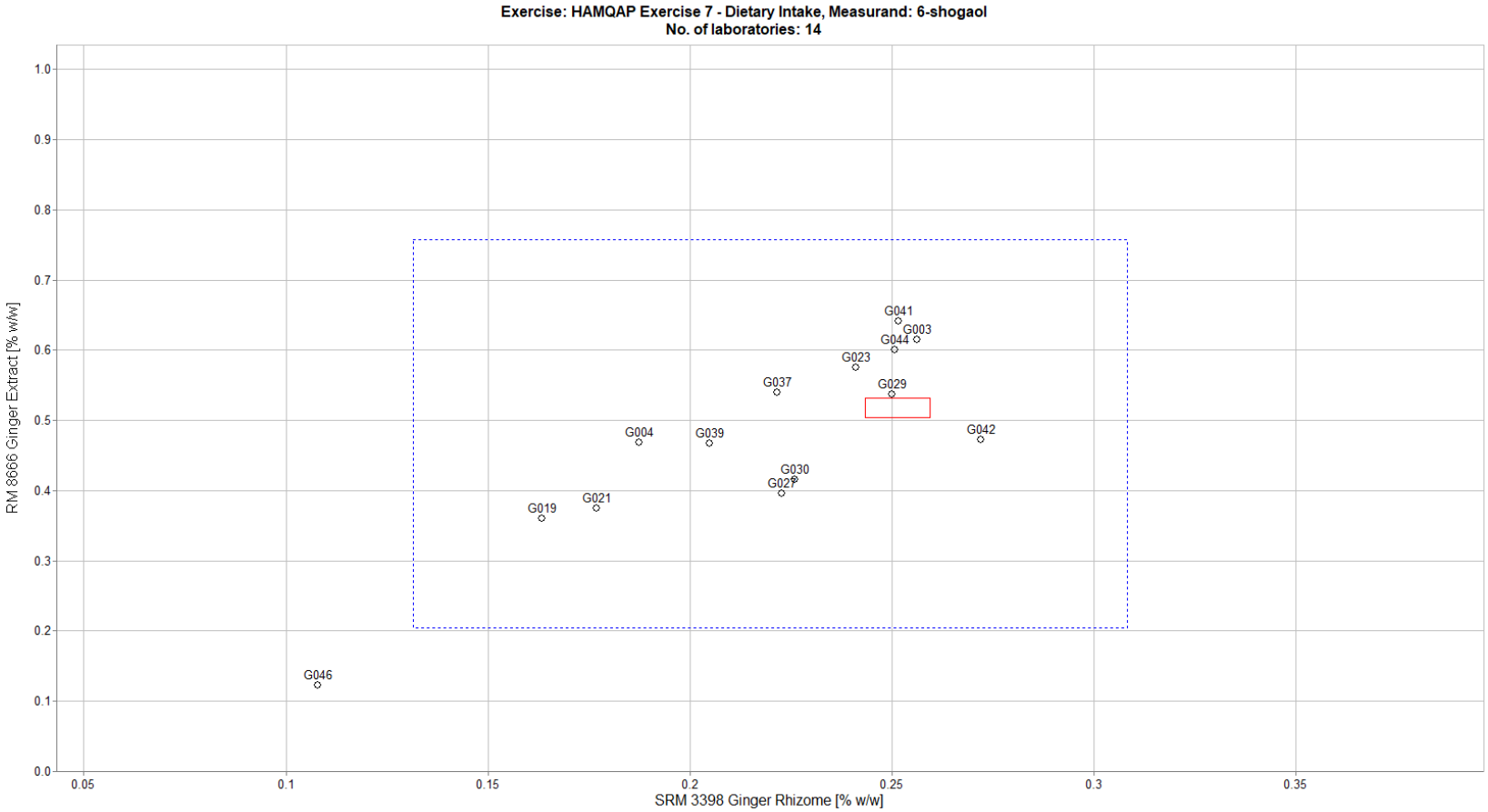

Fig. 5-15. Laboratory means for 6-shogaol in SRM 3398 Ginger (*Zingiber officinale*) Rhizome and RM 8666 Ginger (*Zingiber officinale*) Extract (sample/sample comparison view). In this view, the individual laboratory mean for one sample (SRM 3398) is compared to the individual laboratory mean for a second sample (RM 8666). The solid red box represents the NIST range of tolerance for the two samples, SRM 3398 (x-axis) and RM 8666 (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3398 (x-axis) and RM 8666 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

Table 5-9. Data summary table for 8-shogaol in ginger rhizome and ginger extract. Data points highlighted in blue have been identified as outside the consensus tolerance limits and resulted in an unacceptable Z'_{comm} score, $|Z'_{\text{comm}}| \geq 2$.

		8-shogaol									
		SRM 3398 Ginger Rhizome (% w/w)					RM 8666 Ginger Extract (% w/w)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				0.068	0.002				0.091	0.003
	G001										
	G003	0.0677	0.0677	0.0699	0.068	0.001	0.127	0.127	0.118	0.124	0.005
	G004	0.042	0.0396	0.0416	0.041	0.001	0.0732	0.0726	0.082	0.076	0.005
	G008										
	G009										
	G019	0.06	0.06	0.06	0.06	0	0.1	0.09	0.1	0.097	0.006
	G020										
	G021	0.03	0.032	0.029	0.030	0.002	0.042	0.039	0.043	0.041	0.002
	G023	0.0583	0.0577	0.0571	0.0577	0.0006	0.0938	0.0951	0.0953	0.0947	0.0008
	G026										
	G027	0.6241	0.0601	0.05955	0.248	0.326	0.08171	0.08312	0.08257	0.0825	0.0007
	G029	0.07	0.06	0.07	0.067	0.006	0.09	0.09	0.08	0.087	0.006
	G030	0.0574	0.0573	0.0581	0.0576	0.0004	0.0616	0.0645	0.0735	0.067	0.006
	G033										
	G034										
	G036										
	G037	0.0639	0.0639	0.0645	0.0641	0.0003	0.102	0.107	0.108	0.106	0.003
	G039	0.056	0.055	0.055	0.0553	0.0006	0.086	0.086	0.096	0.089	0.006
	G041	0.0664	0.0662	0.0676	0.0667	0.0008	0.134	0.134	0.133	0.1337	0.0006
	G042	0.0673	0.0675	0.0678	0.0675	0.0003	0.0925	0.0893	0.0995	0.094	0.005
	G044	0.0168	0.0157	0.0161	0.0162	0.0006	< 0.001	< 0.001	< 0.001		
	G046	0.0325	0.0335	0.0327	0.0329	0.0005					
Community Results		Consensus Mean			0.054		Consensus Mean			0.092	
		Consensus Standard Deviation			0.014		Consensus Standard Deviation			0.024	
		Maximum			0.248		Maximum			0.134	
		Minimum			0.016		Minimum			0.041	
		N			14		N			12	

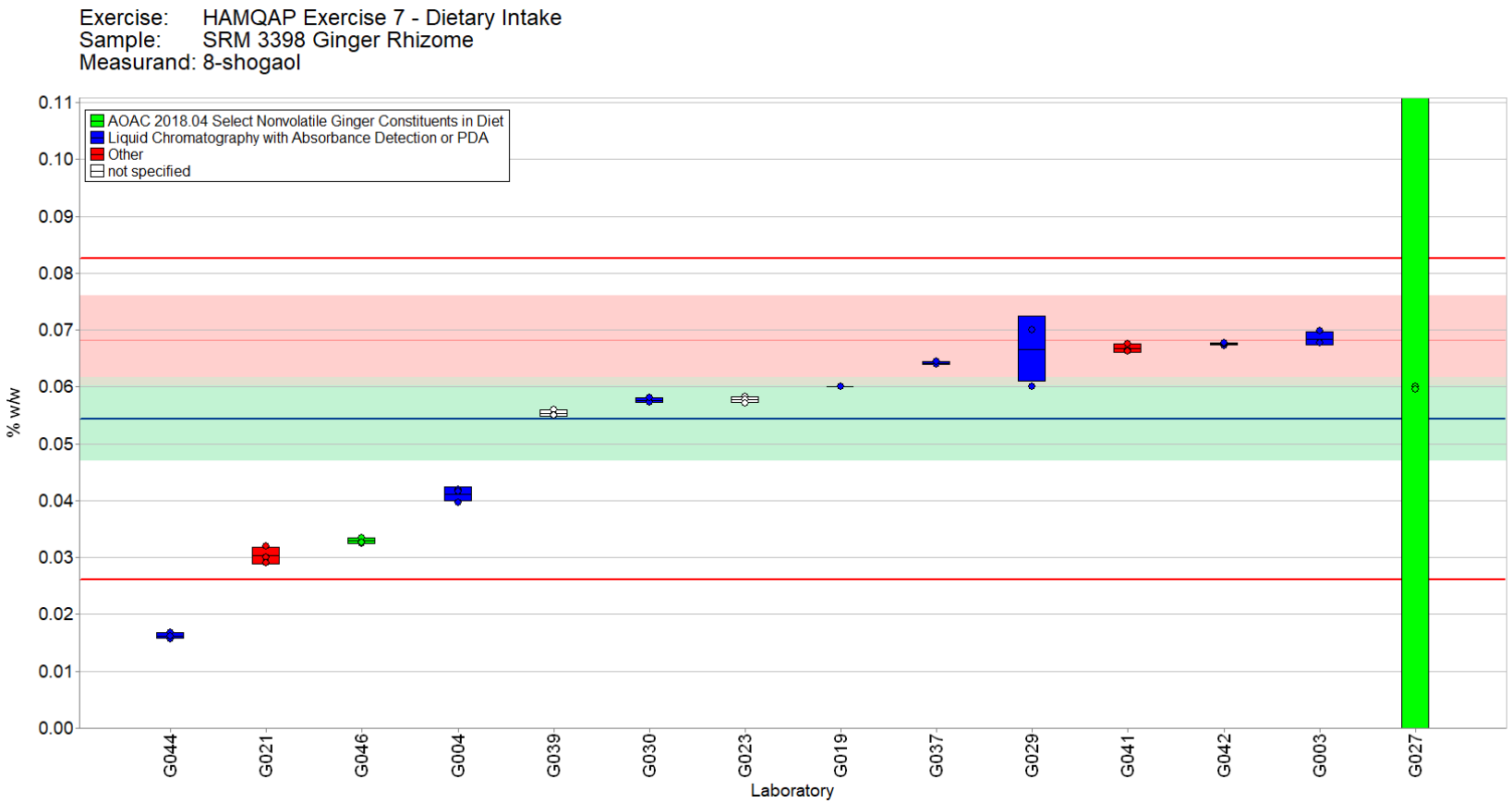

Fig. 5-16. 8-shogaol in SRM 3398 Ginger (*Zingiber officinale*) Rhizome (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

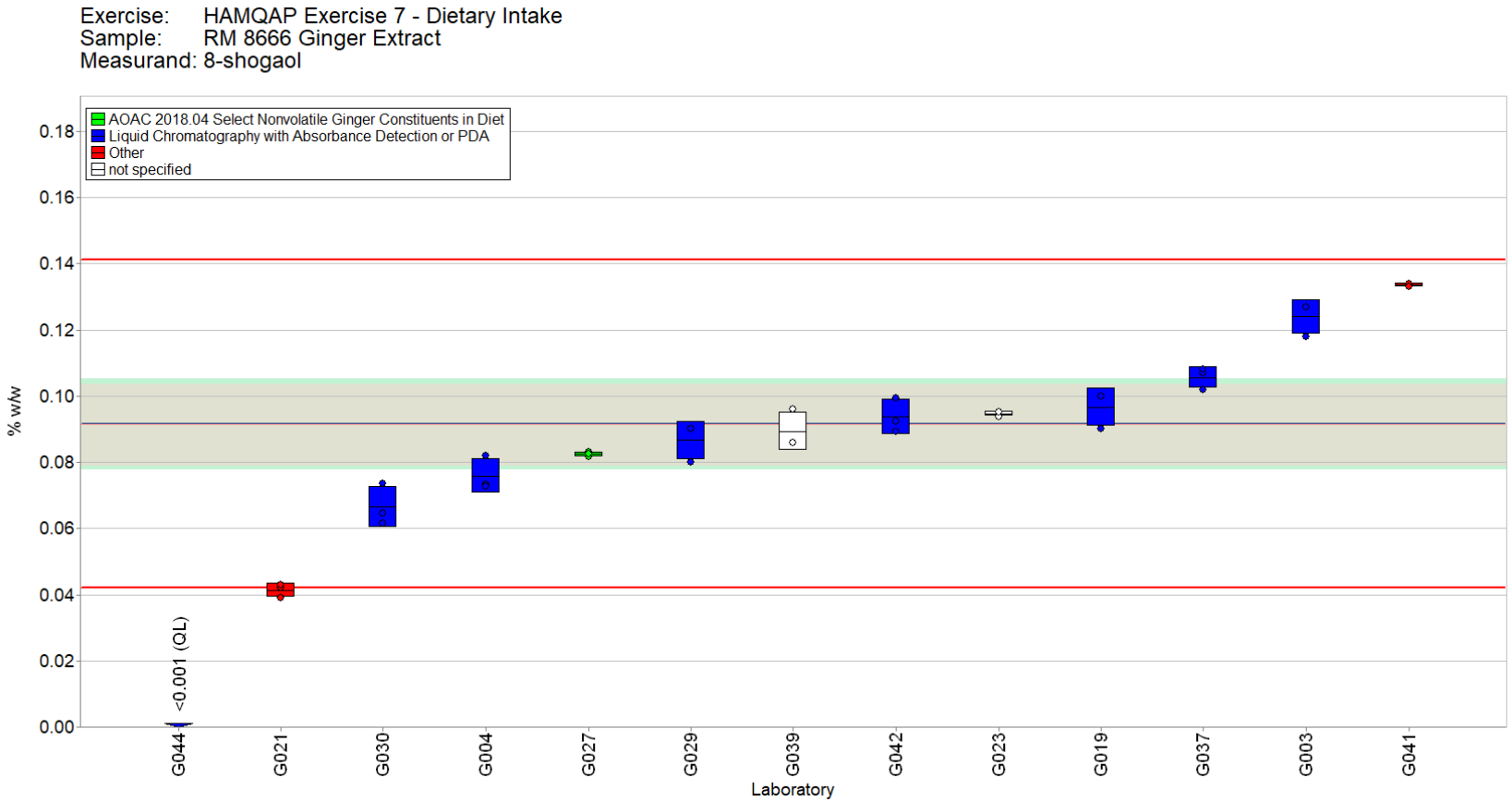

Fig. 5-17. 8-shogaol in RM 8666 Ginger (*Zingiber officinale*) Extract (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The beige shaded region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

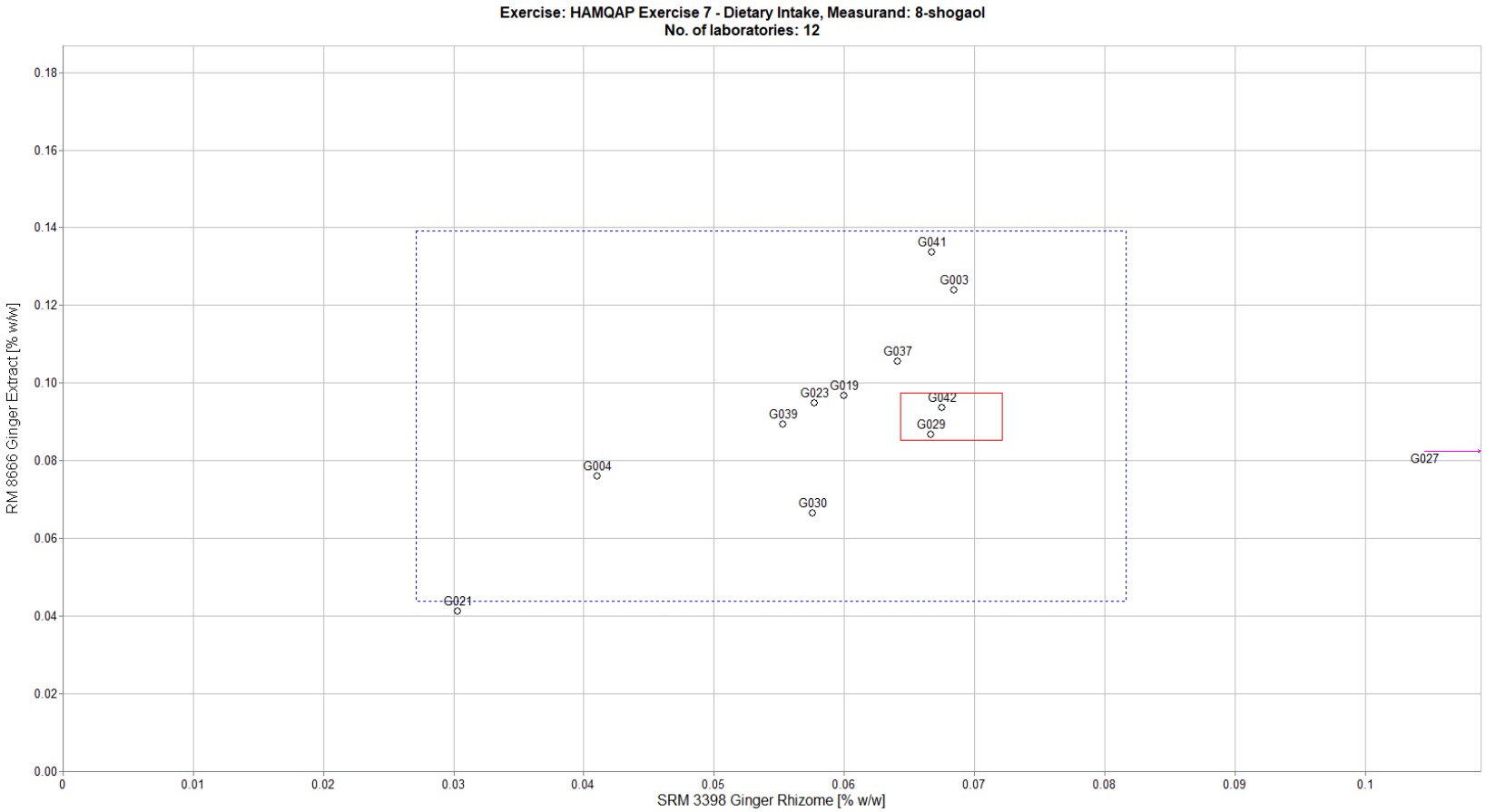

Fig. 5-18. Laboratory means for 8-shogaol in SRM 3398 Ginger (*Zingiber officinale*) Rhizome and RM 8666 Ginger (*Zingiber officinale*) Extract (sample/sample comparison view). In this view, the individual laboratory mean for one sample (SRM 3398) is compared to the individual laboratory mean for a second sample (RM 8666). The solid red box represents the NIST range of tolerance for the two samples, SRM 3398 (x-axis) and RM 8666 (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3398 (x-axis) and RM 8666 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

Table 5-10. Data summary table for 10-shogaol in ginger rhizome and ginger extract. Data points highlighted in blue have been identified as outside the consensus tolerance limits and resulted in an unacceptable Z'_{comm} score, $|Z'_{\text{comm}}| \geq 2$.

		10-shogaol									
		SRM 3398 Ginger Rhizome (% w/w)					RM 8666 Ginger Extract (% w/w)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				0.115	0.002				0.153	0.004
	G001										
	G003	0.104	0.104	0.101	0.103	0.002	0.141	0.137	0.139	0.139	0.002
	G004	0.1095	0.1018	0.1067	0.106	0.004	0.1579	0.1561	0.16	0.158	0.002
	G008										
	G009										
	G019	0.11	0.12	0.12	0.117	0.006	0.16	0.16	0.16	0.16	0
	G020										
	G021	0.088	0.094	0.089	0.090	0.003	0.13	0.143	0.143	0.139	0.008
	G023	0.1017	0.0963	0.096	0.098	0.003	0.1399	0.1391	0.1413	0.140	0.001
	G026										
	G027	0.09621	0.09524	0.09248	0.095	0.002	0.13828	0.14003	0.13617	0.138	0.002
	G029	0.13	0.13	0.13	0.13	0	0.17	0.17	0.17	0.17	0
	G030	0.129	0.126	0.127	0.127	0.002	0.168	0.165	0.166	0.166	0.002
	G033										
	G034										
	G036										
	G037	0.0238	0.024	0.0226	0.0235	0.0008	0.0212	0.0211	0.0206	0.0210	0.0003
	G039	0.135	0.135	0.135	0.1350	0.0000	0.189	0.192	0.209	0.197	0.011
	G041	0.105	0.107	0.104	0.105	0.002	0.152	0.151	0.149	0.151	0.002
	G042	0.106	0.105	0.105	0.1053	0.0006	0.129	0.13	0.175	0.145	0.026
	G044	0.0107	0.0108	0.011	0.0108	0.0002	0.0315	0.0307	0.0298	0.0307	0.0009
	G046	0.0887	0.0899	0.0903	0.0896	0.0008	0.0396	0.0641	0.0509	0.052	0.012
Community Results		Consensus Mean			0.106		Consensus Mean			0.145	
		Consensus Standard Deviation			0.022		Consensus Standard Deviation			0.033	
		Maximum			0.135		Maximum			0.197	
		Minimum			0.011		Minimum			0.021	
		N			14		N			14	

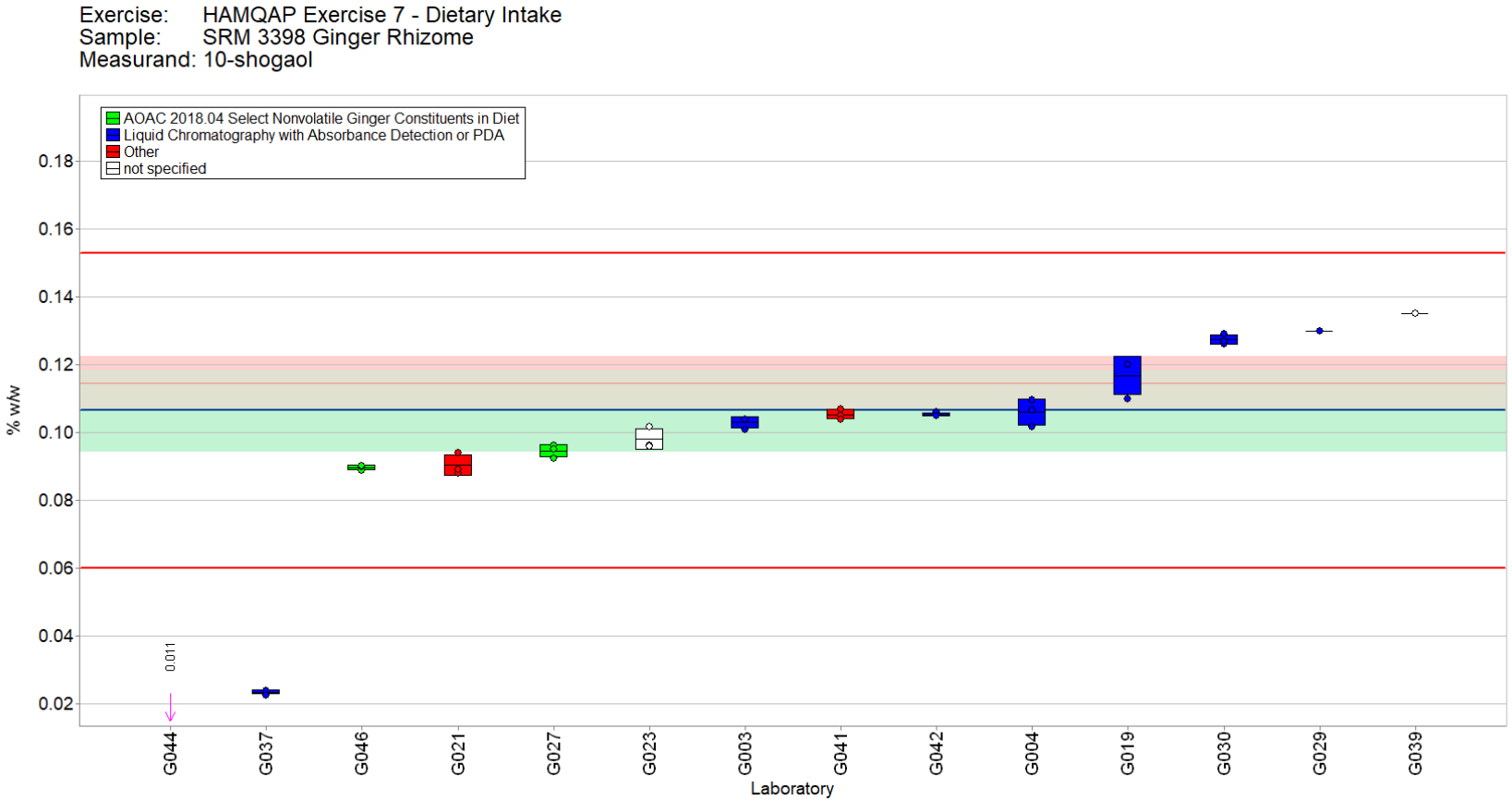

Fig. 5-19. 10-shogaol in SRM 3398 Ginger (*Zingiber officinale*) Rhizome (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The beige shaded region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

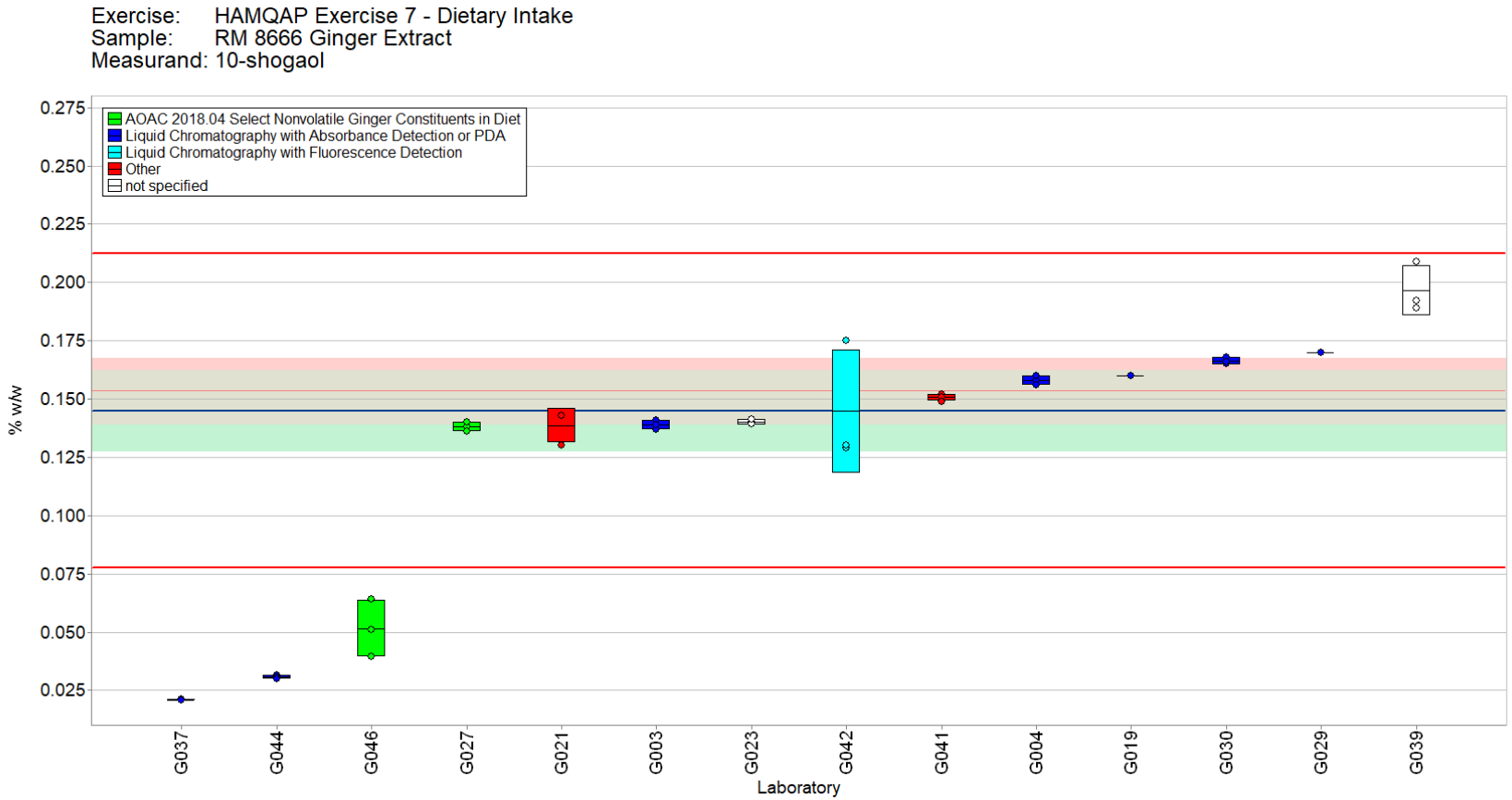

Fig. 5-20. 10-shogaol in RM 8666 Ginger (*Zingiber officinale*) Extract (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The beige shaded region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

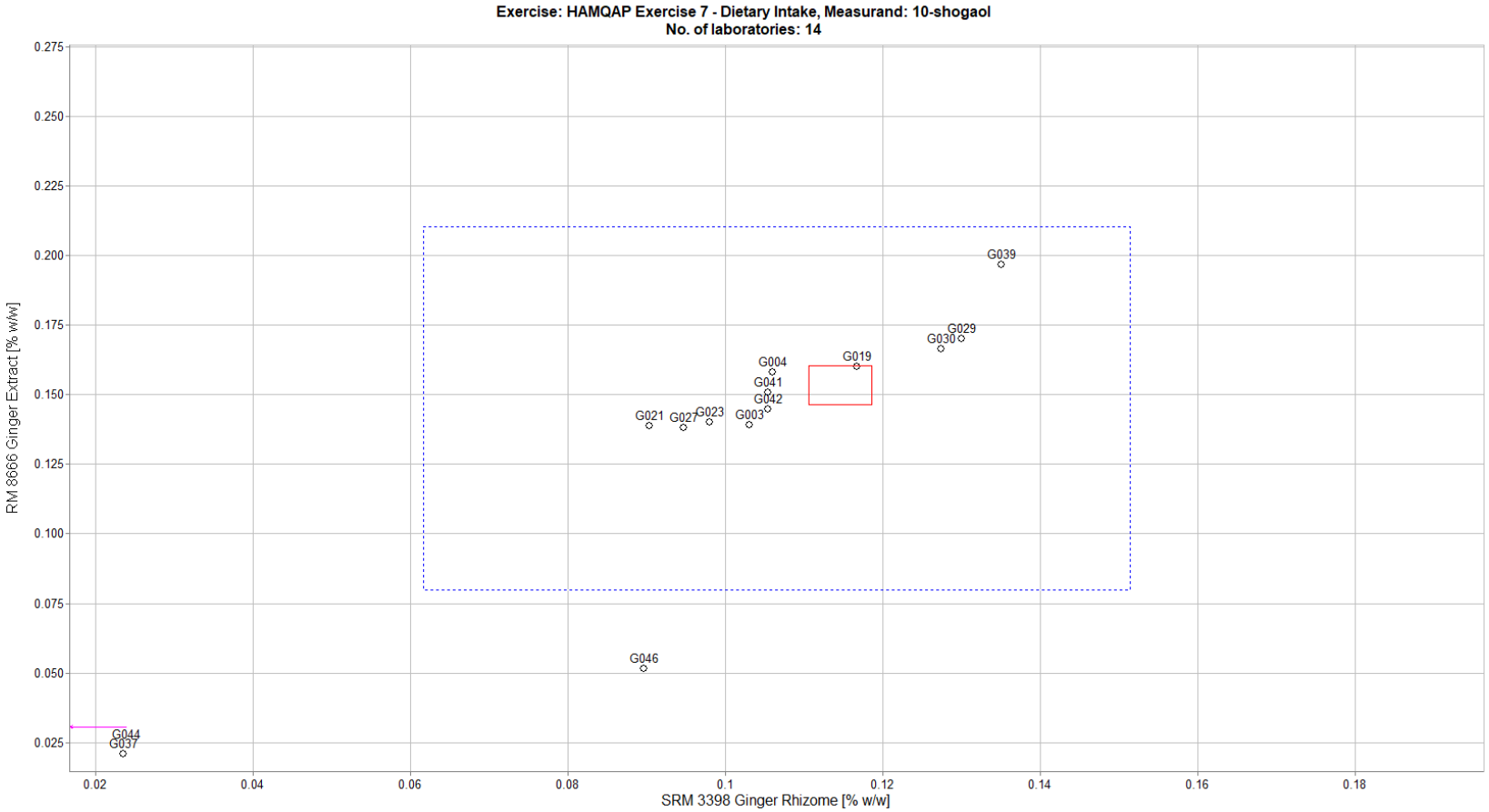

Fig. 5-21. Laboratory means for 10-shogaol in SRM 3398 Ginger (*Zingiber officinale*) Rhizome and RM 8666 Ginger (*Zingiber officinale*) Extract (sample/sample comparison view). In this view, the individual laboratory mean for one sample (SRM 3398) is compared to the individual laboratory mean for a second sample (RM 8666). The solid red box represents the NIST range of tolerance for the two samples, SRM 3398 (x-axis) and RM 8666 (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3398 (x-axis) and RM 8666 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

Table 5-11. Data summary table for 6-paradol in ginger rhizome and ginger extract. Data points highlighted in blue have been identified as outside the consensus tolerance limits and resulted in an unacceptable Z'_{comm} score, $|Z'_{\text{comm}}| \geq 2$. Data points highlighted in red have a zero or non-numeric data point.

		6-paradol									
		SRM 3398 Ginger Rhizome (% w/w)					RM 8666 Ginger Extract (% w/w)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target										
	G001										
	G003										
	G004										
	G008										
	G009										
	G019	0.02	0.03	0.04	0.030	0.010	0.11	0.15	0.16	0.140	0.026
	G020										
	G021										
	G023	0.0957	0.0922	0.109	0.0990	0.0089	0.1496	0.1437	0.1681	0.154	0.013
	G026										
	G027	0.01793	0.01777	0.0161	0.0173	0.0010	0.10056	0.10094	0.10834	0.1033	0.0044
	G029	0.01	0.01	0.01	0.01	0	0.08	0.08	0.07	0.0767	0.0058
	G030	<	<	<			0.0272	0.0295	0.025	0.0272	0.0023
	G033										
	G034										
	G036										
	G037	0.0186	0.019	0.0189	0.0188	0.0002	0.0954	0.0937	0.0954	0.0948	0.0010
	G039	0.011	0.01	0.009	0.0100	0.0010	0.067	0.071	0.073	0.0703	0.0031
	G041										
	G042	0.0132	0.0144	0.0142	0.0139	0.0006	0.0664	0.0618	0.0572	0.0618	0.0046
	G044										
	G046										
Community Results		Consensus Mean			0.017		Consensus Mean			0.091	
		Consensus Standard Deviation			0.010		Consensus Standard Deviation			0.053	
		Maximum			0.099		Maximum			0.154	
		Minimum			0.010		Minimum			0.027	
		N			7		N			8	

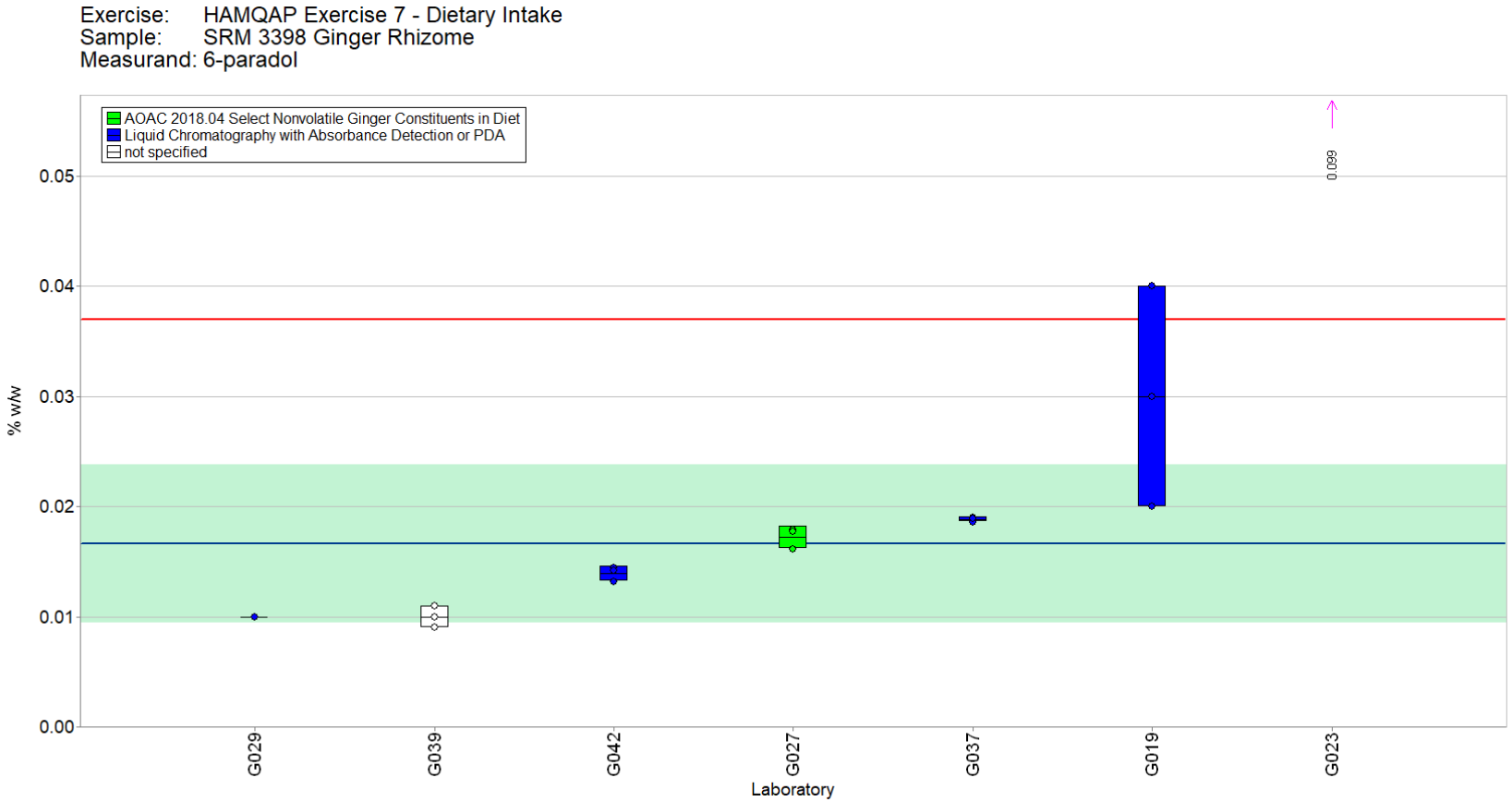

Fig. 5-22. 6-paradol in SRM 3398 Ginger (*Zingiber officinale*) Rhizome (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$, with the lower range set at zero.

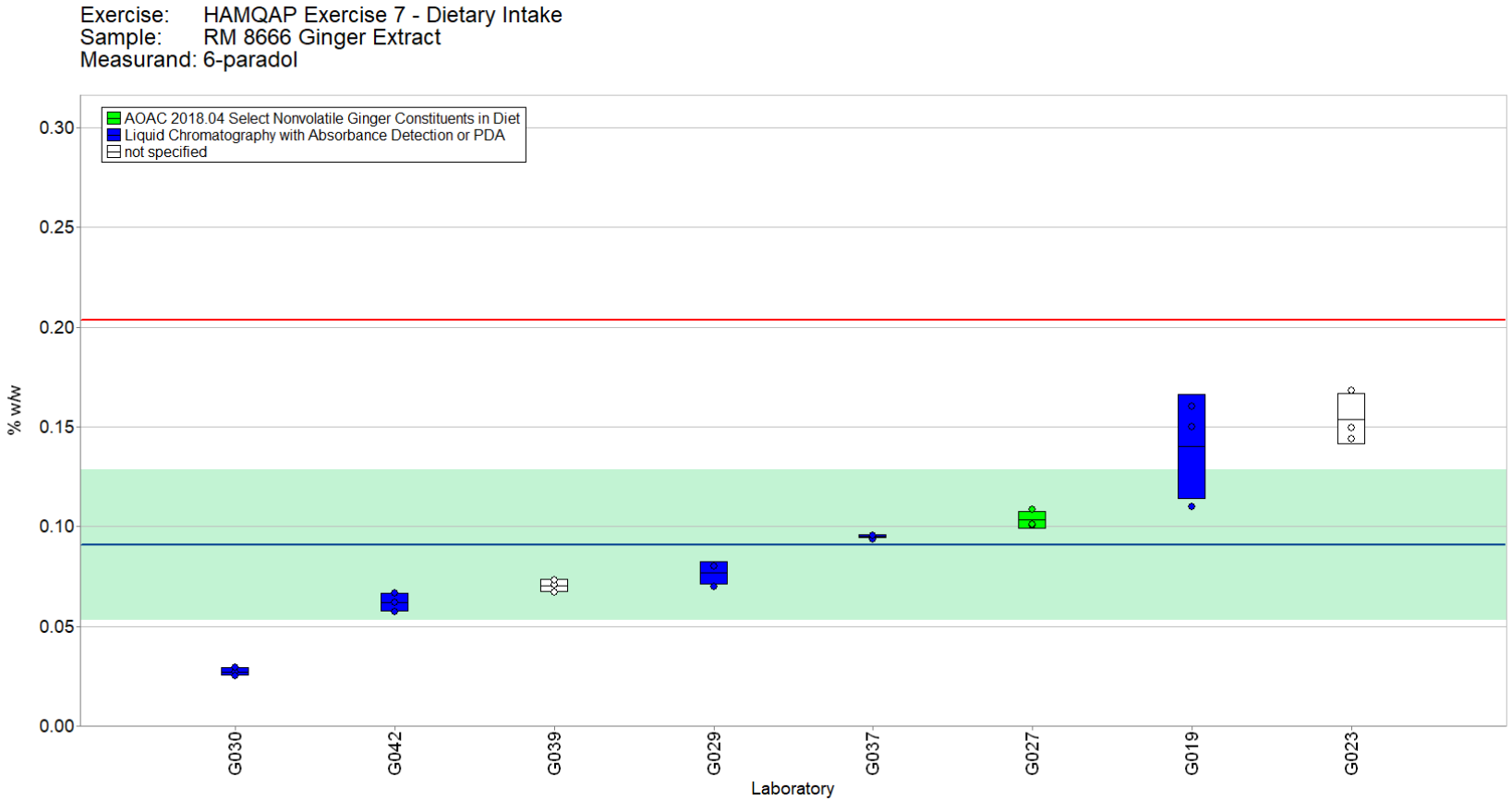

Fig. 5-23. 6-paradol in RM 8666 Ginger (*Zingiber officinale*) Extract (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$, with the lower range set at zero.

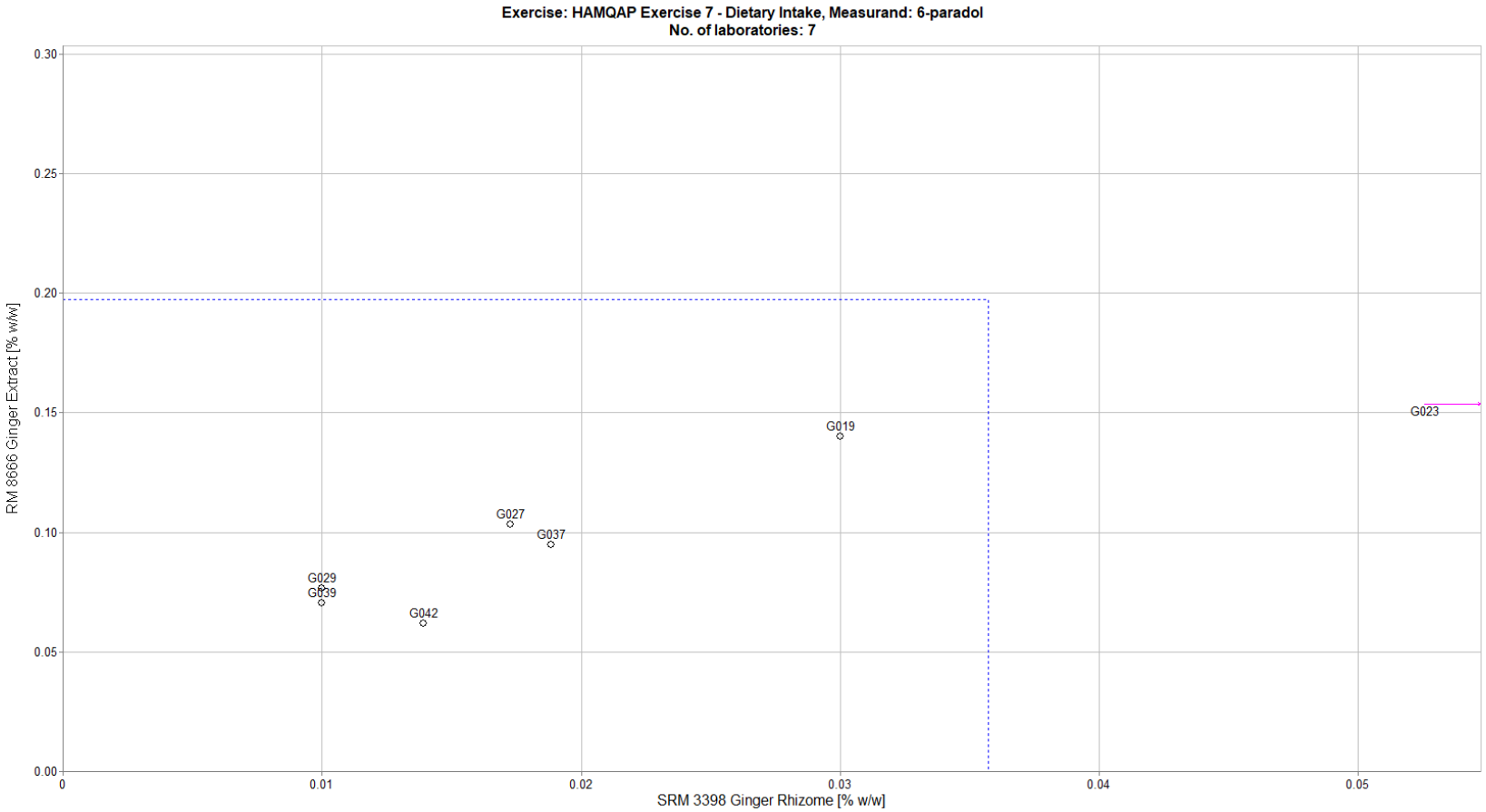

Fig. 5-24. Laboratory means for 6-paradol in SRM 3398 Ginger (*Zingiber officinale*) Rhizome and RM 8666 Ginger (*Zingiber officinale*) Extract (sample/sample comparison view). In this view, the individual laboratory mean for one sample (SRM 3398) is compared to the individual laboratory mean for a second sample (RM 8666). The dotted blue box represents the consensus range of tolerance for SRM 3398 (x-axis) and RM 8666 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

Table 5-12. Data summary table for zingerone in ginger rhizome and ginger extract. Data points highlighted in red have a zero or non-numeric data point.

		zingerone									
		SRM 3398 Ginger Rhizome (% w/w)					RM 8666 Ginger Extract (% w/w)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target										
	G001										
	G003										
	G004	0.01	0.0094	0.0113	0.0102	0.0010	0.0498	0.0516	0.0479	0.0498	0.0019
	G008										
	G009										
	G019	0.01	0.01	0.02	0.0133	0.0058	0.08	0.08	0.08	0.08	0
	G020										
	G021	< 0.030	< 0.030	< 0.030			0.05	0.048	0.043	0.0470	0.0036
	G023						0.0282	0.0283	0.0283	0.0283	0.0001
	G026										
	G027	0.01052	0.00703	0.00493	0.0075	0.0028	0.01436	0.01483	0.01548	0.0149	0.0006
	G029	< 0.010	< 0.010	< 0.010			< 0.010	< 0.010	< 0.010		
	G030	<	<	<			0.0207	0.0239	0.0252	0.0233	0.0023
	G033										
	G034										
	G036										
	G037										
	G039	0.02	0.02	0.021	0.0203	0.0006	0.073	0.073	0.077	0.0743	0.0023
	G041										
	G042	0.0071	0.0071	0.0077	0.0073	0.0003	0.0199	0.0207	0.022	0.0209	0.0011
	G044										
	G046								0.0338	0.0338	
Community Results		Consensus Mean			0.0110		Consensus Mean			0.0410	
		Consensus Standard Deviation			0.0050		Consensus Standard Deviation			0.0250	
		Maximum			0.0203		Maximum			0.0800	
		Minimum			0.0073		Minimum			0.0149	
		N			5		N			8	

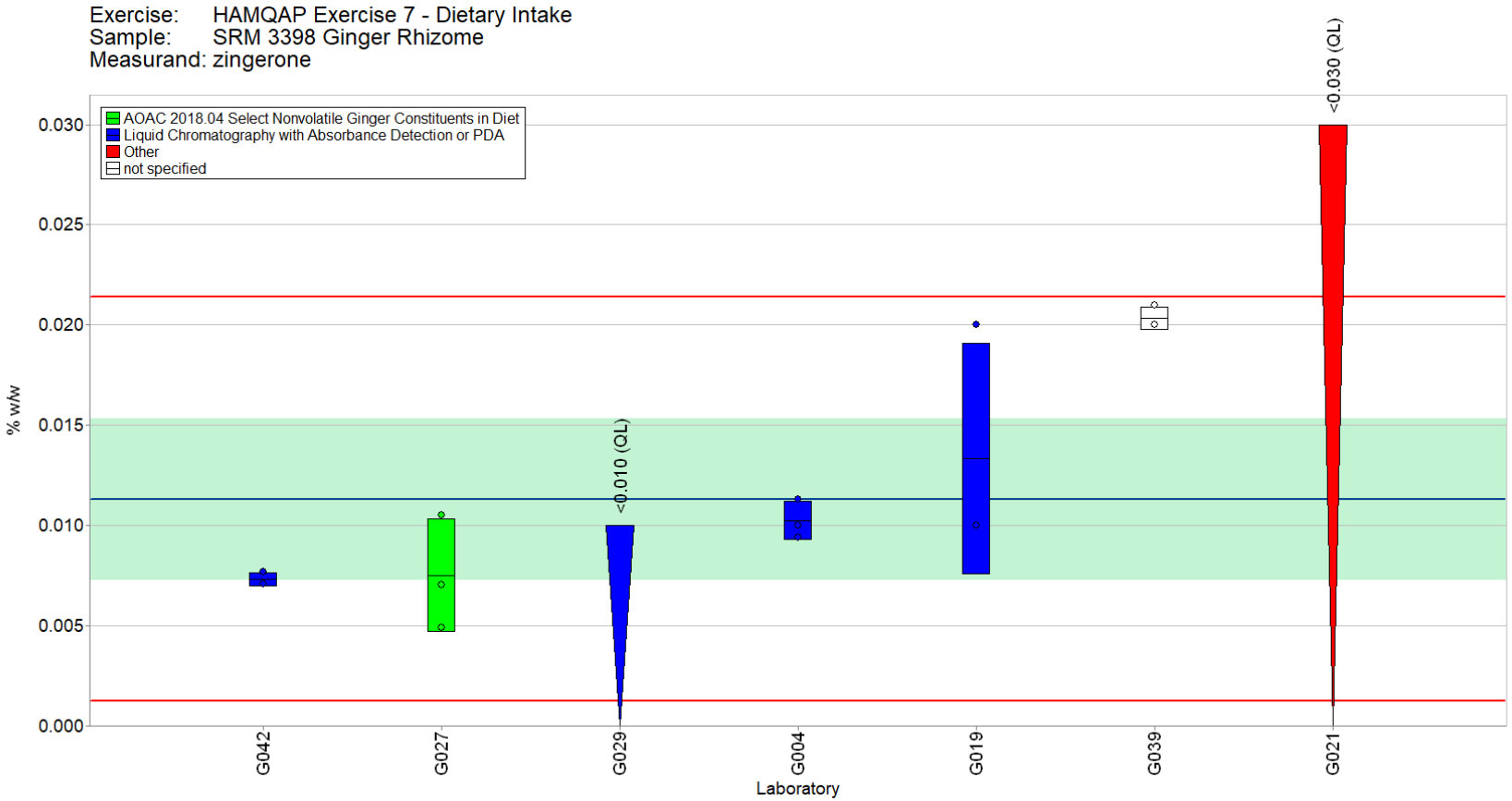

Fig. 5-25. Zingerone in SRM 3398 Ginger (*Zingiber officinale*) Rhizome (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

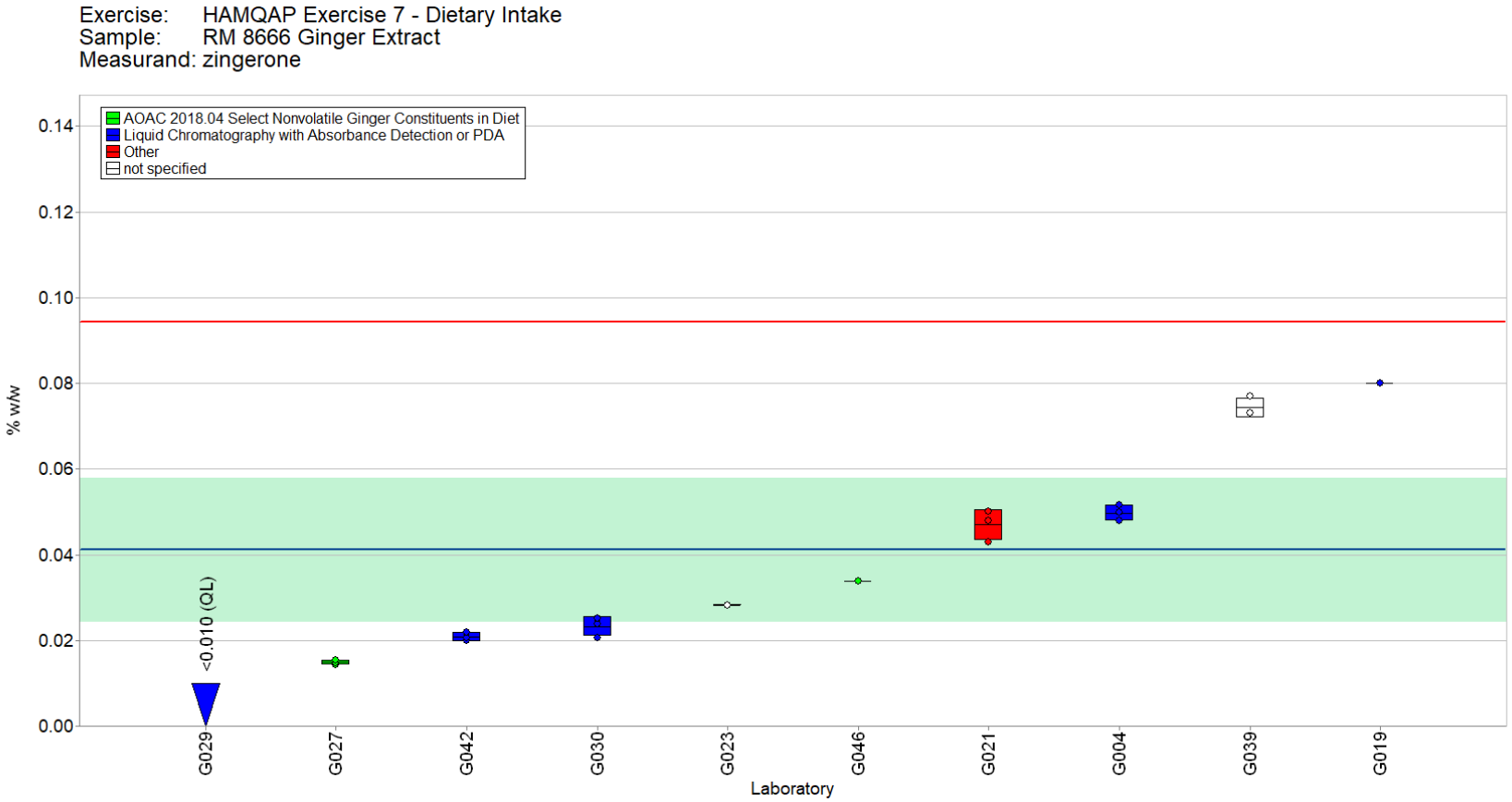

Fig. 5-26. Zingerone in RM 8666 Ginger (*Zingiber officinale*) Extract (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). A downward triangle represents data reported as an LOQ value. The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$, with the lower range set at zero.

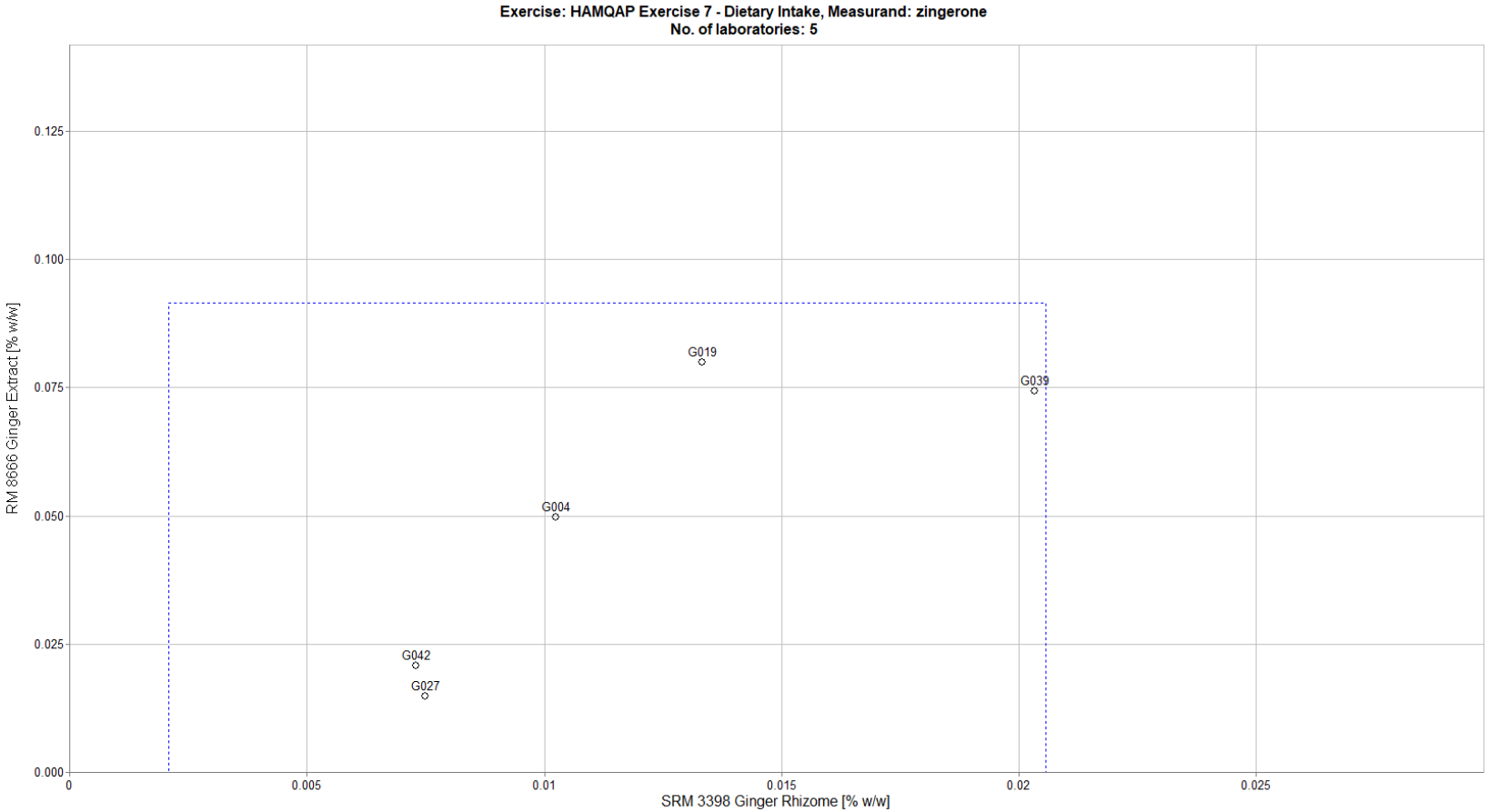

Fig. 5-27. Laboratory means for zingerone in SRM 3398 Ginger (*Zingiber officinale*) Rhizome and RM 8666 Ginger (*Zingiber officinale*) Extract (sample/sample comparison view). In this view, the individual laboratory mean for one sample (SRM 3398) is compared to the individual laboratory mean for a second sample (RM 8666). The dotted blue box represents the consensus range of tolerance for SRM 3398 (x-axis) and RM 8666 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

Table 5-13. Data summary table for Total Ginger Constituents in eight ginger containing materials. Data points highlighted in blue have been identified as outside the consensus tolerance limits and resulted in an unacceptable Z'_{comm} score, $|Z'_{\text{comm}}| \geq 2$.

		Total Ginger Constituents																	
		Individual Results												Community Results					
		Target	G003	G004	G019	G021	G023	G027	G029	G030	G037	G039	G046	Mean	SD	RSD	Max	Min	N
USP 1291446; Ginger Constituent Mixture (% w/w)	A			27.002	8.78		11.0019		22.11	74.5	14.6								
	B			17.1323	8.65		9.6364		25.26	74.51	14.4								
	C			22.8797	8.56		36.0376		25.51	74.81	14.3								
	Avg			22.3	8.66		18.9		24.3	74.61	14.43			22.4	16.8	75%	74.61	8.66	6
	SD			5.0	0.11		14.9		1.9	0.18	0.15								
USP 1291504; Powdered Ginger (% w/w)	A			1.1021	0.96	1.025	1.4057	1.10721	1.04	0.931	1.05	1.152	0.7096						
	B			1.1127	1		1.4449	1.08205	1.09	0.961	1.07	1.132	0.8563						
	C			1.123	1.01		1.4419	1.08317	1.1	0.986	1.09	1.12	0.8685						
	Avg	1.074		1.113	0.990	1.025	1.431	1.091	1.077	0.959	1.070	1.135	0.811	1.06	0.12	12%	1.43	0.81	9
	SD	0.054		0.010	0.026		0.022	0.014	0.032	0.028	0.020	0.016	0.088						
Ginger Supplement A; Tablet (% w/w)	A			0.4361	0.39	0.418	0.7336	0.44844	0.45	0.424	0.418	0.467	0.1626						
	B			0.4341	0.39	0.412	0.7059	0.44873	0.49	0.444	0.409	0.47	0.2077						
	C			0.4249	0.42	0.412	0.6945	0.44809	0.45	0.433	0.399	0.47	0.1679						
	Avg			0.432	0.400	0.414	0.711	0.4484	0.463	0.434	0.409	0.469	0.179	0.434	0.051	12%	0.71	0.18	10
	SD			0.006	0.017	0.003	0.020	0.0003	0.023	0.010	0.010	0.002	0.025						
Ginger Supplement B; Capsule (% w/w)	A			1.4547	1.4	1.459	1.951	1.66323	1.61	1.53	1.4	1.666	1.2267						
	B			1.4494	1.41	1.477	1.9937	1.64663	1.63	1.51	1.46	1.672	1.1303						
	C			1.4515	1.35	1.486	1.9733	1.63479	1.65	1.54	1.43	1.664	1.2264						
	Avg			1.452	1.387	1.474	1.973	1.648	1.630	1.527	1.430	1.667	1.194	1.53	0.22	14%	1.97	1.19	10
	SD			0.003	0.032	0.014	0.021	0.014	0.020	0.015	0.030	0.004	0.056						
Ginger Supplement C; Softgel with Oleoresin (% w/w)	A			9.5402	9.17	14.147	27.0484	8.99007	8.52	6.76	8.8	9.907	7.7953						
	B			9.5	9.14	13.894	27.2777	9.01803	8.72	6.57	8.79	9.899	7.9442						
	C			9.4255	9.45	13.795	26.6017	9.0324	8.68	6.6	8.9	9.901	7.6857						
	Avg			9.49	9.25	13.95	26.98	9.01	8.64	6.64	8.83	9.902	7.81	9.0	1.7	19%	26.98	6.64	10
	SD			0.06	0.17	0.18	0.34	0.02	0.11	0.10	0.06	0.004	0.13						
Ginger Supplement D; Tincture (% w/w)	A			0.251	0.2	0.213	0.5284	0.24972	0.24	0.194	0.221	0.249	0.1673						
	B			0.2507	0.19		0.315	0.24784	0.24	0.223	0.243	0.264	0.1599						
	C			0.25	0.2		0.3185	0.24304	0.23	0.205	0.226	0.254	0.172						
	Avg			0.251	0.197	0.213	0.387	0.247	0.237	0.207	0.230	0.256	0.166	0.227	0.044	19%	0.39	0.17	9
	SD			0.001	0.006		0.122	0.003	0.006	0.015	0.012	0.008	0.006						
RM 8666 Ginger Extract (% w/w)	A			3.5495	3.09	3.263	4.8886	2.16379	3.71	1.93	3.57	3.559	1.8902						
	B			3.559	3.24	3.22	4.8889	2.23784	3.66	2.15	3.57	3.655	2.023						
	C			3.6191	3.21	3.262	4.9533	2.24583	3.63	1.9	3.6	3.675	2.1883						
	Avg	3.791		3.58	3.18	3.25	4.91	2.22	3.67	1.99	3.58	3.63	2.03	3.23	0.57	18%	4.91	1.99	11
	SD	0.038		0.04	0.08	0.02	0.04	0.05	0.04	0.14	0.02	0.06	0.15						
SRM 3398 Ginger Rhizome (% w/w)	A			0.5821	0.57	0.553	1.113	0.65259	0.86	0.609	0.457	0.737	0.3925						
	B			0.5442	0.59	0.6	1.089	0.65443	0.84	0.616	0.463	0.735	0.4005						
	C			0.5819	0.63	0.578	1.1645	0.65547	0.85	0.619	0.477	0.733	0.4019						
	Avg	0.939		0.569	0.597	0.577	1.122	0.654	0.850	0.615	0.466	0.735	0.398	0.63	0.17	27%	1.12	0.40	11
	SD	0.008		0.022	0.031	0.024	0.039	0.001	0.010	0.005	0.010	0.002	0.005						

Table 5-14. Data summary table for 6-gingerol in eight ginger containing materials. Data points highlighted in blue have been identified as outside the consensus tolerance limits and resulted unacceptable Z'_{comm} score, $|Z'_{\text{comm}}| \geq 2$.

		6-gingerol																	
		Individual Results												Community Results					
		Target	G003	G004	G019	G021	G023	G027	G029	G030	G037	G039	G046	Mean	SD	RSD	Max	Min	N
USP 1291446; Ginger Constituent Mixture (% w/w)	**A**			9.2562	4.68		4.9711		10.6	32.02	6.99								
	B			5.0565	4.65		4.3488		12.03	32.05	6.87								
	C			6.7353	4.58		17.2109		12.16	32.19	6.87								
	Avg	8.70		7.02	4.64		8.84		11.60	32.09	6.91			7.80	5.21	67%	32.1	4.6	6
	SD	1.74		2.11	0.05		7.25		0.87	0.09	0.07								
USP 1291504; Powdered Ginger (% w/w)	**A**			0.3937	0.4	0.417	0.5507	0.46616	0.45	0.355	0.447	0.495	0.3588						
	B			0.4135	0.39		0.5603	0.45511	0.46	0.363	0.455	0.489	0.3825						
	C			0.4053	0.4		0.5508	0.45688	0.47	0.357	0.464	0.478	0.3827						
	Avg	0.554		0.404	0.397	0.417	0.554	0.459	0.460	0.358	0.455	0.487	0.375	0.436	0.074	17%	0.554	0.358	9
	SD	0.015		0.010	0.006		0.006	0.006	0.010	0.004	0.009	0.009	0.014						
Ginger Supplement A; Tablet (% w/w)	**A**			0.1792	0.17	0.187	0.2958	0.20255	0.2	0.169	0.199	0.218	0.1161						
	B			0.176	0.17	0.187	0.2851	0.19865	0.21	0.173	0.199	0.221	0.1352						
	C			0.1757	0.18	0.187	0.2898	0.1974	0.2	0.169	0.192	0.221	0.1205						
	Avg			0.177	0.173	0.187	0.290	0.200	0.203	0.170	0.197	0.220	0.124	0.191	0.037	19%	0.290	0.124	10
	SD			0.002	0.006	0	0.005	0.003	0.006	0.002	0.004	0.002	0.010						
Ginger Supplement B; Capsule (% w/w)	**A**			0.686	0.76	0.797	0.9313	0.84419	0.8	0.682	0.745	0.876	0.6938						
	B			0.6819	0.75	0.784	0.9446	0.83489	0.82	0.676	0.779	0.872	0.6247						
	C			0.6889	0.69	0.783	0.9471	0.83647	0.84	0.686	0.763	0.873	0.695						
	Avg			0.686	0.733	0.788	0.941	0.839	0.820	0.681	0.762	0.874	0.671	0.779	0.123	16%	0.941	0.671	10
	SD			0.004	0.038	0.008	0.008	0.005	0.020	0.005	0.017	0.002	0.040						
Ginger Supplement C; Softgel with Oleoresin (% w/w)	**A**			5.4426	5.6	5.203	6.529	5.1614	4.83	3.8	5.18	6.003	5.0421						
	B			5.4554	5.5	5.24	6.5724	5.14159	4.88	3.78	5.1	6.022	5.1923						
	C			5.4507	5.7	5.165	6.4097	5.16738	4.81	3.76	5.26	5.97	4.8964						
	Avg			5.450	5.60	5.20	6.50	5.157	4.84	3.78	5.18	6.00	5.04	5.31	0.66	13%	6.50	3.78	10
	SD			0.006	0.10	0.04	0.08	0.013	0.04	0.02	0.08	0.03	0.15						
Ginger Supplement D; Tincture (% w/w)	**A**			0.107	0.09	0.101	0.2958	0.11029	0.11	0.0832	0.109	0.112	0.0857						
	B			0.1075	0.09		0.1367	0.11036	0.11	0.0918	0.123	0.123	0.0842						
	C			0.1074	0.09		0.1395	0.10842	0.11	0.0864	0.111	0.119	0.0878						
	Avg			0.1073	0.09	0.101	0.191	0.110	0.11	0.087	0.114	0.118	0.086	0.103	0.02	18%	0.2	0.1	9
	SD			0.0003	0		0.091	0.001	0	0.004	0.008	0.006	0.002						
RM 8666 Ginger Extract (% w/w)	**A**		2.11	1.8686	1.75	1.931	2.5753	0.93698	2.12	0.817	2.05	2.021	1.2889						
	B		2.02	1.8775	1.85	1.916	2.583	0.98131	2.08	0.965	2.04	2.072	1.3635						
	C		2.05	1.906	1.8	1.924	2.5936	0.98892	2.08	0.814	2.06	2.065	1.4437						
	Avg	2.230	2.06	1.88	1.80	1.924	2.584	0.97	2.09	0.87	2.05	2.05	1.37	1.90	0.42	22%	2.58	0.87	15
	SD	0.036	0.05	0.02	0.05	0.008	0.009	0.03	0.02	0.09	0.01	0.03	0.08						
SRM 3398 Ginger Rhizome (% w/w)	**A**		0.261	0.121	0.15	0.132	0.2944	0.16896	0.31	0.121	0.0555	0.228	0.1221						
	B		0.275	0.111	0.14	0.138	0.2854	0.16871	0.3	0.121	0.058	0.231	0.1136						
	C		0.274	0.1258	0.15	0.143	0.3054	0.16736	0.3	0.119	0.0576	0.233	0.1166						
	Avg	0.366	0.270	0.119	0.147	0.138	0.295	0.1683	0.303	0.1203	0.0570	0.231	0.117	0.186	0.072	39%	0.303	0.057	15
	SD	0.005	0.008	0.008	0.006	0.006	0.010	0.0009	0.006	0.0012	0.0013	0.003	0.004						

Table 5-15. Data summary table for 8-gingerol in eight ginger containing materials. Data points highlighted in blue have been identified as outside the consensus tolerance limits and resulted in an unacceptable Z'_{comm} score, $|Z'_{\text{comm}}| \geq 2$. Data points highlighted in red have a zero or non-numeric data point.

		8-gingerol																		
		Individual Results												Community Results						
		Target	G003	G004	G019	G021	G023	G027	G029	G030	G037	G039	G046	Mean	SD	RSD	Max	Min	N
USP 1291446; Ginger Constituent Mixture (% w/w)	**A**			4.7726	0				< 0.010	<									
	B			2.3278	0				< 0.010	<									
	C			2.9489	0				< 0.010	<									
	Avg			3.35	0									1.675	5.510	329%	3.350	0.000	2
	SD			1.27	0														
USP 1291504; Powdered Ginger (% w/w)	**A**			0.1444	0.12	0.128	0.2494	0.12259	0.1	0.092	0.121	0.114	0.0842						
	B			0.1436	0.13		0.25	0.11959	0.11	0.093	0.122	0.114	0.0943						
	C			0.1477	0.14		0.254	0.11964	0.11	0.107	0.121	0.109	0.0795						
	Avg	0.140		0.145	0.130	0.128	0.251	0.121	0.107	0.097	0.121	0.112	0.086	0.116	0.027	23%	0.251	0.086	9
	SD	0.019		0.002	0.010		0.003	0.002	0.006	0.008	0.001	0.003	0.008						
Ginger Supplement A; Tablet (% w/w)	**A**			0.0465	0.03	0.04	0.1125	0.03559	0.03	0.03	0.0326	0.031							
	B			0.0456	0.03	0.038	0.1088	0.03622	0.04	0.0307	0.0324	0.031	0.0133						
	C			0.0446	0.04	0.039	0.1101	0.03525	0.03	0.0296	0.0313	0.032	0.0091						
	Avg			0.046	0.033	0.039	0.110	0.036	0.033	0.030	0.032	0.031	0.011	0.033	0.010	30%	0.110	0.011	10
	SD			0.001	0.006	0.001	0.002	0.000	0.006	0.001	0.001	0.001	0.003						
Ginger Supplement B; Capsule (% w/w)	**A**			0.1825	0.15	0.151	0.2749	0.16445	0.15	0.141	0.134	0.145	0.104						
	B			0.1798	0.15	0.151	0.283	0.15941	0.15	0.137	0.14	0.145	0.0926						
	C			0.1809	0.14	0.155	0.278	0.16312	0.15	0.143	0.138	0.143	0.0986						
	Avg			0.181	0.147	0.152	0.279	0.162	0.150	0.140	0.137	0.144	0.098	0.148	0.022	15%	0.279	0.098	10
	SD			0.001	0.006	0.002	0.004	0.003	0.000	0.003	0.003	0.001	0.006						
Ginger Supplement C; Softgel with Oleoresin (% w/w)	**A**			1.1162	1.4	0.949	2.2656	0.96207	0.96	0.915	1.01	0.969	0.7305						
	B			1.122	1.45	0.922	2.3603	0.97453	0.97	0.823	0.997	0.988	0.7132						
	C			1.1173	1.5	0.935	2.2195	0.97081	0.98	0.882	1.05	0.977	0.7114						
	Avg			1.119	1.450	0.935	2.282	0.969	0.970	0.873	1.019	0.978	0.718	0.981	0.177	18%	2.282	0.718	10
	SD			0.003	0.050	0.014	0.072	0.006	0.010	0.047	0.028	0.010	0.011						
Ginger Supplement D; Tincture (% w/w)	**A**			0.0345	0.03	0.026	0.1125	0.02598	0.02	0.0229	0.025	0.022	0.0144						
	B			0.0345	0.02		0.051	0.02639	0.02	0.0219	0.028	0.027	0.0157						
	C			0.0345	0.03		0.0503	0.02537	0.02	0.0219	0.025	0.023	0.0185						
	Avg			0.035	0.027	0.026	0.071	0.026	0.020	0.022	0.026	0.024	0.016	0.025	0.009	36%	0.071	0.016	9
	SD			0.000	0.006		0.036	0.001	0.000	0.001	0.002	0.003	0.002						
RM 8666 Ginger Extract (% w/w)	**A**		0.35	0.3903	0.26	0.287	0.7163	0.16261	0.29	0.145	0.283	0.244	0.1189						
	B		0.336	0.3908	0.26	0.279	0.7436	0.16834	0.29	0.167	0.281	0.252	0.1311						
	C		0.338	0.401	0.27	0.28	0.7291	0.17056	0.29	0.134	0.283	0.248	0.1476						
	Avg	0.355	0.341	0.394	0.263	0.282	0.730	0.167	0.290	0.149	0.282	0.248	0.133	0.301	0.179	59%	0.987	0.133	15
	SD	0.008	0.008	0.006	0.006	0.004	0.014	0.004	0.000	0.017	0.001	0.004	0.014						
SRM 3398 Ginger Rhizome (% w/w)	**A**		0.0507	0.0508	0.02	0.04	0.2247	0.03027	0.03	0.0263	0.0295	0.023							
	B		0.05	0.0438	0.03	0.046	0.2413	0.0303	0.03	0.0259	0.0287	0.024	0.0094						
	C		0.0512	0.046	0.03	0.045	0.2474	0.03374	0.03	0.026	0.0287	0.026	0.0086						
	Avg	0.057	0.051	0.047	0.027	0.044	0.238	0.031	0.030	0.026	0.029	0.024	0.009	0.034	0.017	50%	0.260	0.009	15
	SD	0.001	0.001	0.004	0.006	0.003	0.012	0.002	0.000	0.000	0.000	0.002	0.001						

Table 5-16. Data summary table for 10-gingerol in eight ginger containing materials. Data points highlighted in blue have been identified as outside the consensus tolerance limits and resulted in an unacceptable Z'_{comm} score, $|Z'_{\text{comm}}| \geq 2$. Data points highlighted in red have a zero or non-numeric data point.

		10-gingerol																	
		Individual Results												Community Results					
		Target	G003	G004	G019	G021	G023	G027	G029	G030	G037	G039	G046	Mean	SD	RSD	Max	Min	N
USP 1291446; Ginger Constituent Mixture (% w/w)	**A**			2.508	0				< 0.010	<									
	B			2.1144	0				< 0.010	<									
	C			2.9525	0				< 0.010	<									
	Avg			2.52	0									1.26	4.91	389%	2.52	0.00	2
	SD			0.42	0														
USP 1291504; Powdered Ginger (% w/w)	**A**			0.2566	0.18	0.261	0.2946	0.25097	0.21	0.236	0.192	0.231	0.2128						
	B			0.2528	0.19		0.3103	0.24328	0.22	0.247	0.199	0.228	0.2257						
	C			0.2628	0.18		0.3095	0.24681	0.22	0.246	0.198	0.231	0.2725						
	Avg	0.176		0.257	0.183	0.261	0.305	0.247	0.217	0.243	0.196	0.230	0.237	0.237	0.042	18%	0.305	0.183	9
	SD	0.014		0.005	0.006		0.009	0.004	0.006	0.006	0.004	0.002	0.031						
Ginger Supplement A; Tablet (% w/w)	**A**			0.0502	0.03	0.06	0.0758	0.0431	0.04	0.048	0.0421	0.04	0.0266						
	B			0.0532	0.03	0.059	0.0671	0.04037	0.05	0.06	0.0405	0.04	0.0325						
	C			0.0476	0.04	0.059	0.064	0.0193	0.04	0.058	0.0349	0.041	0.025						
	Avg			0.050	0.033	0.059	0.069	0.034	0.043	0.055	0.039	0.040	0.028	0.045	0.016	36%	0.069	0.028	10
	SD			0.003	0.006	0.001	0.006	0.013	0.006	0.006	0.004	0.001	0.004						
Ginger Supplement B; Capsule (% w/w)	**A**			0.2724	0.17	0.243	0.3514	0.3087	0.24	0.343	0.221	0.262	0.2627						
	B			0.2743	0.18	0.268	0.3608	0.30863	0.23	0.339	0.231	0.269	0.264						
	C			0.2719	0.19	0.275	0.352	0.29806	0.23	0.344	0.228	0.269	0.2692						
	Avg			0.273	0.180	0.262	0.355	0.305	0.233	0.342	0.227	0.267	0.265	0.271	0.072	27%	0.355	0.180	10
	SD			0.001	0.010	0.017	0.005	0.006	0.006	0.003	0.005	0.004	0.003						
Ginger Supplement C; Softgel with Oleoresin (% w/w)	**A**			1.7303	0.76	2.51	14.165	1.71996	1.42	0.611	1.47	1.233	1.2909						
	B			1.7266	0.77	2.26	14.2252	1.74979	1.45	0.604	1.47	1.236	1.2888						
	C			1.6832	0.8	2.255	13.9614	1.74107	1.45	0.621	1.49	1.236	1.2752						
	Avg			1.71	0.78	2.34	14.12	1.74	1.44	0.612	1.48	1.235	1.285	1.40	0.66	47%	14.12	0.61	10
	SD			0.03	0.02	0.15	0.14	0.02	0.02	0.009	0.01	0.002	0.009						
Ginger Supplement D; Tincture (% w/w)	**A**			0.0578	0.04	0.046	0.0646	0.05889	0.05	0.042	0.044	0.053	0.0513						
	B			0.0576	0.04		0.0653	0.0592	0.05	0.0536	0.046	0.053	0.0455						
	C			0.0576	0.04		0.0666	0.05658	0.04	0.0475	0.046	0.053	0.0488						
	Avg			0.0577	0.04	0.046	0.066	0.058	0.047	0.048	0.045	0.0530	0.049	0.051	0.010	20%	0.066	0.040	9
	SD			0.0001	0		0.001	0.001	0.006	0.006	0.001	0.0000	0.003						
RM 8666 Ginger Extract (% w/w)	**A**		0.519	0.5457	0.28	0.446	0.6162	0.30246	0.42	0.306	0.449	0.416	0.3303						
	B		0.498	0.5483	0.29	0.426	0.5849	0.30656	0.41	0.334	0.474	0.439	0.3485						
	C		0.509	0.5421	0.27	0.452	0.6124	0.31184	0.41	0.28	0.474	0.437	0.373						
	Avg	0.443	0.509	0.545	0.280	0.441	0.605	0.307	0.413	0.307	0.466	0.431	0.351	0.410	0.172	42%	0.933	0.113	15
	SD	0.005	0.011	0.003	0.010	0.014	0.017	0.005	0.006	0.027	0.014	0.013	0.021						
SRM 3398 Ginger Rhizome (% w/w)	**A**		0.0569	0.0578	0.04	0.09	0.0996	0.05046	0.06	0.0525	0.0401	0.055	0.0439						
	B		0.0563	0.0561	0.04	0.103	0.0752	0.0517	0.06	0.0595	0.0367	0.055	0.0443						
	C		0.0554	0.0613	0.04	0.101	0.1054	0.05245	0.06	0.0597	0.0479	0.053	0.0448						
	Avg	0.083	0.0562	0.0584	0.0400	0.0980	0.0934	0.0515	0.0600	0.0572	0.0416	0.0543	0.0443	0.058	0.015	26%	0.460	0.040	15
	SD	0.002	0.0008	0.0027	0.0000	0.0070	0.0160	0.0010	0.0000	0.0041	0.0057	0.0012	0.0005						

Table 5-17. Data summary table for 6-shogaol in eight ginger containing materials. Data points highlighted in blue have been identified as outside the consensus tolerance limits and resulted in an unacceptable Z'_{comm} score, $|Z'_{\text{comm}}| \geq 2$.

		6-shogaol																	
		Individual Results												Community Results					
		Target	G003	G004	G019	G021	G023	G027	G029	G030	G037	G039	G046	Mean	SD	RSD	Max	Min	N
USP 1291446; Ginger Constituent Mixture (% w/w)	A			8.7822	4.1		6.0308		11.51	42.48	7.66								
	B			4.8091	4		5.2876		12.23	42.46	7.56								
	C			6.0979	3.98		18.8267		13.35	42.62	7.47								
	Avg	12.30		6.56	4.03		10.05		12.36	42.52	7.56			8.11	7.52	93%	42.52	4.03	6
	SD	2.46		2.03	0.06		7.61		0.93	0.09	0.10								
USP 1291504; Powdered Ginger (% w/w)	A			0.1549	0.12	0.139	0.189	0.16863	0.16	0.161	0.156	0.169	0.0258						
	B			0.1527	0.12		0.1921	0.16538	0.17	0.164	0.16	0.169	0.0855						
	C			0.1566	0.13		0.1942	0.16252	0.17	0.164	0.165	0.17	0.0735						
	Avg	0.116		0.155	0.123	0.139	0.192	0.166	0.167	0.163	0.160	0.169	0.062	0.160	0.021	13%	0.192	0.062	9
	SD	0.007		0.002	0.006		0.003	0.003	0.006	0.002	0.005	0.001	0.032						
Ginger Supplement A; Tablet (% w/w)	A			0.0966	0.08	0.085	0.1251	0.10523	0.11	0.113	0.108	0.1	0.0114						
	B			0.095	0.08	0.083	0.1251	0.10944	0.12	0.116	0.103	0.101	0.0208						
	C			0.0954	0.08	0.082	0.1213	0.10491	0.11	0.113	0.106	0.1	0.0133						
	Avg			0.096	0.08	0.083	0.124	0.107	0.113	0.114	0.106	0.100	0.015	0.101	0.022	22%	0.124	0.015	10
	SD			0.001	0	0.002	0.002	0.003	0.006	0.002	0.003	0.001	0.005						
Ginger Supplement B; Capsule (% w/w)	A			0.2005	0.17	0.19	0.2449	0.23409	0.25	0.232	0.214	0.223	0.1082						
	B			0.1994	0.17	0.189	0.2488	0.23498	0.25	0.228	0.223	0.223	0.0927						
	C			0.1988	0.16	0.189	0.2467	0.23023	0.25	0.232	0.219	0.221	0.1066						
	Avg			0.200	0.167	0.189	0.247	0.233	0.25	0.231	0.219	0.222	0.103	0.211	0.041	19%	0.250	0.103	10
	SD			0.001	0.006	0.001	0.002	0.003	0	0.002	0.005	0.001	0.009						
Ginger Supplement C; Softgel with Oleoresin (% w/w)	A			0.7035	0.56	0.556	0.8753	0.72715	0.66	0.738	0.709	0.777	0.3374						
	B			0.659	0.58	0.567	0.8537	0.72191	0.71	0.675	0.724	0.728	0.354						
	C			0.6673	0.58	0.568	0.8145	0.73333	0.73	0.675	0.687	0.784	0.3784						
	Avg			0.677	0.573	0.564	0.848	0.727	0.700	0.696	0.707	0.763	0.357	0.675	0.122	18%	0.848	0.357	10
	SD			0.024	0.012	0.007	0.031	0.006	0.036	0.036	0.019	0.031	0.021						
Ginger Supplement D; Tincture (% w/w)	A			0.0316	0.02	0.025	0.0369	0.03278	0.03	0.0345	0.0316	0.032	0.0102						
	B			0.0305	0.02		0.0368	0.03168	0.03	0.037	0.0336	0.033	0.0097						
	C			0.0305	0.02		0.0372	0.03185	0.03	0.0345	0.0323	0.033	0.0107						
	Avg			0.031	0.02	0.025	0.037	0.032	0.03	0.035	0.033	0.033	0.010	0.030	0.006	20%	0.037	0.010	9
	SD			0.001	0		0.0002	0.001	0	0.001	0.001	0.001	0.001						
RM 8666 Ginger Extract (% w/w)	A		0.627	0.464	0.35	0.377	0.5693	0.42683	0.54	0.409	0.541	0.463	0.1125						
	B		0.603	0.4621	0.36	0.369	0.5712	0.4427	0.54	0.429	0.534	0.47	0.1159						
	C		0.617	0.4801	0.37	0.377	0.5852	0.3195	0.53	0.408	0.543	0.47	0.1393						
	Avg	0.518	0.616	0.469	0.360	0.374	0.575	0.396	0.537	0.415	0.539	0.468	0.123	0.481	0.138	29%	0.642	0.123	14
	SD	0.007	0.012	0.010	0.010	0.005	0.009	0.067	0.006	0.012	0.005	0.004	0.015						
SRM 3398 Ginger Rhizome (% w/w)	A		0.252	0.191	0.16	0.173	0.2386	0.21581	0.25	0.223	0.219	0.209	0.1054						
	B		0.255	0.1825	0.16	0.187	0.2409	0.22358	0.25	0.226	0.221	0.204	0.1097						
	C		0.262	0.1892	0.17	0.171	0.2442	0.22887	0.25	0.229	0.225	0.202	0.1089						
	Avg	0.252	0.256	0.188	0.163	0.177	0.241	0.223	0.25	0.226	0.222	0.205	0.108	0.220	0.044	20%	0.272	0.108	14
	SD	0.004	0.005	0.004	0.006	0.009	0.003	0.007	0	0.003	0.003	0.004	0.002						

Table 5-18. Data summary table for 8-shogaol in eight ginger containing materials. Data points highlighted in blue have been identified as outside the consensus tolerance limits and resulted in an unacceptable Z'_{comm} score, $|Z'_{\text{comm}}| \geq 2$. Data points highlighted in red have a zero or non-numeric data point.

		8-shogaol																	
		Individual Results											Community Results						
		Target	G003	G004	G019	G021	G023	G027	G029	G030	G037	G039	G046	Mean	SD	RSD	Max	Min	N
USP 1291446; Ginger Constituent Mixture (% w/w)	**A**			0.3539	0				< 0.010	<									
	B			0.4331	0				< 0.010	<									
	C			0.6173	0				< 0.010	<									
	Avg			0.468	0									0.234	0.829	354%	0.468	0.000	2
	SD			0.135	0														
USP 1291504; Powdered Ginger (% w/w)	**A**			0.0338	0.05	0.019	0.0355	0.03706	0.03	0.0245	0.0385	0.042							
	B			0.0331	0.06		0.0365	0.0368	0.04	0.029	0.0367	0.039	0.0221						
	C			0.0327	0.06		0.0371	0.03656	0.04	0.0356	0.0426	0.035	0.0157						
	Avg	0.025		0.033	0.057	0.019	0.036	0.037	0.037	0.030	0.039	0.039	0.019	0.034	0.008	24%	0.057	0.019	9
	SD	0.004		0.001	0.006		0.001	0.0003	0.006	0.006	0.003	0.004	0.005						
Ginger Supplement A; Tablet (% w/w)	**A**			0.0177	0.02	0.012	0.0227	0.02157	0.02	0.0237	0.0241	0.021							
	B			0.0177	0.03	0.012	0.0233	0.02193	0.02	0.0243	0.0251	0.022							
	C			0.0172	0.02	0.012	0.0223	0.02759	0.02	0.0239	0.0246	0.021							
	Avg			0.018	0.023	0.012	0.023	0.024	0.02	0.024	0.025	0.021		0.021	0.005	24%	0.025	0.012	9
	SD			0.0003	0.006	0	0.001	0.003	0	0.0003	0.001	0.001							
Ginger Supplement B; Capsule (% w/w)	**A**			0.0322	0.04	0.022	0.0422	0.04651	0.04	0.0422	0.0438	0.043	0.0168						
	B			0.0322	0.04	0.023	0.0423	0.04628	0.05	0.0419	0.0463	0.044	0.0125						
	C			0.0318	0.04	0.023	0.0429	0.0563	0.05	0.0423	0.045	0.044	0.0161						
	Avg			0.032	0.04	0.023	0.042	0.050	0.047	0.042	0.045	0.044	0.015	0.040	0.009	23%	0.050	0.015	10
	SD			0.0002	0	0.001	0.0004	0.006	0.006	0.0002	0.001	0.001	0.002						
Ginger Supplement C; Softgel with Oleoresin (% w/w)	**A**			0.2118	0.27	4.614	0.2159	0.21727	0.08	0.226	0.286	0.247	0.145						
	B			0.2079	0.26	4.566	0.2109	0.22036	0.13	0.224	0.288	0.242	0.1499						
	C			0.2062	0.25	4.554	0.2072	0.21252	0.13	0.222	0.263	0.25	0.1448						
	Avg			0.209	0.260	4.578	0.211	0.217	0.113	0.224	0.279	0.246	0.147	0.212	0.070	33%	4.578	0.113	10
	SD			0.003	0.010	0.032	0.004	0.004	0.029	0.002	0.014	0.004	0.003						
Ginger Supplement D; Tincture (% w/w)	**A**			0.0054	0.01	0.004	0.0072	0.0077	0.01	<	0.0067	0.008							
	B			0.0054	0.01		0.0071	0.00706	0.01	0.00479	0.0078	0.007							
	C			0.0054	0.01		0.0071	0.00705	0.01	0.003	0.0077	0.008							
	Avg			0.0054	0.01	0.0040	0.0071	0.0073	0.01	0.0039	0.0074	0.0077		0.007	0.003	43%	0.010	0.004	8
	SD			0.0000	0		0.0001	0.0004	0	0.0013	0.0006	0.0006							
RM 8666 Ginger Extract (% w/w)	**A**		0.127	0.0732	0.1	0.042	0.0938	0.08171	0.09	0.0616	0.102	0.086							
	B		0.127	0.0726	0.09	0.039	0.0951	0.08312	0.09	0.0645	0.107	0.086							
	C		0.118	0.082	0.1	0.043	0.0953	0.08257	0.08	0.0735	0.108	0.096							
	Avg	0.091	0.124	0.076	0.097	0.041	0.095	0.082	0.087	0.067	0.106	0.089		0.092	0.024	26%	0.134	0.041	12
	SD	0.003	0.005	0.005	0.006	0.002	0.001	0.001	0.006	0.006	0.003	0.006							
SRM 3398 Ginger Rhizome (% w/w)	**A**		0.0677	0.042	0.06	0.03	0.0583	0.6241	0.07	0.0574	0.0639	0.056	0.0325						
	B		0.0677	0.0396	0.06	0.032	0.0577	0.0601	0.06	0.0573	0.0639	0.055	0.0335						
	C		0.0699	0.0416	0.06	0.029	0.0571	0.05955	0.07	0.0581	0.0645	0.055	0.0327						
	Avg	0.068	0.068	0.041	0.06	0.030	0.058	0.248	0.067	0.058	0.064	0.055	0.033	0.054	0.014	26%	0.248	0.016	14
	SD	0.002	0.001	0.001	0	0.002	0.001	0.326	0.006	0.0004	0.0003	0.001	0.001						

Table 5-19. Data summary table for 10-shogaol in eight ginger containing materials. Data points highlighted in blue have been identified as outside the consensus tolerance limits and resulted in an unacceptable Z'_{comm} score, $|Z'_{\text{comm}}| \geq 2$. Data points highlighted in red have a zero or non-numeric data point.

		10-shogaol																		
		Individual Results												Community Results						
		Target	G003	G004	G019	G021	G023	G027	G029	G030	G037	G039	G046	Mean	SD	RSD	Max	Min	N
USP 1291446; Ginger Constituent Mixture (% w/w)	A			< 0.059	0				< 0.010	<									
	B			0.7237	0				< 0.010	<									
	C			< 0.059	0				< 0.010	<									
	Avg			0.724	0									0.362	0.803	222%	0.724	0.000	1
	SD				0														
USP 1291504; Powdered Ginger (% w/w)	A			0.0712	0.06	0.061	0.0557	0.05135	0.08	0.062	0.0597	0.085	0.0281						
	B			0.0711	0.07		0.0587	0.0509	0.08	0.065	0.0641	0.082	0.0463						
	C			0.0751	0.07		0.0593	0.04993	0.08	0.069	0.0677	0.088	0.0447						
	Avg	0.048		0.072	0.067	0.061	0.058	0.051	0.08	0.065	0.064	0.085	0.040	0.064	0.018	28%	0.085	0.040	9
	SD	0.004		0.002	0.006		0.002	0.001	0	0.004	0.004	0.003	0.010						
Ginger Supplement A; Tablet (% w/w)	A			0.035	0.03	0.029	0.0274	0.02899	0.04	0.0392		0.041	0.0085						
	B			0.0342	0.03	0.028	0.0324	0.03058	0.04	0.0405		0.041	0.0058						
	C			0.0339	0.04	0.028	0.0309	0.0297	0.04	0.0395		0.04							
	Avg			0.034	0.033	0.028	0.030	0.030	0.04	0.040		0.041	0.007	0.033	0.008	24%	0.041	0.007	9
	SD			0.001	0.006	0.001	0.003	0.001	0	0.001		0.001	0.002						
Ginger Supplement B; Capsule (% w/w)	A			0.0677	0.08	0.056	0.0641	0.05332	0.11	0.0692		0.089	0.0413						
	B			0.0677	0.08	0.062	0.064	0.05169	0.11	0.0683		0.091	0.0342						
	C			0.0671	0.09	0.061	0.0653	0.05031	0.11	0.0692		0.088	0.0409						
	Avg			0.068	0.083	0.060	0.064	0.052	0.11	0.069		0.089	0.039	0.070	0.026	37%	0.110	0.039	9
	SD			0.000	0.006	0.003	0.001	0.002	0	0.001		0.002	0.004						
Ginger Supplement C; Softgel with Oleoresin (% w/w)	A			0.2377	0.26	0.191	0.2136	0.13942	0.51	0.19		0.327	0.0766						
	B			0.2563	0.25	0.213	0.205	0.13937	0.52	0.191		0.33	0.0889						
	C			0.2336	0.27	0.194	0.1987	0.13627	0.52	0.189		0.341	0.1079						
	Avg			0.243	0.260	0.199	0.206	0.138	0.517	0.190		0.333	0.091	0.230	0.120	52%	0.517	0.091	9
	SD			0.012	0.010	0.012	0.007	0.002	0.006	0.001		0.007	0.016						
Ginger Supplement D; Tincture (% w/w)	A			0.0131	0.01	0.011	0.0114	0.00973	0.02	0.0117		0.02	0.0058						
	B			0.0135	0.01		0.0113	0.00925	0.02	0.0137		0.017	0.0048						
	C			0.0131	0.01		0.0112	0.00926	0.02	0.0119		0.017	0.0062						
	Avg			0.013	0.01	0.011	0.011	0.009	0.02	0.012		0.018	0.006	0.012	0.004	33%	0.020	0.006	8
	SD			0.000	0		0.000	0.000	0	0.001		0.002	0.001						
RM 8666 Ginger Extract (% w/w)	A		0.141	0.1579	0.16	0.13	0.1399	0.13828	0.17	0.168	0.0212	0.189	0.0396						
	B		0.137	0.1561	0.16	0.143	0.1391	0.14003	0.17	0.165	0.0211	0.192	0.0641						
	C		0.139	0.16	0.16	0.143	0.1413	0.13617	0.17	0.166	0.0206	0.209	0.0509						
	Avg	0.153	0.139	0.158	0.160	0.139	0.140	0.138	0.17	0.166	0.021	0.197	0.052	0.145	0.033	23%	0.197	0.021	14
	SD	0.004	0.002	0.002	0.000	0.008	0.001	0.002	0	0.002	0.000	0.011	0.012						
SRM 3398 Ginger Rhizome (% w/w)	A		0.104	0.1095	0.11	0.088	0.1017	0.09621	0.13	0.129	0.0238	0.135	0.0887						
	B		0.104	0.1018	0.12	0.094	0.0963	0.09524	0.13	0.126	0.024	0.135	0.0899						
	C		0.101	0.1067	0.12	0.089	0.096	0.09248	0.13	0.127	0.0226	0.135	0.0903						
	Avg	0.115	0.103	0.106	0.117	0.090	0.098	0.095	0.13	0.127	0.023	0.135	0.090	0.106	0.022	21%	0.135	0.011	14
	SD	0.002	0.002	0.004	0.006	0.003	0.003	0.002	0	0.002	0.001	0.000	0.001						

Table 5-20. Data summary table for 6-paradol in eight ginger containing materials. Data points highlighted in blue have been identified as outside the consensus tolerance limits and resulted in an unacceptable Z'_{comm} score, $|Z'_{\text{comm}}| \geq 2$. Data points highlighted in red have a zero or non-numeric data point.

		6-paradol																		
		Individual Results												Community Results						
		Target	G003	G004	G019	G021	G023	G027	G029	G030	G037	G039	G046	Mean	SD	RSD	Max	Min	N
USP 1291446; Ginger Constituent Mixture (% w/w)	**A**				0				< 0.010	<									
	B				0				< 0.010	<									
	C				0				< 0.010	<									
	Avg				0														1
	SD				0														
USP 1291504; Powdered Ginger (% w/w)	**A**				0.03		0.0308	0.00826	0.01	<	0.0204	0.016							
	B				0.04		0.037	0.00885	0.01	<	0.0218	0.012							
	C				0.03		0.037	0.00873	0.01	0.00662	0.0234	0.011							
	Avg	0.015			0.033		0.035	0.0086	0.01	0.007	0.022	0.013		0.018	0.011	61%	0.035	0.007	6
	SD	0.003			0.006		0.004	0.0003	0		0.002	0.003							
Ginger Supplement A; Tablet (% w/w)	**A**				0.02		0.0743	0.00727	0.01	<	0.0099	0.006							
	B				0.01		0.0641	0.00679	0.01	<	0.0091	0.006							
	C				0.01		0.0561	0.00715	0.01	<	0.0091	0.007							
	Avg				0.013		0.065	0.0071	0.01		0.0094	0.0063		0.009	0.005	56%	0.065	0.006	6
	SD				0.006		0.009	0.0002	0		0.0005	0.0006							
Ginger Supplement B; Capsule (% w/w)	**A**				0.02		0.0422	0.01146	0.02	0.0165	0.0331	0.016							
	B				0.03		0.0502	0.01025	0.02	0.0162	0.0343	0.016	0.007						
	C				0.03		0.0413	0.01064	0.02	0.0163	0.0339	0.016							
	Avg				0.027		0.045	0.011	0.02	0.016	0.034	0.016	0.007	0.021	0.013	62%	0.045	0.007	7
	SD				0.006		0.005	0.001	0	0.000	0.001	0.000							
Ginger Supplement C; Softgel with Oleoresin (% w/w)	**A**				0.21		2.6575	0.04825	0.06	0.16	0.152	0.351	0.1728						
	B				0.22		2.714	0.05245	0.06	0.157	0.153	0.353	0.157						
	C				0.23		2.6429	0.05303	0.06	0.13	0.152	0.342	0.1716						
	Avg				0.220		2.671	0.051	0.06	0.149	0.152	0.349	0.167	0.164	0.171	104%	2.671	0.051	8
	SD				0.010		0.038	0.003	0	0.017	0.001	0.006	0.009						
Ginger Supplement D; Tincture (% w/w)	**A**				0		< 0.006	0.00333	< 0.010	<	0.0044	0.003							
	B				0		0.0068	0.00283	< 0.010	<	0.0054	0.004							
	C				0		0.0066	0.00284	< 0.010	<	0.0047	0.002							
	Avg				0		0.0067	0.0030			0.0048	0.0030		0.004	0.004	100%	0.007	0.0	5
	SD				0		0.0001	0.0003			0.0005	0.0010							
RM 8666 Ginger Extract (% w/w)	**A**				0.11		0.1496	0.10056	0.08	0.0272	0.0954	0.067							
	B				0.15		0.1437	0.10094	0.08	0.0295	0.0937	0.071							
	C				0.16		0.1681	0.10834	0.07	0.025	0.0954	0.073							
	Avg				0.140		0.154	0.103	0.077	0.027	0.095	0.070		0.091	0.053	58%	0.154	0.027	8
	SD				0.026		0.013	0.004	0.006	0.002	0.001	0.003							
SRM 3398 Ginger Rhizome (% w/w)	**A**				0.02		0.0957	0.01793	0.01	<	0.0186	0.011							
	B				0.03		0.0922	0.01777	0.01	<	0.019	0.01							
	C				0.04		0.109	0.0161	0.01	<	0.0189	0.009							
	Avg				0.030		0.099	0.017	0.01		0.019	0.010		0.017	0.010	59%	0.099	0.010	7
	SD				0.010		0.009	0.001	0.0		0.0002	0.001							

Table 5-21. Data summary table for zingerone in eight ginger containing materials. Data points highlighted in blue have been identified as outside the consensus tolerance limits and resulted in an unacceptable Z'_{comm} score, $|Z'_{\text{comm}}| \geq 2$. Data points highlighted in red have a zero or non-numeric data point.

		zingerone																	
		Individual Results											Community Results						
		Target	G003	G004	G019	G021	G023	G027	G029	G030	G037	G039	G046	Mean	SD	RSD	Max	Min	N
USP 1291446; Ginger Constituent Mixture (% w/w)	A			1.3291	0				< 0.010	10.34									
	B			1.6677	0				< 0.010	4.42									
	C			3.5278	0				< 0.010	9.78									
	Avg			2.17	0					8.18				3.45	5.21	151%	8.18	0.00	3
	SD			1.18	0					3.27									
USP 1291504; Powdered Ginger (% w/w)	A			0.0475	0	< 0.003		0.0022	< 0.010	<		< 0.050							
	B			0.0459	0			0.00215	< 0.010	<		< 0.050							
	C			0.0428	0			0.0021	< 0.010	<		< 0.050							
	Avg			0.045	0			0.0022						0.001	0.005	500%	0.045	0.000	3
	SD			0.002	0			0.0001											
Ginger Supplement A; Tablet (% w/w)	A			0.0109	0.01	0.005		0.00416	< 0.010	<		0.008							
	B			0.0124	0.01	0.005		0.00475	< 0.010	<		0.008							
	C			0.0105	0.01	0.005		0.00418	< 0.010	<		0.007							
	Avg			0.0113	0.01	0.005		0.0044				0.0077		0.008	0.005	63%	0.011	0.004	5
	SD			0.0010	0	0		0.0003				0.0006							
Ginger Supplement B; Capsule (% w/w)	A			0.0134	0.01	< 0.003		0.00052	< 0.010	0.00727		0.013							
	B			0.0141	0.01	< 0.003		0.00052	< 0.010	0.00712		0.012							
	C			0.0121	0.01	< 0.003		0.00036	< 0.010	0.00727		0.011							
	Avg			0.013	0.01			0.00047		0.0072		0.012		0.009	0.006	67%	0.013	0.000	5
	SD			0.001	0			0.00009		0.0001		0.001							
Ginger Supplement C; Softgel with Oleoresin (% w/w)	A			0.0981	0.11	0.124	0.1265	0.01457	< 0.010	0.113		< 0.001							
	B			0.0728	0.11	0.126	0.1362	0.01804	< 0.010	0.119		< 0.001							
	C			0.0672	0.12	0.124	0.1478	0.01799	< 0.010	0.123		< 0.001							
	Avg			0.079	0.113	0.125	0.137	0.017		0.118				0.105	0.030	29%	0.137	0.017	6
	SD			0.016	0.006	0.001	0.011	0.002		0.005									
Ginger Supplement D; Tincture (% w/w)	A			0.0017	0	< 0.003		0.00104	< 0.010	<		< 0.001							
	B			0.0016	0			0.00109	< 0.010	<		< 0.001							
	C			0.0016	0			0.00168	< 0.010	<		< 0.001							
	Avg			0.0016	0			0.0013						0.0015	0.0003	18%	0.002	0.000	3
	SD			0.0001	0			0.0004											
RM 8666 Ginger Extract (% w/w)	A			0.0498	0.08	0.05	0.0282	0.01436	< 0.010	0.0207		0.073							
	B			0.0516	0.08	0.048	0.0283	0.01483	< 0.010	0.0239		0.073							
	C			0.0479	0.08	0.043	0.0283	0.01548	< 0.010	0.0252		0.077	0.0338						
	Avg			0.050	0.080	0.047	0.028	0.015		0.023		0.074	0.034	0.041	0.025	61%	0.080	0.015	8
	SD			0.002	0.000	0.004	0.000	0.001		0.002		0.002							
SRM 3398 Ginger Rhizome (% w/w)	A			0.01	0.01	< 0.003		0.01052	< 0.010	<		0.02							
	B			0.0094	0.01	< 0.003		0.00703	< 0.010	<		0.02							
	C			0.0113	0.02	< 0.003		0.00493	< 0.010	<		0.021							
	Avg			0.010	0.013			0.007				0.020		0.011	0.005	45%	0.020	0.007	5
	SD			0.001	0.006			0.003				0.001							

6. Protein Source Identification (Casein, Whey, Rice, Pea, and Soy)

6.1. Study Overview

The accurate measurement of protein and amino acid content is a necessity for analytical characterization and verification of foods and dietary supplements. However, commonly used methods may not distinguish between proteins, peptides, amino acids, and other non-protein, nitrogen containing compounds. The need for specific detection of certain proteins is further exemplified by increased food allergen concerns. Given these considerations, the use of accurate and reliable measurements that can distinguish between protein, amino acids, and adulterants, as well as differentiate between protein from different sources (e.g., soy versus milk), is a crucial component of manufacturing and QC/QA practices.

In this study, participants were provided with six samples of protein powder supplements. Participants were asked to use in-house analytical methods, and strongly encouraged to use AOAC First Action *Official Methods* 2017.11 and 2017.12, to identify the sources of protein (casein, whey, rice, pea, and soy) present in each sample. Participants were asked to report whether each protein type was Not Detected or Detected, and laboratories using the AOAC methods were asked to also report quantitative information (e.g., peak ratios for specific peptides). The data collected from this method will be used to evaluate method reproducibility and assist in the multilaboratory validation of AOAC 2017.11 and 2017.12. A copy of the method was provided to participants in the study.

6.2. Sample Information

Protein Powders A, B, C, D, E, and F. Participants were provided with one packet of each protein powder, each containing 10 g of material. Participants were asked to store the samples at controlled room temperature, 20 °C to 25 °C in the original unopened packets, to prepare three samples, and to report three results from each packet provided. Before use, participants were instructed to mix the contents of the packet thoroughly, allow contents to settle for one minute prior to opening to minimize the loss of fine particles, and to use a sample size appropriate for their usual in-house method of analysis. For participants following AOAC 2017.11 and/or AOAC 2017.12, participants asked to follow method instructions for recommended sample sizes. The identity of the protein sources present in the samples were not disclosed to participants prior to the study. The target protein sources listed in the table below were based on manufacturer label claims.

Protein Powder	Target Protein Sources				
	Casein	Pea	Rice	Soy	Whey
A	Present	-	-	Present	Present
B	-	Present	Present	-	-
C	-	Present	-	-	-
D	-	Present	Present	Present	-
E	-	-	Present	-	-
F	-	-	-	Present	-

6.3. Study Results

The percent of correct identification of the protein source is displayed in the table below, grouped by protein source (left) and by protein powder sample (right). Table cell color correlates with the percentage of participants that reported the correct answer using a gradient of green, yellow, orange, and red, where Green = 100 %, Yellow = 75 %, Orange = 25 %, and Red = 0 %.

Protein Source	Protein Powder	N	% Correct
Casein	A	4	50%
Casein	B	3	100%
Casein	C	3	100%
Casein	D	3	100%
Casein	E	3	100%
Casein	F	3	100%
Pea	A	4	100%
Pea	B	5	100%
Pea	C	4	75%
Pea	D	4	50%
Pea	E	4	25%
Pea	F	4	100%
Rice	A	4	75%
Rice	B	4	100%
Rice	C	5	40%
Rice	D	4	25%
Rice	E	4	75%
Rice	F	4	100%
Soy	A	4	0%
Soy	B	4	75%
Soy	C	4	75%
Soy	D	4	75%
Soy	E	5	60%
Soy	F	4	100%
Whey	A	3	33%
Whey	B	3	100%
Whey	C	3	100%
Whey	D	4	75%
Whey	E	3	100%
Whey	F	4	75%

Protein Source	Protein Powder	N	% Correct
Casein	A	4	50%
Pea	A	4	100%
Rice	A	4	75%
Soy	A	4	0%
Whey	A	3	33%
Casein	B	3	100%
Pea	B	5	100%
Rice	B	4	100%
Soy	B	4	75%
Whey	B	3	100%
Casein	C	3	100%
Pea	C	4	75%
Rice	C	5	40%
Soy	C	4	75%
Whey	C	3	100%
Casein	D	3	100%
Pea	D	4	50%
Rice	D	4	25%
Soy	D	4	75%
Whey	D	4	75%
Casein	E	3	100%
Pea	E	4	25%
Rice	E	4	75%
Soy	E	5	60%
Whey	E	3	100%
Casein	F	3	100%
Pea	F	4	100%
Rice	F	4	100%
Soy	F	4	100%
Whey	F	4	75%

Ten laboratories enrolled to identify protein sources in the samples. Between 3 and 5 laboratories reported qualitative results for each material and each protein source. Of the 5 laboratories that returned results, the reported method information is listed in the table below.

Lab Code	Sample Preparation	Analytical Method
G014	AOAC 2017.11	LC-MS/MS
G019	Other	Other
G028	Other	Other
G029	Solvent Extraction	HPTLC
G042	Enzymatic Hydrolysis	LC-MS/MS

Through additional method information reporting, one laboratory indicated use of AOAC 2017.11 as written, and one laboratory indicated use of AOAC 2017.11 with a small deviation, and both provided quantitative data. These results are not presented in this report but were provided to the AOAC method authors.

6.4. Protein Source Identification Technical Recommendations

The following recommendations and observations are based on results obtained from the participants in this study. Additional overall technical recommendations can be found on page 6.

- The data collected from this method was intended to help evaluate reproducibility of AOAC 2017.11 and AOAC 2017.12. Additional rounds of this study will be needed to gather enough quantitative data to evaluate reproducibility of the AOAC methods.
- The signup and participation of laboratories for the protein source identification study were low. Ten laboratories registered and received materials and five laboratories returned results. Therefore, the ability to make meaningful observations and recommendations is limited, but the following points are worth mentioning:
  - Some laboratories may have only reported a result when the protein source was detected.
  - Participants were most successful at correctly identifying the protein sources in Protein Powder B (contained pea and rice) and Protein Powder F (contained soy).
  - Certain laboratories had difficulty with specific protein source identification, indicating that the laboratory should focus on improving detection of those proteins.
  - Not enough data was returned to determine if certain proteins are more difficult to identify when in the presence of other protein sources, or if other matrix components cause challenges for the determination of protein sources.

Table 6-1. Individual data table (NIST) for protein source identification. The results are qualitative; Y indicates the protein was detected and N indicates the protein was not detected.

Exercise 7 - Protein Source Identification

		Lab Code: NIST	Your Result	2. Community Results			3. Target
Analyte	Sample	Units	Detected	N	# Correct Responses	% Correct Responses	Present
Casein Protein	Protein Sample A	Detected	Y	4	2	50%	Y
Casein Protein	Protein Sample B	Detected	N	3	3	100%	N
Casein Protein	Protein Sample C	Detected	N	3	3	100%	N
Casein Protein	Protein Sample D	Detected	N	3	3	100%	N
Casein Protein	Protein Sample E	Detected	N	3	3	100%	N
Casein Protein	Protein Sample F	Detected	N	3	3	100%	N
Pea Protein	Protein Sample A	Detected	N	4	4	100%	N
Pea Protein	Protein Sample B	Detected	Y	5	5	100%	Y
Pea Protein	Protein Sample C	Detected	Y	4	3	75%	Y
Pea Protein	Protein Sample D	Detected	Y	4	2	50%	Y
Pea Protein	Protein Sample E	Detected	N	4	1	25%	N
Pea Protein	Protein Sample F	Detected	N	4	4	100%	N
Rice Protein	Protein Sample A	Detected	N	4	3	75%	N
Rice Protein	Protein Sample B	Detected	Y	4	4	100%	Y
Rice Protein	Protein Sample C	Detected	N	5	2	40%	N
Rice Protein	Protein Sample D	Detected	Y	4	1	25%	Y
Rice Protein	Protein Sample E	Detected	Y	4	3	75%	Y
Rice Protein	Protein Sample F	Detected	N	4	4	100%	N
Soy Protein	Protein Sample A	Detected	Y	4	0	0%	Y
Soy Protein	Protein Sample B	Detected	N	4	3	75%	N
Soy Protein	Protein Sample C	Detected	N	4	3	75%	N
Soy Protein	Protein Sample D	Detected	Y	4	3	75%	Y
Soy Protein	Protein Sample E	Detected	N	5	3	60%	N
Soy Protein	Protein Sample F	Detected	Y	4	4	100%	Y
Whey Protein	Protein Sample A	Detected	Y	3	1	33%	Y
Whey Protein	Protein Sample B	Detected	N	3	3	100%	N
Whey Protein	Protein Sample C	Detected	N	3	3	100%	N
Whey Protein	Protein Sample D	Detected	N	4	3	75%	N
Whey Protein	Protein Sample E	Detected	N	3	3	100%	N
Whey Protein	Protein Sample F	Detected	N	4	3	75%	N
			Y = Detected N = Not Detected	N = Number of labs that returned results			Y = Detected N = Not Detected

Table 6-2. Data summary table for casein protein identification in protein powder samples. The results are qualitative; 0 indicates the protein was not detected and 1 indicates the protein was detected. Data points highlighted in blue are considered incorrect based on the target result from manufacturer label claims.

		Casein													
		Individual Results (0 = Not Detected, 1 = Detected)											Community Results		
	Lab	Target	G014	G019	G021	G028	G029	G036	G038	G040	G042	G045	# of Correct Reponses	% Correct Reponses	N
Protein Sample A	A		0	1		1					0				
	B		0	1		1					0				
	C		0	1		1					0				
	Avg	1	0	1		1					0		2	50%	4
	SD														
Protein Sample B	A		0	0							0				
	B		0	0							0				
	C		0	0							0				
	Avg	0	0	0							0		3	100%	3
	SD														
Protein Sample C	A		0	0							0				
	B		0	0							0				
	C		0	0							0				
	Avg	0	0	0							0		3	100%	3
	SD														
Protein Sample D	A		0	0							0				
	B		0	0							0				
	C		0	0							0				
	Avg	0	0	0							0		3	100%	3
	SD														
Protein Sample E	A		0	0							0				
	B		0	0							0				
	C		0	0							0				
	Avg	0	0	0							0		3	100%	3
	SD														
Protein Sample F	A		0	0							0				
	B		0	0							0				
	C		0	0							0				
	Avg	0	0	0							0		3	100%	3
	SD														

Table 6-3. Data summary table for pea protein identification in protein powder samples. The results are qualitative; 0 indicates the protein was not detected and 1 indicates the protein was detected. Data points highlighted in blue are considered incorrect based on the target result from manufacturer label claims.

		Pea													
		Individual Results (0 = Not Detected, 1 = Detected)											Community Results		
	Lab	Target	G014	G019	G021	G028	G029	G036	G038	G040	G042	G045	# of Correct Reponses	% Correct Reponses	N
Protein Sample A	A		0	0			0				0				
	B		0								0				
	C		0												
	Avg	0	0	0			0				0		4	100%	4
	SD										0				
Protein Sample B	A		1	1		1	1				1				
	B		1			1					1				
	C		1			1									
	Avg	1	1	1		1	1				1		5	100%	5
	SD														
Protein Sample C	A		1	1			1				0				
	B		1								0				
	C		1								0				
	Avg	1	1	1			1				0		3	75%	4
	SD														
Protein Sample D	A		0	1			1				0				
	B		0								0				
	C		0								0				
	Avg	1	0	1			1				0		2	50%	4
	SD														
Protein Sample E	A		0	1			1				1				
	B		0								1				
	C		0								1				
	Avg	0	0	1			1				1		1	25%	4
	SD														
Protein Sample F	A		0	0			0				0				
	B		0								0				
	C		0												
	Avg	0	0	0			0				0		4	100%	4
	SD														

Table 6-4. Data summary table for rice protein identification in protein powder samples. The results are qualitative; 0 indicates the protein was not detected and 1 indicates the protein was detected. Data points highlighted in blue are considered incorrect based on the target result from manufacturer label claims.

		Rice													
		Individual Results (0 = Not Detected, 1 = Detected)											Community Results		
	Lab	Target	G014	G019	G021	G028	G029	G036	G038	G040	G042	G045	# of Correct Reponses	% Correct Reponses	N
Protein Sample A	A		0	0			1				0				
	B		0								0				
	C		0								0				
	Avg	0	0	0			1				0		3	75%	4
	SD														
Protein Sample B	A		1	1			1				1				
	B		1								1				
	C		1								1				
	Avg	1	1	1			1				1		4	100%	4
	SD														
Protein Sample C	A		0	1		1	0				1				
	B		0			1					1				
	C		0			1					1				
	Avg	0	0	1		1	0				1		2	40%	5
	SD														
Protein Sample D	A		0	0			1				0				
	B		0	0							0				
	C		0	0							0				
	Avg	1	0	0			1				0		1	25%	4
	SD														
Protein Sample E	A		1	1			1				0				
	B		1								0				
	C		1								0				
	Avg	1	1	1			1				0		3	75%	4
	SD														
Protein Sample F	A		0	0			0				0				
	B		0								0				
	C		0								0				
	Avg	0	0	0			0				0		4	100%	4
	SD														

Table 6-5. Data summary table for soy protein identification in protein powder samples. The results are qualitative; 0 indicates the protein was not detected and 1 indicates the protein was detected. Data points highlighted in blue are considered incorrect based on the target result from manufacturer label claims.

		Soy													
		Individual Results (0 = Not Detected, 1 = Detected)											Community Results		
	Lab	Target	G014	G019	G021	G028	G029	G036	G038	G040	G042	G045	# of Correct Reponses	% Correct Reponses	N
Protein Sample A	A		0	0			0				0				
	B		0								0				
	C		0								0				
	Avg	1	0	0			0				0		0	0%	4
	SD														
Protein Sample B	A		0	1			0				0				
	B		0								0				
	C		0								0				
	Avg	0	0	1			0				0		3	75%	4
	SD														
Protein Sample C	A		0	1			0				0				
	B		0	1							0				
	C		0	1							0				
	Avg	0	0	1			0				0		3	75%	4
	SD														
Protein Sample D	A		1	1			0				1				
	B		1	1							1				
	C		1	1							1				
	Avg	1	1	1			0				1		3	75%	4
	SD														
Protein Sample E	A		0	1		1	0				0				
	B		0	1		1					0				
	C		0	1		1					0				
	Avg	0	0	1		1	0				0		3	60%	5
	SD														
Protein Sample F	A		1	1			1				1				
	B		1	1							1				
	C		1	1							1				
	Avg	1	1	1			1				1		4	100%	4
	SD														

Table 6-6. Data summary table for whey protein identification in protein powder samples. The results are qualitative; 0 indicates the protein was not detected and 1 indicates the protein was detected. Data points highlighted in blue are considered incorrect based on the target result from manufacturer label claims.

		Whey													
		Individual Results (0 = Not Detected, 1 = Detected)											Community Results		
	Lab	Target	G014	G019	G021	G028	G029	G036	G038	G040	G042	G045	# of Correct Reponses	% Correct Reponses	N
Protein Sample A	A		0	1							0				
	B		0	1							0				
	C		0	1							0				
	Avg	1	0	1							0		1	33%	3
	SD														
Protein Sample B	A		0	0							0				
	B		0	0							0				
	C		0	0							0				
	Avg	0	0	0							0		3	100%	3
	SD														
Protein Sample C	A		0	0							0				
	B		0	0							0				
	C		0	0							0				
	Avg	0	0	0							0		3	100%	3
	SD														
Protein Sample D	A		0	0		1					0				
	B		0	0		1					0				
	C		0	0		1					0				
	Avg	0	0	0		1					0		3	75%	4
	SD														
Protein Sample E	A		0	0							0				
	B		0	0							0				
	C		0	0							0				
	Avg	0	0	0							0		3	100%	3
	SD		0	0							0				
Protein Sample F	A		0	0		1					0				
	B		0	0		1					0				
	C		0	0		1					0				
	Avg	0	0	0		1					0		3	75%	4
	SD														

7. Human Metabolism Studies

7.1. Study Overview

A goal of HAMQAP is to provide samples representing total human health, with dietary intake samples linked with human metabolism samples. Exercise 7 offered participants the opportunity to assess their in-house measurements of nutritional elements (calcium, magnesium, and zinc), toxic elements (arsenic, cadmium, lead, and mercury), water-soluble vitamins (vitamins B_2 and B_6 and homocysteine), and fat-soluble vitamins (vitamin K) in blood and serum samples. Accurate and reliable determinations of clinically relevant analytes are essential for the association of status to health outcomes and for medical recommendations. Due to the participation rates for the individual human metabolite studies, all descriptions, observations, and recommendations will be jointly summarized in this section.

Informed in part by low participation rates and stakeholder engagement activities, NIST has concluded that the HAMQAP program has not fully met the needs of the clinical measurement community. Future programs will aim to hold more workshops and presentations to increase stakeholder awareness of NIST QAPs and engagement for the planning and administration of upcoming exercises. The design of NIST QAPs will also shift to more matrix targeted exercises with the revitalization of the Clinical Measurements Quality Assurance Program (ClinQAP).

Study	Analytes	Samples
Nutritional Elements	Ca, Mg, Zn	Human Serum A Animal Serum B
Toxic Elements	As, Cd, Pb, Hg	Human Blood A Animal Serum B
Water-Soluble Vitamins	vitamin B_2 (riboflavin) flavin mononucleotide (FMN) flavin adenine dinucleotide (FAD) pyridoxal 5′-phosphate (PLP) pyridoxal (PL) 4-pyridoxic acid (PA) homocysteine	Human Serum C Human Serum D
Fat-Soluble Vitamins	total vitamin K_1 (phylloquinone) *cis*-vitamin K_1 *trans*-vitamin K_1 total vitamin K_2 vitamin K_2 MK-4 vitamin K_2 MK-7 vitamin K_2 MK-9	Human Serum E Human Serum F

7.2. Sample Information

Human intake samples were intended for research use only and not for human consumption. Human output samples were human-source and/or animal-source biohazardous materials capable of transmitting infectious disease. Participants were advised to handle these materials at the Biosafety Level 2 or higher as recommended for any potentially infectious human source materials by the Centers for Disease Control and Prevention (CDC) Office of Safety, Health, and Environment and the National Institutes of Health (NIH). The supplier of the source materials for the blood, serum, and/or plasma used to prepare the sample materials found the materials to be non-reactive when tested for hepatitis B surface antigen (HBsAg), human immunodeficiency virus (HIV), hepatitis C virus (HCV), and human immunodeficiency virus 1 antigen (HIV-1Ag) by FDA licensed tests.

Human Blood A. Participants were provided with three vials of SRM 955d Toxic Elements and Metabolites in Frozen Human Blood (Level 1) for the determination of arsenic (As), cadmium (Cd), lead (Pb), and mercury (Hg) mass fractions. Each vial contained approximately 1.6 mL of material. Participants were asked to store the material at ultracold freezer (–70 °C or colder) conditions in the original unopened vials, to prepare one sample, and to report one value for each measurand from each vial provided. Before use, participants were instructed to allow the material to thaw at room temperature for at least 30 min, and then to gently mix each vial prior to removal of a test portion for analysis. Participants were asked to use a sample size appropriate for their normal in-house method of analysis and report the toxic element mass fractions in µg/L. Approximate analyte levels were not disclosed to participants prior to the study. The target values for As, Cd, Pb, and Hg in SRM 955d were determined at NIST and the values and uncertainties from the COA at the time of this report are provided in the table below.

Analyte	Target Mass Fractions in SRM 955d Level 1 (µg/L)		
Arsenic (As)	5.31	±	0.76
Cadmium (Cd)	0.33	±	0.01
Lead (Pb)	14.8	±	0.26
Mercury (Hg)	1.37	±	0.081

Human Serum A. Participants were provided with three vials of SRM 909c Frozen Human Serum for the determination of calcium (Ca), magnesium (Mg), and zinc (Zn) mass fractions. Each vial contained approximately 2 mL of material. Participants were asked to store the material at ultracold freezer (-70 °C or colder) conditions in the original unopened vials, to prepare one sample, and to report one value for each measurand from each vial provided. Before use, participants were instructed to allow the material to thaw at room temperature for at least 30 min, and then to gently mix each vial prior to removal of a test portion for analysis. Participants were asked to use a sample size appropriate for their normal in-house method of analysis and report the nutritional element mass fractions in mg/dL. Approximate analyte levels were not disclosed to participants prior to the study. The target values for Ca and Mg in SRM 909c were determined at NIST the values and uncertainties from the COA at the time of this report are provided in the table below. A target value for Zn in SRM 909c was not available at the time of this report.

Analyte	Target Mass Fractions in SRM 909c (mg/dL)		
Calcium (Ca)	10.10	±	0.11
Magnesium (Mg)	2.176	±	0.016

Animal Serum B. Participants were provided with one vial of SRM 1598a Inorganic Constituents in Animal Serum for the determination of calcium (Ca), magnesium (Mg), and zinc (Zn) and one

vial for the determination of arsenic (As), cadmium (Cd), lead (Pb), and mercury (Hg) mass fractions, depending on the participants' signup for Nutritional Elements, Toxic Elements, or both. Each vial contained approximately 5 mL of material. Participants were asked to store the material at ultracold freezer (–70 °C or colder) conditions in the original unopened vials, to prepare one sample, and to report one value for each measurand from each vial provided. Before use, participants were instructed to allow the material to thaw at room temperature for at least 30 min, and then to gently mix each vial prior to removal of a test portion for analysis. Participants were asked to use a sample size appropriate for their normal in-house methods of analysis and report the nutritional element mass fractions in mg/dL and the toxic element mass fractions in µg/L. Approximate analyte levels were not disclosed to participants prior to the study. The target values for As, Cd, Hg, Ca, and Mg in SRM 1598a were determined at NIST and the values and uncertainties from the COA at the time of this report are provided in the table below. Target values for Pb and Mg in SRM 1598a were not available at the time of this report.

Analyte	Target Mass Fractions in SRM 1598a
	(µg/L)
Arsenic (As)	0.3
Cadmium (Cd)	0.048 ± 0.004
Mercury (Hg)	0.32 ± 0.19
	(mg/dL)
Calcium (Ca)	9.6 ± 0.7
Zinc (Zn)	0.088 ± 0.0024

Human Serum C and D. Participants were provided with three vials each of SRM 3950 Vitamin B_6 in Frozen Human Serum (Level 1 and Level 2) for the determination of vitamin B_2 (as riboflavin, flavin mononucleotide (FMN) and flavin adenine dinucleotide (FAD)), vitamin B_6 (as pyridoxal 5′-phosphate (PLP), pyridoxal (PL), and 4-pyridoxic acid (PA)) and homocysteine mass fractions. Each vial contained approximately 1 mL of material. Participants were asked to store the material at ultracold freezer (–70 °C or colder) conditions in the original unopened vials, and to prepare one sample and report one value for each measurand from each vial provided. Before use, participants were instructed to allow the material to thaw at room temperature for at least 30 min, taking precautions to avoid exposure to direct UV light, and then to gently mix each vial prior to removal of a test portion for analysis. Participants were asked to use a sample size appropriate for their normal in-house method of analysis and report the analyte mass fractions in ng/mL. Approximate analyte levels were not disclosed to participants prior to the study. The target values for pyridoxal 5′-phosphate (PLP) and 4-pyridoxic acid (PA) in SRM 3950 were determined at NIST. The values and uncertainties from the COA at the time of this report are provided in the table below. Target values for riboflavin, flavin mononucleotide (FMN) and flavin adenine dinucleotide (FAD), pyridoxal (PL), and homocysteine in SRM 3950 were not available at the time of this report.

	Target Mass Concentrations in SRM 3950 (ng/mL)	
Analyte	Level 1	Level 2
pyridoxal 5′-phosphate (PLP)	4.59 ± 0.16	9.0 ± 0.29
4-pyridoxic acid (PA)	22.2	37.1

Human Serum E and F. Participants were provided with three vials each of SRM 968f Fat-Soluble Vitamins in Frozen Human Serum (Level 1 and Level 2) for the determination of vitamin K_1 (as total phylloquinone, *cis*-vitamin K_1, *trans*-vitamin K_1) and vitamin K_2 (as total vitamin K_2,

MK-4, MK-7, MK-9) mass fractions. Each vial contained approximately 1 mL of material. Participants were asked to store the material at ultracold freezer (–70 °C or colder) conditions in the original unopened vials, to prepare one sample, and to report one value for each measurand from each vial provided. Before use, participants were instructed to allow the material to thaw at room temperature for at least 30 min, taking precautions to avoid exposure to direct UV light, and then to gently mix each vial prior to removal of a test portion for analysis. Participants were asked to use a sample size appropriate for their normal in-house method of analysis and report the fat-soluble vitamin mass fractions in ng/mL. Approximate analyte levels were not disclosed to participants prior to the study. The target value for total vitamin K_1 (phylloquinone) in SRM 968f was determined by results of previous QAPs. The value and standard deviation is provided in the table below. Target values for the additional vitamin K_1 and vitamin K_2 analytes in SRM 968f were not available at the time of this report.

	Target Mass Fractions in SRM 968f (ng/mL)	
Analyte	Level 1	Level 2
Total Vitamin K_1 (phylloquinone)	0.227 ± 0.047	0.69 ± 0.14

7.3. Human Metabolites Study Results

Nine laboratories enrolled and received samples to measure analytes in the combined human metabolism studies. In past HAMQAP exercises, when study enrollment was below 10, the study was cancelled. For Exercise 7, the requesting participants were notified of the low enrollment and asked if they would still like to participate. Some labs agreed to still receive samples and return results. The enrollment and reporting statistics for each of the studies are described in the tables below.

Study	Number of Laboratories Requesting Samples	Number of Laboratories Reporting Results Range for Individual Analytes
Nutritional Elements	1	0 to 1
Toxic Elements	2	0 to 1
Water-Soluble Vitamins	4	0 to 3
Fat-Soluble Vitamins	2	0

- The enrollment and participation in the human metabolism studies were too low to make meaningful observations and recommendations.
- One laboratory returned results for nutritional and toxic elements, with several resulting in acceptable Z_{NIST} scores.
- Three laboratories returned results for water-soluble vitamins, and all labs did well for the measurement of pyridoxal 5′-phosphate (PLP) and 4-pyridoxic acid (PA). One lab returned results outside the target ranges, but it is very likely there were unit errors when reporting. Two labs also returned results for homocysteine and were in agreement with each other.
- There were no results returned for the fat-soluble vitamin study.

Table 7-1. Individual data table (NIST) for calcium, magnesium, and zinc in human and animal serums.

HAMQAP Exercise 7 - Nutritional Elements

	Lab Code:	**NIST**	**1. Your Results**				**2. Community Results**			**3. Target**	
Analyte	Sample	Units	x_i	s_i	Z'_{comm}	Z_{NIST}	N	x*	s*	x_{NIST}	*U*
Calcium	SRM 909c Frozen Human Serum	mg/dL	10.01	0.11			0			10.01	0.11
Calcium	SRM 1598a Inorganic Constituents in Animal Serum	ug/L	96000	7000			0			96000	7000
Magnesium	SRM 909c Frozen Human Serum	mg/dL	2.176	0.015			0			2.176	0.015
Magnesium	SRM 1598a Inorganic Constituents in Animal Serum	ug/L					0				
Zinc	SRM 909c Frozen Human Serum	mg/dL					1	0.06			
Zinc	SRM 1598a Inorganic Constituents in Animal Serum	ug/L	880	24			1	660		880	24

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

Table 7-2. Individual data table (NIST) for arsenic, cadmium, mercury, and lead in human blood and human serum.

	Lab Code:	**NIST**	**1. Your Results**				**2. Community Results**			**3. Target**	
Analyte	Sample	Units	x_i	s_i	Z'_{comm}	Z_{NIST}	N	x*	s*	x_{NIST}	*U*
Arsenic	SRM 955d Toxic Elements and Metabolites in Frozen Human Blood (L1)	ug/L	5.31	0.76			1	5.11		5.31	0.76
Arsenic	SRM 1598a Inorganic Constituents in Animal Serum	ug/L	0.3				1	0.29		0.3	
Cadmium	SRM 955d Toxic Elements and Metabolites in Frozen Human Blood (L1)	ug/L	0.326	0.01			1	0.3		0.326	0.01
Cadmium	SRM 1598a Inorganic Constituents in Animal Serum	ug/L	0.048	0.004			0			0.048	0.004
Mercury	SRM 955d Toxic Elements and Metabolites in Frozen Human Blood (L1)	ug/L	1.37	0.081			1	1.5		1.37	0.081
Mercury	SRM 1598a Inorganic Constituents in Animal Serum	ug/L	0.32	0.19			0			0.32	0.19
Lead	SRM 955d Toxic Elements and Metabolites in Frozen Human Blood (L1)	ug/L	14.8	0.26			1	12.15		14.8	0.26
Lead	SRM 1598a Inorganic Constituents in Animal Serum	ug/L					0				

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

Table 7-3. Individual data table (NIST) for vitamins B_2, B_6, and homocysteine in human serums.

HAMQAP Exercise 7 - Water-Soluble Vitamins

		Lab Code:	**NIST**	**1. Your Results**				**2. Community Results**			**3. Target**	
Analyte	Sample		Units	x_i	s_i	Z'_{comm}	Z_{NIST}	N	x*	s*	x_{NIST}	*U*
Ribofavin (Vitamin B2)	SRM 3950 Vitamin B6 in Frozen Human Serum (L1)		ng/mL					2	5891	15041		
Ribofavin (Vitamin B2)	SRM 3950 Vitamin B6 in Frozen Human Serum (L2)		ng/mL					2	13172	32811		
FAD	SRM 3950 Vitamin B6 in Frozen Human Serum (L1)		ng/mL					0				
FAD	SRM 3950 Vitamin B6 in Frozen Human Serum (L2)		ng/mL					0				
flavin mononucleotide (FMN)	SRM 3950 Vitamin B6 in Frozen Human Serum (L1)		ng/mL					0				
flavin mononucleotide (FMN)	SRM 3950 Vitamin B6 in Frozen Human Serum (L2)		ng/mL					0				
4-pyridoxic acid (PA)	SRM 3950 Vitamin B6 in Frozen Human Serum (L1)		ng/mL	22.2				1			22.2	
4-pyridoxic acid (PA)	SRM 3950 Vitamin B6 in Frozen Human Serum (L2)		ng/mL	37.1				2			37.1	
Pyridoxal (PL)	SRM 3950 Vitamin B6 in Frozen Human Serum (L1)		ng/mL					1				
Pyridoxal (PL)	SRM 3950 Vitamin B6 in Frozen Human Serum (L2)		ng/mL					2				
pyridoxal 5'-phosphate (PLP)	SRM 3950 Vitamin B6 in Frozen Human Serum (L1)		ng/mL	4.59	0.16			3	8.90	30.01	4.59	0.16
pyridoxal 5'-phosphate (PLP)	SRM 3950 Vitamin B6 in Frozen Human Serum (L2)		ng/mL	9.00	0.29			6	20.52	58.25	9.00	0.29
Homocysteine	SRM 3950 Vitamin B6 in Frozen Human Serum (L1)		ng/mL					2	630	2759		
Homocysteine	SRM 3950 Vitamin B6 in Frozen Human Serum (L2)		ng/mL					2	659	2166		

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

Table 7-4. Data summary table for 4-pyridoxic acid (PA) in human serums. One laboratory returned data, and reported using protein precipitation and LC-FLD.

		4-pyridoxic acid (PA)									
		SRM 3950 Vitamin B6 in Frozen Human Serum (L1) (ng/mL)					SRM 3950 Vitamin B6 in Frozen Human Serum (L2) (ng/mL)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				22.20					37.10	
	G051	22908	22664	22851	22808	128	37811	37587	37811	37736	129
	G052										
	G053										
Community Results		Consensus Mean					Consensus Mean				
		Consensus Standard Deviation					Consensus Standard Deviation				
		Maximum			22808		Maximum			37736	
		Minimum			22808		Minimum			37736	
		N			1		N			1	

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