NISTIR 8249

Health Assessment Measurements Quality Assurance Program: Exercise 2 Final Report

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National Institute of Standards and Technology
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ABSTRACT

The NIST Health Assessment Measurements Quality Assurance Program (HAMQAP) was launched in collaboration with the National Institutes of Health (NIH) Office of Dietary Supplements (ODS) in 2017. HAMQAP was established to enable laboratories to improve the accuracy of measurements for demonstration of compliance with various regulations by measuring samples that represent human intake (e.g., foods, dietary supplements, tobacco) and samples that represent human output (e.g., blood, serum, plasma, urine) for demonstration of proficiency and/or compliance with various regulations. Analytes are paired where possible to represent the full spectrum of health assessment. Exercise 2 of this program offered the opportunity for laboratories to assess their in-house measurements of nutritional elements (iodine), contaminants (arsenic, cadmium, lead, mercury, and selenium), water-soluble vitamins (folates), fat-soluble vitamins (vitamins A and E), fatty acids, and natural product compounds (eleutherosides and co-enzyme Q10) in foods and dietary supplements, and biomarkers/metabolites in clinical specimens (including human serum, blood, and urine).

INTRODUCTION

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

The HAMQAP offers the opportunity for laboratories to assess their in-house measurements of nutritional and toxic elements, fat- and water-soluble vitamins, fatty acids, active and/or marker compounds, and contaminants in samples distributed by NIST. Samples that represent human intake (e.g., food, dietary supplements, tobacco) are paired with samples that represent human output (e.g., blood, serum, plasma, urine)[1], where possible, to represent the full spectrum of intake and metabolism for health assessment. Reports and certificates of participation are provided and may be used to demonstrate compliance with the current good manufacturing practices (cGMPs) or to fulfill requirements established by related accreditation bodies. In addition, NIST and the HAMQAP assist the ODS Analytical Methods and Reference Materials program (AMRM) at the NIH in supporting the development and dissemination of analytical tools and reference materials. In the future, results from HAMQAP exercises could be used by ODS and NIST to identify problematic matrices and analytes for which consensus-based methods of analysis would benefit the dietary supplements and clinical communities.

[1] Human intake samples were intended for research use only and not for human consumption. Human output samples were human-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 Food and Drug Administration (FDA) licensed tests.

NIST has decades of experience in the administration of QAPs, and the HAMQAP builds on the approach taken by the former DSQAP by providing a wide range of matrices and analytes. The HAMQAP design emphasizes emerging and challenging measurements in the dietary supplement, food, and clinical matrix categories. Participating laboratories are interested in evaluating in-house methods on a wide variety of challenging, real-world matrices 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, the 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.

This report summarizes the results from the second exercise of the HAMQAP. Forty-five laboratories responded to the dietary intake portion and twenty-six laboratories responded to the human metabolites portion of the call for participants distributed in May 2018 (see table below). One dietary intake study and three human metabolites studies were cancelled prior to shipment due to low enrollment. Samples were shipped to participants in September 2018 and results were returned to NIST by October 2018. This report contains the final data and information that was disseminated to the participants in May 2019.

Study Group	Dietary Intake Study	Human Metabolites Study
Nutritional Elements	Iodine Multivitamin, Nutritional Formula	~~Iodine, TSH, T3, T4~~* ~~Human Milk, Urine~~
Toxic Elements	As, Cd, Pb, Hg, Se Kudzu Extract, Eleuthero Extract	~~As, Cd, Pb, Hg, Se~~* ~~Whole Human Blood~~
Water-Soluble Vitamins	Folates Meat Homogenate, Egg Powder	~~Folates~~* ~~Human Serum~~
Fat-Soluble Vitamins	Retinol, Tocopherols Multivitamin, Nutritional Formula	Retinol, Tocopherols Human Serum
Fatty Acids	Fatty Acids Egg Powder, Spirulina, Palm Oil Powder	Fatty Acids Human Milk, Lyophilized Human Serum
Natural Products	Ubiquinone Commercial Supplements	~~Ubiquinone, Ubiquinol~~* ~~Human Serum~~
Botanicals	Eleutherosides Eleuthero Root, Eleuthero Extract	Not offered
Contaminants	~~Acrylamide~~* ~~Coffee, Peanut Butter~~	Not offered

* Cancelled due to low enrollment.

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

Individualized data tables and certificates are provided to the participants that have submitted data in each study, in addition to this report. Examples of the data tables using NIST data 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 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).[2] The consensus means and standard deviations are calculated according to the robust Q/Hampel method outlined in ISO 13528:2015(E), Annex C.[3]

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, reference, or estimated values, when available). The upper left of the data table includes the randomized laboratory code. Example individualized data tables are included in this report; participating laboratories received uniquely coded individualized data tables in a separate distribution.

Section 1 of the data table (*Your Results*) contains the laboratory results as reported, including the mean and standard deviation when multiple values were reported. A blank indicates that NIST does not have data on file for that laboratory for the corresponding analyte or matrix. An empty box for standard deviation indicates that the participant reported a single value or a value below the limit of quantification (LOQ) and therefore that value was not included in the calculation of the consensus data.[3] Example individualized data tables are included in this report using NIST data in Section 1 to protect the identity and performance of participants.

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

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

[2] 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.

[3] ISO 13528:2015(E), *Statistical methods for use in proficiency testing by interlaboratory comparisons*, pp. 53–54.

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

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

or

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

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

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

Section 2 of the data table (*Community Results*) contains the consensus results, including the number of laboratories reporting more than a single quantitative value for each analyte, the mean value determined for each analyte, and a robust estimate of the standard deviation of the reported values.[3] Consensus means and standard deviations are calculated using the laboratory means; if a laboratory reported a single value, the reported value is not included in determination of the consensus values.[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 possible, the target value is a certified value, a reference value, or a value determined at NIST. Certified values and the associated expanded uncertainty (U_{95}) have been determined with two independent analytical methods at NIST, one Joint Committee for Traceability in Laboratory Medicine (JCTLM)-recognized Reference Measurement Procedure (RMP) at NIST, or by combination of a single method at NIST and results from collaborating laboratories. Reference values are assigned using NIST values obtained from the average and standard deviation of measurements made using a single analytical method at NIST or by measurements obtained from collaborating laboratories. For both certified and reference values, at least six samples have been tested and duplicate preparations from the sample package have been included, allowing the uncertainty to encompass variability due to inhomogeneity within and between packages. For samples in which a NIST certified or reference value is not available, the analytes may be measured at NIST using a validated method or data from a partner laboratory may be used to establish a NIST-assessed value. The NIST-assessed value represents the mean of at least three replicates. For materials acquired from another interlaboratory study or proficiency testing program, the consensus value and uncertainty from the completed round is used as the target range. Within each section of this report, the exact methods for determination of the study target values are outlined in detail.

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 points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package. The standard deviation (SD) for the target value in this table is the uncertainty (U_{NIST}) around the target value.

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 method quantitation limit (QL) 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 black 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. Where appropriate, two consensus means may be calculated for the same sample if bimodality is identified in the data. In this case, two consensus means and ranges will be displayed in the data summary view.

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

The red shaded region represents the target zone for "acceptable" performance, which encompasses the NIST target value bounded by twice its uncertainty (U_{95} or U_{NIST}). The solid red lines represent the range of tolerance (values that result in an acceptable Z' score, $|Z'| \leq 2$). If the lower limit is below zero, the lower limit has been set to zero. In this view, the relative locations of individual laboratory data and consensus zones with respect to the target zone can be compared easily. In most cases, the target zone and the consensus zone overlap, which is the expected result. The major program goals are to reduce the size of the consensus zone and center the consensus zone about the target value. Analysis of an appropriate reference material as part of a quality control scheme can help to identify sources of bias for laboratories reporting results that are significantly different from the target zone. In the case in which a method comparison is relevant, different colored data points may be used to indicate 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 (NIST Standard Reference Material (SRM) with a certified, reference, or NIST-determined value) are compared to the results for another sample (e.g., another NIST SRM with a more challenging matrix, a commercial sample). The solid red box represents the target zone for the first sample (x-axis) and the second sample (y-axis). The dotted blue box represents the consensus zone for the first sample (x-axis) and the second sample (y-axis). The axes of this graph are centered about the consensus mean values for each sample or control, to a limit of twice the range of tolerance (values that result in an acceptable Z' score, $|Z'| \leq 2$). 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 % relative standard deviation (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.

SECTION 1: NUTRITIONAL ELEMENTS (Iodine)

Study Overview

In this study, participants were provided with two NIST SRMs for dietary intake, SRM 1869 Infant/Adult Nutritional Formula II (milk/whey/soy-based) and SRM 3280 Multivitamin/Multielement Tablets. Participants were asked to use in-house analytical methods to determine the mass fraction (mg/kg) of iodine (I) in each matrix. Iodine is an essential mineral required in the synthesis of thyroid hormones that regulate metabolism; yet, an accurate assessment of the element in supplement samples is challenged throughout sample preparation and instrumental measurement. Accurate measurement of iodine in foods and supplements is necessary for understanding daily intake of iodine and related health outcomes.

Dietary Intake Sample Information

Nutritional Formula. Participants were provided with three packets, each containing 10 g of nutritional formula powder. Before use, participants were instructed to thoroughly mix the contents by shaking the unopened packet prior to removal of a test sample for analysis, and to use a sample size of at least 0.5 g. Participants were asked to store the material at –20 °C before use, and to prepare one sample and report one value from each packet provided. The approximate analyte level was not reported to participants prior to the study. The certified value for iodine in SRM 1869 was assigned using results from NIST by inductively coupled plasma mass spectrometry (ICP-MS), results from the manufacturer, and results from collaborating laboratories. The certified value and uncertainty for iodine is provided in the table below on an as-received basis.

	Certified Mass Fraction in SRM 1869 (mg/kg)
Analyte	(as-received basis)
Iodine (I)	1.28 ± 0.15

Multivitamin. Participants were provided with three bottles, each containing 30 multivitamin/multielement tablets. Before use, participants were instructed to grind all tablets within a bottle, mix the resulting powder thoroughly, and to use a sample size of at least 0.2 g. Participants were asked to store the material at controlled room temperature, 10 °C to 30 °C, and to prepare one sample and report one value from each bottle provided. The approximate analyte level was not reported to participants prior to the study. The certified value for iodine in SRM 3280 was assigned using results from NIST by ICP-MS and instrumental neutron activation analysis (INAA). The certified value and uncertainty for iodine is provided in the table below, both on a dry-mass basis, as shown on the Certificate of Analysis (COA), and on an as-received basis accounting for moisture of the material (1.37 %).

	Certified Mass Fraction in SRM 3280 (mg/kg)	
Analyte	(dry-mass basis)	(as-received basis)
Iodine (I)	132.7 ± 6.6	130.9 ± 6.5

Dietary Intake Study Results

- Eighteen laboratories enrolled in this study and received samples to measure iodine. Nine laboratories reported results for the nutritional formula (50 % participation) and ten laboratories reported results for the multivitamin (56 % participation).
- The consensus means for iodine in both the nutritional formula and the multivitamin were within the target ranges. The between-laboratory variability was good for the nutritional formula but poor for the multivitamin (18 % RSD and 43 % RSD, respectively).
- Five laboratories reported using ICP-MS as their analytical technique (50 % of participants), one reported using ion chromatography with electrochemical detection (10 %), one reported using isotope dilution ICP-MS (10 %), and three did not specify an analytical method used or specified other.

Dietary Intake Technical Recommendations

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

- The low participation in this study could be due to a greater challenge posed by analysis of iodine compared to other nutritional elements, due to a lack of interest in iodine measurements, or to a lack of established protocols for iodine measurements.
- With a small number of laboratories reporting data, identification of strong trends in the data based on the information reported by participants is difficult.
- When laboratories report data biased for both samples, either both high or both low, the source may be a calibration issue. Standards used for calibration should be of known quality.
- Some general suggestions regarding iodine sample preparation are provided below.
  - Iodine is a volatile element and can form hydrogen iodide (HI) during acid digestion; care must be taken to retain iodine during sample preparation.
  - Iodine is light sensitive and at some stages of sample preparation solutions may need to be kept covered or in amber vessels.
  - When using ICP-MS, samples prepared in an acidic solution can result in carryover between analyses. Addition of a surfactant such as Triton X-100 to sample solutions will improve washout of iodine. The wash solution used between sample readings should also be slightly basic and contain Triton X-100.
  - Tetramethylammonium hydroxide (TMAH) is very effective solvent for iodine sample preparation, and many protocols call for the use of TMAH. However, TMAH is a very strong base with high toxicity and extreme caution must be taken when used. A safer alternative may be to use an acid digestion and neutralize sample solutions with a base such as ammonium hydroxide before analysis.
  - During sample preparation, iodine can adhere to modified tetrafluoroethylene (TFM) vessels, so perfluoroalkoxy (PFA) vessels or quartz/glass vessels are recommended to eliminate erratic results.

Table 1-1. Individualized data summary table (NIST) for iodine in nutritional formula and multivitamin.

National Institute of Standards & Technology

HAMQAP Exercise 2 - 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*
Iodine	SRM 1869 Infant/Adult Nutritional Formula II	mg/kg	1.28	0.08			9	1.20	0.22	1.28	0.15
Iodine	SRM 3280 Multivitamin/Multielement Tablets	mg/kg	130.9	3.3			10	121.3	52.7	130.9	6.5

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 1-2. Data summary table for iodine in nutritional formula and multivitamin. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		Iodine									
		SRM 1869 Infant/Adult Nutritional Formula II (mg/kg)					SRM 3280 Multivitamin/Multielement Tablets (mg/kg)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				1.28	0.15				130.9	6.5
	B001										
	B005	1.27	1.28	1.29	1.28	0.01					
	B007						136	134	130	133	3
	B008	0.002	0.002	0.002	0.002	0.000	0.15	0.18	0.18	0.17	0.02
	B011	1.50	1.30	1.30	1.37	0.12	145	119	96	120	25
	B012										
	B021	1.04	1.08	1.14	1.09	0.05	83.1	88.5	105	92.2	11.4
	B022										
	B024										
	B028	1.00	0.97	1.06	1.01	0.05	78.3	75.6	84	79.3	4.3
	B031	11.02	10.97	10.58	10.86	0.24	224	215	243	227	14
	B032										
	B035	1.23	1.22	1.21	1.22	0.01	109	142	148	133	21
	B036	1.23	1.23	1.19	1.22	0.02	133	129	122	128	6
	B037										
	B038	1.17	1.23	1.17	1.19	0.03	133	120	107	120	13
	B042						192	119	181	164	39
	B044										
Community Results		Consensus Mean			1.20		Consensus Mean			121	
		Consensus Standard Deviation			0.22		Consensus Standard Deviation			53	
		Maximum			10.86		Maximum			227	
		Minimum			0.002		Minimum			0.2	
		N			9		N			10	

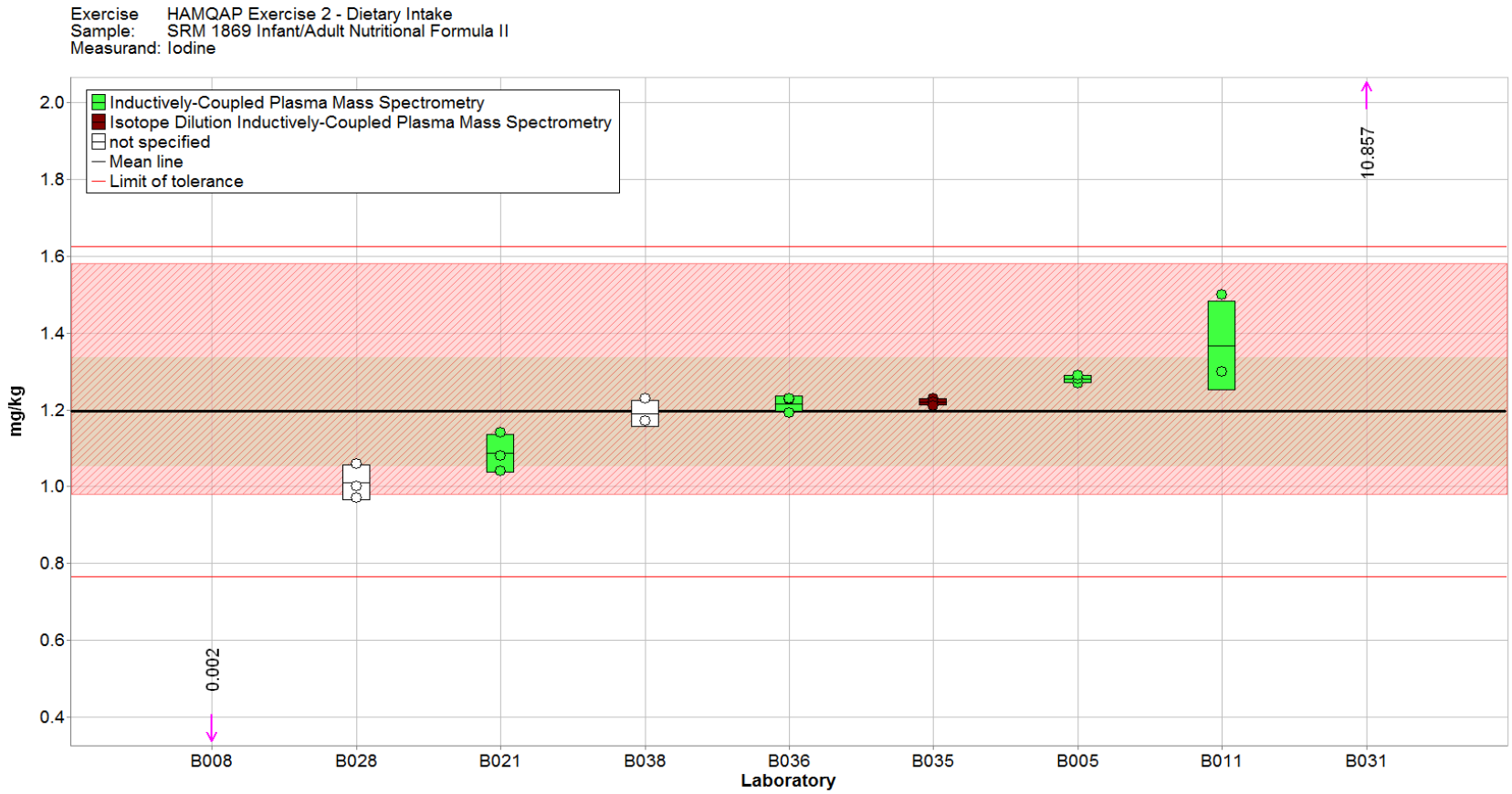

Figure 1-1. Iodine in SRM 1869 Infant/Adult Nutritional Formula II (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black 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, $|Z_{\text{NIST}}| \leq 2$.

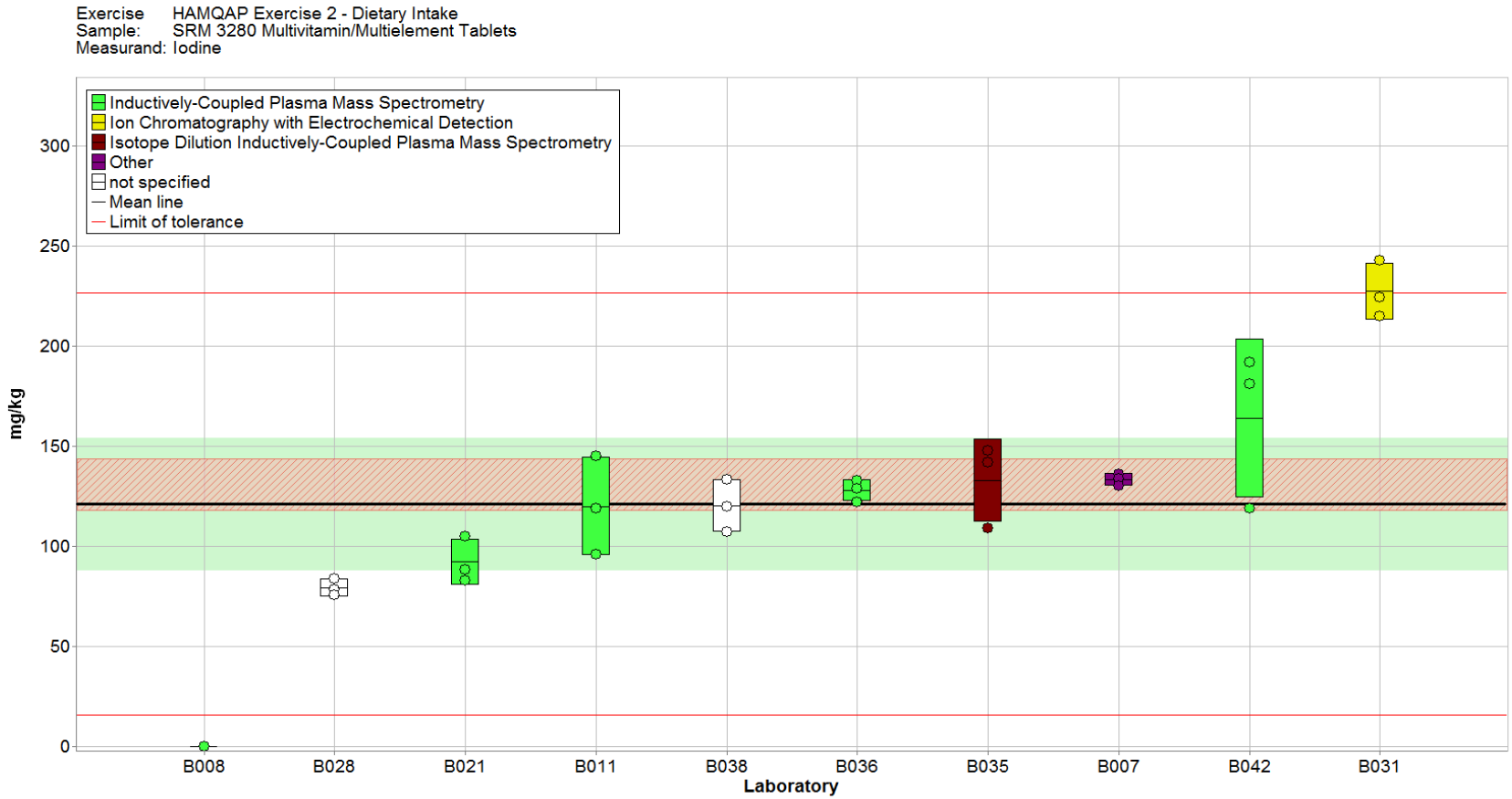

Figure 1-2. Iodine in SRM 3280 Multivitamin/Multielement Tablets (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black 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, $|Z_{\text{NIST}}| \leq 2$.

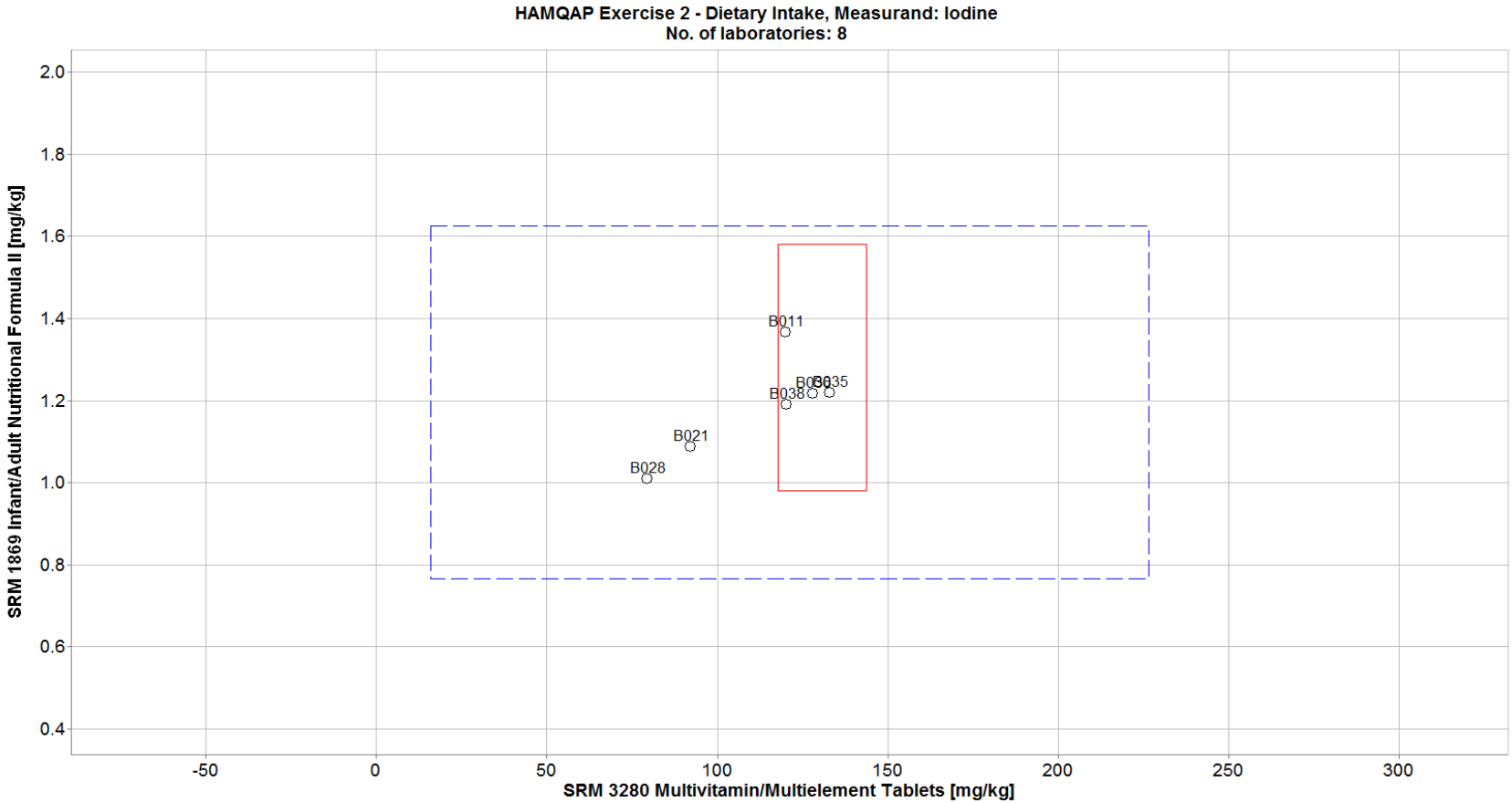

Figure 1-3. Laboratory means for iodine in SRM 1869 Infant/Adult Nutritional Formula II and SRM 3280 Multivitamin/Multielement Tablets (sample/sample comparison view). In this view, the individual laboratory mean for one sample (SRM 3280) is compared to the individual laboratory mean for a second sample (SRM 1869). The solid red box represents the NIST range of tolerance for the two samples, SRM 3280 (x-axis) and SRM 1869 (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 3280 (x-axis) and SRM 1869 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

SECTION 2: TOXIC ELEMENTS (Arsenic, Cadmium, Lead, Mercury, Selenium)

Study Overview

In this study, participants were provided with two samples for dietary intake, Siberian ginseng root extract and kudzu extract. Participants were asked to use in-house analytical methods to determine the mass fractions (ng/g) of arsenic (As), cadmium (Cd), lead (Pb), mercury (Hg), and selenium (Se) in each matrix. The United States' cGMPs require dietary supplement manufacturers to establish limits on contaminants, therefore laboratories must establish scientifically valid methods for the determination of toxic elements to demonstrate the products meet the specifications in 21 CFR 111.70(b)(3). Selenium is an essential element but is toxic at elevated levels, and therefore should only be included in supplements at safe concentrations. Monitoring these and other toxic substances in foods and dietary supplements prevents exposure of consumers and related negative health outcomes.

Dietary Intake Sample Information

Siberian Ginseng Root Extract. Participants were provided with three packets, each containing approximately 1 g of powdered Siberian ginseng root extract. Before use, participants were instructed to thoroughly mix the contents of each packet and to use a sample size of at least 0.5 g. Participants were asked to store the material at controlled room temperature, 10 °C to 30 °C, and to prepare a single sample and to report a single value from each packet provided. Approximate analyte levels were not reported to participants prior to the study. A target value for Hg in the Siberian ginseng root extract was assigned using results from NIST by cold vapor isotope dilution inductively coupled plasma mass spectrometry (CV ID ICP-MS). The NIST-determined value and uncertainty for Hg in Siberian ginseng root extract is provided in the table below on an as-received basis. Target values have not been determined for As, Cd, Pb, and Se in the Siberian ginseng root extract.

Analyte	NIST-Determined Mass Fraction in Siberian Ginseng Root Extract (ng/g)
Mercury (Hg)	397 ± 104

Kudzu Extract. Participants were provided with three packets, each containing approximately 1 g of powdered kudzu extract. Before use, participants were instructed to thoroughly mix the contents of each packet and to use a sample size of at least 0.5 g. Participants were asked to store the material at controlled room temperature, 10 °C to 30 °C, and to prepare a single sample and to report a single value from each packet provided. Approximate analyte levels were not reported to participants prior to the study. Target values for As, Cd, Pb, and Se in the kudzu extract were assigned using results from NIST by ICP-MS, and a target value for Hg in the kudzu extract was assigned using results from NIST by CV ID ICP-MS. The NIST-determined values and uncertainty for As, Cd, Pb, Hg, and Se in kudzu extract are provided in the table below on an as-received basis.

Analyte	NIST-Determined Mass Fraction in Kudzu Extract (ng/g)
Arsenic (As)	771 ± 47
Cadmium (Cd)	81 ± 4
Lead (Pb)	1040 ± 790
Mercury (Hg)	10 ± 5
Selenium (Se)	117 ± 5

Dietary Intake Study Results

- The enrollment and reporting statistics for the toxic elements study is described in the table below. Some of the reported values were non-quantitative (zero or below LOQ) but are included in the participation and reporting statistics.

		Number of Laboratories Reporting Results (Percent Participation)	
Analyte	Number of Laboratories Requesting Samples	Siberian Ginseng Root Extract	Kudzu Extract
As	35	25 (71 %)	24 (69 %)
Cd	35	24 (69 %)	23 (66 %)
Pb	35	24 (69 %)	22 (63 %)
Hg	33	20 (61 %)	17 (52 %)
Se	30	19 (63 %)	20 (67 %)

- The consensus means were within the target ranges for all analytes except Se in kudzu, in which the consensus mean was above the target range.
- The between-laboratory variabilities for each sample-analyte pair are summarized below, showing that performance was best for As, Cd, and Pb.

	Between-laboratory variability (% RSD)	
Analyte	Siberian Ginseng Root Extract	Kudzu Extract
As	18 %	16 %
Cd	15 %	17 %
Pb	8 %	27 %
Hg	41 %	57 %
Se	80 %	52 %

- The sample preparation methods reported by participating laboratories are summarized in the table below. Most laboratories reported using either microwave digestion or hot block digestion for all five analytes.

	Percent Reporting				
Reported Method	As	Cd	Pb	Hg	Se
Microwave digestion	57 %	62 %	61 %	62 %	53 %
Hot Block digestion	23 %	19 %	22 %	24 %	21 %
Acid hydrolysis	4 %	5 %	4 %	5 %	5 %
Open beaker digestion	4 %	5 %	4 %	5 %	5 %
Other or no response	9 %	10 %	9 %	5 %	16 %

Technical Recommendations

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

- For all analytes, no pattern was observed between reported result and analytical method, but a correlation was identified between reported results and sample preparation method for some sample analyte pairs.
  - Laboratories using a microwave digestion generally reported lower values for most sample-analyte pairs (**Figures 2-8 through 2-14** and **Figures 2-19 through 2-24**).
  - Laboratories reporting the highest values reported using hot block digestion (**Figures 2-14, 2-23, and 2-24**).
- Sample preparation methods should be well established before analyzing unknown samples. Established quality control materials (SRMs, CRMs, RMs, and in-house materials) and accepted methods of analysis can assist in this process.
- Detection of the analyte in the sample may be improved by limiting the number of dilutions performed, however matrix effects may become more significant. A matrix-matched calibration curve may reduce some of the matrix interferences.
- For arsenic, most laboratories reported data that were within the consensus ranges, as shown in **Figures 2-1 and 2-2**. Several laboratories reported data outside the NIST target range for As in the kudzu extract (**Figure 2-2**).
  - In **Figure 2-5**, a strong linear trend is apparent between the reported results for As in the two materials, which may indicate a calibration issue. Calibration curves must be linear and include the lowest and highest values expected to be measured in the sample solutions. Extrapolation of the curve may cause incorrect results.
    - Difficulty in the digestion of samples can cause bias and/or increased variability between samples.
      - Neither extract should be difficult to digest; 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.
        - The high temperatures of a vigorous microwave digestion should convert all volatile organoarsenic species to arsenic acid (AsV), at which point subsequent heating will not result in loss of arsenic.

        - Open-beaker digestion should not be used for As sample preparation. Closed-vessel digestions should be opened with care ensuring that no As is lost as a result of inadvertent venting.
      - Incomplete sample digestion may yield interferences that cause signal enhancement or suppression, thereby introducing measurement bias in one of the matrices.
      - Collision cell technology can be used to minimize the molecular ion interferences that may be found when analyzing As in these two materials.
    - ID ICP-MS is not a practicable method because As is monoisotopic. Measurement methods should be reported correctly and completely.
- For cadmium, most laboratories reported data that were within the consensus ranges (**Figures 2-6 through 2-9**). Several laboratories reported data outside the NIST target range for Cd in the kudzu extract (**Figure 2-9**) with a large with-in laboratory variability.
  - In **Figure 2-10**, a linear trend is apparent between the reported results for Cd the two materials, which may indicate a calibration issue. As with arsenic, calibration curves must be linear and include the lowest and highest values expected to be measured in the sample solutions. Extrapolation of the curve may cause incorrect results.
    - The boiling point of Cd is high and volatile loss of Cd is not a concern. Spectral interferences can make Cd difficult to measure accurately by ICP-MS.
    - High concentrations of certain elements, mainly Mo, Sn, or Zr, are known to cause interferences in the analysis of Cd by ICP-MS. A scan of the sample before analysis will indicate any potential interferences in the sample that will need to be addressed.
      - NIST scans of both materials indicated high levels of Mo relative to Cd. The kudzu extract also contains a high level of Sn that may interfere with Cd analysis.
    - Anion exchange separation of matrix elements prior to ICP-MS can reduce interferences.
    - Collision cell technology can be used to minimize molecular interferences that may be found in these two materials.
- For lead, **Figures 2-11 through 2-14** show that most laboratories reported data that were within the consensus ranges. Several laboratories reported data outside the consensus range for Pb in the kudzu extract with very large with-in laboratory variability, as shown in **Figures 2-12 and 2-14**.
  - No linear trend was observed in **Figure 2-15** between the reported results for Pb in the two materials. Although the levels of Pb in the two samples were comparable, the between-laboratory variability for the kudzu extract (27 %) was significantly higher than for the Siberian ginseng extract (8 %), indicating a greater difficulty with the analysis of the kudzu extract.
    - Some laboratories reported high sample-to-sample variability (59 % to > 100 %), which may be caused by difficulties in sample preparation, incomplete sample digestion, or calibration curves which do not encompass all sample solutions measured.
    - Lead is easily digested and volatile loss of Pb is not a concern. Digestion with HCl may form insoluble $PbCl_2$ precipitate, so digestion with HNO_3 is recommended.
    - The kudzu material may be more inhomogeneous for Pb in comparison to the Siberian ginseng material, causing larger observed sample-to-sample variability in the kudzu extract Pb data.

- Analysis of an appropriate number of procedural blanks is always important and can be critical when sample concentrations are near the DLs or, as in this case, when trying to determine the cause of sample-to-sample variability. Analysis of many blanks can provide information about whether the variability is arising from the sample preparation method itself. The suggested minimum number of blanks to prepare is equal to the number of samples being prepared.

- For mercury, **Figures 2-16 through 2-19** show that most laboratories reported data that were within the consensus ranges. Several laboratories reported data outside the NIST target range for Hg in the kudzu extract (**Figure 2-17**), with very large with-in laboratory variability.
  - No linear trend was observed in **Figure 2-20** between the reported results for mercury in the two materials.
- Mercury is volatile, so care must be taken to not lose Hg during sample preparation. Microwave digestion is the best method for sample preparation for mercury analysis.
- The low levels of mercury in the kudzu extract may be close to method detection limits (MDL) for some techniques.
  - A sufficient number of procedural blanks must be used to determine an accurate MDL and LOQ. Blanks and backgrounds for Hg measurements may be large, leading to high detection limits and making determination of low-level samples difficult.
  - Low concentrations of Hg are not stable in solution over time. Samples should be prepared as near as possible to the time of analysis. Samples containing low concentrations of Hg may be more stable in dilute HCl than in dilute HNO_3.
  - The sensitivity of ICP-MS is low for Hg. Using cold vapor mercury generation increases sensitivity of ICP-MS and allows lower levels of Hg to be measured.
  - Low level measurements are often challenged by contamination from sources such as poorly cleaned glassware or other laboratory materials.
  - Mercury carryover between samples is common on many instruments, which can lead to erratic results if an adequate washout time is not used after each measurement. Use of dilute HCl in the rinse solution may decrease the length of necessary washout time.
  - Laboratories reporting measured values at the higher end of the range also reported larger within-laboratory variability.
- For selenium, **Figures 2-21 through 2-24** show that only a few laboratories reported data that were within the consensus ranges. Laboratories reporting data below the 95 % confidence interval (**Figures 2-22 and 2-24**) were also the laboratories that reported data within the NIST range of tolerance and reported a smaller within-laboratory variability (3 % to 9 %).
  - In **Figure 2-25**, a linear trend is apparent between the reported results for Se in the two materials, which may indicate a calibration issue. Calibration curves must be linear and include the lowest and highest values expected to be measured in the sample solutions. Extrapolation of the curve may cause incorrect results. Calibration standards must also be of known quality and from a trusted source.
  - The most abundant isotope of Se (^{80}Se) is often not used for ICP-MS determination of Se to avoid interference from $^{40}Ar_2^+$. When a less abundant isotope is selected, the detection limit is increased.
  - Due to the low concentrations of Se in the materials, a large number of sample procedural blanks should be prepared along with the samples.

- To avoid calculation errors for all measurements, use a quality assurance material (CRM, SRM, RM), or in-house prepared quality control material, to help identify calculation errors and to be sure results are reported in the correct units.

Table 2-1. Individualized data summary table (NIST) for toxic elements in Siberian ginseng extract and kudzu extract.

National Institute of Standards & Technology

HAMQAP Exercise 2 - 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*
Cadmium	Siberian Ginseng Root Extract	ng/g					24	63.7	9.5		
Cadmium	Kudzu Extract	ng/g	81	2			23	80	13	81	4
Mercury	Siberian Ginseng Root Extract	ng/g	397	52			20	320	130	397	104
Mercury	Kudzu Extract	ng/g	10	3			18	10.9	6.2	10	5
Lead	Siberian Ginseng Root Extract	ng/g					24	840	66		
Lead	Kudzu Extract	ng/g	1040	400			22	1200	320	1040	790
Selenium	Siberian Ginseng Root Extract	ng/g					19	90	73		
Selenium	Kudzu Extract	ng/g	117	3			20	200	110	117	5
Total Arsenic	Siberian Ginseng Root Extract	ng/g					25	520	95		
Total Arsenic	Kudzu Extract	ng/g	771	24			24	820	130	771	47

x_i Mean of reported values

s_i Standard deviation of reported values

Z'_{comm} Z'-score with respect to community consensus

Z_{NIST} Z-score with respect to NIST value

N Number of quantitative values reported

x* Robust mean of reported values

s* Robust standard deviation

x_{NIST} NIST-assessed value

U expanded uncertainty about the NIST-assessed value

Table 2-2. Data summary table for total arsenic in Siberian ginseng extract and kudzu extract. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		Total Arsenic									
		Siberian Ginseng Root Extract (ng/g)					Kudzu Extract (ng/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target									771	47
	B001										
	B002										
	B003	0.500	0.500	0.512	0.504	0.007	0.821	0.822	0.818	0.820	0.002
	B005		528	515	533	20	871	848	787	835	43
	B007	508	540	515	521	17	833	841	874	849	22
	B008	568	538	551	552	15	871	913	912	899	24
	B011	547	542	594	561	29	812	826	831	823	10
	B012										
	B013										
	B019	580	571	488	546	50	743	912	979	878	122
	B020	440	440	430	437	6	670	670	690	677	12
	B021	326	330	319	325	6	655	568	619	614	44
	B022	380	340	320	347	31	580	560	550	563	15
	B023	506	491	525	507	17	858	813	792	821	34
	B024	600	550	580	577	25	870	870	880	873	6
	B025										
	B026	538	568	551	552	15	909	906	905	907	2
	B027	589	593	599	594	5	904	937	902	914	20
	B028	696	757	666	706	46	933	969	893	932	38
	B029										
	B031	637	629	637	634	5	962	958	956	959	3
	B032	546	533	548	542	8	867	886	869	874	11
	B033	618	675	650	648	28	980	983	996	986	9
	B034										
	B035	438	465	490	465	26	713	759	785	752	36
	B036	470	475	470	472	3	757	758	764	760	4
	B037										
	B038	546	518	520	528	16	851	846	818	838	18
	B039										
	B040	510	490	480	493	15	800	780	790	790	10
	B042	473	417	461	450	29	507	520	511	512	6
	B043	538	555	456	516	53	880	933	943	919	34
	B044										
	B045	514	506	511	510	4	813	742	740	765	42
	B046	340	329	334	334	6					
Community Results		Consensus Mean			518		Consensus Mean			824	
		Consensus Standard Deviation			95		Consensus Standard Deviation			129	
		Maximum			706		Maximum			986	
		Minimum			0.504		Minimum			0.820	
		N			25		N			24	

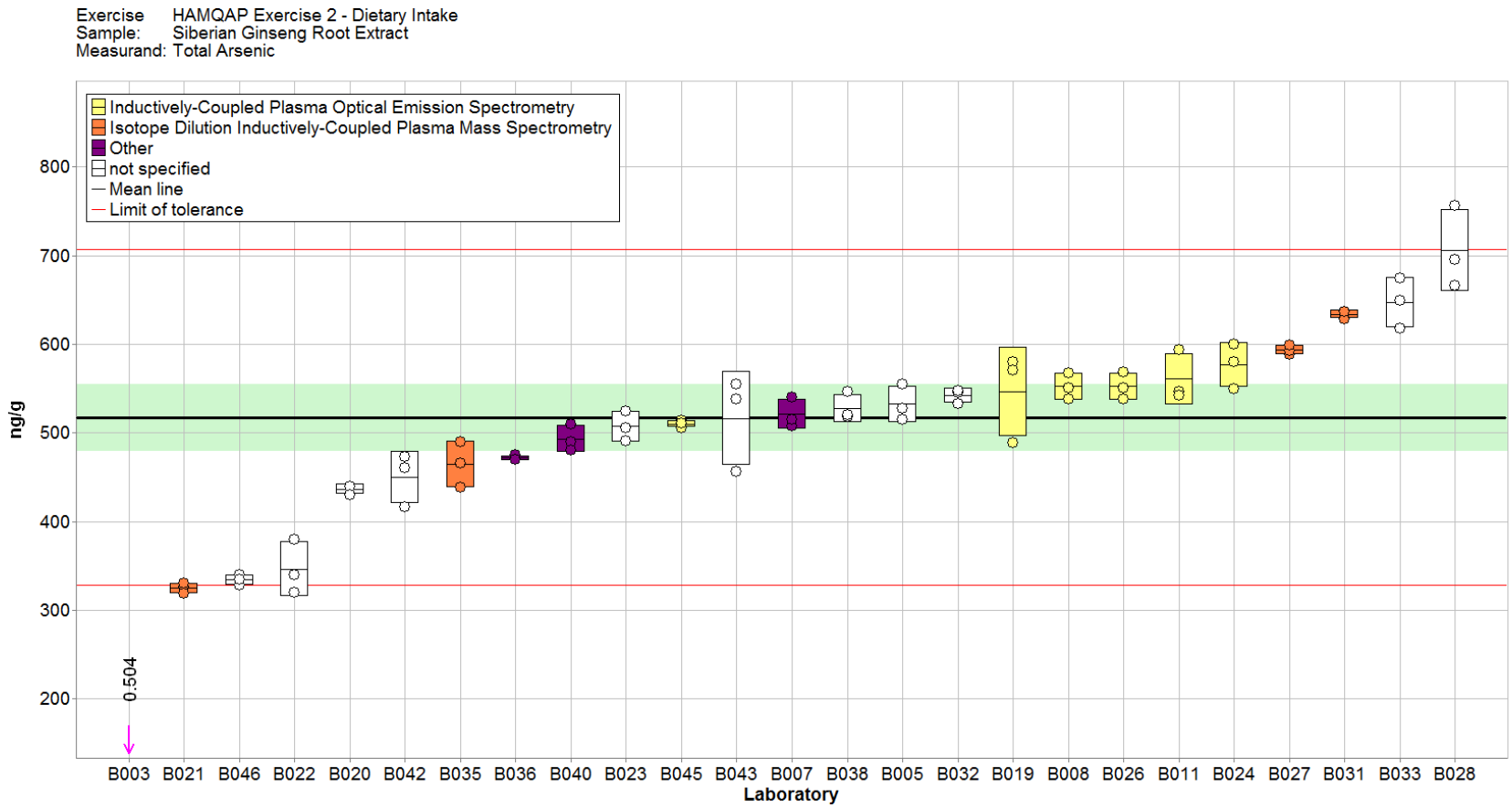

Figure 2-1. Total arsenic in Siberian Ginseng Root Extract (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black 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 NIST value has not been determined in this material.

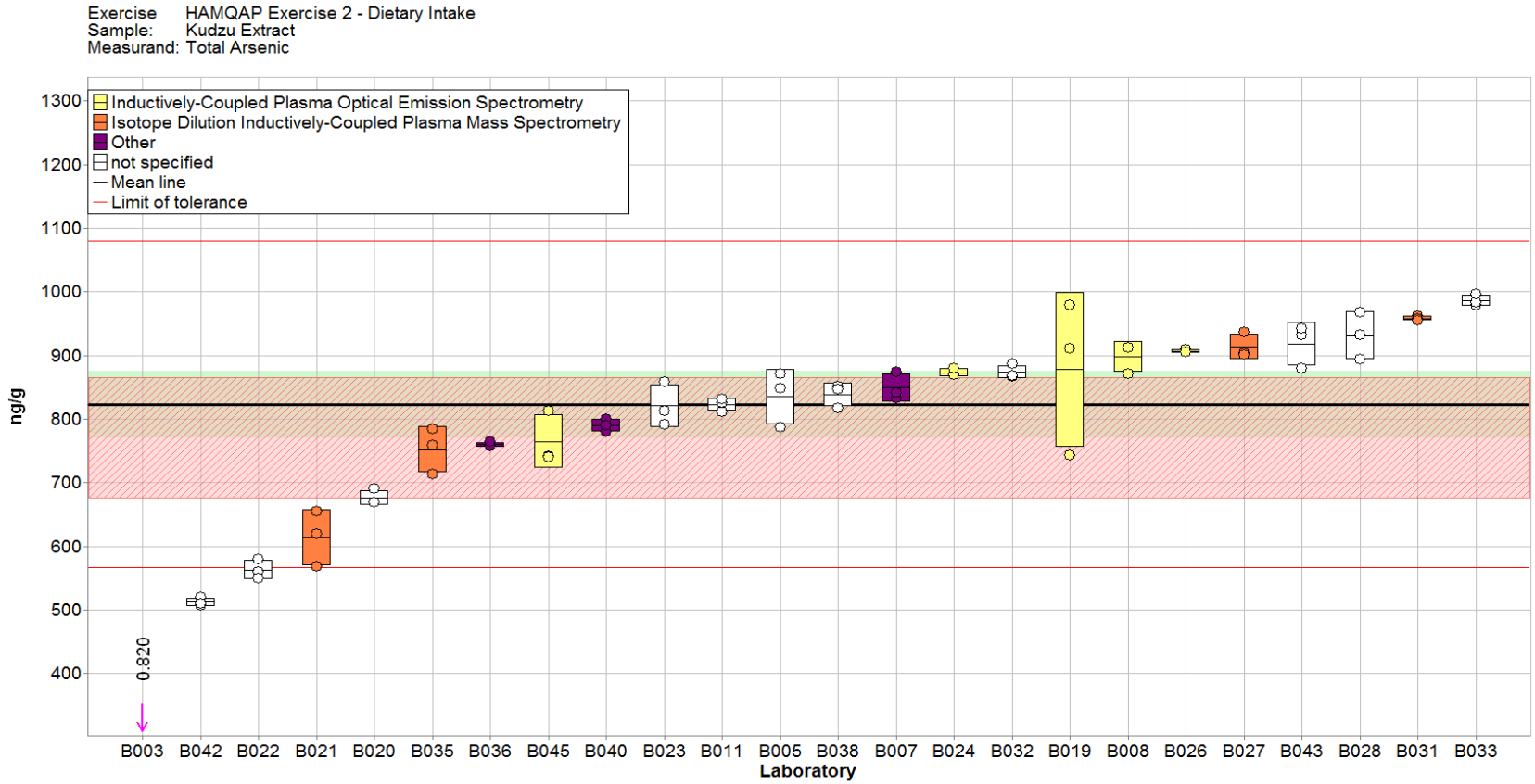

Figure 2-2. Total arsenic in Kudzu Extract (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

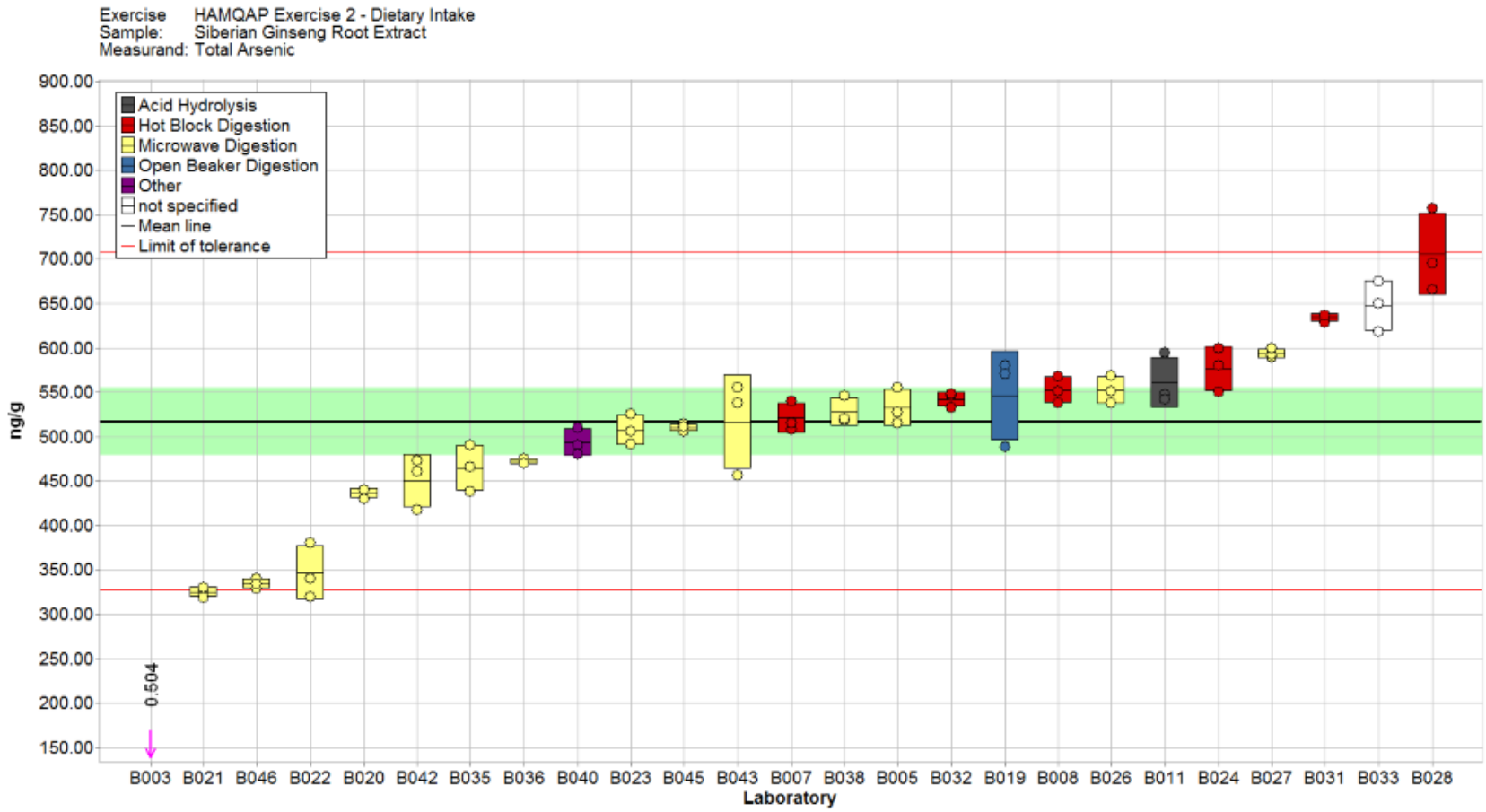

Figure 2-3. Total arsenic in Siberian Ginseng Root Extract (data summary view – sample preparation method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the sample preparation method employed. The solid black 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 NIST value has not been determined in this material.

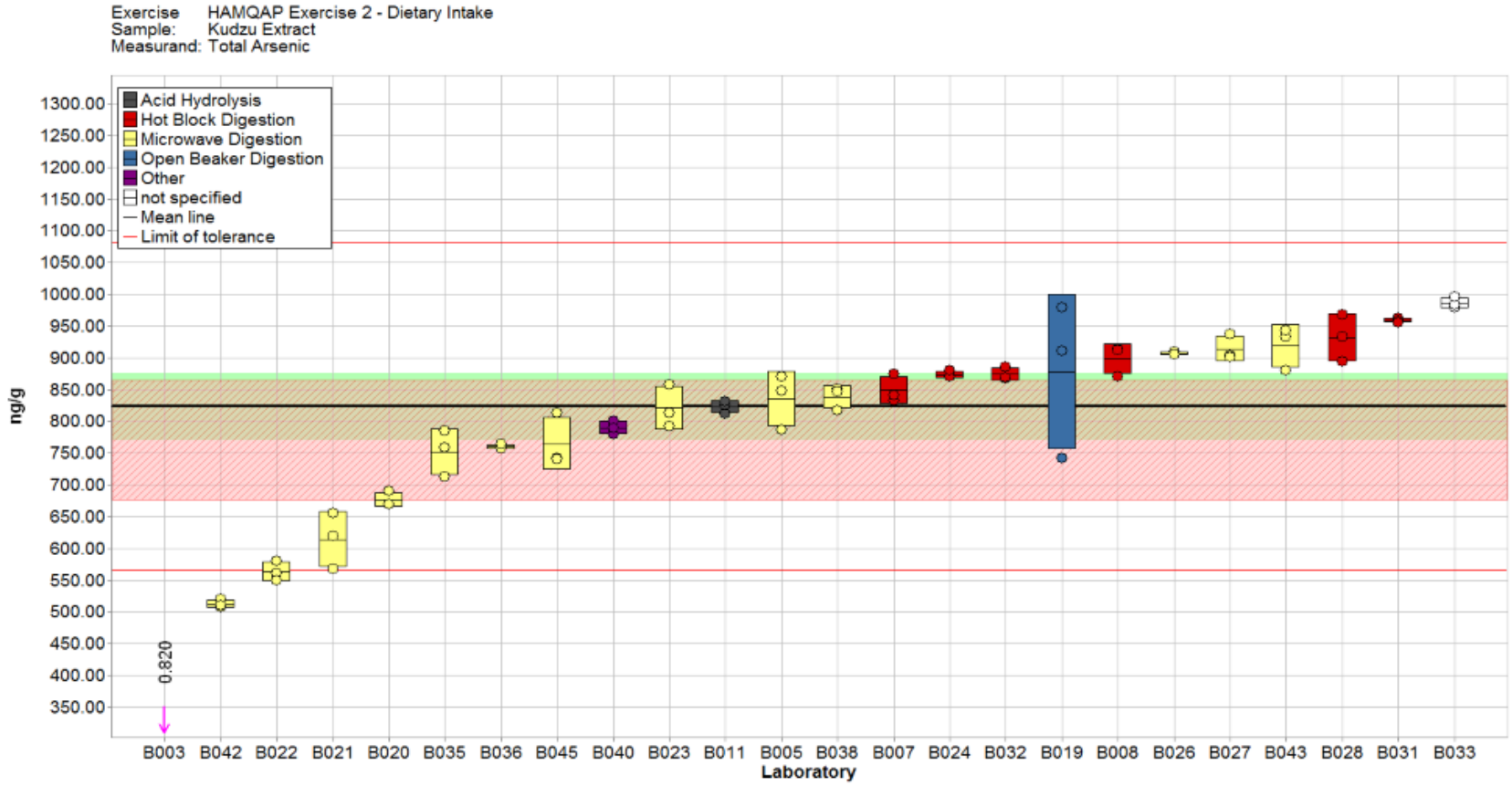

Figure 2-4. Total arsenic in Kudzu Extract (data summary view – sample preparation method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the sample preparation method employed. The solid black 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

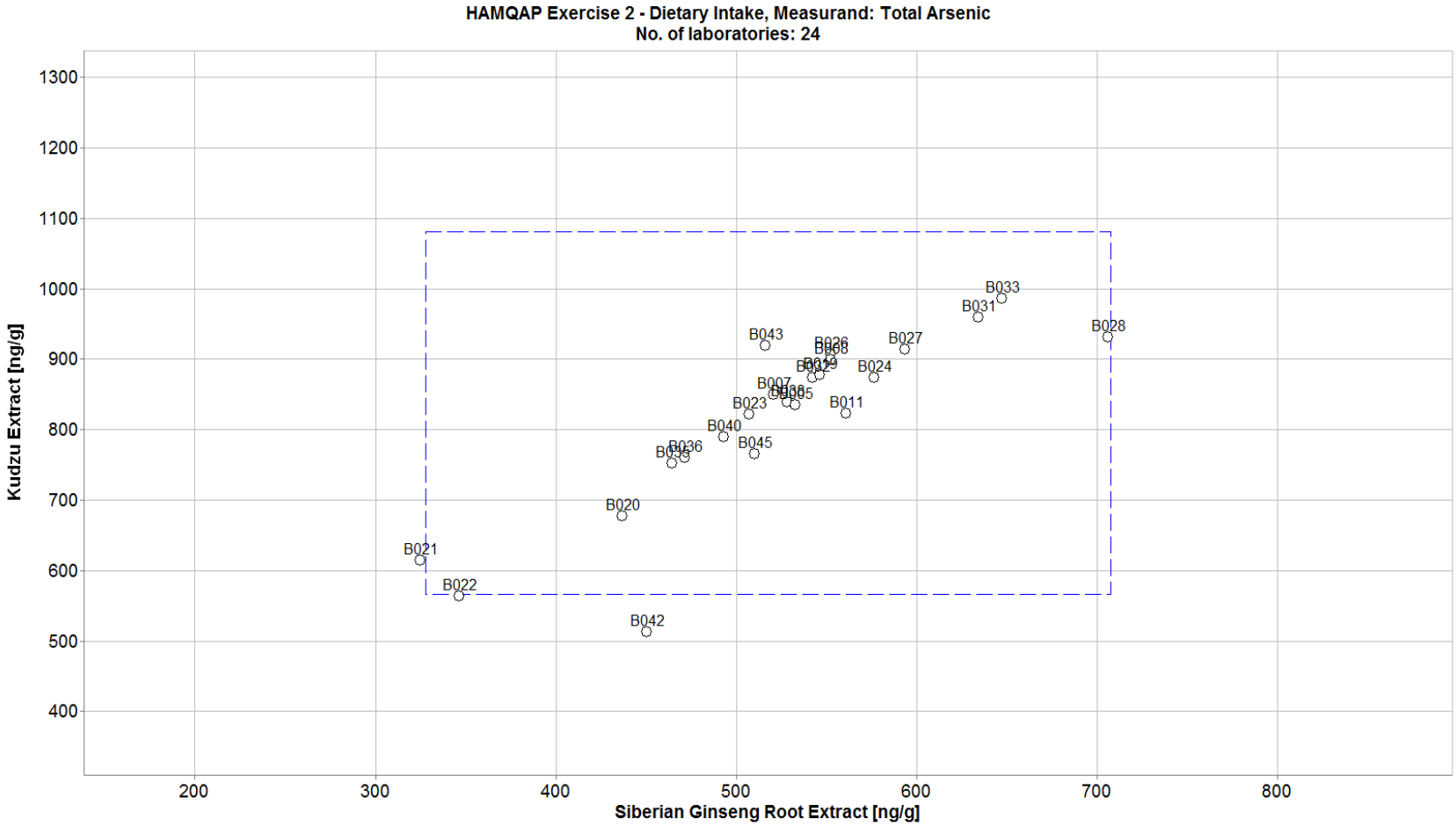

Figure 2-5. Laboratory means for total arsenic in Siberian Ginseng Root Extract and Kudzu Extract (sample/sample comparison view). In this view, the individual laboratory mean for one sample (Siberian Ginseng Root Extract) is compared to the mean for a second sample (Kudzu Extract). The dotted blue box represents the consensus range of tolerance for Siberian Ginseng Root Extract (x-axis) and Kudzu 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 Siberian ginseng extract and kudzu extract. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		Cadmium									
		Siberian Ginseng Root Extract (ng/g)					Kudzu Extract (ng/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target									81	4
	B001										
	B002										
	B003	0.063	0.062	0.062	0.062	0.001	0.079	0.076	0.077	0.077	0.001
	B005	61.9	60.7	59.0	60.5	1.5	71.3	70.2	70.3	70.6	0.6
	B007	62.1	67.6	66.1	65.3	2.8	78.0	78.3	83.7	80.0	3.2
	B008	68.5	69.3	66.8	68.2	1.3	82.1	81.0	90.3	84.5	5.1
	B011	69.0	72.0	74.0	71.7	2.5	90.0	87.0	93.0	90.0	3.0
	B012										
	B013										
	B019	59.9	60.8	55.9	58.9	2.6	47.4	72.9	66.8	62.4	13.3
	B020	61.0	62.0	59.0	60.7	1.5	66.0	72.0	70.0	69.3	3.1
	B021	43.0	48.0	46.0	45.7	2.5	63.0	62.0	64.0	63.0	1.0
	B022	62.0	60.0	56.0	59.3	3.1	72.0	76.0	76.0	74.7	2.3
	B023	63.6	64.6	66.0	64.7	1.2	80.1	75.5	76.9	77.5	2.4
	B024	120.0	110.0	100.0	110.0	10.0	120.0	130.0	140.0	130.0	10.0
	B025										
	B026	67.7	66.7	68.8	67.7	1.0	68.6	68.6	68.9	68.7	0.2
	B027	77.1	68.9	71.8	72.6	4.1	80.4	81.0	79.2	80.2	0.9
	B028										
	B029										
	B031	67.9	64.3	65.2	65.8	1.9	98.2	99.3	150.2	115.9	29.7
	B032	62.7	68.1	68.4	66.4	3.2	80.3	75.6	78.2	78.0	2.3
	B033	64.3	67.5	67.2	66.3	1.7	83.3	85.5	89.8	86.2	3.3
	B034										
	B035	56.0	58.4	62.0	58.8	3.0	74.9	77.7	78.6	77.0	1.9
	B036	55.0	57.0	56.0	56.0	1.0	75.0	74.0	71.0	73.3	2.1
	B037										
	B038	85.3	73.4	72.8	77.2	7.0	156.4	98.3	106.9	120.5	31.3
	B039										
	B040	72.0	68.0	64.0	68.0	4.0	79.0	81.0	79.0	79.7	1.2
	B042	0.0	0.0	0.0	0.0	0.0	60.9	65.9	65.8	64.2	2.9
	B043	57.9	57.5	58.7	58.0	0.6	71.4	73.4	72.6	72.5	1.0
	B044										
	B045	72.0	64.0	62.0	66.0	5.3	71.0	80.0	66.0	72.3	7.1
	B046	57.1	50.7	59.7	55.9	4.7					
Community Results		Consensus Mean			63.7		Consensus Mean			77	
		Consensus Standard Deviation			9.5		Consensus Standard Deviation			13	
		Maximum			110		Maximum			130	
		Minimum			0		Minimum			0.08	
		N			24		N			23	

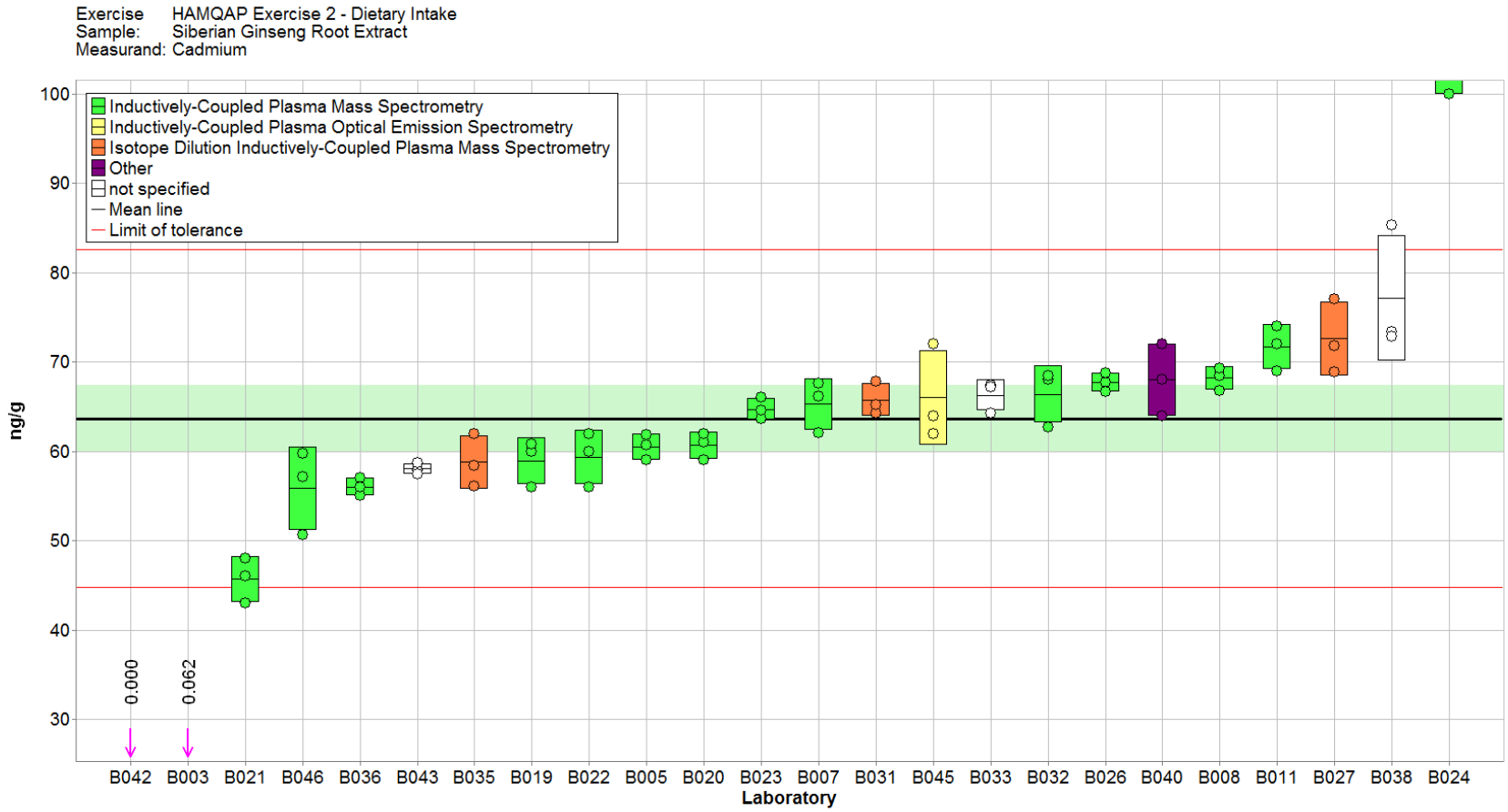

Figure 2-6. Cadmium in Siberian Ginseng Root Extract (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black 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 NIST value has not been determined in this material.

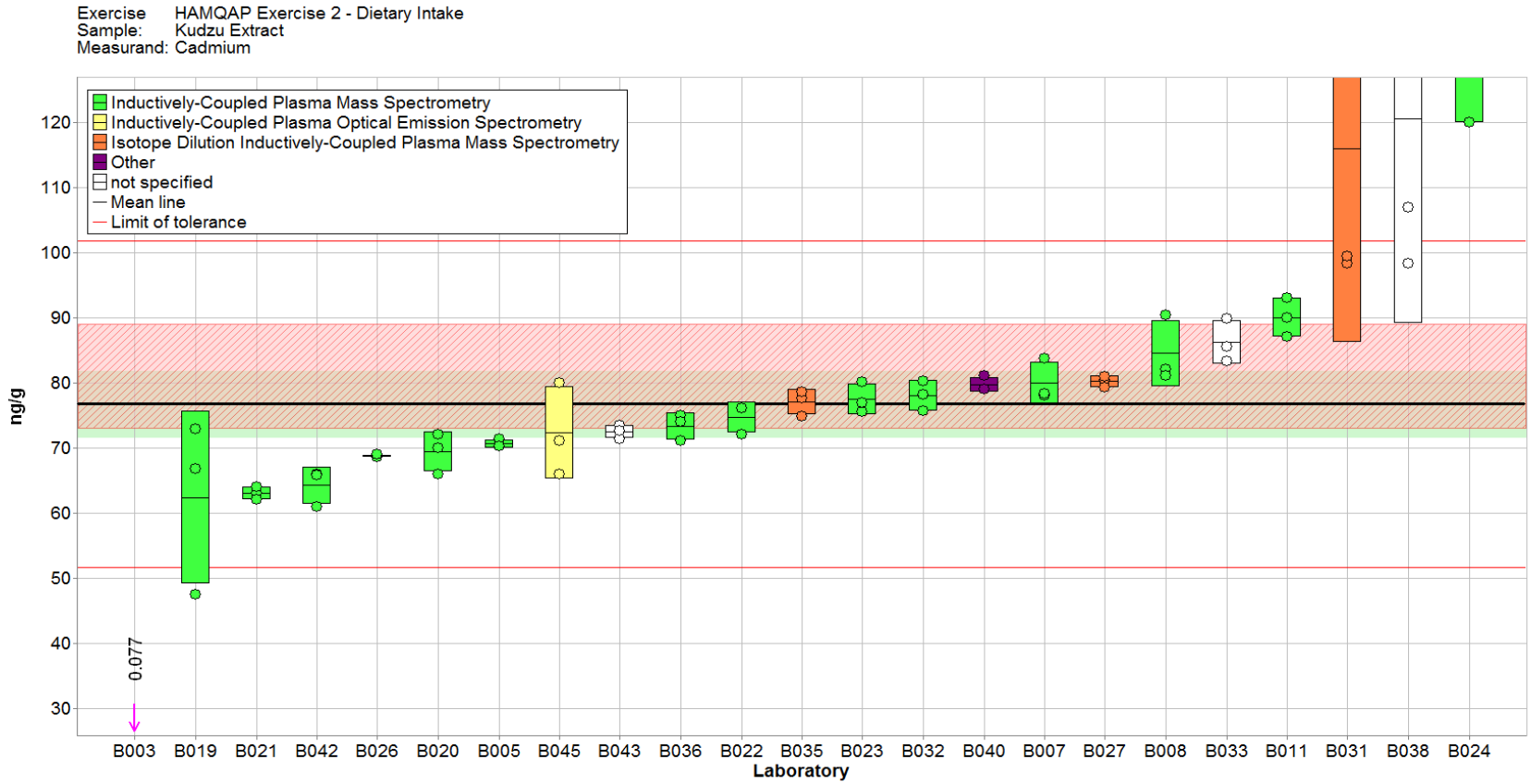

Figure 2-7. Cadmium in Kudzu Extract (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

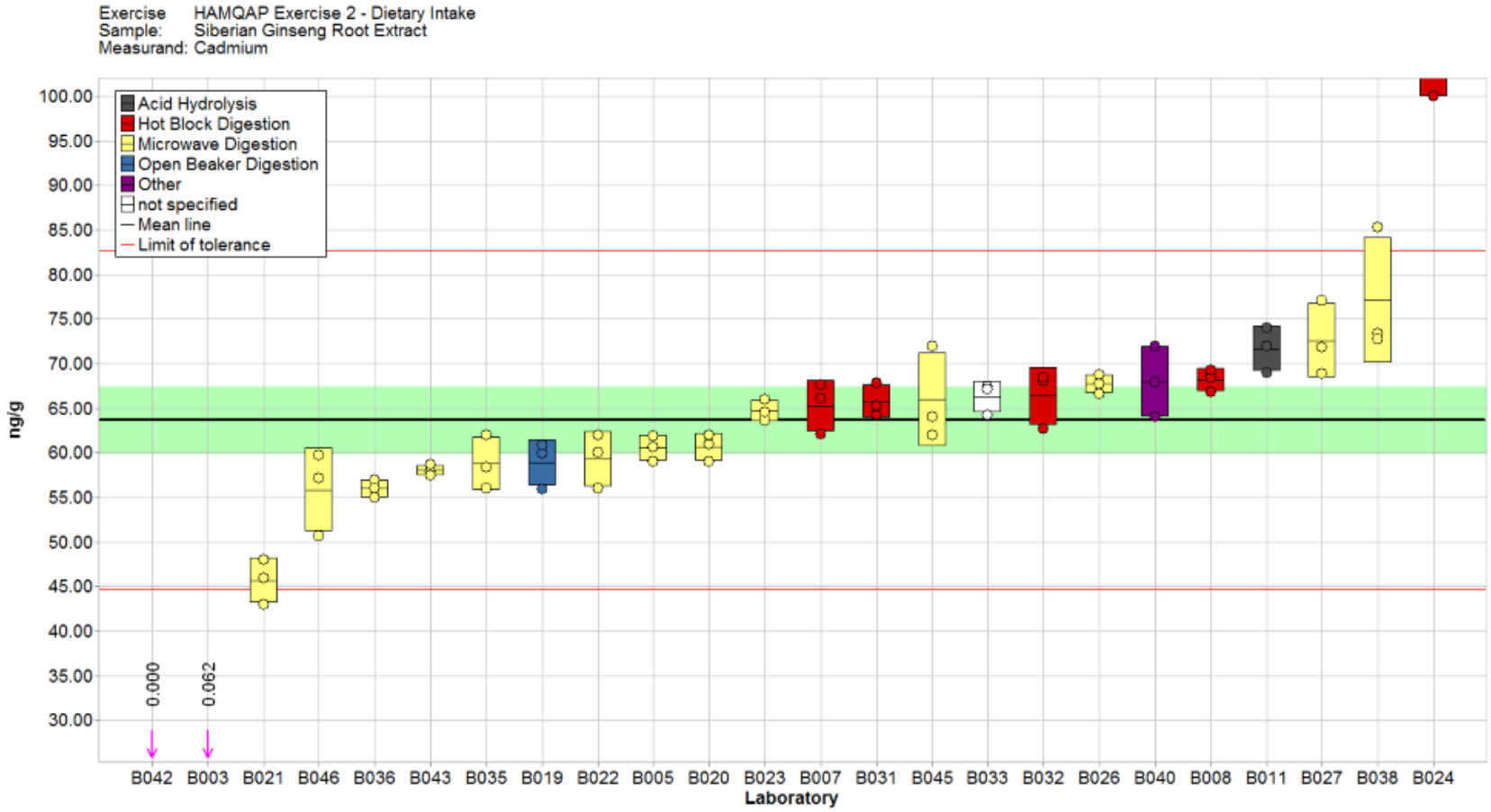

Figure 2-8. Cadmium in Siberian Ginseng Root Extract (data summary view – sample preparation method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the sample preparation method employed. The solid black 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 NIST value has not been determined in this material.

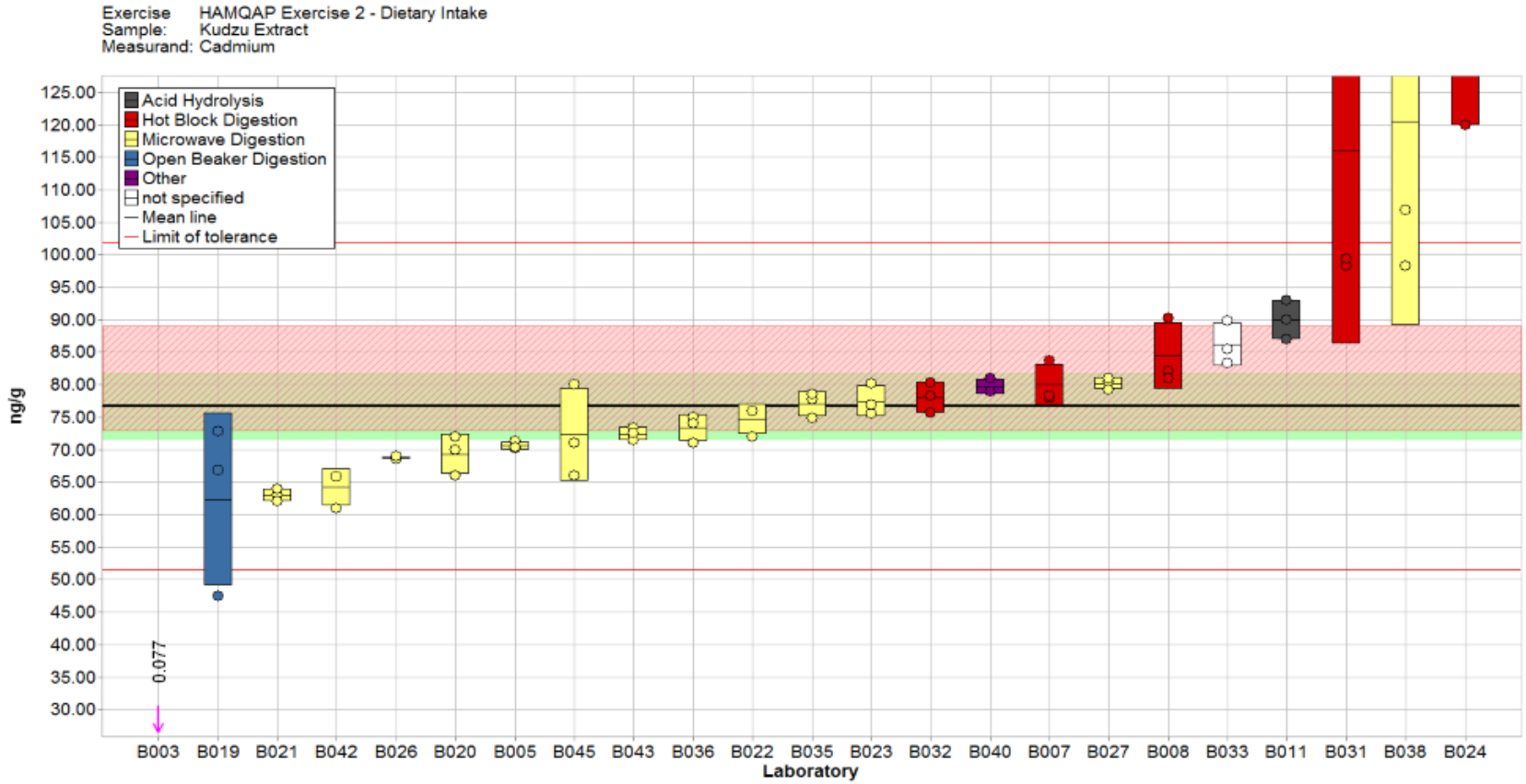

Figure 2-9. Cadmium in Kudzu Extract (data summary view – sample preparation method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the sample preparation method employed. The solid black 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

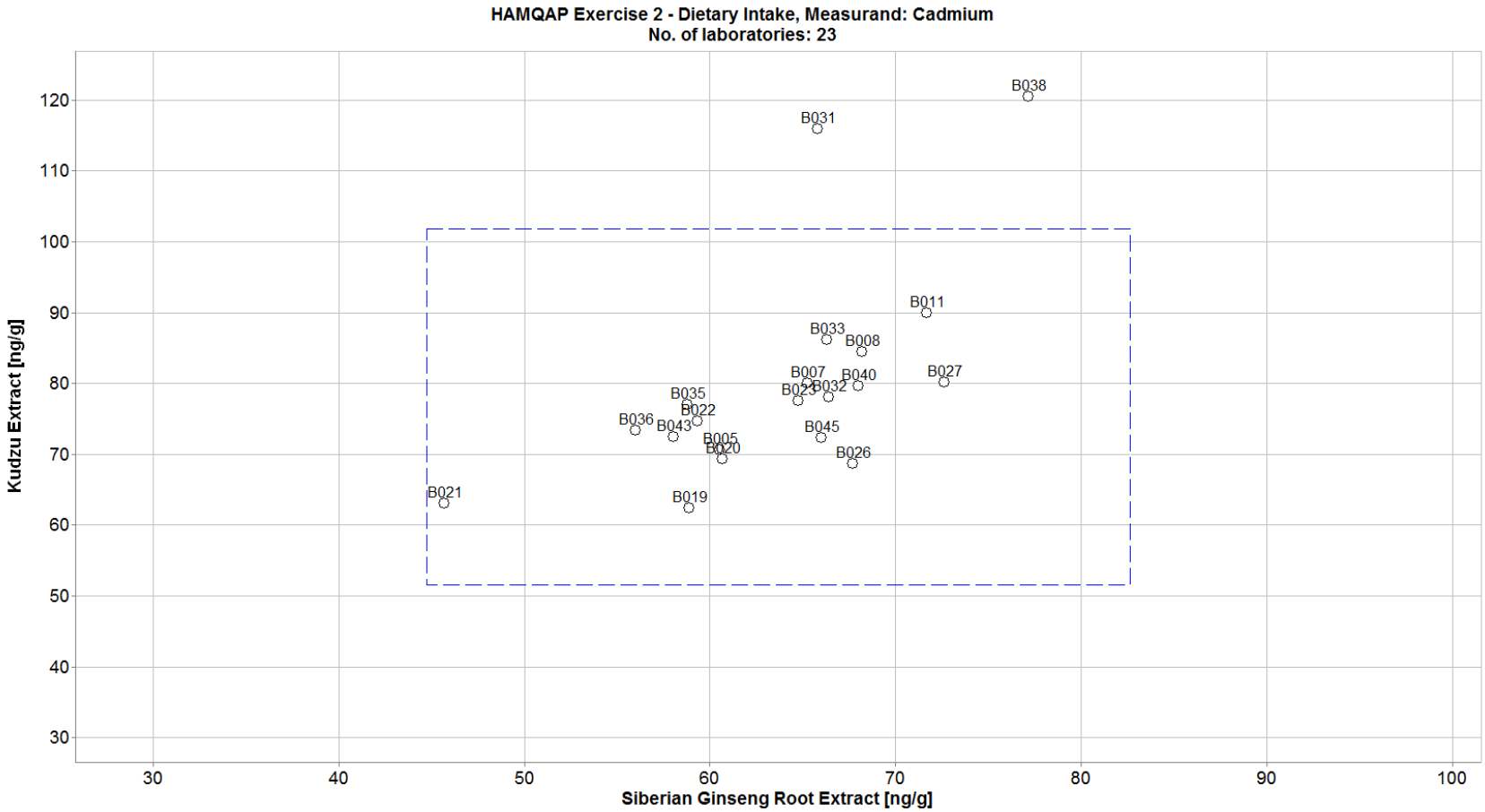

Figure 2-10. Laboratory means for cadmium in Siberian Ginseng Root Extract and Kudzu Extract (sample/sample comparison view). In this view, the individual laboratory mean for one sample (Siberian Ginseng Root Extract) is compared to the mean for a second sample (Kudzu Extract). The dotted blue box represents the consensus range of tolerance for Siberian Ginseng Root Extract (x-axis) and Kudzu 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 Siberian ginseng extract and kudzu extract. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		Lead									
		Siberian Ginseng Root Extract (ng/g)					Kudzu Extract (ng/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target									1040	790
	B001										
	B002										
	B003	0.889	0.884	0.889	0.887	0.003					
	B005	933	958	961	951	15	1220	1206	1154	1193	35
	B007	956	971	930	952	21	1908	1005	967	1293	533
	B008	832	830	853	838	13	928	892	1070	963	94
	B011	843	849	861	851	9	1037	962	841	947	99
	B012										
	B013										
	B019	889	857	755	834	70	655	1561	971	1062	460
	B020	850	850	840	847	6	1550	1240	1480	1423	163
	B021	801	842	846	830	25	1164	1099	1123	1129	33
	B022	890	880	870	880	10	1600	4900	1900	2800	1825
	B023	869	887	858	871	15	840	911	787	846	62
	B024	840	820	800	820	20	2600	1060	810	1490	969
	B025										
	B026	872	863	877	871	7	1467	1462	1480	1470	9
	B027	911	761	751	807	90	1590	903	870	1121	407
	B028										
	B029										
	B031	797	783	789	790	7	1140	1119	2101	1453	561
	B032	797	818	825	814	14	1319	776	6200	2765	2987
	B033	814	812	836	821	13	997	920	948	955	39
	B034										
	B035	823	833	844	833	10	931	969	1064	988	69
	B036	769	764	762	765	4	858	687	1962	1169	692
	B037										
	B038	1074	999	970	1014	54	3304	1123	1343	1923	1201
	B039										
	B040	837	837	833	836	2	2430	1240	1450	1707	635
	B042	608	739	791	713	94	1118	1208	1514	1280	208
	B043	859	854	862	858	4	900	846	920	889	38
	B044										
	B045	860	870	880	870	10	881	930	1010	940	65
	B046	784	696	758	746	45					
Community Results		Consensus Mean			841		Consensus Mean			1198	
		Consensus Standard Deviation			66		Consensus Standard Deviation			320	
		Maximum			1014		Maximum			2800	
		Minimum			0.89		Minimum			846	
		N			24		N			22	

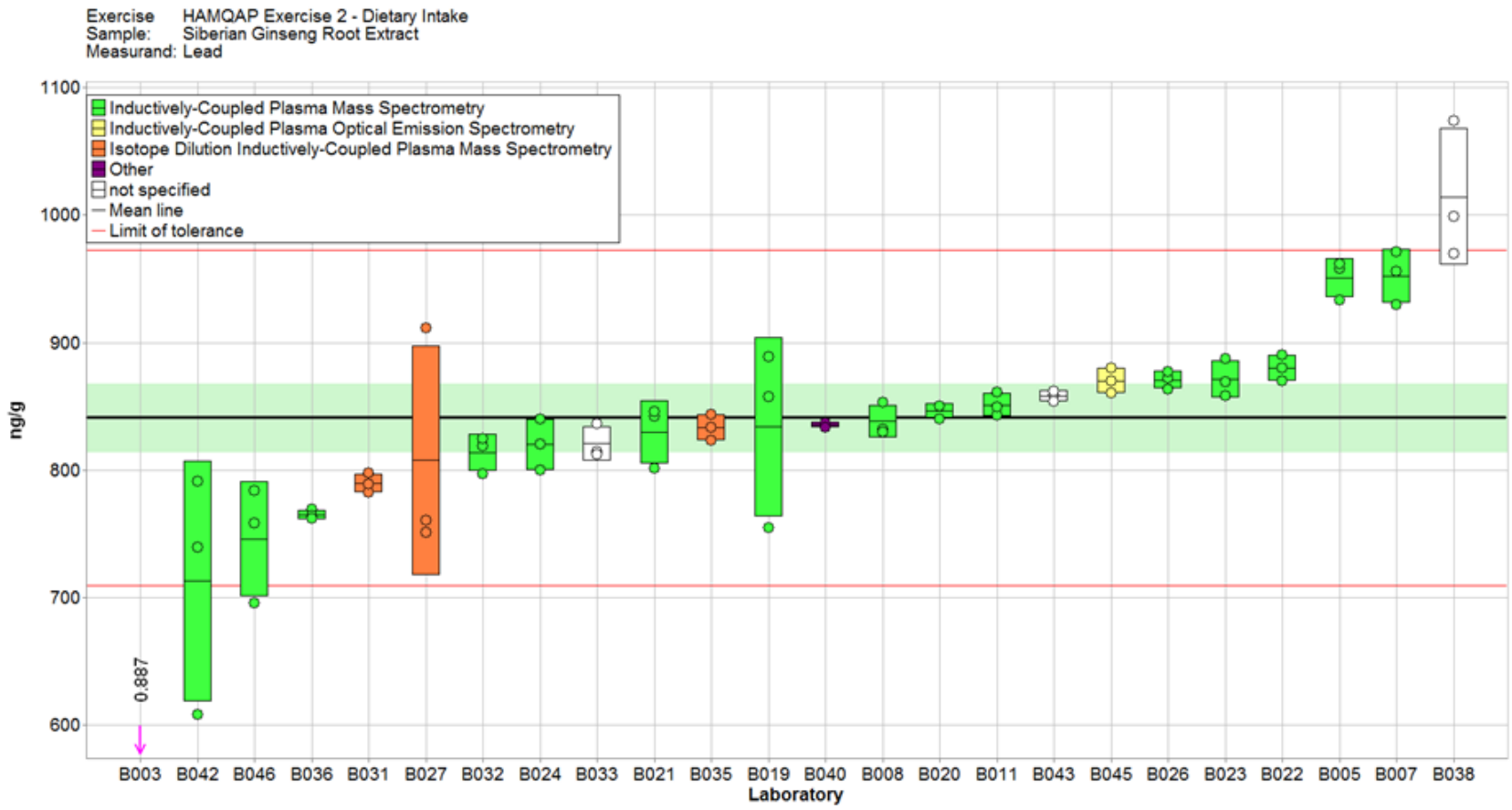

Figure 2-11. Lead in Siberian Ginseng Root Extract (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black 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 NIST value has not been determined in this material.

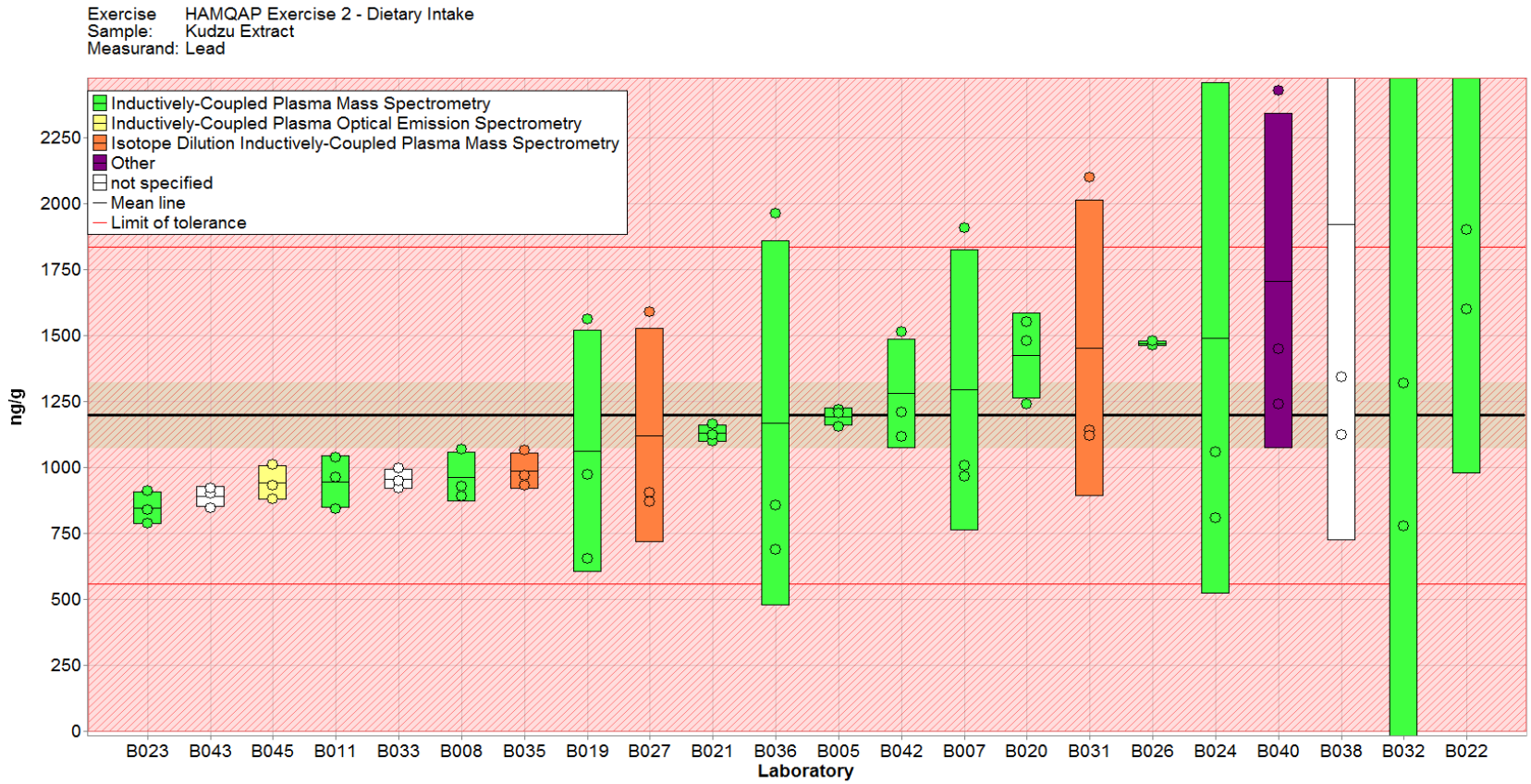

Figure 2-12. Lead in Kudzu Extract (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

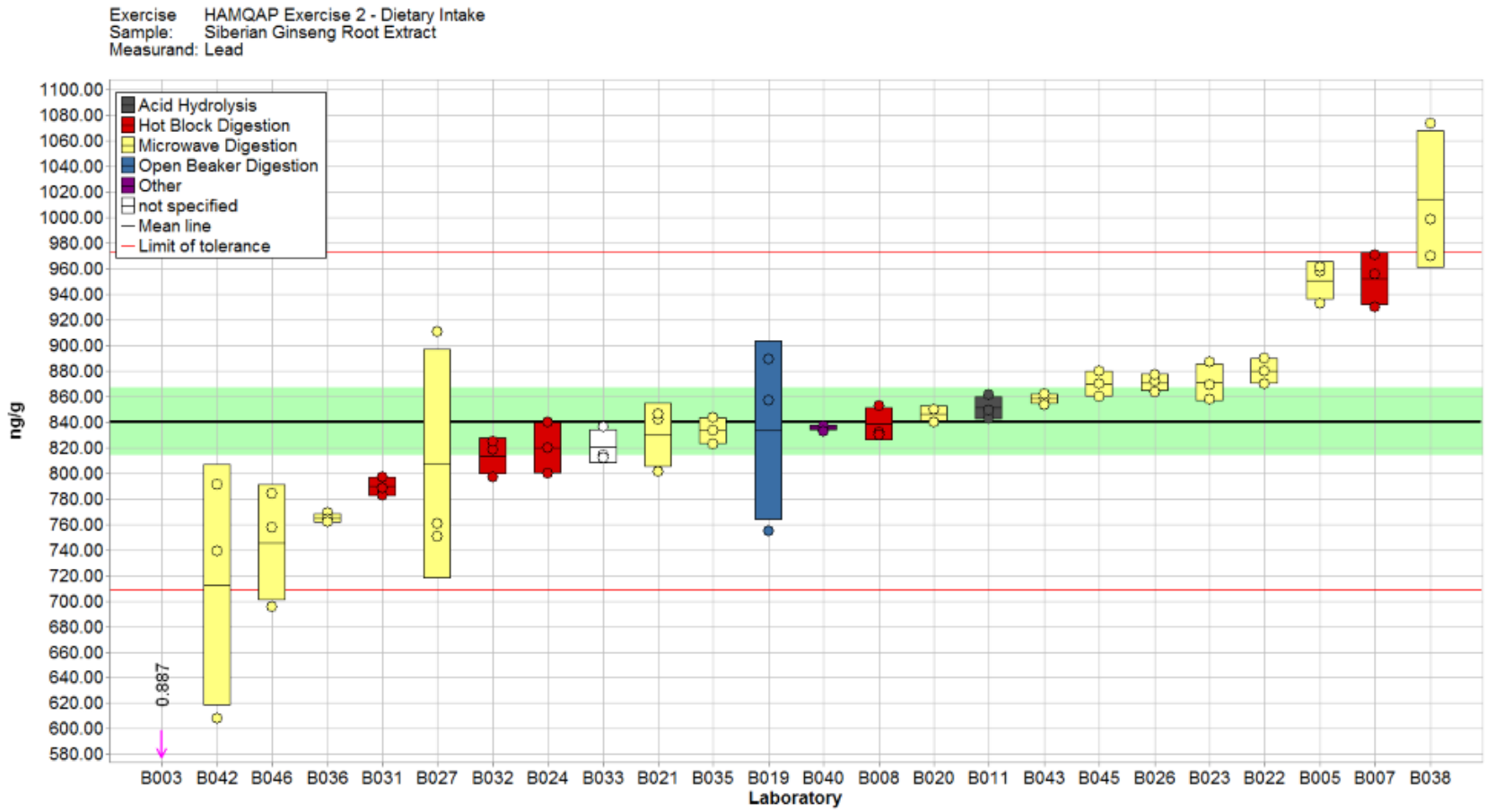

Figure 2-13. Lead in Siberian Ginseng Root Extract (data summary view – sample preparation method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the sample preparation method employed. The solid black 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 NIST value has not been determined in this material.

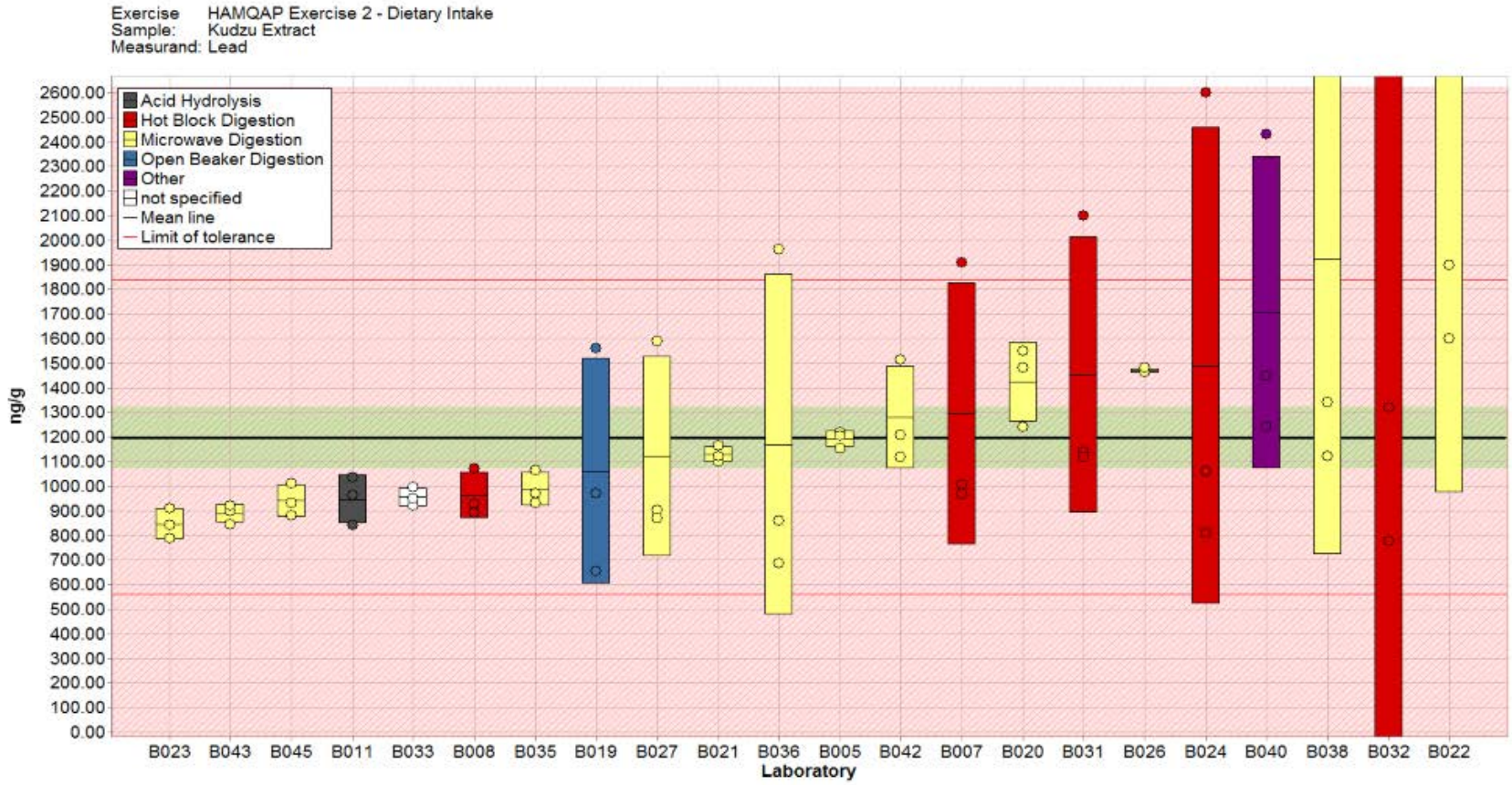

Figure 2-14. Lead in Kudzu Extract (data summary view – sample preparation method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the sample preparation method employed. The solid black 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

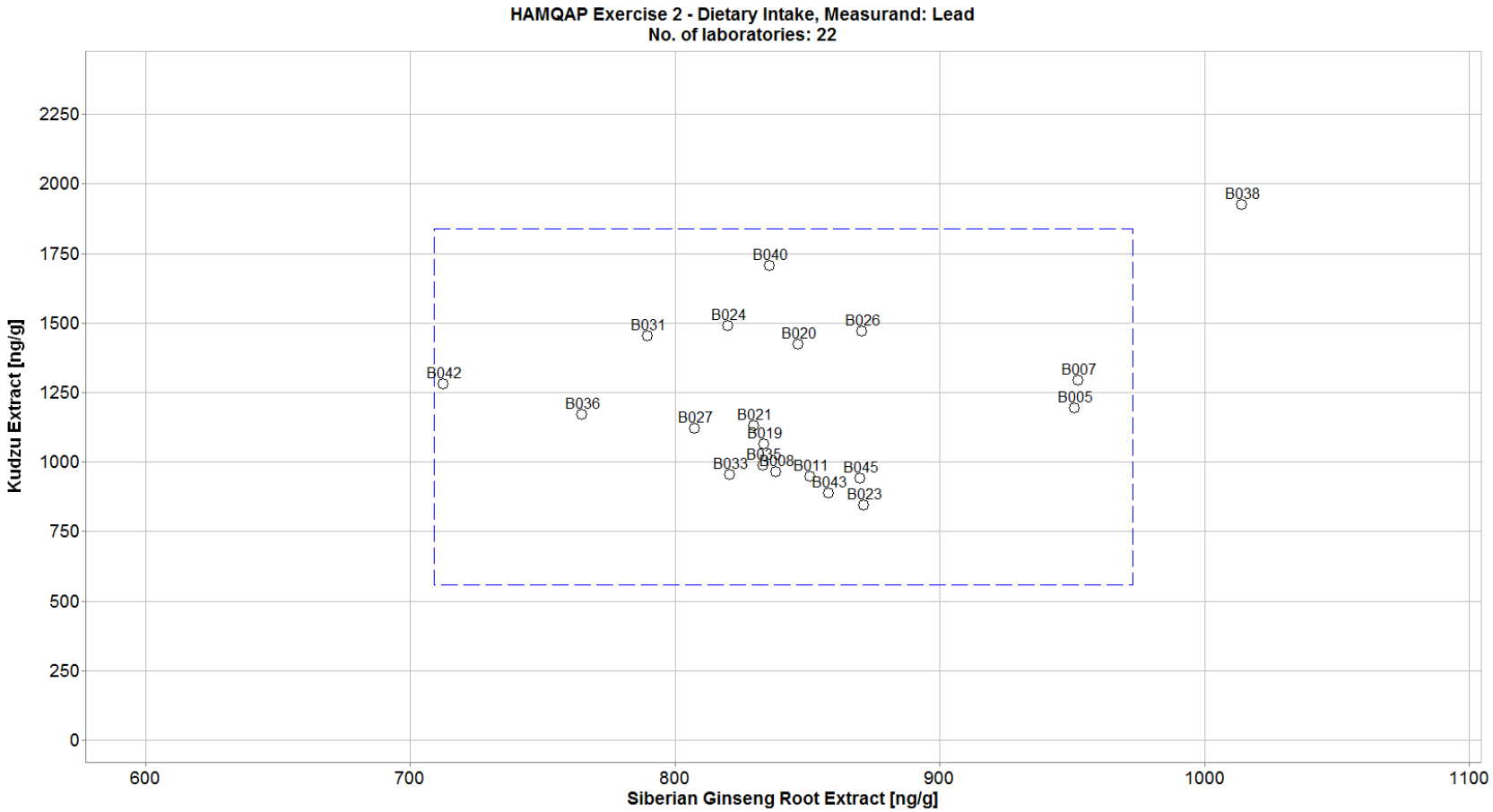

Figure 2-15. Laboratory means for lead in Siberian Ginseng Root Extract and Kudzu Extract (sample/sample comparison view). In this view, the individual laboratory mean for one sample (Siberian Ginseng Root Extract) is compared to the mean for a second sample (Kudzu Extract). The dotted blue box represents the consensus range of tolerance for Siberian Ginseng Root Extract (x-axis) and Kudzu 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 Siberian ginseng extract and kudzu extract. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		Mercury									
		Siberian Ginseng Root Extract (ng/g)					Kudzu Extract (ng/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				397	104				10	5
	B001										
	B002										
	B003										
	B005	437	409	439	428	17	8	11	10	10	2
	B007	346	318	418	360	52	27	23	20	23	3
	B008	238	141	379	253	120	< 19.8	< 19.8	< 19.8		
	B011	212	201	249	221	25	14	15	16	15	1
	B013										
	B019	19.8	24.9	25.4	23.4	3.1	7.6	11.2	12.3	10.4	2.5
	B020	320	430	560	437	120	20	10	10	13	6
	B021	300	353	210	288	72	8.80	8.10	8.20	8.37	0.38
	B022	260	360	330	317	51	4.1	7.6	5.3	5.7	1.8
	B023	341	359	330	343	15	5.80	5.62	5.57	5.66	0.12
	B024	280	318	186	261	68	7.3	9.5	4.6	7.1	2.5
	B025										
	B026	428	529	431	463	58	21.8	17.0	12.0	16.9	4.9
	B027	438	271	277	329	95	4.5	2.3	0.5	2.4	2.0
	B029										
	B031	195	210	197	201	8	21.71	22.10	20.32	21.38	0.94
	B032	442	468	704	538	144	6.60	6.52	5.94	6.35	0.36
	B033										
	B034										
	B035	244	376	272	297	70	<20	<20	<20		
	B036	284	373	534	397	127	7.00	6.00	6.00	6.33	0.58
	B037										
	B038	1204	1302	237	914	589	13.4	4.6	11.1	9.7	4.6
	B039										
	B040	237	274	258	256	19	<15	<15	13	13	
	B042	256	197	247	233	32	6.6	3.4	6.4	5.5	1.8
	B043	236	333	297	289	49	23.87	25.57	24.40	24.61	0.87
	B044										
	B045	<20	<20	<20			<20	<20	<20		
	B046										
Community Results		Consensus Mean			316		Consensus Mean			10.9	
		Consensus Standard Deviation			130		Consensus Standard Deviation			6.2	
		Maximum			914		Maximum			24.6	
		Minimum			23		Minimum			2.4	
		N			20		N			17	

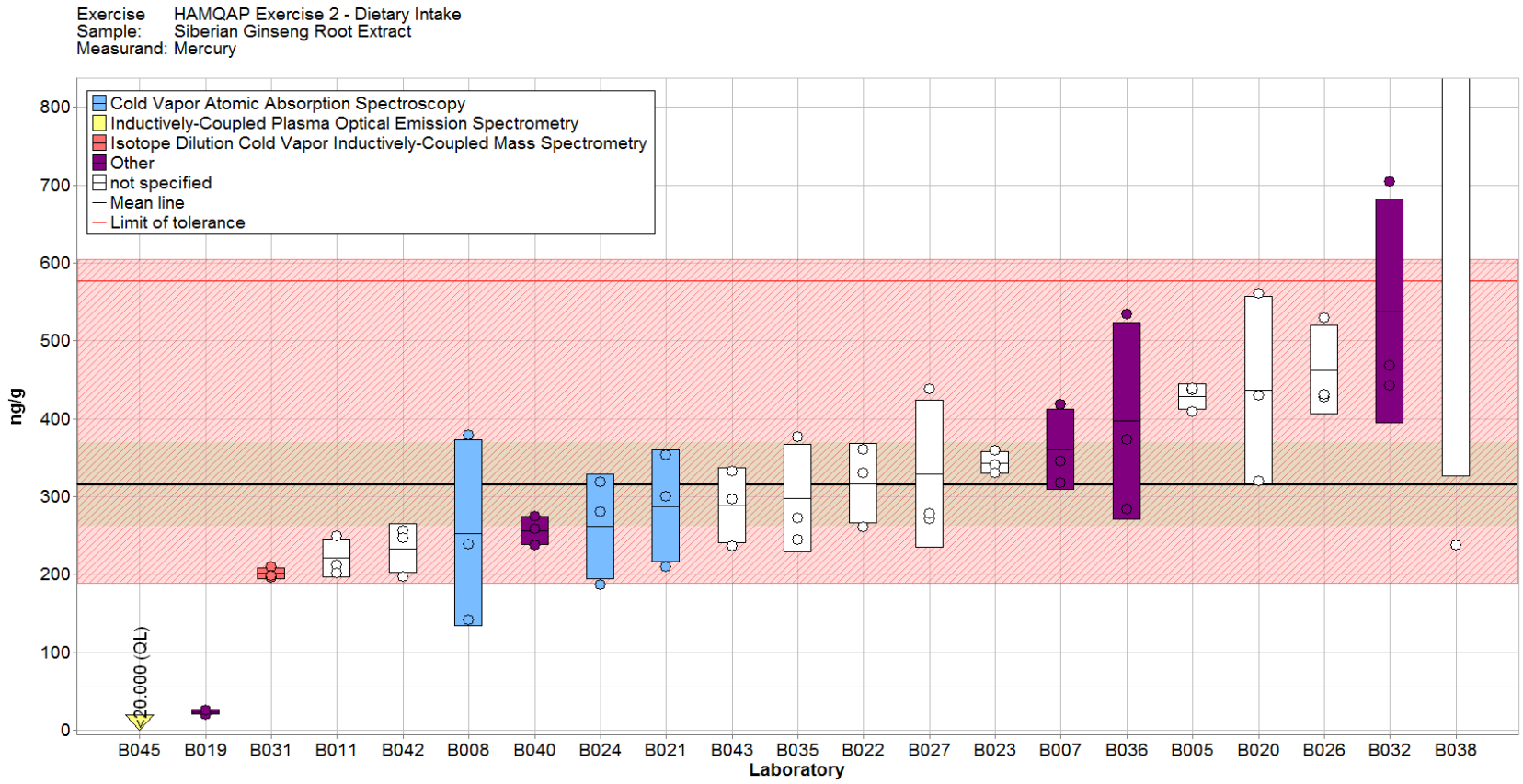

Figure 2-16. Mercury in Siberian Ginseng Root Extract (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

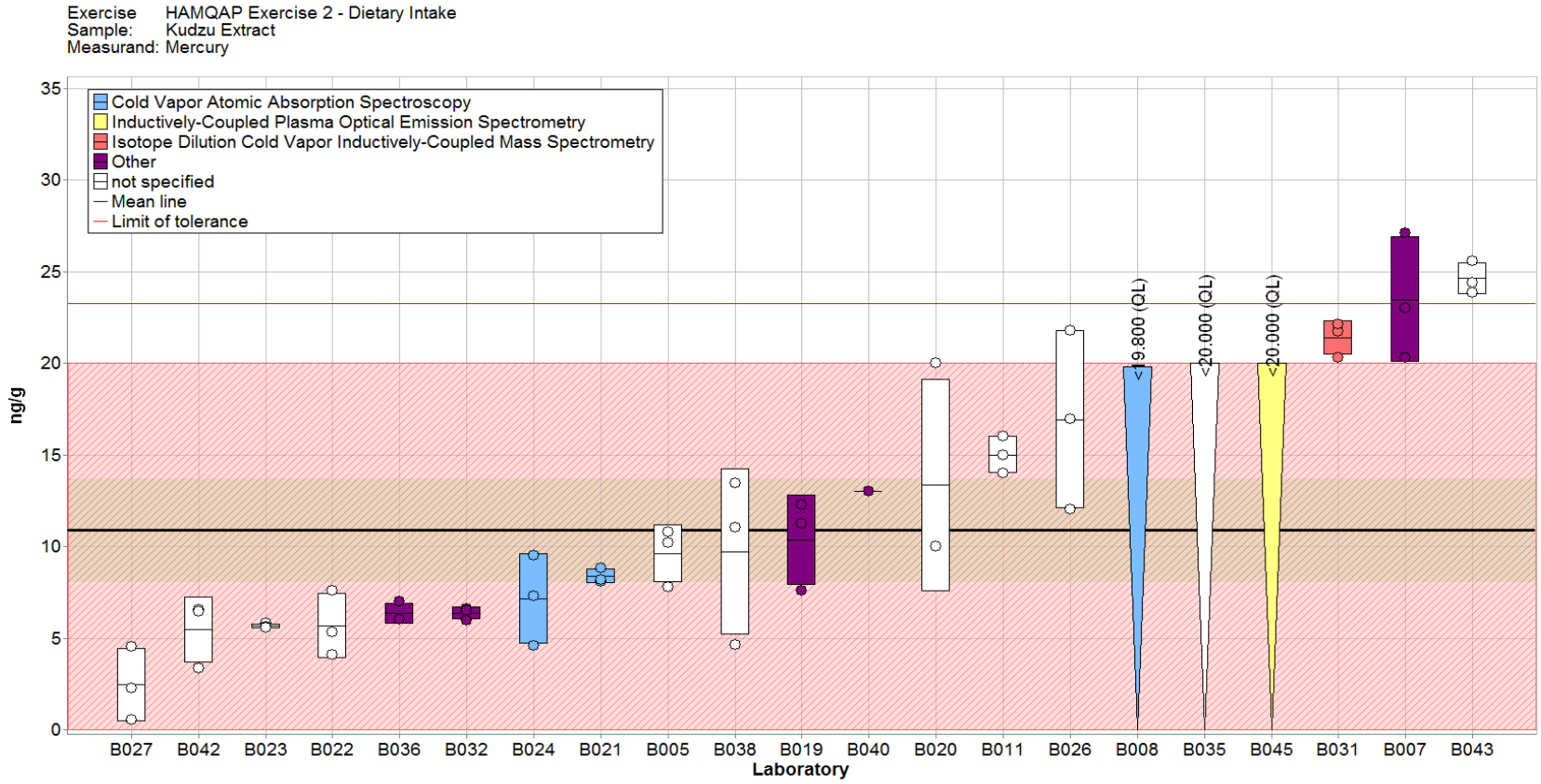

Figure 2-17. Mercury in Kudzu Extract (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

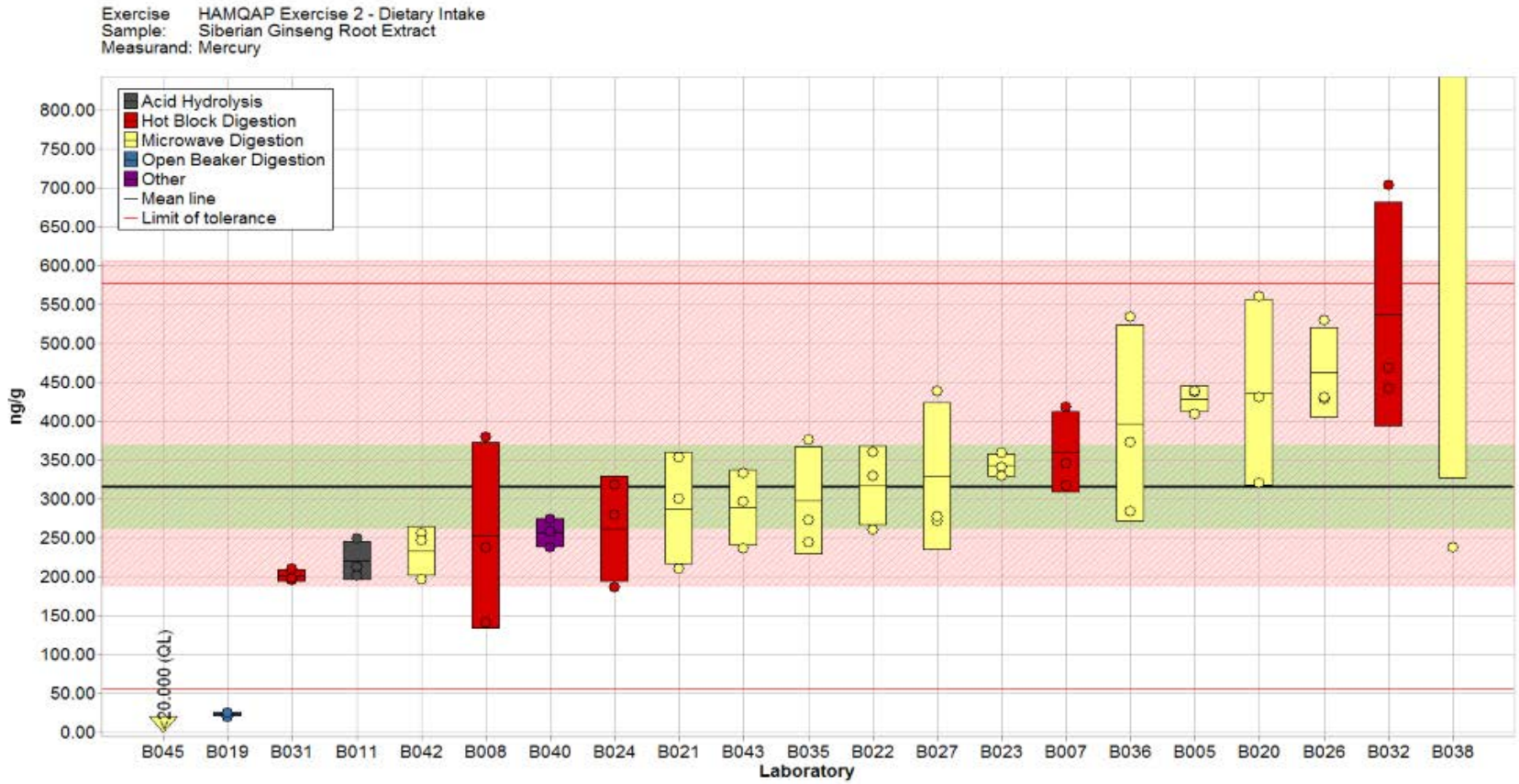

Figure 2-18. Mercury in Siberian Ginseng Root Extract (data summary view – sample preparation method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the sample preparation method employed. The solid black 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

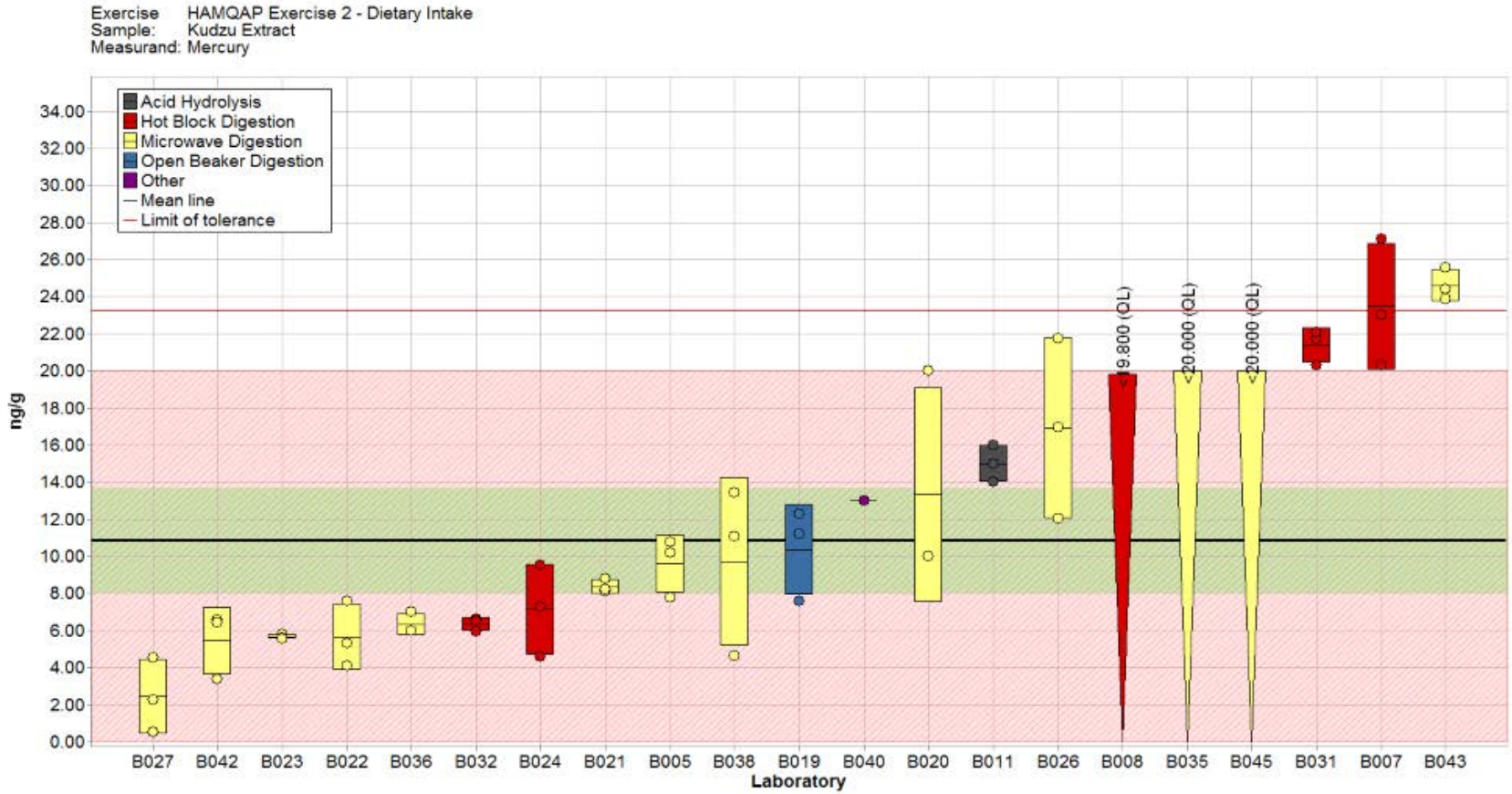

Figure 2-19. Mercury in Kudzu Extract (data summary view – sample preparation method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the sample preparation method employed. The solid black 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

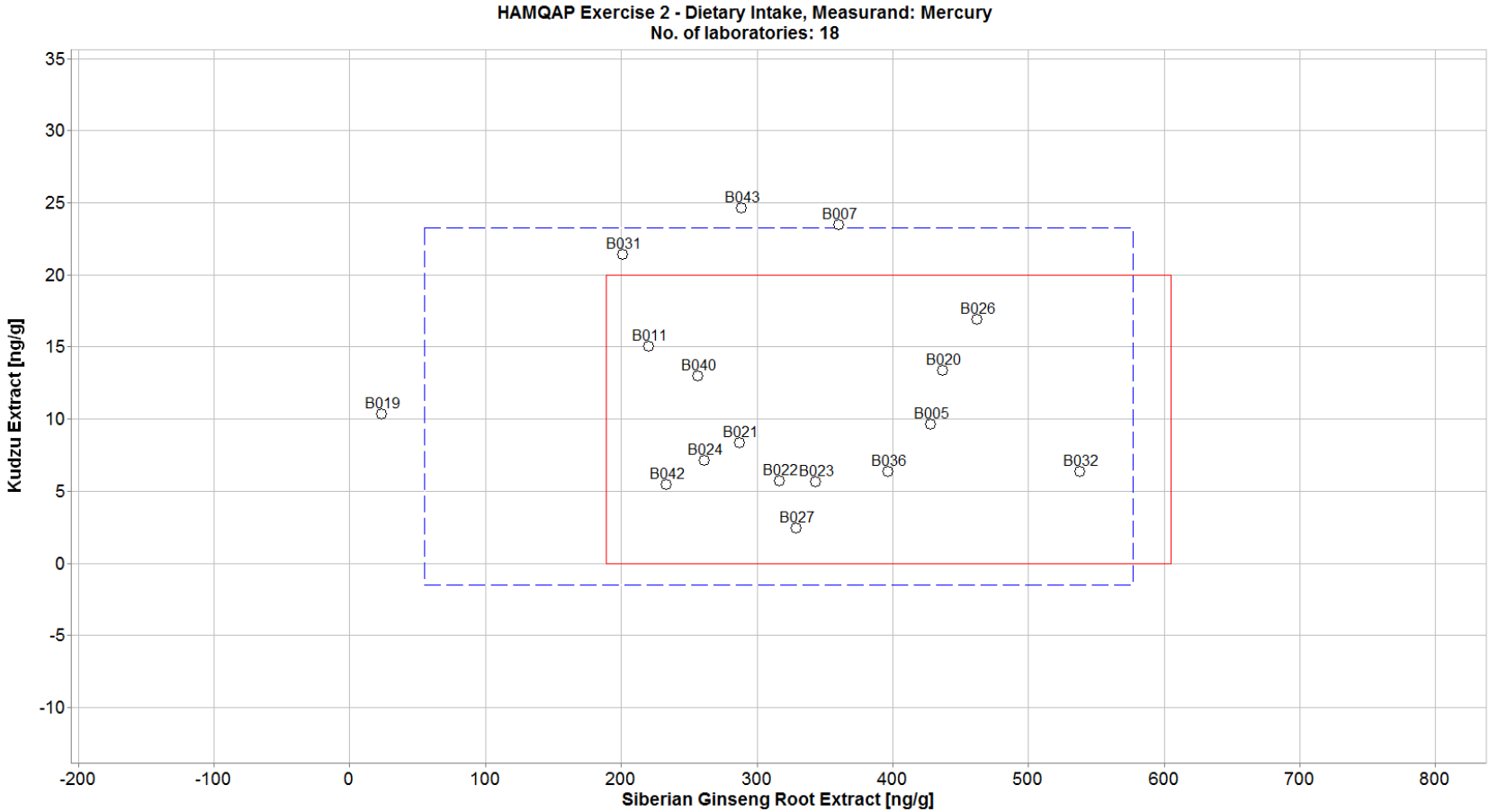

Figure 2-20. Laboratory means for mercury in Siberian Ginseng Root Extract and Kudzu Extract (sample/sample comparison view). In this view, the individual laboratory mean for one sample (Siberian Ginseng Root Extract) is compared to the mean for a second sample (Kudzu Extract). The solid red box represents the NIST range of tolerance for the two samples, Siberian Ginseng Root Extract (x-axis) and Kudzu Extract (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 Siberian Ginseng Root Extract (x-axis) and Kudzu Extract (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 2-6. Data summary table for selenium in Siberian ginseng extract and kudzu extract. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		Selenium									
		Siberian Ginseng Root Extract (ng/g)					Kudzu Extract (ng/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target									117	5
	B003	0.012	0.009	0.001	0.007	0.006	0.160	0.144	0.149	0.151	0.008
	B005	82	60	57	66	14	174	167	149	163	12
	B007	268	277	238	261	20	428	447	461	445	17
	B008	< 989	< 989	< 989			< 992	< 992	< 992		
	B011	113	85	134	111	25	212	358	149	240	107
	B013										
	B019	172	134	144	150	20	259	315	321	298	34
	B020	41.0	38.0	43.0	40.7	2.5	105	116	104	108	7
	B021	65	73	59	66	7	152	120	163	145	22
	B022	43.0	41.0	43.0	42.3	1.2	130	130	140	133	6
	B023	44.5	41.1	43.4	43.0	1.7	114	109	112	112	3
	B024	240	200	170	203	35	220	230	290	247	38
	B025										
	B026	77	68	70	72	5	298	290	278	289	10
	B027	40	50	45	45	5	133	138	128	133	5
	B028										
	B029										
	B031	225	261	271	252	24	229	289	305	274	40
	B032	90	52	99	80	25	176	180	199	185	12
	B033	134.1	132.9	131.3	132.8	1.4	426	397	484	436	44
	B034										
	B035	38	41	40	39	2	106	118	115	113	6
	B036	0	0	0	0	0	110	109	129	116	11
	B039										
	B040	110	140	120	123	15	250	240	200	230	26
	B042						259	276	288	274	14
	B043										
	B044										
	B045	116	110	115	114	3	222	228	203	218	13
	B046										
Community Results		Consensus Mean			91		Consensus Mean			202	
		Consensus Standard Deviation			73		Consensus Standard Deviation			106	
		Maximum			261		Maximum			445	
		Minimum			0		Minimum			0.15	
		N			19		N			20	

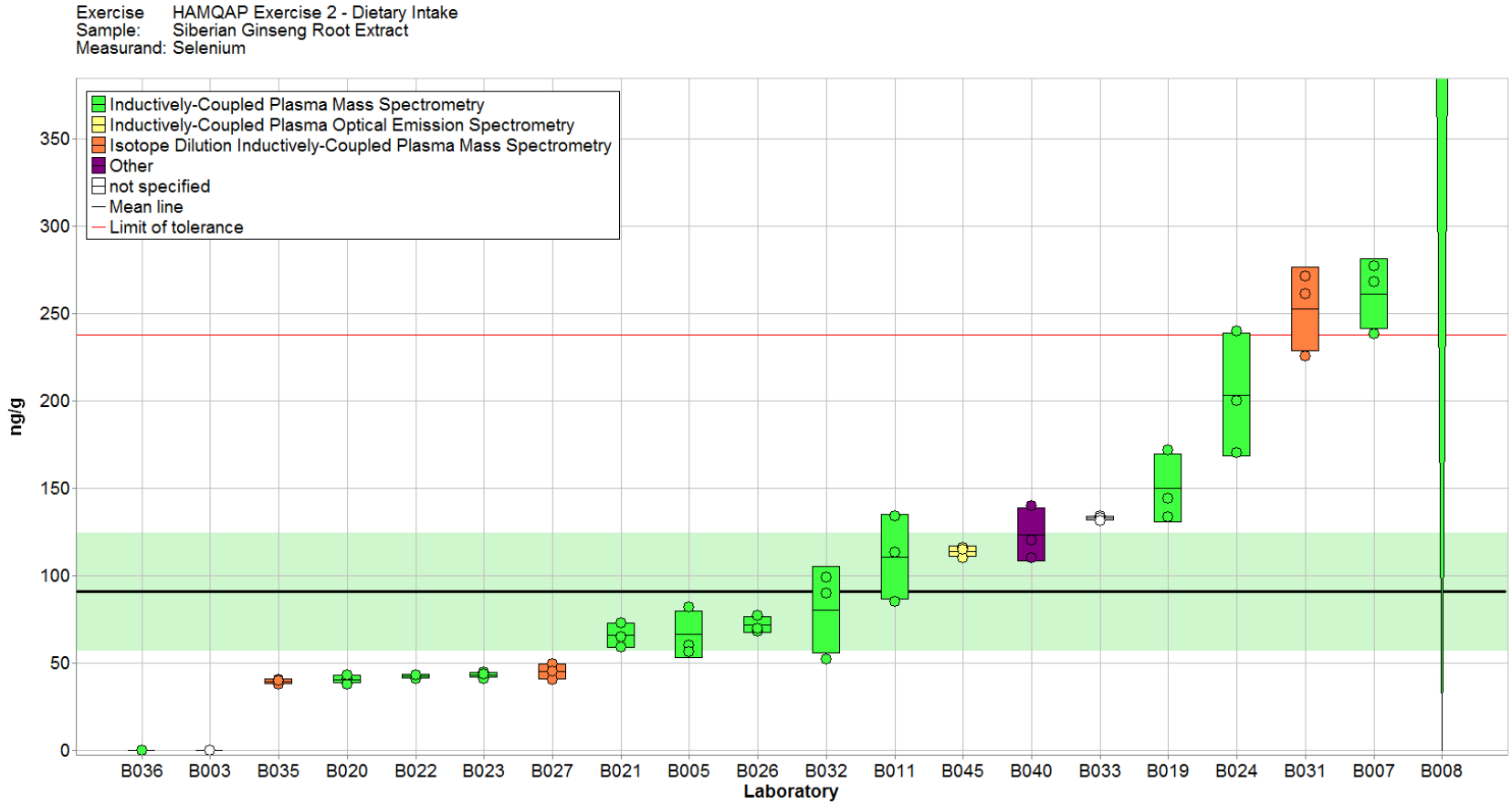

Figure 2-21. Selenium in Siberian Ginseng Root Extract (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black 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 NIST value has not been determined in this material.

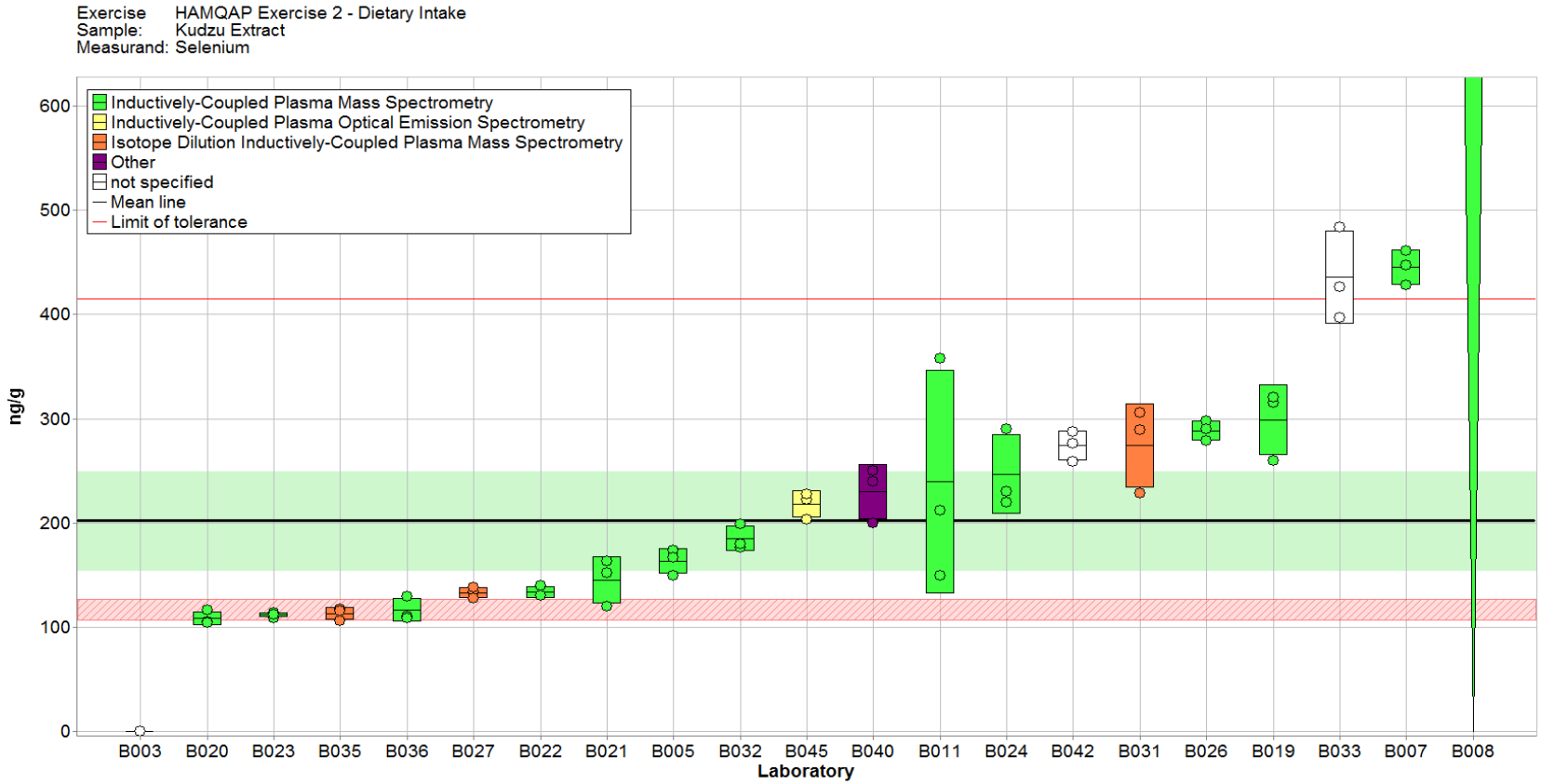

Figure 2-22. Selenium in Kudzu Extract (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

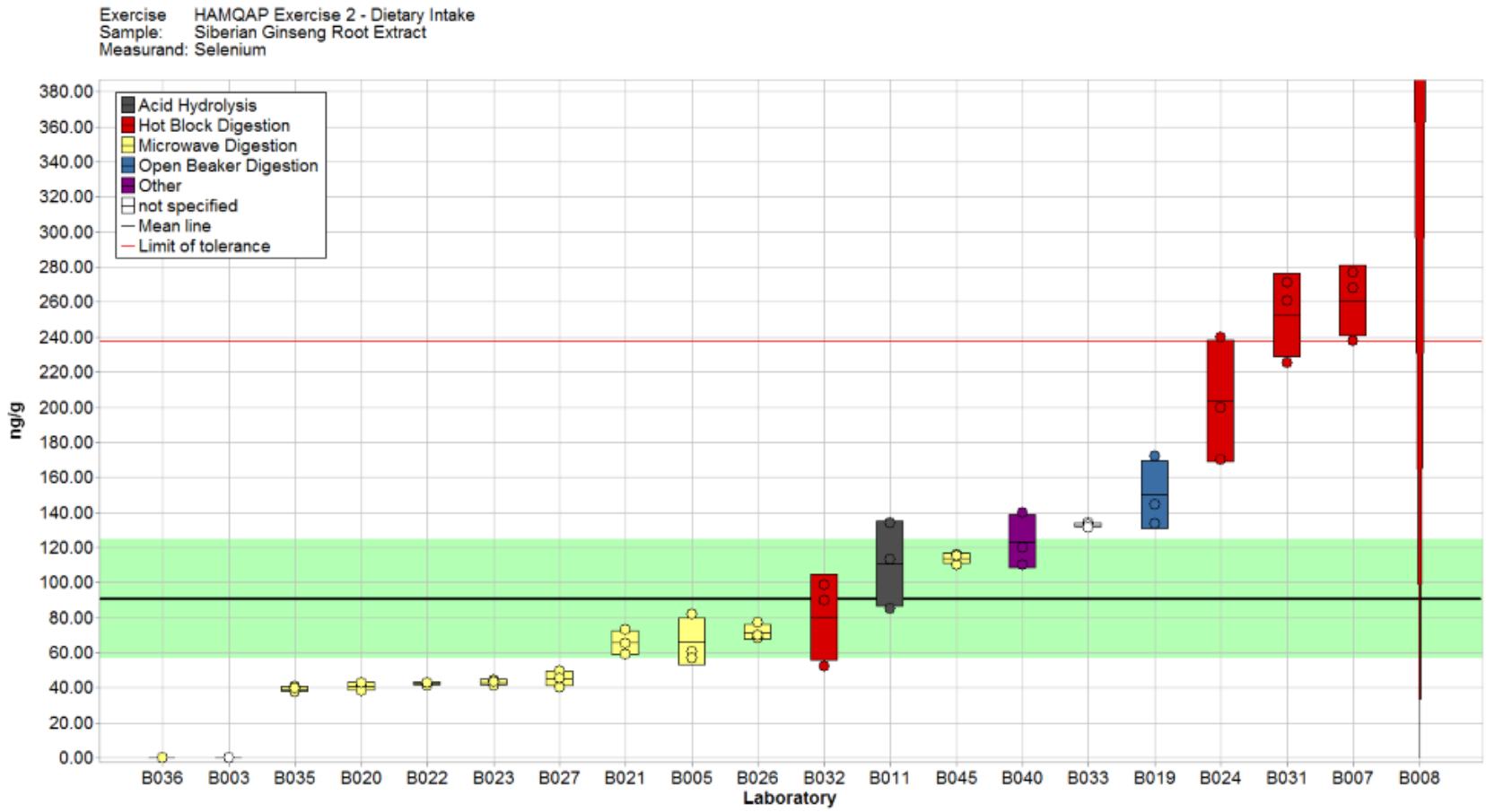

Figure 2-23. Selenium in Siberian Ginseng Root Extract (data summary view – sample preparation method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the sample preparation method employed. The solid black 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 NIST value has not been determined in this material.

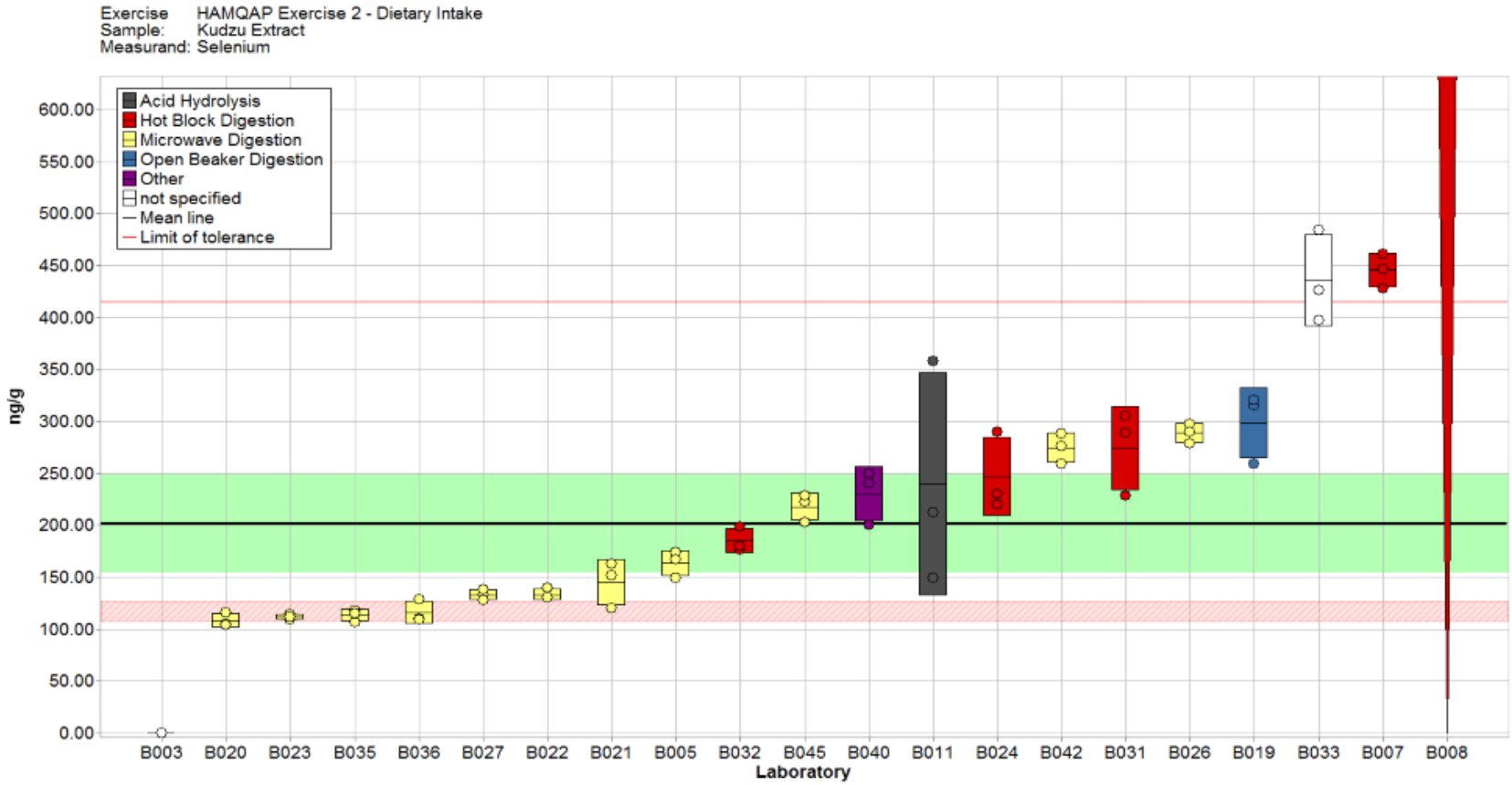

Figure 2-24. Selenium in Kudzu Extract (data summary view – sample preparation method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the sample preparation method employed. The solid black 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

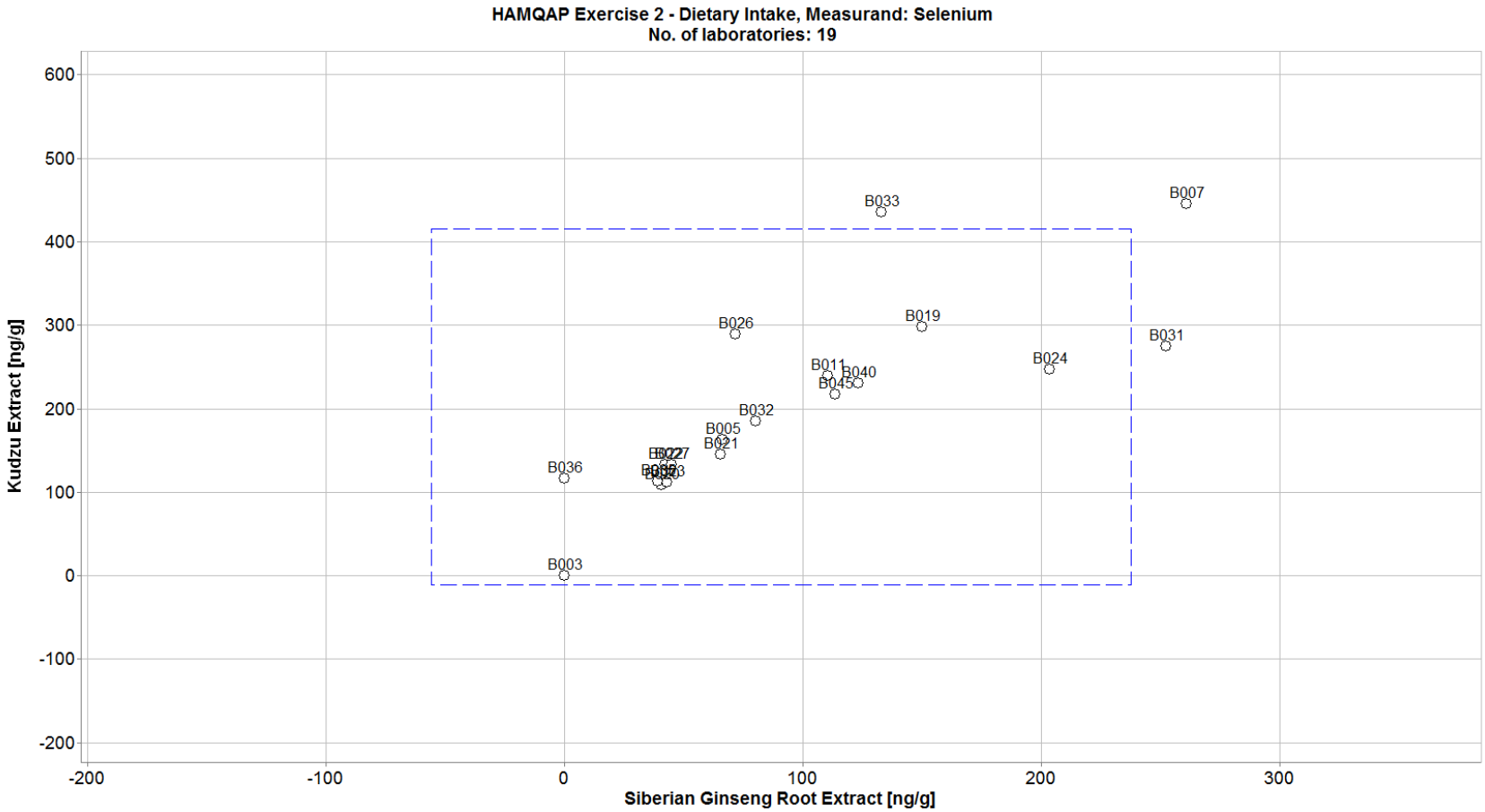

Figure 2-25. Laboratory means for selenium in Siberian Ginseng Root Extract and Kudzu Extract (sample/sample comparison view). In this view, the individual laboratory mean for one sample (Siberian Ginseng Root Extract) is compared to the mean for a second sample (Kudzu Extract). The dotted blue box represents the consensus range of tolerance for Siberian Ginseng Root Extract (x-axis) and Kudzu Extract (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

SECTION 3: WATER-SOLUBLE VITAMINS (Folates)

Study Overview

In this study, participants were provided with two NIST SRMs for dietary intake, SRM 1845a Whole Egg Powder and SRM 1546a Meat Homogenate. Participants were asked to use in-house analytical methods to determine the mass fraction (mg/kg) of total folate and/or the individual folate vitamers (folic acid, 5-methyltetrahydrofolate, 5-formyltetrahydrofolate, tetrahydrofolate, and 5,10-methenyltetrahydrofolate) in each matrix. Folate is an essential vitamin, critical for the production and maintenance of new cells as well as synthesis of DNA and RNA, and adequate folate intake during pregnancy is important for the prevention of neural tube defects.[4] Naturally occurring folates in food are in the tetrahydrofolate form, and humans obtain folic acid through via fortified foods and supplements, and other forms of folate occur naturally in some foods. Folate health status is evaluated through determination of folate metabolites in serum.

Dietary Intake Sample Information

Egg Powder. Participants were provided with three packets, each containing approximately 10 g of egg powder. Before use, participants were instructed to allow the packets to warm to room temperature, mix the contents thoroughly, and allow the contents to settle for one minute prior to opening to minimize the loss of fine particles. Participants were instructed to use a sample size appropriate for their usual in-house method of analysis. Participants were asked to store the material at 4 °C in the original unopened packets and to prepare one sample and report one value from each packet provided. The approximate analyte level was not reported to participants prior to the study. The reference value for total folate in SRM 1845a was assigned using results from collaborating laboratories. The reference value and uncertainty for total folate in SRM 1845a is provided in the table below on an as-received basis.

Analyte	Reference Mass Fraction in SRM 1845a (mg/kg)
Total folate	1.300 ± 0.069

Meat Homogenate. Participants were provided with one can containing approximately 85 g of material. Before use, participants were instructed to thoroughly mix the contents of the can; the suggested technique was to transfer the entire contents of a can to a plastic bag, then manually squeeze the bag to blend the material, taking care to avoid separating fat from the material to preserve homogeneity. Participants were instructed to use a sample size appropriate for their usual in-house method of analysis. Participants were asked to store the material at controlled room temperature, 10 °C to 30 °C, in the original unopened can and to prepare three samples and report three values from the single can provided. The approximate analyte level was not reported to participants prior to the study. A target value for folic acid in SRM 1546a was assigned using results from collaborating laboratories. The NIST-determined value and uncertainty for folic acid in SRM 1546a is provided in the table below on an as-received basis.

Analyte	NIST-Determined Mass Fraction in SRM 1546a (mg/kg)
Folic acid	0.125 ± 0.166

[4] Folate Fact Sheet for Health Professionals. National Institutes of Health Office of Dietary Supplements. https://ods.od.nih.gov/factsheets/folate-healthprofessional/ (accessed February 20, 2019).

Dietary Intake Study Results

- Twenty-two laboratories enrolled in this exercise and received samples to measure folic acid. Six laboratories reported results for the egg powder (27 % participation) and five laboratories reported results for meat homogenate (23 % participation).
- For egg powder, the between-laboratory variability for folic acid measurements was high (100 % RSD) (**Table 3-2, Figure 3-1**). No target range was available for folic acid in egg powder. Two laboratories reported using liquid chromatography with absorbance detection (LC-absorbance), two laboratories reported using liquid chromatography with mass spectrometry (LC-MS), one laboratory reported using liquid chromatography with tandem mass spectrometry (LC-MS/MS), and one laboratory did not report the measurement method.
- For meat homogenate, the consensus mean for folic acid was significantly above the target range and the between-laboratory variability was very high (300 % RSD) (**Table 3-2, Figure 3-2**). Two laboratories reported using LC-absorbance, one laboratory reported using LC-MS, one laboratory reported using LC-MS/MS, and one laboratory did not report the measurement method.
- Most laboratories that reported values for folic acid in both materials were within the consensus range of tolerance, which is quite wide given the small number of laboratories reporting results (**Figure 3-3**).
- Twelve laboratories enrolled in this exercise and received samples to measure total folate. Two laboratories reported results for both egg powder and meat homogenate (17 % participation), including one laboratory reporting 0 mg/kg total folate for both materials (**Table 3-3, Figure 3-4, Figure 3-5**). For both materials, one laboratory reported using LC-MS and one laboratory reported using LC-MS/MS.
- Fourteen laboratories enrolled in this exercise and received samples to measure 5-methyltetrahydrofolate (5-mTHF). Two laboratories reported results for both egg powder and meat homogenate (14 % participation).
  - The between-laboratory variability was very high for 5-mTHF in egg powder (420 % RSD). No target range was available for 5-mTHF in egg powder. Both laboratories that submitted results for meat homogenate reported 0 mg/kg 5-mTHF.
  - For both egg powder and meat homogenate, one laboratory reported using LC-absorbance and one laboratory reported using LC-MS/MS.
- For 5-formyltetrahydrofolate and 5,10-methenyltetrahydrofolate, nine laboratories enrolled in this exercise and received samples. However, only one laboratory reported results (11 % participation).
- For tetrahydrofolate, eleven laboratories enrolled in this exercise and received samples. Both laboratories that submitted results reported 0 mg/kg for both materials (18 % participation).

Dietary Intake Technical Recommendations

The following recommendations are based on results obtained from the participants in this study and are focused on the methods reported by those participants (liquid chromatography separation with either absorbance or mass spectrometry-based detection).

- Both materials used in this exercise, egg powder and meat homogenate, are unfortified food matrices. Unfortified foods from animal sources are expected to contain endogenous folates (i.e., 5-mTHF), but are not expected to have detectable levels of synthetic folic acid.
- Given that many participants reported values for folic acid, some participants may be measuring other folate forms and reporting them as "folic acid." The NIST-determined value for folic acid in meat homogenate is based on collaborator data which may also be "total folate" reported as folic acid.
- The very large between-laboratory variabilities for folic acid (100 % RSD and 300 % RSD for egg powder and meat homogenate, respectively) indicate that measuring folic acid in unfortified food matrices is clearly a challenge (**Figure 3-1, Figure 3-2**).
- The use of liquid chromatographic approaches provides the opportunity for participating laboratories to separate most or all folate vitamers prior to detection. However, many laboratories are reporting folic acid in these unfortified food materials, which may result from non-specific matrix interferences affecting folic acid identification and quantification.
- Folate calibration solutions that are value assigned based on UV absorbance spectrophotometry may contain significant impurities that impact quantification. Additional purity correction by LC-UV analysis of calibration solutions may resolve some biases.
- The various folates have different stabilities in solution. Solution pH or the addition of antioxidants, such as ascorbic acid, should be considered to ensure all folate calibration solutions are stable throughout the duration of the sample analysis.
- The release of endogenous folates typically requires complete processing of the food matrix by digestion with protease, amylase, and deconjugase, especially for analysis by mass spectrometry methods. Incomplete digestion will lead to biased results. Trienzyme digestion optimization may be required for complete release and quantification of folates.
- Use of matrix-matched CRMs for method validation and quality assurance of the measurement process is recommended.
- Next steps may include quantitative analysis of folates in a defined solution to discriminate between variation from separation and detection of folates and variation from incomplete extraction.
- Several laboratories reported values of zero for measurements in all materials. "Zero" is not a quantity that can be measured, and therefore a more appropriate result would be to report that a value is below the MDL, LOQ, or QL.

Table 3-1. Individualized data summary table (NIST) for folates in egg powder and meat homogenate.

National Institute of Standards & Technology

HAMQAP Exercise 2 - 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*
5,10-Methenyltetrahydrofolate	SRM 1845a Whole Egg Powder	mg/kg						1				
5,10-Methenyltetrahydrofolate	SRM 1546a Meat Homogenate	mg/kg						1				
5-Formyltetrahydrofolate	SRM 1845a Whole Egg Powder	mg/kg						1				
5-Formyltetrahydrofolate	SRM 1546a Meat Homogenate	mg/kg						1				
5-Methyltetrahydrofolate	SRM 1845a Whole Egg Powder	mg/kg						2	40	190		
5-Methyltetrahydrofolate	SRM 1546a Meat Homogenate	mg/kg						2	0	0		
Folic Acid	SRM 1845a Whole Egg Powder	mg/kg						6	2	2.1		
Folic Acid	SRM 1546a Meat Homogenate	mg/kg		0.125	0.083			5	0	10	0.125	0.166
MeFox	SRM 1845a Whole Egg Powder	mg/kg						1				
MeFox	SRM 1546a Meat Homogenate	mg/kg						1				
Tetrahydrofolate	SRM 1845a Whole Egg Powder	mg/kg						2	0	0		
Tetrahydrofolate	SRM 1546a Meat Homogenate	mg/kg						2	0	0		
Total Folates	SRM 1845a Whole Egg Powder	mg/kg		1.300	0.035			2	0.23	0.87	1.300	0.069
Total Folates	SRM 1546a Meat Homogenate	mg/kg						2	70	140		

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 3-2. Data summary table for folic acid in egg powder and meat homogenate. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		Folic Acid									
		SRM 1845a Whole Egg Powder (mg/kg)					SRM 1546a Meat Homogenate (mg/kg)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target									0.13	0.17
	B001										
	B006										
	B007										
	B008										
	B013										
	B016										
	B019	8.3	8.3	9.5	8.7	0.7	5.6	4.5	5.3	5.1	0.6
	B021										
	B026										
	B027	0.19	0.14	0.09	0.14	0.05	181	42.3	21.9	81.7	86.6
	B030										
	B031	0	0	0	0	0	0	0	0	0	0
	B032										
	B033	1.1	1.0	0.9	1.0	0.1	7.6	8.4	7.9	8.0	0.4
	B035										
	B036										
	B038										
	B039										
	B042	7.71			7.71						
	B044										
	B045										
	B046	1.221	1.350	1.250	1.274	0.068	0.201	0.216	0.217	0.211	0.009
Community Results		Consensus Mean			2.1		Consensus Mean			3.3	
		Consensus Standard Deviatio			2.1		Consensus Standard Deviatio			10.0	
		Maximum			8.7		Maximum			81.7	
		Minimum			0		Minimum			0.0	
		N			5		N			5	

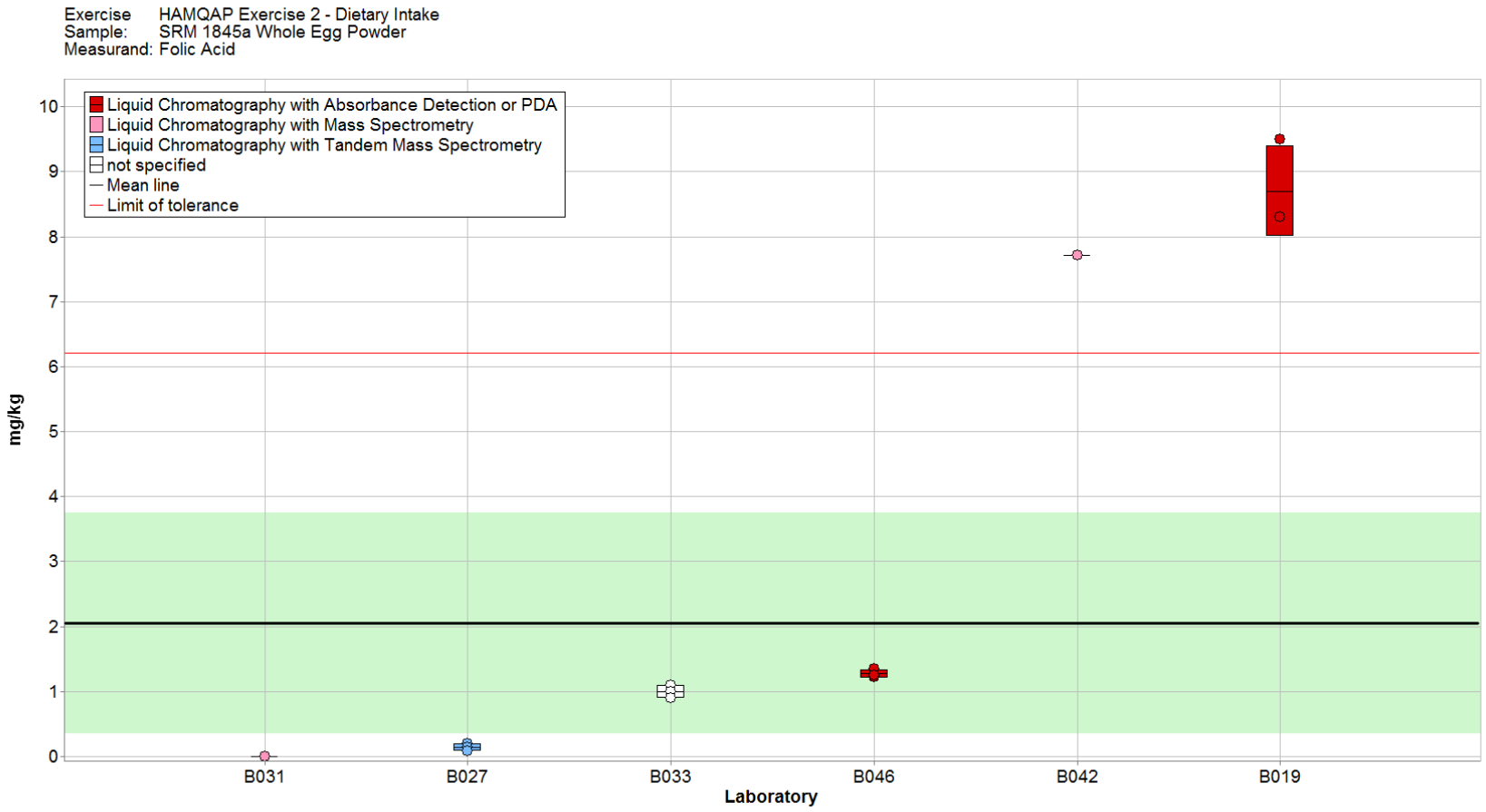

Figure 3-1. Folic acid in SRM 1845a Whole Egg Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the consensus mean bounded by twice the consensus standard error. The solid red line represents the upper 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 NIST value has not been determined in this material.

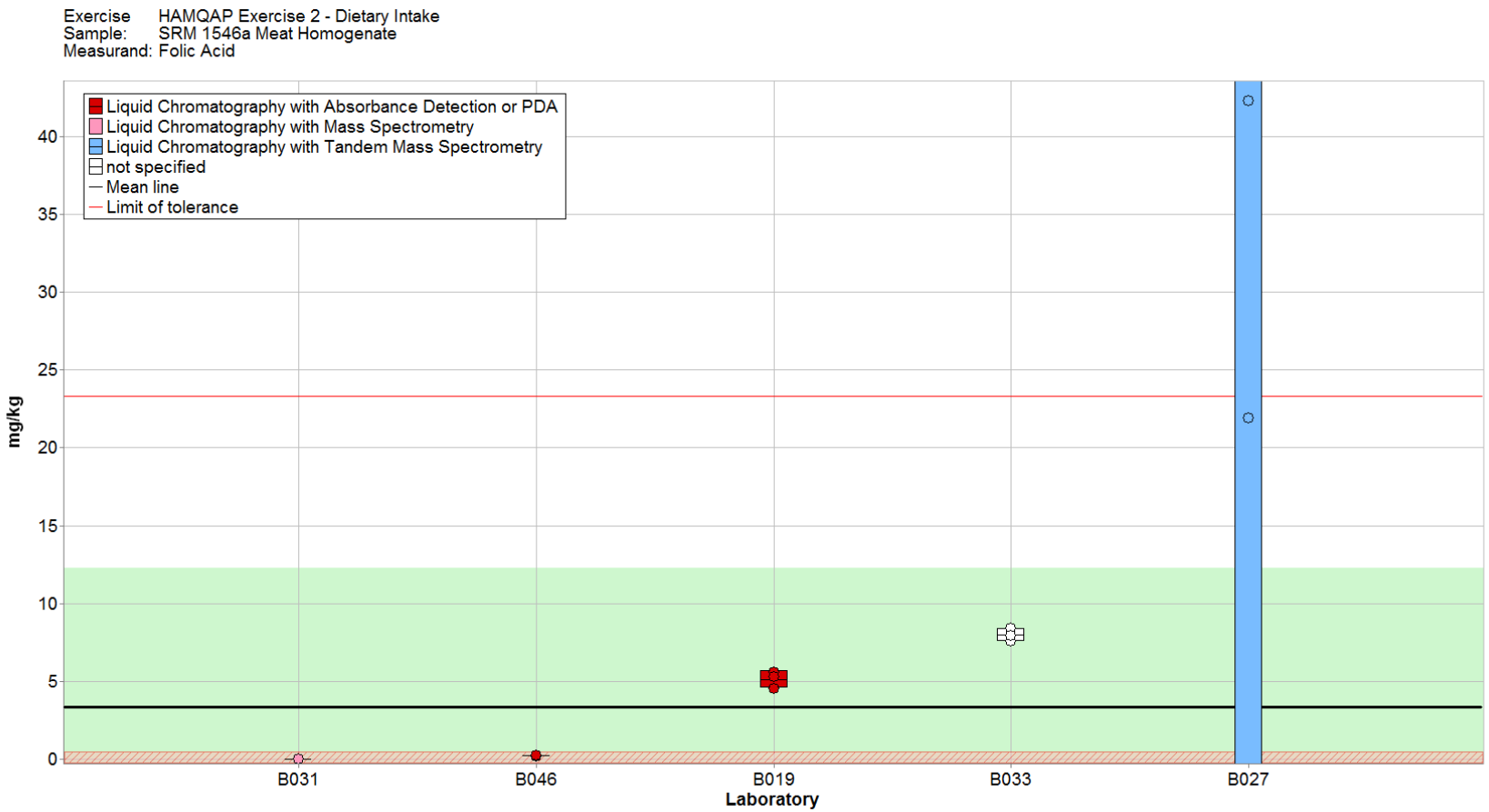

Figure 3-2. Folic acid in SRM 1546a Meat Homogenate (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the consensus mean bounded by twice the consensus standard error. The solid red line represents the upper 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

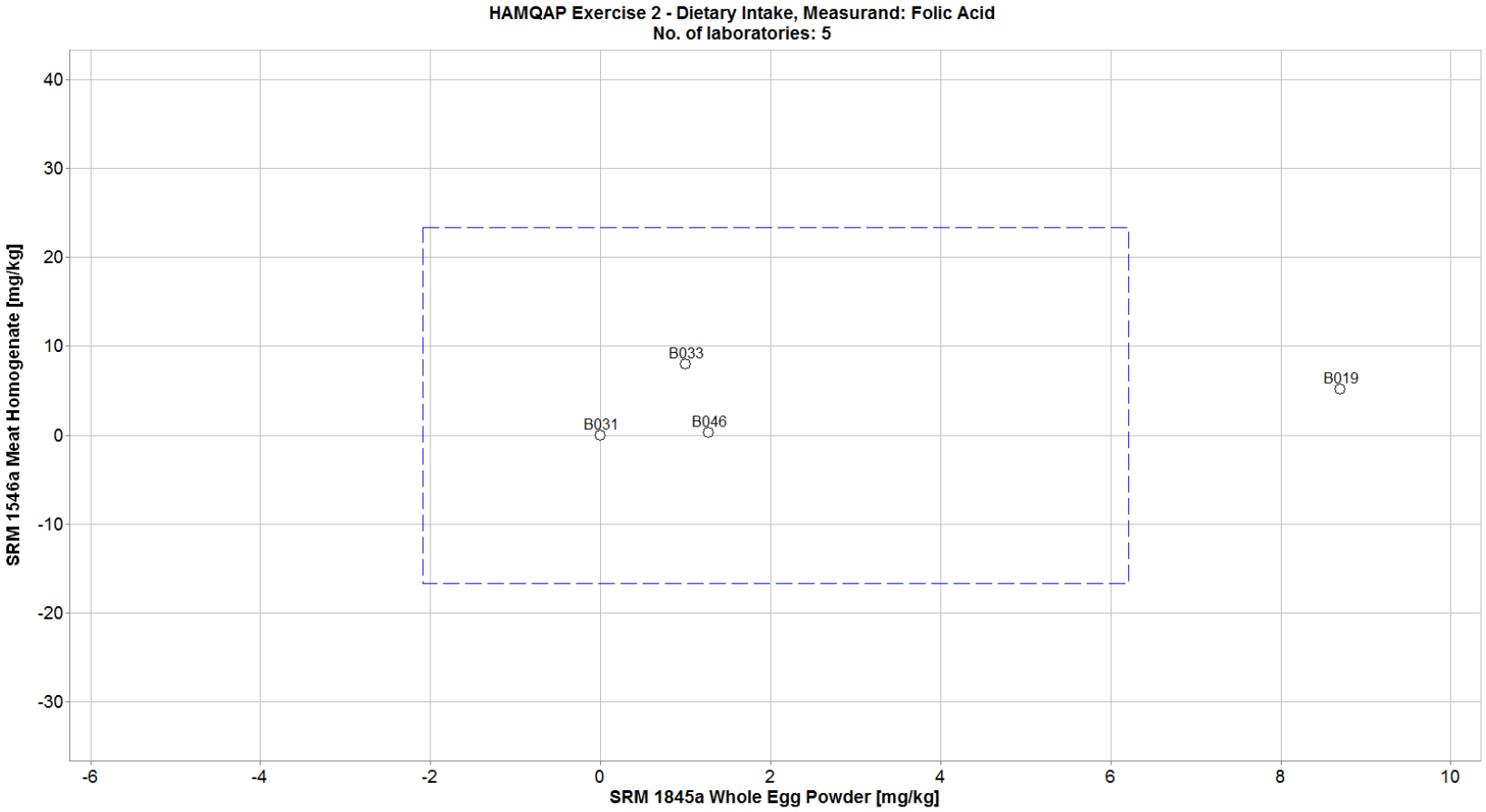

Figure 3-3. Laboratory means for folic acid in SRM 1845a Whole Egg Powder and SRM 1546a Meat Homogenate (sample/sample comparison view). In this view, the individual laboratory mean for one sample (SRM 1845a) is compared to the mean for a second sample (SRM 1546a). The dotted blue box represents the consensus range of tolerance for SRM 1845a (x-axis) and SRM 1546a (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 3-3. Data summary table for total folates in egg powder and meat homogenate.

		Total Folates									
		SRM 1845a Whole Egg Powder (mg/kg)					SRM 1546a Meat Homogenate (mg/kg)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				1.30	0.07					
	B001										
	B006										
	B010										
	B016										
	B027	0.52	0.49	0.36	0.46	0.09	301.7	70.5	36.5	136.2	144.3
	B030										
	B031	0	0	0	0	0	0	0	0	0	0
	B032										
	B035										
	B039										
	B042										
	B044										
Community Results		Consensus Mean			0.23		Consensus Mean			68.1	
		Consensus Standard Deviatio			0.87		Consensus Standard Deviatio			142.1	
		Maximum			0.46		Maximum			136.2	
		Minimum			0		Minimum			0	
		N			2		N			2	

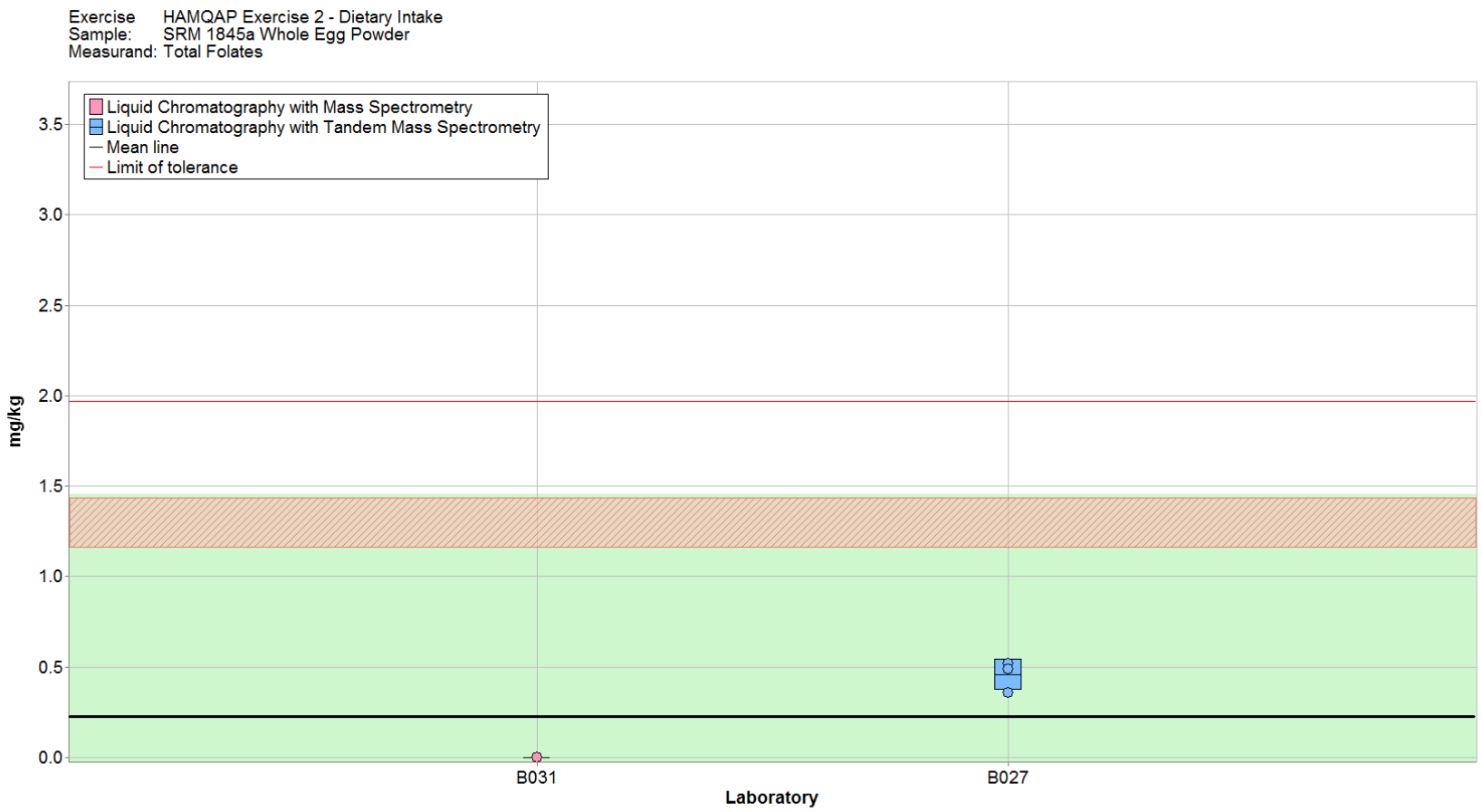

Figure 3-4. Total folates in SRM 1845a Whole Egg Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the consensus mean bounded by twice the consensus standard error. The solid red line represents the upper 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

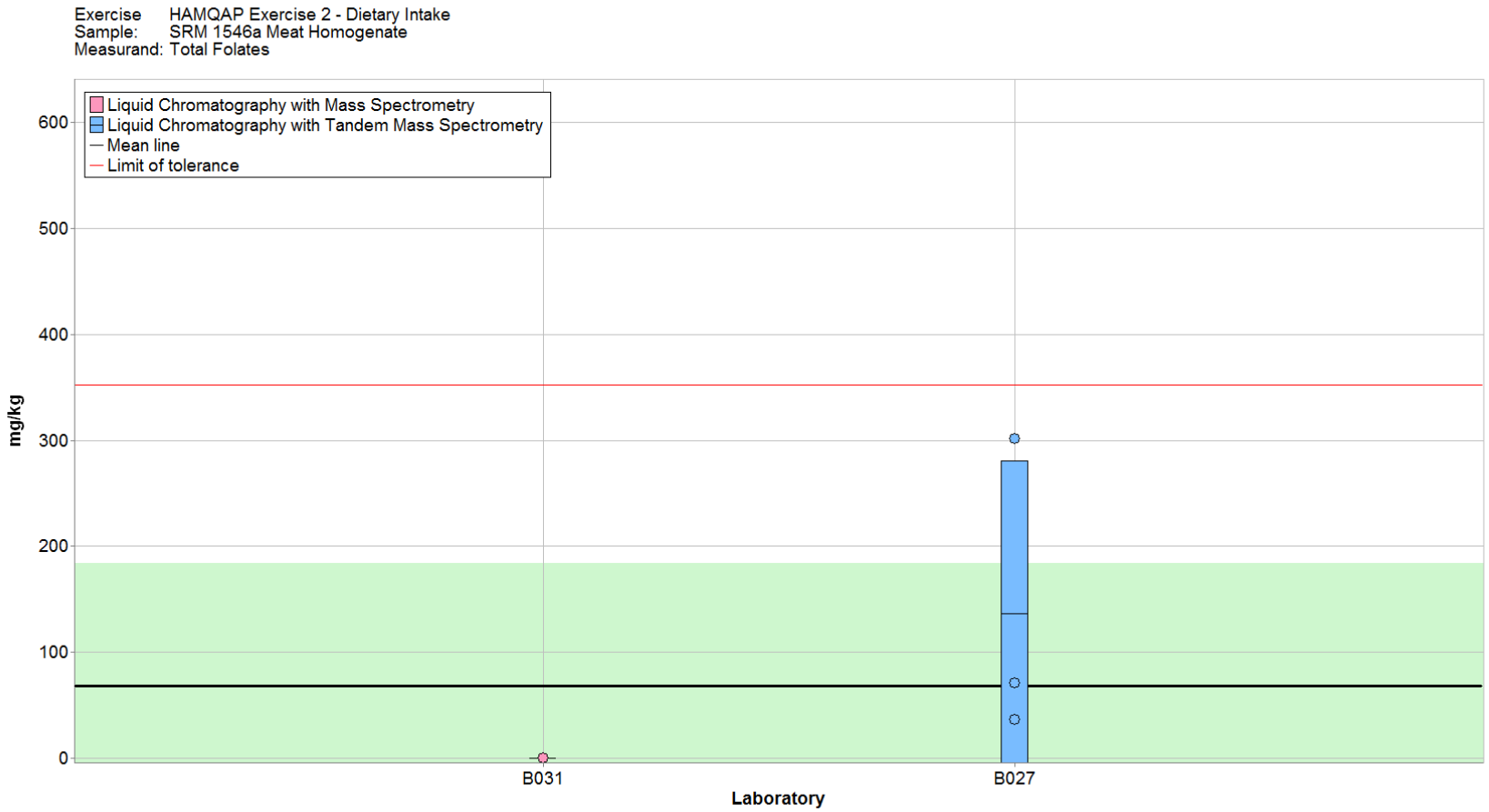

Figure 3-5. Total folates in SRM 1546a Meat Homogenate (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the consensus mean bounded by twice the consensus standard error. The solid red line represents the upper 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 NIST value has not been determined in this material.

SECTION 4: FAT-SOLUBLE VITAMINS (Vitamins A and E)

Study Overview

In this study, participants were provided with SRM 1869 Infant/Adult Nutritional Formula II and a multivitamin for dietary intake, and two samples of human serum for human metabolites. Participants were asked to use in-house analytical methods to determine and report the mass fraction (mg/kg) of as many forms of vitamin A (retinol, retinyl acetate, retinyl palmitate) and vitamin E (α-tocopherol, α-tocopheryl acetate, β-tocopherol, γ-tocopherol, δ-tocopherol) as possible in each matrix. Vitamins A and E are fat-soluble vitamins and act as antioxidants in the human body. Consumption of vitamin A is important to maintain normal human vision, the function of the immune and reproductive systems, as well as the heart, lungs, kidneys, and other organs.[5] Vitamin E consumption also supports immune system function and helps to dilate blood vessels to reduce formation of blood clots.[6] Vitamins A and E are very common in foods and supplements and can each be found in numerous forms. Esterified forms of vitamins A and E are converted in the body, where health status is measured by quantitative determination of retinol and α-tocopherol in serum. To maintain proper growth and function, health professionals may recommend dietary changes or supplementation to individuals with low serum levels of these vitamers.

Dietary Intake Sample Information

Nutritional Formula. Participants were provided with three packets, each containing 10 g of powdered nutritional formula. Before use, participants were instructed to thoroughly mix the contents by shaking the unopened packet prior to removal of a test sample for analysis, and to use a sample size of at least 1 g. Participants were asked to store the material at –20 °C before use, and to prepare one sample and report one value from each packet provided. The approximate analyte levels were not reported to participants prior to the study. The reference values for vitamins A and E in SRM 1869 were assigned using results from the manufacturer and results from collaborating laboratories. The reference values and uncertainties for vitamin A (retinol, retinyl acetate, retinyl palmitate) and vitamin E (α-tocopherol, α-tocopheryl acetate, β-tocopherol, γ-tocopherol, δ-tocopherol) in SRM 1869 are provided in the table below on an as-received basis.

Analyte	Reference Mass Fraction in SRM 1869 (mg/kg)
Retinol	19.27 ± 0.32
Retinyl Acetate	11.1 ± 1.3
Retinyl Palmitate	17.1 ± 2.9
α-Tocopherol (Free)	55.9 ± 5.3
α-Tocopherol (Total)	217.2 ± 6.2
α-Tocopheryl Acetate	174 ± 17
β-Tocopherol	4.22 ± 0.69
γ-Tocopherol	99.4 ± 5.1
δ-Tocopherol	32.5 ± 2.9

[5] Vitamin A Fact Sheet for Consumers. National Institutes of Health Office of Dietary Supplements. https://ods.od.nih.gov/factsheets/VitaminA-Consumer/ (accessed February 20, 2019).

[6] Vitamin E Fact Sheet for Consumers. National Institutes of Health Office of Dietary Supplements. https://ods.od.nih.gov/factsheets/VitaminE-Consumer/ (accessed February 20, 2019).

Multivitamin. Participants were provided with three bottles, each containing 30 multivitamin tablets. Before use, participants were instructed to grind all tablets within a bottle, mix the resulting powder thoroughly, and to use a sample size of at least 0.2 g. Participants were asked to store the material at controlled room temperature, 10 °C to 30 °C, and to prepare one sample and report one value from each bottle provided. The approximate analyte levels were not reported to participants prior to the study. The NIST-determined values for vitamins A and E in Multivitamin B were assigned using results from the manufacturer. The NIST-determined values and uncertainties for vitamin A (as retinol and retinyl acetate) and vitamin E (as α-tocopheryl acetate) in the multivitamin are provided in the table below on an as-received basis.

Analyte	NIST-Determined Mass Fraction in Multivitamin B (mg/kg)
Vitamin A (as Retinol)	783 ± 38
Vitamin A (as Retinyl Acetate)	898 ± 42
Vitamin E (as α-Tocopheryl Acetate)	18019 ± 432

Dietary Intake Study Results

- The table below summarizes the participation statistics for the study of vitamin A and vitamin E in nutritional formula and multivitamin.

Analyte	Number of Laboratories Requesting Samples	Number of Laboratories Reporting Results (Percent Participation) SRM 1869 Infant/Adult Nutritional Formula II	Multivitamin B
Total Retinol	19	6 (32 %)	7 (37 %)
Retinyl Acetate	22	10 (45 %)	11 (50 %)
Retinyl Palmitate	22	10 (45 %)	7 (32 %)
α-Tocopherol	20	9 (45 %)	5 (25 %)
α-Tocopheryl Acetate	21	8 (38 %)	11 (52 %)
Total α-Tocopherol	23	10 (43 %)	10 (43 %)
β-Tocopherol	15	4 (27 %)	4 (27 %)
γ-Tocopherol	16	5 (31 %)	5 (31 %)
β- plus γ-Tocopherol	16	3 (19 %)	3 (19 %)

- The consensus means were within the target ranges for retinyl palmitate in the nutritional formula and total retinol and α-tocopheryl acetate in the multivitamin.
- The between-laboratory variabilities for total retinol and α-tocopheryl acetate in the multivitamin were good at 7 % and 10 % RSD, respectively.
- The between-laboratory variability for retinyl palmitate in the nutritional formula was unacceptable at 150 % RSD.
- The consensus means were below the target ranges for total retinol, α-tocopheryl acetate, total α-tocopherol, β-tocopherol, γ-tocopherol, and β- plus γ-tocopherol in the nutritional formula, and for retinyl acetate in the multivitamin.

- The between-laboratory variabilities for total retinol, total α-tocopherol, and γ-tocopherol in the nutritional formula were acceptable between 31 % and 44 % RSD, respectively.
- The between-laboratory variabilities for α-tocopheryl acetate, β-tocopherol, and β- plus γ-tocopherol in the nutritional formula and for retinyl acetate in the multivitamin were unacceptable at over 75 % RSD.
- The consensus means were above the target ranges for retinyl acetate and α-tocopherol in the nutritional formula, with between-laboratory variabilities over 100 % RSD.
- The between-laboratory variability for total α-tocopherol in the multivitamin was acceptable at 19 % RSD.
- For all other sample/measurand combinations, the between-laboratory variability was either very high (> 75 % RSD) or sufficient participation was not attained to allow discussion of performance.
- Most laboratories reported using either solvent extraction or saponification (base hydrolysis of fat) as the sample preparation approach for vitamin A and vitamin E. One laboratory reported using dilution for the nutritional formula sample, and one laboratory did not specify a sample preparation approach.
- Most laboratories reported using LC-absorbance as their analytical method for the determination of vitamin A and vitamin E in both samples. Two laboratories reported using LC-fluorescence as their analytical method for the determination of total retinol, α-tocopherol, total α-tocopherol, β-tocopherol, and γ-tocopherol in both samples. The remaining laboratories did not specify an analytical method.

Dietary Intake Technical Recommendations

The following recommendations are based on results obtained from the participants in this study. In some cases, too few data were reported to allow for meaningful conclusions to be drawn. Corresponding figures were chosen to show results according to analytical method or sample preparation method depending on observed trends.

- The overall results for vitamin A indicate that laboratories can measure various forms of vitamin A in these types of samples. Reporting values for vitamin A can be complicated, and understanding the requested information (e.g., form, units) as well as the vitamer forms measured by the analytical method used is critical for accurate reporting.
- The vitamin A in SRM 1869 was fortified using both retinyl acetate and retinyl palmitate according to the manufacturer, while endogenous retinol was present from the other ingredients in the formulation. The vitamin A in Multivitamin B was fortified using retinyl acetate, according to the manufacturer, and no retinyl palmitate was expected in this sample.
    - Laboratories using a solvent extraction approach (without hydrolysis) may identify measurable quantities of retinol, retinyl acetate, and retinyl palmitate in SRM 1869 and retinyl acetate in Multivitamin B. These mass fractions can be reported individually and can also be normalized using the relative molecular weights of the vitamers and summed to provide a value for total retinol.
    - Laboratories using a saponification or hydrolysis sample preparation approach, in which esterified forms of vitamin A are converted to retinol, should report a single value for total retinol.
    - The data for total retinol in Multivitamin B was of bimodal distribution, with a group of three laboratories reporting mass fractions of approximately 700 mg/kg and another group of four laboratories reporting mass fractions of approximately 875 mg/kg.

    - The four laboratories reporting at the higher level all used an extraction-based sample preparation approach, which would not convert retinyl acetate to retinol. The high bias may be from incorrect reporting of measured values, from incorrect conversion (or no conversion) of the mass fraction of retinyl acetate to retinol using the molecular weights of the two vitamers. This type of bias may also be the result of improper calibration (see below) or a chromatographic coelution causing the results to be higher than expected.
    - Two of the three laboratories reporting at the lower level used a hydrolysis or saponification-based extraction approach, which would convert retinyl acetate in the sample to retinol. The bias may be the result of incomplete extraction or saponification causing the results to be lower than expected. Some degradation of the analyte may also be caused by the saponification step, resulting in a low bias in the overall results.
- The overall results for vitamin E indicate that laboratories can measure various forms of vitamin E in these types of samples. Reporting values for vitamin E can be complicated, and understanding the requested information (e.g., form, units) as well as the vitamer forms measured by the analytical method used is critical for accurate reporting.
    - The vitamin E in SRM 1869 was fortified using α-tocopheryl acetate according to the manufacturer, while non-esterified α-tocopherol as well as β-tocopherol, γ-tocopherol, and δ-tocopherol were present from the other ingredients in the formulation. The vitamin E in Multivitamin B was fortified using α-tocopheryl acetate according to the manufacturer, and no other tocopherols were expected in this sample.
    - Laboratories using a solvent extraction approach (without hydrolysis) may identify measurable quantities of α-tocopherol, α-tocopheryl acetate, β-tocopherol, γ-tocopherol, and δ-tocopherol in SRM 1869 and α-tocopheryl acetate in Multivitamin B. The mass fractions of α-tocopherol and α-tocopheryl acetate can be reported individually and can also be normalized using the relative molecular weights of the vitamers and summed to provide a value for total α-tocopherol.
    - Laboratories using a saponification or hydrolysis sample preparation approach, in which esterified forms of vitamin E would be converted to α-tocopherol, should report a single value for total α-tocopherol.
    - Not enough laboratories reported data for β-tocopherol, γ-tocopherol, and δ-tocopherol to allow meaningful conclusions to be drawn from the data.
- Several laboratories reported values of zero for measurements in all materials. "Zero" is not a quantity that can be measured, and therefore a more appropriate result would be to report that a value is below the MDL, LOQ, or QL.
- The concentration of calibration solutions for vitamins A and E must be determined spectrophotometrically to prevent bias in the final analytical result. Future studies on vitamins A and E may include a request for information about the calibration approach used by participants in order to understand potential bias related to calibration.
- In many analytical approaches for the determination of vitamins A and E, a saponification step is necessary to remove fat from the sample. An adequate amount of acid or base must be added, based on the fat content of the sample, to ensure that the fat removal is exhaustive. Often, post-extraction cleanup is also necessary (e.g., liquid-liquid extraction, solid-phase extraction) and can affect the overall analytical result, particularly if the cleanup results in analyte loss (even when a recovery calculation is used).

Table 4-1. Individualized data summary table (NIST) for vitamins A and E in nutritional formula and multivitamin.

National Institute of Standards & Technology

HAMQAP Exercise 2 - 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 Retinol	SRM 1869 Infant/Adult Nutritional Formula II		mg/kg	19.27	0.16			6	20	7	19.27	0.32
Total Retinol	Multivitamin B		mg/kg	783	19			7	810	55	783	38
Retinyl Acetate	SRM 1869 Infant/Adult Nutritional Formula II		mg/kg	11.1	0.7			10	10	22	11.1	1.3
Retinyl Acetate	Multivitamin B		mg/kg	898	21			11	550	410	898	42
Retinyl Palmitate	SRM 1869 Infant/Adult Nutritional Formula II		mg/kg	17.1	1.5			10	10	15	17.1	2.9
Retinyl Palmitate	Multivitamin B		mg/kg					7	40	130		
alpha-Tocopherol	SRM 1869 Infant/Adult Nutritional Formula II		mg/kg	55.9	2.7			9	130	180	55.9	5.3
alpha-Tocopherol	Multivitamin B		mg/kg					5	40	190		
alpha-Tocopheryl Acetate	SRM 1869 Infant/Adult Nutritional Formula II		mg/kg	174	9			8	130	110	174	17
alpha-Tocopheryl Acetate	Multivitamin B		mg/kg	18019	216			11	18800	1800	18019	432
Total alpha-Tocopherol	SRM 1869 Infant/Adult Nutritional Formula II		mg/kg	217.2	3.1			10	200	61	217.2	6.2
Total alpha-Tocopherol	Multivitamin B		mg/kg					10	18300	3500		
beta-Tocopherol	SRM 1869 Infant/Adult Nutritional Formula II		mg/kg	4.22	0.35			4	1.4	6.4	4.22	0.69
beta-Tocopherol	Multivitamin B		mg/kg					4	0	0		
gamma-Tocopherol	SRM 1869 Infant/Adult Nutritional Formula II		mg/kg	99.4	2.6			5	80	35	99.4	5.1
gamma-Tocopherol	Multivitamin B		mg/kg					5	0	0		
gamma plus beta-Tocopherol	SRM 1869 Infant/Adult Nutritional Formula II		mg/kg	103.6	2.6			3	60	170	104.0	5.1
gamma plus beta-Tocopherol	Multivitamin B		mg/kg					3	0	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 4-2. Data summary table for total retinol in nutritional formula and multivitamin. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		Total Retinol									
		SRM 1869 Infant/Adult Nutritional Formula II (mg/kg)					Multivitamin B (mg/kg)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				19.27	0.32				783	38
	B001										
	B002										
	B006										
	B010										
	B016	15.78	17.82	16.37	16.66	1.05	633	700	683	672	35
	B017										
	B019										
	B020	17	17.6	17.5	17.37	0.32	702	703	702	702	1
	B021										
	B025										
	B026	6.3	6	6.5	6.27	0.25	873	866	905	882	21
	B027	17.87	18.79	18.39	18.35	0.46	717	686	761	722	38
	B028	21.43	21.61	20.97	21.34	0.33	893	865	867	875	16
	B030										
	B031	87.28	103.03	78.76	89.69	12.31	852	869	892	871	20
	B035										
	B036										
	B038						867	877	832	859	24
	B039										
	B042										
	B044										
Community Results		Consensus Mean			16.0		Consensus Mean			809	
		Consensus Standard Deviation			7.0		Consensus Standard Deviation			55	
		Maximum			89.7		Maximum			882	
		Minimum			6.3		Minimum			672	
		N			6		N			7	

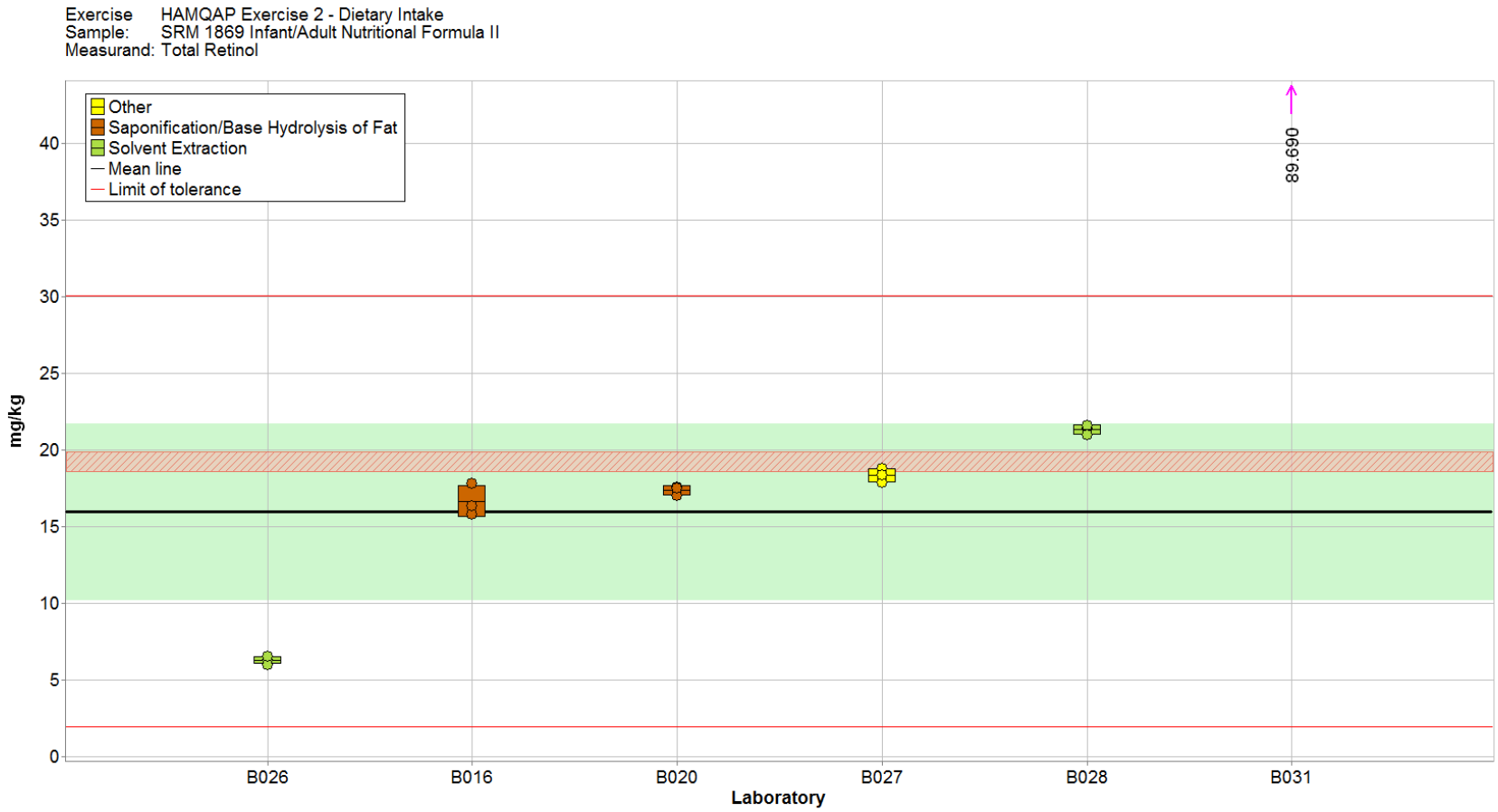

Figure 4-1. Total retinol in SRM 1869 Infant/Adult Nutritional Formula II (data summary view – sample preparation method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the sample preparation method employed. The solid black 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

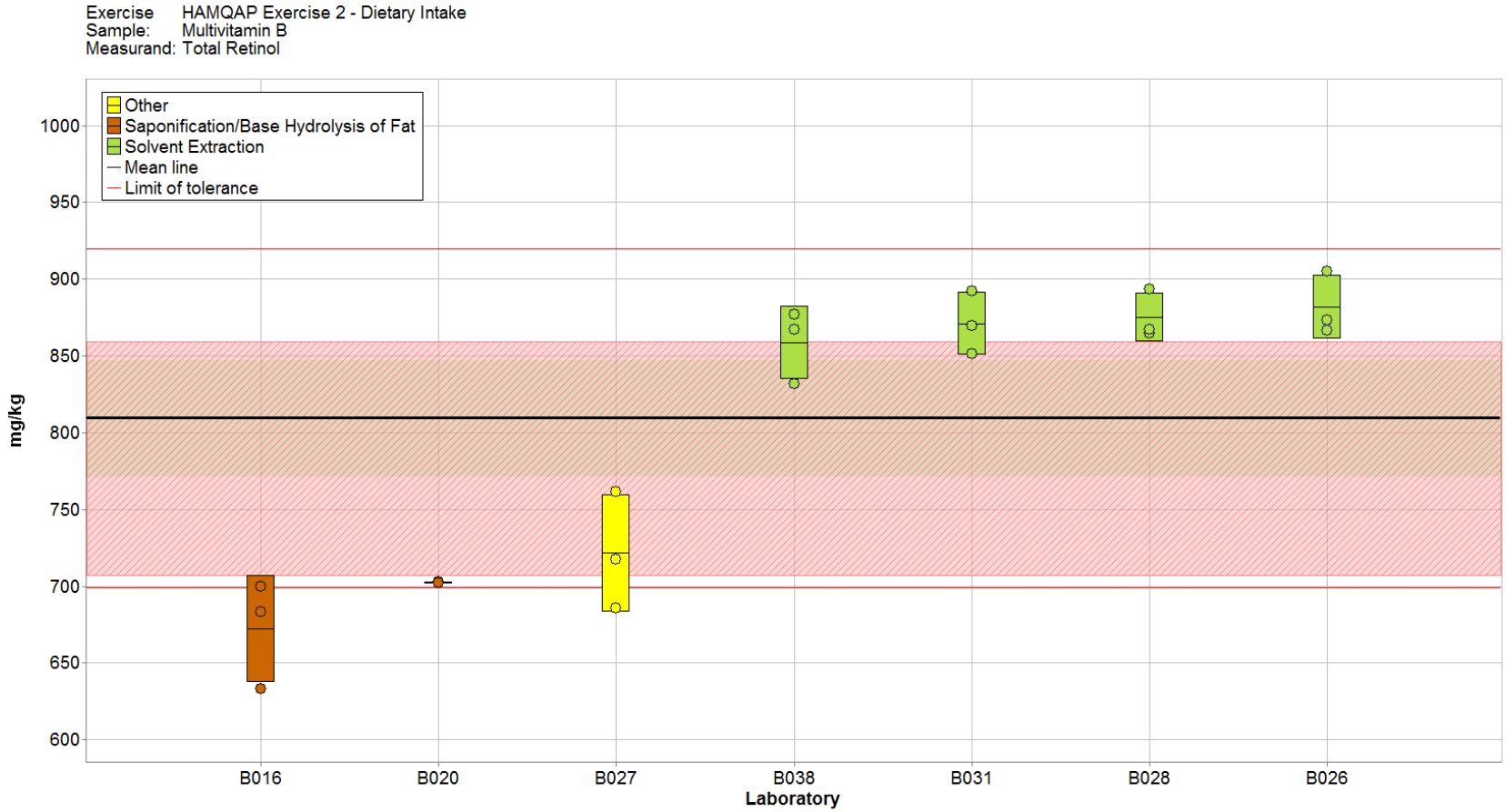

Figure 4-2. Total retinol in Multivitamin B (data summary view – sample preparation method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the sample preparation method employed. The solid black 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

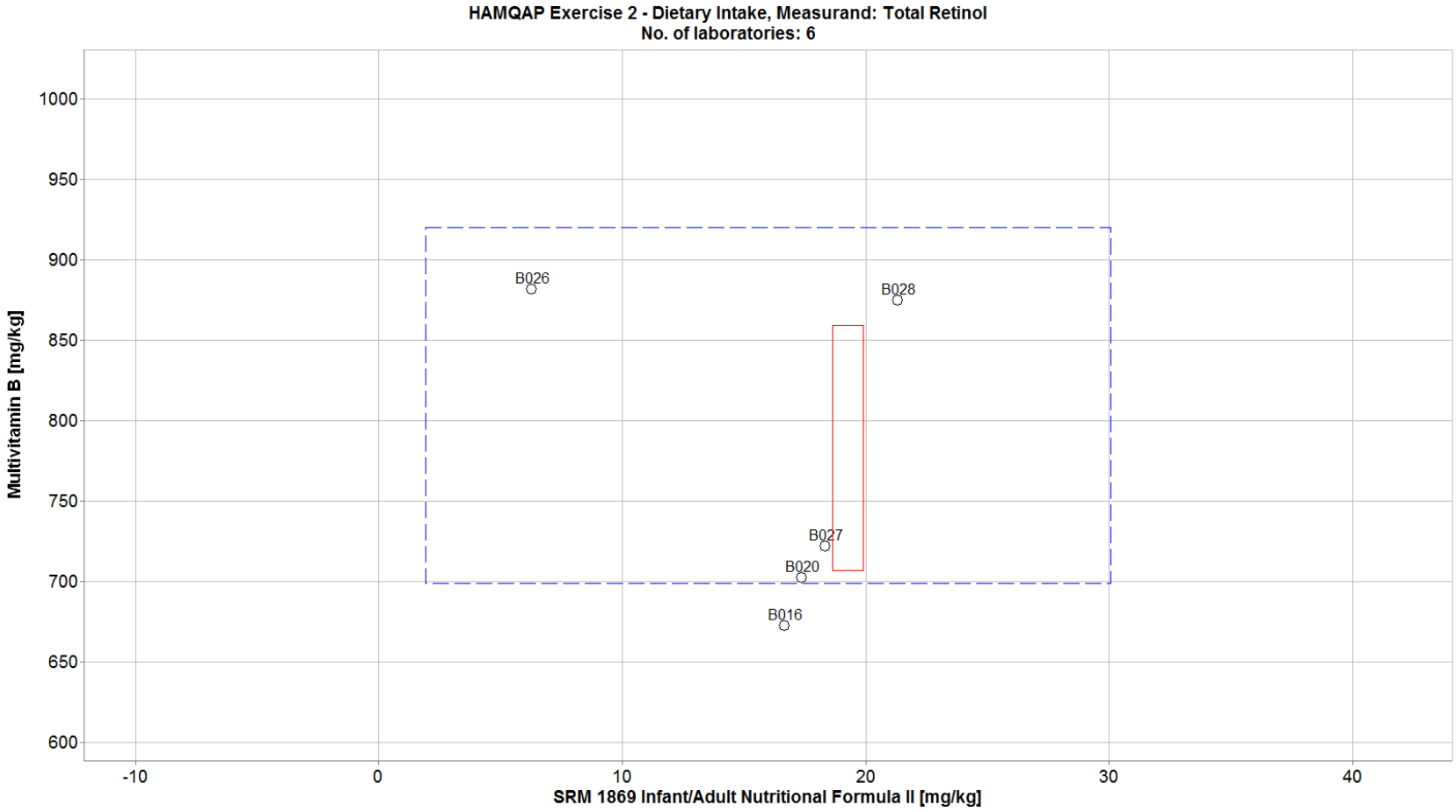

Figure 4-3. Laboratory means for total retinol in SRM 1869 Infant/Adult Nutritional Formula II and Multivitamin B (sample/sample comparison view). In this view, the individual laboratory mean for one sample (SRM 1869) is compared to the mean for a second sample (Multivitamin B). The solid red box represents the NIST range of tolerance for the two samples, SRM 1869 (x-axis) and Multivitamin 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 1869 (x-axis) and Multivitamin 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 4-3. Data summary table for retinyl acetate in nutritional formula and multivitamin. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		Retinyl Acetate									
		SRM 1869 Infant/Adult Nutritional Formula II (mg/kg)					Multivitamin B (mg/kg)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				11.10	1.30				898	42
	B001										
	B002										
	B005	10.97	10.91	10.68	10.85	0.15	141	137	145	141	4
	B006										
	B008	0	0	0	0	0	0	0	0	0	0
	B013										
	B016						786	817	843	815	28
	B019	11.5	11.6	11.3	11.47	0.15	807	556	563	642	143
	B020										
	B021	564	619	636	606.33	37.63	1650	1636	1836	1707	112
	B025										
	B026	0	0	0	0.00	0.00	0	0	0	0	0
	B028										
	B030										
	B031	56.46	64.84	63.04	61.45	4.41	816	869	883	856	36
	B033	50	30	40	40	10	620	600	580	600	20
	B034										
	B035										
	B036										
	B038	11	11.1	10.9	11.0	0.1	867	877	832	859	24
	B039										
	B042	0	0	0	0	0	0	0	0	0	0
	B044										
	B046	10.509	10.145	10.361	10.34	0.18	958	898	978	945	42
Community Results		Consensus Mean			14.59		Consensus Mean			548	
		Consensus Standard Deviatio			22.01		Consensus Standard Deviatio			414	
		Maximum			606.33		Maximum			1707	
		Minimum			0.00		Minimum			0	
		N			10		N			11	

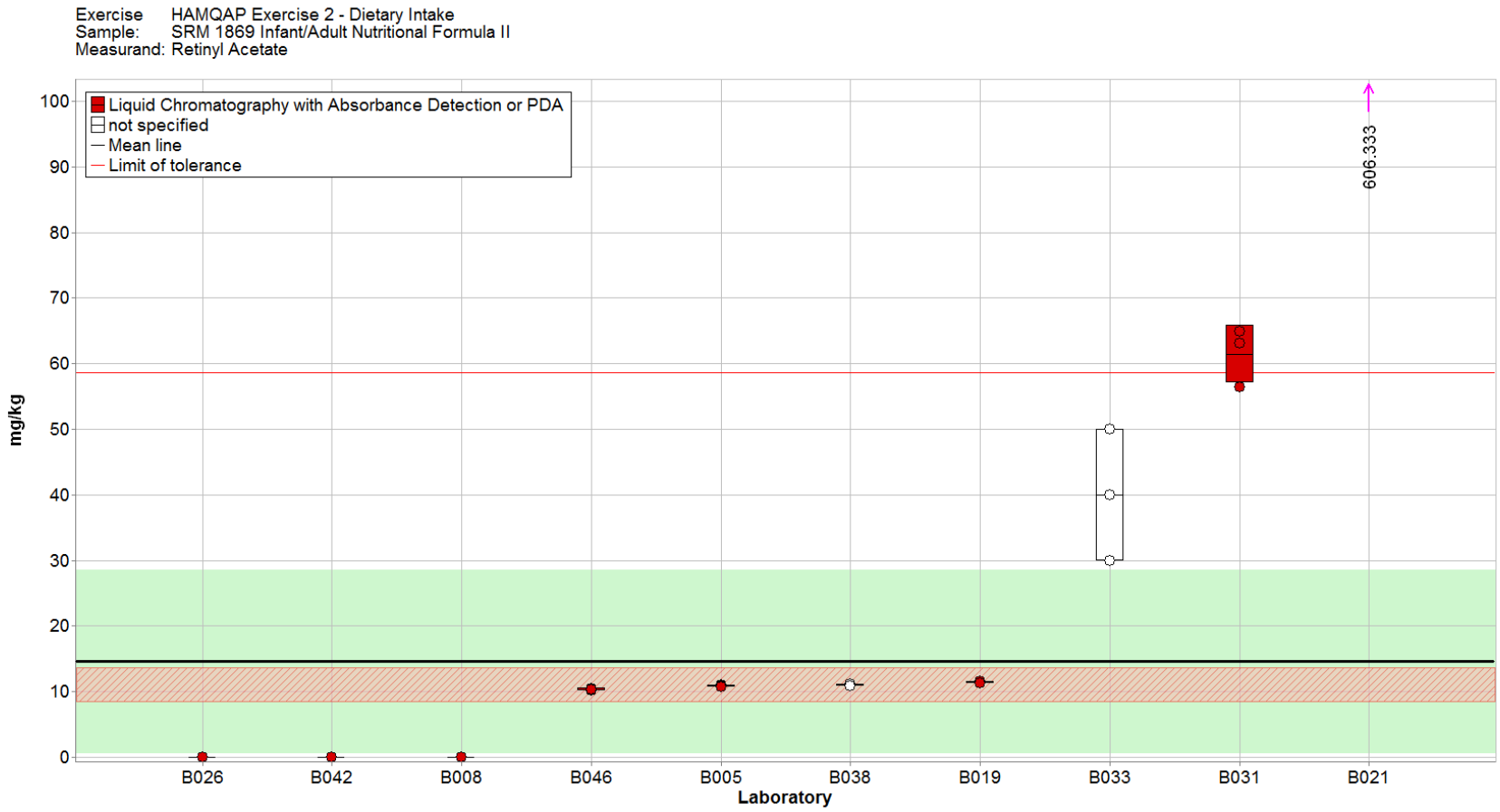

Figure 4-4. Retinyl acetate in SRM 1869 Infant/Adult Nutritional Formula II (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

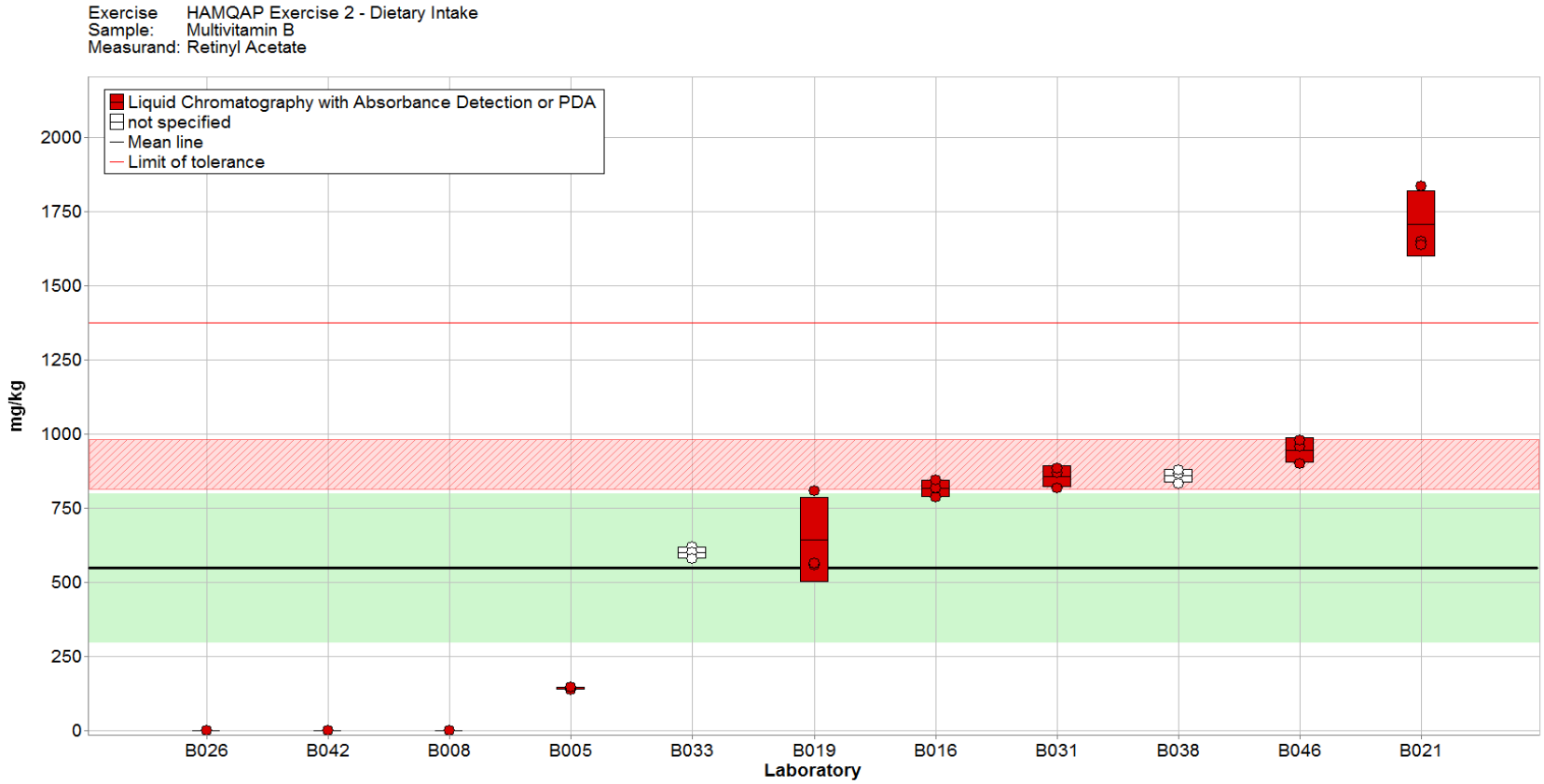

Figure 4-5. Retinyl acetate in Multivitamin B (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

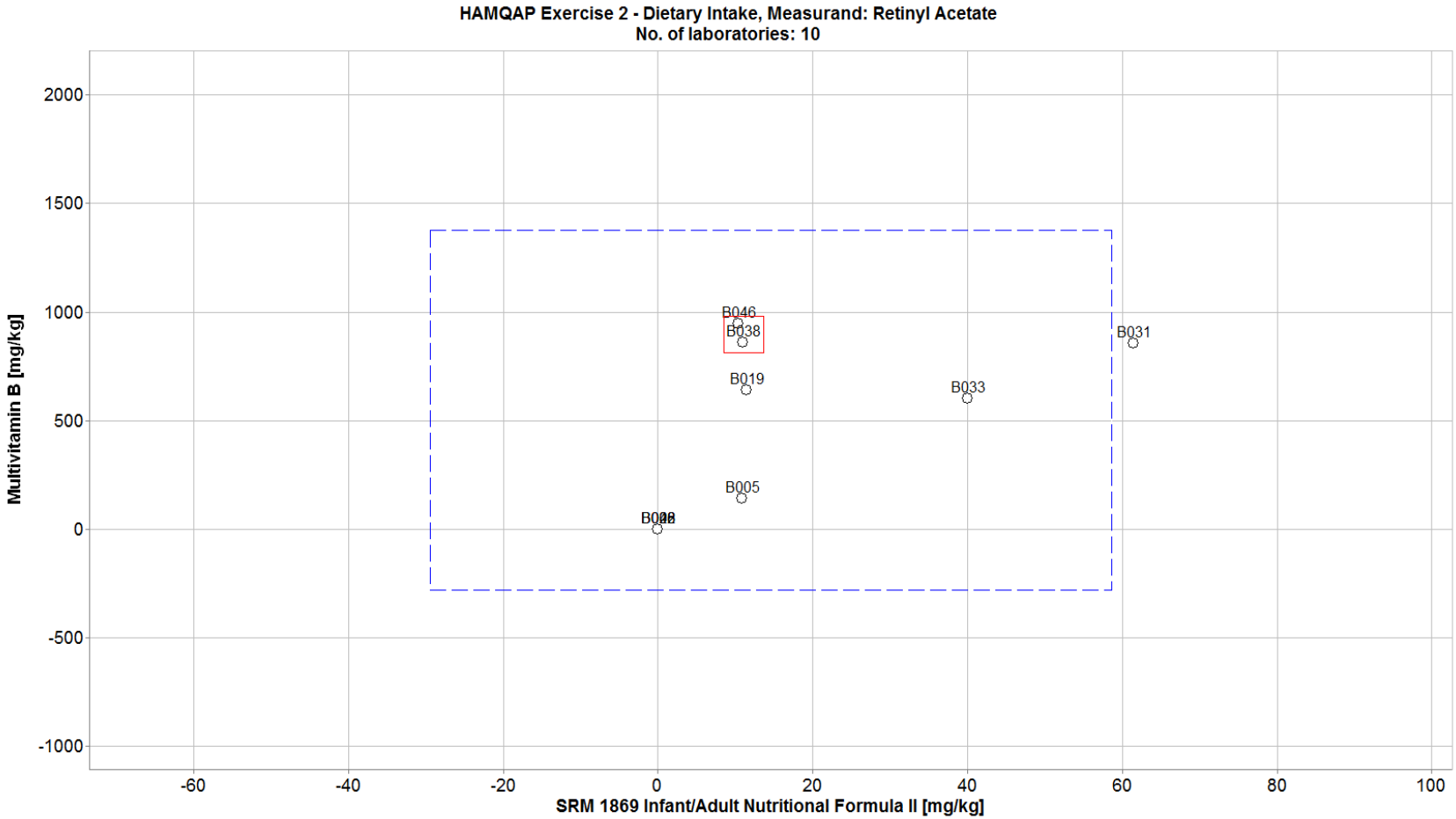

Figure 4-6. Laboratory means for retinyl acetate in SRM 1869 Infant/Adult Nutritional Formula II and Multivitamin B (sample/sample comparison view). In this view, the individual laboratory mean for one sample (SRM 1869) is compared to the mean for a second sample (Multivitamin B). The solid red box represents the NIST range of tolerance for the two samples, SRM 1869 (x-axis) and Multivitamin 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 1869 (x-axis) and Multivitamin 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 4-4. Data summary table for retinyl palmitate in nutritional formula and multivitamin. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		Retinyl Palmitate									
		SRM 1869 Infant/Adult Nutritional Formula II (mg/kg)					Multivitamin B (mg/kg)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				17.1	2.9					
	B001										
	B002										
	B005	21.38	20.85	19.33	20.52	1.06	< 0.001	< 0.001	< 0.001		
	B006										
	B008	0	0	0	0	0	< 0.000	< 0.000	< 0.000		
	B013										
	B016						0	0	0	0	0
	B019										
	B020										
	B021	366	400	410	392	23	200	223	208	210	12
	B025										
	B026	6.3	6	6.5	6.3	0.3	873.3	866.3	905.3	881.6	20.8
	B028										
	B030										
	B031	14.06	19.98	13.19	15.74	3.69	0	0	0	0	0
	B033	30	20	20	23	6					
	B035										
	B036	15	15	15	15	0					
	B038	172	174	154	167	11	0	0	0	0	0
	B039										
	B042	19.42	18.63	20.57	19.54	0.98	0	0	0	0	0
	B044										
	B046	1.134	1.133	1.235	1.167	0.059	0.547	0.528	0.405	0.493	0.077
Community Results		Consensus Mean			12.7		Consensus Mean			35	
		Consensus Standard Deviation			15.1		Consensus Standard Deviation			132	
		Maximum			392		Maximum			882	
		Minimum			0		Minimum			0	
		N			10		N			7	

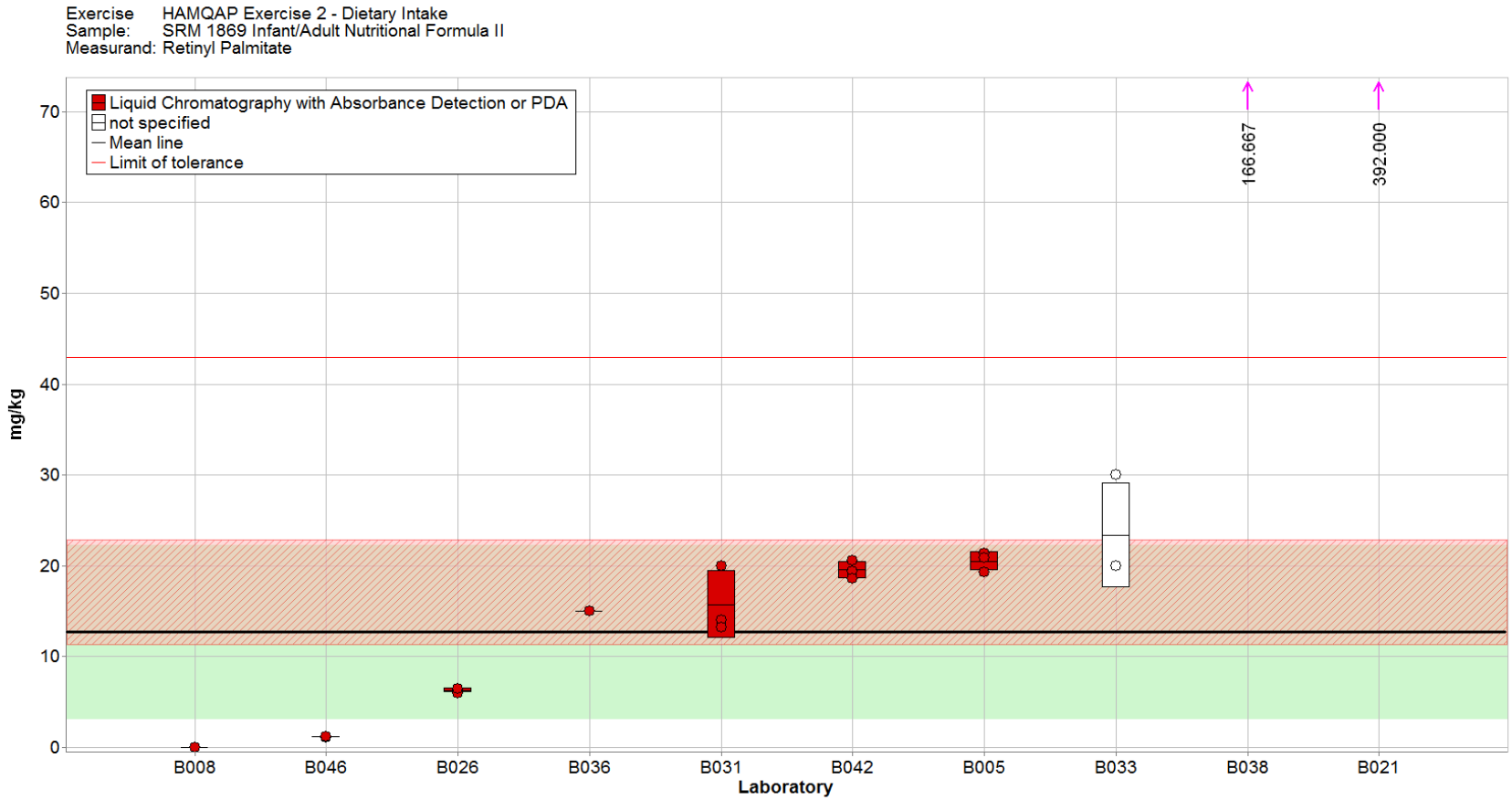

Figure 4-7. Retinyl palmitate in SRM 1869 Infant/Adult Nutritional Formula II (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

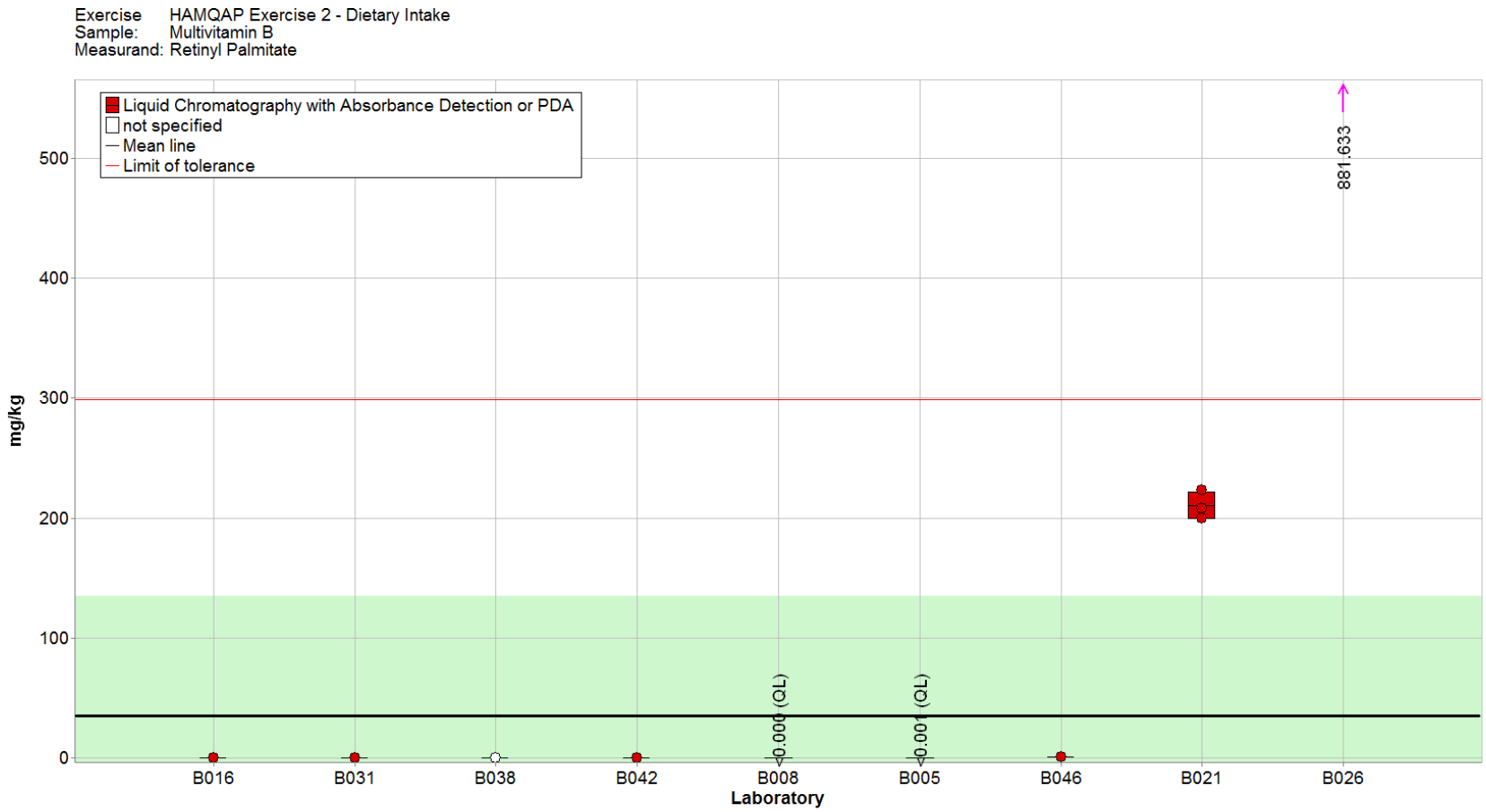

Figure 4-8. Retinyl palmitate in Multivitamin B (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

Table 4-5. Data summary table for α-tocopherol in nutritional formula and multivitamin. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		alpha-Tocopherol									
		SRM 1869 Infant/Adult Nutritional Formula II (mg/kg)					Multivitamin B (mg/kg)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				55.9	5.3					
	B001										
	B002										
	B006										
	B007	1730	1710	2110	1850	225.4					
	B008										
	B010										
	B016	228	228	221	225.7	4.0	16738	16756	17106	16867	207.5
	B019	60.1	59.3	60.1	59.8	0.46					
	B020										
	B021	484	482	473	479.7	5.9					
	B025										
	B026	0	0	0	0.00	0	0	0	0	0.00	0
	B027										
	B028										
	B030										
	B031	188.32	164.32	160.32	171.0	15	0	0	0	0.00	0
	B033	140	120	100	120	20					
	B034										
	B035										
	B036	< 76	< 76	< 76			< 4113	< 4113	< 4113		
	B038	0	0	0	0.00	0	0	0	0	0.00	0
	B039										
	B042										
	B044										
	B046	52.41	53.36	54.15	53.31	0.87	157.08	164.17	183.17	168.14	13
Community Results		Consensus Mean			128		Consensus Mean			42	
		Consensus Standard Deviation			176		Consensus Standard Deviation			188	
		Maximum			1850		Maximum			16867	
		Minimum			0		Minimum			0	
		N			9		N			5	

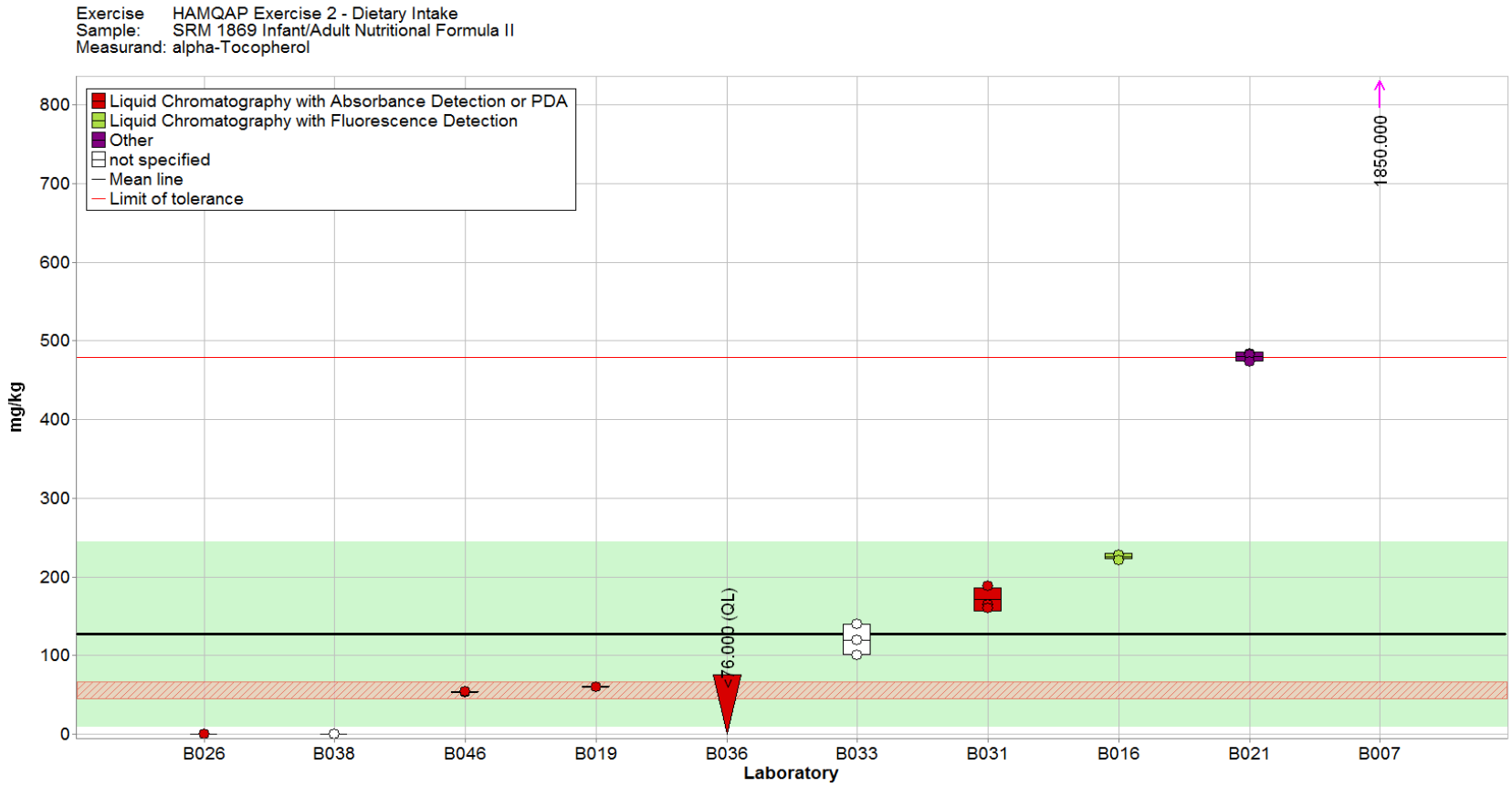

Figure 4-9. α-Tocopherol in SRM 1869 Infant/Adult Nutritional Formula II (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

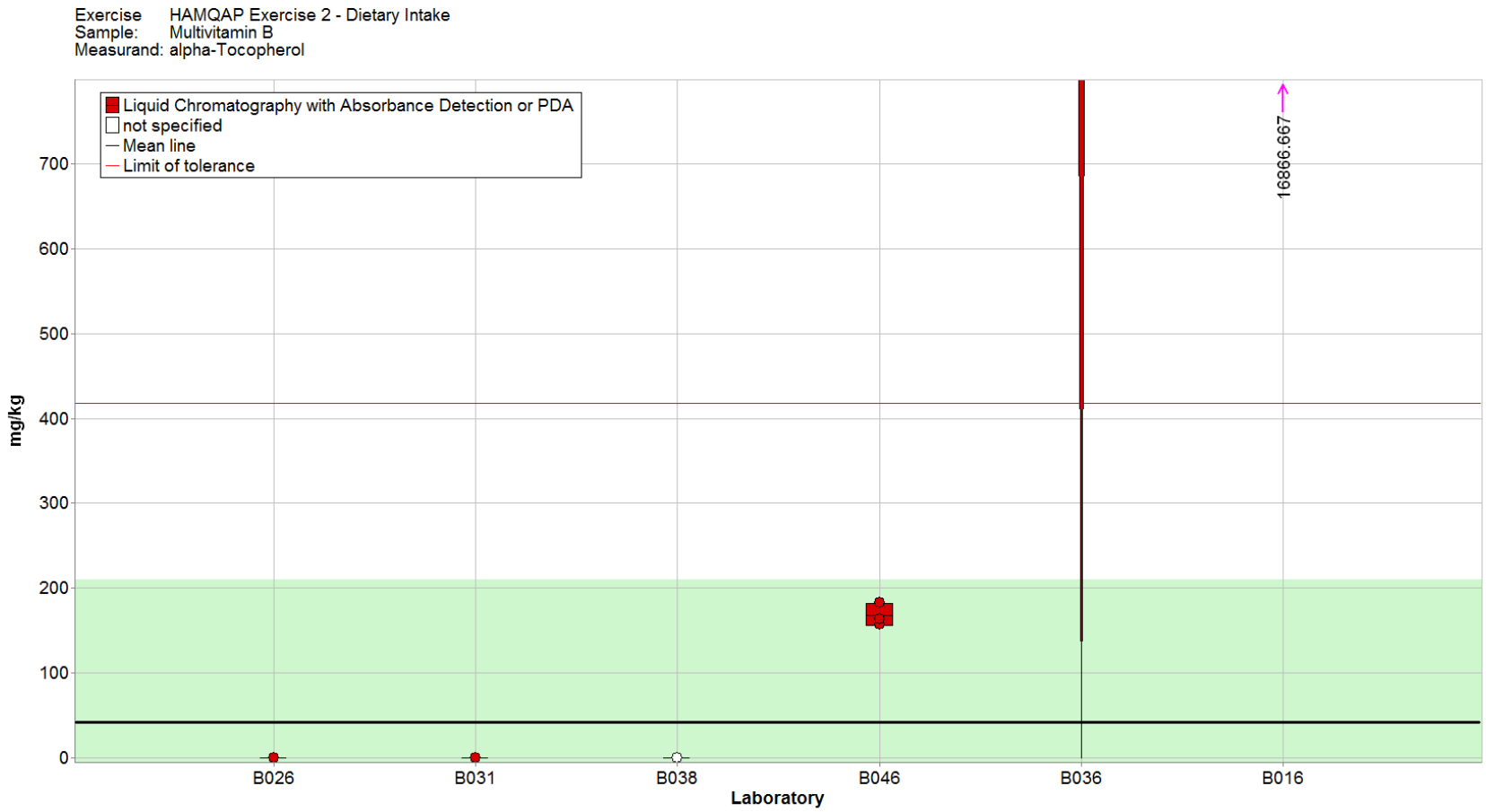

Figure 4-10. α-Tocopherol in Multivitamin B (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

Table 4-6. Data summary table for α-tocopheryl acetate in nutritional formula and multivitamin. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		alpha-Tocopheryl Acetate									
		SRM 1869 Infant/Adult Nutritional Formula II (mg/kg)					Multivitamin B (mg/kg)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				174	17				18019	432
	B001										
	B002										
	B006										
	B007						19710	20580	19820	20037	474
	B008	106	105	91.3	100.8	8.2	13700	12900	13500	13367	416
	B013										
	B016										
	B019	127.4	127.6	130.3	128.4	1.6	19731	20451	19874	20018	381
	B020										
	B021						16595	17873	18516	17661	978
	B025										
	B026	0	0	0	0	0	17246	17112	15102	16487	1201
	B028										
	B031	183.89	213.25	186.38	194.51	16.28	19229	19736	20289	19751	531
	B033	350	330	300	327	25	25400	25570	24390	25120	638
	B035										
	B036	< 494	< 494	< 494			17863	17586	17870	17773	162
	B038	163	154	161	159	5	19500	19900	20100	19833	306
	B039										
	B042	0	0	0	0	0	19800	19300	18700	19267	551
	B044										
	B046	157.02	159.35	155.09	157.15	2.13	19088	19767	19735	19530	383
Community Results		Consensus Mean			129		Consensus Mean			18839	
		Consensus Standard Deviation			110		Consensus Standard Deviatio			1771	
		Maximum			327		Maximum			25120	
		Minimum			0		Minimum			13367	
		N			8		N			11	

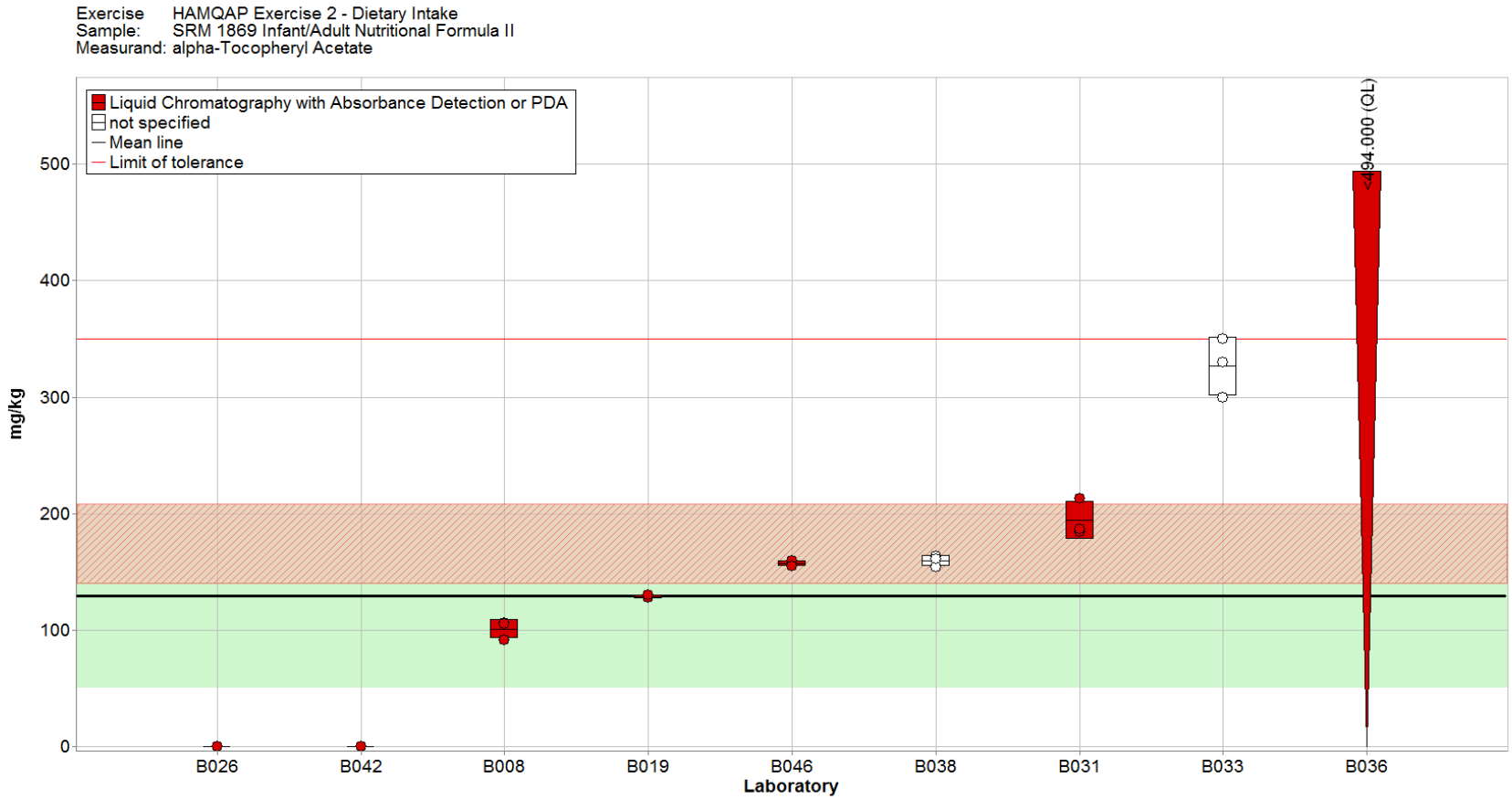

Figure 4-11. α-Tocopheryl acetate in SRM 1869 Infant/Adult Nutritional Formula II (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

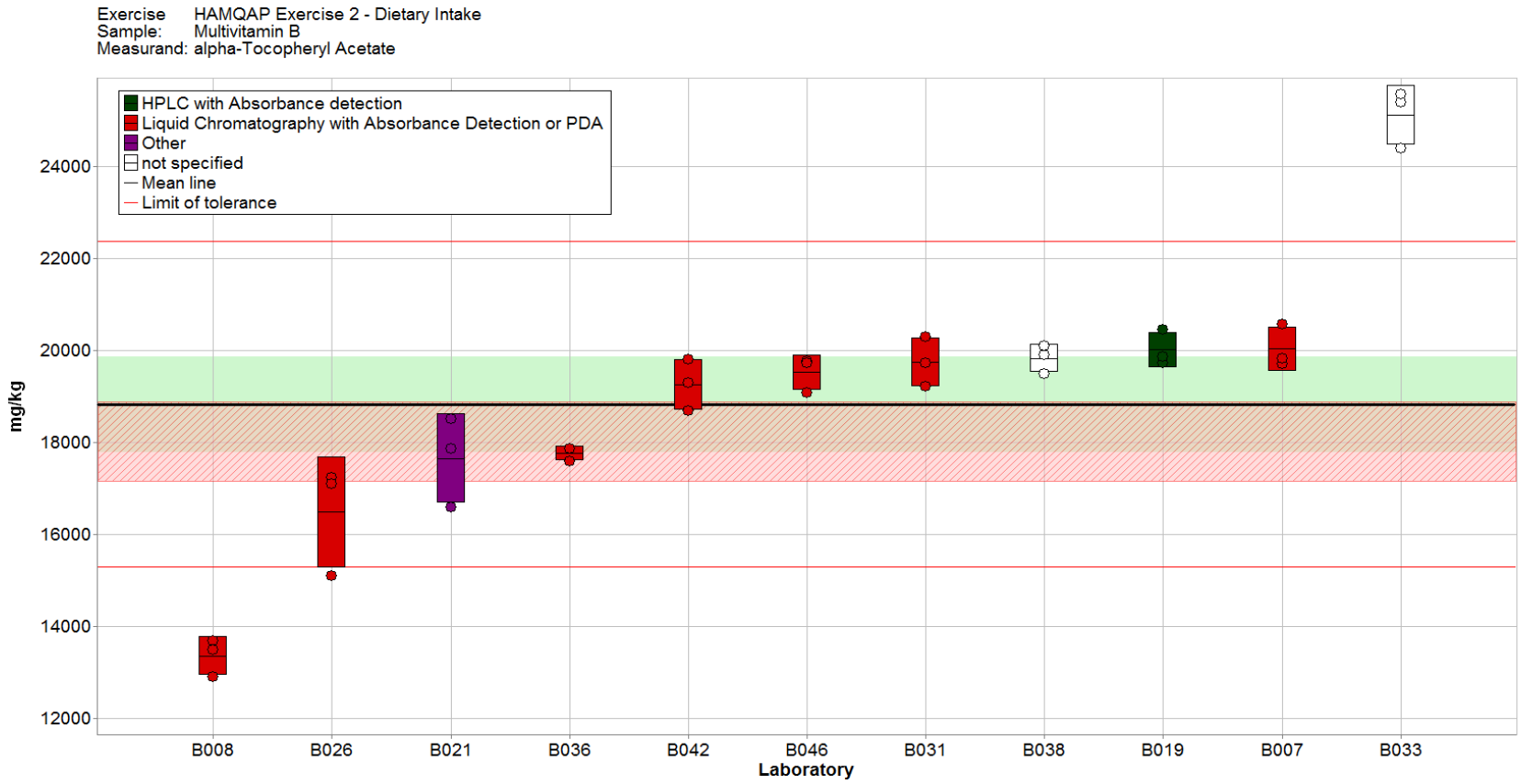

Figure 4-12. α-Tocopheryl acetate in Multivitamin B (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

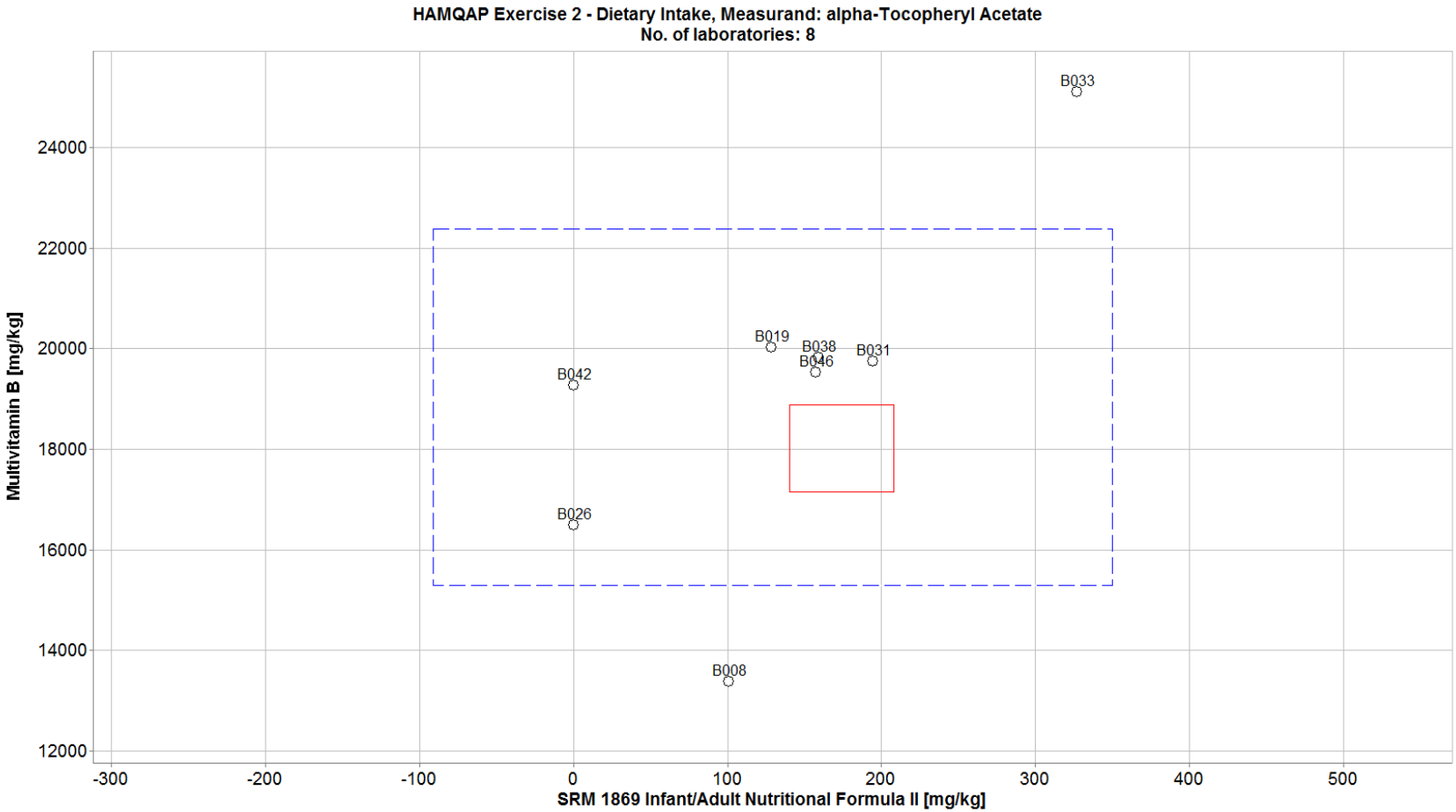

Figure 4-13. Laboratory means for α-tocopheryl acetate in SRM 1869 Infant/Adult Nutritional Formula II and Multivitamin B (sample/sample comparison view). In this view, the individual laboratory mean for one sample (SRM 1869) is compared to the mean for a second sample (Multivitamin B). The solid red box represents the NIST range of tolerance for the two samples, SRM 1869 (x-axis) and Multivitamin 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 1869 (x-axis) and Multivitamin 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 4-7. Data summary table for total α-tocopherol in nutritional formula and multivitamin. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		Total alpha-Tocopherol									
		SRM 1869 Infant/Adult Nutritional Formula II (mg/kg)					Multivitamin B (mg/kg)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				217.2	6.2					
	B001										
	B002										
	B004	257	258	259	258	1	21500	21600	21100	21400	265
	B006										
	B007										
	B008	139.8	138.3	123.4	133.8	9.1	13863	13073	13667	13534	411
	B016	228	228	221	226	4	16738	16756	17106	16867	207
	B017										
	B019	223.8	222	227.3	224.4	2.7					
	B020	227	227.3	227	227.1	0.2	18000	18007	18010	18006	5
	B021	484	482	473	480	6					
	B025										
	B026	0	0	0	0	0	22489	22096	19567	21384	1586
	B027	224.9	227.8	202.8	218.5	13.7	16067	17484	17905	17152	963
	B028	228.8	236.7	238.6	234.7	5.2	20162	18595	18535	19097	923
	B030										
	B031	0	0	0	0	0	0	0	0	0	0
	B035										
	B036	< 494	< 494	< 494			17863	17586	17870	17773	162
	B038						19500	19900	20100	19833	306
	B039										
	B042										
	B044										
	B045										
Community Results		Consensus Mean			195		Consensus Mean			18338	
		Consensus Standard Deviation			61		Consensus Standard Deviation			3477	
		Maximum			480		Maximum			21400	
		Minimum			0		Minimum			0	
		N			10		N			10	

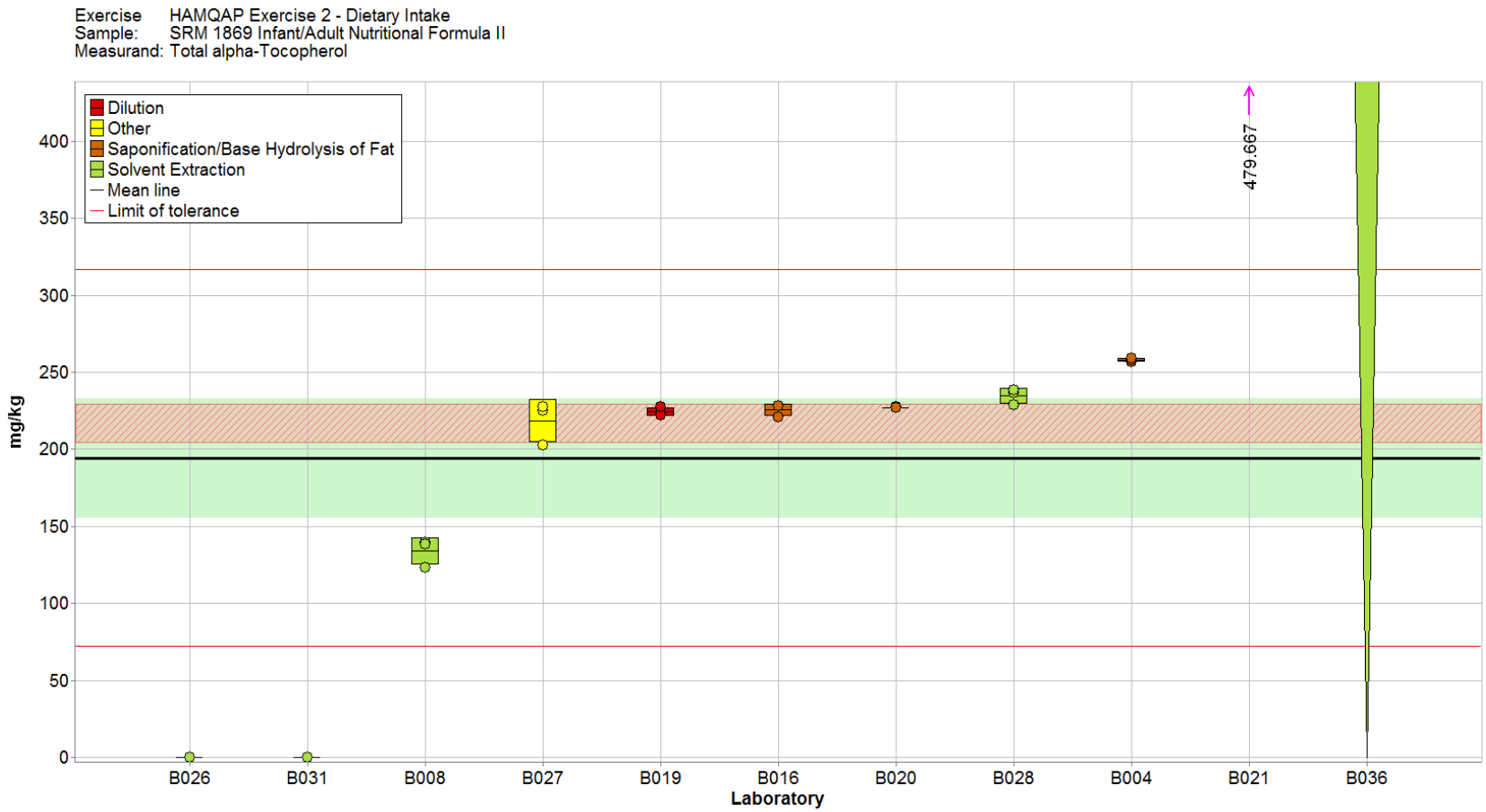

Figure 4-14. Total α-tocopherol in SRM 1869 Infant/Adult Nutritional Formula II (data summary view – sample preparation method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the sample preparation employed. The solid black 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

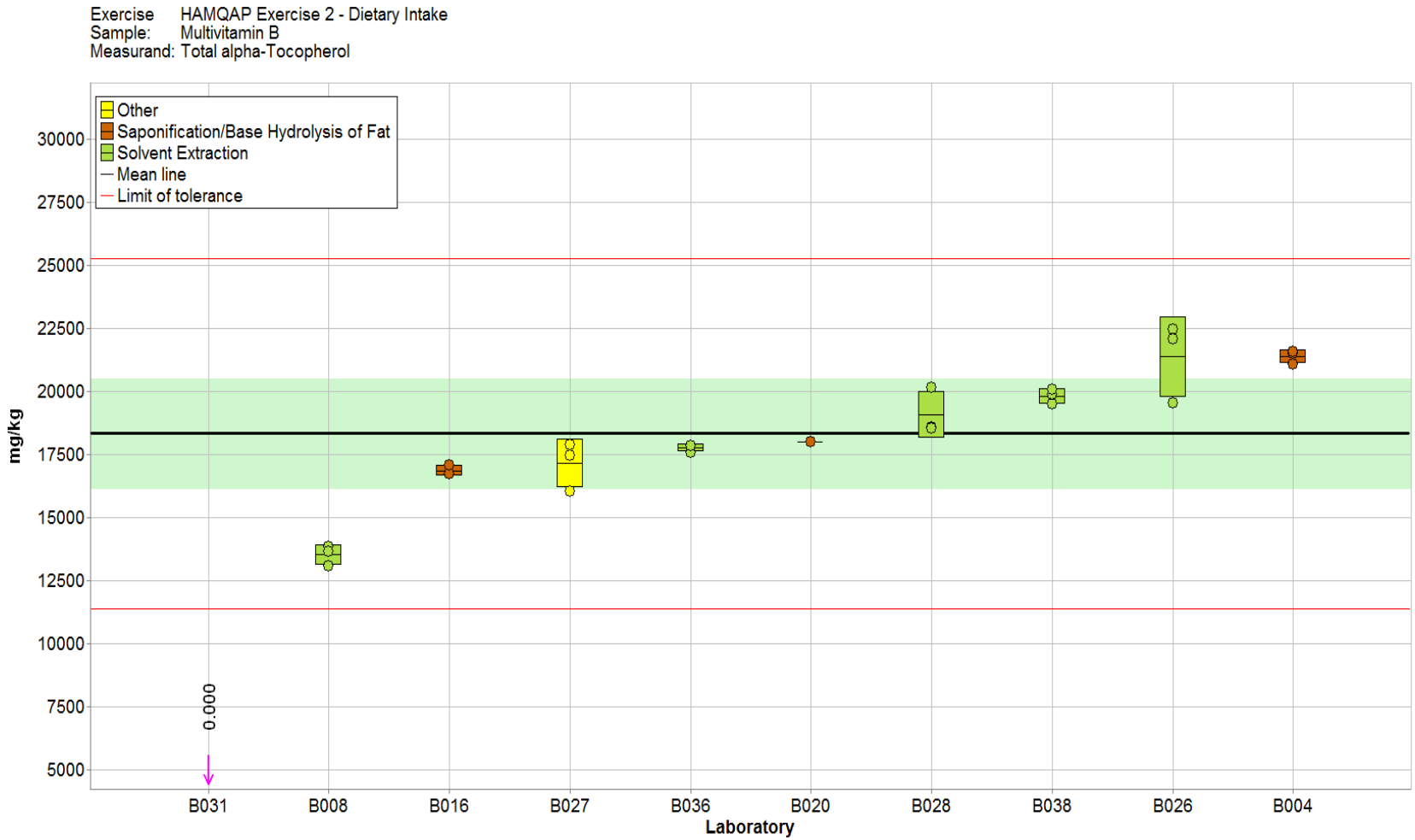

Figure 4-15. Total α-tocopherol in Multivitamin B (data summary view – sample preparation method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the sample preparation employed. The solid black 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 NIST value has not been determined in this material.

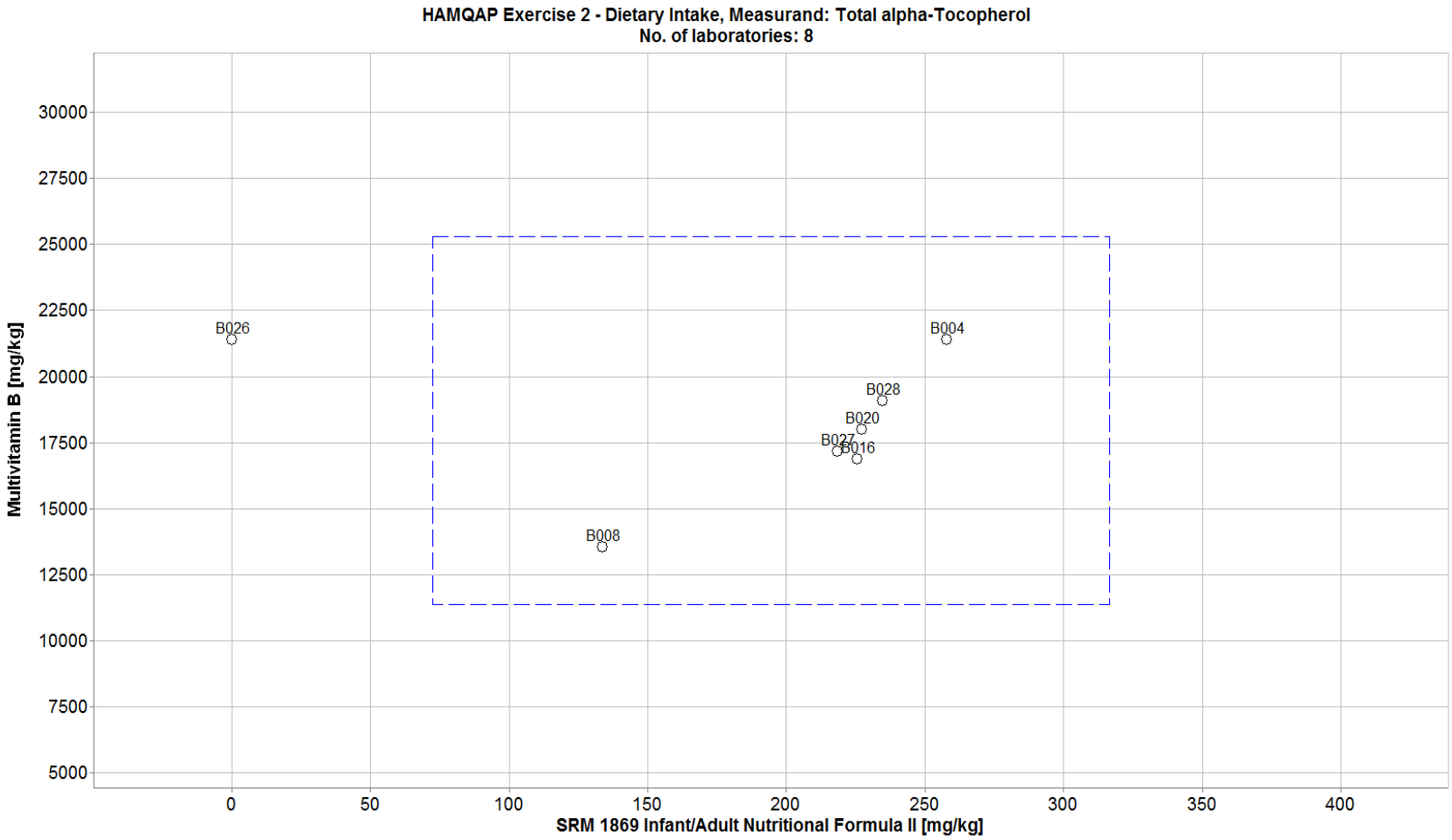

Figure 4-16. Laboratory means for total α-tocopherol in SRM 1869 Infant/Adult Nutritional Formula II and Multivitamin B (sample/sample comparison view). In this view, the individual laboratory mean for one sample (SRM 1869) is compared to the mean for a second sample (Multivitamin B). The dotted blue box represents the consensus range of tolerance for SRM 1869 (x-axis) and Multivitamin B (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 4-8. Data summary table for β-tocopherol in nutritional formula and multivitamin. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		beta-Tocopherol									
		SRM 1869 Infant/Adult Nutritional Formula II (mg/kg)					Multivitamin B (mg/kg)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				4.22	0.69					
	B001										
	B002										
	B006										
	B007										
	B008										
	B016	4.86	4.11	4.07	4.35	0.45	0	0	0	0	0
	B020										
	B021										
	B025										
	B026	0	0	0	0	0	0	0	0	0	0
	B031	39.48	40.45	42.02	40.65	1.28	0	0	0	0	0
	B035										
	B036										
	B038	0	0	0	0	0	0	0	0	0	0
	B039										
	B042										
	B044										
Community Results		Consensus Mean			1.45		Consensus Mean			0	
		Consensus Standard Deviation			6.35		Consensus Standard Deviation			0	
		Maximum			40.65		Maximum			0	
		Minimum			0		Minimum			0	
		N			4		N			4	

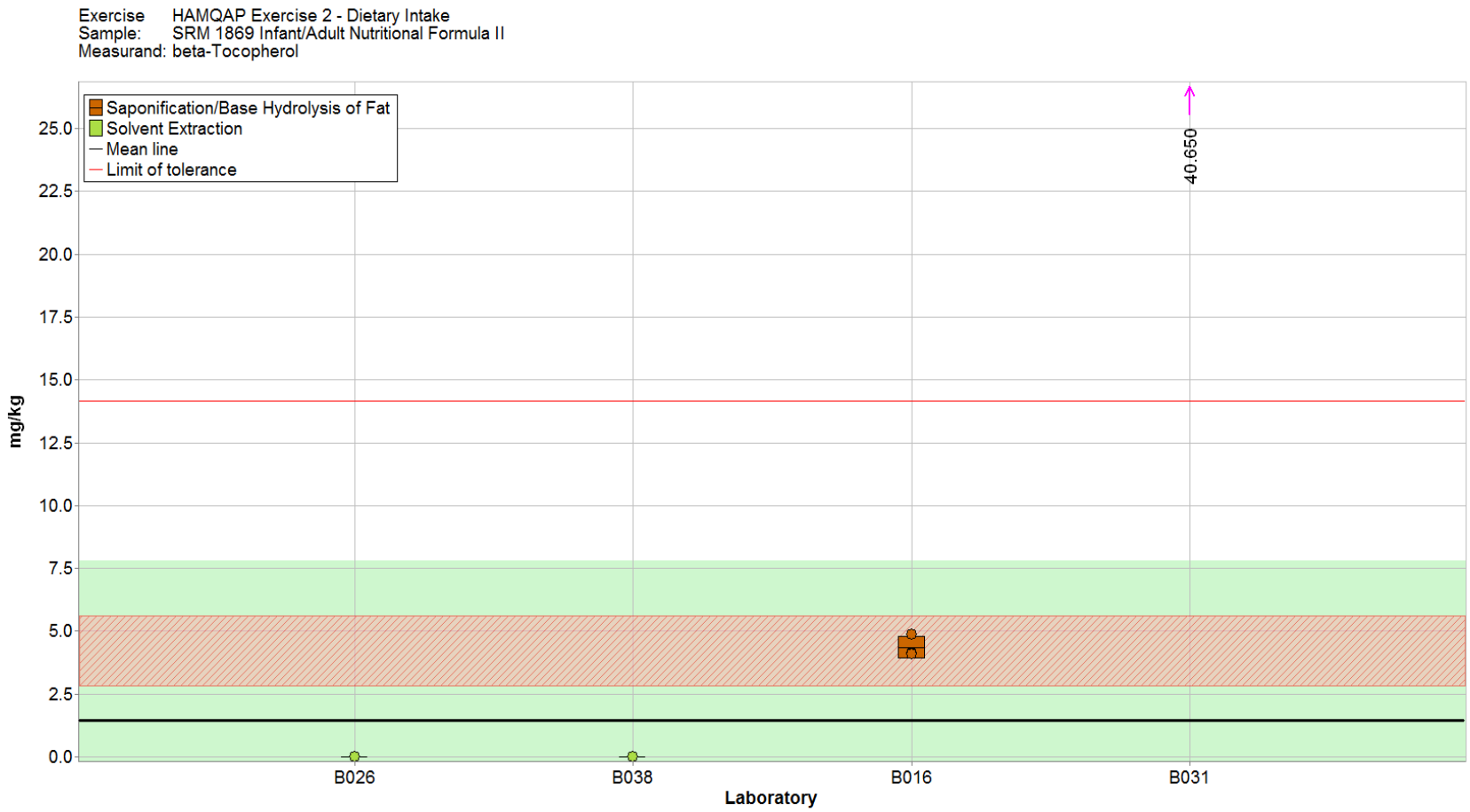

Figure 4-17. β-Tocopherol in SRM 1869 Infant/Adult Nutritional Formula II (data summary view – sample preparation method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the sample preparation employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

Table 4-9. Data summary table for γ-tocopherol in nutritional formula and multivitamin. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		gamma-Tocopherol									
		SRM 1869 Infant/Adult Nutritional Formula II (mg/kg)					Multivitamin B (mg/kg)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				99.4	5.1					
	B001										
	B002										
	B006										
	B007										
	B008										
	B016	112.0	111.0	108.0	110.3	2.1	0	0	0	0	0
	B020										
	B021										
	B025										
	B026	0	0	0	0	0	0	0	0	0	0
	B031	123.7	126.7	128.7	126.3	2.5	0	0	0	0	0
	B035										
	B036	< 95	< 95	< 95							
	B038	0	0	0	0	0	0	0	0	0	0
	B039										
	B042										
	B044										
	B046	105.2	103.7	105.2	104.7	0.9	0	0	0	0	0
Community Results		Consensus Mean			79.2		Consensus Mean			0	
		Consensus Standard Deviation			34.6		Consensus Standard Deviation			0	
		Maximum			126.3		Maximum			0	
		Minimum			0		Minimum			0	
		N			5		N			5	

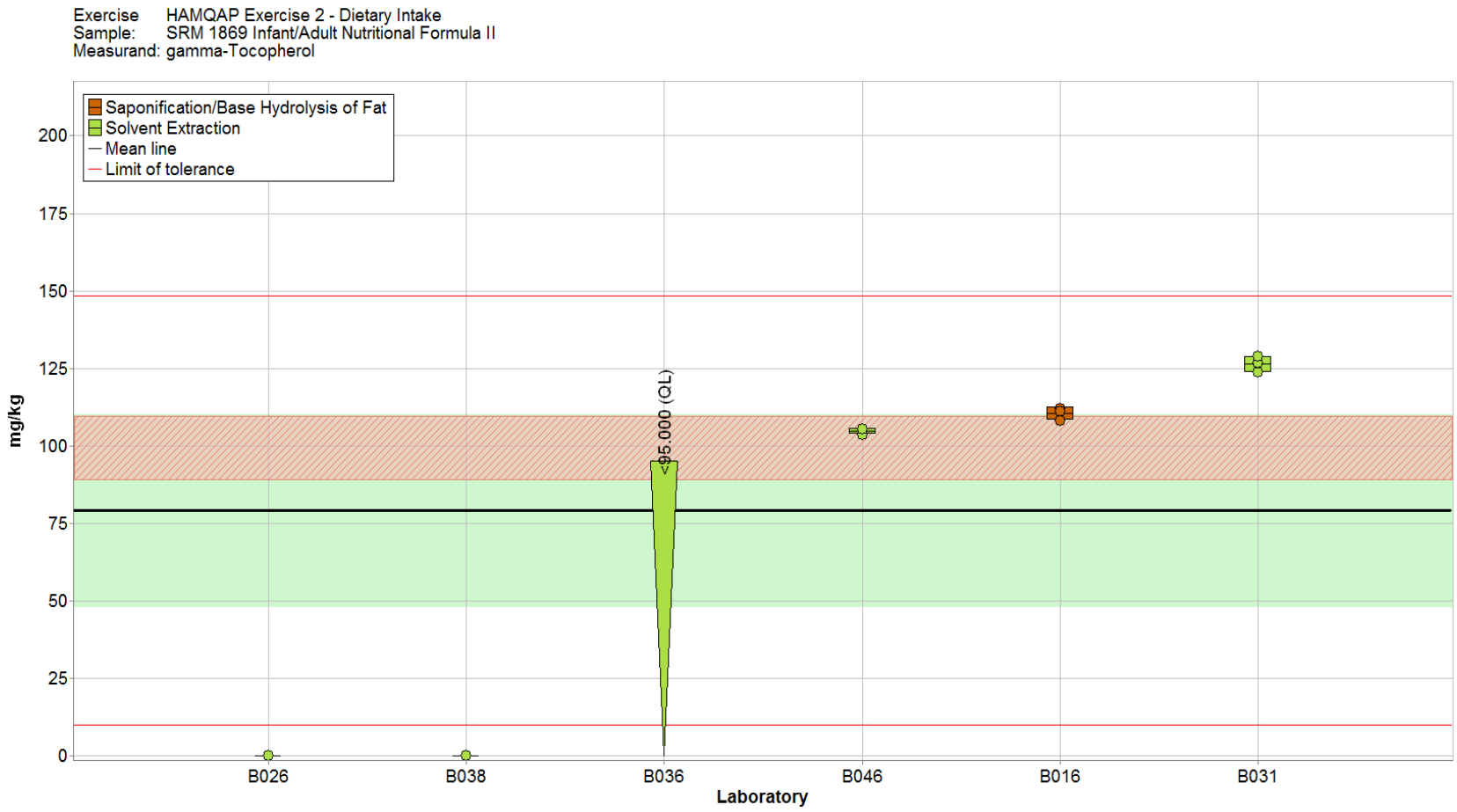

Figure 4-18. γ-Tocopherol in SRM 1869 Infant/Adult Nutritional Formula II (data summary view – sample preparation method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the sample preparation employed. The solid black 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

Table 4-10. Data summary table for β- plus γ-tocopherol in nutritional formula and multivitamin.

		gamma plus beta-Tocopherol									
		SRM 1869 Infant/Adult Nutritional Formula II (mg/kg)					Multivitamin B (mg/kg)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				103.6	5.1					
	B001										
	B002										
	B006										
	B007										
	B016										
	B017										
	B020										
	B021										
	B025										
	B026	0	0	0	0	0	0	0	0	0	0
	B030										
	B031	163.16	167.13	170.7	167.0	3.8	0	0	0	0	0
	B035										
	B036	< 95	< 95	< 95							
	B038	0	0	0	0	0	0	0	0	0	0
	B039										
	B042										
	B044										
Community Results		Consensus Mean			55.7		Consensus Mean			0	
		Consensus Standard Deviation			172		Consensus Standard Deviation			0	
		Maximum			167		Maximum			0	
		Minimum			0		Minimum			0	
		N			3		N			3	

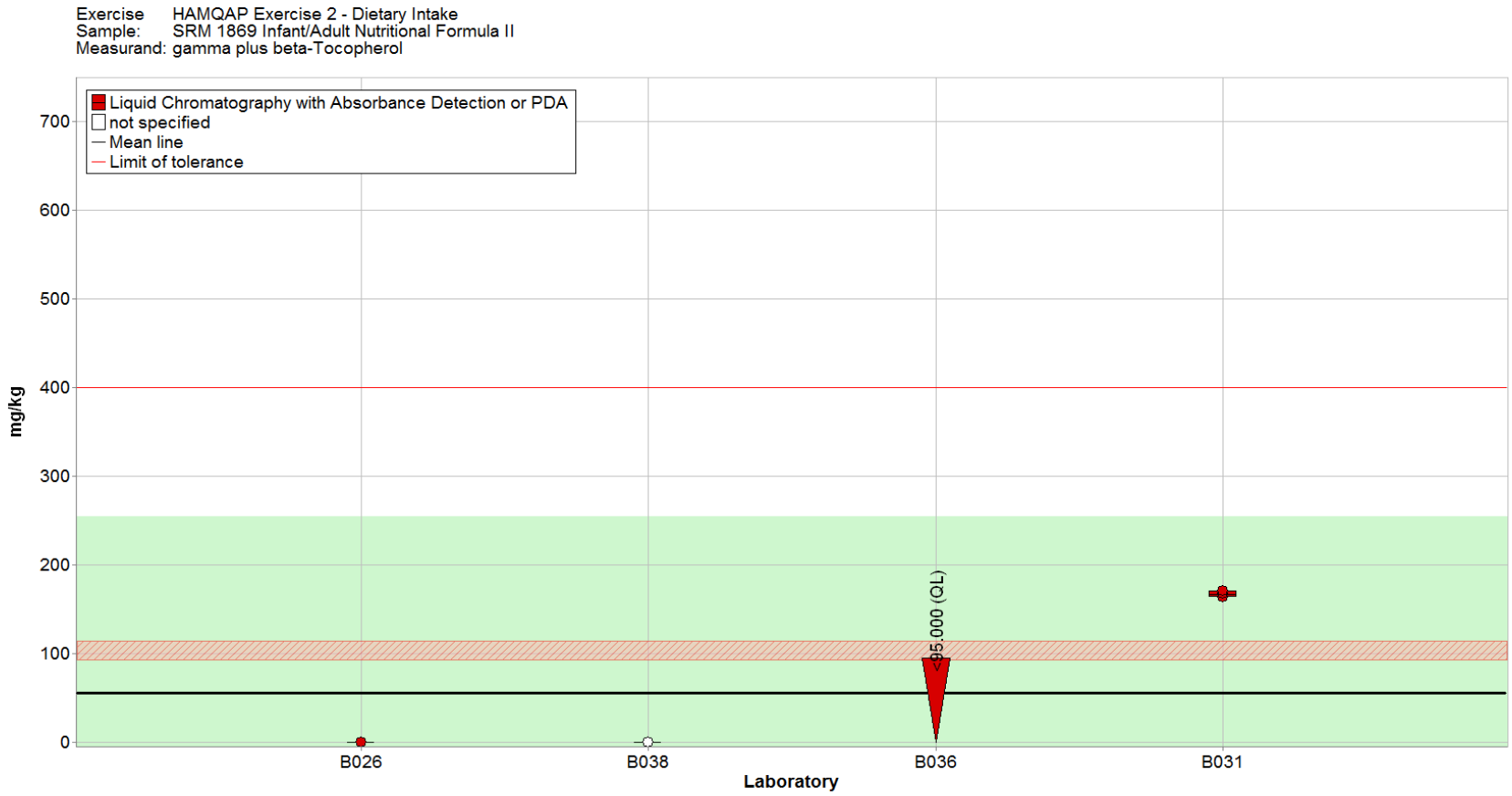

Figure 4-19. γ- plus β-Tocopherol in SRM 1869 Infant/Adult Nutritional Formula II (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

Human Metabolites Sample Information

Human Serum C. Participants were provided with three vials, each containing 1 mL of frozen human serum. Bovine thrombin and calcium chloride were added to convert the plasma to serum. The serum was dialyzed to remove bovine thrombin, calcium chloride, and anticoagulants. Salts were added back into the serum, and the material was pooled along with isotonic saline, blended, bottled in 1 mL aliquots, and stored at –80 °C. Before use, participants were instructed to allow the material to thaw at room temperature for at least 30 min prior to sampling, use the material immediately after thawing, gently mix the contents prior to removal of a test portion for analysis, and use a sample size appropriate for their usual in-house method of analysis. Participants were asked to avoid exposing the material to direct UV light, to store the material at or below –80 °C, and to prepare one sample and report one value from each vial provided. The approximate analyte levels were not reported to participants prior to the study. The NIST-determined values for total retinol, α-tocopherol, and γ- plus β-tocopherol in Human Serum C were assigned using results from two NIST LC methods and results from collaborating laboratories. The NIST-determined values and uncertainties for total retinol, α-tocopherol, and γ- plus β-tocopherol in Human Serum C are provided in the table below.

Analyte	NIST-Determined Mass Concentration in Human Serum C (µg/mL)
Total Retinol	0.341 ± 0.016
α-Tocopherol	6.53 ± 0.86
γ- plus β-Tocopherol	1.86 ± 0.16

Human Serum D. Participants were provided with three vials, each containing 1 mL of frozen human serum. Bovine thrombin and calcium chloride were added to convert the plasma to serum. The serum was dialyzed to remove bovine thrombin, calcium chloride, and anticoagulants. Salts were added back into the serum, and the material was pooled along with isotonic saline, blended, bottled in 1 mL aliquots, and stored at –80 °C. Before use, participants were instructed to allow the material to thaw at room temperature for at least 30 min prior to sampling, use the material immediately after thawing, gently mix the contents prior to removal of a test portion for analysis, and use a sample size appropriate for their usual in-house method of analysis. Participants were asked to avoid exposing the material to direct UV light, to store the material at or below –80 °C, and to prepare one sample and report one value from each vial provided. The approximate analyte levels were not reported to participants prior to the study. The NIST-determined values for total retinol, α-tocopherol, and γ- plus β-tocopherol were assigned using results from two NIST LC methods and results from collaborating laboratories. The NIST-determined values and uncertainties for total retinol, α-tocopherol, and γ- plus β-tocopherol in Human Serum D are provided in the table below.

Analyte	NIST-Determined Mass Concentration in Human Serum D (µg/mL)
Total Retinol	0.482 ± 0.030
α-Tocopherol	10.33 ± 0.14
γ- plus β-Tocopherol	1.432 ± 0.081

Human Metabolites Study Results

- Seventeen laboratories enrolled in this exercise and received samples. Not all laboratories measured and reported results for every analyte in the study.
  - The highest participation rates were for total retinol and total α-tocopherol, with 71 % and 65 % of laboratories returning results, respectively.
  - The consensus means for total retinol were within the target ranges for both samples, with between-laboratory variability of approximately 20 % RSD and 14 % RSD, respectively, for Human Serum C and Human Serum D.
  - The consensus means for total α-tocopherol were within the target ranges for both samples, with between-laboratory variability of approximately 11 % RSD.
  - The participation rate for γ- plus β-tocopherol was significantly lower, with only three of nine laboratories returning results (33 % participation). The between-laboratory variability for γ- plus β-tocopherol was good at 6 % and 7 % in Human Serum C and D, respectively.
  - Two of nine laboratories reported results for γ-tocopherol (22 % participation).
- Most laboratories used LC-absorbance, LC with photodiode array detection, or LC-fluorescence to determine total retinol in both serum samples. Two laboratories used unspecified methods to measure total retinol in the samples.
- To determine tocopherols in the samples, most laboratories used LC-absorbance, LC with photodiode array detection, LC-fluorescence, or LC-MS/MS.

Human Metabolites Technical Recommendations

The following recommendations are based on results obtained from the participants in this study. In some cases, too few data were reported to allow for meaningful conclusions to be drawn.

- For Human Serum C and D, a few laboratories reported data significantly outside of the target and consensus ranges. The use of appropriate calibration materials and quality assurance samples to establish that a method is in control and performing correctly may reduce the likelihood of outlying data. Quality assurance samples can be commercially available reference materials (CRMs, SRMs, or RMs) or prepared in-house.
- A linear calibration curve which surrounds the expected sample concentration values should be used for calculations. This curve should include both the lowest and highest expected concentration values of the sample solutions. Extrapolation of results beyond calibration curves may result in incorrect values.
- In the sample/sample comparison plot for total retinol (**Figure 4-22**), a linear trend is apparent. This type of trend often indicates a common error is present for both samples, frequently a calibration error.
- Some laboratories reported data for γ-tocopherol, while others reported data for γ- plus β-tocopherol. The NIST-determined values in these materials are the sum of γ- and β-tocopherol, based on LC methods where γ- and β-tocopherol are not chromatographically separated. Some commercial LC columns can separate these two analytes; however, optimization of the chromatographic method and confirming the identification of the analyte(s) would help assure the accuracy of measurements. Despite an apparent discrepancy between the identification of γ-tocopherol and γ- plus β-tocopherol, laboratories were within the consensus range of tolerance or the NIST range of tolerance.
- In general, all results should be checked closely to avoid calculation errors and to be sure that results are reported in the requested units and as the requested vitamer.

Table 4-11. Individualized data summary table (NIST) for vitamins A and E in human serum.

National Institute of Standards & Technology

HAMQAP Exercise 2 - 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*
beta-Tocopherol	Human Serum C	µg/mL						0				
beta-Tocopherol	Human Serum D	µg/mL						0				
delta-Tocopherol	Human Serum C	µg/mL						0				
delta-Tocopherol	Human Serum D	µg/mL						0				
gamma plus beta-Tocopherol	Human Serum C	µg/mL		1.86	0.08			3	1.59	0.10	1.86	0.16
gamma plus beta-Tocopherol	Human Serum D	µg/mL		1.432	0.041			3	1.16	0.08	1.432	0.081
gamma-Tocopherol	Human Serum C	µg/mL						2	1.77	0.25		
gamma-Tocopherol	Human Serum D	µg/mL						2	1.37	0.34		
Total alpha-Tocopherol	Human Serum C	µg/mL		6.53	0.43			11	7.03	0.75	6.53	0.86
Total alpha-Tocopherol	Human Serum D	µg/mL		10.33	0.07			10	10.6	1.1	10.33	0.14
Total Retinol	Human Serum C	µg/mL		0.341	0.008			10	0.345	0.068	0.341	0.016
Total Retinol	Human Serum D	µg/mL		0.482	0.015			9	0.475	0.068	0.482	0.030

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 4-12. Data summary table for total retinol in human serum. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		Total Retinol									
		Human Serum C (µg/mL)					Human Serum D (µg/mL)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				0.341	0.016				0.482	0.030
	B016										
	B017										
	B028	0.346	0.344	0.341	0.344	0.003	0.475	0.502	0.492	0.490	0.014
	B031										
	B034										
	B047	0.390	0.383	0.404	0.392	0.011	0.535	0.571	0.562	0.556	0.018
	B048	0.450	0.443	0.443	0.445	0.004					
	B050	0.377	0.377	0.372	0.375	0.003	0.506	0.520	0.541	0.522	0.018
	B051	0.19	0.20	0.20	0.20	0.01	0	0	0	0	0
	B053	0.315	0.316	0.323	0.318	0.004	0.457	0.459	0.453	0.456	0.003
	B054	0.368	0.373	0.341	0.361	0.017	0.435	0.494	0.486	0.472	0.032
	B056	0.36	0.36	0.37	0.36	0.01	0.52	0.53	0.52	0.52	0.01
	B057	0.271	0.308	0.308	0.296	0.021	0.461	0.427	0.445	0.444	0.017
Community Results		Consensus Mean			0.345		Consensus Mean			0.475	
		Consensus Standard Deviation			0.068		Consensus Standard Deviation			0.068	
		Maximum			0.445		Maximum			0.556	
		Minimum			0.20		Minimum			0.34	
		N			10		N			9	

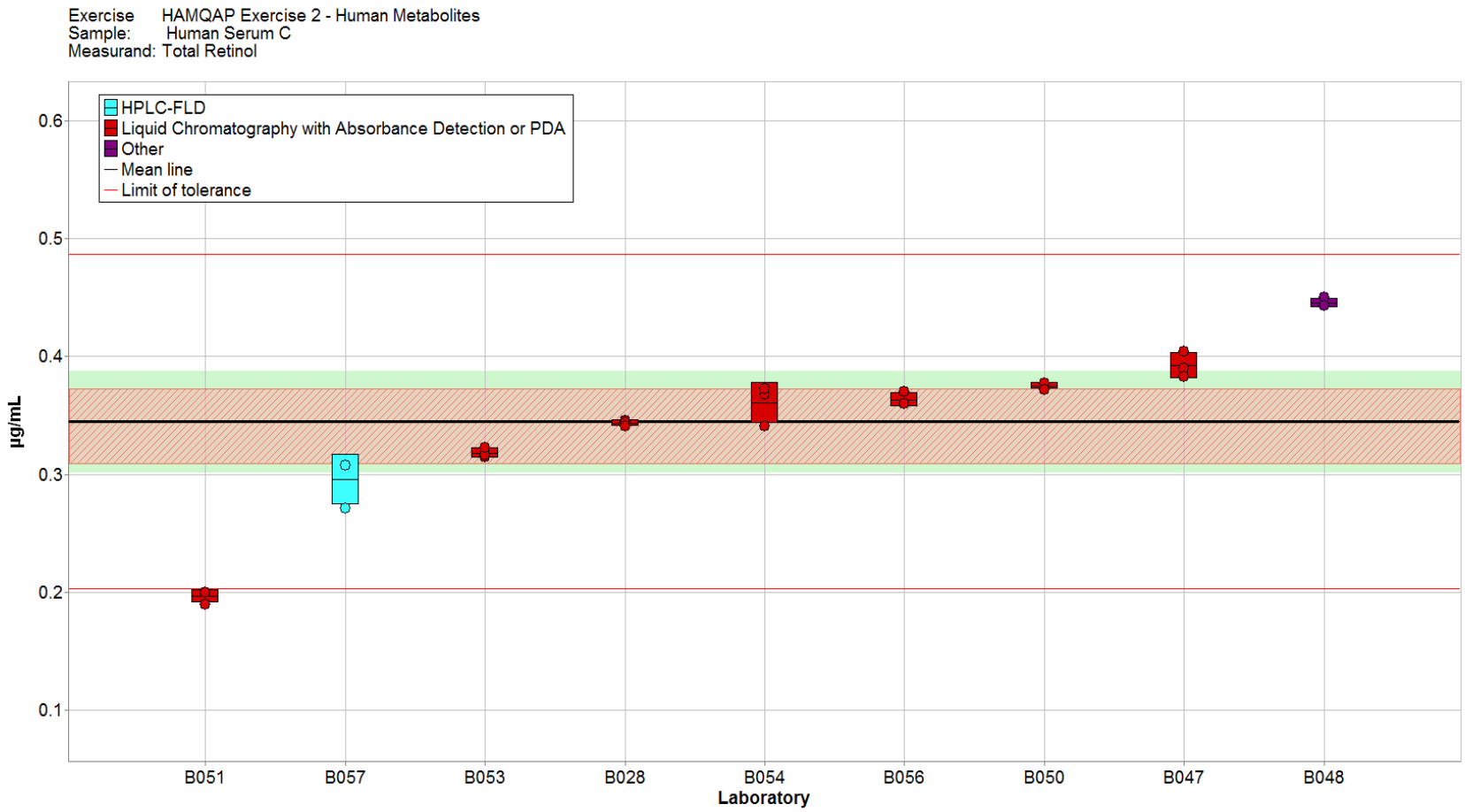

Figure 4-20. Total retinol in Human Serum C (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

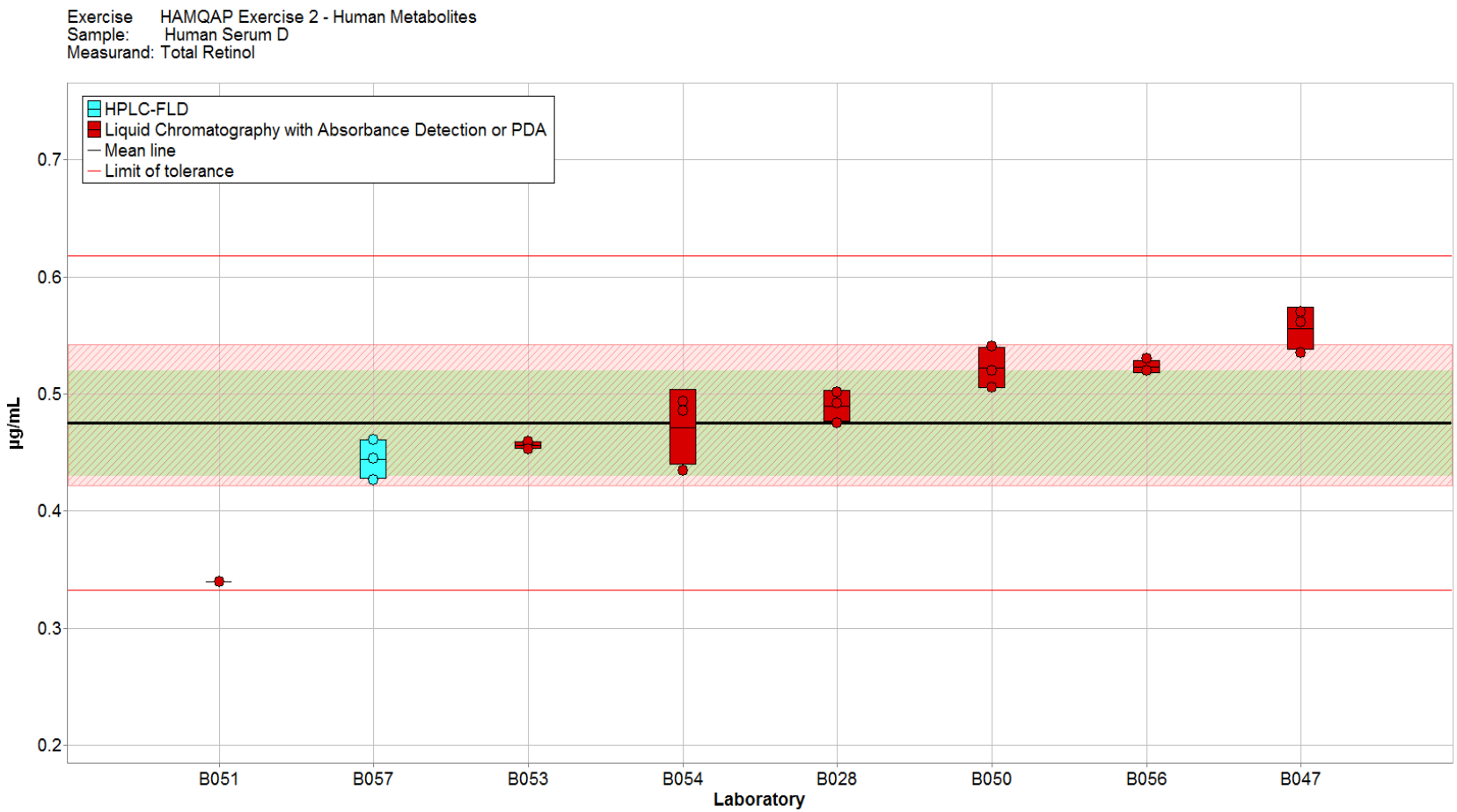

Figure 4-21. Total retinol in Human Serum D (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

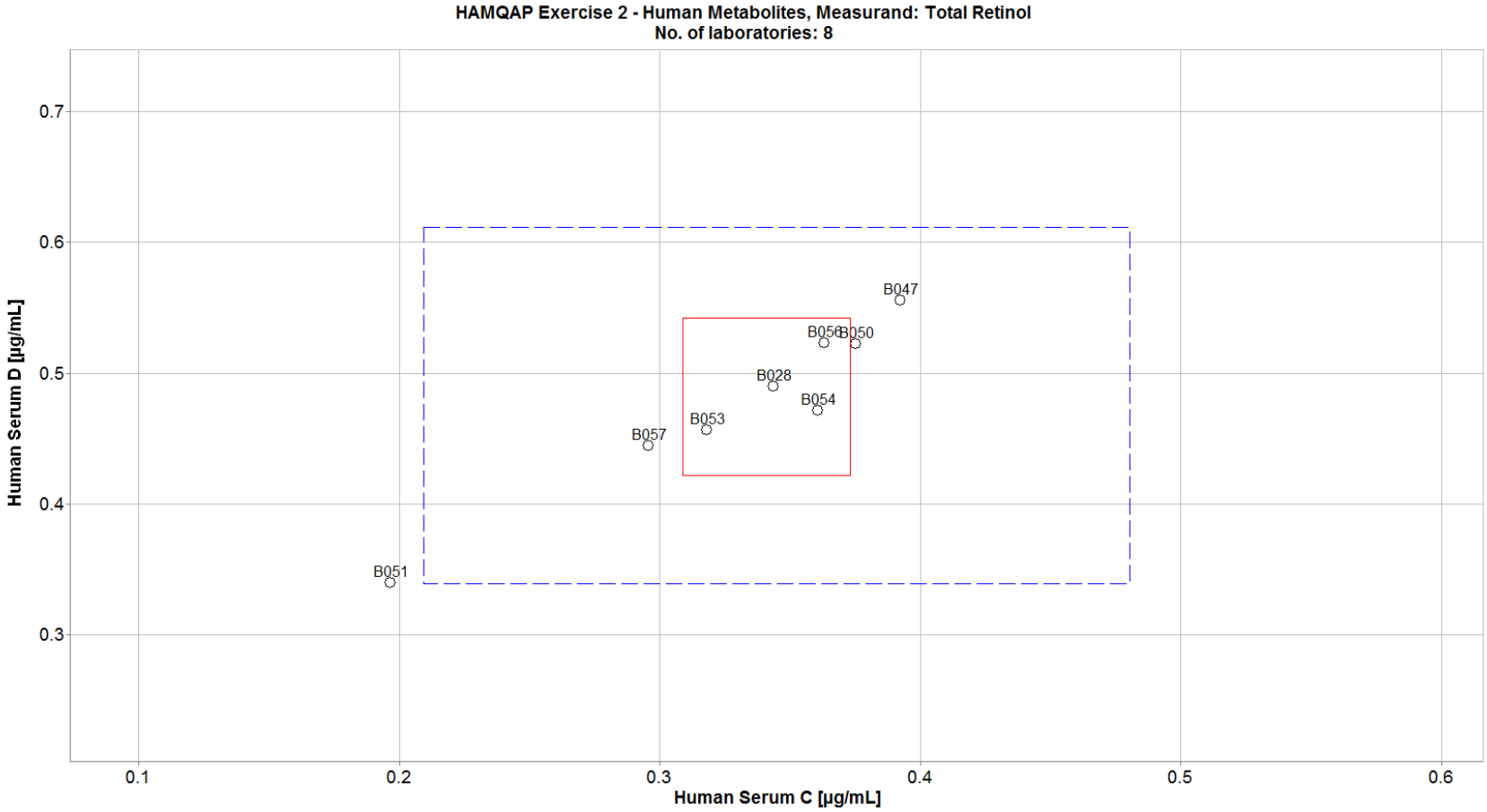

Figure 4-22. Laboratory means for total retinol in Human Serum C and Human Serum D (sample/sample comparison view). In this view, the individual laboratory mean for one sample (Human Serum C) is compared to the mean for a second sample (Human Serum D). The solid red box represents the NIST range of tolerance for the two samples, Human Serum C (x-axis) and Human Serum 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 Human Serum C (x-axis) and Human Serum 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 4-13. Data summary table for total α-tocopherol in human serum. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		Total alpha-Tocopherol									
		Human Serum C (µg/mL)					Human Serum D (µg/mL)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				6.53	0.86				10.33	0.14
	B008										
	B016										
	B017										
	B028	7.37	7.35	7.31	7.34	0.03	11.35	11.24	10.11	10.90	0.68
	B031										
	B034										
	B045										
	B047	8.01	5.98	7.75	7.24	1.11	12.51	9.53	10.75	10.93	1.50
	B048	9.90	9.75	9.67	9.77	0.12					
	B050	6.9	6.5	6.3	6.6	0.3	10.1	10.0	10.5	10.2	0.3
	B051	7.19	7.05	6.92	7.05	0.14	11.18	11.26	11.25	11.23	0.04
	B052	8.08	7.29	7.51	7.63	0.41	12.23	11.68	11.75	11.89	0.30
	B053	7.05	6.99	7.14	7.06	0.08	10.70	10.90	10.70	10.77	0.12
	B054	7.46	7.19	7.04	7.23	0.21	8.23	8.93	8.15	8.43	0.43
	B056	6.7	6.7	7.0	6.8	0.2	10.7	10.8	10.7	10.7	0.1
	B057	5.97	6.40	6.36	6.24	0.24	10.47	9.87	10.29	10.21	0.31
Community Results		Consensus Mean			7.03		Consensus Mean			10.56	
		Consensus Standard Deviation			0.75		Consensus Standard Deviation			1.14	
		Maximum			9.773		Maximum			11.89	
		Minimum			6.244		Minimum			8.43	
		N			11		N			10	

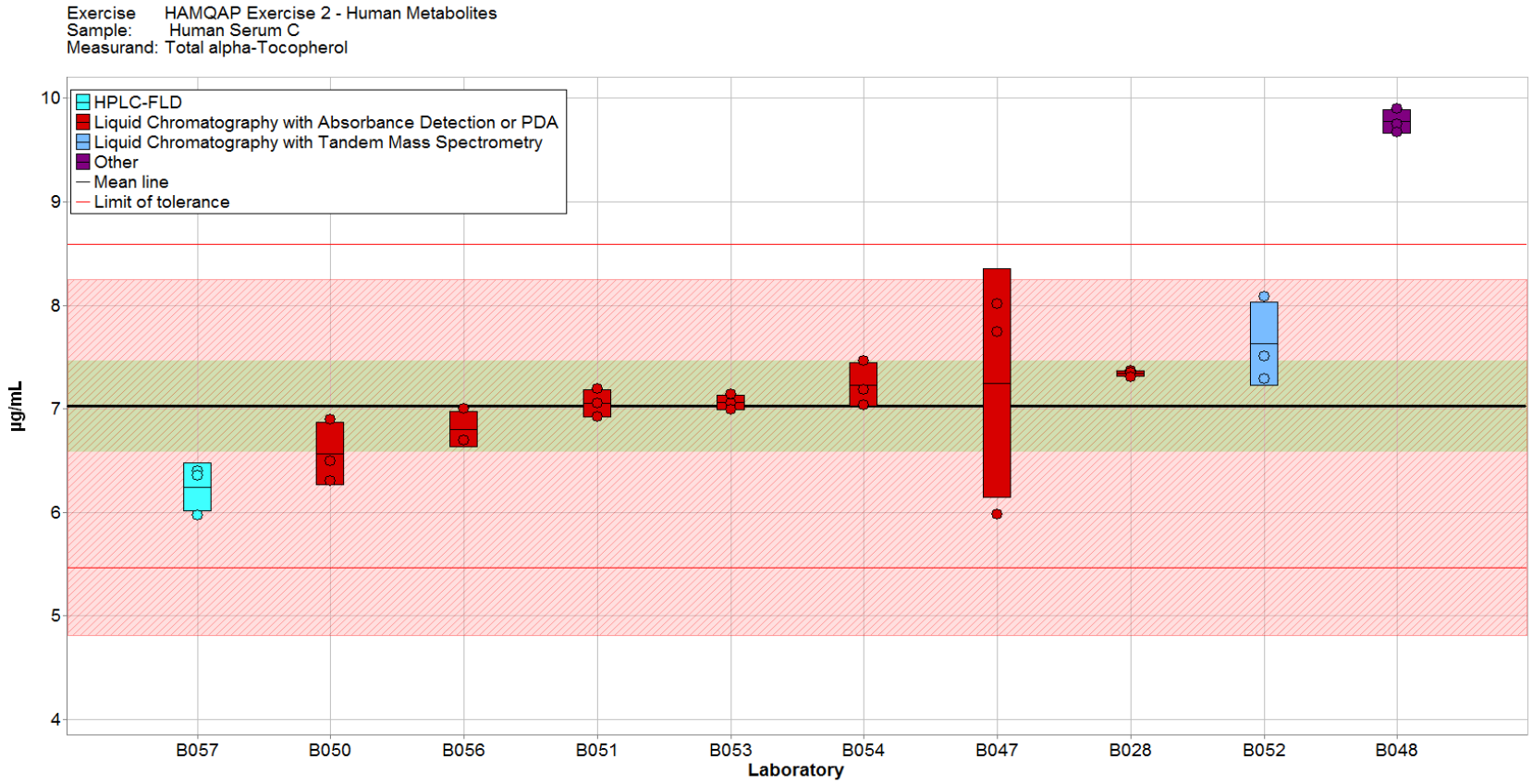

Figure 4-23. Total α-tocopherol in Human Serum C (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black 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 NIST-determined value bounded by its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

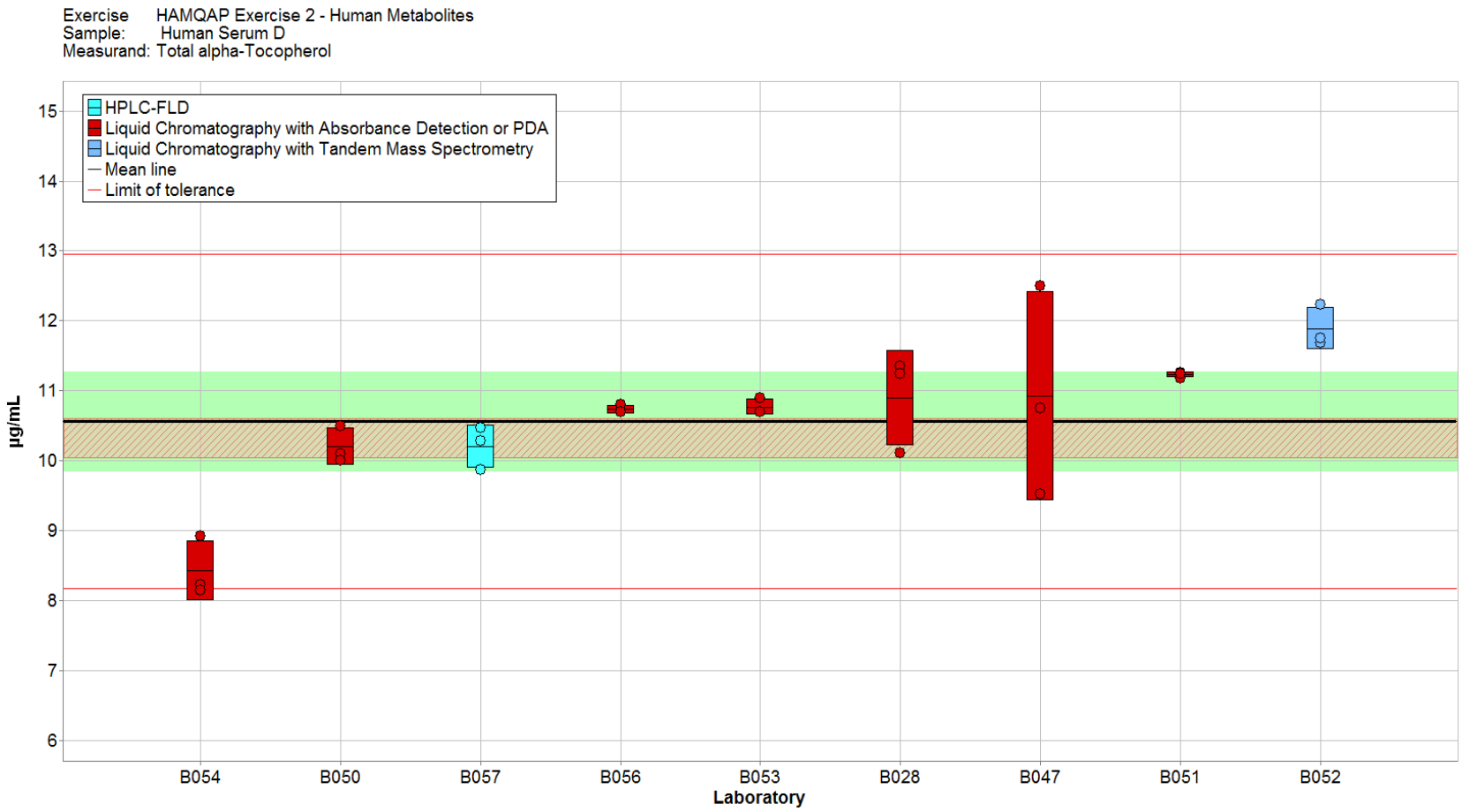

Figure 4-24. Total α-tocopherol in Human Serum D (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

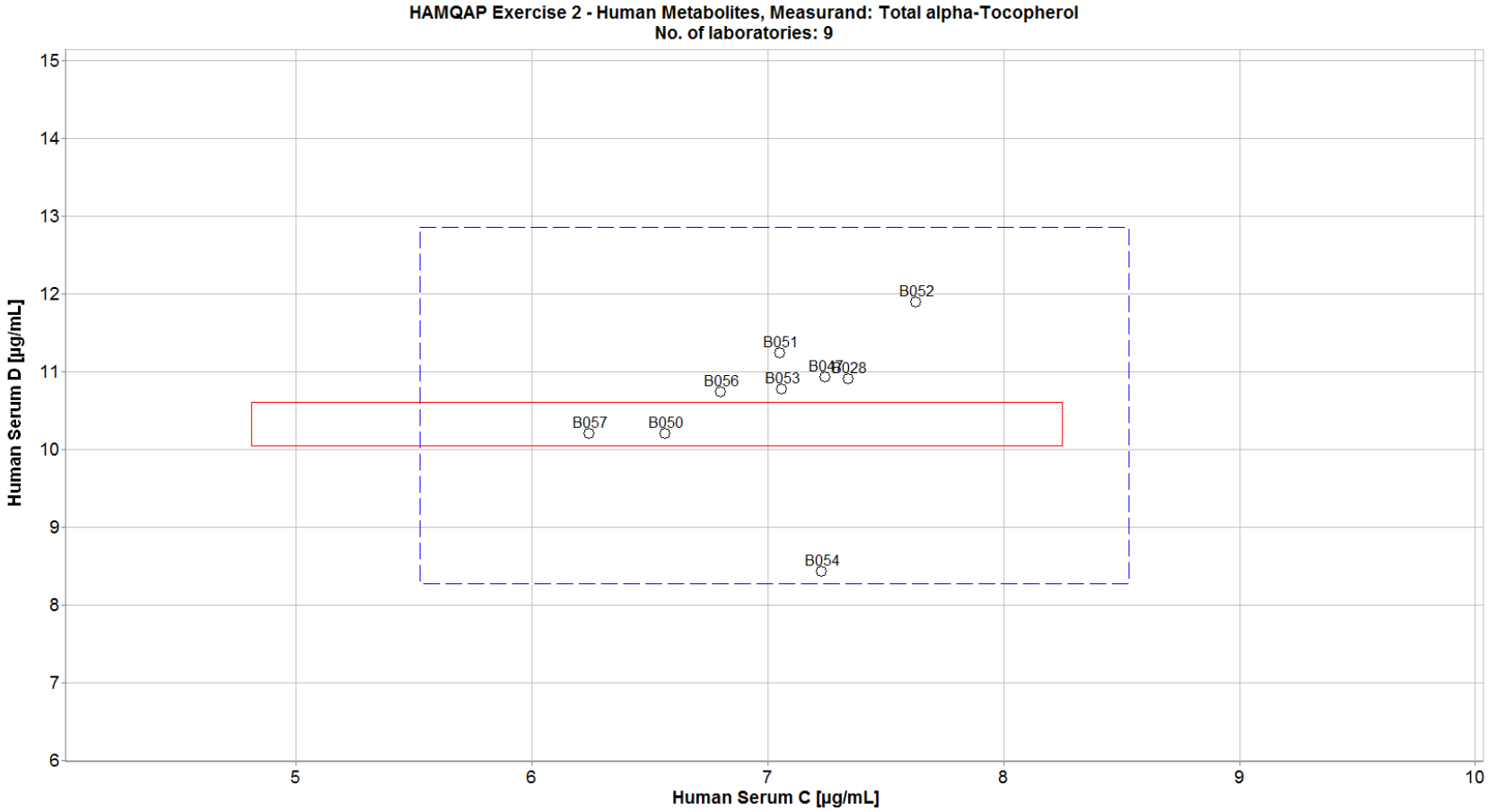

Figure 4-25. Laboratory means for total α-tocopherol in Human Serum C and in Human Serum D (sample/sample comparison view). In this view, the individual laboratory mean for one sample (Human Serum C) is compared to the mean for a second sample (Human Serum D). The solid red box represents the NIST range of tolerance for the two samples, Human Serum C (x-axis) and Human Serum 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 the samples, calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

Table 4-14. Data summary table for γ-tocopherol in human serum.

		gamma-Tocopherol									
		Human Serum C (µg/mL)					Human Serum D (µg/mL)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target										
	B008										
	B016										
	B031										
	B034										
	B047	1.77	1.36	1.80	1.64	0.24	1.35	1.05	1.34	1.25	0.17
	B053										
	B056	1.9	1.9	1.9	1.9	0	1.5	1.5	1.5	1.5	0
	B057										
Community Results		Consensus Mean			1.77		Consensus Mean			1.37	
		Consensus Standard Deviation			0.25		Consensus Standard Deviation			0.34	
		Maximum			1.9		Maximum			1.5	
		Minimum			1.64		Minimum			1.25	
		N			2		N			2	

Table 4-15. Data summary table for γ- plus β-tocopherol in human serum.

		gamma plus beta-Tocopherol									
		Human Serum C (µg/mL)					Human Serum D (µg/mL)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				1.86	0.16				1.43	0.08
	B016										
	B017										
	B031										
	B034										
	B047										
	B053										
	B054	1.86	1.80	1.86	1.84	0.03	1.01	1.05	1.00	1.02	0.02
	B057	1.47	1.52	1.53	1.50	0.03	1.25	1.20	1.24	1.23	0.03
Community Results		Consensus Mean			1.59		Consensus Mean			1.16	
		Consensus Standard Deviation			0.10		Consensus Standard Deviation			0.08	
		Maximum			1.84		Maximum			1.23	
		Minimum			1.50		Minimum			1.02	
		N			3		N			3	

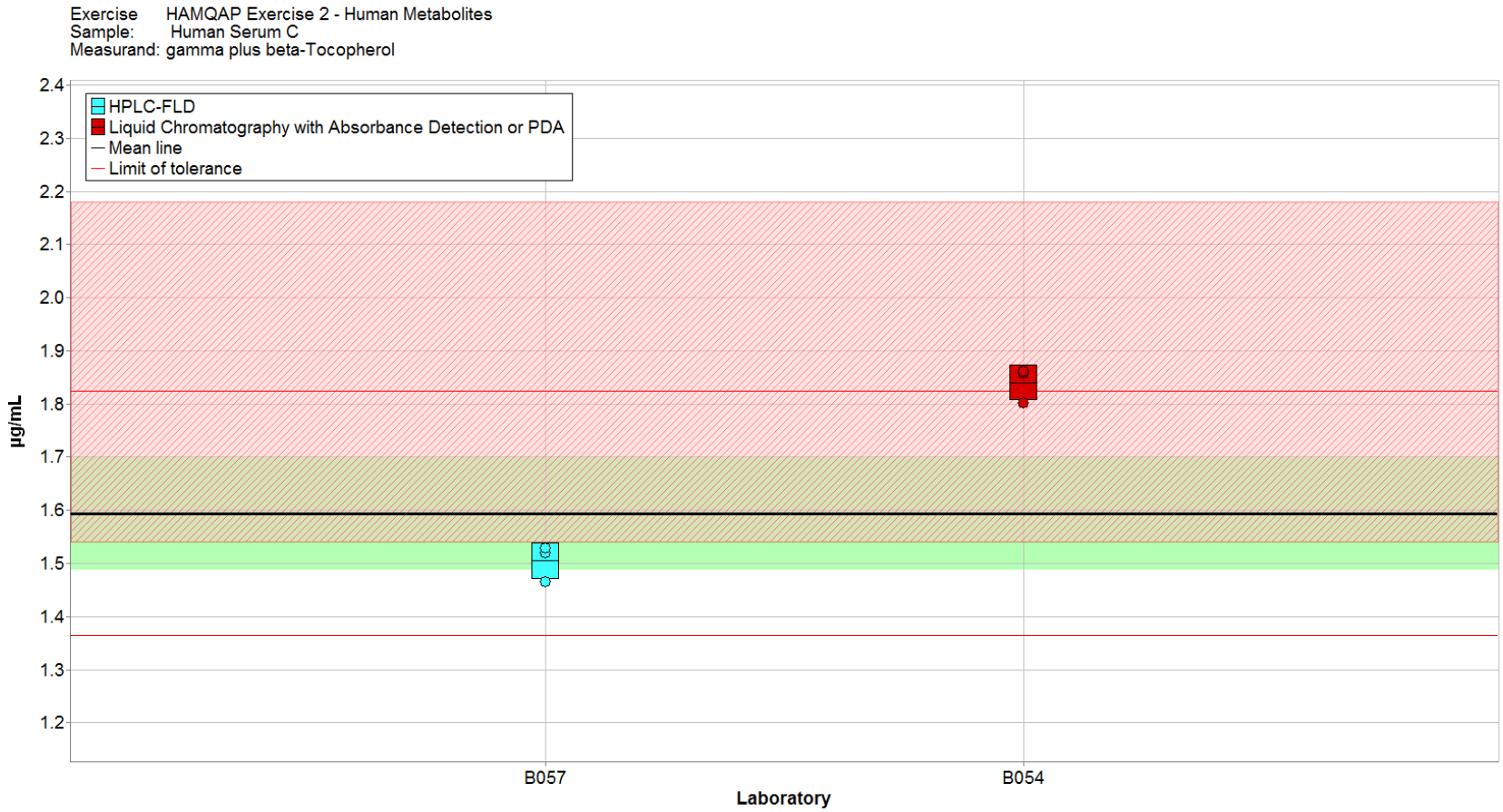

Figure 4-26. γ- plus β-tocopherol in Human Serum C (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

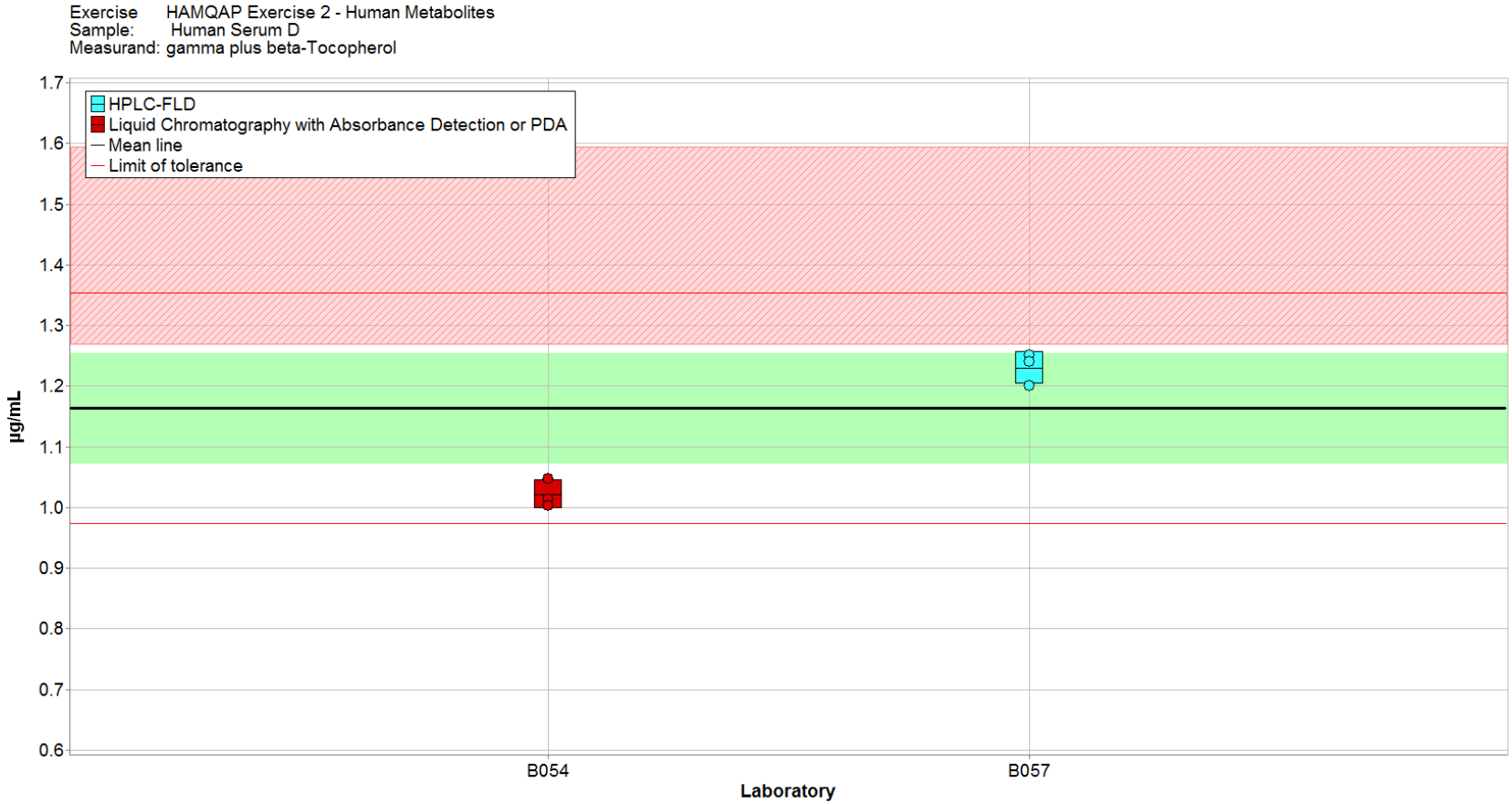

Figure 4-27. γ- plus β-tocopherol in Human Serum D (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

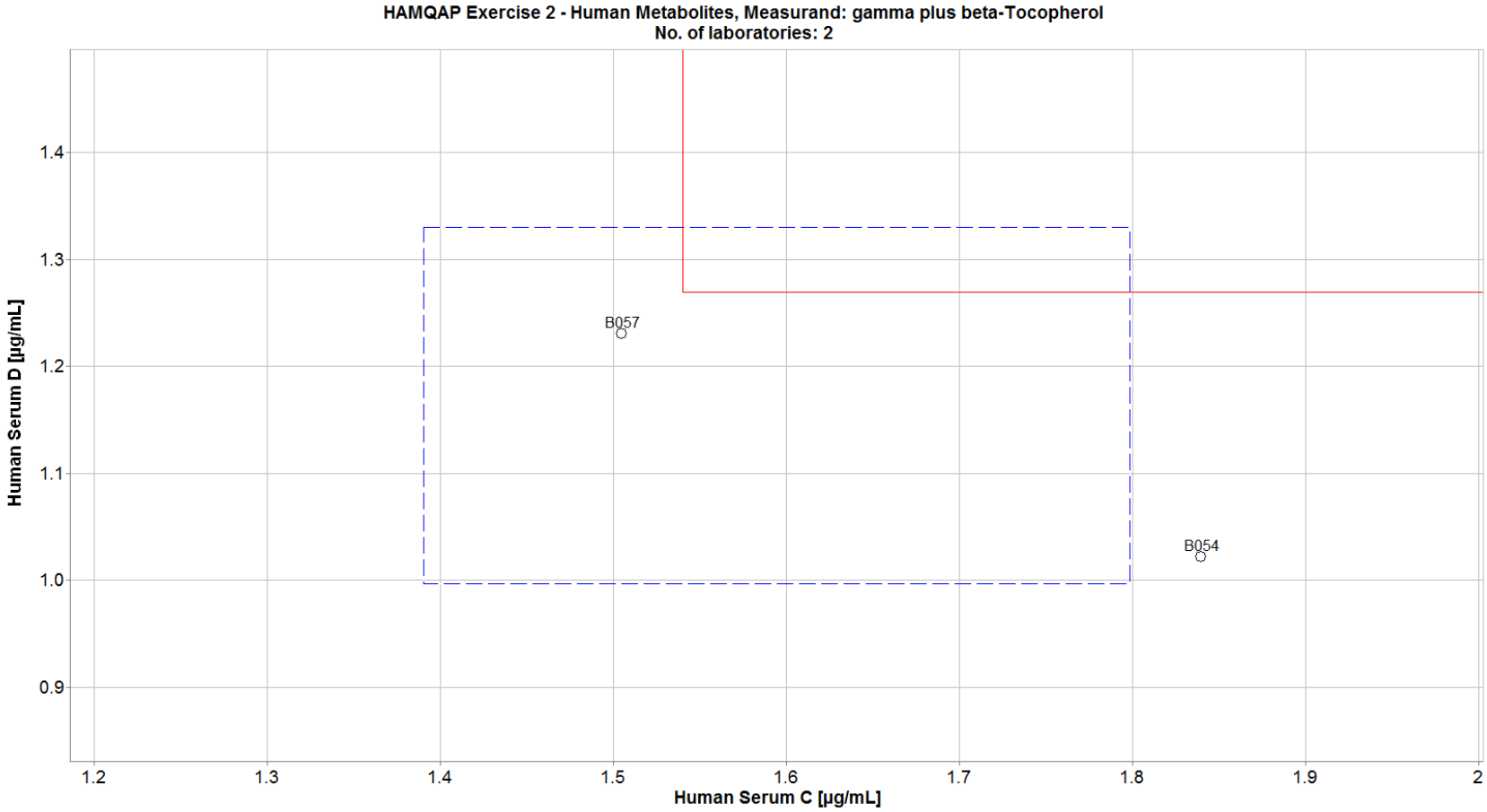

Figure 4-28. Laboratory means for γ- plus β-tocopherol in Human Serum C and in Human Serum D (sample/sample comparison view). In this view, the individual laboratory mean for one sample (Human Serum C) is compared to the mean for a second sample (Human Serum D). The solid red box represents the NIST range of tolerance for the two samples, Human Serum C (x-axis) and Human Serum 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 the samples, calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

Fat-Soluble Vitamins Overall Study Comparison

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

- Food laboratories have extensive experience in measuring total vitamins A and E in various food commodities, typically using a saponification or hydrolysis approach. With improvements in technology for sample preparation, separation, and detection, many laboratories are interested in measuring individual forms of each vitamin (fortified and endogenous, esterified, etc.) to better understand the health impact of foods and supplements. The biggest challenge for these laboratories seems to be in understanding the forms of each vitamin being measured and how to report the values appropriately.
- Clinical laboratories demonstrated good performance in the determination of total retinol and total alpha-tocopherol in serum. Better measurement performance of clinical laboratories is expected for these two analytes given the long history of vitamin A and E measurements and the existence of numerous interlaboratory comparisons, proficiency tests, and reference materials with values assigned for fat-soluble vitamins in human serum.
- Clinical laboratories had lower participation for the determination of γ-tocopherol. The discrepancy among data reported for γ-tocopherol and β- plus γ-tocopherol indicates that measurement challenges still exist in the clinical community. This could be an area of future study.

SECTION 5: FATTY ACIDS

Study Overview

In this study, participants were asked to measure 21 fatty acids (as total fatty acids) in three dietary intake samples and in two human metabolite samples. Total fatty acids are defined as the fatty acid content after acid and/or base hydrolysis of the sample that converts the lipids into their individual fatty acid constituents (typically as fatty acid methyl esters). For the dietary intake study, participants were provided with three materials, NIST SRM 1845a Whole Egg Powder, Palm Oil Powder, and Spirulina. For the human metabolites study, participants were provided with two materials, NIST SRM 1953 Organic Contaminants in Non-Fortified Human Milk, and a lyophilized human serum sample (Human Serum E). Participants were asked to use in-house analytical methods to determine the mass fractions (mg/g as received) of total fatty acids for the dietary intake study and the mass concentrations (µmol/L as reconstituted) of total fatty acids for the human metabolites study. Fatty acids from the diet can have beneficial and detrimental effects on human health depending on the degree and type of saturation and the fatty acid content of human serum and human milk can serve as indicators of health status and diet quality. The dietary intake samples have varying phospholipid content and were chosen to identify potential measurement biases with samples based on the relative fat composition (triglyceride or phospholipid). The Human Serum E sample consists of lyophilized material collected in the late 1980s,[7] and was included to gather information on trans-fatty acid measurements. Trans-fatty acids, which were listed as not generally recognized as safe (GRAS) by the U.S. Food and Drug Administration in 2013,[8] no longer regularly appear in the U.S. food supply and therefore are not found in human serum samples collected from U.S. subjects.

Dietary Intake Sample Information

Whole Egg Powder. Participants were provided with three packets, each containing 10 g of whole powdered egg. Before use, participants were instructed to allow the powder to warm to room temperature, to thoroughly mix the contents by shaking the unopened packet prior to removal of a test sample for analysis, and to use a sample size of 0.5 g to 0.8 g. Participants were asked to store the material at 4 °C in the original unopened packets before use, and to prepare one sample and report one value from each packet provided. The approximate analyte levels were not reported to the participants prior to the study. The NIST-determined values for the individual total fatty acids in SRM 1845a were assigned using results from NIST using pressurized fluid extraction (PFE) followed by gas chromatography with flame ionization detection (GC-FID) and Soxhlet extraction followed by gas chromatography with mass spectrometry (GC-MS). The NIST-determined values and uncertainties for the individual total fatty acids in SRM 1845a are listed in the table below.

[7] Rasberry, S.D. Z. *Anal. Chem.* (1988) 332: 528. https://doi.org/10.1007/BF00472636.

[8] Department of Health and Human Services. Tentative determination regarding partially hydrogenated oils; request for comments and for scientific data and information. *Fed Regist* 2013;78:67169-67175

Analyte	NIST-Determined Mass Fraction in SRM 1845a (mg/g)
Total Myristic Acid (C14:0)	0.1094 ± 0.0048
Total Myristoleic Acid (C14:1 n-5)	0.0185 ± 0.0008
Total Palmitic Acid (C16:0)	8.22 ± 0.26
Total Palmitoleic Acid (C16:1)	0.831 ± 0.023
Total Stearic Acid (C18:0)	2.802 ± 0.095
Total *cis*-Vaccenic Acid (C18:1 n-7)	0.532 ± 0.015
Total Oleic Acid (C18:1 n-9)	11.0 ± 1.4
Total Linoleic Acid (C18:2 n-6)	5.43 ± 0.12
Total α-Linolenic Acid (C18:3 n-3)	0.1643 ± 0.0047
Total γ-Linolenic Acid (C18:3 n-6)	0.0452 ± 0.0018
Total Arachidonic Acid (C20:4 n-6)	0.643 ± 0.017
Total DPA (C22:5 n-3)	0.0202 ± 0.0007
Total DHA (C22:6 n-3)	0.1701 ± 0.0077

Palm Oil Powder. Participants were provided with three packets, each containing approximately 3 g of commercial palm oil powder. The commercial powder was heat-sealed inside 4 mil polyethylene bags, which were then sealed inside nitrogen-flushed aluminized plastic bags along with two packets of silica gel. Before use, participants were instructed to allow the powder to warm to room temperature, to mix the contents of the packet thoroughly, and to use a sample size appropriate for their usual in-house methods. Participants were asked to store the material at 4 °C in the original unopened packets, and to prepare one sample and report one value from each packet provided. The approximate analyte levels were not reported to the participants prior to the study, and target values for the individual total fatty acids in the palm oil powder have not been determined.

Spirulina. Participants were provided with three packets, each containing approximately 3 g of commercial spirulina powder. The commercial powder was heat-sealed inside 4 mil polyethylene bags, which were then sealed inside nitrogen-flushed aluminized plastic bags along with two packets of silica gel. Before use, participants were instructed to allow the powder to warm to room temperature, to mix the contents of the packet thoroughly, and to use a sample size appropriate for their usual in-house methods. Participants were asked to store the material at 4 °C in the original unopened packets, and to prepare one sample and report one value from each packet provided. The approximate analyte levels were not reported to the participants prior to the study, and target values for the individual total fatty acids in the spirulina powder have not been determined.

Dietary Intake Study Results

- Fifteen to twenty laboratories enrolled in this exercise to measure total fatty acids. The table below lists the participation statistics for total fatty acids in the dietary intake samples.

Analyte	Number of Laboratories Requesting Samples	Number of Laboratories Reporting Results (Percent Participation) SRM 1845a Whole Egg Powder	Palm Oil Powder	Spirulina
Total Caprylic Acid (C8:0)	17	4 (24 %)	4 (24 %)	6 (35 %)
Total Capric Acid (C10:0)	17	4 (24 %)	5 (29 %)	6 (35 %)
Total Lauric Acid (C12:0)	18	6 (33 %)	5 (28 %)	9 (50 %)
Total Myristic Acid (C14:0)	19	8 (42 %)	7 (37 %)	9 (47 %)
Total Myristoleic Acid (C14:1 n-5)	16	5 (31 %)	3 (19 %)	4 (25 %)
Total Palmitic Acid (C16:0)	19	8 (42 %)	8 (42 %)	9 (47 %)
Total Palmitoleic Acid (C16:1)	18	7 (39 %)	7 (39 %)	8 (44 %)
Total Stearic Acid (C18:0)	20	9 (45 %)	9 (45 %)	11 (55 %)
Total *cis*-Vaccenic Acid (C18:1 n-7)	15	5 (33 %)	3 (20 %)	5 (33 %)
Total Oleic Acid (C18:1 n-9)	20	9 (45 %)	9 (45 %)	11 (55 %)
Total Linoleic Acid (C18:2 n-6)	20	9 (45 %)	9 (45 %)	11 (55 %)
Total *trans*-Vaccenic Acid (C18:1 n-7t)	15	2 (13 %)	2 (13 %)	3 (20 %)
Total Elaidic Acid (C18:1 n-9t)	15	4 (27 %)	3 (20 %)	3 (20 %)
Total α-Linolenic Acid (C18:3 n-3)	20	9 (45 %)	6 (30 %)	11 (55 %)
Total γ-Linolenic Acid (C18:3 n-6)	19	9 (47 %)	8 (42 %)	9 (47 %)
Total Arachidic Acid (C20:0)	15	4 (27 %)	2 (13 %)	4 (27 %)
Total Dihomo-γ-Linolenic Acid (C20:3 n-6)	16	2 (13 %)	2 (13 %)	2 (13 %)
Total Arachidonic Acid (C20:4 n-6)	17	6 (35 %)	3 (18 %)	4 (24 %)
Total EPA (C20:5 n-3)	20	5 (25 %)	5 (25 %)	6 (30 %)
Total DPA (C22:5 n-3)	18	4 (22 %)	2 (11 %)	3 (17 %)
Total DHA (C22:6 n-3)	20	9 (45 %)	5 (25 %)	6 (30 %)

- The consensus means for most of the fatty acids in SRM 1845a Whole Egg Powder were below the target ranges, but the consensus ranges overlapped with the target ranges (see table below). The exceptions included total myristic acid and oleic acid, with consensus values within the target ranges. The between-laboratory variability is also summarized below and ranged from good for dihomo-γ-linolenic acid (13 % RSD) to very high for caprylic acid and lauric acid (> 200 % RSD). For most fatty acids in this sample, the between-laboratory variability was over 75 % RSD.

Performance Summary for SRM 1845a Whole Egg Powder

Analyte	Relative Position of Consensus and Target Ranges	Between-Laboratory Variability
Total Caprylic Acid (C8:0)	Not Applicable	264 %
Total Capric Acid (C10:0)	Not Applicable	NA
Total Lauric Acid (C12:0)	Consensus mean *slightly below* target range Consensus range *overlaps* target range	300 %
Total Myristic Acid (C14:0)	Consensus mean *within* target range Consensus range *overlaps* target range	79 %
Total Myristoleic Acid (C14:1 n-5)	Consensus mean *below* target range Consensus range *does not overlap* target range	104 %
Total Palmitic Acid (C16:0)	Consensus mean *slightly below* target Consensus range *overlaps* target range	116 %
Total Palmitoleic Acid (C16:1)	Consensus mean *below* target range Consensus range *slightly overlaps* target range	122 %
Total Stearic Acid (C18:0)	Consensus mean *below* target range Consensus range *overlaps* target range	100 %
Total *cis*-Vaccenic Acid (C18:1 n-7)	Consensus mean *above* target range Consensus range *overlaps* target range	51 %
Total Oleic Acid (C18:1 n-9)	Consensus mean *within* target range Consensus range *overlaps* target range	86 %
Total Linoleic Acid (C18:2 n-6)	Consensus mean *below* target range Consensus range *overlaps* target range	89 %
Total *trans*-Vaccenic Acid (C18:1 n-7t)	Consensus mean *above* target range Consensus range *overlaps* target range	288 %
Total Elaidic Acid (C18:1 n-9t)	Consensus mean *below* target range Consensus range *overlaps* target range	96 %
Total α-Linolenic Acid (C18:3 n-3)	Consensus mean *below* target range Consensus range *does not overlap* target range	109 %
Total γ-Linolenic Acid (C18:3 n-6)	Consensus mean *below* target range Consensus range *overlaps* target range	120 %

Analyte	Relative Position of Consensus and Target Ranges	Between-Laboratory Variability
Total Arachidic Acid (C20:0)	Consensus mean *slightly below* target range Consensus range *overlaps* target range	150 %
Total Dihomo-γ-Linolenic Acid (C20:3 n-6)	Consensus mean *below* target range Consensus range *does not overlap* target range	13 %
Total Arachidonic Acid (C20:4 n-6)	Consensus mean *slightly above* target range Consensus range *overlaps* target range	43 %
Total EPA (C20:5 n-3)	Not Applicable	NA
Total DPA (C22:5 n-3)	Consensus mean *slightly above* target range Consensus range *overlaps* target range	55 %
Total DHA (C22:6 n-3)	Consensus mean *below* target range Consensus range *overlaps* target range	130 %

- The between-laboratory variability for Palm Oil Powder is summarized below and was good for *cis*-vaccenic acid (14 % RSD), while most between-laboratory variability for fatty acids was poor (> 75% RSD).

Performance Summary for
Palm Oil Powder

Analyte	Between-Laboratory Variability
Total Caprylic Acid (C8:0)	189%
Total Capric Acid (C10:0)	240%
Total Lauric Acid (C12:0)	283%
Total Myristic Acid (C14:0)	67%
Total Myristoleic Acid (C14:1 n-5)	NA
Total Palmitic Acid (C16:0)	88%
Total Palmitoleic Acid (C16:1)	93%
Total Stearic Acid (C18:0)	67%
Total *cis*-Vaccenic Acid (C18:1 n-7)	14%
Total Oleic Acid (C18:1 n-9)	83%
Total Linoleic Acid (C18:2 n-6)	94%
Total *trans*-Vaccenic Acid (C18:1 n-7t)	200%
Total Elaidic (C18:1 n-9t)	333%
Total α-Linolenic Acid (C18:3 n-3)	59%
Total γ-Linolenic Acid (C18:3 n-6)	417%
Total Arachidic Acid (C20:0)	42%
Total Dihomo-γ-Linolenic Acid (C20:3 n-6)	NA
Total Arachidonic Acid (C20:4 n-6)	276%
Total EPA (C20:5 n-3)	209%
Total DPA (C22:5 n-3)	240%
Total DHA (C22:6 n-3)	200%

- The between-laboratory variability for Spirulina is summarized below and was poor for all fatty acids (> 75 % RSD).

	Performance Summary for Spirulina
Analyte	Between-Laboratory Variability
Total Caprylic Acid (C8:0)	254 %
Total Capric Acid (C10:0)	167 %
Total Lauric Acid (C12:0)	600 %
Total Myristic Acid (C14:0)	172 %
Total Myristoleic Acid (C14:1 n-5)	228 %
Total Palmitic Acid (C16:0)	154 %
Total Palmitoleic Acid (C16:1)	135 %
Total Stearic Acid (C18:0)	100 %
Total *cis*-Vaccenic Acid (C18:1 n-7)	93 %
Total Oleic Acid (C18:1 n-9)	122 %
Total Linoleic Acid (C18:2 n-6)	134 %
Total *trans*-Vaccenic Acid (C18:1 n-7t)	NA
Total Elaidic Acid (C18:1 n-9t)	289 %
Total α-Linolenic Acid (C18:3 n-3)	325 %
Total γ-Linolenic Acid (C18:3 n-6)	103 %
Total Arachidic Acid (C20:0)	NA
Total Dihomo-γ-Linolenic Acid (C20:3 n-6)	138 %
Total Arachidonic Acid (C20:4 n-6)	200 %
Total EPA (C20:5 n-3)	85 %
Total DPA (C22:5 n-3)	NA
Total DHA (C22:6 n-3)	NA

- Seven laboratories reported using GC or GC-FID for determination of total fatty acids. No other laboratories specified the analytical methods used.

Dietary Intake Technical Recommendations

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

- No general trend was apparent between the consensus means and the NIST-determined values for total fatty acids in SRM 1845a Whole Egg Powder. Most consensus ranges overlapped the target range based on the NIST-determined value, except for myristoleic acid, α-linolenic acid, and dihomo-γ-linolenic acid.
- The average between-laboratory variabilities for SRM 1845a Whole Egg Powder, Palm Oil Powder, and Spirulina were 122 %, 168 %, and 194 % respectively.
- The greatest between-laboratory variability was observed for fatty acids present at less than 0.1 mg/g, for which significant zero values were reported by some laboratories.
- The homogeneity of the Palm Oil Powder and Spirulina materials has not been thoroughly evaluated, and inhomogeneity may be contributing to the greater variability about the consensus means in these materials.
- For many of the fatty acids in the Palm Oil Powder, a sample in which the fat is present primarily as triglycerides, participant data clustered into two groups. One possible reason for this bimodal distribution would be based on the sample preparation approach used by participating laboratories. Given the limited number of results (only two to nine laboratories reporting data), no conclusions can be made. However, exploration of this distribution could be the focus of a follow-up study in which detailed sample preparation information is collected from each participating laboratory to glean meaningful insight from the two distinct groups.
- "Zero" is not a quantity that can be measured, and therefore a more appropriate result would be to report that a value is below the MDL, LOQ, or QL.
- All results should be checked closely to avoid calculation errors and to be sure that results are reported in the requested units.

Table 5-1. Individualized data summary table (NIST) for total fatty acids in SRM 1845a Whole Egg Powder, Palm Oil Powder, and Spirulina.

National Institute of Standards & Technology

HAMQAP Exercise 2 - Fatty Acids

		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 Caprylic Acid (C8:0)	SRM 1845a Whole Egg Powder	mg/g						4	0.025	0.066		
Total Caprylic Acid (C8:0)	Palm Oil Powder	mg/g						6	0.019	0.036		
Total Caprylic Acid (C8:0)	Spirulina	mg/g						4	0.013	0.033		
Total Capric Acid (C10:0)	SRM 1845a Whole Egg Powder	mg/g						4	0	0		
Total Capric Acid (C10:0)	Palm Oil Powder	mg/g						6	0.030	0.072		
Total Capric Acid (C10:0)	Spirulina	mg/g						5	0.09	0.15		
Total Lauric Acid (C12:0)	SRM 1845a Whole Egg Powder	mg/g						6	0.003	0.009		
Total Lauric Acid (C12:0)	Palm Oil Powder	mg/g						9	0.06	0.17		
Total Lauric Acid (C12:0)	Spirulina	mg/g						5	0.011	0.066		
Total Myristic Acid (C14:0)	SRM 1845a Whole Egg Powder	mg/g		1.094	0.024			8	1.08	0.85	1.094	0.048
Total Myristic Acid (C14:0)	Palm Oil Powder	mg/g						9	0	3		
Total Myristic Acid (C14:0)	Spirulina	mg/g						7	0.1	0.18		
Total Myristoleic Acid (C14:1 n-5)	SRM 1845a Whole Egg Powder	mg/g		0.185	0.004			5	0.24	0.25	0.185	0.008
Total Myristoleic Acid (C14:1 n-5)	Palm Oil Powder	mg/g						4	0	0		
Total Myristoleic Acid (C14:1 n-5)	Spirulina	mg/g						3	0.039	0.089		
Total Palmitic Acid (C16:0)	SRM 1845a Whole Egg Powder	mg/g		82.2	1.3			8	80	88	82.2	2.6
Total Palmitic Acid (C16:0)	Palm Oil Powder	mg/g						9	160	140		
Total Palmitic Acid (C16:0)	Spirulina	mg/g						8	10	20		
Total Palmitoleic Acid (C16:1 n-7)	SRM 1845a Whole Egg Powder	mg/g		8.31	0.12			7	7.9	9.6	8.31	0.23
Total Palmitoleic Acid (C16:1 n-7)	Palm Oil Powder	mg/g						8	0.59	0.55		
Total Palmitoleic Acid (C16:1 n-7)	Spirulina	mg/g						7	1.1	1.5		
Total Stearic Acid (C18:0)	SRM 1845a Whole Egg Powder	mg/g		28.02	0.48			9	20	23	28.02	0.95
Total Stearic Acid (C18:0)	Palm Oil Powder	mg/g						11	20	12		
Total Stearic Acid (C18:0)	Spirulina	mg/g						9	0.31	0.31		
Total cis-Vaccenic Acid (C18:1 n-7)	SRM 1845a Whole Egg Powder	mg/g		5.32	0.08			5	6.9	3.5	5.32	0.15
Total cis-Vaccenic Acid (C18:1 n-7)	Palm Oil Powder	mg/g						5	4.26	0.58		
Total cis-Vaccenic Acid (C18:1 n-7)	Spirulina	mg/g						3	0.44	0.41		
Total Oleic Acid (C18:1 n-9)	SRM 1845a Whole Egg Powder	mg/g		110	7			9	100	89	110	14
Total Oleic Acid (C18:1 n-9)	Palm Oil Powder	mg/g						11	150	120		
Total Oleic Acid (C18:1 n-9)	Spirulina	mg/g						9	0.9	1.1		
Total Transvaccenic Acid (C18:1 n-7t)	SRM 1845a Whole Egg Powder	mg/g		0.123	0.020			2	0.8	2.3	0.123	0.039
Total Transvaccenic Acid (C18:1 n-7t)	Palm Oil Powder	mg/g						3	0.43	0.86		
Total Transvaccenic Acid (C18:1 n-7t)	Spirulina	mg/g						2	0	0		
Total Elaidic Acid (C18:1 n-9t)	SRM 1845a Whole Egg Powder	mg/g		0.053	0.008			4	0.25	0.24	0.053	0.016
Total Elaidic Acid (C18:1 n-9t)	Palm Oil Powder	mg/g						3	0	1		
Total Elaidic Acid (C18:1 n-9t)	Spirulina	mg/g						3	0.9	2.6		
Total Linoleic Acid (C18:2 n-6)	SRM 1845a Whole Egg Powder	mg/g		54.3	0.6			9	50	41	54.3	1.2
Total Linoleic Acid (C18:2 n-6)	Palm Oil Powder	mg/g						11	40	34		
Total Linoleic Acid (C18:2 n-6)	Spirulina	mg/g						9	6.5	8.7		
Total alpha-Linolenic Acid (C18:3 n-3)	SRM 1845a Whole Egg Powder	mg/g		1.643	0.024			9	0.8	0.87	1.643	0.047
Total alpha-Linolenic Acid (C18:3 n-3)	Palm Oil Powder	mg/g						11	0.56	0.33		
Total alpha-Linolenic Acid (C18:3 n-3)	Spirulina	mg/g						6	0.8	2.6		
Total gamma-Linolenic Acid (C18:3 n-6)	SRM 1845a Whole Egg Powder	mg/g		0.452	0.009			9	0.30	0.36	0.452	0.018
Total gamma-Linolenic Acid (C18:3 n-6)	Palm Oil Powder	mg/g						9	0.01	0.05		
Total gamma-Linolenic Acid (C18:3 n-6)	Spirulina	mg/g						8	3.7	3.8		
Total Arachidic Acid (C20:0)	SRM 1845a Whole Egg Powder	mg/g		0.047	0.010			4	0.10	0.15	0.047	0.020
Total Arachidic Acid (C20:0)	Palm Oil Powder	mg/g						4	2.1	0.9		
Total Arachidic Acid (C20:0)	Spirulina	mg/g						2	0	0		
Total Dihomo-gamma-linolenic acid (C20:3 n-6)	SRM 1845a Whole Egg Powder	mg/g		1.10	0.20			2	0.88	0.11	1.08	0.40
Total Dihomo-gamma-linolenic acid (C20:3 n-6)	Palm Oil Powder	mg/g						2	0	0		
Total Dihomo-gamma-linolenic acid (C20:3 n-6)	Spirulina	mg/g						2	0.20	0.27		
Total Arachidonic Acid (C20:4 n-6)	SRM 1845a Whole Egg Powder	mg/g		6.43	0.09			6	6.8	2.9	6.43	0.17
Total Arachidonic Acid (C20:4 n-6)	Palm Oil Powder	mg/g						4	0.021	0.058		
Total Arachidonic Acid (C20:4 n-6)	Spirulina	mg/g						3	0.14	0.28		
Total EPA (C20:5 n-3)	SRM 1845a Whole Egg Powder	mg/g						5	0	0		
Total EPA (C20:5 n-3)	Palm Oil Powder	mg/g						6	0.04	0.09		
Total EPA (C20:5 n-3)	Spirulina	mg/g						5	0.041	0.035		
Total DPA (C22:5 n-3)	SRM 1845a Whole Egg Powder	mg/g		0.202	0.004			4	0.22	0.12	0.202	0.007
Total DPA (C22:5 n-3)	Palm Oil Powder	mg/g						3	0.010	0.024		
Total DPA (C22:5 n-3)	Spirulina	mg/g						2	0	0		
Total DHA (C22:6 n-3)	SRM 1845a Whole Egg Powder	mg/g		1.701	0.039			9	1	1.3	1.701	0.077
Total DHA (C22:6 n-3)	Palm Oil Powder	mg/g						6	0.007	0.014		
Total DHA (C22:6 n-3)	Spirulina	mg/g						5	0	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 5-2. Data summary table for total caprylic acid in SRM 1845a Whole Egg Powder, Palm Oil Powder, and Spirulina.

		Total Caprylic Acid (C8:0)													
		SRM 1845a Whole Egg Powder (mg/g)					Palm Oil Powder (mg/g)					Spirulina (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target															
	B001															
	B002															
	B006															
	B014	< 0.300	< 0.300	< 0.300			< 0.500	< 0.500	< 0.500			< 0.300	< 0.300	< 0.300		
	B016						0.066	0.058	0.064	0.0627	0.0042					
	B017															
	B018	< 0.003	< 0.003	< 0.003			< 0.012	< 0.012	< 0.012			< 0.003	< 0.003	< 0.003		
	B019	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
	B027	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
	B031	0.1	0.1	0.1	0.1	0	0.05	0.05	0.05	0.05	0	0.05	0.05	0.05	0.05	0
	B034															
	B036						0	0	0	0	0					
	B038	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
	B039															
	B041															
	B042															
	B044															
Community Results		Consensus Mean			0.025		Consensus Mean			0.019		Consensus Mean			0.013	
		Consensus Standard Deviation			0.066		Consensus Standard Deviation			0.036		Consensus Standard Deviation			0.033	
		Maximum			0.1		Maximum			0.0627		Maximum			0.05	
		Minimum			0		Minimum			0		Minimum			0	
		N			4		N			6		N			4	

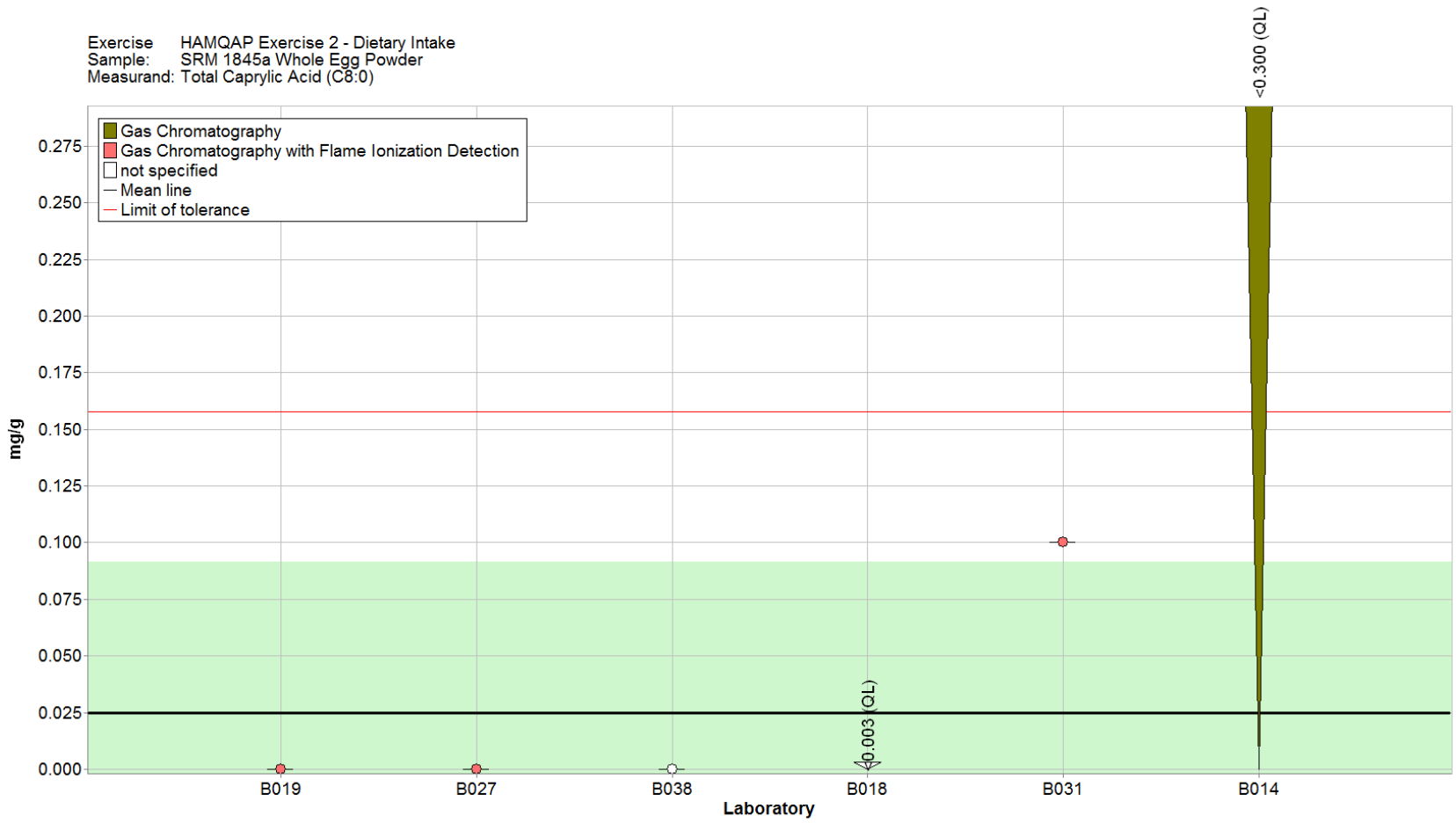

Figure 5-1. Total caprylic acid (C8:0) in SRM 1845a Whole Egg Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

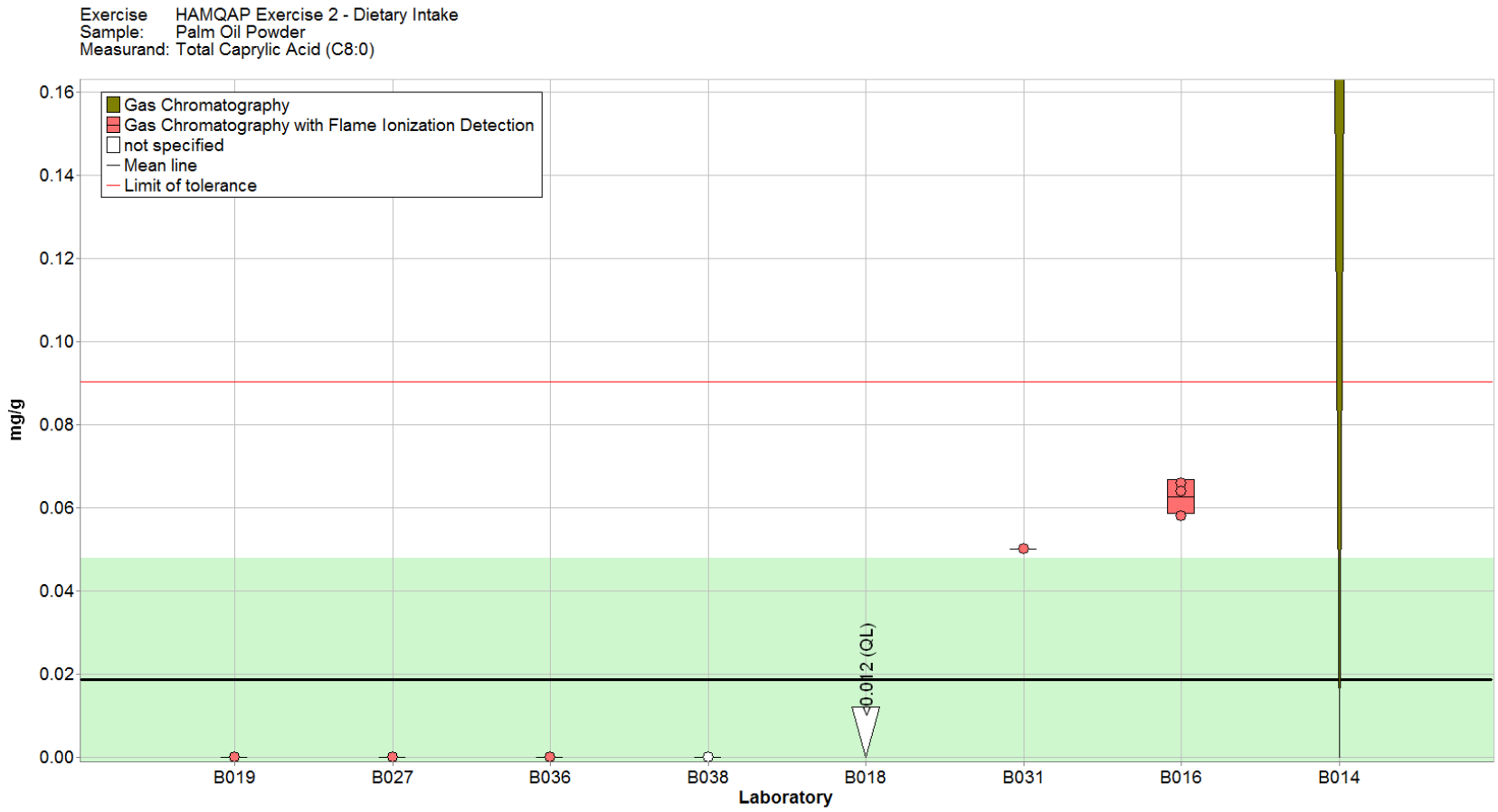

Figure 5-2. Total caprylic acid (C8:0) in Palm Oil Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

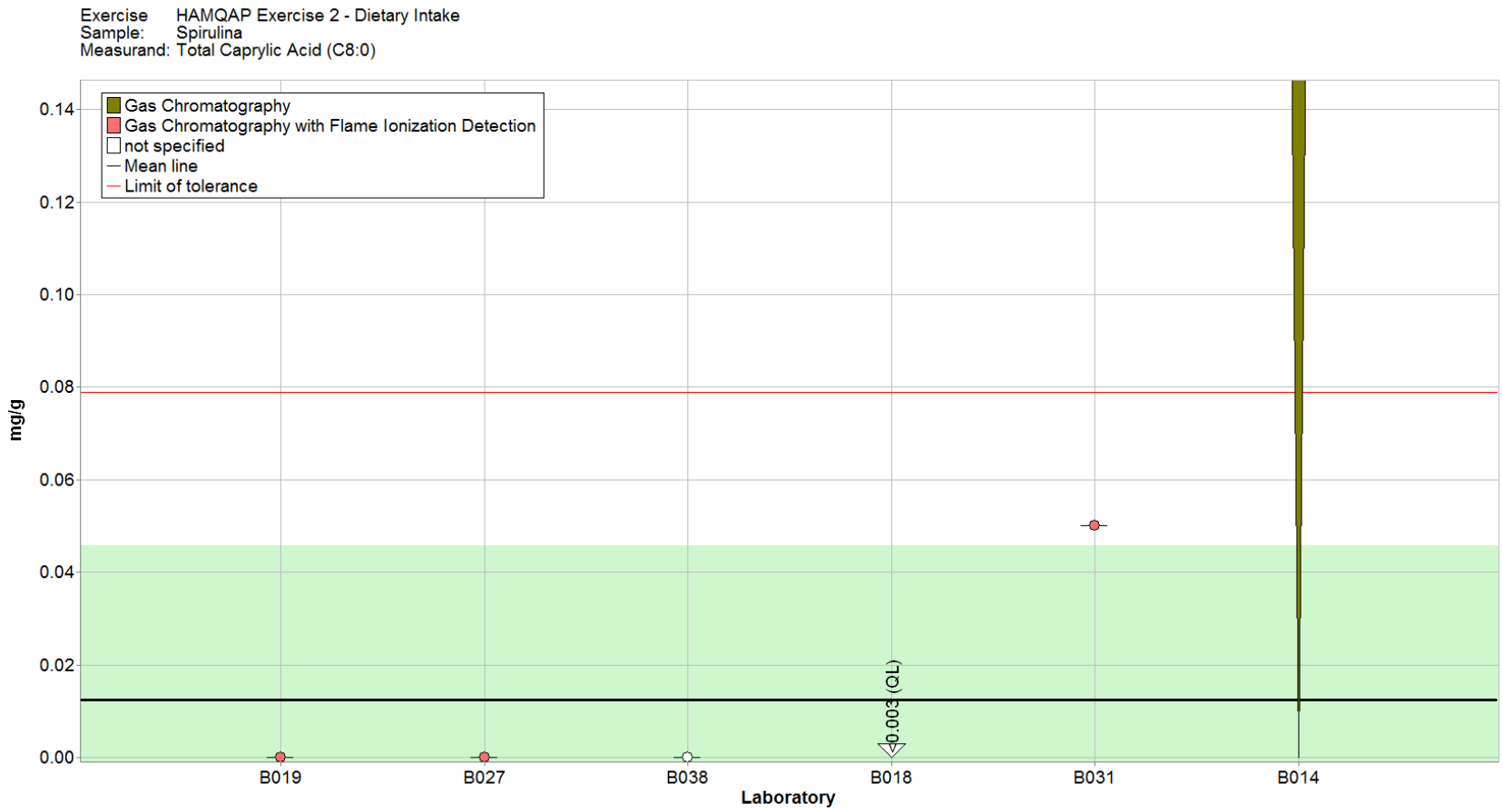

Figure 5-3. Total caprylic acid (C8:0) in Spirulina (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

Table 5-3. Data summary table for total capric acid in SRM 1845a Whole Egg Powder, Palm Oil Powder, and Spirulina.

		Total Capric Acid (C10:0)													
		SRM 1845a Whole Egg Powder (mg/g)					Palm Oil Powder (mg/g)					Spirulina (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target															
	B001															
	B002															
	B006															
	B014	< 0.300	< 0.300	< 0.300			< 0.500	< 0.500	< 0.500			< 0.300	< 0.300	< 0.300		
	B016						0.078	0.078	0.076	0.0773	0.0012	0.241	0.261	0.204	0.235	0.029
	B017															
	B018	< 0.003	< 0.003	< 0.003			< 0.012	< 0.012	< 0.012			< 0.003	< 0.003	< 0.003		
	B019	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
	B027	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
	B031	0	0	0	0	0	0.1	0.1	0.1	0.1	0	0.2	0.2	0.2	0.2	0
	B034															
	B036						0	0	0	0	0					
	B038	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
	B039															
	B041															
	B042															
	B044															
Community Results		Consensus Mean			0		Consensus Mean			0.030		Consensus Mean			0.09	
		Consensus Standard Deviation			0		Consensus Standard Deviation			0.072		Consensus Standard Deviation			0.15	
		Maximum			0		Maximum			0.1		Maximum			0.235	
		Minimum			0		Minimum			0		Minimum			0	
		N			4		N			6		N			5	

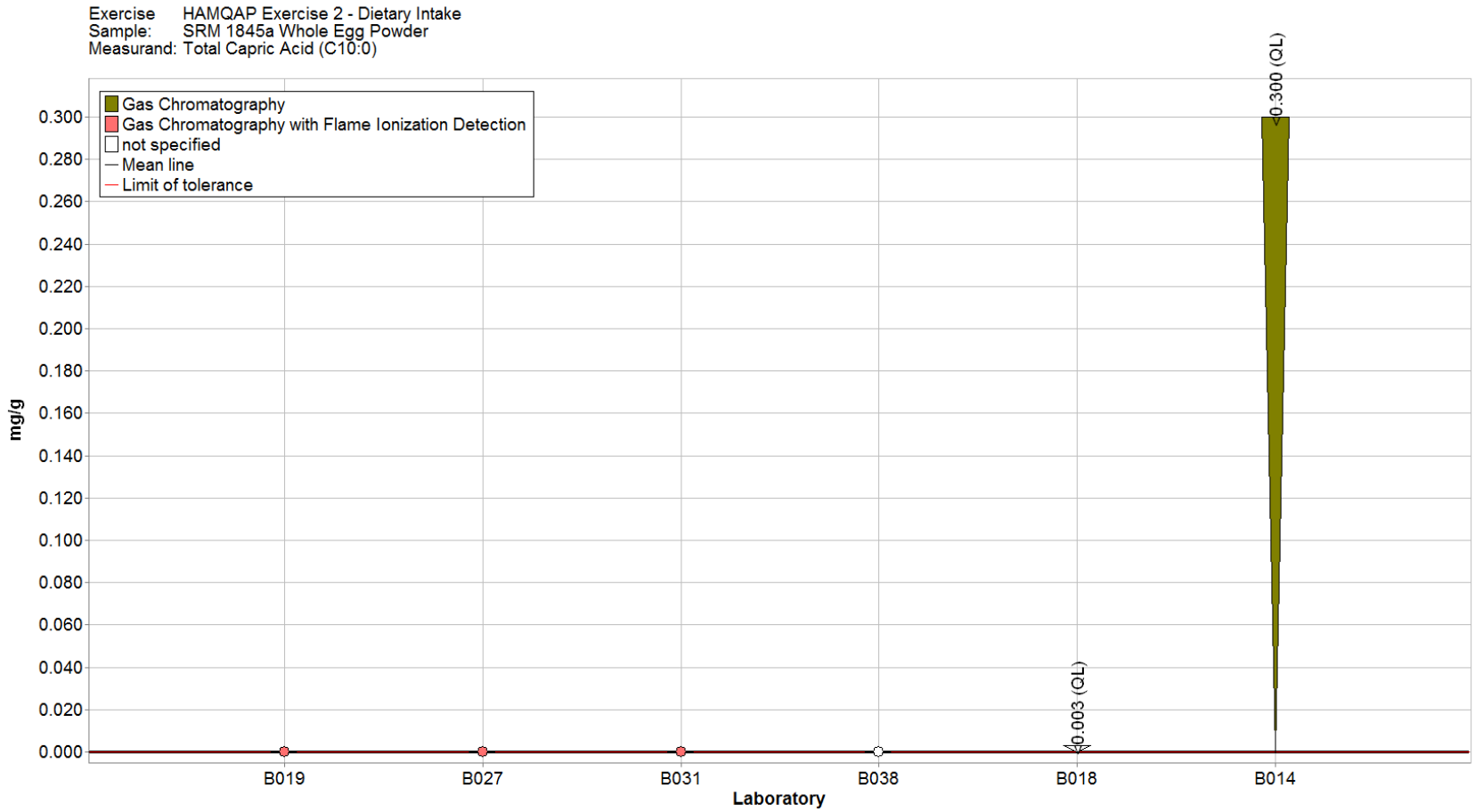

Figure 5-4. Total capric acid (C10:0) in SRM 1845 Whole Egg Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. A NIST value has not been determined in this material.

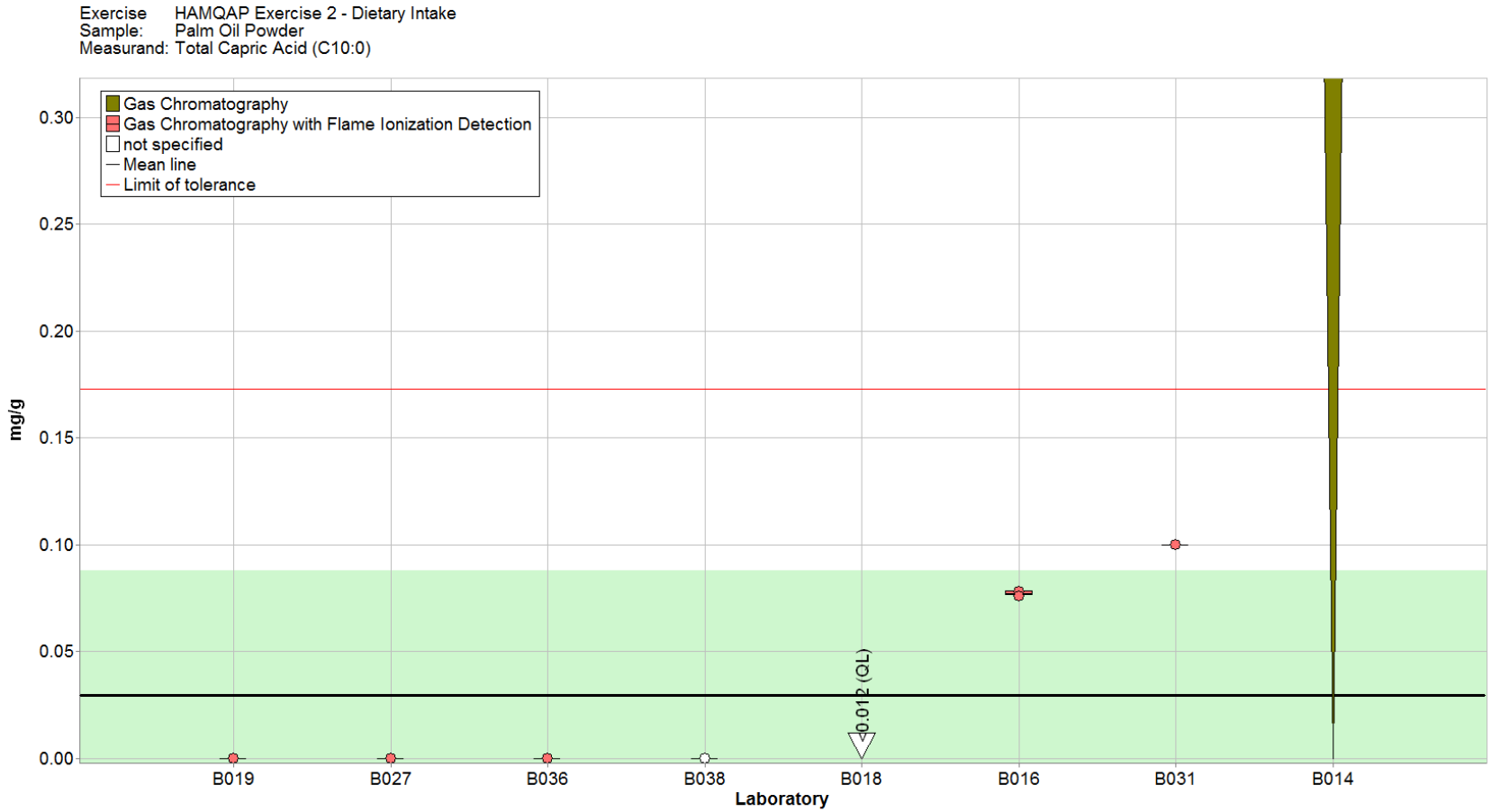

Figure 5-5. Total capric acid (C10:0) in Palm Oil Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

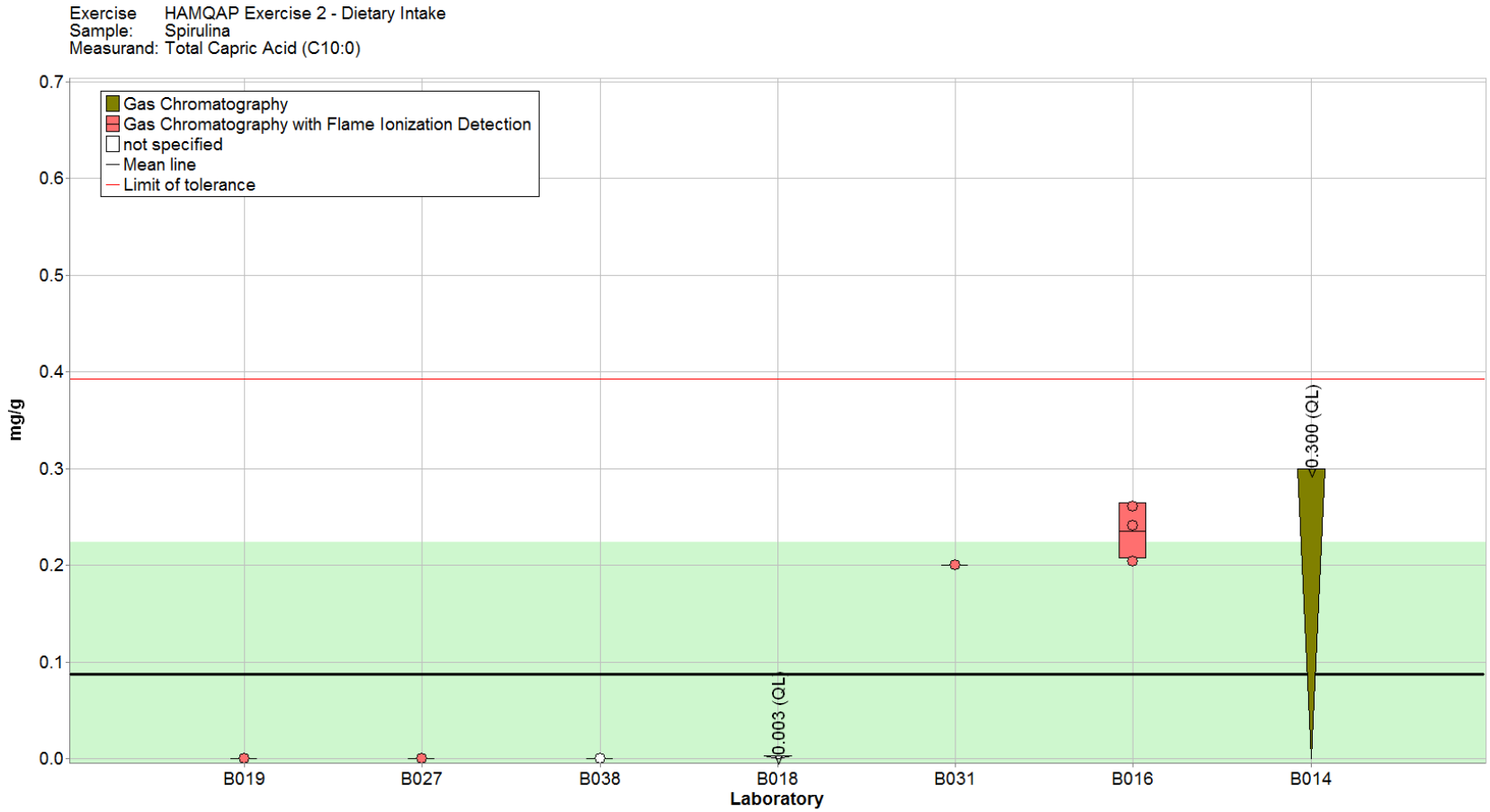

Figure 5-6. Total capric acid (C10:0) in Spirulina (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

Table 5-4. Data summary table for total lauric acid in SRM 1845a Whole Egg Powder, Palm Oil Powder, and Spirulina. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		Total Lauric Acid (C12:0)														
		SRM 1845a Whole Egg Powder (mg/g)					Palm Oil Powder (mg/g)					Spirulina (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target															
	B001															
	B002															
	B006															
	B014	< 0.300	< 0.300	< 0.300			0.84	0.85	0.85	0.8467	0.0058	< 0.300	< 0.300	< 0.300		
	B015															
	B016	0.015	0.019	0.015	0.0163	0.0023	0.073	0.072	0.072	0.07233	0.00058					
	B017															
	B018	< 0.003	< 0.003	< 0.003			0.936	0.908	0.95	0.931	0.021	< 0.003	< 0.003	< 0.003		
	B019	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
	B027	0	0	0	0	0	0.91	0.89	0.89	0.897	0.012	0.16	0	0	0.053	0.092
	B031	0	0	0	0	0	0.8	0.75	0.8	0.783	0.029	0	0	0	0	0
	B033	0	0	0	0	0	0.089	0.083	0.08	0.0838	0.0044	0	0	0	0	0
	B034															
	B036						0.132	0.136	0.132	0.1332	0.0025					
	B038	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
	B039															
	B041															
	B042															
	B044															
Community Results		Consensus Mean			0.003		Consensus Mean			0.06		Consensus Mean			0.011	
		Consensus Standard Deviation			0.009		Consensus Standard Deviation			0.17		Consensus Standard Deviation			0.066	
		Maximum			0.0163		Maximum			0.93133		Maximum			0.0533	
		Minimum			0		Minimum			0		Minimum			0	
		N			6		N			9		N			5	

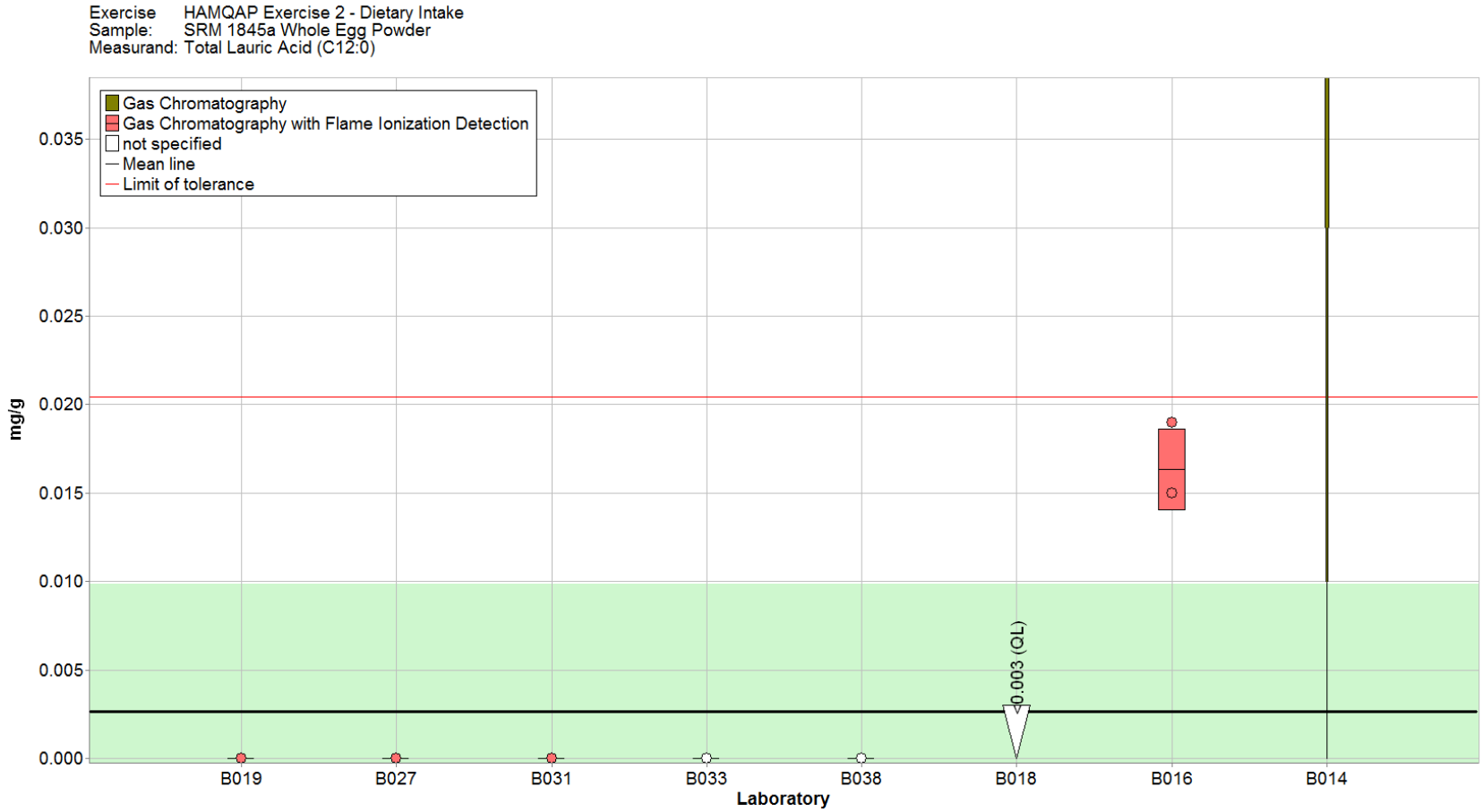

Figure 5-7. Total lauric acid (C12:0) in SRM 1845a Whole Egg Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

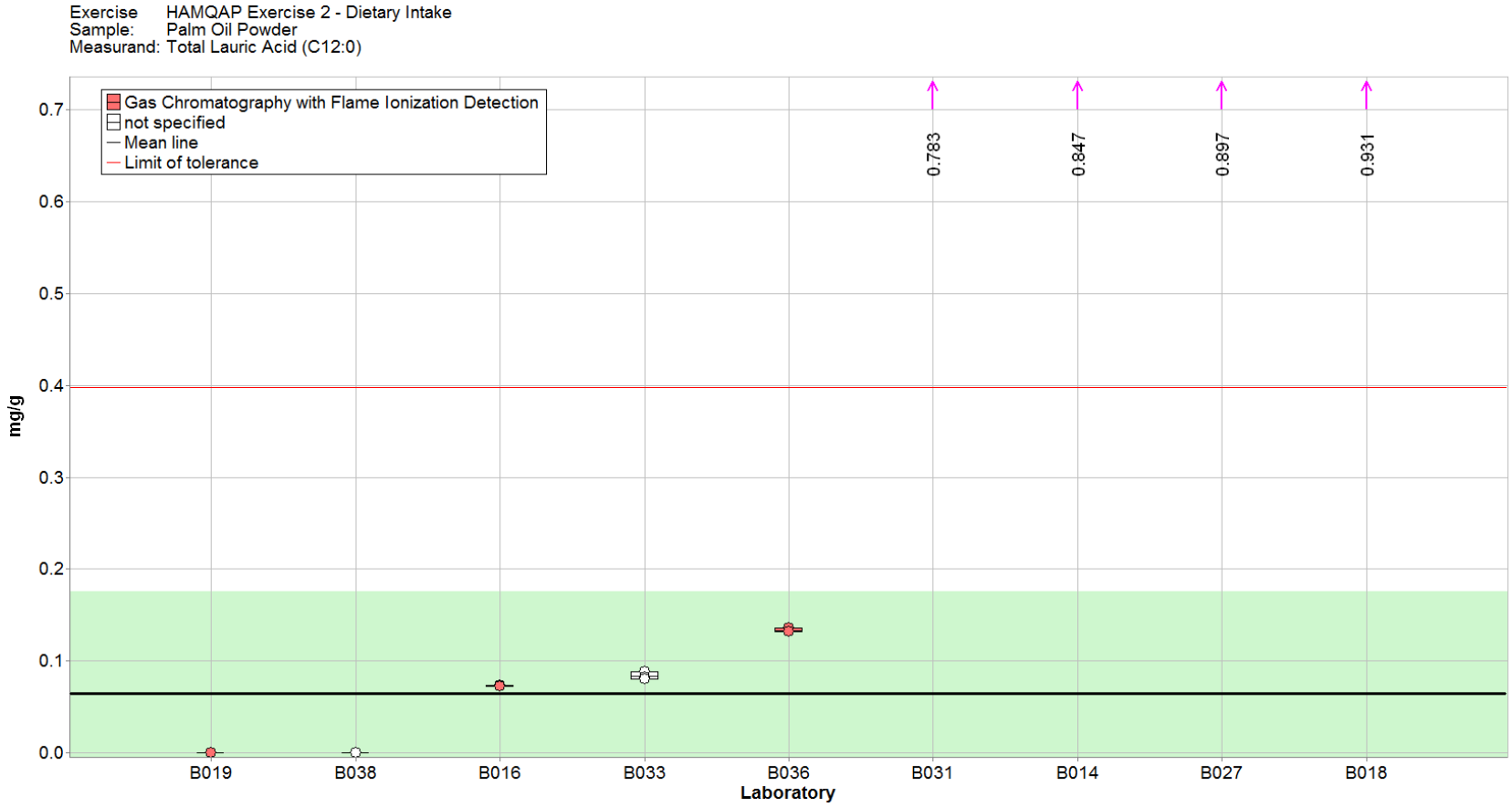

Figure 5-8. Total lauric acid (C12:0) in Palm Oil Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

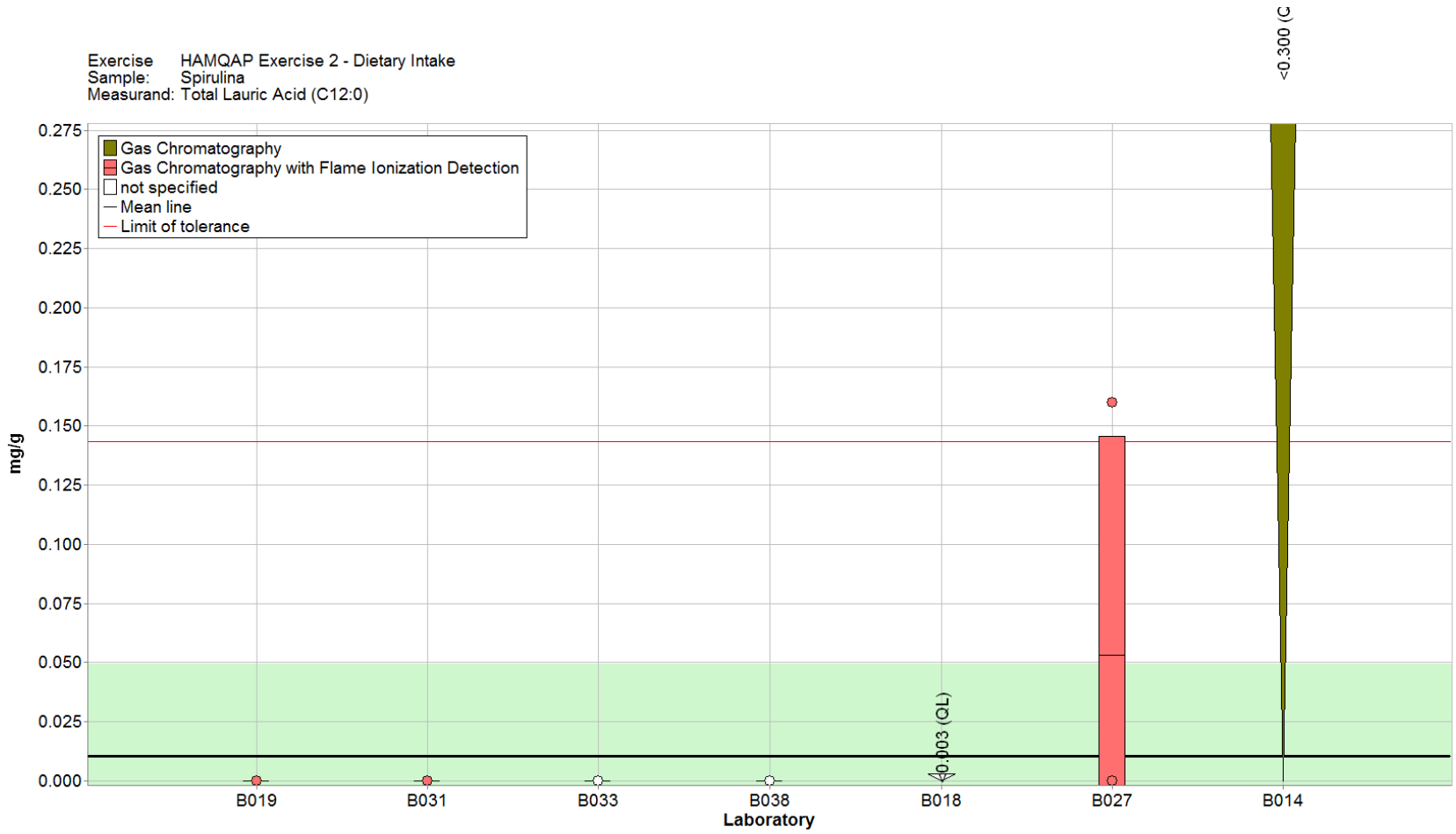

Figure 5-9. Total lauric acid (C12:0) in Spirulina (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

Table 5-5. Data summary table for total myristic acid in SRM 1845a Whole Egg Powder, Palm Oil Powder, and Spirulina. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		Total Myristic Acid (C14:0)														
		SRM 1845a Whole Egg Powder (mg/g)					Palm Oil Powder (mg/g)					Spirulina (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				1.09	0.05										
	B001															
	B002															
	B006															
	B014	1.17	1.23	1.18	1.193	0.032	6.54	6.69	6.74	6.66	0.10	< 0.300	< 0.300	< 0.300		
	B015															
	B016	0.791	0.796	0.782	0.7897	0.0071	6.01	6	5.94	5.983	0.038	0.204	0.143	0.12	0.156	0.043
	B017															
	B018	2.25	2.178	2.312	2.247	0.067	8.648	8.73	8.67	8.683	0.042	1.598	1.35	1.501	1.48	0.12
	B019	0.13	0.13	0.13	0.13	0	0.69	0.69	0.68	0.6867	0.0058	0.04	0.04	0.04	0.04	0
	B027	1.39	1.39	1.39	1.39	0	7.05	7.08	6.96	7.030	0.062	0	0	0.13	0.043	0.075
	B031	1.4	1.6	1.4	1.47	0.12	7.35	6.95	7.2	7.17	0.20	0.15	0.15	0.15	0.15	0
	B034															
	B036						1.073	1.082	1.073	1.0761	0.0053					
	B038	0	0	0	0	0	0.543	0.531	0.53	0.5347	0.0072	0	0	0	0	0
	B039															
	B041															
	B042															
	B044															
	B045	1.42	1.38	1.36	1.387	0.031	2.92	3.2	3.15	3.09	0.15	0.21	0.26	0.23	0.233	0.025
Community Results		Consensus Mean			1.08		Consensus Mean			4.5		Consensus Mean			0.104	
		Consensus Standard Deviation			0.85		Consensus Standard Deviation			3.0		Consensus Standard Deviation			0.179	
		Maximum			2.247		Maximum			8.683		Maximum			1.48	
		Minimum			0		Minimum			0.5347		Minimum			0	
		N			8		N			9		N			7	

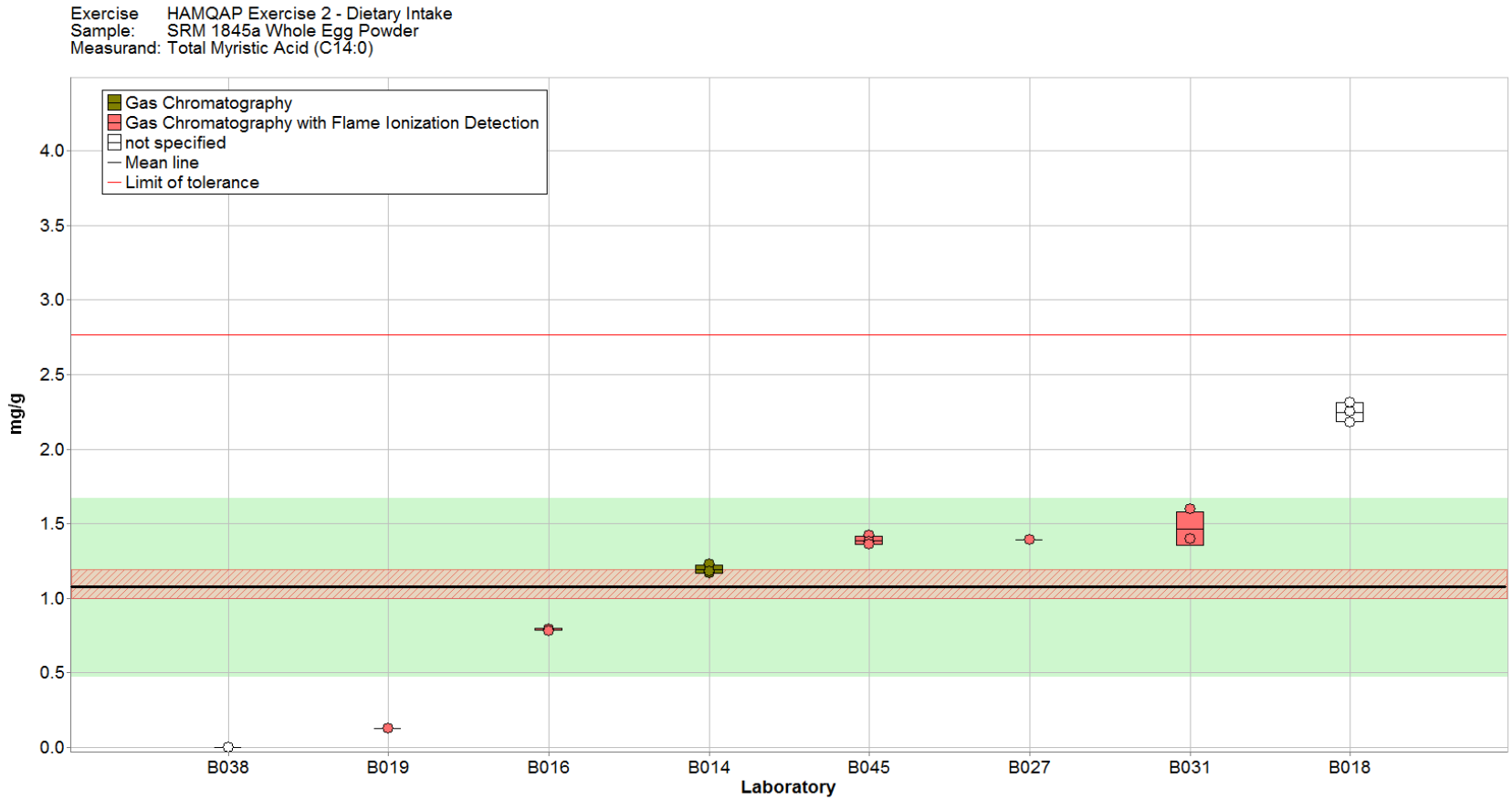

Figure 5-10. Total myristic acid (C14:0) in SRM 1845a Whole Egg Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

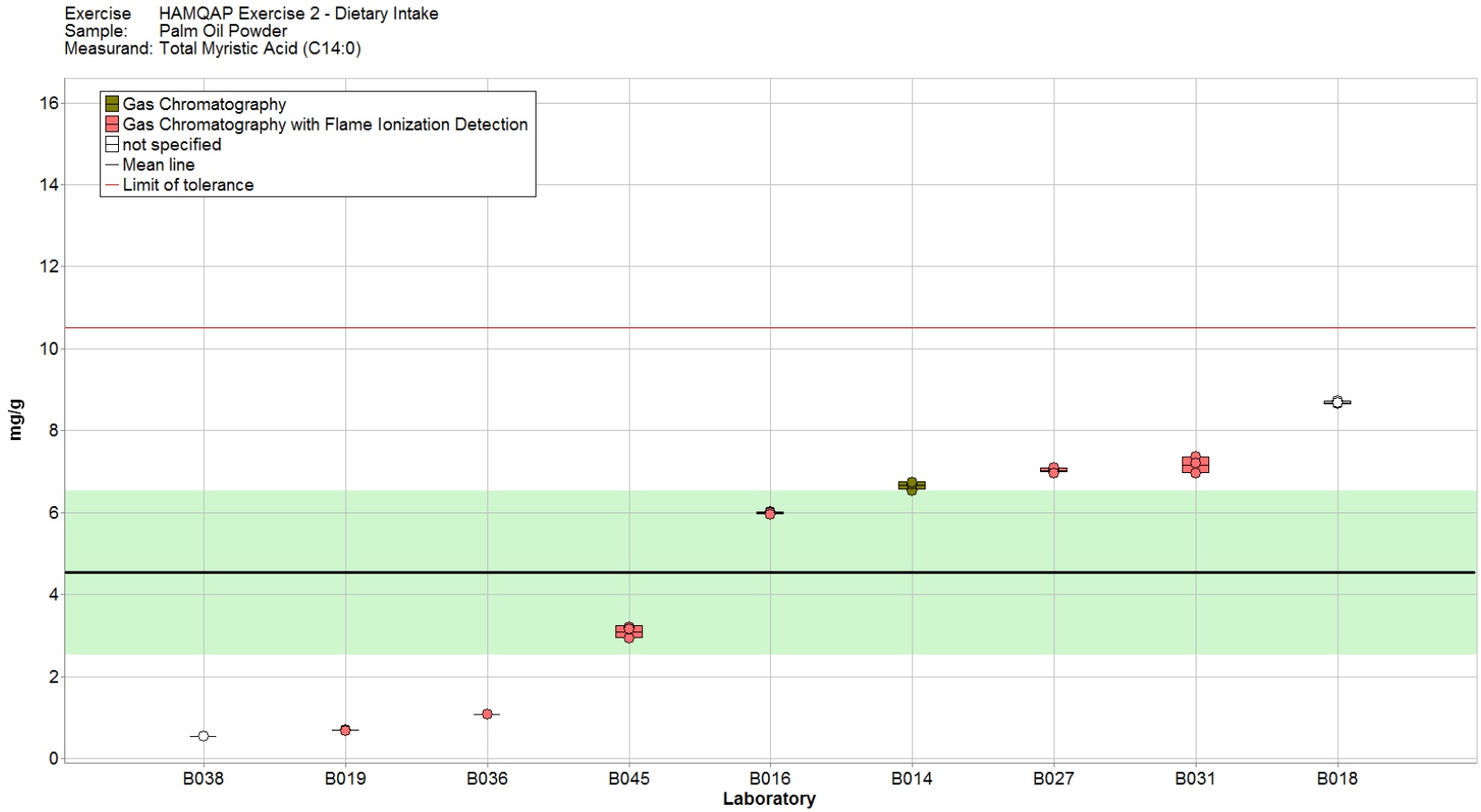

Figure 5-11. Total myristic acid (C14:0) in Palm Oil Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

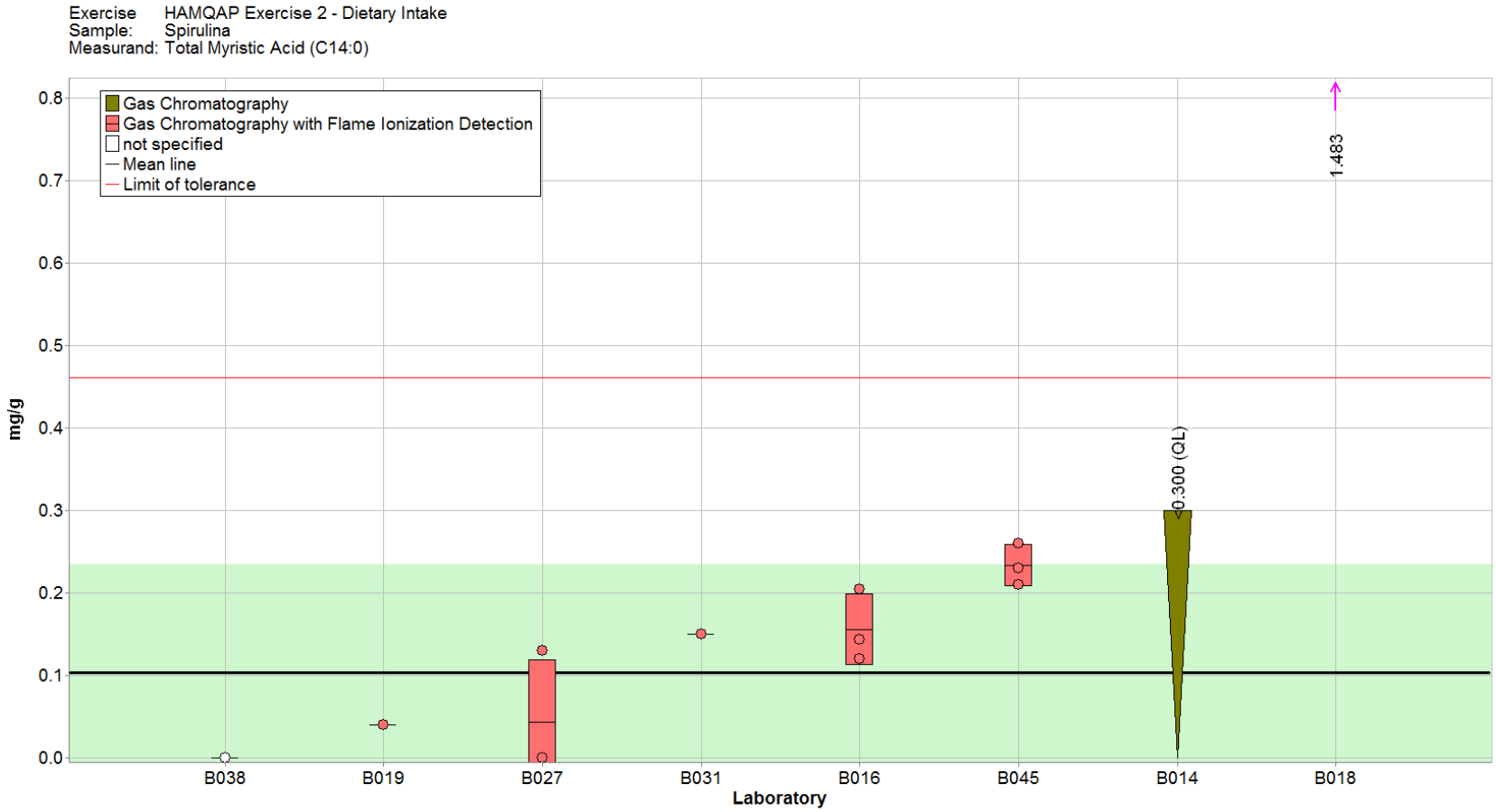

Figure 5-12. Total myristic acid (C14:0) in Spirulina (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

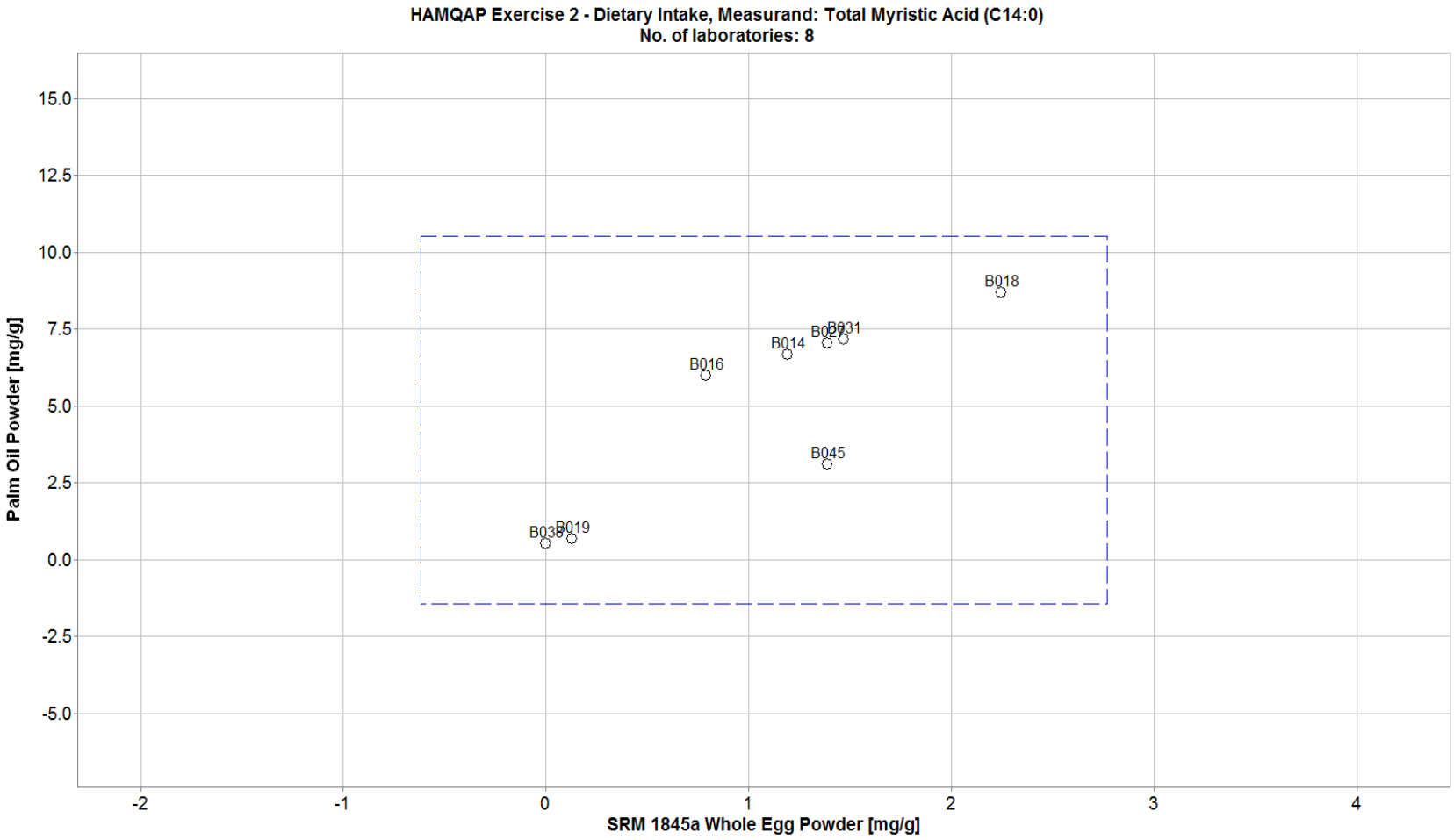

Figure 5-13. Laboratory means for total myristic acid in SRM 1845a Whole Egg Powder and Palm Oil Powder (sample/sample comparison view). In this view, the individual laboratory mean for one sample (SRM 1845a) is compared to the mean for a second sample (Palm Oil Powder). The dotted blue box represents the consensus range of tolerance for SRM 1845a (x-axis) and Palm Oil Powder (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

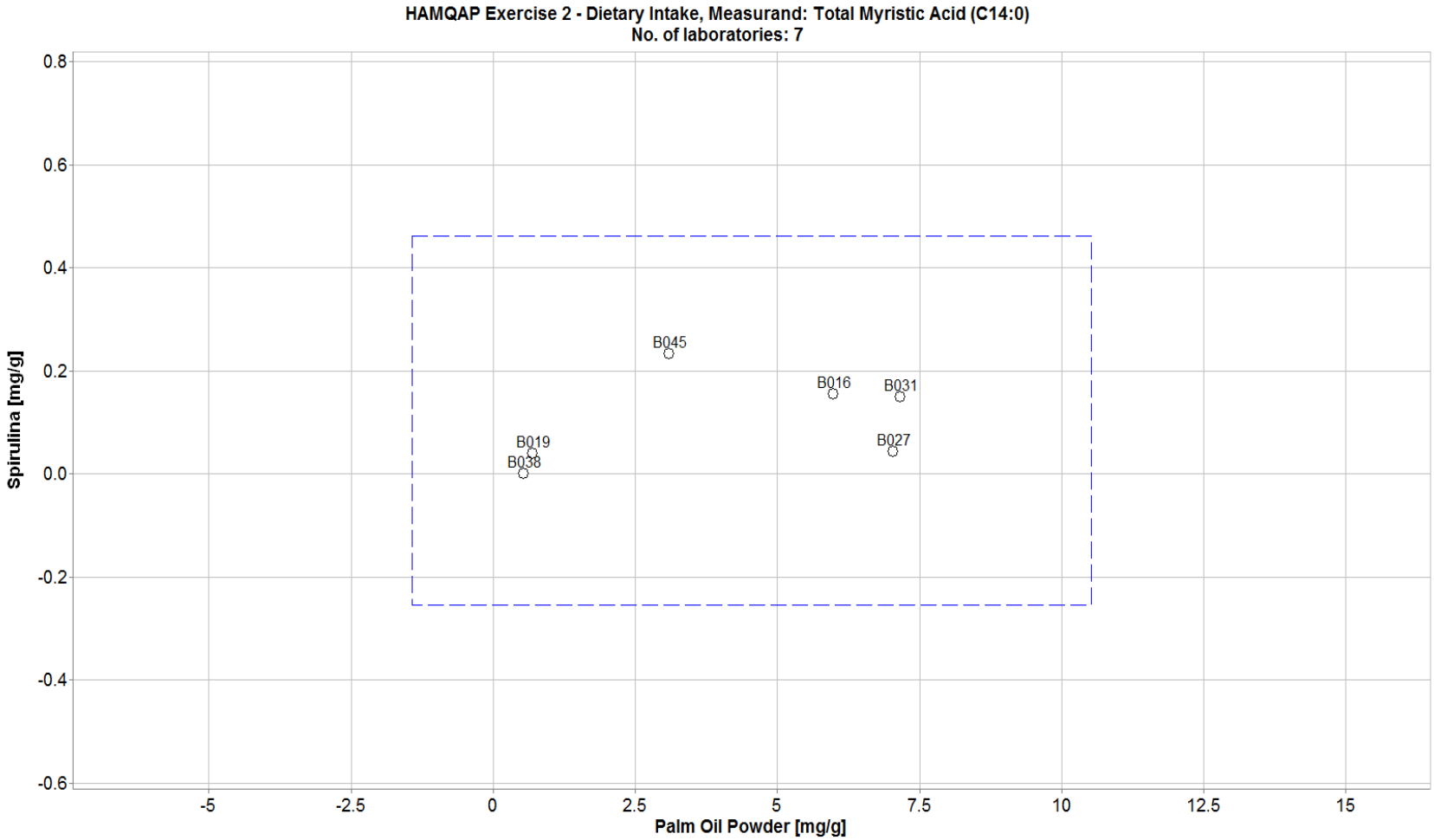

Figure 5-14. Laboratory means for total myristic acid in Palm Oil Powder and Spirulina (sample/sample comparison view). In this view, the individual laboratory mean for one sample (Palm Oil Powder) is compared to the mean for a second sample (Spirulina). The dotted blue box represents the consensus range of tolerance for Palm Oil Powder (x-axis) and Spirulina (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 5-6. Data summary table for total myristoleic acid in SRM 1845a Whole Egg Powder, Palm Oil Powder, and Spirulina.

		Total Myristoleic Acid (C14:1 n-5)														
		SRM 1845a Whole Egg Powder (mg/g)					Palm Oil Powder (mg/g)					Spirulina (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				0.19	0.01										
	B001															
	B002															
	B006															
	B014	< 0.300	< 0.300	< 0.300			< 0.500	< 0.500	< 0.500			< 0.300	< 0.300	< 0.300		
	B016	0.156	0.152	0.15	0.1527	0.0031										
	B017															
	B018	0.324	0.311	0.373	0.34	0.03	< 0.003	< 0.003	< 0.003			< 0.003	< 0.003	< 0.003		
	B019	0.03	0.03	0.03	0.03	0	0	0	0	0	0	0.05	0.05	0.05	0.05	0
	B027	0.26	0.26	0.26	0.26	0	0	0	0	0	0	0.2	0	0	0.07	0.12
	B031	0.4	0.5	0.4	0.433	0.058	0	0	0	0	0	0	0	0	0	0
	B034															
	B036						0	0	0	0	0					
	B039															
	B041															
	B042															
	B044															
Community Results		Consensus Mean			0.24		Consensus Mean			0		Consensus Mean			0.039	
		Consensus Standard Deviation			0.25		Consensus Standard Deviation			0		Consensus Standard Deviation			0.089	
		Maximum			0.433		Maximum			0		Maximum			0.06667	
		Minimum			0.03		Minimum			0		Minimum			0	
		N			5		N			4		N			3	

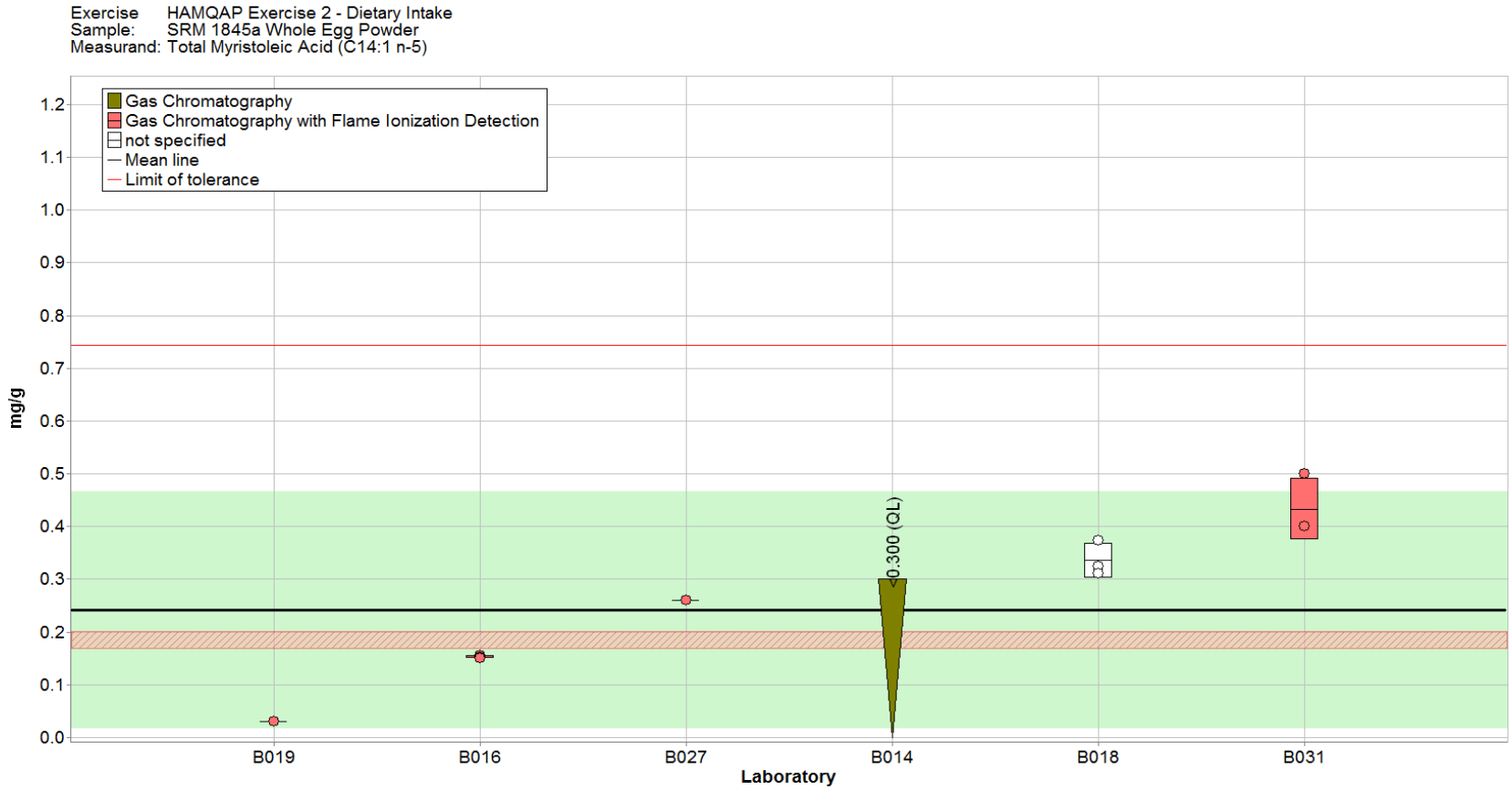

Figure 5-15. Total myristoleic acid (C14:1 n-5) in SRM 1845a Whole Egg Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

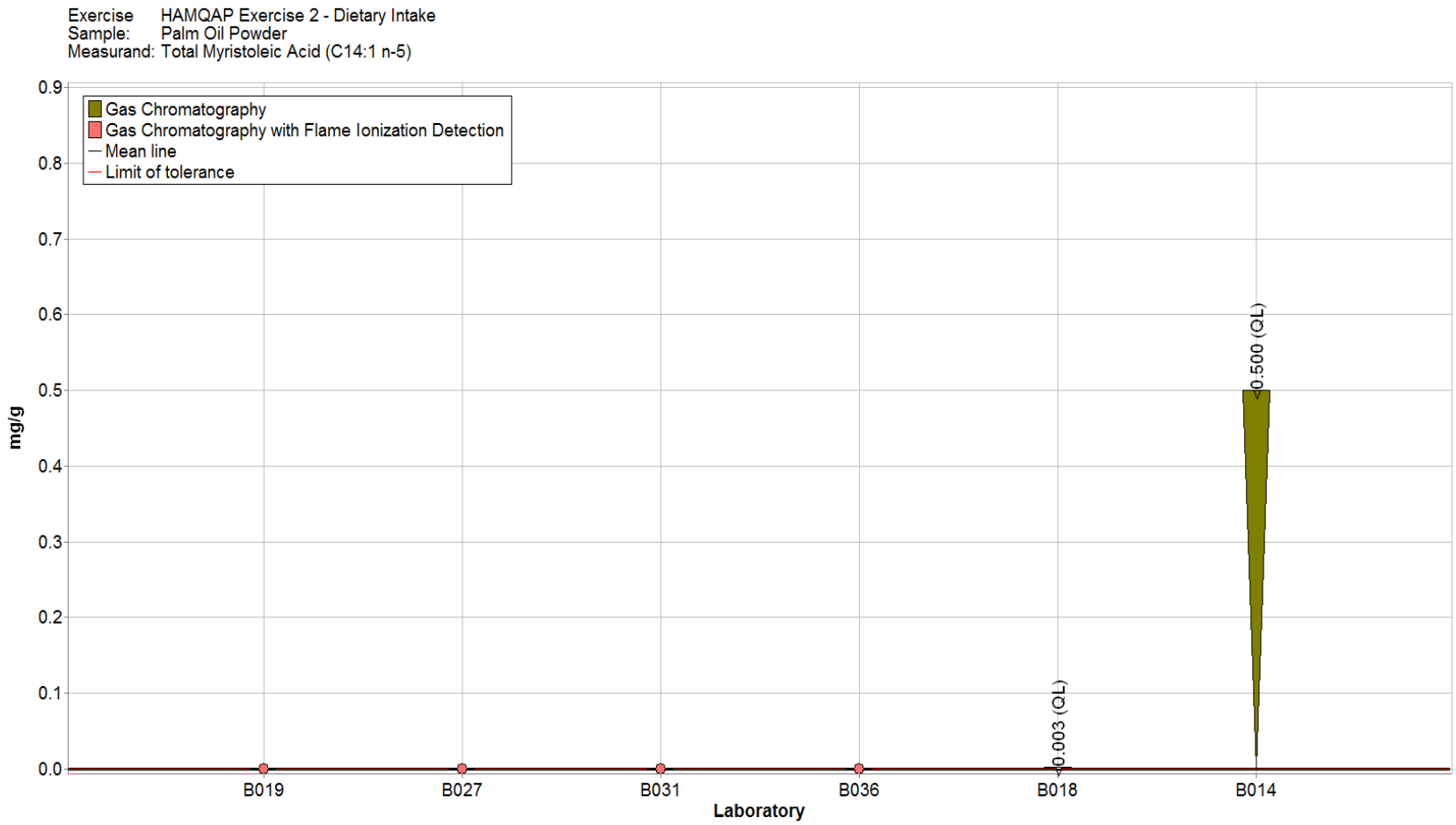

Figure 5-16. Total myristoleic acid (C14:1 n-5) in Palm Oil Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. A NIST value has not been determined in this material.

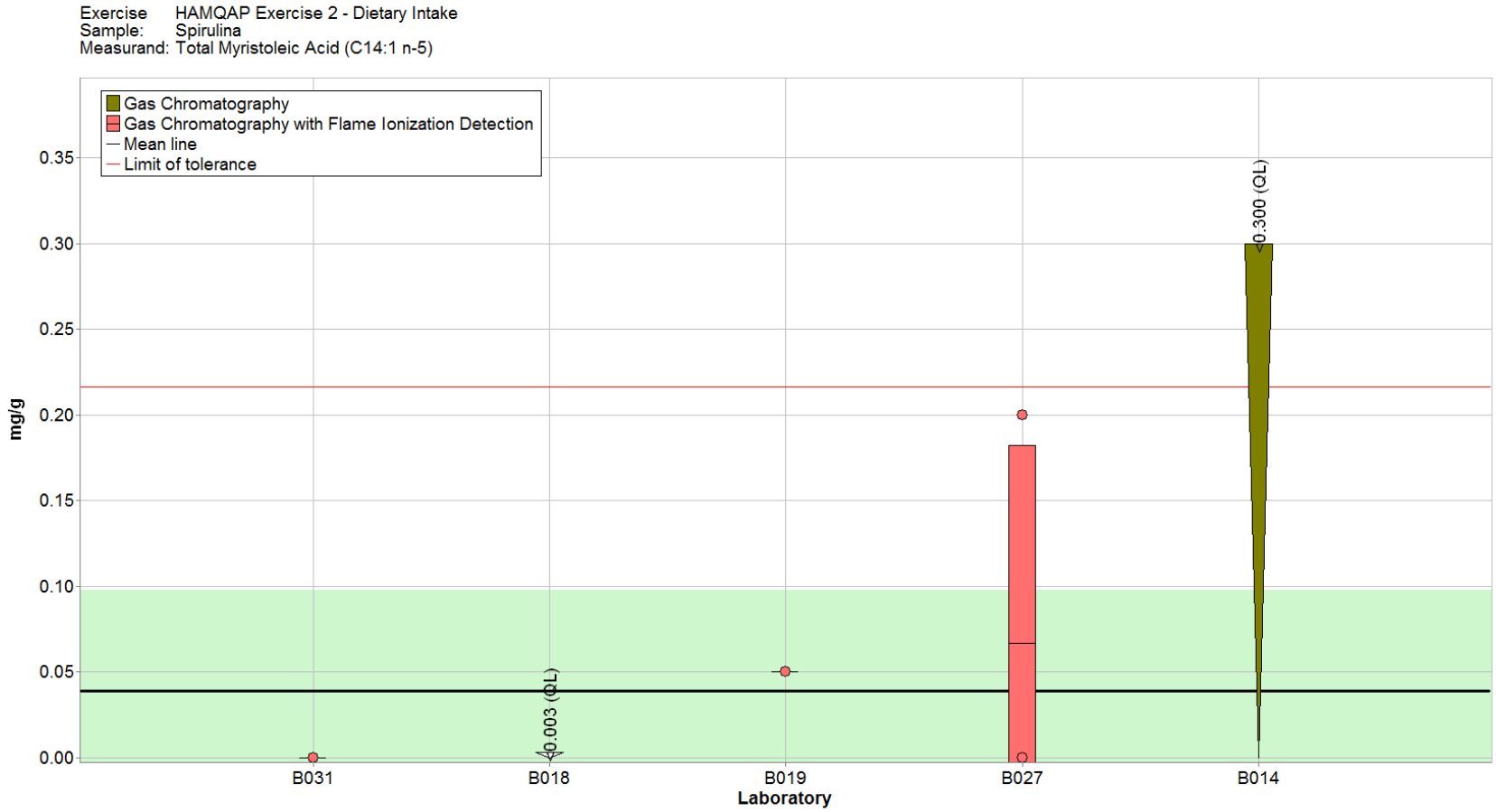

Figure 5-17. Total myristoleic acid (C14:1 n-5) in Spirulina (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

Table 5-7. Data summary table for total palmitic acid in SRM 1845a Whole Egg Powder, Palm Oil Powder, and Spirulina. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		Total Palmitic Acid (C16:0)														
		SRM 1845a Whole Egg Powder (mg/g)					Palm Oil Powder (mg/g)					Spirulina (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				82.20	2.60										
	B001															
	B002															
	B006															
	B014	81.9	86.1	83.6	83.9	2.1	259	266	269	264.7	5.1	20.3	20.6	20.4	20.43	0.15
	B015															
	B016	57.53	57.42	56.99	57.31	0.29	255	253.3	251.5	253.3	1.8	18.25	19.35	18.46	18.69	0.58
	B017															
	B018	284.129	284	280.5	282.9	2.1	472.1	466.9	471	470.0	2.7	545.2	537.7	538.88	540.6	4.0
	B019	9.614	9.809	9.839	9.75	0.12	30.996	29.628	30.234	30.29	0.69	2.574	2.538	2.605	2.572	0.034
	B027	11.7	11.9	11.76	11.79	0.10	0.98	0.99	0.99	0.9867	0.0058	2.6	2.68	3.22	2.83	0.34
	B031	116	111.45	112.4	113.3	2.4	353.85	332.2	345.9	344	11	27.5	29.65	29.65	28.9	1.2
	B034															
	B036						44.566	44.508	44.566	44.547	0.033					
	B038	9.77	9.21	9.29	9.42	0.30	29	28.9	28.8	28.9	0.1	2.16	2.22	2.36	2.25	0.10
	B039															
	B041															
	B042															
	B044															
	B045	113	110.2	112.7	112.0	1.5	91.2	93.4	92.5	92.4	1.1	13.86	18.93	15.29	16.0	2.6
Community Results		Consensus Mean			76		Consensus Mean			158		Consensus Mean			13	
		Consensus Standard Deviation			88		Consensus Standard Deviation			139		Consensus Standard Deviation			20	
		Maximum			282.9		Maximum			470.00		Maximum			540.6	
		Minimum			9.42		Minimum			0.9867		Minimum			2.25	
		N			8		N			9		N			8	

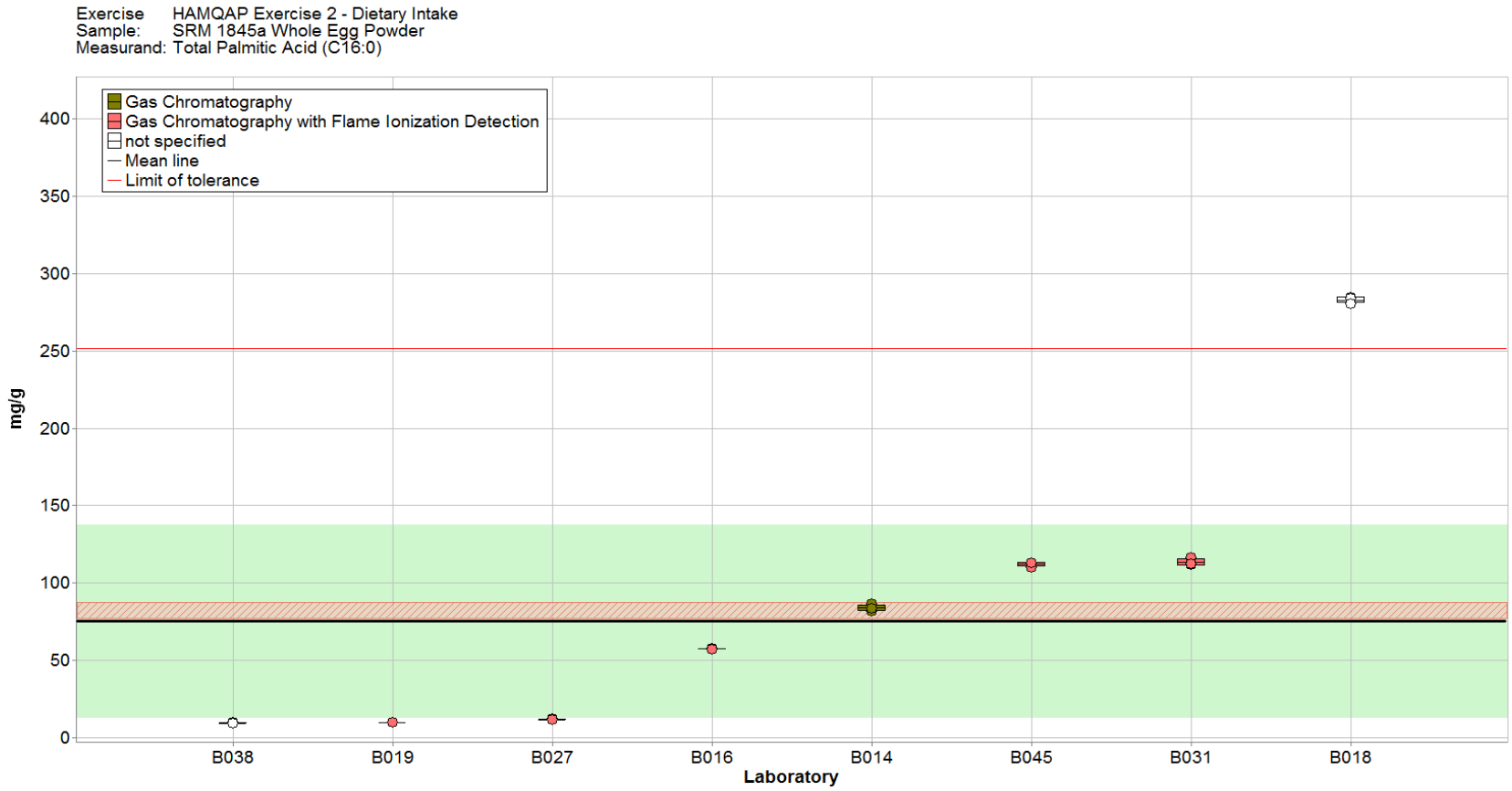

Figure 5-18. Total palmitic acid (C16:0) in SRM 1845a Whole Egg Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

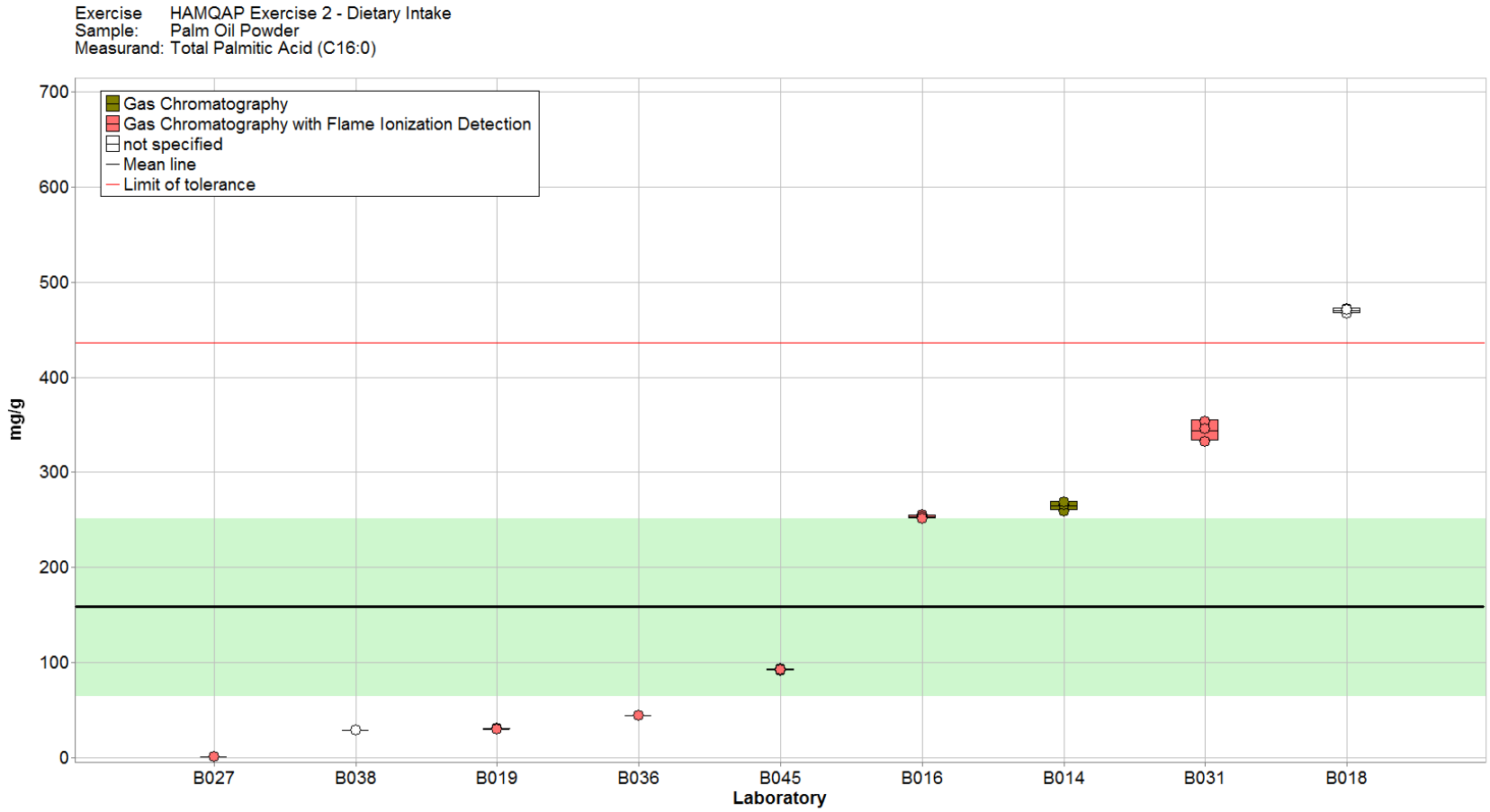

Figure 5-19. Total palmitic acid (C16:0) in Palm Oil Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

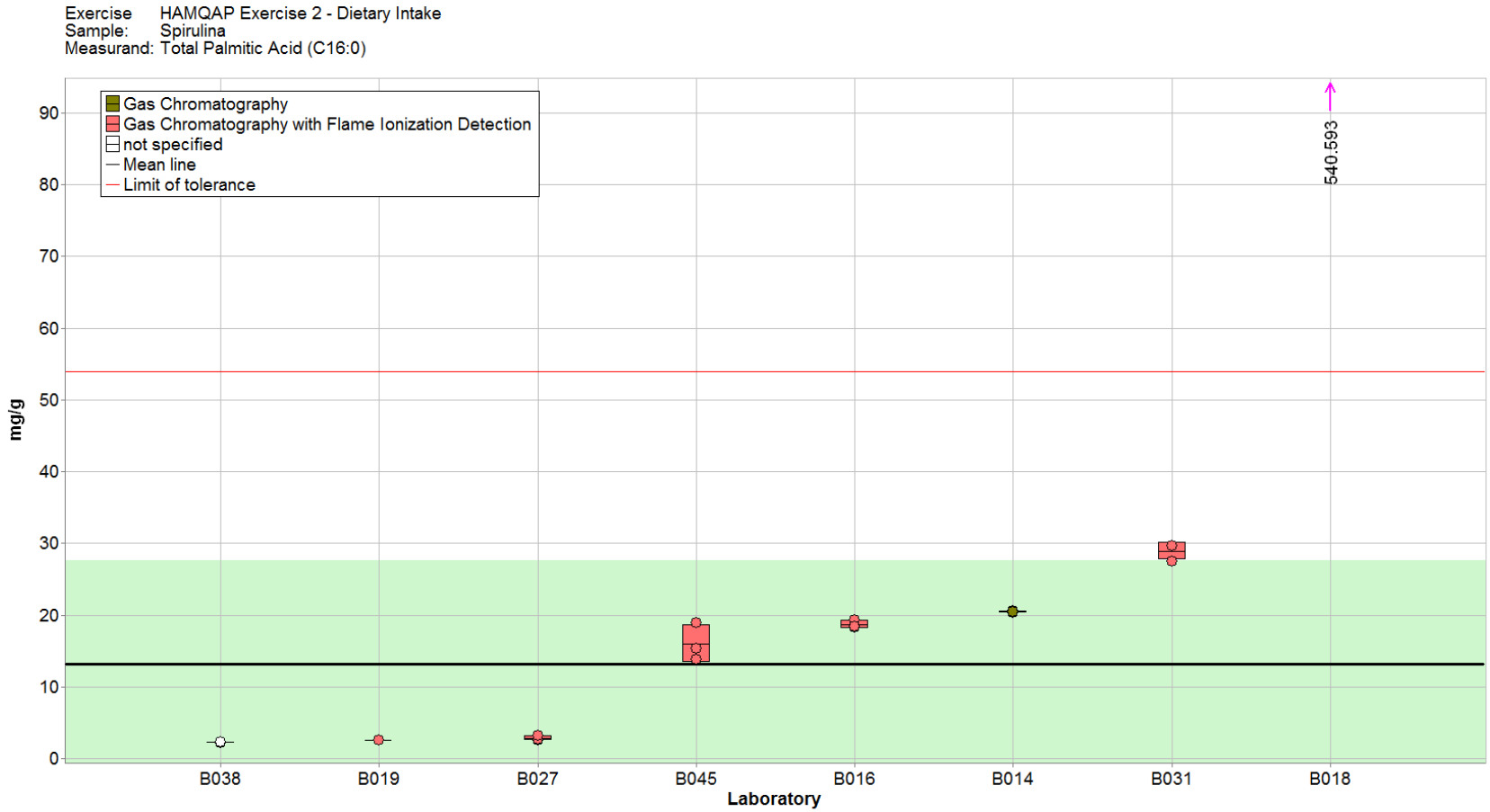

Figure 5-20. Total palmitic acid (C16:0) in Spirulina (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

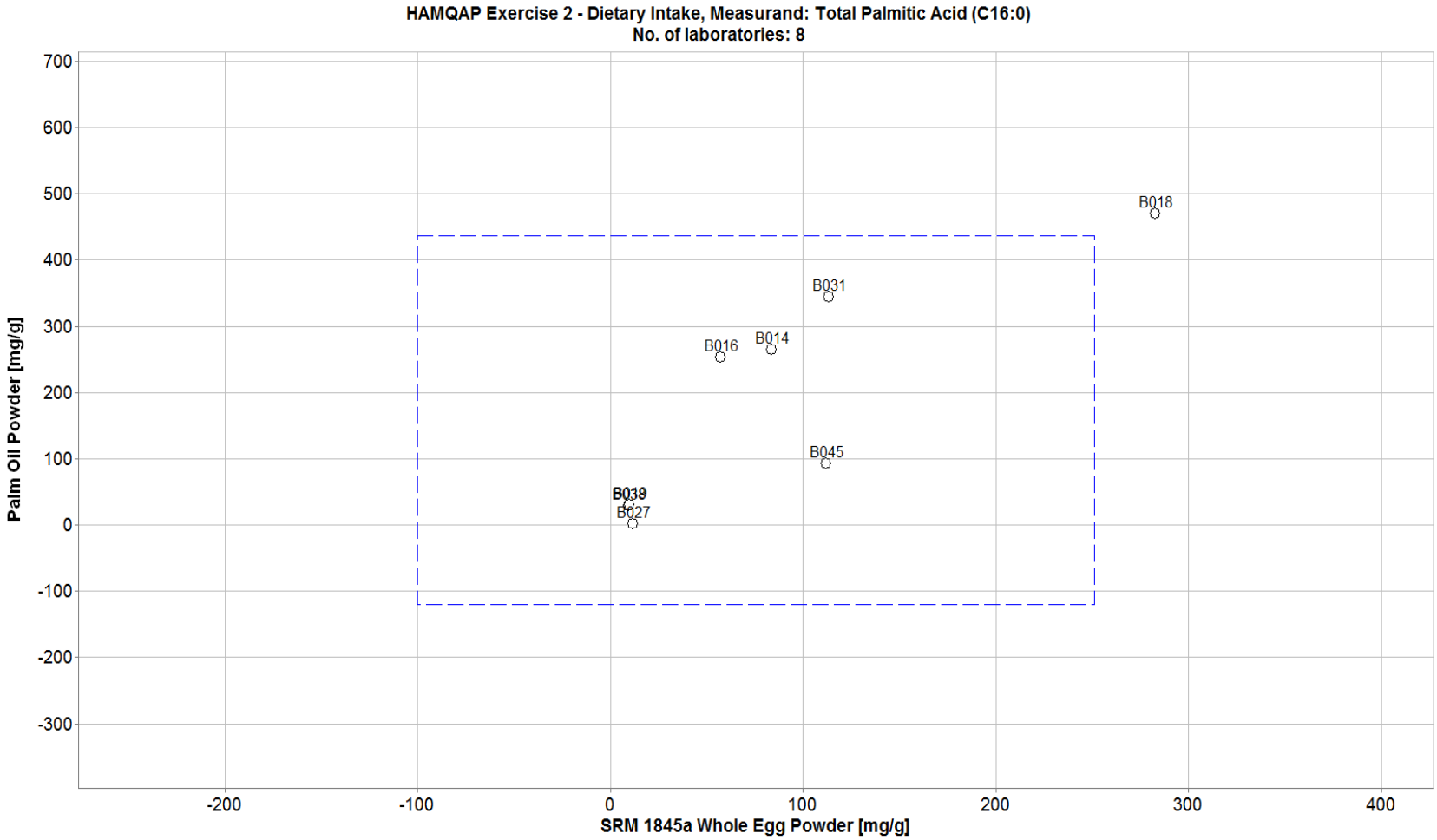

Figure 5-21. Laboratory means for total palmitic acid in SRM 1845a Whole Egg Powder and Palm Oil Powder (sample/sample comparison view). In this view, the individual laboratory mean for one sample (SRM 1845a) is compared to the mean for a second sample (Palm Oil Powder). The dotted blue box represents the consensus range of tolerance for SRM 1845a (x-axis) and Palm Oil Powder (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

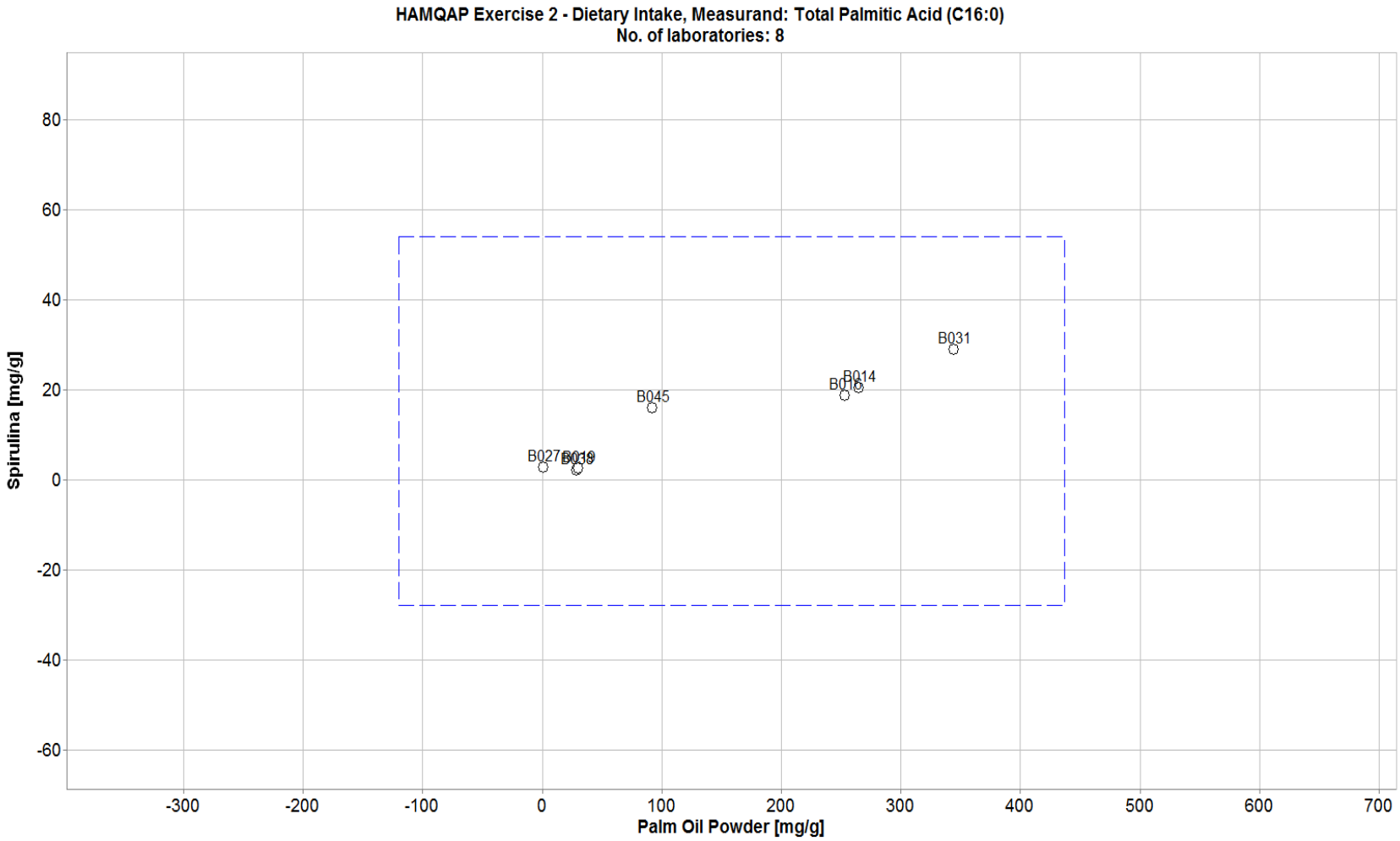

Figure 5-22. Laboratory means for total palmitic acid in Palm Oil Powder and Spirulina (sample/sample comparison view). In this view, the individual laboratory mean for one sample (Palm Oil Powder) is compared to the mean for a second sample (Spirulina). The dotted blue box represents the consensus range of tolerance for Palm Oil Powder (x-axis) and Spirulina (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 total palmitoleic acid in SRM 1845a Whole Egg Powder, Palm Oil Powder, and Spirulina. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		Total Palmitoleic Acid (C16:1 n-7)														
		SRM 1845a Whole Egg Powder (mg/g)					Palm Oil Powder (mg/g)					Spirulina (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				8.31	0.23										
	B001															
	B002															
	B006															
	B014	8.01	8.4	8.1	8.17	0.20	0.86	0.89	0.89	0.880	0.017	1.62	1.65	1.63	1.633	0.015
	B015															
	B016	5.41	5.39	5.29	5.363	0.064	0.867	0.858	0.854	0.8597	0.0067	1.5	1.56	1.49	1.517	0.038
	B017															
	B018	20.55	20.084	22.46	21.0	1.3	1.331	1.441	1.299	1.357	0.074	50.42	50	50.83	50.42	0.42
	B019	0.905	0.931	0.934	0.923	0.016	0.106	0.096	0.094	0.0987	0.0064	0.209	0.206	0.212	0.209	0.003
	B027	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
	B031	10.45	9.95	10.05	10.15	0.26	1.05	0.95	1	1	0.05	2.15	2.3	2.3	2.25	0.09
	B034															
	B036						0.152	0.151	0.152	0.15208	0.00062					
	B039															
	B041															
	B042															
	B044															
	B045	10.27	9.83	9.71	9.94	0.29	0.36	0.4	0.38	0.380	0.020	1.02	1.37	1.12	1.2	0.2
Community Results		Consensus Mean			7.939		Consensus Mean			0.591		Consensus Mean			1.13	
		Consensus Standard Deviation			9.647		Consensus Standard Deviation			0.552		Consensus Standard Deviation			1.526	
		Maximum			21.0313		Maximum			1.357		Maximum			50.4167	
		Minimum			0		Minimum			0		Minimum			0	
		N			7		N			8		N			7	

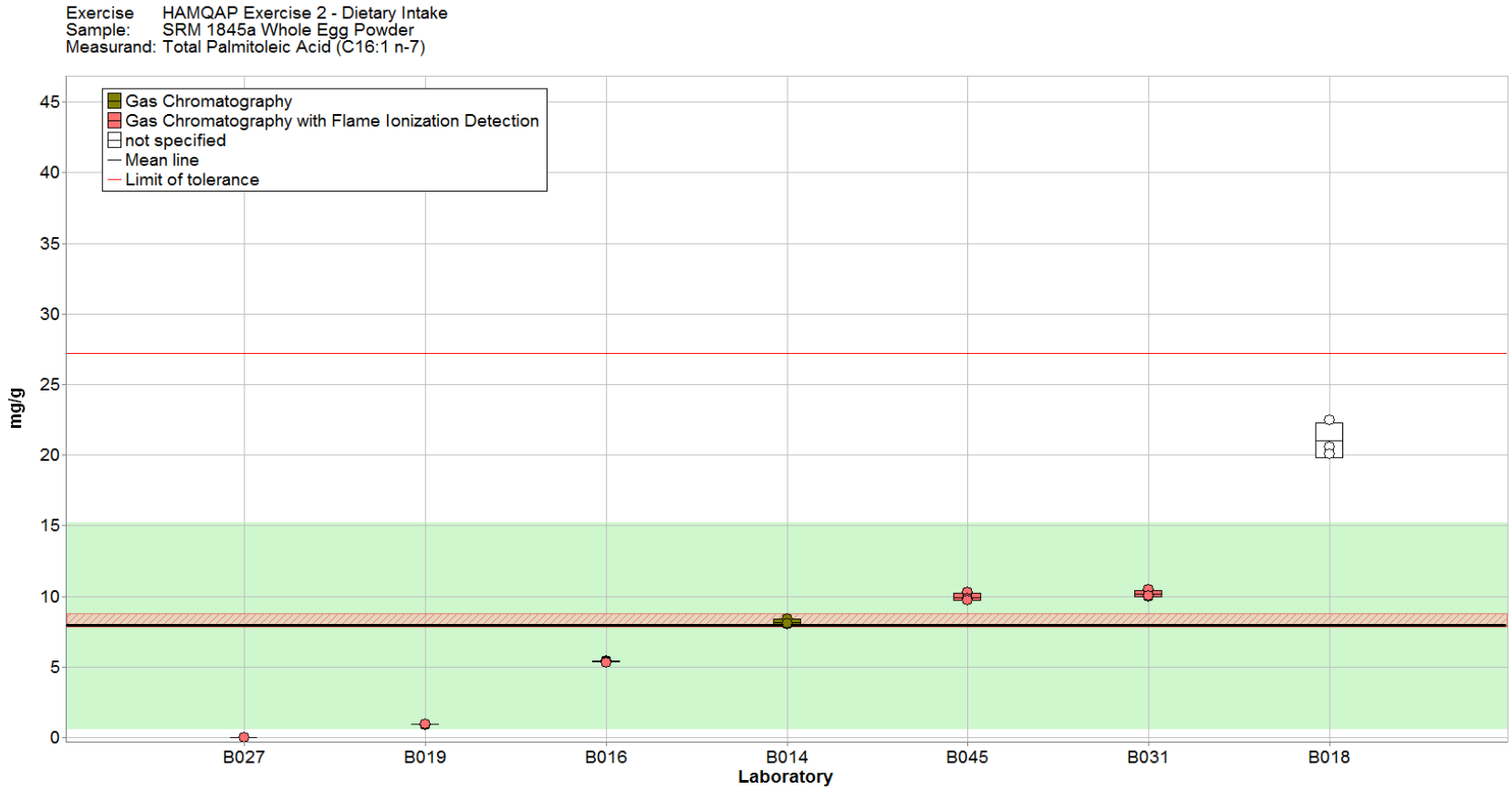

Figure 5-23. Total palmitoleic acid (C16:1 n-7) in SRM 1845a Whole Egg Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

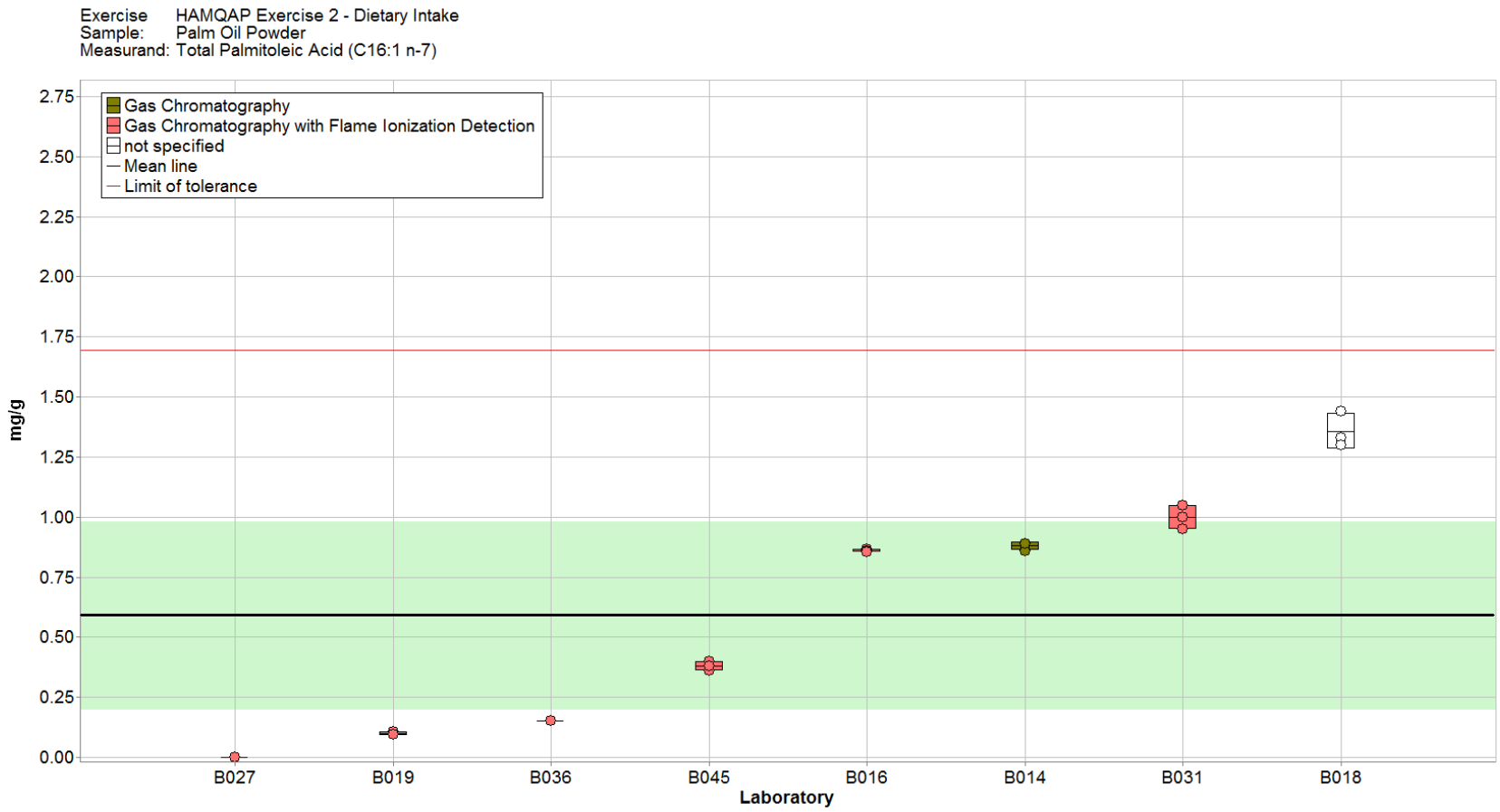

Figure 5-24. Total palmitoleic acid (C16:1 n-7) in Palm Oil Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

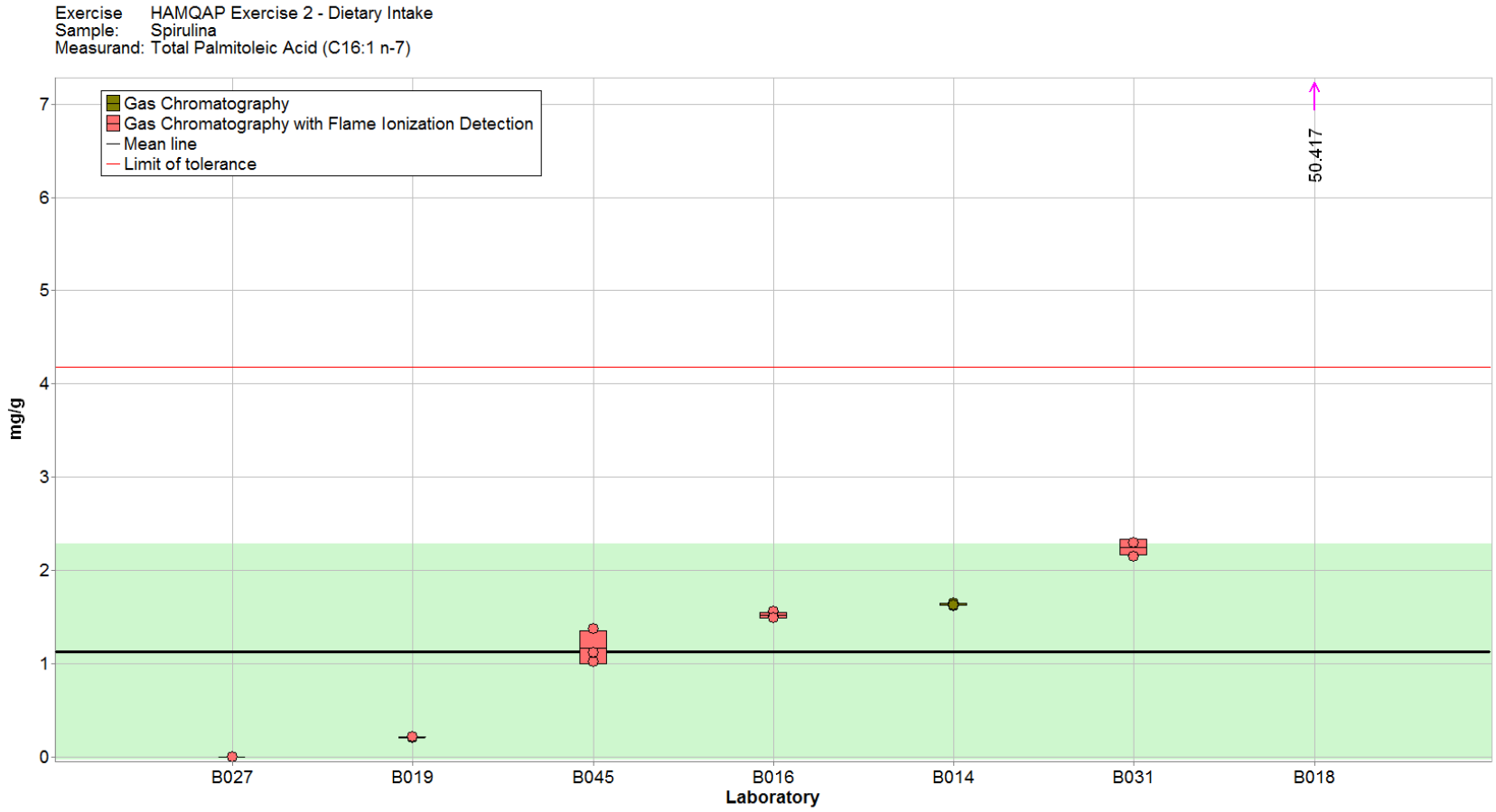

Figure 5-25. Total palmitoleic acid (C16:1 n-7) in Spirulina (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

Table 5-9. Data summary table for total stearic acid in SRM 1845a Whole Egg Powder, Palm Oil Powder, and Spirulina. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		Total Stearic Acid (C18:0)														
		SRM 1845a Whole Egg Powder (mg/g)					Palm Oil Powder (mg/g)					Spirulina (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				28.02	0.95										
	B001															
	B002															
	B006															
	B014	29.2	30.5	29.4	29.70	0.70	27.2	28.8	29.1	28.4	1.0	0.37	0.37	0.34	0.360	0.017
	B015						16.64	19.28	17.96	18.0	1.3					
	B016	20.05	20.4	20.3	20.25	0.18	26.83	26.7	26.5	26.68	0.17	0.495	0.558	0.474	0.509	0.044
	B017															
	B018	115.141	115.6	108	112.9	4.3	41.13	41.61	41.35	41.36	0.24	4.795	4.63	4.581	4.67	0.11
	B019	3.31	3.38	3.3	3.330	0.044	3.04	3.02	2.94	3.000	0.053	0.05	0.05	0.05	0.05	0
	B027	33.92	34.07	34.04	34.010	0.079	30.01	30.05	29.77	29.94	0.15	0.45	0.46	0.56	0.490	0.061
	B031	39.85	38	38.5	38.78	0.96	34.8	32.75	34.05	33.9	1.0	0.6	0.6	0.6	0.6	0
	B033	3.024	3.123	3.285	3.14	0.13	3.1	2.979	2.9	2.99	0.10	0.057	0.056	0.057	0.05690	0.00069
	B034															
	B036						4.736	4.725	4.736	4.732	0.007					
	B038	3.37	3.17	3.2	3.25	0.11	2.99	2.96	2.95	2.967	0.021	0	0	0	0	0
	B039															
	B041															
	B042															
	B044															
	B045	41.95	39.5	40.06	40.5	1.3	7.74	8.21	7.95	7.97	0.24	0.33	0.44	0.4	0.390	0.056
Community Results		Consensus Mean			23		Consensus Mean			18		Consensus Mean			0.31	
		Consensus Standard Deviation			23		Consensus Standard Deviation			12		Consensus Standard Deviation			0.31	
		Maximum			112.9		Maximum			41.36		Maximum			4.67	
		Minimum			3.14		Minimum			2.967		Minimum			0	
		N			9		N			11		N			9	

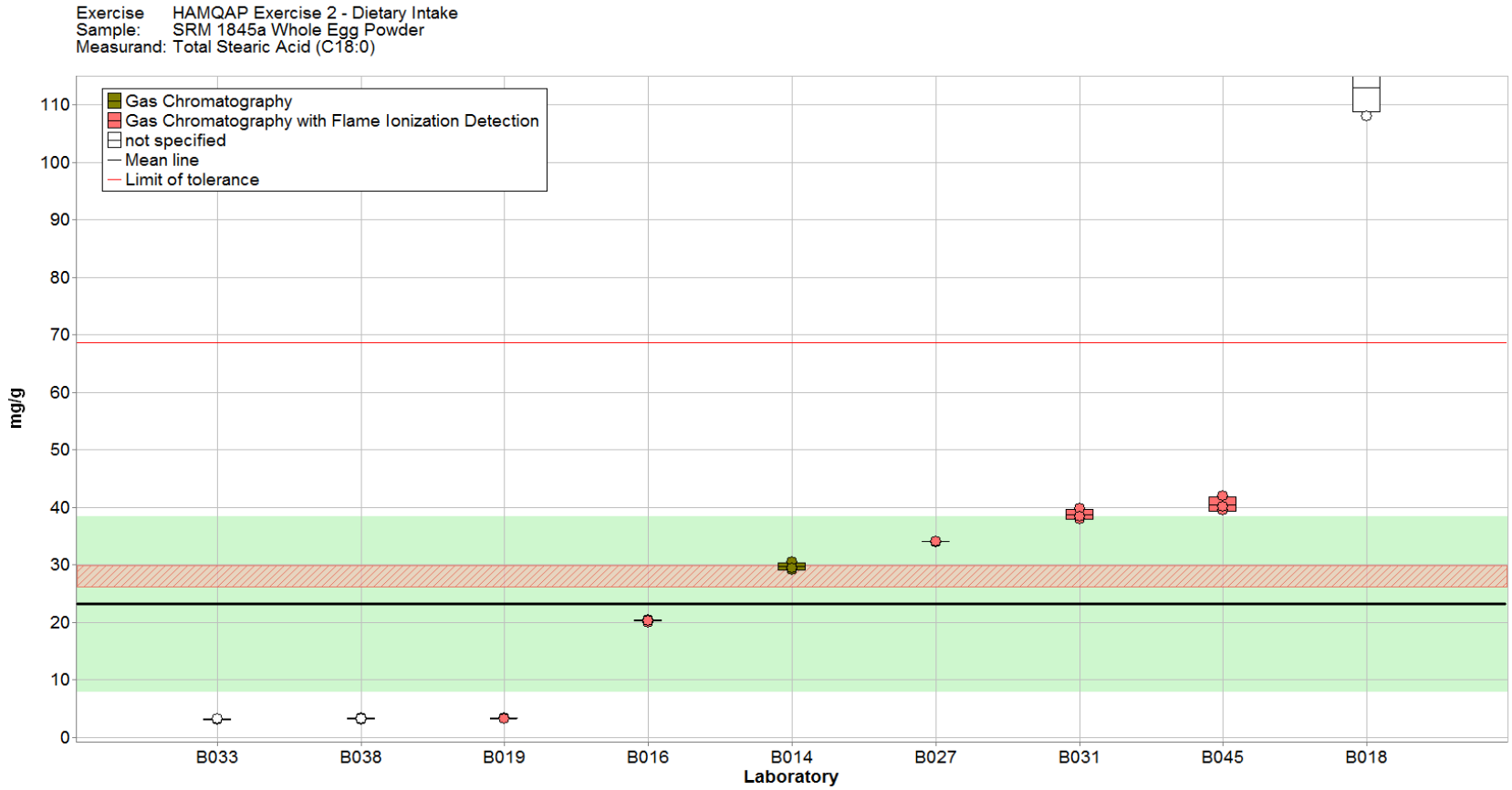

Figure 5-26. Total stearic acid (C18:0) in SRM 1845a Whole Egg Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

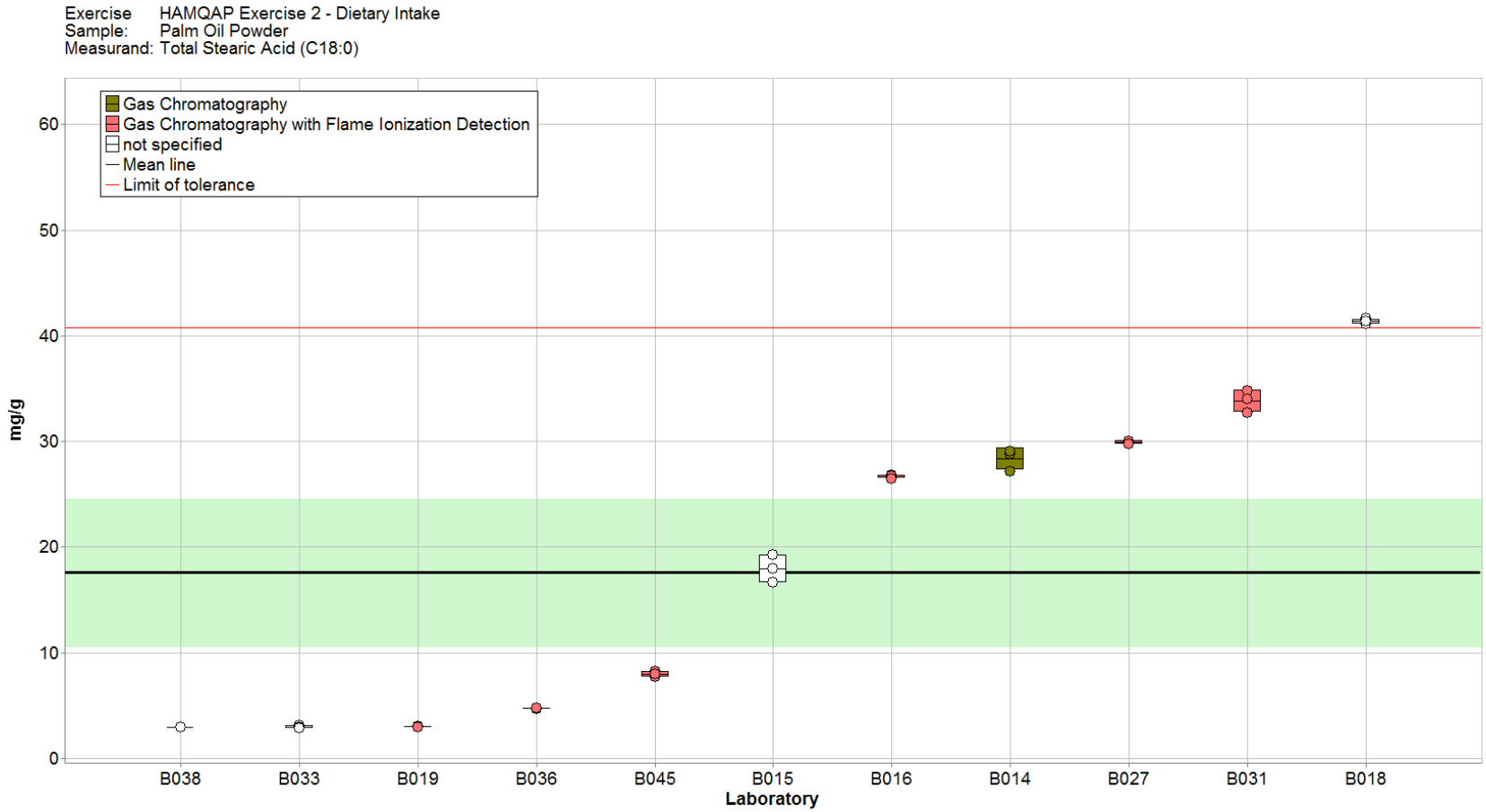

Figure 5-27. Total stearic acid (C18:0) in Palm Oil Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

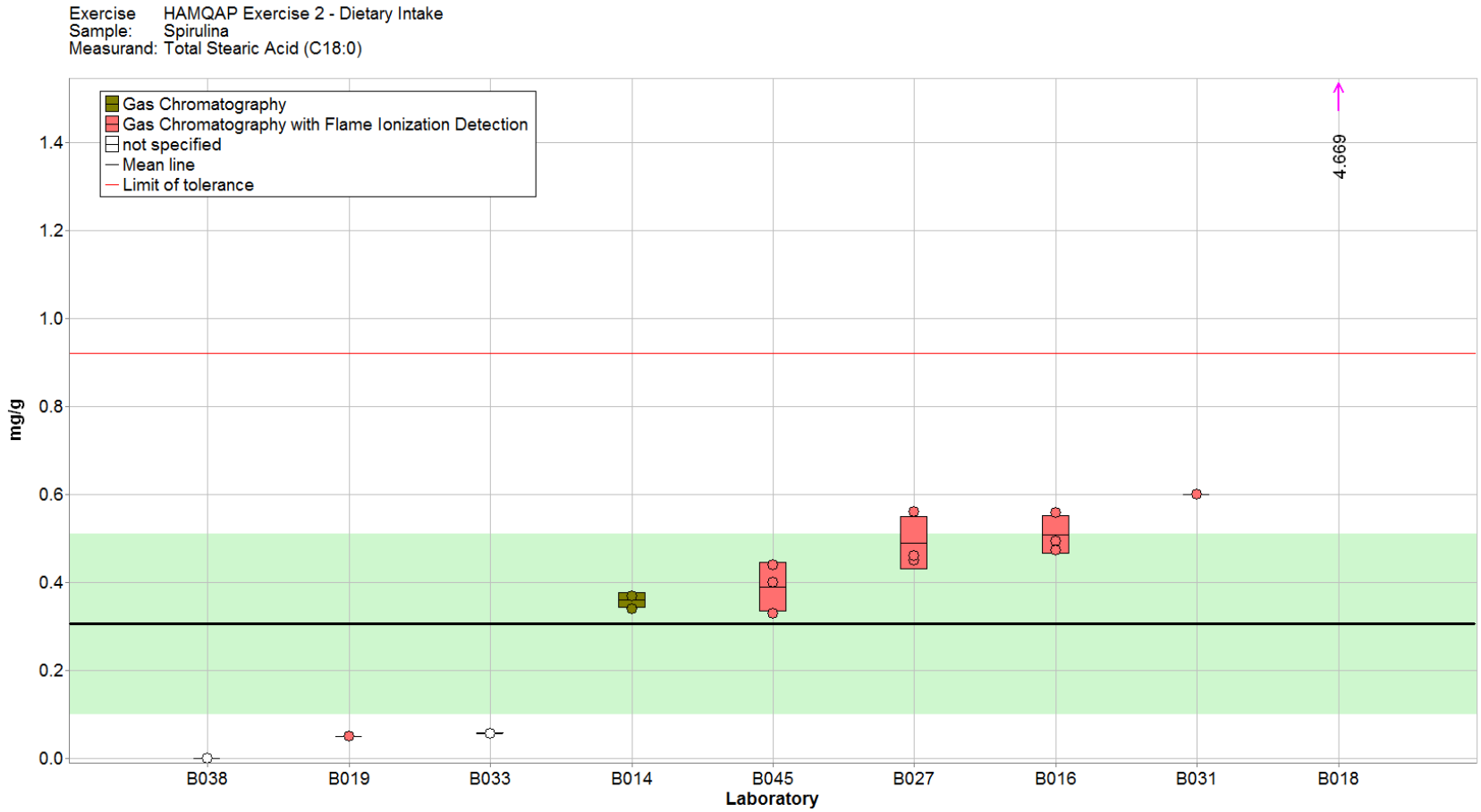

Figure 5-28. Total stearic acid (C18:0) in Spirulina (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

Table 5-10. Data summary table for total *cis*-vaccenic acid in SRM 1845a Whole Egg Powder, Palm Oil Powder, and Spirulina. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		Total cis-Vaccenic Acid (C18:1 n-7)														
		SRM 1845a Whole Egg Powder (mg/g)					Palm Oil Powder (mg/g)					Spirulina (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				5.32	0.15										
	B001															
	B002															
	B006															
	B014	5.19	5.45	5.25	5.30	0.14	3.74	3.93	3.93	3.87	0.11	< 0.300	< 0.300	< 0.300		
	B016	3.66	3.72	3.64	3.673	0.042	3.75	3.76	3.74	3.75	0.01					
	B017															
	B018	11.89	11.7	12.33	11.97	0.32	5.175	5.18	5.11	5.155	0.039	1.93	1.57	2.11	1.87	0.27
	B027	6.54	7.16	6.83	6.84	0.31	3.97	4.18	4.01	4.05	0.11	0.27	0.27	0.35	0.297	0.046
	B031	7.1	6.75	6.8	6.88	0.19	4.7	4.35	4.45	4.50	0.18	0.15	0.15	0.15	0.15	0
	B034															
	B036															
	B039															
	B041															
	B042															
	B044															
Community Results		Consensus Mean			6.9		Consensus Mean			4.26		Consensus Mean			0.44	
		Consensus Standard Deviation			3.5		Consensus Standard Deviation			0.58		Consensus Standard Deviation			0.41	
		Maximum			11.97		Maximum			5.155		Maximum			1.87	
		Minimum			3.673		Minimum			3.75		Minimum			0.15	
		N			5		N			5		N			3	

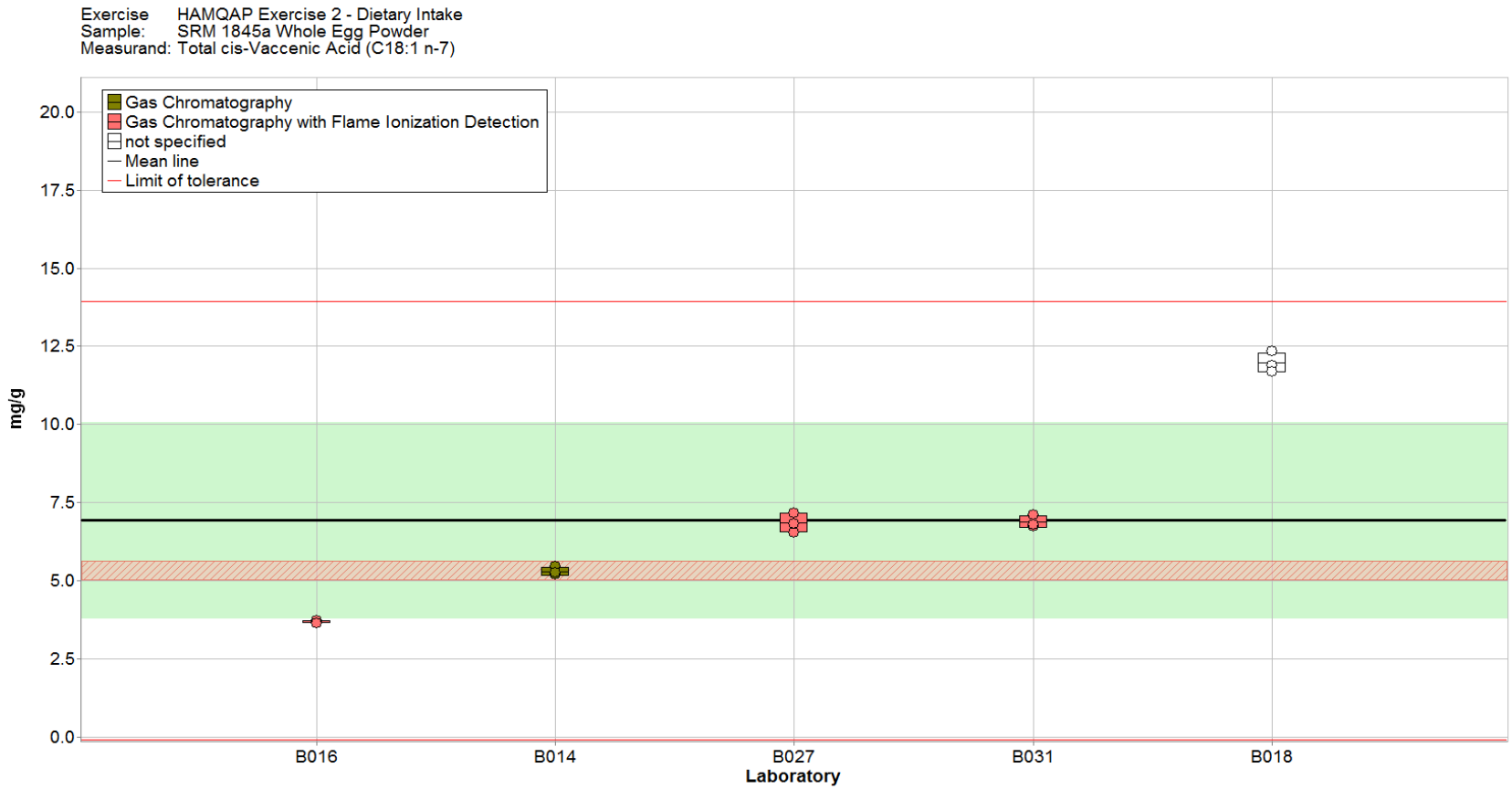

Figure 5-29. Total *cis*-vaccenic acid (C18:1 n-7) in SRM 1845a Whole Egg Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. A NIST value has not been determined in this material.

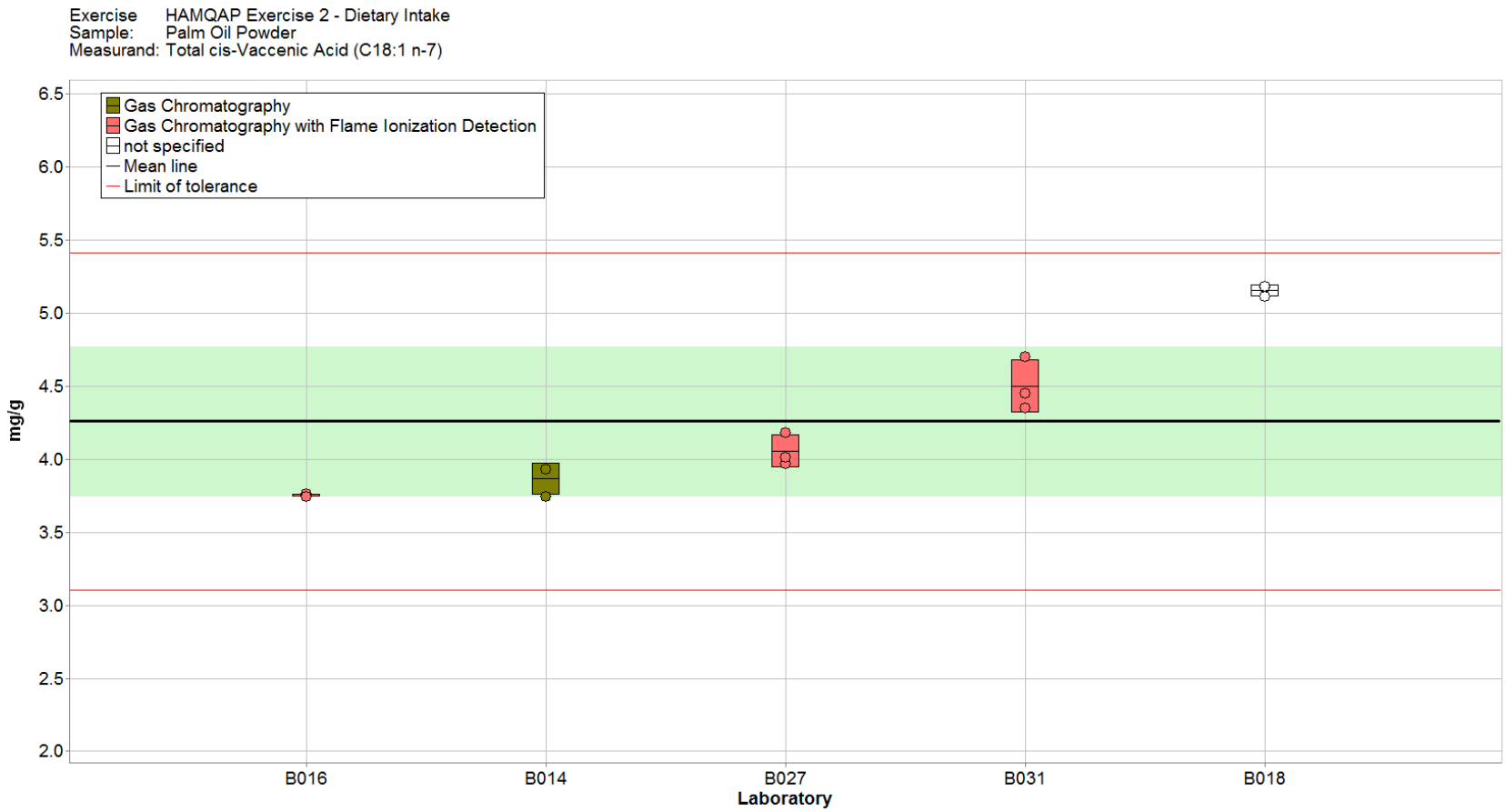

Figure 5-30. Total *cis*-vaccenic acid (C18:1 n-7) in Palm Oil Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

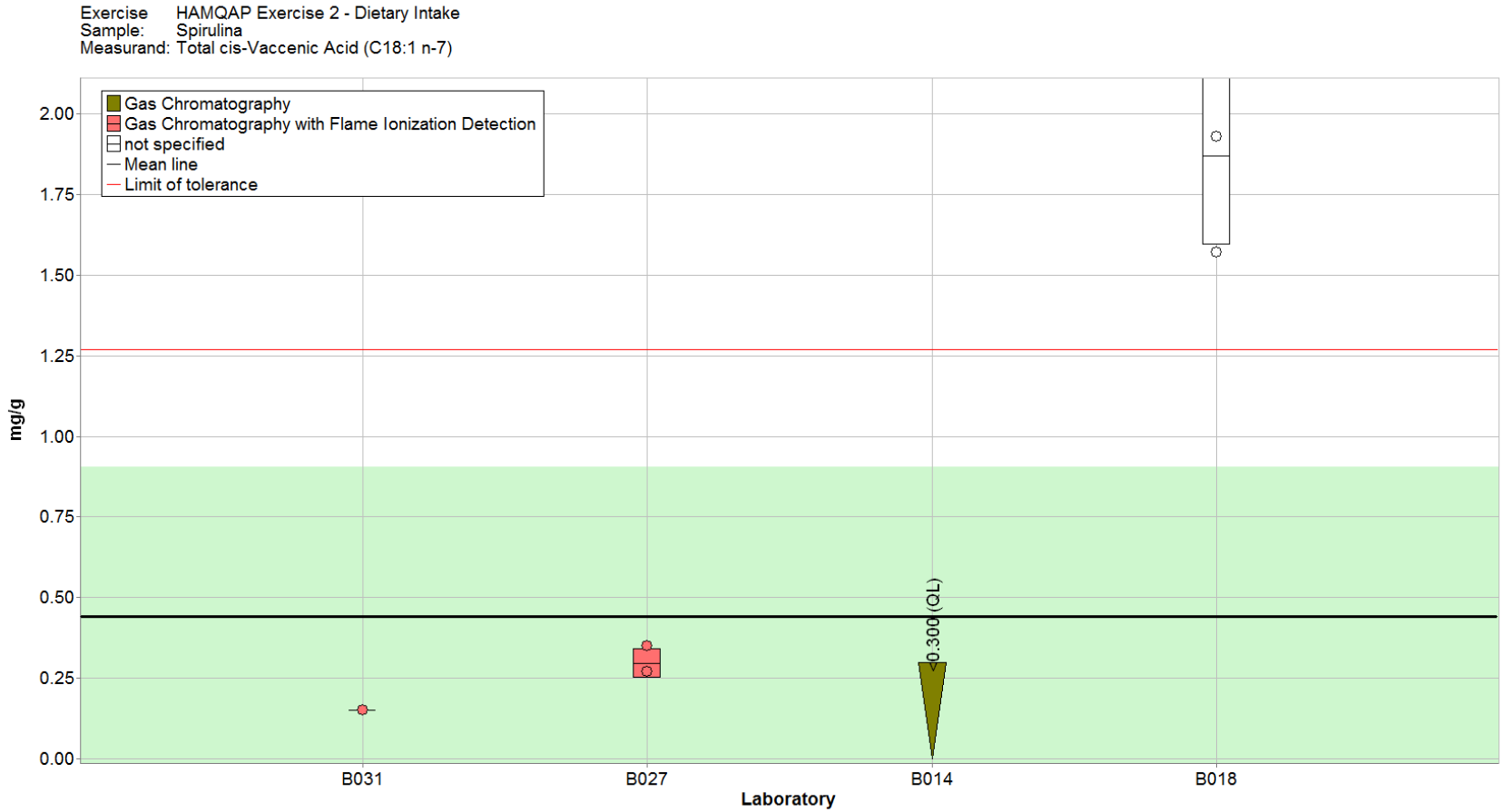

Figure 5-31. Total *cis*-vaccenic acid (C18:1 n-7) in Spirulina (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

Table 5-11. Data summary table for total oleic acid in SRM 1845a Whole Egg Powder, Palm Oil Powder, and Spirulina. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		Total Oleic Acid (C18:1 n-9)														
		SRM 1845a Whole Egg Powder (mg/g)					Palm Oil Powder (mg/g)					Spirulina (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				110.00	14.00										
	B001															
	B002															
	B006															
	B014	116	122	118	118.7	3.1	213	222	224	219.7	5.9	1.27	1.32	1.18	1.257	0.071
	B015						131.78	150.16	141.87	141.3	9.2					
	B016	84.78	86.05	84.98	85.27	0.68	217.6	216.6	215.4	216.5	1.1	1.4	1.72	1.39	1.50	0.19
	B017															
	B018	346.4	346.66	358.89	350.7	7.1	375.995	378.8	376.59	377.1	1.5	23.99	23.04	23.1	23.38	0.53
	B019	13.76	14.01	13.75	13.84	0.15	24.36	24.16	23.55	24.02	0.42	0.15	0.15	0.13	0.143	0.012
	B027	137.61	138.76	137.91	138.09	0.60	257.69	236.28	255.92	250	12	1.43	1.47	2.03	1.64	0.34
	B031	172.05	164.55	166.85	167.8	3.8	297.9	280.6	292.2	290.2	8.8	1.45	1.55	1.65	1.55	0.10
	B033	13.236	12.245	12.348	12.61	0.54	25.921	24.177	25.178	25.09	0.87	0.143	0.142	0.139	0.141267	0.0021
	B034															
	B036						38.098	38.079	38.098	38.091	0.011					
	B038	13.1	12.4	12.5	12.67	0.38	21.9	21.6	21.6	21.70	0.17	0	0	0	0	0
	B039															
	B041															
	B042															
	B044															
	B045	146.32	144.16	142.08	144.2	2.1	74.26	77.38	75.15	75.6	1.6	0.65	0.86	0.72	0.74	0.11
Community Results		Consensus Mean			103		Consensus Mean			149		Consensus Mean			0.9	
		Consensus Standard Deviation			89		Consensus Standard Deviation			124		Consensus Standard Deviation			1.1	
		Maximum			350.7		Maximum			377.1		Maximum			23.38	
		Minimum			12.61		Minimum			21.70		Minimum			0	
		N			9		N			11		N			9	

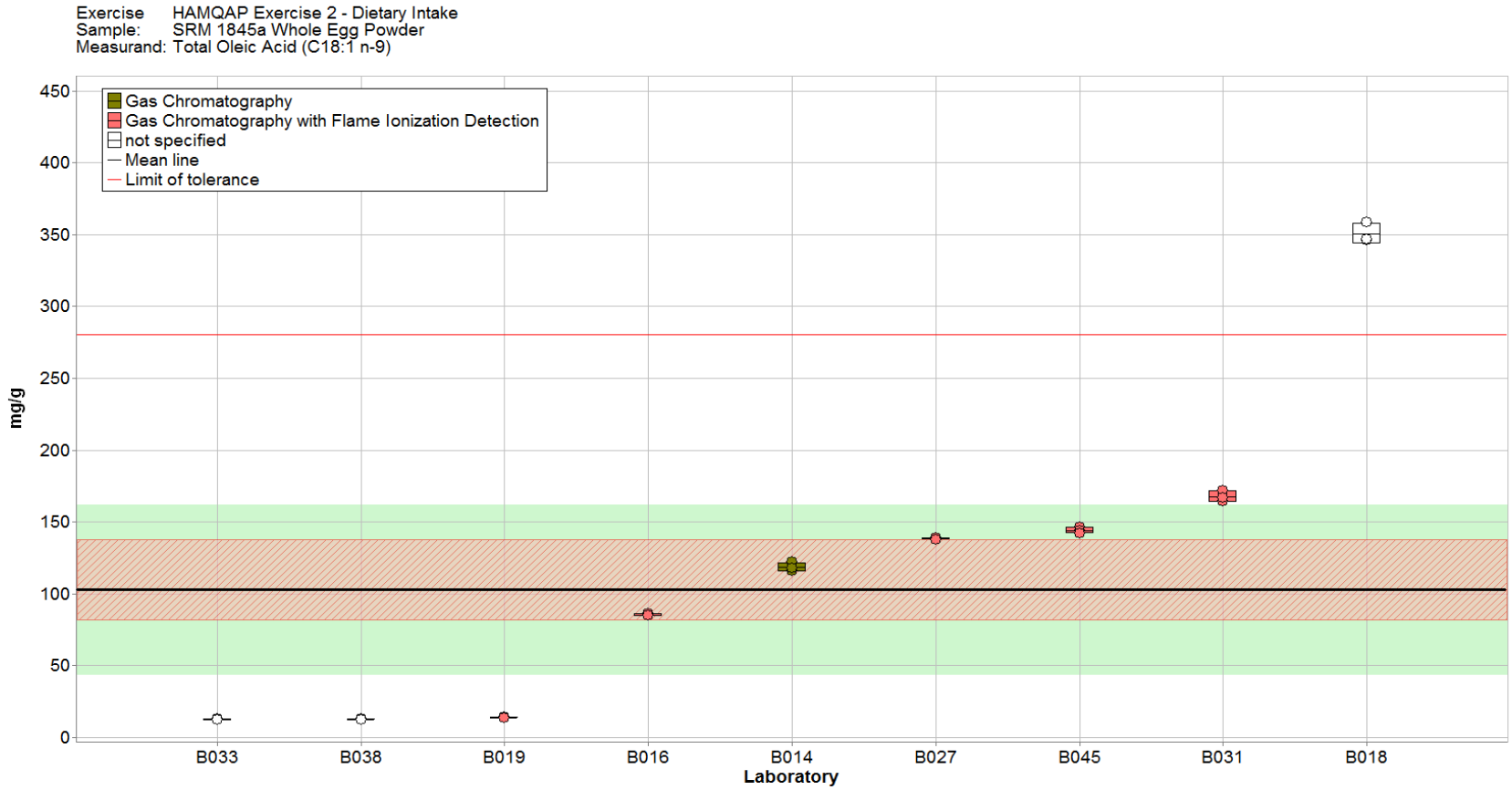

Figure 5-32. Total oleic acid (C18:1 n-9) in SRM 1845a Whole Egg Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

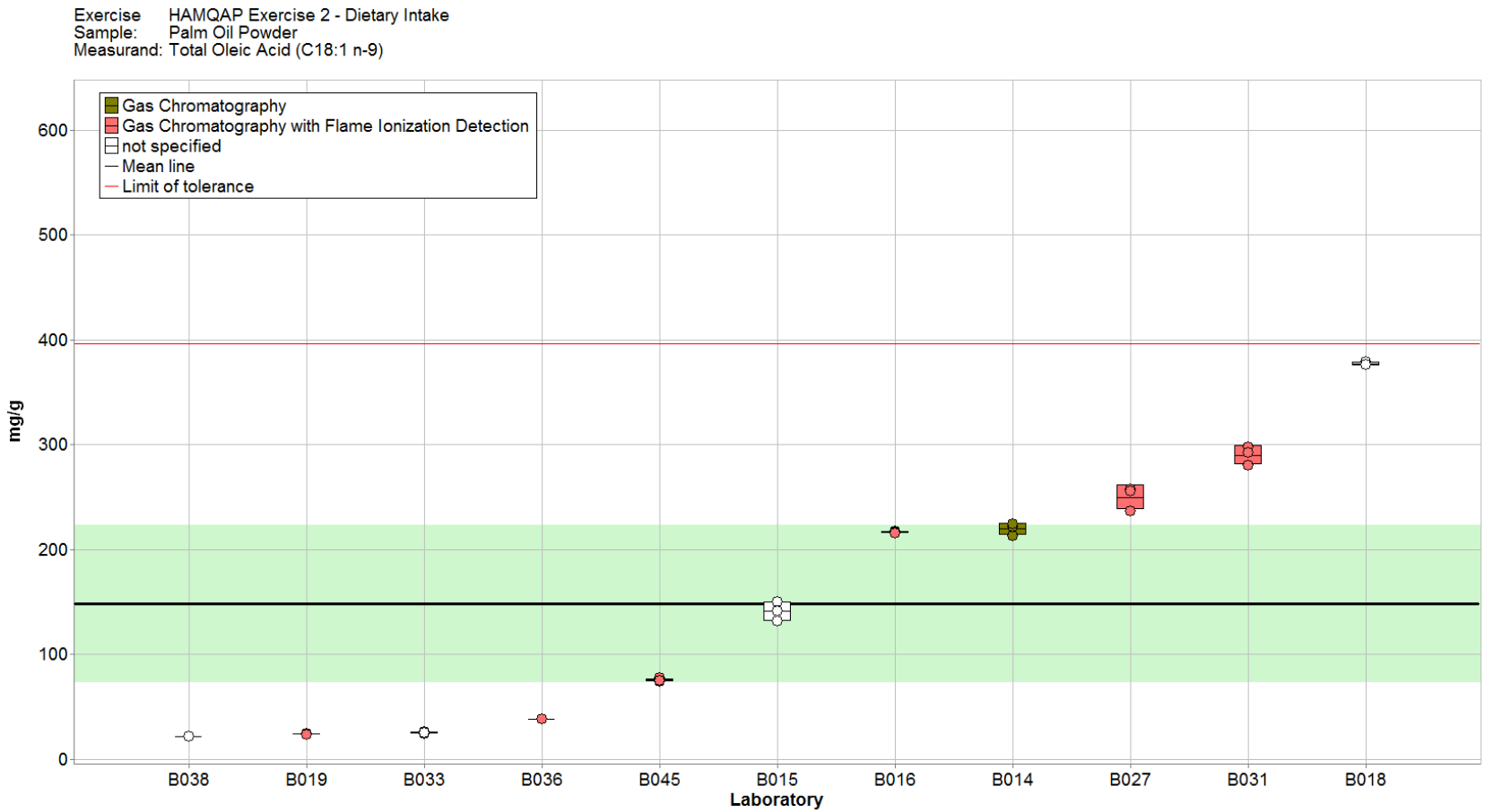

Figure 5-33. Total oleic acid (C18:1 n-9) in Palm Oil Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

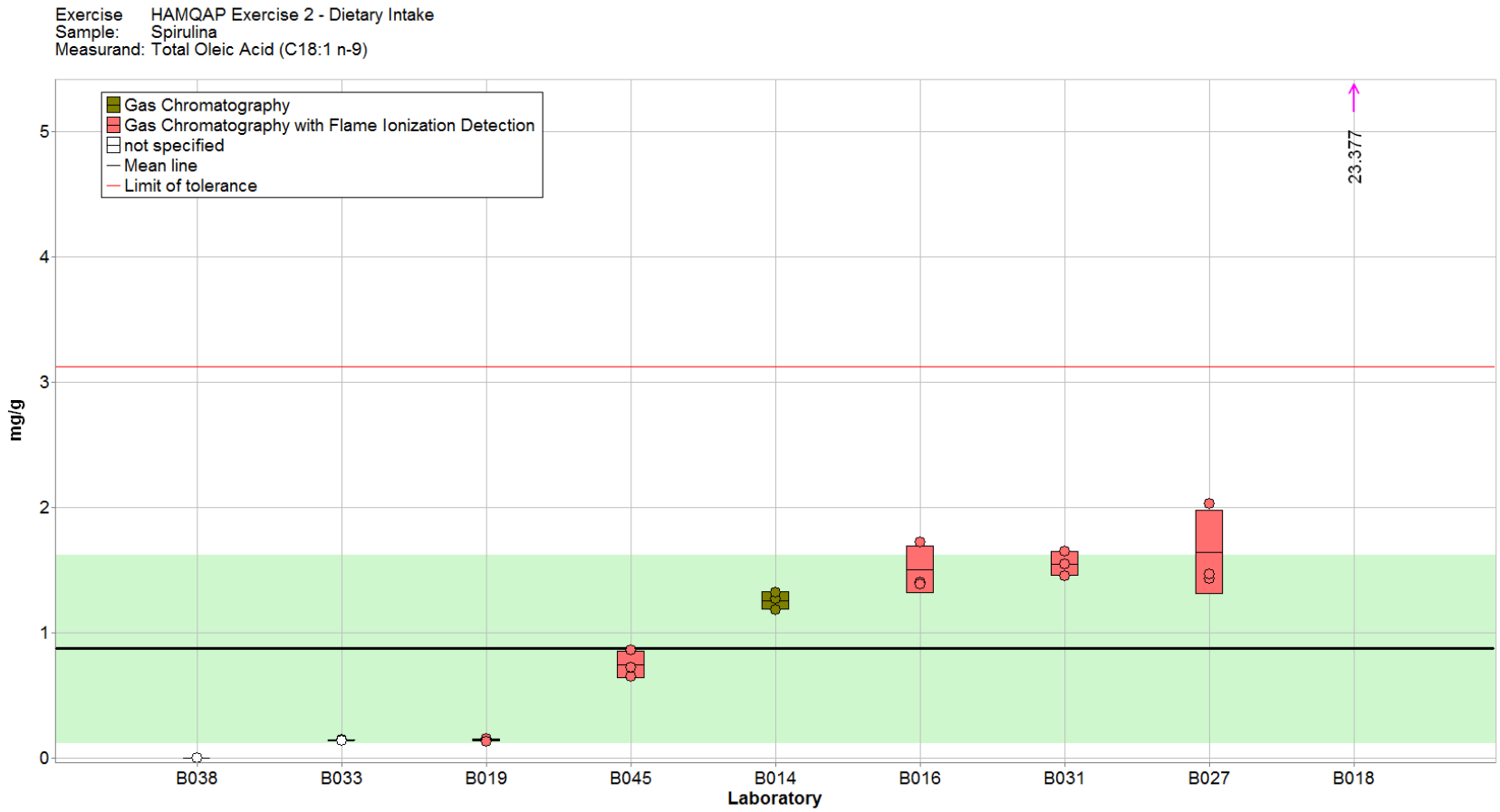

Figure 5-34. Total oleic acid (C18:1 n-9) in Spirulina (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

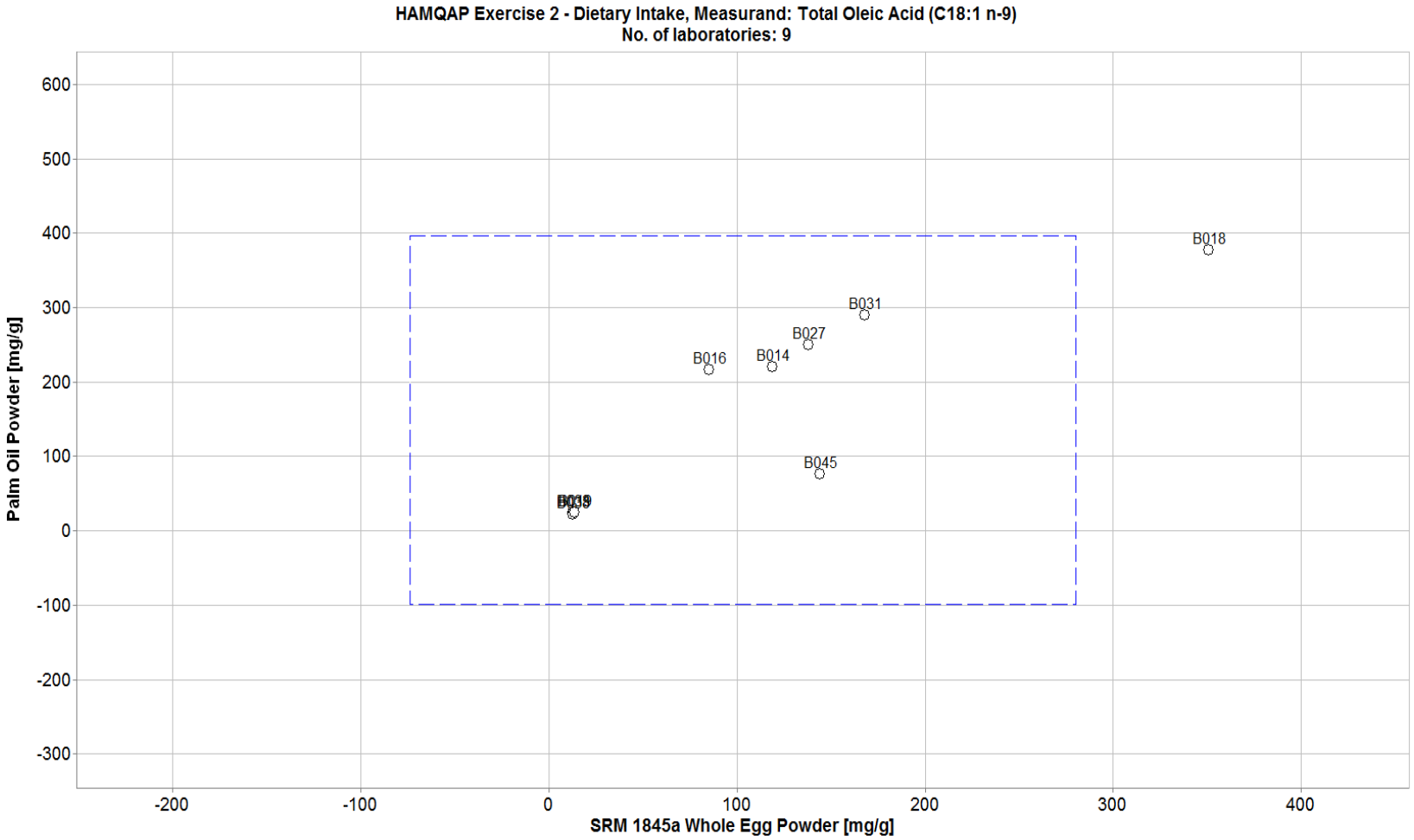

Figure 5-35. Laboratory means for total oleic acid in SRM 1845a Whole Egg Powder and Palm Oil Powder (sample/sample comparison view). In this view, the individual laboratory mean for one sample (SRM 1845a) is compared to the mean for a second sample (Palm Oil Powder). The dotted blue box represents the consensus range of tolerance for SRM 1845a (x-axis) and Palm Oil Powder (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

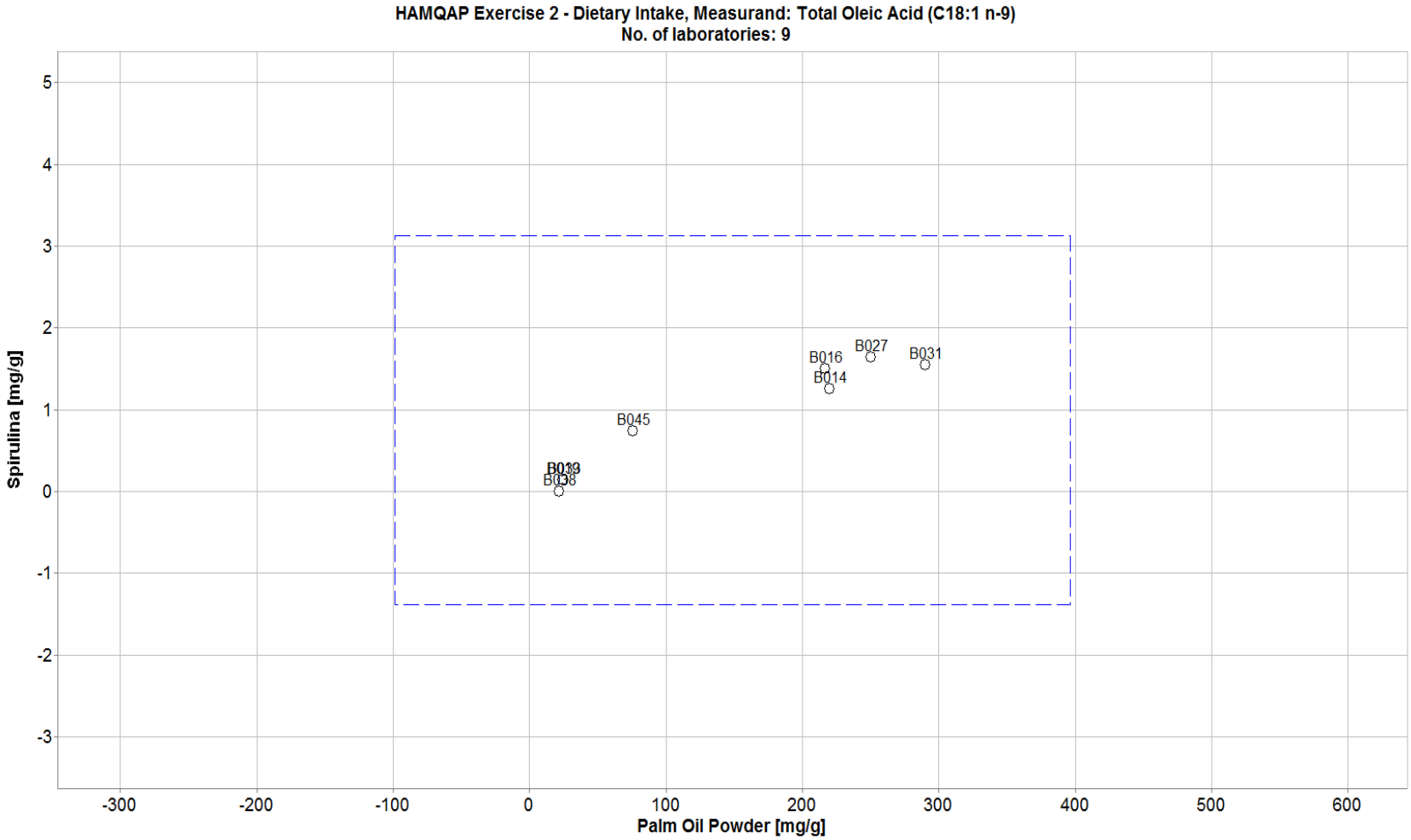

Figure 5-36. Laboratory means for total oleic acid in Palm Oil Powder and Spirulina (sample/sample comparison view). In this view, the individual laboratory mean for one sample (Palm Oil Powder) is compared to the mean for a second sample (Spirulina). The dotted blue box represents the consensus range of tolerance for Palm Oil Powder (x-axis) and Spirulina (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 5-12. Data summary table for total *trans*-vaccenic acid in SRM 1845a Whole Egg Powder, Palm Oil Powder, and Spirulina.

		Total Transvaccenic Acid (C18:1 n-7t)														
		SRM 1845a Whole Egg Powder (mg/g)					Palm Oil Powder (mg/g)					Spirulina (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				0.12	0.04										
	B001															
	B002															
	B006															
	B014	< 0.300	< 0.300	< 0.300			< 0.500	< 0.500	< 0.500			< 0.300	< 0.300	< 0.300		
	B016															
	B017															
	B018	< 0.003	< 0.003	< 0.003			0.968	1.006	1.044	1.006	0.038	< 0.003	< 0.003	< 0.003		
	B027	1.24	1.4	1.5	1.38	0.13	0	0.59	0	0.20	0.34	0	0	0	0	0
	B031	0.25	0.25	0.25	0.25	0	0.1	0.1	0.1	0.1	0	0	0	0	0	0
	B034															
	B036															
	B039															
	B041															
	B042															
	B044															
Community Results		Consensus Mean			0.8		Consensus Mean			0.43		Consensus Mean			0	
		Consensus Standard Deviation			2.3		Consensus Standard Deviation			0.86		Consensus Standard Deviation			0	
		Maximum			1.38		Maximum			1.006		Maximum			0	
		Minimum			0.25		Minimum			0.1		Minimum			0	
		N			2		N			3		N			2	

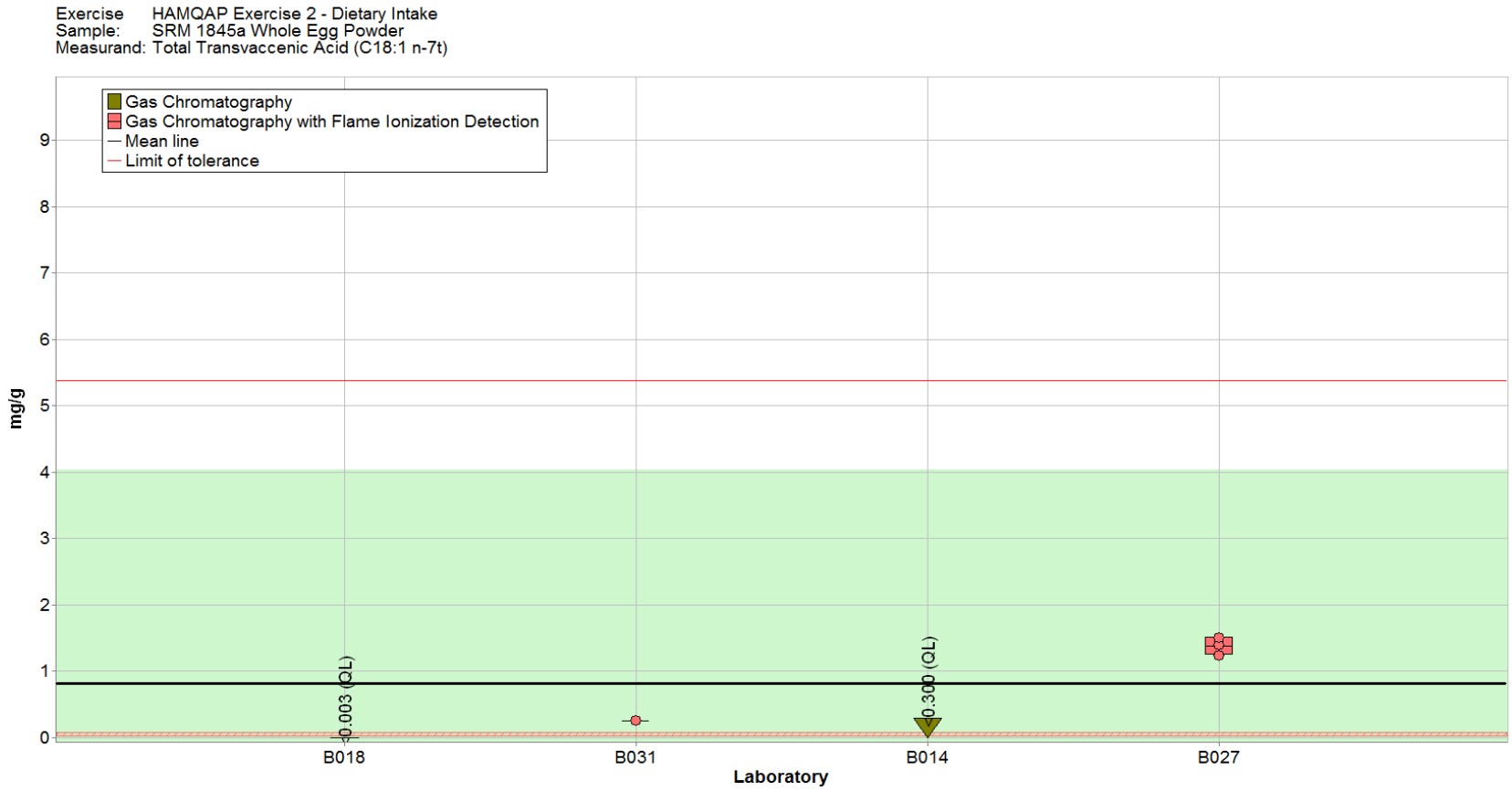

Figure 5-37. Total *trans*-vaccenic acid (C18:1 n-7t) in SRM 1845a Whole Egg Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

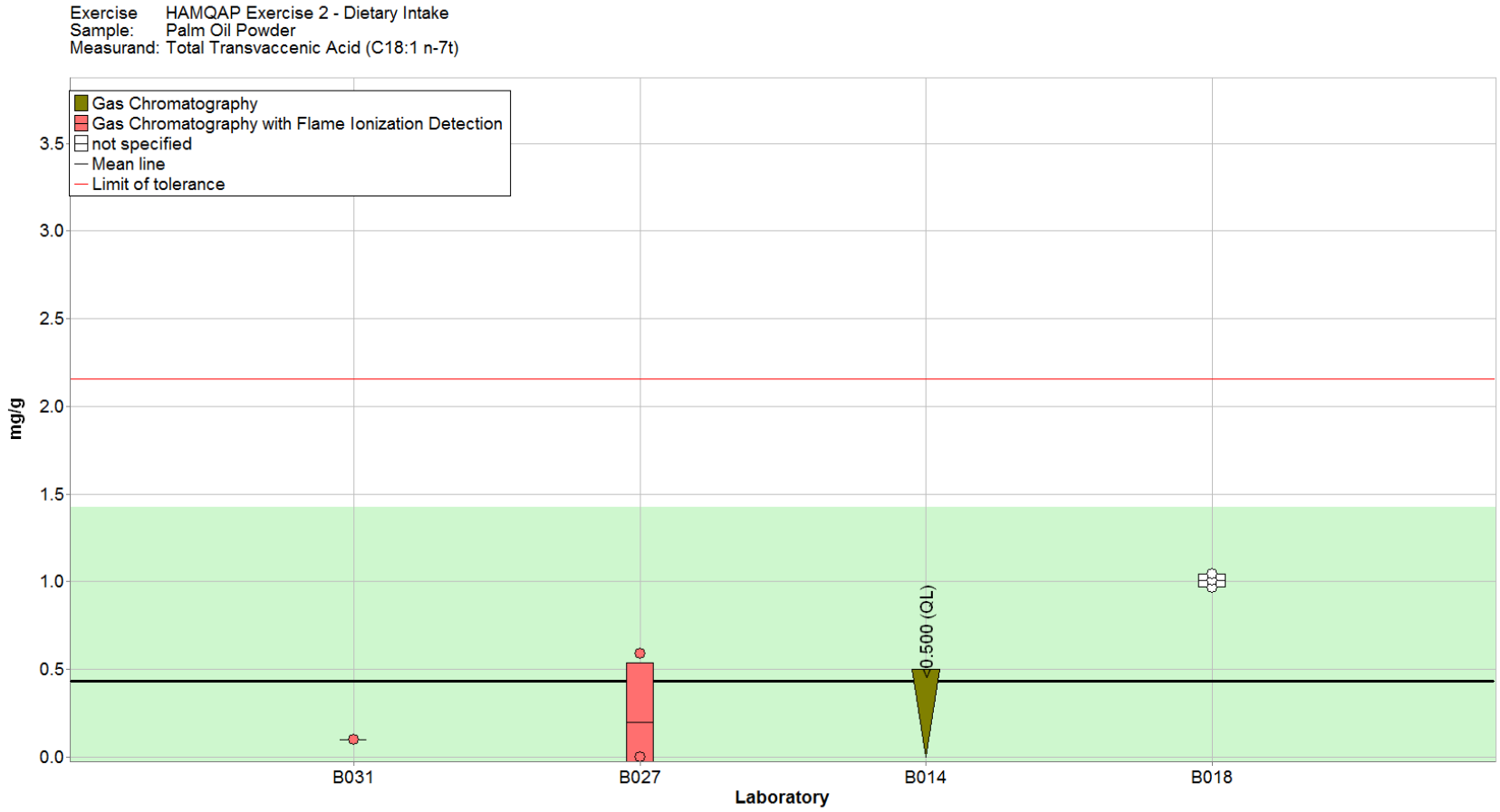

Figure 5-38. Total *trans*-vaccenic acid (C18:1 n-7t) in Palm Oil Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

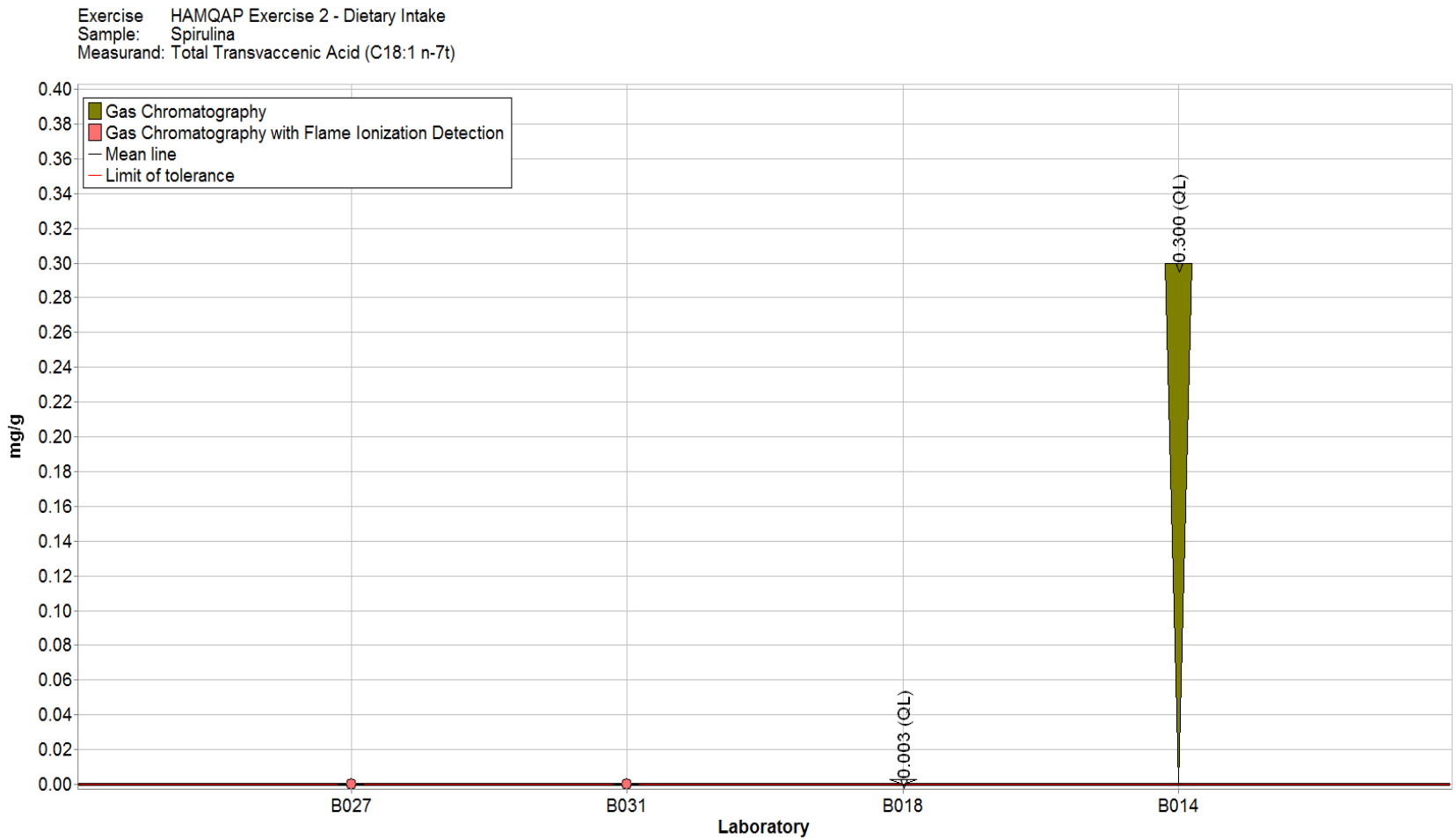

Figure 5-39. Total *trans*-vaccenic acid (C18:1 n-7t) in Spirulina (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. A NIST value has not been determined in this material.

Table 5-13. Data summary table for total linoleic acid in SRM 1845a Whole Egg Powder, Palm Oil Powder, and Spirulina. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		Total Linoleic Acid (C18:2 n-6)														
		SRM 1845a Whole Egg Powder (mg/g)					Palm Oil Powder (mg/g)					Spirulina (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				54.30	1.20										
	B001															
	B002															
	B006															
	B014	50.8	52.9	51.3	51.7	1.1	52	54.3	54.3	53.5	1.3	9.19	9.23	9.2	9.207	0.021
	B015						32.01	36.47	34.67	34.4	2.2					
	B016	34.54	34.98	34.28	34.60	0.35	50.26	50.17	49.96	50.13	0.15	8.09	8.46	8.18	8.24	0.19
	B017															
	B018	162.768	162.23	164.1	163.03	0.96	85.1	86.7	85.3	85.70	0.87	213.66	216.39	215.61	215.2	1.4
	B019	6.01	6.13	6.01	6.050	0.069	5.94	5.88	5.74	5.85	0.10	1.18	1.3	1.09	1.19	0.11
	B027	61.38	63.27	62.52	62.39	0.95	60.57	60.13	60.22	60.31	0.23	11.67	10.99	13.64	12.1	1.4
	B031	71.75	68.6	69.7	70.0	1.6	68.25	64.6	67	66.6	1.9	12.25	13.35	13.25	12.95	0.61
	B033	6	5.932	6.24	6.06	0.16	5.83	5.602	6.01	5.81	0.20	1.221	1.146	1.174	1.1804	0.03752
	B034															
	B036						9.249	9.253	9.249	9.251	0.002					
	B038	5.47	5.18	5.23	5.29	0.16	4.05	3.94	4.01	4.000	0.056	0.679	0.684	0.733	0.699	0.030
	B039															
	B041															
	B042															
	B044															
	B045	69.87	68.1	69	68.99	0.89	21.06	24.12	23.1	22.8	1.6	5.81	7.82	6.73	6.8	1.0
Community Results		Consensus Mean			46		Consensus Mean			36		Consensus Mean			6.5	
		Consensus Standard Deviation			41		Consensus Standard Deviation			34		Consensus Standard Deviation			8.7	
		Maximum			163.03		Maximum			85.70		Maximum			215.2	
		Minimum			5.29		Minimum			4.000		Minimum			0.699	
		N			9		N			11		N			9	

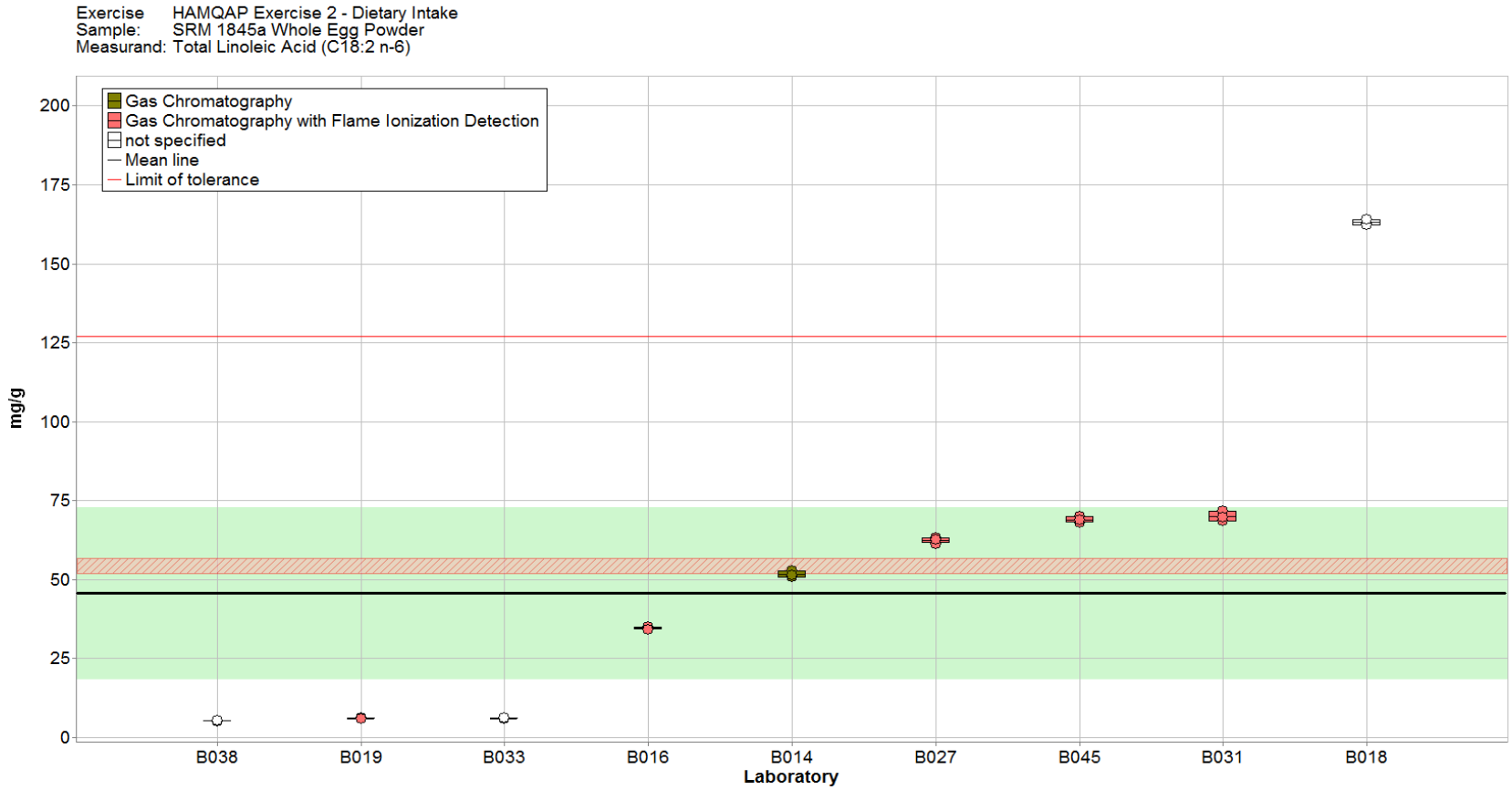

Figure 5-40. Total linoleic acid (C18:2 n-6) in SRM 1845a Whole Egg Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

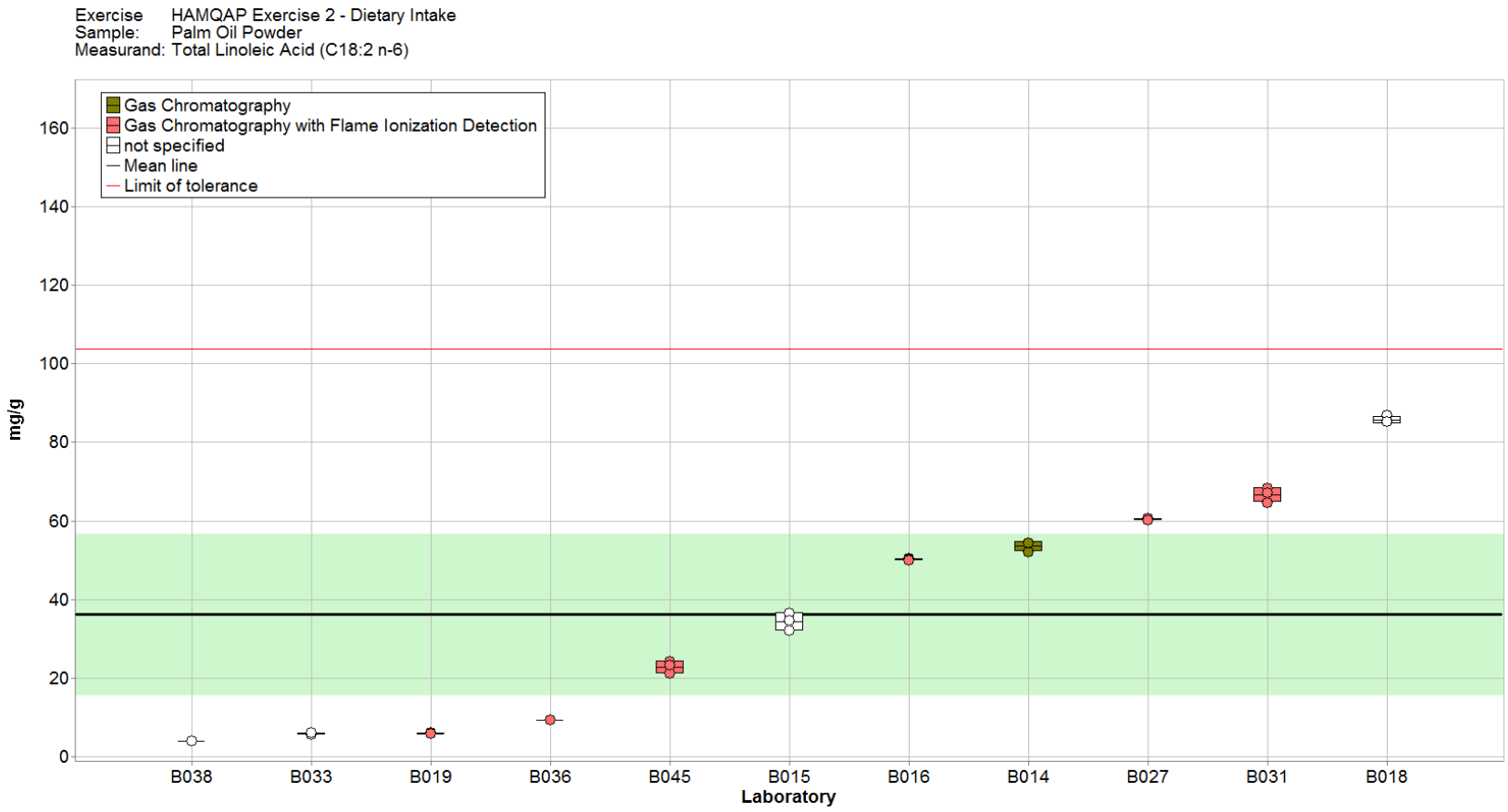

Figure 5-41. Total linoleic acid (C18:2 n-6) in Palm Oil Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

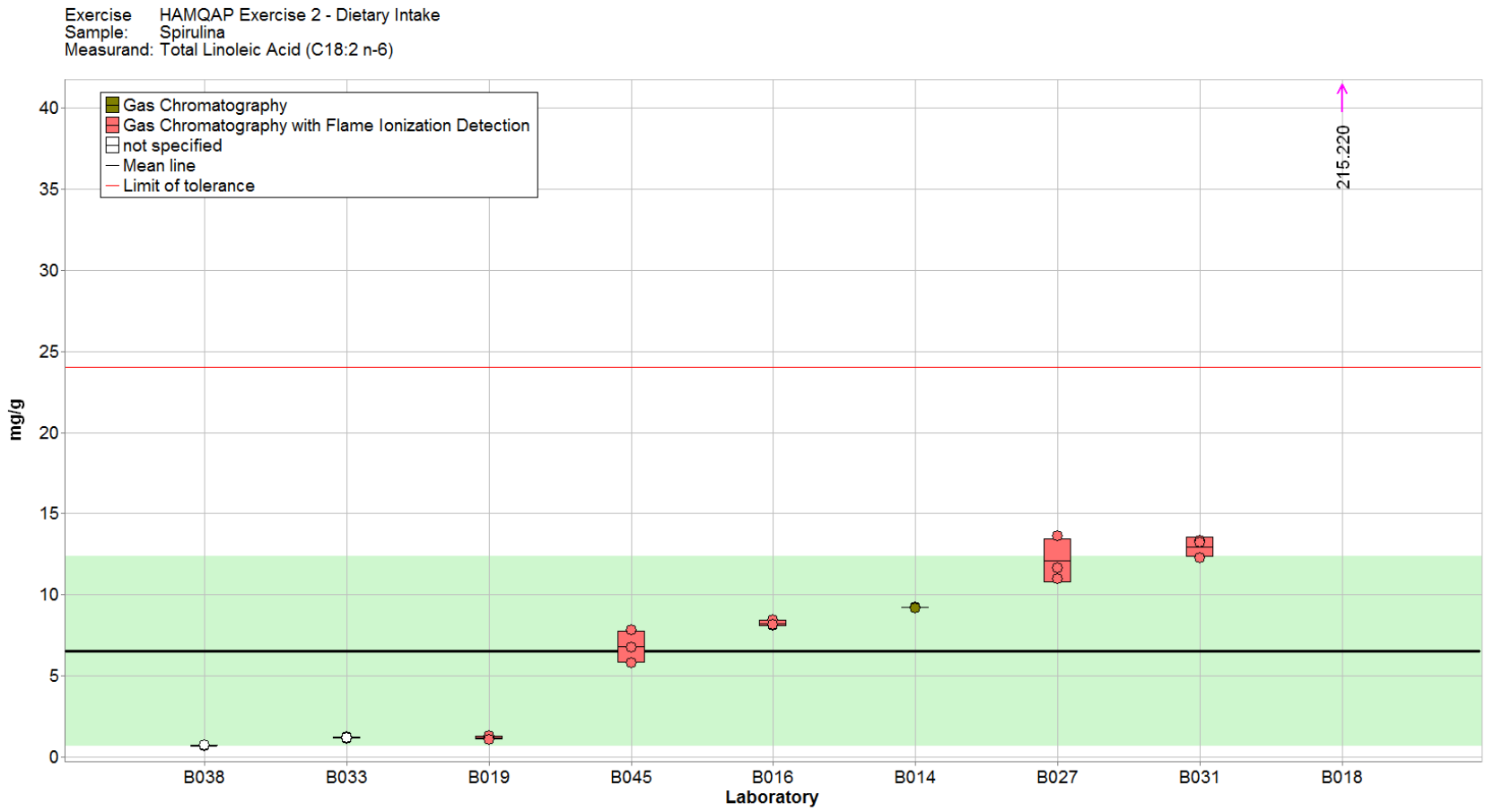

Figure 5-42. Total linoleic acid (C18:2 n-6) in Spirulina (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

Table 5-14. Data summary table for total elaidic acid in SRM 1845a Whole Egg Powder, Palm Oil Powder, and Spirulina.

		Total Elaidic Acid (C18:1 n-9t)														
		SRM 1845a Whole Egg Powder (mg/g)					Palm Oil Powder (mg/g)					Spirulina (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				0.05	0.02										
	B001															
	B002															
	B006															
	B014	0.49	0.51	0.5	0.50	0.01	< 0.500	< 0.500	< 0.500			< 0.300	< 0.300	< 0.300		
	B016															
	B017															
	B018	0.449	0.401	0.357	0.402	0.046	0.968	1.006	1.044	1.006	0.038	2.44	2.539	2.678	2.55	0.12
	B027	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
	B031	0.1	0.1	0.1	0.1	0	0	0	0	0	0	0	0	0	0	0
	B034															
	B036															
	B039															
	B041															
	B042															
	B044															
Community Results		Consensus Mean			0.25		Consensus Mean			0.3		Consensus Mean			0.9	
		Consensus Standard Deviation			0.24		Consensus Standard Deviation			1.0		Consensus Standard Deviation			2.6	
		Maximum			0.50		Maximum			1.006		Maximum			2.55	
		Minimum			0		Minimum			0		Minimum			0	
		N			4		N			3		N			3	

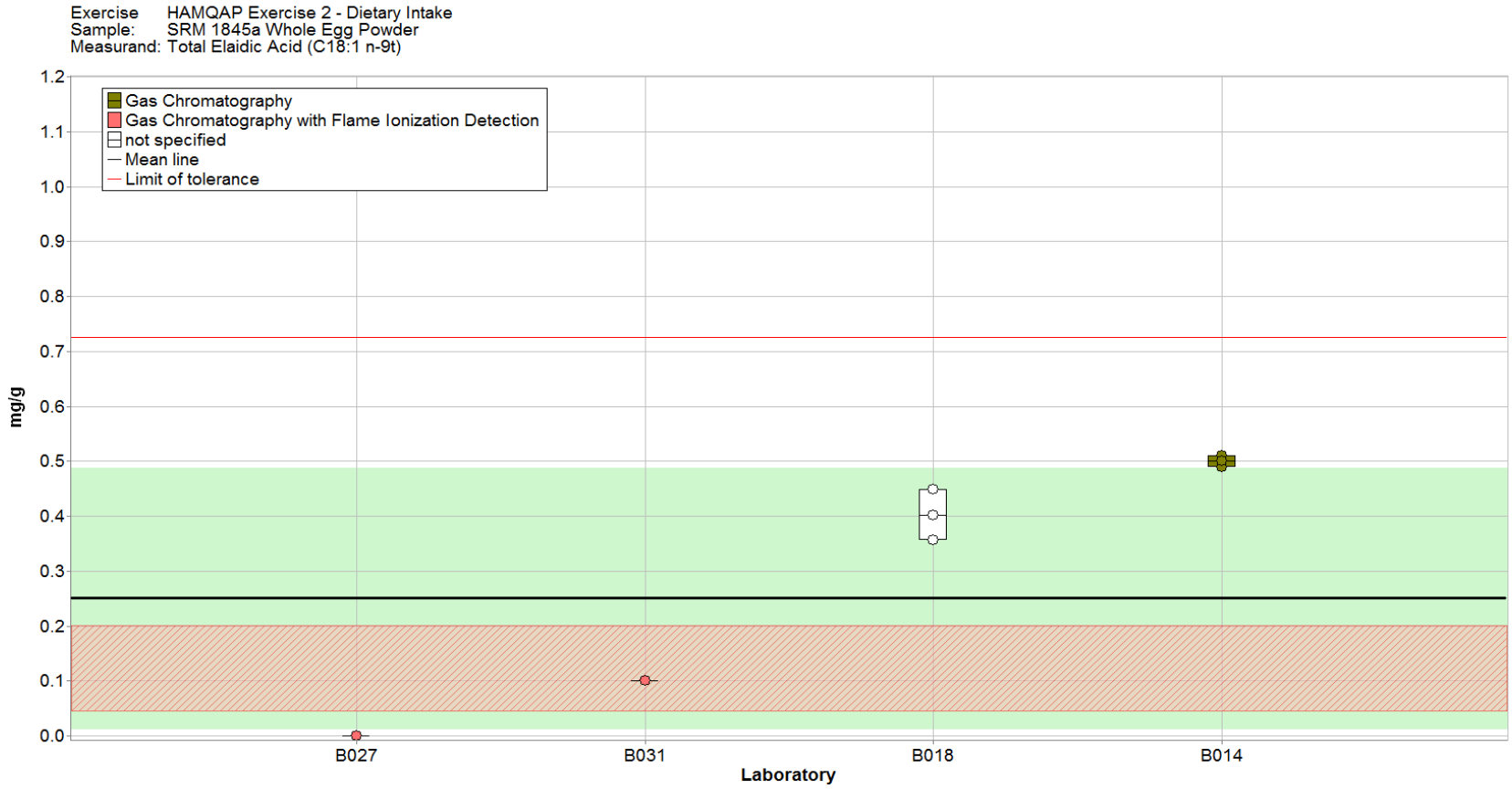

Figure 5-43. Total elaidic acid (C18:1 n-9t) in SRM 1845a Whole Egg Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

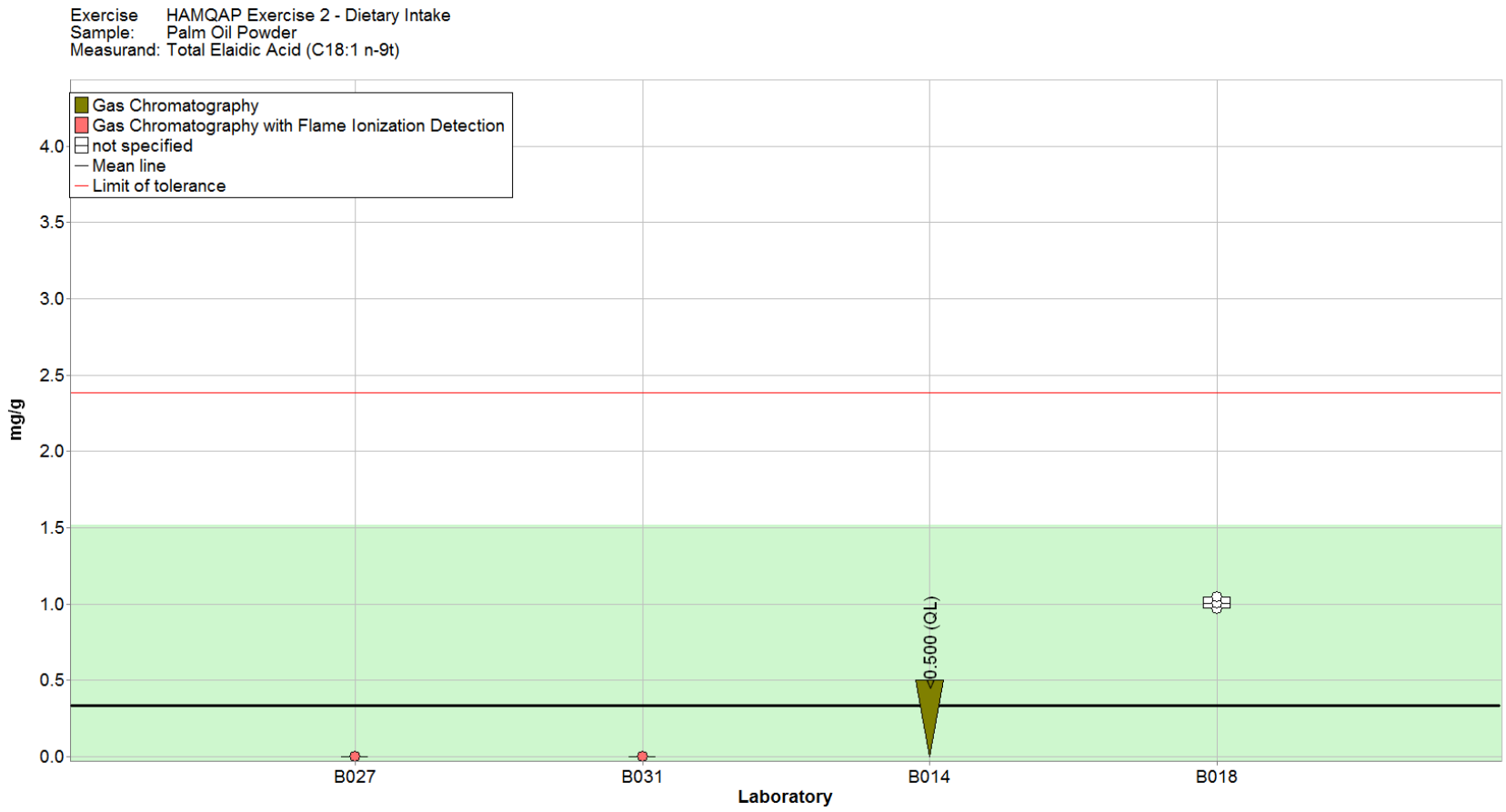

Figure 5-44. Total elaidic acid (C18:1 n-9t) in Palm Oil Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

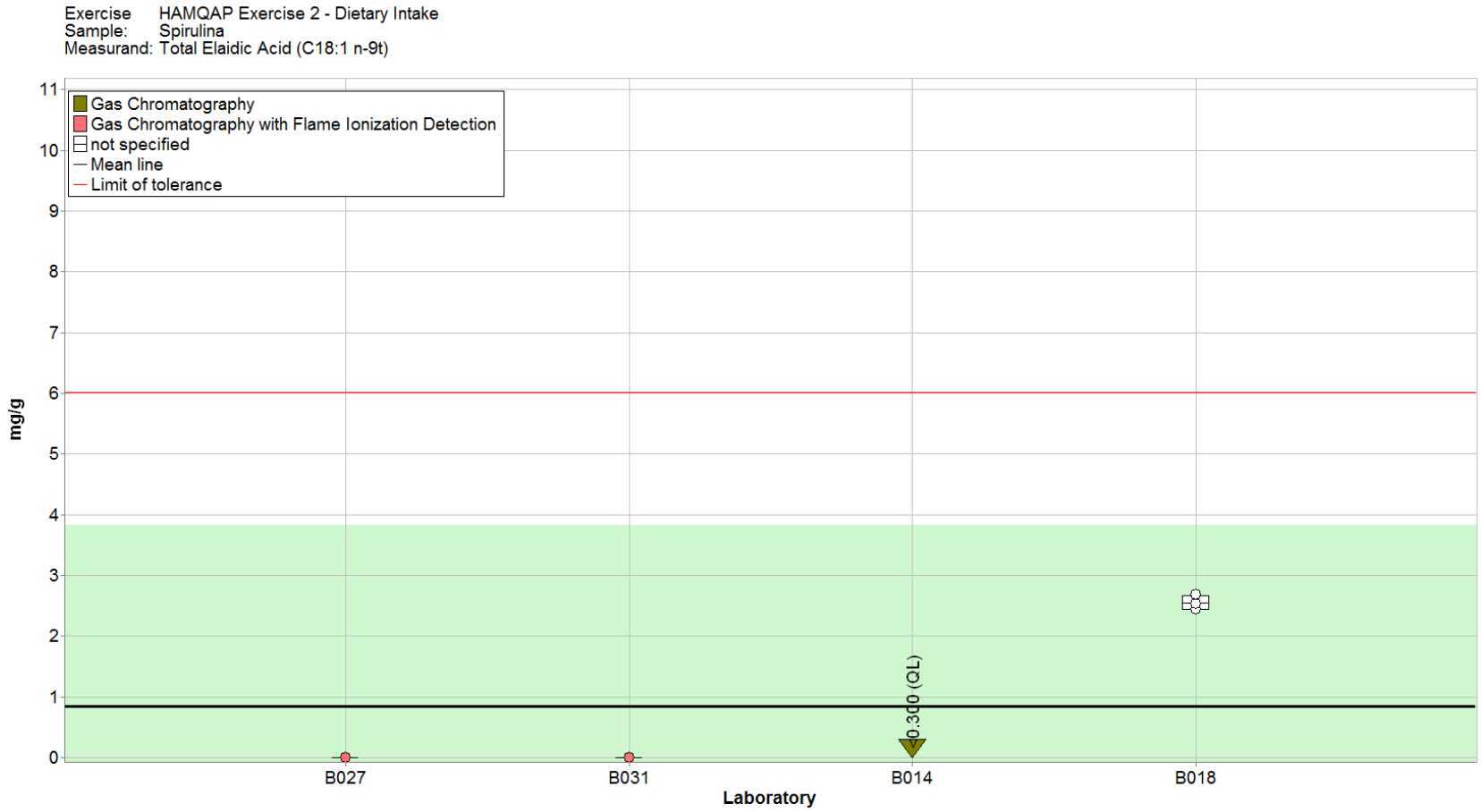

Figure 5-45. Total elaidic acid (C18:1 n-9t) in Spirulina (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

Table 5-15. Data summary table for total α-linolenic acid in SRM 1845a Whole Egg Powder, Palm Oil Powder, and Spirulina. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		Total alpha-Linolenic Acid (C18:3 n-3)														
		SRM 1845a Whole Egg Powder (mg/g)					Palm Oil Powder (mg/g)					Spirulina (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				1.64	0.05										
	B001															
	B002															
	B006															
	B014	1.06	1.07	1.08	1.07	0.01	0.76	0.77	0.76	0.7633	0.0058	< 0.300	< 0.300	< 0.300		
	B015						0.84	0.73	0.7	0.757	0.074					
	B016	0.741	0.771	0.762	0.758	0.015	0.954	0.96	0.945	0.9530	0.0075					
	B017															
	B018	1.966	1.965	2.102	2.011	0.079	1.274	1.211	1.238	1.241	0.032	< 0.003	< 0.003	< 0.003		
	B019	0.13	0.13	0.13	0.13	0	0.11	0.11	0.11	0.11	0	0	0	0	0	0
	B027	1.28	1.28	1.3	1.287	0.012	1.02	1.04	1.01	1.023	0.015	0	0	0	0	0
	B031	1.3	1.2	1.25	1.25	0.05	1.05	1	1	1.017	0.029	0	0	0	0	0
	B033	0.152	0.147	0.142	0.1471	0.0051	0.104	0.095	0.1	0.0996	0.0046	0	0	0	0	0
	B034															
	B036						0.151	0.153	0.151	0.1513	0.0011					
	B038	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
	B039															
	B041															
	B042															
	B044															
	B045	0.49	0.53	0.57	0.53	0.04	0.11	0.13	0.11	0.117	0.012	4.17	5.55	4.38	4.7	0.74357
Community Results		Consensus Mean			0.80		Consensus Mean			0.56		Consensus Mean			0.8	
		Consensus Standard Deviation			0.87		Consensus Standard Deviation			0.33		Consensus Standard Deviation			2.6	
		Maximum			2.011		Maximum			1.241		Maximum			4.7	
		Minimum			0		Minimum			0		Minimum			0	
		N			9		N			11		N			6	

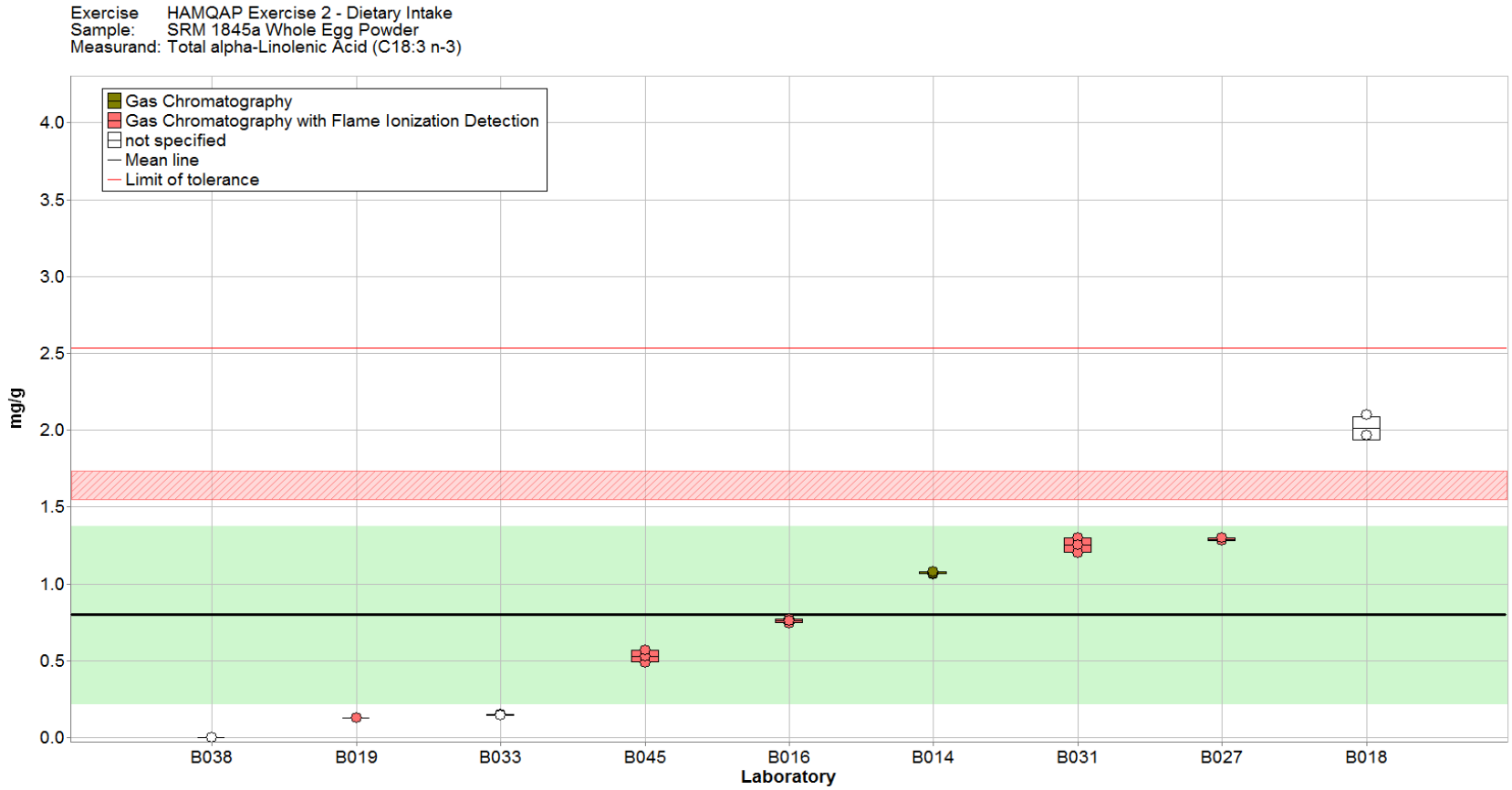

Figure 5-46. Total α-linolenic acid (C18:3 n-3) in SRM 1845a Whole Egg Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

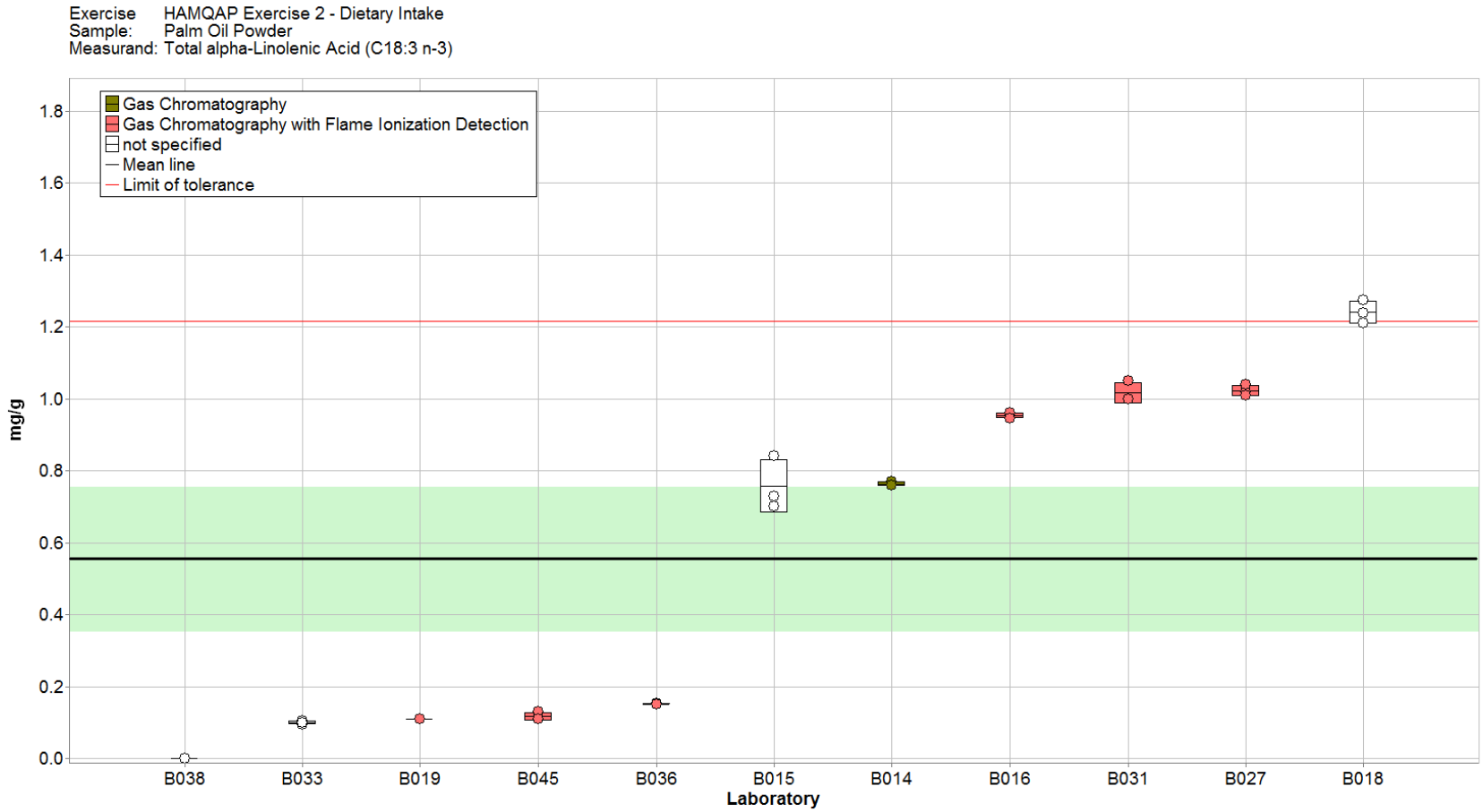

Figure 5-47. Total α-linolenic acid (C18:3 n-3) in Palm Oil Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

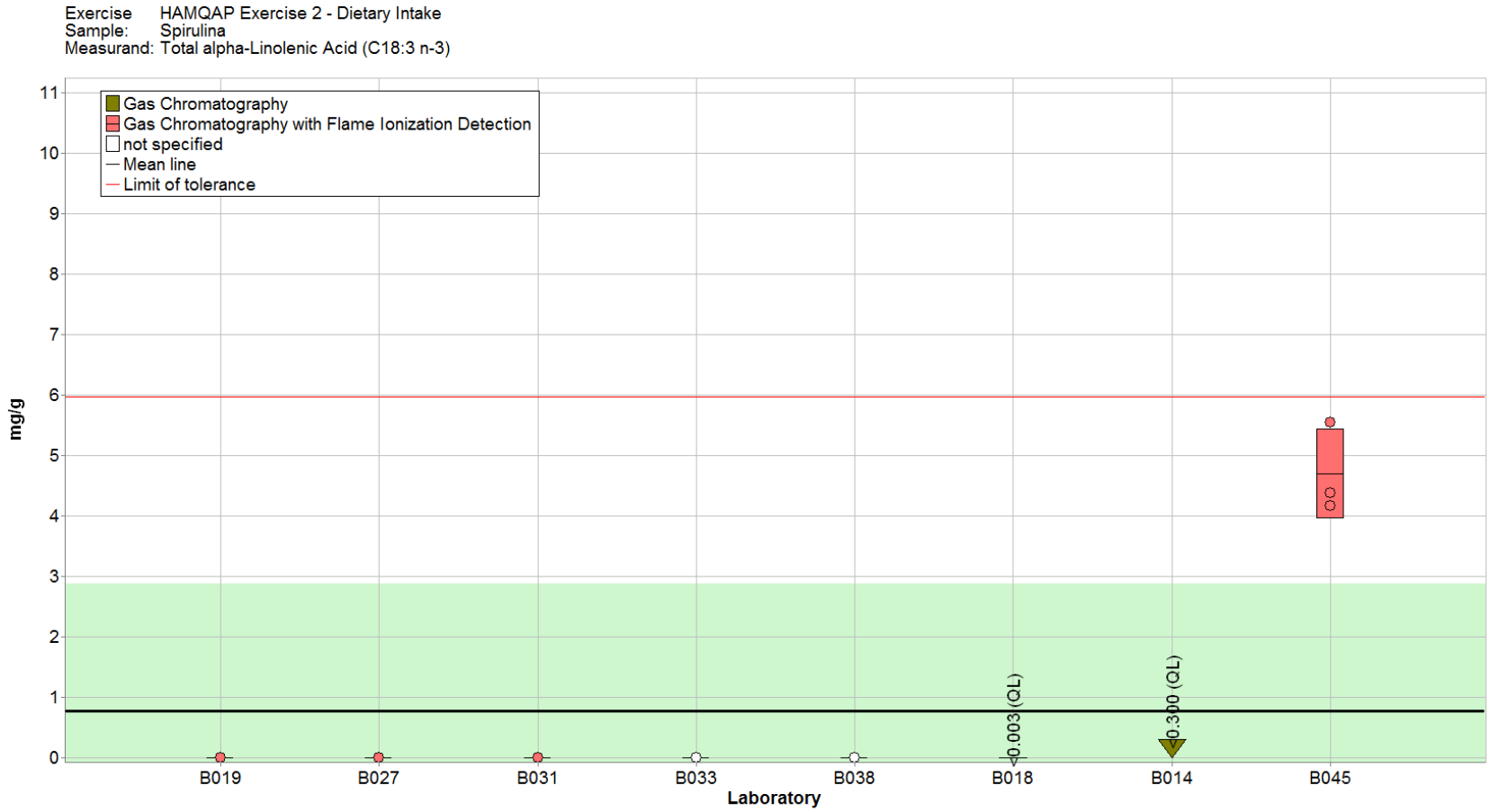

Figure 5-48. Total α-linolenic acid (C18:3 n-3) in Spirulina (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

Table 5-16. Data summary table for total γ-linolenic acid in SRM 1845a Whole Egg Powder, Palm Oil Powder, and Spirulina. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		Total gamma-Linolenic Acid (C18:3 n-6)														
		SRM 1845a Whole Egg Powder (mg/g)					Palm Oil Powder (mg/g)					Spirulina (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				0.45	0.02										
	B001															
	B002															
	B006															
	B014	0.5	0.53	0.51	0.513	0.015	2.03	2.17	2.18	2.127	0.084	6.8	6.7	6.81	6.770	0.061
	B016	0.27	0.258	0.255	0.2610	0.0079	0.052	0.045	0.051	0.0493	0.0038	5.38	5.68	5.49	5.52	0.15
	B017															
	B018	1.124	1.117	1.1	1.114	0.012	< 0.003	< 0.003	< 0.003			154.4	160	157.84	157.4	2.8
	B019	0.05	0.05	0.05	0.05	0	0	0	0	0	0	0.87	0.93	0.85	0.883	0.042
	B027	0.54	0.54	0.56	0.547	0.012	0	0	0	0	0	7.84	8.11	9.98	8.6	1.2
	B031	0.1	0.1	0.1	0.1	0	0	0	0	0	0	2.3	2.5	2.4	2.40	0.10
	B033	0.086	0.088	0.088	0.0872	0.0014	0.032	0.034	0.031	0.0322	0.0017	0.865	0.858	0.874	0.866	0.008
	B034															
	B036						0	0	0	0	0					
	B038	0	0	0	0	0	0	0	0	0	0	0.611	0.623	0.652	0.629	0.021
	B039															
	B041															
	B042															
	B044															
	B045	0.37	0.32	0.29	0.327	0.040	0.34	0.36	0.33	0.343	0.015	< 0.100	< 0.100	< 0.100		
Community Results		Consensus Mean			0.30		Consensus Mean			0.012		Consensus Mean			3.7	
		Consensus Standard Deviation			0.36		Consensus Standard Deviation			0.050		Consensus Standard Deviation			3.8	
		Maximum			1.114		Maximum			2.127		Maximum			157.4	
		Minimum			0		Minimum			0		Minimum			0.629	
		N			9		N			9		N			8	

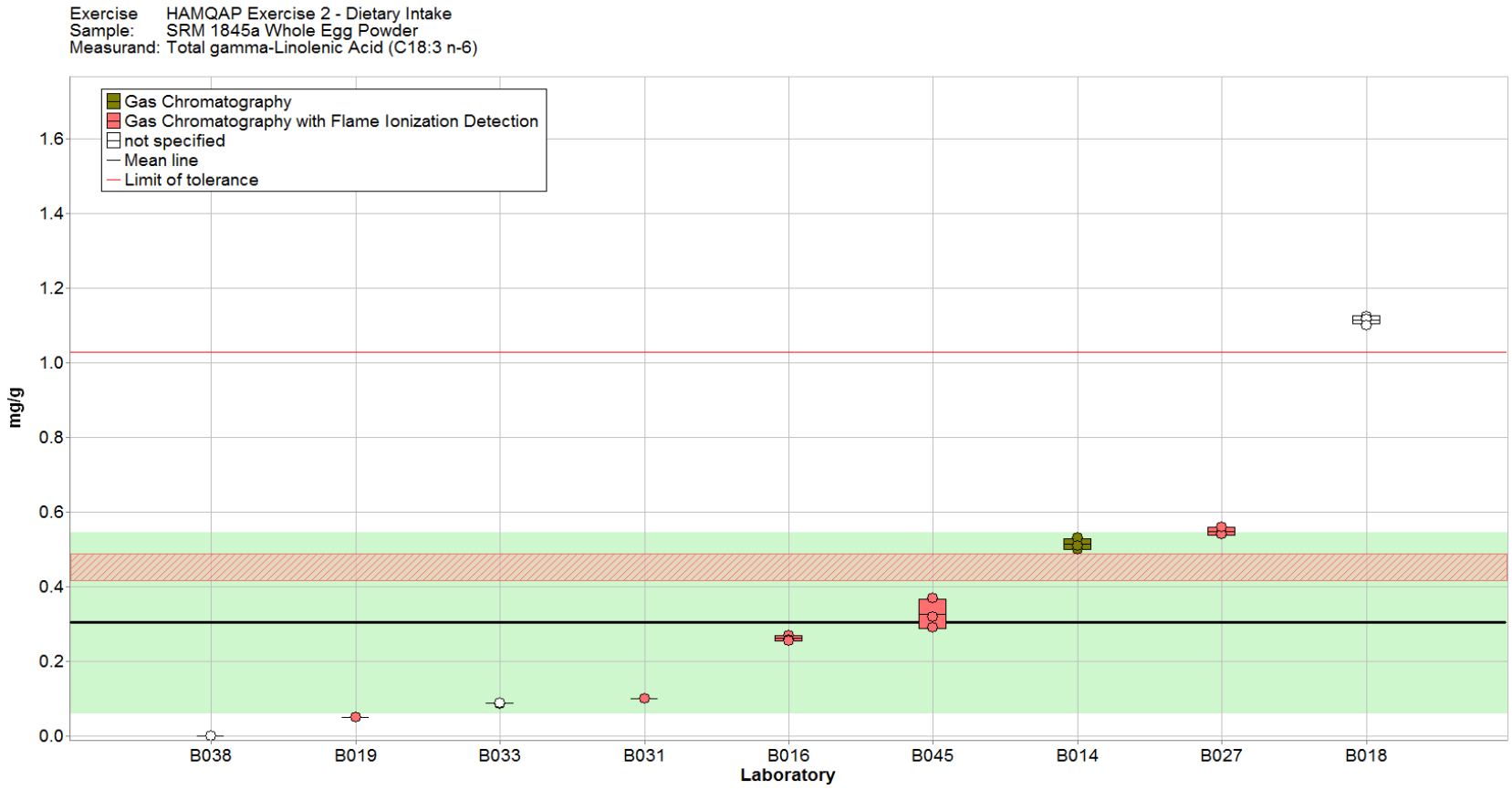

Figure 5-49. Total γ-linolenic acid (C18:3 n-6) in SRM 1845a Whole Egg Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

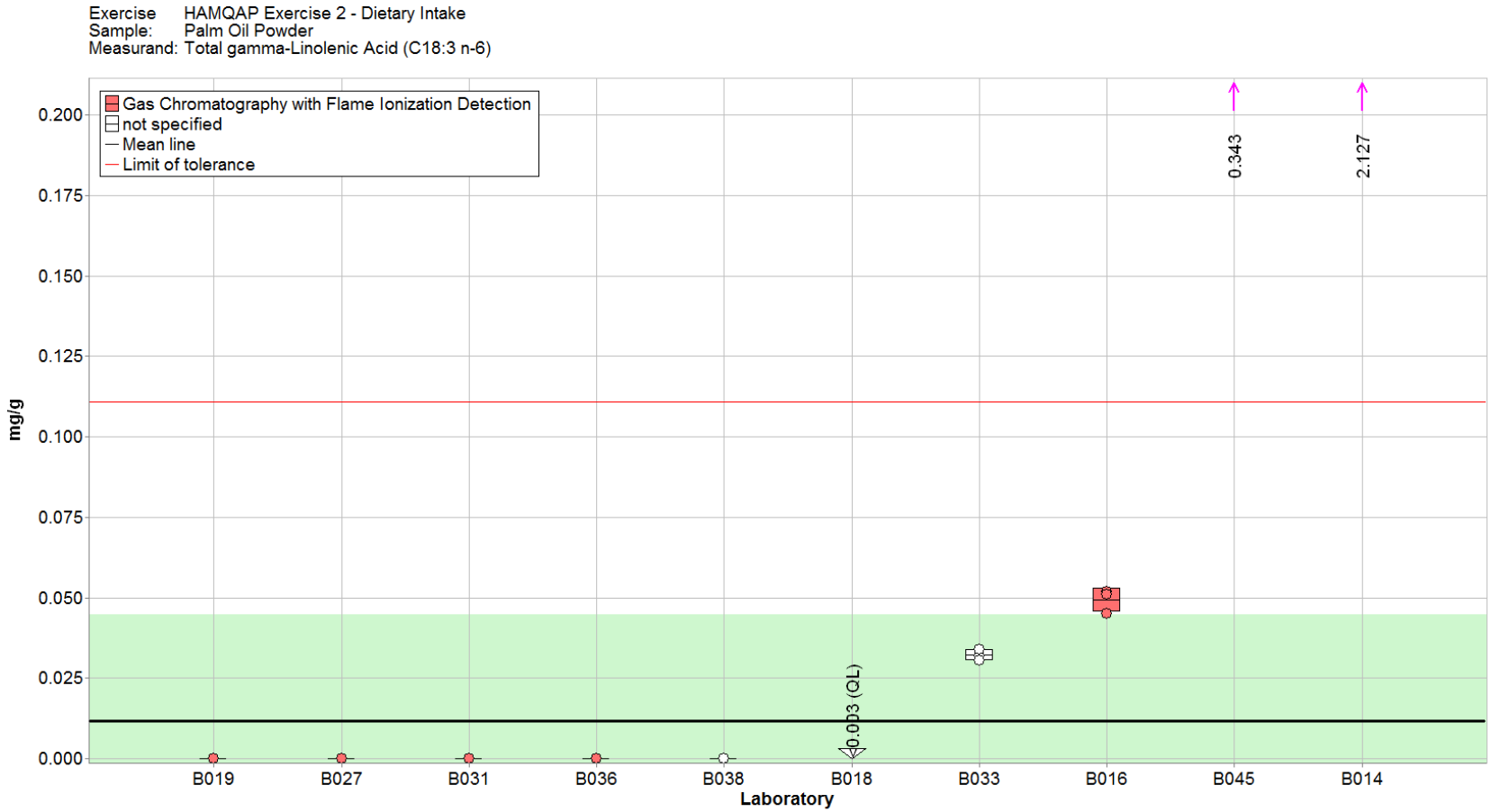

Figure 5-50. Total γ-linolenic acid (C18:3 n-6) in Palm Oil Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

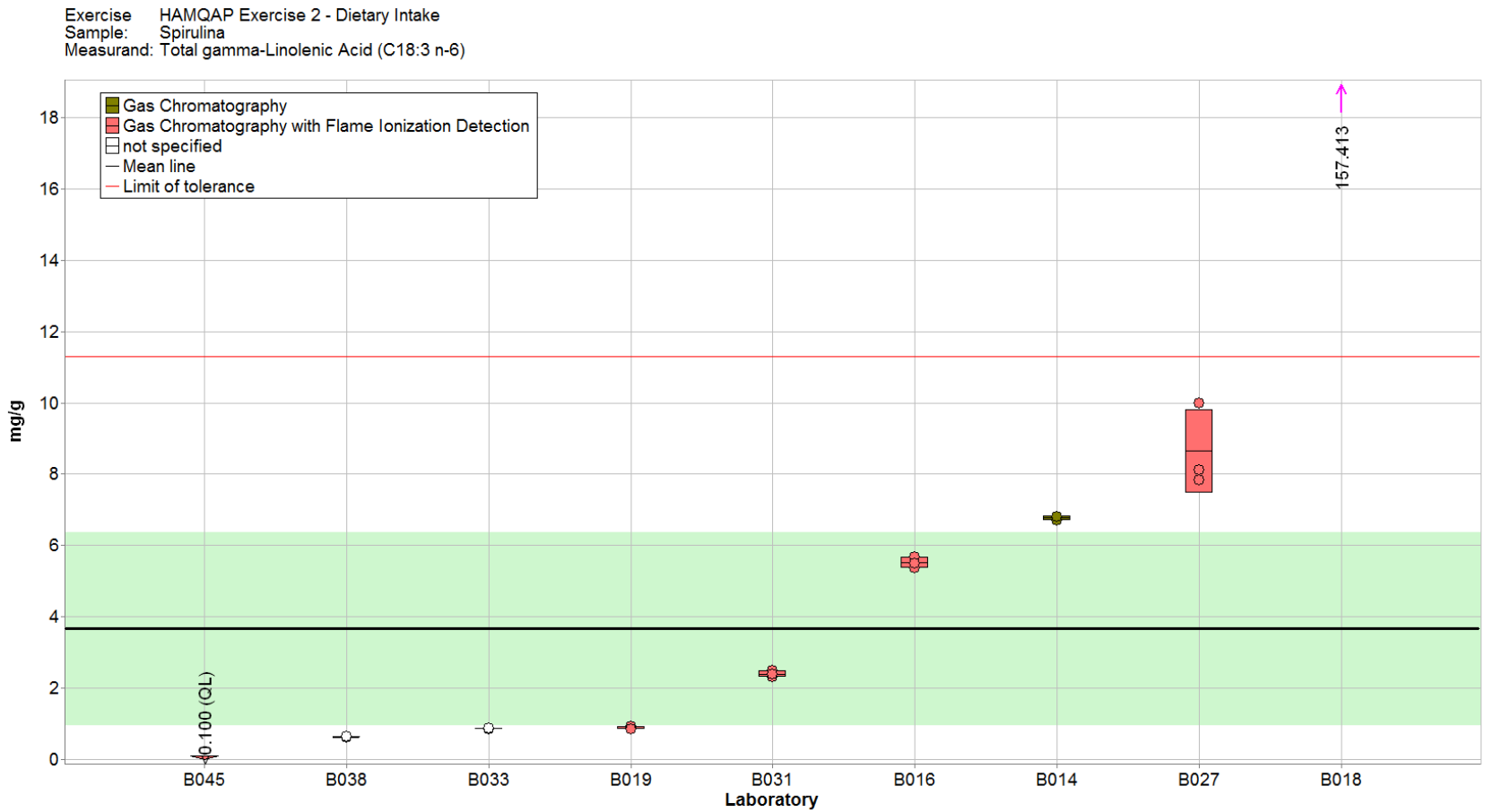

Figure 5-51. Total γ-linolenic acid (C18:3 n-6) in Spirulina (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

Table 5-17. Data summary table for total arachidic acid in SRM 1845a Whole Egg Powder, Palm Oil Powder, and Spirulina.

		Total Arachidic Acid (C20:0)													
		SRM 1845a Whole Egg Powder (mg/g)					Palm Oil Powder (mg/g)					Spirulina (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				0.05	0.02										
	B001															
	B002															
	B006															
	B014	< 0.300	< 0.300	< 0.300			< 0.500	< 0.500	< 0.500			< 0.300	< 0.300	< 0.300		
	B016	0.065	0.08	0.066	0.0703	0.0084										
	B017															
	B018	0.242	0.235	0.249	0.242	0.007	2.868	2.849	2.856	2.8577	0.0096	< 0.003	< 0.003	< 0.003		
	B027	0	0	0	0	0	2.37	2.37	2.33	2.357	0.023	0	0	0	0	0
	B031	0.1	0.1	0.1	0.1	0	2.6	2.4	2.45	2.48	0.10	0	0	0	0	0
	B034															
	B036						0.375	0.381	0.375	0.3767	0.0033					
	B039															
	B041															
	B042															
	B044															
Community Results		Consensus Mean			0.10		Consensus Mean			2.12		Consensus Mean			0	
		Consensus Standard Deviation			0.15		Consensus Standard Deviation			0.90		Consensus Standard Deviation			0	
		Maximum			0.242		Maximum			2.8577		Maximum			0	
		Minimum			0		Minimum			0.3767		Minimum			0	
		N			4		N			4		N			2	

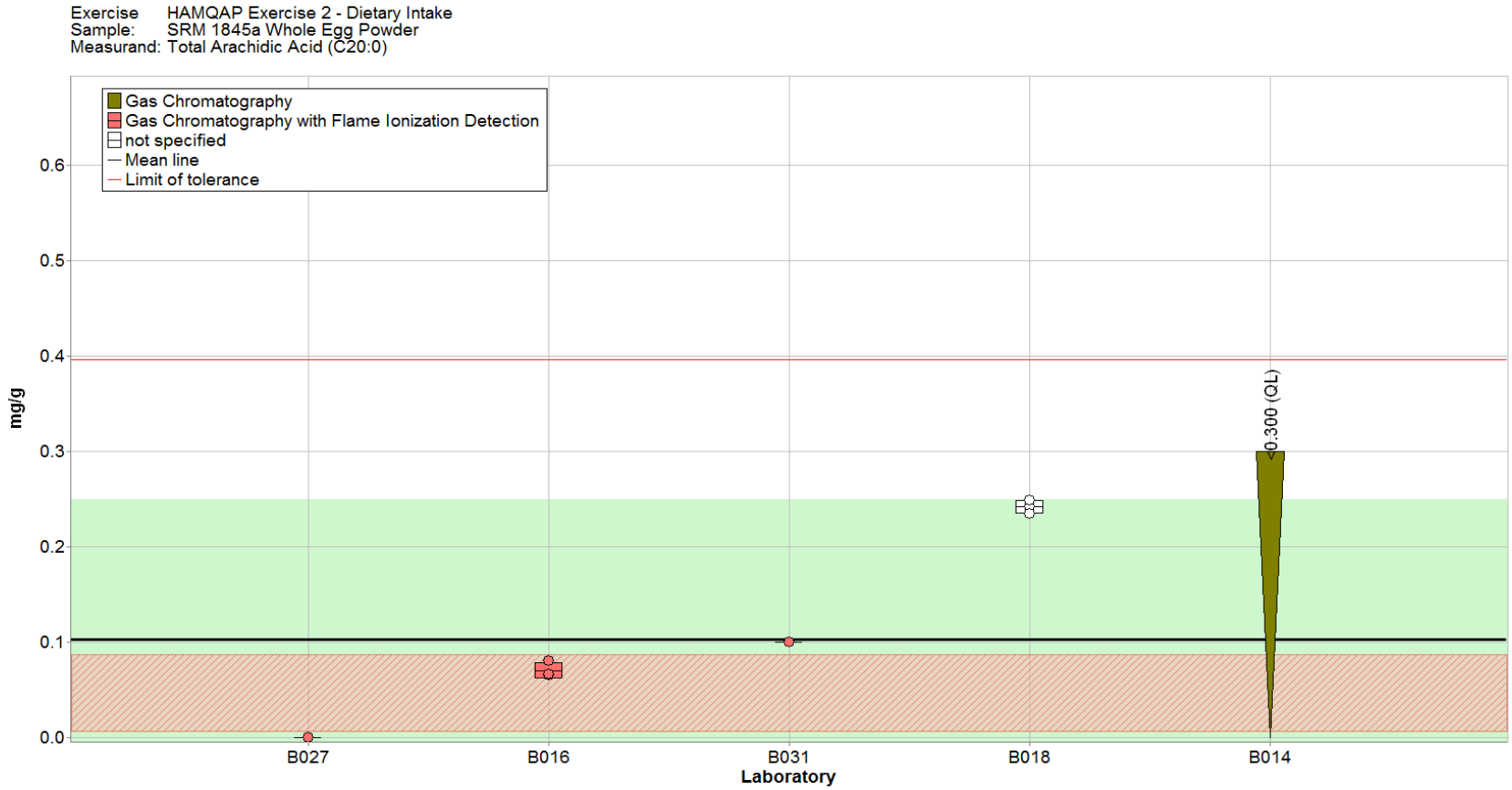

Figure 5-52. Total arachidic acid (C20:0) in SRM 1845a Whole Egg Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

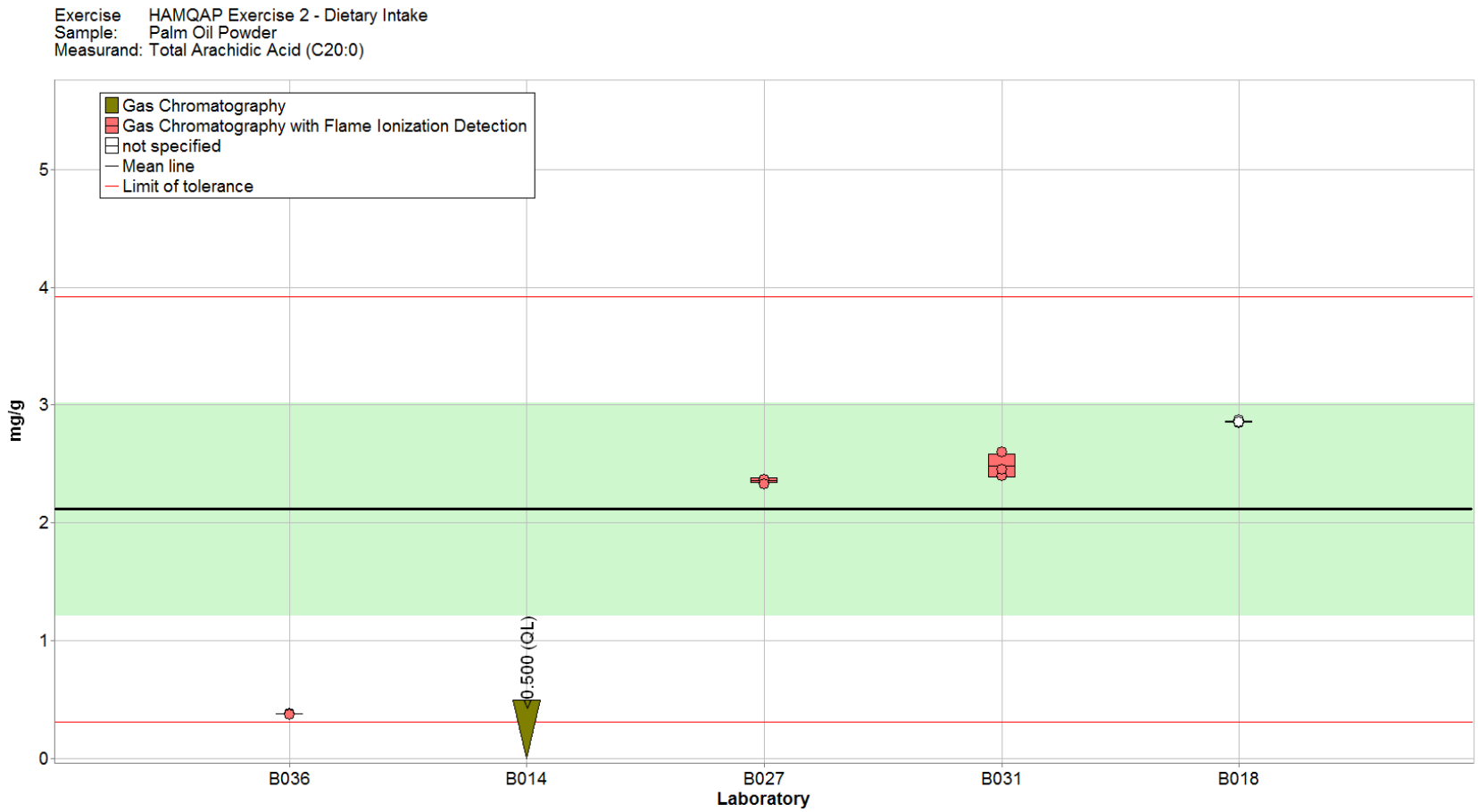

Figure 5-53. Total arachidic acid (C20:0) in Palm Oil Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

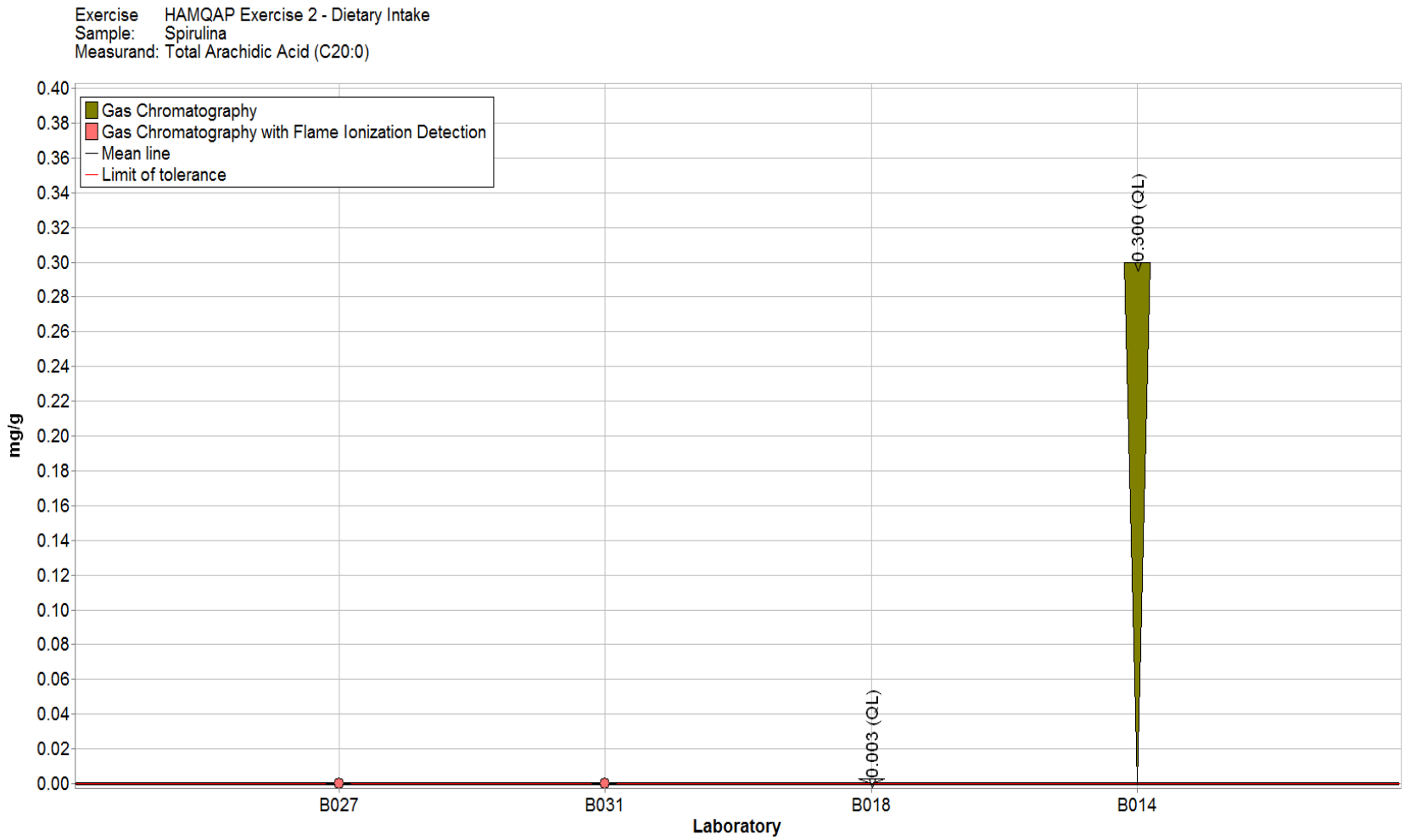

Figure 5-54. Total arachidic acid (C20:0) in Spirulina (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. A NIST value has not been determined in this material.

Table 5-18. Data summary table for total dihomo-γ-linolenic acid in SRM 1845a Whole Egg Powder, Palm Oil Powder, and Spirulina.

		Total Dihomo-gamma-linolenic acid (C20:3 n-6)														
		SRM 1845a Whole Egg Powder (mg/g)					Palm Oil Powder (mg/g)					Spirulina (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				1.08	0.4										
	B001															
	B002															
	B006															
	B014						< 0.500	< 0.500	< 0.500			< 0.300	< 0.300	< 0.300		
	B016															
	B017															
	B018						< 0.003	< 0.003	< 0.003			< 0.003	< 0.003	< 0.003		
	B027	0.92	0.92	0.92	0.92	0	0	0	0	0	0	0.23	0.22	0.43	0.29333	0.11846
	B031						0	0	0	0	0	0.1	0.1	0.1	0.1	0
	B034															
	B036															
	B039															
	B041															
	B042															
	B044															
	B045	0.84	0.88	0.8	0.84	0.04	< 0.100	< 0.100	< 0.100			< 0.100	< 0.100	< 0.100		
Community Results		Consensus Mean			0.88		Consensus Mean			0		Consensus Mean			0.197	
		Consensus Standard Deviation			0.11		Consensus Standard Deviation			0		Consensus Standard Deviation			0.272	
		Maximum			0.92		Maximum			0		Maximum			0.29333	
		Minimum			0.84		Minimum			0		Minimum			0.1	
		N			2		N			2		N			2	

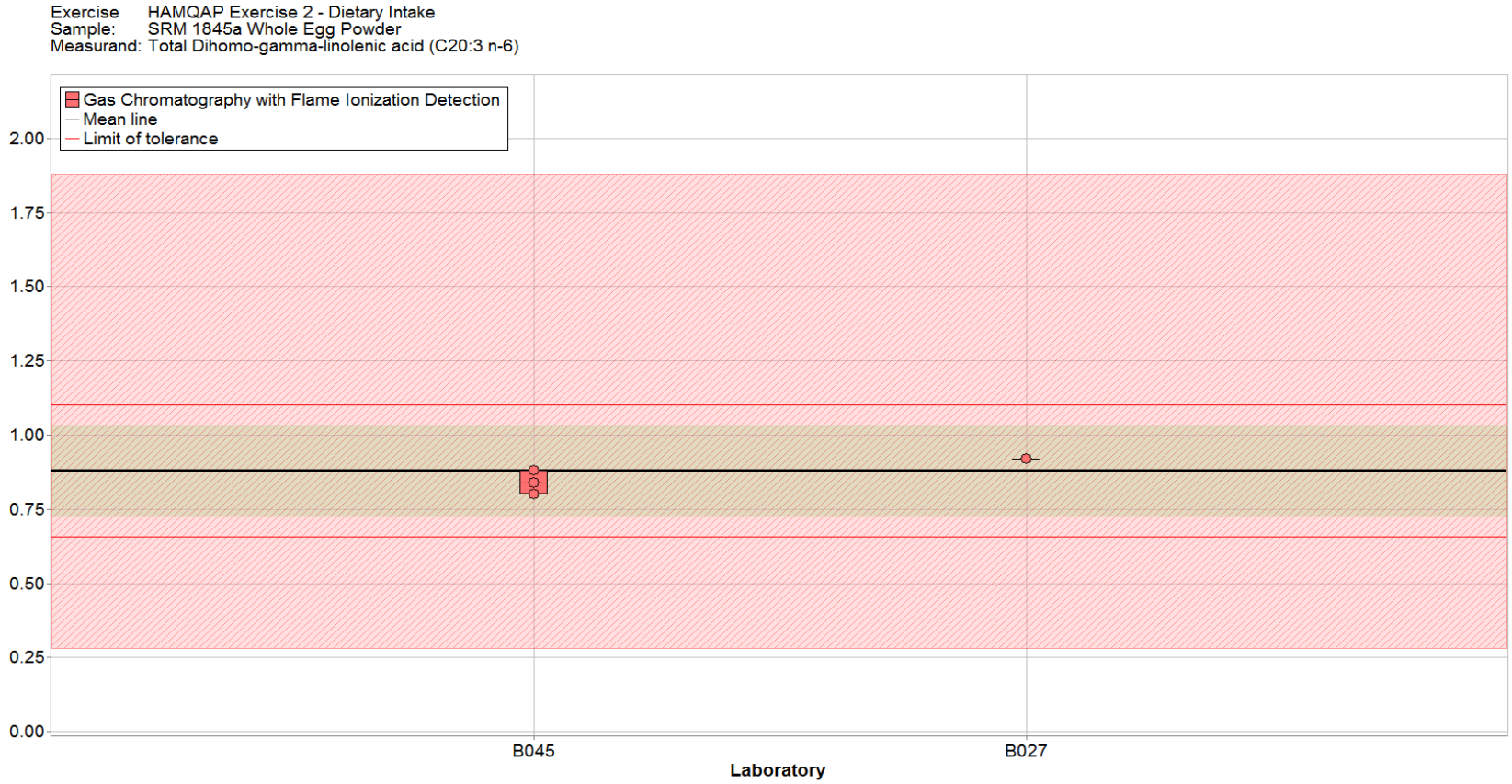

Figure 5-55. Total dihomo-γ-linolenic acid (C20:3 n-6) in SRM 1845a Whole Egg Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

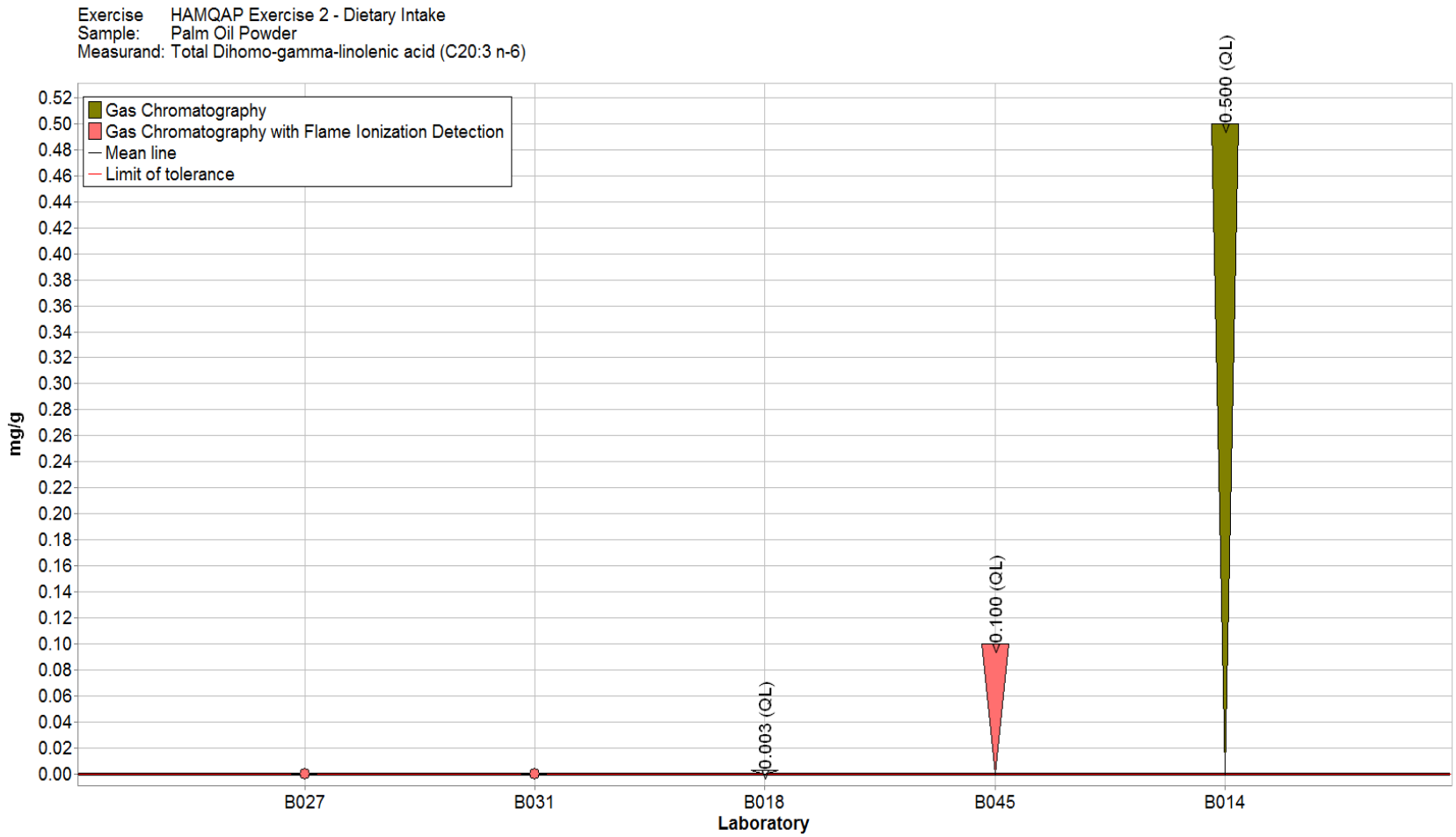

Figure 5-56. Total dihomo-γ-linolenic acid (C20:3 n-6) in Palm Oil Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. A NIST value has not been determined in this material.

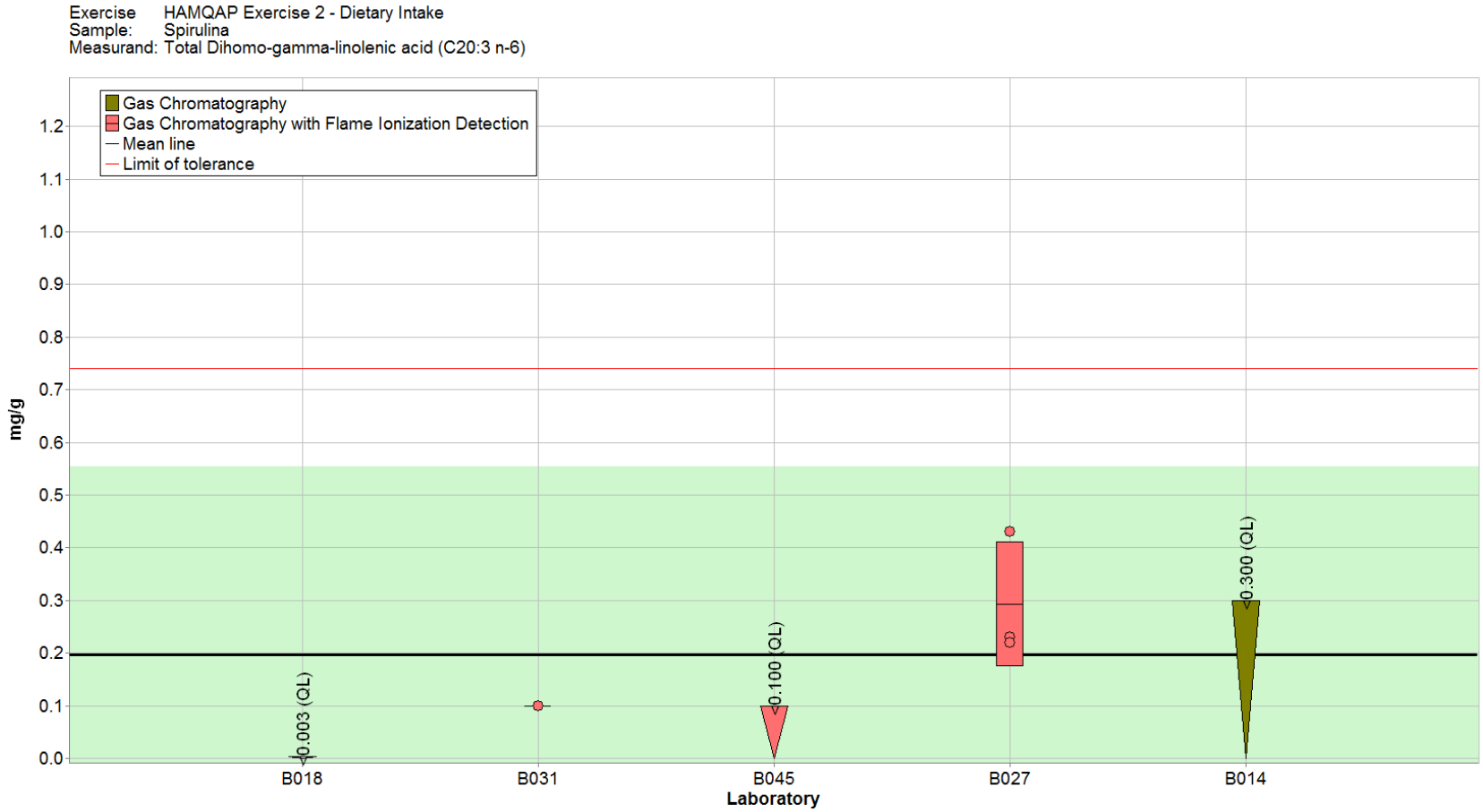

Figure 5-57. Total dihomo-γ-linolenic acid (C20:3 n-6) in Spirulina (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

Table 5-19. Data summary table for total arachidonic acid in SRM 1845a Whole Egg Powder, Palm Oil Powder, and Spirulina. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		Total Arachidonic Acid (C20:4 n-6)														
		SRM 1845a Whole Egg Powder (mg/g)					Palm Oil Powder (mg/g)					Spirulina (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				6.43	0.17										
	B001															
	B002															
	B006															
	B014	6.28	6.5	6.27	6.35	0.13	< 0.500	< 0.500	< 0.500			0.34	0.35	0.34	0.3433	0.0058
	B015						0.09	0.07	0.09	0.083	0.012					
	B016	4.4	3.92	3.85	4.06	0.30										
	B017															
	B018	32.55	32.58	28.4	31.2	2.4	< 0.003	< 0.003	< 0.003			< 0.003	< 0.003	< 0.003		
	B027	7.66	7.73	7.82	7.737	0.080	0	0	0	0	0	0	0	0.2	0.07	0.12
	B031	8.2	7.65	7.9	7.92	0.28	0	0	0	0	0	0	0	0	0	0
	B034															
	B036						0	0	0	0	0					
	B039															
	B041															
	B042															
	B044															
	B045	8.43	7.73	7.82	7.99	0.38	< 0.100	< 0.100	< 0.100			< 0.100	< 0.100	< 0.100		
Community Results		Consensus Mean			6.8		Consensus Mean			0.021		Consensus Mean			0.14	
		Consensus Standard Deviation			2.9		Consensus Standard Deviation			0.058		Consensus Standard Deviation			0.28	
		Maximum			31.2		Maximum			0.08333		Maximum			0.3433	
		Minimum			4.06		Minimum			0		Minimum			0	
		N			6		N			4		N			3	

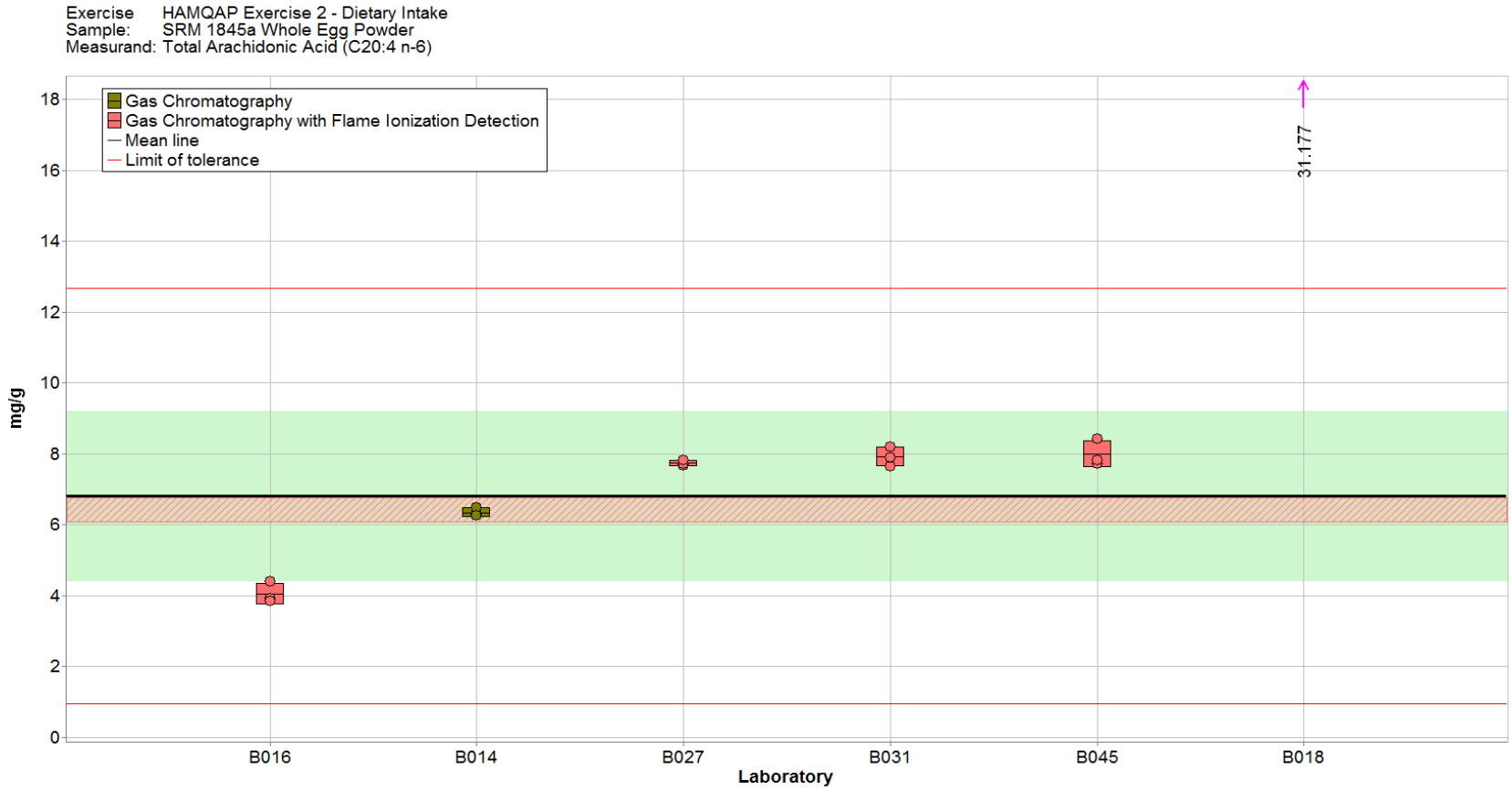

Figure 5-58. Total arachidonic acid (C20:4 n-6) in SRM 1845a Whole Egg Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

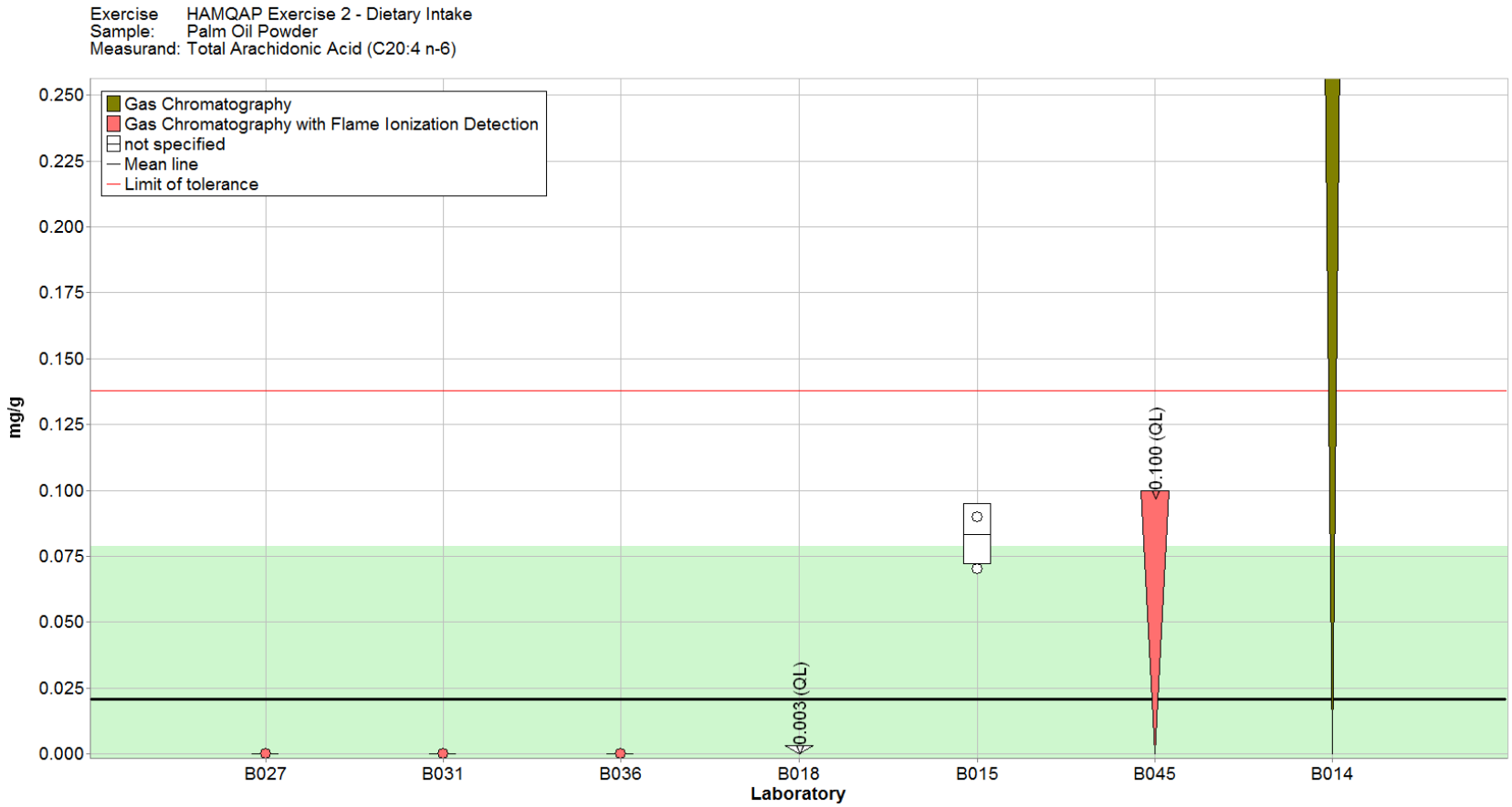

Figure 5-59. Total arachidonic acid (C20:4 n-6) in Palm Oil Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

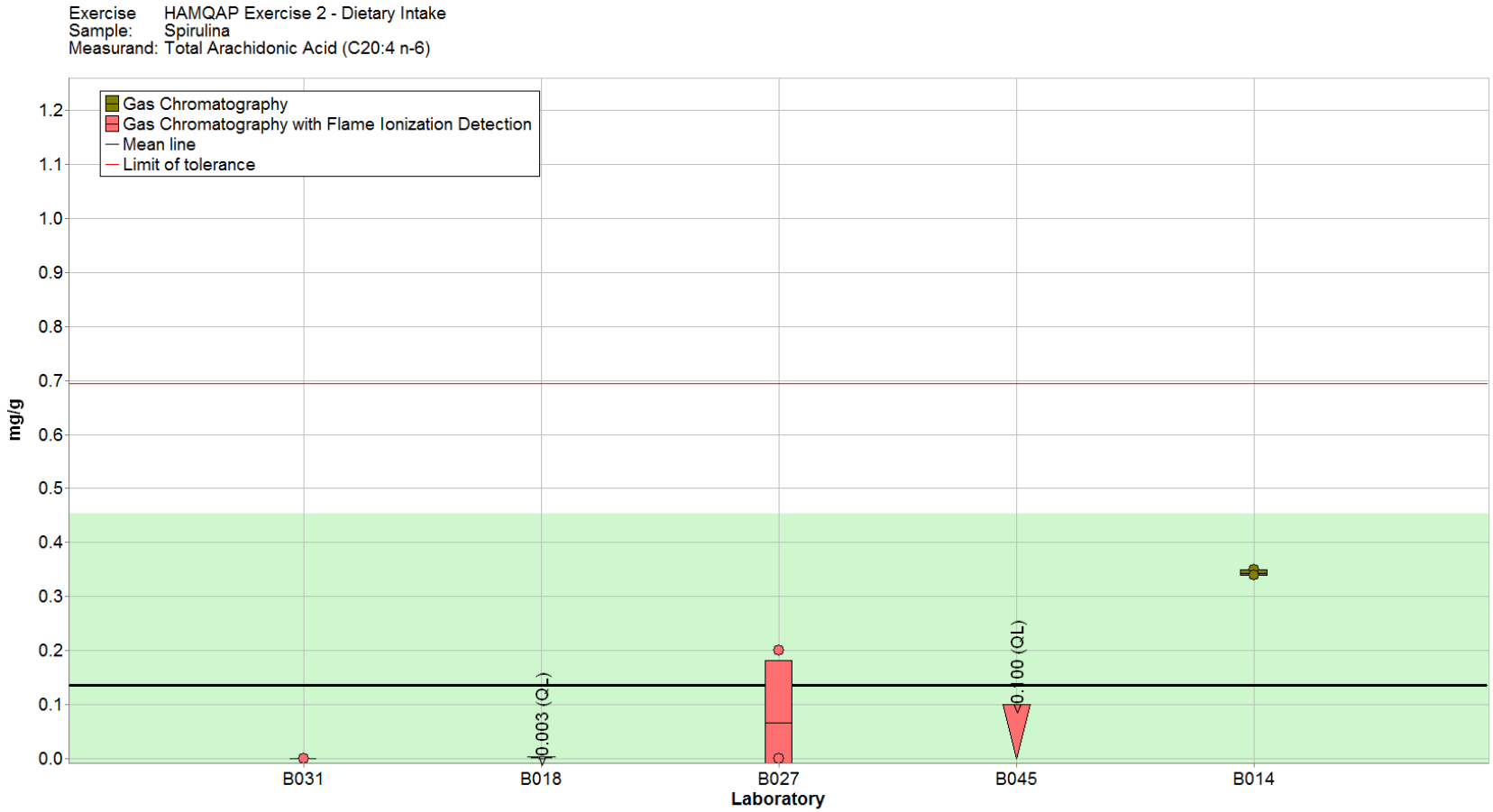

Figure 5-60. Total arachidonic acid (C20:4 n-6) in Spirulina (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

Table 5-20. Data summary table for total EPA in SRM 1845a Whole Egg Powder, Palm Oil Powder, and Spirulina. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		Total EPA (C20:5 n-3)														
		SRM 1845a Whole Egg Powder (mg/g)					Palm Oil Powder (mg/g)					Spirulina (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target															
	B001															
	B002															
	B006															
	B014	< 0.300	< 0.300	< 0.300			< 0.500	< 0.500	< 0.500			< 0.300	< 0.300	< 0.300		
	B015						0.08	0.08	0.08	0.08	0					
	B016															
	B017															
	B018	< 0.003	< 0.003	< 0.003			< 0.003	< 0.003	< 0.003			< 0.003	< 0.003	< 0.003		
	B019															
	B027	0	0	0	0	0	0	0	0	0	0	0	0	0.26	0.09	0.15
	B031	0	0	0	0	0	0	0	0	0	0	0.1	0.1	0.1	0.1	0
	B033	0	0	0	0	0	0.33	0.304	0.314	0.316	0.013	0.025	0.023	0.029	0.0259	0.0034
	B034															
	B036															
	B038	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
	B039															
	B041															
	B042	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
	B044															
	B045	< 0.100	< 0.100	< 0.100			< 0.100	< 0.100	< 0.100			< 0.100	< 0.100	< 0.100		
Community Results		Consensus Mean			0		Consensus Mean			0.043		Consensus Mean			0.041	
		Consensus Standard Deviation			0		Consensus Standard Deviation			0.090		Consensus Standard Deviation			0.035	
		Maximum			0		Maximum			0.31583		Maximum			0.1	
		Minimum			0		Minimum			0		Minimum			0	
		N			5		N			6		N			5	

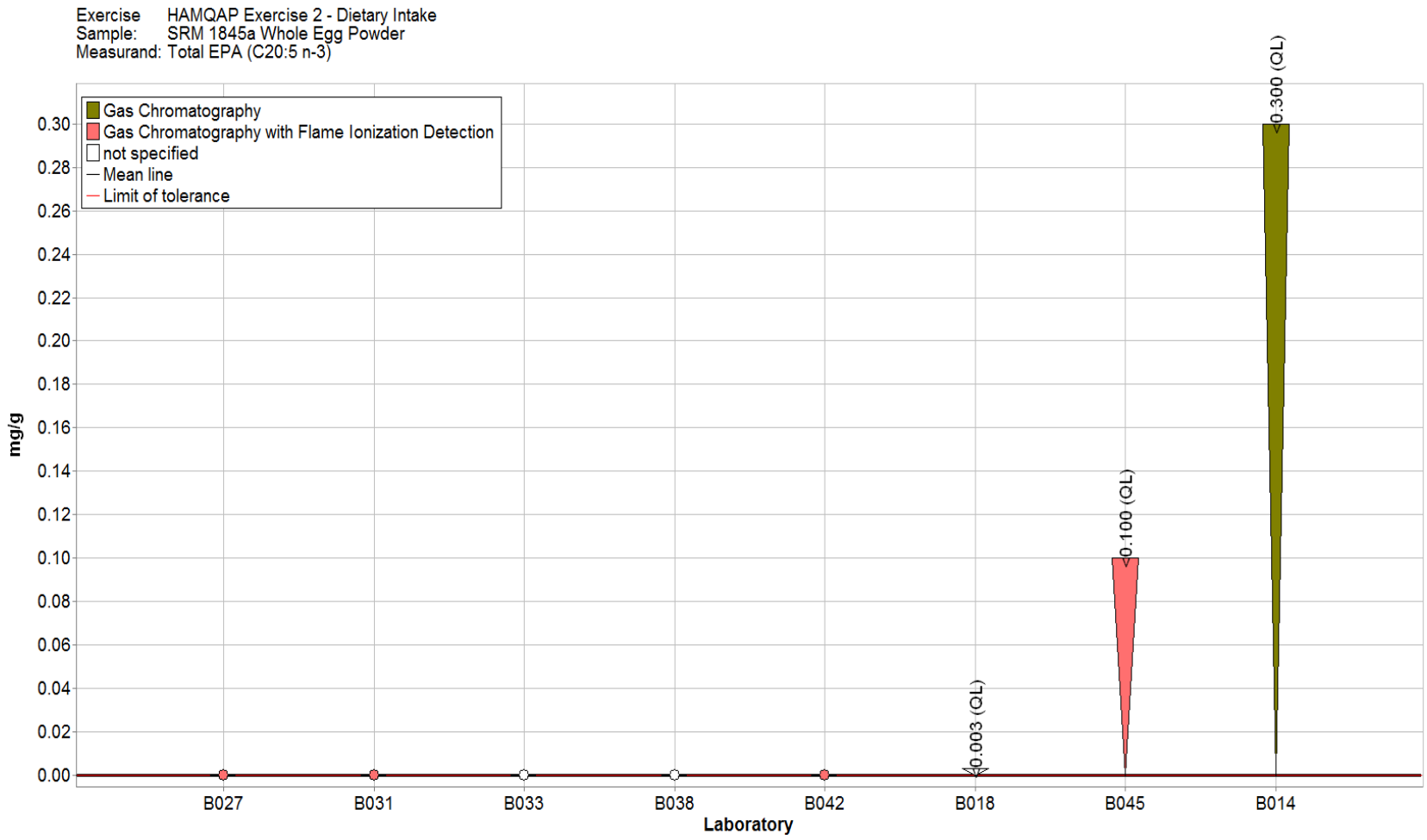

Figure 5-61. Total EPA (C20:5 n-3) in SRM 1845a Whole Egg Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. A NIST value has not been determined in this material.

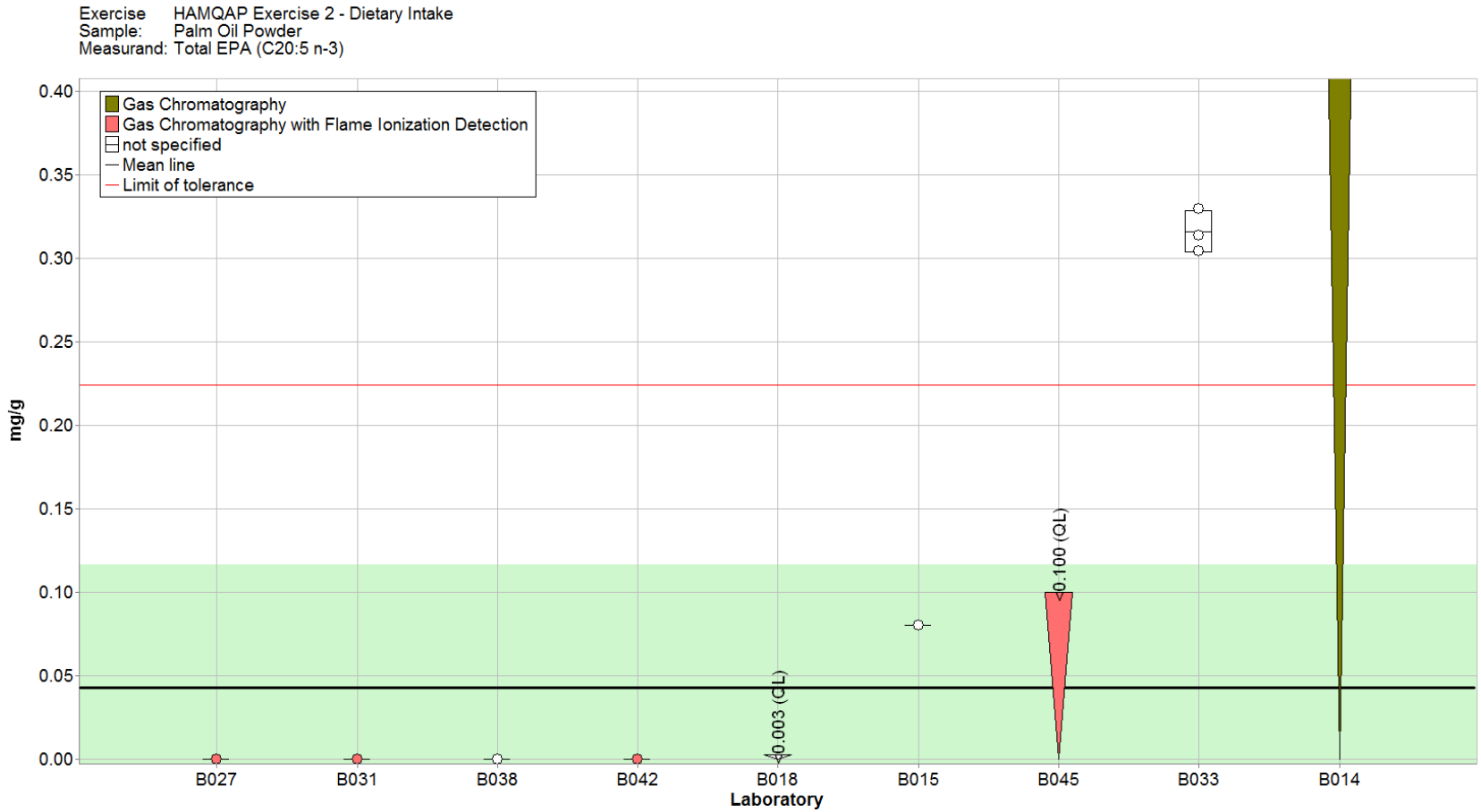

Figure 5-62. Total EPA (C20:5 n-3) in Palm Oil Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

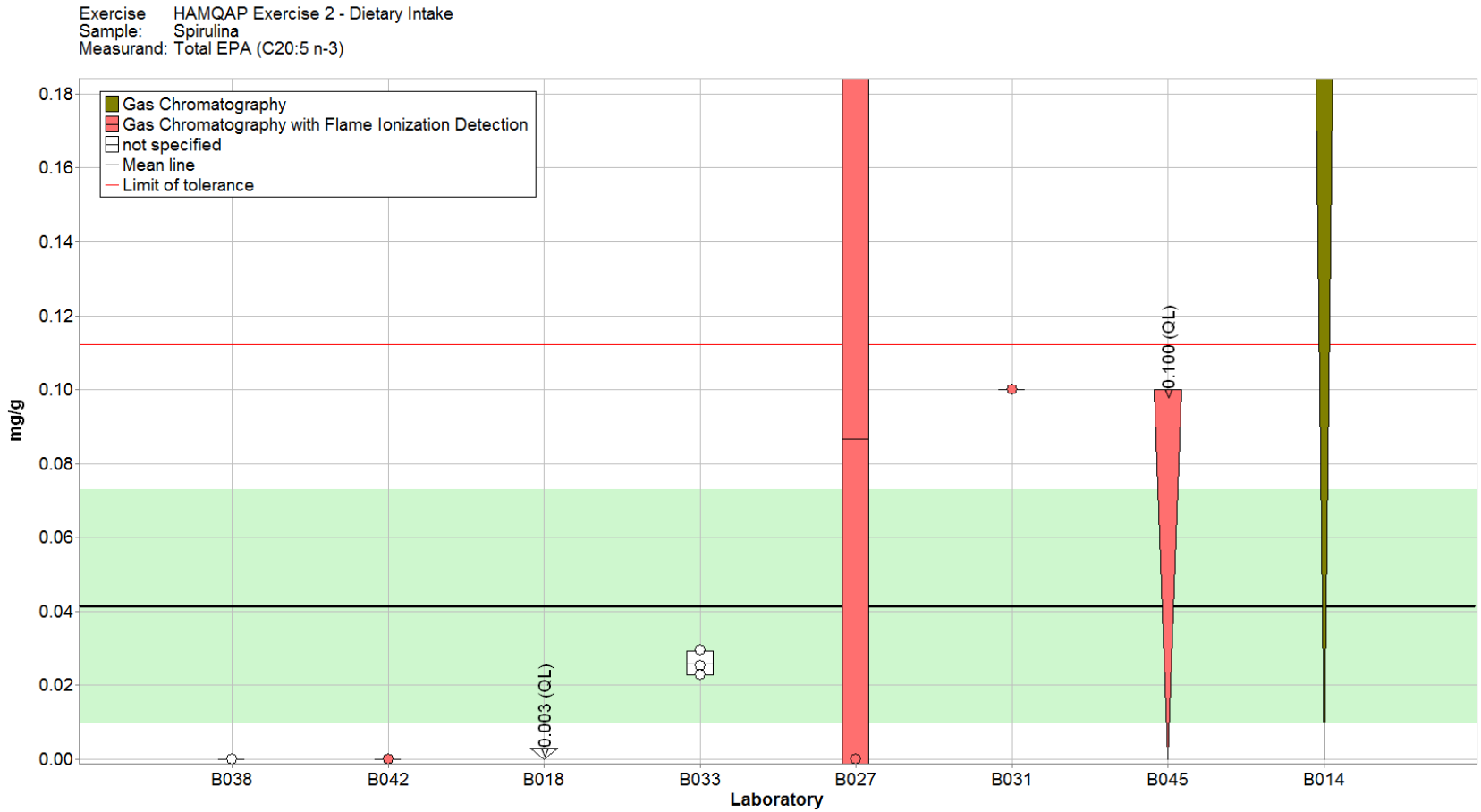

Figure 5-63. Total EPA (C20:5 n-3) in Spirulina (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

Table 5-21. Data summary table for total DPA in SRM 1845a Whole Egg Powder, Palm Oil Powder, and Spirulina.

		Total DPA (C22:5 n-3)														
		SRM 1845a Whole Egg Powder (mg/g)					Palm Oil Powder (mg/g)					Spirulina (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				0.20	0.01										
	B001															
	B002															
	B006															
	B014	< 0.300	< 0.300	< 0.300			< 0.500	< 0.500	< 0.500			< 0.300	< 0.300	< 0.300		
	B015						0.03	0.03	0.03	0.03	0					
	B016	0.132	0.165	0.146	0.148	0.017										
	B017															
	B018	< 0.003	< 0.003	< 0.003			< 0.003	< 0.003	< 0.003			< 0.003	< 0.003	< 0.003		
	B019															
	B027	0.28	0.28	0.29	0.2833	0.0058	0	0	0	0	0	0	0	0	0	0
	B031	0.2	0.2	0.15	0.183	0.029	0	0	0	0	0	0	0	0	0	0
	B034															
	B036															
	B039															
	B041															
	B042															
	B044															
	B045	0.28	0.27	0.28	0.2767	0.0058	< 0.100	< 0.100	< 0.100			< 0.100	< 0.100	< 0.100		
Community Results		Consensus Mean			0.22		Consensus Mean			0.010		Consensus Mean			0	
		Consensus Standard Deviation			0.12		Consensus Standard Deviation			0.024		Consensus Standard Deviation			0	
		Maximum			0.2833		Maximum			0.03		Maximum			0	
		Minimum			0.148		Minimum			0		Minimum			0	
		N			4		N			3		N			2	

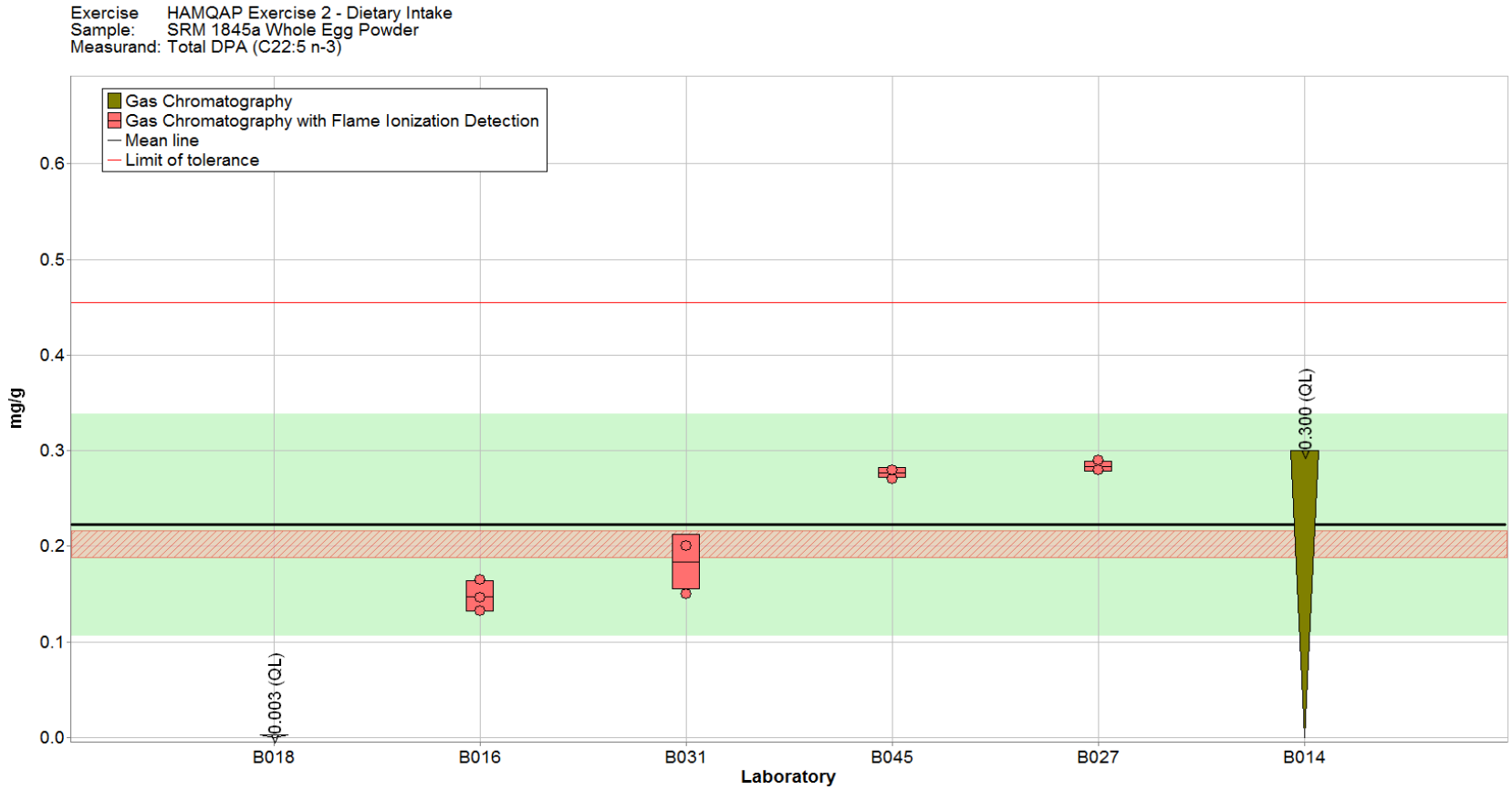

Figure 5-64. Total DPA (C22:5 n-3) in SRM 1845a Whole Egg Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

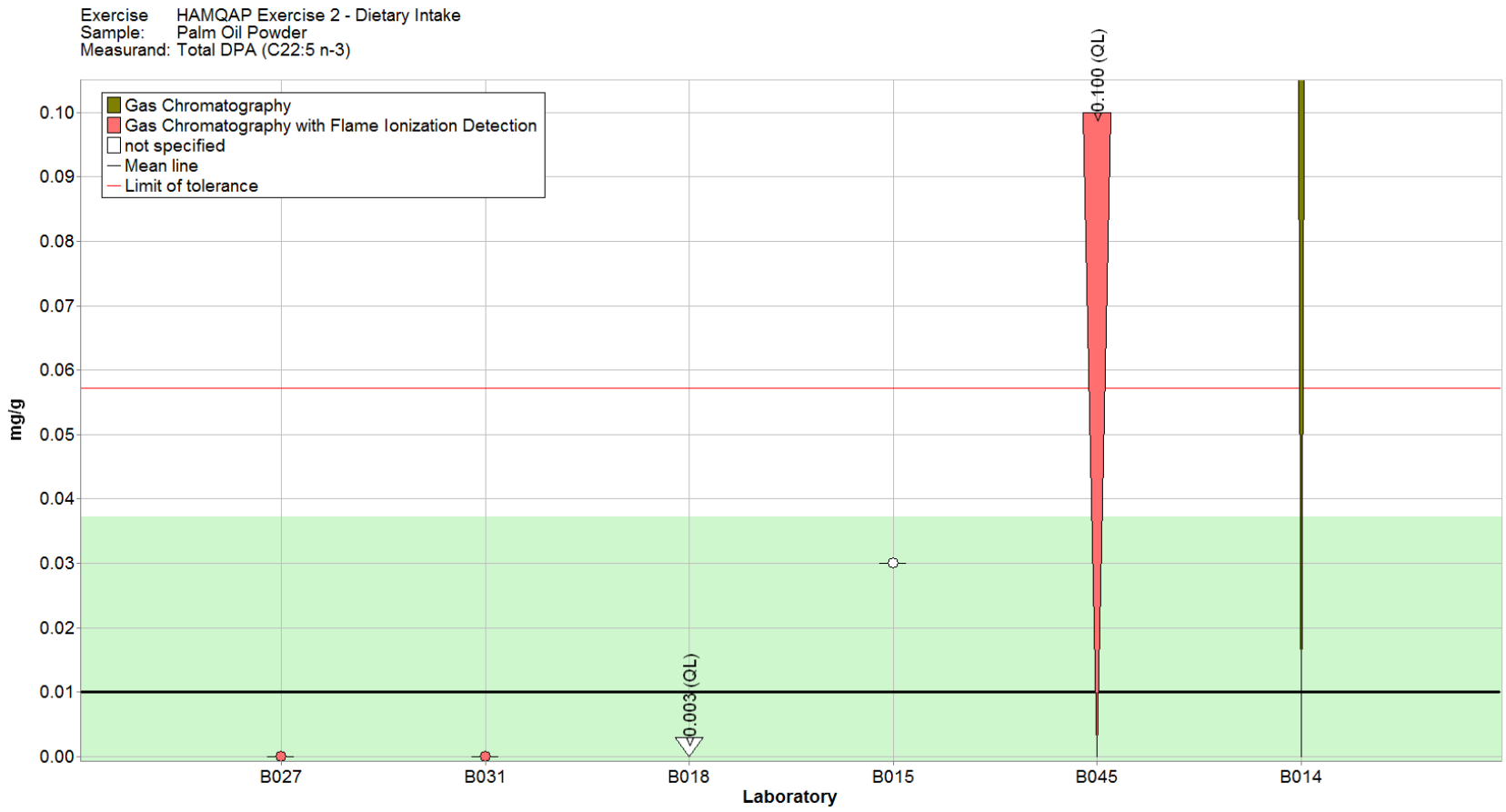

Figure 5-65. Total DPA (C22:5 n-3) in Palm Oil Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

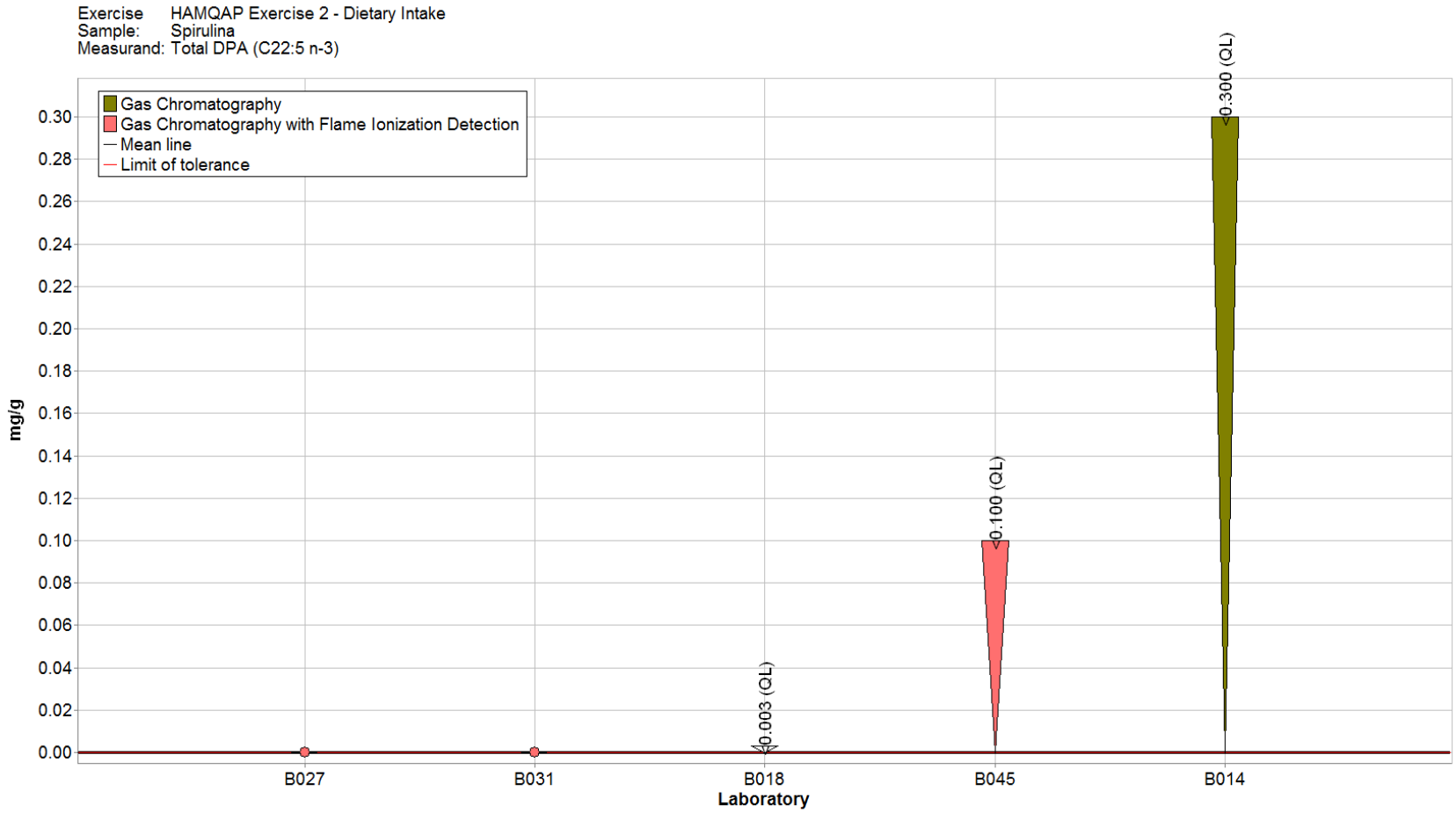

Figure 5-66. Total DPA (C22:5 n-3) in Spirulina (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. A NIST value has not been determined in this material.

Table 5-22. Data summary table for total DHA in SRM 1845a Whole Egg Powder, Palm Oil Powder, and Spirulina. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		Total DHA (C22:6 n-3)														
		SRM 1845a Whole Egg Powder (mg/g)					Palm Oil Powder (mg/g)					Spirulina (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				1.70	0.08										
	B001															
	B002															
	B006															
	B014	1.61	1.66	1.61	1.627	0.029	< 0.500	< 0.500	< 0.500			< 0.300	< 0.300	< 0.300		
	B015						0.02	0.02	0.02	0.0200	0					
	B016	1.07	0.712	0.742	0.84	0.20										
	B017															
	B018	7.927	7.95	6.93	7.60	0.58	< 0.003	< 0.003	< 0.003			< 0.003	< 0.003	< 0.003		
	B019															
	B027	2.08	2.09	2.14	2.103	0.032	0	0	0	0	0	0	0	0	0	0
	B031	1.6	1.5	1.55	1.550	0.050	0	0	0	0	0	0	0	0	0	0
	B033	0.098	0.113	0.108	0.106	0.008	0.022	0.025	0.024	0.0237	0.0012	0	0	0	0	0
	B034															
	B036															
	B038	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
	B039															
	B041															
	B042	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
	B044															
	B045	1.36	1.4	1.37	1.377	0.021	< 0.100	< 0.100	< 0.100			< 0.100	< 0.100	< 0.100		
Community Results		Consensus Mean			1.0		Consensus Mean			0.007		Consensus Mean			0	
		Consensus Standard Deviation			1.3		Consensus Standard Deviation			0.014		Consensus Standard Deviation			0	
		Maximum			7.60		Maximum			0.0237		Maximum			0	
		Minimum			0		Minimum			0		Minimum			0	
		N			9		N			6		N			5	

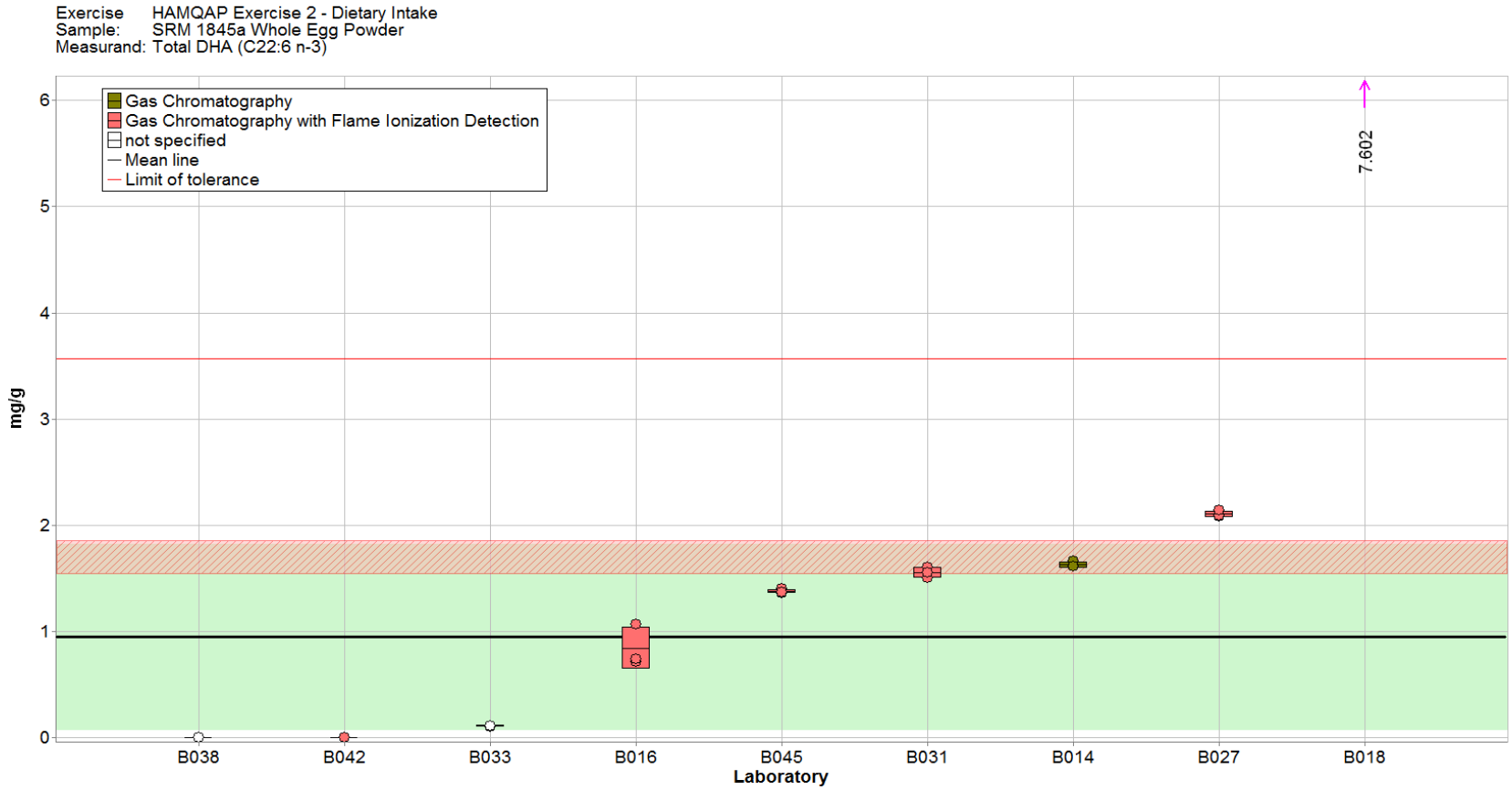

Figure 5-67. Total DHA (C22:6 n-3) in SRM 1845a Whole Egg Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

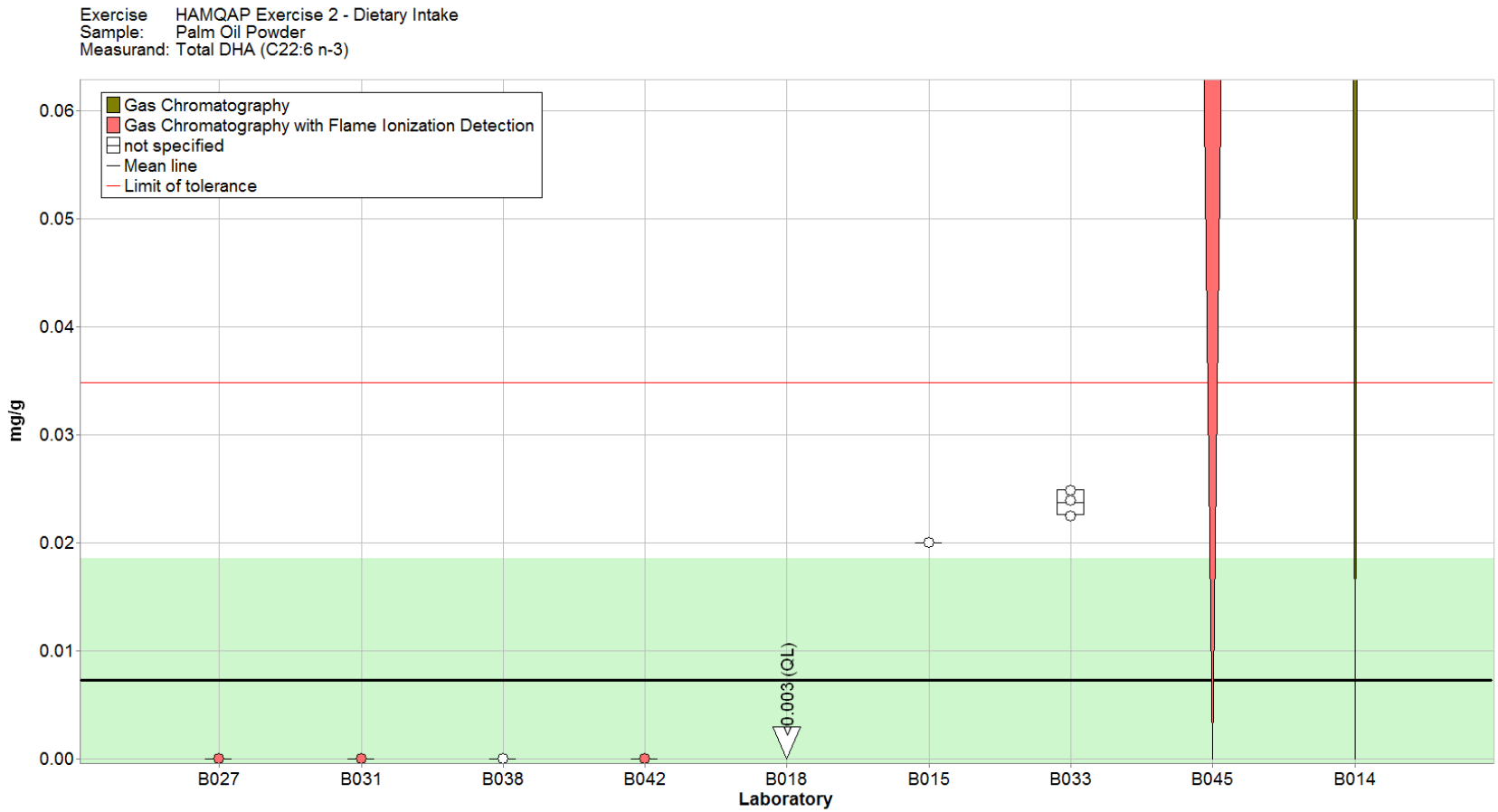

Figure 5-68. Total DHA (C22:6 n-3) in Palm Oil Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

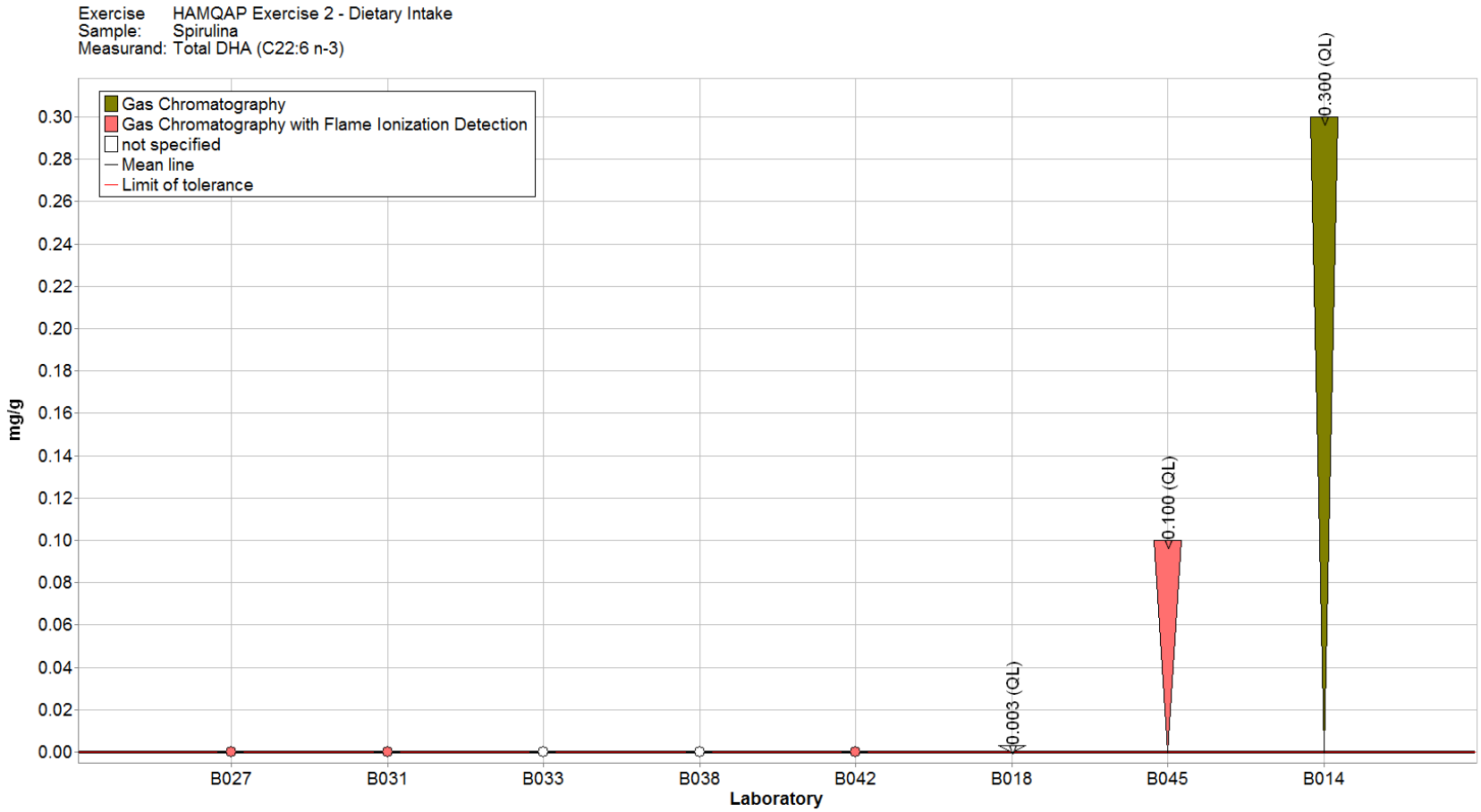

Figure 5-69. Total DHA (C22:6 n-3) in Spirulina (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. A NIST value has not been determined in this material.

Human Metabolites Sample Information

Human Milk. Participants were provided with three vials, each containing 5 mL of frozen human milk. Before use, participants were instructed to allow the material to thaw at room temperature for at least 30 min prior to sampling, use the material immediately after thawing, gently mix the contents prior to removal of a test portion for analysis, and use a sample size appropriate for their usual in-house method of analysis. Participants were asked to avoid exposing the material to direct UV light, to store the material at temperatures between –20 °C and –80 °C, and to prepare one sample and report one value from each vial provided. The approximate analyte levels were not reported to participants prior to the study, and target values for individual total fatty acids in SRM 1953 have not been determined.

Human Serum E. Participants were provided with three vials, each containing approximately 0.28 g of freeze-dried human serum. Before use, participants were instructed to reconstitute the serum in each vial with 3.00 mL of distilled water, use the material with 8 h, and use a sample size appropriate for their usual in-house method of analysis. Participants were asked to store the material at temperatures between 2 °C to 8 °C in the original unopened vials and to prepare one sample and report one value from each vial provided. The approximate analyte levels were not reported to participants prior to the study, and target values for individual total fatty acids in Human Serum E have not been determined.

Human Metabolites Study Results

- Eight to twelve laboratories enrolled in this exercise to measure total fatty acids. The table below lists the participation statistics for total fatty acids in the human metabolites study.

Analyte	Number of Laboratories Requesting Samples	Number of Laboratories Reporting Results (Percent Participation)	
		SRM 1953	Human Serum E
Total Caprylic Acid (C8:0)	10	1 (10 %)	1 (10 %)
Total Capric Acid (C10:0)	10	2 (20 %)	3 (30 %)
Total Lauric Acid (C12:0)	11	2 (18 %)	3 (27 %)
Total Myristic Acid (C14:0)	12	4 (33 %)	7 (58 %)
Total Myristoleic Acid (C14:1 n-5)	10	3 (30 %)	5 (50 %)
Total Palmitic Acid (C16:0)	12	4 (33 %)	7 (58 %)
Total Palmitoleic Acid (C16:1)	12	4 (33 %)	7 (58 %)
Total Stearic Acid (C18:0)	12	6 (50 %)	9 (75 %)
Total *cis*-Vaccenic Acid (C18:1 n-7)	9	3 (33 %)	4 (44 %)
Total Oleic Acid (C18:1 n-9)	12	5 (42 %)	9 (75 %)
Total *trans*-Vaccenic Acid (C18:1 n-7t)	8	1 (13 %)	1 (13 %)
Total Elaidic Acid (C18:1 n-9t)	8	2 (25 %)	2 (25 %)

Analyte	Number of Laboratories Requesting Samples	Number of Laboratories Reporting Results (Percent Participation)	
		SRM 1953	Human Serum E
Total Linoleic Acid (C18:2 n-6)	12	5 (42 %)	9 (75 %)
Total α-Linolenic Acid (C18:3 n-3)	12	5 (42 %)	7 (58 %)
Total γ-Linolenic Acid (C18:3 n-6)	11	5 (45 %)	7 (64 %)
Total Arachidic Acid (C20:0)	10	3 (30 %)	6 (60 %)
Total Dihomo-γ-Linolenic Acid (C20:3 n-6)	11	4 (36 %)	5 (45 %)
Total Arachidonic Acid (C20:4 n-6)	12	6 (50 %)	9 (75 %)
Total EPA (C20:5 n-3)	12	4 (33 %)	8 (67 %)
Total DPA (C22:5 n-3)	11	3 (27 %)	5 (45 %)
Total DHA (C22:6 n-3)	12	4 (33 %)	8 (67 %)

- The between-laboratory variability about the consensus means for total fatty acids, for both SRM 1953 and Human Serum E, ranged between 6 % RSD and 172 % RSD (see table below). The between-laboratory variability for most total fatty acids was good (< 75 % RSD), except for γ-linolenic acid, arachidic acid, dihomo-γ-linolenic acid, EPA, and DPA in SRM 1953 and myristoleic acid, *cis*-vaccenic acid, elaidic acid, α-linolenic acid, and DPA in Human Serum E. For all of the values with high between-laboratory variability (> 75%), the consensus means for the individual fatty acids was below 200 µmol/L.

Analyte	Between-Laboratory Variability	
	SRM 1953	Human Serum E
Total Caprylic Acid (C8:0)	NA	NA
Total Capric Acid (C10:0)	6 %	46 %
Total Lauric Acid (C12:0)	28 %	40 %
Total Myristic Acid (C14:0)	48 %	23 %
Total Myristoleic Acid (C14:1 n-5)	58 %	75 %
Total Palmitic Acid (C16:0)	66 %	56 %
Total Palmitoleic Acid (C16:1)	6 %	40 %
Total Stearic Acid (C18:0)	72 %	46 %
Total *cis*-Vaccenic Acid (C18:1 n-7)	67 %	117 %
Total Oleic Acid (C18:1 n-9)	7 %	68 %
Total *trans*-Vaccenic Acid (C18:1 n-7t)	NA	NA
Total Elaidic Acid (C18:1 n-9t)	40 %	150 %

Analyte	Between-Laboratory Variability SRM 1953	Human Serum E
Total Linoleic Acid (C18:2 n-6)	25 %	71 %
Total α-Linolenic Acid (C18:3 n-3)	40 %	110 %
Total γ-Linolenic Acid (C18:3 n-6)	85 %	52 %
Total Arachidic Acid (C20:0)	172 %	14 %
Total Dihomo-γ-Linolenic Acid (C20:3 n-6)	96 %	23 %
Total Arachidonic Acid (C20:4 n-6)	69 %	63 %
Total EPA (C20:5 n-3)	157 %	53 %
Total DPA (C22:5 n-3)	116 %	94 %
Total DHA (C22:6 n-3)	38 %	38 %

- Up to three laboratories reported using GC-FID, four laboratories reported using GC-MS, and one laboratory reported using GC (detector unspecified) for determination of total fatty acids in SRM 1953 and Human Serum E.

Human Metabolites Technical Recommendations

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

- The between-laboratory variability was good for most fatty acids in these two samples. Lower levels of individual total fatty acids (below 200 µmol/L) typically resulted in higher between-laboratory variability (> 75 % RSD). Future studies should continue to include both high and low levels of individual fatty acids, allowing laboratories to understand the concentration-dependent variability of their individual methods.
- The use of atypical matrices (human milk and lyophilized human serum) did not impact the precision of the measurements. For a future study, a more typical sample for clinical analysis (such as frozen human serum) could be provided alongside unusual materials to verify this conclusion.
- All results should be checked closely to avoid calculation errors and to ensure that results are reported in the requested units.
- Generally, fewer participants reported results for the two *trans*-fatty acids (*trans*-vaccenic and elaidic acids). Trans-fatty acids should be included in future studies only if the goal is to specifically evaluate laboratory performance of *trans*-fatty acid measurements.

Table 5-23. Individualized data summary table (NIST) for total fatty acids in SRM 1953 Organic Contaminants in Non-Fortified Human Milk and Human Serum E.

National Institute of Standards & Technology

HAMQAP Exercise 2 - Fatty Acids

		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 Caprylic Acid (C8:0)	SRM 1953 Organic Contaminants in Non-Fortified Human Milk	µmol/L					1				
Total Caprylic Acid (C8:0)	Human Serum E	µmol/L					1				
Total Capric Acid (C10:0)	SRM 1953 Organic Contaminants in Non-Fortified Human Milk	µmol/L					2	510	28		
Total Capric Acid (C10:0)	Human Serum E	µmol/L					3	3.9	1.8		
Total Lauric Acid (C12:0)	SRM 1953 Organic Contaminants in Non-Fortified Human Milk	µmol/L					2	1820	510		
Total Lauric Acid (C12:0)	Human Serum E	µmol/L					3	11.7	4.7		
Total Myristic Acid (C14:0)	SRM 1953 Organic Contaminants in Non-Fortified Human Milk	µmol/L					4	1930	880		
Total Myristic Acid (C14:0)	Human Serum E	µmol/L					7	160	36		
Total Myristoleic Acid (C14:1 n-5)	SRM 1953 Organic Contaminants in Non-Fortified Human Milk	µmol/L					3	80	49		
Total Myristoleic Acid (C14:1 n-5)	Human Serum E	µmol/L					5	10	6		
Total Palmitic Acid (C16:0)	SRM 1953 Organic Contaminants in Non-Fortified Human Milk	µmol/L					4	5460	3600		
Total Palmitic Acid (C16:0)	Human Serum E	µmol/L					7	3480	2000		
Total Palmitoleic Acid (C16:1 n-7)	SRM 1953 Organic Contaminants in Non-Fortified Human Milk	µmol/L					4	580	33		
Total Palmitoleic Acid (C16:1 n-7)	Human Serum E	µmol/L					7	320	130		
Total Stearic Acid (C18:0)	SRM 1953 Organic Contaminants in Non-Fortified Human Milk	µmol/L					6	1870	1400		
Total Stearic Acid (C18:0)	Human Serum E	µmol/L					9	1520	700		
Total cis-Vaccenic Acid (C18:1 n-7)	SRM 1953 Organic Contaminants in Non-Fortified Human Milk	µmol/L					3	410	280		
Total cis-Vaccenic Acid (C18:1 n-7)	Human Serum E	µmol/L					4	180	220		
Total Oleic Acid (C18:1 n-9)	SRM 1953 Organic Contaminants in Non-Fortified Human Milk	µmol/L					5	8930	640		
Total Oleic Acid (C18:1 n-9)	Human Serum E	µmol/L					9	2540	1700		
Total Transvaccenic Acid (C18:1 n-7t)	SRM 1953 Organic Contaminants in Non-Fortified Human Milk	µmol/L					1				
Total Transvaccenic Acid (C18:1 n-7t)	Human Serum E	µmol/L					1				
Total Elaidic Acid (C18:1 n-9t)	SRM 1953 Organic Contaminants in Non-Fortified Human Milk	µmol/L					2	280	110		
Total Elaidic Acid (C18:1 n-9t)	Human Serum E	µmol/L					2	40	66		
Total Linoleic Acid (C18:2 n-6)	SRM 1953 Organic Contaminants in Non-Fortified Human Milk	µmol/L					5	4290	1100		
Total Linoleic Acid (C18:2 n-6)	Human Serum E	µmol/L					9	3150	2200		
Total alpha-Linolenic Acid (C18:3 n-3)	SRM 1953 Organic Contaminants in Non-Fortified Human Milk	µmol/L					5	350	140		
Total alpha-Linolenic Acid (C18:3 n-3)	Human Serum E	µmol/L					7	40	44		
Total gamma-Linolenic Acid (C18:3 n-6)	SRM 1953 Organic Contaminants in Non-Fortified Human Milk	µmol/L					5	120	100		
Total gamma-Linolenic Acid (C18:3 n-6)	Human Serum E	µmol/L					7	20	11		
Total Arachidic Acid (C20:0)	SRM 1953 Organic Contaminants in Non-Fortified Human Milk	µmol/L					3	110	190		
Total Arachidic Acid (C20:0)	Human Serum E	µmol/L					6	33.2	4.7		
Total Dihomo-gamma-linolenic acid (C20:3 n-6)	SRM 1953 Organic Contaminants in Non-Fortified Human Milk	µmol/L					4	150	150		
Total Dihomo-gamma-linolenic acid (C20:3 n-6)	Human Serum E	µmol/L					5	210	48		
Total Arachidonic Acid (C20:4 n-6)	SRM 1953 Organic Contaminants in Non-Fortified Human Milk	µmol/L					6	280	190		
Total Arachidonic Acid (C20:4 n-6)	Human Serum E	µmol/L					9	690	430		
Total EPA (C20:5 n-3)	SRM 1953 Organic Contaminants in Non-Fortified Human Milk	µmol/L					4	30	44		
Total EPA (C20:5 n-3)	Human Serum E	µmol/L					8	30	17		
Total DPA (C22:5 n-3)	SRM 1953 Organic Contaminants in Non-Fortified Human Milk	µmol/L					3	60	73		
Total DPA (C22:5 n-3)	Human Serum E	µmol/L					5	30	32		
Total DHA (C22:6 n-3)	SRM 1953 Organic Contaminants in Non-Fortified Human Milk	µmol/L					4	80	32		
Total DHA (C22:6 n-3)	Human Serum E	µmol/L					8	100	38		

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 5-24. Data summary table for total caprylic acid in SRM 1953 Organic Contaminants in Non-Fortified Human Milk and Human Serum E.

		Total Caprylic Acid (C8:0)									
		SRM 1953 Organic Contaminants in Non-Fortified Human Milk (µmol/L)					Human Serum E (µmol/L)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target										
	B014	< 110	< 110	< 110			< 200	< 200	< 200		
	B016	45.42	48.54	56.86	50.3	5.9					
	B017										
	B031										
	B034										
	B041										
	B047										
	B053										
	B055										
	B058						5.3	4.8	5.6	5.23	0.40
Community Results		Consensus Mean					Consensus Mean				
		Consensus Standard Deviation					Consensus Standard Deviation				
		Maximum			50.3		Maximum			5.2	
		Minimum			50.3		Minimum			5.2	
		N			1		N			1	

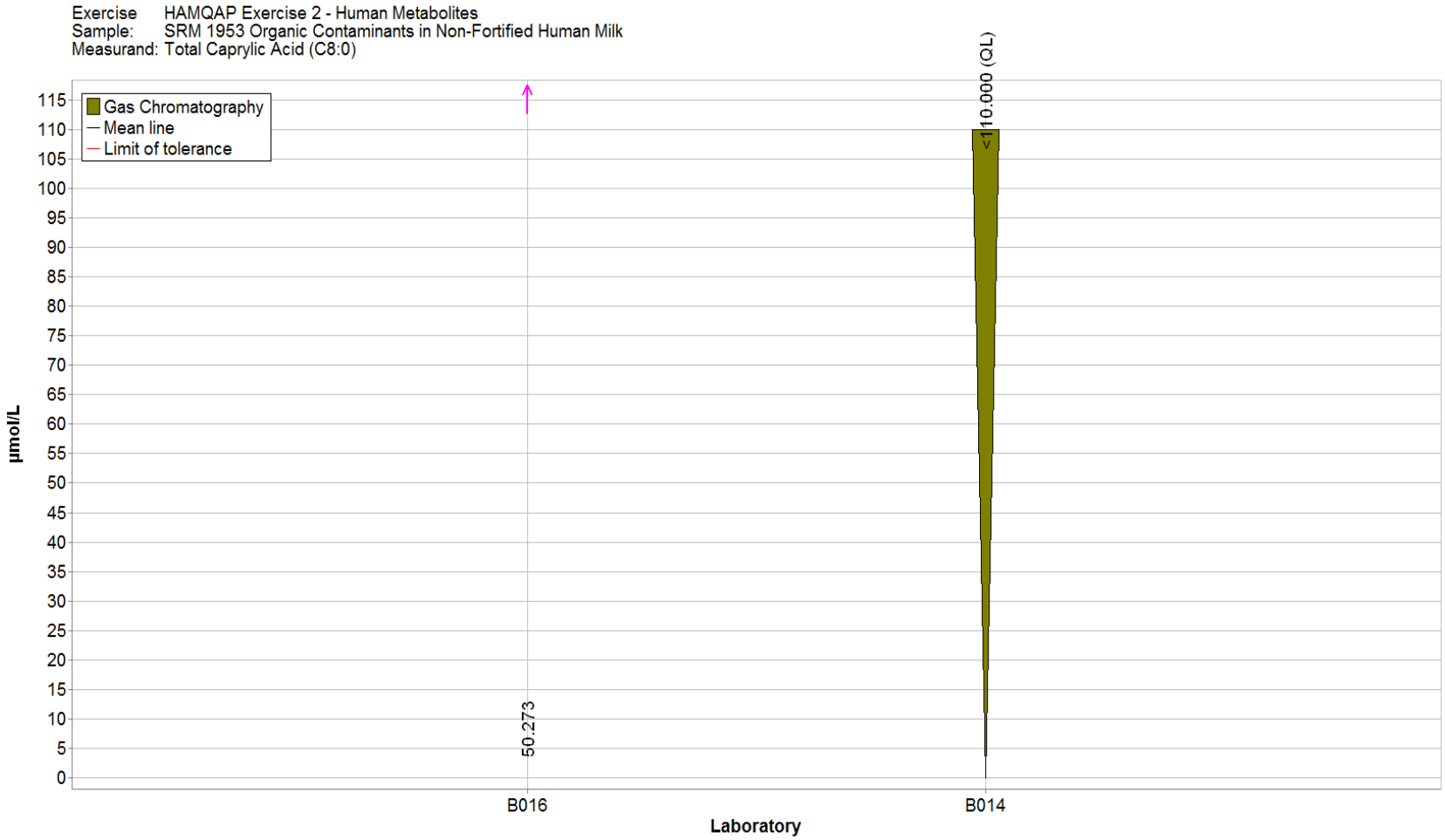

Figure 5-70. Total caprylic acid (C8:0) in SRM 1953 Organic Contaminants in Non-Fortified Human Milk (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A NIST value has not been determined in this material.

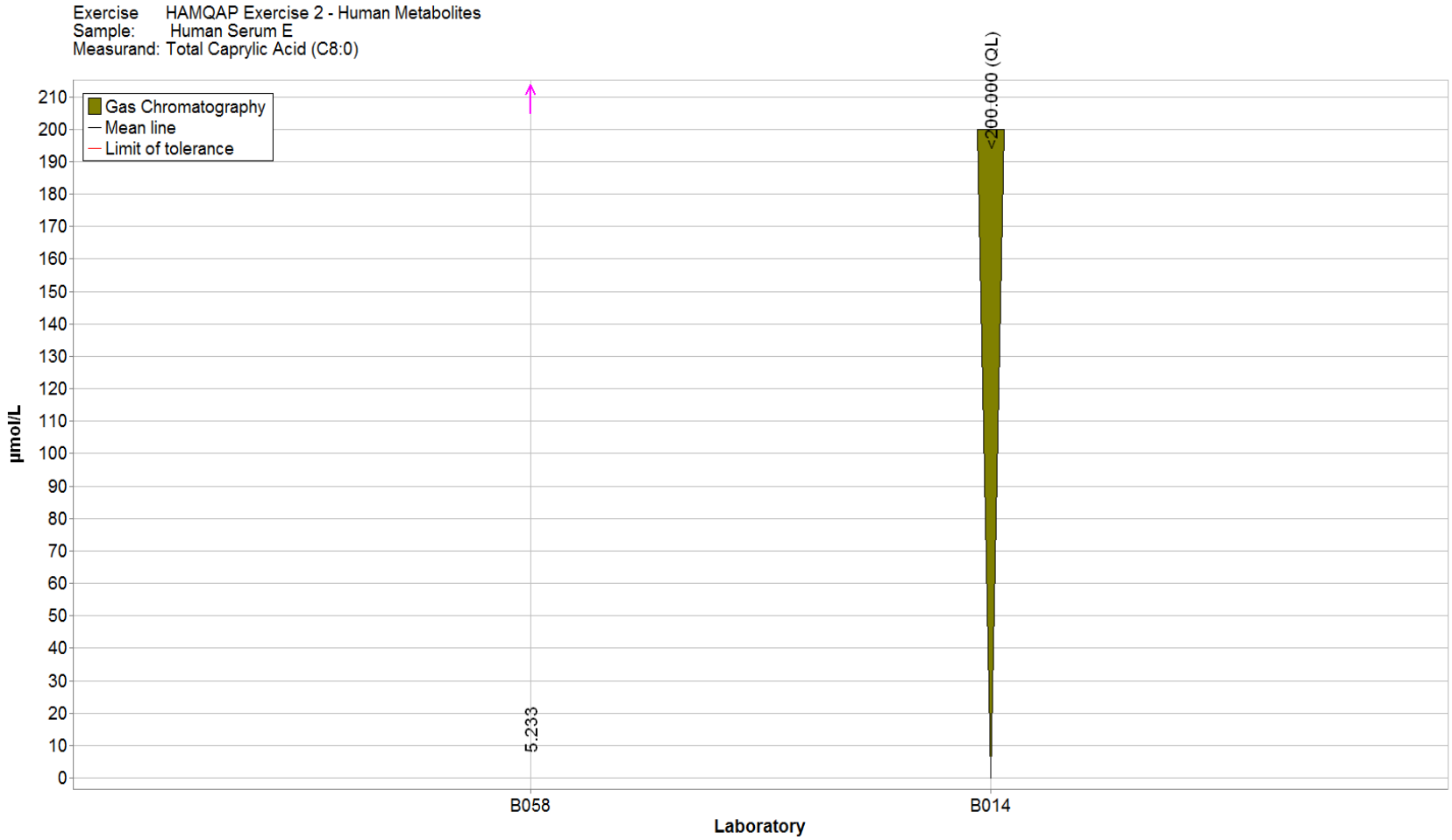

Figure 5-71. Total caprylic acid (C8:0) in Human Serum E (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. A NIST value has not been determined in this material.

Table 5-25. Data summary table for total capric acid in SRM 1953 Organic Contaminants in Non-Fortified Human Milk and Human Serum E.

		Total Caprylic Acid (C8:0)									
		SRM 1953 Organic Contaminants in Non-Fortified Human Milk (µmol/L)					Human Serum E (µmol/L)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target										
	B014	< 110	< 110	< 110			< 200	< 200	< 200		
	B016	45.42	48.54	56.86	50.3	5.9					
	B017										
	B031										
	B034										
	B041										
	B047										
	B053										
	B055										
	B058						5.3	4.8	5.6	5.2	0.40
Community Results		Consensus Mean					Consensus Mean				
		Consensus Standard Deviation					Consensus Standard Deviation				
		Maximum			50.3		Maximum			5.2	
		Minimum			50.3		Minimum			5.2	
		N			1		N			1	

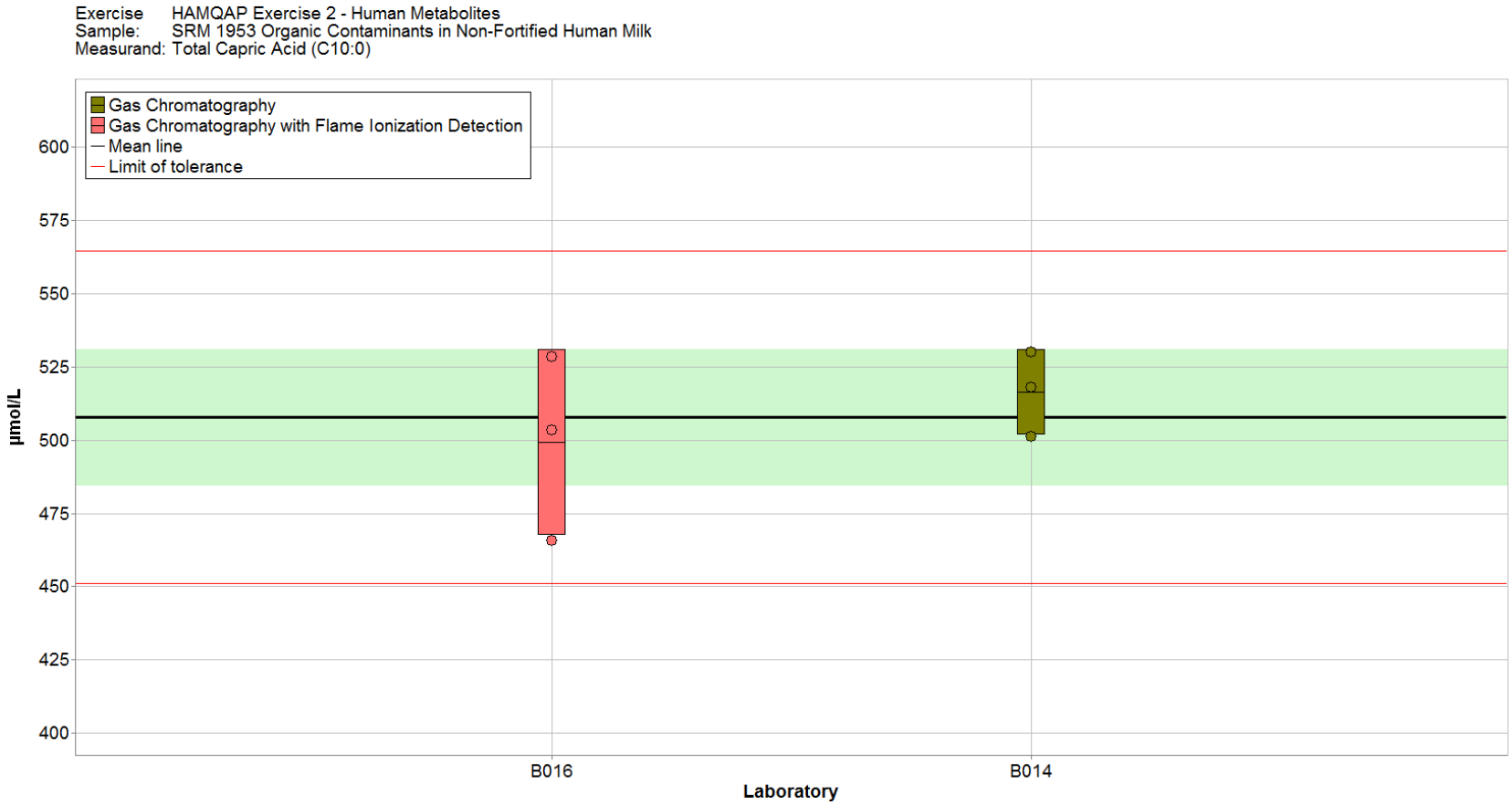

Figure 5-72. Total capric acid (C10:0) in SRM 1953 Organic Contaminants in Non-Fortified Human Milk (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

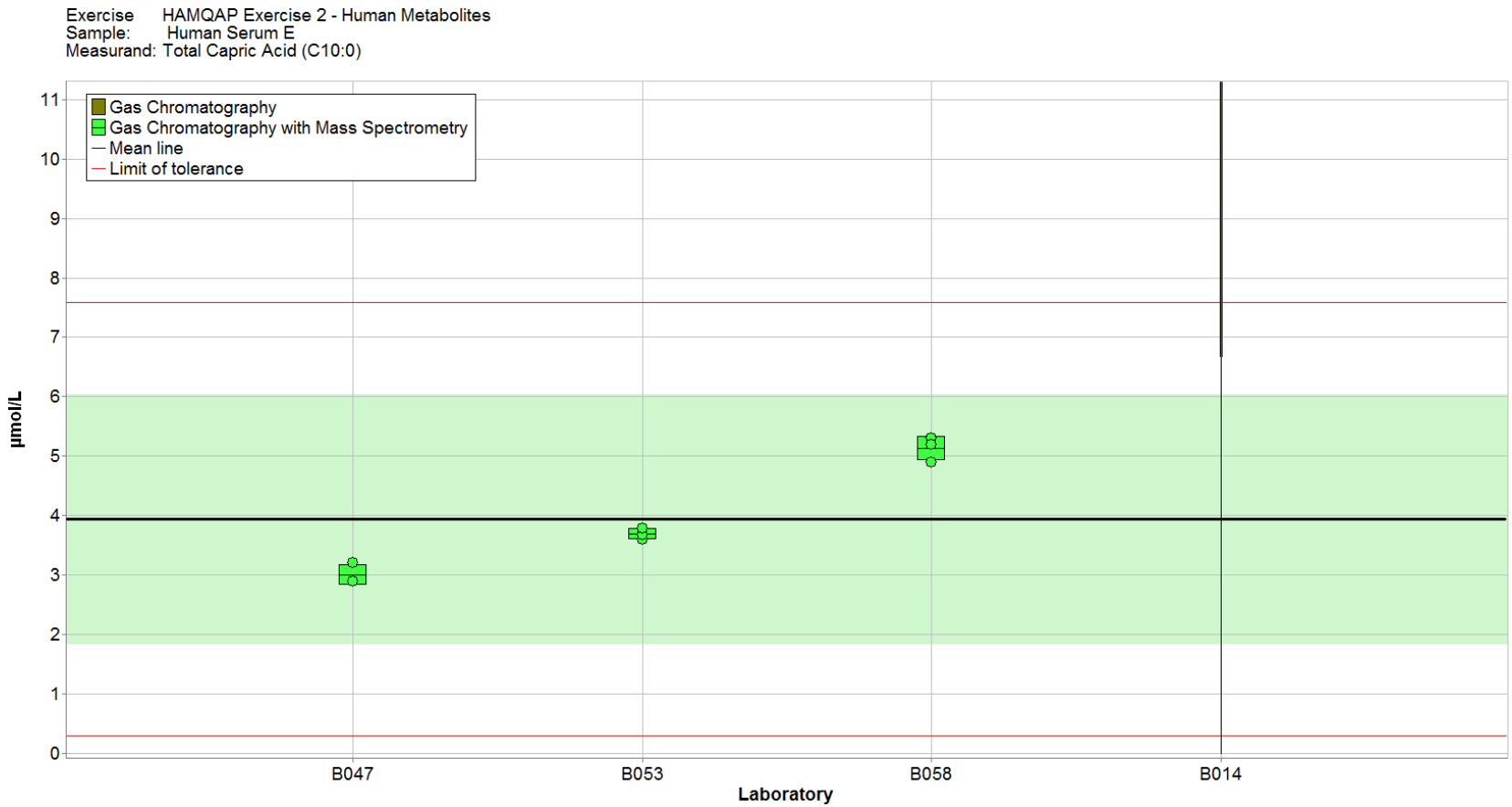

Figure 5-73. Total capric acid (C10:0) in Human Serum E (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

Table 5-26. Data summary table for total lauric acid in SRM 1953 Organic Contaminants in Non-Fortified Human Milk and Human Serum E.

		Total Lauric Acid (C12:0)									
		SRM 1953 Organic Contaminants in Non-Fortified Human Milk (µmol/L)					Human Serum E (µmol/L)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target										
	B014	1934	1982	2001	1972	35	< 200	< 200	< 200		
	B015										
	B016	1709	1572	1752	1678	94					
	B017										
	B031										
	B034										
	B041										
	B047						8.3	7.4	7.2	7.63	0.59
	B053						12.9	13.4	15.4	13.9	1.3
	B055										
	B058						15	14.2	11.2	13.5	2.0
Community Results		Consensus Mean			1825		Consensus Mean			11.7	
		Consensus Standard Deviation			508		Consensus Standard Deviation			4.7	
		Maximum			1972		Maximum			13.9	
		Minimum			1678		Minimum			7.63	
		N			2		N			3	

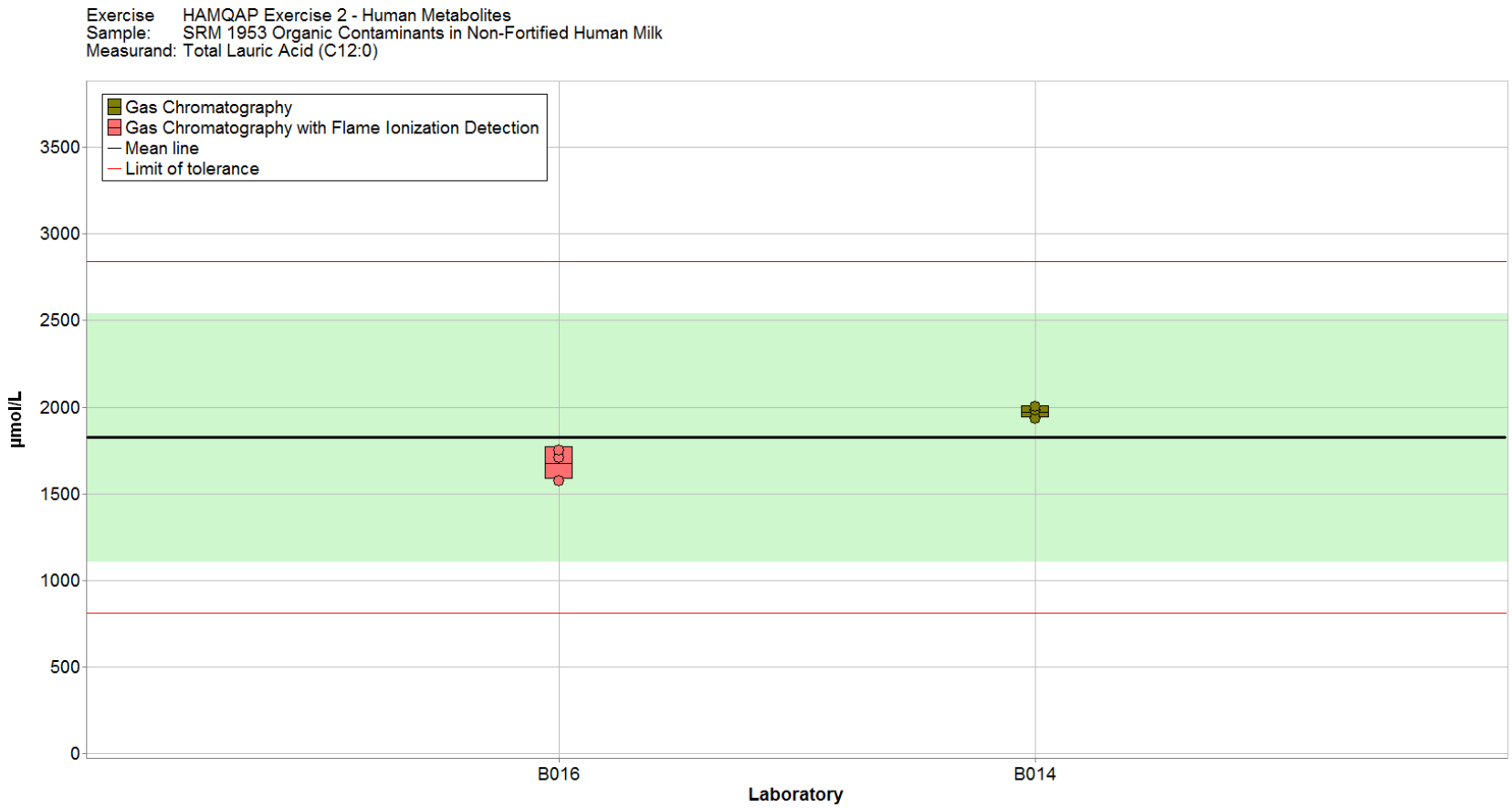

Figure 5-74. Total lauric acid (C12:0) in SRM 1953 Organic Contaminants in Non-Fortified Human Milk (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

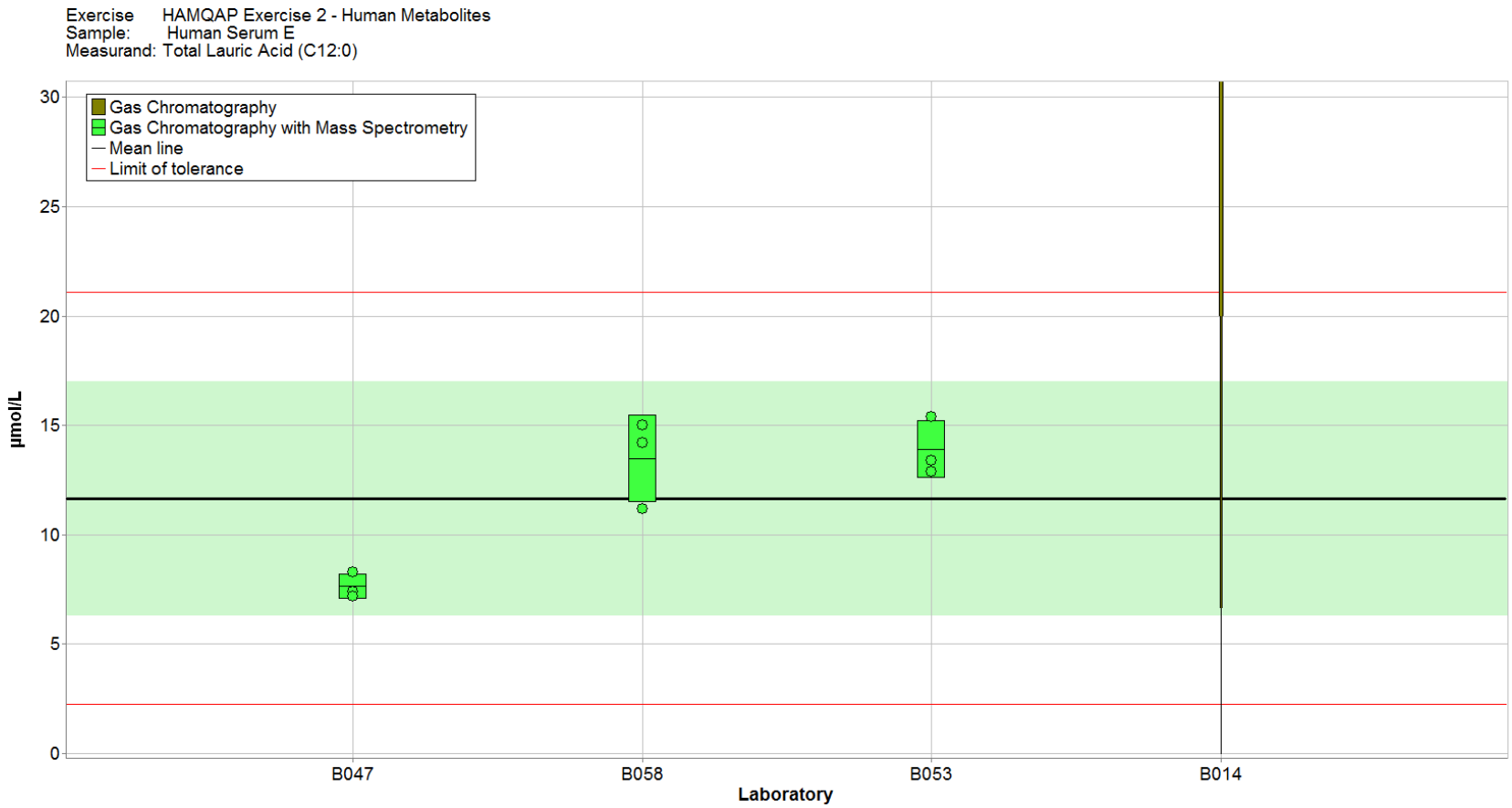

Figure 5-75. Total lauric acid (C12:0) in Human Serum E (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

Table 5-27. Data summary table for total myristic acid in SRM 1953 Organic Contaminants in Non-Fortified Human Milk and Human Serum E. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		Total Myristic Acid (C14:0)									
		SRM 1953 Organic Contaminants in Non-Fortified Human Milk (µmol/L)					Human Serum E (µmol/L)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target										
	B014	2083	2132	2145	2120	33	< 200	< 200	< 200		
	B015										
	B016	1647	1521	1682	1617	85	49.92	49.04	48.6	49.19	0.67
	B017										
	B031										
	B034										
	B041						188.68	189.69	200.85	193.1	6.8
	B045	2005	2073	2048	2042	34	189.42	191.12	188.96	189.8	1.1
	B047						65	60	59	61.3	3.2
	B053						153	156	192	167	22
	B055	6023.1	6673.4	7249.6	6649	614	194.9	196.2	204.5	198.5	5.2
	B058						153	114	139	135	20
Community Results		Consensus Mean			1926		Consensus Mean			155	
		Consensus Standard Deviation			882		Consensus Standard Deviation			36	
		Maximum			6649		Maximum			198.5	
		Minimum			1617		Minimum			49.19	
		N			4		N			7	

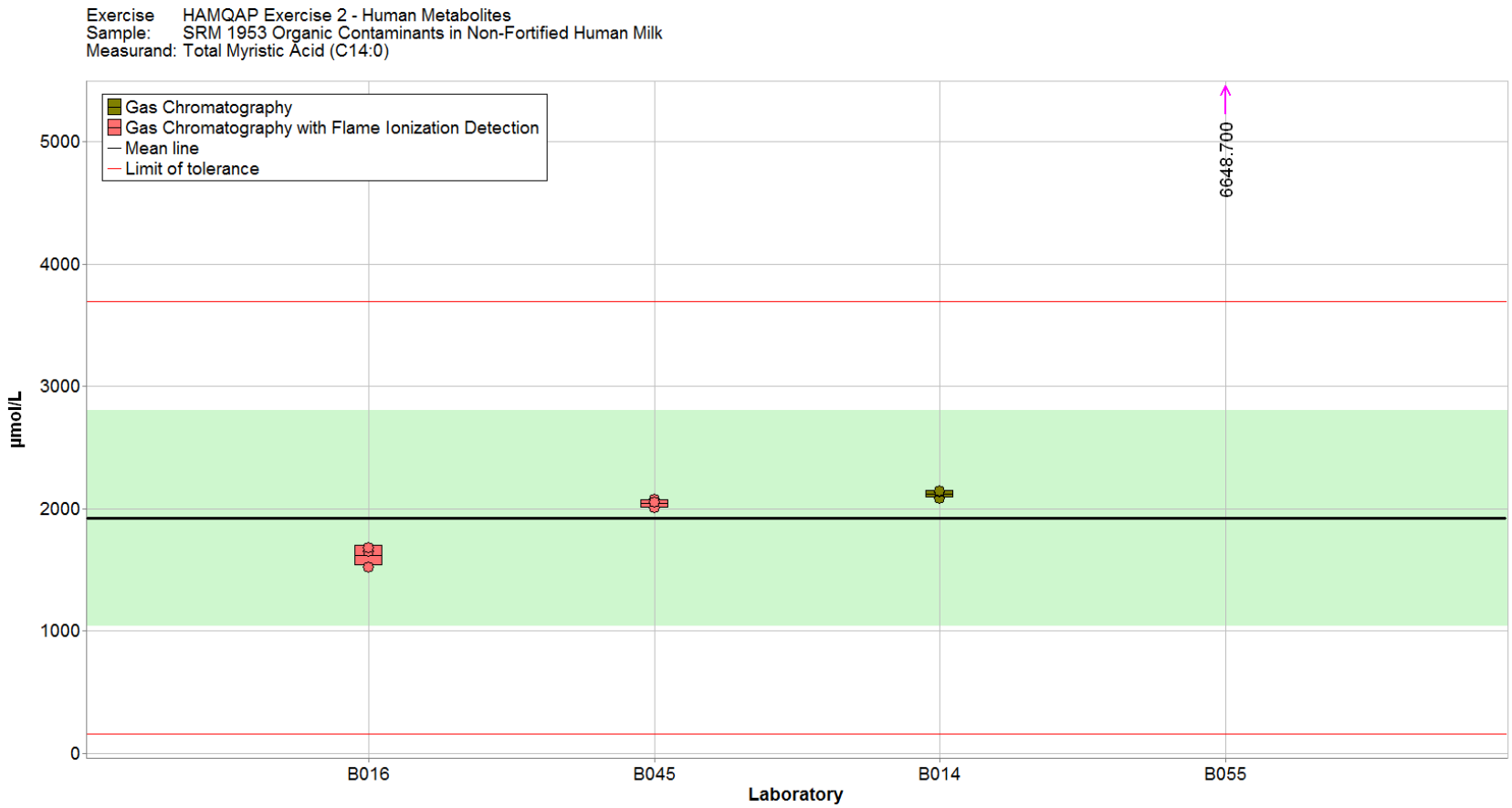

Figure 5-76. Total myristic acid (C14:0) in SRM 1953 Organic Contaminants in Non-Fortified Human Milk (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

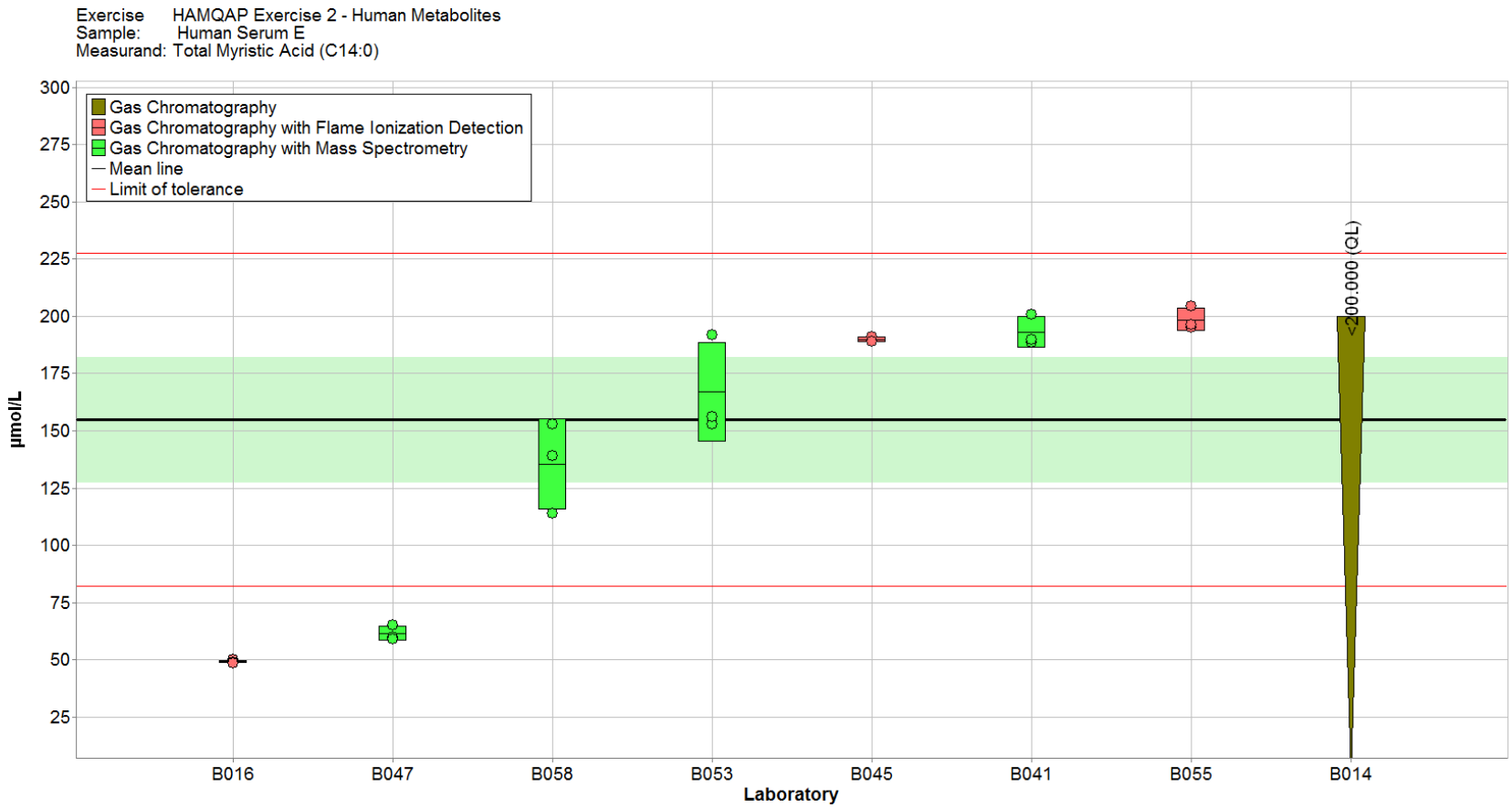

Figure 5-77. Total myristic acid (C14:0) in Human Serum E (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

Table 5-28. Data summary table for total myristoleic acid in SRM 1953 Organic Contaminants in Non-Fortified Human Milk and Human Serum E.

		Total Myristoleic Acid (C14:1 n-5)									
		SRM 1953 Organic Contaminants in Non-Fortified Human Milk (µmol/L)					Human Serum E (µmol/L)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target										
	B014	82.6	82.6	86.9	84.0	2.5	< 200	< 200	< 200		
	B016	61.62	56.54	63.39	60.5	3.6	3.13	3.13	3.13	3.13	0
	B017										
	B031										
	B034										
	B041						19.43	16.05	16.99	17.5	1.7
	B047	106.4	107	106.8	106.7	0.3	4.6	3.7	4	4.10	0.46
	B053						8.55	8.53	10.9	9.3	1.4
	B055										
	B058						7.05	6.2	5.8	6.35	0.64
Community Results		Consensus Mean			84		Consensus Mean			8.0	
		Consensus Standard Deviation			49		Consensus Standard Deviation			6.0	
		Maximum			106.7		Maximum			17.5	
		Minimum			60.5		Minimum			3.13	
		N			3		N			5	

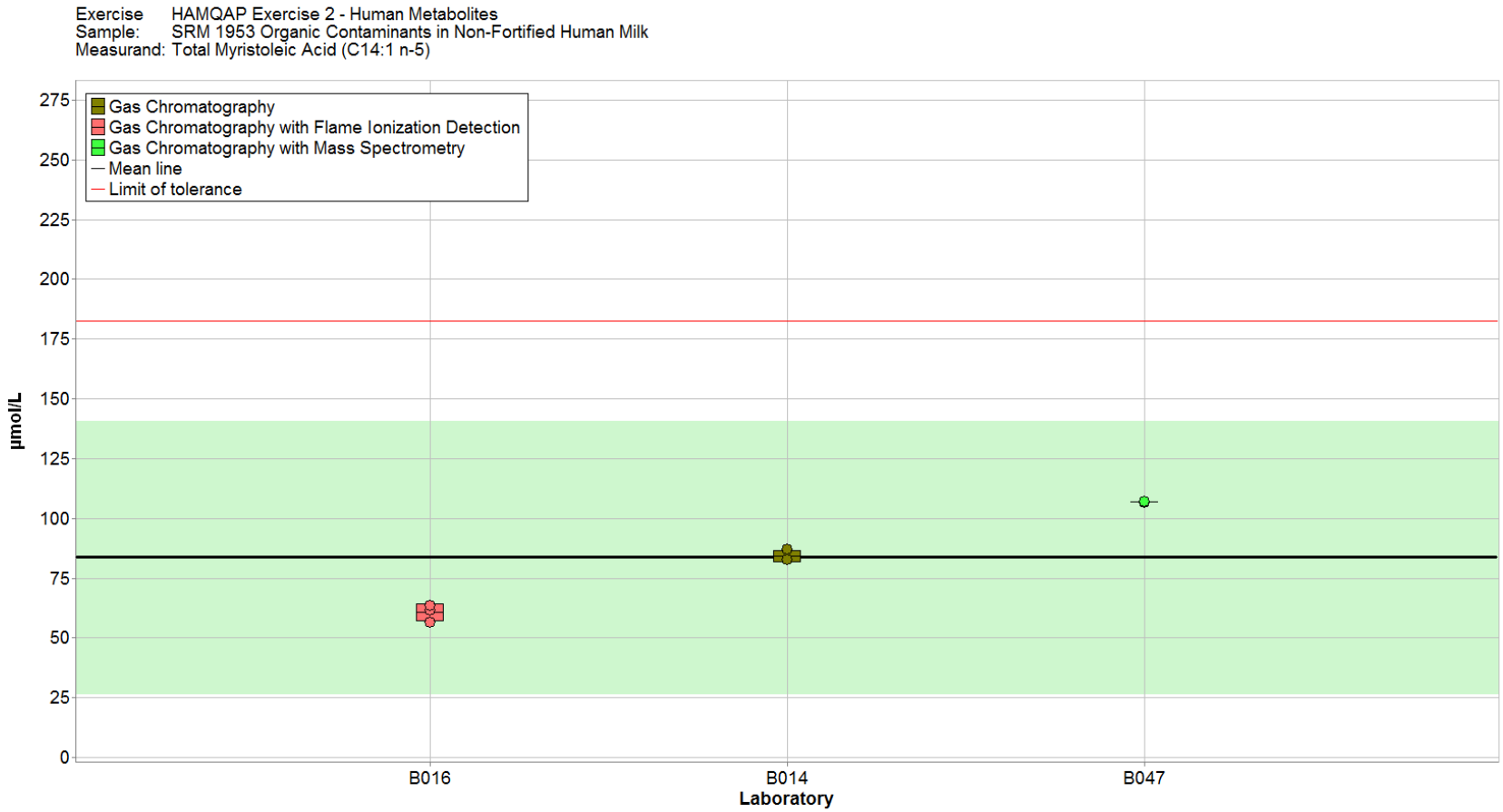

Figure 5-78. Total myristoleic acid (C14:1 n-5) in SRM 1953 Organic Contaminants in Non-Fortified Human Milk (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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. A NIST value has not been determined in this material.

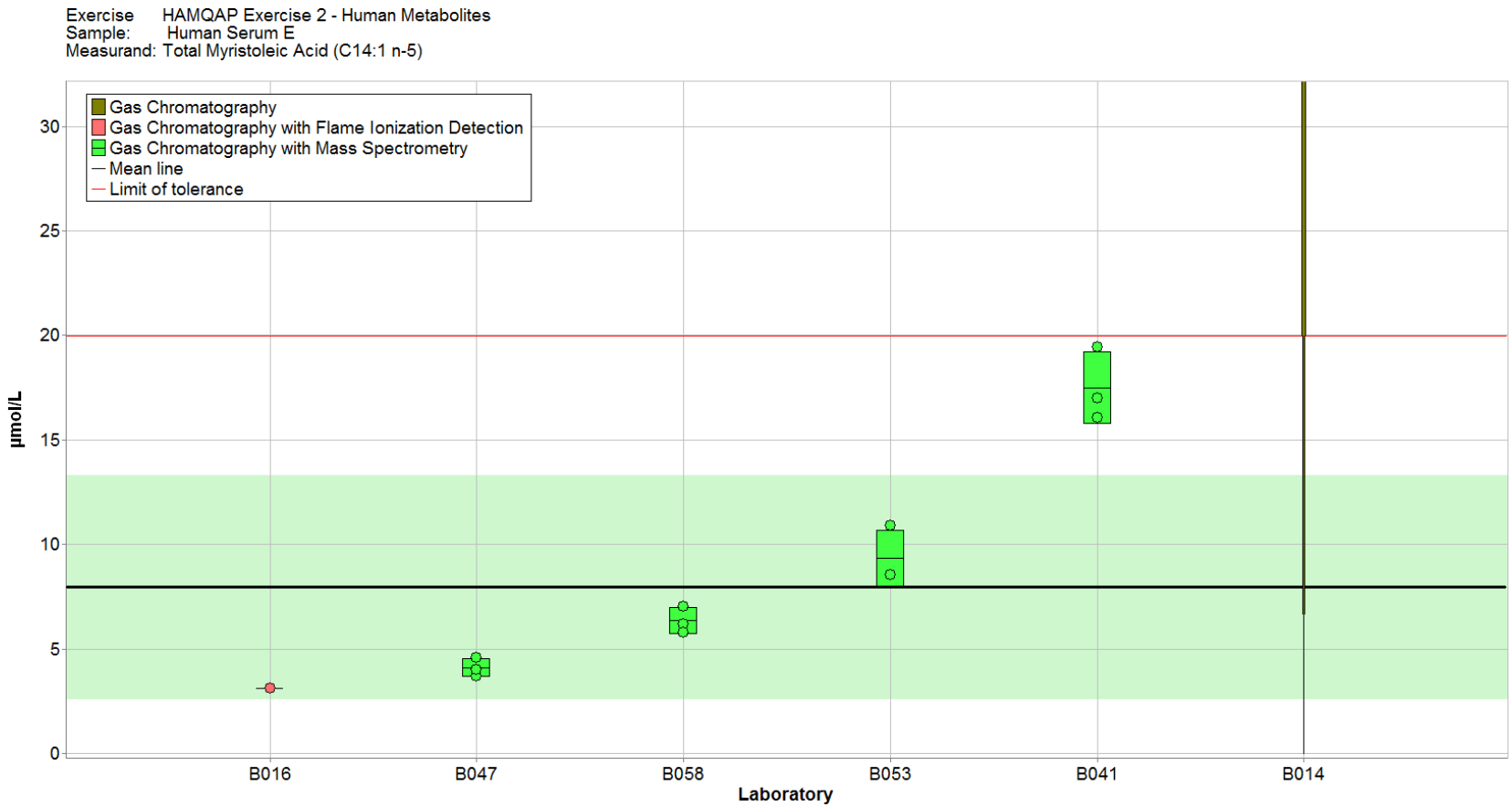

Figure 5-79. Total myristoleic acid (C14:1 n-5) in Human Serum E (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

Table 5-29. Data summary table for total palmitic acid in SRM 1953 Organic Contaminants in Non-Fortified Human Milk and Human Serum E.

		Total Palmitic Acid (C16:0)									
		SRM 1953 Organic Contaminants in Non-Fortified Human Milk (µmol/L)					Human Serum E (µmol/L)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target										
	B014	7539	7672	7699	7637	86	1319	1270	1294	1294	25
	B015										
	B016	5930	5469	6058	5819	310	1420	1414	1384	1406	19
	B017										
	B031										
	B034										
	B041						5230.81	5410.58	5707.23	5450	241
	B045	5180	5040	5120	5113	70	4964.17	5021.12	5112.27	5033	75
	B047	3419	3477	2944	3280	292	2296	2270	2266	2277	16
	B053						4778	4687	5200	4888	274
	B055										
	B058						4259	3963	3789	4004	238
Community Results		Consensus Mean			5462		Consensus Mean			3479	
		Consensus Standard Deviation			3597		Consensus Standard Deviation			1959	
		Maximum			7637		Maximum			5450	
		Minimum			3280		Minimum			1294	
		N			4		N			7	

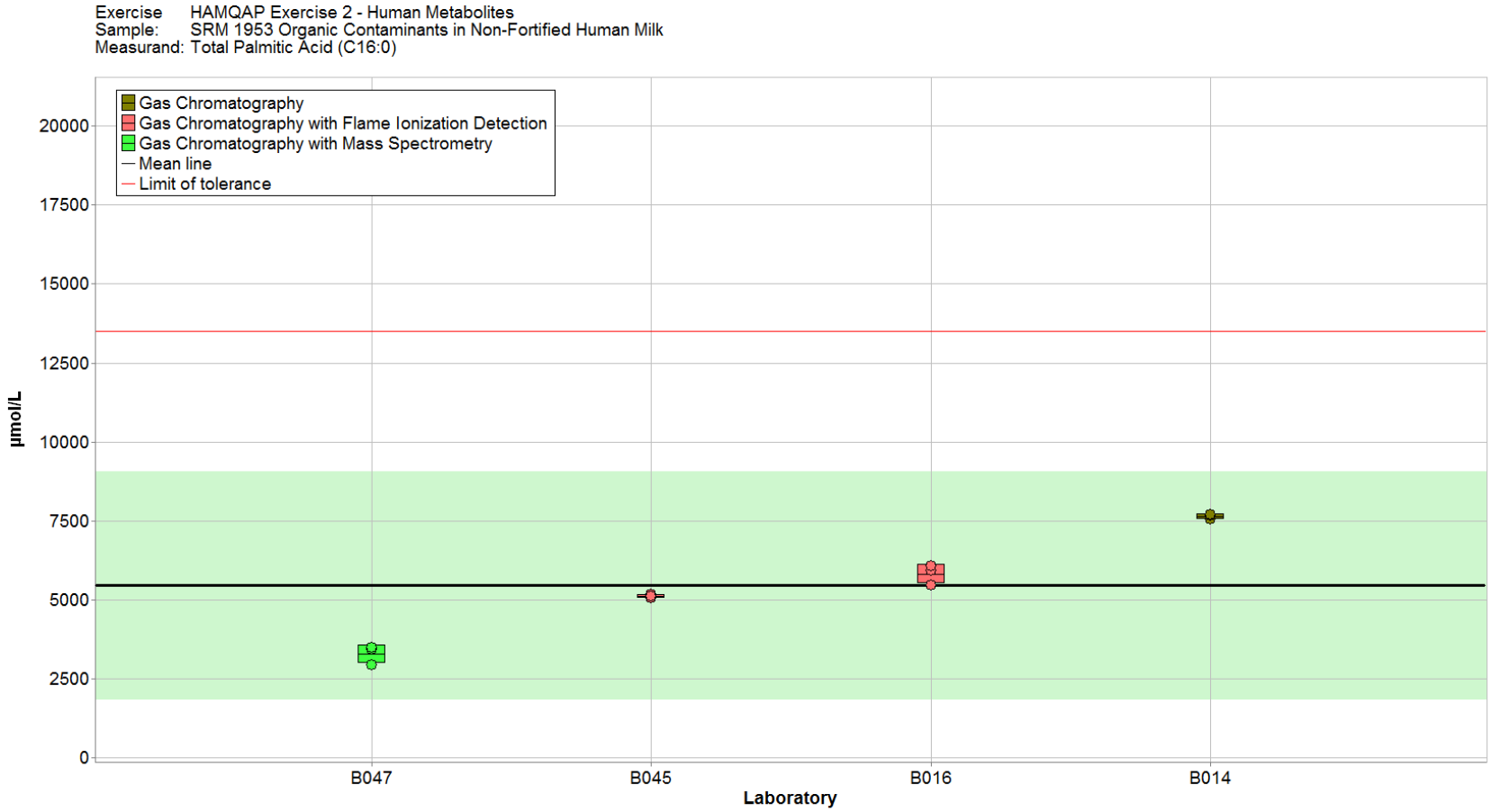

Figure 5-80. Total palmitic acid (C16:0) in SRM 1953 Organic Contaminants in Non-Fortified Human Milk (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

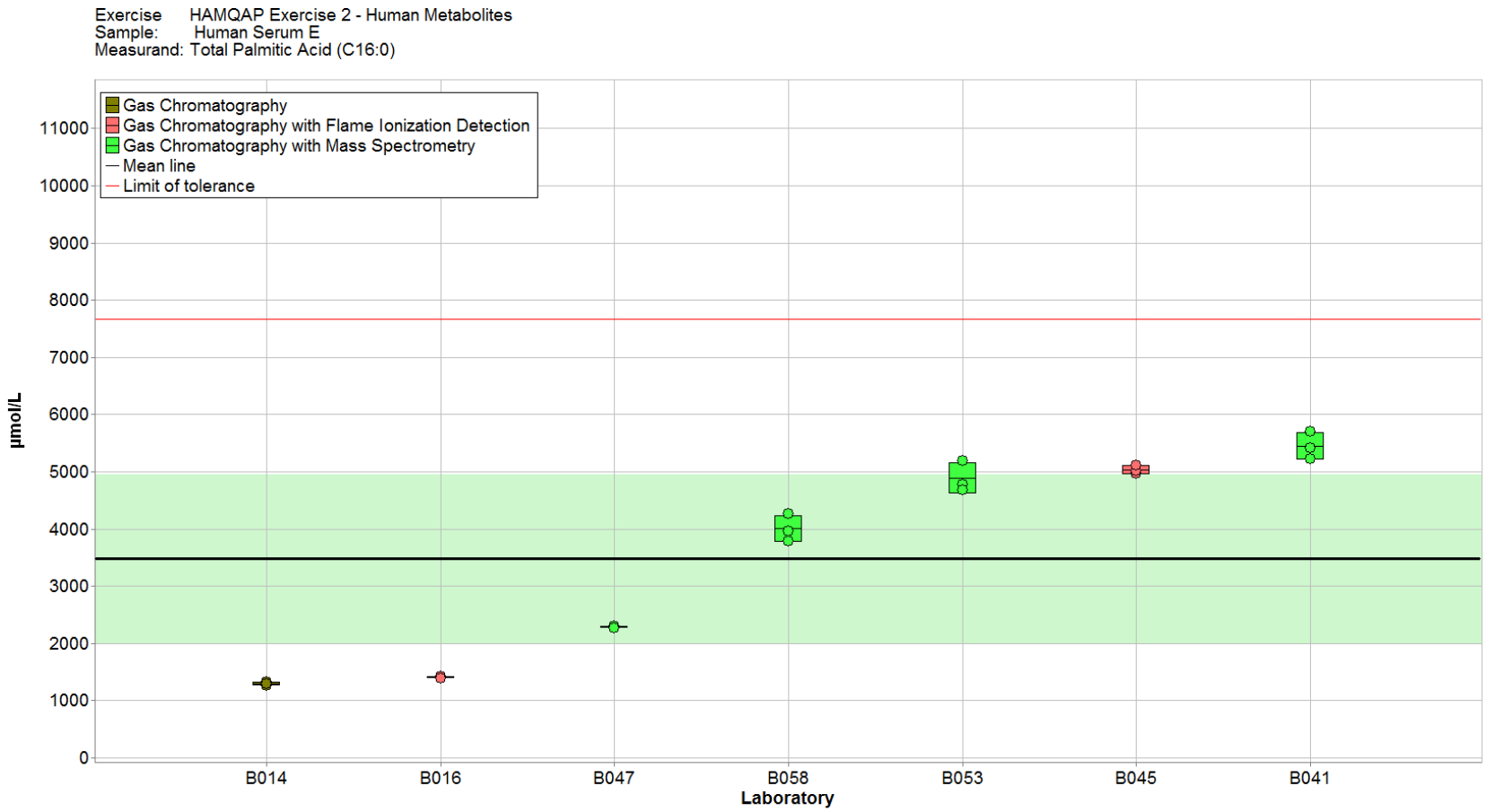

Figure 5-81. Total palmitic acid (C16:0) in Human Serum E (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

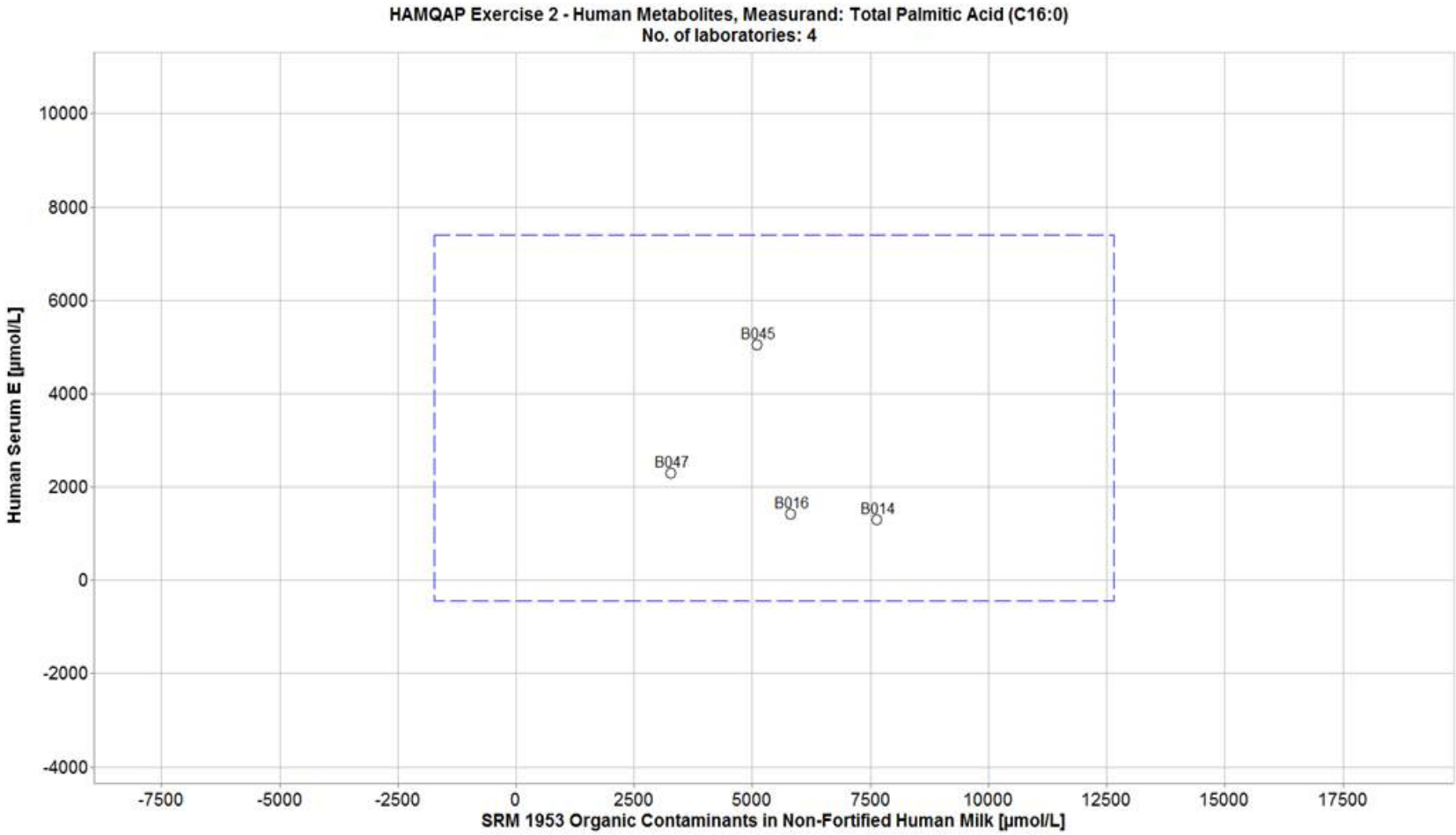

Figure 5-82. Laboratory means for total palmitic acid in SRM 1953 Organic Contaminants in Non-Fortified Human Milk and Human Serum E (sample/sample comparison view). In this view, the individual laboratory mean for one sample (SRM 1953) is compared to the mean for a second sample (Human Serum E). The dotted blue box represents the consensus range of tolerance for SRM 1953 (x-axis) and Human Serum E (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 5-30. Data summary table for total palmitoleic acid in SRM 1953 Organic Contaminants in Non-Fortified Human Milk and Human Serum E. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		Total Palmitoleic Acid (C16:1 n-7)									
		SRM 1953 Organic Contaminants in Non-Fortified Human Milk (µmol/L)					Human Serum E (µmol/L)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target										
	B014	580	592	597	589.7	8.7	< 200	< 200	< 200		
	B015										
	B016	575	525.7	586.6	562	32	120.5	118.1	114.4	117.7	3.1
	B017										
	B031										
	B034										
	B041						452.04	450.62	478.91	461	16
	B045	582	563	578	574	10	425.04	422.12	420.73	422.6	2.2
	B047						161	151	149	153.7	6.4
	B053						310	319	430	353	67
	B055	1931.5	2103.7	2266.8	2101	168	410.4	451.6	465	442	28
	B058						280.1	223	296	266	38
Community Results		Consensus Mean			575		Consensus Mean			318	
		Consensus Standard Deviation			33		Consensus Standard Deviation			128	
		Maximum			2101		Maximum			461	
		Minimum			562		Minimum			117.7	
		N			4		N			7	

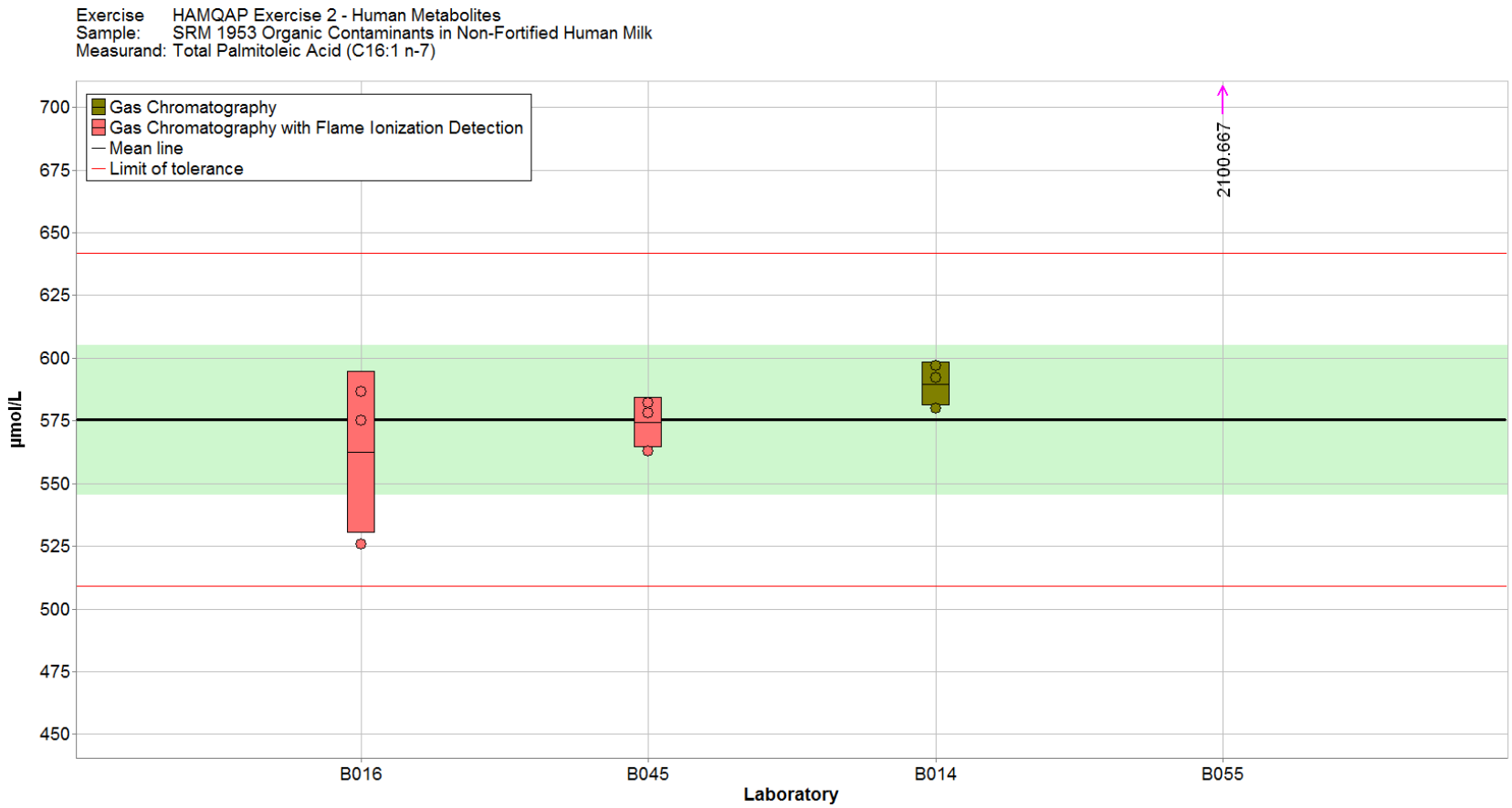

Figure 5-83. Total palmitoleic acid (C16:1 n-7) in SRM 1953 Organic Contaminants in Non-Fortified Human Milk (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

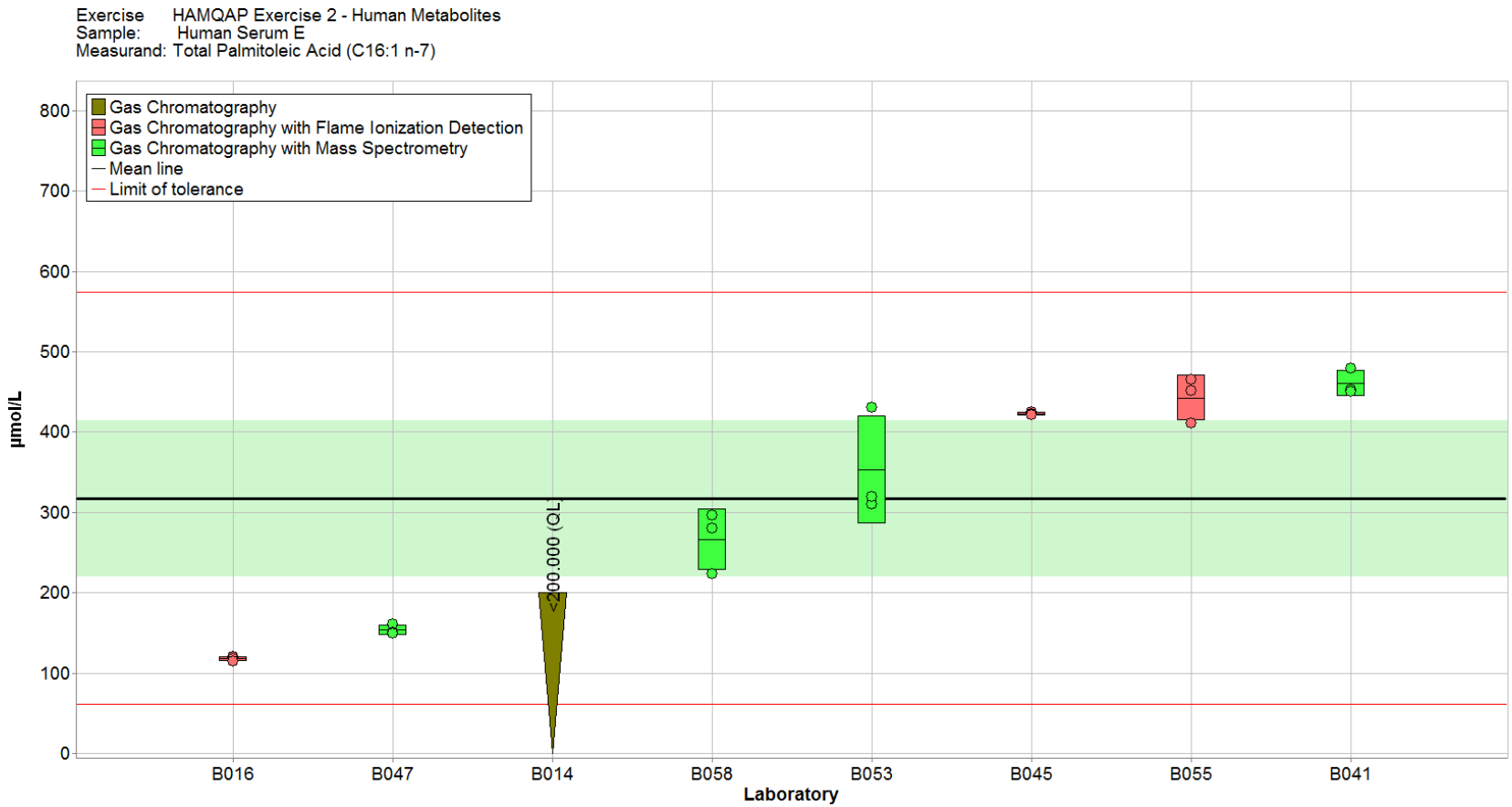

Figure 5-84. Total palmitoleic acid (C16:1 n-7) in Human Serum E (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

Table 5-31. Data summary table for total stearic acid in SRM 1953 Organic Contaminants in Non-Fortified Human Milk and Human Serum E. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		Total Stearic Acid (C18:0)									
		SRM 1953 Organic Contaminants in Non-Fortified Human Milk (µmol/L)					Human Serum E (µmol/L)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target										
	B014	2538	2579	2586	2568	26	480	462	470	470.7	9.0
	B015	1385.91	1316.76	1311.45	1338	42	2906.3	2753.7	2665.5	2775	122
	B016	1925	1781	1969	1892	98	515.2	513.4	500.4	509.7	8.1
	B017										
	B031										
	B034										
	B041						1862.44	1929.84	2030.6	1941	85
	B045	1528.35	1545.13	1538.28	1537.3	8.4	1855.63	1873.81	1888.21	1873	16
	B047	878	888	886	884.0	5.3	784	783	795	787.3	6.7
	B053						1576	1526	1594	1565	35
	B055	6247.6	6873.3	7378.5	6833	567	1746.7	1752.7	1799.4	1766	29
	B058						2350	2010	2122	2161	173
Community Results		Consensus Mean			1866		Consensus Mean			1515	
		Consensus Standard Deviation			1351		Consensus Standard Deviation			698	
		Maximum			6833		Maximum			2775	
		Minimum			884.0		Minimum			470.7	
		N			6		N			9	

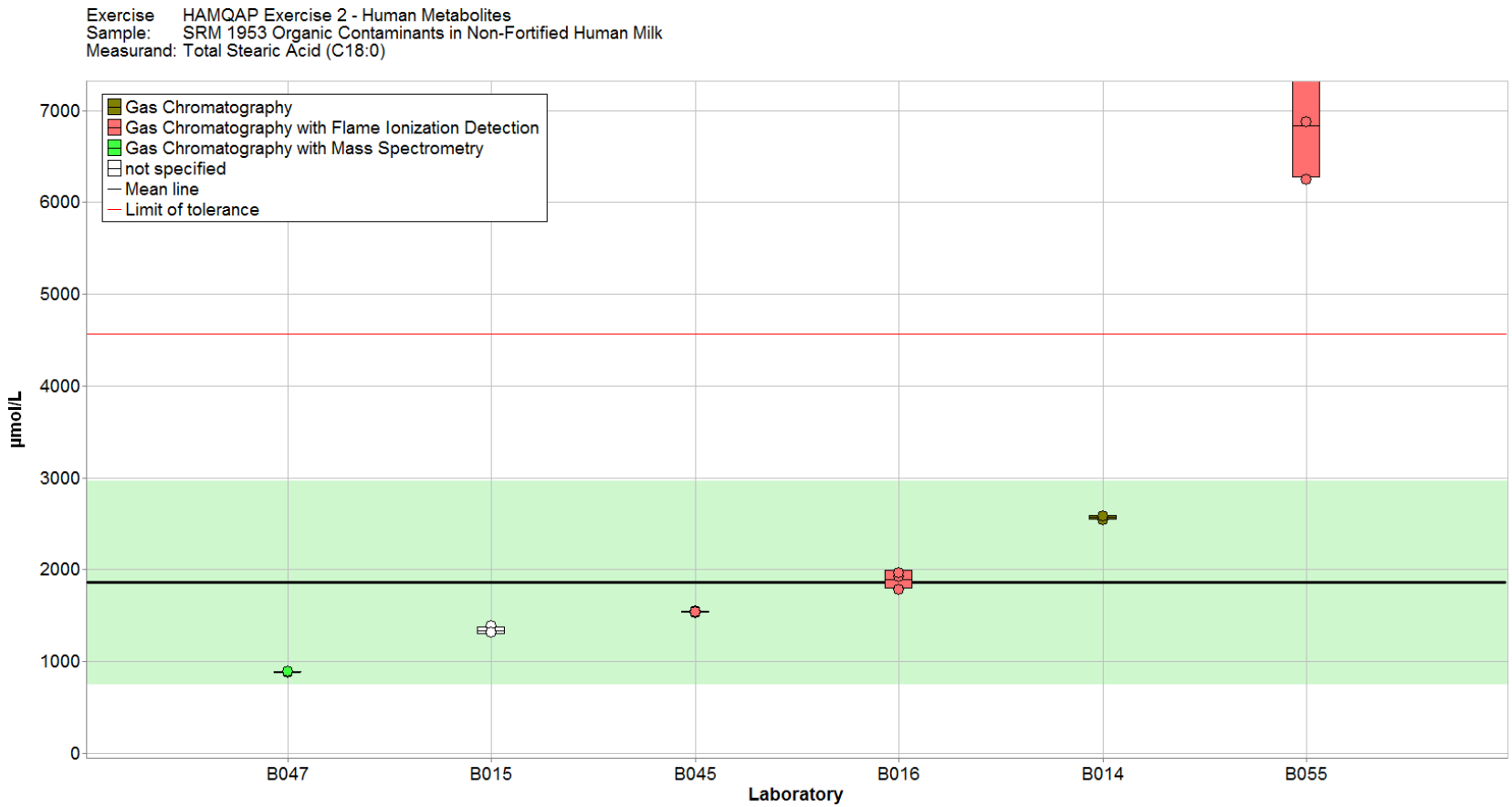

Figure 5-85. Total stearic acid (C18:0) in SRM 1953 Organic Contaminants in Non-Fortified Human Milk (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

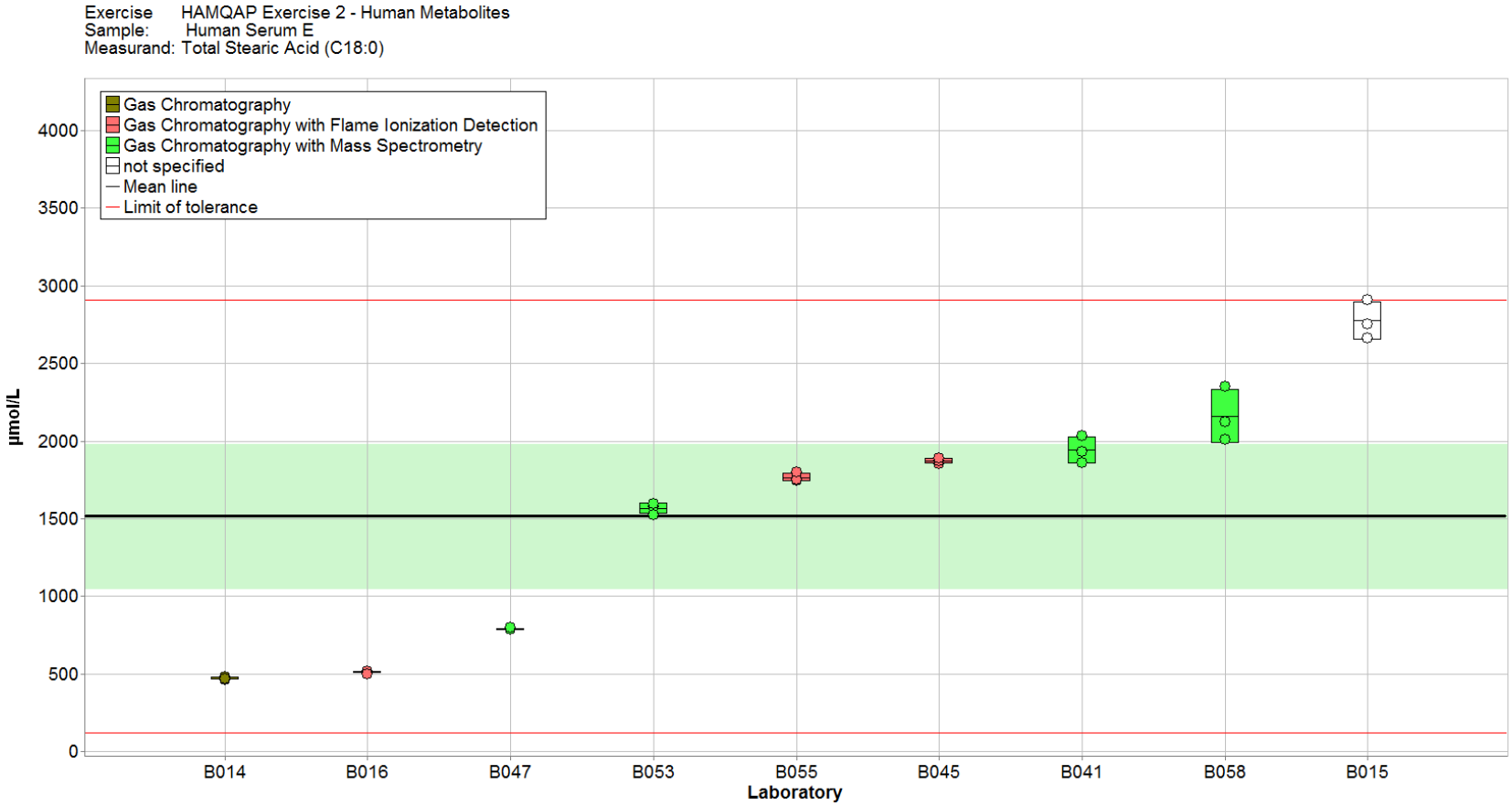

Figure 5-86. Total stearic acid (C18:0) in Human Serum E (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

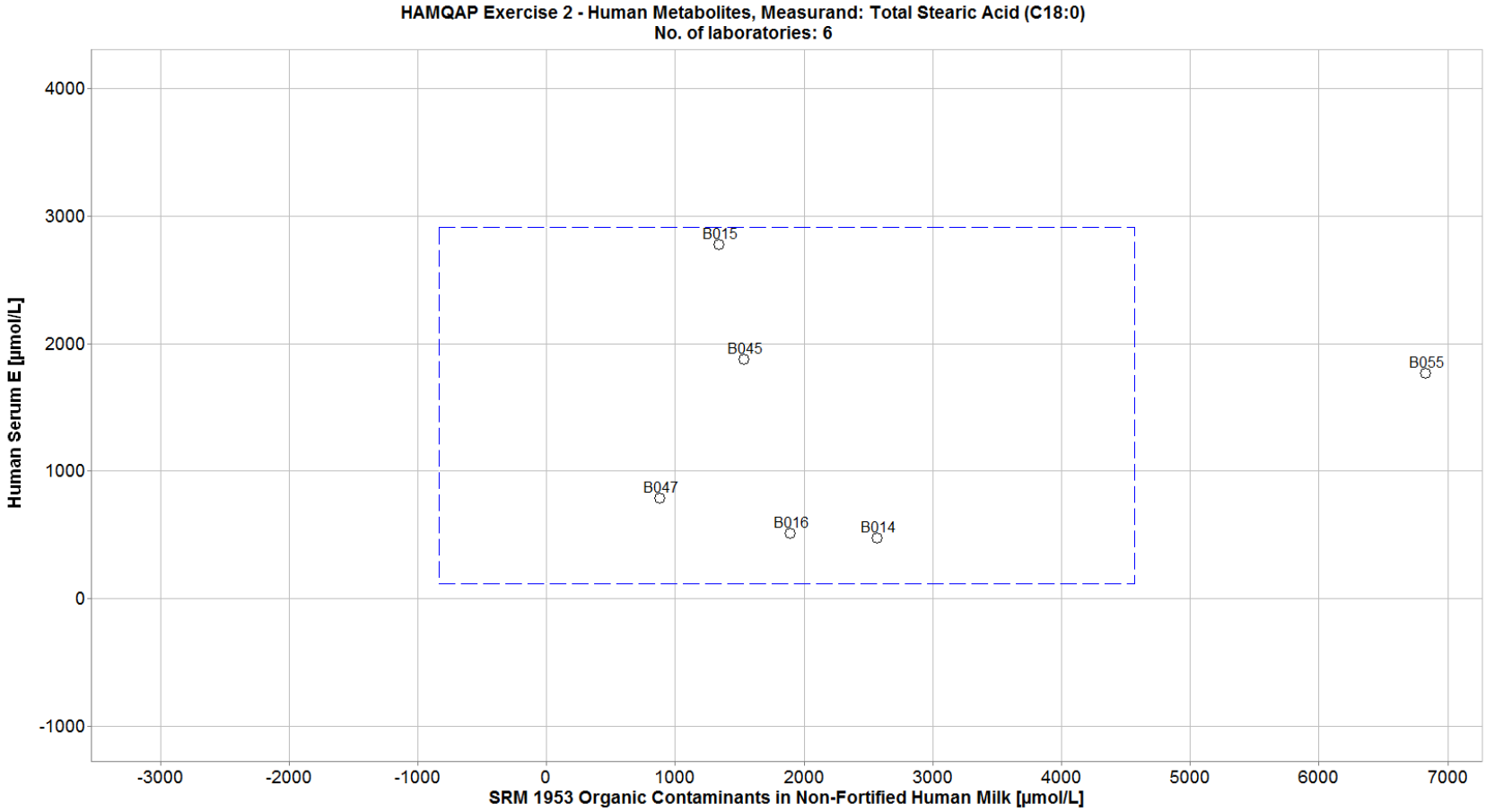

Figure 5-87. Laboratory means for total stearic acid in SRM 1953 Organic Contaminants in Non-Fortified Human Milk and Human Serum E (sample/sample comparison view). In this view, the individual laboratory mean for one sample (SRM 1953) is compared to the mean for a second sample (Human Serum E). The dotted blue box represents the consensus range of tolerance for SRM 1953 (x-axis) and Human Serum E (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 5-32. Data summary table for total *cis*-vaccenic acid in SRM 1953 Organic Contaminants in Non-Fortified Human Milk and Human Serum E.

		Total cis-Vaccenic Acid (C18:1 n-7)									
		SRM 1953 Organic Contaminants in Non-Fortified Human Milk (µmol/L)					Human Serum E (µmol/L)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target										
	B014	561	575	576	570.7	8.4	< 100	< 100	< 100		
	B016	484.4	440.4	479	468	24	87.97	89.39	100.5	92.6	6.9
	B017										
	B031										
	B034										
	B041						307.89	310.39	326.62	315	10
	B047	172	196		184	17	107	102	104	104.3	2.5
	B053						204	213	245	221	22
	B055										
Community Results		Consensus Mean			408		Consensus Mean			183	
		Consensus Standard Deviation			275		Consensus Standard Deviation			215	
		Maximum			570.7		Maximum			315	
		Minimum			184		Minimum			92.6	
		N			3		N			4	

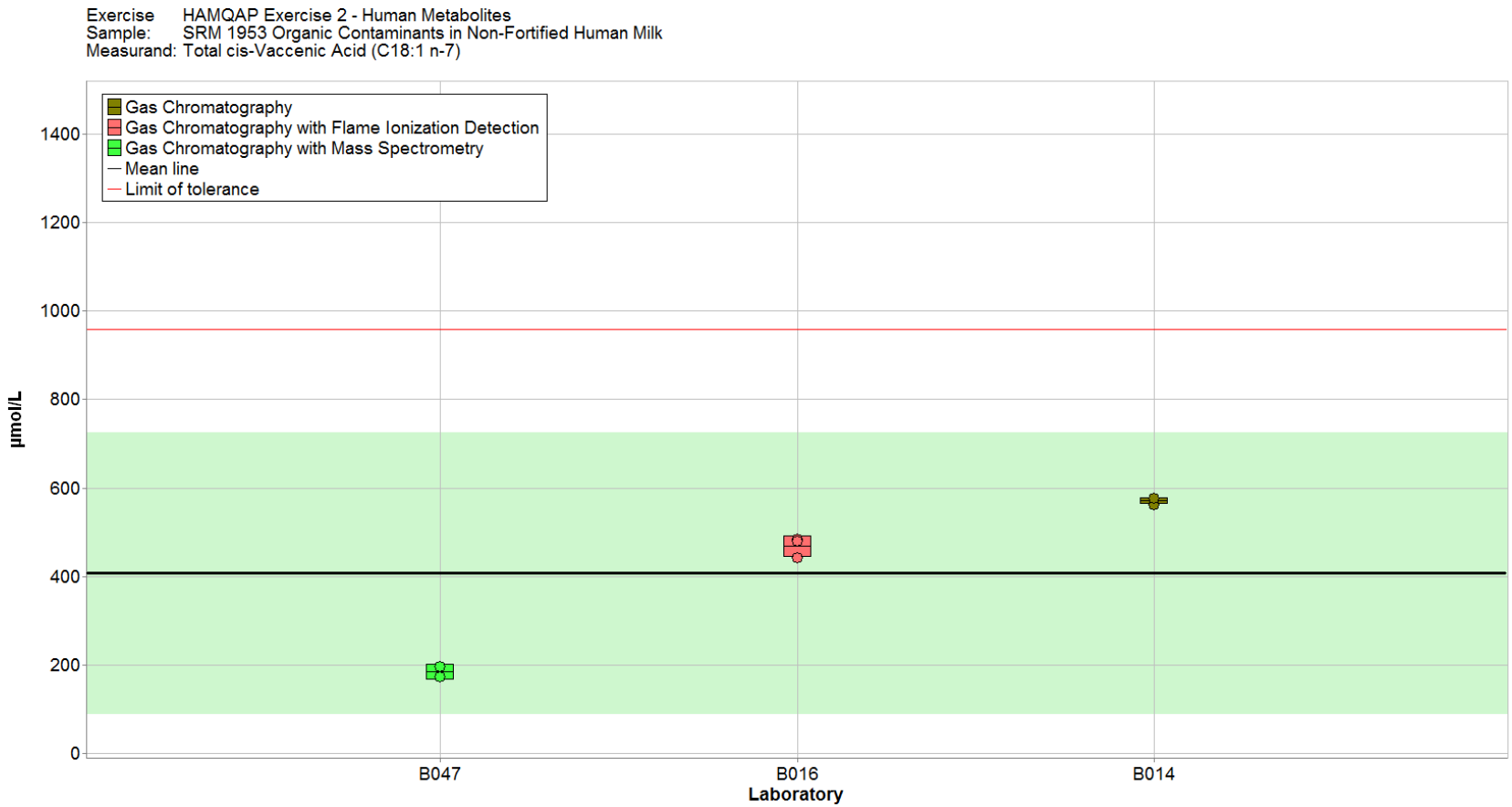

Figure 5-88. Total *cis*-vaccenic acid (C18:1 n-7) in SRM 1953 Organic Contaminants in Non-Fortified Human Milk (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

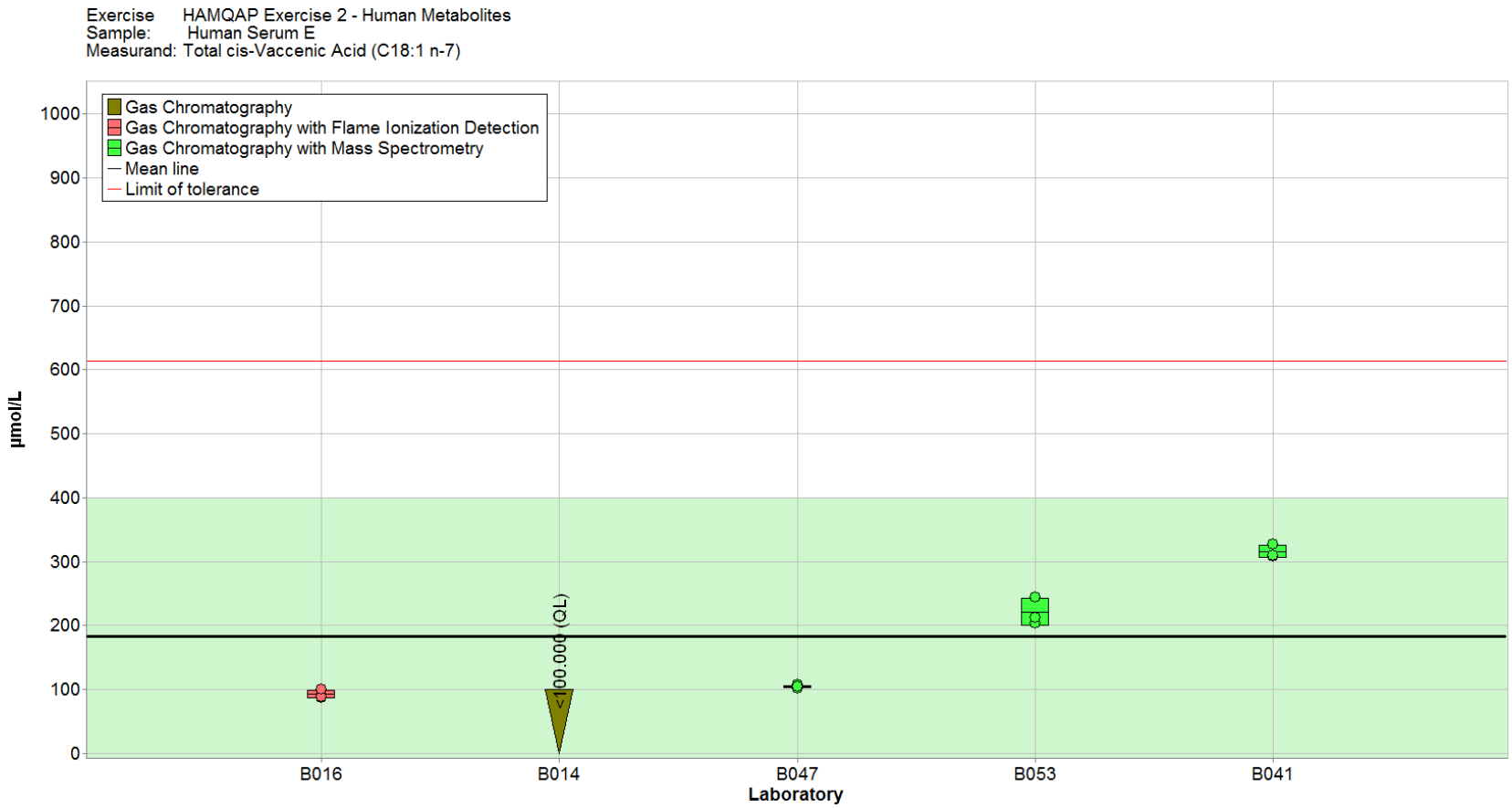

Figure 5-89. Total *cis*-vaccenic acid (C18:1 n-7) in Human Serum E (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

Table 5-33. Data summary table for total oleic acid in SRM 1953 Organic Contaminants in Non-Fortified Human Milk and Human Serum E. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		Total Oleic Acid (C18:1 n-9)									
		SRM 1953 Organic Contaminants in Non-Fortified Human Milk (µmol/L)					Human Serum E (µmol/L)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target										
	B014	8883	9075	9096	9018	117	855	819	837	837	18
	B015	8874.61	8321.88	8214.75	8470	354	5755.2	5576.3	5397.6	5576	179
	B016	9315	8489	9426	9077	512	1008	1004	1049	1020	25
	B017										
	B031										
	B034										
	B041						3378.94	3433.36	3635.84	3483	135
	B045	9120	9163	9124	9136	24	3098.09	3114.52	3138.21	3117	20
	B047						1247	1197	1179	1208	35
	B053						2488	2532	3273	2764	441
	B055	26896	30401	32537	29945	2848	3237.6	3251.4	3322.6	3271	46
	B058						2014	1980	2140	2045	84
Community Results		Consensus Mean			8925		Consensus Mean			2540	
		Consensus Standard Deviation			640		Consensus Standard Deviation			1720	
		Maximum			29945		Maximum			5576	
		Minimum			8470		Minimum			837	
		N			5		N			9	

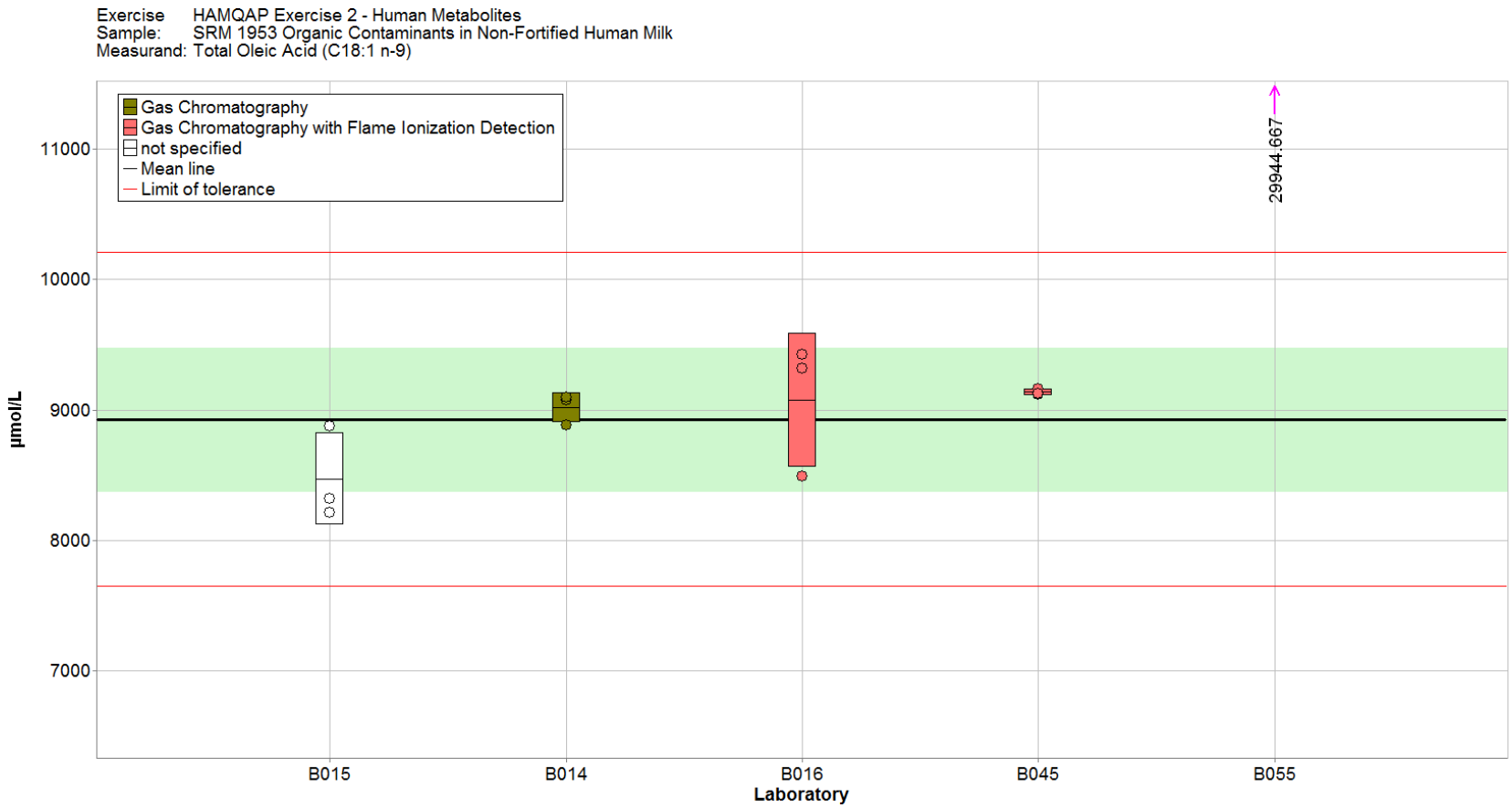

Figure 5-90. Total oleic acid (C18:1 n-9) in SRM 1953 Organic Contaminants in Non-Fortified Human Milk (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

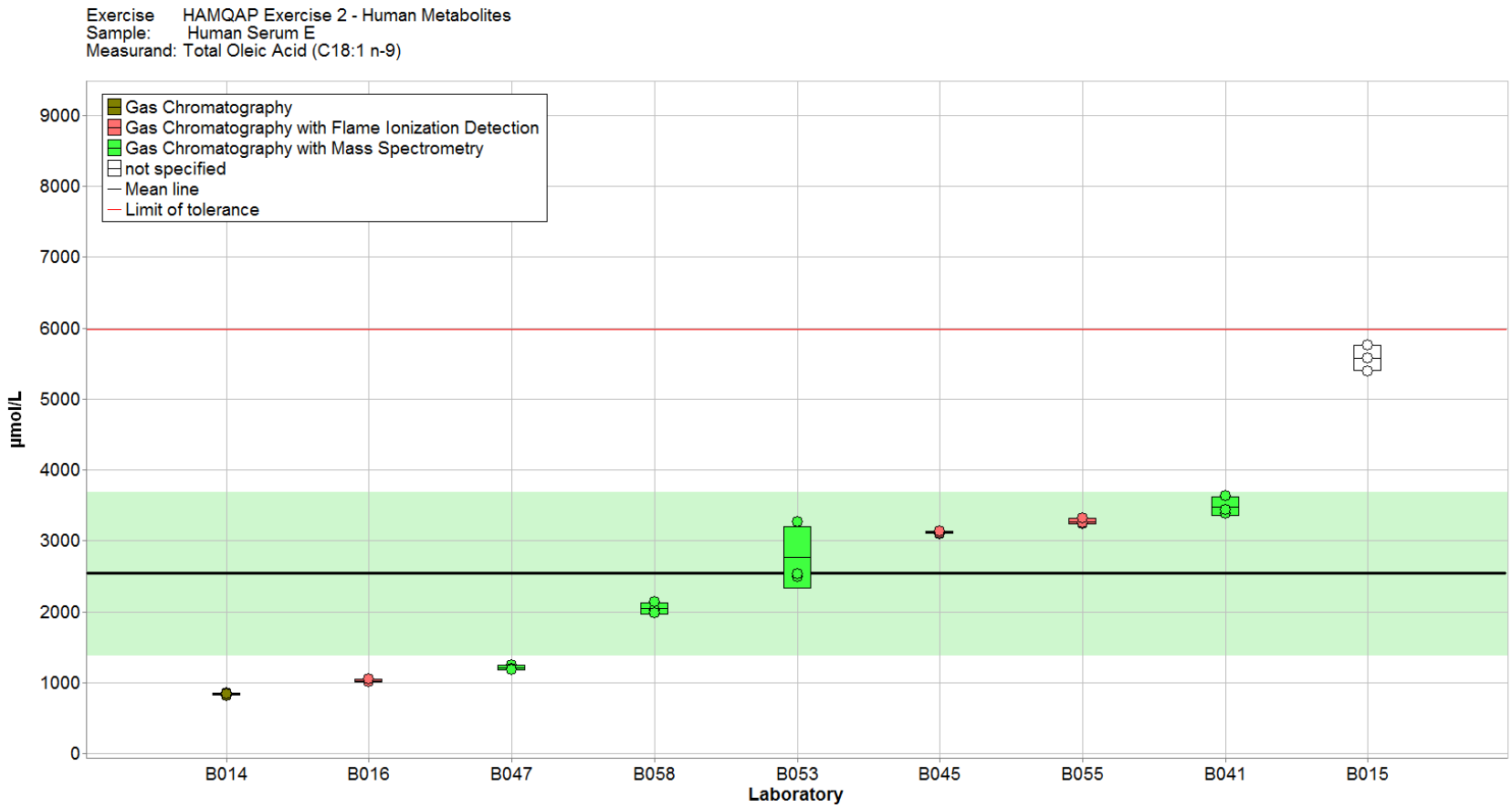

Figure 5-91. Total oleic acid (C18:1 n-9) in Human Serum E (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

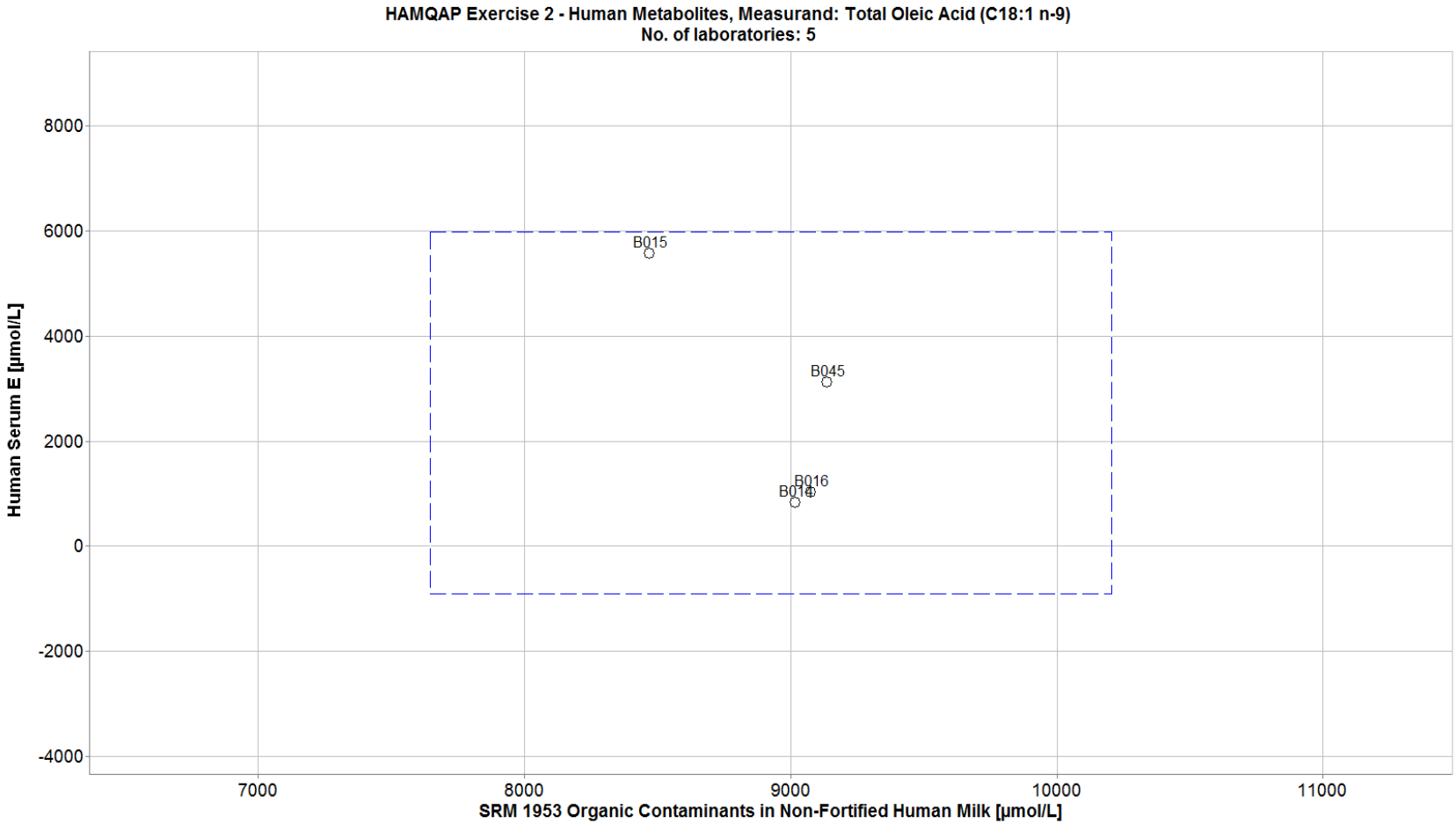

Figure 5-92. Laboratory means for total oleic acid (C18:1 n-9) in SRM 1953 Organic Contaminants in Non-Fortified Human Milk and Human Serum E (sample/sample comparison view). In this view, the individual laboratory mean for one sample (SRM 1953) is compared to the mean for a second sample (Human Serum E). The dotted blue box represents the consensus range of tolerance for SRM 1953 (x-axis) and Human Serum E (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 5-34. Data summary table for total *trans*-vaccenic acid in SRM 1953 Organic Contaminants in Non-Fortified Human Milk and Human Serum E.

		Total Transvaccenic Acid (C18:1 n-7t)									
		SRM 1953 Organic Contaminants in Non-Fortified Human Milk (µmol/L)					Human Serum E (µmol/L)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
Individual Results	Target										
	B014	321	330	327	326.0	4.6	< 110	< 110	< 110		
	B016										
	B017										
	B031										
	B034										
	B041						75.36	74.96	79.58	76.6	2.6
	B053										
	B055										
Community Results		Consensus Mean					Consensus Mean				
		Consensus Standard Deviation					Consensus Standard Deviation				
		Maximum			326		Maximum			76.6	
		Minimum			326		Minimum			76.6	
		N			1		N			1	

Table 5-35. Data summary table for total linoleic acid in SRM 1953 Organic Contaminants in Non-Fortified Human Milk and Human Serum E. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		Total Linoleic Acid (C18:2 n-6)									
		SRM 1953 Organic Contaminants in Non-Fortified Human Milk (µmol/L)					Human Serum E (µmol/L)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target										
	B014	4420	4525	4556	4500	71	1067	974	1032	1024	47
	B015	45549	42210.5	41687.3	43149	2095	6428	5966.9	5164.1	5853	640
	B016	4414	4042	4513	4323	248	1039	1186	1023	1083	90
	B017										
	B031										
	B034										
	B041						4414.42	4225.72	4556.95	4399	166
	B045	4028	4073	4035	4045	24	4130.76	4112.28	4138.73	4127	14
	B047						1375	1447	1551	1458	88
	B053						2875	2983	3639	3166	413
	B055	15218	17122	18313	16884	1561	4334.5	4329.9	4127.7	4264	118
	B058						3150	2896	2987	3011	129
Community Results		Consensus Mean			4290		Consensus Mean			3154	
		Consensus Standard Deviation			1087		Consensus Standard Deviation			2235	
		Maximum			43149		Maximum			5853	
		Minimum			4045		Minimum			1024	
		N			5		N			9	

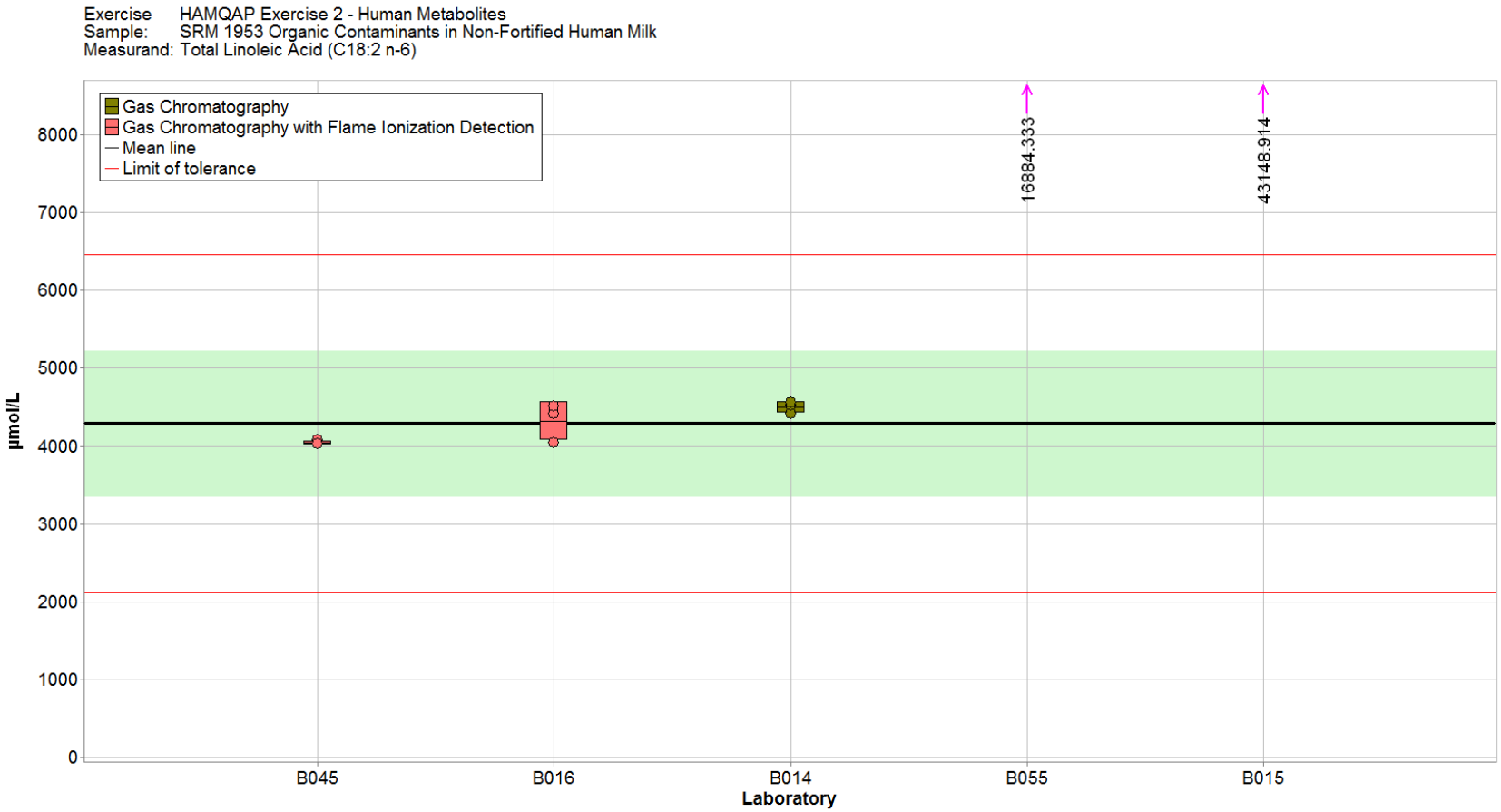

Figure 5-93. Total linoleic acid (C18:2 n-6) in SRM 1953 Organic Contaminants in Non-Fortified Human Milk (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

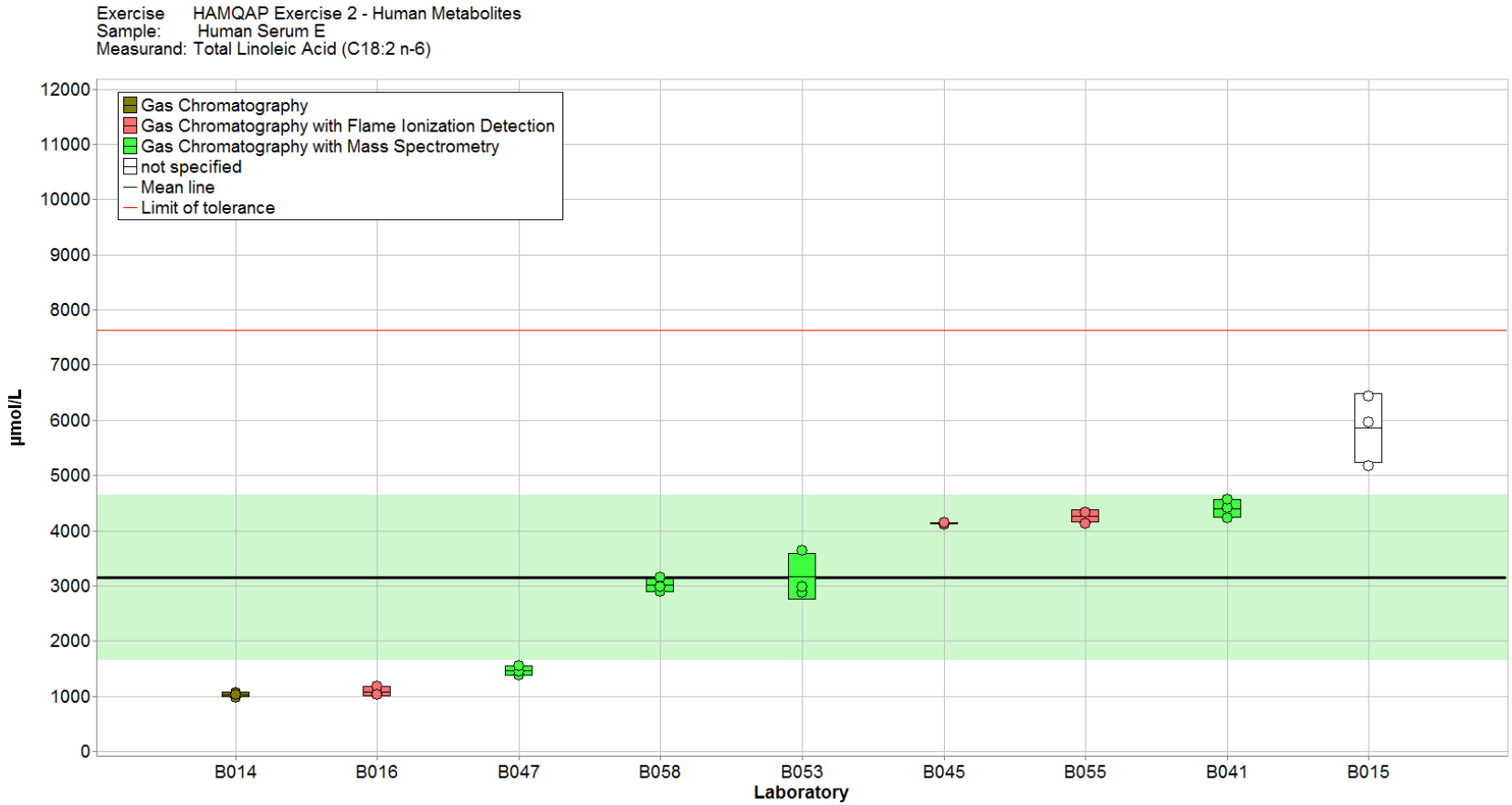

Figure 5-94. Total linoleic acid (C18:2 n-6) in Human Serum E (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

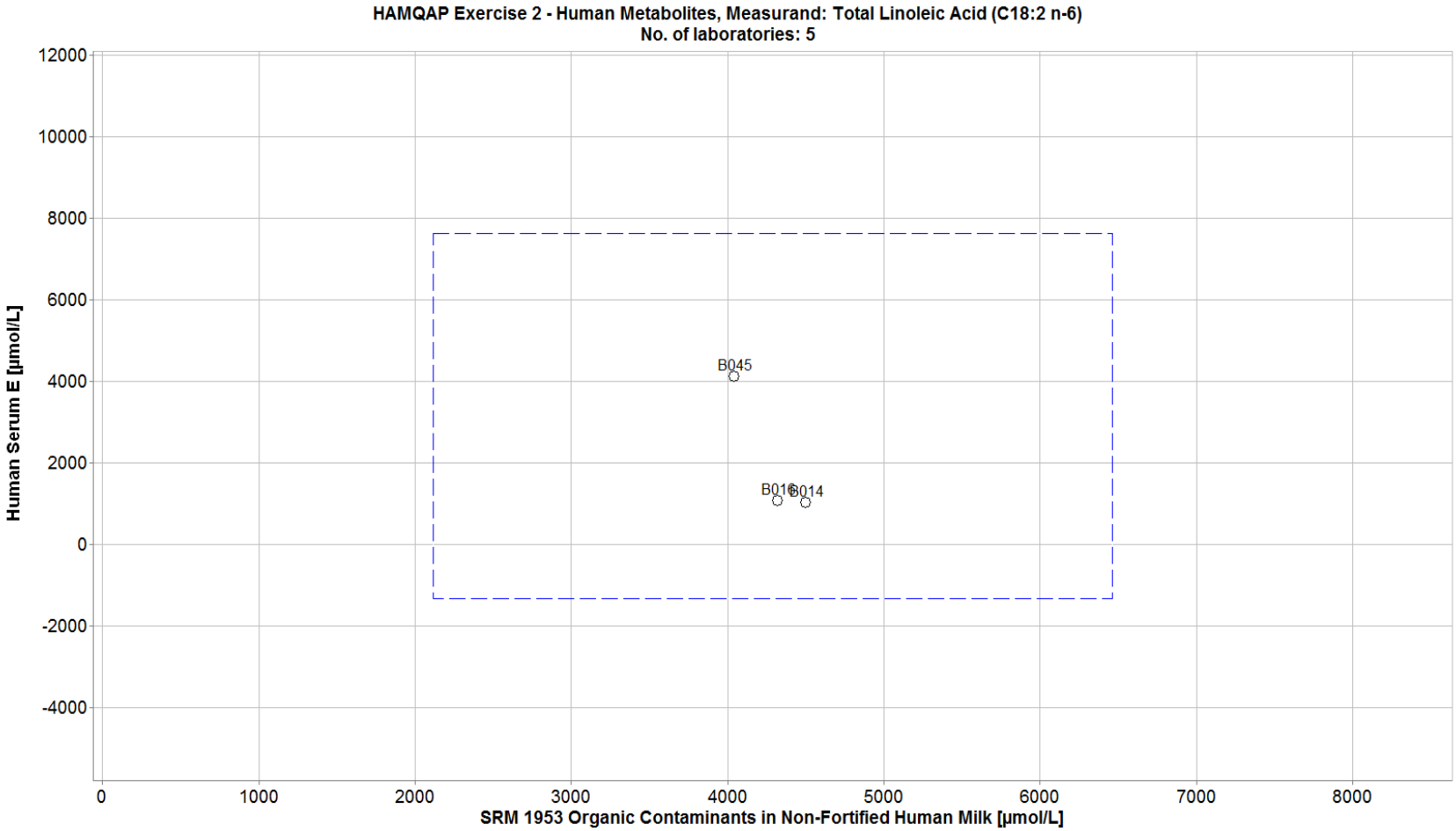

Figure 5-95. Laboratory means for total linoleic acid (C18:2 n-6) in SRM 1953 Organic Contaminants in Non-Fortified Human Milk and Human Serum E (sample/sample comparison view). In this view, the individual laboratory mean for one sample (SRM 1953) is compared to the mean for a second sample (Human Serum E). The dotted blue box represents the consensus range of tolerance for SRM 1953 (x-axis) and Human Serum E (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 5-36. Data summary table for total elaidic acid in SRM 1953 Organic Contaminants in Non-Fortified Human Milk and Human Serum E.

		Total Elaidic Acid (C18:1 n-9t)									
		SRM 1953 Organic Contaminants in Non-Fortified Human Milk (µmol/L)					Human Serum E (µmol/L)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target										
	B014	242	240	235	239.0	3.6	< 110	< 110	< 110		
	B016	294.7	286.7	373.6	318	48	27.26	28.32	29.74	28.4	1.2
	B017										
	B031										
	B034										
	B041						58.72	58.45	61.36	59.5	1.6
	B053										
	B055										
Community Results		Consensus Mean			279		Consensus Mean			44	
		Consensus Standard Deviation			112		Consensus Standard Deviation			66	
		Maximum			318		Maximum			59.5	
		Minimum			239.0		Minimum			28.4	
		N			2		N			2	

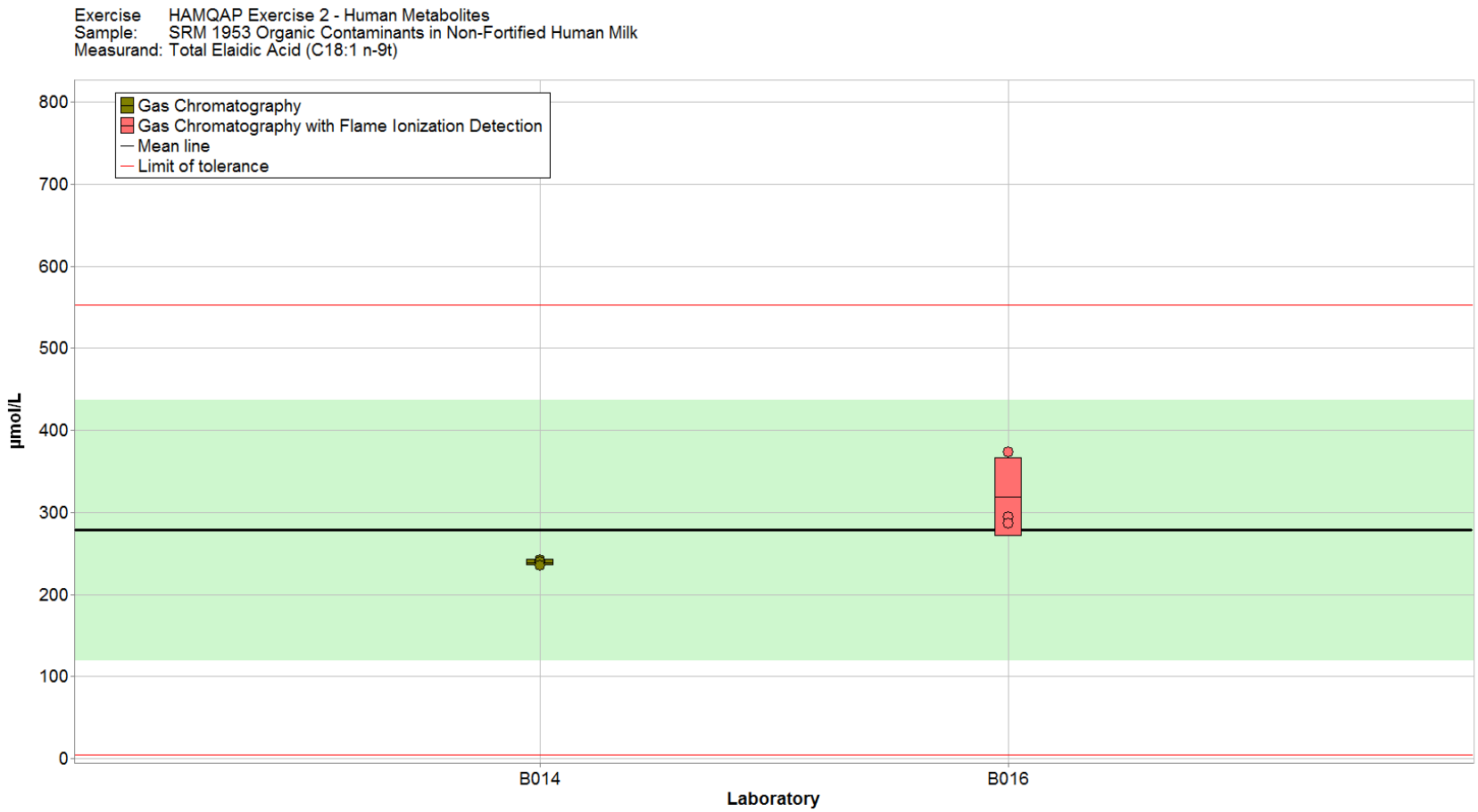

Figure 5-96. Total elaidic acid (C18:1 n-9t) in SRM 1953 Organic Contaminants in Non-Fortified Human Milk (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$. A NIST value has not been determined in this material.

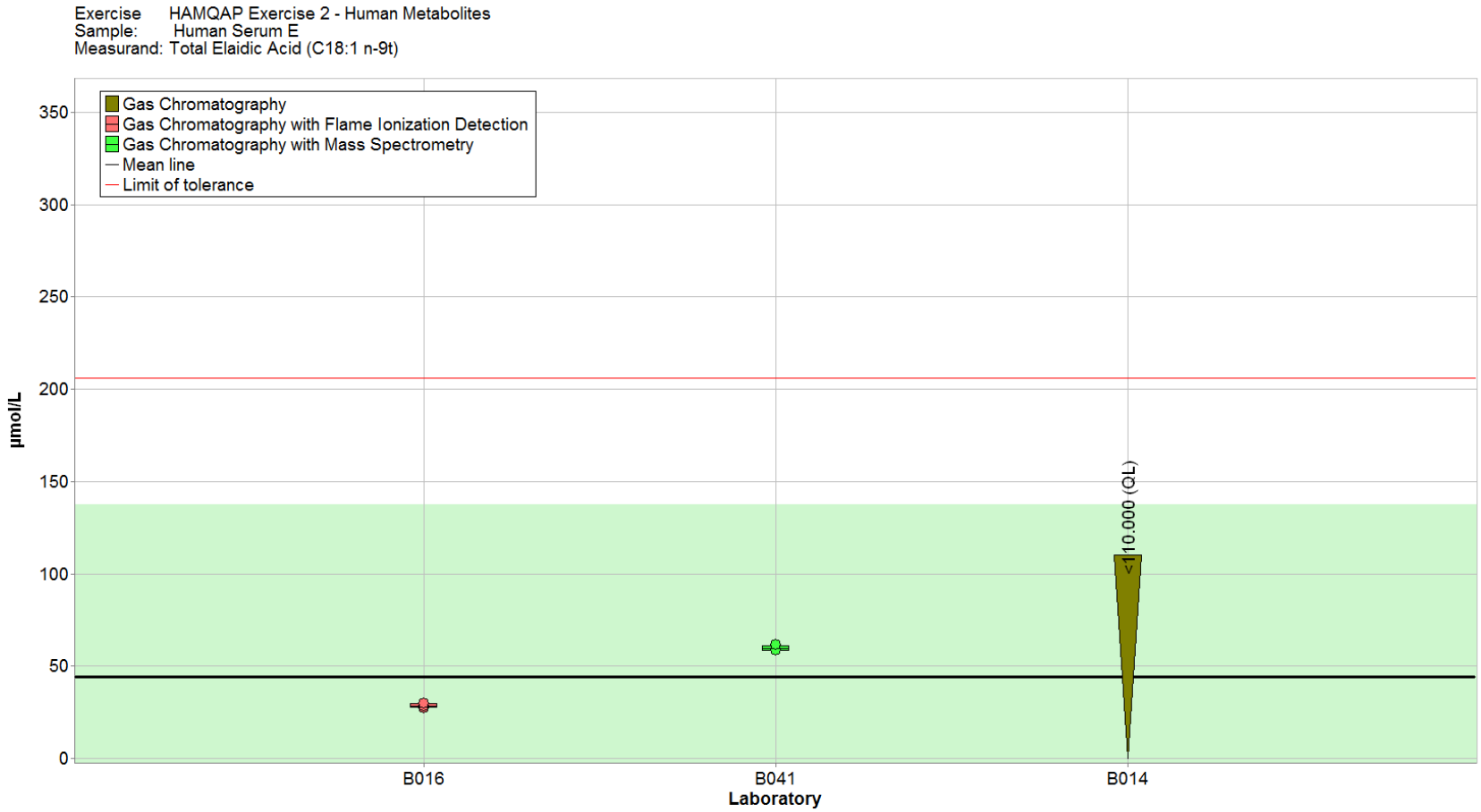

Figure 5-97. Total elaidic acid (C18:1 n-9t) in Human Serum E (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

Table 5-36. Data summary table for total α-linolenic acid in SRM 1953 Organic Contaminants in Non-Fortified Human Milk and Human Serum E. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		Total alpha-Linolenic Acid (C18:3 n-3)									
		SRM 1953 Organic Contaminants in Non-Fortified Human Milk (µmol/L)					Human Serum E (µmol/L)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target										
	B014	310	313	324	315.7	7.4	< 110	< 110	< 110		
	B015	21241	19649.9	19443.6	20112	984	76.5	70	61.4	69.3	7.6
	B016	354.4	324.6	362.7	347	20	8.08	10.23	9.34	9.2	1.1
	B017										
	B031										
	B034										
	B041						33.83	30.6	33.62	32.7	1.8
	B045	374	382	376	377.3	4.2	33.61	32.23	33.15	33.00	0.70
	B047						12	13	14	13.0	1.0
	B053										
	B055	1304.5	1469.3	1569.9	1448	134	38.4	38.4	34.5	37.1	2.3
	B058						91.4	86.5	89.6	89.2	2.5
Community Results		Consensus Mean			347		Consensus Mean			40	
		Consensus Standard Deviation			140		Consensus Standard Deviation			44	
		Maximum			20112		Maximum			89.2	
		Minimum			315.7		Minimum			9.2	
		N			5		N			7	

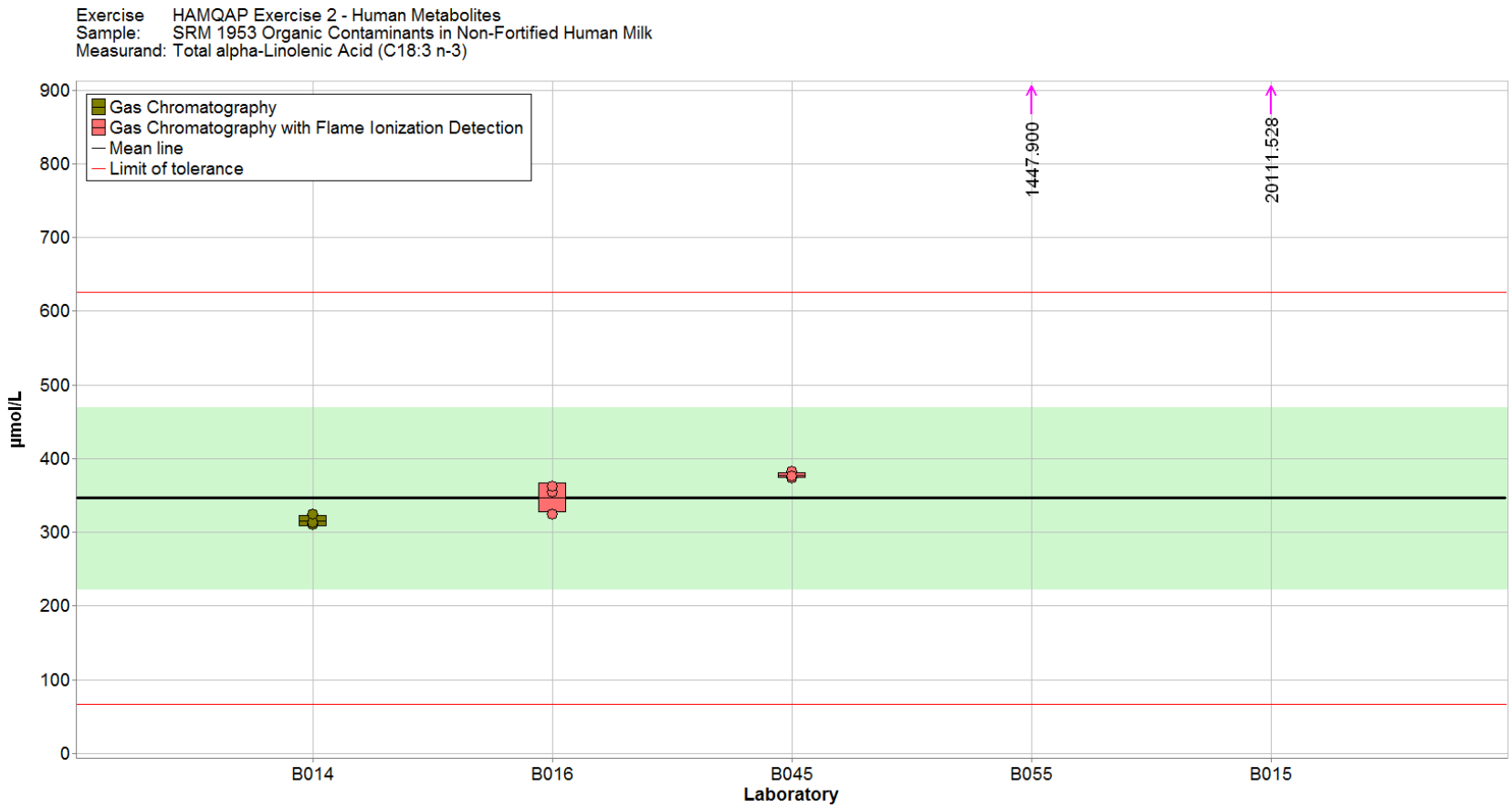

Figure 5-98. Total α-linolenic acid (C18:3 n-3) in SRM 1953 Organic Contaminants in Non-Fortified Human Milk (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

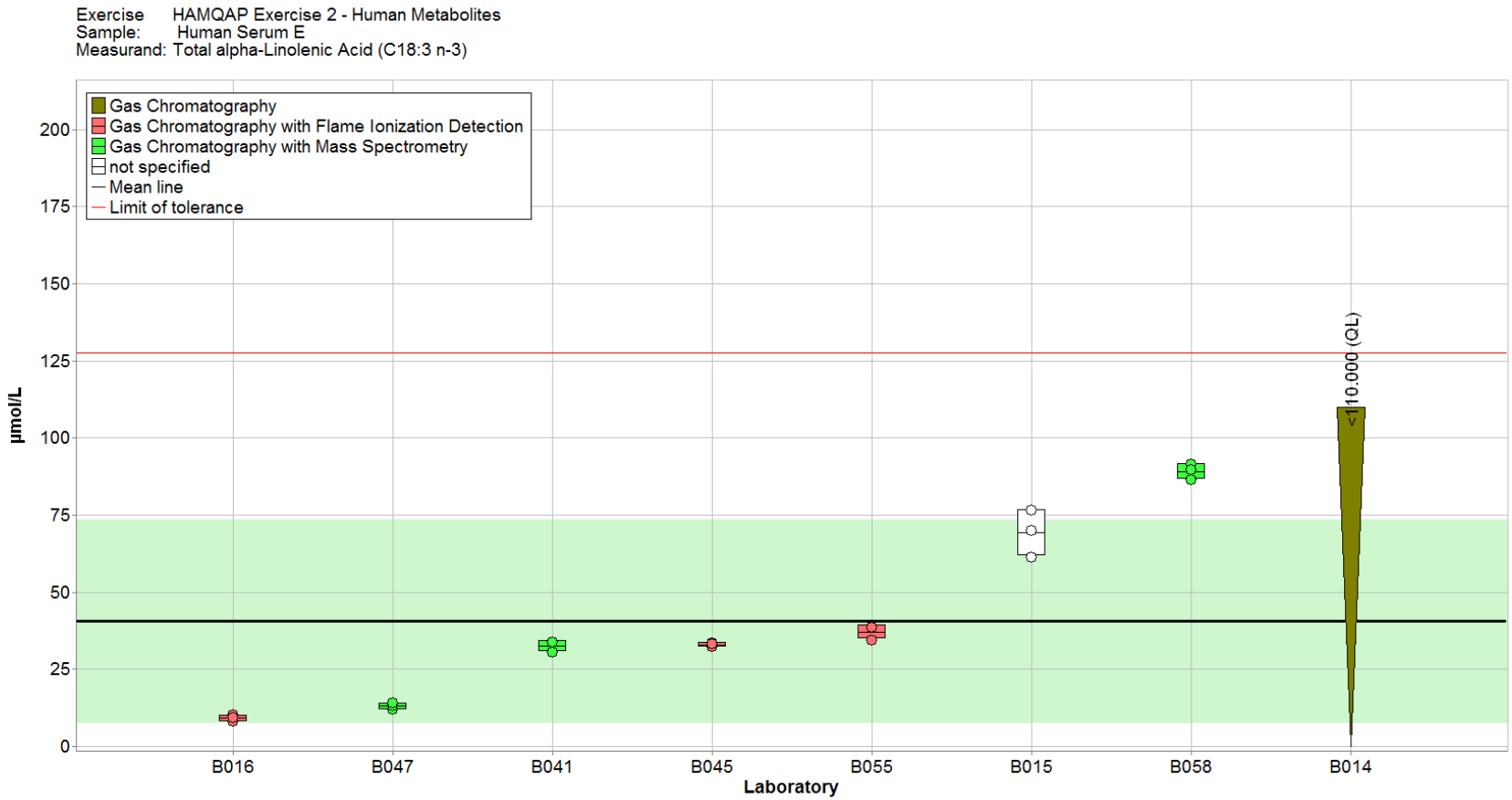

Figure 5-99. Total α-linolenic acid (C18:3 n-3) in Human Serum E (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

Table 5-38. Data summary table for total γ-linolenic acid in SRM 1953 Organic Contaminants in Non-Fortified Human Milk and Human Serum E.

		Total gamma-Linolenic Acid (C18:3 n-6)									
		SRM 1953 Organic Contaminants in Non-Fortified Human Milk (µmol/L)					Human Serum E (µmol/L)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target										
	B014	89.6	91.8	92.8	91.4	1.6	< 110	< 110	< 110		
	B016	32.14	29.51	32.68	31.4	1.7	6.82	8.62	7.9	7.78	0.91
	B017										
	B031										
	B034										
	B041						33.03	28.86	32.18	31.4	2.2
	B045	321	313	312	315.3	4.9	29.14	28.93	29.02	29.03	0.11
	B047	71	70	69	70.0	1.0	9	10	11	10.0	1.0
	B053						17.4	18.7	24.2	20.1	3.6
	B055	121.4	138.3	146.5	135.4	12.8	33.4	33.8	28.4	31.9	3.0
	B058						16.2	15.9	16.9	16.33	0.51
Community Results		Consensus Mean			121		Consensus Mean			21	
		Consensus Standard Deviation			103		Consensus Standard Deviation			11	
		Maximum			315.3		Maximum			31.9	
		Minimum			31.4		Minimum			7.78	
		N			5		N			7	

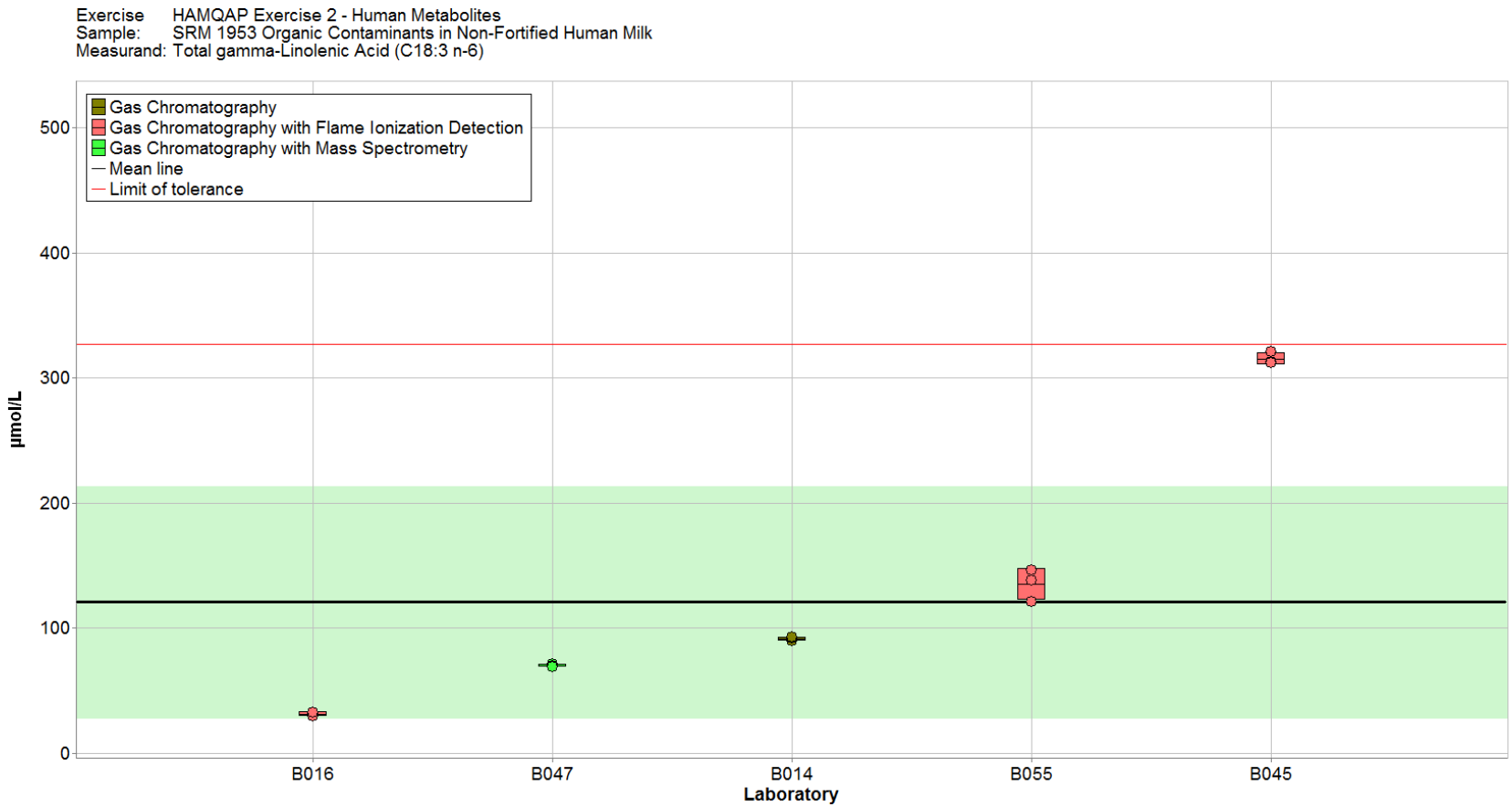

Figure 5-100. Total γ-linolenic acid (C18:3 n-6) in SRM 1953 Organic Contaminants in Non-Fortified Human Milk (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

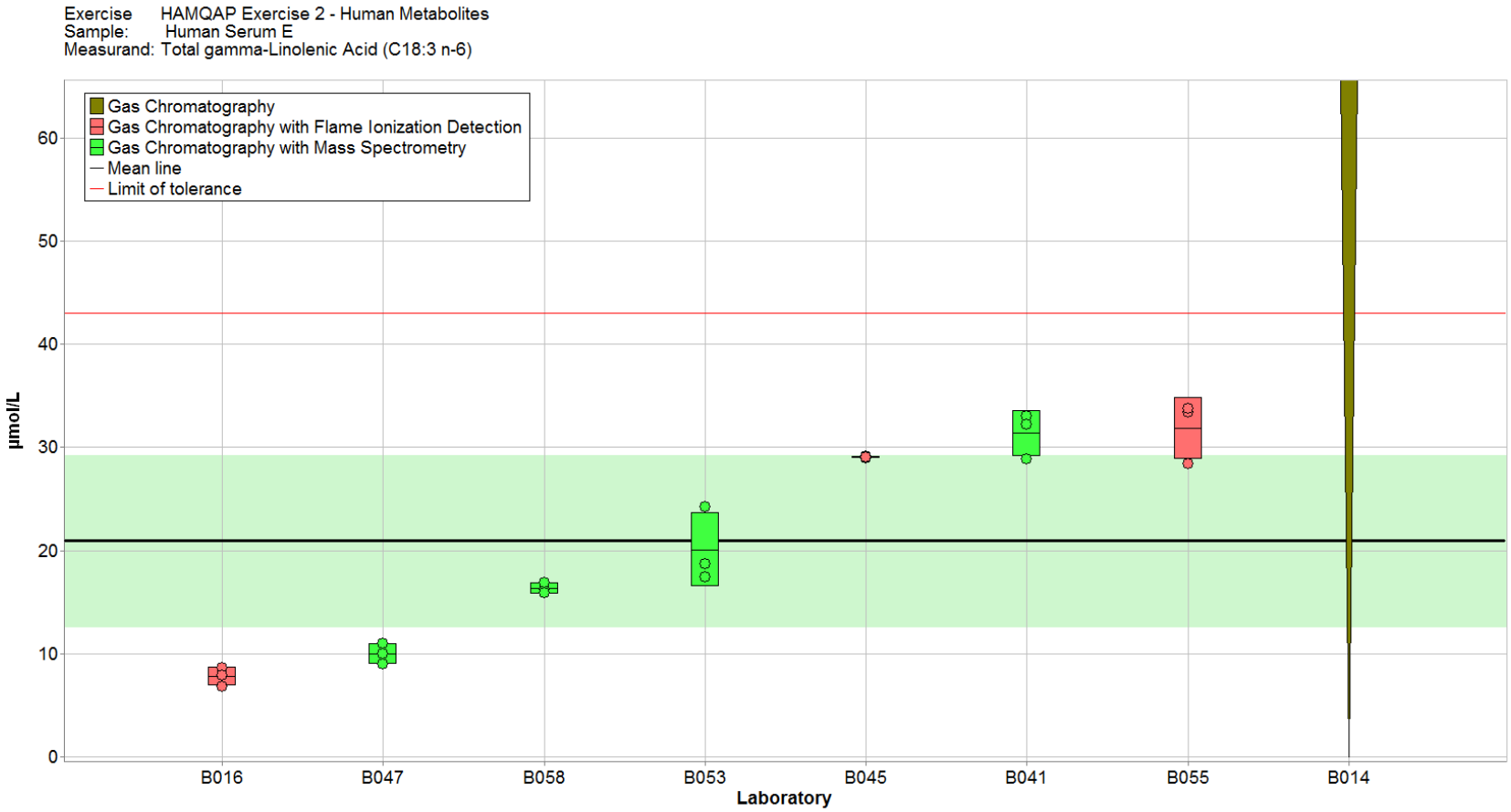

Figure 5-101. Total γ-linolenic acid (C18:3 n-6) in Human Serum E (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

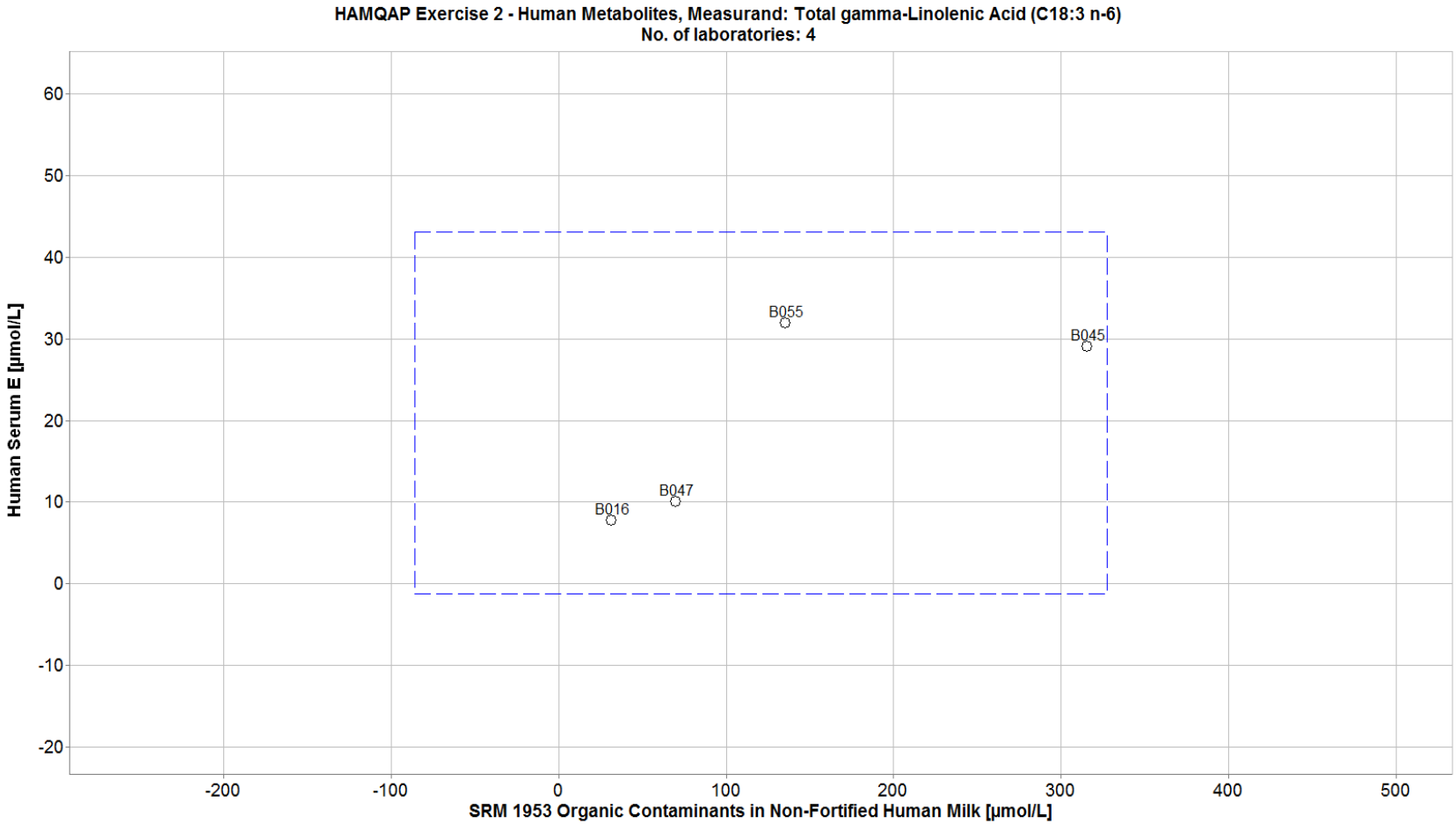

Figure 5-102. Laboratory means for total γ-linolenic acid (C18:3 n-6) in SRM 1953 Organic Contaminants in Non-Fortified Human Milk and Human Serum E (sample/sample comparison view). In this view, the individual laboratory mean for one sample (SRM 1953) is compared to the mean for a second sample (Human Serum E). The dotted blue box represents the consensus range of tolerance for SRM 1953 (x-axis) and Human Serum E (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 5-39. Data summary table for total arachidic acid in SRM 1953 Organic Contaminants in Non-Fortified Human Milk and Human Serum E. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		Total Arachidic Acid (C20:0)									
		SRM 1953 Organic Contaminants in Non-Fortified Human Milk (µmol/L)					Human Serum E (µmol/L)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target										
	B014	< 100	< 100	< 100			< 100	< 100	< 100		
	B016	119.9	113	125.1	119.3	6.1	2.08	2.08	1.92	2.027	0.092
	B017										
	B031										
	B034										
	B041						35.3	35.51	37.61	36.1	1.3
	B047	15.3	15.6	15.6	15.5	0.2	3.4	3.2	3.2	3.27	0.12
	B053						32.9	31.9	33.2	32.67	0.68
	B055	176.9	195.2	207.3	193	15	32.3	32.6	33.6	32.83	0.68
	B058						32.1	30.4	31.2	31.23	0.85
Community Results		Consensus Mean			109		Consensus Mean			33.2	
		Consensus Standard Deviation			188		Consensus Standard Deviation			4.7	
		Maximum			193		Maximum			36.1	
		Minimum			15.5		Minimum			2.027	
		N			3		N			6	

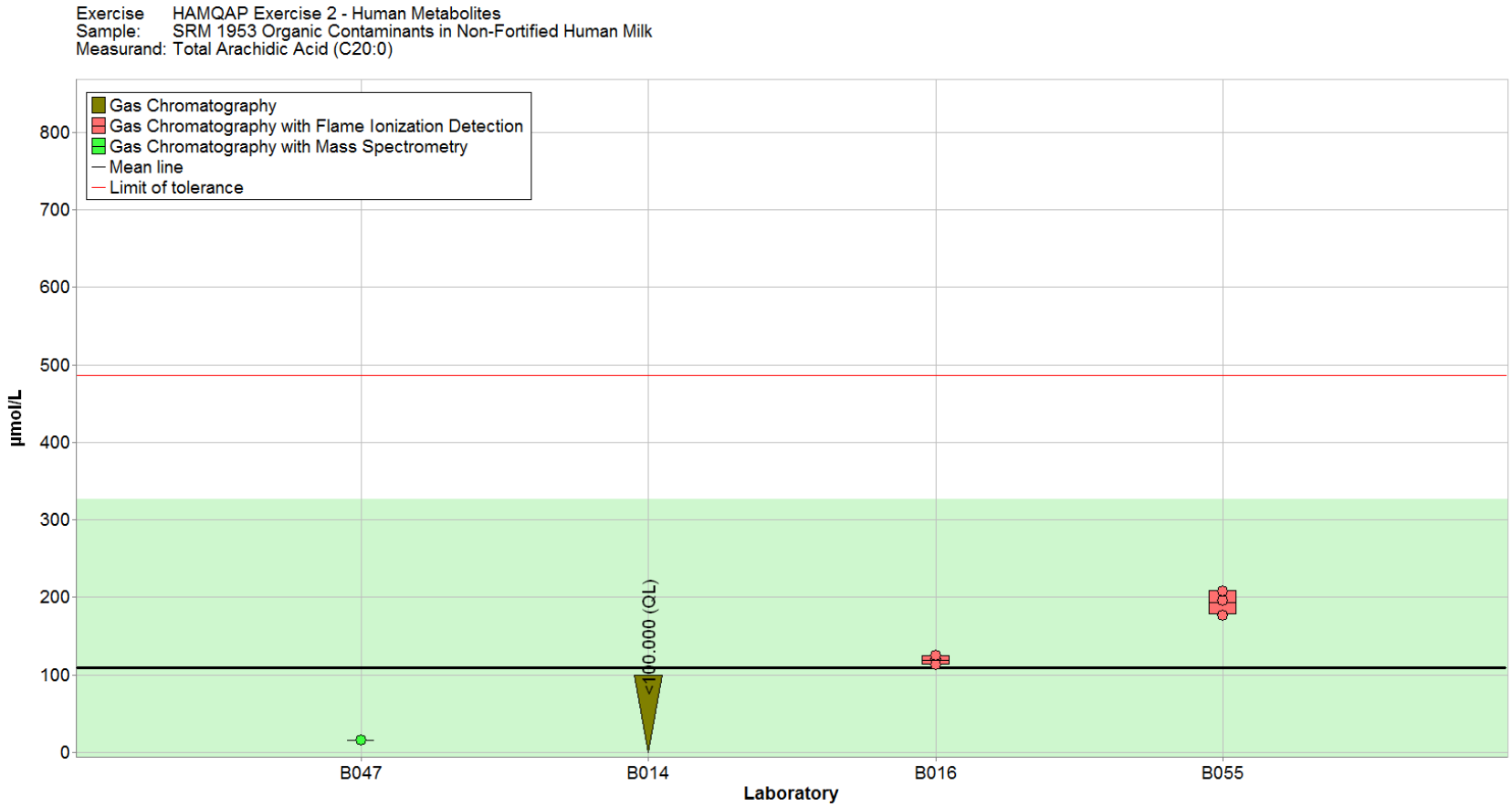

Figure 5-103. Total arachidic acid (C20:0) in SRM 1953 Organic Contaminants in Non-Fortified Human Milk (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

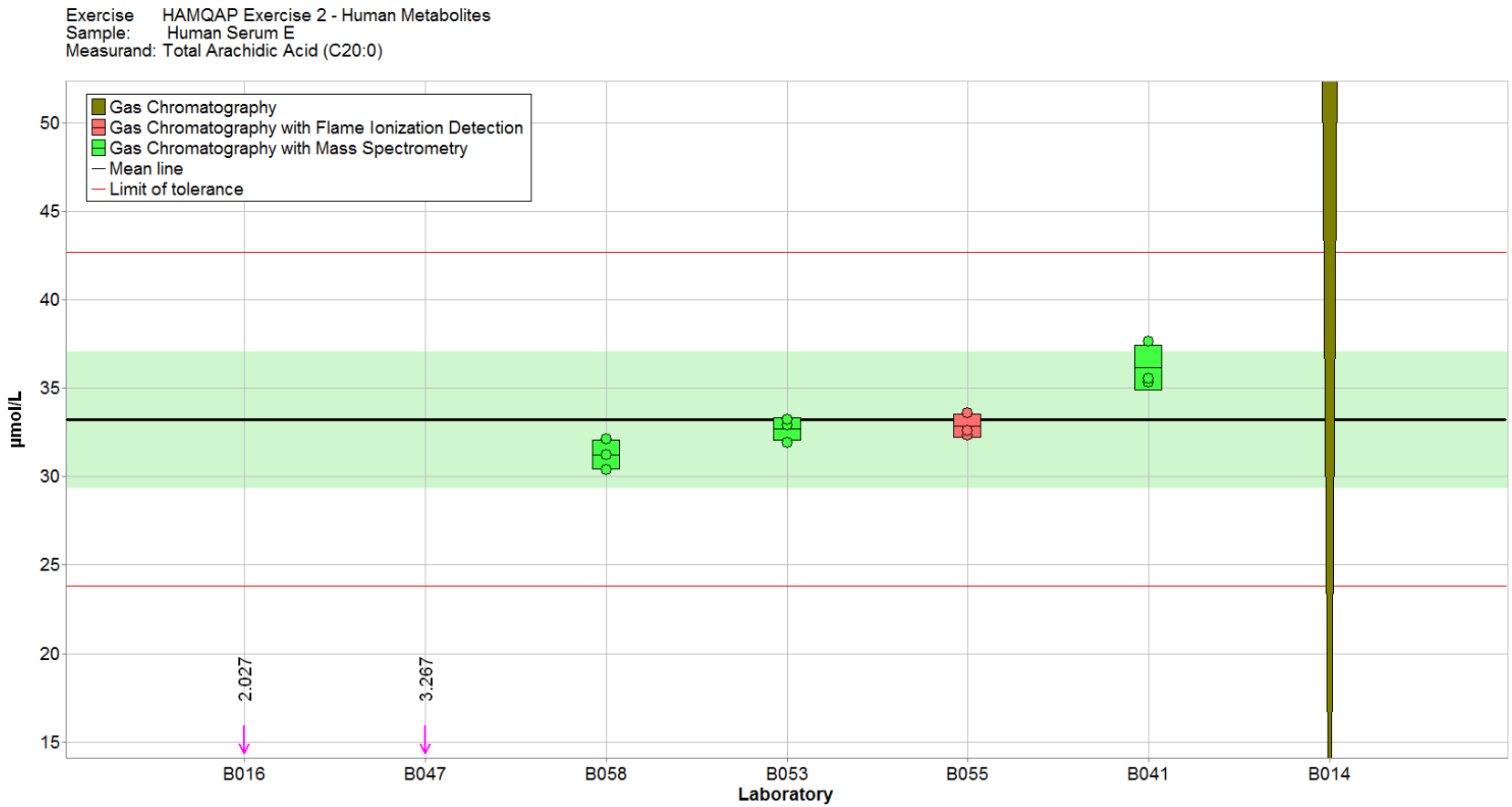

Figure 5-104. Total arachidic acid (C20:0) in Human Serum E (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

Table 5-40. Data summary table for total dihomo-γ-linolenic acid in SRM 1953 Organic Contaminants in Non-Fortified Human Milk and Human Serum E. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		Total Dihomo-gamma-linolenic acid (C20:3 n-6)									
		SRM 1953 Organic Contaminants in Non-Fortified Human Milk (µmol/L)					Human Serum E (µmol/L)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target										
	B014	81.5	81.5	83.1	82.03	0.92	< 100	< 100	< 100		
	B016	17.29	14.03	15.66	15.7	1.6					
	B017										
	B031										
	B034										
	B041						205.85	217.11	239.25	221	17
	B045	< 100	< 100	< 100			208.806	212.13	210.72	210.6	1.7
	B047	143	141	140	141.3	1.5	66	76	82	74.7	8.1
	B053										
	B055	353.7	397.4	425.8	392	36	264.6	260	245.7	256.8	9.9
	B058						242.6	232.1	227.5	234.1	7.7
Community Results		Consensus Mean			153		Consensus Mean			213	
		Consensus Standard Deviation			147		Consensus Standard Deviation			48	
		Maximum			392		Maximum			256.8	
		Minimum			15.7		Minimum			74.7	
		N			4		N			5	

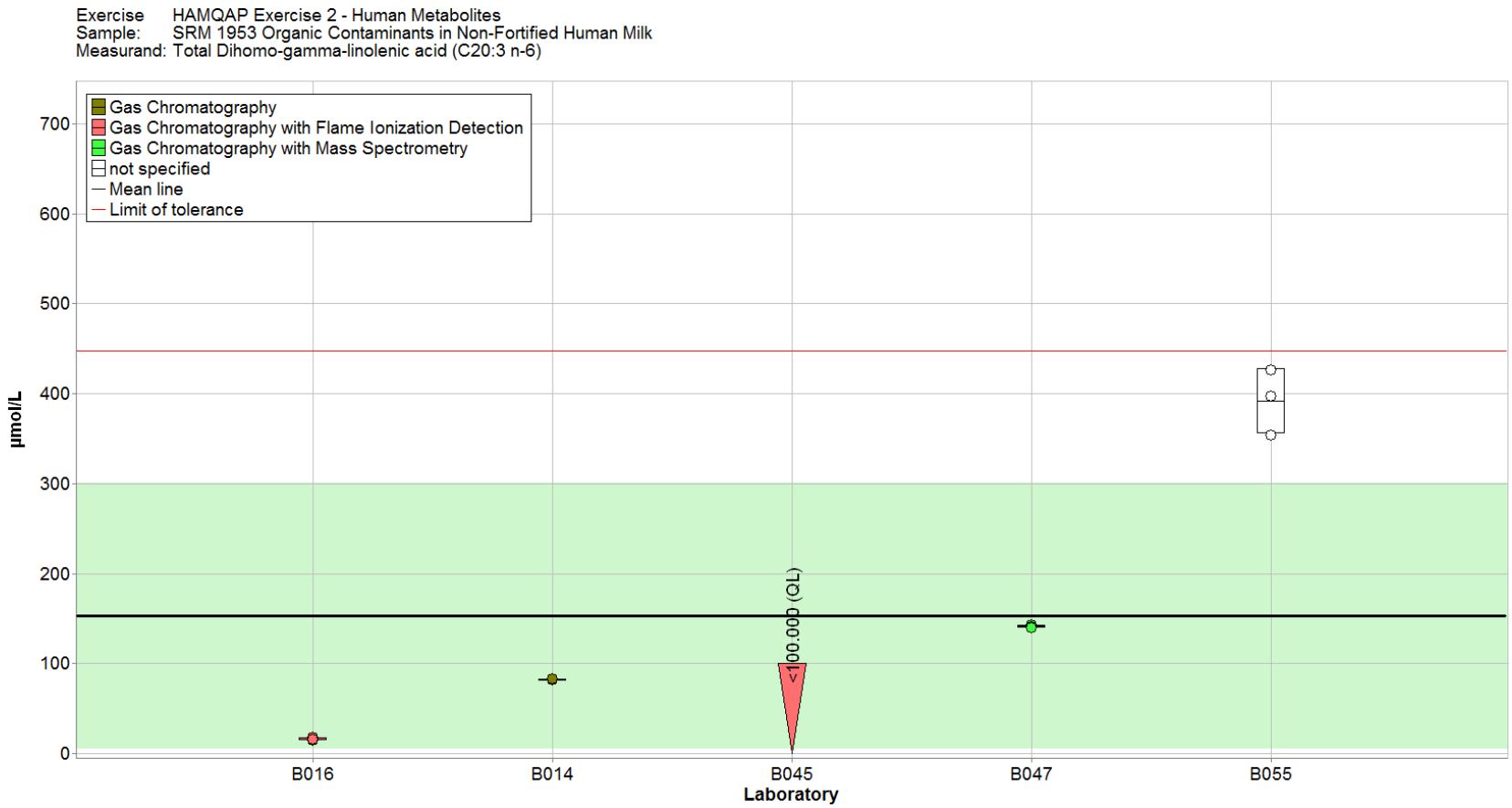

Figure 5-105. Total dihomo-γ-linolenic acid (C20:3 n-6) in SRM 1953 Organic Contaminants in Non-Fortified Human Milk (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

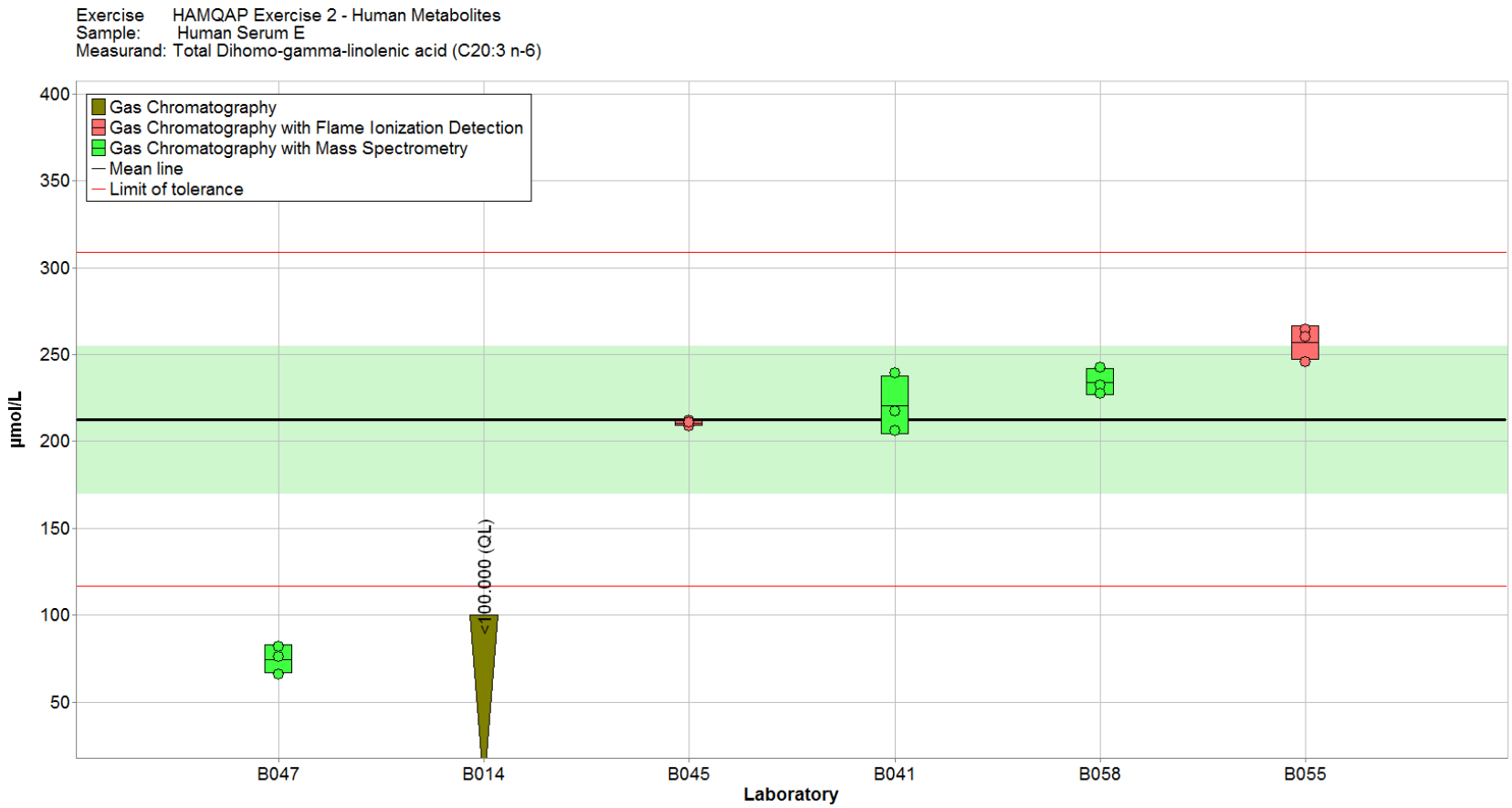

Figure 5-106. Total dihomo-γ-linolenic acid (C20:3 n-6) in Human Serum E (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

Table 5-41. Data summary table for total arachidonic acid in SRM 1953 Organic Contaminants in Non-Fortified Human Milk and Human Serum E. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		Total Arachidonic Acid (C20:4 n-6)									
		SRM 1953 Organic Contaminants in Non-Fortified Human Milk (µmol/L)					Human Serum E (µmol/L)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target										
	B014	141	143	141	141.7	1.2	252	229	244	242	12
	B015	423.274	391.978	382.093	399	21	1345.6	1247.1	924.9	1173	220
	B016	119.9	113	125.1	119.3	6.1	176.7	243.5	220.7	214	34
	B017										
	B031										
	B034										
	B041						821.43	720.9	822.64	788	58
	B045	207	214	212	211.0	3.6	757.39	763.28	756.13	758.9	3.8
	B047	234	228	227	229.7	3.8	202	230	262	231	30
	B053						652	675	671	666	12
	B055	734.1	840.5	594.2	723	124	1150.2	1132.1	988.9	1090	88
	B058						1052	1003	987	1014	34
Community Results		Consensus Mean			278		Consensus Mean			686	
		Consensus Standard Deviation			193		Consensus Standard Deviation			433	
		Maximum			722.9		Maximum			1173	
		Minimum			119.3		Minimum			214	
		N			6		N			9	

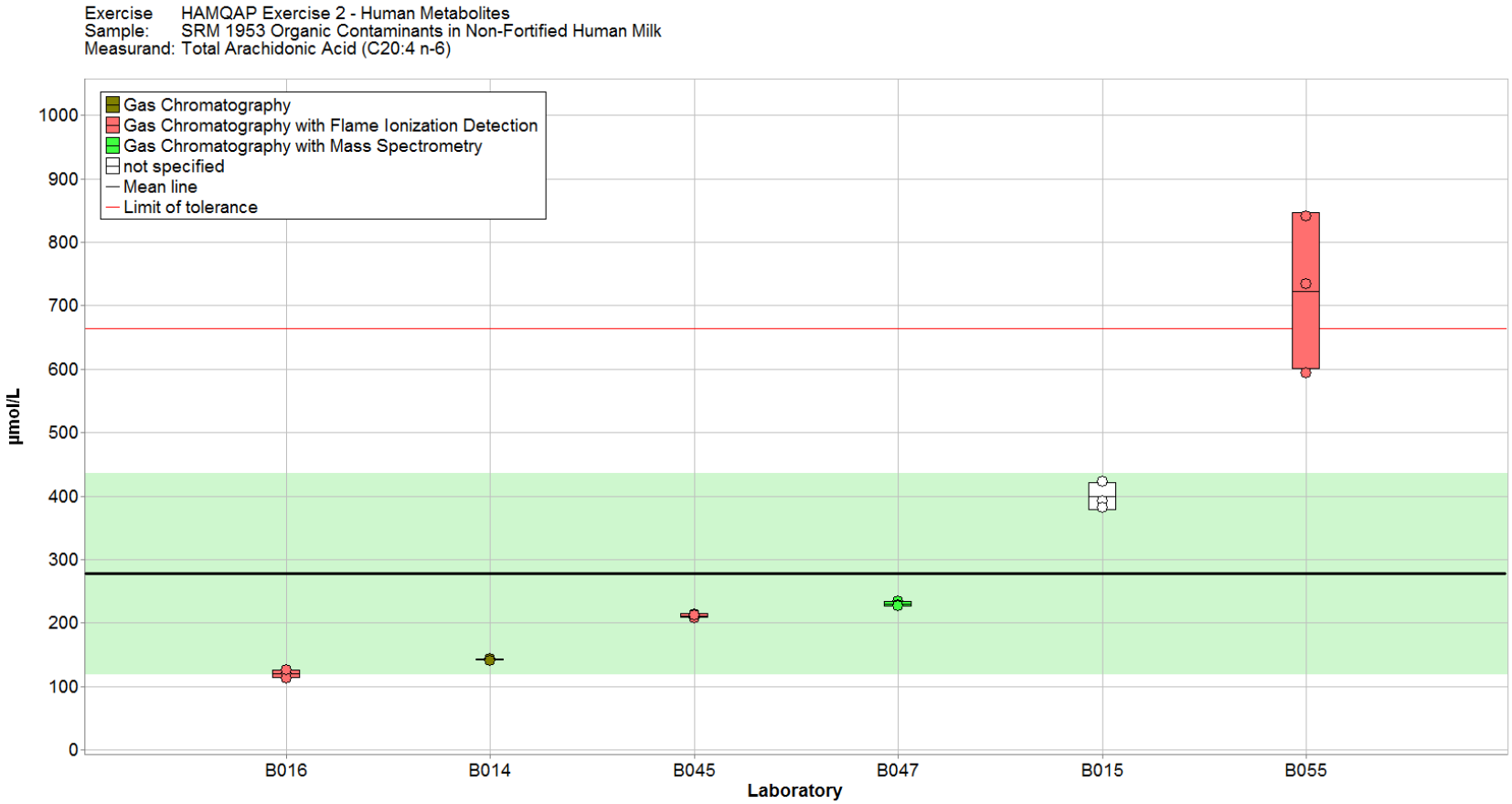

Figure 5-107. Total arachidonic acid (C20:4 n-6) in SRM 1953 Organic Contaminants in Non-Fortified Human Milk (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

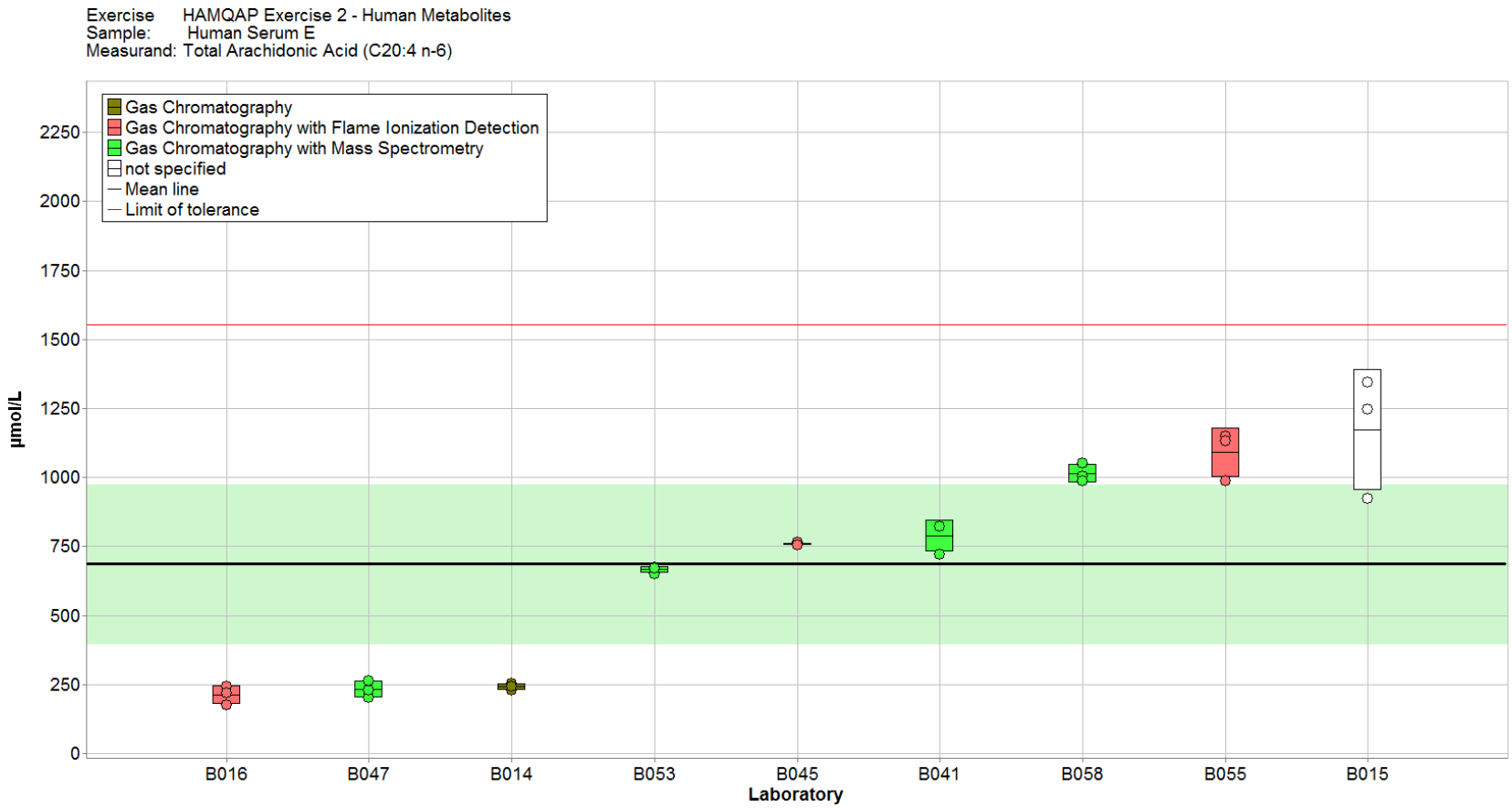

Figure 5-108. Total arachidonic acid (C20:4 n-6) in Human Serum E (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

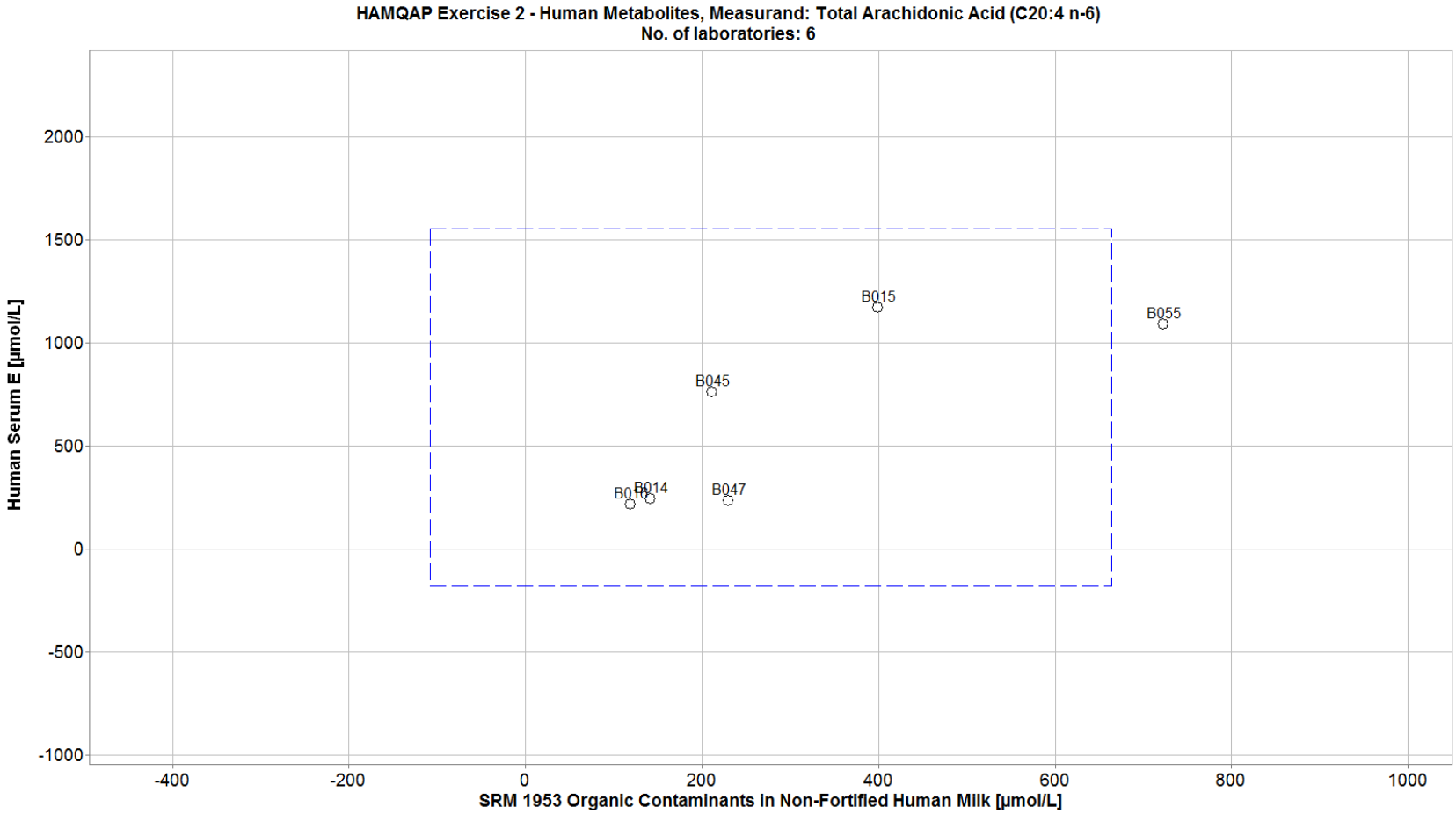

Figure 5-109. Laboratory means for total arachidonic acid (C20:4 n-6) in SRM 1953 Organic Contaminants in Non-Fortified Human Milk and Human Serum E (sample/sample comparison view). In this view, the individual laboratory mean for one sample (SRM 1953) is compared to the mean for a second sample (Human Serum E). The dotted blue box represents the consensus range of tolerance for SRM 1953 (x-axis) and Human Serum E (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 5-42. Data summary table for total EPA in SRM 1953 Organic Contaminants in Non-Fortified Human Milk and Human Serum E. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		Total EPA (C20:5 n-3)									
		SRM 1953 Organic Contaminants in Non-Fortified Human Milk (µmol/L)					Human Serum E (µmol/L)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target										
	B014	< 100	< 100	< 100			< 100	< 100	< 100		
	B015	693.682	642.111	624.419	653	36	167.2	156.8	153.9	159.3	7.0
	B016	13.39	13.72	15.37	14.2	1.1	12.56	14.54	13.55	13.55	0.99
	B017										
	B031										
	B034										
	B041						34.59	29.33	34.17	32.7	2.9
	B045	< 100	< 100	< 100			32.08	31.88	33.05	32.34	0.63
	B047	25	26	25	25.33	0.58	24	22	23	23.0	1.0
	B053						29.4	30.6	31.5	30.5	1.1
	B055	38.4	45	48.6	44.00	5.17	37	37	34.1	36.0	1.7
	B058						58.9	48.6	50.2	52.6	5.5
Community Results		Consensus Mean			28		Consensus Mean			32	
		Consensus Standard Deviation			44		Consensus Standard Deviation			17	
		Maximum			653		Maximum			159.3	
		Minimum			14.2		Minimum			13.55	
		N			4		N			8	

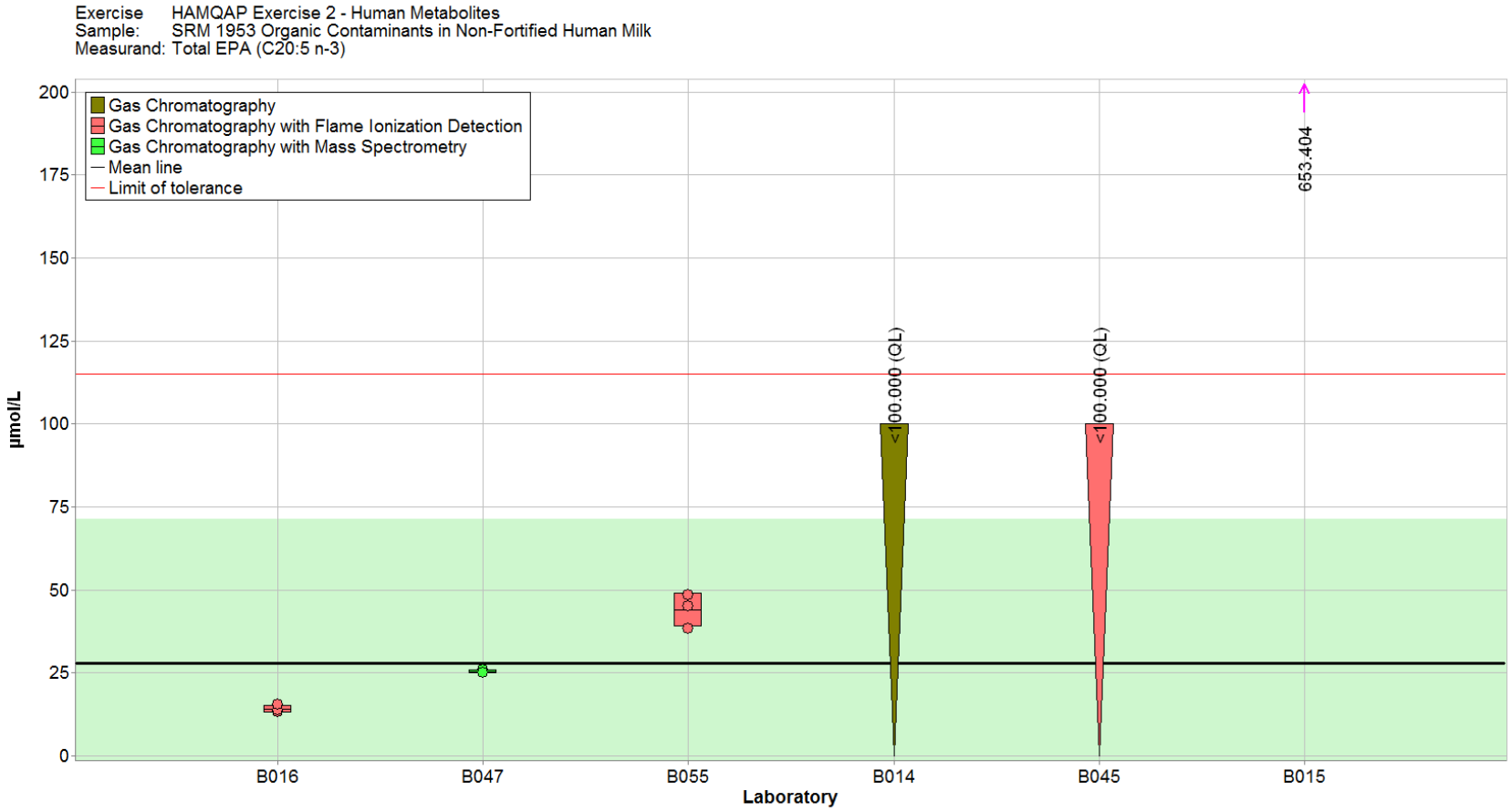

Figure 5-110. Total EPA (C20:5 n-3) in SRM 1953 Organic Contaminants in Non-Fortified Human Milk (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

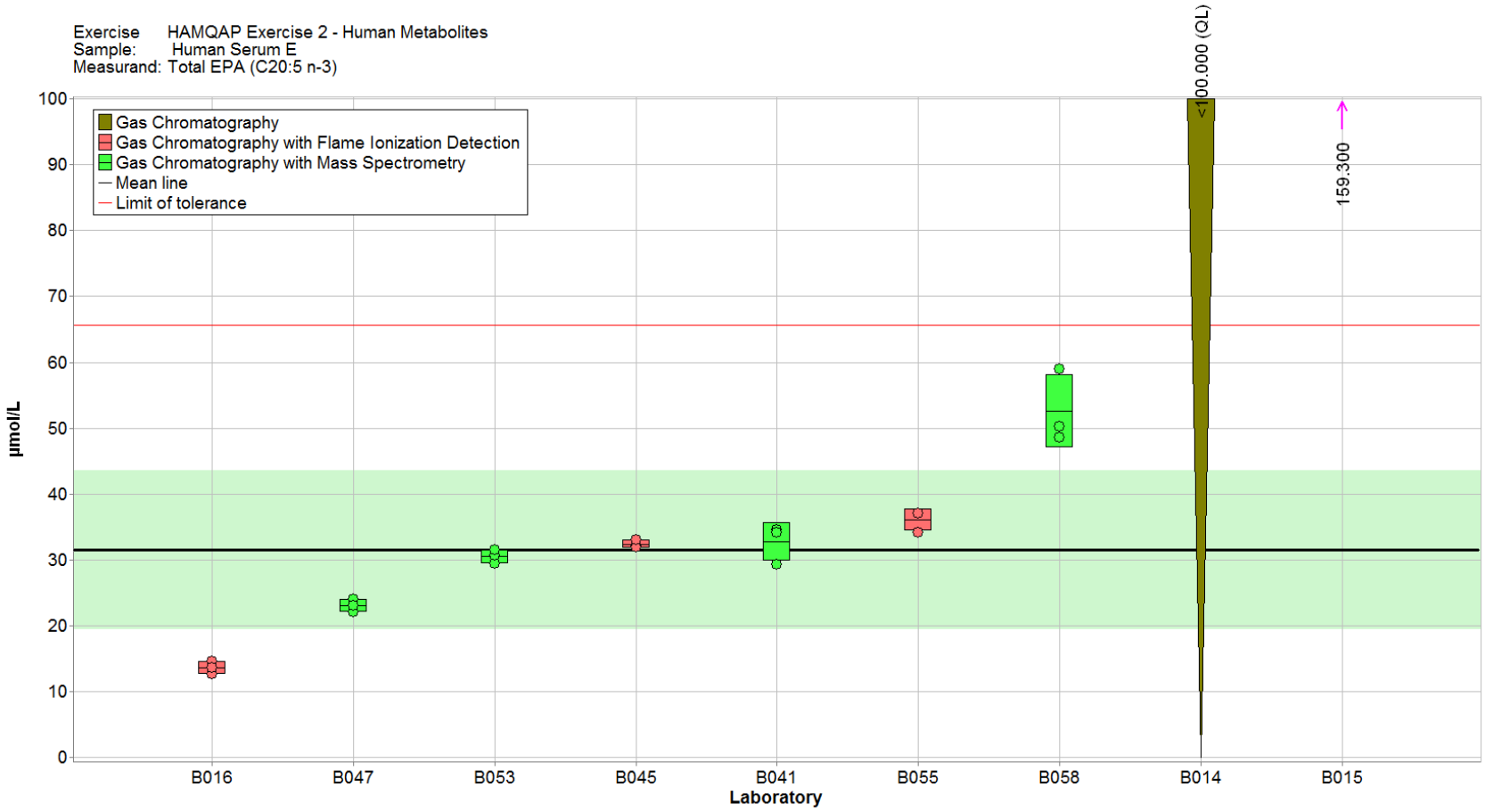

Figure 5-111. Total EPA (C20:5 n-3) in Human Serum E (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

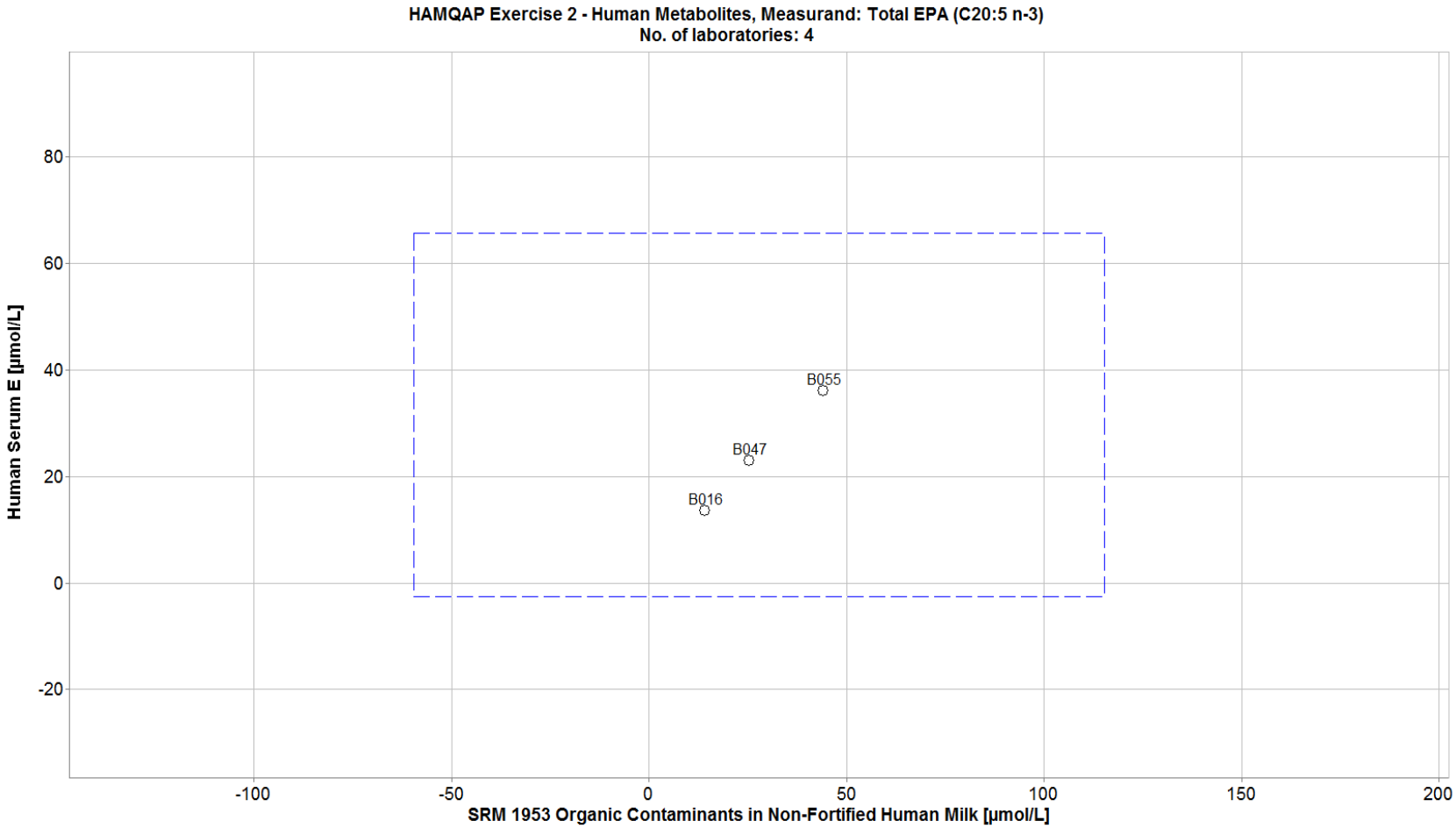

Figure 5-112. Laboratory means for total EPA (C20:5 n-3) in SRM 1953 Organic Contaminants in Non-Fortified Human Milk and Human Serum E (sample/sample comparison view). In this view, the individual laboratory mean for one sample (SRM 1953) is compared to the mean for a second sample (Human Serum E). The dotted blue box represents the consensus range of tolerance for SRM 1953 (x-axis) and Human Serum E (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 5-43. Data summary table for total DPA in SRM 1953 Organic Contaminants in Non-Fortified Human Milk and Human Serum E.

		Total DPA (C22:5 n-3)									
		SRM 1953 Organic Contaminants in Non-Fortified Human Milk (µmol/L)					Human Serum E (µmol/L)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target										
	B014	< 100	< 100	< 100			< 100	< 100	< 100		
	B015	103.542	100.014	95.919	99.8	3.8	73	68.2	51.4	64	11
	B016	29.5	26.32	29.65	28.5	1.9	7.56	11.19	18	12.3	5.3
	B017										
	B031										
	B034										
	B041						33.75	34.82	40.43	36.3	3.6
	B045	< 100	< 100	< 100			36.3	37.12	37.29	36.90	0.53
	B047	60	59	60	59.67	0.58	16	18	21	18.3	2.5
	B053										
	B055										
Community Results		Consensus Mean			63		Consensus Mean			34	
		Consensus Standard Deviation			73		Consensus Standard Deviation			32	
		Maximum			99.8		Maximum			64	
		Minimum			28.5		Minimum			12.3	
		N			3		N			5	

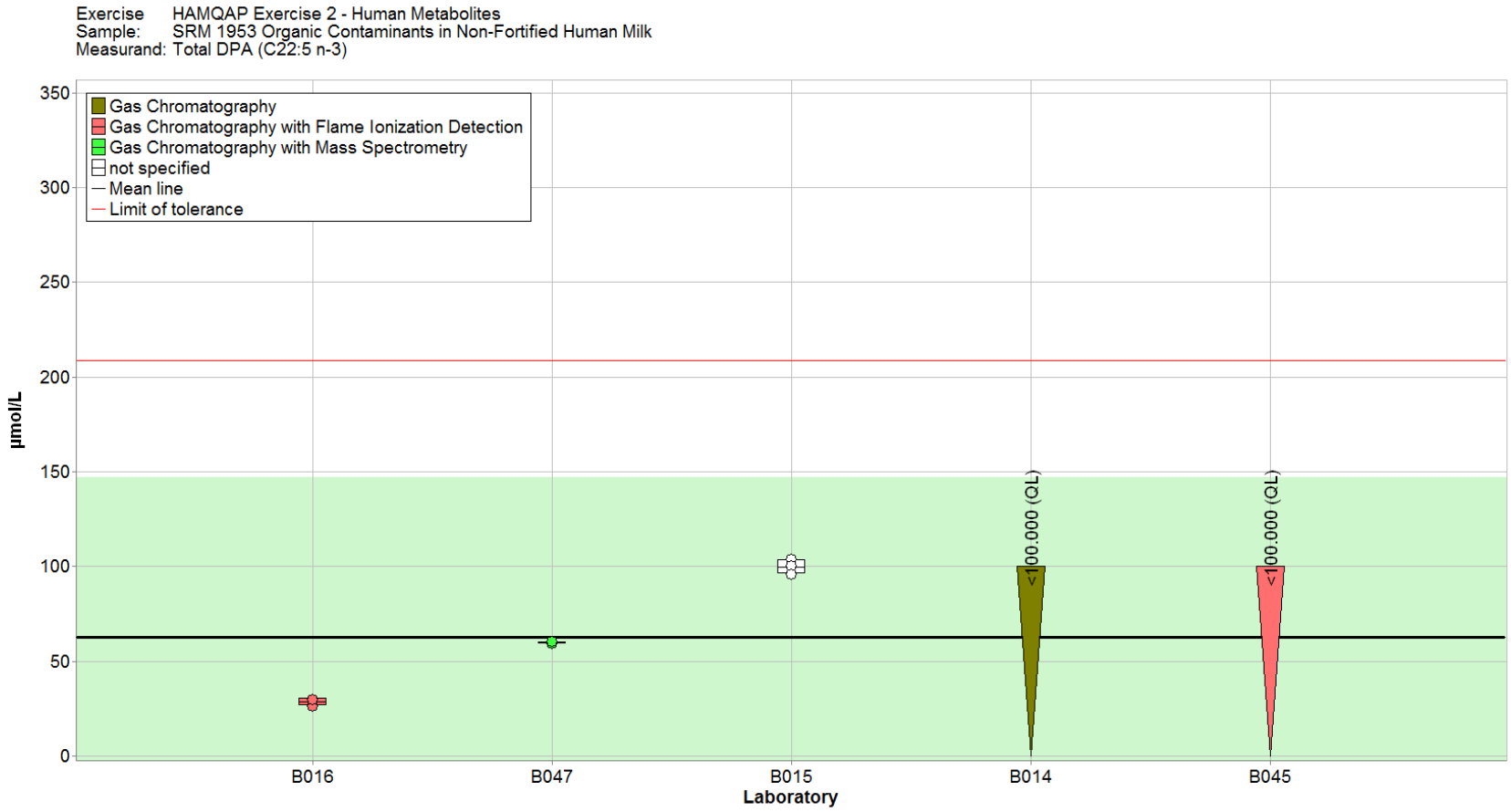

Figure 5-113. Total DPA (C22:5 n-3) in SRM 1953 Organic Contaminants in Non-Fortified Human Milk (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

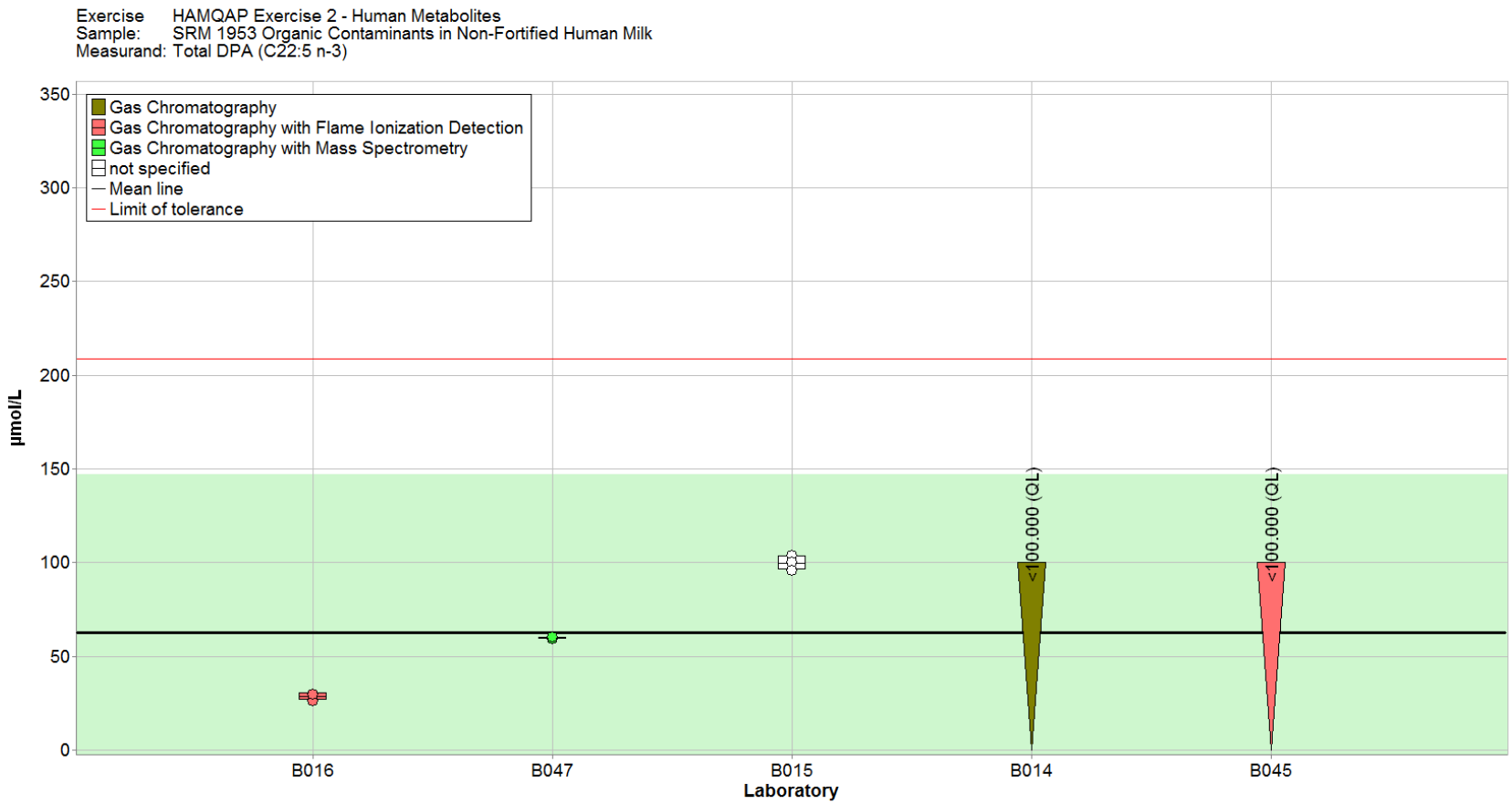

Figure 5-114. Total DPA (C22:5 n-3) in Human Serum E (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

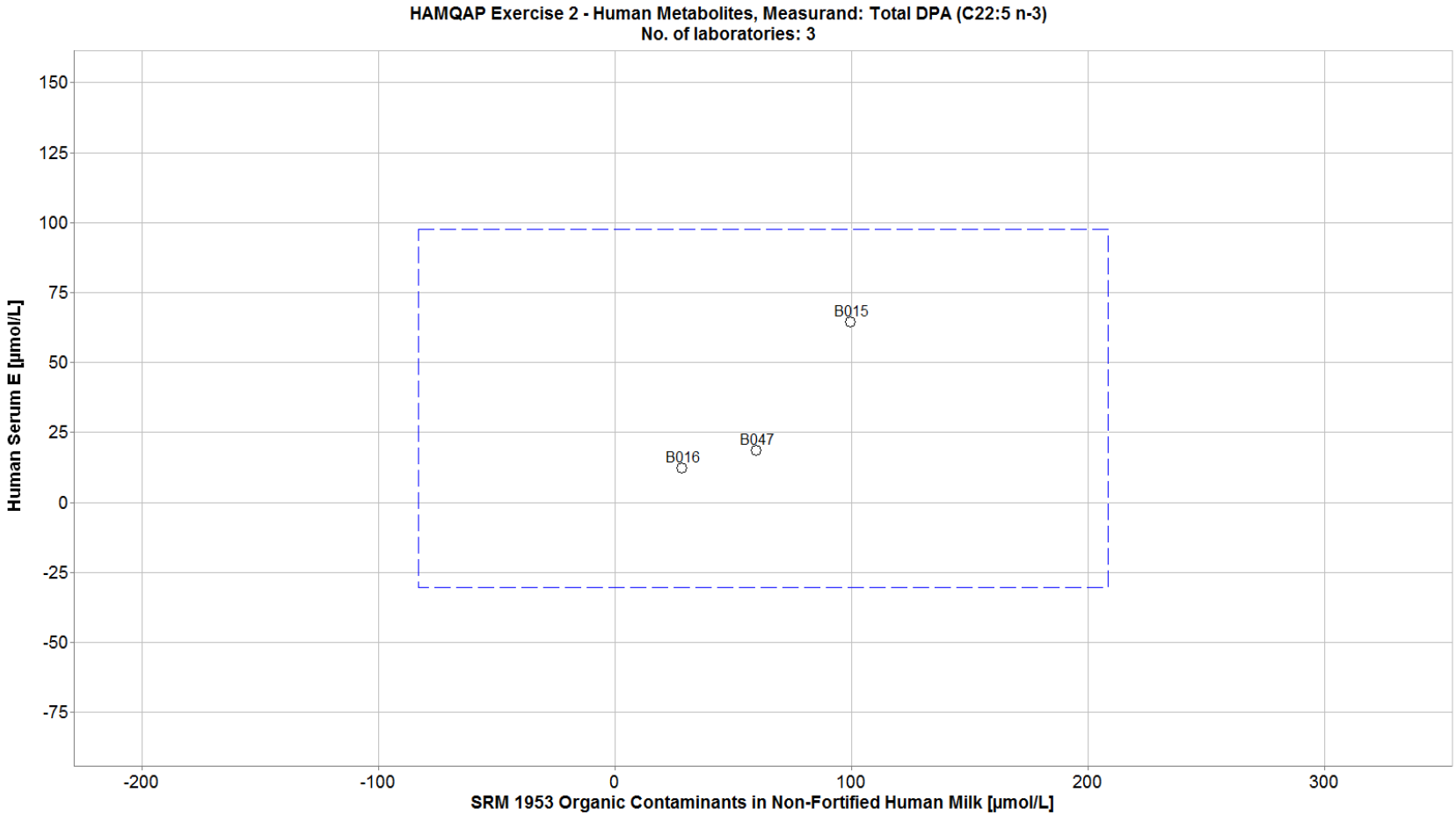

Figure 5-115. Laboratory means for total DPA (C22:5 n-3) in SRM 1953 Organic Contaminants in Non-Fortified Human Milk and Human Serum E (sample/sample comparison view). In this view, the individual laboratory mean for one sample (SRM 1953) is compared to the mean for a second sample (Human Serum E). The dotted blue box represents the consensus range of tolerance for SRM 1953 (x-axis) and Human Serum E (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 5-44. Data summary table for total DHA in SRM 1953 Organic Contaminants in Non-Fortified Human Milk and Human Serum E.

		Total DHA (C22:6 n-3)									
		SRM 1953 Organic Contaminants in Non-Fortified Human Milk (µmol/L)					Human Serum E (µmol/L)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target										
	B014	< 100	< 100	< 100			< 100	< 100	< 100		
	B015	129.468	118.711	114.355	120.8	7.8	177.7	167.1	118.1	154	32
	B016	40.33	37.74	42	40.0	2.1	21.3	36.83	57.83	39	18
	B017										
	B031										
	B034										
	B041						118.76	101.983	118.7	113	10
	B045	< 100	< 100	< 100			101.31	100.87	101.81	101.33	0.47
	B047	53	52	52	52.33	0.58	36	44	50	43.3	7.0
	B053						99	105	96.6	100.2	4.3
	B055	113.9	127.9	134.3	125	10	123.6	113.6	109.9	115.7	7.1
	B058						142.1	133.1	120.4	132	11
Community Results		Consensus Mean			85		Consensus Mean			101	
		Consensus Standard Deviation			32		Consensus Standard Deviation			38	
		Maximum			125		Maximum			154	
		Minimum			40.0		Minimum			39	
		N			4		N			8	

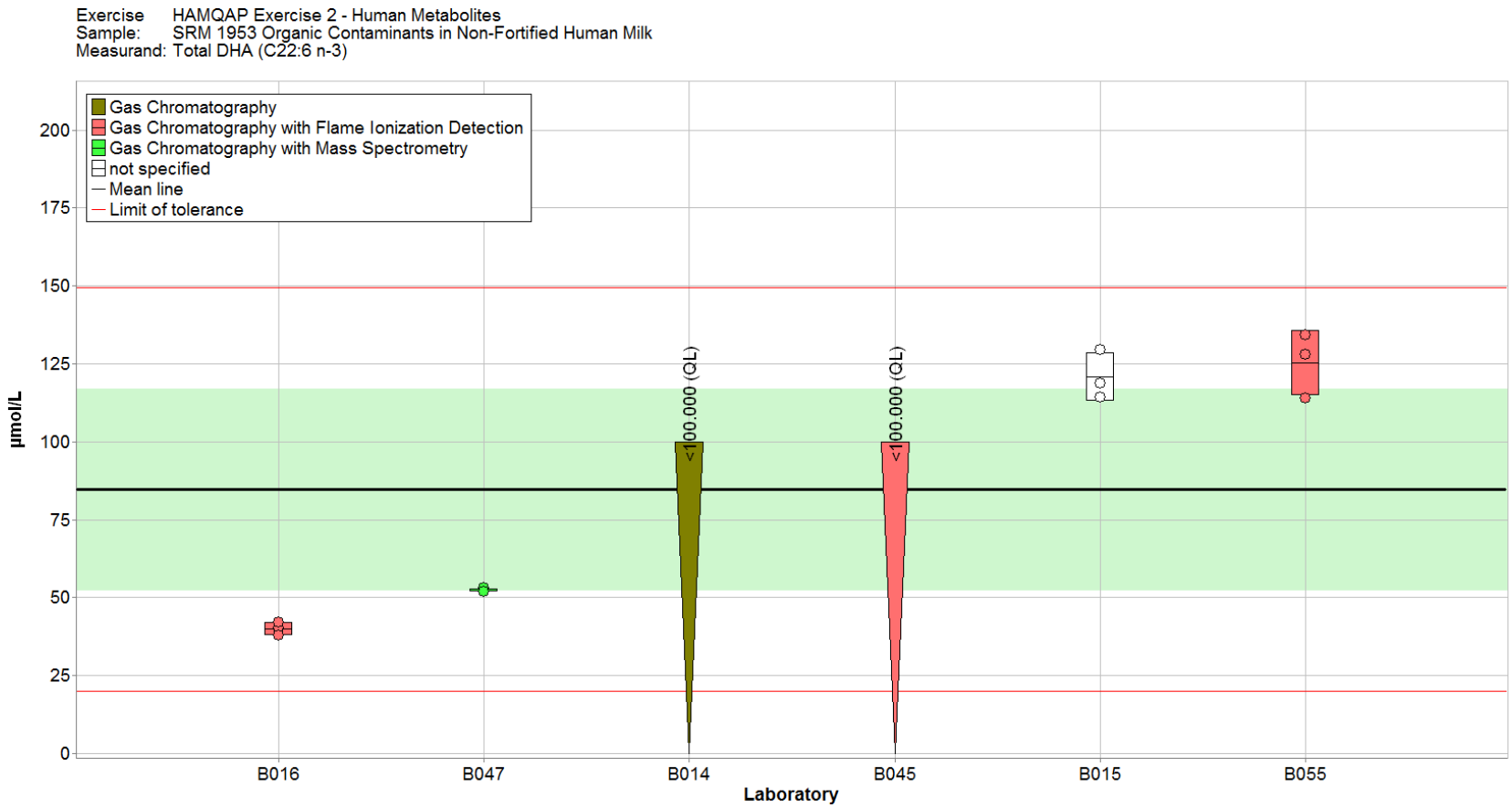

Figure 5-116. Total DHA (C22:6 n-3) in SRM 1953 Organic Contaminants in Non-Fortified Human Milk (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

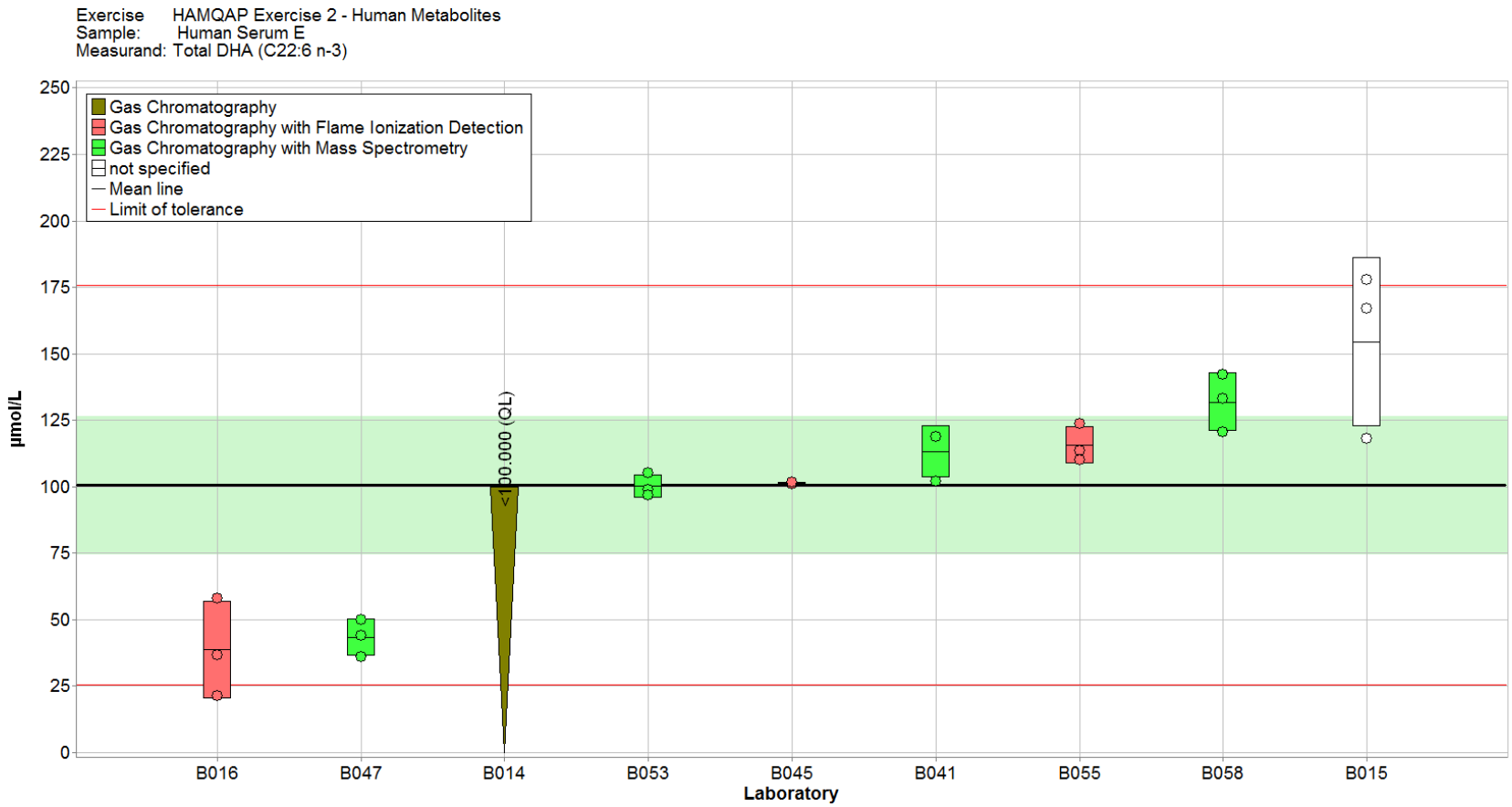

Figure 5-117. Total DHA (C22:6 n-3) in Human Serum E (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

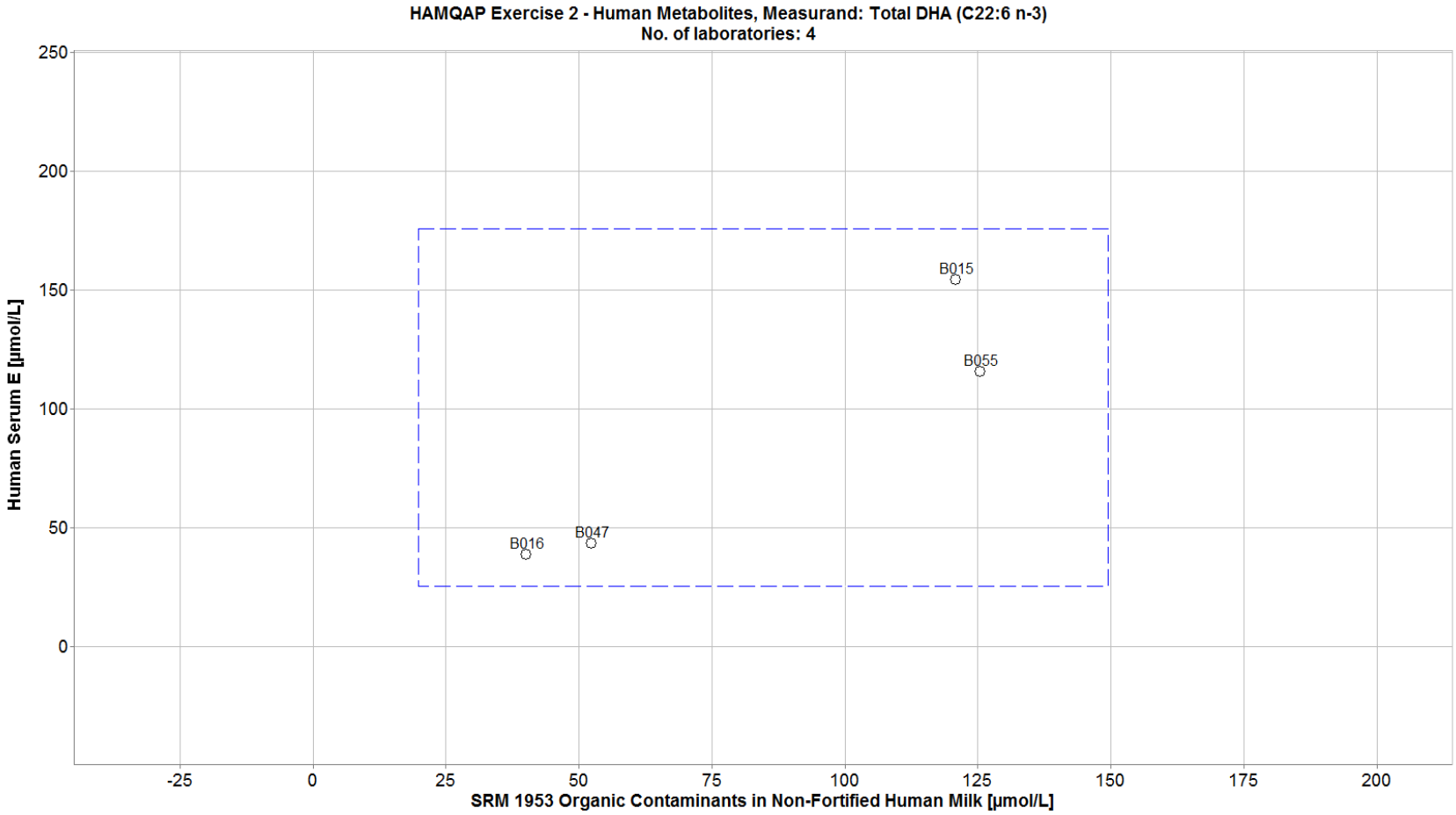

Figure 5-118. Laboratory means for total DHA (C22:6 n-3) in SRM 1953 Organic Contaminants in Non-Fortified Human Milk and Human Serum E (sample/sample comparison view). In this view, the individual laboratory mean for one sample (SRM 1953) is compared to the mean for a second sample (Human Serum E). The dotted blue box represents the consensus range of tolerance for SRM 1953 (x-axis) and Human Serum E (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

Fatty Acids Overall Samples Comparison

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

- The between-laboratory variability for measured total fatty acids was generally higher in the dietary intake study than the human metabolism study. Specific precision needs may be different for laboratories testing dietary intake samples compared to those testing human metabolism samples.
- Participation was low for the fatty acids in both the dietary intake and human metabolites studies. Participating laboratories may not have analytical methods to measure all individual fatty acids, or the measurement of all indicated fatty acids is not a current concern of the participating laboratories.
    - Participation was especially low for *trans*-fatty acids (*trans*-vaccenic and elaidic acids).
    - Future studies may want to target a smaller number of fatty acids over a range of concentrations.

SECTION 6: NATURAL PRODUCTS (Co-Enzyme Q10)

Study Overview

In this study, participants were provided with two commercial coenzyme Q10 (CoQ10) supplements. Commercial supplement A was labeled as containing only CoQ10 at approximately 400 mg/tablet and commercial supplement B was labeled as containing CoQ10 at approximately 30 mg/tablet, plus garlic. Participants were asked to use in-house analytical methods to determine the mass fraction (mg/g) of total CoQ10, ubiquinol, and/or ubiquinone in each matrix. CoQ10 is naturally found in the body and is used in the cellular production of energy and from damage.[9] Due to the critical function of CoQ10, supplementation has been studied for use in prevention of cardiac disease and reduction of neurological disease symptoms as well as multiple other disease states.

Dietary Intake Sample Information

CoQ10 Supplement A. Participants were provided with three packets, each containing 20 gelcaps. The gelcaps were heat-sealed inside aluminized bags. Before use, participants were instructed to composite the contents of the packet and mix thoroughly and to use a sample size appropriate for their in-house method. After compositing, participants were asked to store the material at controlled room temperature, 10 °C to 30 °C, to analyze the material within two days, and to prepare one sample and report one value from each packet provided. The approximate analyte levels were not reported to participants prior to the study, and target values for CoQ10 in commercial supplement A have not been determined at NIST.

CoQ10 Supplement B. Participants were provided with three packets, each containing 20 gelcaps. The gelcaps were heat-sealed inside aluminized bags. Before use, participants were instructed to composite the contents of the packet and mix thoroughly and to use a sample size appropriate for their in-house method. After compositing, participants were asked to store the material at controlled room temperature, 10 °C to 30 °C, to analyze the material within two days, and to prepare one sample and report one value from each packet provided. The approximate analyte levels were not reported to participants prior to the study, and target values for CoQ10 in commercial supplement B have not been determined at NIST.

Dietary Intake Study Results

- Sixteen laboratories enrolled in this exercise and received samples to measure CoQ10 as ubiquinone, ubiquinol and/or total CoQ10. Eleven laboratories reported results for each sample (69% participation).
- The between-laboratory variability was good for the determination of ubiquinone in commercial supplement A (15 % RSD) and commercial supplement B (21% RSD).
- All laboratories that reported analytical method information indicated using LC-absorbance.
- Tables and figures for ubiquinone are provided to represent the data for this study. Limited value was found from tables and figures for ubiquinol and CoQ10.
- Six laboratories reported results for ubiquinol in supplement A, however, all but one reported zero as a result.

[9] Coenzyme Q10 (PDQ®)–Health Professional Version. National Institutes of Health National Cancer Institute. https://www.cancer.gov/about-cancer/treatment/cam/hp/coenzyme-q10-pdq (accessed February 20, 2019).

- For both samples, the values reported for total CoQ10 were virtually identical to the results for ubiquinone (**Figure 6-2**).
- Seven laboratories reported results for ubiquinol in supplement B, and two of those laboratories reported zero as a result (**Figure 6-3**).

Dietary Intake Technical Recommendations

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

- Ubiquinol was not present in the samples at levels above the limit of detection for laboratories using LC-absorbance methods.
- The determination of ubiquinone, at levels of 15 mg/g to 400 mg/g, with or without the presence of garlic, does not appear to be a challenge for most laboratories (**Figures 6-1 and 6-4**).
- Laboratories reporting large within-laboratory variability should investigate the completeness of the extraction during sample preparation.
    - Any extraction procedure should be optimized to determine the most effective extraction solvent to ensure exhaustive extraction of the analyte from the matrix.
    - The optimum number of extraction cycles must be determined by sequential re-extraction of the sample matrix until no further increase in yield is observed. Sequential extractions may be needed if the extraction solvent becomes saturated during the first (or only) extraction cycle.
- Laboratories reporting results flagged as outliers should check for calculation errors. One example is to confirm that factors for all dilutions have been properly tabulated.
- Several laboratories reported values of zero for measurements in all materials. "Zero" is not a quantity that can be measured, and therefore a more appropriate result would be to report that a value is below the MDL, LOQ, or QL.

Table 6-1. Individualized data summary table (NIST) for total CoQ10, ubiquinol, and ubiquinone in CoQ10 supplements.

National Institute of Standards & Technology

HAMQAP Exercise 2 - Natural Products

		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 CoQ10	CoQ10 Supplement A		mg/g					2	400	32		
Total CoQ10	CoQ10 Supplement B		mg/g					2	20	14		
Ubiquinol	CoQ10 Supplement A		mg/g					6	1.7	5.7		
Ubiquinol	CoQ10 Supplement B		mg/g					7	0.35	0.58		
Ubiquinone	CoQ10 Supplement A		mg/g					11	370	55		
Ubiquinone	CoQ10 Supplement B		mg/g					11	14.9	3.1		

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 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 6-2. Data summary table for total CoQ10 in CoQ10 dietary supplements.

		Total CoQ10									
		CoQ10 Supplement A (mg/g)					CoQ10 Supplement B (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target										
	B011	334	453	396	394	60	20.6	20.6	20.6	20.6	0.0
	B030										
	B039										
	B044										
	B046	397	392	396	395	2	14.4	14.3	14.5	14.4	0.1
Community Results		Consensus Mean			395		Consensus Mean			17.5	
		Consensus Standard Deviation			32		Consensus Standard Deviation			13.7	
		Maximum			395		Maximum			20.6	
		Minimum			394		Minimum			14.4	
		N			2		N			2.0	

Table 6-3. Data summary table for ubiquinol in CoQ10 dietary supplements. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		Ubiquinol									
		CoQ10 Supplement A (mg/g)					CoQ10 Supplement B (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target										
	B006						4.4	4.4	4.4	4.4	0.0
	B007										
	B011	0	0	0	0	0	0.30	0.30	0.30	0.30	0.00
	B016	0	0	0	0	0	0	0	0	0	0
	B019	0	0	0	0	0	0.80	0.80	0.50	0.70	0.17
	B025				0						
	B031	0	0	0	0	0	0	0	0	0	0
	B033	0	0	0	0	0	0.90	1.10	0.90	0.97	0.12
	B036										
	B038										
	B039										
	B044										
	B046	14.5	11.8	8.5	11.6	3.0	0.13	0.12	0.18	0.14	0.03
Community Results		Consensus Mean			1.7		Consensus Mean			0.35	
		Consensus Standard Deviation			5.7		Consensus Standard Deviation			0.58	
		Maximum			11.6		Maximum			4.40	
		Minimum			0		Minimum			0	
		N			6		N			7	

Table 6-4. Data summary table for ubiquinone in CoQ10 dietary supplements. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		Ubiquinone									
		CoQ10 Supplement A (mg/g)					CoQ10 Supplement B (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target										
	B005	444	435	434	437	6	16	15.6	15.9	15.8	0.2
	B006	206	223	225	218	10	10.2	10.1	9.8	10.0	0.2
	B007										
	B008	354	319	348	340	19	26.4	23.8	25.7	25.3	1.3
	B011	334	453	396	394	60	20.3	20.3	20	20.2	0.2
	B016	367130	370790	371940	369953	2512	16454	15893	15079	15809	691
	B019	395	384	385	388	6	14.7	14.7	14.6	14.7	0.1
	B025										
	B026	374	363	371	369	6	15.0	15.0	15.0	15.0	0.0
	B031	355	352	353	353	2	9.7	10.5	11.8	10.7	1.0
	B033	387	392	399	393	6	15.6	15.4	16.0	15.7	0.3
	B036	395	390	393	393	3	14.4	14.6	14.5	14.5	0.1
	B038										
	B039										
	B044										
	B046	382	380	387	383	4	14.2	14.2	14.3	14.2	0.1
Community Results		Consensus Mean			374		Consensus Mean			14.9	
		Consensus Standard Deviation			55		Consensus Standard Deviation			3.1	
		Maximum			369953		Maximum			15809	
		Minimum			218		Minimum			10.0	
		N			11		N			11	

Figure 6-1. Ubiquinone in CoQ10 Supplement 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 black 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, $|Z'_{\text{comm}}| \leq 2$. A NIST value has not been determined in this material.

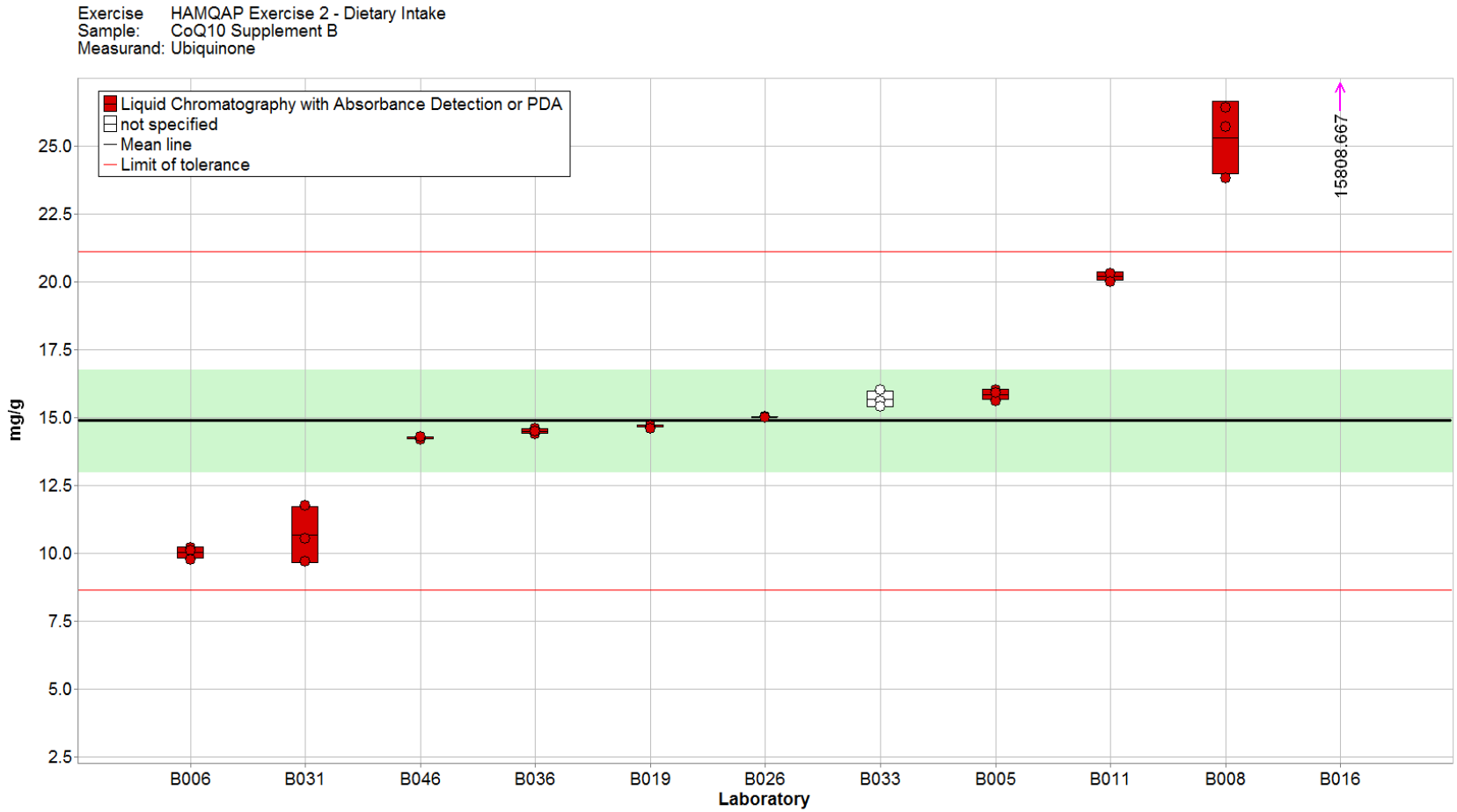

Figure 6-2. Ubiquinone in CoQ10 Supplement B (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid black line 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, $|Z'_{\text{comm}}| \leq 2$. A NIST value has not been determined in this material.

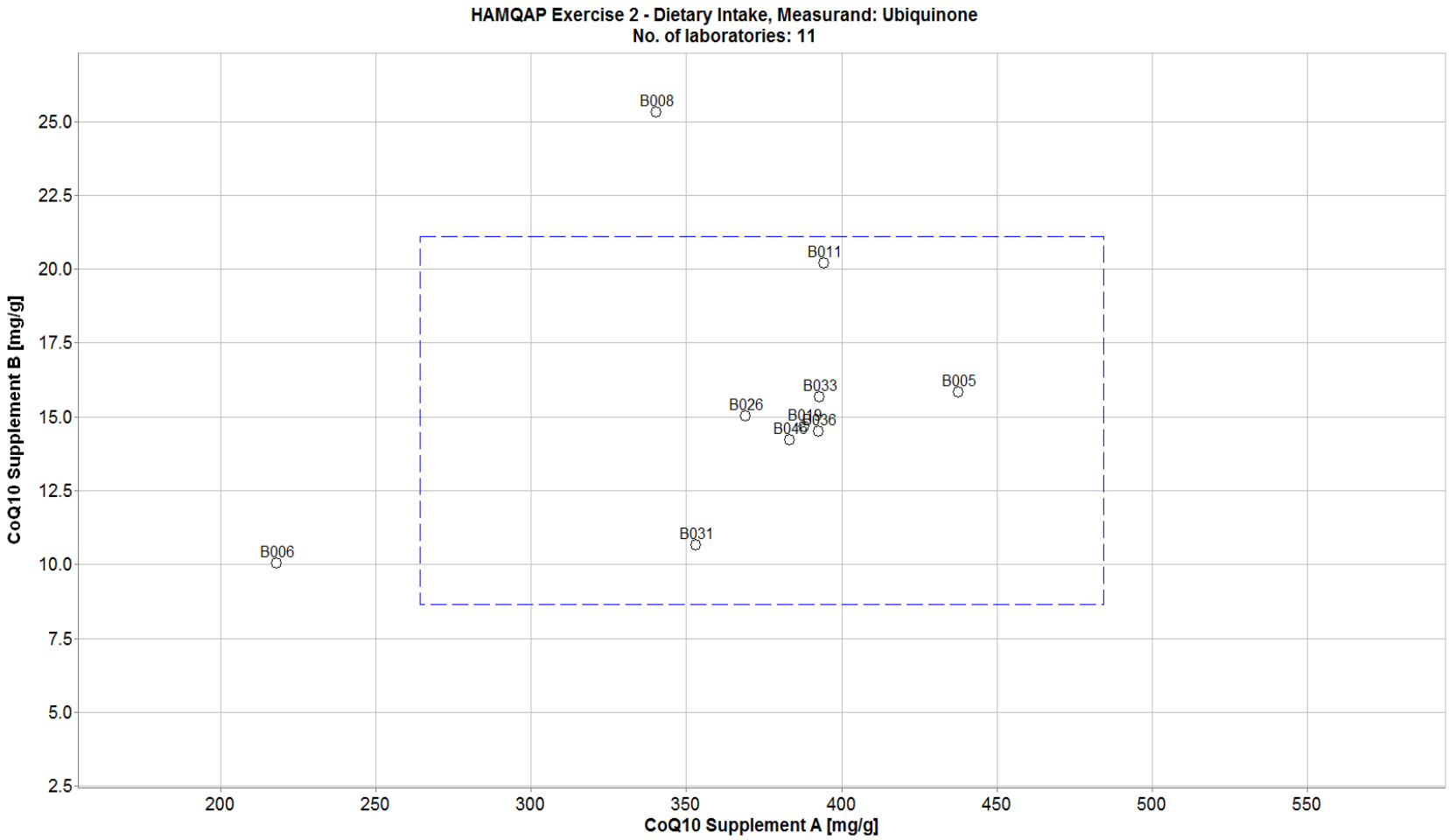

Figure 6-3. Laboratory means for ubiquinone in Supplement A and Supplement B (sample/sample comparison view). In this view, the individual laboratory mean for one sample (Supplement A) is compared to the individual laboratory mean for a second sample (Supplement B). The dotted blue box represents the consensus range of tolerance for Supplement A (x-axis) and Supplement B (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

SECTION 7: BOTANICALS (Eleutherosides)

Study Overview

In this study, participants were provided with two *Eleuthrococcus senticosus* containing dietary supplement samples, Siberian Ginseng Root Extract and Siberian Ginseng Root. Participants were asked to use in-house analytical methods to determine the mass fraction (mg/g) of Eleutheroside A, Eleutheroside B, Eleutheroside C, Eleutheroside D, and Eleutheroside E in each matrix. The purportedly positive health effects attributed to the consumption of *Eleuthrococcus senticosus* supplements include cardioprotective effects, stress reduction, and increased athletic performance.[10] The quality of ginseng products varies significantly and the accurate and precise determination of eleutherosides and ginsenosides will lead to a better understanding of eleutheroside properties, as well as ensuring safety and quality of dietary supplement products.

Dietary Intake Sample Information

Siberian Ginseng Root Extract. Participants were provided with three packets, each containing 1 g of Siberian ginseng root extract powder. The extract material was ground, homogenized, and heat-sealed inside 4 mil polyethylene bags, which were then sealed inside nitrogen-flushed aluminized plastic bags along with two packets of silica gel. Before use, participants were instructed to mix the contents of the packet thoroughly, and to use a sample size appropriate for their usual in-house methods. Participants were asked to store the material at controlled room temperature, 10 °C to 30 °C, and to prepare one sample and report one value from each packet provided. The approximate analyte levels were not reported to participants prior to the study, and target values for eleutherosides in the Siberian Ginseng Root Extract have not been determined.

Siberian Ginseng Root. Participants were provided with three packets, each containing 3 g of powdered Siberian ginseng root powder. The root was ground, homogenized, and heat-sealed inside 4 mil polyethylene bags, which were then sealed inside nitrogen-flushed aluminized plastic bags along with two packets of silica gel. Before use, the contents of each packet should be mixed thoroughly. Participants should use a sample size appropriate for their usual in-house methods. Participants were asked to store the material at controlled room temperature, 10 °C to 30 °C, and to prepare one sample and report one value from each packet provided. The approximate analyte levels were not reported to participants prior to the study, and target values for eleutherosides in the Siberian Ginseng Root have not been determined.

[10] Siberian Ginseng. National Institutes of Health, US National Library of Medicine Medline Plus. https://medlineplus.gov/druginfo/natural/985.html (accessed February 20, 2019).

Dietary Intake Study Results

- Nine to thirteen laboratories enrolled in this exercise and received samples to measure each of the eleutherosides. The table below lists the participation statistics for each eleutheroside.

Analyte	Number of Laboratories Requesting Samples	Number of Laboratories Reporting Results (Percent Participation) Siberian Ginseng Root Extract	Siberian Ginseng Root
Eleutheroside A*	9	1 (11 %)	1 (11 %)
Eleutheroside B	13	7 (54 %)	7 (54 %)
Eleutheroside C*	9	1 (11 %)	1 (11 %)
Eleutheroside D*	9	1 (11 %)	1 (11 %)
Eleutheroside E	13	7 (54 %)	7 (54 %)

*The presence of Eleutheroside A, Eleutheroside C, and Eleutheroside D are not confirmed in these samples. The low reporting rate of these analytes may reflect that they were not detected.

- The between-laboratory variability was acceptable for Eleutheroside B in the Siberian Ginseng Root Extract and in the Siberian Ginseng Root (39 % RSD and 32 % RSD, respectively).
- The between-laboratory variability was good for Eleutheroside E in the Siberian Ginseng Root Extract and in the Siberian Ginseng Root (27 % RSD and 22 % RSD, respectively).
- Five participating laboratories reported the use of LC-absorbance as the analytical method. Remaining laboratories did not report the analytical method used.

Dietary Intake Technical Recommendations

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

- Eleutherosides A, C, and D were not present in the samples at levels above the LOQ for laboratories performing LC-absorbance.
- Eleutherosides B and E were reported by all laboratories returning results.
  - The sample/sample comparison plot comparing the performance of the measurement of Eleutheroside B in the two samples (**Figure 7-3**) demonstrates a linear correlation in the results, which is often indicative of calibration issues.
    - Calibration curves must be linear and include the lowest and highest values expected to be measured in the sample solutions. Extrapolation of the curve may cause incorrect results.
    - The purity of all calibrant materials should be checked carefully using multiple complimentary approaches, and the measured purity should be used to correct concentrations of solutions used for calibration.
  - The sample/sample comparison plot comparing the performance of the measurement of Eleutheroside E in the two samples (**Figure 7-6**) demonstrates randomly scattered results, indicating no systematic bias among the submitted results.
- Laboratories reporting large sample-to-sample variability should investigate the completeness of the extraction during sample preparation.
  - Any extraction procedure should be optimized to determine the most effective extraction solvent to ensure exhaustive extraction of the analyte from the matrix.

    - The optimum number of extraction cycles must be determined by sequential re-extraction of the sample matrix until no further increase in yield is observed. Sequential extractions may be needed if the extraction solvent becomes saturated during the first (or only) extraction cycle.
- Laboratories reporting results flagged as outliers should check for calculation errors. One example is to confirm that factors for all dilutions have been properly tabulated.

Table 7-1. Individualized data summary table (NIST) for eleutherosides in Siberian ginseng samples.

National Institute of Standards & Technology

HAMQAP Exercise 2 - Botanicals

		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*
Eleutheroside A	Siberian Ginseng Root Extract	mg/g						1				
Eleutheroside A	Siberian Ginseng Root	mg/g						1				
Eleutheroside B	Siberian Ginseng Root Extract	mg/g						7	6.4	2.5		
Eleutheroside B	Siberian Ginseng Root	mg/g						7	0.222	0.071		
Eleutheroside C	Siberian Ginseng Root Extract	mg/g						1				
Eleutheroside C	Siberian Ginseng Root	mg/g						1				
Eleutheroside D	Siberian Ginseng Root Extract	mg/g						1				
Eleutheroside D	Siberian Ginseng Root	mg/g						1				
Eleutheroside E	Siberian Ginseng Root Extract	mg/g						7	4.9	1.3		
Eleutheroside E	Siberian Ginseng Root	mg/g						7	0.49	0.11		

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. Individualized data summary table (NIST) for Eleutheroside B in Siberian ginseng samples. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		Eleutheroside B									
		Siberian Ginseng Root Extract (mg/g)					Siberian Ginseng Root (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target										
	B002										
	B006	8.85	8.50	8.74	8.70	0.18	0.666	0.675	0.671	0.671	0.005
	B007										
	B009	5.47	5.34	5.13	5.31	0.17	0.232	0.211	0.222	0.222	0.011
	B011	5.82	5.76	5.75	5.78	0.04	0.249	0.245	0.237	0.244	0.006
	B012										
	B026										
	B029	5.82	5.82	5.80	5.81	0.01	0.230	0.230	0.240	0.233	0.006
	B030										
	B031	4.20	4.07	4.14	4.14	0.06	0.159	0.160	0.149	0.156	0.006
	B033	15.77	15.87	16.16	15.93	0.20	0.550	0.560	0.560	0.557	0.006
	B038	6.80	6.86	6.87	6.84	0.04	0.255	0.255	0.253	0.254	0.001
	B039										
Community Results		Consensus Mean			6.36		Consensus Mean			0.222	
		Consensus Standard Deviation			2.48		Consensus Standard Deviation			0.071	
		Maximum			15.93		Maximum			0.671	
		Minimum			4.14		Minimum			0.156	
		N			7		N			7	

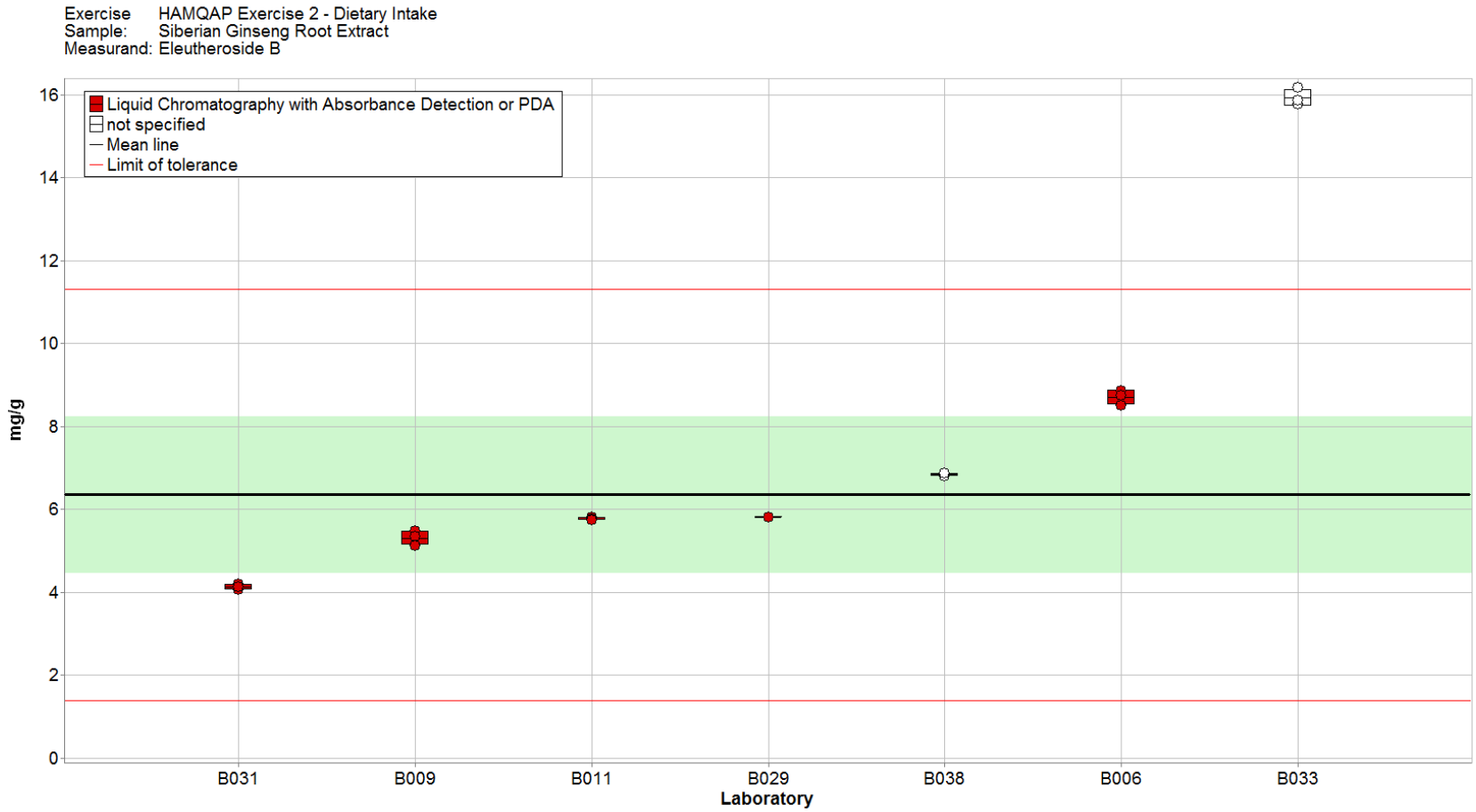

Figure 7-1. Eleutheroside B in Siberian Ginseng Root Extract (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

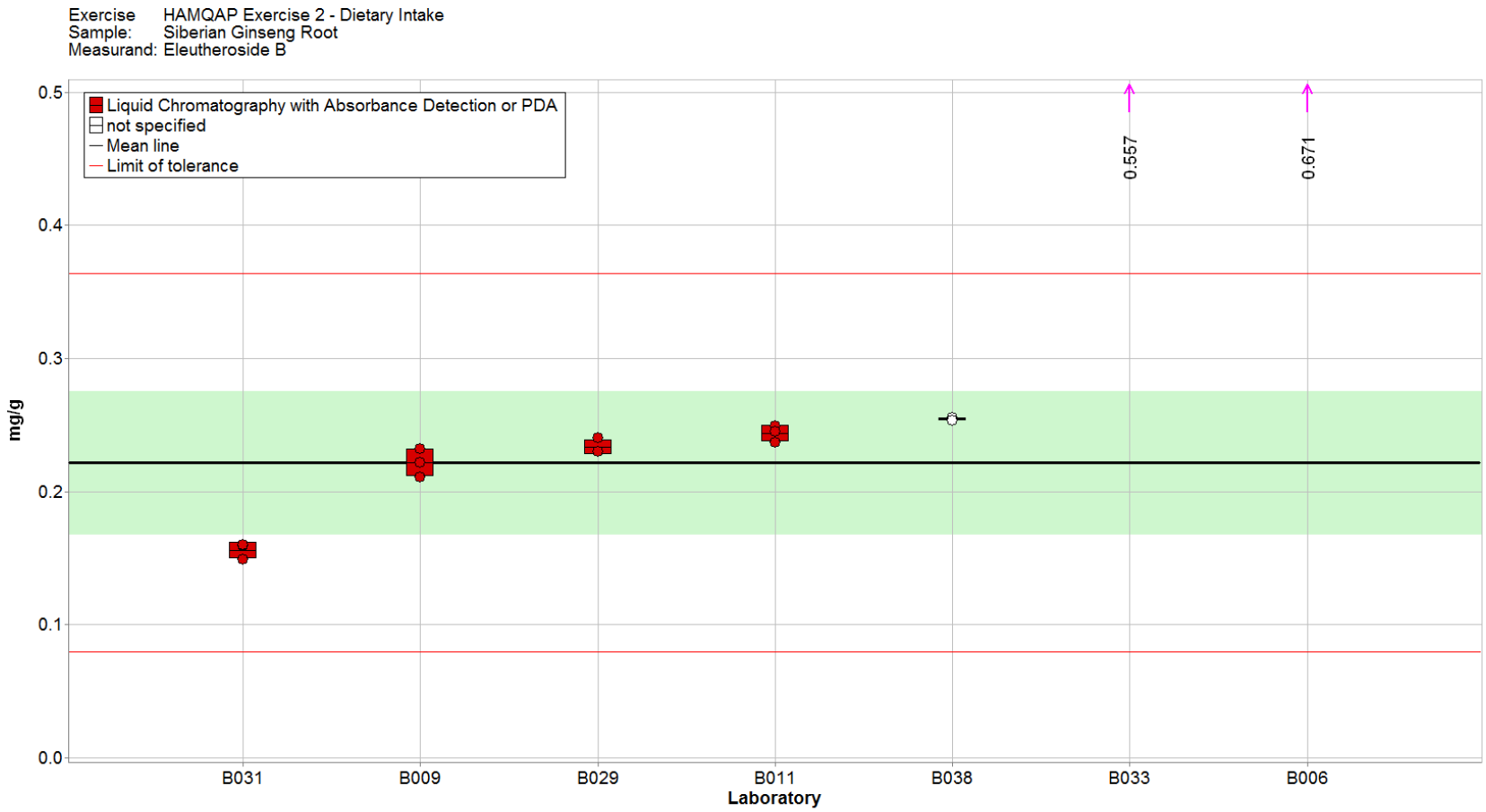

Figure 7-2. Eleutheroside B in Siberian Ginseng Root (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

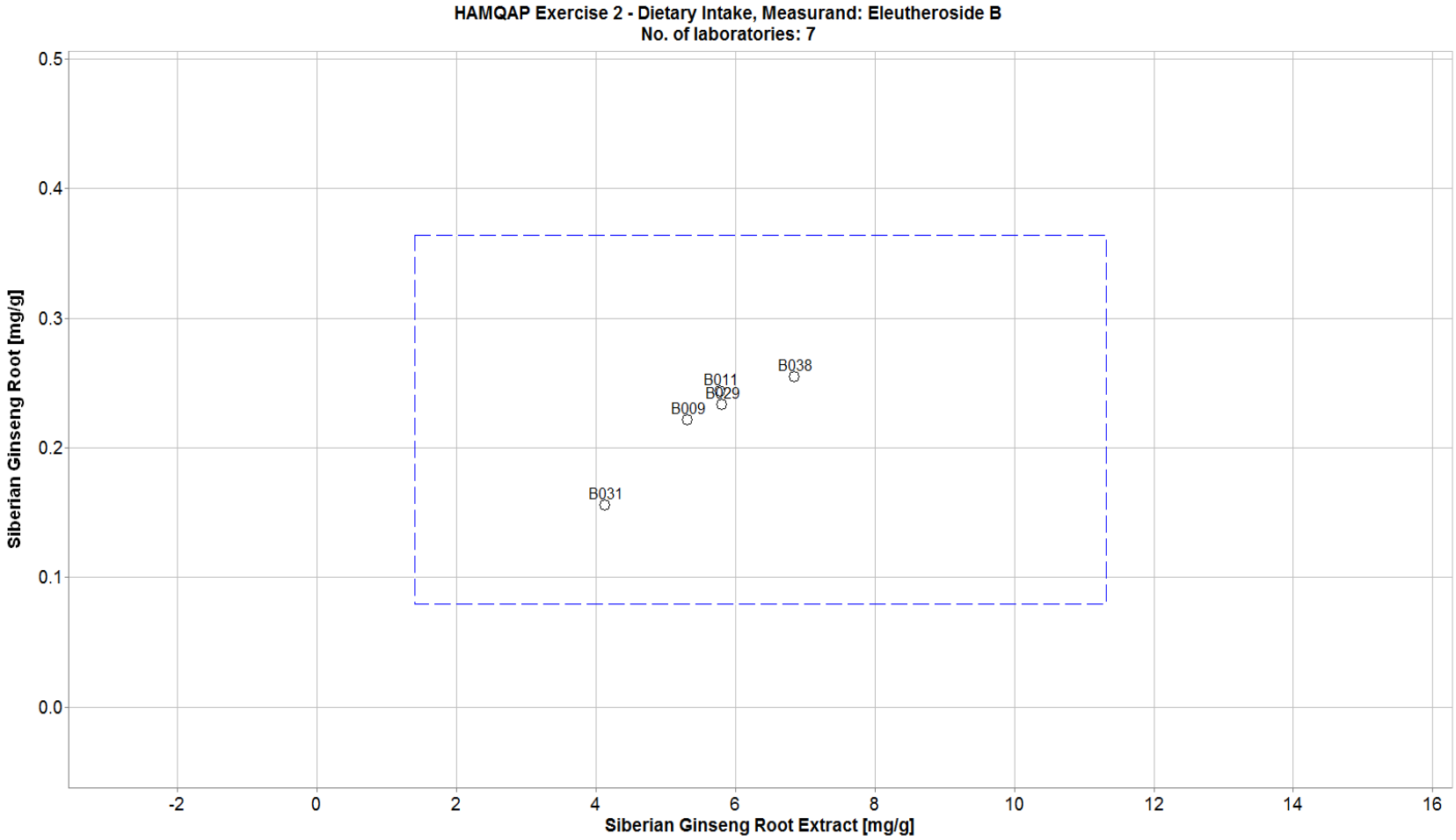

Figure 7-3. Laboratory means for Eleutheroside B in Siberian Ginseng Root Extract and Siberian Ginseng Root (sample/sample comparison view). In this view, the individual laboratory mean for one sample (Siberian Ginseng Root Extract) is compared to the individual laboratory mean for a second sample (Siberian Ginseng Root). The dotted blue box represents the consensus range of tolerance for Siberian Ginseng Root Extract (x axis) and Siberian Ginseng Root (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 7-3. Individualized data summary table (NIST) for Eleutheroside E in Siberian ginseng samples. Data points highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

		Eleutheroside E									
		Siberian Ginseng Root Extract (mg/g)					Siberian Ginseng Root (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target										
	B002										
	B006	5.49	5.28	5.33	5.37	0.11	0.342	0.342	0.345	0.343	0.002
	B007										
	B009	4.05	3.94	3.74	3.91	0.16	0.474	0.460	0.470	0.468	0.007
	B011	5.92	5.80	5.77	5.83	0.08	0.546	0.559	0.557	0.554	0.007
	B012										
	B026										
	B029	5.07	5.02	5.01	5.03	0.03	0.650	0.610	0.470	0.577	0.095
	B030										
	B031	4.60	4.47	4.54	4.54	0.06	0.519	0.513	0.502	0.511	0.009
	B033	18.44	18.52	18.74	18.57	0.16	1.750	1.850	1.880	1.827	0.068
	B038	4.66	4.66	4.68	4.67	0.01	0.515	0.518	0.508	0.514	0.005
	B039										
Community Results		Consensus Mean			4.89		Consensus Mean			0.494	
		Consensus Standard Deviation			1.33		Consensus Standard Deviation			0.111	
		Maximum			18.57		Maximum			1.827	
		Minimum			3.91		Minimum			0.343	
		N			7		N			7	

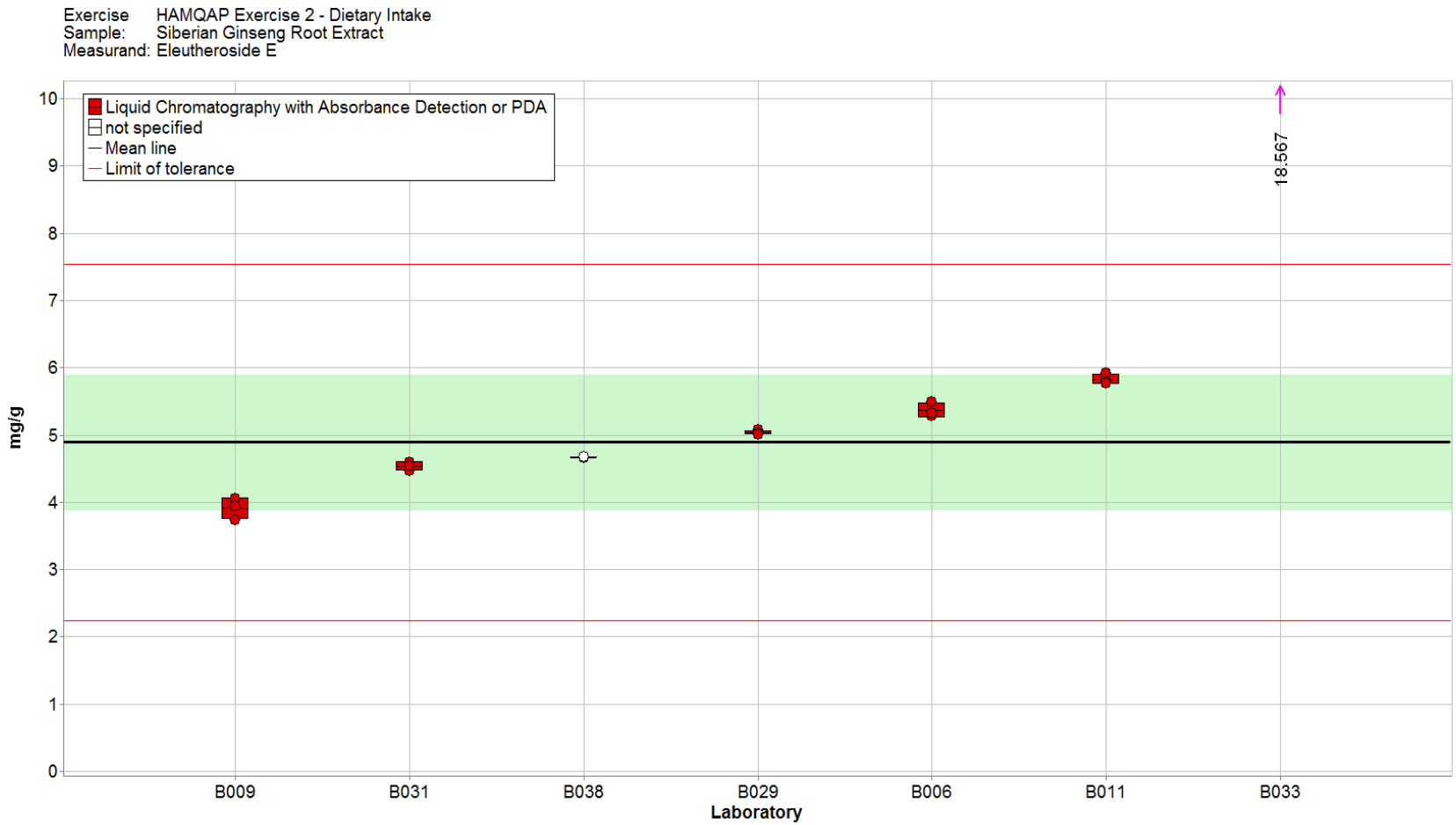

Figure 7-4. Eleutheroside E in Siberian Ginseng Root Extract (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

Figure 7-5. Eleutheroside E in Siberian Ginseng Root (data summary view – analytical method). In this view, individual laboratory data are plotted (circles) with the individual laboratory standard deviation (rectangle). The solid black line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper 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 NIST value has not been determined in this material.

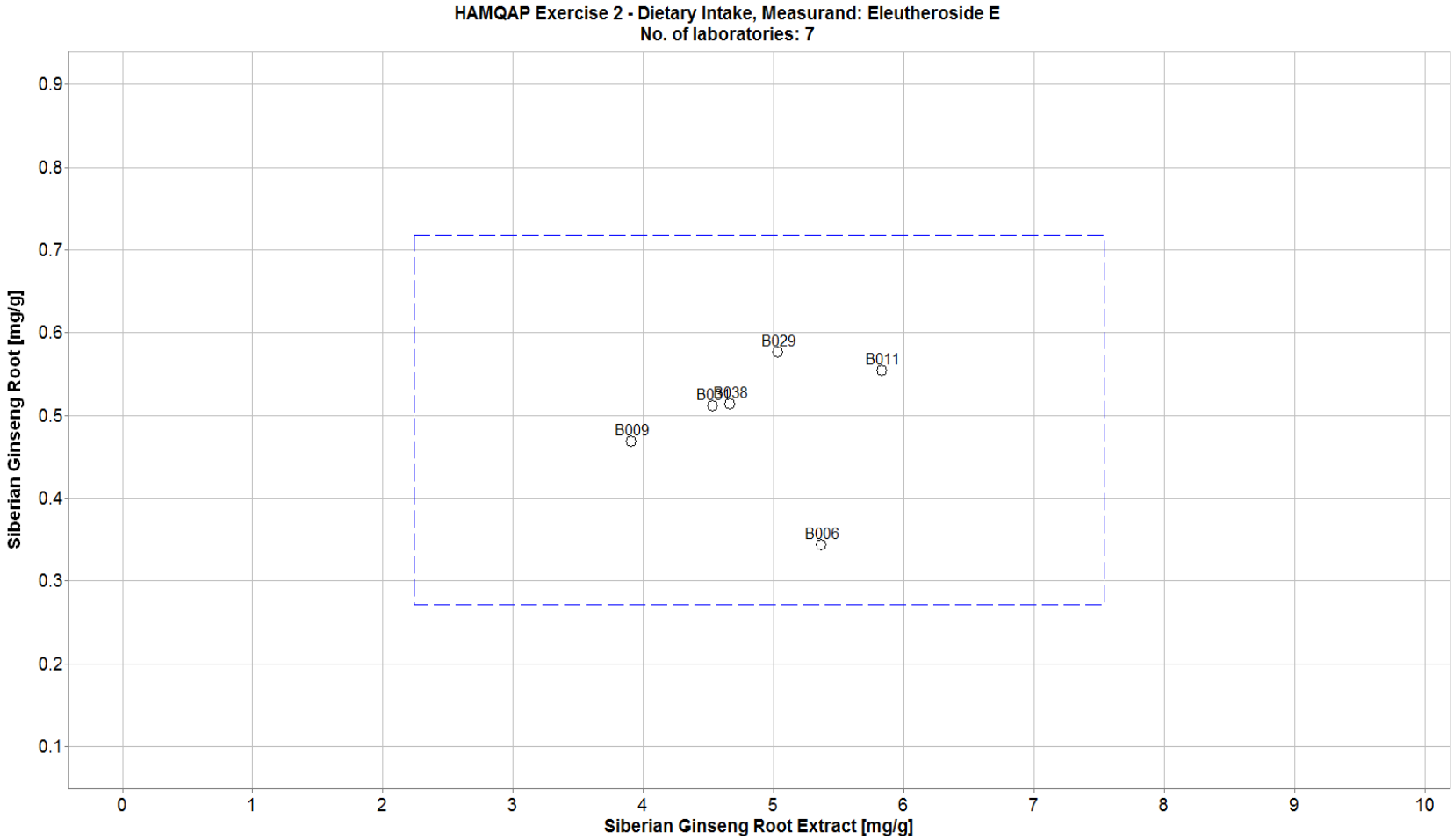

Figure 7-6. Laboratory means for Eleutheroside E in Siberian Ginseng Root Extract and Siberian Ginseng Root (sample/sample comparison view). In this view, the individual laboratory mean for one sample (Siberian Ginseng Root Extract) is compared to the individual laboratory mean for a second sample (Siberian Ginseng Root). The dotted blue box represents the consensus range of tolerance for Siberian Ginseng Root Extract (x axis) and Siberian Ginseng Root (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.