NIST IR 8163

Dietary Supplement Laboratory Quality Assurance Program: Exercise G Final Report

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National Institute of Standards and Technology
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ABSTRACT

The NIST Dietary Supplement Laboratory Quality Assurance Program (DSQAP) was established in collaboration with the National Institutes of Health (NIH) Office of Dietary Supplements (ODS) in 2007 to enable members of the dietary supplements community to improve the accuracy of measurements made in compliance with various regulations including the dietary supplement current good manufacturing practices (cGMPs). Exercise G of this program offered the opportunity for laboratories to assess their in-house measurements of nutritional elements (Na), contaminants (Pb), water-soluble vitamins (folic acid), fat-soluble vitamins (ß-carotene), and anthocyanins in foods and/or botanical dietary supplement ingredients and finished products.

INTRODUCTION

The dietary supplement industry in the U.S. is booming, with two-thirds of adults considering themselves to be supplement users.[1] Consumption of dietary supplements, which includes vitamin and mineral supplements, represents an annual US expenditure of more than $25 billion. These figures represent an increasing American trend, and as a result, it is critically important that both the quality and safety of these products are verified and maintained.

The Dietary Supplement Health and Education Act of 1994 (DSHEA) amended the Federal Food, Drug and Cosmetic Act to create the regulatory category called dietary supplements. The DSHEA also gave the FDA authority to write current Good Manufacturing Practices (cGMPs) that require manufacturers to evaluate the identity, purity, and composition of their ingredients and finished products. In addition, the DSHEA authorized the establishment of the Office of Dietary Supplements at the National Institutes of Health (NIH ODS). To enable members of the dietary supplements community to improve the accuracy of the measurements made in compliance with these and other regulations, NIST established the Dietary Supplement Laboratory Quality Assurance Program (DSQAP) in collaboration with the NIH ODS in 2007.

The program offers the opportunity for laboratories to assess their in-house measurements of active or marker compounds, nutritional elements, contaminants (toxic elements, pesticides, mycotoxins), and fat- and water-soluble vitamins in foods as well as botanical dietary supplement ingredients and finished products. Reports and certificates of participation are provided and can be used to demonstrate compliance with the cGMPs. In addition, NIST and the DSQAP 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 DSQAP exercises could be used by ODS to identify problematic matrices and analytes for which an AOAC INTERNATIONAL Official Method of Analysis would benefit the dietary supplement community

NIST has experience in the area of quality assurance programs, but the DSQAP takes a unique approach. In other NIST quality assurance programs, a set of analytes is measured repeatedly over time in the same or similar matrices to demonstrate laboratory performance. In contrast, the wide range of matrices and analytes under the “dietary supplement” umbrella means that not every

[1] Walsh, T. (2012) *Supplement Usage, Consumer Confidence Remain Steady According to New Annual Survey from CRN*. Council for Responsible Nutrition, Washington, DC.

laboratory is interested in every sample or analyte. The constantly changing dietary supplement market, and the enormous diversity of finished products, makes repeated determinations of a few target compounds in a single matrix of little use to participants. Instead, participating laboratories are interested in testing in-house methods on a wide variety of challenging, real-world matrices to demonstrate that their performance is comparable to that of the community. In an area where there are few standard methods, the DSQAP offers a unique tool for assessment of the quality of measurements, provides feedback about performance, and can assist participants in improving laboratory operations.

This report contains the results from the seventh exercise of the DSQAP, Exercise G. Seventy-eight laboratories responded to the call for participants distributed in May 2011. Samples were shipped to participants in July 2011, and results were returned to NIST by October 2011. The information and data contained in this report was disseminated to the participants in June 2011.

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 graphs 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 graphs 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. The consensus mean and standard deviation are calculated according to the robust algorithm outlined in ISO 13528:2005(E), Annex C.[2] The algorithm is summarized here in simplified form.

Initial values of the consensus mean, x^*, and consensus standard deviation, s^*, are estimated as

$$x^* = \text{median of } x_i \qquad (i = 1, 2, \ldots, n)$$
$$s^* = 1.483 \times \text{median of } |x_i - x^*| \qquad (i = 1, 2, \ldots, n).$$

These initial values for x^* and s^* are updated by first calculating the expanded standard deviation, δ, as

$$\delta = 1.5 \times s^*.$$

Then each x_i is compared to the expanded range and adjusted to x_i^* as described below to reduce the effect of outliers.

If $x_i < x^* - \delta$, then $x_i^* = x^* - \delta$.
If $x_i > x^* + \delta$, then $x_i^* = x^* + \delta$.

[2] ISO 13528:2005(E), *Statistical methods for use in proficiency testing by interlaboratory comparisons*, pp. 14-15.

Otherwise, $x_i^* = x_i$.

New values of x^*, s^*, and δ are calculated iteratively until the process converges. Convergence is taken as no change from one iteration to the next in the third significant figure of s^* and in the equivalent digit in x^*:

$$x^* = \frac{\sum_{i=1}^{n} x_i^*}{n}$$

$$s^* = 1.134 \times \sqrt{\frac{\sum_{i=1}^{n} (x_i^* - x^*)}{n-1}}.$$

Individualized Data Table

The data in this table is individualized to each participating laboratory and is provided to allow participants to directly compare their data to the summary statistics (consensus or community data as well as NIST certified, reference, or estimated values). The upper left of the data table includes the randomized laboratory code. Tables included in this report are generated using NIST data to protect the identity and performance of participants.

Section 1 of the data table 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 a particular analyte or matrix. An empty box for standard deviation indicates that only a single value was reported and therefore that value was not included in the calculation of the consensus data.[2]

Also in Section 1 are two Z-scores. The first Z-score, Z_{comm}, is calculated with respect to the community consensus value, using x* and s*:

$$Z_{comm} = \frac{x_i - x^*}{s^*}.$$

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

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

or

$$Z_{NIST} = \frac{x_i - x_{NIST}}{s_{NIST}}.$$

The significance of the 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 contains the community results, including the number of laboratories reporting more than a single value for a given analyte[1], 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.[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 contains the target values for each analyte. When possible, the target value is a certified or reference value determined at NIST. Certified values and the associated expanded uncertainty (U_{95}) have been determined with two independent analytical methods at NIST, by collaborating laboratories, or in some combination. Reference values are assigned using NIST values obtained from the average and standard deviation of measurements made using a single analytical method 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 are measured at NIST using an appropriate method. The NIST-assessed value represents the mean of at least three replicates. For materials acquired from another proficiency testing program, the consensus value and uncertainty from the completed round is used as the target range.

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 a single laboratory to data reported by the other participating laboratories or to the consensus data. A blank indicates that the laboratory signed up and received samples for that particular analyte and matrix, but NIST does not have data on file for that laboratory.

Graphs

Data Summary View (Method Comparison Data Summary View)

In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). Data points that are unfilled represent laboratories that reported a single value for that analyte and therefore were not included in the consensus mean. The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean. 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. The gray shaded region represents the target zone for "acceptable" performance, which encompasses the NIST certified, reference, or estimated value bounded by twice its uncertainty (U_{95}) or standard deviation. For the purpose of the DSQAP, a target range spanning twice the uncertainty in the NIST value is selected because participants are only asked to make a limited number of

[3] ISO 13528:2005(E), *Statistical methods for use in proficiency testing by interlaboratory comparisons*, Annex C.

observations. The size of the y-axis on the data summary view graph represents the consensus mean bounded by 2δ. 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 SRM with a certified or reference value) are compared to the results for another sample (another NIST SRM with a more challenging matrix, a commercial sample, etc.). The error bars represent the individual laboratory standard deviation. 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 zero and twice the consensus mean. 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. This view emphasizes 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.

NUTRITIONAL ELEMENTS (Na) IN POWDERED MATRICES

Study Overview

In this study, participants were provided with two NIST SRMs, SRM 1573a Tomato Leaves and SRM 3281 Cranberry (Fruit). Participants were asked to use in-house analytical methods to determine the mass fraction of sodium in each matrix and report values on an as-received basis.

Sample Information

Tomato Leaves. Participants were provided with one bottle containing approximately 50 g of dried, powdered tomato leaves. The material was prepared from tomato leaves collected in Pennsylvania that had been picked from an untreated area, rinsed, then dried at 60 °C to 70 °C. The dried leaves were ground and sieved prior to packaging. Before use, participants were instructed to thoroughly mix the contents of the bottle and use a sample size of at least 0.5 g. Participants were asked to store the material at controlled room temperature, 20 °C to 25 °C, and to prepare three samples and report three values from the single bottle provided. Prior to the study, the approximate analyte level was given as 100 mg/kg to 200 mg/kg. The certified value for sodium in SRM 1573a was determined at NIST using instrumental neutron activation analysis (INAA) and flame atomic emission spectrometry (FAES). The certified values and uncertainties for Na are provided in the table below, both on a dry-mass basis and on an as-received basis accounting for moisture of the material (0.97 %).

	Certified Mass Fraction in SRM 1573a (mg/kg)	
	(dry-mass basis)	(as-received basis)
Sodium (Na)	136 ± 4	132 ± 4

Cranberries. Participants were provided with one packet containing approximately 6 g of freeze-dried, powdered cranberries. The cranberry powder was blended, aliquotted, 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 thoroughly mix the contents of each packet and use a sample size of at least 0.5 g. Participants were asked to store the material at controlled room temperature, 20 °C to 25 °C, and to prepare three samples and report three values from the single packet provided. Prior to the study, the approximate analyte level was given as 200 mg/kg to 300 mg/kg. The reference value for sodium in SRM 3281 Cranberry (Fruit) was determined at NIST using inductively coupled plasma optical emission spectroscopy (ICP-OES). The reference values and uncertainties for Na are provided in the table below, both on a dry-mass basis and on an as-received basis accounting for moisture of the material (2.39 %).

	Reference Mass Fraction in SRM 3281 (mg/kg)	
	(dry-mass basis)	(as-received basis)
Sodium (Na)	259 ± 3	253 ± 3

Study Results

- Thirty-seven laboratories enrolled in this study and received samples. Twenty-four laboratories reported results for both the samples (65 % participation).

- For both samples, the consensus ranges were wide but encompassed the NIST target ranges.
  - The consensus mean for sodium in tomato leaves was on the upper edge of the target range.
  - The consensus mean for sodium in the cranberries was above the target range.
- The between-laboratory variability for was high in both materials (29 % and 28 % RSD for the tomato leaves and cranberries, respectively).
- A majority of the laboratories reported using either open-beaker digestion (48 %) or microwave digestion (32 % to 36 %) for sample preparation. The remaining laboratories reported using hot block digestion, dry ashing, or dilution. One laboratory did not report the type of sample preparation technique that was used.
- A majority of the laboratories reported using either ICP-OES (56 %) or ICP-MS (28 %) as their analytical method. Two laboratories reported using atomic absorption spectroscopy, and one laboratory reported using ion chromatography with conductivity detection. One laboratory did not report the type of analytical technique that was used.
- A majority of the laboratories reported using an external standard approach to calibration (88 %). One laboratory reported using a standard addition approach, and one laboratory reported using an internal standard approach. One laboratory did not report the type of calibration approach that was used.

Technical Recommendations

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

- A significant difference was apparent between results obtained using open beaker and those using microwave digestion. Too few results were reported by other methods to identify any additional trends.
  - As shown in **Figure 1** and **Figure 2**, laboratories that reported high sodium values also reported using open beaker digestion. This trend may indicate contamination of samples with sodium from the environment during digestion.
    - To minimize contamination, work areas should be cleaned prior to sample preparation, glassware should be cleaned by acid wash prior to use, and exposure of samples to the laboratory environment should be limited.
    - If a soap solution is used for cleaning, sodium levels in blank solutions must be checked, as some soaps will give high sodium blank levels.
    - Extra procedural reagent blanks should be prepared along with samples to know the extent of sodium contamination from the analysis.
- No difference was apparent in the sodium results based on analytical method used (ICP-OES or ICP-MS). Too few results were reported by other methods to identify any additional trends.
- Additional sources for potential error in the final results may be errors in calibration and dilution.
- Samples with very low sodium levels may be inadvertently diluted below the calibration range and possibly out of the quantification or detection range of the instrument. Care should be taken to evaluate a more concentrated sample if the signal appears to be lower than expected or is below the lowest calibration point.

- Many of the calibration curves reported by participants extended far above the reported working range of sodium in the solutions to be analyzed. While the calibration curve may appear linear over a wide concentration range, a measured value near an end of the calibration curve may have significant bias. For the most accurate results, use calibration points closely surrounding the expected solution concentration, and ensure that the calibration curve is linear along the region of your expected solution concentrations.
  - The sample/sample comparison graph does not reflect an obvious calibration error. Some laboratories reported values that were high for one sample but were within range for the second sample. This type of trend may indicate individualized problems with the digestion of one matrix compared to another.
  - Quality assurance samples should always be used. These can be commercially available reference materials (CRMs, SRMs, or RMs) or prepared in-house, but need to be of known concentration.
    - They are used to ensure that the method is performing as expected.
    - They are useful in finding where errors are occurring, including calculation errors.
    - After checking for calculation errors, make sure results are reported correctly.

Table 1. Individual data table (NIST) for sodium in foods and dietary supplements.

National Institute of Standards & Technology

Exercise G - July 2011 - Nutritional Elements

	Lab Code:	**NIST**	**1. Your Results**				**2. Community Results**			**3. Target Value**	
Analyte	Sample	Units	Mean	s_{total}	Z_{comm}	Z_{NIST}	N	Mean	Std Dev	Value	U_{95}
Na	Tomato Leaves	mg/kg	132	3.9	-0.1	0.1	24	134	40	132	3.9
Na	Cranberry (Fruit)	mg/kg	253	2.9	-0.2	0.1	24	265	73	253	2.9

Mean	Average of reported values
s_{total}	Standard deviation of reported values
Z_{comm}	Z-score: (Lab Mean - Consensus Mean)/ Consensus Standard Deviation
Z_{NIST}	Z-score: (Lab Mean - NIST Value or Label Claim)/ NIST or Label Claim Standard Deviation
NR	No data reported

N	Number of quantitative values reported
Mean	Robust mean of the reported values
Std Dev	Robust standard deviation
Value	NIST-assessed value
U_{95}	±95% confidence interval about the assessed value

Table 2. Data summary table for sodium in foods and dietary supplements.

		Sodium									
		SRM 1573a Tomato Leaves (mg/kg)					SRM 3281 Cranberry (Fruit) (mg/kg)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	NIST				132	4				253	3
	G701	80	79	77	79	1	210	215	215	213	3
	G703	113	106	111	110	4	139	146	136	140	5
	G704										
	G709										
	G710	112	113	116	114	2	233	234	240	236	4
	G711	103	106	103	104	1	214	214	148	192	38
	G714	151	140	133	142	9	231	223	243	232	10
	G720										
	G721	707	391	452	517	167	911	902	889	901	11
	G723	119	116	118	117	2	232	230	224	229	4
	G724	130	131	142	134	7	260	252	248	253	6
	G728										
	G729										
	G734	219	220	218	219	1	572	573	554	566	11
	G735	157	162	166	162	5	418	410	432	420	11
	G736	194	181	187	187	6	403	436	416	418	17
	G737	163	163	169	165	3	502	491	485	493	9
	G738	138	138	139	138	1					
	G739										
	G741	107	109	108	108	1	240	238	239	239	1
	G742										
	G743	180	175	200	185	13	299	286	296	294	7
	G747	305	245	271	274	30	228	229	225	227	2
	G749	116	130		123	9	294	314		304	14
	G752	122	122	117	120	3	287	257	263	269	16
	G754										
	G755										
	G756						260	233	246	246	14
	G757	111	113	110	111	2	223	232	230	228	5
	G758										
	G759	122	122	118	121	2	263	262	265	263	1
	G761										
	G762	113	113	112	113	1	237	237	238	237	1
	G763	105	100	102	102	2	230	232	230	231	1
	G764										
	G770	128	123	120	124	4	183	184	181	183	1
	G778										
Community Results		Consensus Mean			136		Consensus Mean			268	
		Consensus Standard Deviation			39		Consensus Standard Deviation			83	
		Maximum			517		Maximum			901	
		Minimum			79		Minimum			140	
		N			23		N			23	

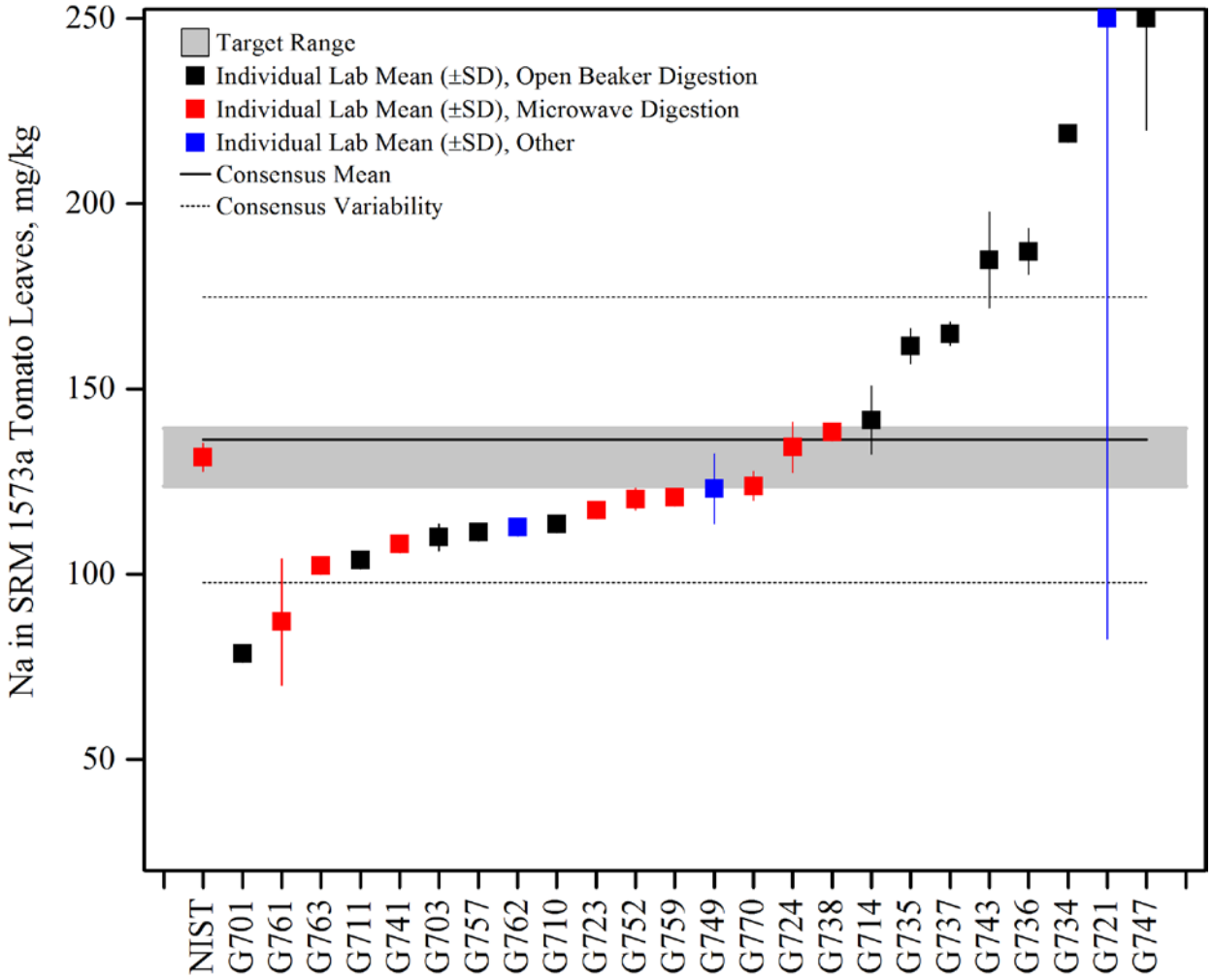

Figure 1. Sodium in SRM 1573a Tomato Leaves (data summary view – digestion method). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The color of the data point represents the sample preparation (digestion) procedure employed. The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean. The gray shaded region represents the target zone for "acceptable" performance, which encompasses the NIST reference value bounded by twice its uncertainty (U_{95}).

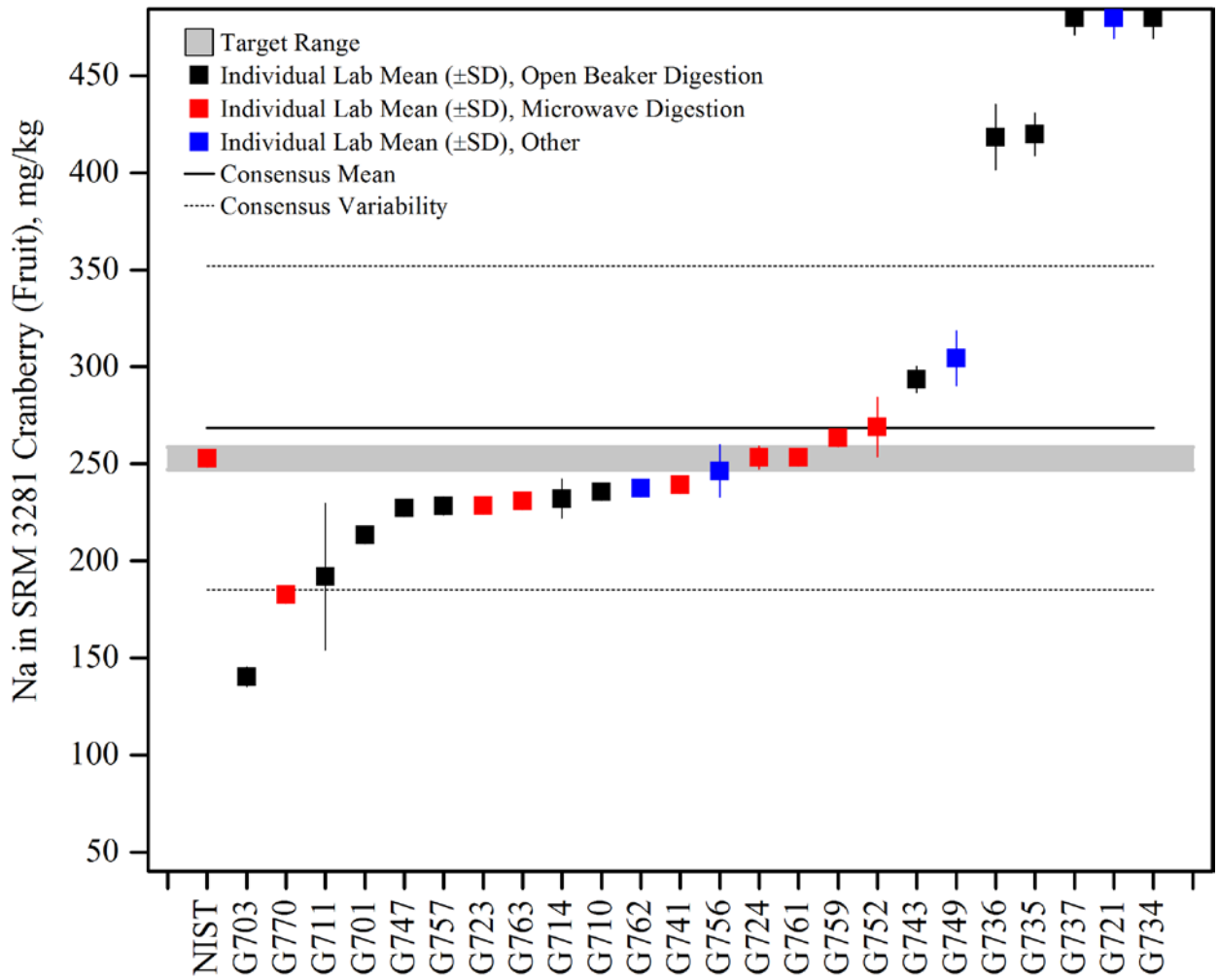

Figure 2. Sodium in SRM 3281 Cranberry (Fruit) (data summary view – digestion method). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The data are identified by digestion method in this graph. The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean. The gray shaded region represents the target zone for "acceptable" performance, which encompasses the NIST certified value bounded by twice its uncertainty (U_{95}).

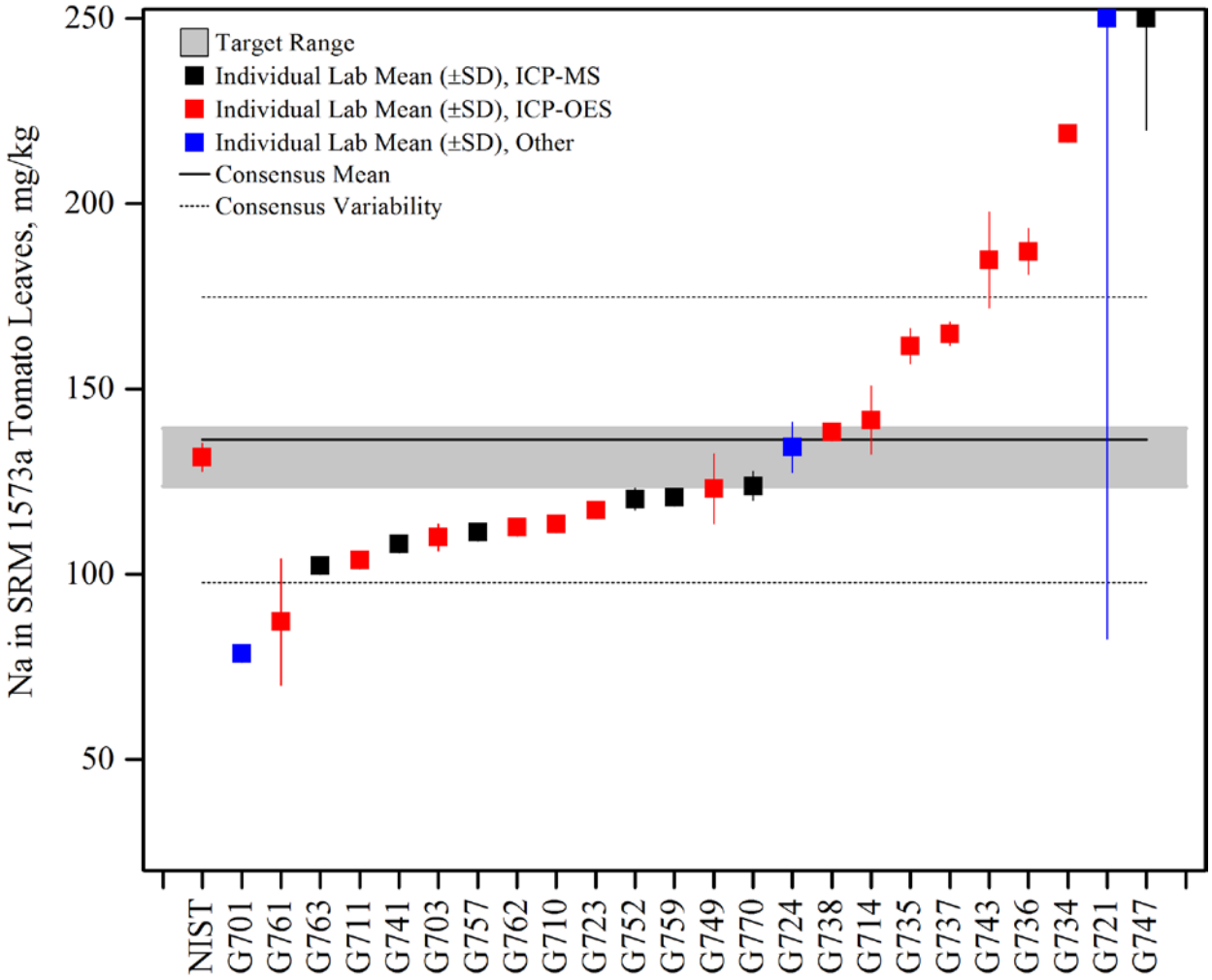

Figure 3. Sodium in SRM 1573a Tomato Leaves (data summary view – instrumental methods). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The data are identified by instrumental method in this graph. The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean. The gray shaded region represents the target zone for "acceptable" performance, which encompasses the NIST certified value bounded twice by its uncertainty (U_{95}).

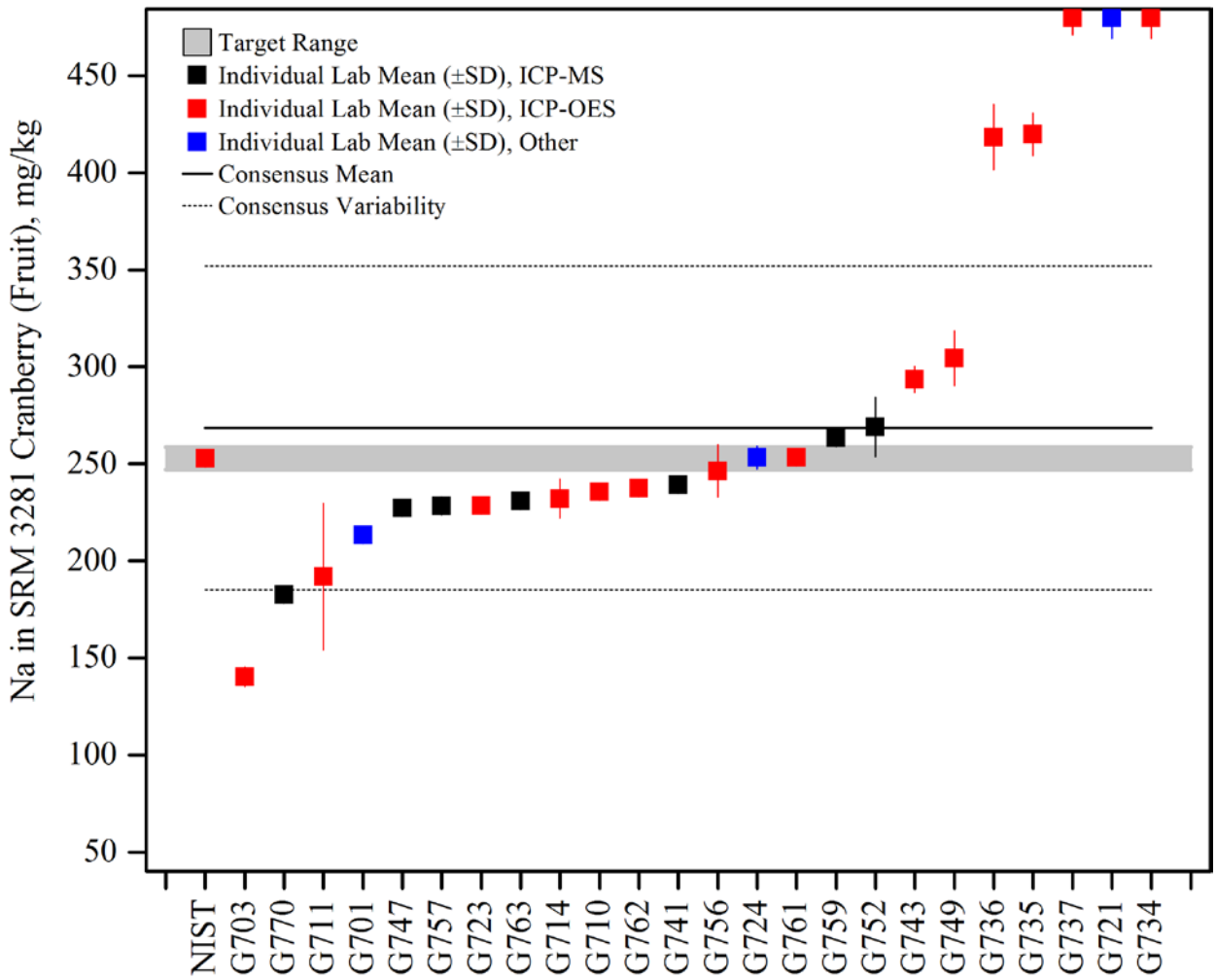

Figure 4. Sodium in SRM 3281 Cranberry (Fruit) (data summary view – instrumental methods). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The data are identified by instrumental method in this graph. The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean. The gray shaded region represents the target zone for "acceptable" performance, which encompasses the NIST certified value bounded by twice its uncertainty (U_{95}).

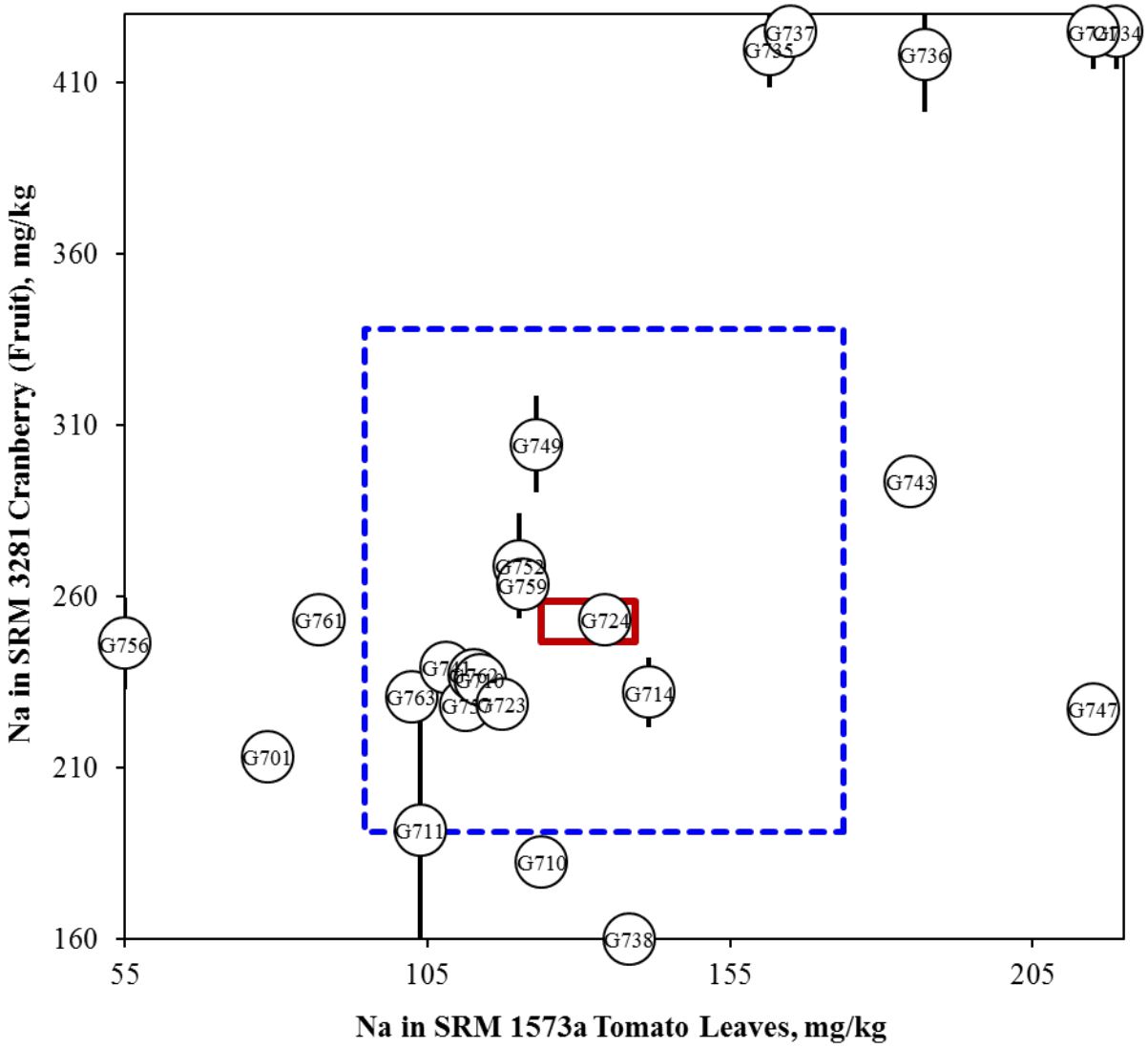

Figure 5. Sodium in SRM 1573a Tomato Leaves and SRM 3281 Cranberry (Fruit) (sample/sample comparison view). In this view, the individual laboratory results for one sample (tomato leaves) are compared to the results for a second sample (cranberry). The solid red box represents the target zone for the two samples, tomato leaves (x-axis) and cranberry (y-axis). The dotted blue box represents the consensus zone for tomato leaves (x-axis) and cranberry (y-axis).

TOXIC ELEMENTS (Pb) IN DIETARY SUPPLEMENTS

Study Overview

In this study, participants were provided with two NIST SRMs, SRM 3243 Ephedra-Containing Solid Oral Dosage Form and SRM 3280 Multivitamin/Multielement Tablets. Participants were asked to use in-house analytical methods to determine the mass fractions of lead (Pb) in each of the matrices and report values on an as-received basis.

Sample Information

Ephedra-Containing Tablets. Participants were provided with three bottles containing approximately 2.5 g of dried, powdered ephedra-containing tablets. The ephedra tablets were ground, homogenized, and packaged in amber high-density polyethylene bottles. Before use, participants were asked to thoroughly mix the contents of the bottles and use a sample size of at least 1.0 g. Participants were asked to store the material at controlled room temperature, 20 °C to 25 °C, and to prepare one sample and report one value from each bottle provided. Prior to the study, the approximate analyte level was given as 600 ng/g to 800 ng/g. The certified value for lead in SRM 3243 was determined at NIST using isotope dilution inductively coupled plasma mass spectrometry (ID-ICP-MS), in combination with data from two collaborating laboratories. The certified value and uncertainty for Pb are provided in the table below, both on a dry-mass basis and on an as-received basis accounting for moisture of the material (4.63 %).

	Certified Mass Fraction in SRM 3243 (ng/g)	
Analyte	(dry-mass basis)	(as-received basis)
Lead (Pb)	692 ± 56	660 ± 53

Multivitamin/Multielement Tablets. Participants were provided with three vials containing approximately 2.5 g of ground multivitamin/multielement tablets. The multivitamin/multielement tablets were ground, sieved, and packaged in amber glass vials. Before use, participants were asked to thoroughly mix the contents of the vial and to use a sample size of at least 0.25 g. Participants were asked to store the material at controlled room temperature, 20 °C to 25 °C, and to prepare one sample and report one value from each of the vials provided. Prior to the study, the approximate analyte level was given as 200 ng/g to 300 ng/g. The certified value for lead in SRM 3280 was determined at NIST using isotope dilution inductively coupled plasma mass spectrometry (ID-ICP-MS). The certified values and uncertainties are reported in the table below, both on a dry-mass basis and on an as-received basis accounting for moisture of the material (1.37 %).

	Certified Mass Fraction in SRM 3280 (ng/g)	
Analyte	(dry-mass basis)	(as-received basis)
Lead (Pb)	273 ± 2	269 ± 2

Study Results

- Forty-eight laboratories enrolled in this exercise and received samples. Thirty-six laboratories reported results for lead in Ephedra tablets (75 % participation). Thirty-seven laboratories reported results for lead in multivitamin tablets (77 % participation).
- The consensus means for lead in both matrices were within the target ranges. The between-laboratory variability for lead in the Ephedra tablets was acceptable (13 % RSD), but variability for lead in the multivitamin tablets was high (23 % RSD).
- A majority of the laboratories reported using microwave digestion (58 %) or open beaker digestion (36 %) for sample preparation. Hot block digestion was also reported as a method of sample preparation by two laboratories (6 %). One laboratory did not report the sample preparation technique used.
- Most laboratories reported using ICP-MS as their analytical method for analysis (78 %). Laboratories also reported using ICP-OES (17 %) and AAS (6 %). One laboratory did not report the analytical method used.
- A majority of the laboratories reported using an external standard approach to calibration (89 %). One laboratory reported using a standard addition approach (6 %), and one laboratory reported using an internal standard approach (6 %). One laboratory did not report the type of calibration approach that was used.

Technical Recommendations

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

- Lead is generally easily digested, and digestion with HNO_3 is recommended. Digestion with HCl may form a $PbCl_2$ precipitate that is difficult to dissolve. While the sample solution may look clear, results may be biased low if solid $PbCl_2$ remains. The Cl can be removed by repeatedly drying the sample solution using HNO_3.
- Some laboratories reported high values for one sample, but not both, which may indicate more difficulty in digestion of one matrix over the other. Because each matrix is different, some may digest more readily, and the digestion quality must be evaluated for each sample prior to analysis. A quality assurance sample of a similar matrix prepared alongside unknown samples will help to determine if the digestion method is appropriate for the determination of lead in these matrices.
- ICP-OES has low sensitivity for lead, making it difficult to measure low-level samples. The concentration of lead in solutions prepared from the multivitamin samples may be near the method detection limit of many ICP-OES systems, leading to inaccurate results. Sufficient procedural reagent blanks should be prepared along with samples to determine the method detection limit for lead.
- Many of the calibration curves reported by participants extended far below the reported working range of lead in the solutions to be analyzed. While the calibration curve may appear linear over a wide concentration range, a measured value outside of the calibration curve may have significant bias. For the most accurate results, use calibration points closely surrounding the expected solution concentration, and ensure that the calibration curve is linear along the region of your expected solution concentrations.
    - The sample/sample comparison graph does not reflect an obvious calibration error. Some laboratories reported values that were high for one sample but were within range for the second sample. This type of trend may indicate individualized problems with the digestion of one matrix compared to another.

- Quality assurance samples should always be used. These can be commercially available reference materials (CRMs, SRMs, or RMs) or prepared in-house, but need to be of known concentration.
  - They are used to ensure that the method is performing as expected.
  - They are useful in finding where errors are occurring, including calculation errors.
  - After checking for calculation errors, make sure results are reported correctly.

Table 3. Individual data table (NIST) for lead in dietary supplements.

National Institute of Standards & Technology

Exercise G - July 2011 - Toxic Elements

	Lab Code:	**NIST**	**1. Your Results**				**2. Community Results**			**3. Target Value**	
Analyte	Sample	Units	Mean	s_{total}	Z_{comm}	Z_{NIST}	N	Mean	Std Dev	Value	U_{95}
Pb	Ephedra Tablets	ng/g	660	53	0.4	0.0	36	626	84	660	53
Pb	Multivitamin Tablets	ng/g	269	2.4	-0.3	0.0	36	291	66	269	2

Mean	Average of reported values
s_{total}	Standard deviation of reported values
Z_{comm}	Z-score: (Lab Mean - Consensus Mean)/ Consensus Standard Deviation
Z_{NIST}	Z-score: (Lab Mean - NIST Value or Label Claim)/ NIST or Label Claim Standard Deviation
NR	No data reported

N	Number of quantitative values reported
Mean	Robust mean of the reported values
Std Dev	Robust standard deviation
Value	NIST-assessed value
U_{95}	±95% confidence interval about the assessed value

Table 4. Data summary table for lead in dietary supplements.

		Lead									
		SRM 3243 EphedraTablets (ng/g)					SRM 3280 Multivitamin Tablets (ng/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	NIST				660	53				269	2
	G703	661	678	621	653	29	279	288	275	281	7
	G706	690	717	728	712	20	228	232	240	233	6
	G709	636	641	627	634	7	265	253	273	264	10
	G710	665	644	584	631	42	282	277	277	279	3
	G711										
	G713	620	649	624	631	16	271	283	280	278	6
	G714						219	235	341	265	66
	G718	587	601	549	579	27	319	297	269	295	25
	G719	500	559	549	536	32	210	225	214	216	8
	G720										
	G723	704	687	664	685	20	226	229	226	227	2
	G724	711	681	674	689	20	258	257	252	256	3
	G725	682	615	614	637	39	279	288	287	285	5
	G726	711	672	693	692	20	324	328	292	315	20
	G728										
	G729										
	G734	472	466	505	481	21	616	627	658	634	22
	G735	538	569	522	543	24	618	554	554	576	37
	G736	659	489	553	567	86	769	818	811	799	27
	G737	960	678	946	861	159	657	628	638	641	15
	G738	670	610	680	653	38	290	280	290	287	6
	G739										
	G742										
	G743	531	546	524	534	11	233	230	225	230	4
	G745	667	560	576	601	58	275	285	272	277	7
	G746	614	614	614	614	0	326	370	360	352	23
	G747	606	596	573	592	17	242	245	222	236	13
	G748	699	644	736	693	46	299	200	333	277	69
	G749	1610		3400	2505	1266	530			530	
	G750	757	658	634	683	65	211	208	213	211	3
	G751	614	689	589	631	52	267	270	267	268	2
	G752	1042	718	849	870	163	649	706	824	727	89
	G754	532	562	543	546	15	328	305	324	319	12
	G755										
	G756	574	534	521	543	28	233	239	243	238	5
	G757	647	601	614	621	24	249	257	249	252	5
	G758										
	G759	637	646	642	642	4	281	280	280	280	0
	G762	640	630	650	640	10	280	280	290	283	6
	G763	606	627	645	626	19	365	327	299	330	33
	G764										
	G766	549	538	567	551	15	239	246	238	241	4
	G769	163	153	504	273	200	467	456	389	437	42
	G770	667	689	689	682	13	298	302	300	300	2
	G771	421	507	391	440	60	94	91	83	89	5
	G773	929	778	774	827	88	392	357	443	397	43
	G774										
	G778										
Community Results		Consensus Mean			626		Consensus Mean			291	
		Consensus Standard Deviation			84		Consensus Standard Deviation			66	
		Maximum			2505		Maximum			799	
		Minimum			273		Minimum			89	
		N			36		N			36	

		Lead									
		SRM 3243 EphedraTablets (ng/g)					SRM 3280 Multivitamin Tablets (ng/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	NIST				660	53				269	2
	G703	661	678	621	653	29	279	288	275	281	7
	G706	690	717	728	712	20	228	232	240	233	6
	G709	636	641	627	634	7	265	253	273	264	10
	G710	665	644	584	631	42	282	277	277	279	3
	G711										
	G713	620	649	624	631	16	271	283	280	278	6
	G714						219	235	341	265	66
	G718	587	601	549	579	27	319	297	269	295	25
	G719	500	559	549	536	32	210	225	214	216	8
	G720										
	G723	704	687	664	685	20	226	229	226	227	2
	G724	711	681	674	689	20	258	257	252	256	3
	G725	682	615	614	637	39	279	288	287	285	5
	G726	711	672	693	692	20	324	328	292	315	20
	G728										
	G729										
	G734	472	466	505	481	21	616	627	658	634	22
	G735	538	569	522	543	24	618	554	554	576	37
	G736	659	489	553	567	86	769	818	811	799	27
	G737	960	678	946	861	159	657	628	638	641	15
	G738	670	610	680	653	38	290	280	290	287	6
	G739										
	G742										
	G743	531	546	524	534	11	233	230	225	230	4
	G745	667	560	576	601	58	275	285	272	277	7
	G746	614	614	614	614	0	326	370	360	352	23
	G747	606	596	573	592	17	242	245	222	236	13
	G748	699	644	736	693	46	299	200	333	277	69
	G749	1610		3400	2505	1266	530			530	
	G750	757	658	634	683	65	211	208	213	211	3
	G751	614	689	589	631	52	267	270	267	268	2
	G752	1042	718	849	870	163	649	706	824	727	89
	G754	532	562	543	546	15	328	305	324	319	12
	G755										
	G756	574	534	521	543	28	233	239	243	238	5
	G757	647	601	614	621	24	249	257	249	252	5
	G758										
	G759	637	646	642	642	4	281	280	280	280	0
	G762	640	630	650	640	10	280	280	290	283	6
	G763	606	627	645	626	19	365	327	299	330	33
	G764										
	G766	549	538	567	551	15	239	246	238	241	4
	G769	163	153	504	273	200	467	456	389	437	42
	G770	667	689	689	682	13	298	302	300	300	2
	G771	421	507	391	440	60	94	91	83	89	5
	G773	929	778	774	827	88	392	357	443	397	43
	G774										
	G778										
Community Results		Consensus Mean			626		Consensus Mean			291	
		Consensus Standard Deviation			84		Consensus Standard Deviation			66	
		Maximum			2505		Maximum			799	
		Minimum			273		Minimum			89	
		N			36		N			36	

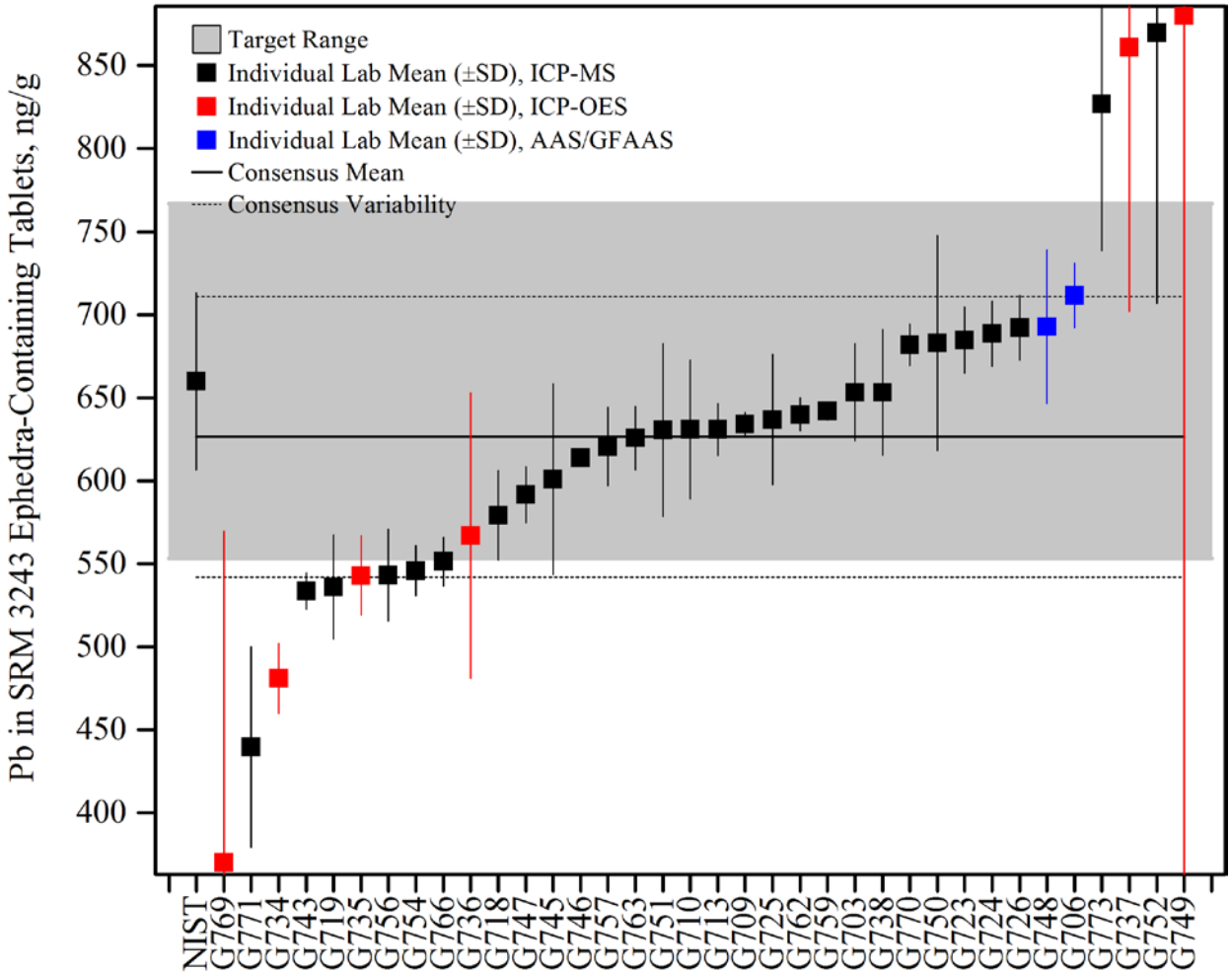

Figure 6. Lead in SRM 3243 Ephedra-Containing Tablets (data summary view – instrumental method). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The data are identified by instrumental method in this graph. The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean. The gray shaded region represents the target zone for "acceptable" performance, which encompasses the NIST certified value bounded by twice its uncertainty (U_{95}).

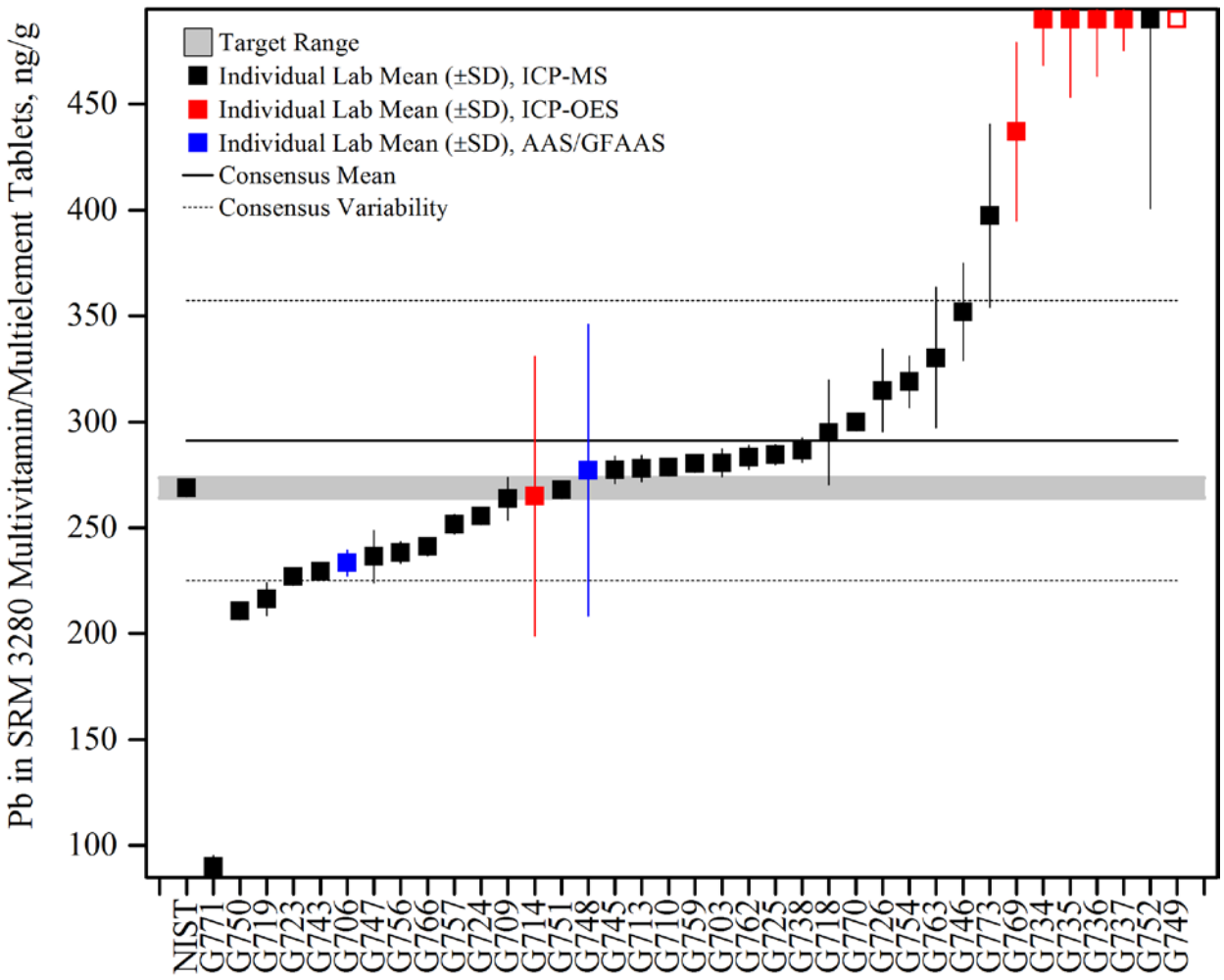

Figure 7. Lead in SRM 3280 Multivitamin/Multielement Tablets (data summary view – instrumental method). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The data are identified by instrumental method in this graph. The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean. The gray shaded region represents the target zone for "acceptable" performance, which encompasses the NIST certified value bounded by twice its uncertainty (U_{95}).

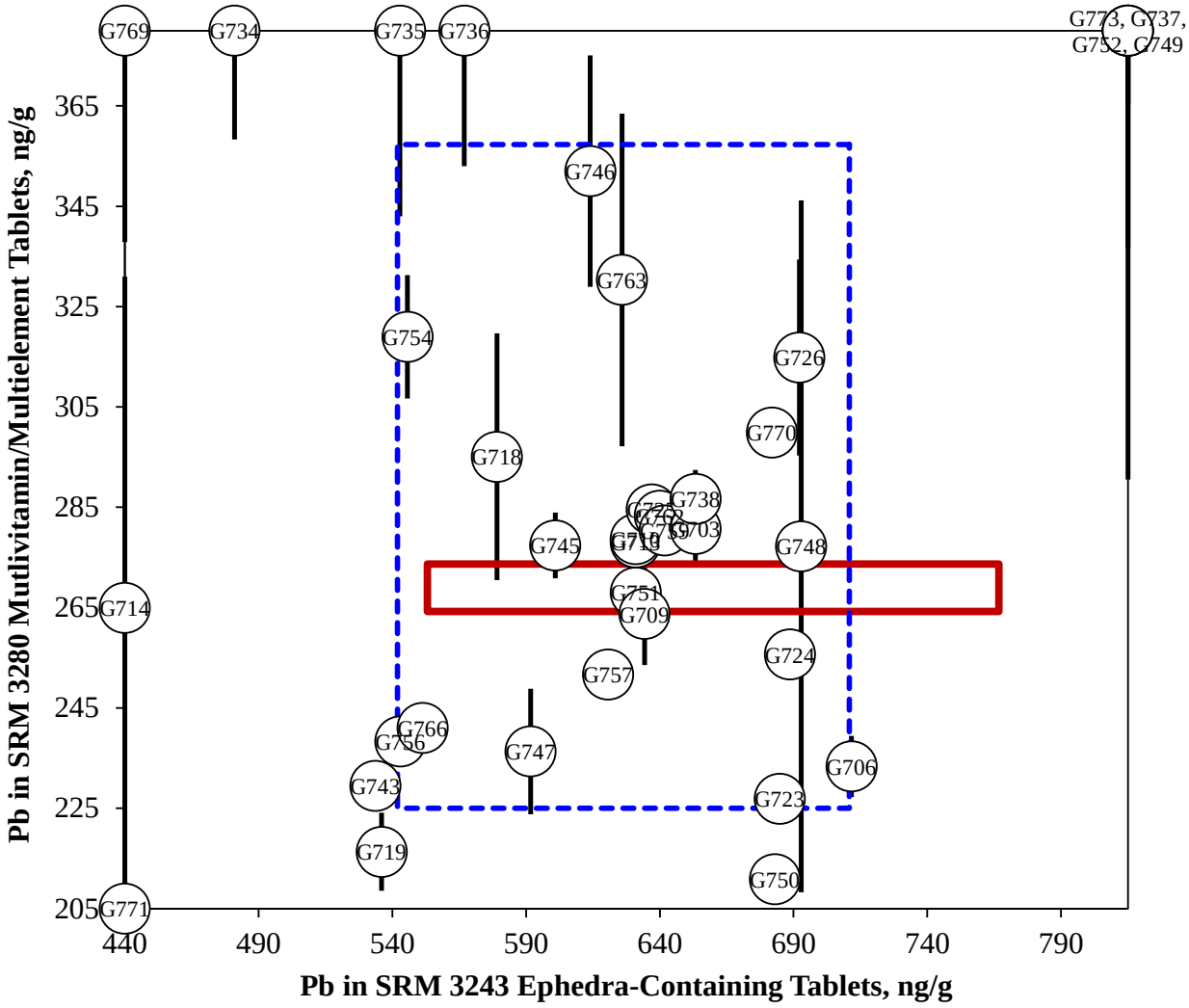

Figure 8. Lead in SRM 3243 Ephedra-Containing Tablets and SRM 3280 Multivitamin/Multielement Tablets (sample/control comparison view). In this view, the individual laboratory results for the one sample (SRM 3243 Ephedra-Containing Tablets) are compared to the results for a second sample (SRM 3280 Multivitamin/Multielement Tablets). The solid red box represents the target zone for the two samples, ephedra tablets (x-axis) and multivitamin (y-axis). The dotted blue box represents the consensus zone for the ephedra tablets (x-axis) and the multivitamin (y-axis).

FOLIC ACID IN FOOD MATRICES

Study Overview

In this study, participants were provided with one NIST SRM, SRM 3233 Fortified Breakfast Cereal, and a well-characterized fortified milk powder sample. Participants were asked to use in-house analytical methods to determine the mass fraction of folic acid in each of the matrices and report values on an as-received basis.

Sample Information

Fortified Breakfast Cereal. Participants were provided with one bottle containing approximately 60 g of dried, powdered breakfast cereal. The cereal flakes were ground, homogenized, and packaged in amber glass bottles, and the bottles were capped, sealed with heat-shrink tape, and individually sealed in mylar bags. Before use, participants were instructed to thoroughly mix the contents of the bottle and use a sample size of at least 1.0 g. Participants were asked to store the material at controlled room temperature, 20 °C to 25 °C, and to prepare three samples and report three values from the single bottle provided. Prior to the study, the approximate analyte level was given as 15 mg/kg. The certified value for folic acid in SRM 3233 was determined at NIST by ID-LC-MS/MS following solvent extraction, in combination with data from numerous collaborating laboratories. The certified values and uncertainties are reported in the table below, both on a dry-mass basis and on an as-received basis accounting for moisture of the material (1.7 %).

	Certified Mass Fraction in SRM 3233 (mg/kg)	
Analyte	(dry-mass basis)	(as-received basis)
Folic Acid	15.1 ± 1.2	14.8 ± 1.2

Milk Powder. Participants were provided with three packets containing approximately 10 g of freeze-dried, powdered milk. The fortified milk powder was homogenized and packaged in nitrogen-flushed foil pouches with a paper over-wrap. Before use, participants were instructed to thoroughly mix the contents of the packet and use a sample size of at least 1.0 g. Participants were asked to store the material at –20 °C, and to prepare one sample and report one value from each packet of milk powder provided. Prior to the study, the approximate analyte level was given as 2.4 mg/kg. The target value for folic acid in the fortified milk powder was determined at NIST by ID-LC-MS/MS following solvent extraction and hydrolysis, in combination with data from two collaborating laboratories. The NIST-determined values and uncertainties are reported in the table below, both on a dry-mass basis and on an as-received basis accounting for moisture of the material (1.6 %).

	Mass Fraction in Milk Powder (mg/kg)	
Analyte	(dry-mass basis)	(as-received basis)
Folic Acid	2.35 ± 0.06	2.31 ± 0.06

Study Results

- Twenty-three laboratories enrolled in this exercise and received samples. Eleven laboratories reported results for folic acid in both the breakfast cereal and the milk powder

(48 % participation). One laboratory reported a single value for folic acid in the breakfast cereal and was therefore excluded from calculation of summary statistics.[2]

- The consensus ranges for folic acid in both matrices were wide but contained the target ranges. The consensus means for folic acid in both matrices were slightly above the target ranges.
  - For folic acid in the breakfast cereal, the between-laboratory variability was high (32 % RSD).
  - For folic acid in the milk powder, the between-laboratory variability was acceptable (15 % RSD).
- A majority of the laboratories reported using an enzymatic hydrolysis approach (50 %). A solvent extraction approach (25 %) and a shaking or sonication extraction approach (25 %) were also reported as methods of sample preparation.
- Most laboratories reported using LC with absorbance detection as the analytical method for analysis (67 %). Laboratories also reported using microbiological assay (17 %), LC-MS (8 %), and protein binding assay (8 %) as the instrumental approach.
- All participating laboratories reported the use of an external standard approach to quantitation.

Technical Recommendations

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

- No sample preparation approach or analytical method was identified as exceptionally good or problematic.
- Some high values were reported for either the sample or control (but not both). This may indicate chromatographic coelutions with matrix components. Because each matrix is different, the chromatographic method should be evaluated to confirm any potential biases from coeluting matrix components.
- NIST values in SRM 3233 and the fortified milk powder were determined using a gentle extraction procedure designed for determination of fortified folic acid in foods. As a result, the consensus means may be slightly higher than the NIST target ranges, reflecting the small fraction of endogenous folic acid that could be extracted by participating laboratories using more extensive extraction procedures such as enzymatic treatments. This contribution is expected to be relatively small, perhaps only 2 % of the total folic acid present in these highly fortified samples.

Table 5. Individual data table (NIST) for folic acid in foods.

National Institute of Standards and Technology

Exercise G - July 2011 - Folic Acid

	Lab Code:	**NIST**	**1. Your Results**				**2. Community Results**			**3. Target Value**	
Analyte	Sample	Units	Mean	s_{total}	Z_{comm}	Z_{NIST}	N	Mean	Std Dev	Value	U_{95}
Folic Acid	Breakfast Cereal	mg/kg	14.8	1.2	-0.5	0.0	10	17.8	5.7	14.8	1.2
Folic Acid	Milk Powder	mg/kg	2.31	0.06	-0.4	0.0	11	2.44	0.37	2.31	0.06

Mean	Average of reported values
s_{total}	Standard deviation of reported values
Z_{comm}	Z-score: (Lab Mean - Consensus Mean)/ Consensus Standard Deviation
Z_{NIST}	Z-score: (Lab Mean - NIST Value or Label Claim)/ NIST or Label Claim Standard Deviation
NR	No data reported

N	Number of quantitative values reported
Mean	Robust mean of the reported values
Std Dev	Robust standard deviation
Value	NIST-assessed value
U_{95}	±95% confidence interval about the assessed value

Table 6. Data summary table for folic acid in dietary supplements.

		Folic Acid									
		SRM 3233 Fortified Breakfast Cereal (mg/kg)					Fortified Milk Powder (mg/kg)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	NIST				14.8	1.2				2.31	0.06
	G703	13.1	13.1	13.9	13.4	0.5	2.33	2.22	2.32	2.29	0.06
	G705										
	G706										
	G709	24.6	26.2	26.0	25.6	0.9					
	G711										
	G721										
	G727	14.0	13.9	13.6	13.8	0.2	2.20	2.14	2.15	2.16	0.03
	G728										
	G729	15.8	15.5	14.8	15.4	0.5	2.61	1.90	2.72	2.41	0.44
	G738	16.1			16.1		3.10	3.20	3.20	3.17	0.06
	G739										
	G740										
	G744	14.9	15.6	14.1	14.9	0.8	2.97	2.70	2.59	2.75	0.20
	G746	24.4	22.5	22.3	23.1	1.2	5.00	4.90	4.80	4.90	0.10
	G749	40.6	44.4	51.6	45.5	5.6	1.23	1.59	3.65	2.16	1.31
	G752										
	G753	15.2	15.0	14.9	15.0	0.1	2.19	1.93	2.16	2.09	0.14
	G754	14.8	15.6	15.2	15.2	0.4	2.27	2.28	2.29	2.28	0.01
	G758										
	G759	15.5	15.7	15.4	15.5	0.2	2.02	2.33	2.28	2.21	0.16
	G767										
	G768						2.74	2.72	2.24	2.57	0.28
	G778										
Community Results		Consensus Mean			17.8		Consensus Mean			2.44	
		Consensus Standard Deviation			5.7		Consensus Standard Deviation			0.37	
		Maximum			45.5		Maximum			4.9	
		Minimum			13.4		Minimum			2.1	
		N			10		N			11	

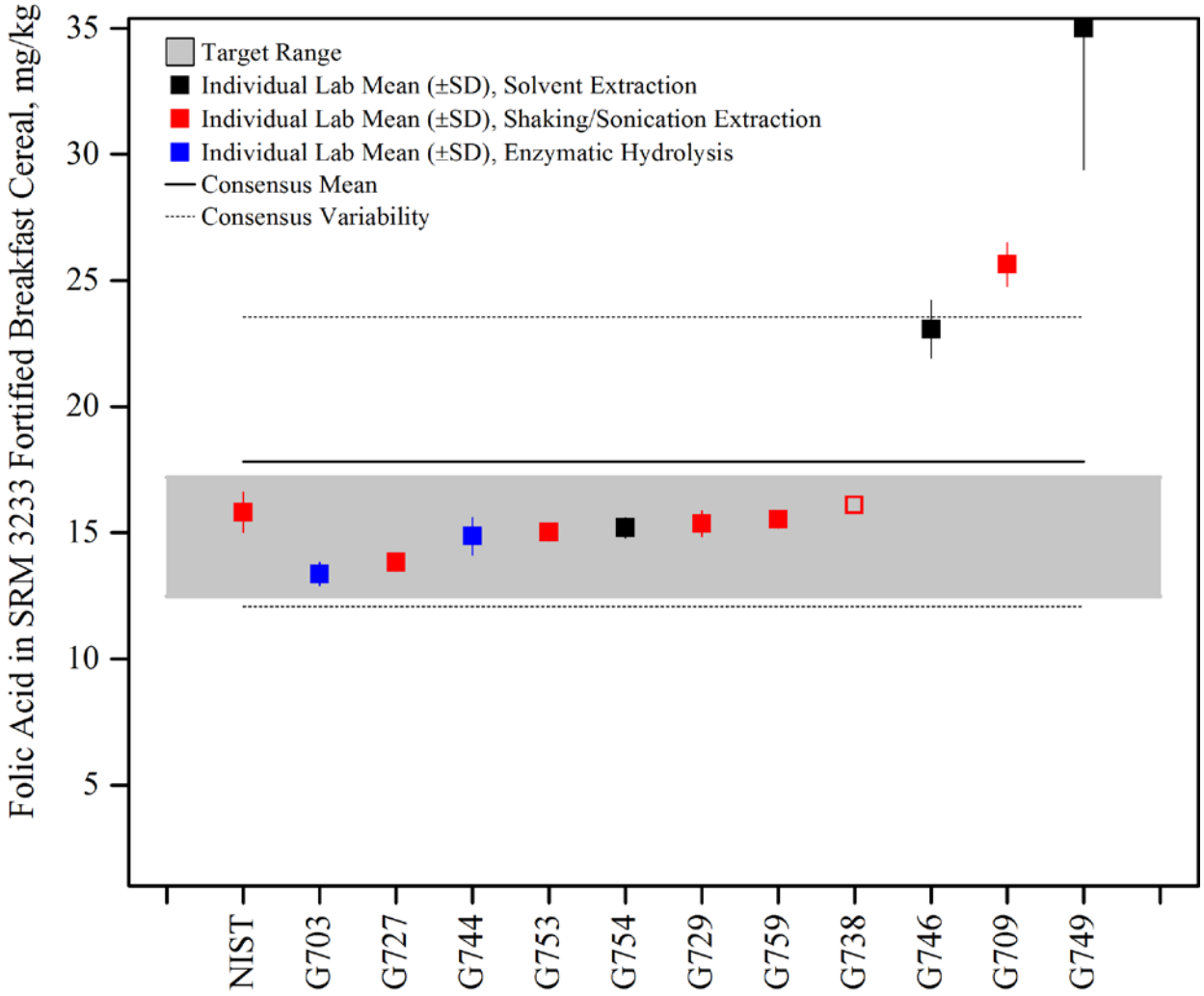

Figure 9. Folic acid in SRM 3233 Fortified Breakfast Cereal (data summary view – sample preparation method). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The data are identified by sample preparation method in this graph. The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean. The gray shaded region represents the target zone for "acceptable" performance, which encompasses the NIST certified value bounded by twice its uncertainty (U_{95}).

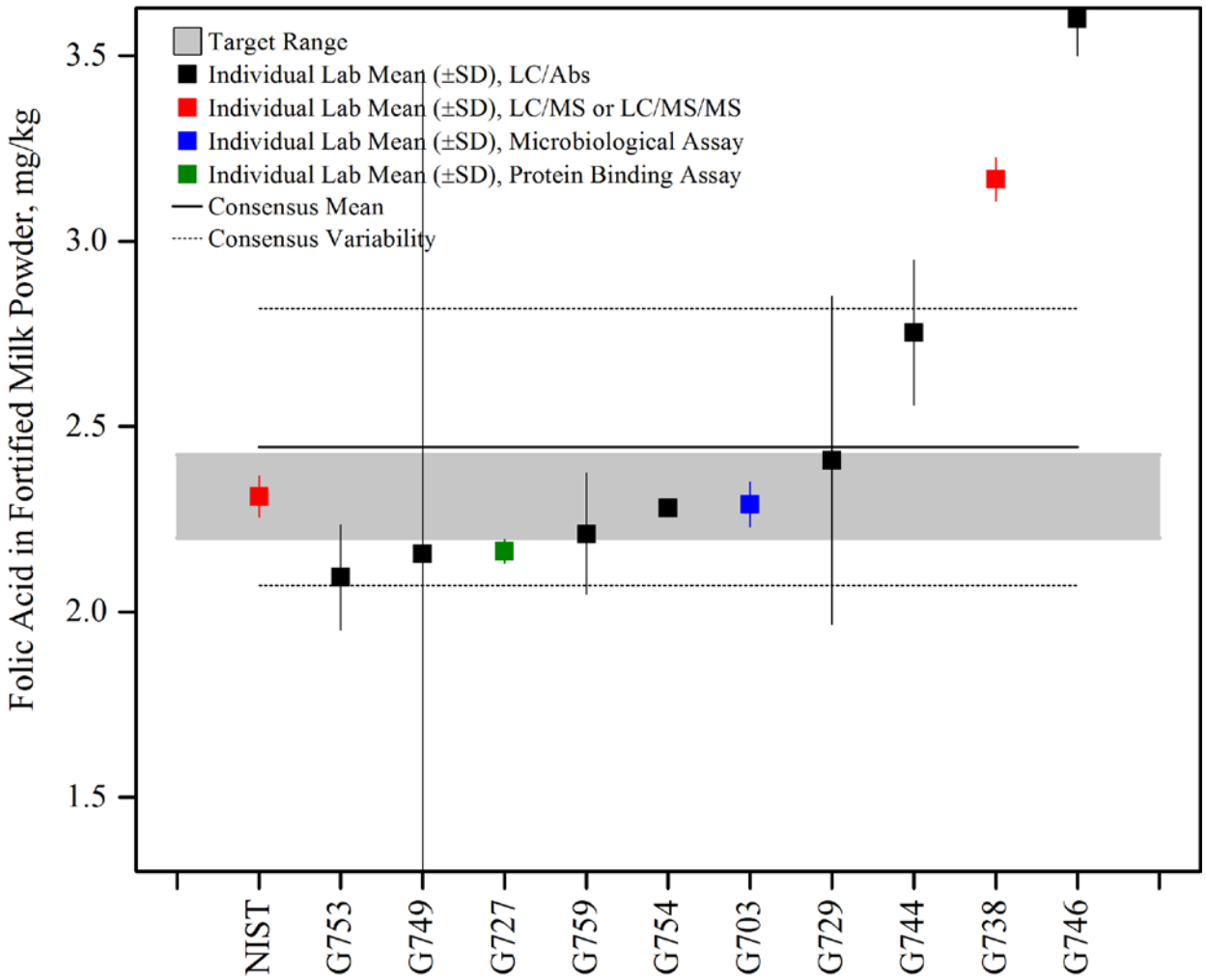

Figure 10. Folic acid in Fortified Milk Powder (data summary view – sample preparation method). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The data are identified by sample preparation method in this graph. The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean. The gray shaded region represents the target zone for "acceptable" performance, which encompasses the NIST assigned value bounded by twice its standard deviation.

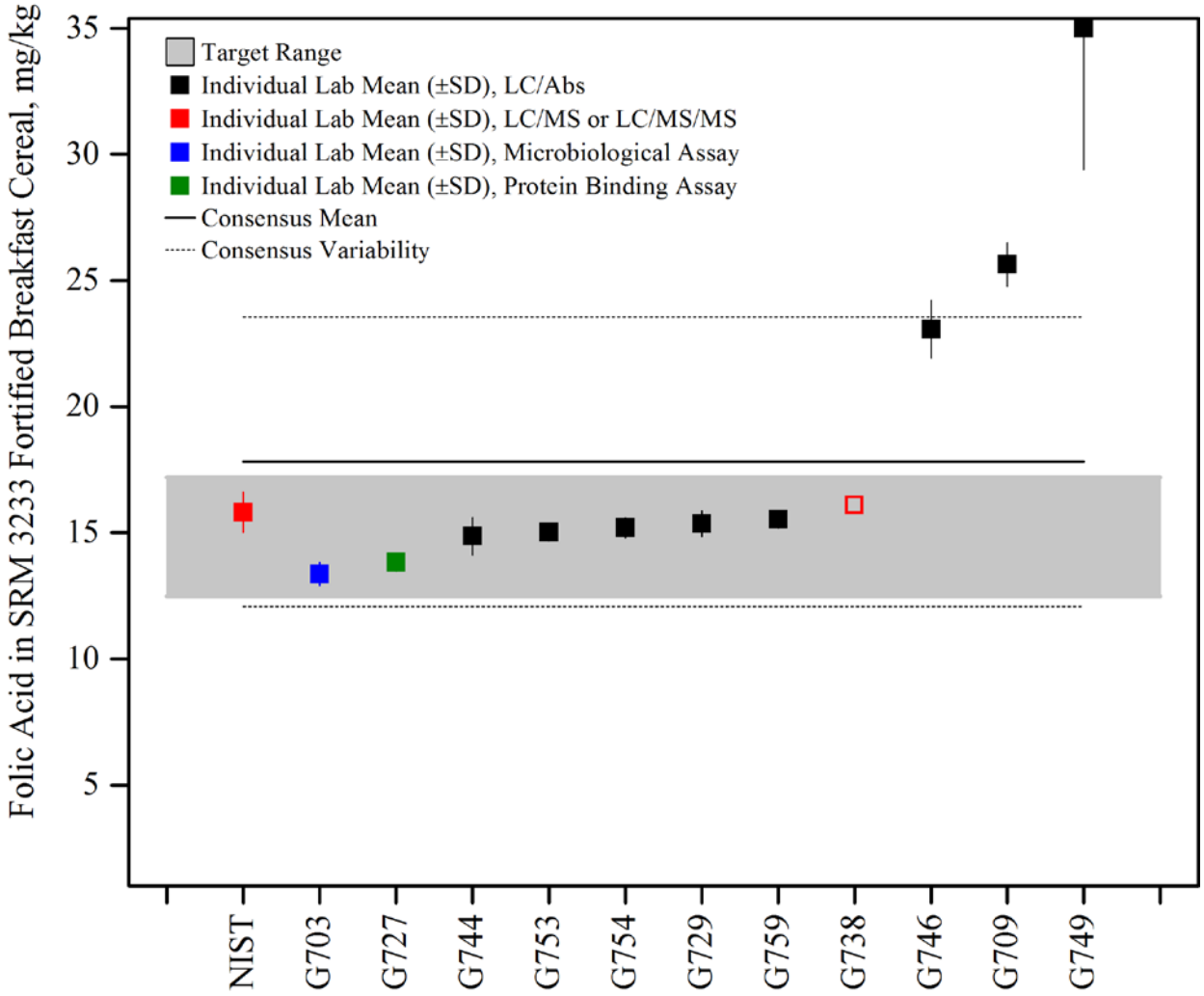

Figure 11. Folic acid in SRM 3233 Fortified Breakfast Cereal (data summary view – instrumental method). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The data are identified by instrumental method in this graph. The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean. The gray shaded region represents the target zone for "acceptable" performance, which encompasses the NIST certified value bounded by twice its uncertainty (U_{95}).

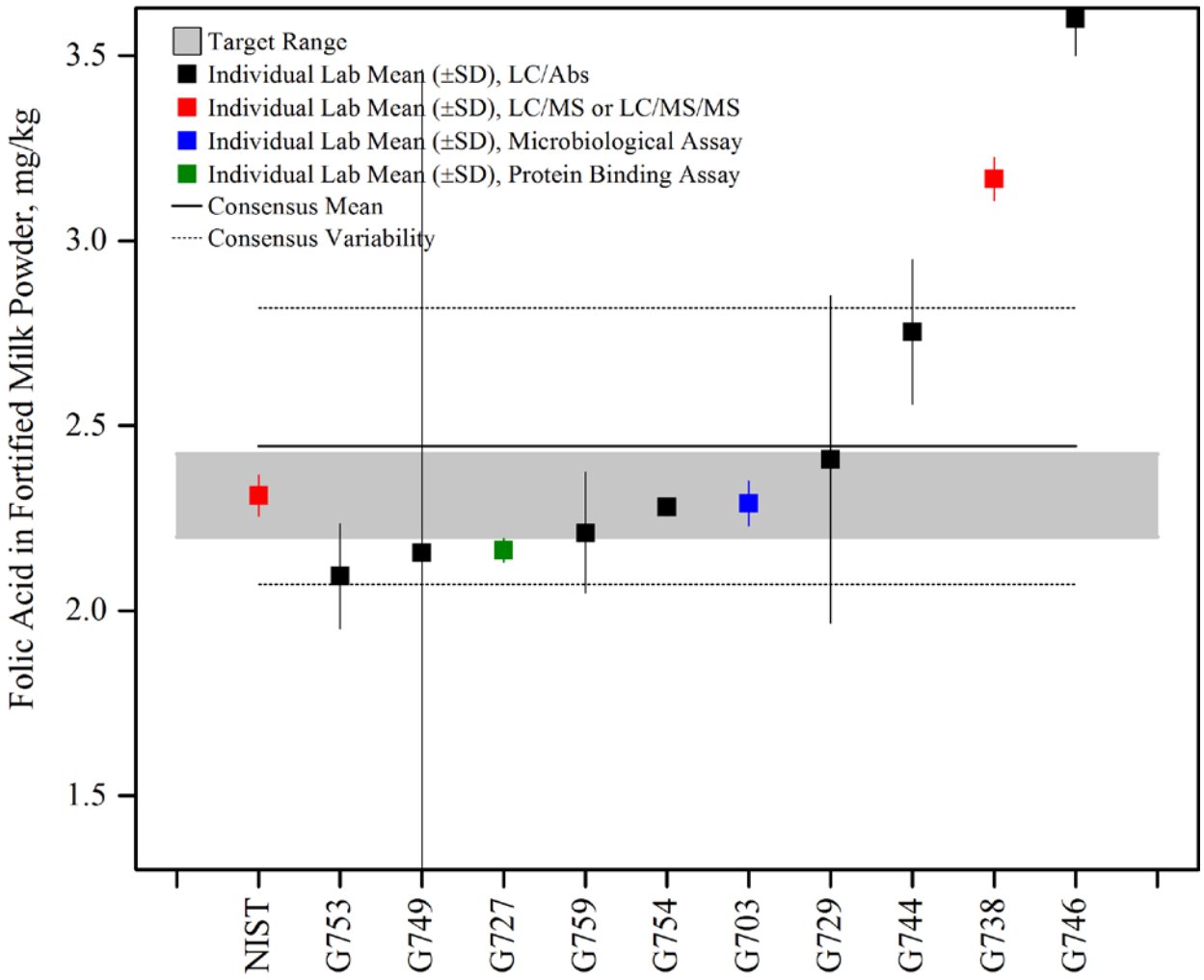

Figure 12. Folic acid in Fortified Milk Powder (data summary view – instrumental method). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The data are identified by instrumental method in this graph. The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean. The gray shaded region represents the target zone for "acceptable" performance, which encompasses the NIST assigned value bounded by twice its standard deviation.

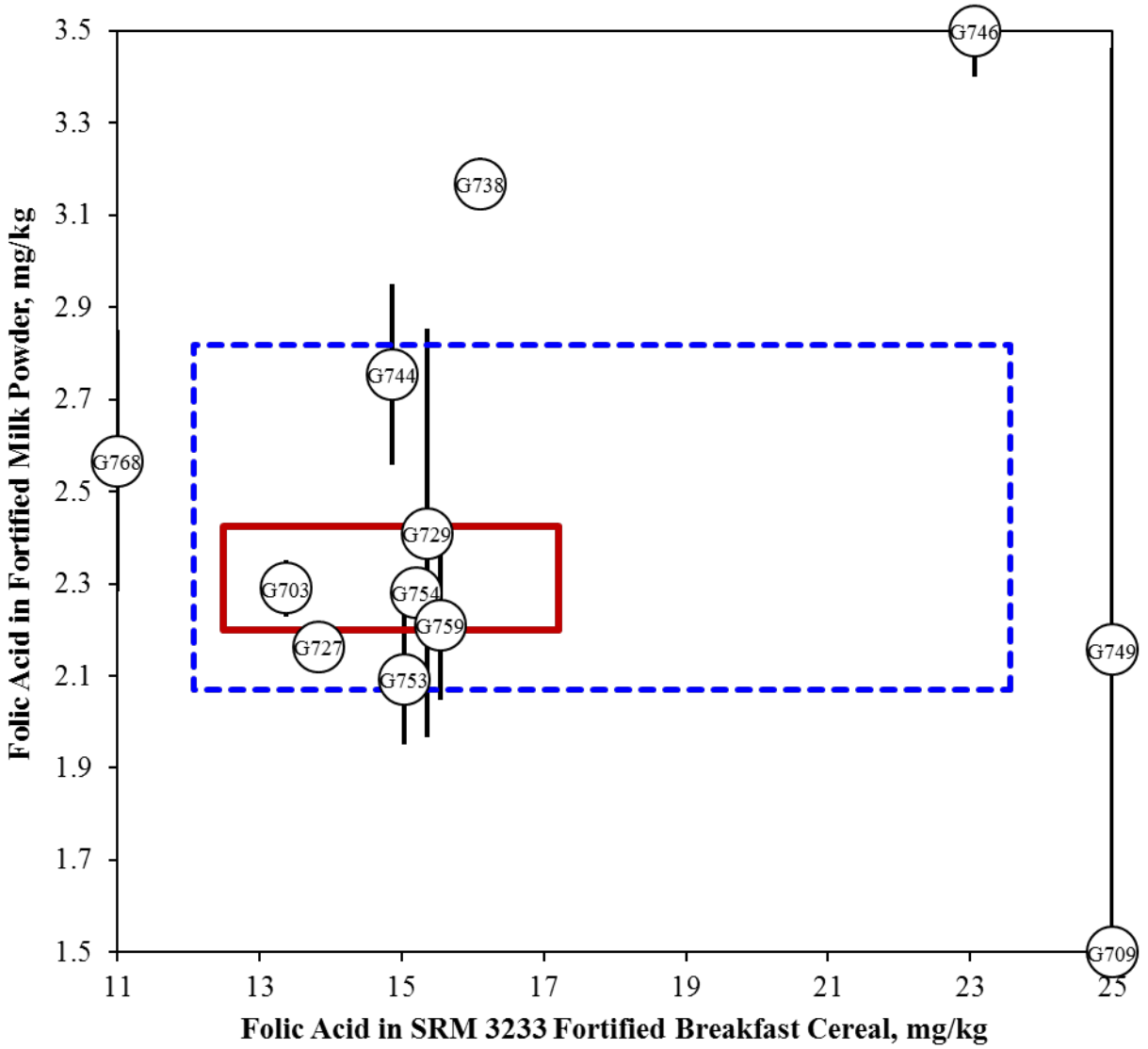

Figure 13. Folic acid in SRM 3233 Fortified Breakfast Cereal and Fortified Milk Powder (sample/sample comparison view). In this view, the individual laboratory results for one sample (SRM 3233 Fortified Breakfast Cereal) are compared to the results for a second sample (Fortified Milk Powder). The solid red box represents the target zone for the breakfast cereal (x-axis) and the milk powder (y-axis). The dotted blue box represents the consensus zone for the breakfast cereal (x-axis) and the milk powder (y-axis).

β-CAROTENE IN SOLUTIONS AND DIETARY SUPPLEMENTS

Study Overview

In this study, participants were provided with one NIST SRM, SRM 3251 *Serenoa repens* Extract, and two ethanolic solutions of β-carotene. Participants were asked to use in-house analytical methods to determine the mass fraction of total β-carotene and isomers in each of the matrices and report values on an as-received basis.

Sample Information

β-carotene Solution 1. Participants were provided with three vials containing approximately 2 mL of all-*trans*-β-carotene dissolved in ethanol containing 30 ppm BHT. Solution 1 did not contain significant amounts of *cis*-β-carotene isomers. Before use, participants were instructed to thoroughly mix the contents of the vials and use a sample size of at least 50 mg. Participants were asked to store the material in a freezer at -20 °C, and to prepare one sample and report one value per analyte from each vial provided. Prior to the study, the approximate level of total β-carotene was given as 0.9 mg/kg. The NIST value and uncertainty for total β-carotene in solution 1 were determined spectrophotometrically in ethanol with a molar absorptivity of 2620 $dLg^{-1}cm^{-1}$, and the β-carotene concentration decreased approximately 10 % from July 2011 until February 2012. The NIST-determined values and uncertainties are reported in the table below.

β-carotene Solution 2. Participants were provided with three vials containing approximately 2 mL of *Dunaliella* extract dissolved in ethanol containing 30 ppm BHT. In solution 2, approximately 30 % of the total β-carotene was in the form of the 9-*cis*-β-carotene isomer. Before use, participants were instructed to thoroughly mix the contents of the vials and use a sample size of at least 50 mg. Participants were asked to store the material in a freezer at -20 °C, and to prepare one sample and report one value per analyte from each vial provided. Prior to the study, the approximate level of total β-carotene was given as 4 mg/kg. The NIST value and uncertainty for total β-carotene in solution 2 were determined spectrophotometrically in ethanol with a molar absorptivity of 2620 $dLg^{-1}cm^{-1}$; the total β-carotene content of solution 2 was stabile throughout the time period of the study. The NIST-determined values and uncertainties are reported in the table below.

Saw Palmetto Extract. Participants were provided with three ampoules, each containing approximately 1 mL of *Serenoa repens* extract. The extract was packaged under nitrogen in amber glass ampoules. Before use, participants were instructed to thoroughly mix the contents of the ampoules and use a sample size of at least 50 mg. Participants were asked to store the material at controlled room temperature, 20 °C to 25 °C, and to prepare one sample and report one value per analyte from each ampoule provided. Prior to the study, the approximate level of total β-carotene was given as 50 mg/kg. The NIST certified values and uncertainties for *trans*-β-carotene, 9-*cis*-β-carotene, and total β-carotene in SRM 3251 were determined at NIST by LC-absorbance and LC-fluorescence (using different column chemistries), following gravimetric dilution and addition of an internal standard. The NIST-determined values and uncertainties are reported in the table below.

Analyte	Mass Fraction in Solution 1 (mg/kg)	Mass Fraction in Solution 2 (mg/kg)	Mass Fraction in SRM 3251 (mg/kg)
trans-β-carotene			36.4 ± 5.6
9-*cis*-β-carotene			10.40 ± 1.20
Total β-carotene	0.900 ± 0.045	4.000 ± 0.200	46.8 ± 4.6

Study Results

- Forty-one laboratories enrolled in this exercise and received samples. Twenty-three laboratories reported results for total β-carotene in both of the β-carotene solutions (56 % participation). Twenty-four laboratories reported results for total β-carotene in the saw palmetto extract (59 % participation). One laboratory reported a single value for total β-carotene in the saw palmetto extract and was therefore excluded from calculation of summary statistics.[2]
- Few laboratories (6 or fewer) reported values for *cis* and *trans* isomers of β-carotene in any sample.
- The consensus ranges for total β-carotene in both solutions were wide (21 % to 23 % relative standard deviation, (RSD) respectively) but overlapped with the target ranges. The consensus means for total β-carotene in both solutions were significantly above the target ranges.
- The consensus range for total β-carotene in SRM 3251 was excellent (10 % RSD) and overlapped with the target range. The consensus mean for total β-carotene was within the target range.
- The consensus range for *trans*-β-carotene in SRM 3251 was excellent (7 % RSD) and overlapped with the target range. The consensus mean for *trans*-β-carotene was within the target range.
- The consensus range for 9-*cis*-β-carotene in SRM 3251 was reasonable (17 % RSD) and overlapped with the target range. The consensus mean for 9-*cis*-β-carotene was within the target range.
- A majority of the laboratories reported using simple dilution (48 %) or saponification with extraction (40 %). Other solvent extraction approaches (8 %) and hydrolysis extraction approaches (4 %) were also reported as methods of sample preparation.
- Most laboratories reported using LC with absorbance detection as the analytical method for analysis (92 %). Laboratories also reported using LC-MS or LC-MS/MS (4 %) and spectrophotometry (4 %) as the instrumental approach.
- All participating laboratories reported the use of an external standard approach to quantitation.

Technical Recommendations

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

- No sample preparation approach or analytical method was identified as exceptionally good or problematic.
- Although no sample pretreatment was necessary for the β-carotene solutions, many laboratories subjected the samples to saponification, enzymatic hydrolysis, and solvent extraction. Sample preparation can isomerize the β-carotene, which would not be evident

in the total β-carotene measurements, but would affect the ratio of the *cis/trans* isomers observed.

- As shown in **Figure 25, Figure 26**, and **Figure 27**, calibration errors may be possible. Laboratories tended to report consistently biased results for the samples and solutions. The most common calibration error in the measurement of carotenoids is caused by either neglecting to assign the concentration spectrophotometrically (carotenoid measurements are traceable to molar absorptivity) or using the wrong molar absorption coefficient (wrong solvent or temperature).

Table 7. Individual data table (NIST) for β-carotene in solutions and dietary supplements.

National Institute of Standards & Technology

Exercise G - July 2011 - β-carotene

		Lab Code:	**NIST**	**1. Your Results**				**2. Community Results**			**3. Target Value**	
Analyte	Sample	Units		Mean	s_{total}	Z_{comm}	Z_{NIST}	N	Mean	Std Dev	Value	U_{95}
trans-β-carotene	Solution 1	mg/kg						6	1.05	0.227	NR	NR
trans-β-carotene	Solution 2	mg/kg						6	2.48	0.548	NR	NR
trans-β-carotene	Saw Palmetto Extract	mg/kg		36.4	5.6	0.6	0.0	7	34.9	2.35	36.4	5.6
9-*cis*-β-carotene	Solution 1	mg/kg						2	0.0113	0.00135	NR	NR
9-*cis*-β-carotene	Solution 2	mg/kg						6	2.21	0.74	NR	NR
9-*cis*-β-carotene	Saw Palmetto Extract	mg/kg		10.4	1.2	-0.7	0.0	6	11.8	2.04	10.4	1.2
13-*cis*-β-carotene	Solution 1	mg/kg						1	0.0364		NR	NR
13-*cis*-β-carotene	Solution 2	mg/kg						3	0.199	0.0427	NR	NR
13-*cis*-β-carotene	Saw Palmetto Extract	mg/kg						4	2.59	2.33	NR	NR
15-*cis*-β-carotene	Solution 1	mg/kg						3	0.0341	0.0347	NR	NR
15-*cis*-β-carotene	Solution 2	mg/kg						4	0.202	0.156	NR	NR
15-*cis*-β-carotene	Saw Palmetto Extract	mg/kg						4	2.75	2.37	NR	NR
Total β-carotene	Solution 1	mg/kg		0.9	0.045	-1.0	0.0	20	1.17	0.271	0.900	0.045
Total β-carotene	Solution 2	mg/kg		4	0.2	-1.4	0.0	21	5.61	1.19	4.00	0.20
Total β-carotene	Saw Palmetto Extract	mg/kg		46.8	4.6	-1.0	0.0	20	52.2	5.38	46.8	4.6

Mean — Average of reported values

s_{total} — Standard deviation of reported values

Z_{comm} — Z-score: (Lab Mean - Consensus Mean)/ Consensus Standard Deviation

Z_{NIST} — Z-score: (Lab Mean - NIST Value or Label Claim)/ NIST or Label Claim Standard Deviation

NR — No data reported

N — Number of quantitative values reported

Mean — Robust mean of the reported values

Std Dev — Robust standard deviation

Value — NIST-assessed value

U_{95} — ±95% confidence interval about the assessed value

Table 8. Data summary table for *trans*-β-carotene in solutions and dietary supplements.

		trans-β-carotene														
		Solution 1 (mg/kg)					Solution 2 (mg/kg)					SRM 3251 Saw Palmetto Extract (mg/kg)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	NIST														36.4	5.6
	G703	1.34	1.60	1.17	1.37	0.22	2.98	3.23	3.26	3.16	0.15	35.8	34.4	34.4	34.9	0.8
	G705															
	G706															
	G707															
	G709	1.13	1.00	0.95	1.03	0.10	3.48	2.51	2.26	2.75	0.64	33.0	33.8	34.0	33.6	0.5
	G710															
	G711															
	G712															
	G714															
	G716	0.75	0.92	1.00	0.89	0.13	1.33	2.16	1.78	1.76	0.42	34.8	34.7	41.8	37.1	4.1
	G717															
	G721															
	G722															
	G724															
	G727	18.61	22.90	17.56	19.69	2.83	53.80	47.72	50.17	50.56	3.06		37.2	37.5	37.4	0.2
	G728															
	G729															
	G730															
	G731															
	G732															
	G733															
	G738															
	G739															
	G740															
	G742															
	G744															
	G746															
	G749															
	G752															
	G753	1.06	1.11	1.09	1.09	0.03	2.48	2.54	2.67	2.56	0.10	35.0	35.5	37.0	35.8	1.0
	G755															
	G758															
	G760															
	G762															
	G764															
	G766	0.78	0.75	0.85	0.79	0.05	2.18	2.03	2.25	2.15	0.11	28.4	29.0	28.1	28.5	0.4
	G767															
	G770															
	G773															
	G774															
	G775															
Community Results		Consensus Mean			1.14		Consensus Mean			2.76		Consensus Mean			35.0	
		Consensus Standard Deviation			0.37		Consensus Standard Deviation			0.96		Consensus Standard Deviation			2.6	
		Maximum			19.69		Maximum			50.56		Maximum			37.4	
		Minimum			0.79		Minimum			1.76		Minimum			28.5	
		N			6		N			6		N			6	

Table 9. Data summary table for 9-*cis*-β-carotene in solutions and dietary supplements.

		9-cis-β-carotene														
		Solution 1 (mg/kg)					Solution 2 (mg/kg)					SRM 3251 Saw Palmetto Extract (mg/kg)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	NIST														10.4	1.2
	G703	0.0108	0.0146	0.0110	0.0121	0.0021	2.63	2.67	2.67	2.66	0.02	15.3	15.0	14.8	15.1	0.3
	G705															
	G706															
	G707															
	G709						1.81	2.46	2.44	2.23	0.37	11.5	12.2	12.2	12.0	0.4
	G710															
	G711															
	G712															
	G714															
	G716						0.28	0.41	0.34	0.34	0.07	10.3	10.1	11.7	10.7	0.8
	G717															
	G721															
	G722															
	G724															
	G727															
	G728															
	G729															
	G730															
	G731															
	G732															
	G733															
	G738															
	G739															
	G740															
	G742															
	G744															
	G746															
	G749															
	G752															
	G753						2.76	2.71	2.72	2.73	0.03	9.3	9.7	9.0	9.3	0.4
	G755															
	G758															
	G760															
	G762															
	G764															
	G766						1.80	1.77	1.82	1.80	0.03	12.9	16.3	10.3	13.2	3.0
	G767															
	G770															
	G773															
	G774															
	G775															
Community Results		Consensus Mean			0.0121		Consensus Mean			1.95		Consensus Mean			12.0	
		Consensus Standard Deviation					Consensus Standard Deviation			1.10		Consensus Standard Deviation			2.4	
		Maximum					Maximum			2.73		Maximum			15.1	
		Minimum					Minimum			0.34		Minimum			9.3	
		N			1		N			5		N			5	

Table 10. Data summary table for 13-*cis*-β-carotene in solutions and dietary supplements.

		13-cis-β-carotene														
		Solution 1 (mg/kg)					Solution 2 (mg/kg)					SRM 3251 Saw Palmetto Extract (mg/kg)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	NIST															
	G703															
	G705															
	G706															
	G707															
	G709											8.00	6.40	6.51	6.97	0.89
	G710															
	G711															
	G712															
	G714															
	G716						0.160	0.220	0.190	0.190	0.030	0.73	0.66	0.70	0.70	0.04
	G717															
	G721															
	G722															
	G724															
	G727															
	G728															
	G729															
	G730															
	G731															
	G732															
	G733															
	G738															
	G739															
	G740															
	G742															
	G744															
	G746															
	G749															
	G752															
	G753						0.360	0.340	0.340	0.347	0.012	3.20	2.90	2.60	2.90	0.30
	G755															
	G758															
	G760															
	G762															
	G764															
	G766															
	G767															
	G770															
	G773															
	G774															
	G775															
Community Results		Consensus Mean					Consensus Mean			0.268		Consensus Mean			3.52	
		Consensus Standard Deviation					Consensus Standard Deviation			0.126		Consensus Standard Deviation			3.61	
		Maximum					Maximum			0.347		Maximum			7.0	
		Minimum					Minimum			0.190		Minimum			0.7	
		N			0		N			2		N			3	

Table 11. Data summary table for 15-*cis*-β-carotene in solutions and dietary supplements.

		15-cis-β-carotene														
		Solution 1 (mg/kg)					Solution 2 (mg/kg)					SRM 3251 Saw Palmetto Extract (mg/kg)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	NIST															
	G703															
	G705															
	G706															
	G707															
	G709															
	G710															
	G711															
	G712															
	G714															
	G716	0.0300	0.0400	0.0400	0.0367	0.0058	0.150	0.190	0.170	0.170	0.020	1.93	1.90	2.23	2.02	0.18
	G717															
	G721															
	G722															
	G724															
	G727															
	G728															
	G729															
	G730															
	G731															
	G732															
	G733															
	G738															
	G739															
	G740															
	G742															
	G744															
	G746															
	G749															
	G752															
	G753	0.0500	0.0600	0.0800	0.0633	0.0153	0.270	0.270	0.270	0.270	0.000	3.50	4.70	3.40	3.87	0.72
	G755															
	G758															
	G760															
	G762															
	G764															
	G766						0.350	0.310	0.370	0.343	0.031	5.63	4.88	4.26	4.92	0.69
	G767															
	G770															
	G773															
	G774															
	G775															
Community Results		Consensus Mean			0.0500		Consensus Mean			0.261		Consensus Mean			3.60	
		Consensus Standard Deviation			0.0214		Consensus Standard Deviation			0.099		Consensus Standard Deviation			1.67	
		Maximum			0.0633		Maximum			0.343		Maximum			4.9	
		Minimum			0.0367		Minimum			0.170		Minimum			2.0	
		N			2		N			3		N			3	

Table 12. Data summary table for total β-carotene in solutions and dietary supplements.

		Total β-carotene														
		Solution 1 (mg/kg)					Solution 2 (mg/kg)					SRM 3251 Saw Palmetto Extract (mg/kg)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	NIST				0.90	0.05				4.00	0.20				46.8	4.6
	G703	1.46	1.73	1.27	1.48	0.24	6.47	6.76	6.79	6.67	0.18	57.7	55.4	54.5	55.9	1.7
	G705															
	G706	0.88	1.13	1.02	1.01	0.13	4.91	5.19	4.68	4.93	0.26	46.2	46.0	46.5	46.2	0.3
	G707															
	G709	1.13	1.00	0.95	1.03	0.10	5.29	4.97	4.69	4.98	0.30	52.5	52.4	52.7	52.6	0.2
	G710															
	G711	1.13	0.94	0.91	0.99	0.12	4.81	4.60	5.58	5.00	0.52	48.3	47.8	51.5	49.2	2.0
	G712															
	G714	0.94	0.77	1.18	0.96	0.21	4.33	6.44	6.74	5.84	1.31					
	G716	0.78	0.96	1.04	0.93	0.13	1.92	2.98	2.48	2.46	0.53	47.8	47.4	56.4	50.5	5.1
	G717															
	G721															
	G722	2.82	2.87	2.76	2.82	0.06	8.74	9.84	8.82	9.13	0.61	163.3	176.9	182.5	174.2	9.9
	G724	1.16	1.28	1.43	1.29	0.14	6.28	6.68	6.16	6.38	0.27	50.8	49.3	49.9	50.0	0.8
	G727															
	G728															
	G729	1.16	0.79	0.69	0.88	0.25	5.13	5.04	3.66	4.61	0.82	62.3	38.8	57.6	52.9	12.4
	G730	1.50	1.30	1.30	1.37	0.12	6.30	6.10	6.20	6.20	0.10	57.5	56.9	58.4	57.6	0.8
	G731	1.19	1.63	1.20	1.34	0.25	5.56	5.57	5.44	5.52	0.07	54.5	53.5	53.2	53.7	0.6
	G732	1.18	1.25	1.15	1.19	0.05	5.67	5.88	5.65	5.73	0.13	49.8	50.7	51.8	50.7	1.0
	G733	1.31	1.45	1.35	1.37	0.07	5.95	6.39	6.23	6.19	0.22	52.8	55.5	51.5	53.3	2.0
	G738	2.40	1.30	1.10	1.60	0.70	6.20	7.30	7.00	6.83	0.57	89.0	119.0	150.0	119.3	30.5
	G739															
	G740															
	G742															
	G744	1.04	0.86	0.85	0.92	0.11	4.50	4.20	4.20	4.30	0.17	55.5	42.0	45.8	47.8	7.0
	G746	1.20	1.90	1.20	1.43	0.40	3.40	3.30	4.90	3.87	0.90	69.6	45.8	56.7	57.4	11.9
	G749	2.22			2.22		8.33	5.83	6.27	6.81	1.33	113.6	106.4	124.3	114.8	9.0
	G752															
	G753	1.11	1.17	1.17	1.15	0.03	6.35	6.37	6.50	6.41	0.08	51.0	52.8	52.0	51.9	0.9
	G755															
	G758															
	G760	1.05	1.30	1.15	1.16	0.12	4.89	5.09	4.97	4.98	0.10	64.5			64.5	
	G762	1.32	1.19	1.08	1.20	0.12	6.37	6.29	6.25	6.30	0.06	56.2	54.5	55.6	55.4	0.9
	G764															
	G766											47.0	50.1	42.7	46.6	3.7
	G767															
	G770											49.4	43.6	43.3	45.4	3.4
	G773	0.10	0.11	0.08	0.10	0.02	3.90	3.83	3.68	3.80	0.11	0.1	0.1	0.1	0.1	0.0
	G774															
	G775	0.90	0.95		0.93	0.04	6.29	5.91	6.60	6.27	0.35	54.2	52.0	51.4	52.5	1.5
Community Results		Consensus Mean			1.17		Consensus Mean			5.58		Consensus Mean			52.5	
		Consensus Standard Deviation			0.28		Consensus Standard Deviation			1.22		Consensus Standard Deviation			5.3	
		Maximum			2.82		Maximum			9.13		Maximum			174.2	
		Minimum			0.88		Minimum			2.46		Minimum			46.2	
		N			19		N			20		N			19	

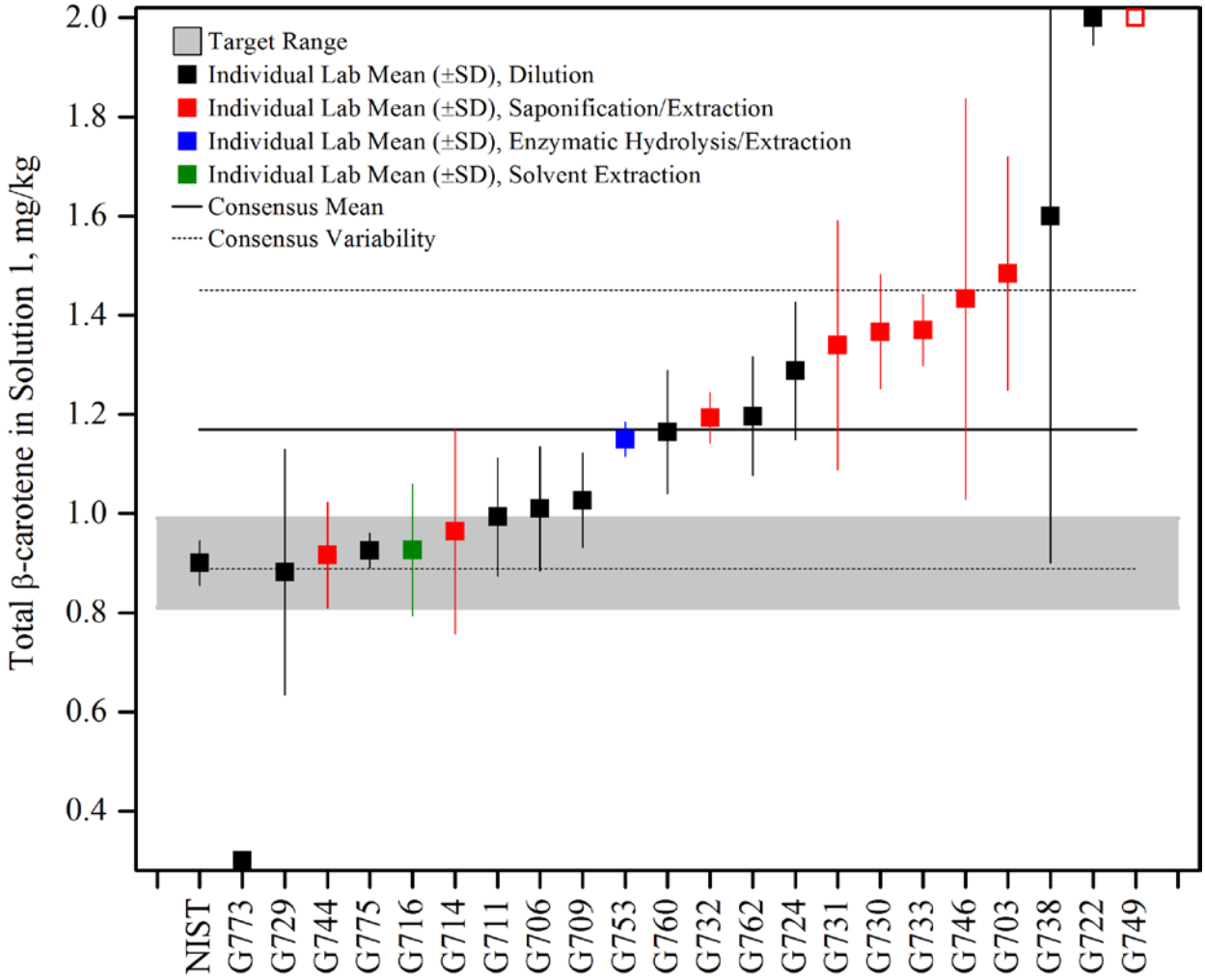

Figure 14. Total β-carotene in Solution 1 (data summary view – sample preparation method). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The data are identified by sample preparation method in this graph. The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean. The gray shaded region represents the target zone for "acceptable" performance, which encompasses the NIST assigned value bounded by twice its standard deviation.

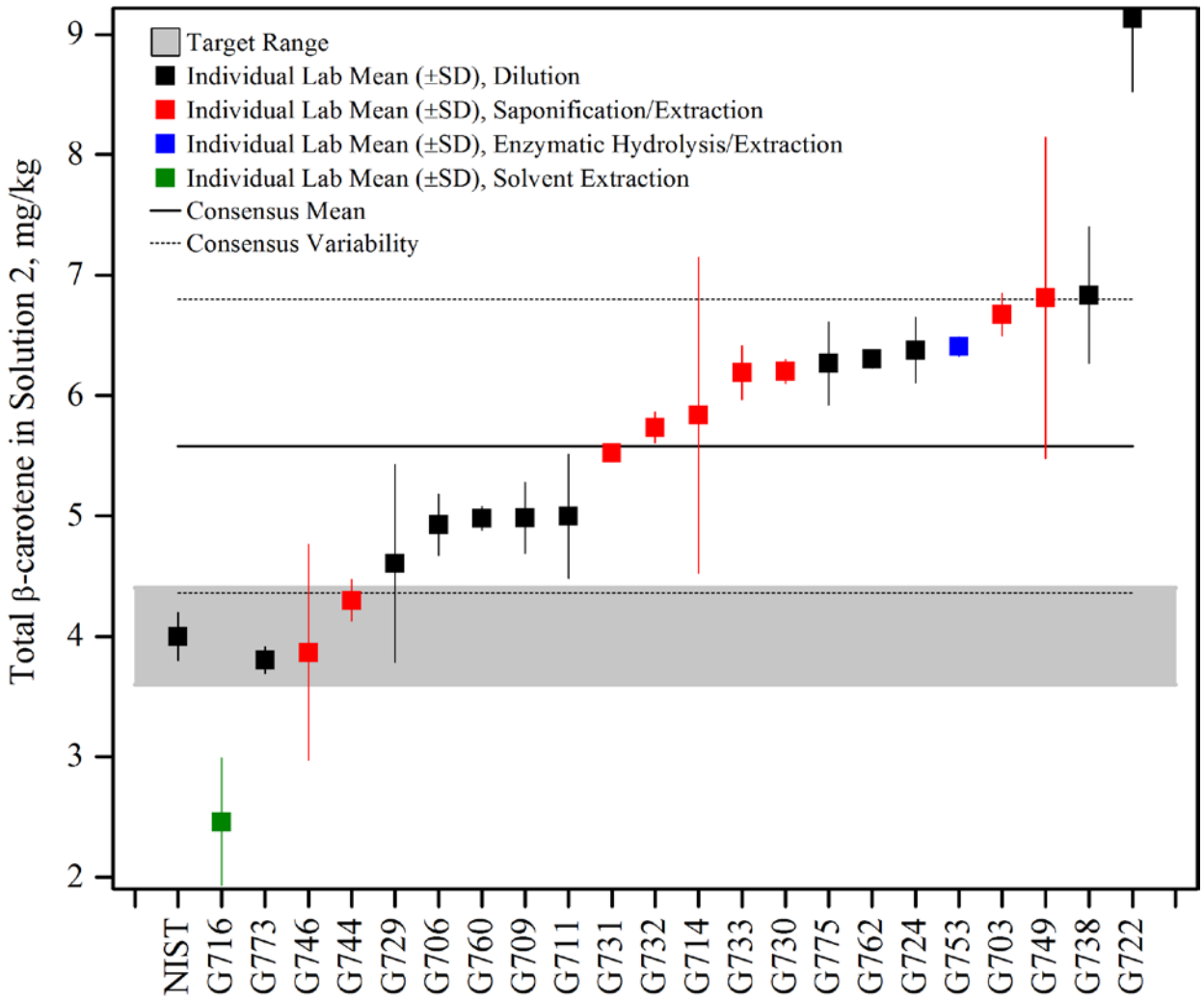

Figure 15. Total β-carotene in Solution 2 (data summary view – sample preparation method). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The data are identified by sample preparation method in this graph. The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean. The gray shaded region represents the target zone for "acceptable" performance, which encompasses the NIST assigned value bounded by twice its standard deviation.

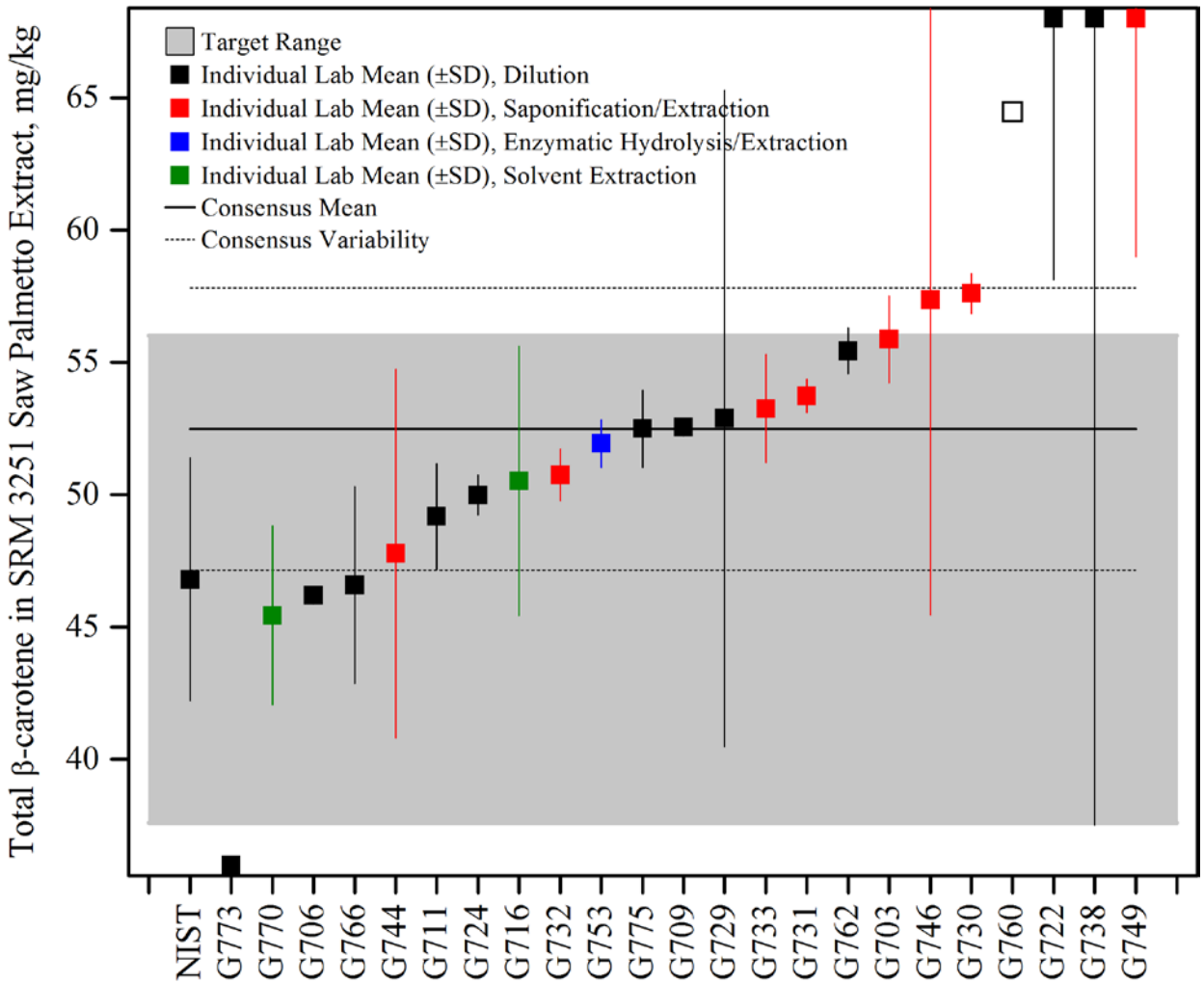

Figure 16. Total β-carotene in SRM 3251 *Serenoa repens* Extract (data summary view – sample preparation method). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The data are identified by sample preparation method in this graph. The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean. The gray shaded region represents the target zone for "acceptable" performance, which encompasses the NIST certified value bounded by twice its uncertainty (U_{95}).

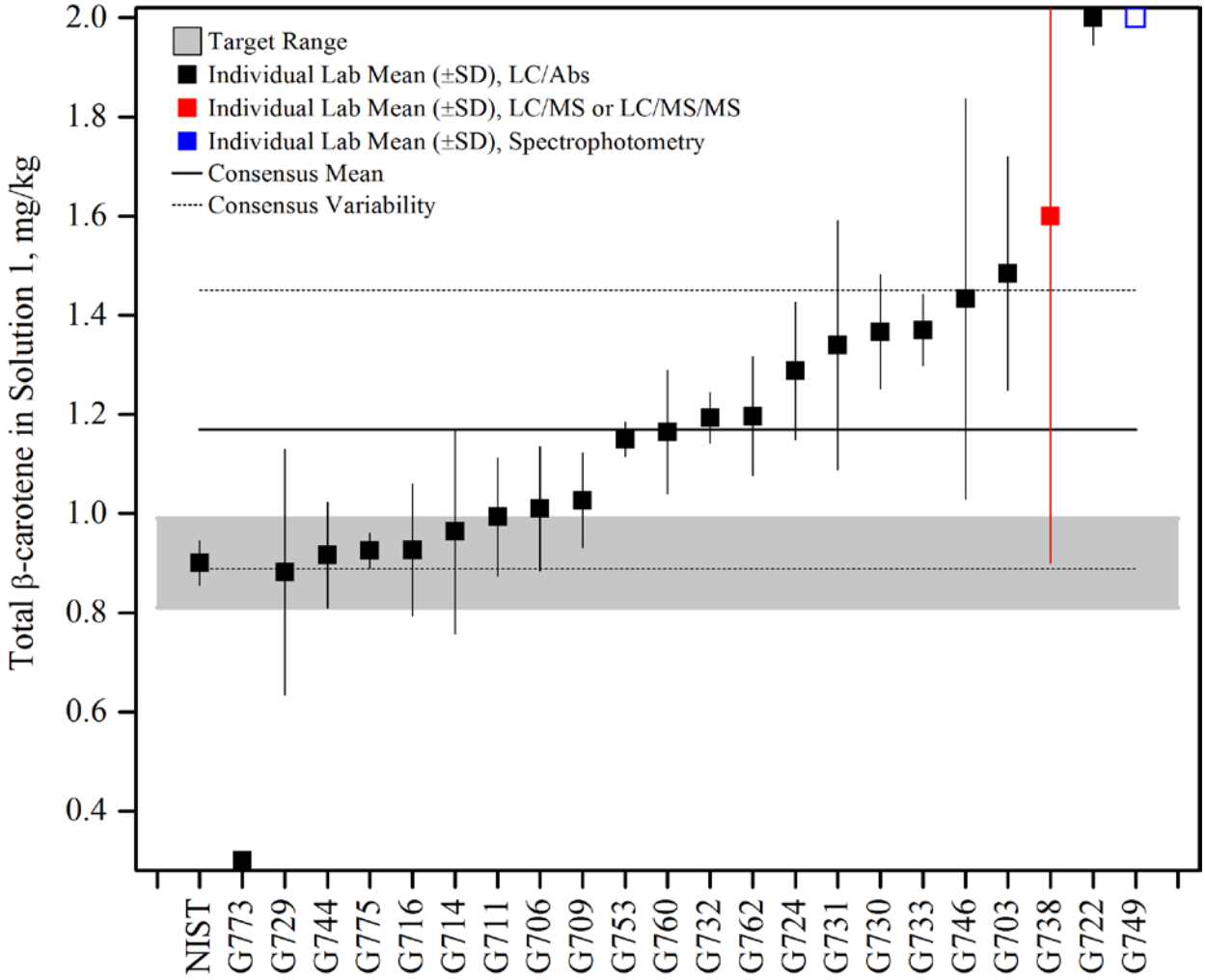

Figure 17. Total β-carotene in Solution 1 (data summary view – instrumental method). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The data are identified by instrumental method in this graph. The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean. The gray shaded region represents the target zone for "acceptable" performance, which encompasses the NIST assigned value bounded by twice its standard deviation.

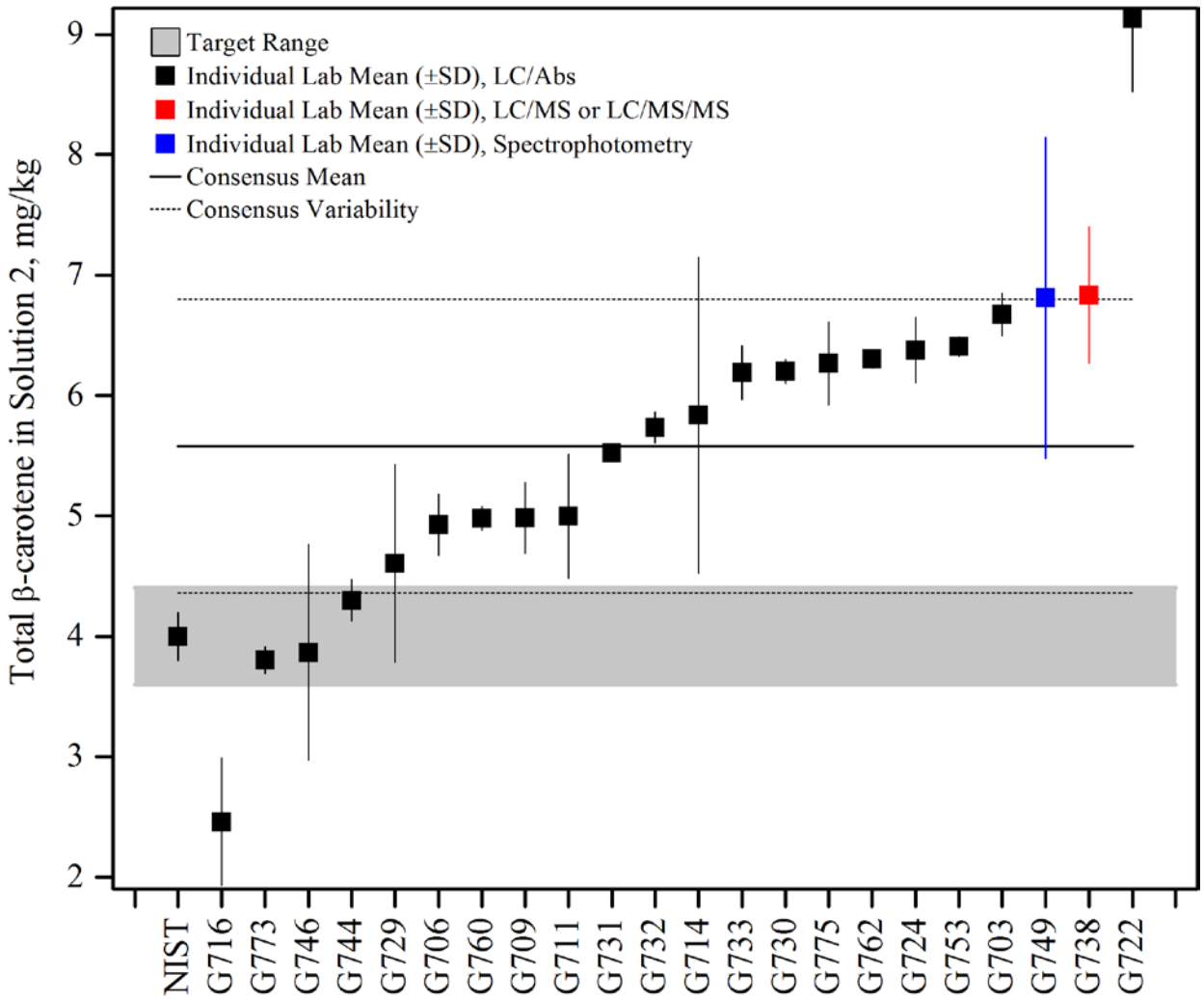

Figure 18. Total β-carotene in Solution 2 (data summary view – instrumental method). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The data are identified by instrumental method in this graph. The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean. The gray shaded region represents the target zone for "acceptable" performance, which encompasses the NIST assigned value bounded by twice its standard deviation.

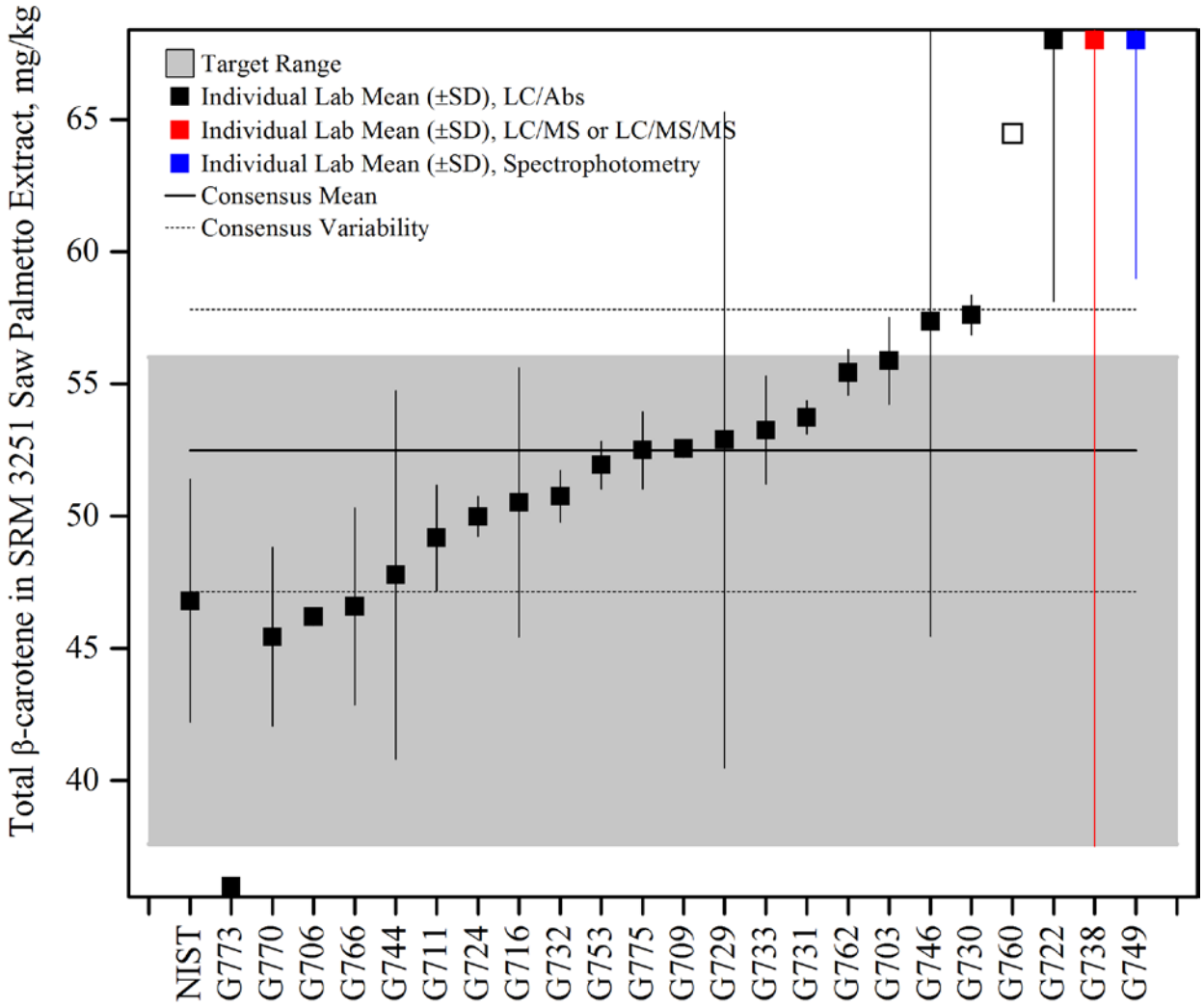

Figure 19. Total β-carotene in SRM 3251 *Serenoa repens* Extract (data summary view – instrumental method). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The data are identified by instrumental method in this graph. The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean. The gray shaded region represents the target zone for "acceptable" performance, which encompasses the NIST certified value bounded by twice its uncertainty (U_{95}).

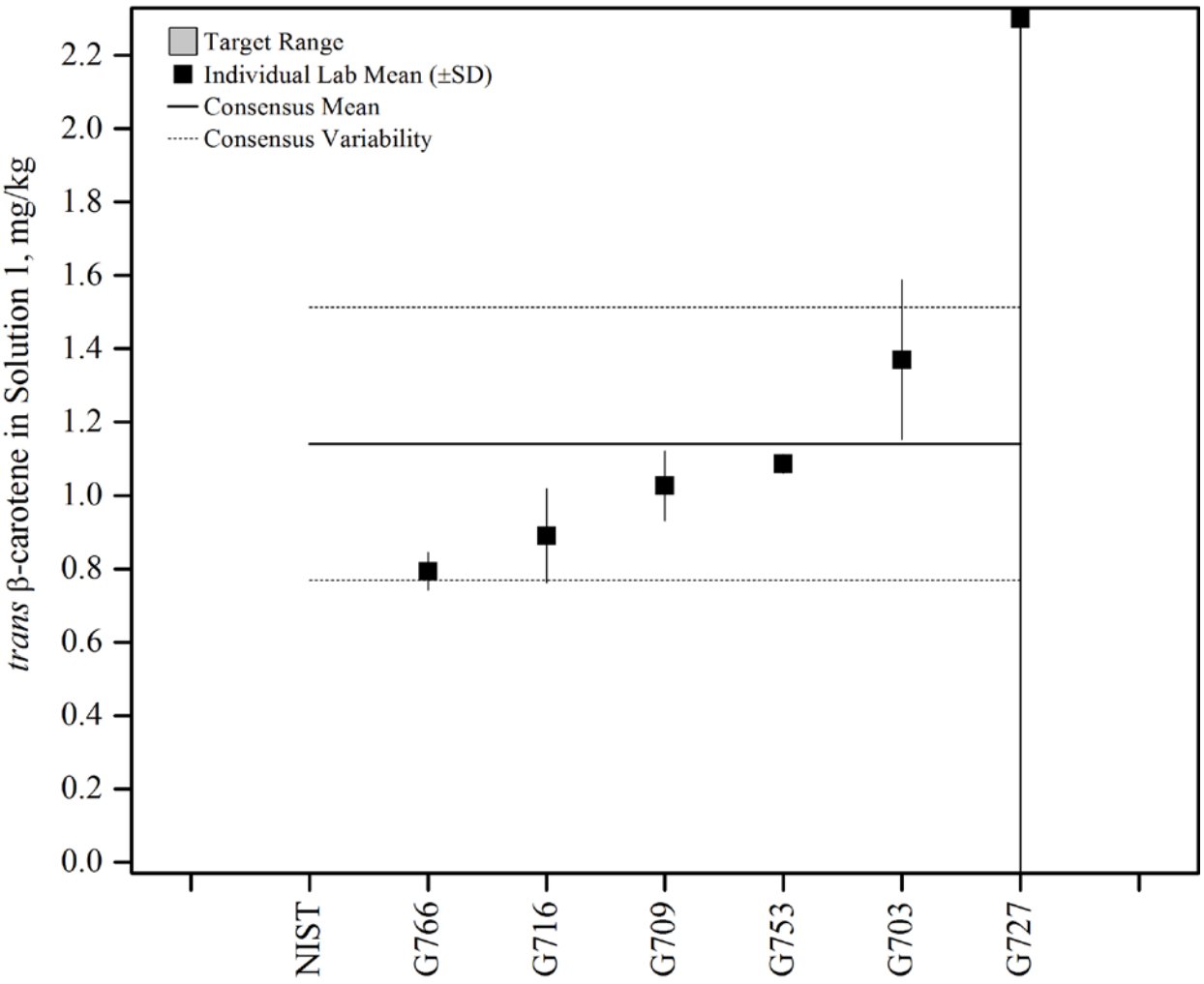

Figure 20. *trans*-β-carotene in Solution 1 (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

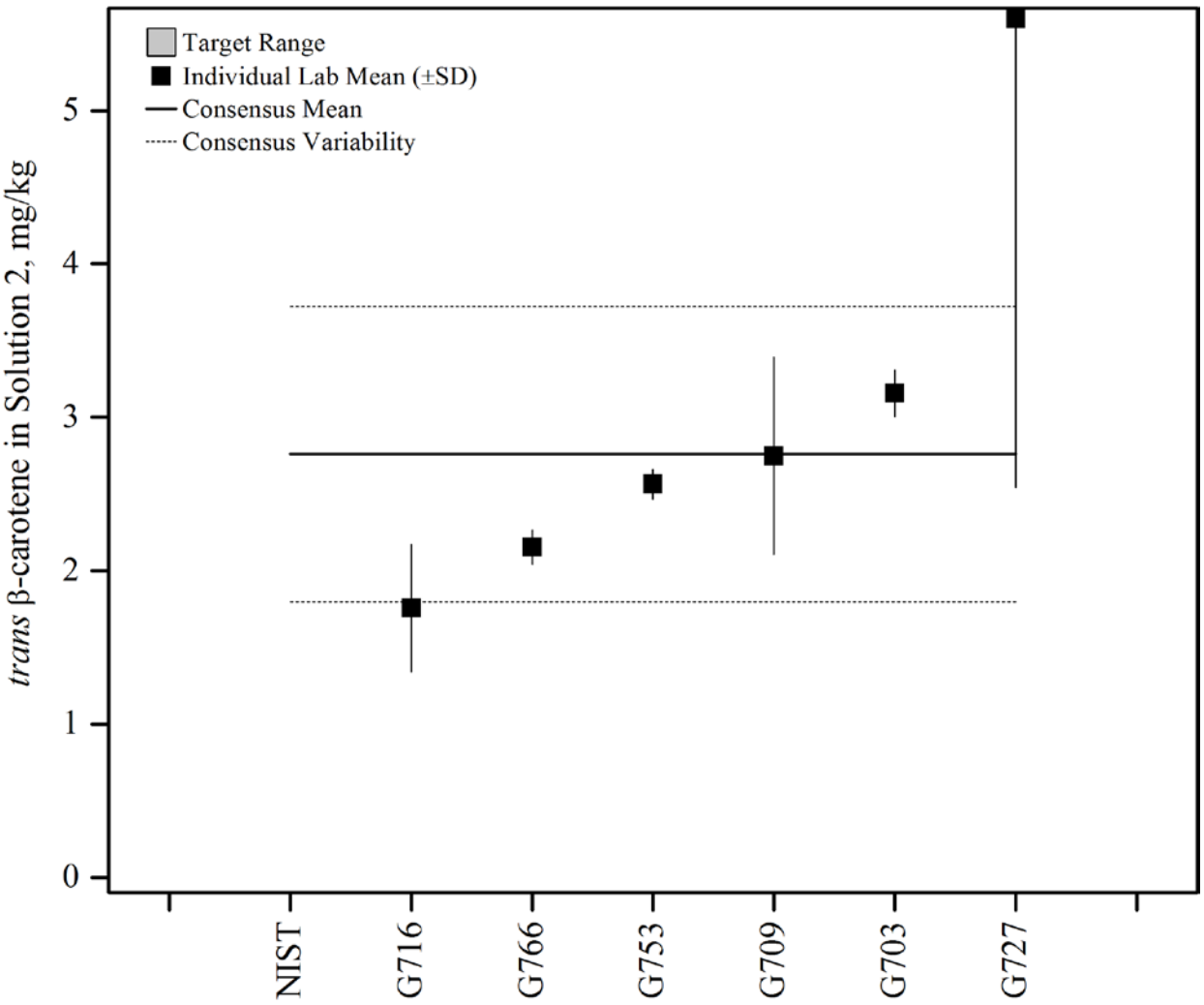

Figure 21. *trans*-β-carotene in Solution 2 (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

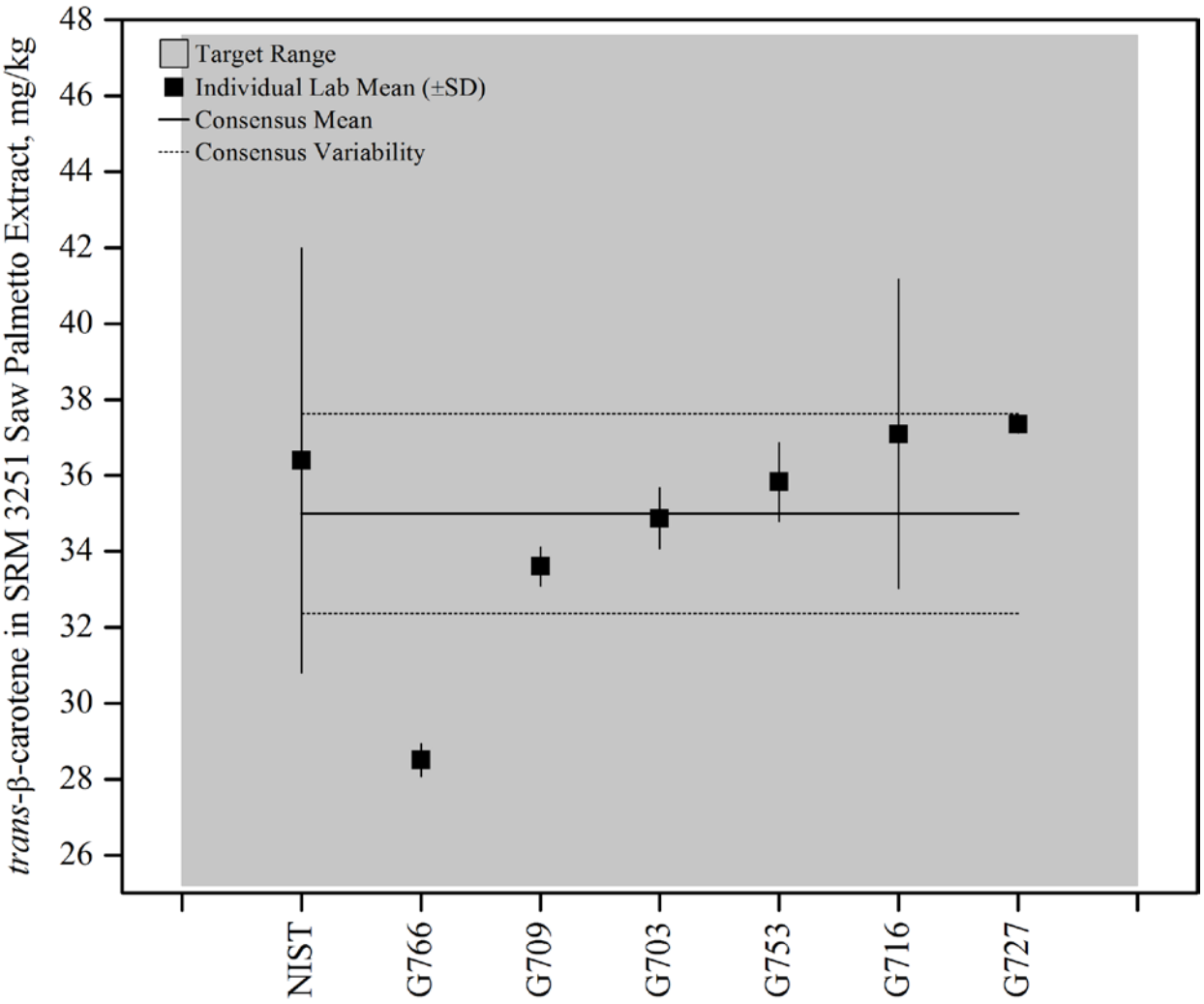

Figure 22. *trans*-β-carotene in SRM 3251 *Serenoa repens* Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean. The gray shaded region represents the target zone for "acceptable" performance, which encompasses the NIST certified value bounded by twice its uncertainty (U_{95}).

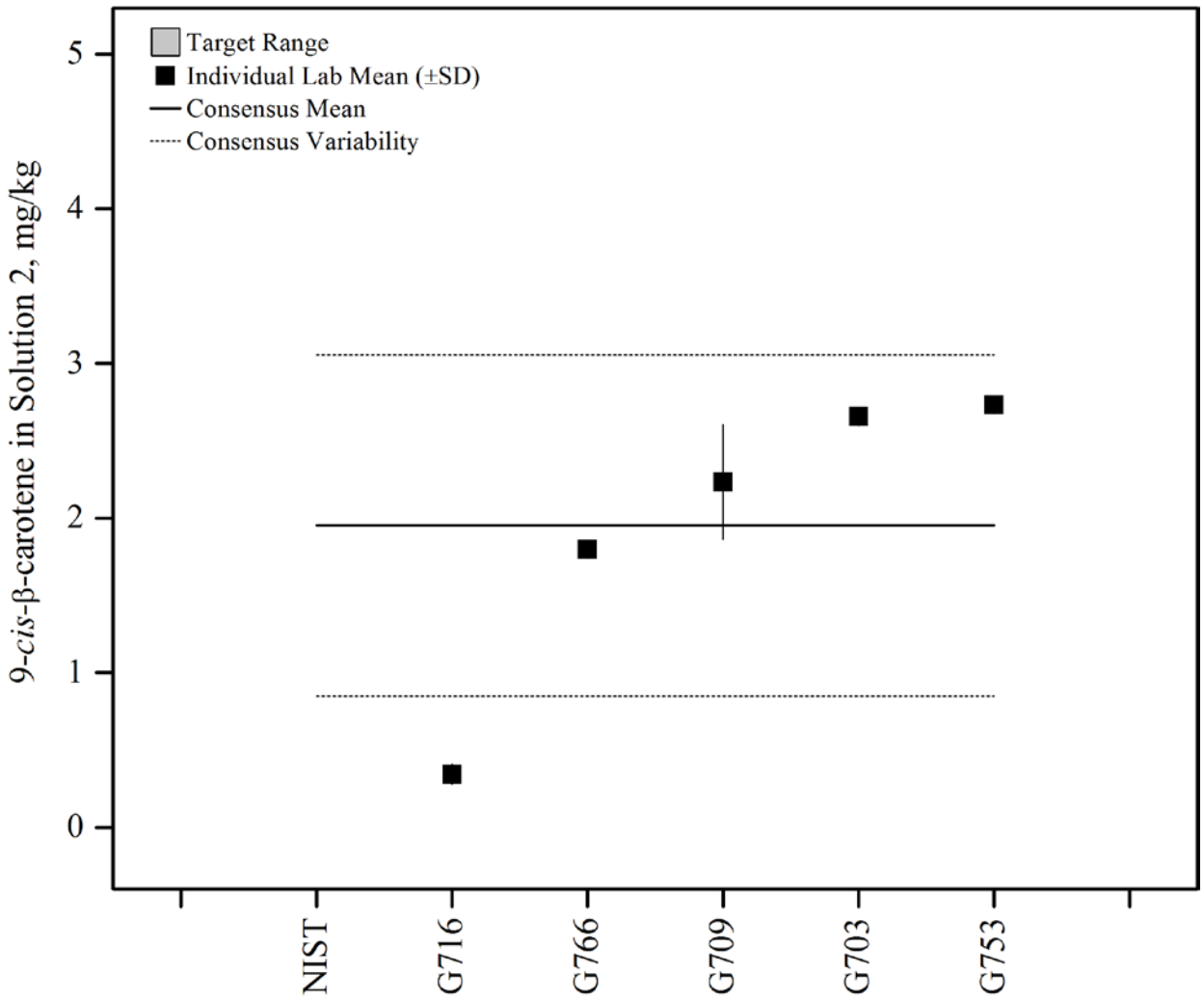

Figure 23. 9-*cis*-β-carotene in Solution 2 (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

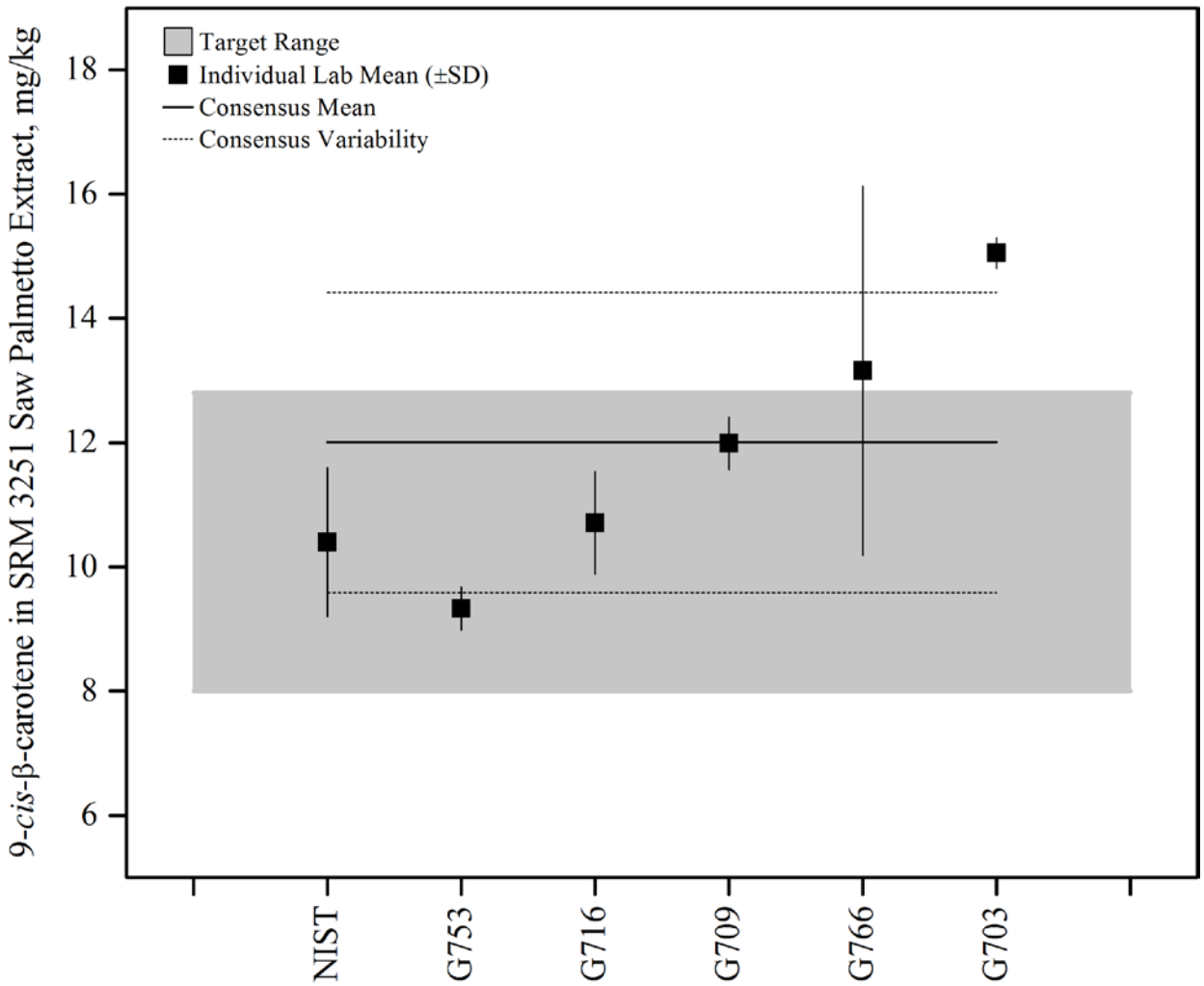

Figure 24. 9-*cis*--β-carotene in SRM 3251 *Serenoa repens* Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean. The gray shaded region represents the target zone for "acceptable" performance, which encompasses the NIST certified value bounded by twice its uncertainty (U_{95}).

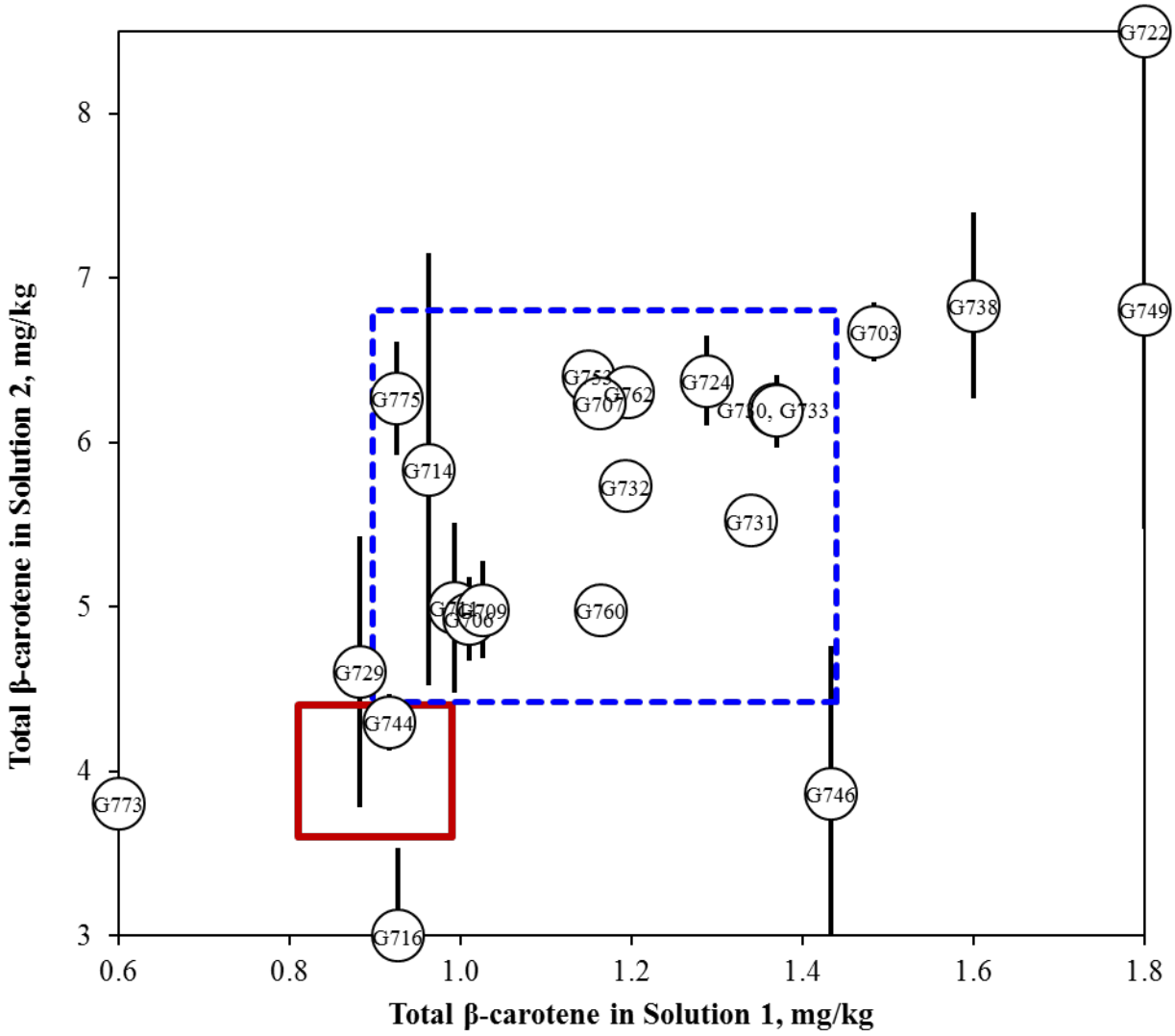

Figure 25. Total β-carotene in Solution 1 and Solution 2 (sample/sample comparison view). In this view, the individual laboratory results for one sample (β-carotene Solution 1) are compared to the results for a second sample (β-carotene Solution 2). The solid red box represents the target zone for solution 1 (x-axis) and solution 2 (y-axis). The dotted blue box represents the consensus zone for solution 1 (x-axis) and solution 2 (y-axis).

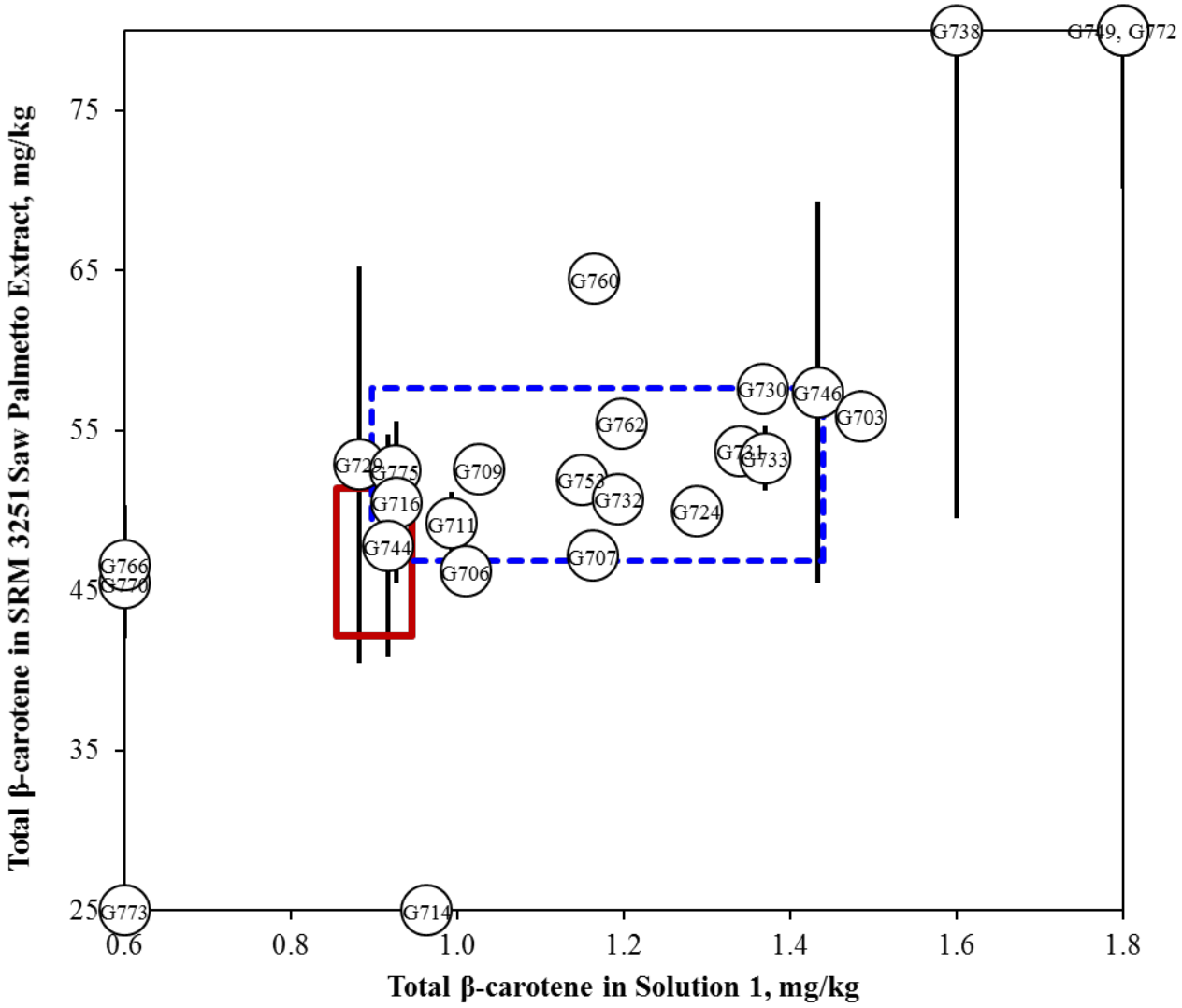

Figure 26. Total β-carotene in Solution 1 and SRM 3251 *Serenoa repens* Extract (sample/sample comparison view). In this view, the individual laboratory results for one sample (β-carotene Solution 1) are compared to the results for a second sample (saw palmetto extract). The solid red box represents the target zone for solution 1 (x-axis) and saw palmetto extract (y-axis). The dotted blue box represents the consensus zone for solution 1 (x-axis) and saw palmetto extract (y-axis).

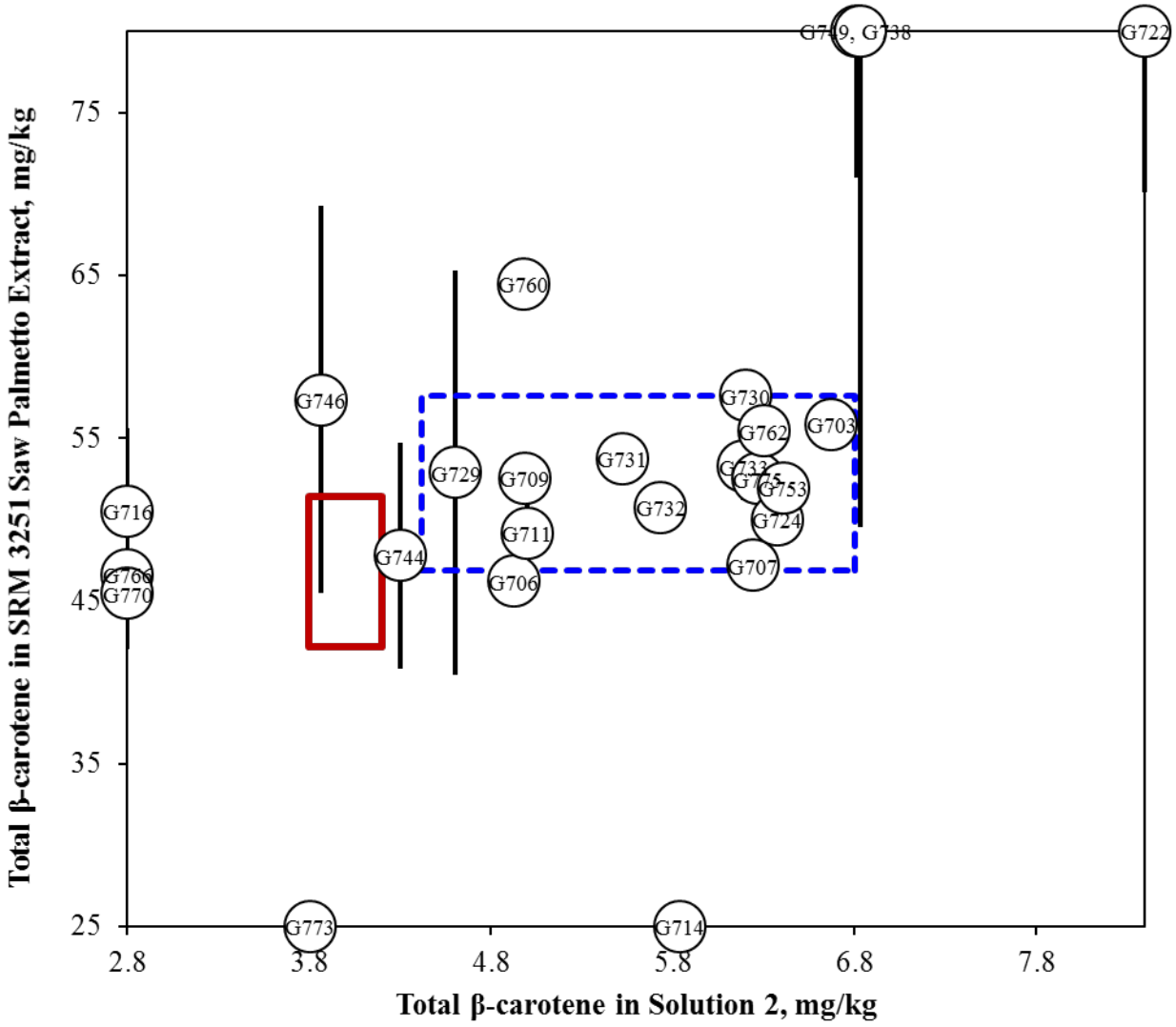

Figure 27. Total β-carotene in Solution 2 and SRM 3251 *Serenoa repens* Extract (sample/sample comparison view). In this view, the individual laboratory results for one sample (β-carotene Solution 2) are compared to the results for a second sample (saw palmetto extract). The solid red box represents the target zone for solution 2 (x-axis) and saw palmetto extract (y-axis). The dotted blue box represents the consensus zone for solution 2 (x-axis) and saw palmetto extract (y-axis).

ANTHOCYANINS & ANTHOCYANIDINS IN DIETARY SUPPLEMENTS

Study Overview

In this study, participants were provided with two NIST SRMs, SRM 3283 Cranberry Extract and SRM 3291 Bilberry Extract. Participants were asked to use in-house analytical methods to determine the mass fractions of anthocyanins and/or anthocyanidins in each of the matrices and report values on an as-received basis.

Sample Information

Cranberry Extract. Participants were provided with one packet containing approximately 2.5 g of cranberry extract. The cranberry extract was heat-sealed inside nitrogen-flushed 4 mil polyethylene bags, which were then sealed inside aluminized plastic bags with 2 packets of silica gel. Before use, participants were instructed to thoroughly mix the contents of the packet and use a sample size of at least 0.1 g. Participants were asked to store the material at controlled room temperature, 20 °C to 25 °C, and to prepare three samples and report three values from the single packet provided. Prior to the study, the approximate analyte levels were given as 1.8 mg/g cyanidin, 0.1 mg/g delphinidin, and 0.7 mg/g peonidin. NIST assigned values for anthocyanins and anthocyanidins were not available at the time of this report.

Bilberry Extract. Participants were provided with one packet containing approximately 1 g of bilberry extract. The bilberry extract was heat-sealed inside nitrogen-flushed 4 mil polyethylene bags, which were then sealed inside aluminized plastic bags with 2 packets of silica gel. Before use, participants were instructed to thoroughly mix the contents of the packet and use a sample size of at least 0.1 g. Participants were asked to store the material at controlled room temperature, 20 °C to 25 °C, and to prepare three samples and report three values from the single packet provided. Prior to the study, the approximate analyte levels were given as 40 mg/g cyanidin, 45 mg/g delphinidin, 25 mg/g malvidin, 10 mg/g peonidin, and 15 mg/g petunidin. NIST assigned values for anthocyanins and anthocyanidins were not available at the time of this report.

Study Results

- Thirty-four laboratories enrolled in this exercise and received samples.
- Five to ten laboratories reported results for anthocyanidins in both the cranberry extract and the bilberry extract (21 % to 38 % participation). One laboratory reported a single value for anthocyanidins in both samples and was therefore excluded from calculation of summary statistics.[2] Further discussion will only include studies in which at least eight laboratories reported data.
  - The consensus ranges for anthocyanidins in both matrices were wide (46 % to over 100 % RSD).
  - The consensus means were very low compared to the expected values reported prior to the study.
- Nine to fourteen laboratories reported results for anthocyanins in both the cranberry extract and the bilberry extract (27 % to 41 % participation).
  - The consensus ranges for anthocyanins in the cranberry extract ranged from acceptable (21 % RSD) to wide (29 % to over 100 % RSD).
  - The consensus ranges for anthocyanins in the bilberry extract ranged from acceptable (13 % to 23 % RSD) to wide (38 % to 56 % RSD).

- Nineteen laboratories reported results for total anthocyanins in both the cranberry extract and the bilberry extract (56 % participation). One laboratory reported a single value for total anthocyanins in both samples and was therefore excluded from calculation of summary statistics.[2]
  - The consensus range for total anthocyanins in the cranberry extract was wide (38 % RSD).
  - The consensus range for total anthocyanins in the bilberry extract was acceptable (22 % RSD).
- A majority of the laboratories reported using a solvent extraction approach (50 %) or a shaking or sonication extraction approach (39 %). Hydrolysis was also reported as a method of sample preparation by two laboratories (11 %).
- Most laboratories reported using LC with absorbance detection as the analytical method for analysis (78 %). Laboratories also reported using spectrophotometry (17 %) and a microbiological assay (6 %) as the instrumental approach.
- Most laboratories reported the use of an external standard approach to quantitation (89 %), while two laboratories reported calibration against a molar absorptivity.

Technical Recommendations

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

- The wide consensus ranges for the anthocyanidins may be related to the extraction approach utilized.
  - Laboratories using a hydrolysis approach were not included in the consensus calculations.
  - The variability in the data could be attributed to the extraction approach. The glycosides can be broken down into the aglycones, and differences in the extraction time, temperature, or solvent used could result in increased variability in the amount of glycosides converted to aglycones.
  - Many laboratories are not calibrating using reference standards for each of these compounds, and are instead using a single compound (e.g. cyanidin) for calibration of all five aglycones. If this were a major factor, the variability in the cyanidin consensus should be lower than that for the other aglycones, which was not observed.
- The wide consensus ranges for the anthocyanins may also be related to the extraction approach utilized.
  - Laboratories using a hydrolysis approach were not included in the consensus calculations.
  - As described above, the variability in the data could be attributed to the extraction approach. The glycosides can be broken down into the aglycones, and differences in the extraction time, temperature, or solvent used could result in increased variability in the amount of glycosides converted to aglycones.
  - Many laboratories are not calibrating using reference standards for each of these compounds, and are instead using a single compound (e.g. cyanidin glucoside) or a small subset of the glycosides for calibration of all fifteen glycosides. If this were a major factor, the variability in the cyanidin consensus should be lower than that for the other aglycones, which was not observed.

- After molar mass conversion to totals for each aglycone, the data for both hydrolysis and extraction approaches could be compared. In many cases, the consensus variability for a total aglycone was decreased compared to that for any of the other forms individually.
  - The value reported for each of the three glycosides was converted to the aglycone equivalent and summed, along with the value reported for the aglycone, to give a value for total aglycone.
  - The decrease in variability using this approach is further evidence in support of the theory provided above, of the differences in extraction procedure leading to increased variability. By reducing all forms to the base aglycone yields a sum that has a tighter consensus.
  - **Figure 73** does not reflect an obvious calibration error. Some laboratories reported values that were high for the one sample but were within range for the other sample. This type of trend may indicate individualized problems with the extraction or separation from one matrix compared to another.
- The consistency of the data is improved when all values are converted to a value for "equivalents" of each compound, and even further improved when converted to a value for total anthocyanidins.
  - The degree of sample pretreatment may affect the individual compounds measured, but would affect the total less. Harsher extraction conditions (elevated temperature, high acid content, etc.) could cause increased, unintended hydrolysis. Extraction conditions should be thoroughly optimized to ensure that conversion is not occurring unintentionally during the extraction process.
  - Lack of authentic standards for the large number of compounds may cause difficulties in chromatographic peak identification and quantitation. The best solution is to acquire as many standards as possible and use retention time (and *m/z*) to confirm peak identifications. Relying on literature or official methods has limitations, as variations in column chemistry, mobile phase composition, and temperature can all affect the chromatographic selectivity and therefore the retention times of all compounds.

Table 13. Individual data table (NIST) for anthocyanins and anthocyanidins in dietary supplements.

National Institute of Standards & Technology

Exercise G - July 2011 - Anthocyanins and Anthocyanidins

	Lab Code:	NIST	1. Your Results				2. Community Results			3. Target Value	
Analyte	Sample	Units	Mean	s_{total}	Z_{comm}	Z_{NIST}	N	Mean	Std Dev	Value	U_{95}
Cyanidin	Cranberry	mg/g					8	0.0859	0.0504	NR	NR
Cyanidin	Bilberry	mg/g					12	3.08	2.68	NR	NR
Delphinidin	Cranberry	mg/g					6	0.116	0.18	NR	NR
Delphinidin	Bilberry	mg/g					12	4.23	4.64	NR	NR
Malvidin	Cranberry	mg/g					2	0.0493	0.0269	NR	NR
Malvidin	Bilberry	mg/g					10	1.11	0.823	NR	NR
Peonidin	Cranberry	mg/g					6	0.0505	0.0045	NR	NR
Peonidin	Bilberry	mg/g					10	0.669	0.608	NR	NR
Petunidin	Cranberry	mg/g					2	0.133	0.0589	NR	NR
Petunidin	Bilberry	mg/g					11	0.757	0.348	NR	NR
Cyanidin Ara	Cranberry	mg/g					11	0.198	0.0574	NR	NR
Cyanidin Ara	Bilberry	mg/g					14	29.9	11.3	NR	NR
Cyanidin Gal	Cranberry	mg/g					13	0.132	0.0394	NR	NR
Cyanidin Gal	Bilberry	mg/g					14	35.5	7.32	NR	NR
Cyanidin Glu	Cranberry	mg/g					8	0.0586	0.0778	NR	NR
Cyanidin Glu	Bilberry	mg/g					13	43.2	13.3	NR	NR
Delphinidin Ara	Cranberry	mg/g					2	0.0615	0.0815	NR	NR
Delphinidin Ara	Bilberry	mg/g					13	37.3	7.64	NR	NR
Delphinidin Gal	Cranberry	mg/g					3	0.0187	0.029	NR	NR
Delphinidin Gal	Bilberry	mg/g					13	41.9	6.45	NR	NR
Delphinidin Glu	Cranberry	mg/g					4	0.0568	0.0621	NR	NR
Delphinidin Glu	Bilberry	mg/g					14	45.8	7.8	NR	NR
Malvidin Ara	Cranberry	mg/g					2	0.0514	0.0776	NR	NR
Malvidin Ara	Bilberry	mg/g					13	8.19	3.22	NR	NR
Malvidin Gal	Cranberry	mg/g					3	0.0595	0.0865	NR	NR
Malvidin Gal	Bilberry	mg/g					13	18.4	8.76	NR	NR
Malvidin Glu	Cranberry	mg/g					4	0.0881	0.0908	NR	NR
Malvidin Glu	Bilberry	mg/g					12	29.7	13.4	NR	NR
Peonidin Ara	Cranberry	mg/g					11	0.148	0.0479	NR	NR
Peonidin Ara	Bilberry	mg/g					12	2.44	0.921	NR	NR
Peonidin Gal	Cranberry	mg/g					12	0.201	0.0428	NR	NR
Peonidin Gal	Bilberry	mg/g					14	5.29	2.96	NR	NR
Peonidin Glu	Cranberry	mg/g					9	0.0467	0.0454	NR	NR
Peonidin Glu	Bilberry	mg/g					13	17.5	2.96	NR	NR
Petunidin Ara	Cranberry	mg/g					0			NR	NR
Petunidin Ara	Bilberry	mg/g					12	10.3	1.31	NR	NR
Petunidin Gal	Cranberry	mg/g					2	0.0993	0.109	NR	NR
Petunidin Gal	Bilberry	mg/g					11	16.8	3.84	NR	NR
Petunidin Glu	Cranberry	mg/g					4	0.0618	0.0998	NR	NR
Petunidin Glu	Bilberry	mg/g					14	32.7	5.96	NR	NR
Total Anthocyanins	Cranberry	mg/g					18	0.934	0.358	NR	NR
Total Anthocyanins	Bilberry	mg/g					18	354	78.2	NR	NR
Cyanidin	Cranberry	mg/g					15	0.259	0.103	NR	NR
Cyanidin	Bilberry	mg/g					15	76.5	23.7	NR	NR
Delphinidin	Cranberry	mg/g					9	0.134	0.166	NR	NR
Delphinidin	Bilberry	mg/g					15	84.7	14.4	NR	NR
Malvidin	Cranberry	mg/g					8	0.0624	0.0576	NR	NR
Malvidin	Bilberry	mg/g					15	34.2	12.7	NR	NR
Peonidin	Cranberry	mg/g					15	0.252	0.118	NR	NR
Peonidin	Bilberry	mg/g					15	17.6	4.37	NR	NR
Petunidin	Cranberry	mg/g					6	0.0942	0.0979	NR	NR
Petunidin	Bilberry	mg/g					15	38.4	7.06	NR	NR

Mean — Average of reported values

s_{total} — Standard deviation of reported values

Z_{comm} — Z-score: (Lab Mean - Consensus Mean)/ Consensus Standard Deviation

Z_{NIST} — Z-score: (Lab Mean - NIST Value or Label Claim)/ NIST or Label Claim Standard Deviation

NR — No data reported

N — Number of quantitative values reported

Mean — Robust mean of the reported values

Std Dev — Robust standard deviation

Value — NIST-assessed value

U_{95} — ±95% confidence interval about the assessed value

Table 14. Data summary table for cyanidin in dietary supplements.

		Cyanidin									
		SRM 3283 Cranberry Extract (mg/g)					SRM 3291 Bilberry Extract (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	NIST										
	G701										
	G702										
	G703	0.068	0.067	0.069	0.068	0.001	3.33	3.31	3.36	3.33	0.03
	G704										
	G705	0.039	0.043	0.038	0.040	0.003	25.60	25.40	25.50	25.50	0.10
	G707										
	G708										
	G715	0.095	0.098		0.097	0.002	1.81	1.75		1.78	0.05
	G716										
	G717	0.053	0.051	0.048	0.050	0.002	0.25	0.25	0.25	0.25	0.00
	G718	0.060	0.059	0.057	0.059	0.002	0.74	0.64	0.67	0.68	0.05
	G722										
	G724										
	G728						2.10	1.83	1.82	1.92	0.16
	G729										
	G738										
	G739										
	G740										
	G749										
	G752										
	G753										
	G757										
	G760										
	G762	0.126	0.128	0.126	0.127	0.001	3.88	3.60	3.41	3.63	0.24
	G765						1.26	1.20	1.30	1.25	0.05
	G766										
	G767										
	G771										
	G772										
	G773						4.73	4.86	4.47	4.69	0.20
	G774										
	G775						2.22	2.23	2.28	2.25	0.03
	G776										
	G777	0.173	0.169	0.172	0.171	0.002	48.45	48.35	48.58	48.46	0.11
Community Results		Consensus Mean			0.0859		Consensus Mean			3.08	
		Consensus Standard Deviation			0.0504		Consensus Standard Deviation			2.68	
		Maximum			0.1713		Maximum			48.46	
		Minimum			0.0402		Minimum			0.25	
		N			8		N			12	

Table 15. Data summary table for cyanidin-3-arabinoside in dietary supplements.

		Cyanidin-3-Arabinoside									
		SRM 3283 Cranberry Extract (mg/g)					SRM 3291 Bilberry Extract (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	NIST										
	G701	0.150	0.160	0.160	0.157	0.006	53.2	53.1	48.4	51.6	2.7
	G702										
	G703	0.134	0.132	0.133	0.133	0.001	20.0	20.3	20.3	20.2	0.2
	G704										
	G705	0.157	0.175	0.154	0.162	0.011	42.8	42.5	42.5	42.6	0.2
	G707										
	G708	0.217	0.211	0.217	0.215	0.003	26.7	26.5	26.6	26.6	0.1
	G715						20.9	19.5		20.2	0.9
	G716										
	G717	0.210	0.211	0.215	0.212	0.003	21.5	20.9	19.3	20.6	1.2
	G718	0.235	0.236	0.235	0.235	0.001	25.2	24.2	25.1	24.8	0.6
	G722										
	G724										
	G728	0.362	0.283	0.369	0.338	0.048	34.5	39.0	32.7	35.4	3.2
	G729										
	G738										
	G739										
	G740										
	G749										
	G752										
	G753										
	G757										
	G760										
	G762	0.162	0.175	0.171	0.169	0.007	28.5	28.4	26.0	27.6	1.4
	G765	0.231	0.237	0.253	0.240	0.011	43.7	41.9	42.5	42.7	0.9
	G766	0.242	0.240	0.228	0.237	0.008	27.5	26.6	26.7	26.9	0.5
	G767										
	G771										
	G772										
	G773		0.130	0.130	0.130	0.000	19.7	19.7	17.5	19.0	1.3
	G774										
	G775						23.0	23.0	23.0	23.0	0.0
	G776										
	G777						42.3	42.4	42.3	42.3	0.1
Community Results		Consensus Mean			0.198		Consensus Mean			29.9	
		Consensus Standard Deviation			0.057		Consensus Standard Deviation			11.3	
		Maximum			0.338		Maximum			51.6	
		Minimum			0.130		Minimum			19.0	
		N			11		N			14	

Table 16. Data summary table for cyanidin-3-galactoside in dietary supplements.

		Cyanidin-3-Galactoside									
		SRM 3283 Cranberry Extract (mg/g)					SRM 3291 Bilberry Extract (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	NIST										
	G701	0.090	0.087	0.090	0.089	0.002	58.9	50.5	48.1	52.5	5.7
	G702										
	G703	0.099	0.099	0.097	0.098	0.001	27.5	27.9	27.6	27.7	0.2
	G704										
	G705	0.022	0.024	0.020	0.022	0.002	36.8	36.4	36.3	36.5	0.3
	G707										
	G708	0.149	0.150	0.155	0.151	0.003	26.3	27.1	27.0	26.8	0.4
	G715	0.140	0.148		0.144	0.006	35.0	35.0		35.0	0.0
	G716										
	G717	0.149	0.144	0.145	0.146	0.002	34.3	34.0	33.7	34.0	0.3
	G718	0.152	0.159	0.159	0.157	0.004	34.3	33.2	34.5	34.0	0.7
	G722										
	G724										
	G728	0.207	0.164	0.189	0.187	0.022	43.8	42.9	44.6	43.7	0.9
	G729										
	G738										
	G739										
	G740										
	G749										
	G752										
	G753										
	G757										
	G760										
	G762	0.110	0.113	0.111	0.111	0.002	37.4	37.2	34.4	36.3	1.7
	G765	0.155	0.159	0.178	0.164	0.012	58.5	55.8	56.7	57.0	1.4
	G766	0.173	0.152	0.156	0.160	0.011	38.1	37.7	37.8	37.9	0.2
	G767										
	G771										
	G772										
	G773	0.130	0.140	0.160	0.143	0.015	27.3	28.3	25.4	27.0	1.5
	G774										
	G775						31.1	31.1	31.1	31.1	0.0
	G776										
	G777	0.094	0.092	0.096	0.094	0.002	34.7	34.9	34.8	34.8	0.1
Community Results		Consensus Mean			0.132		Consensus Mean			35.5	
		Consensus Standard Deviation			0.039		Consensus Standard Deviation			7.3	
		Maximum			0.187		Maximum			57.0	
		Minimum			0.022		Minimum			26.8	
		N			13		N			14	

Table 17. Data summary table for cyanidin-3-glucoside in dietary supplements.

		Cyanidin-3-Glucoside									
		SRM 3283 Cranberry Extract (mg/g)					SRM 3291 Bilberry Extract (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	NIST										
	G701	0.0086	0.0062	0.0068	0.0072	0.0012	93.4	83.5	88.0	88.3	4.9
	G702										
	G703	0.0136			0.0136		31.1	31.5	31.5	31.4	0.2
	G704										
	G705										
	G707										
	G708	0.0102	0.0100	0.0100	0.0101	0.0001	38.6	38.4	38.5	38.5	0.1
	G715						38.0	37.8		37.9	0.1
	G716										
	G717	0.0111	0.0108	0.0116	0.0112	0.0004	38.1	37.6	37.3	37.7	0.4
	G718	0.1110	0.1110	0.0930	0.1050	0.0104	93.1	90.0	93.3	92.1	1.9
	G722										
	G724										
	G728						47.2	46.0	48.3	47.2	1.1
	G729										
	G738										
	G739										
	G740										
	G749										
	G752										
	G753										
	G757										
	G760										
	G762	0.0064	0.0068	0.0080	0.0071	0.0008	40.9	40.8	37.7	39.8	1.8
	G765	0.0180	0.0180	0.0160	0.0173	0.0012	62.8	60.1	60.7	61.2	1.4
	G766						42.0	41.0	41.1	41.4	0.5
	G767										
	G771										
	G772										
	G773						30.3	30.2	27.2	29.2	1.8
	G774										
	G775	0.1711	0.1740	0.1643	0.1698	0.0050	33.9	34.0	34.0	34.0	0.1
	G776										
	G777	0.1400	0.1390	0.1440	0.1410	0.0026	38.1	38.2	38.2	38.2	0.0
Community Results		Consensus Mean			0.0586		Consensus Mean			43.2	
		Consensus Standard Deviation			0.0778		Consensus Standard Deviation			13.3	
		Maximum			0.1698		Maximum			92.1	
		Minimum			0.0071		Minimum			29.2	
		N			8		N			13	

Table 18. Data summary table for cyanidin equivalents in dietary supplements.

		Cyanidin Equivalents									
		SRM 3283 Cranberry Extract (mg/g)					SRM 3291 Bilberry Extract (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	NIST										
	G701	0.166	0.169	0.171	0.169	0.003	133.7	122.0	120.1	125.3	7.4
	G702										
	G703	0.232	0.221	0.222	0.225	0.006	54.5	55.2	55.0	54.9	0.4
	G704										
	G705	0.161	0.179	0.156	0.165	0.012	78.4	77.8	77.8	78.0	0.4
	G707										
	G708	0.250	0.247	0.254	0.250	0.004	59.8	60.0	60.1	60.0	0.2
	G715	0.184	0.193		0.189	0.006	62.7	61.7		62.2	0.8
	G716										
	G717	0.299	0.294	0.295	0.296	0.002	61.3	60.3	58.8	60.1	1.2
	G718	0.389	0.393	0.379	0.387	0.007	99.4	95.9	99.6	98.3	2.1
	G722										
	G724										
	G728	0.380	0.299	0.374	0.351	0.045	83.9	85.3	83.6	84.3	0.9
	G729										
	G738	0.297			0.297		34.5			34.5	
	G739										
	G740										
	G749										
	G752										
	G753										
	G757										
	G760										
	G762	0.311	0.325	0.319	0.318	0.007	73.5	72.9	67.3	71.2	3.4
	G765	0.269	0.275	0.297	0.281	0.015	108.7	104.0	105.4	106.1	2.4
	G766	0.276	0.262	0.256	0.265	0.011	70.0	68.5	68.7	69.1	0.8
	G767										
	G771										
	G772	1.174	1.172	1.133	1.160	0.023	64.2	67.5	67.7	66.5	2.0
	G773	0.083	0.179	0.191	0.151	0.059	55.0	55.7	50.1	53.6	3.1
	G774										
	G775	0.109	0.111	0.105	0.109	0.003	59.5	59.6	59.7	59.6	0.1
	G776										
	G777	0.323	0.317	0.325	0.322	0.004	124.0	124.1	124.2	124.1	0.1
Community Results		Consensus Mean			0.259		Consensus Mean			76.5	
		Consensus Standard Deviation			0.103		Consensus Standard Deviation			23.7	
		Maximum			1.160		Maximum			125.3	
		Minimum			0.109		Minimum			34.5	
		N			15		N			15	

Table 19. Data summary table for delphinidin in dietary supplements.

		Delphinidin									
		SRM 3283 Cranberry Extract (mg/g)					SRM 3291 Bilberry Extract (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	NIST										
	G701										
	G702										
	G703	0.011	0.015		0.013	0.003	4.50	4.39	4.48	4.46	0.06
	G704										
	G705						4.08	3.36	3.29	3.58	0.44
	G707										
	G708										
	G715						0.63	1.33		0.98	0.49
	G716										
	G717	0.007	0.007	0.006	0.007	0.000	0.91	0.83	0.92	0.89	0.05
	G718						0.97	0.92	0.96	0.95	0.03
	G722										
	G724										
	G728						1.17	1.21	1.23	1.20	0.03
	G729										
	G738										
	G739										
	G740										
	G749										
	G752										
	G753										
	G757										
	G760										
	G762	0.038	0.039	0.037	0.038	0.001	3.38	3.54	3.12	3.35	0.21
	G765						0.47	0.43	0.45	0.45	0.02
	G766										
	G767										
	G771										
	G772										
	G773	0.150	0.130		0.140	0.014	10.30	10.60	7.98	9.63	1.43
	G774										
	G775						2.92	3.00	3.01	2.97	0.05
	G776										
	G777	0.557	0.547	0.573	0.559	0.013	17.37	17.36	17.20	17.31	0.10
Community Results		Consensus Mean			0.116		Consensus Mean			4.23	
		Consensus Standard Deviation			0.180		Consensus Standard Deviation			4.64	
		Maximum			0.559		Maximum			17.31	
		Minimum			0.007		Minimum			0.45	
		N			6		N			12	

Table 20. Data summary table for delphinidin-3-arabinoside in dietary supplements.

		Cyanidin Equivalents									
		SRM 3283 Cranberry Extract (mg/g)					SRM 3291 Bilberry Extract (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	NIST										
	G701	0.166	0.169	0.171	0.169	0.003	133.7	122.0	120.1	125.3	7.4
	G702										
	G703	0.232	0.221	0.222	0.225	0.006	54.5	55.2	55.0	54.9	0.4
	G704										
	G705	0.161	0.179	0.156	0.165	0.012	78.4	77.8	77.8	78.0	0.4
	G707										
	G708	0.250	0.247	0.254	0.250	0.004	59.8	60.0	60.1	60.0	0.2
	G715	0.184	0.193		0.189	0.006	62.7	61.7		62.2	0.8
	G716										
	G717	0.299	0.294	0.295	0.296	0.002	61.3	60.3	58.8	60.1	1.2
	G718	0.389	0.393	0.379	0.387	0.007	99.4	95.9	99.6	98.3	2.1
	G722										
	G724										
	G728	0.380	0.299	0.374	0.351	0.045	83.9	85.3	83.6	84.3	0.9
	G729										
	G738	0.297			0.297		34.5			34.5	
	G739										
	G740										
	G749										
	G752										
	G753										
	G757										
	G760										
	G762	0.311	0.325	0.319	0.318	0.007	73.5	72.9	67.3	71.2	3.4
	G765	0.269	0.275	0.297	0.281	0.015	108.7	104.0	105.4	106.1	2.4
	G766	0.276	0.262	0.256	0.265	0.011	70.0	68.5	68.7	69.1	0.8
	G767										
	G771										
	G772	1.174	1.172	1.133	1.160	0.023	64.2	67.5	67.7	66.5	2.0
	G773	0.083	0.179	0.191	0.151	0.059	55.0	55.7	50.1	53.6	3.1
	G774										
	G775	0.109	0.111	0.105	0.109	0.003	59.5	59.6	59.7	59.6	0.1
	G776										
	G777	0.323	0.317	0.325	0.322	0.004	124.0	124.1	124.2	124.1	0.1
Community Results		Consensus Mean			0.259		Consensus Mean			76.5	
		Consensus Standard Deviation			0.103		Consensus Standard Deviation			23.7	
		Maximum			1.160		Maximum			125.3	
		Minimum			0.109		Minimum			34.5	
		N			15		N			15	

Table 21. Data summary table for delphinidin-3-galactoside in dietary supplements.

		Delphinidin-3-Galactoside									
		SRM 3283 Cranberry Extract (mg/g)					SRM 3291 Bilberry Extract (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	NIST										
	G701										
	G702										
	G703						35.2	35.7	35.6	35.5	0.3
	G704										
	G705						47.5	47.0	47.2	47.2	0.3
	G707										
	G708						43.6	43.6	43.7	43.6	0.1
	G715						42.2	42.5		42.4	0.2
	G716										
	G717						44.4	43.9	43.6	44.0	0.4
	G718						42.7	41.4	43.0	42.3	0.9
	G722										
	G724										
	G728						48.7	47.7	48.3	48.2	0.5
	G729										
	G738										
	G739										
	G740										
	G749										
	G752										
	G753										
	G757										
	G760										
	G762	0.0020	0.0069	0.0030	0.0040	0.0026	45.1	45.3	41.6	44.0	2.1
	G765						61.1	58.4	59.2	59.6	1.4
	G766						31.0	30.9	31.0	30.9	0.1
	G767										
	G771										
	G772										
	G773						34.7	34.8	31.4	33.6	1.9
	G774										
	G775	0.0488	0.0495	0.0464	0.0482	0.0016	38.0	38.3	38.3	38.2	0.2
	G776										
	G777	0.0040	0.0040	0.0040	0.0040	0.0000	42.1	42.1	42.1	42.1	0.0
Community Results		Consensus Mean			0.0187		Consensus Mean			41.9	
		Consensus Standard Deviation			0.0290		Consensus Standard Deviation			6.4	
		Maximum			0.0482		Maximum			59.6	
		Minimum			0.0040		Minimum			30.9	
		N			3		N			13	

Table 22. Data summary table for delphinidin-3-glucoside in dietary supplements.

		Delphinidin-3-Glucoside									
		SRM 3283 Cranberry Extract (mg/g)					SRM 3291 Bilberry Extract (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	NIST										
	G701						64.7	52.3	53.2	56.7	6.9
	G702										
	G703						38.1	38.6	38.6	38.4	0.3
	G704										
	G705						50.9	50.5	50.6	50.7	0.2
	G707										
	G708						46.5	46.2	46.5	46.4	0.2
	G715						45.2	45.2		45.2	0.0
	G716										
	G717						47.0	46.4	46.2	46.5	0.4
	G718	0.0530	0.0510	0.0570	0.0537	0.0031	45.7	44.2	45.9	45.2	0.9
	G722										
	G724										
	G728						52.2	51.1	51.3	51.5	0.6
	G729										
	G738										
	G739										
	G740										
	G749										
	G752										
	G753										
	G757										
	G760										
	G762	0.0242	0.0202	0.0198	0.0214	0.0024	48.3	48.4	44.5	47.1	2.2
	G765						64.7	61.8	62.7	63.1	1.5
	G766						33.1	32.8	33.0	33.0	0.1
	G767										
	G771										
	G772										
	G773						36.7	36.9	33.4	35.7	2.0
	G774										
	G775	0.1363	0.1370	0.1324	0.1352	0.0025	40.3	40.7	40.7	40.6	0.2
	G776										
	G777	0.0170	0.0160	0.0180	0.0170	0.0010	44.9	45.1	45.1	45.1	0.1
Community Results		Consensus Mean			0.0568		Consensus Mean			45.8	
		Consensus Standard Deviation			0.0621		Consensus Standard Deviation			7.8	
		Maximum			0.1352		Maximum			63.1	
		Minimum			0.0170		Minimum			33.0	
		N			4		N			14	

Table 23. Data summary table for delphinidin equivalents in dietary supplements.

		Delphinidin Equivalents									
		SRM 3283 Cranberry Extract (mg/g)					SRM 3291 Bilberry Extract (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	NIST										
	G701						42.2	34.0	34.7	37.0	4.5
	G702										
	G703	0.011	0.015		0.013	0.003	72.8	73.6	73.8	73.4	0.5
	G704										
	G705	0.076	0.085	0.074	0.078	0.006	96.8	95.2	95.5	95.9	0.8
	G707										
	G708						92.3	91.3	91.9	91.8	0.5
	G715						83.5	84.5		84.0	0.7
	G716										
	G717	0.007	0.007	0.006	0.007	0.000	87.3	86.0	85.7	86.3	0.8
	G718	0.035	0.033	0.037	0.035	0.002	83.8	81.1	84.2	83.0	1.7
	G722										
	G724										
	G728						96.9	94.6	95.3	95.6	1.2
	G729										
	G738	0.087			0.087		41.7			41.7	
	G739										
	G740										
	G749										
	G752										
	G753										
	G757										
	G760										
	G762	0.055	0.056	0.052	0.054	0.002	92.2	92.4	84.9	89.8	4.3
	G765						120.4	114.9	116.6	117.3	2.8
	G766						60.0	59.5	59.8	59.7	0.2
	G767										
	G771										
	G772	0.384	0.383	0.369	0.379	0.008	83.3	86.8	86.9	85.7	2.0
	G773	0.150	0.130		0.140	0.014	77.5	77.7	68.5	74.6	5.2
	G774										
	G775	0.121	0.122	0.117	0.120	0.003	76.8	77.5	77.6	77.3	0.4
	G776										
	G777	0.580	0.566	0.594	0.580	0.014	100.4	100.6	100.3	100.4	0.1
Community Results		Consensus Mean			0.134		Consensus Mean			84.7	
		Consensus Standard Deviation			0.166		Consensus Standard Deviation			14.4	
		Maximum			0.580		Maximum			117.3	
		Minimum			0.007		Minimum			37.0	
		N			9		N			15	

Table 24. Data summary table for malvidin in dietary supplements.

		Malvidin									
		SRM 3283 Cranberry Extract (mg/g)					SRM 3291 Bilberry Extract (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	NIST										
	G701										
	G702										
	G703						1.19	1.19	1.21	1.20	0.01
	G704										
	G705						0.99	0.90	0.93	0.94	0.04
	G707										
	G708										
	G715	0.0300	0.0350		0.0325	0.0035	0.75	0.71		0.73	0.03
	G716										
	G717						0.34	0.34	0.32	0.33	0.01
	G718	0.0680	0.0640	0.0660	0.0660	0.0020	0.26	0.25	0.25	0.25	0.00
	G722										
	G724										
	G728						0.76	0.64	0.62	0.67	0.08
	G729										
	G738										
	G739										
	G740										
	G749										
	G752										
	G753										
	G757										
	G760										
	G762										
	G765										
	G766										
	G767										
	G771										
	G772										
	G773						2.06	1.60	2.51	2.06	0.46
	G774										
	G775						1.57	1.57	1.24	1.46	0.19
	G776										
	G777						4.72	4.68	4.81	4.74	0.07
Community Results		Consensus Mean			0.0493		Consensus Mean			1.11	
		Consensus Standard Deviation			0.0269		Consensus Standard Deviation			0.82	
		Maximum			0.0660		Maximum			4.74	
		Minimum			0.0325		Minimum			0.25	
		N			2		N			10	

Table 25. Data summary table for malvidin-3-arabinoside in dietary supplements.

		Malvidin-3-Arabinoside									
		SRM 3283 Cranberry Extract (mg/g)					SRM 3291 Bilberry Extract (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	NIST										
	G701										
	G702										
	G703						6.89	7.12	7.08	7.03	0.12
	G704										
	G705						9.25	9.14	9.19	9.19	0.06
	G707										
	G708	0.0959	0.0966	0.1070	0.0998	0.0062	11.20	11.00	11.10	11.10	0.10
	G715						8.87	9.00		8.94	0.09
	G716										
	G717						8.43	8.23	8.40	8.35	0.11
	G718						9.21	8.48	8.71	8.80	0.37
	G722										
	G724										
	G728						12.34	11.36	12.04	11.91	0.50
	G729										
	G738										
	G739										
	G740										
	G749										
	G752										
	G753										
	G757										
	G760										
	G762						10.50	10.20	9.47	10.06	0.53
	G765						11.52	11.11	11.79	11.47	0.34
	G766						5.03	4.84	4.91	4.93	0.10
	G767										
	G771										
	G772										
	G773						1.24	1.04	0.96	1.08	0.14
	G774										
	G775						7.87	7.85	7.82	7.85	0.03
	G776										
	G777	0.0030	0.0030	0.0030	0.0030	0.0000	1.50	1.49	1.52	1.50	0.01
Community Results		Consensus Mean			0.0514		Consensus Mean			8.19	
		Consensus Standard Deviation			0.0776		Consensus Standard Deviation			3.22	
		Maximum			0.0998		Maximum			11.91	
		Minimum			0.0030		Minimum			1.08	
		N			2		N			13	

Table 26. Data summary table for malvidin-3-galactoside in dietary supplements.

		Malvidin-3-Galactoside									
		SRM 3283 Cranberry Extract (mg/g)					SRM 3291 Bilberry Extract (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	NIST										
	G701	0.1400	0.1400	0.1600	0.1467	0.0115	26.1	23.1	21.0	23.4	2.5
	G702										
	G703	0.0125			0.0125		12.1	12.3	12.3	12.2	0.1
	G704										
	G705						16.3	16.1	16.2	16.2	0.1
	G707										
	G708										
	G715						12.5	12.6		12.5	0.1
	G716										
	G717						38.1	37.1	38.6	37.9	0.8
	G718	0.0520			0.0520		14.9	14.2	14.7	14.6	0.3
	G722										
	G724										
	G728						19.2	18.1	19.4	18.9	0.7
	G729										
	G738										
	G739										
	G740										
	G749										
	G752										
	G753										
	G757										
	G760										
	G762	0.0252	0.0270	0.0270	0.0264	0.0010	14.0	14.0	13.0	13.7	0.6
	G765						18.3	17.4	17.8	17.8	0.4
	G766						6.7	6.6	6.8	6.7	0.1
	G767										
	G771										
	G772										
	G773						27.9	27.9	25.3	27.0	1.5
	G774										
	G775						33.3	33.3	33.2	33.3	0.1
	G776										
	G777	0.0060	0.0050	0.0050	0.0053	0.0006	13.3	13.3	13.3	13.3	0.0
Community Results		Consensus Mean			0.0595		Consensus Mean			18.4	
		Consensus Standard Deviation			0.0865		Consensus Standard Deviation			8.8	
		Maximum			0.1467		Maximum			37.9	
		Minimum			0.0053		Minimum			6.7	
		N			3		N			13	

Table 27. Data summary table for malvidin-3-glucoside in dietary supplements.

		Malvidin-3-Glucoside									
		SRM 3283 Cranberry Extract (mg/g)					SRM 3291 Bilberry Extract (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	NIST										
	G701										
	G702										
	G703						30.2	30.6	30.6	30.5	0.2
	G704										
	G705										
	G707										
	G708	0.1720	0.1780	0.1800	0.1767	0.0042	37.3	37.0	37.2	37.2	0.2
	G715	0.0270	0.0320		0.0295	0.0035	34.7	34.8		34.8	0.0
	G716										
	G717	0.0128	0.0110	0.0117	0.0118	0.0009	14.5	14.3	14.2	14.3	0.2
	G718						36.5	35.4	36.6	36.2	0.7
	G722										
	G724										
	G728						43.8	42.5	43.3	43.2	0.6
	G729										
	G738										
	G739										
	G740										
	G749										
	G752										
	G753										
	G757										
	G760										
	G762						35.8	35.6	32.9	34.8	1.6
	G765						45.9	43.9	44.4	44.7	1.0
	G766	0.1323	0.1340	0.1370	0.1344	0.0024	21.6	21.2	21.2	21.3	0.2
	G767										
	G771										
	G772										
	G773						10.2	11.4	9.7	10.4	0.9
	G774										
	G775						13.4	13.5	13.5	13.4	0.0
	G776										
	G777						35.7	35.8	35.8	35.8	0.1
Community Results		Consensus Mean			0.0881		Consensus Mean			29.7	
		Consensus Standard Deviation			0.0908		Consensus Standard Deviation			13.4	
		Maximum			0.1767		Maximum			44.7	
		Minimum			0.0118		Minimum			10.4	
		N			4		N			12	

Table 28. Data summary table for malvidin equivalents in dietary supplements.

		Malvidin Equivalents									
		SRM 3283 Cranberry Extract (mg/g)					SRM 3291 Bilberry Extract (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	NIST										
	G701	0.0940	0.0940	0.1074	0.0985	0.0078	17.5	15.5	14.1	15.7	1.7
	G702										
	G703	0.0084			0.0084		34.5	35.1	35.1	34.9	0.3
	G704										
	G705						18.5	18.2	18.4	18.4	0.1
	G707										
	G708	0.1840	0.1886	0.1974	0.1900	0.0068	33.1	32.7	32.9	32.9	0.2
	G715	0.0481	0.0565		0.0523	0.0059	38.8	38.9		38.9	0.1
	G716										
	G717	0.0086	0.0074	0.0079	0.0079	0.0006	41.7	40.7	41.8	41.4	0.6
	G718	0.1029	0.0640	0.0660	0.0776	0.0219	41.4	39.7	40.9	40.7	0.9
	G722										
	G724										
	G728						51.8	49.4	51.3	50.9	1.3
	G729										
	G738						6.1			6.1	
	G739										
	G740										
	G749										
	G752										
	G753										
	G757										
	G760										
	G762	0.0169	0.0181	0.0181	0.0177	0.0007	40.9	40.6	37.6	39.7	1.8
	G765						51.3	49.1	50.2	50.2	1.1
	G766	0.0888	0.0900	0.0920	0.0903	0.0016	22.6	22.1	22.3	22.3	0.2
	G767										
	G771										
	G772						38.3	41.2	42.8	40.8	2.3
	G773						28.5	28.7	26.7	28.0	1.1
	G774										
	G775						38.6	38.5	38.1	38.4	0.2
	G776										
	G777	0.0062	0.0055	0.0055	0.0057	0.0004	38.7	38.8	38.9	38.8	0.1
Community Results		Consensus Mean			0.0624		Consensus Mean			34.2	
		Consensus Standard Deviation			0.0576		Consensus Standard Deviation			12.7	
		Maximum			0.1900		Maximum			50.9	
		Minimum			0.0057		Minimum			6.1	
		N			8		N			15	

Table 29. Data summary table for peonidin in dietary supplements.

		Peonidin									
		SRM 3283 Cranberry Extract (mg/g)					SRM 3291 Bilberry Extract (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	NIST										
	G701										
	G702										
	G703	0.0507	0.0526	0.0549	0.0527	0.0021	0.468	0.479	0.491	0.479	0.012
	G704										
	G705	0.0372	0.0411	0.0355	0.0379	0.0029	0.427	0.491	0.418	0.445	0.040
	G707										
	G708										
	G715	0.0510	0.0460		0.0485	0.0035	0.234	0.234		0.234	0.000
	G716										
	G717	0.0507	0.0507	0.0498	0.0504	0.0005	0.200	0.206	0.223	0.210	0.012
	G718		0.0510		0.0510		0.242	0.229	0.229	0.233	0.008
	G722										
	G724										
	G728						0.299	0.316	0.323	0.313	0.012
	G729										
	G738										
	G739										
	G740										
	G749										
	G752										
	G753										
	G757										
	G760										
	G762										
	G765										
	G766										
	G767										
	G771										
	G772										
	G773						1.270	1.490	1.220	1.327	0.144
	G774										
	G775						1.307	1.082	1.452	1.280	0.186
	G776										
	G777	0.1260	0.1220	0.1320	0.1267	0.0050	1.491	1.476	1.534	1.500	0.030
Community Results		Consensus Mean			0.0505		Consensus Mean			0.669	
		Consensus Standard Deviation			0.0045		Consensus Standard Deviation			0.608	
		Maximum			0.1267		Maximum			1.500	
		Minimum			0.0379		Minimum			0.210	
		N			6		N			10	

Table 30. Data summary table for peonidin-3-arabinoside in dietary supplements.

		Peonidin-3-Arabinoside									
		SRM 3283 Cranberry Extract (mg/g)					SRM 3291 Bilberry Extract (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	NIST										
	G701	0.168	0.163	0.174	0.168	0.006	23.97	23.36	20.93	22.75	1.61
	G702										
	G703	0.103	0.101	0.107	0.104	0.003	1.63	1.67	1.65	1.65	0.02
	G704										
	G705	0.129	0.138	0.123	0.130	0.008	2.54	2.46	2.50	2.50	0.04
	G707										
	G708										
	G715	0.134	0.148		0.141	0.010	2.12	2.15		2.14	0.02
	G716										
	G717	0.165	0.165	0.171	0.167	0.003	1.77	1.69	1.58	1.68	0.10
	G718	0.192	0.195	0.192	0.193	0.002	2.12	2.02	2.09	2.08	0.05
	G722										
	G724										
	G728	0.245	0.183	0.225	0.218	0.032	3.89	3.58	3.89	3.78	0.18
	G729										
	G738										
	G739										
	G740										
	G749										
	G752										
	G753										
	G757										
	G760										
	G762	0.140	0.144	0.140	0.141	0.002	2.61	2.58	2.41	2.53	0.11
	G765	0.176	0.179	0.182	0.179	0.003	3.37	3.40	3.51	3.43	0.07
	G766										
	G767										
	G771										
	G772										
	G773			0.150	0.150		1.61	1.54	1.35	1.50	0.13
	G774										
	G775	0.045	0.045	0.050	0.046	0.003	2.01	1.99	1.93	1.98	0.04
	G776										
	G777	0.108	0.106	0.112	0.109	0.003	2.21	2.23	2.24	2.23	0.01
Community Results		Consensus Mean			0.148		Consensus Mean			2.44	
		Consensus Standard Deviation			0.048		Consensus Standard Deviation			0.92	
		Maximum			0.218		Maximum			22.75	
		Minimum			0.046		Minimum			1.50	
		N			11		N			12	

Table 31. Data summary table for peonidin-3-galactoside in dietary supplements.

		Peonidin-3-Galactoside									
		SRM 3283 Cranberry Extract (mg/g)					SRM 3291 Bilberry Extract (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	NIST										
	G701	0.177	0.173	0.177	0.176	0.002	1.67	0.76	0.82	1.08	0.51
	G702										
	G703	0.156	0.157	0.160	0.158	0.002	3.73	3.81	3.80	3.78	0.04
	G704										
	G705	0.178	0.199	0.176	0.184	0.013	5.72	5.70	5.68	5.70	0.02
	G707										
	G708	0.211	0.211	0.218	0.213	0.004	4.56	4.49	4.54	4.53	0.04
	G715	0.177	0.204		0.191	0.019	8.55	8.19		8.37	0.25
	G716										
	G717	0.221	0.223	0.224	0.223	0.002	3.76	3.78	3.97	3.84	0.12
	G718	0.241	0.260	0.251	0.251	0.010	4.65	4.49	4.64	4.59	0.09
	G722										
	G724										
	G728	0.456	0.211	0.404	0.357	0.129	9.67	9.08	10.32	9.69	0.62
	G729										
	G738										
	G739										
	G740										
	G749										
	G752										
	G753										
	G757										
	G760										
	G762	0.172	0.176	0.173	0.174	0.002	5.15	5.17	4.68	5.00	0.28
	G765	0.235	0.240	0.243	0.239	0.004	7.47	7.06	7.23	7.25	0.21
	G766	0.197	0.218	0.199	0.205	0.012	4.40	3.98	4.00	4.13	0.24
	G767										
	G771										
	G772										
	G773	0.077	0.078	0.100	0.085	0.013	2.28	2.18	1.84	2.10	0.23
	G774										
	G775						4.25	4.25	4.20	4.23	0.03
	G776										
	G777						13.02	13.02	13.09	13.04	0.04
Community Results		Consensus Mean			0.201		Consensus Mean			5.29	
		Consensus Standard Deviation			0.043		Consensus Standard Deviation			2.96	
		Maximum			0.357		Maximum			13.04	
		Minimum			0.085		Minimum			1.08	
		N			12		N			14	

Table 32. Data summary table for peonidin-3-glucoside in dietary supplements.

		Peonidin-3-Glucoside									
		SRM 3283 Cranberry Extract (mg/g)					SRM 3291 Bilberry Extract (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	NIST										
	G701										
	G702										
	G703	0.0286	0.0267	0.0235	0.0263	0.0026	14.2	14.4	14.4	14.3	0.1
	G704										
	G705	0.0245	0.0252	0.0224	0.0240	0.0015	18.9	18.7	18.7	18.8	0.1
	G707										
	G708	0.0367	0.0385	0.0710	0.0487	0.0193	31.7	31.6	31.6	31.6	0.1
	G715						16.2	16.3		16.2	0.1
	G716										
	G717	0.0298	0.0313	0.0296	0.0302	0.0009	16.9	16.8	16.6	16.8	0.1
	G718	0.1290	0.1350	0.1260	0.1300	0.0046	17.3	16.6	17.1	17.0	0.3
	G722										
	G724										
	G728						23.9	23.6	24.1	23.9	0.3
	G729										
	G738										
	G739										
	G740										
	G749										
	G752										
	G753										
	G757										
	G760										
	G762	0.0084	0.0089	0.0080	0.0084	0.0005	18.1	18.1	16.6	17.6	0.9
	G765	0.0320	0.0340	0.0450	0.0370	0.0070	28.0	26.6	27.7	27.4	0.7
	G766						16.8	16.3	16.1	16.4	0.4
	G767										
	G771										
	G772										
	G773						15.5	14.6	13.6	14.6	1.0
	G774										
	G775	0.1090	0.1194	0.1148	0.1144	0.0052	15.8	15.8	15.7	15.8	0.0
	G776										
	G777	0.0180	0.0160	0.0170	0.0170	0.0010	17.0	17.1	13.0	15.7	2.3
Community Results		Consensus Mean			0.0467		Consensus Mean			17.5	
		Consensus Standard Deviation			0.0454		Consensus Standard Deviation			3.0	
		Maximum			0.1300		Maximum			31.6	
		Minimum			0.0084		Minimum			14.3	
		N			9		N			13	

Table 33. Data summary table for peonidin equivalents in dietary supplements.

		Peonidin Equivalents									
		SRM 3283 Cranberry Extract (mg/g)					SRM 3291 Bilberry Extract (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	NIST										
	G701	0.232	0.226	0.236	0.231	0.005	17.7	16.7	15.1	16.5	1.3
	G702										
	G703	0.242	0.242	0.249	0.244	0.004	13.3	13.5	13.5	13.4	0.1
	G704										
	G705	0.259	0.283	0.250	0.264	0.017	18.2	18.1	18.0	18.1	0.1
	G707										
	G708	0.161	0.162	0.188	0.170	0.015	23.6	23.5	23.5	23.5	0.1
	G715	0.259	0.281		0.270	0.016	17.8	17.6		17.7	0.1
	G716										
	G717	0.328	0.331	0.333	0.331	0.003	14.9	14.7	14.7	14.8	0.1
	G718	0.374	0.443	0.379	0.399	0.039	16.0	15.4	15.8	15.7	0.3
	G722										
	G724										
	G728	0.467	0.264	0.419	0.383	0.106	24.8	24.0	25.4	24.8	0.7
	G729										
	G738	0.092			0.092		2.5			2.5	
	G739										
	G740										
	G749										
	G752										
	G753										
	G757										
	G760										
	G762	0.215	0.220	0.215	0.217	0.003	16.9	16.9	15.5	16.5	0.8
	G765	0.296	0.303	0.314	0.304	0.009	25.4	24.2	25.1	24.9	0.6
	G766	0.128	0.142	0.129	0.133	0.008	13.8	13.2	13.0	13.3	0.4
	G767										
	G771										
	G772	0.642	0.641	0.600	0.628	0.024	15.5	16.6	17.3	16.5	0.9
	G773	0.050	0.051	0.169	0.090	0.069	13.9	13.5	12.2	13.2	0.9
	G774										
	G775	0.102	0.109	0.109	0.107	0.004	15.7	15.5	15.7	15.6	0.1
	G776										
	G777	0.213	0.206	0.221	0.213	0.007	22.6	22.6	20.1	21.7	1.4
Community Results		Consensus Mean			0.252		Consensus Mean			17.6	
		Consensus Standard Deviation			0.118		Consensus Standard Deviation			4.4	
		Maximum			0.628		Maximum			24.9	
		Minimum			0.090		Minimum			2.5	
		N			15		N			15	

Table 34. Data summary table for petunidin in dietary supplements.

		Petunidin									
		SRM 3283 Cranberry Extract (mg/g)					SRM 3291 Bilberry Extract (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	NIST										
	G701										
	G702										
	G703						1.230	1.210	1.230	1.223	0.012
	G704										
	G705						0.750	0.742	0.765	0.752	0.012
	G707										
	G708										
	G715	0.086	0.107		0.097	0.015	0.766	0.724		0.745	0.030
	G716										
	G717						0.576	0.556	0.586	0.573	0.015
	G718						0.350	0.324	0.343	0.339	0.013
	G722										
	G724										
	G728						0.724	0.562	0.531	0.606	0.104
	G729										
	G738										
	G739										
	G740										
	G749										
	G752										
	G753										
	G757										
	G760										
	G762										
	G765						0.460	0.540	0.490	0.497	0.040
	G766										
	G767										
	G771										
	G772										
	G773	0.150	0.180	0.180	0.170	0.017	0.540	0.670	0.610	0.607	0.065
	G774										
	G775						1.487	1.478	1.471	1.479	0.008
	G776										
	G777						0.961	0.959	0.941	0.954	0.011
Community Results		Consensus Mean			0.133		Consensus Mean			0.757	
		Consensus Standard Deviation			0.059		Consensus Standard Deviation			0.348	
		Maximum			0.170		Maximum			1.479	
		Minimum			0.097		Minimum			0.339	
		N			2		N			11	

Table 35. Data summary table for petunidin-3-arabinoside in dietary supplements.

		Petunidin-3-Arabinoside									
		SRM 3283 Cranberry Extract (mg/g)					SRM 3291 Bilberry Extract (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	NIST										
	G701										
	G702										
	G703						8.3	8.3	8.4	8.3	0.1
	G704										
	G705						11.1	11.0	11.1	11.1	0.1
	G707										
	G708						10.9	10.8	10.8	10.8	0.1
	G715						10.3	10.3		10.3	0.0
	G716										
	G717						9.9	9.7	9.7	9.8	0.1
	G718						10.2	9.9	10.2	10.1	0.2
	G722										
	G724										
	G728						11.7	12.0	12.2	11.9	0.3
	G729										
	G738										
	G739										
	G740										
	G749										
	G752										
	G753										
	G757										
	G760										
	G762						10.9	10.9	10.0	10.6	0.5
	G765						14.3	13.7	13.9	14.0	0.3
	G766						11.1	10.6	10.6	10.7	0.3
	G767										
	G771										
	G772										
	G773						9.1	9.2	7.9	8.7	0.7
	G774										
	G775						9.3	9.4	9.3	9.3	0.0
	G776										
	G777										
Community Results		Consensus Mean					Consensus Mean			10.3	
		Consensus Standard Deviation					Consensus Standard Deviation			1.3	
		Maximum					Maximum			14.0	
		Minimum					Minimum			8.3	
		N			0		N			12	

Table 36. Data summary table for petunidin-3-galactoside in dietary supplements.

		Petunidin-3-Galactoside									
		SRM 3283 Cranberry Extract (mg/g)					SRM 3291 Bilberry Extract (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	NIST										
	G701										
	G702										
	G703						13.1	13.3	13.3	13.2	0.1
	G704										
	G705										
	G707										
	G708						16.7	16.5	16.7	16.6	0.1
	G715	0.1700	0.1640		0.1670	0.0042	20.5	21.9		21.2	1.1
	G716										
	G717						21.2	21.3	22.7	21.7	0.9
	G718						16.2	15.6	16.2	16.0	0.3
	G722										
	G724										
	G728						18.6	12.8	20.6	17.3	4.1
	G729										
	G738										
	G739										
	G740										
	G749										
	G752										
	G753										
	G757										
	G760										
	G762	0.0297	0.0330	0.0319	0.0315	0.0017	16.8	16.8	15.5	16.4	0.8
	G765						22.1	21.1	21.5	21.6	0.5
	G766						13.2	12.4	12.5	12.7	0.5
	G767										
	G771										
	G772										
	G773						13.1	13.5	12.0	12.9	0.8
	G774										
	G775						15.2	15.2	15.2	15.2	0.0
	G776										
	G777										
Community Results		Consensus Mean			0.0993		Consensus Mean			16.8	
		Consensus Standard Deviation			0.1086		Consensus Standard Deviation			3.8	
		Maximum			0.1670		Maximum			21.7	
		Minimum			0.0315		Minimum			12.7	
		N			2		N			11	

Table 37. Data summary table for petunidin-3-glucoside in dietary supplements.

		Petunidin-3-Glucoside									
		SRM 3283 Cranberry Extract (mg/g)					SRM 3291 Bilberry Extract (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	NIST										
	G701						57.8	49.6	53.0	53.5	4.1
	G702										
	G703						27.3	27.7	27.6	27.5	0.2
	G704										
	G705						33.5	34.5	34.6	34.2	0.6
	G707										
	G708						34.0	34.0	34.2	34.1	0.1
	G715						29.4	30.6		30.0	0.8
	G716										
	G717						32.6	33.7	33.6	33.3	0.6
	G718	0.0440		0.0440	0.0440	0.0000	33.2	32.0	33.2	32.8	0.7
	G722										
	G724										
	G728						39.9	37.5	39.6	39.0	1.3
	G729										
	G738										
	G739										
	G740										
	G749										
	G752										
	G753										
	G757										
	G760										
	G762	0.0056	0.0057	0.0060	0.0058	0.0002	34.0	33.9	31.0	33.0	1.7
	G765						44.3	42.3	43.0	43.2	1.0
	G766						26.2	25.2	25.3	25.6	0.6
	G767										
	G771										
	G772										
	G773		0.0180		0.0180		23.8	23.0	21.5	22.8	1.2
	G774										
	G775	0.1866	0.1918	0.1945	0.1910	0.0040	30.1	30.2	30.2	30.2	0.1
	G776										
	G777	0.0070	0.0060	0.0060	0.0063	0.0006	31.9	32.0	32.0	31.9	0.0
Community Results		Consensus Mean			0.0618		Consensus Mean			32.7	
		Consensus Standard Deviation			0.0998		Consensus Standard Deviation			6.0	
		Maximum			0.1910		Maximum			53.5	
		Minimum			0.0058		Minimum			22.8	
		N			4		N			14	

Table 38. Data summary table for petunidin equivalents in dietary supplements.

		Petunidin Equivalents									
		SRM 3283 Cranberry Extract (mg/g)					SRM 3291 Bilberry Extract (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	NIST										
	G701						38.3	32.8	35.1	35.4	2.7
	G702										
	G703						33.8	34.2	34.2	34.1	0.2
	G704										
	G705						30.8	31.3	31.5	31.2	0.4
	G707										
	G708						41.2	41.0	41.3	41.2	0.1
	G715	0.1985	0.2155		0.2070	0.0120	41.0	42.8		41.9	1.2
	G716										
	G717						43.1	43.8	44.7	43.9	0.8
	G718	0.0291		0.0291	0.0291	0.0000	40.3	38.9	40.2	39.8	0.8
	G722										
	G724										
	G728						47.7	42.3	49.0	46.3	3.6
	G729										
	G738						12.1			12.1	
	G739										
	G740										
	G749										
	G752										
	G753										
	G757										
	G760										
	G762	0.0233	0.0256	0.0251	0.0247	0.0012	41.3	41.2	37.8	40.1	2.0
	G765						54.5	52.2	53.0	53.2	1.2
	G766						34.0	32.4	32.4	32.9	0.9
	G767										
	G771										
	G772						41.9	44.4	45.3	43.9	1.8
	G773	0.1500	0.1919	0.1800	0.1740	0.0216	31.4	31.3	28.4	30.4	1.7
	G774										
	G775	0.1235	0.1269	0.1287	0.1264	0.0027	38.0	38.2	38.1	38.1	0.1
	G776										
	G777	0.0046	0.0040	0.0040	0.0042	0.0004	22.1	22.1	22.1	22.1	0.0
Community Results		Consensus Mean			0.0942		Consensus Mean			38.4	
		Consensus Standard Deviation			0.0979		Consensus Standard Deviation			7.1	
		Maximum			0.2070		Maximum			53.2	
		Minimum			0.0042		Minimum			12.1	
		N			6		N			15	

Table 39. Data summary table for total anthocyanins in dietary supplements.

		Total Anthocyanins									
		SRM 3283 Cranberry Extract (mg/g)					SRM 3291 Bilberry Extract (mg/g)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	NIST										
	G701	0.734	0.729	0.768	0.744	0.021	380	336	334	350	26
	G702										
	G703	0.676	0.651	0.644	0.657	0.017	310	314	314	313	2
	G704										
	G705	0.696	0.768	0.675	0.713	0.049	390	387	387	388	2
	G707										
	G708	0.891	0.895	0.958	0.915	0.038	376	374	376	375	1
	G715	0.910	0.982		0.946	0.051	366	368		367	2
	G716	1.700	1.800	1.900	1.800	0.100	290	280	270	280	10
	G717	0.798	0.796	0.808	0.801	0.006	371	367	367	368	2
	G718	1.337	1.321	1.280	1.313	0.029	424	409	424	419	9
	G722										
	G724	0.730	0.770	0.770	0.757	0.023	282	281	282	282	1
	G728	1.270	0.841	1.187	1.099	0.228	457	443	457	453	8
	G729										
	G738	0.476			0.476		97			97	
	G739										
	G740										
	G749										
	G752										
	G753										
	G757	10.100	11.100	10.200	10.467	0.551	433	444	419	432	13
	G760										
	G762	0.685	0.710	0.699	0.698	0.013	388	387	357	377	18
	G765	0.847	0.867	0.917	0.877	0.036	543	519	528	530	12
	G766	0.745	0.745	0.720	0.737	0.014	303	296	297	298	4
	G767										
	G771										
	G772	2.200	2.196	2.102	2.166	0.055	243	257	260	253	9
	G773	0.507	0.676	0.720	0.634	0.112	302	303	272	292	17
	G774										
	G775	0.696	0.717	0.702	0.705	0.010	340	341	341	340	1
	G776										
	G777	1.267	1.233	1.292	1.264	0.030	428	428	424	427	2
Community Results		Consensus Mean			0.934		Consensus Mean			354	
		Consensus Standard Deviation			0.358		Consensus Standard Deviation			78	
		Maximum			10.467		Maximum			530	
		Minimum			0.476		Minimum			97	
		N			18		N			18	

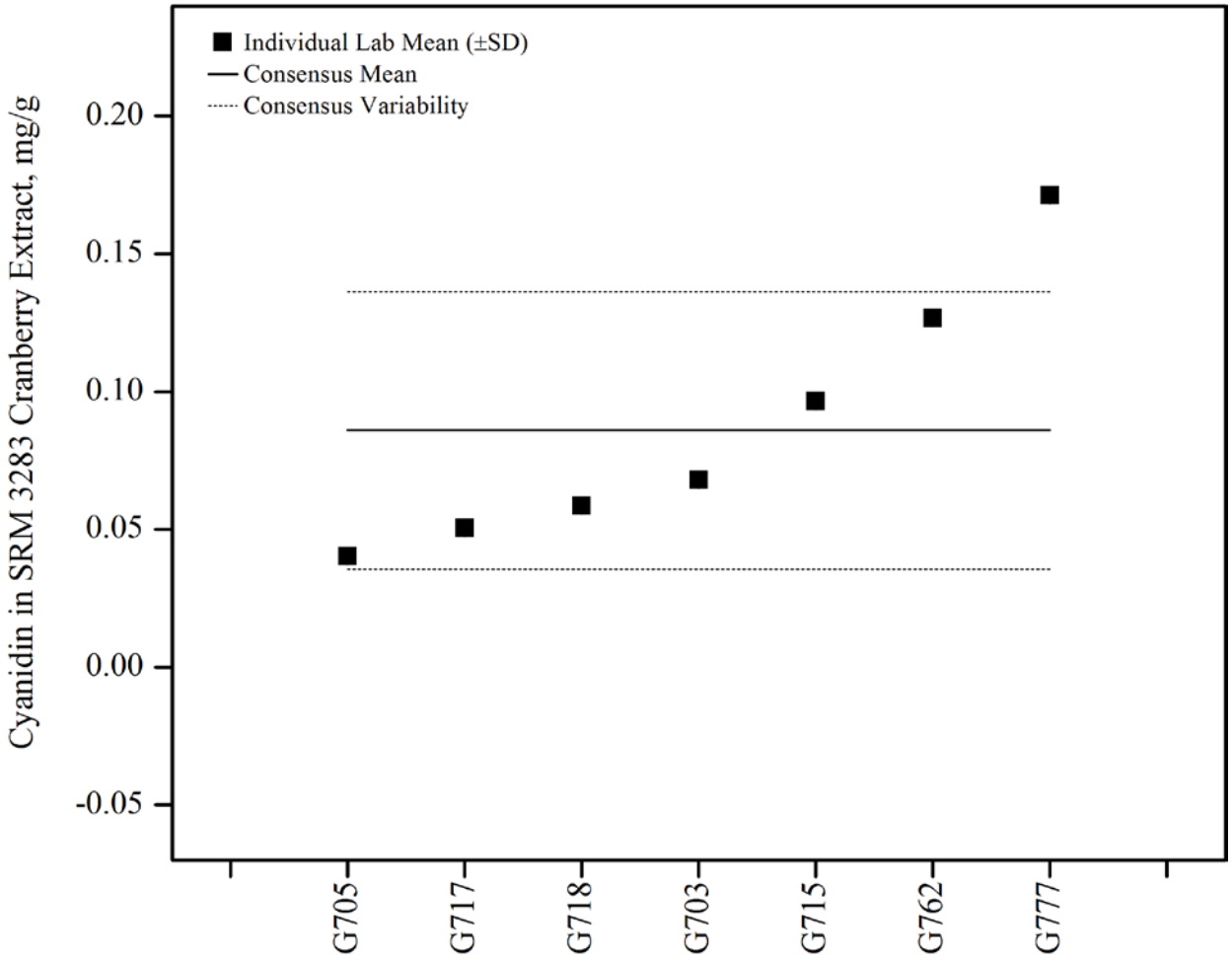

Figure 28. Cyanidin in SRM 3283 Cranberry Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

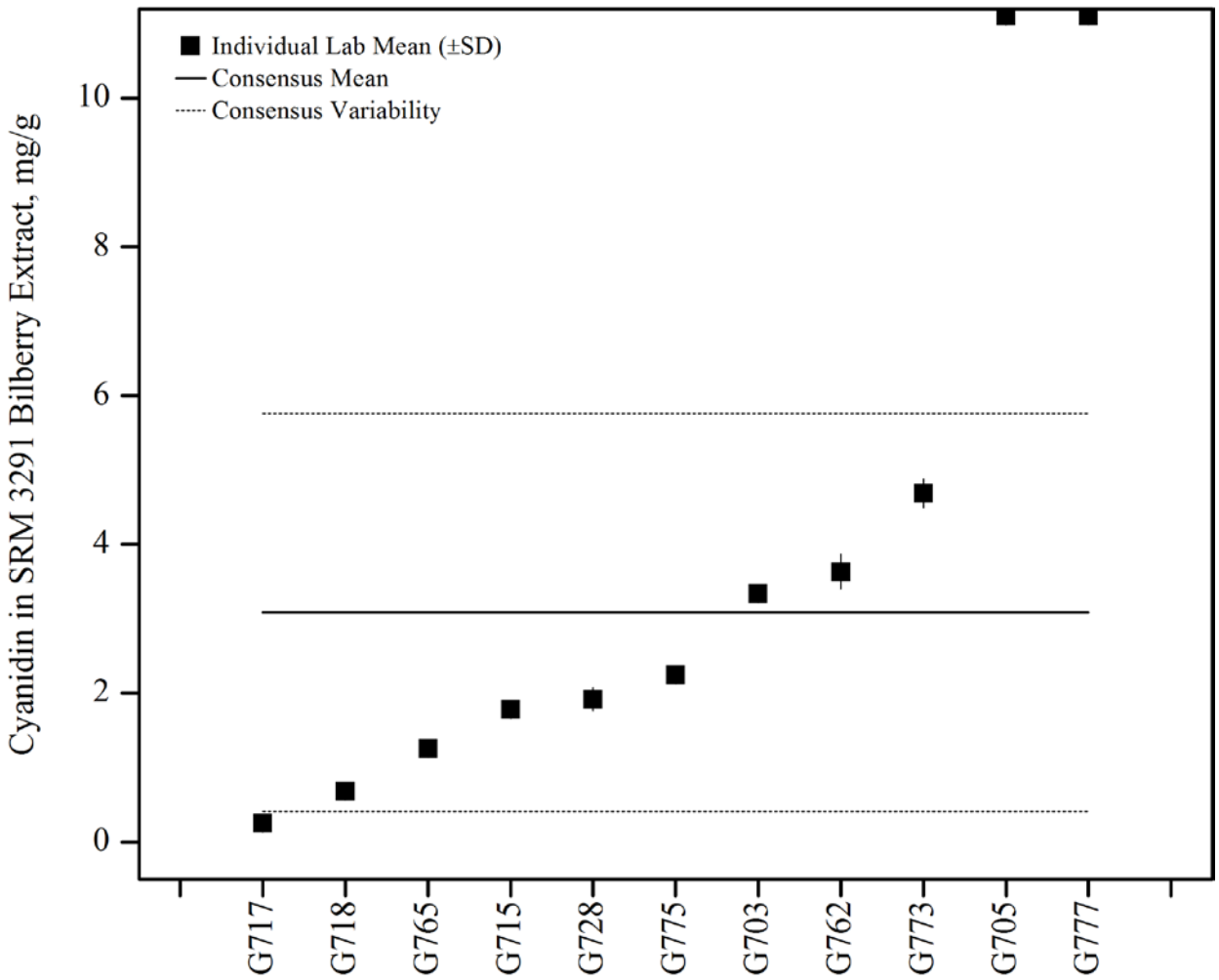

Figure 29. Cyanidin in SRM 3291 Bilberry Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

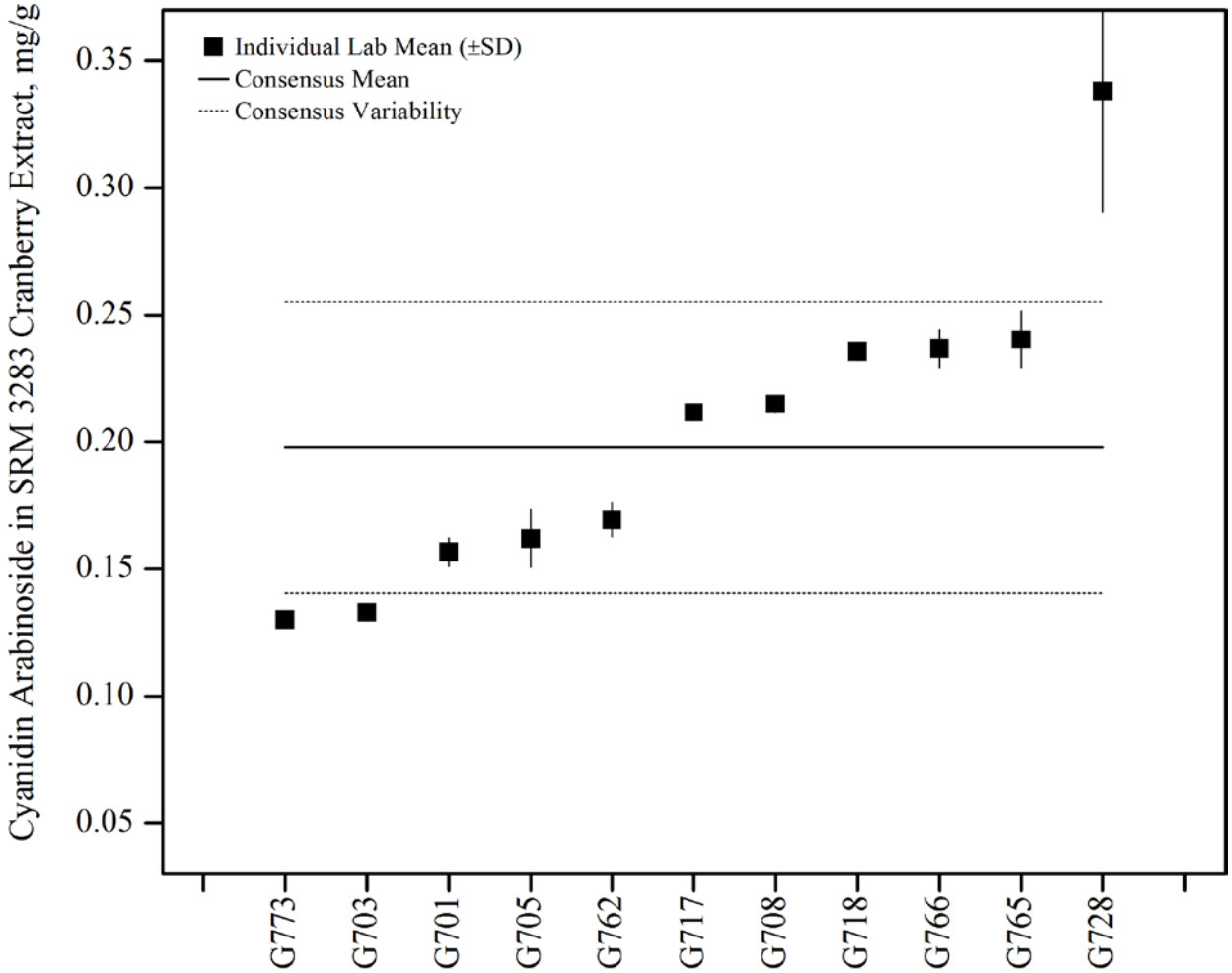

Figure 30. Cyanidin-3-arabinoside in SRM 3283 Cranberry Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

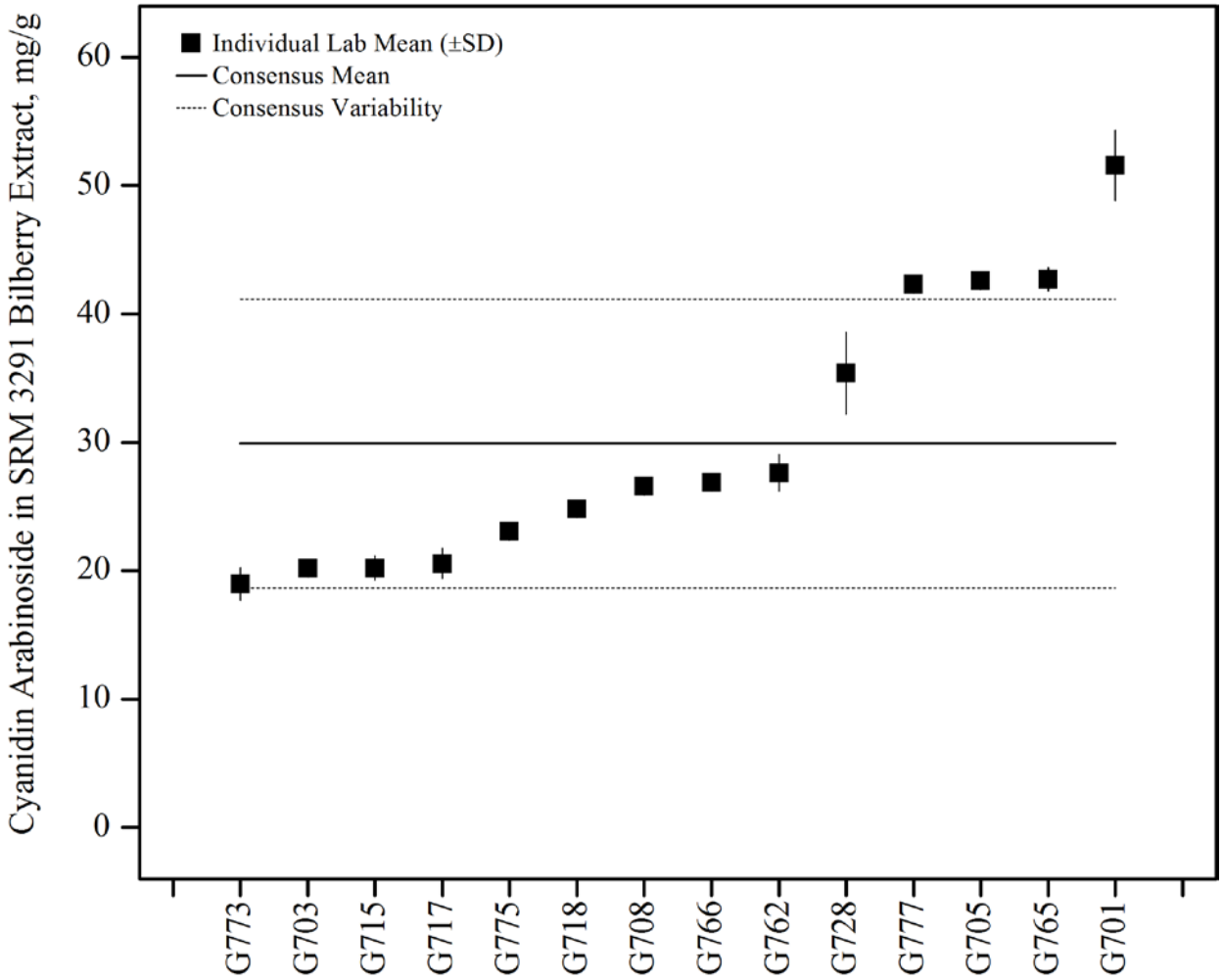

Figure 31. Cyanidin-3-arabinoside in SRM 3291 Bilberry Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

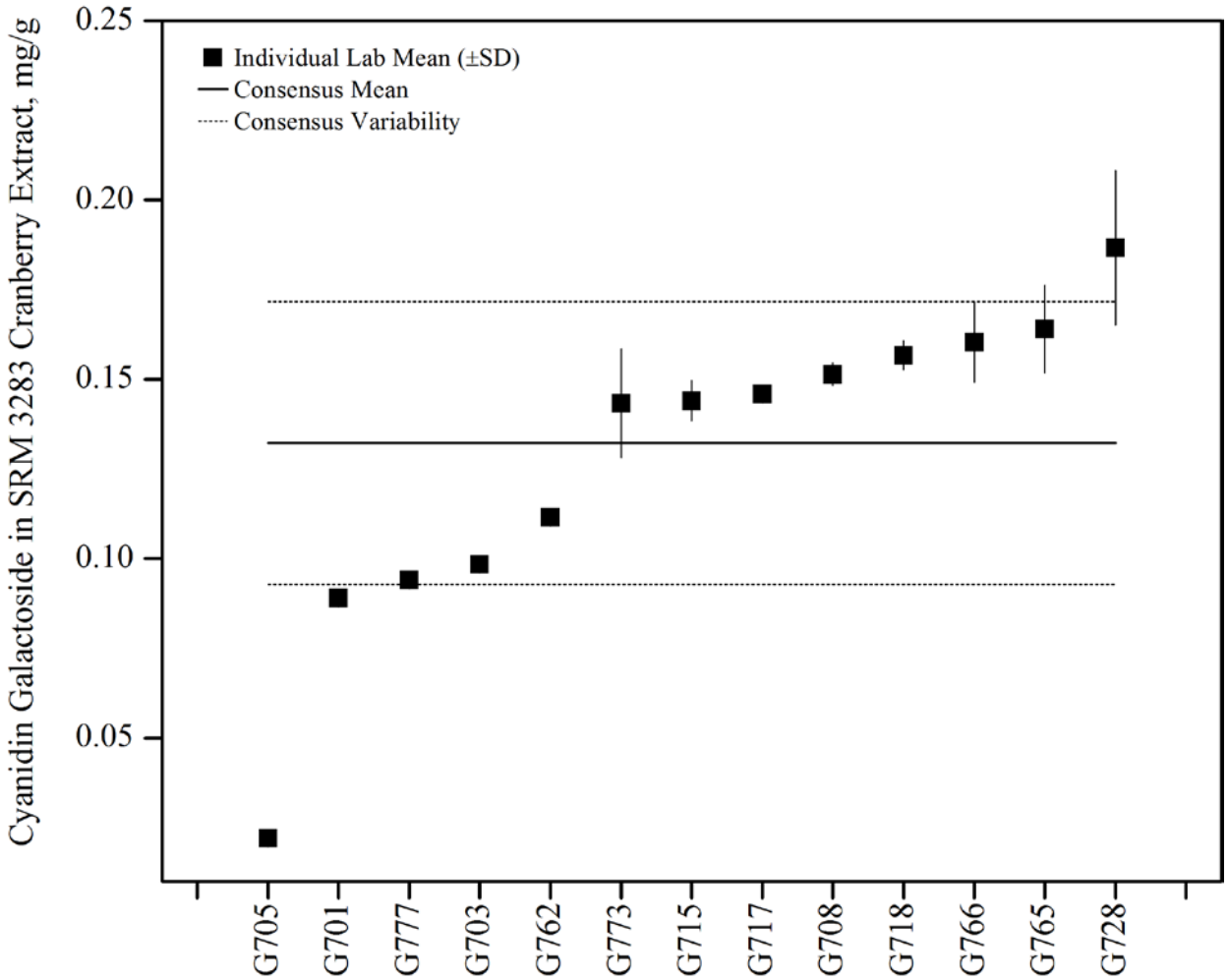

Figure 32. Cyanidin-3-galactoside in SRM 3283 Cranberry Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

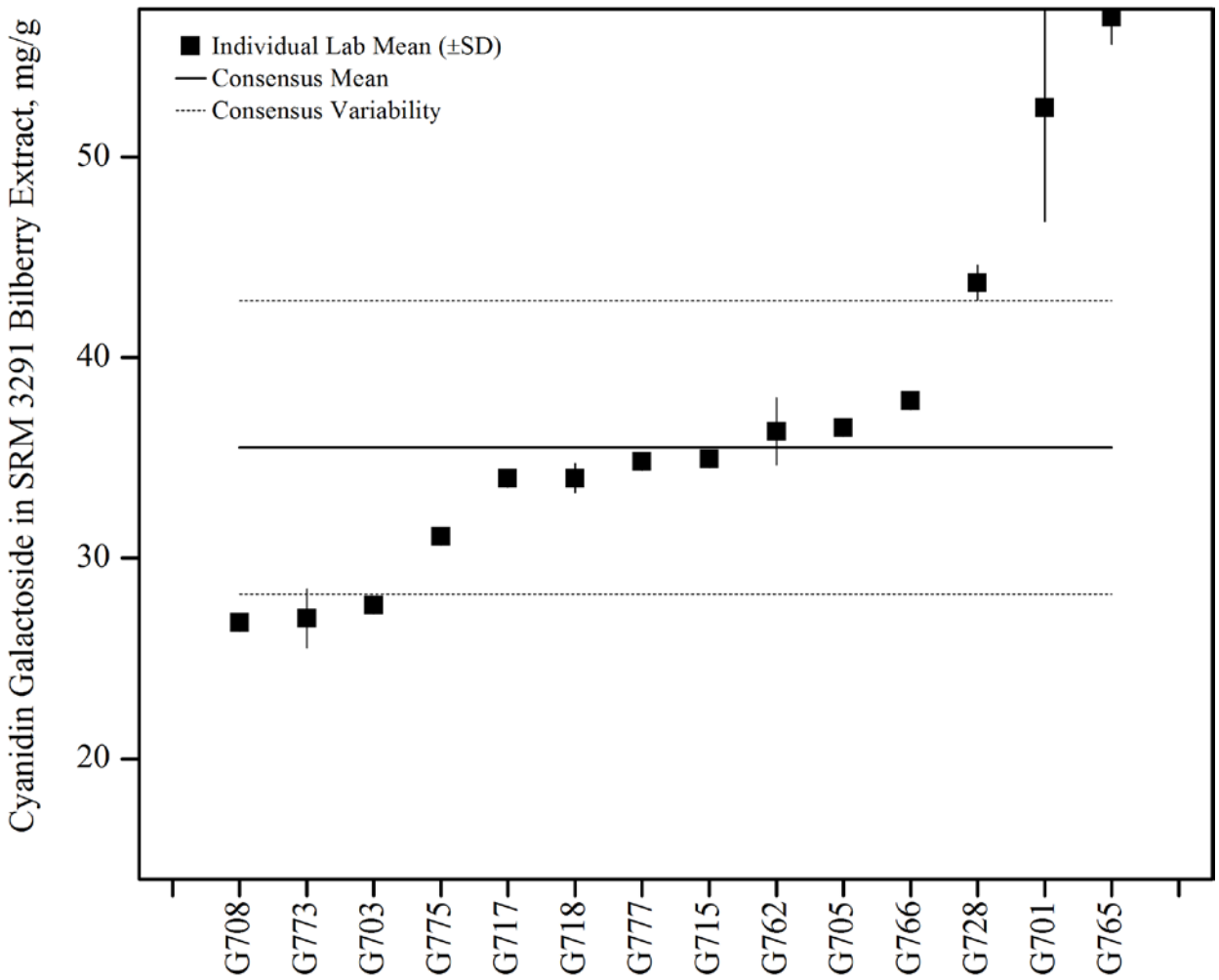

Figure 33. Cyanidin-3-galactoside in SRM 3291 Bilberry Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

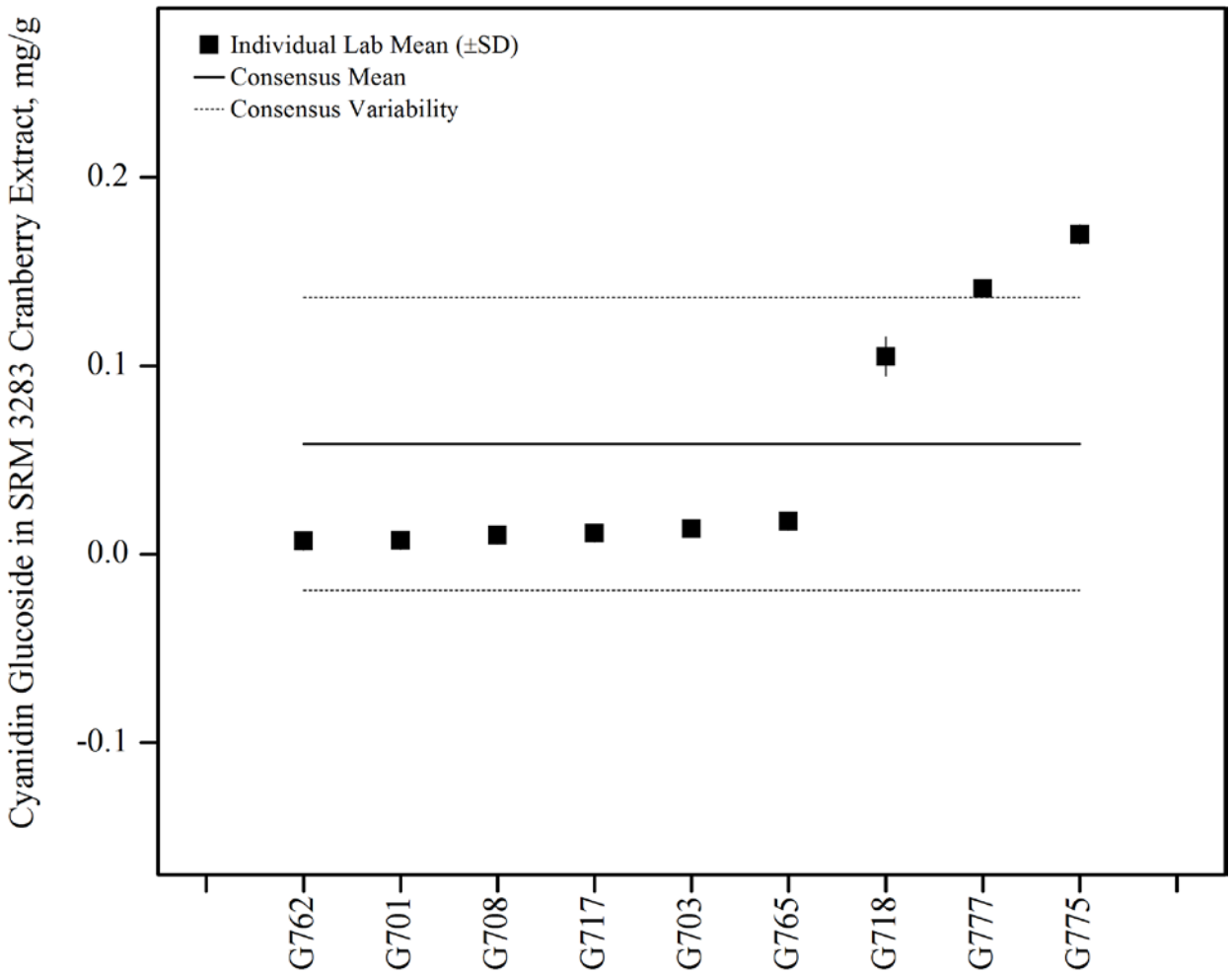

Figure 34. Cyanidin-3-glucoside in SRM 3283 Cranberry Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

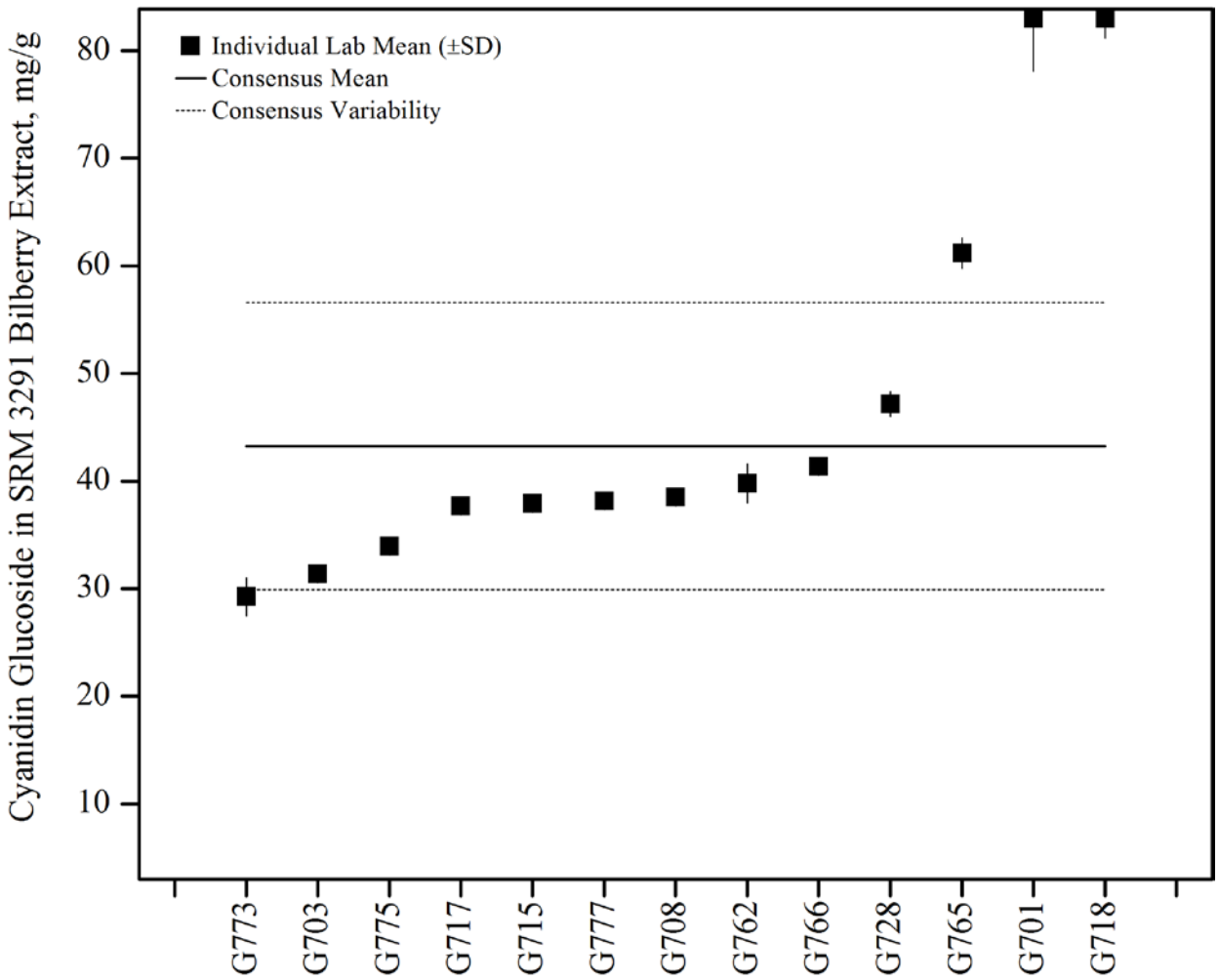

Figure 35. Cyanidin-3-glucoside in SRM 3291 Bilberry Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

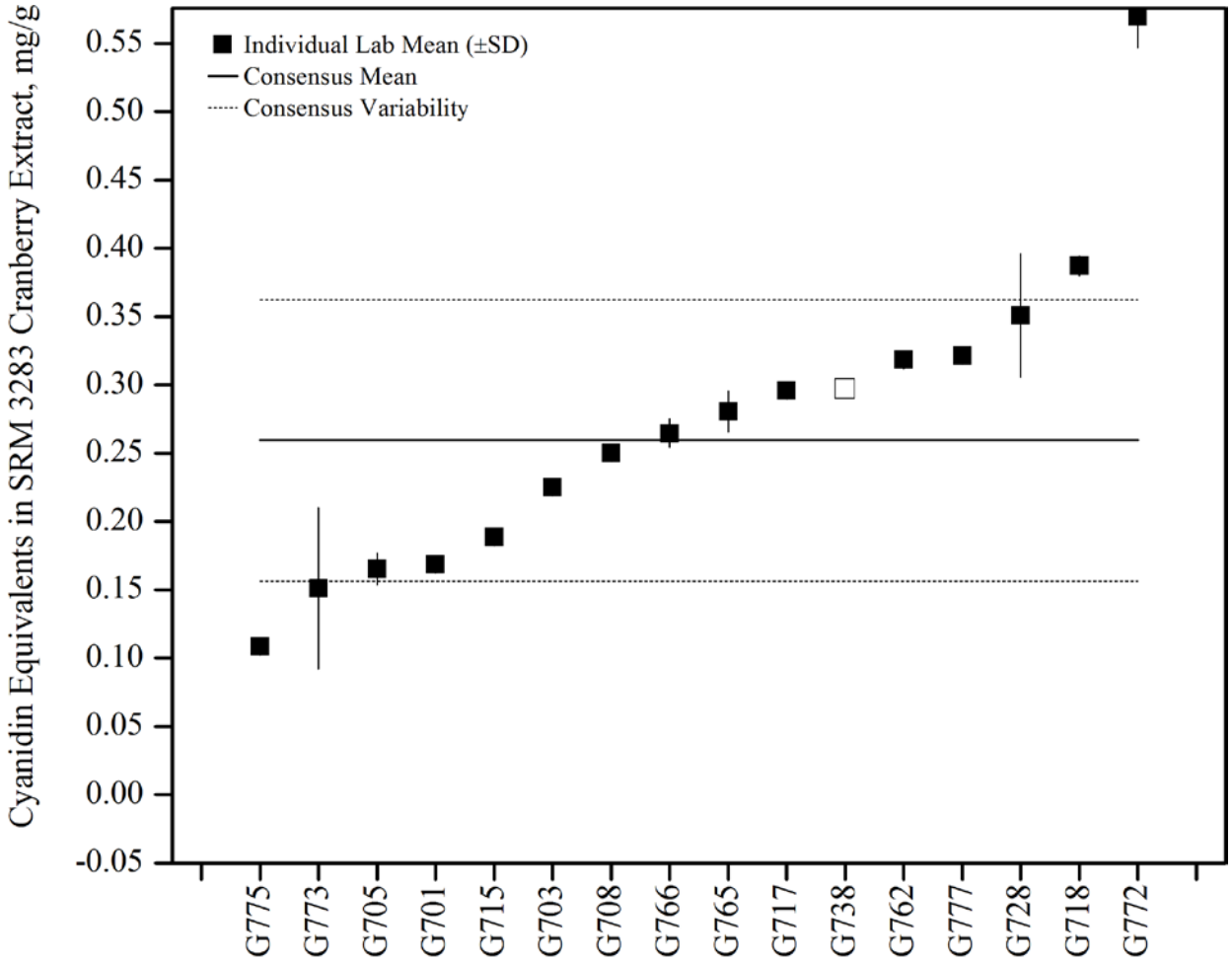

Figure 36. Cyanidin equivalents in SRM 3283 Cranberry Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

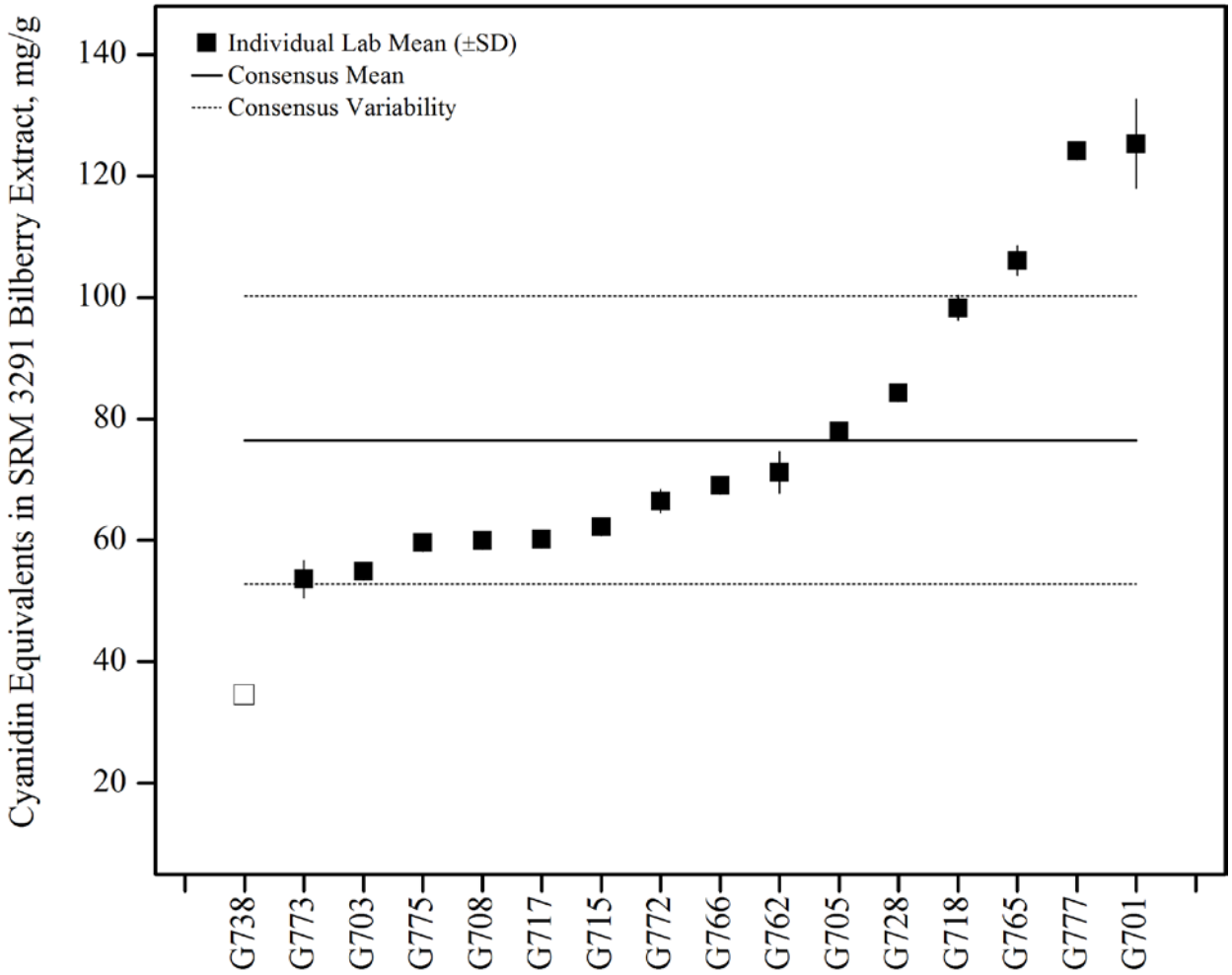

Figure 37. Cyanidin equivalents in SRM 3291 Bilberry Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

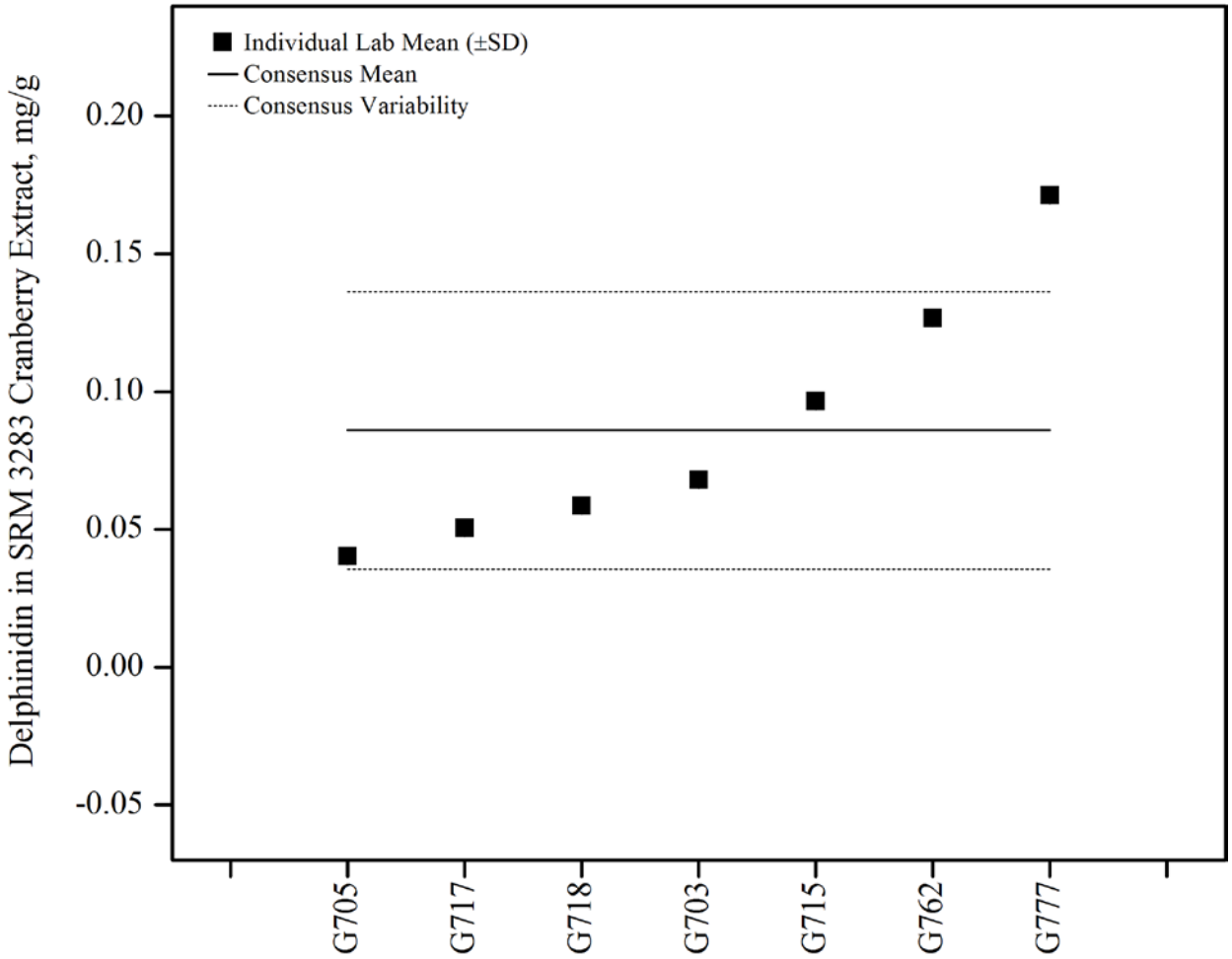

Figure 38. Delphinidin in SRM 3283 Cranberry Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

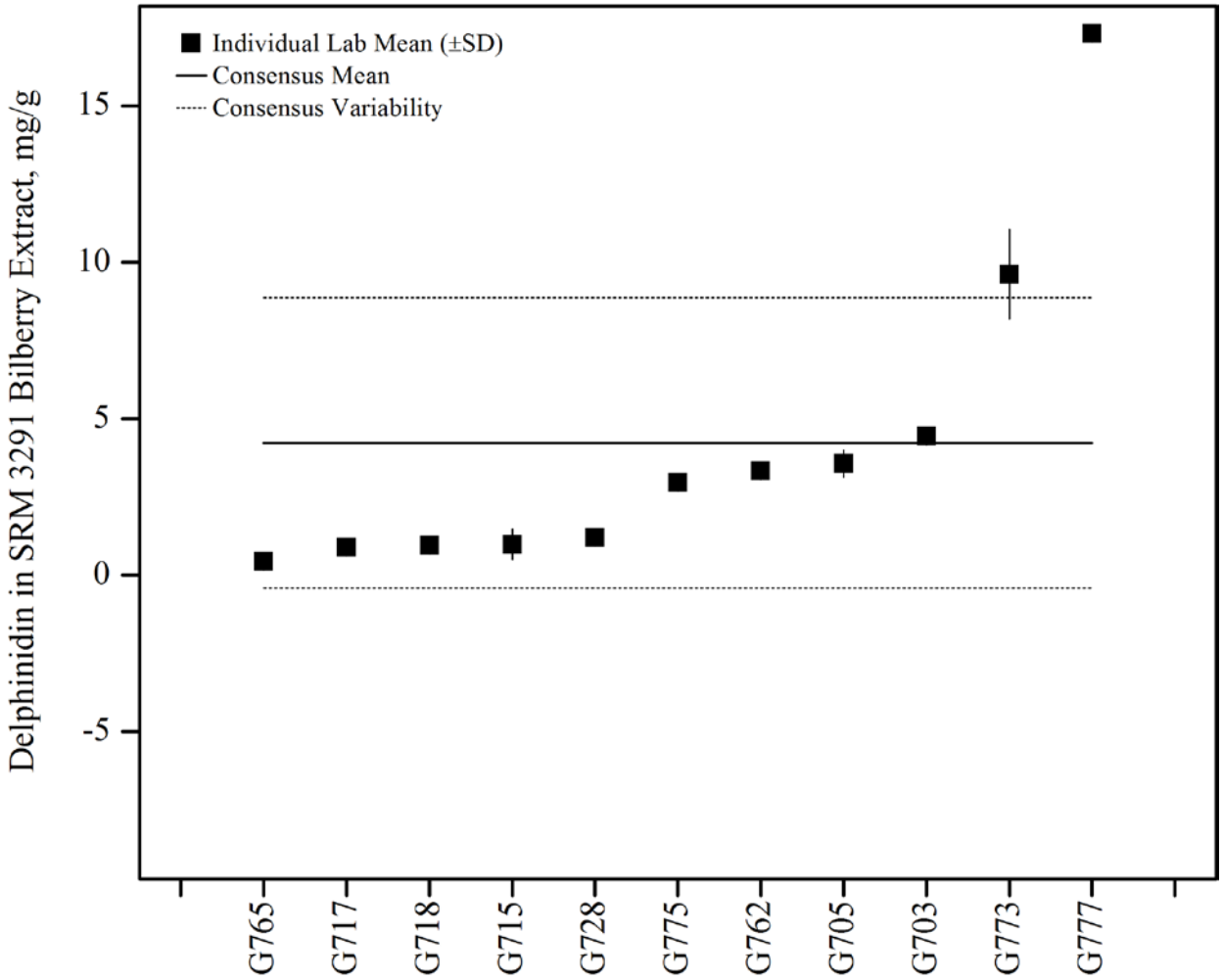

Figure 39. Delphinidin in SRM 3291 Bilberry Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

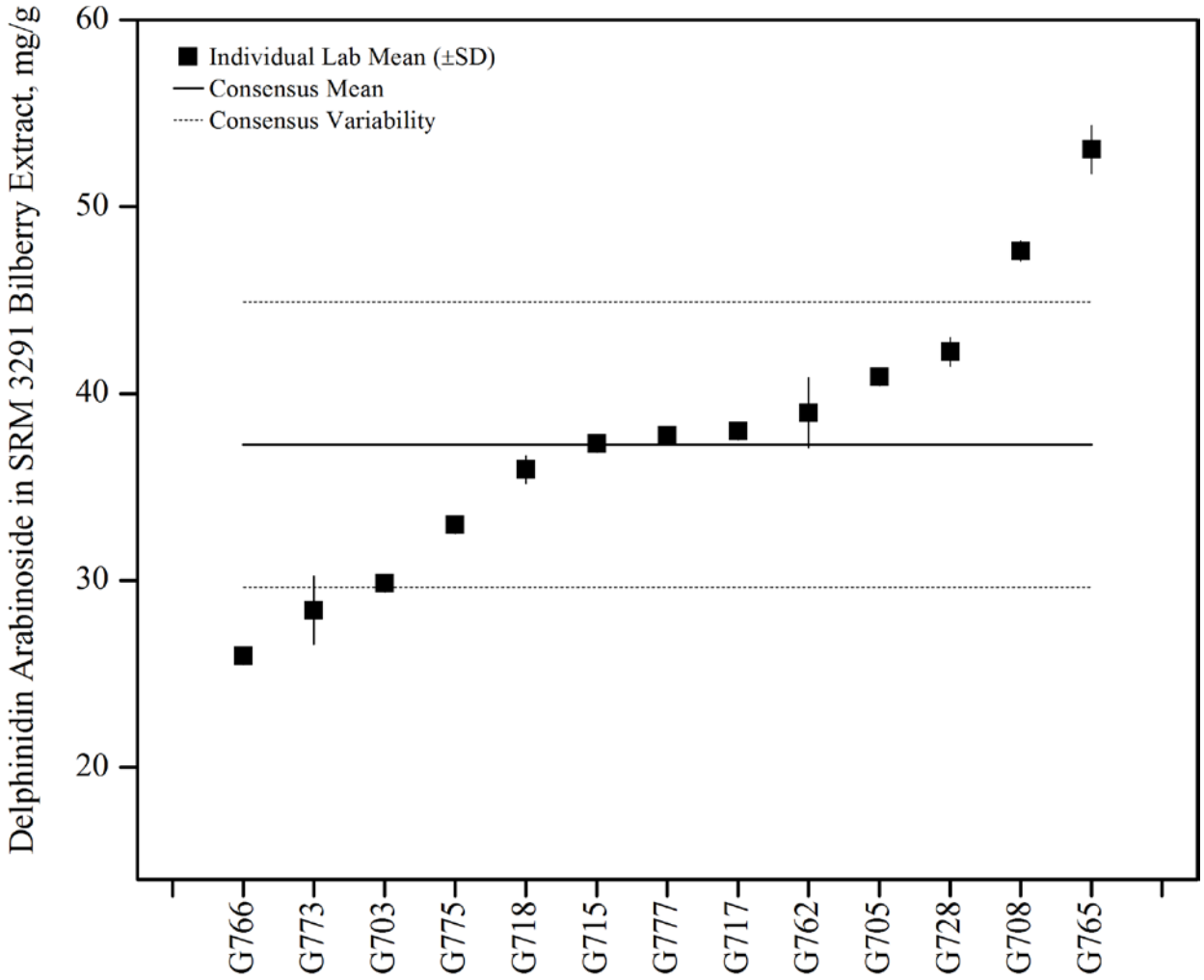

Figure 40. Delphinidin-3-arabinoside in SRM 3291 Bilberry Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

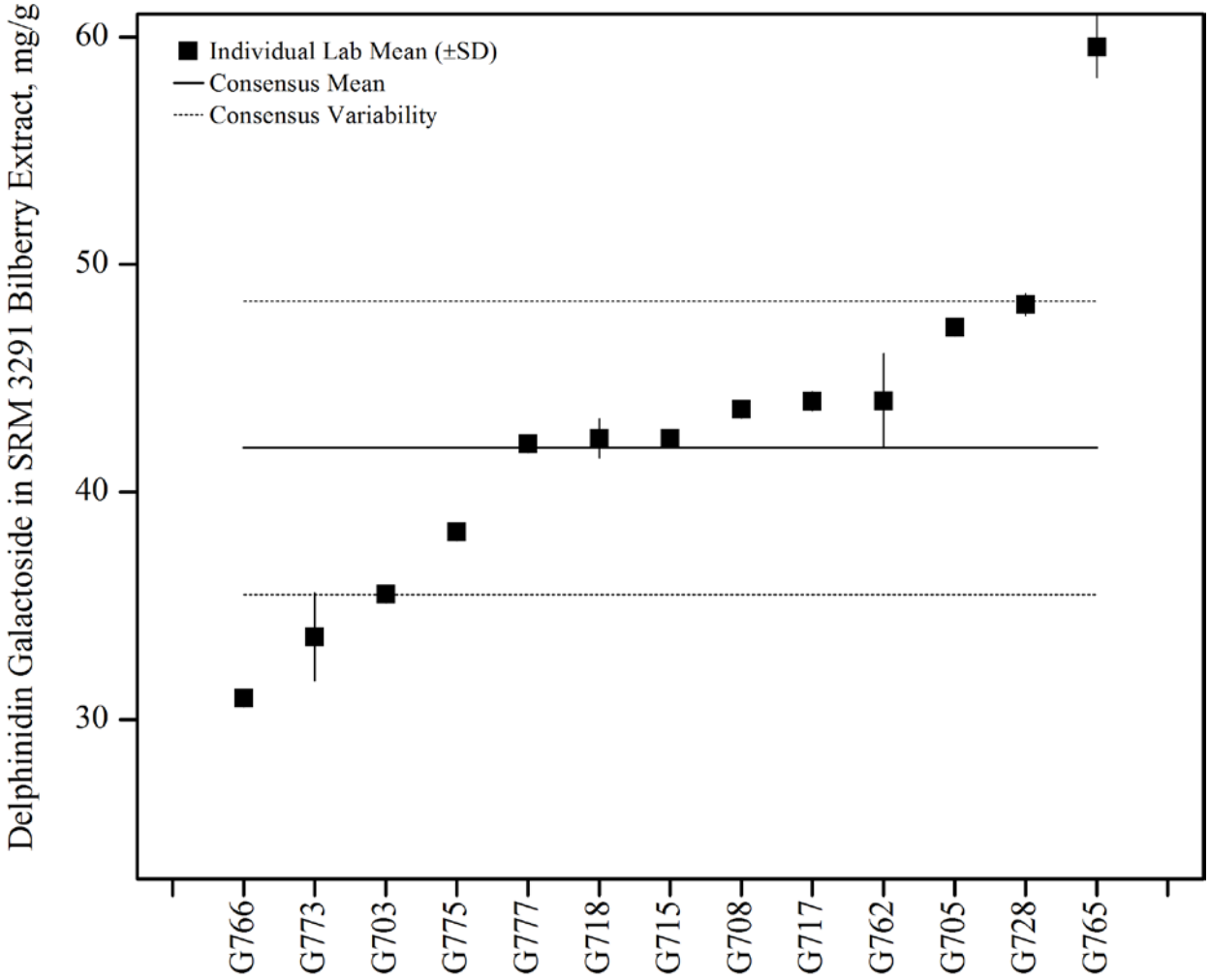

Figure 41. Delphinidin-3-galactoside in SRM 3291 Bilberry Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

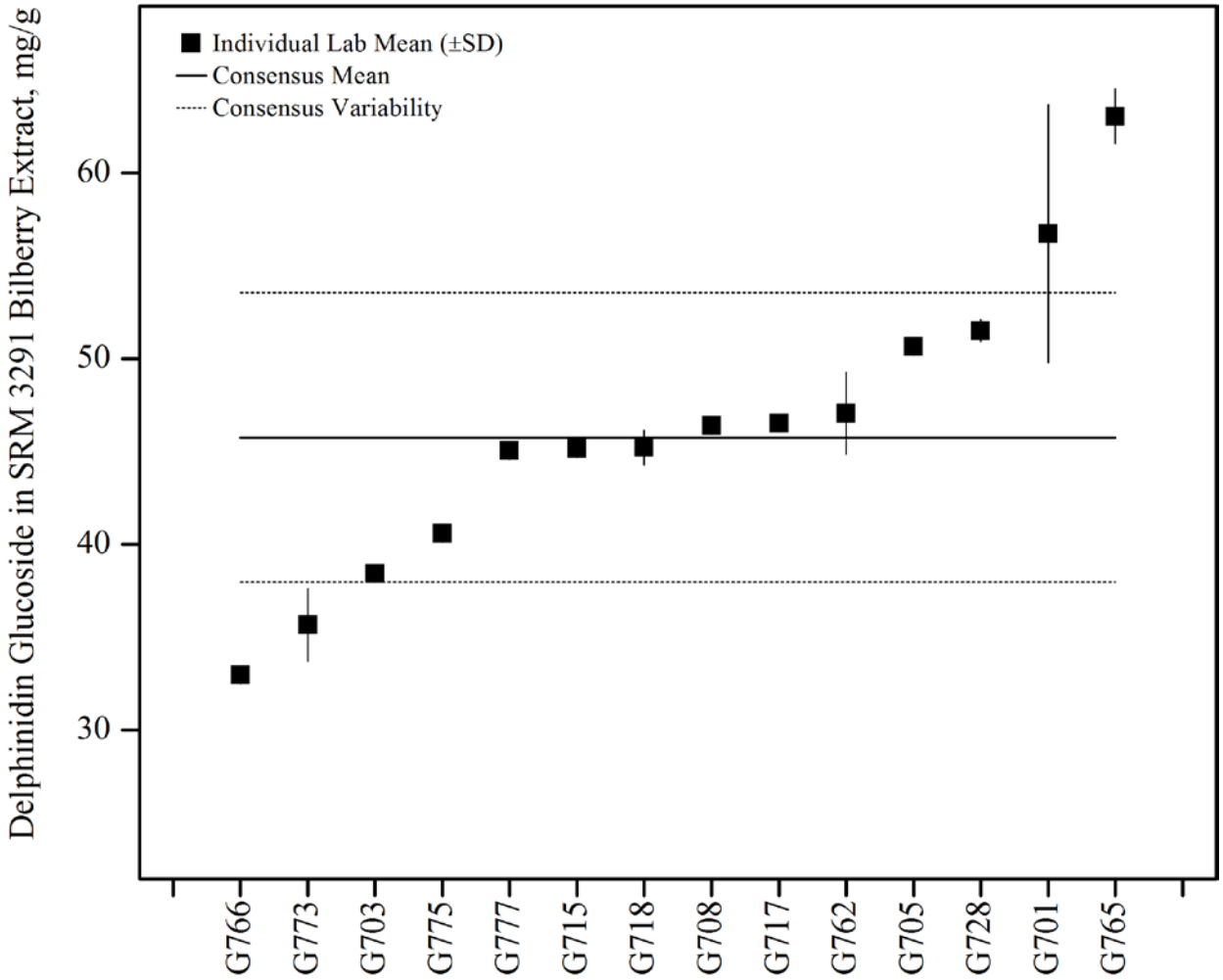

Figure 42. Delphinidin-3-glucoside in SRM 3291 Bilberry Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

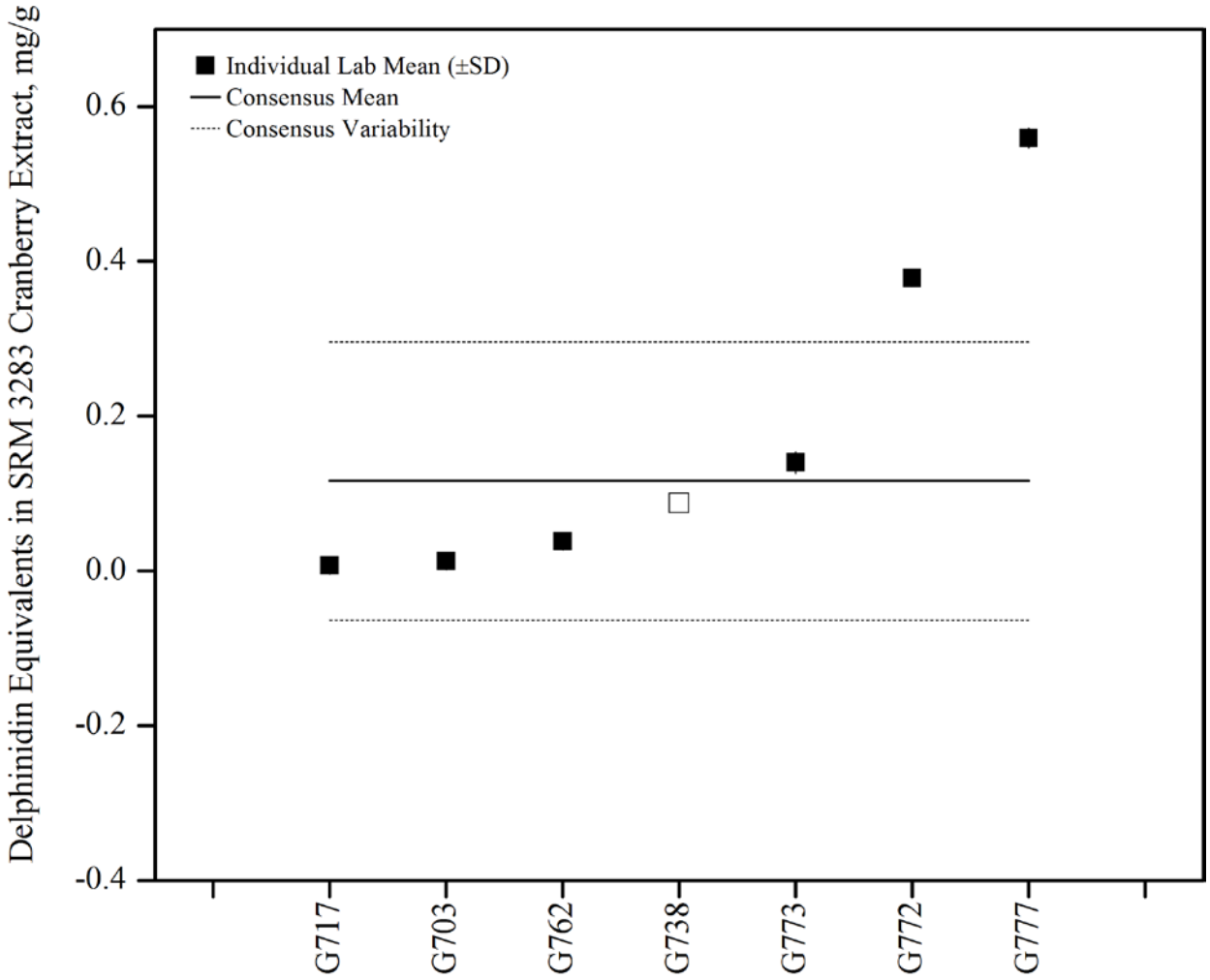

Figure 43. Delphinidin equivalents in SRM 3283 Cranberry Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

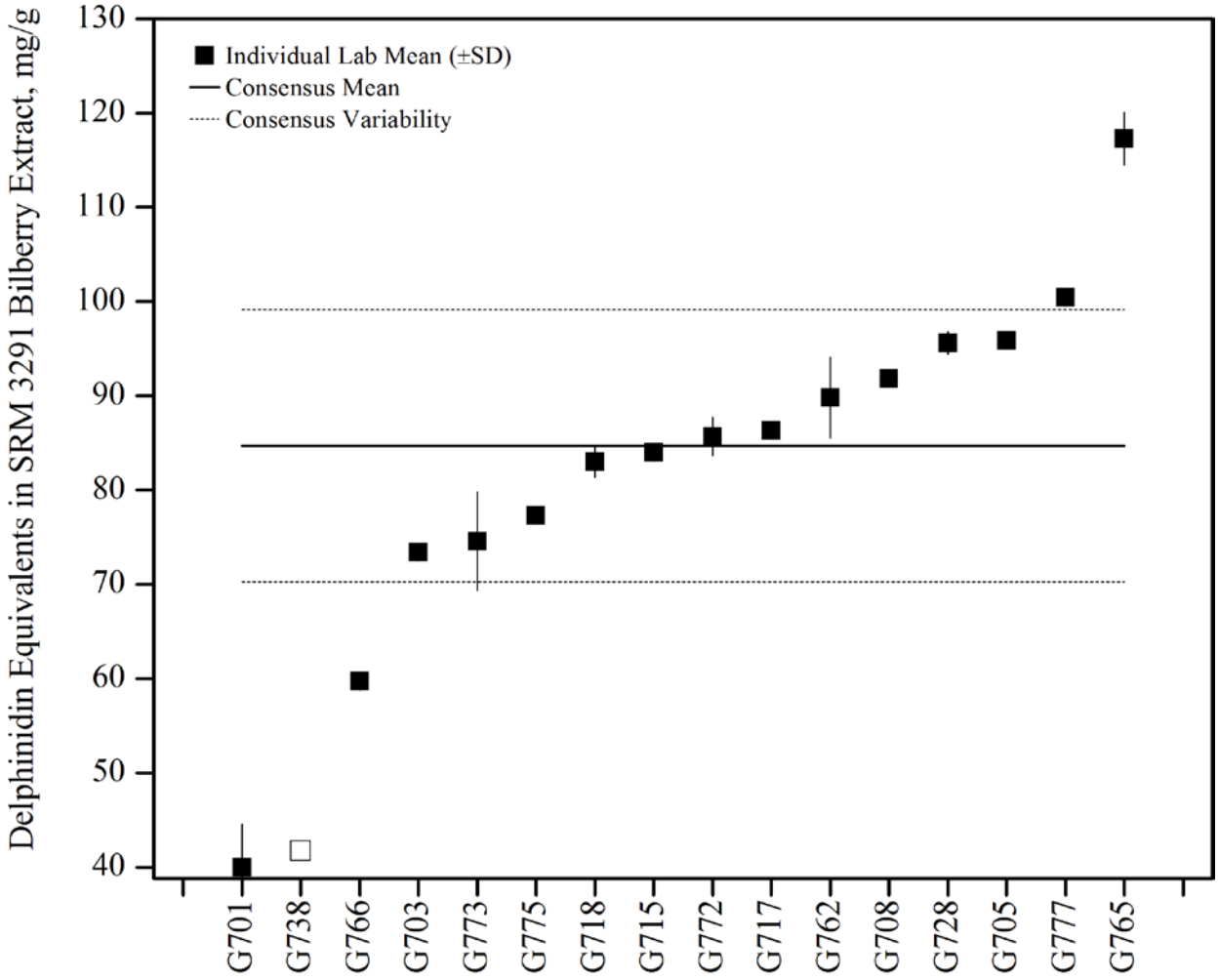

Figure 44. Delphinidin equivalents in SRM 3291 Bilberry Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

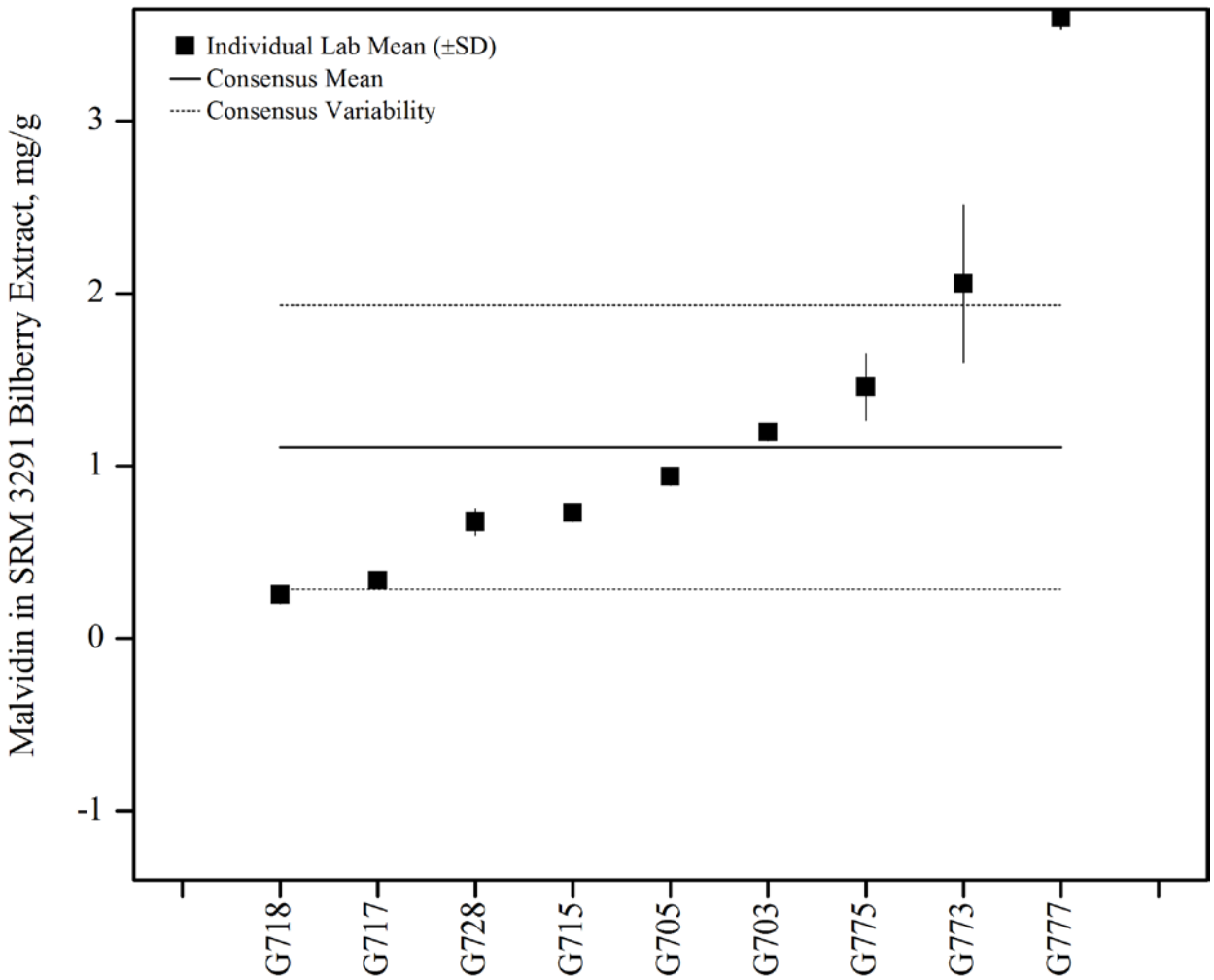

Figure 45. Malvidin in SRM 3291 Bilberry Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

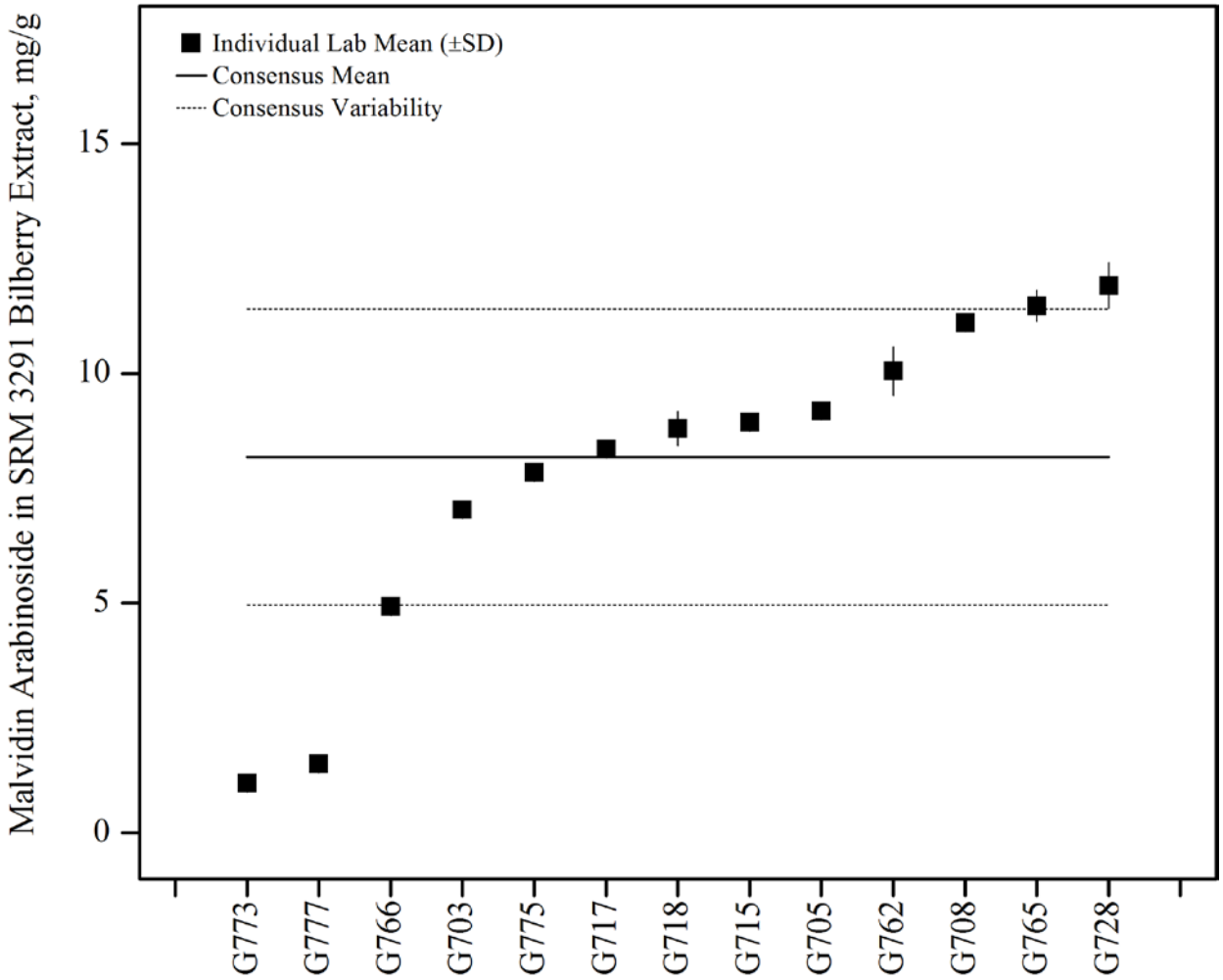

Figure 46. Malvidin-3-arabinoside in SRM 3291 Bilberry Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

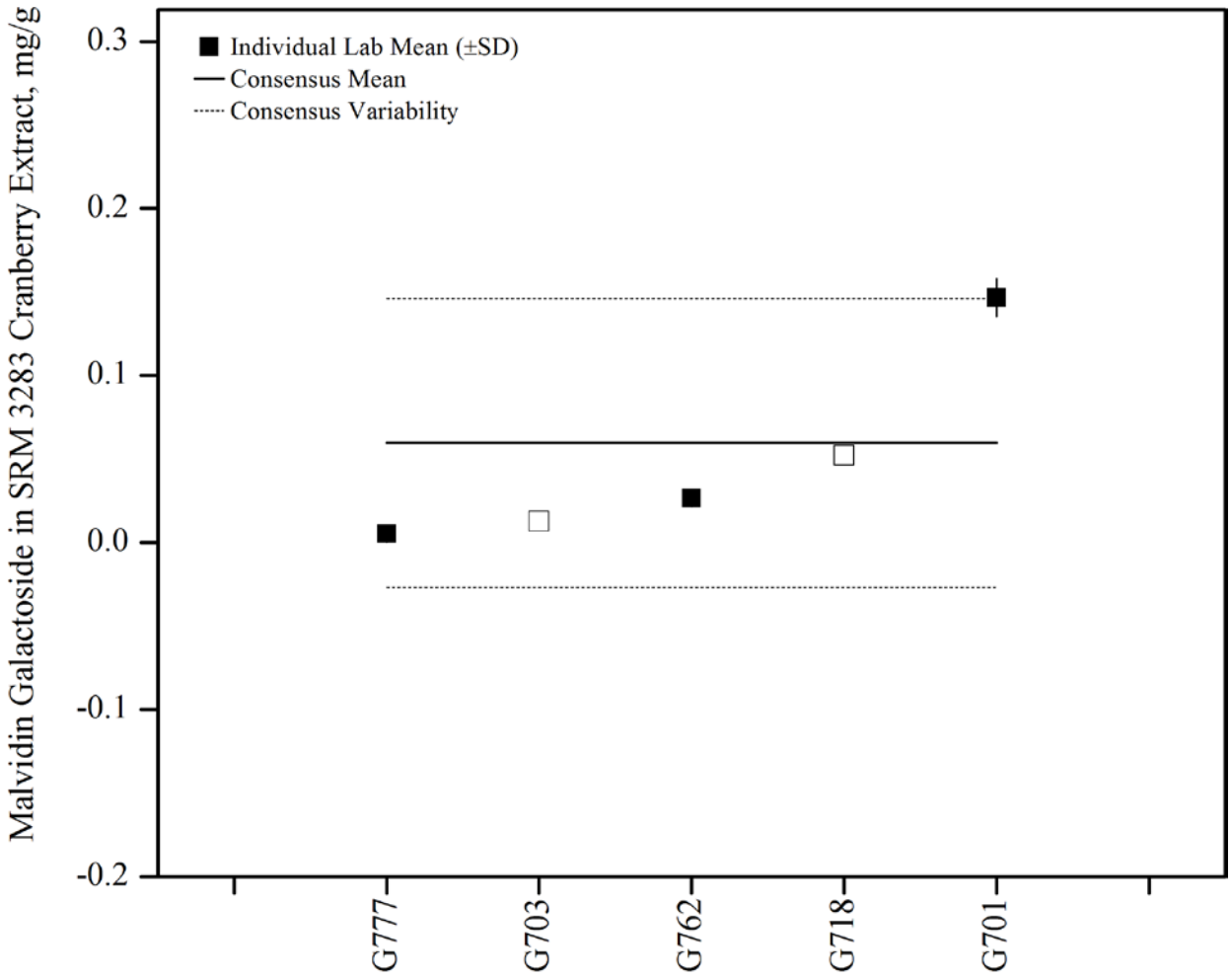

Figure 47. Malvidin-3-galactoside in SRM 3283 Cranberry Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

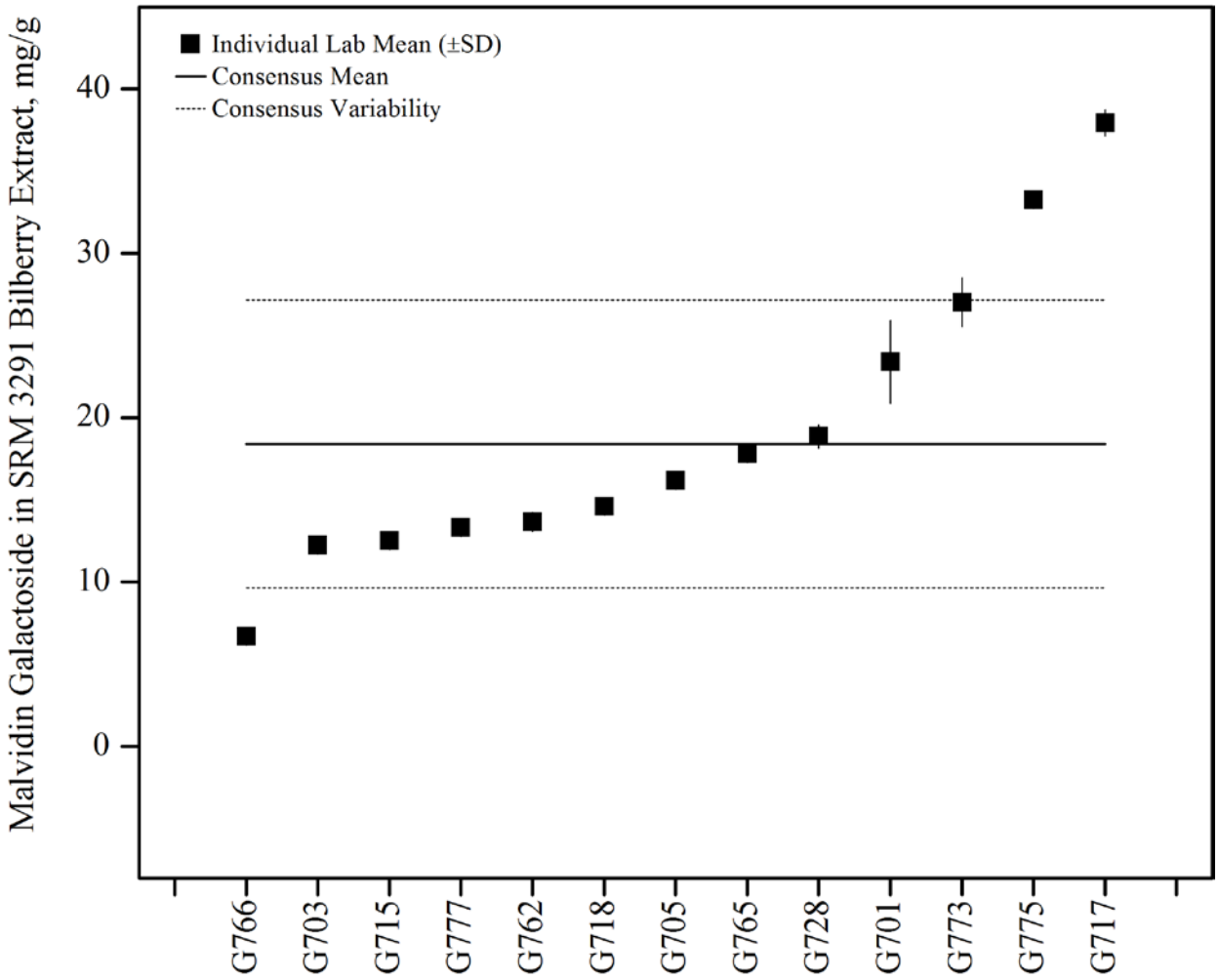

Figure 48. Malvidin-3-galactoside in SRM 3291 Bilberry Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

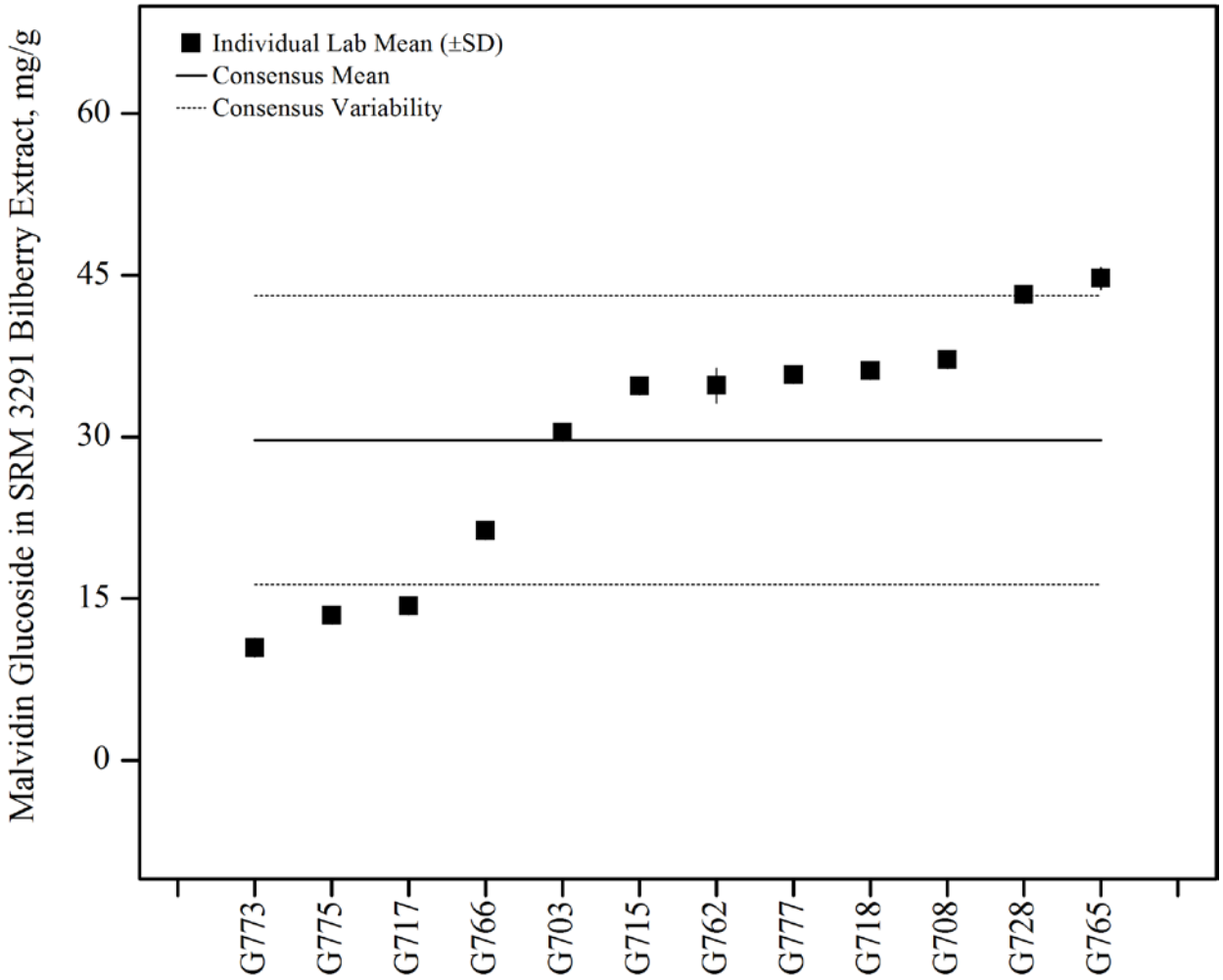

Figure 49. Malvidin-3-glucoside in SRM 3291 Bilberry Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

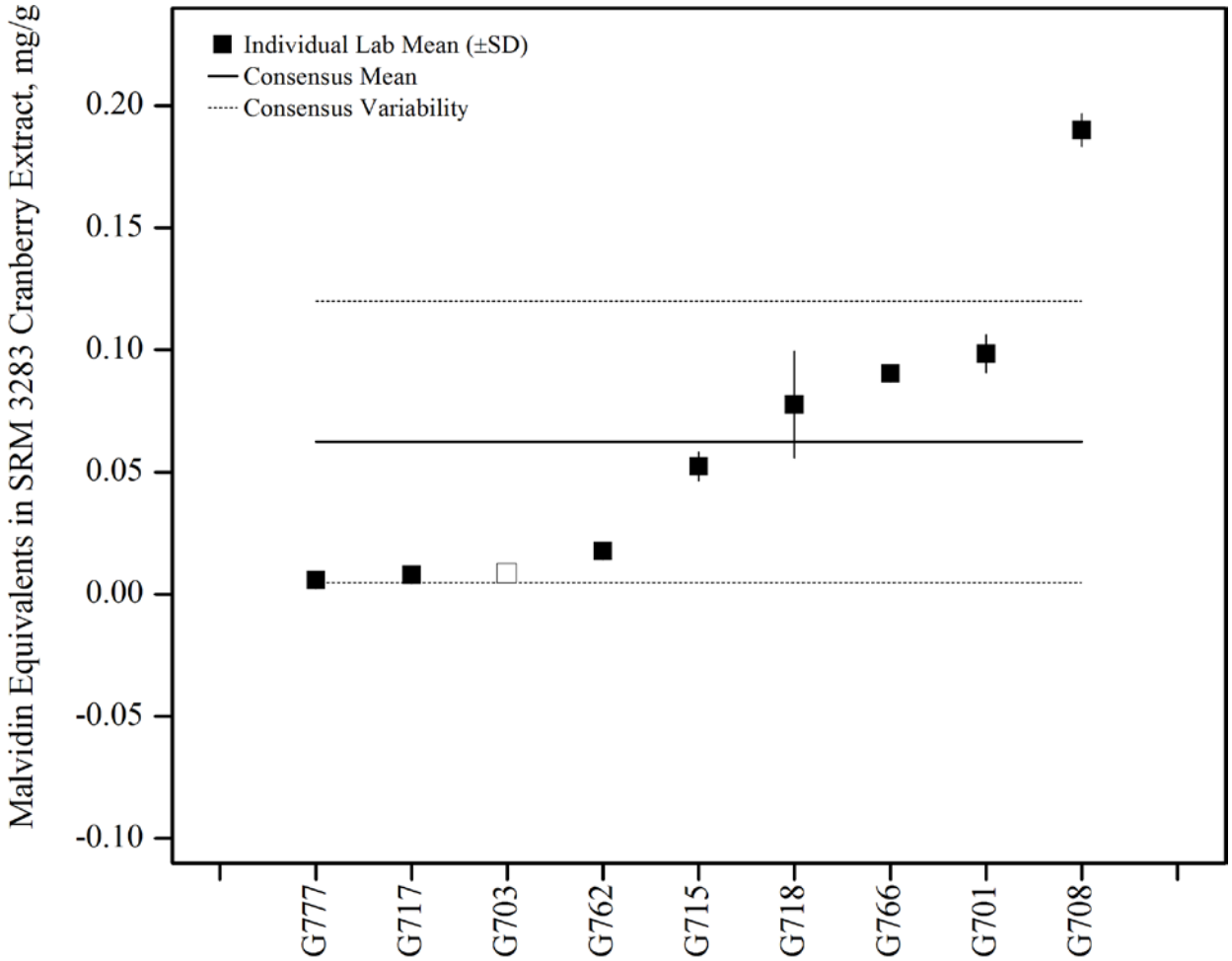

Figure 50. Malvidin equivalents in SRM 3283 Cranberry Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

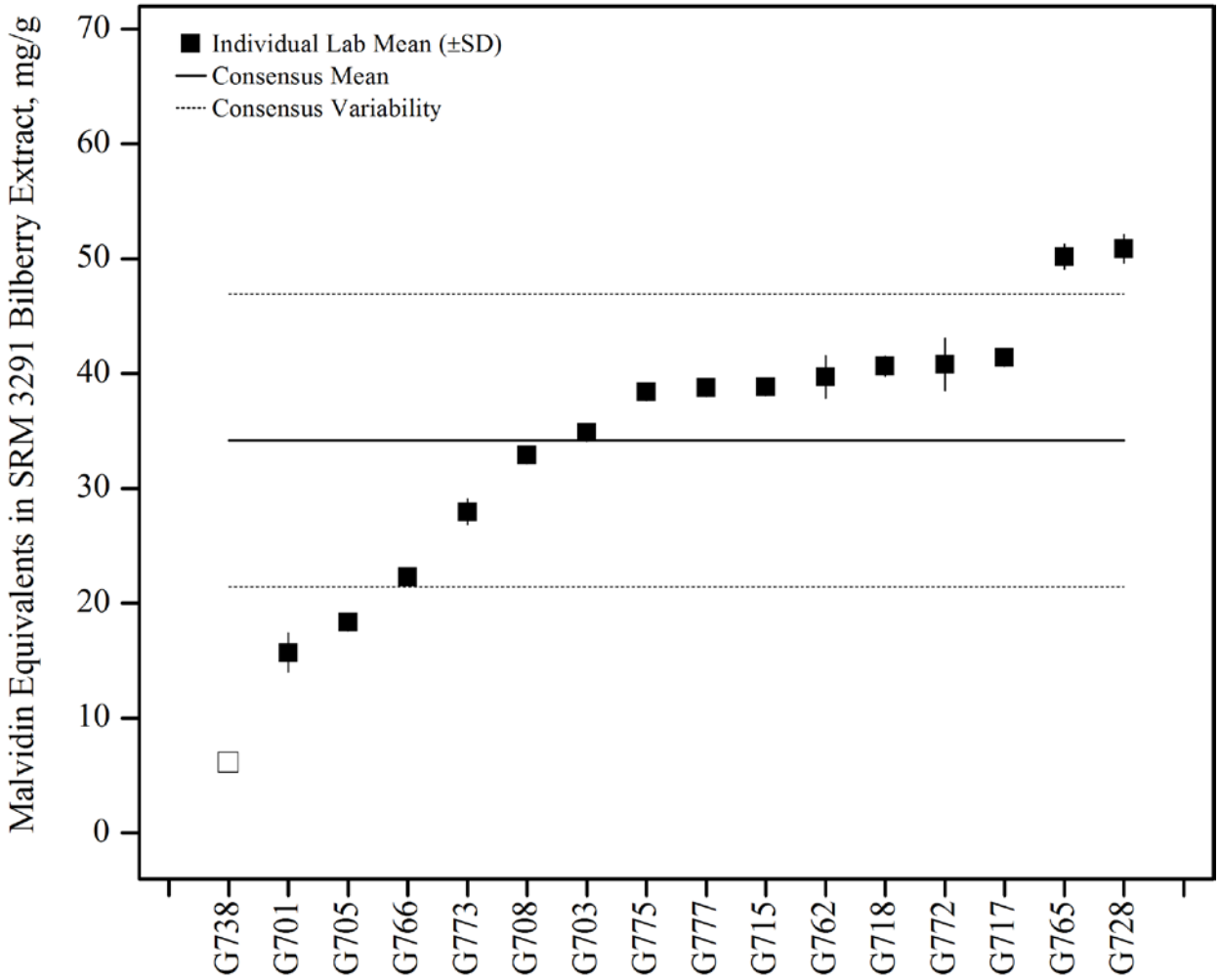

Figure 51. Malvidin equivalents in SRM 3291 Bilberry Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

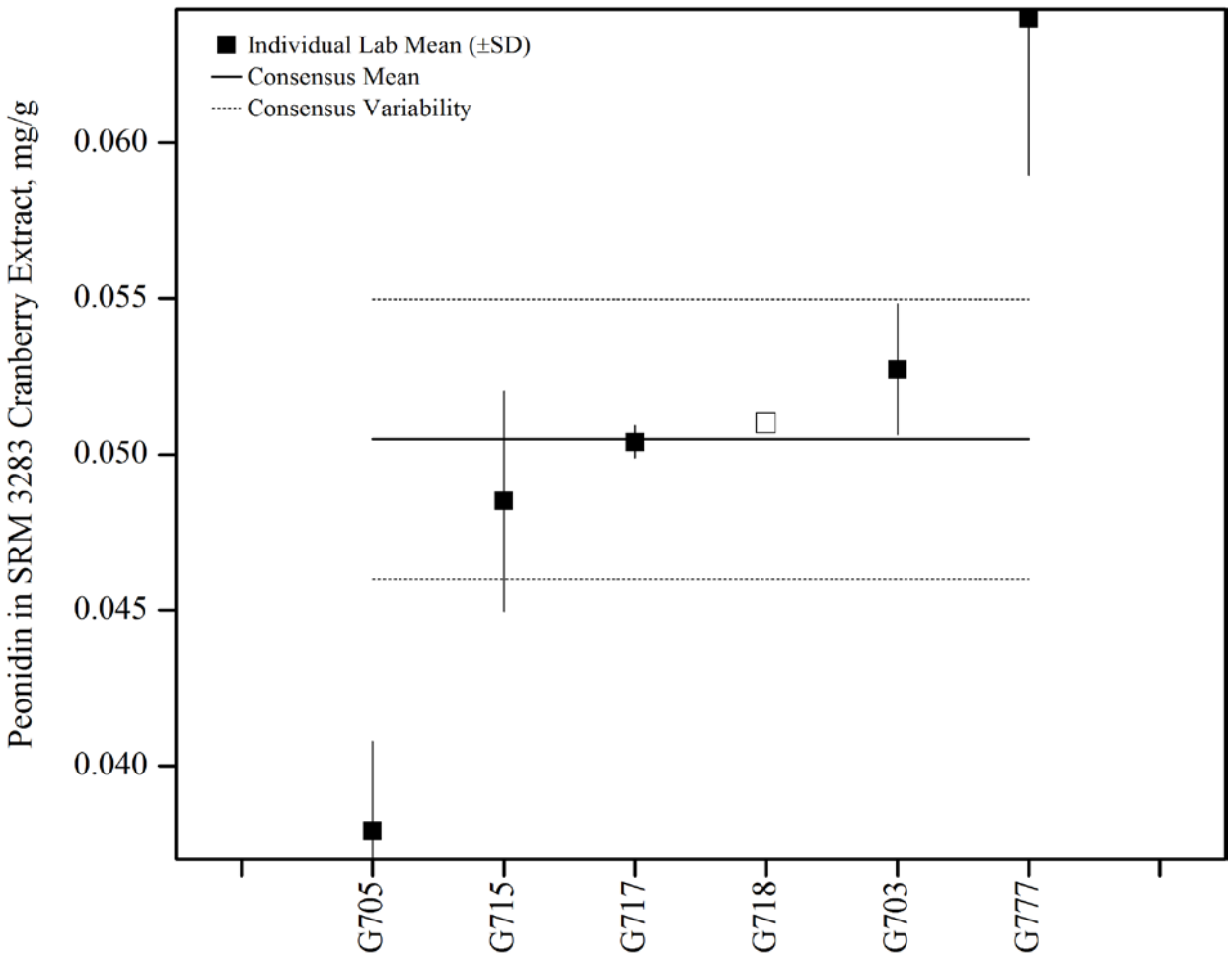

Figure 52. Peonidin in SRM 3283 Cranberry Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

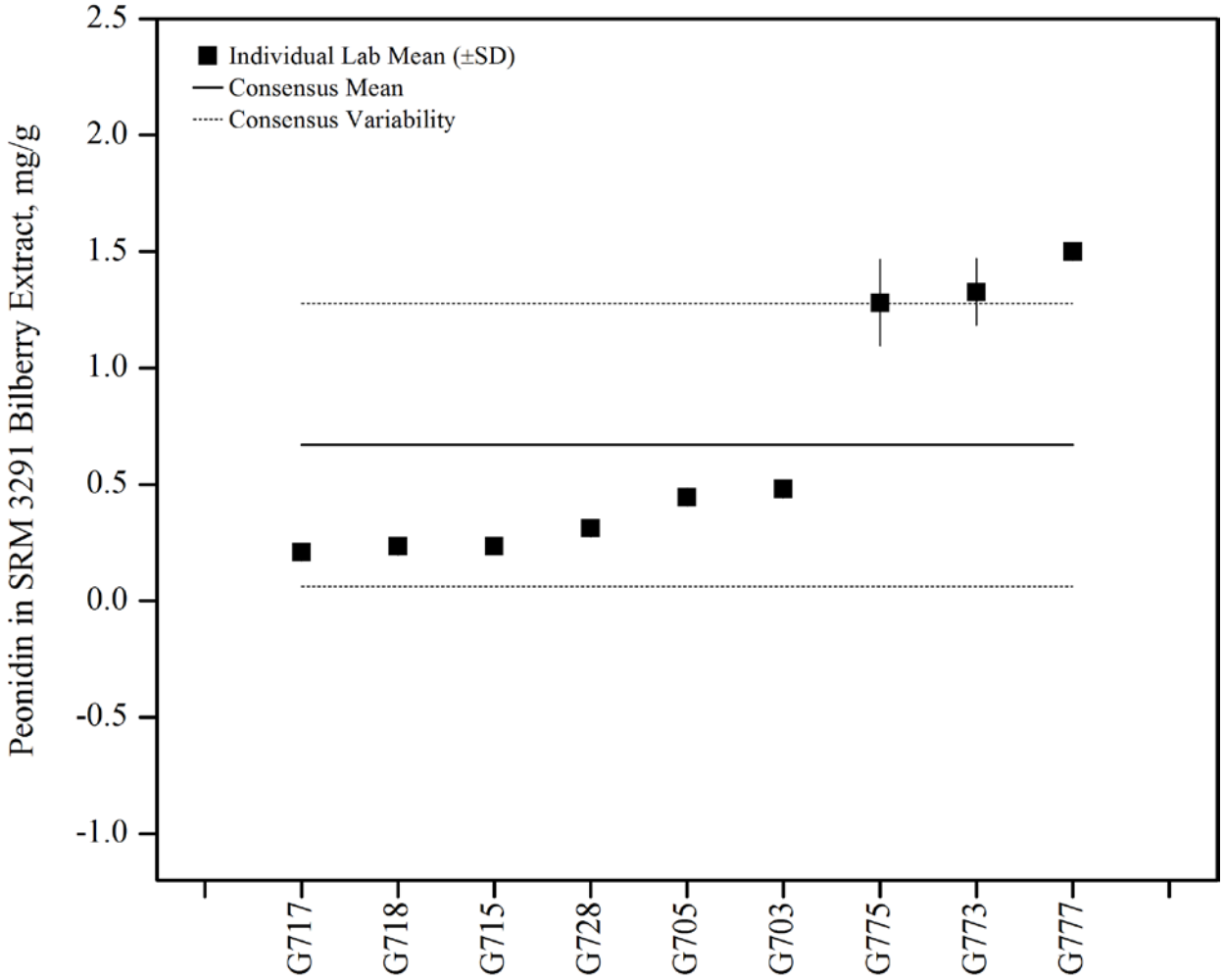

Figure 53. Peonidin in SRM 3291 Bilberry Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

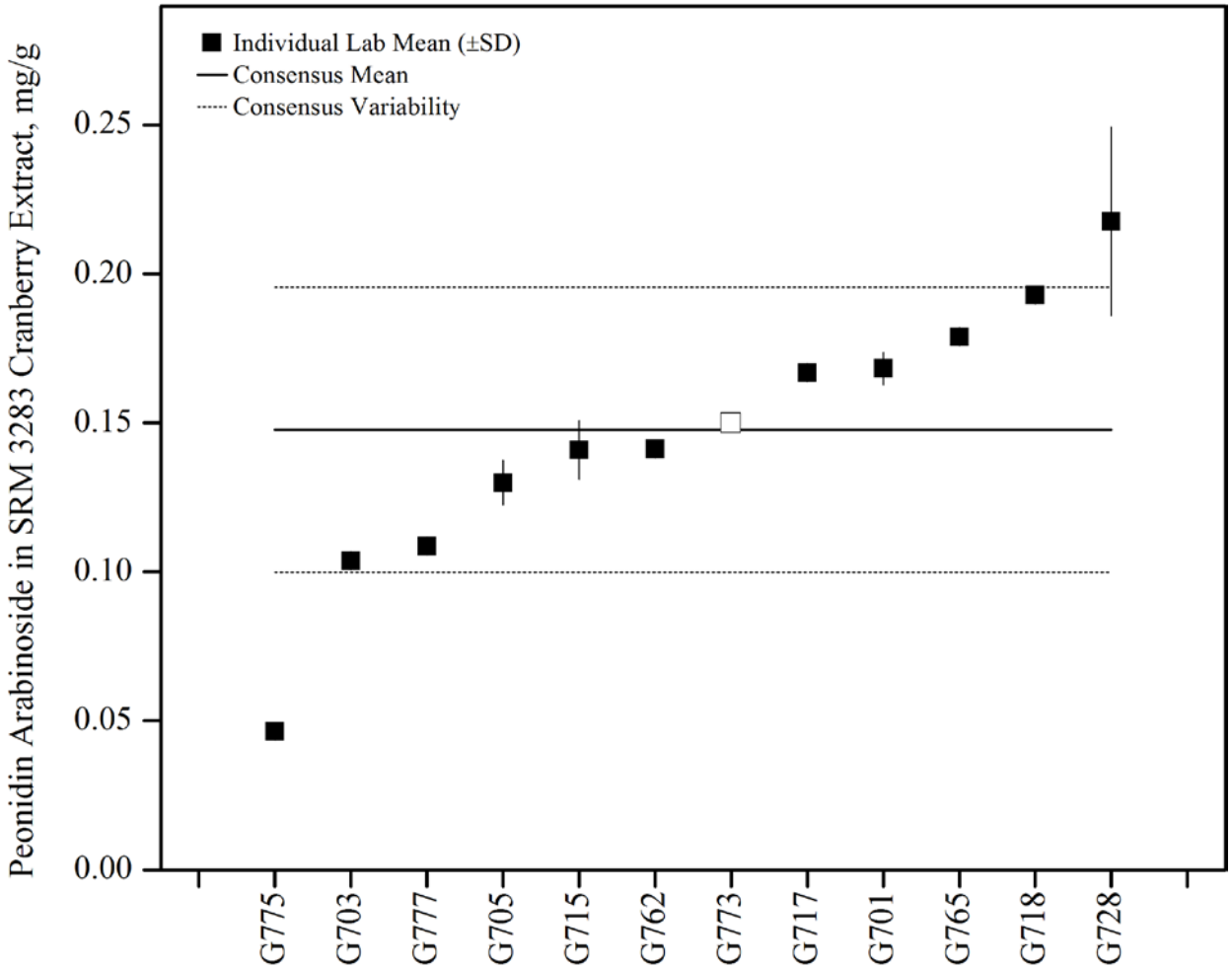

Figure 54. Peonidin-3-arabinoside in SRM 3283 Cranberry Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

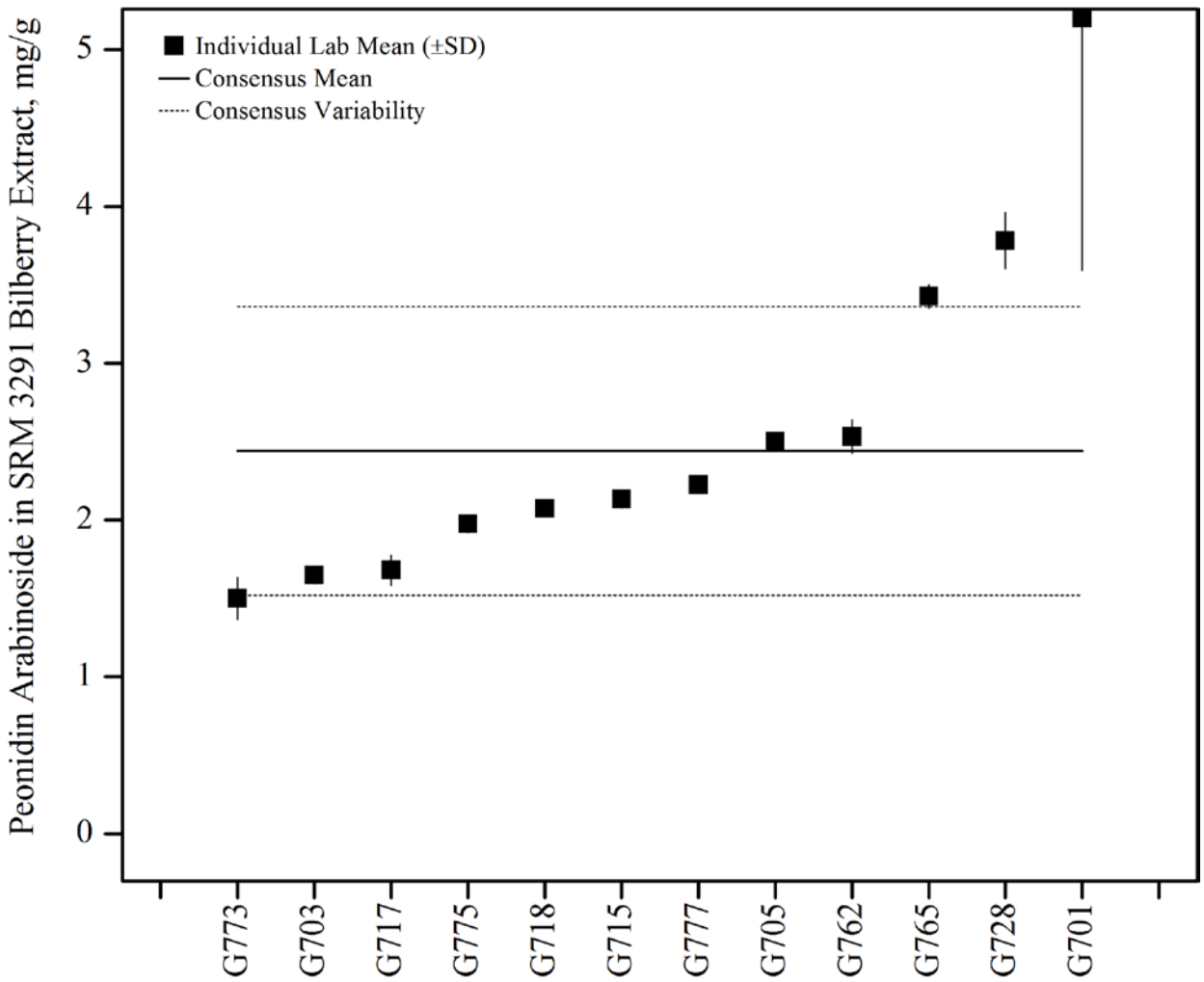

Figure 55. Peonidin-3-arabinoside in SRM 3291 Bilberry Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

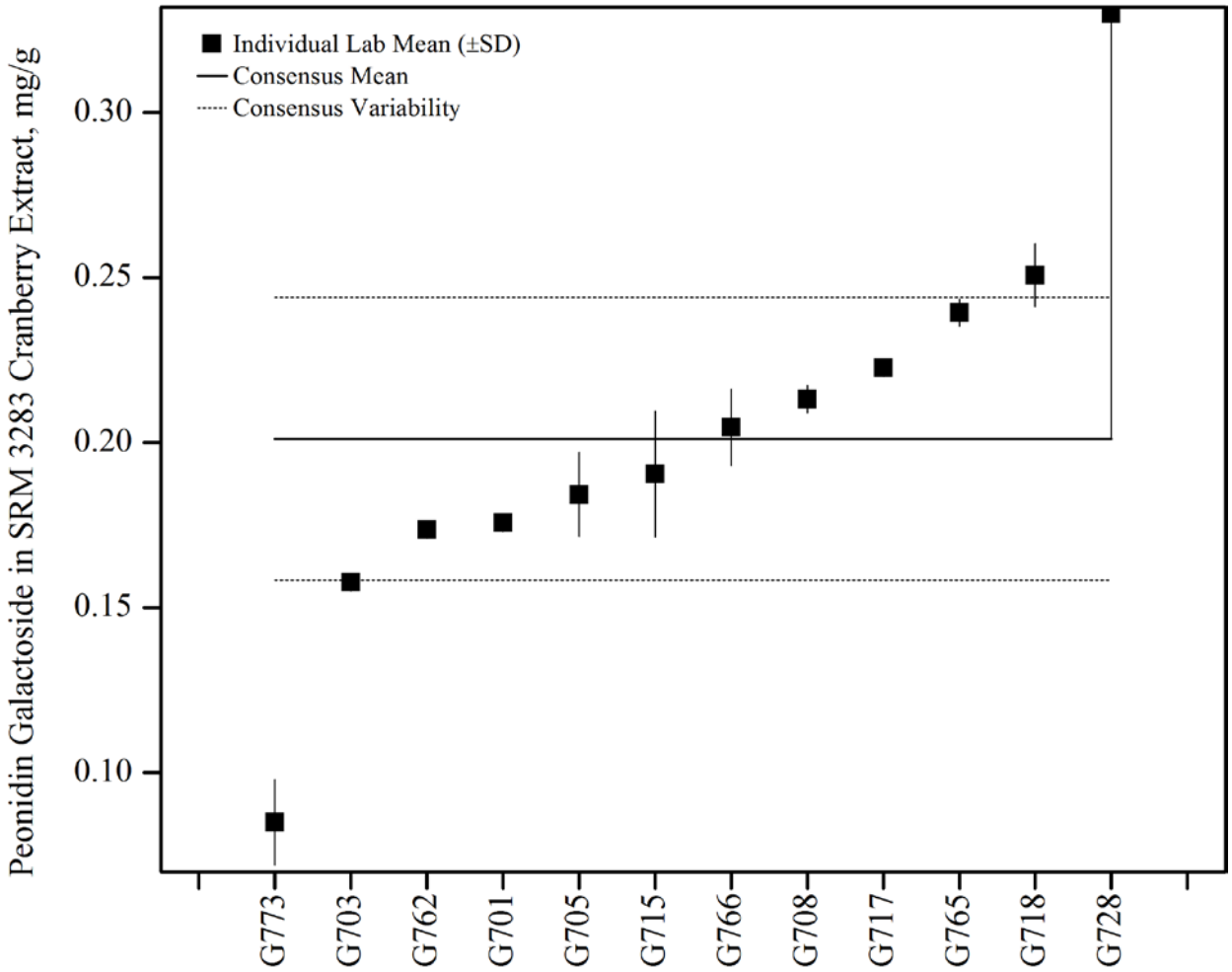

Figure 56. Peonidin-3-galactoside in SRM 3283 Cranberry Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

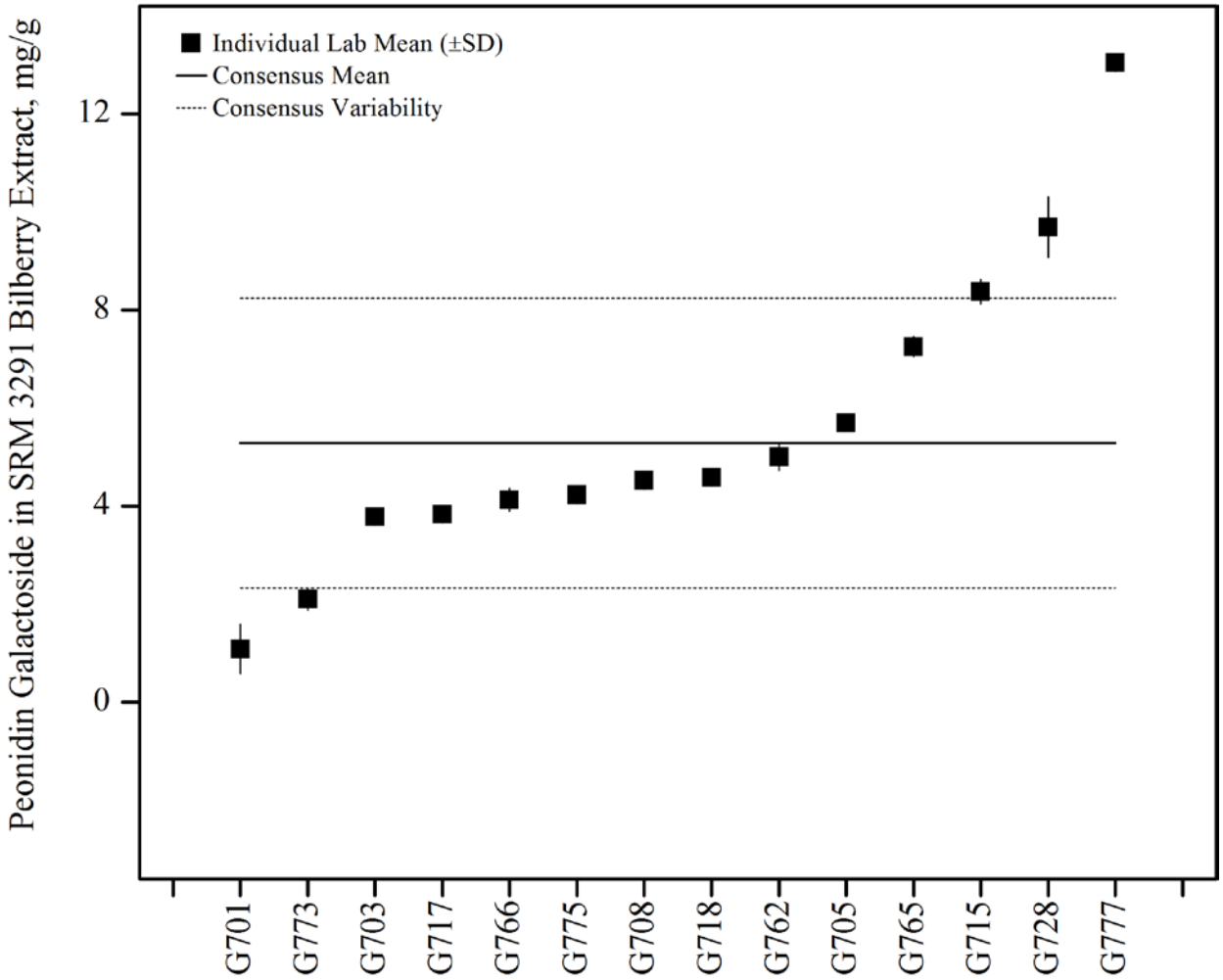

Figure 57. Peonidin-3-galactoside in SRM 3291 Bilberry Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

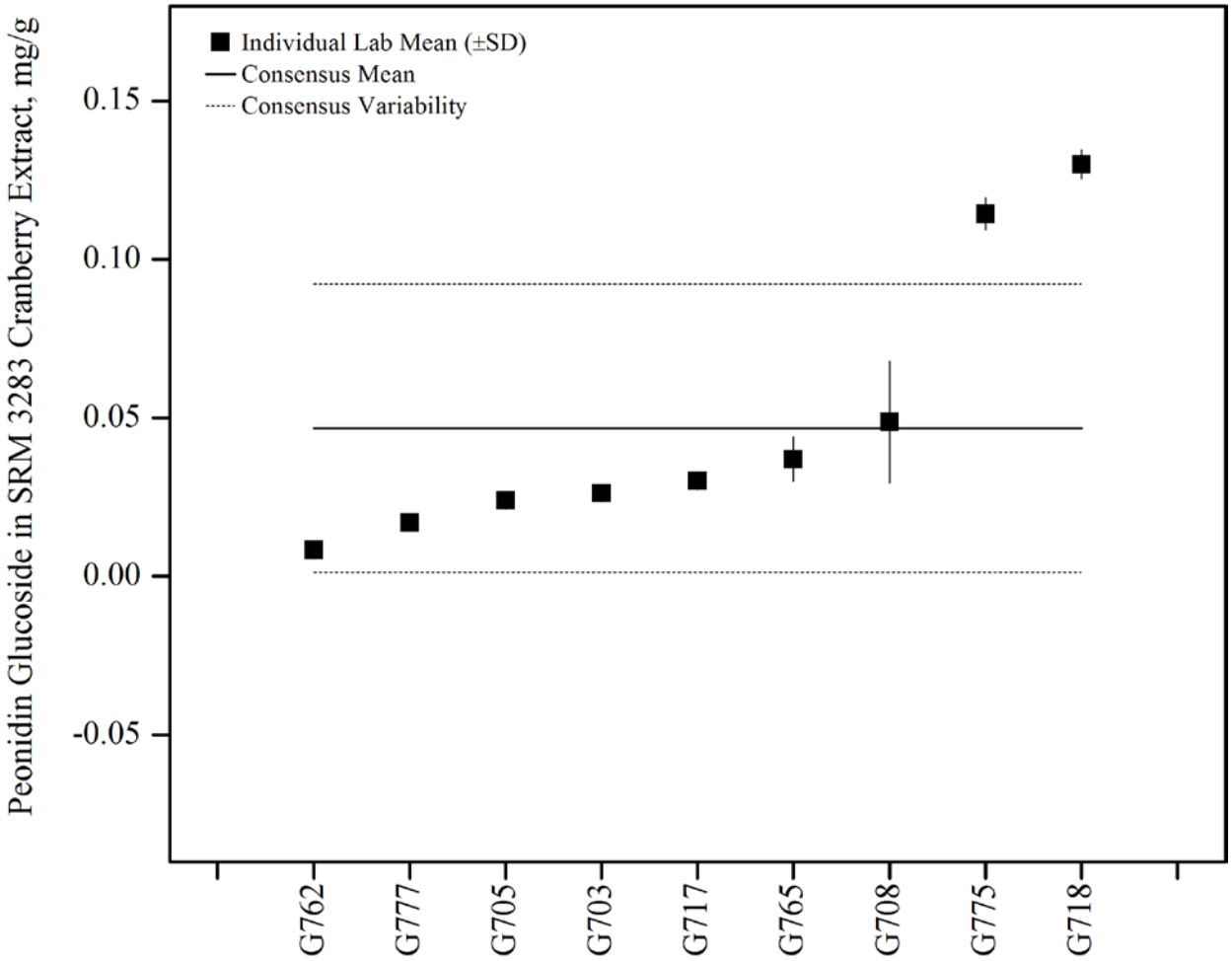

Figure 58. Peonidin-3-glucoside in SRM 3283 Cranberry Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

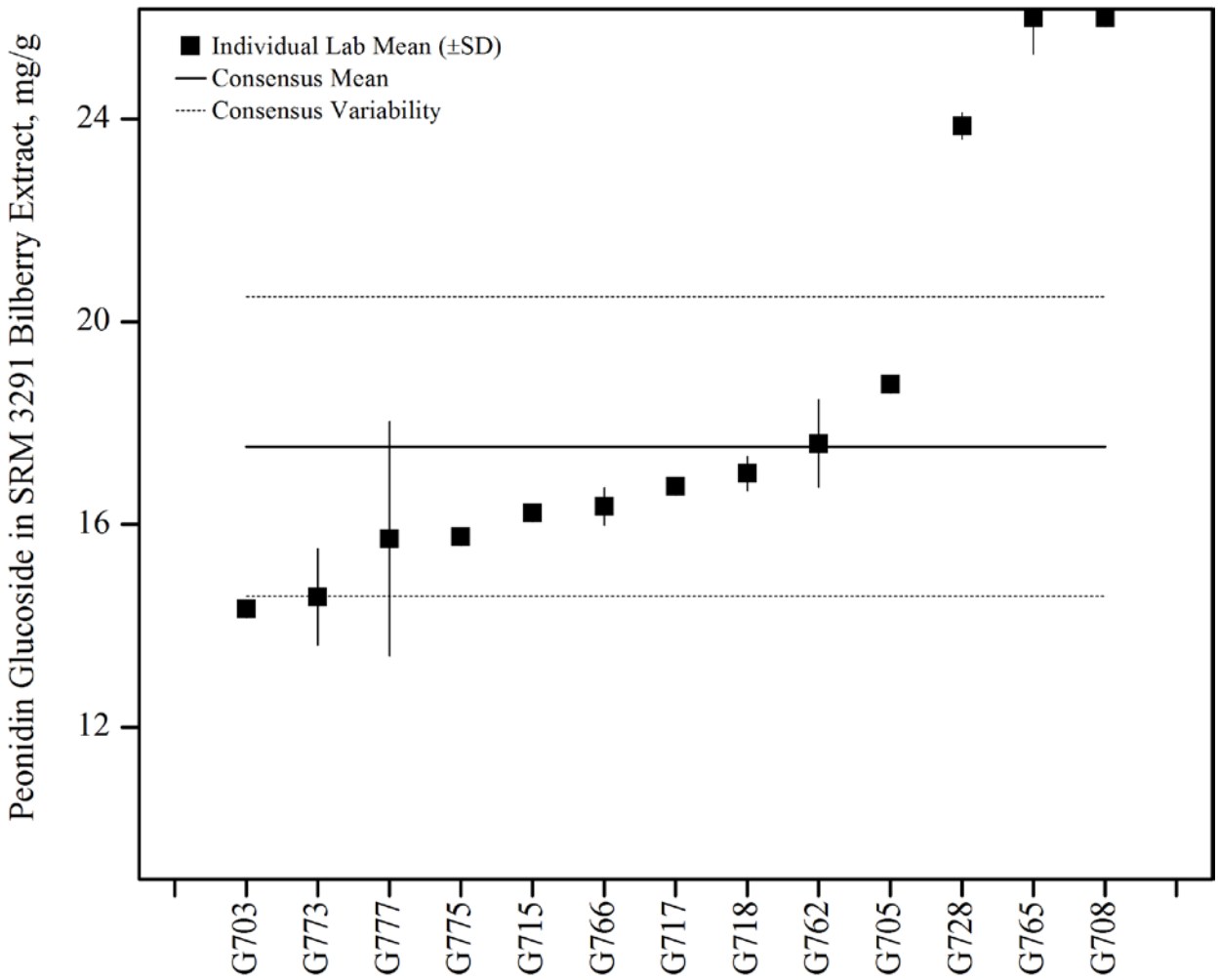

Figure 59. Peonidin-3-glucoside in SRM 3291 Bilberry Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

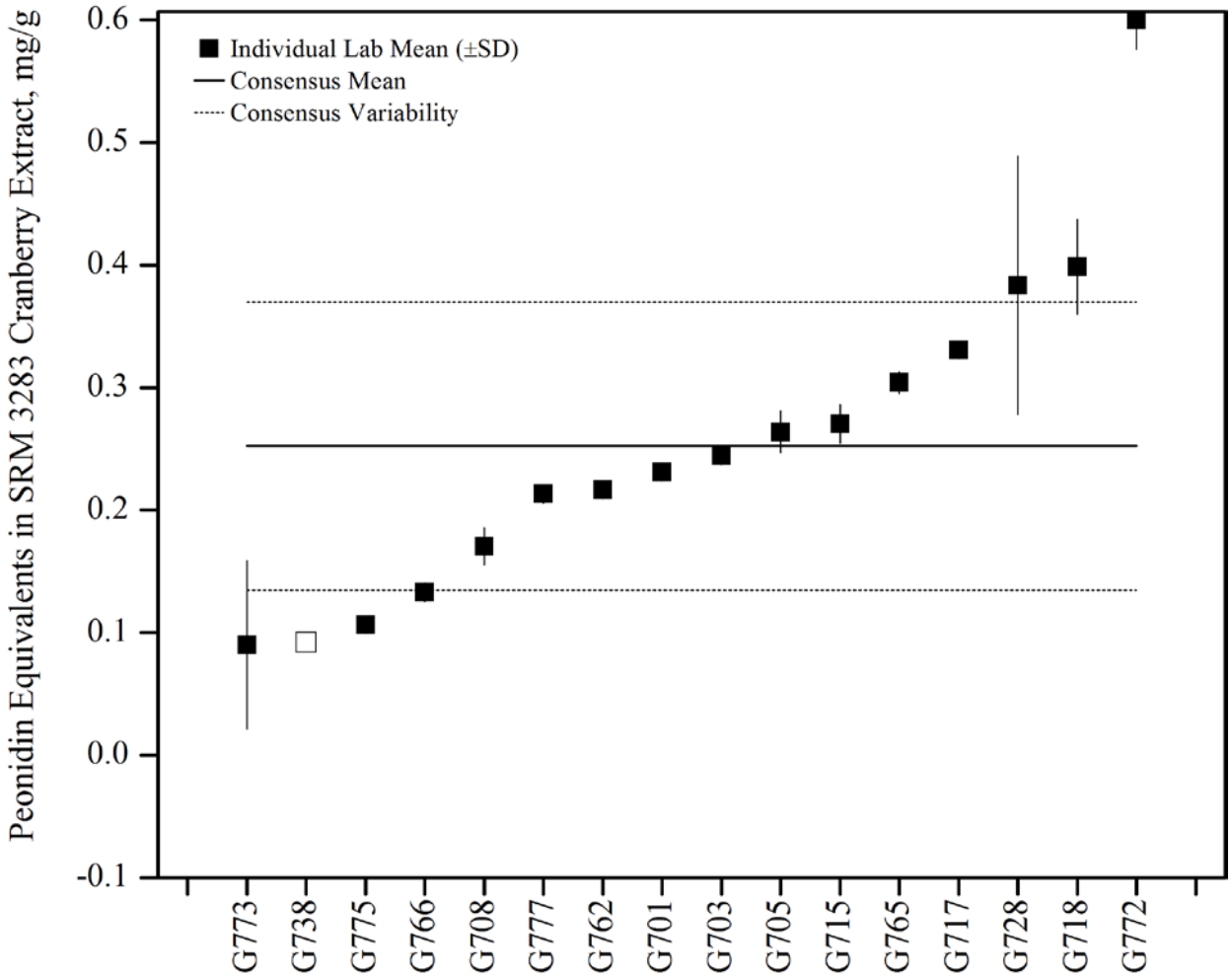

Figure 60. Peonidin equivalents in SRM 3283 Cranberry Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

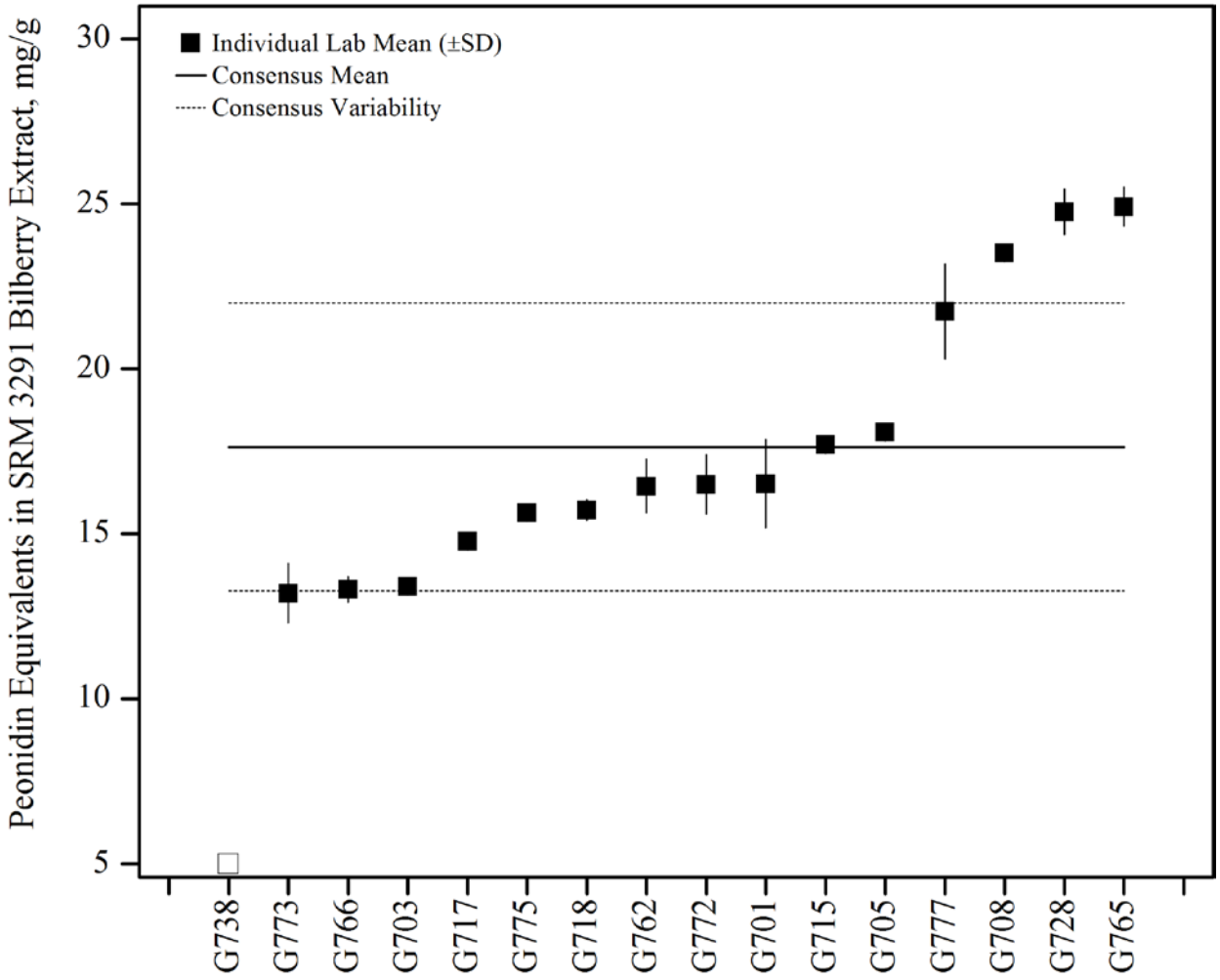

Figure 61. Peonidin equivalents in SRM 3291 Bilberry Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

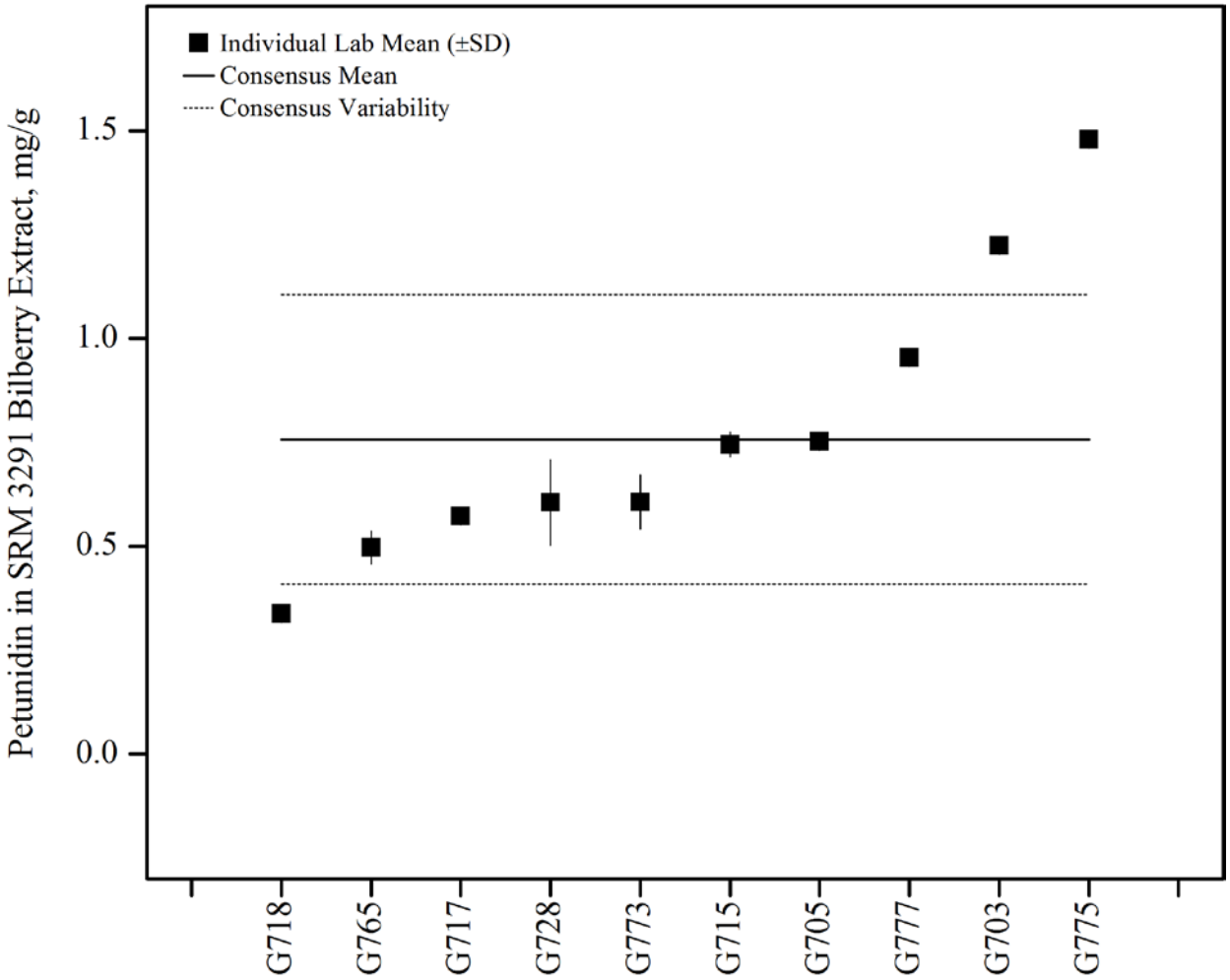

Figure 62. Petunidin in SRM 3291 Bilberry Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

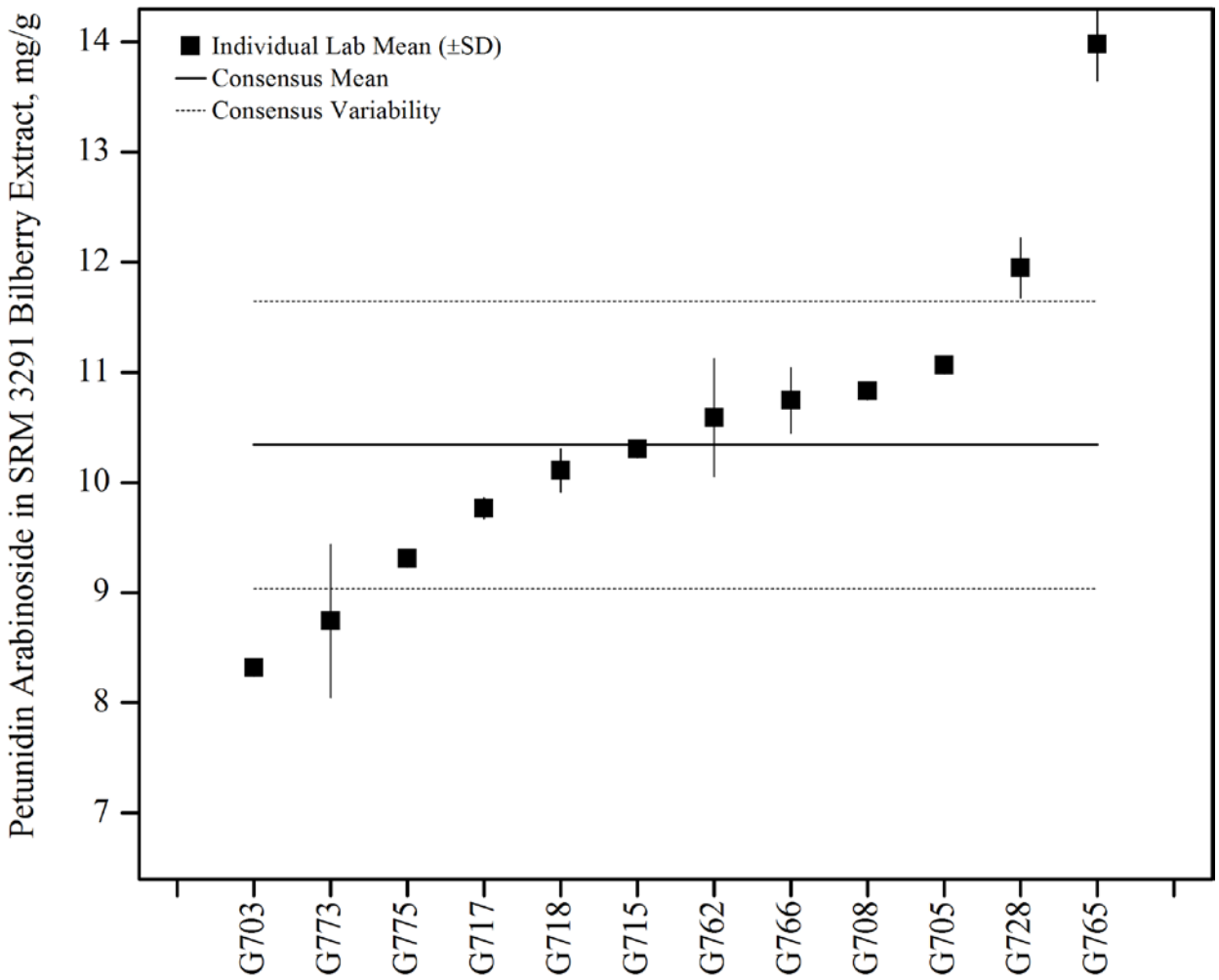

Figure 63. Petunidin-3-arabinoside in SRM 3291 Bilberry Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

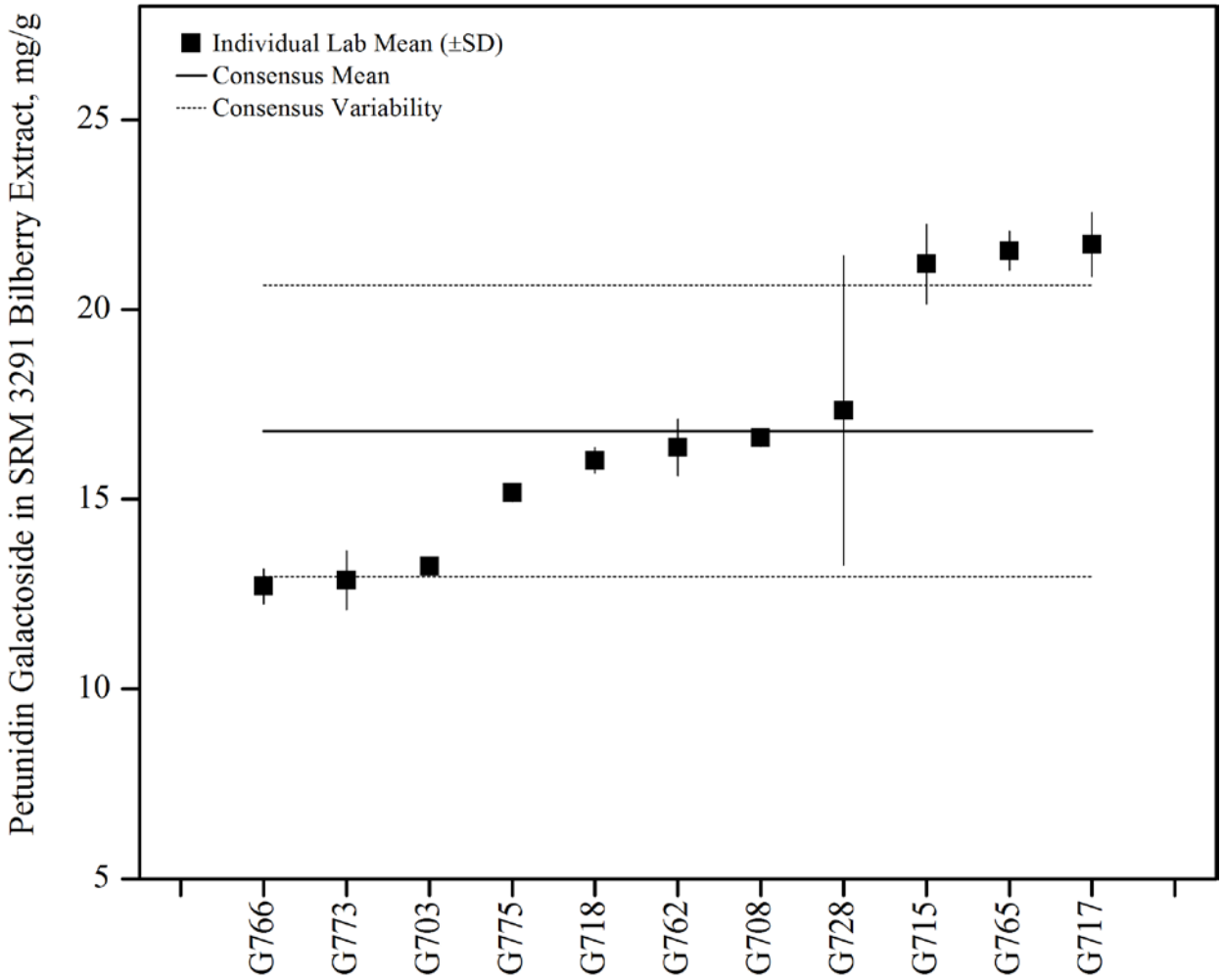

Figure 64. Petunidin-3-galactoside in SRM 3291 Bilberry Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

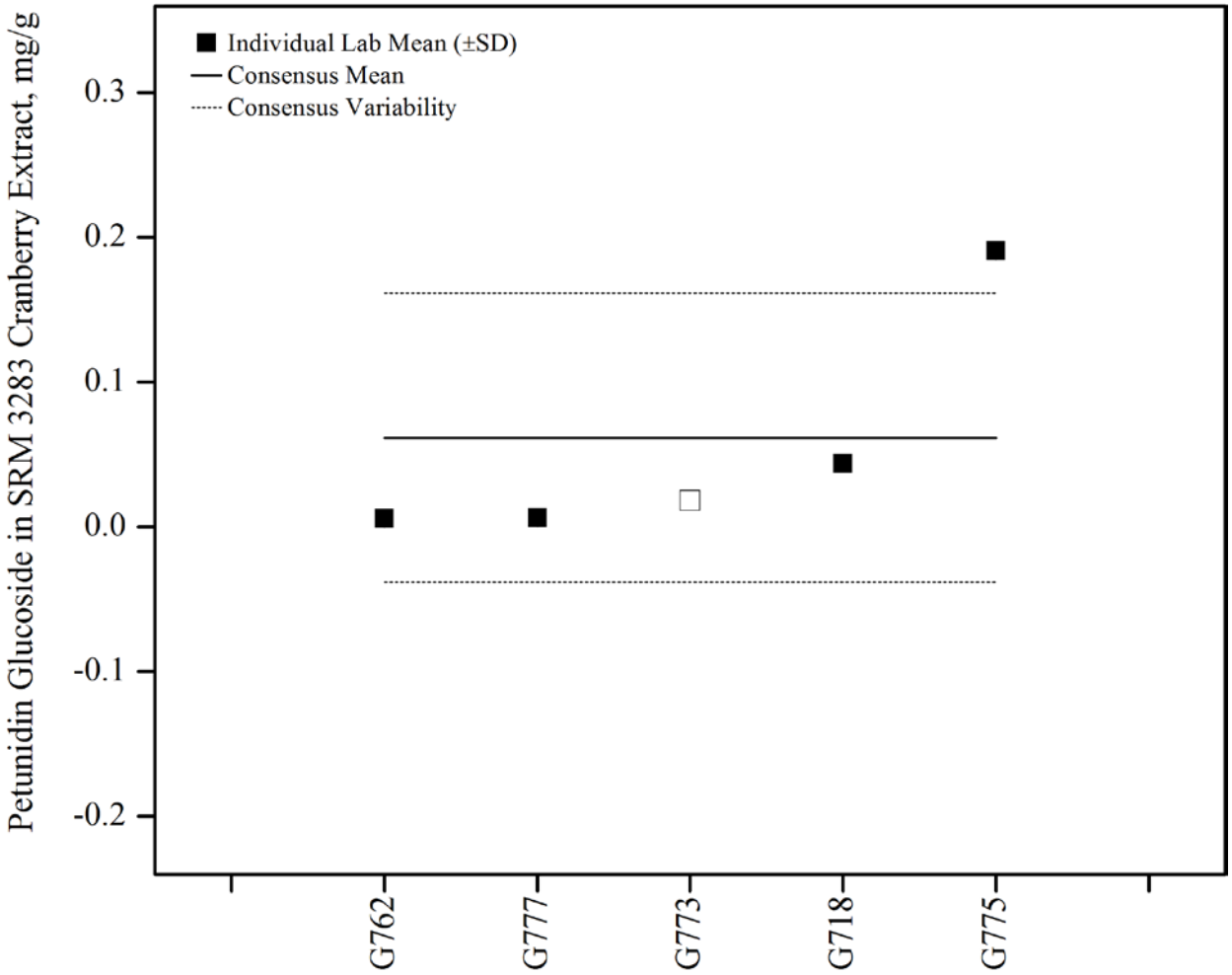

Figure 65. Petunidin-3-glucoside in SRM 3283 Cranberry Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

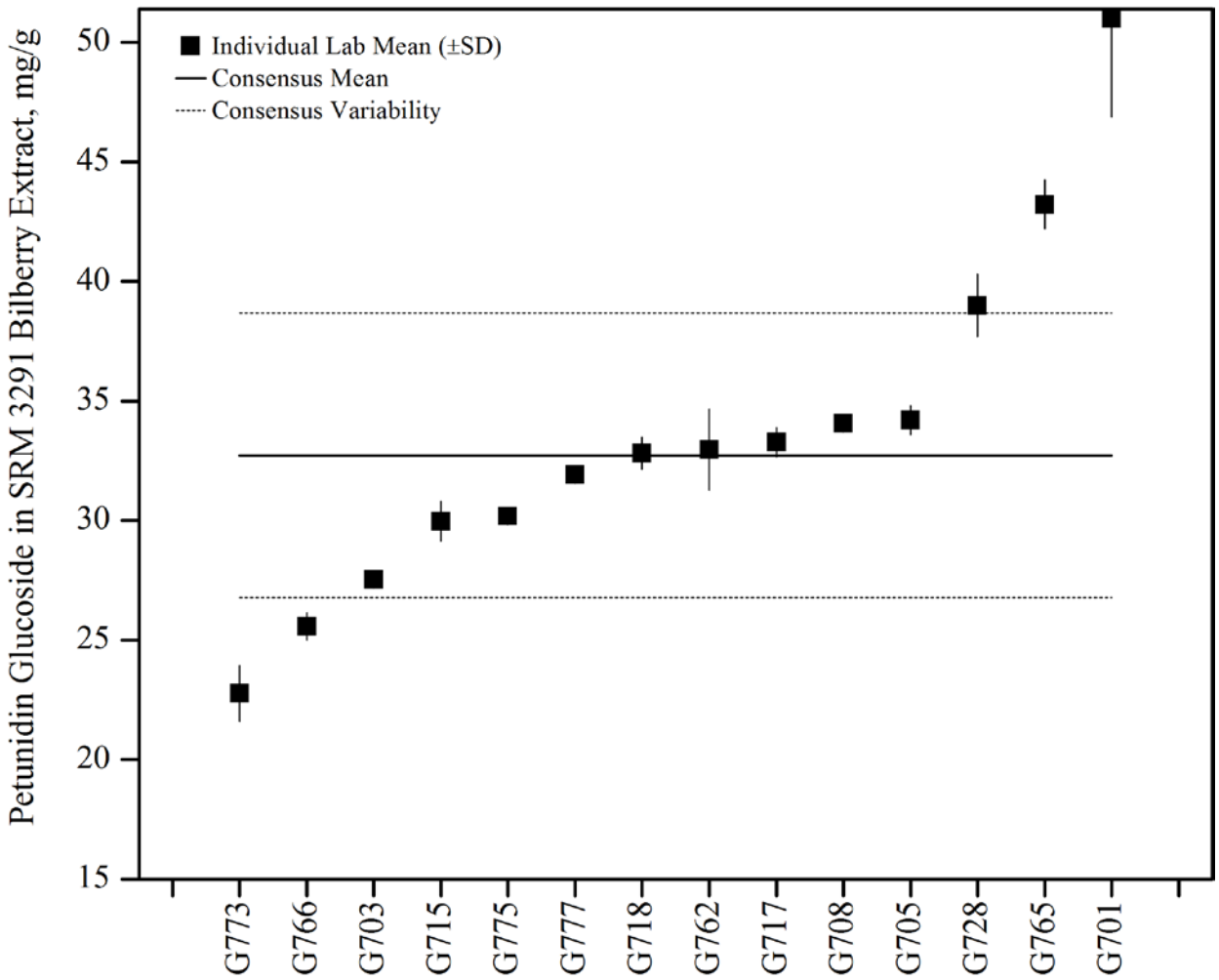

Figure 66. Petunidin-3-glucoside in SRM 3291 Bilberry Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

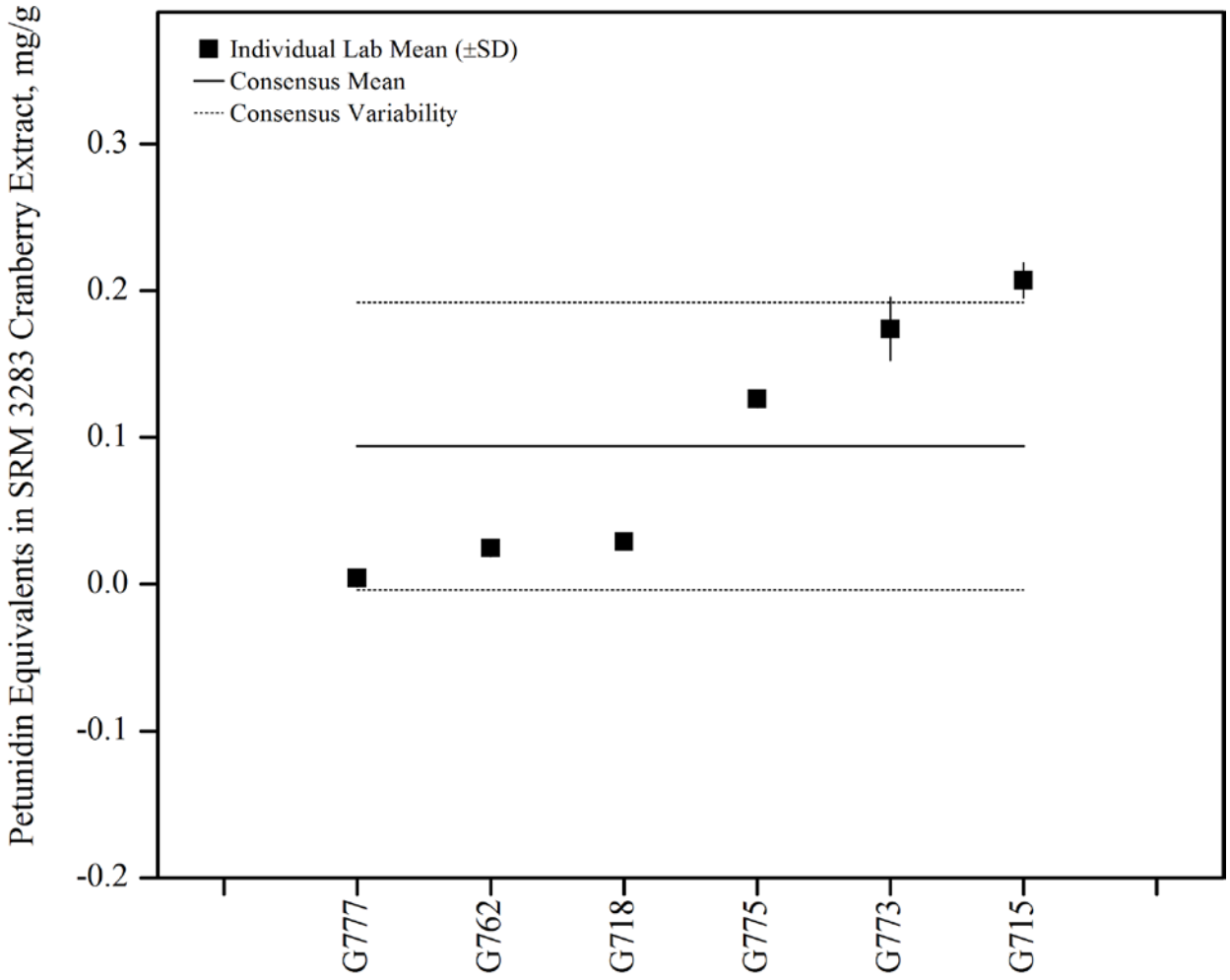

Figure 67. Petunidin equivalents in SRM 3283 Cranberry Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

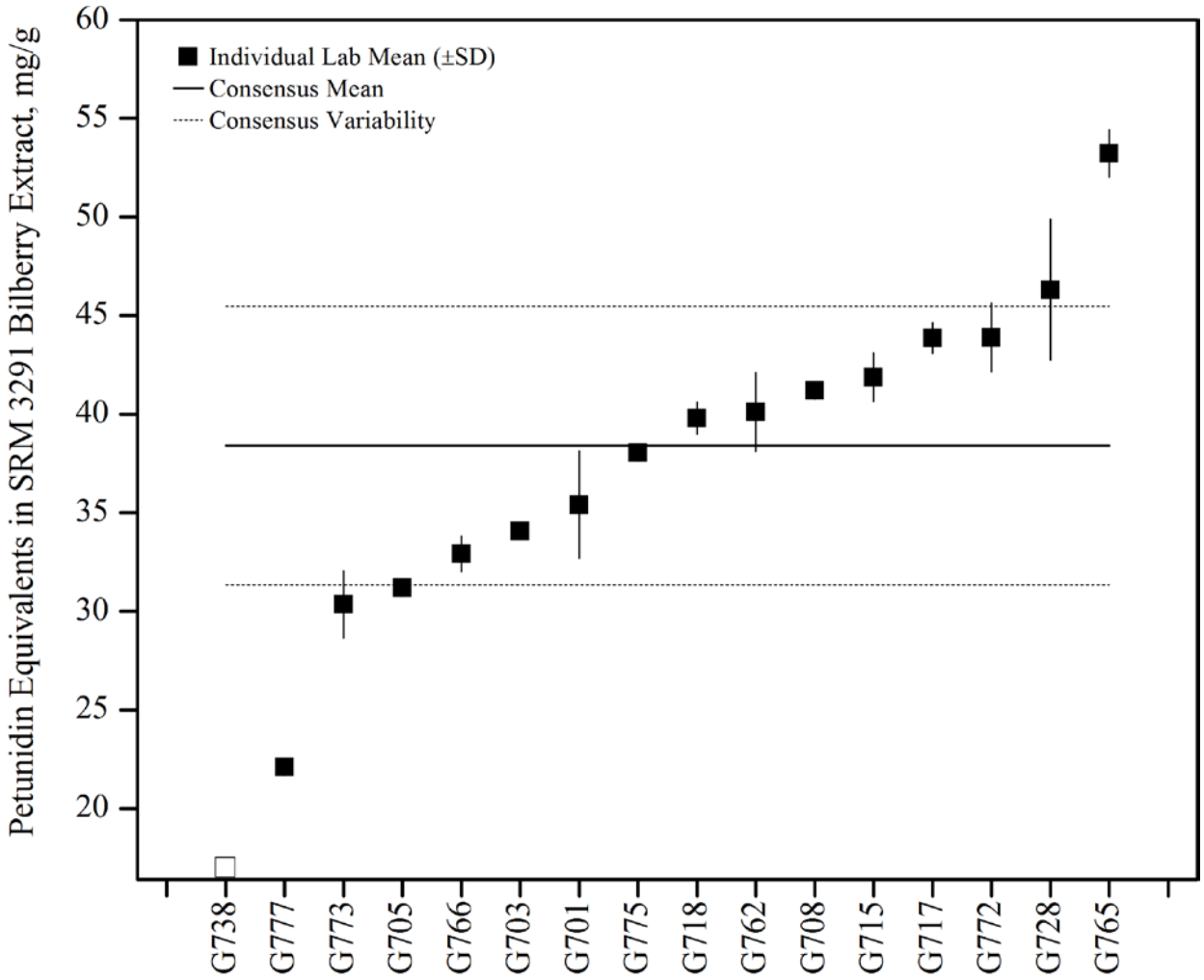

Figure 68. Petunidin equivalents in SRM 3291 Bilberry Extract (data summary view). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

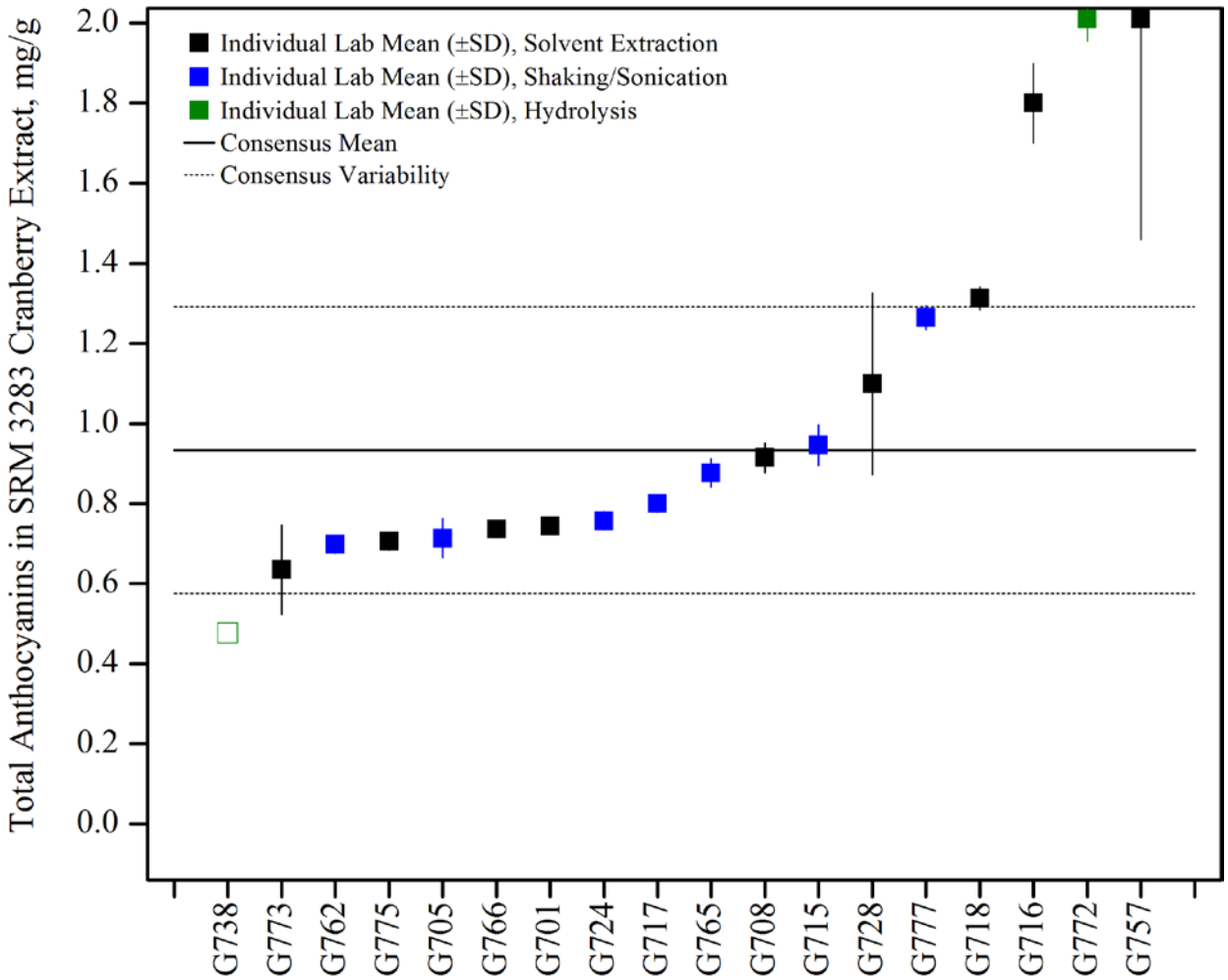

Figure 69. Total anthocyanins in SRM 3283 Cranberry Extract (data summary view – sample preparation method). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The data are identified by sample preparation method in this graph. The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

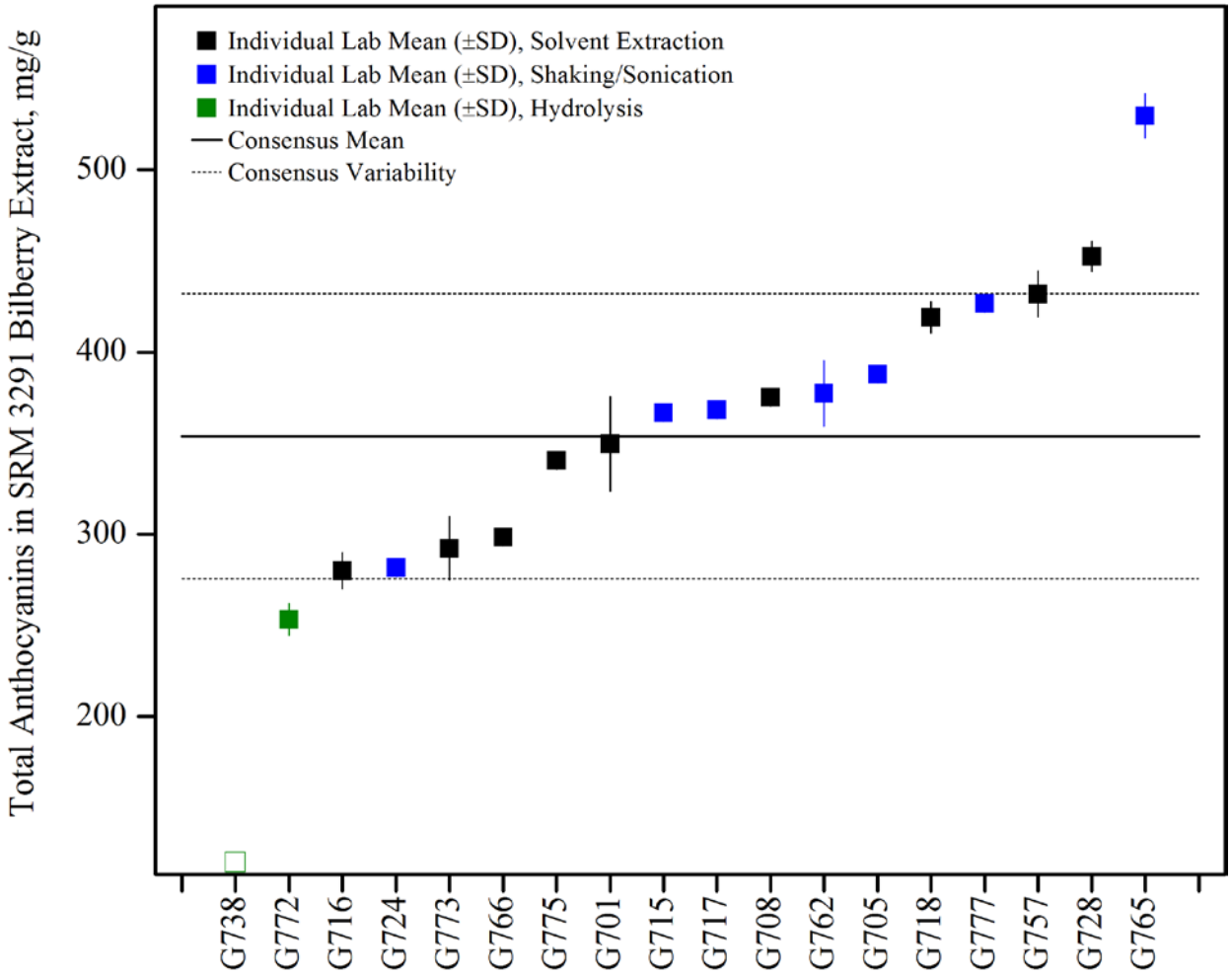

Figure 70. Total anthocyanins in SRM 3291 Bilberry Extract (data summary view – sample preparation method). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The data are identified by sample preparation method in this graph. The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

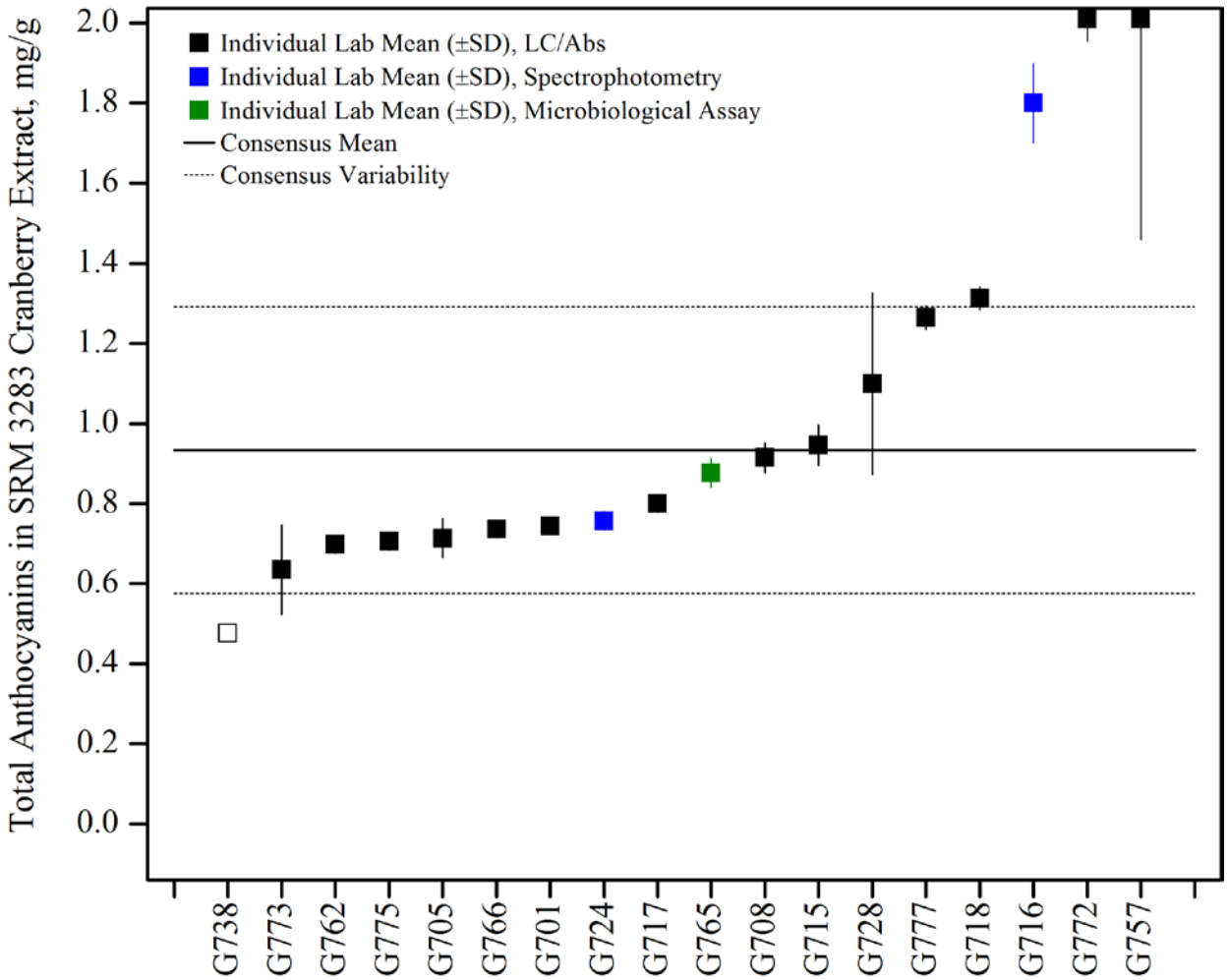

Figure 71. Total anthocyanins in SRM 3283 Cranberry Extract (data summary view – instrumental method). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The data are identified by instrumental method in this graph. The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

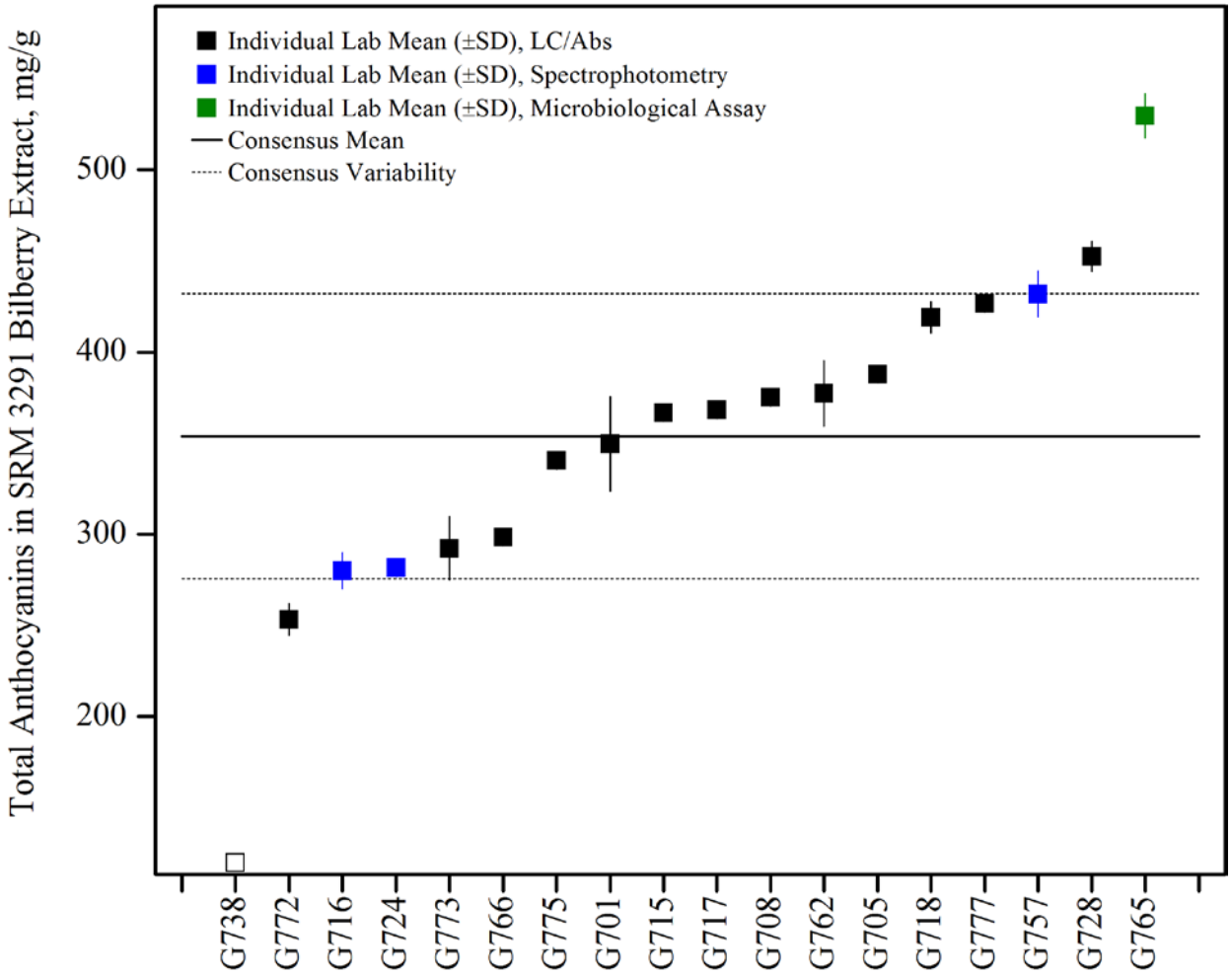

Figure 72. Total anthocyanins in SRM 3291 Bilberry Extract (data summary view – instrumental method). In this view, individual laboratory data are plotted with the individual laboratory standard deviation (error bars). The data are identified by instrumental method in this graph. The black solid line represents the consensus mean, and the black dotted lines represent the consensus variability calculated as one standard deviation about the consensus mean.

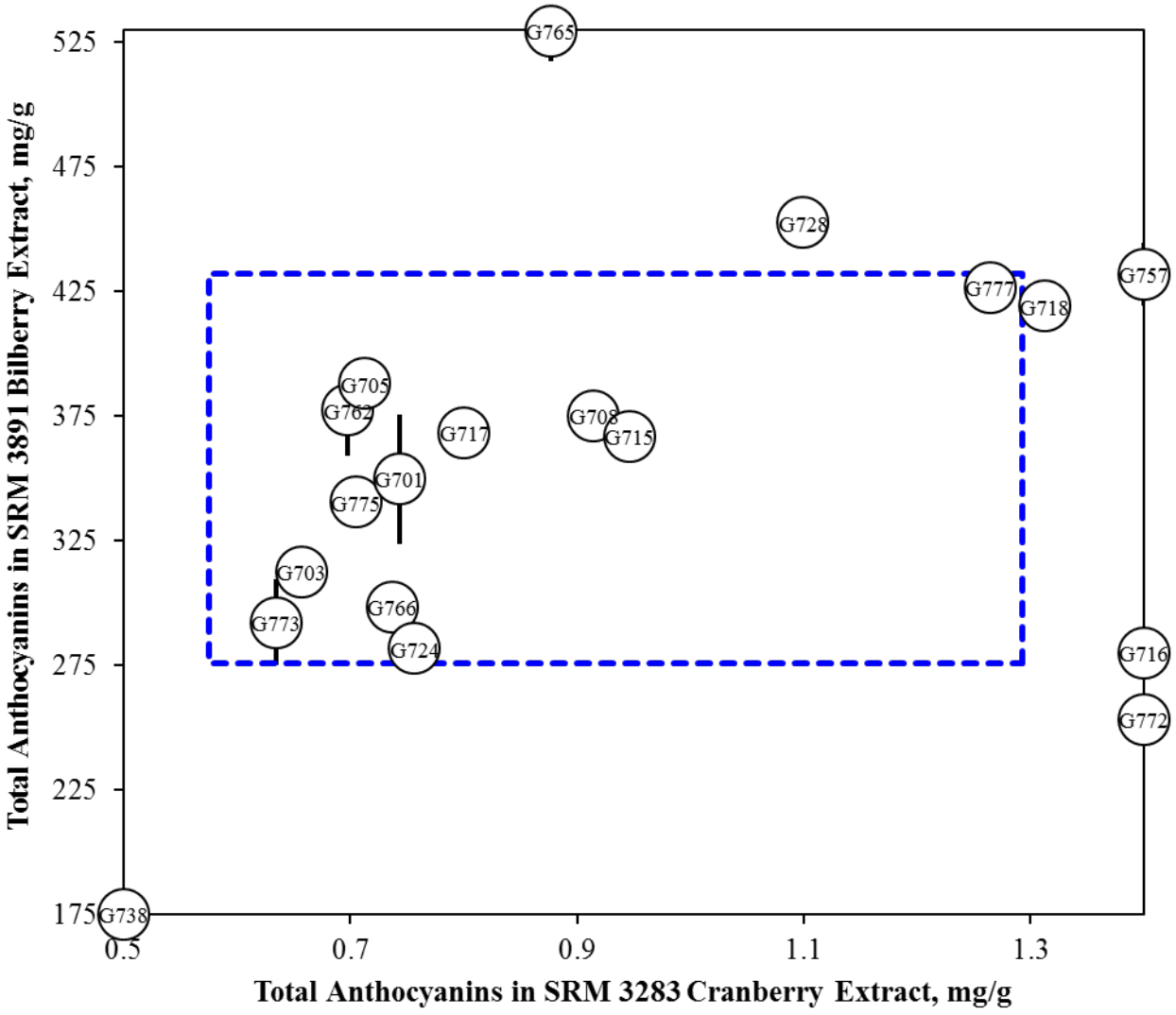

Figure 73. Total anthocyanins in SRM 3283 Cranberry Extract and SRM 3291 Bilberry Extract (sample/sample comparison view). In this view, the individual laboratory results for one sample (SRM 3283 Cranberry Extract) are compared to the results for a second sample (SRM 3291 Bilberry Extract). The dotted blue box represents the consensus zone for the cranberry extract (x-axis) and the bilberry extract (y-axis).