**NISTIR 8266**

# Dietary Supplement Laboratory Quality Assurance Program: Exercise O Final Report

Charles A. Barber
Melissa M. Phillips
Catherine A. Rimmer
Laura J. Wood
Maria R. Ale
Stephen E. Long
Elizabeth Mudge
Shannon L. Whitehead

This publication is available free of charge from:
https://doi.org/10.6028/NIST.IR.8266

National Institute of Standards and Technology
U.S. Department of Commerce

**NISTIR 8266**

# Dietary Supplement Laboratory Quality Assurance Program: Exercise O Final Report

Charles A. Barber
Melissa M. Phillips
Catherine A. Rimmer
Laura J. Wood
Maria R. Ale
Stephen E. Long
Elizabeth Mudge
Shannon L. Whitehead
*Chemical Sciences Division*
*Material Measurement Laboratory*

This publication is available free of charge from:
https://doi.org/10.6028/NIST.IR.8266

September 2019


U.S. Department of Commerce
*Wilbur L. Ross, Jr., Secretary*

National Institute of Standards and Technology
*Walter Copan, NIST Director and Under Secretary of Commerce for Standards and Technology*

**National Institute of Standards and Technology Interagency or Internal Report 8266**
**Natl. Inst. Stand. Technol. Interag. Intern. Rep. 8266, 151 pages (September 2019)**

**This publication is available free of charge from:**
**https://doi.org/10.6028/NIST.IR.8266**

**TABLE OF CONTENTS**

## 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 supplement research and industry communities to improve the accuracy of their measurements and for demonstration of compliance with various regulations, including the dietary supplement current good manufacturing practices (cGMPs). Exercise O of this program offered the opportunity for laboratories to assess their in-house measurements of contaminants (arsenic, cadmium, lead, mercury), marker compounds in botanicals (curcuminoids) and natural products (chondroitin sulfate), and authenticity of *Ginkgo biloba* materials in botanical dietary supplement ingredients and finished products.

## INTRODUCTION

The dietary supplement industry in the US is booming, with over 75 % of adults considering themselves to be supplement users.[1] Sales of dietary supplements, which includes vitamin and mineral supplements, are estimated at annual U.S. expenditure of more than $35 billion. These figures represent a trend, in America and worldwide, of increasing supplement consumption, and as a result, the verification and maintenance of both the quality and safety of these products is critically important.

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 supplement community to improve the accuracy of the measurements required for compliance with these and other regulations, NIST established the Dietary Supplements Laboratory Quality Assurance Program (DSQAP) in collaboration with the NIH ODS in 2007.

The program offered 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.

NIST has experience in the administration of multiple 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 and improve laboratory performance. In contrast, the wide range of matrices and analytes under the “dietary supplements” umbrella means that not every laboratory is interested in every sample or analyte. The constantly changing dietary supplement market, and the enormous diversity of finished products, makes

[1] *2018 CRN Consumer Survey on Dietary Supplements*. Council for Responsible Nutrition, Washington, DC; accessed https://www.crnusa.org/CRNConsumerSurvey (August 2019).

repeated determination 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 and that their methods provide accurate results. In an area where there are few generally accepted 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. In the future, the Health Assessment Measurements Quality Assurance Program (HAMQAP) that was formed in 2017, in part as a collaboration with the NIH ODS, will represent the ongoing efforts at NIST that were supported previously via historical quality assurance programs (QAPs), including DSQAP, Micronutrients Measurement QAP (MMQAP), Fatty Acids in Human Serum QAP (FAQAP), and Vitamin D Metabolites QAP (VitDQAP).

This report summarizes the results from the fifteenth and final exercise of the DSQAP, Exercise O. Sixty-four laboratories responded to the call for participants distributed in September 2017. The first set of samples, which included only half of the commercial turmeric samples, were shipped to participants in December 2017 and results were returned to NIST by February 2018. Given the limited number of data sets that were received from laboratories using AOAC First Action *Official Method of Analysis 2016.16 Determination of Curcuminoids in Turmeric Raw Materials and Dietary Supplements by HPLC*, controls as well as the alternate four commercial turmeric samples were shipped to participants in July 2018 and results were returned to NIST by August 2018. This report contains the final data and information that was disseminated to the participants in August 2019.

## 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. All calculations are performed in PROLab Plus (QuoData GmbH, Dresden, Germany).[2] The consensus mean and standard deviation are calculated according to the robust algorithm outlined in ISO 13528:2015(E), Annex C.[3] The algorithm is summarized here in simplified form.

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

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

[2] Certain commercial equipment, instruments or materials are identified in this certificate to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

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

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

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

Each $x_i$ is then 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 $x_i^* = x^* + \delta$.
Otherwise, $x_i^* = x_i$.

New values of $x^*$, $s^*$, and $\delta$ 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, when available). The upper left of the data table includes the randomized laboratory code. Example individualized data tables are included in this report; participating laboratories received uniquely coded individualized data tables in a separate distribution.

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

Also included in Section 1 are two Z-scores. The first Z-score, $Z'_{comm}$, is calculated with respect to the community consensus value, taking into consideration bias that may result from the uncertainty in the assigned consensus value, using x* and s*:

$$Z'_{comm} = \frac{x_i - x*}{\sqrt{2}s*}.$$

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

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

or

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

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

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

Section 2 of the data table (*Community Results*) contains the consensus results, including the number of laboratories reporting more than a single quantitative value for a given analyte, the mean value determined for each analyte, and a robust estimate of the standard deviation of the reported values.[3] Consensus means and standard deviations are calculated using the laboratory means; if a laboratory reported a single value, the reported value is not included.[3] Additional information on calculation of the consensus mean and standard deviation can be found in the previous section.

Section 3 of the data table (*Target*) contains the target values for each analyte, when available. When possible, the target value is a certified value, a reference value, or a value determined at NIST. Certified values and the associated expanded uncertainty ($U_{95}$) have been determined with two independent analytical methods at NIST, or by combination of a single method at NIST and results from collaborating laboratories. Reference values are assigned using NIST values obtained from the average and standard deviation of measurements made using a single analytical method at NIST or by measurements obtained from collaborating laboratories. For both certified and reference values, at least six samples have been tested and duplicate preparations from the sample package have been included, allowing the uncertainty to encompass variability due to inhomogeneity within and between packages. For samples in which a NIST certified or reference value is not available, the analytes may be measured at NIST using a validated method or data from a partner laboratory may be used to establish a NIST-assessed value. The NIST-assessed value represents the mean of at least three replicates. For materials acquired from another interlaboratory study or proficiency testing program, the consensus value and uncertainty from the completed round is used as the target range. Within each section of this report, the exact methods for determination of the study target values are outlined in detail.

Summary Data Table

This data table includes a summary of all reported data for a particular analyte in a particular study. Participants can compare the raw data for their laboratory to data reported by the other participating laboratories and to the consensus data. A blank indicates that the laboratory signed up and received samples for that particular analyte and matrix, but NIST does not have data on file for that laboratory. Data points highlighted in red have been flagged as potential outliers (e.g., Grubb

and/or Cochran) by the NIST software package. The standard deviation (SD) for the target value in this table is the uncertainty ($U_{NIST}$) around the target value.

Graphs

*Data Summary View (Method Comparison Data Summary View)*

In this view, individual laboratory data (diamonds) are plotted with the individual laboratory standard deviation (rectangles). Laboratories reporting values below the method quantitation limit are shown in this view as downward triangles beginning at the limit of quantitation (LOQ), reported as quantitation limit (QL) on the figures. Laboratories reporting values as "below LOQ" can still be successful in the study if the target value is also below the laboratory LOQ. The blue solid line represents the consensus mean, and the green shaded area represents the 95 % confidence interval for the consensus mean, based on the standard error of the consensus mean. The red shaded region represents the target zone for "acceptable" performance, which encompasses the NIST target value bounded by twice its uncertainty ($U_{95}$ or $U_{NIST}$). The solid red lines represent the range of tolerance (values that result in an acceptable $Z'$-score, $|Z'| \leq 2$). If the lower limit is below zero, the lower limit has been set to zero. In this view, the relative locations of individual laboratory data and consensus zones with respect to the target zone can be compared easily. In most cases, the target zone and the consensus zone overlap, which is the expected result. The major program goals are to reduce the size of the consensus zone and center the consensus zone about the target value. Analysis of an appropriate reference material as part of a quality control scheme can help to identify sources of bias for laboratories reporting results that are significantly different from the target zone. In the case in which a method comparison is relevant, different colored data points may be used to indicate laboratories that used a specific approach to sample preparation, analysis, or quantitation.

*Sample/Sample Comparison View*

In this view, the individual laboratory results for one sample (NIST SRM with a certified, reference, or NIST-determined value) are compared to the results for another sample (another NIST SRM with a more challenging matrix, a commercial sample, etc.). The solid red box represents the target zone for the first sample (x-axis) and the second sample (y-axis). The dotted blue box represents the consensus zone for the first sample (x-axis) and the second sample (y-axis). The axes of this graph are centered about the consensus mean values for each sample or control, to a limit of twice the range of tolerance (values that result in an acceptable $Z'$-score, $|Z'| \leq 2$). Depending on the variability in the data, the axes may be scaled proportionally to better display the individual data points for each laboratory. In some cases, when the consensus and target ranges have limited overlap, the solid red box may only appear partially on the graph. If the variability in the data is high (greater than 100 % relative standard deviation, or RSD), the dotted blue box may also only appear partially on the graph. These views emphasize trends in the data that may indicate potential calibration issues or method biases. One program goal is to identify such calibration or method biases and assist participants in improving analytical measurement capabilities. In some cases, when two equally challenging materials are provided, the same view (sample/sample comparison) can be helpful in identifying commonalities or differences in the analysis of the two materials.

## SECTION 1: TOXIC ELEMENTS (As, Cd, Pb, Hg) IN BLACK COHOSH AND TURMERIC DIETARY SUPPLEMENTS

Study Overview

In this study, participants were provided with samples of black cohosh rhizome and turmeric rhizome and were asked to use in-house analytical methods to determine the mass fractions (ng/g) of As, Cd, Pb, and Hg in each matrix. Black cohosh and turmeric are popular dietary supplements used to alleviate menopausal symptoms[4] and reduce inflammation[5]. In the United States, cGMPs require dietary supplement manufacturers to establish limits on reasonably anticipated contaminants, therefore laboratories must establish scientifically valid methods for the determination of toxic elements to demonstrate the products meet their specifications in 21 CFR 111.70(b)(3). Monitoring toxic substances in foods and dietary supplements helps prevent exposure to consumers and reduces the risk of related negative health outcomes.

Sample Information

*Black Cohosh Rhizome.* Participants were provided with one packet containing 3 g of black cohosh rhizome powder. Before use, participants were instructed to mix the contents of the packet thoroughly, and to 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. The approximate analyte levels were not reported to participants prior to the study. The target values for As, Cd, and Pb were determined at NIST using inductively coupled plasma mass spectroscopy (ICP-MS). The target value for Hg was determined at NIST using cold-vapor inductively coupled plasma mass spectrometry (CV ICP-MS). The NIST-determined values and uncertainties for toxic elements in black cohosh rhizome are provided in the table below.

| Analyte | NIST-Determined Mass Fractions in Black Cohosh Rhizome (ng/g) |
|---|---|
| Arsenic (As) | 300 ± 20 |
| Cadmium (Cd) | 243 ± 8 |
| Lead (Pb) | 2236 ± 46 |
| Mercury (Hg) | 12.8 ± 0.1 |

*Turmeric Rhizome.* Participants were provided with one packet containing 3 g of turmeric rhizome powder. Before use, participants were instructed to mix the contents of the packet thoroughly, and to 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. The approximate analyte levels were not reported to participants prior to the study. The target values for As, Cd, and Pb were determined at NIST using ICP-MS. The target value for Hg was determined at NIST using CV ICP-MS. The NIST-determined values and uncertainties for toxic elements in turmeric rhizome are provided in the table below.

[4] *Black Cohosh: Fact Sheet for Health Professionals.* https://ods.od.nih.gov/factsheets/BlackCohosh-HealthProfessional/ (accessed August 2019).
[5] *Turmeric*. https://nccih.nih.gov/health/turmeric/ataglance.htm (accessed August 2019).

| Analyte | NIST-Determined Mass Fractions in Turmeric Rhizome (ng/g) |
|---|---|
| Arsenic (As) | 323 ± 23 |
| Cadmium (Cd) | 1700 ± 160 |
| Lead (Pb) | 1143 ± 38 |
| Mercury (Hg) | 54.1 ± 3.7 |



Study Results

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

| Analyte | Number of Laboratories Requesting Samples | Number of Laboratories Reporting Results (Percent Participation) | |
|---|---|---|---|
| | | Black Cohosh Rhizome | Turmeric Rhizome |
| Arsenic (As) | 37 | 27 (73 %) | 27 (73 %) |
| Cadmium (Cd) | 39 | 28 (72 %) | 28 (72 %) |
| Lead (Pb) | 39 | 28 (72 %) | 28 (72 %) |
| Mercury (Hg) | 38 | 23 (61 %) | 27 (71 %) |

- The consensus means for As and Pb in the black cohosh rhizome and for Pb in the turmeric rhizome were below the target ranges with no overlap of the target range and the consensus range.
- The consensus means for Hg in both black cohosh rhizome and turmeric rhizome were above the target ranges with no overlap of the target range and the consensus range.
- The target range and the consensus range for As and Cd in the turmeric rhizome and for Cd in the black cohosh rhizome did overlap.
- The between-laboratory variabilities were all reasonable and are reported below.

| Analyte | Between-Laboratory Variability (Percent RSD) | |
|---|---|---|
| | Black Cohosh Rhizome | Turmeric Rhizome |
| Arsenic (As) | 24 % | 19 % |
| Cadmium (Cd) | 16 % | 21 % |
| Lead (Pb) | 17 % | 18 % |
| Mercury (Hg) | 37 % | 30 % |

- Most laboratories reported using ICP-MS (90 % to 93 %) as their analytical method for all analytes. One laboratory reported using atomic absorption spectroscopy (AAS), and another laboratory did not specify a method used.

- The sample preparation methods reported by participating laboratories are summarized in the table below. Most laboratories reported using microwave digestion for all four analytes.

| Reported Method | As | Cd | Pb | Hg |
|---|---|---|---|---|
| Microwave digestion | 70 % | 75 % | 68 % | 78 % |
| Hot Block digestion | 22 % | 18 % | 24 % | 11 % |
| Open beaker digestion | 7 % | 7 % | 8 % | 11 % |

Technical Recommendations

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

- For all analytes, no pattern or trend was observed between reported results and analytical methods or sample preparation methods used.
- Sample preparation methods should be well established before analyzing unknown samples. Established quality control materials (SRMs, CRMs, RMs, and in-house materials) and accepted methods of analysis can assist in this process.
- Detection of the analyte in the sample may be improved by limiting the number of dilutions performed, however matrix effects may become more significant. A matrix-matched calibration curve may reduce some matrix interferences.
- For arsenic, the majority of the laboratories reported data below the NIST target range for the black cohosh rhizome and less than half of the laboratories reported data below the NIST target range for As in the turmeric rhizome, as shown in **Figures 1-1 through 1-4**.
  - Arsenic is volatile and can be lost during sample preparation, resulting in data that is biased low as seen in **Figure 1-5**.
    - The high temperatures of a vigorous microwave digestion should convert all volatile organoarsenic species to arsenic acid (AsV), at which point subsequent heating will not result in loss of arsenic.
    - The use of an open-beaker digestion may cause loss of As during sample preparation. Closed-vessel digestions should be opened with care ensuring that no As is lost as a result of inadvertent venting.
  - **Figure 1-5** shows that more laboratories had difficulty measuring As in the black cohosh rhizome than in the turmeric rhizome. The black cohosh material may require a more rigorous sample preparation than the turmeric material, or arsenic may be lost from volatilization.
  - Higher temperatures or the use of a small amount of HF may be needed to ensure complete digestion of plant materials for analysis.
- The boiling point of Cd is high and volatile loss of Cd is not a concern, but Cd can be difficult to measure by ICP-MS due to spectral interferences or by ICP-OES due to low sensitivity.
  - As seen in **Figure 1-10**, approximately half of the laboratories fell within the target range for both the black cohosh rhizome and the turmeric rhizome indicating Cd in these materials may have been less difficult to analyze than As.
  - Some laboratories that reported low values for Cd in one material also reported low values for Cd in the second material, but laboratories reporting high values for Cd in the black cohosh did not always report high values for Cd in turmeric.
    - For laboratories reporting low values for both samples there could be a possible calibration issue or incomplete sample digestion.

- For laboratories reporting high values for black cohosh only, challenges in the sample preparation could cause suppression or enhancement of the Cd signal.

- For ICP-MS, the most used method for Cd, the presence of high concentrations of certain elements, mainly Mo, Sn, or Zr, can cause interferences in the measurement of Cd. A scan of the sample beforehand will identify potential interferences in the sample that will need to be addressed.
  - Commonly used masses of Cd ($^{111}$Cd, $^{112}$Cd, $^{113}$Cd, and$^{114}$Cd) can have molecular interferences such as $^{95, 96, 97 \text{ and } 98}Mo^{16}O^{+}$, $^{94, 95, 96, \text{ and } 97}Mo^{16}O^{1}H^{+}$, $^{96}Zr^{16}O^{+}$, $^{94 \text{ and } 96}Zr^{16}O^{1}H^{+}$, $^{40}Ar_2{}^{16}O_2$, $^{40}Ca_2{}^{16}O_2$, or $^{40}Ca_2{}^{16}O_2{}^{1}H^{+}$ as well as elemental isobaric interferences such as $^{112}$Sn, $^{113}$In, and $^{114}$Sn. Interferences can cause signal suppression or signal enhancement.
  - Chemical separations by anion chromatography can reduce or remove interferences but are usually impractical for laboratories due to the labor-intensive work required.
  - Collision cell technology, available on most ICP-MS instruments, can be used to remove many of the molecular interferences that may be found in these two materials.
  - Interference equations inherent to the software provided on some ICP-MS instruments are designed to correct for interferences, and these equations can also be applied off-line. Both are less labor-intensive alternatives to chemical separations.

- Lead is easily digested and volatile loss of Pb is not a concern. However, digestion with $HNO_3$ is recommended since use of HCl may form a highly insoluble $PbCl_2$ precipitate. Dry ashing with a small volume of acid is another recommended technique, though this technique can be time-consuming.
  - Since both sample materials contained high levels of lead, as shown in **Figures 1-11 through 1-14**, the consensus value for both rhizomes should easily have fallen within the NIST target range, providing HCl was not used for digestion. Since the consensus values did not overlap the NIST target ranges, a calibration problem is suspected (**Figure 1-15**).
  - Only two laboratories overlapped the NIST target range for lead in black cohosh and most did not fall within the consensus range. The laboratories performed better when reporting results for lead in turmeric.
  - The concentrations of lead are high in these samples and when analyzed by ICP-MS, larger dilutions may be necessary for improved accuracy.
  - Calibration curves must be checked before sample analysis to ensure that expected sample values will fall between the lowest and highest calibration points and that the calibration curve is linear at the point where the sample values fall. A calibration curve using calibration standards of (0, 1, 10, and 100) ng/kg may appear to give a linear curve but for sample values near the 1 ng/kg range, the calibration curve may no longer be linear when using only the lower calibration standards. In this case the final Pb values will be wrong and can be either too high or too low.
- Mercury is volatile, so care must be taken to not lose Hg during sample preparation.
  - Microwave digestion is the best method for sample preparation.
  - Low concentrations of Hg are not stable in solution over time. Samples are best prepared close to the time of analysis. Samples containing low concentrations of Hg may be more stable in dilute HCl than in dilute $HNO_3$.
  - Mercury levels are very low in the black cohosh rhizome and may be close to method detection limits (MDL) in both materials. A sufficient number of blanks are required to

determine an accurate MDL and LOQ. Mercury blanks and backgrounds may be large, making determination of Hg values in samples containing low levels of Hg difficult.
    - Mercury has a poor washout (long memory effect) and can give erratic answers if an adequate washout time is not used after each measurement.
    - Values reported at the higher end of the range had more within-laboratory variability, most likely due to contamination issues or problems with sample analysis such as memory effects. Use of dilute HCl may decrease the length of necessary washout time.
    - The sensitivity of ICP-MS is low for Hg. Using cold vapor mercury generation increases sensitivity allowing for lower levels of Hg to be measured.
- To summarize, measurement of toxic elements in plant materials is challenging for most laboratories.
    - An appropriate quality control material is needed and is one that will mirror both the sample matrix and the mass fraction levels expected to be found in the sample.
    - For complete digestion of plant materials, the use of a small amount of HF may be necessary even if particulates are not visible.
    - Calibration curves must be linear for all analytes, including the lowest and highest values expected to be measured in the samples. Extrapolation of the curve may cause incorrect results.
    - Analysis of an appropriate number of procedural blanks is important and can be critical when sample concentrations are near the detection limit.
    - All results should be checked closely to avoid calculation errors and to be sure that results are reported in the requested units.
    - For both rhizomes, a few laboratories reported data significantly outside of the target and consensus ranges. Calculation errors are often a cause for incorrect results. Using a quality assurance material (CRM, SRM, RM) or in-house prepared material to establish that a method is in control will also help find calculation errors. Once a method and quality assurance material appear to be in control, be sure results are reported in the correct units.

**Table 1-1.** NIST data summary table for arsenic, cadmium, mercury, and lead in black cohosh and turmeric rhizomes.

## *National Institute of Standards and Technology*

**DSQAP Exercise O - Toxic Elements**

| | | **Lab Code:** | **NIST** | **1. Your Results** | | | | **2. Community Results** | | | **3. Target** | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Analyte | Sample | Units | | $x_i$ | $s_i$ | $Z'_{comm}$ | $Z_{NIST}$ | N | x* | s* | $x_{NIST}$ | *U* |
| Total arsenic | Black Cohosh Rhizome | ng/g | | 300 | 20 | | | 29 | 240 | 12 | 300 | 20 |
| Total arsenic | (A) SRM 3299 Turmeric Rhizome | ng/g | | 320 | 23 | | | 29 | 290 | 10 | 323 | 23 |
| Cadmium | Black Cohosh Rhizome | ng/g | | 240 | 8 | | | 30 | 230 | 6.9 | 243 | 8 |
| Cadmium | (A) SRM 3299 Turmeric Rhizome | ng/g | | 1700 | 160 | | | 30 | 1430 | 55 | 1700 | 160 |
| Mercury | Black Cohosh Rhizome | ng/g | | 12.8 | 0.1 | | | 25 | 15.3 | 1.1 | 12.8 | 0.1 |
| Mercury | (A) SRM 3299 Turmeric Rhizome | ng/g | | 54.1 | 3.7 | | | 30 | 69.9 | 3.6 | 54.1 | 3.7 |
| Lead | Black Cohosh Rhizome | ng/g | | 2240 | 46 | | | 30 | 1800 | 53 | 2240 | 46 |
| Lead | (A) SRM 3299 Turmeric Rhizome | ng/g | | 1140 | 36 | | | 30 | 1000 | 32 | 1140 | 36 |

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

**Table 1-2.** Data summary table for total arsenic in black cohosh and turmeric rhizomes. Data points highlighted in red have been flagged as potential outliers (e.g., Grubb and/or Cochran) by the NIST software package.

| | | Total Arsenic | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | SRM 3295 Black Cohosh Rhizome (ng/g) | | | | | SRM 3299 Turmeric Rhizome (ng/g) | | | | |
| | Lab | A | B | C | Avg | SD | A | B | C | Avg | SD |
| Individual Results | NIST | | | | 300 | 20 | | | | 323 | 23 |
| | O405 | | | | | | | | | | |
| | O407 | 238 | 241 | 236 | 238 | 3 | 268 | 254 | 308 | 277 | 28 |
| | O408 | 374 | 370 | 388 | 377 | 9 | 341 | 345 | 337 | 341 | 4 |
| | O409 | | | | | | | | | | |
| | O411 | 229 | 220 | 212 | 220 | 9 | 216 | 267 | 209 | 231 | 32 |
| | O412 | | | | | | | | | | |
| | O413 | 240 | 240 | 240 | 240 | 0 | 340 | 310 | 320 | 323 | 15 |
| | O414 | 330 | | | 330 | | 270 | | | 270 | |
| | O416 | 246 | 240 | 233 | 240 | 6 | 346 | 322 | 318 | 329 | 15 |
| | O417 | 267 | 266 | 275 | 269 | 5 | 320 | 304 | 303 | 309 | 10 |
| | O418 | 223 | 236 | 232 | 230 | 7 | 259 | 262 | 258 | 260 | 2 |
| | O419 | 199 | 195 | 200 | 198 | 3 | 224 | 242 | 226 | 231 | 10 |
| | O420 | 265 | 250 | 251 | 255 | 8 | 285 | 306 | 336 | 309 | 26 |
| | O423 | | | | | | | | | | |
| | O425 | 340 | 340 | 320 | 333 | 12 | 410 | 360 | 390 | 387 | 25 |
| | O427 | 269 | 275 | 256 | 267 | 10 | 295 | 332 | 289 | 305 | 23 |
| | O428 | 312 | 314 | 325 | 317 | 7 | 325 | 335 | 327 | 329 | 5 |
| | O429 | | | | | | | | | | |
| | O430 | 201 | 208 | 202 | 204 | 4 | 224 | 205 | 204 | 211 | 11 |
| | O431 | 259 | 267 | 266 | 264 | 4 | 268 | 268 | 261 | 266 | 4 |
| | O433 | 156 | 122 | 109 | 129 | 24 | 141 | 280 | 135 | 186 | 82 |
| | O434 | | | | | | | | | | |
| | O437 | | | | | | | | | | |
| | O440 | 294 | 246 | 251 | 264 | 26 | 284 | 301 | 288 | 291 | 9 |
| | O441 | 221 | 206 | 219 | 215 | 8 | 288 | 280 | 258 | 275 | 15 |
| | O442 | 267 | 250 | 265 | 261 | 9 | 271 | 392 | 469 | 377 | 100 |
| | O445 | 264 | 270 | 257 | 264 | 7 | 281 | 293 | 289 | 288 | 6 |
| | O447 | | | | | | | | | | |
| | O449 | 173 | 172 | 177 | 174 | 3 | 255 | 306 | 260 | 273 | 28 |
| | O452 | 0.280 | 0.290 | 0.390 | 0.320 | 0.061 | 0.300 | 0.300 | 0.320 | 0.307 | 0.012 |
| | O454 | 233 | 240 | 261 | 245 | 14 | 247 | 248 | 246 | 247 | 1 |
| | O455 | 192 | 203 | 194 | 196 | 6 | 345 | 311 | 313 | 323 | 19 |
| | O457 | 200 | 200 | 210 | 203 | 6 | 280 | 270 | 290 | 280 | 10 |
| | O458 | | | | | | | | | | |
| | O462 | 190 | 180 | 200 | 190 | 10 | 320 | 280 | 250 | 283 | 35 |
| | O463 | 204 | 215 | 216 | 212 | 7 | 229 | 247 | 235 | 237 | 9 |
| | O464 | 139 | 92 | 72 | 101 | 34 | 934 | 920 | 961 | 938 | 21 |
| Community Results | | Consensus Mean | | | 235 | | Consensus Mean | | | 286 | |
| | | Consensus Standard Deviation | | | 57 | | Consensus Standard Deviation | | | 55 | |
| | | Maximum | | | 377 | | Maximum | | | 938 | |
| | | Minimum | | | 0.320 | | Minimum | | | 0.307 | |
| | | N | | | 27 | | N | | | 27 | |

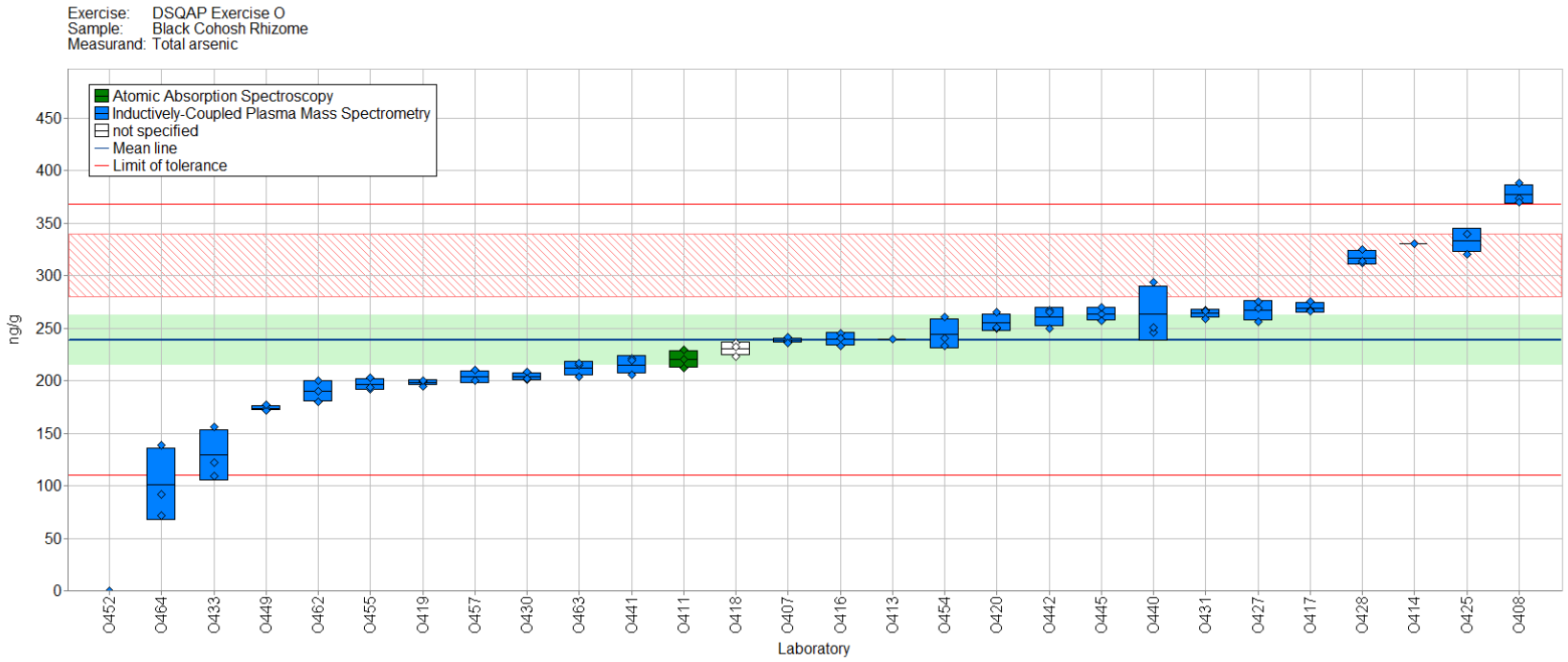


**Figure 1-1.** Total arsenic in black cohosh rhizome (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid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

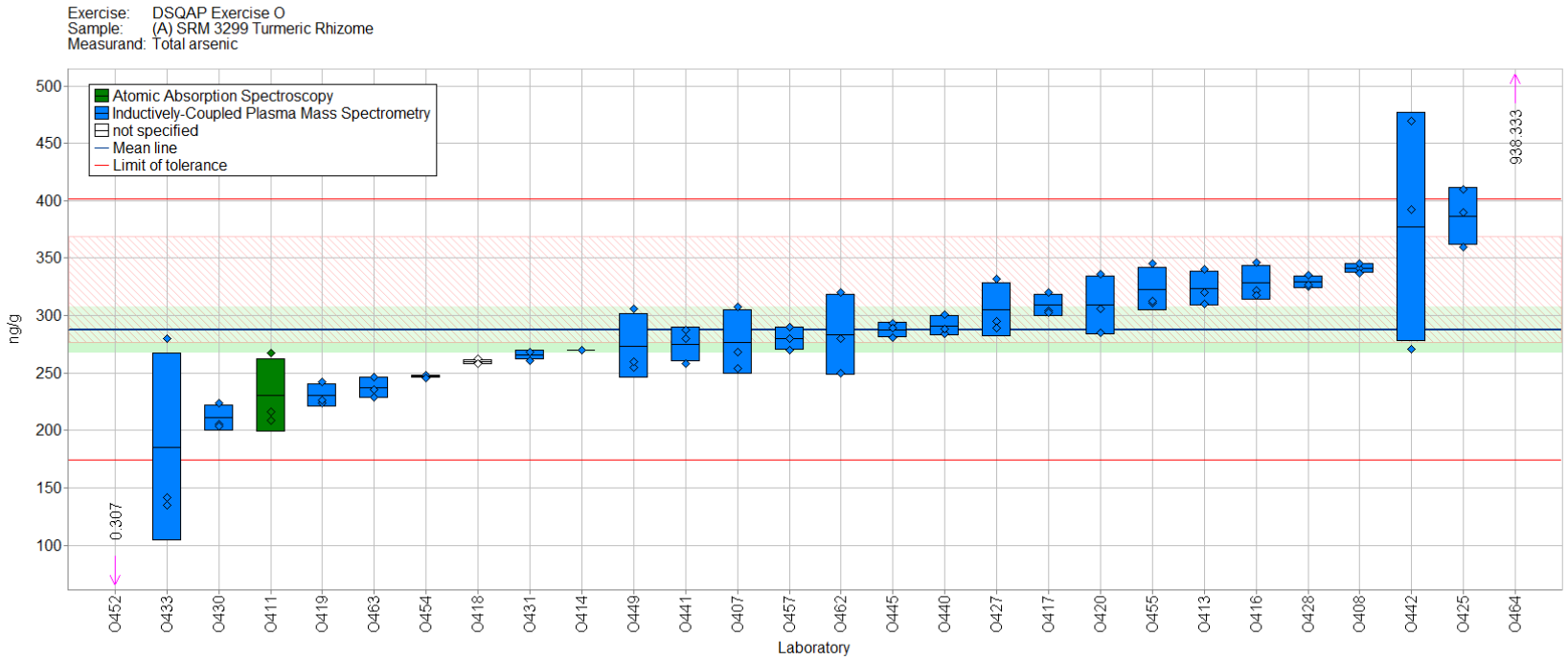


**Figure 1-2.** Total arsenic in turmeric rhizome (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue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

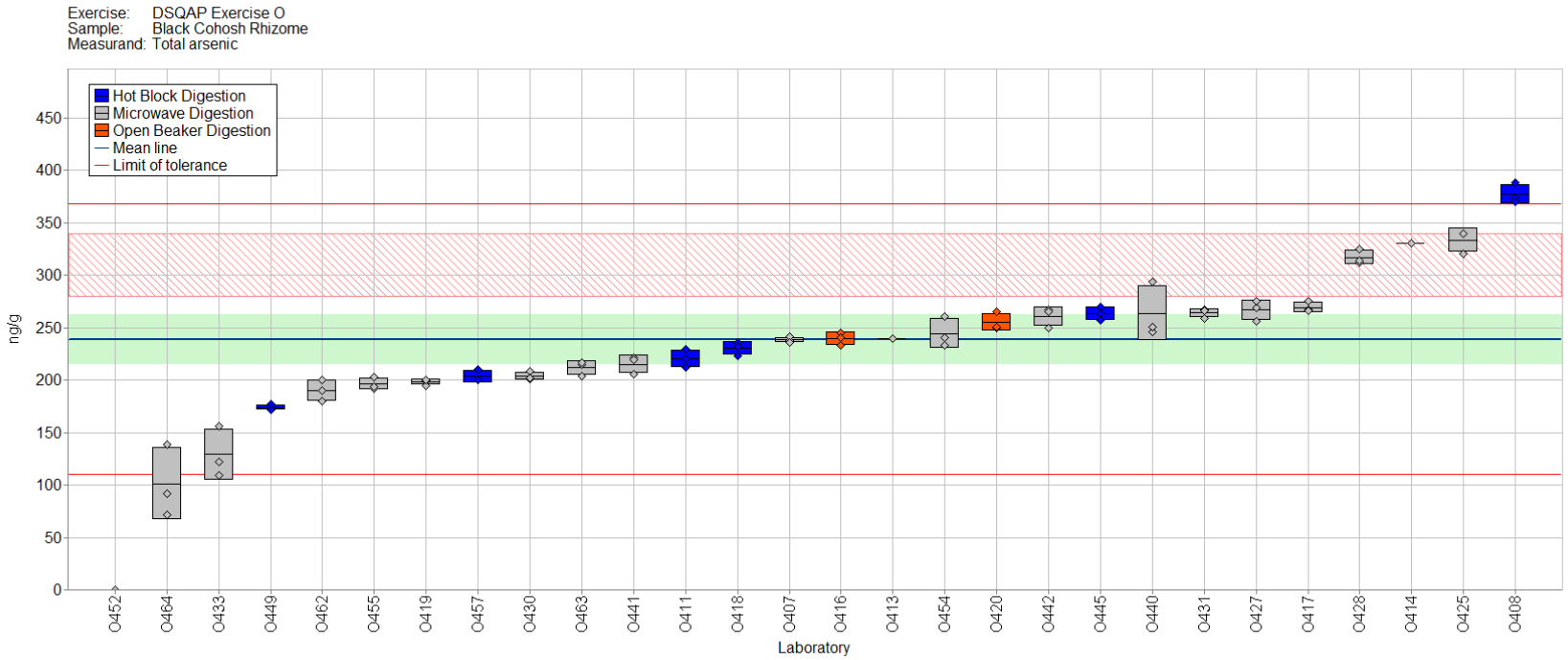


**Figure 1-3.** Total arsenic in black cohosh rhizome (data summary view – sample preparation method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the sample preparation method employed.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

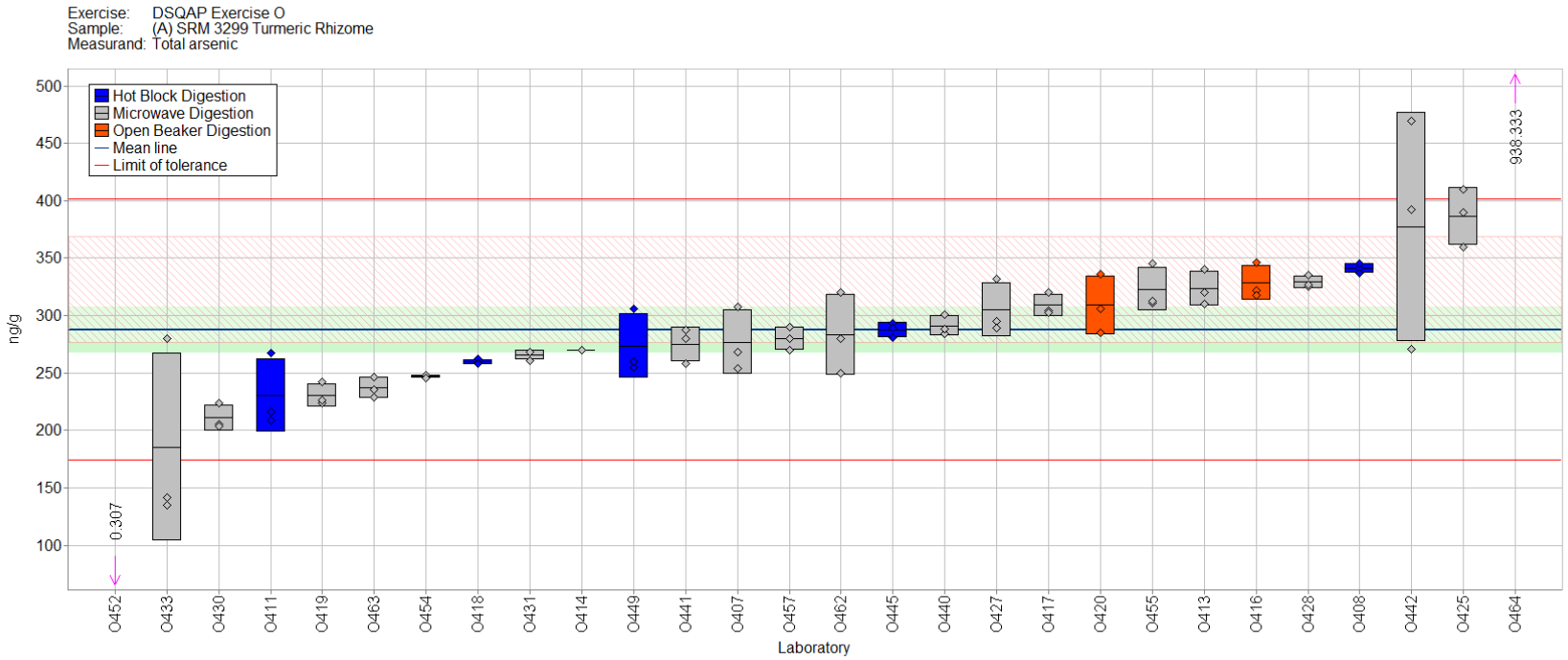


**Figure 1-4.** Total arsenic in turmeric rhizome (data summary view – sample preparation method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the sample preparation method employed.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

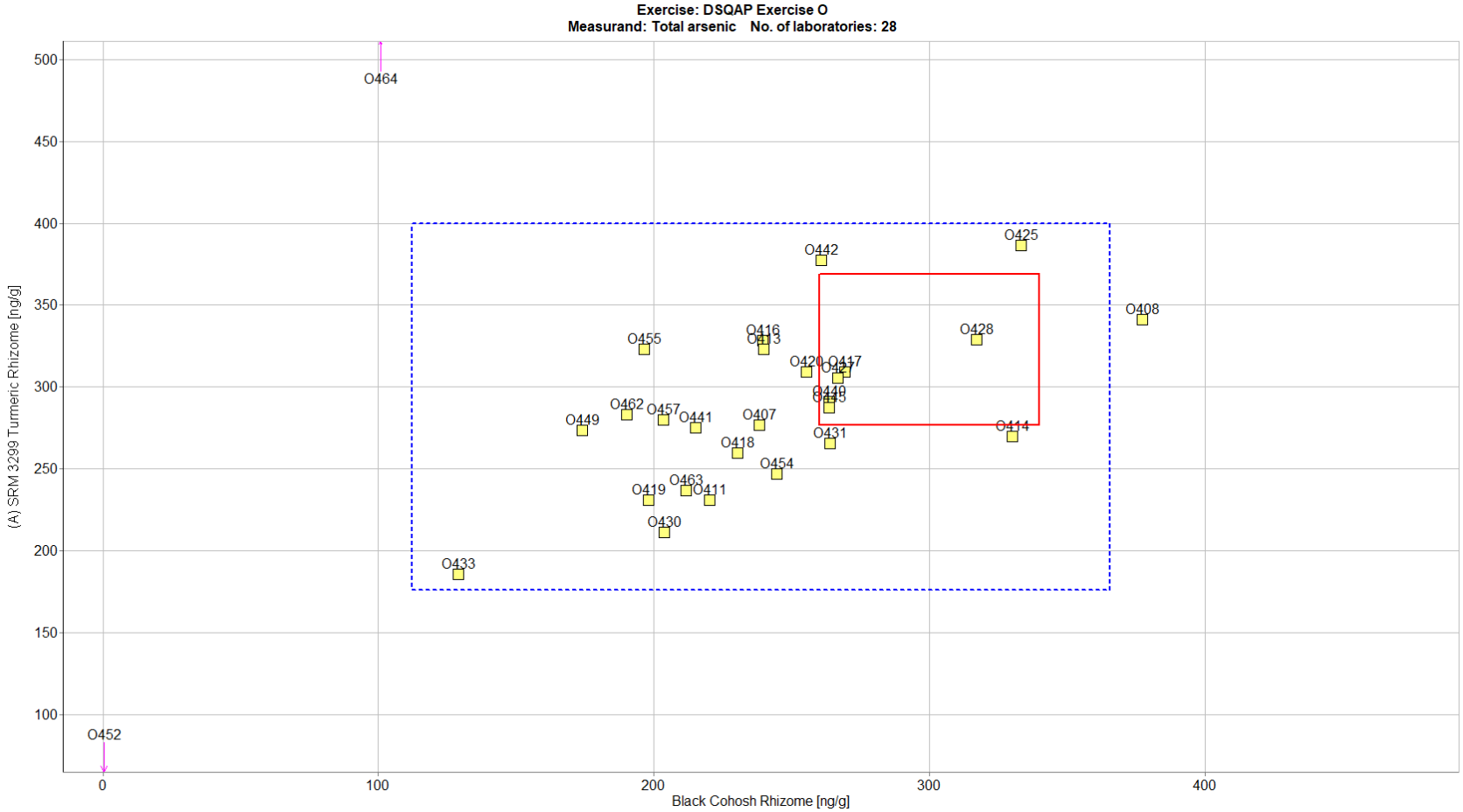


**Figure 1-5.** Laboratory means for total arsenic in black cohosh rhizome and turmeric rhizome (sample/sample comparison view). In this view, the individual laboratory mean for one sample (black cohosh) is compared to the mean for a second sample (turmeric). The solid red box represents the NIST range of tolerance for the two samples, black cohosh (x-axis) and turmeric (y-axis), which encompasses the NIST-determined values bounded by their uncertainties ($U_{\text{NIST}}$) and represents the range that results in an acceptable $Z_{\text{NIST}}$ score, $|Z_{\text{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for black cohosh (x-axis) and turmeric (y-axis), calculated as the values above and below the consensus means that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$.

**Table 1-3.** Data summary table for cadmium in black cohosh and turmeric rhizomes. Data points highlighted in red have been flagged as potential outliers (e.g., Grubb and/or Cochran) by the NIST software package.

| | | Cadmium | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | SRM 3295 Black Cohosh Rhizome (ng/g) | | | | | SRM 3299 Turmeric Rhizome (ng/g) | | | | |
| | Lab | A | B | C | Avg | SD | A | B | C | Avg | SD |
| Individual Results | NIST | | | | 243 | 8 | | | | 1700 | 160 |
| | O405 | | | | | | | | | | |
| | O407 | 247 | 277 | 263 | 262 | 15 | 1600 | 1610 | 1980 | 1730 | 217 |
| | O408 | 291 | 282 | 288 | 287 | 5 | 1760 | 1810 | 1760 | 1777 | 29 |
| | O409 | | | | | | | | | | |
| | O411 | 230 | 229 | 239 | 233 | 6 | 1782 | 1756 | 1863 | 1800 | 56 |
| | O412 | | | | | | | | | | |
| | O413 | 230 | 230 | 240 | 233 | 6 | 1600 | 1500 | 1500 | 1533 | 58 |
| | O414 | 259 | | | 259 | | 1320 | | | 1320 | |
| | O415 | 230 | 236 | 233 | 233 | 3 | 1780 | 1720 | 1670 | 1723 | 55 |
| | O416 | 248 | 224 | 224 | 232 | 14 | 1502 | 1419 | 1499 | 1473 | 47 |
| | O417 | 237 | 239 | 249 | 242 | 6 | 1488 | 1477 | 1511 | 1492 | 17 |
| | O418 | 229 | 240 | 236 | 235 | 6 | 1580 | 1630 | 1540 | 1583 | 45 |
| | O419 | 199 | 194 | 200 | 198 | 3 | 1280 | 1322 | 1267 | 1290 | 29 |
| | O420 | 272 | 266 | 247 | 262 | 13 | 1658 | 1385 | 1353 | 1465 | 168 |
| | O423 | | | | | | | | | | |
| | O425 | 240 | 250 | 230 | 240 | 10 | 1.60 | 1.47 | 1.56 | 1.54 | 0.07 |
| | O426 | | | | | | | | | | |
| | O427 | 265 | 267 | 270 | 267 | 3 | 1750 | 1680 | 1730 | 1720 | 36 |
| | O428 | 242 | 246 | 246 | 245 | 2 | 1.52 | 1.51 | 1.53 | 1.52 | 0.01 |
| | O429 | | | | | | | | | | |
| | O430 | 226 | 231 | 231 | 229 | 3 | 1472 | 1434 | 1515 | 1474 | 41 |
| | O431 | 263 | 235 | 221 | 240 | 21 | 1434 | 1572 | 1546 | | 73 |
| | O433 | 207 | 208 | 207 | 208 | 1 | 1331 | 1360 | 1296 | 1329 | 32 |
| | O434 | | | | | | | | | | |
| | O437 | | | | | | | | | | |
| | O440 | 246 | 271 | 253 | 257 | 13 | 1526 | 1583 | 1590 | 1566 | 35 |
| | O441 | 180 | 159 | 176 | 172 | 11 | 1130 | 1300 | 1270 | 1233 | 91 |
| | O442 | 223 | 224 | 256 | 234 | 19 | 1430 | 1480 | 1480 | 1463 | 29 |
| | O445 | 238 | 230 | 235 | 234 | 4 | 1499 | 1482 | 1496 | 1492 | 9 |
| | O447 | | | | | | | | | | |
| | O449 | 144 | 135 | 132 | 137 | 6 | 927 | 916 | 942 | 928 | 13 |
| | O452 | 0.2 | 0.2 | 0.3 | 0.240 | 0.010 | 1.51 | 1.56 | 1.51 | 1.53 | 0.03 |
| | O454 | 244 | 260 | 250 | 251 | 8 | 1436 | 1409 | 1455 | 1433 | 23 |
| | O455 | 177 | 168 | 171 | 172 | 5 | 1043 | 1000 | 1025 | 1023 | 22 |
| | O457 | 250 | 250 | 250 | 250 | 0 | 1900 | 1900 | 1900 | 1900 | 0 |
| | O458 | | | | | | | | | | |
| | O462 | 200 | 180 | 200 | 193 | 12 | 1280 | 1130 | 1320 | 1243 | 100 |
| | O463 | 162 | 168 | 163 | 165 | 3 | 1314 | 1347 | 1267 | 1309 | 40 |
| | O464 | 216 | 218 | 208 | 214 | 5 | 1312 | 1304 | 1309 | 1308 | 4 |
| Community Results | | Consensus Mean | | | 228 | | Consensus Mean | | | 1415 | |
| | | Consensus Standard Deviation | | | 36 | | Consensus Standard Deviation | | | 299 | |
| | | Maximum | | | 287 | | Maximum | | | 1900 | |
| | | Minimum | | | 0.240 | | Minimum | | | 1.52 | |
| | | N | | | 28 | | N | | | 28 | |

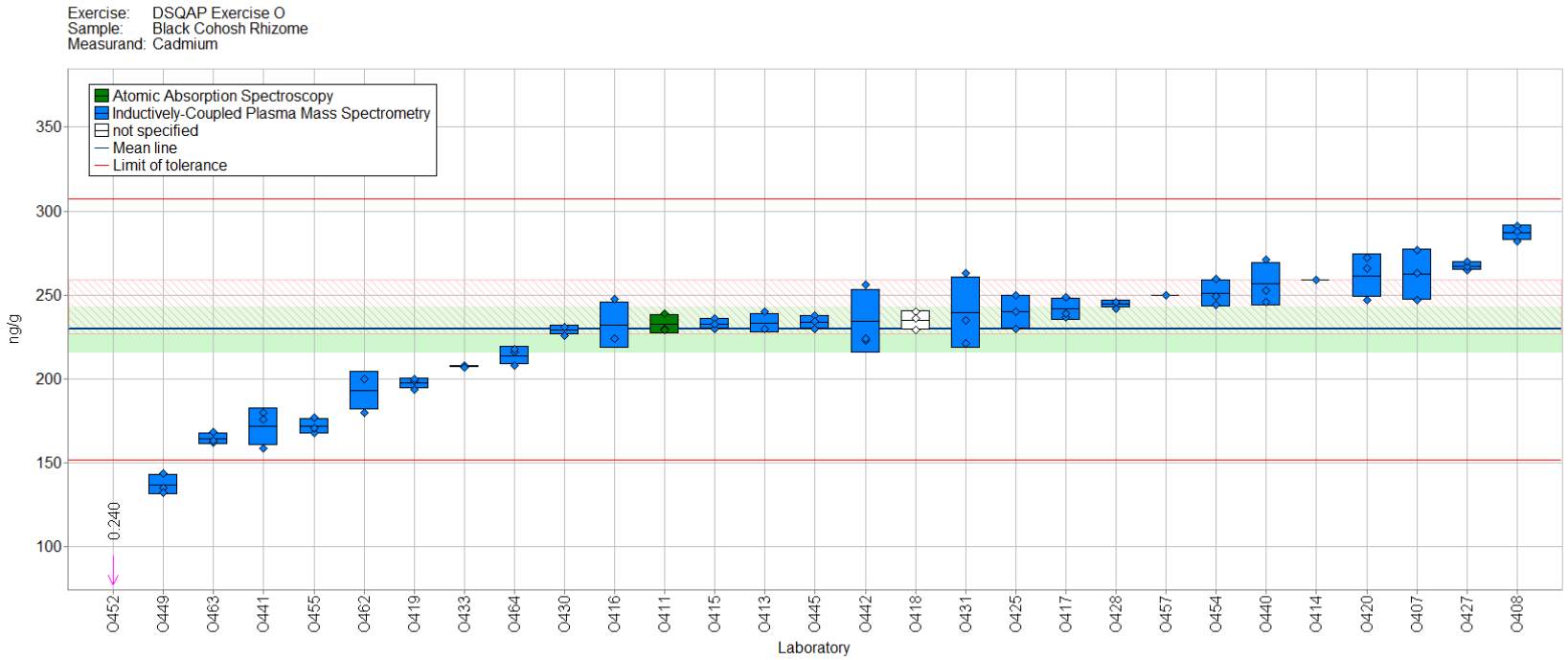


**Figure 1-6.** Cadmium in black cohosh rhizome (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by its uncertainty ($U_{\text{NIST}}$) and represents the range that results in an acceptable $Z_{\text{NIST}}$ score, $|Z_{\text{NIST}}| \leq 2$.

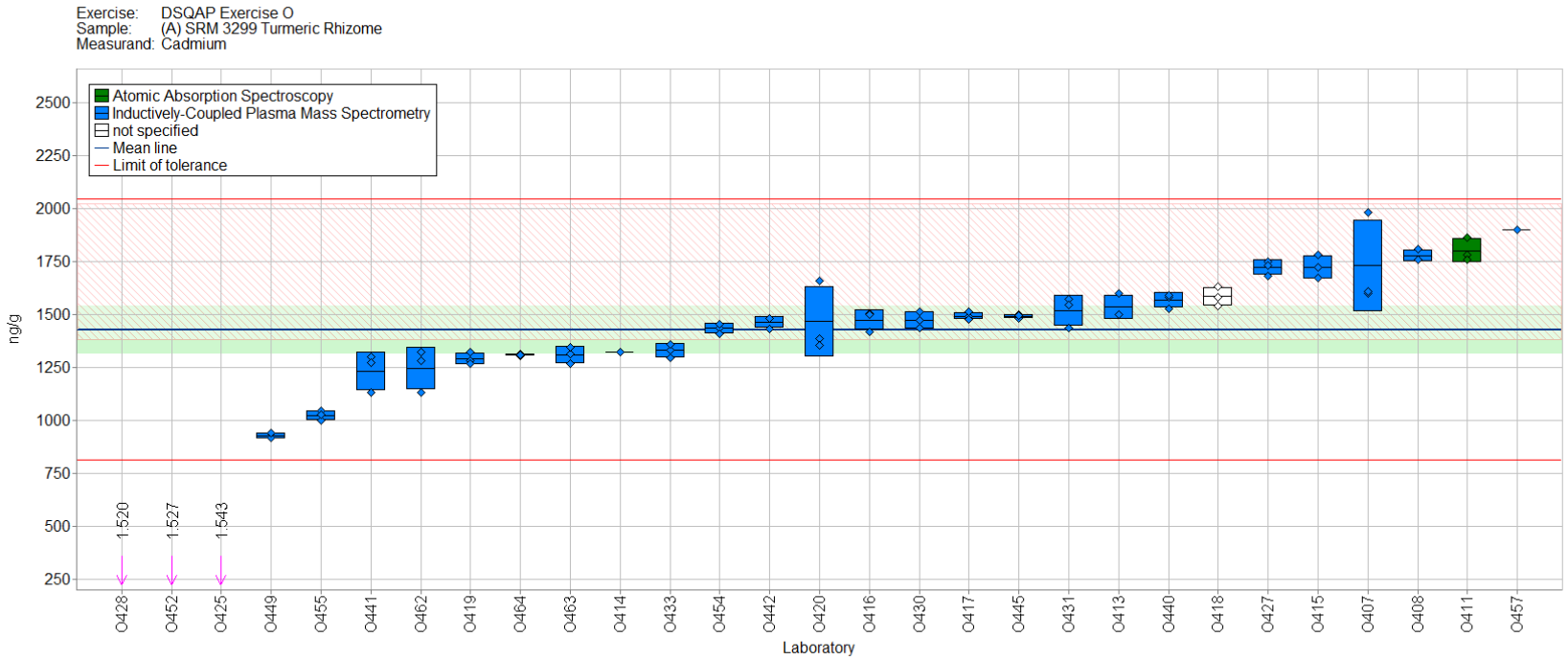


**Figure 1-7.** Cadmium in turmeric rhizome (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by its uncertainty ($U_{\text{NIST}}$) and represents the range that results in an acceptable $Z_{\text{NIST}}$ score, $|Z_{\text{NIST}}| \leq 2$.

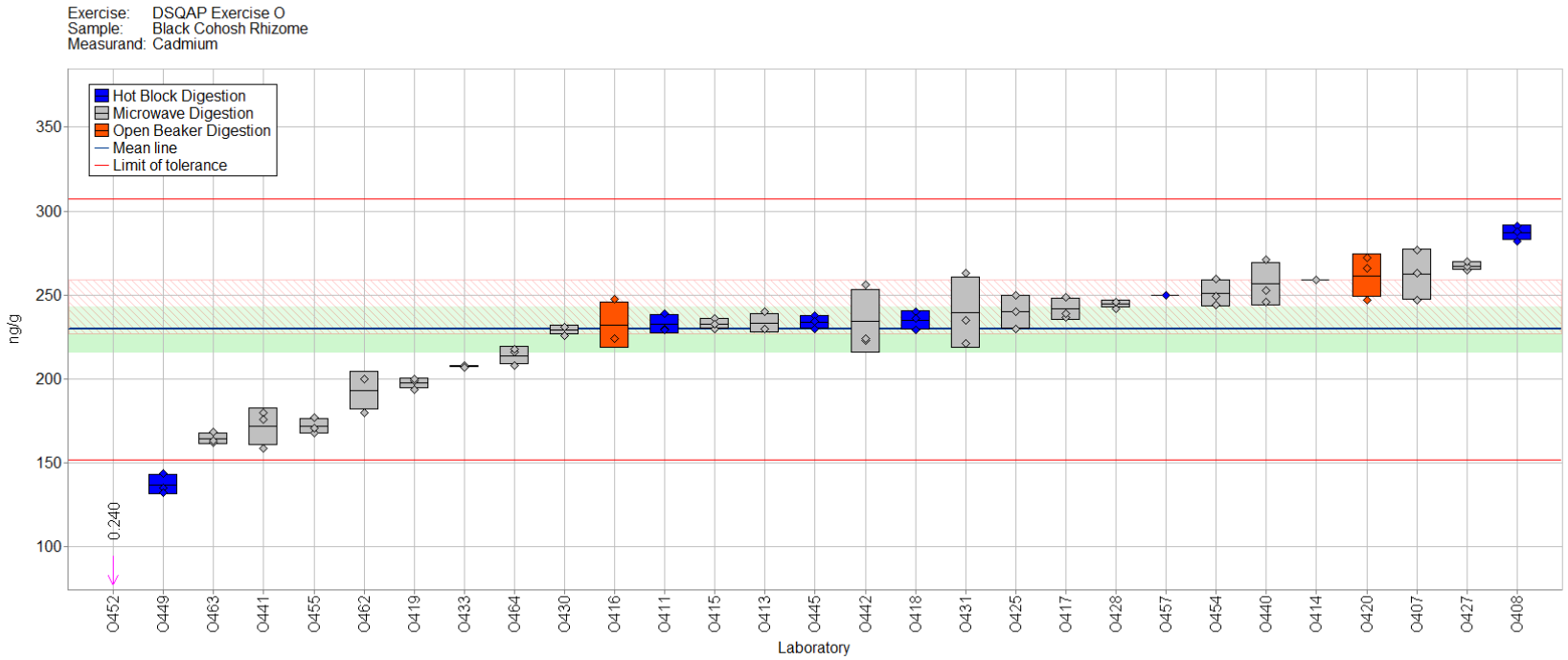


**Figure 1-8.** Cadmium in black cohosh rhizome (data summary view – sample preparation method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the sample preparation method employed.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

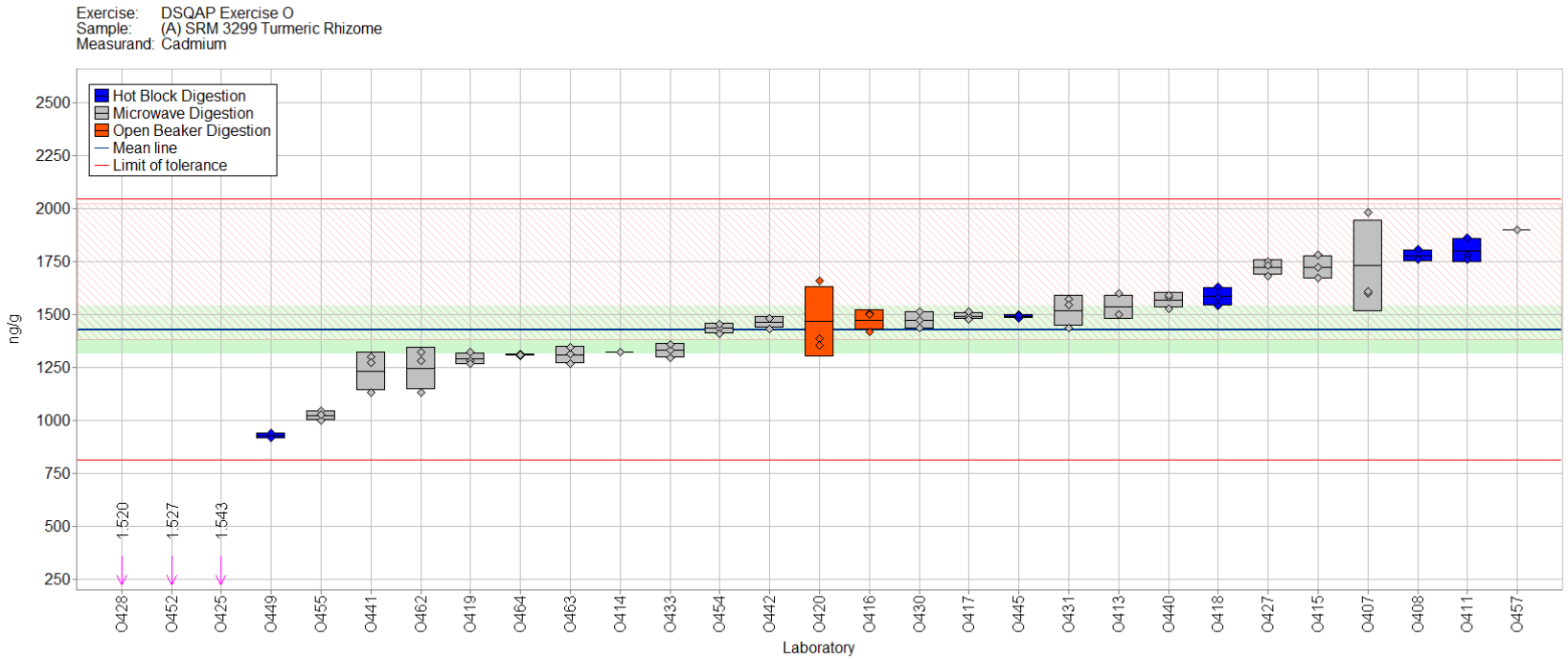


**Figure 1-9.** Cadmium in turmeric rhizome (data summary view – sample preparation method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the sample preparation method employed. The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

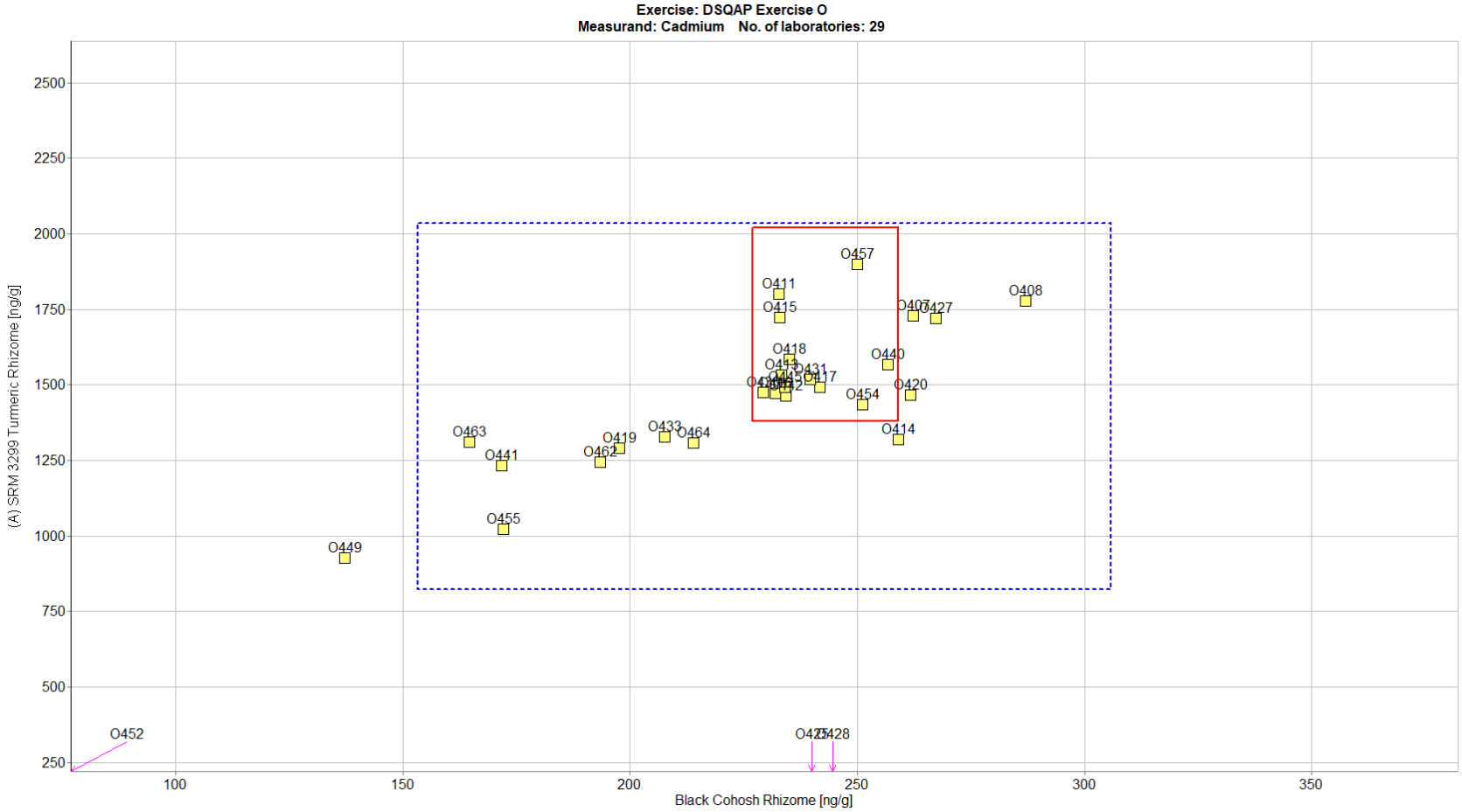


**Figure 1-10.** Laboratory means for cadmium in black cohosh rhizome and turmeric rhizome (sample/sample comparison view). In this view, the individual laboratory mean for one sample (black cohosh) is compared to the mean for a second sample (turmeric). The solid red box represents the NIST range of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

**Table 1-4.** Data summary table for lead in black cohosh and turmeric rhizomes. Data points highlighted in red have been flagged as potential outliers (e.g., Grubb and/or Cochran) by the NIST software package.

| | | Lead | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | SRM 3295 Black Cohosh Rhizome (ng/g) | | | | | SRM 3299 Turmeric Rhizome (ng/g) | | | | |
| | Lab | A | B | C | Avg | SD | A | B | C | Avg | SD |
| Individual Results | NIST | | | | 2236 | 46 | | | | 1143 | 38 |
| | O405 | | | | | | | | | | |
| | O407 | 1970 | 2040 | 2050 | 2020 | 44 | 1090 | 1180 | 1400 | 1223 | 159 |
| | O408 | 2080 | 2090 | 2080 | 2083 | 6 | 1080 | 1100 | 1120 | 1100 | 20 |
| | O409 | | | | | | | | | | |
| | O411 | 1679 | 1633 | 1743 | 1685 | 55 | 864 | 739 | 678 | 760 | 95 |
| | O412 | | | | | | | | | | |
| | O413 | 2000 | 2000 | 2000 | 2000 | 0 | 1100 | 1100 | 1100 | 1100 | 0 |
| | O414 | 1900 | | | 1900 | | 880 | | | 880 | |
| | O415 | 1930 | 1950 | 1910 | 1930 | 20 | 1160 | 1110 | 1190 | 1153 | 40 |
| | O416 | 1764 | 1745 | 1733 | 1747 | 15 | 1062 | 1010 | 1056 | 1043 | 29 |
| | O417 | 1981 | 1989 | 2041 | 2004 | 33 | 1065 | 1029 | 1096 | 1063 | 34 |
| | O418 | 1780 | 1990 | 1970 | 1913 | 116 | 1080 | 1070 | 1040 | 1063 | 21 |
| | O419 | 1604 | 1595 | 1601 | 1600 | 5 | 918 | 878 | 872 | 889 | 25 |
| | O420 | 1986 | 2031 | 2045 | 2021 | 31 | 1073 | 1007 | 1057 | 1046 | 34 |
| | O423 | | | | | | | | | | |
| | O425 | 1.63 | 1.75 | 1.74 | 1.71 | 0.07 | 1.07 | 1.02 | 1.04 | 1.04 | 0.03 |
| | O426 | | | | | | | | | | |
| | O427 | 1850 | 1800 | 1910 | 1853 | 55 | 1170 | 1100 | 1100 | 1123 | 40 |
| | O428 | 2.85 | 2.07 | 2.12 | 2.35 | 0.43 | 1.17 | 1.16 | 1.18 | 1.17 | 0.01 |
| | O429 | | | | | | | | | | |
| | O430 | 1708 | 1682 | 1653 | 1681 | 28 | 985 | 958 | 949 | 964 | 19 |
| | O431 | 2019 | 2022 | 2056 | 2032 | 21 | 1099 | 1042 | 1553 | 1231 | 280 |
| | O433 | 1889 | 1862 | 1870 | 1873 | 14 | 1148 | 1262 | 1080 | 1163 | 92 |
| | O434 | | | | | | | | | | |
| | O437 | | | | | | | | | | |
| | O440 | 1903 | 1991 | 1871 | 1922 | 62 | 1162 | 1048 | 1050 | 1087 | 65 |
| | O441 | 1737 | 1653 | 1590 | 1660 | 74 | 862 | 994 | 955 | 937 | 68 |
| | O442 | 1830 | 1800 | 1890 | 1840 | 46 | 992 | 1100 | 1010 | 1034 | 58 |
| | O445 | 1904 | 1881 | 1853 | 1879 | 26 | 1035 | 1031 | 1000 | 1022 | 19 |
| | O447 | | | | | | | | | | |
| | O449 | 1614 | 1744 | 1770 | 1709 | 83 | 949 | 1064 | 1078 | 1030 | 71 |
| | O452 | 1.95 | 1.97 | 2.05 | 1.99 | 0.05 | 1.08 | 1.07 | 1.18 | 1.11 | 0.06 |
| | O454 | 1906 | 2189 | 2138 | 2077 | 150 | 961 | 932 | 974 | 955 | 22 |
| | O455 | 1577 | 1494 | 1422 | 1498 | 78 | 832 | 753 | 763 | 783 | 43 |
| | O457 | 2100 | 2000 | 2000 | 2033 | 58 | 1000 | 1100 | 1100 | 1067 | 58 |
| | O458 | | | | | | | | | | |
| | O462 | 2070 | 2280 | 2860 | 2403 | 409 | 1050 | 1100 | 1510 | 1220 | 252 |
| | O463 | 1169 | 1082 | 1294 | 1182 | 107 | 966 | 994 | 989 | 983 | 15 |
| | O464 | 197 | 193 | 187 | 192 | 5 | 188 | 197 | 200 | 195 | 6 |
| Community Results | | Consensus Mean | | | 1790 | | Consensus Mean | | | 994 | |
| | | Consensus Standard Deviation | | | 300 | | Consensus Standard Deviation | | | 182 | |
| | | Maximum | | | 2403 | | Maximum | | | 1231 | |
| | | Minimum | | | 1.707 | | Minimum | | | 1.043 | |
| | | N | | | 28 | | N | | | 28 | |

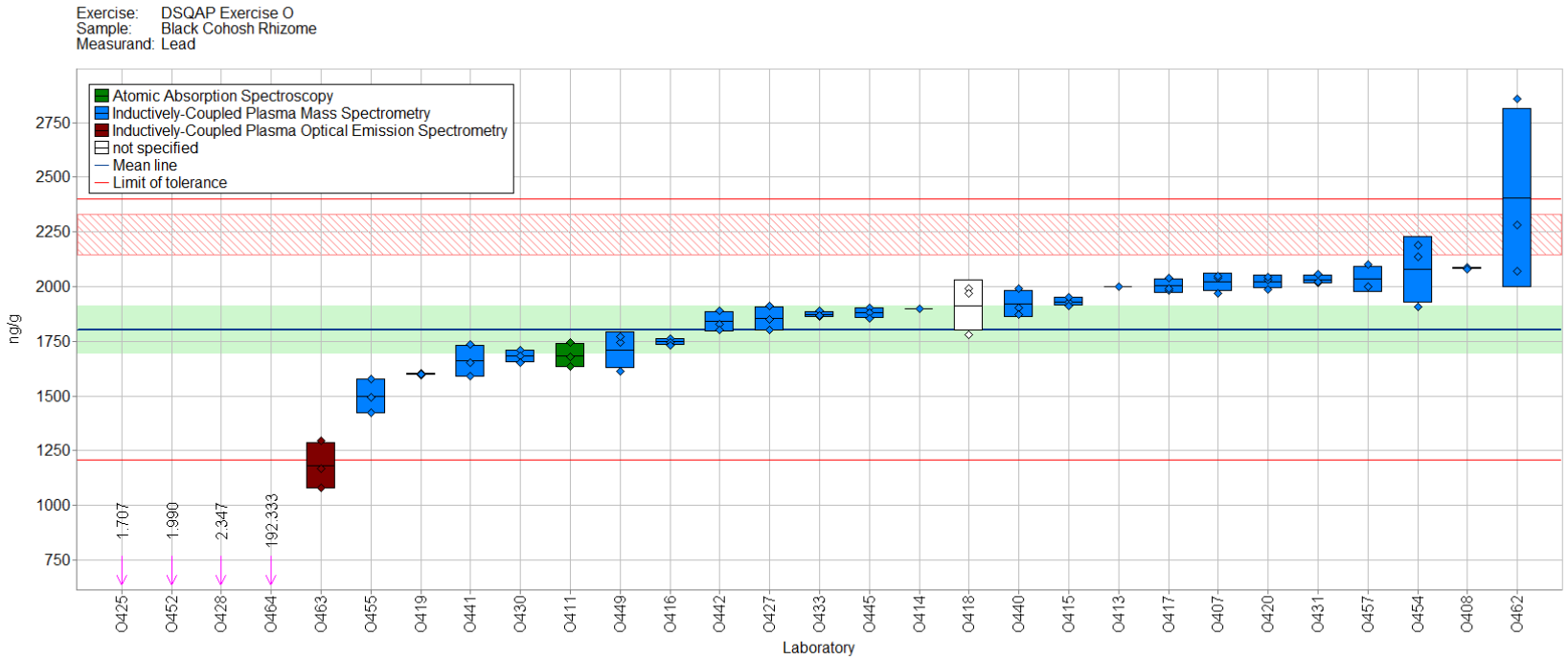


**Figure 1-11.** Lead in candidate black cohosh rhizome (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by its uncertainty ($U_{\text{NIST}}$) and represents the range that results in an acceptable $Z_{\text{NIST}}$ score, $|Z_{\text{NIST}}| \leq 2$.

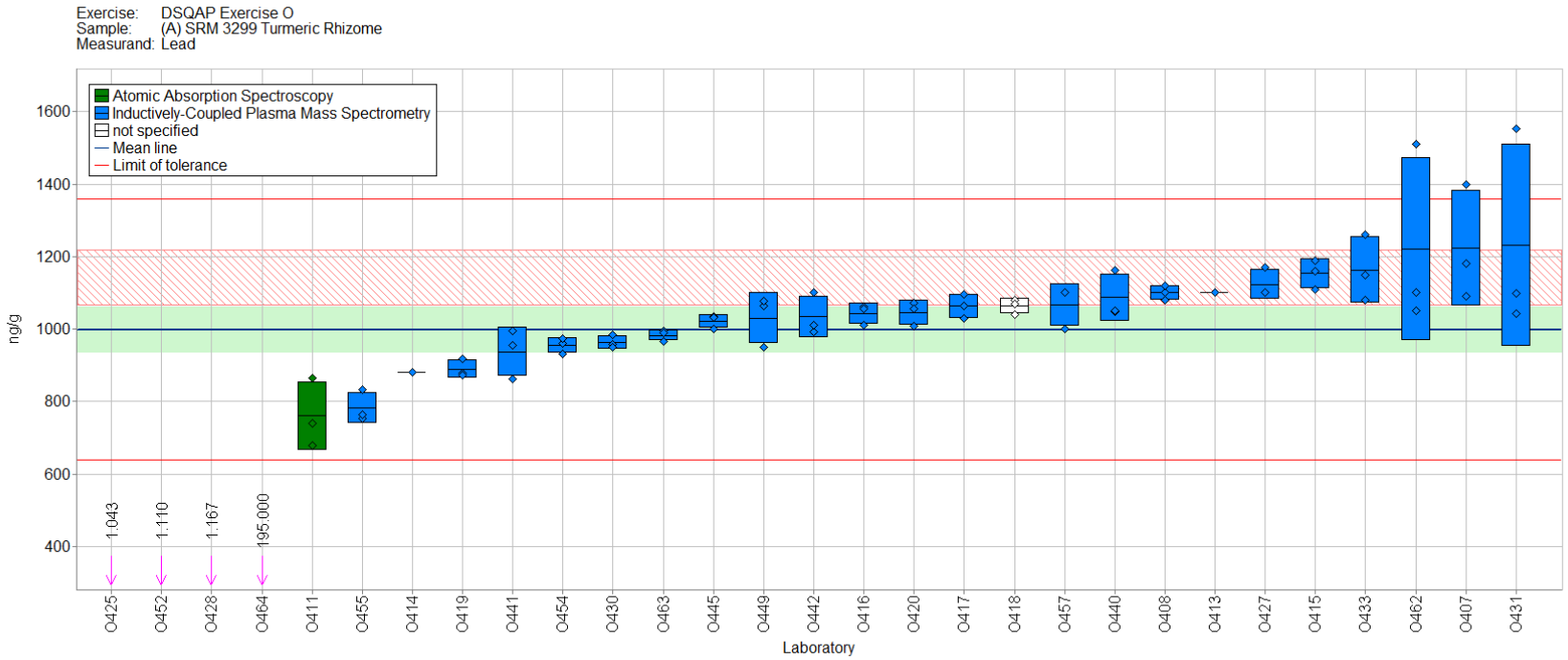


**Figure 1-12.** Lead in turmeric rhizome (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by its uncertainty ($U_{\text{NIST}}$) and represents the range that results in an acceptable $Z_{\text{NIST}}$ score, $|Z_{\text{NIST}}| \leq 2$.

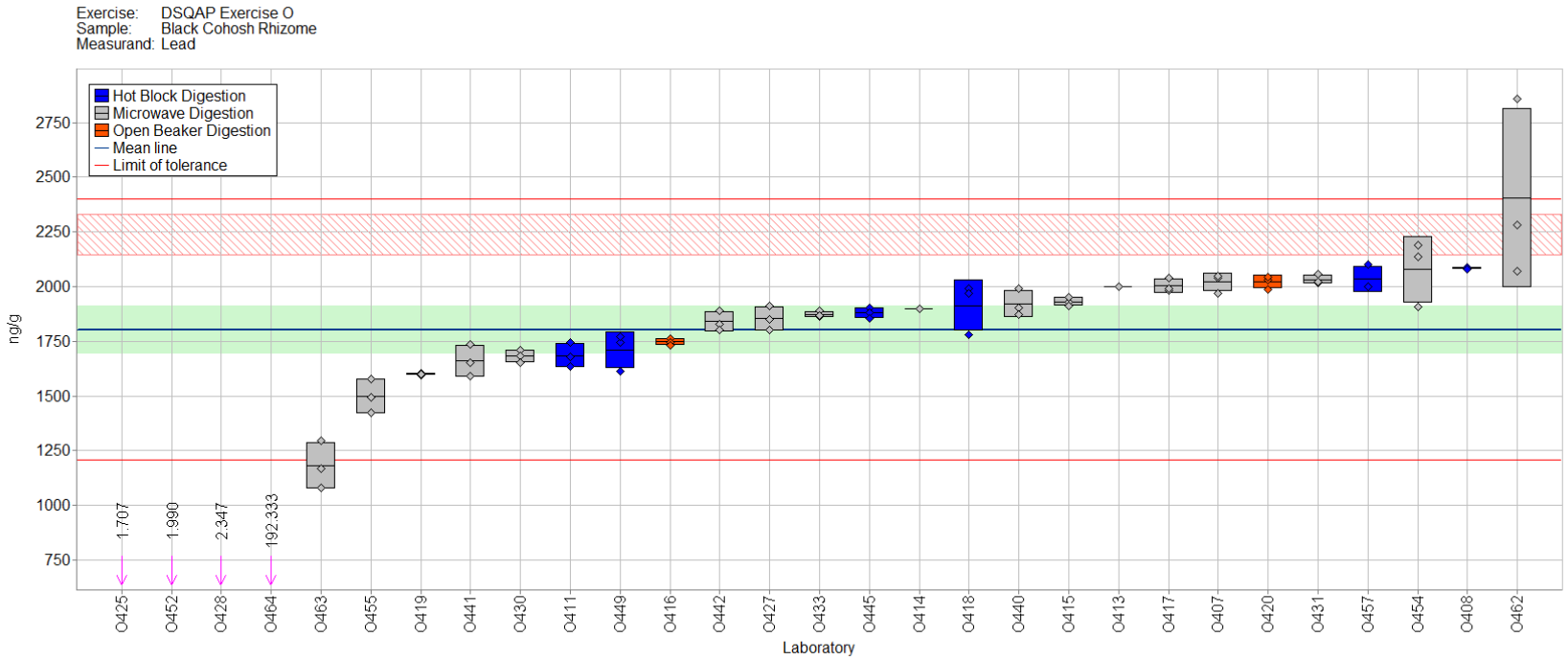


**Figure 1-13.** Lead in black cohosh rhizome (data summary view – sample preparation method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the sample preparation method employed.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

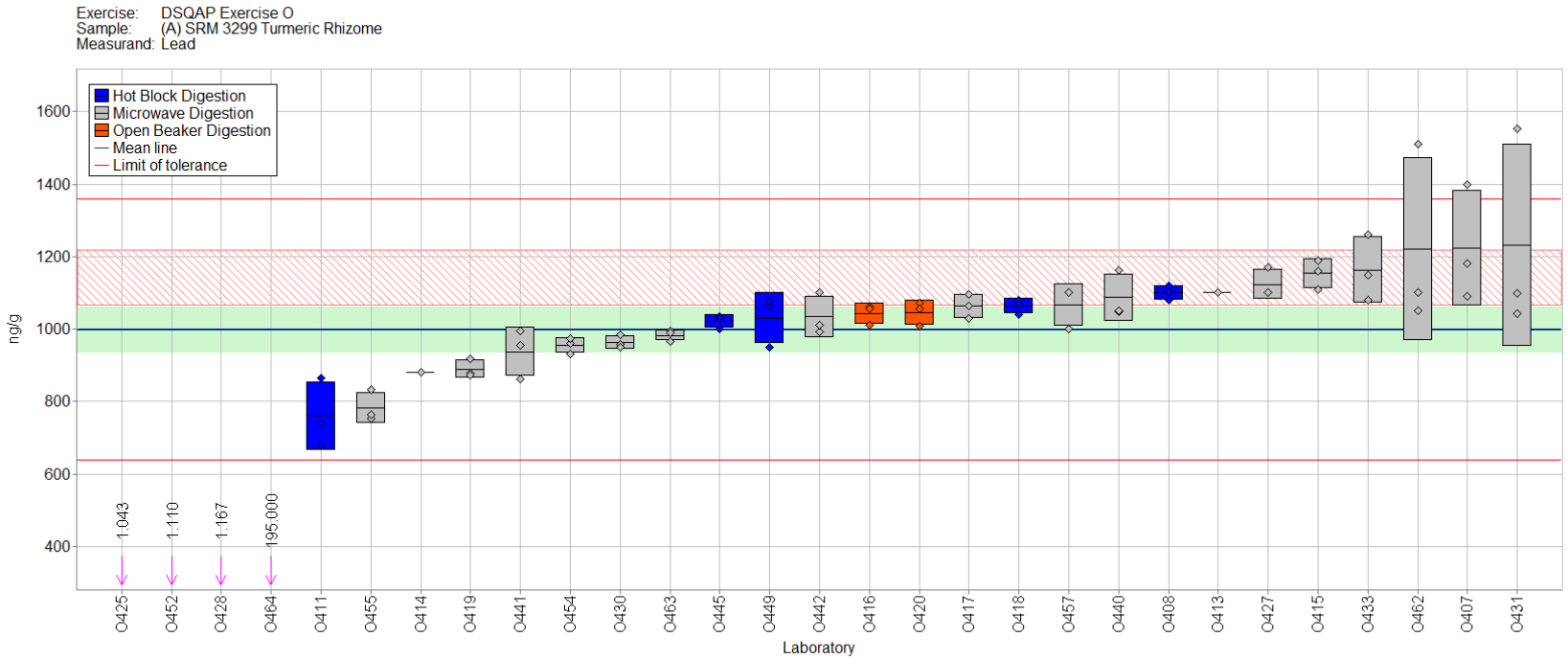


**Figure 1-14.** Lead in turmeric rhizome (data summary view – sample preparation method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the sample preparation method employed. The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

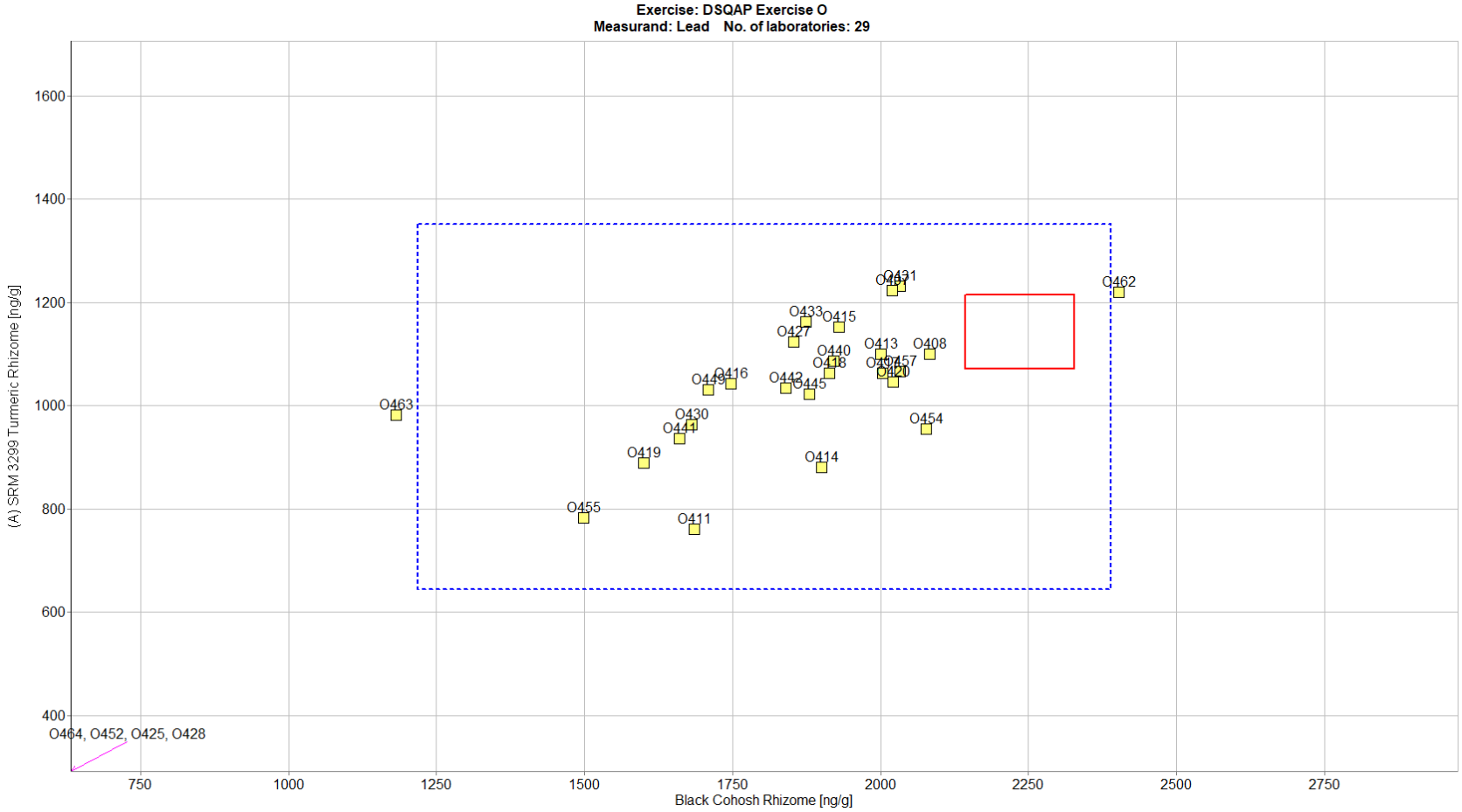


**Figure 1-15.** Laboratory means for lead in black cohosh rhizome and turmeric rhizome (sample/sample comparison view). In this view, the individual laboratory mean for one sample (black cohosh) is compared to the mean for a second sample (turmeric). The solid red box represents the NIST range of tolerance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

**Table 1-5.** Data summary table for mercury in black cohosh and turmeric rhizomes. Data points highlighted in red have been flagged as potential outliers (e.g., Grubb and/or Cochran) by the NIST software package.

| | | Mercury | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | SRM 3295 Black Cohosh Rhizome (ng/g) | | | | | SRM 3299 Turmeric Rhizome (ng/g) | | | | |
| | Lab | A | B | C | Avg | SD | A | B | C | Avg | SD |
| Individual Results | NIST | | | | 12.8 | 0.1 | | | | 54.1 | 3.7 |
| | O405 | | | | | | | | | | |
| | O407 | 10.0 | 13.0 | 12.0 | 11.7 | 1.5 | 72.0 | 90.0 | 68.0 | 76.7 | 11.7 |
| | O408 | 18.0 | 17.0 | 15.0 | 16.7 | 1.5 | 81.0 | 91.0 | 150.0 | 107.3 | 37.3 |
| | O409 | | | | | | | | | | |
| | O412 | | | | | | | | | | |
| | O413 | 16.0 | 15.0 | 13.0 | 14.7 | 1.5 | 62.0 | 94.0 | 62.0 | 72.7 | 18.5 |
| | O414 | 11.0 | | | 11.0 | | 47.0 | | | 47.0 | |
| | O415 | 13.4 | 13.3 | 10.1 | 12.3 | 1.9 | 65.7 | 84.5 | 53.2 | 67.8 | 15.8 |
| | O416 | 12.4 | 18.3 | 23.3 | 18.0 | 5.5 | 15.5 | 17.7 | 18.0 | 17.1 | 1.4 |
| | O417 | 13.6 | 13.1 | 13.7 | 13.5 | 0.3 | 60.0 | 68.5 | 67.3 | 65.3 | 4.6 |
| | O418 | < 19 | < 19 | < 19 | < 19 | | 53.5 | 42.1 | 85.8 | 60.5 | 22.7 |
| | O419 | 9.0 | 12.0 | 11.0 | 10.7 | 1.5 | 65.0 | 65.0 | 50.0 | 60.0 | 8.7 |
| | O420 | 16.0 | 16.0 | 15.0 | 15.7 | 0.6 | 60.0 | 60.0 | 72.0 | 64.0 | 6.9 |
| | O423 | | | | | | | | | | |
| | O425 | 30.0 | 50.0 | 40.0 | 40.0 | 10.0 | 90.0 | 90.0 | 90.0 | 90.0 | 0.0 |
| | O426 | 10.0 | 13.0 | 10.0 | 11.0 | 1.7 | 50.0 | 52.5 | 50.7 | 51.1 | 1.3 |
| | O427 | 12.1 | 12.0 | 12.5 | 12.2 | 0.3 | 77.9 | 59.0 | 59.1 | 65.3 | 10.9 |
| | O428 | 21.0 | 21.0 | 23.0 | 21.7 | 1.2 | 102.0 | 72.0 | 79.0 | 84.3 | 15.7 |
| | O429 | | | | | | | | | | |
| | O430 | 11.7 | 12.4 | 11.3 | 11.8 | 0.5 | 101.0 | 76.2 | 98.3 | 91.8 | 13.6 |
| | O431 | 13.0 | 14.0 | 14.0 | 13.7 | 0.6 | 87.0 | 98.0 | 49.0 | 78.0 | 25.7 |
| | O433 | 19.9 | 9.3 | 9.9 | 13.0 | 5.9 | 86.7 | 54.7 | 72.0 | 71.1 | 16.0 |
| | O434 | | | | | | | | | | |
| | O437 | | | | | | | | | | |
| | O440 | 25.0 | 24.8 | 23.6 | 24.5 | 0.8 | 74.8 | 87.0 | 151.0 | 104.3 | 40.9 |
| | O441 | | | | | | 62.5 | 49.1 | 51.7 | 54.4 | 7.1 |
| | O442 | 12.0 | 13.0 | 14.0 | 13.0 | 1.0 | 46.0 | 107.0 | 64.0 | 72.3 | 31.3 |
| | O445 | 9.4 | 7.3 | 9.9 | 8.9 | 1.4 | 63.9 | 57.9 | 47.8 | 56.5 | 8.1 |
| | O447 | | | | | | | | | | |
| | O449 | 27.8 | 24.8 | 22.4 | 25.0 | 2.7 | 77.9 | 69.8 | 76.7 | 74.8 | 4.4 |
| | O452 | < 0.01 | < 0.01 | < 0.01 | < 0.01 | | 0.040 | 0.030 | 0.070 | 0.047 | 0.021 |
| | O454 | 14.3 | 15.0 | 12.9 | 14.1 | 1.1 | 59.7 | 57.4 | 81.2 | 66.1 | 13.1 |
| | O455 | 26.0 | 26.0 | 27.0 | 26.3 | 0.6 | 77.0 | 73.0 | 77.0 | 75.7 | 2.3 |
| | O457 | < 10 | < 10 | < 10 | < 10 | | 69.0 | 82.0 | 68.0 | 73.0 | 7.8 |
| | O458 | | | | | | | | | | |
| | O462 | < 30 | < 30 | < 30 | < 30 | | < 30 | 30.0 | < 30 | 30.0 | |
| | O463 | 10.8 | 8.9 | 8.9 | 9.5 | 1.1 | 59.3 | 61.4 | 54.1 | 58.3 | 3.8 |
| | O464 | 22.0 | 19.0 | 20.0 | 20.3 | 1.5 | 1724 | 1769 | 1753 | 1749 | 23 |
| Community Results | | Consensus Mean | | | 15.3 | | Consensus Mean | | | 68.6 | |
| | | Consensus Standard Deviation | | | 5.6 | | Consensus Standard Deviation | | | 20.3 | |
| | | Maximum | | | 40.0 | | Maximum | | | 1749 | |
| | | Minimum | | | 8.9 | | Minimum | | | 0.047 | |
| | | N | | | 23 | | N | | | 27 | |

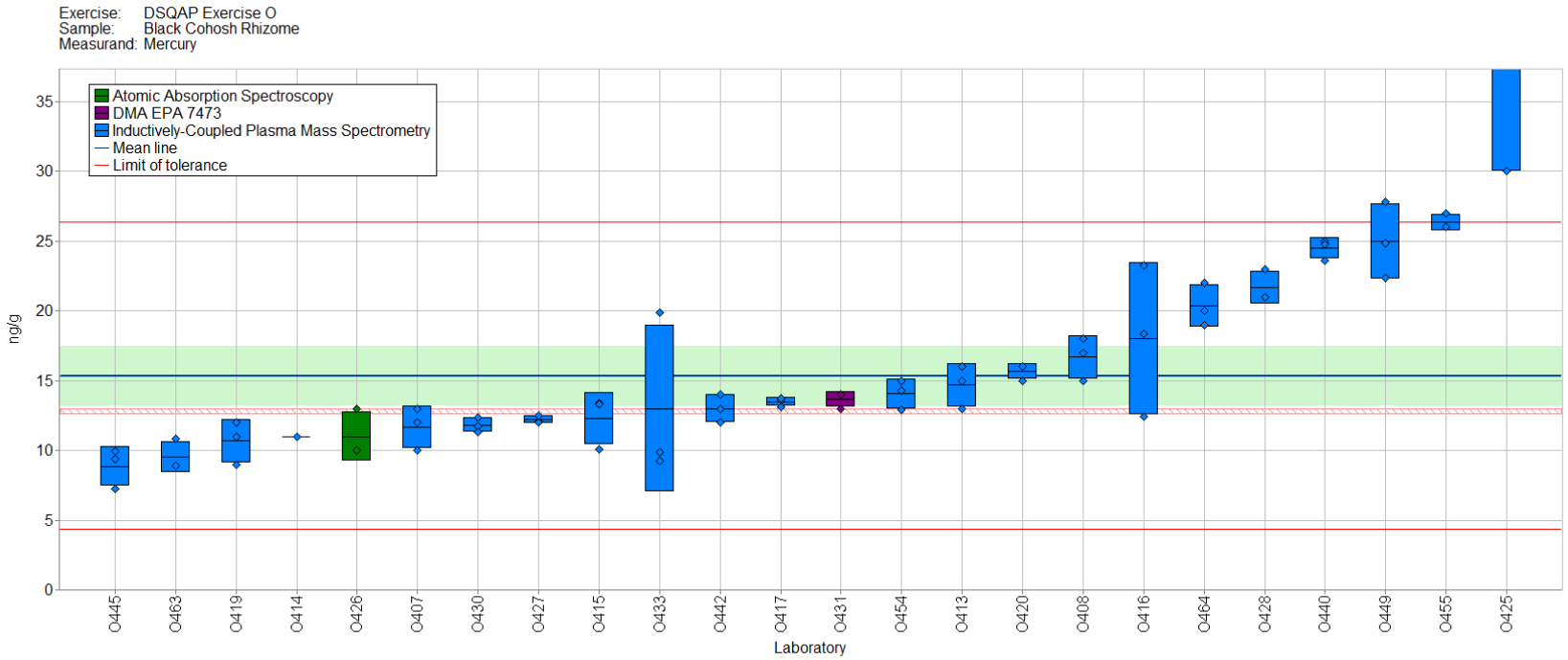


**Figure 1-16.** Mercury in black cohosh rhizome (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by its uncertainty ($U_{\text{NIST}}$) and represents the range that results in an acceptable $Z_{\text{NIST}}$ score, $|Z_{\text{NIST}}| \leq 2$.

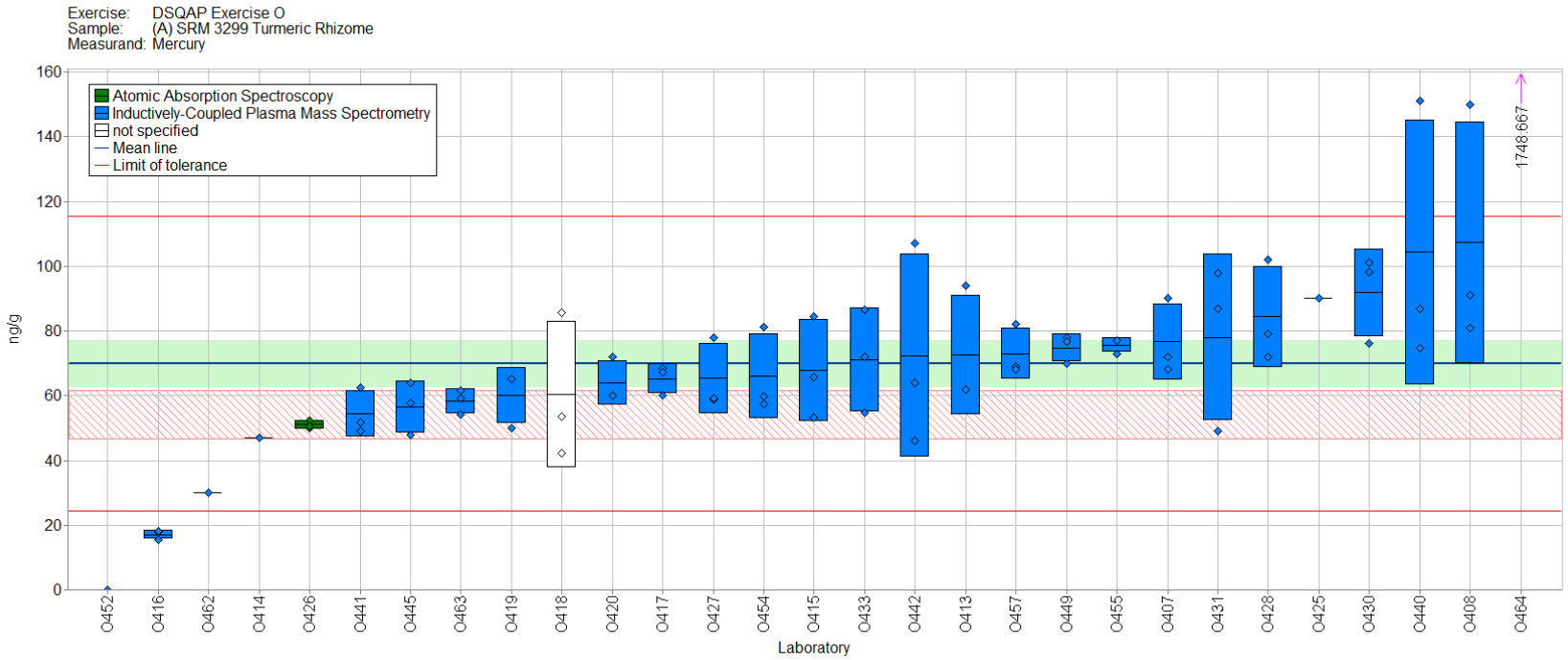


**Figure 1-17.** Mercury in turmeric rhizome (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by its uncertainty ($U_{\text{NIST}}$) and represents the range that results in an acceptable $Z_{\text{NIST}}$ score, $|Z_{\text{NIST}}| \leq 2$.

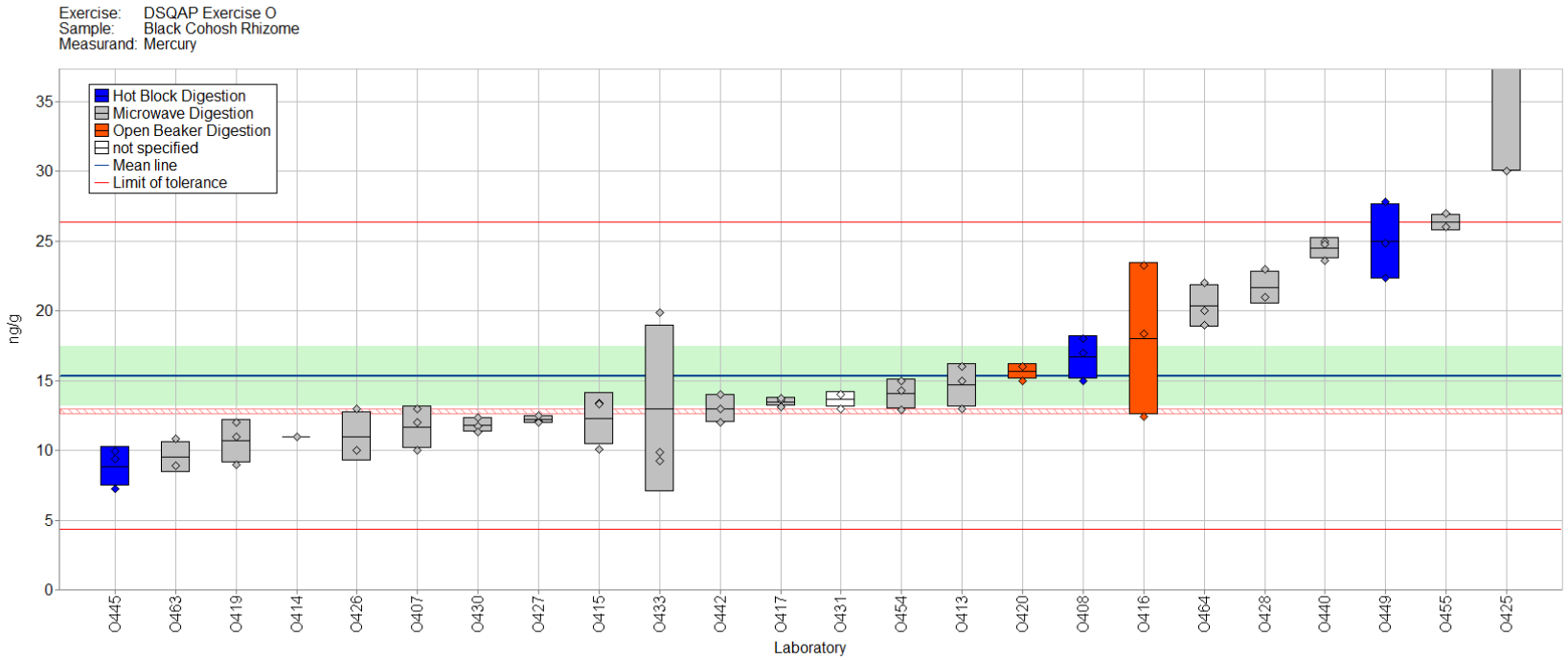


**Figure 1-18.** Mercury black cohosh rhizome (data summary view – sample preparation method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the sample preparation method employed. The solid blue line represents the consensus mean and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by its uncertainty ($U_{\text{NIST}}$) and represents the range that results in an acceptable $Z_{\text{NIST}}$ score, $|Z_{\text{NIST}}| \leq 2$.

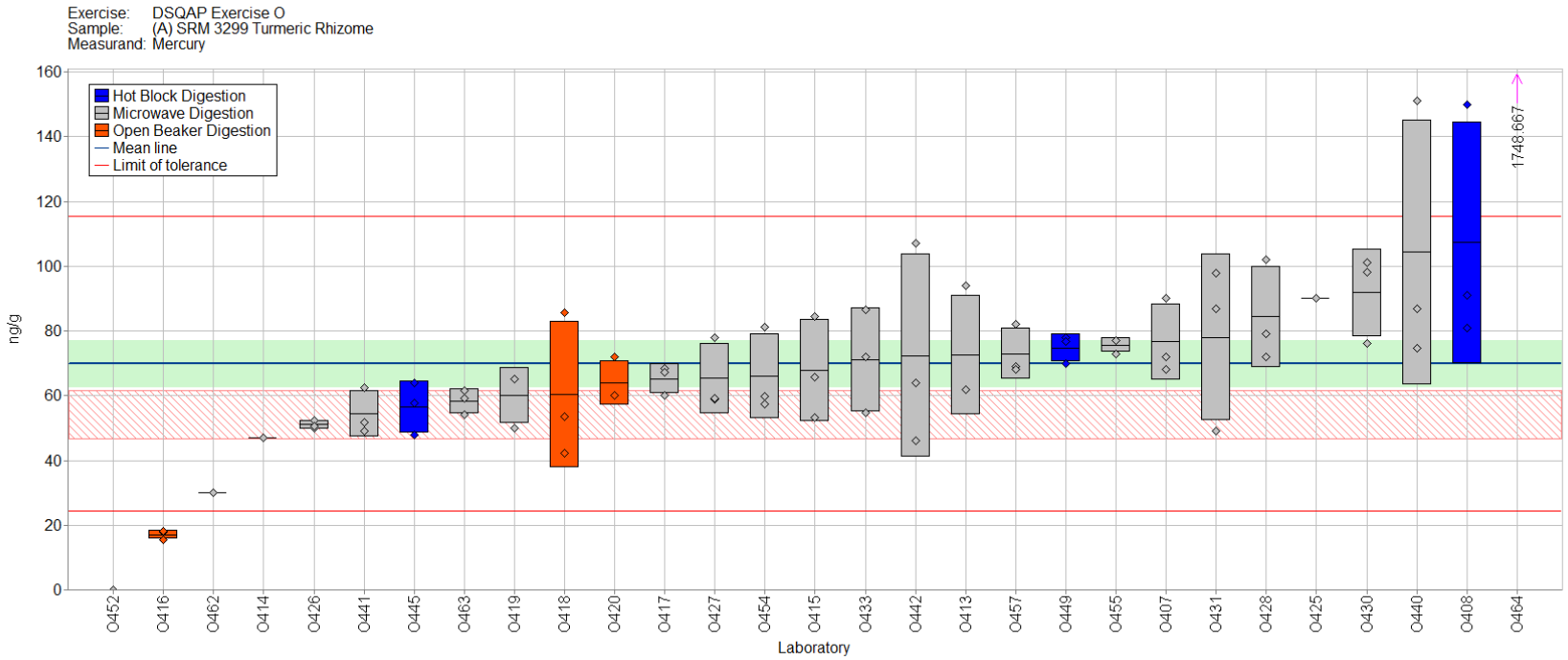


**Figure 1-19.** Mercury in turmeric rhizome (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by its uncertainty ($U_{\text{NIST}}$) and represents the range that results in an acceptable $Z_{\text{NIST}}$ score, $|Z_{\text{NIST}}| \leq 2$.

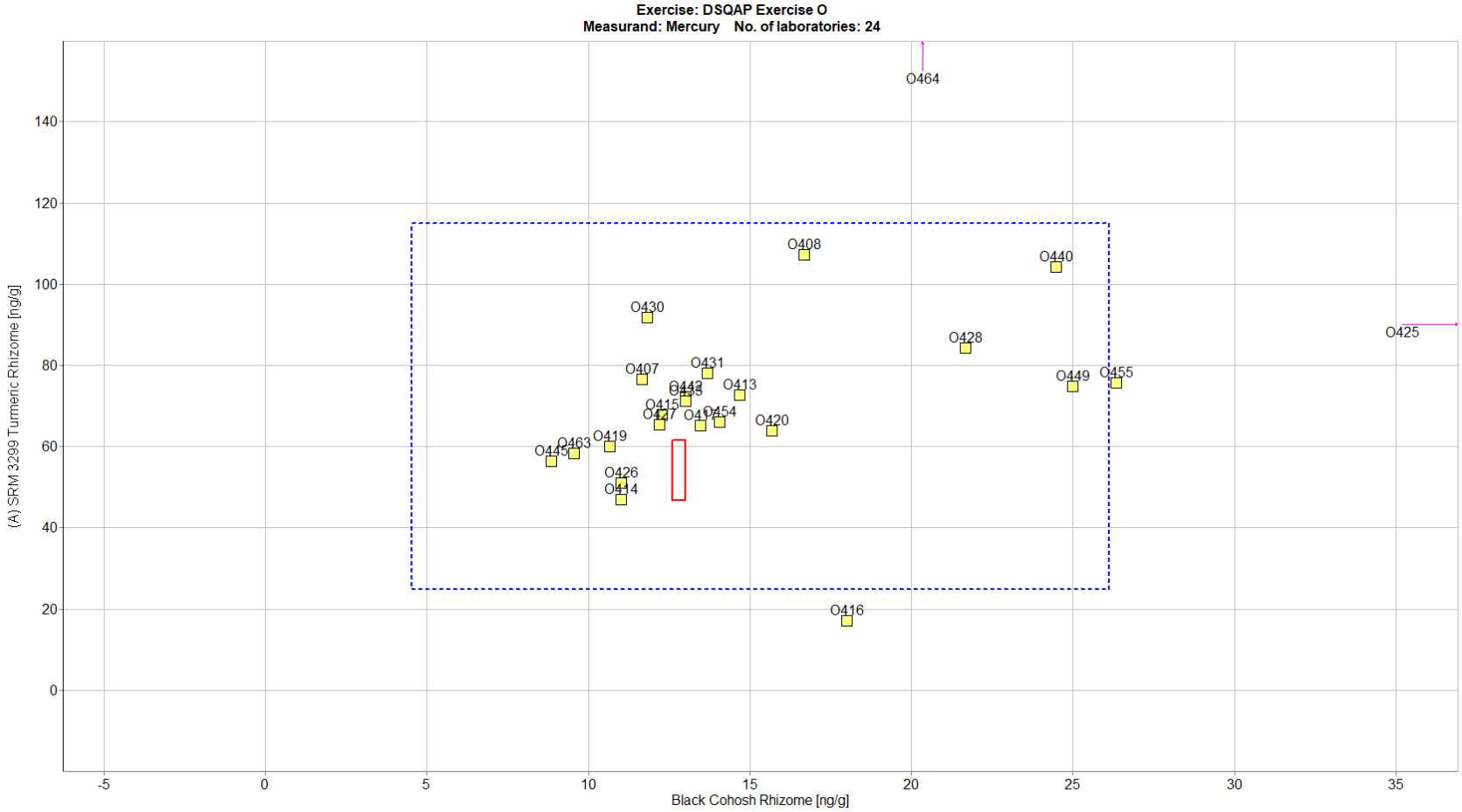


**Figure 1-20.** Laboratory means for mercury in black cohosh rhizome and turmeric rhizome (sample/sample comparison view). In this view, the individual laboratory mean for one sample (black cohosh) is compared to the mean for a second sample (turmeric). The solid red box represents the NIST range of tolerance for the two samples, black cohosh (x-axis) and turmeric (y-axis), which encompasses the NIST-determined values bounded by their uncertainties ($U_{\mathrm{NIST}}$) and represents the range that results in an acceptable $Z_{\mathrm{NIST}}$ score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for black cohosh (x-axis) and turmeric (y-axis), calculated as the values above and below the consensus means that result in an acceptable $Z'_{\mathrm{comm}}$ score, $|Z'_{\mathrm{comm}}| \leq 2$.

## SECTION 2: CURCUMINOIDS IN TURMERIC COMMERICAL PRODUCTS

Study Overview

For this two-part curcuminoid study, participants initially were provided with two NIST candidate SRMs, SRM 3299 Ground Turmeric (*Curcuma longa* L.) Rhizome and SRM 3300 Curcumin Extract of Turmeric (*Curcuma longa* L.) Rhizome, and four of eight turmeric commercial products. Participants were asked to use the AOAC First Action *Official Method of Analysis 2016.16 Determination of Curcuminoids in Turmeric Raw Materials and Dietary Supplements by HPLC*[6] or in-house methods to determine the mass fractions (mg/g or mg/L) of curcumin, bisdemethoxycurcumin (BDMC), and desmethoxycurcumin (DMC) in each matrix. For those laboratories interested in using the AOAC method, a copy of the method was enclosed, and participants were advised to follow the method exactly. For the second part of this study, participants using the AOAC method received the same two candidate NIST SRMs and the remaining four products not received in the first part of the study, such that all of the selected laboratories received two sets of the candidate NIST SRMs and all eight commercial products. Data from laboratories using the AOAC method was included in a collaborative study effort to evaluate the reproducibility of the method to support *Final Action* status. For participants using an in-house method, results were compared with the consensus data.

Sample Information

*Turmeric Rhizome.* Participants were provided with 1 packet of ground turmeric rhizome. Before use, participants were instructed to thoroughly mix the contents of the packet and were instructed to use a minimum sample size as described in AOAC 2016.16. 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. Approximate analyte levels were not reported to participants prior to the study. The target values for curcuminoids in the turmeric rhizome were determined at NIST using liquid chromatography with absorbance detection (LC-absorbance). The NIST-determined values and uncertainties for curcuminoids in the turmeric rhizome are provided in the table below.

| Analyte | NIST-Determined Mass Fraction in Candidate SRM 3299 (mg/g) |
|---|---|
| Curcumin | 11.04 ± 0.21 |
| Bisdemethoxycurcumin | 2.84 ± 0.05 |
| Desmethoxycurcumin | 3.14 ± 0.06 |

*Turmeric Extract.* Participants were provided with 1 packet of turmeric extract powder. Before use, participants were instructed to thoroughly mix the contents of the packet and were instructed to use a minimum sample size as described in AOAC 2016.16. 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. Approximate analyte levels were not reported to participants prior to the study. The target values for curcuminoids in the turmeric extract were

[6]Mudge, E.M.; Brown, P. N. (2018) *Determination of Curcuminoids in Turmeric Raw Materials and Dietary Supplements by HPLC: Single-Laboratory Validation, First Action 2016.16.* J AOAC Int. 101 (1), pp 203-207.

determined at NIST using LC-absorbance. The NIST-determined values and uncertainties for curcuminoids in the turmeric extract are provided in the table below.

| Analyte | NIST-Determined Mass Fraction in Candidate SRM 3300 (mg/g) |
|---|---|
| Curcumin | 822 ± 11 |
| Bisdemethoxycurcumin | 18.25 ± 0.49 |
| Desmethoxycurcumin | 117.1 ± 1.1 |

*Turmeric Commercial Products.* Participants received some or all the samples listed in the table below. Before use, participants were instructed to thoroughly mix the contents of each packet or vial and were instructed to use a minimum sample size as described in AOAC 2016.16. Participants were asked to store the materials at controlled room temperature, 20 °C to 25 °C, and to prepare the number of samples and report the number of values as described in the table below. The approximate analyte levels were not reported to participants prior to the study, and no values for curcuminoids in these products were determined by NIST prior to the study.

| Sample ID | Quantity and Packaging | Quantity per Package | How to Report |
|---|---|---|---|
| Sample C: Turmeric Root Powder | 3 packets | 3 g of powder | Prepare 1 sample and report 1 value per packet |
| Sample D: Turmeric Smoothie Additive | 3 packets | 3 g of powder | Prepare 1 sample and report 1 value per packet |
| Sample E: Turmeric Root Capsule | 3 packets | 20 capsules | Prepare 1 sample and report 1 value per packet |
| Sample F: Turmeric Extract/Root Capsule with Black Pepper | 3 packets | 20 capsules | Prepare 1 sample and report 1 value per packet |
| Sample G: Turmeric Extract/Root Capsule with Black Pepper & Coconut Oil | 3 packets | 20 capsules | Prepare 1 sample and report 1 value per packet |
| Sample H: Turmeric Tincture | 3 vials | 3 mL of liquid | Prepare 1 sample and report 1 value per vial |
| Sample I: Turmeric Gelcap with Coconut | 1 packet | 20 capsules | Prepare 3 samples and report 3 values from the single packet |
| Sample J: Turmeric Gelcap, Liquid Curcumin | 1 packet | 20 capsules | Prepare 3 samples and report 3 values from the single packet |

Study Results

- Twenty-four of the thirty-four laboratories enrolled in the exercise reported results for the samples that they received (71 % participation).
- For curcumin, the 95 % confidence intervals for the consensus mean in both turmeric candidate SRMs overlapped the NIST target ranges, as illustrated in **Figures 2-1 and 2-2**. The consensus mean for candidate SRM 3299 was within the NIST target range while the consensus mean for candidate SRM 3300 fell below the NIST target range.
- For BDMC, the 95 % confidence interval for the consensus mean in candidate SRM 3299 overlapped the NIST target range as illustrated in **Figure 2-22**, but the consensus mean was above the target range. The consensus range overlapped the NIST target range for candidate SRM 3300 and the consensus mean was within the NIST target range as illustrated in **Figure 2-23**.
- For DMC, the 95 % confidence intervals for the consensus mean in both turmeric candidate SRMs overlapped the NIST target ranges as illustrated in **Figures 2-43 and 2-44**. The consensus mean was above the NIST target range for candidate SRM 3299 and was within the NIST target range for candidate SRM 3300.
- The between-laboratory variability was acceptable for most analyte-sample pairs, as indicated in the table below. The variability generally decreased when considering only the laboratories using AOAC 2016.16.

| | Between-Laboratory Variability (RSD) | | | | | |
|---|---|---|---|---|---|---|
| | All Laboratories | | | Laboratories using AOAC 2016.16 | | |
| Sample | Curcumin | BDMC | DMC | Curcumin | BDMC | DMC |
| Sample A: SRM 3299 Turmeric Rhizome | 16.1 % | 20.9 % | 21.3 % | 9.4 % | 7.8 % | 7.3 % |
| Sample B: SRM 3300 Turmeric Extract | 7.2 % | 15.3 % | 8.1 % | 5.8 % | 9.1 % | 6.7 % |
| Sample C: Turmeric Root Powder | 12.0 % | 10.3 % | 16.2 % | 4.6 % | 5.5 % | 6.9 % |
| Sample D: Turmeric Smoothie Additive | 10.9 % | 27.7 % | 14.6 % | 13.8 % | 37.4 % | 15.8 % |
| Sample E: Turmeric Root Capsule | 7.8 % | 18.8 % | 12.1 % | 7.2 % | 10.4 % | 6.1 % |
| Sample F: Turmeric Extract/Root Capsule with Black Pepper | 8.3 % | 22.7 % | 20.1 % | 3.9 % | 12.6 % | 8.1 % |
| Sample G: Turmeric Extract/Root Capsule with Black Pepper & Coconut Oil | 10.0 % | 21.3 % | 18.9 % | 9.4 % | 19.2 % | 18.7 % |
| Sample H: Turmeric Tincture | 86.4 % | 85.7 % | 77.3 % | 21.9 % | 23.6 % | 21.4 % |
| Sample I: Turmeric Gelcap with Coconut | 12.4 % | 35.7 % | 15.2 % | 7.0 % | 13.7 % | 8.0 % |
| Sample J: Turmeric Gelcap, Liquid Curcumin | 9.6 % | 19.8 % | 8.4 % | 8.2 % | 17.6 % | 5.7 % |

- Variability was highest for the tincture sample (over 75 % RSD), but was reduced when laboratories were using the same method.
- In general, variability was lowest for curcumin, which is present in the highest concentration in most samples.

- A variety of analytical methods were reported for determination of curcuminoids in the turmeric samples.
  - Twenty-two laboratories (65 %) reported using LC-absorbance approaches for determination of curcumin in the turmeric samples.
    - Ten laboratories (29 %) reported using AOAC 2016.16, an LC-absorbance technique, as their analytical method for the turmeric samples.
    - Twelve additional laboratories (35 %) reported using a different LC-absorbance technique.
  - One laboratory (3 %) reported using an LC approach but did not specify the detection method.
  - One laboratory (3 %) reported using LC with fluorescence detection for determination of curcumin in the turmeric samples.
  - One laboratory (3 %) reported using high performance thin-layer chromatography (HPTLC) for determination of curcumin in the turmeric samples.



Technical Recommendations

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

- No specific trends could be noted based on the analytical methods used by participants.
  - Most laboratories reported using an LC-absorbance approach for determination of the curcuminoids in the various turmeric samples. In general, use of AOAC 2016.16 or another LC-absorbance approach gave comparable results.
  - Results reported using an LC-fluorescence approach were biased high with respect to the consensus for one sample, and biased low for a second sample.
  - Results reported using HPTLC were also biased high and low for different samples.
- Several of the sample/sample comparison view plots indicate an upward linear trend, in which the bias of laboratory values is consistent among multiple samples.
  - Such a trend may indicate overall calibration issues within each laboratory.
  - The purity of calibration standards should be evaluated or confirmed in-house prior to quantitative measurements. For best results, use a combination of methods that can provide information about various types of possible impurities (LC-absorbance, mass spectrometry, Karl-Fischer or thermogravimetry to determine moisture content, etc.).
- The quality of the separation is critical for commercial samples, to ensure that potential coeluting compounds in each unique matrix are identified and removed prior to final analysis. Coeluting compounds are a common source of a positive bias in results.
- Inefficient extraction is a common reason for values biased low with respect to the target or consensus ranges.
  - For samples that originate from turmeric rhizome or root, extraction of curcuminoids may require significant sample preparation to isolate compounds of interest. Steps to consider include sample homogenization, extraction time, extraction solvent, and extraction temperature, as well as number of required extraction cycles. Low results may be the result of curcuminoids not being fully isolated from the matrix.

    - For highly concentrated samples such as extracts, the solubility limit for curcuminoids (particularly curcumin) may easily be reached during sample preparation. Additional extraction cycles may be useful to achieve maximum accuracy.
- The largest variability was observed for the smoothie additive, the capsules containing black pepper, and the tincture.
    - Measurement of curcuminoids in these matrices may be more challenging than in other matrices.
    - Inhomogeneity of the sample matrix may also result in higher variability. To avoid issues with sample homogeneity, samples should be thoroughly blended prior to sampling. For curcumin, the within-laboratory repeatability was high for the smoothie additive and the black pepper-containing samples, which supports sample inhomogeneity as a cause for higher variability.
- Use of matrix-matched CRMs for method validation and quality assurance of the measurement process is recommended.

**Table 2-1.** NIST data summary table for curcumin, bisdemethoxycurcumin, and desmethoxycurcumin in turmeric commercial products.

***National Institute of Standards and Technology***

**DSQAP Exercise O - Botanicals**

| | | **Lab Code:** | **NIST** | **1. Your Results** | | | | **2. Community Results** | | | **3. Target** | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Analyte | Sample | | Units | $x_i$ | $s_i$ | $Z'_{comm}$ | $Z_{NIST}$ | N | x* | s* | $x_{NIST}$ | *U* |
| Bisdemethoxycurcumin | (A) SRM 3299 Turmeric Rhizome | | mg/g | 3.39 | 0.11 | | | 22 | 3.16 | 0.16 | 3.39 | 0.109 |
| Bisdemethoxycurcumin | (B) SRM 3300 Turmeric Extract | | mg/g | 18.25 | 0.98 | | | 23 | 17.3 | 0.58 | 18.2 | 0.98 |
| Bisdemethoxycurcumin | (C) Turmeric Root Powder | | mg/g | | | | | 14 | 10.8 | 0.33 | | |
| Bisdemethoxycurcumin | (D) Turmeric Smoothie Additive | | mg/g | | | | | 10 | 0.763 | 0.085 | | |
| Bisdemethoxycurcumin | (E) Turmeric Root Capsule | | mg/g | | | | | 12 | 4.41 | 0.29 | | |
| Bisdemethoxycurcumin | (F) Turmeric Extract/Root Capsule with Black Pepper | | mg/g | | | | | 12 | 3.23 | 0.27 | | |
| Bisdemethoxycurcumin | (G) Turmeric Extract/Root Capsule with Black Pepper & Coconut Oil | | mg/g | | | | | 13 | 11.9 | 0.74 | | |
| Bisdemethoxycurcumin | (H) Turmeric Tincture | | mg/L | | | | | 13 | 160 | 39 | | |
| Bisdemethoxycurcumin | (I) Turmeric Gelcap with Coconut | | mg/g | | | | | 13 | 1.29 | 0.14 | | |
| Bisdemethoxycurcumin | (J) Turmeric Gelcap, Liquid Curcumin | | mg/g | | | | | 12 | 1.88 | 0.13 | | |
| CURCUMIN | (A) SRM 3299 Turmeric Rhizome | | mg/g | 11.17 | 0.43 | | | 24 | 11.2 | 0.38 | 11.2 | 0.428 |
| CURCUMIN | (B) SRM 3300 Turmeric Extract | | mg/g | 820 | 22 | | | 25 | 790 | 10 | 822 | 22 |
| CURCUMIN | (C) Turmeric Root Powder | | mg/g | | | | | 16 | 16.4 | 0.56 | | |
| CURCUMIN | (D) Turmeric Smoothie Additive | | mg/g | | | | | 11 | 9.6 | 0.27 | | |
| CURCUMIN | (E) Turmeric Root Capsule | | mg/g | | | | | 12 | 18.1 | 0.4 | | |
| CURCUMIN | (F) Turmeric Extract/Root Capsule with Black Pepper | | mg/g | | | | | 12 | 48.3 | 1.1 | | |
| CURCUMIN | (G) Turmeric Extract/Root Capsule with Black Pepper & Coconut Oil | | mg/g | | | | | 15 | 295 | 7.9 | | |
| CURCUMIN | (H) Turmeric Tincture | | mg/L | | | | | 14 | 370 | 85 | | |
| CURCUMIN | (I) Turmeric Gelcap with Coconut | | mg/g | | | | | 15 | 24 | 0.77 | | |
| CURCUMIN | (J) Turmeric Gelcap, Liquid Curcumin | | mg/g | | | | | 12 | 44.6 | 1.2 | | |
| Desmethoxycurcumin | (A) SRM 3299 Turmeric Rhizome | | mg/g | 3.63 | 0.13 | | | 22 | 3.39 | 0.16 | 3.63 | 0.128 |
| Desmethoxycurcumin | (B) SRM 3300 Turmeric Extract | | mg/g | 117 | 2.2 | | | 23 | 118 | 2.5 | 117 | 2.2 |
| Desmethoxycurcumin | (C) Turmeric Root Powder | | mg/g | | | | | 14 | 8.2 | 0.49 | | |
| Desmethoxycurcumin | (D) Turmeric Smoothie Additive | | mg/g | | | | | 11 | 1.92 | 0.088 | | |
| Desmethoxycurcumin | (E) Turmeric Root Capsule | | mg/g | | | | | 12 | 5.74 | 0.24 | | |
| Desmethoxycurcumin | (F) Turmeric Extract/Root Capsule with Black Pepper | | mg/g | | | | | 12 | 4.34 | 0.29 | | |
| Desmethoxycurcumin | (G) Turmeric Extract/Root Capsule with Black Pepper & Coconut Oil | | mg/g | | | | | 13 | 60.6 | 3.1 | | |
| Desmethoxycurcumin | (H) Turmeric Tincture | | mg/L | | | | | 13 | 160 | 38 | | |
| Desmethoxycurcumin | (I) Turmeric Gelcap with Coconut | | mg/g | | | | | 13 | 5.06 | 0.36 | | |
| Desmethoxycurcumin | (J) Turmeric Gelcap, Liquid Curcumin | | mg/g | | | | | 12 | 10.3 | 0.25 | | |

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

**Table 2-2.1.** Data summary table for curcumin in turmeric commercial products. Individual results are displayed in this table for ten of the laboratories that requested samples (O404 through O415), while community results are shown for all laboratories participating the study. Results for additional laboratories can be found in Tables 2-2.2 through 2-2.4. Data highlighted in red have been flagged as potential outliers (e.g., Grubb and/or Cochran) by the NIST software package.

| | | Curcumin | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Individual Results - Page 1 of 4 | | | | | | | | | | | Community Results | | | | | |
| | Lab | NIST | O404 | O405 | O406 | O407 | O409 | O410 | O411 | O412 | O414 | O415 | Mean | SD | % RSD | Max | Min | N |
| Sample A: SRM 3299 Turmeric Rhizome (mg/g) | A | | 11.26 | | 10.60 | 9.71 | | 23.83 | | | 11.00 | 14.00 | | | | | | |
| | B | | 11.30 | | 10.50 | 9.71 | | 23.63 | | | 11.00 | 13.90 | | | | | | |
| | C | | 11.41 | | 10.70 | 9.73 | | 23.71 | | | 11.00 | 13.90 | | | | | | |
| | D | | 10.26 | | | 10.80 | | | | | | | | | | | | |
| | E | | 11.12 | | | 10.20 | | | | | | | | | | | | |
| | F | | 10.58 | | | 10.20 | | | | | | | | | | | | |
| | Avg | 11.04 | 10.99 | | 10.60 | 10.06 | | 23.72 | | | 11.00 | 13.93 | 11.1 | 1.8 | 16.1% | 23.7 | 5.6 | 21 |
| | SD | 0.21 | 0.46 | | 0.10 | 0.43 | | 0.10 | | | 0.00 | 0.06 | | | | | | |
| Sample B: SRM 3300 Turmeric Extract (mg/g) | A | | 802 | | 858 | 871 | | 507 | | | 795 | 780 | | | | | | |
| | B | | 750 | | 864 | 793 | | 501 | | | 795 | 777 | | | | | | |
| | C | | 731 | | 870 | 845 | | 492 | | | 802 | 774 | | | | | | |
| | D | | 801 | | | 872 | | | | | | | | | | | | |
| | E | | 801 | | | 883 | | | | | | | | | | | | |
| | F | | 816 | | | 875 | | | | | | | | | | | | |
| | Avg | 822 | 784 | | 864 | 857 | | 500 | | | 797 | 777 | 791 | 57 | 7.2% | 867 | 81 | 22 |
| | SD | 11 | 34 | | 6 | 34 | | 7 | | | 4 | 3 | | | | | | |
| Sample C: Turmeric Root Powder (mg/g) | A | | 15.52 | | 15.30 | 13.80 | | 33.96 | 16.98 | | | 17.90 | | | | | | |
| | B | | 15.52 | | 14.90 | 14.90 | | 34.22 | 16.56 | | | 17.80 | | | | | | |
| | C | | 15.76 | | 15.30 | 15.10 | | 33.65 | 15.92 | | | 18.10 | | | | | | |
| | Avg | | 15.60 | | 15.17 | 14.60 | | 33.94 | 16.49 | | | 17.93 | 16.1 | 1.9 | 12.0% | 33.9 | 13.6 | 18 |
| | SD | | 0.14 | | 0.23 | 0.70 | | 0.29 | 0.53 | | | 0.15 | | | | | | |
| Sample D: Turmeric Smoothie Additive (mg/g) | A | | 11.55 | | | 8.83 | | | | | 9.00 | | | | | | | |
| | B | | 11.13 | | | 9.11 | | | | | 10.00 | | | | | | | |
| | C | | 10.97 | | | 9.41 | | | | | 10.00 | | | | | | | |
| | Avg | | 11.22 | | | 9.12 | | | | | 9.67 | | 9.4 | 1.0 | 10.9% | 12.7 | 6.7 | 14 |
| | SD | | 0.30 | | | 0.29 | | | | | 0.58 | | | | | | | |
| Sample E: Turmeric Root Capsule (mg/g) | A | | 19.56 | | | 18.80 | | | | | 18.00 | | | | | | | |
| | B | | 19.77 | | | 18.90 | | | | | 18.00 | | | | | | | |
| | C | | 19.51 | | | 18.40 | | | | | 18.00 | | | | | | | |
| | Avg | | 19.61 | | | 18.70 | | | | | 18.00 | | 18.0 | 1.4 | 7.8% | 22.2 | 14.0 | 15 |
| | SD | | 0.14 | | | 0.26 | | | | | 0.00 | | | | | | | |
| Sample F: Turmeric Extract/Root Capsule with Black Pepper (mg/g) | A | | 50.60 | | | 52.40 | | | | | 50.00 | | | | | | | |
| | B | | 48.58 | | | 52.20 | | | | | 51.00 | | | | | | | |
| | C | | 49.23 | | | 52.60 | | | | | 50.00 | | | | | | | |
| | Avg | | 49.47 | | | 52.40 | | | | | 50.33 | | 48.4 | 4.0 | 8.3% | 54.6 | 27.6 | 15 |
| | SD | | 1.03 | | | 0.20 | | | | | 0.58 | | | | | | | |
| Sample G: Turmeric Extract/Root Capsule with Black Pepper & Coconut Oil (mg/g) | A | | | | 317 | 154 | | 307 | | | | 278 | | | | | | |
| | B | | | | 316 | 234 | | 174 | | | | 304 | | | | | | |
| | C | | | | 306 | 276 | | 286 | | | | 293 | | | | | | |
| | Avg | | | | 313 | 221 | | 255 | | | | 292 | 295 | 30 | 10.0% | 352 | 221 | 16 |
| | SD | | | | 6 | 62 | | 71 | | | | 13 | | | | | | |
| Sample H: Turmeric Tincture (mg/L) | A | | 666 | | 0.586 | 502 | | 1.35 | | | | 4790 | | | | | | |
| | B | | 802 | | 0.584 | 497 | | 1.30 | | | | 4940 | | | | | | |
| | C | | 705 | | 0.613 | 500 | | 1.28 | | | | 4520 | | | | | | |
| | Avg | | 725 | | 0.594 | 500 | | 1.31 | | | | 4750 | 371 | 321 | 86.4% | 4750 | 1 | 16 |
| | SD | | 70 | | 0.016 | 3 | | 0.04 | | | | 213 | | | | | | |
| Sample I: Turmeric Gelcap with Coconut (mg/g) | A | | | | 25.2 | 22.5 | | 48.2 | | | | 26.5 | | | | | | |
| | B | | | | 24.2 | 23.7 | | 49.2 | | | | 26.2 | | | | | | |
| | C | | | | 23.9 | 24.5 | | 45.3 | | | | 28.1 | | | | | | |
| | Avg | | | | 24.4 | 23.6 | | 47.6 | | | | 26.9 | 24.0 | 3.0 | 12.4% | 47.6 | 15.5 | 15 |
| | SD | | | | 0.7 | 1.0 | | 2.0 | | | | 1.0 | | | | | | |
| Sample J: Turmeric Gelcap, Liquid Curcumin (mg/g) | A | | 47.1 | | | 45.3 | | | | | 46.0 | | | | | | | |
| | B | | 47.2 | | | 47.0 | | | | | 47.0 | | | | | | | |
| | C | | 46.1 | | | 46.7 | | | | | 46.0 | | | | | | | |
| | Avg | | 46.8 | | | 46.3 | | | | | 46.3 | | 44.3 | 4.3 | 9.6% | 54.1 | 37.6 | 15 |
| | SD | | 0.6 | | | 0.9 | | | | | 0.6 | | | | | | | |

**Table 2-2.2.** Data summary table for curcumin in turmeric commercial products. Individual results are displayed in this table for ten of the laboratories that requested samples (O416 through O431), while community results are shown for all laboratories participating the study. Results for additional laboratories can be found in Tables 2-2.1, 2-2.3, and 2-2.4. Data highlighted in red have been flagged as potential outliers (e.g., Grubb and/or Cochran) by the NIST software package.

| | | Curcumin | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Individual Results - Page 2 of 4 | | | | | | | | | | | Community Results | | | | | |
| | Lab | NIST | O416 | O419 | O420 | O421 | O423 | O425 | O426 | O428 | O429 | O431 | Mean | SD | % RSD | Max | Min | N |
| Sample A: SRM 3299 Turmeric Rhizome (mg/g) | A | | 12.19 | 11.11 | 12.05 | 5.61 | | | 9.25 | | | | | | | | | |
| | B | | 12.92 | 11.50 | 12.13 | 5.54 | | | 9.50 | | | | | | | | | |
| | C | | 12.61 | 11.54 | 12.70 | 5.78 | | | 9.70 | | | | | | | | | |
| | D | | 11.51 | 10.86 | | | | | | | | | | | | | | |
| | E | | 11.20 | 10.88 | | | | | | | | | | | | | | |
| | F | | 11.88 | 10.98 | | | | | | | | | | | | | | |
| | Avg | 11.04 | 12.05 | 11.15 | 12.29 | 5.64 | | | 9.48 | | | | 11.1 | 1.8 | 16.1% | 23.7 | 5.6 | 21 |
| | SD | 0.21 | 0.65 | 0.30 | 0.35 | 0.12 | | | 0.23 | | | | | | | | | |
| Sample B: SRM 3300 Turmeric Extract (mg/g) | A | | 796 | 807 | 817 | 727 | | 777 | 420 | | | | | | | | | |
| | B | | 866 | 799 | 816 | 722 | | | 418 | | | | | | | | | |
| | C | | 755 | 831 | 815 | 728 | | 777 | 418 | | | | | | | | | |
| | D | | 835 | 791 | | | | | | | | | | | | | | |
| | E | | 846 | 802 | | | | | | | | | | | | | | |
| | F | | 825 | 807 | | | | | | | | | | | | | | |
| | Avg | 822 | 820 | 806 | 816 | 726 | | 777 | 419 | | | | 791 | 57 | 7.2% | 867 | 419 | 22 |
| | SD | 11 | 40 | 14 | 1 | 3 | | 0 | 1 | | | | | | | | | |
| Sample C: Turmeric Root Powder (mg/g) | A | | 15.2 | 15.28 | 17.52 | | | | 16.40 | | | | | | | | | |
| | B | | 16.4 | 15.43 | 18.20 | | | | 16.40 | | | | | | | | | |
| | C | | 15.0 | 15.38 | 17.97 | | | | 17.00 | | | | | | | | | |
| | Avg | | 15.5 | 15.36 | 17.90 | | | | 16.60 | | | | 16.1 | 1.9 | 12.0% | 33.9 | 13.6 | 18 |
| | SD | | 0.8 | 0.07 | 0.35 | | | | 0.35 | | | | | | | | | |
| Sample D: Turmeric Smoothie Additive (mg/g) | A | | 9.20 | 9.83 | | 6.88 | | | | | | | | | | | | |
| | B | | 8.47 | 9.94 | | 6.61 | | | | | | | | | | | | |
| | C | | 8.10 | 9.99 | | 6.64 | | | | | | | | | | | | |
| | Avg | | 8.59 | 9.92 | | 6.71 | | | | | | | 9.4 | 1.0 | 10.9% | 11.2 | 6.7 | 14 |
| | SD | | 0.56 | 0.08 | | 0.15 | | | | | | | | | | | | |
| Sample E: Turmeric Root Capsule (mg/g) | A | | 18.70 | 19.21 | | 14.19 | | 18.17 | | | | | | | | | | |
| | B | | 18.00 | 19.32 | | 13.89 | | 17.27 | | | | | | | | | | |
| | C | | 17.62 | 19.36 | | 13.84 | | 17.38 | | | | | | | | | | |
| | Avg | | 18.11 | 19.30 | | 13.97 | | 17.61 | | | | | 18.0 | 1.4 | 7.8% | 19.6 | 14.0 | 15 |
| | SD | | 0.55 | 0.08 | | 0.19 | | 0.49 | | | | | | | | | | |
| Sample F: Turmeric Extract/Root Capsule with Black Pepper (mg/g) | A | | 52.78 | 50.89 | | 28.10 | | 44.23 | | | | | | | | | | |
| | B | | 50.03 | 51.86 | | 26.69 | | 44.43 | | | | | | | | | | |
| | C | | 50.00 | 51.96 | | 28.06 | | 44.87 | | | | | | | | | | |
| | Avg | | 50.94 | 51.57 | | 27.62 | | 44.51 | | | | | 48.4 | 4.0 | 8.3% | 53.4 | 27.6 | 15 |
| | SD | | 1.60 | 0.59 | | 0.80 | | 0.33 | | | | | | | | | | |
| Sample G: Turmeric Extract/Root Capsule with Black Pepper & Coconut Oil (mg/g) | A | | 249 | 304 | 309 | | | | 315 | | | | | | | | | |
| | B | | 299 | 306 | 305 | | | | 312 | | | | | | | | | |
| | C | | 292 | 306 | 310 | | | | 321 | | | | | | | | | |
| | Avg | | 280 | 305 | 308 | | | | 316 | | | | 295 | 30 | 10.0% | 352 | 221 | 16 |
| | SD | | 27 | 1 | 3 | | | | 5 | | | | | | | | | |
| Sample H: Turmeric Tincture (mg/L) | A | | 466 | 372 | 1.53 | | | | 580 | | | | | | | | | |
| | B | | 522 | 372 | 1.49 | | | | 620 | | | | | | | | | |
| | C | | 410 | 366 | 1.53 | | | | 570 | | | | | | | | | |
| | Avg | | 466 | 370 | 1.52 | | | | 590 | | | | 371 | 321 | 86.4% | 4750 | 1 | 16 |
| | SD | | 56 | 3 | 0.02 | | | | 26 | | | | | | | | | |
| Sample I: Turmeric Gelcap with Coconut (mg/g) | A | | 22.4 | 24.2 | 23.8 | | | | 26.5 | | | | | | | | | |
| | B | | 22.8 | 24.2 | 24.4 | | | | 28.8 | | | | | | | | | |
| | C | | 20.3 | 23.9 | 23.6 | | | | 29.7 | | | | | | | | | |
| | Avg | | 21.8 | 24.1 | 23.9 | | | | 28.3 | | | | 24.0 | 3.0 | 12.4% | 47.6 | 15.5 | 15 |
| | SD | | 1.3 | 0.2 | 0.4 | | | | 1.7 | | | | | | | | | |
| Sample J: Turmeric Gelcap, Liquid Curcumin (mg/g) | A | | 42.0 | 47.5 | | 38.4 | | 35.8 | | | | | | | | | | |
| | B | | 44.9 | 47.4 | | 38.2 | | 38.7 | | | | | | | | | | |
| | C | | 43.0 | 46.9 | | 38.1 | | 38.4 | | | | | | | | | | |
| | Avg | | 43.3 | 47.3 | | 38.2 | | 37.6 | | | | | 44.3 | 4.3 | 9.6% | 48.5 | 37.6 | 15 |
| | SD | | 1.5 | 0.4 | | 0.1 | | 1.6 | | | | | | | | | | |

**Table 2-2.3.** Data summary table for curcumin in turmeric commercial products. Individual results are displayed in this table for ten of the laboratories that requested samples (O433 through O458), while community results are shown for all laboratories participating the study. Results for additional laboratories can be found in Tables 2-2.1, 2-2.2, and 2-2.4.

| | | Curcumin | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Individual Results - Page 3 of 4 | | | | | | | | | | | Community Results | | | | | |
| | Lab | NIST | O433 | O434 | O437 | O440 | O443 | O446 | O449 | O452 | O455 | O458 | Mean | SD | % RSD | Max | Min | N |
| Sample A: SRM 3299 Turmeric Rhizome (mg/g) | A | | 10.22 | | 8.78 | 10.65 | | 10.93 | 11.59 | 12.49 | 10.87 | | | | | | | |
| | B | | 9.99 | | 8.92 | | | 10.65 | 12.10 | 12.45 | 10.63 | | | | | | | |
| | C | | 9.82 | | 9.16 | | | 10.76 | 11.00 | 11.98 | 11.01 | | | | | | | |
| | D | | | | | | | 9.71 | 10.13 | | 11.04 | | | | | | | |
| | E | | | | | | | 9.24 | 10.38 | | 11.17 | | | | | | | |
| | F | | | | | | | 8.91 | 10.89 | | 11.15 | | | | | | | |
| | Avg | 11.04 | 10.01 | | 8.95 | 10.65 | | 10.03 | 11.01 | 12.30 | 10.98 | | 11.1 | 1.8 | 16.1% | 23.7 | 5.6 | 21 |
| | SD | 0.21 | 0.20 | | 0.19 | | | 0.86 | 0.73 | 0.29 | 0.20 | | | | | | | |
| Sample B: SRM 3300 Turmeric Extract (mg/g) | A | | 787 | | 745 | 732 | | 765 | 852 | 851 | 841 | | | | | | | |
| | B | | 784 | | 774 | | | 799 | 867 | 820 | 850 | | | | | | | |
| | C | | 781 | | 769 | | | 772 | 871 | 827 | 860 | | | | | | | |
| | D | | | | | | | 570 | 807 | | 827 | | | | | | | |
| | E | | | | | | | 610 | 822 | | 832 | | | | | | | |
| | F | | | | | | | 604 | 802 | | 830 | | | | | | | |
| | Avg | 822 | 784 | | 763 | 732 | | 687 | 837 | 833 | 840 | | 791 | 57 | 7.2% | 867 | 419 | 22 |
| | SD | 11 | 3 | | 15 | | | 103 | 31 | 16 | 13 | | | | | | | |
| Sample C: Turmeric Root Powder (mg/g) | A | | 14.39 | | 13.50 | 15.00 | | 15.06 | 15.08 | | 15.04 | | | | | | | |
| | B | | 14.03 | | 13.50 | 14.80 | | 14.90 | 16.16 | | 14.96 | | | | | | | |
| | C | | 14.27 | | 13.70 | 14.80 | | 14.83 | 15.10 | | 15.24 | | | | | | | |
| | Avg | | 14.23 | | 13.57 | 14.87 | | 14.93 | 15.44 | | 15.08 | | 16.1 | 1.9 | 12.0% | 33.9 | 13.6 | 18 |
| | SD | | 0.18 | | 0.12 | 0.12 | | 0.12 | 0.62 | | 0.14 | | | | | | | |
| Sample D: Turmeric Smoothie Additive (mg/g) | A | | 9.39 | | 8.83 | | | 7.68 | 9.21 | 9.82 | 9.82 | | | | | | | |
| | B | | 9.33 | | 8.22 | | | 7.54 | 9.58 | 9.87 | 9.26 | | | | | | | |
| | C | | 13.20 | | 8.31 | | | 7.42 | 10.07 | 9.76 | 9.33 | | | | | | | |
| | Avg | | 10.64 | | 8.45 | | | 7.55 | 9.62 | 9.81 | 9.47 | | 9.4 | 1.0 | 10.9% | 11.2 | 6.7 | 14 |
| | SD | | 2.22 | | 0.33 | | | 0.13 | 0.43 | 0.06 | 0.30 | | | | | | | |
| Sample E: Turmeric Root Capsule (mg/g) | A | | 17.32 | | 16.70 | | | 15.54 | 18.82 | 19.19 | 19.90 | | | | | | | |
| | B | | 17.94 | | 16.80 | | | 15.65 | 18.61 | 19.55 | 18.08 | | | | | | | |
| | C | | 17.65 | | 16.50 | | | 15.31 | 18.84 | 19.20 | 17.94 | | | | | | | |
| | Avg | | 17.64 | | 16.67 | | | 15.50 | 18.76 | 19.32 | 18.64 | | 18.0 | 1.4 | 7.8% | 19.6 | 14.0 | 15 |
| | SD | | 0.31 | | 0.15 | | | 0.18 | 0.13 | 0.20 | 1.09 | | | | | | | |
| Sample F: Turmeric Extract/Root Capsule with Black Pepper (mg/g) | A | | 49.16 | | 54.20 | | | 41.73 | 48.07 | 46.87 | 47.28 | | | | | | | |
| | B | | 50.04 | | 50.30 | | | 42.07 | 49.96 | 46.12 | 50.63 | | | | | | | |
| | C | | 47.86 | | 50.30 | | | 42.27 | 49.85 | 47.67 | 50.52 | | | | | | | |
| | Avg | | 49.02 | | 51.60 | | | 42.02 | 49.29 | 46.89 | 49.48 | | 48.4 | 4.0 | 8.3% | 53.4 | 27.6 | 15 |
| | SD | | 1.10 | | 2.25 | | | 0.27 | 1.06 | 0.77 | 1.90 | | | | | | | |
| Sample G: Turmeric Extract/Root Capsule with Black Pepper & Coconut Oil (mg/g) | A | | 302 | | 289 | 275 | | 296 | 347 | | 272 | | | | | | | |
| | B | | 246 | | 282 | 271 | | 294 | 349 | | 285 | | | | | | | |
| | C | | 293 | | 281 | 283 | | 293 | 361 | | 277 | | | | | | | |
| | Avg | | 280 | | 284 | 276 | | 294 | 352 | | 278 | | 295 | 30 | 10.0% | 352 | 221 | 16 |
| | SD | | 30 | | 4 | 6 | | 2 | 8 | | 6 | | | | | | | |
| Sample H: Turmeric Tincture (mg/L) | A | | 390 | | 439 | 16.6 | | 496 | 468 | | 252 | | | | | | | |
| | B | | 393 | | 428 | 16.4 | | 487 | 487 | | 259 | | | | | | | |
| | C | | 391 | | 422 | 16.3 | | 492 | 523 | | 250 | | | | | | | |
| | Avg | | 391 | | 430 | 16.4 | | 492 | 493 | | 253 | | 371 | 321 | 86.4% | 4750 | 1 | 16 |
| | SD | | 1 | | 9 | 0.1 | | 4 | 28 | | 5 | | | | | | | |
| Sample I: Turmeric Gelcap with Coconut (mg/g) | A | | 23.3 | | 21.5 | 22.9 | | 24.8 | 23.6 | | 22.3 | | | | | | | |
| | B | | 23.4 | | 22.0 | | | 23.2 | 26.1 | | 21.0 | | | | | | | |
| | C | | 21.5 | | 21.5 | | | 22.7 | 23.9 | | 19.6 | | | | | | | |
| | Avg | | 22.7 | | 21.7 | 22.9 | | 23.6 | 24.5 | | 21.0 | | 24.0 | 3.0 | 12.4% | 47.6 | 15.5 | 15 |
| | SD | | 1.1 | | 0.3 | | | 1.1 | 1.4 | | 1.4 | | | | | | | |
| Sample J: Turmeric Gelcap, Liquid Curcumin (mg/g) | A | | 41.7 | | 40.5 | | | 38.6 | 48.4 | 47.9 | 47.9 | | | | | | | |
| | B | | 42.8 | | 40.9 | | | 37.7 | 45.4 | 48.0 | 48.7 | | | | | | | |
| | C | | 42.9 | | 42.4 | | | 37.7 | 46.9 | 48.2 | 48.9 | | | | | | | |
| | Avg | | 42.5 | | 41.3 | | | 38.0 | 46.9 | 48.0 | 48.5 | | 44.3 | 4.3 | 9.6% | 48.5 | 37.6 | 15 |
| | SD | | 0.6 | | 1.0 | | | 0.5 | 1.5 | 0.1 | 0.5 | | | | | | | |

**Table 2-2.4.** Data summary table for curcumin in turmeric commercial products. Individual results are displayed in this table for ten of the laboratories that requested samples (O433 through O458), while community results are shown for all laboratories participating the study. Results for additional laboratories can be found in Tables 2-2.1 through 2-2.3. Data highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

| | | Curcumin | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Individual Results - Page 4 of 4 | | | | | Community Results | | | | | |
| | Lab | NIST | O459 | O460 | O461 | O462 | Mean | SD | % RSD | Max | Min | N |
| Sample A: SRM 3299 Turmeric Rhizome (mg/g) | A | | 6.92 | 11.46 | 13.22 | 16.40 | | | | | | |
| | B | | 5.95 | 11.30 | 12.69 | 13.10 | | | | | | |
| | C | | 5.82 | 11.47 | 12.84 | | | | | | | |
| | D | | | | | | | | | | | |
| | E | | | | | | | | | | | |
| | F | | | | | | | | | | | |
| | Avg | 11.04 | 6.23 | 11.41 | 12.92 | 14.75 | 11.1 | 1.8 | 16.1% | 23.7 | 5.6 | 21 |
| | SD | 0.21 | 0.60 | 0.10 | 0.27 | 2.33 | | | | | | |
| Sample B: SRM 3300 Turmeric Extract (mg/g) | A | | 779 | 803 | 871 | 800 | | | | | | |
| | B | | 768 | 802 | 872 | 745 | | | | | | |
| | C | | 774 | 816 | 858 | | | | | | | |
| | D | | | | | | | | | | | |
| | E | | | | | | | | | | | |
| | F | | | | | | | | | | | |
| | Avg | 822 | 774 | 807 | 867 | 773 | 791 | 57 | 7.2% | 867 | 419 | 22 |
| | SD | 11 | 5 | 8 | 8 | 39 | | | | | | |
| Sample C: Turmeric Root Powder (mg/g) | A | | | | 17.76 | 32.30 | | | | | | |
| | B | | | | 19.45 | 14.10 | | | | | | |
| | C | | | | 18.66 | 24.60 | | | | | | |
| | Avg | | | | 18.62 | 23.67 | 16.1 | 1.9 | 12.0% | 33.9 | 13.6 | 18 |
| | SD | | | | 0.85 | 9.14 | | | | | | |
| Sample D: Turmeric Smoothie Additive (mg/g) | A | | 8.89 | 9.50 | | | | | | | | |
| | B | | 9.21 | 9.92 | | | | | | | | |
| | C | | 9.64 | 9.40 | | | | | | | | |
| | Avg | | 9.25 | 9.61 | | | 9.4 | 1.0 | 10.9% | 12.7 | 6.7 | 14 |
| | SD | | 0.38 | 0.27 | | | | | | | | |
| Sample E: Turmeric Root Capsule (mg/g) | A | | 17.15 | 18.94 | | | | | | | | |
| | B | | 15.80 | 18.98 | | | | | | | | |
| | C | | 16.20 | 19.68 | | | | | | | | |
| | Avg | | 16.38 | 19.20 | | | 18.0 | 1.4 | 7.8% | 22.2 | 14.0 | 15 |
| | SD | | 0.69 | 0.42 | | | | | | | | |
| Sample F: Turmeric Extract/Root Capsule with Black Pepper (mg/g) | A | | 47.82 | 53.67 | | | | | | | | |
| | B | | 46.46 | 52.25 | | | | | | | | |
| | C | | 47.10 | 54.42 | | | | | | | | |
| | Avg | | 47.13 | 53.45 | | | 48.4 | 4.0 | 8.3% | 54.6 | 27.6 | 15 |
| | SD | | 0.68 | 1.10 | | | | | | | | |
| Sample G: Turmeric Extract/Root Capsule with Black Pepper & Coconut Oil (mg/g) | A | | | | 352 | 313 | | | | | | |
| | B | | | | 348 | 305 | | | | | | |
| | C | | | | 349 | 319 | | | | | | |
| | Avg | | | | 350 | 312 | 295 | 30 | 10.0% | 352 | 4 | 16 |
| | SD | | | | 2 | 7 | | | | | | |
| Sample H: Turmeric Tincture (mg/L) | A | | | | 745 | | | | | | | |
| | B | | | | 707 | | | | | | | |
| | C | | | | 745 | | | | | | | |
| | Avg | | | | 732 | | 371 | 321 | 86.4% | 4750 | 1 | 16 |
| | SD | | | | 22 | | | | | | | |
| Sample I: Turmeric Gelcap with Coconut (mg/g) | A | | | | 26.9 | 19.1 | | | | | | |
| | B | | | | 27.0 | 11.9 | | | | | | |
| | C | | | | 27.5 | | | | | | | |
| | Avg | | | | 27.1 | 15.5 | 24.0 | 3.0 | 12.4% | 47.6 | 11.9 | 15 |
| | SD | | | | 0.3 | 5.1 | | | | | | |
| Sample J: Turmeric Gelcap, Liquid Curcumin (mg/g) | A | | 44.7 | 45.9 | | | | | | | | |
| | B | | 43.7 | 45.8 | | | | | | | | |
| | C | | 43.3 | 44.5 | | | | | | | | |
| | Avg | | 43.9 | 45.4 | | | 44.3 | 4.3 | 9.6% | 54.1 | 37.6 | 15 |
| | SD | | 0.7 | 0.8 | | | | | | | | |

**Table 2-3.** Data summary table for curcumin in turmeric commercial products. Individual results are displayed in this table for the laboratories that reported using AOAC 2016.16 for analysis. Data highlighted in red have been flagged as potential outliers (e.g., Grubb and/or Cochran) by the NIST software package. Data shown in italicized font were collected in the second part of the study. Data for laboratory O411 was not included in the collaborative study because only a single sample was analyzed.

| | | Curcumin by AOAC 2016.16 | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Individual Results | | | | | | | | | | | Community Results | | | | | |
| | Lab | NIST | O404 | O407 | ~~O411~~ | O416 | O419 | O433 | O437 | O446 | O449 | O455 | Mean | SD | % RSD | Max | Min | N |
| Sample A: SRM 3299 Turmeric Rhizome (mg/g) | A | | 11.3 | 9.7 | | 12.2 | 11.1 | 10.2 | 8.8 | 10.9 | 11.6 | 10.9 | | | | | | |
| | B | | 11.3 | 9.7 | | 12.9 | 11.5 | 10.0 | 8.9 | 10.7 | 12.1 | 10.6 | | | | | | |
| | C | | 11.4 | 9.7 | | 12.6 | 11.5 | 9.8 | 9.2 | 10.8 | 11.0 | 11.0 | | | | | | |
| | D | | *10.3* | *10.8* | | *11.5* | *10.9* | | | *9.7* | *10.1* | *11.0* | | | | | | |
| | E | | *11.1* | *10.2* | | *11.2* | *10.9* | | | *9.2* | *10.4* | *11.2* | | | | | | |
| | F | | *10.6* | *10.2* | | *11.9* | *11.0* | | | *8.9* | *10.9* | *11.1* | | | | | | |
| | Avg | 11.04 | 11.0 | 10.1 | | 12.1 | 11.1 | 10.0 | 9.0 | 10.0 | 11.0 | 11.0 | 10.6 | 1.0 | 9.4% | 12.1 | 9.0 | 9 |
| | SD | 0.21 | 0.5 | 0.4 | | 0.7 | 0.3 | 0.2 | 0.2 | 0.9 | 0.7 | 0.2 | | | | | | |
| Sample B: SRM 3300 Tumeric Extract (mg/g) | A | | 802 | 871 | | 796 | 807 | 787 | 745 | 765 | 852 | 841 | | | | | | |
| | B | | 750 | 793 | | 866 | 799 | 784 | 774 | 799 | 867 | 850 | | | | | | |
| | C | | 731 | 845 | | 755 | 831 | 781 | 769 | 772 | 871 | 860 | | | | | | |
| | D | | *801* | *872* | | *835* | *791* | | | *570* | *807* | *827* | | | | | | |
| | E | | *801* | *883* | | *846* | *802* | | | *610* | *822* | *832* | | | | | | |
| | F | | *816* | *875* | | *825* | *807* | | | *604* | *802* | *830* | | | | | | |
| | Avg | 822 | 784 | 857 | | 820 | 806 | 784 | 763 | 687 | 837 | 840 | 803 | 46 | 5.8% | 857 | 687 | 9 |
| | SD | 11 | 34 | 34 | | 40 | 14 | 3 | 15 | 103 | 31 | 13 | | | | | | |
| Sample C: Turmeric Root Powder (mg/g) | A | | *15.52* | 13.80 | ~~16.98~~ | 15.20 | *15.28* | *14.39* | 13.50 | 15.06 | 15.08 | *15.04* | | | | | | |
| | B | | *15.52* | 14.90 | ~~16.56~~ | 16.40 | *15.43* | *14.03* | 13.50 | 14.90 | 16.16 | *14.96* | | | | | | |
| | C | | *15.76* | 15.10 | ~~15.92~~ | 15.00 | *15.38* | *14.27* | 13.70 | 14.83 | 15.10 | *15.24* | | | | | | |
| | Avg | | *15.60* | 14.60 | ~~16.49~~ | 15.53 | *15.36* | *14.23* | 13.57 | 14.93 | 15.44 | *15.08* | 14.97 | 0.69 | 4.6% | 16.49 | 13.57 | 10 |
| | SD | | *0.14* | 0.70 | ~~0.53~~ | 0.76 | *0.07* | *0.18* | 0.12 | 0.12 | 0.62 | *0.14* | | | | | | |
| Sample D: Turmeric Smoothie Additive (mg/g) | A | | 11.5 | *8.8* | | *9.2* | 9.8 | 9.4 | *8.8* | *7.7* | *9.2* | 9.8 | | | | | | |
| | B | | 11.1 | *9.1* | | *8.5* | 9.9 | 9.3 | *8.2* | *7.5* | *9.6* | 9.3 | | | | | | |
| | C | | 11.0 | *9.4* | | *8.1* | 10.0 | 13.2 | *8.3* | *7.4* | *10.1* | 9.3 | | | | | | |
| | Avg | | 11.2 | *9.1* | | *8.6* | 9.9 | 10.6 | *8.5* | *7.5* | *9.6* | 9.5 | 9.4 | 1.3 | 13.8% | 11.2 | 7.5 | 9 |
| | SD | | 0.3 | *0.3* | | *0.6* | 0.1 | 2.2 | *0.3* | *0.1* | *0.4* | 0.3 | | | | | | |
| Sample E: Turmeric Root Capsule (mg/g) | A | | 19.6 | *18.8* | | *18.7* | 19.2 | 17.3 | *16.7* | *15.5* | *18.8* | 19.9 | | | | | | |
| | B | | 19.8 | *18.9* | | *18.0* | 19.3 | 17.9 | *16.8* | *15.7* | *18.6* | 18.1 | | | | | | |
| | C | | 19.5 | *18.4* | | *17.6* | 19.4 | 17.6 | *16.5* | *15.3* | *18.8* | 17.9 | | | | | | |
| | Avg | | 19.6 | *18.7* | | *18.1* | 19.3 | 17.6 | *16.7* | *15.5* | *18.8* | 18.6 | 18.2 | 1.3 | 7.2% | 19.6 | 15.5 | 9 |
| | SD | | 0.1 | *0.3* | | *0.5* | 0.1 | 0.3 | *0.2* | *0.2* | *0.1* | 1.1 | | | | | | |
| Sample F: Turmeric Extract/Root Capsule with Black Pepper (mg/g) | A | | 50.6 | *52.4* | | *52.8* | 50.9 | 49.2 | *54.2* | *41.7* | *48.1* | 47.3 | | | | | | |
| | B | | 48.6 | *52.2* | | *50.0* | 51.9 | 50.0 | *50.3* | *42.1* | *50.0* | 50.6 | | | | | | |
| | C | | 49.2 | *52.6* | | *50.0* | 52.0 | 47.9 | *50.3* | *42.3* | *49.9* | 50.5 | | | | | | |
| | Avg | | 49.5 | *52.4* | | *50.9* | 51.6 | 49.0 | *51.6* | *42.0* | *49.3* | 49.5 | 50.1 | 1.9 | 3.9% | 52.4 | 42.0 | 9 |
| | SD | | 1.0 | *0.2* | | *1.6* | 0.6 | 1.1 | *2.3* | *0.3* | *1.1* | 1.9 | | | | | | |
| Sample G: Turmeric Extract/Root Capsule with Black Pepper & Coconut Oil (mg/g) | A | | | 154 | | 249 | *304* | *302* | 289 | 296 | 347 | *272* | | | | | | |
| | B | | | 234 | | 299 | *306* | *246* | 282 | 294 | 349 | *285* | | | | | | |
| | C | | | 276 | | 292 | *306* | *293* | 281 | 293 | 361 | *277* | | | | | | |
| | Avg | | | 221 | | 280 | *305* | *280* | 284 | 294 | 352 | *278* | 287 | 27 | 9.4% | 352 | 221 | 8 |
| | SD | | | 62 | | 27 | *1* | *30* | 4 | 2 | 8 | *6* | | | | | | |
| Sample H: Turmeric Tincture (mg/L) | A | | *666* | 502 | | 466 | *372* | *390* | 439 | 496 | 468 | *252* | | | | | | |
| | B | | *802* | 497 | | 522 | *372* | *393* | 428 | 487 | 486 | *259* | | | | | | |
| | C | | *705* | 500 | | 410 | *366* | *391* | 422 | 492 | 523 | *250* | | | | | | |
| | Avg | | *725* | 500 | | 466 | *370* | *391* | 430 | 492 | 492 | *253* | 449 | 98 | 21.9% | 725 | 253 | 9 |
| | SD | | *70* | 3 | | 56 | *3* | *1* | 9 | 4 | 28 | *5* | | | | | | |
| Sample I: Turmeric Gelcap with Coconut (mg/g) | A | | | 22.5 | | 22.4 | *24.2* | *23.3* | 21.5 | 24.8 | 23.6 | *22.3* | | | | | | |
| | B | | | 23.7 | | 22.8 | *24.2* | *23.4* | 22.0 | 23.2 | 26.1 | *21.0* | | | | | | |
| | C | | | 24.5 | | 20.3 | *23.9* | *21.5* | 21.5 | 22.7 | 23.9 | *19.6* | | | | | | |
| | Avg | | | 23.6 | | 21.8 | *24.1* | *22.7* | 21.7 | 23.6 | 24.5 | *21.0* | 22.9 | 1.6 | 7.0% | 24.5 | 21.0 | 8 |
| | SD | | | 1.0 | | 1.3 | *0.2* | *1.1* | 0.3 | 1.1 | 1.4 | *1.4* | | | | | | |
| Sample J: Turmeric Gelcap, Liquid Curcumin (mg/g) | A | | 47.1 | *45.3* | | *42.0* | 47.5 | 41.7 | *40.5* | *38.6* | *48.4* | 47.9 | | | | | | |
| | B | | 47.2 | *47.0* | | *44.9* | 47.4 | 42.8 | *40.9* | *37.7* | *45.4* | 48.7 | | | | | | |
| | C | | 46.1 | *46.7* | | *43.0* | 46.9 | 42.9 | *42.4* | *37.7* | *46.9* | 48.9 | | | | | | |
| | Avg | | 46.8 | *46.3* | | *43.3* | 47.3 | 42.5 | *41.3* | *38.0* | *46.9* | 48.5 | 44.7 | 3.7 | 8.2% | 48.5 | 38.0 | 9 |
| | SD | | 0.6 | *0.9* | | *1.5* | 0.4 | 0.6 | *1.0* | *0.5* | *1.5* | 0.5 | | | | | | |

**Table 2-4.1.** Data summary table for bisdemethoxycurcumin (BDMC) in turmeric commercial products. Individual results are displayed in this table for eleven of the laboratories that requested samples (O404 through O419), while community results are shown for all laboratories participating the study. Results for additional laboratories can be found in Tables 2-4.2 and 2-4.3. Data highlighted in red have been flagged as potential outliers (e.g., Grubb and/or Cochran) by the NIST software package.

| | | Bisdemethoxycurcumin | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Individual Results - Page 1 of 3 | | | | | | | | | | | | Community Results | | | | | |
| | Lab | NIST | O404 | O405 | O406 | O407 | O409 | O410 | O411 | O414 | O415 | O416 | O419 | Mean | SD | % RSD | Max | Min | N |
| Sample A: SRM 3299 Turmeric Rhizome (mg/g) | A | | 3.14 | | 2.77 | 2.46 | | 11.44 | | 4.00 | 4.59 | 3.75 | 2.91 | | | | | | |
| | B | | 3.14 | | 2.74 | 2.47 | | 11.68 | | 4.00 | 4.53 | 2.65 | 2.97 | | | | | | |
| | C | | 3.18 | | 2.81 | 2.48 | | 11.56 | | 4.00 | 4.38 | 3.30 | 2.99 | | | | | | |
| | D | | 2.42 | | | 3.35 | | | | | | 2.78 | | | | | | | |
| | E | | 2.55 | | | 3.34 | | | | | | 2.80 | | | | | | | |
| | F | | 2.45 | | | 3.23 | | | | | | 2.97 | | | | | | | |
| | Avg | 2.84 | 2.81 | | 2.77 | 2.89 | | 11.56 | | 4.00 | 4.50 | 3.04 | 2.96 | 3.14 | 0.66 | 20.9% | 11.56 | 1.16 | 18 |
| | SD | 0.05 | 0.38 | | 0.04 | 0.46 | | 0.12 | | 0.00 | 0.11 | 0.41 | 0.04 | | | | | | |
| Sample B: SRM 3300 Turmeric Extract (mg/g) | A | | 16.23 | | 17.20 | 16.60 | | 89.84 | | 19.00 | 17.80 | 19.09 | 15.35 | | | | | | |
| | B | | 16.55 | | 17.30 | 15.10 | | 84.63 | | 19.00 | 17.30 | 19.18 | 15.30 | | | | | | |
| | C | | 16.06 | | 17.40 | 16.40 | | 82.72 | | 19.00 | 17.00 | 17.62 | 15.17 | | | | | | |
| | D | | 13.99 | | | 18.00 | | | | | | 14.08 | 15.73 | | | | | | |
| | E | | 13.61 | | | 18.40 | | | | | | 14.40 | 15.73 | | | | | | |
| | F | | 14.31 | | | 18.20 | | | | | | 15.12 | 15.72 | | | | | | |
| | Avg | 18.25 | 15.12 | | 17.30 | 17.12 | | 85.73 | | 19.00 | 17.37 | 16.58 | 15.50 | 17.1 | 2.6 | 15.3% | 85.7 | 1.9 | 19 |
| | SD | 0.49 | 1.30 | | 0.10 | 1.30 | | 3.69 | | 0.00 | 0.40 | 2.34 | 0.25 | | | | | | |
| Sample C: Turmeric Root Powder (mg/g) | A | | 9.93 | | 10.60 | 9.40 | | 38.65 | 11.15 | | 10.70 | 11.80 | 10.31 | | | | | | |
| | B | | 9.97 | | 10.20 | 10.00 | | 39.38 | 11.98 | | 11.00 | 11.26 | 10.42 | | | | | | |
| | C | | 10.11 | | 10.70 | 10.20 | | 38.83 | 12.10 | | 10.70 | 10.84 | 10.34 | | | | | | |
| | Avg | | 10.00 | | 10.50 | 9.87 | | 38.95 | 11.74 | | 10.80 | 11.30 | 10.35 | 10.7 | 1.1 | 10.3% | 39.0 | 9.2 | 15 |
| | SD | | 0.10 | | 0.26 | 0.42 | | 0.38 | 0.52 | | 0.17 | 0.48 | 0.05 | | | | | | |
| Sample D: Turmeric Smoothie Additive (mg/g) | A | | 1.394 | | | 0.610 | | | | < 1.000 | | 1.430 | 0.630 | | | | | | |
| | B | | 1.404 | | | 0.701 | | | | < 1.000 | | 1.330 | 0.630 | | | | | | |
| | C | | 1.369 | | | 0.681 | | | | < 1.000 | | 1.160 | 0.650 | | | | | | |
| | Avg | | 1.389 | | | 0.664 | | | | | | 1.307 | 0.637 | 0.78 | 0.22 | 27.7% | 1.31 | 0.50 | 12 |
| | SD | | 0.018 | | | 0.048 | | | | | | 0.137 | 0.012 | | | | | | |
| Sample E: Turmeric Root Capsule (mg/g) | A | | 4.74 | | | 5.42 | | | | 6.00 | | 4.650 | 4.29 | | | | | | |
| | B | | 4.86 | | | 5.46 | | | | 5.00 | | 4.930 | 4.34 | | | | | | |
| | C | | 4.78 | | | 5.21 | | | | 6.00 | | 4.880 | 4.35 | | | | | | |
| | Avg | | 4.79 | | | 5.36 | | | | 5.67 | | 4.820 | 4.33 | 4.49 | 0.84 | 18.8% | 5.97 | 1.48 | 14 |
| | SD | | 0.06 | | | 0.13 | | | | 0.58 | | 0.149 | 0.03 | | | | | | |
| Sample F: Turmeric Extract/Root Capsule with Black Pepper (mg/g) | A | | 3.519 | | | 3.410 | | | | 4.000 | | 3.220 | 2.940 | | | | | | |
| | B | | 3.450 | | | 3.250 | | | | 4.000 | | 3.100 | 2.970 | | | | | | |
| | C | | 3.465 | | | 3.410 | | | | 4.000 | | 2.920 | 2.990 | | | | | | |
| | Avg | | 3.478 | | | 3.357 | | | | 4.000 | | 3.080 | 2.967 | 3.18 | 0.72 | 22.7% | 5.15 | 0.96 | 14 |
| | SD | | 0.036 | | | 0.092 | | | | 0.000 | | 0.151 | 0.025 | | | | | | |
| Sample G: Turmeric Extract/Root Capsule with Black Pepper & Coconut Oil (mg/g) | A | | | | 11.70 | 6.05 | | 43.01 | | | 11.20 | 12.91 | 10.82 | | | | | | |
| | B | | | | 11.70 | 8.51 | | 41.11 | | | 12.40 | 12.45 | 10.91 | | | | | | |
| | C | | | | 11.40 | 9.75 | | 44.68 | | | 12.30 | 12.57 | 10.95 | | | | | | |
| | Avg | | | | 11.60 | 8.10 | | 42.93 | | | 11.97 | 12.64 | 10.89 | 11.7 | 2.5 | 21.3% | 42.9 | 8.1 | 14 |
| | SD | | | | 0.17 | 1.88 | | 1.79 | | | 0.67 | 0.24 | 0.07 | | | | | | |
| Sample H: Turmeric Tincture (mg/L) | A | | 22.94 | | 0.26 | 235.00 | | 0.89 | | | 3350 | 242.6 | 198.11 | | | | | | |
| | B | | 30.19 | | 0.25 | 236.00 | | 0.88 | | | 3370 | 270.0 | 199.66 | | | | | | |
| | C | | 26.44 | | 0.27 | 236.00 | | 0.87 | | | 2840 | 213.0 | 197.98 | | | | | | |
| | Avg | | 26.52 | | 0.26 | 235.67 | | 0.88 | | | 3187 | 241.9 | 198.58 | 156 | 133 | 85.7% | 3187 | 0.26 | 14 |
| | SD | | 3.62 | | 0.01 | 0.58 | | 0.01 | | | 300 | 28.5 | 0.93 | | | | | | |
| Sample I: Turmeric Gelcap with Coconut (mg/g) | A | | | | 1.14 | 0.98 | | 3.64 | | | 3.56 | 1.530 | 1.08 | | | | | | |
| | B | | | | 1.09 | 1.02 | | 3.80 | | | 3.08 | 1.600 | 1.08 | | | | | | |
| | C | | | | 1.08 | 1.06 | | 3.47 | | | 3.32 | 1.290 | 1.07 | | | | | | |
| | Avg | | | | 1.10 | 1.02 | | 3.63 | | | 3.32 | 1.473 | 1.07 | 1.26 | 0.45 | 35.7% | 3.63 | 0.60 | 13 |
| | SD | | | | 0.03 | 0.04 | | 0.17 | | | 0.24 | 0.163 | 0.01 | | | | | | |
| Sample J: Turmeric Gelcap, Liquid Curcumin (mg/g) | A | | 2.134 | | | 1.970 | | | | 2.000 | | 2.34 | 1.750 | | | | | | |
| | B | | 2.160 | | | 1.940 | | | | 2.000 | | 2.22 | 1.770 | | | | | | |
| | C | | 2.172 | | | 1.980 | | | | 2.000 | | 2.27 | 1.740 | | | | | | |
| | Avg | | 2.155 | | | 1.963 | | | | 2.000 | | 2.28 | 1.753 | 1.89 | 0.37 | 19.8% | 2.76 | 0.58 | 14 |
| | SD | | 0.019 | | | 0.021 | | | | 0.000 | | 0.06 | 0.015 | | | | | | |

**Table 2-4.2.** Data summary table for bisdemethoxycurcumin (BDMC) in turmeric commercial products. Individual results are displayed in this table for ten of the laboratories that requested samples (O420 through O437), while community results are shown for all laboratories participating the study. Results for additional laboratories can be found in Tables 2-4.1 and 2-4.3. Data highlighted in red have been flagged as potential outliers (e.g., Grubb and/or Cochran) by the NIST software package.

| | | Bisdemethoxycurcumin | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Individual Results - Page 2 of 3 | | | | | | | | | | | | Community Results | | | | | |
| | Lab | NIST | O420 | O421 | O423 | O425 | O428 | O429 | O431 | O433 | O434 | O437 | | Mean | SD | % RSD | Max | Min | N |
| Sample A: SRM 3299 Turmeric Rhizome (mg/g) | A | | 4.65 | 2.48 | | | | | 3.21 | 3.14 | | 2.35 | | | | | | | |
| | B | | 4.68 | 2.43 | | | | | 3.26 | 2.85 | | 2.35 | | | | | | | |
| | C | | 4.79 | 2.53 | | | | | 3.27 | 3.05 | | 2.40 | | | | | | | |
| | D | | | | | | | | | | | | | | | | | | |
| | E | | | | | | | | | | | | | | | | | | |
| | F | | | | | | | | | | | | | | | | | | |
| | Avg | 2.84 | 4.71 | 2.48 | | | | | 3.247 | 3.01 | | 2.37 | | 3.14 | 0.66 | 20.9% | 11.56 | 1.16 | 19 |
| | SD | 0.05 | 0.07 | 0.05 | | | | | 0.032 | 0.15 | | 0.03 | | | | | | | |
| Sample B: SRM 3300 Turmeric Extract (mg/g) | A | | 18.16 | 21.78 | | 9.09 | | | 17.10 | 17.55 | | 15.10 | | | | | | | |
| | B | | 18.05 | 18.56 | | | | | 15.30 | 17.55 | | 15.80 | | | | | | | |
| | C | | 18.12 | 20.80 | | 10.04 | | | 15.40 | 17.24 | | 15.40 | | | | | | | |
| | D | | | | | | | | | | | | | | | | | | |
| | E | | | | | | | | | | | | | | | | | | |
| | F | | | | | | | | | | | | | | | | | | |
| | Avg | 18.25 | 18.11 | 20.38 | | 9.57 | | | 15.933 | 17.45 | | 15.43 | | 17.1 | 2.6 | 15.3% | 85.7 | 1.9 | 20 |
| | SD | 0.49 | 0.06 | 1.65 | | 0.67 | | | 1.012 | 0.18 | | 0.35 | | | | | | | |
| Sample C: Turmeric Root Powder (mg/g) | A | | 14.53 | | | | | | | 10.31 | | 9.07 | | | | | | | |
| | B | | 15.03 | | | | | | | 9.90 | | 9.27 | | | | | | | |
| | C | | 15.05 | | | | | | | 10.16 | | 9.29 | | | | | | | |
| | Avg | | 14.87 | | | | | | | 10.13 | | 9.21 | | 10.7 | 1.1 | 10.3% | 39.0 | 9.2 | 15 |
| | SD | | 0.29 | | | | | | | 0.21 | | 0.12 | | | | | | | |
| Sample D: Turmeric Smoothie Additive (mg/g) | A | | | 0.600 | | | | | 0.787 | 0.794 | | 0.751 | | | | | | | |
| | B | | | 0.570 | | | | | 0.789 | 0.766 | | 0.737 | | | | | | | |
| | C | | | 0.580 | | | | | 0.810 | 1.005 | | 0.737 | | | | | | | |
| | Avg | | | 0.583 | | | | | 0.795 | 0.855 | | 0.742 | | 0.78 | 0.22 | 27.7% | 1.31 | 0.50 | 13 |
| | SD | | | 0.015 | | | | | 0.013 | 0.131 | | 0.008 | | | | | | | |
| Sample E: Turmeric Root Capsule (mg/g) | A | | | 3.76 | | 1.48 | | | 5.16 | 4.48 | | 3.97 | | | | | | | |
| | B | | | 3.71 | | 1.39 | | | 5.07 | 4.76 | | 4.04 | | | | | | | |
| | C | | | 3.70 | | 1.56 | | | 5.09 | 4.46 | | 3.97 | | | | | | | |
| | Avg | | | 3.72 | | 1.48 | | | 5.11 | 4.57 | | 3.99 | | 4.49 | 0.84 | 18.8% | 5.97 | 1.48 | 15 |
| | SD | | | 0.03 | | 0.09 | | | 0.05 | 0.17 | | 0.04 | | | | | | | |
| Sample F: Turmeric Extract/Root Capsule with Black Pepper (mg/g) | A | | | 2.830 | | 0.850 | | | 4.360 | 3.711 | | 2.980 | | | | | | | |
| | B | | | 2.690 | | 1.000 | | | 4.320 | 3.641 | | 2.940 | | | | | | | |
| | C | | | 2.780 | | 1.040 | | | 4.190 | 3.480 | | 2.880 | | | | | | | |
| | Avg | | | 2.767 | | 0.963 | | | 4.290 | 3.611 | | 2.933 | | 3.18 | 0.72 | 22.7% | 5.15 | 0.96 | 15 |
| | SD | | | 0.071 | | 0.100 | | | 0.089 | 0.118 | | 0.050 | | | | | | | |
| Sample G: Turmeric Extract/Root Capsule with Black Pepper & Coconut Oil (mg/g) | A | | 12.79 | | | | | | | 11.70 | | 10.30 | | | | | | | |
| | B | | 13.52 | | | | | | | 9.67 | | 10.10 | | | | | | | |
| | C | | 13.76 | | | | | | | 10.71 | | 10.00 | | | | | | | |
| | Avg | | 13.36 | | | | | | | 10.69 | | 10.13 | | 11.7 | 2.5 | 21.3% | 42.9 | 8.1 | 14 |
| | SD | | 0.51 | | | | | | | 1.02 | | 0.15 | | | | | | | |
| Sample H: Turmeric Tincture (mg/L) | A | | 1.25 | | | | | | | 209.6 | | 212.1 | | | | | | | |
| | B | | 1.21 | | | | | | | 211.5 | | 209.1 | | | | | | | |
| | C | | 1.24 | | | | | | | 210.1 | | 205.3 | | | | | | | |
| | Avg | | 1.23 | | | | | | | 210.4 | | 208.8 | | 156 | 133 | 85.7% | 3187 | 0.26 | 14 |
| | SD | | 0.02 | | | | | | | 1.0 | | 3.4 | | | | | | | |
| Sample I: Turmeric Gelcap with Coconut (mg/g) | A | | 1.86 | | | | | | | 1.05 | | 1.05 | | | | | | | |
| | B | | 1.89 | | | | | | | 1.05 | | 1.11 | | | | | | | |
| | C | | 1.85 | | | | | | | 0.97 | | 1.06 | | | | | | | |
| | Avg | | 1.87 | | | | | | | 1.02 | | 1.07 | | 1.26 | 0.45 | 35.7% | 3.63 | 0.60 | 13 |
| | SD | | 0.02 | | | | | | | 0.05 | | 0.03 | | | | | | | |
| Sample J: Turmeric Gelcap, Liquid Curcumin (mg/g) | A | | | 1.520 | | 0.610 | | | 2.010 | 1.770 | | 2.220 | | | | | | | |
| | B | | | 1.500 | | 0.460 | | | 1.800 | 1.793 | | 2.160 | | | | | | | |
| | C | | | 1.870 | | 0.670 | | | 1.810 | 1.796 | | 2.250 | | | | | | | |
| | Avg | | | 1.630 | | 0.580 | | | 1.873 | 1.786 | | 2.210 | | 1.89 | 0.37 | 19.8% | 2.76 | 0.58 | 15 |
| | SD | | | 0.208 | | 0.108 | | | 0.118 | 0.014 | | 0.046 | | | | | | | |

**Table 2-4.3.** Data summary table for bisdemethoxycurcumin (BDMC) in turmeric commercial products. Individual results are displayed in this table for ten of the laboratories that requested samples (O440 through O461), while community results are shown for all laboratories participating the study. Results for additional laboratories can be found in Tables 2-4.1 and 2-4.2. Data highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

| | | Bisdemethoxycurcumin | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Individual Results - Page 3 of 3 | | | | | | | | | | | Community Results | | | | | | |
| | Lab | NIST | O440 | O443 | O446 | O449 | O452 | O455 | O458 | O459 | O460 | O461 | Mean | SD | % RSD | Max | Min | N |
| Sample A: SRM 3299 Turmeric Rhizome (mg/g) | A | | 3.04 | | 3.09 | 3.44 | 3.39 | 2.69 | | 1.26 | 3.64 | 2.94 | | | | | | |
| | B | | | | 3.04 | 3.64 | 3.43 | 2.69 | | 1.12 | 3.66 | 2.90 | | | | | | |
| | C | | | | 3.04 | 3.22 | 3.32 | 2.67 | | 1.10 | 3.79 | 2.97 | | | | | | |
| | D | | | | 2.62 | 3.00 | | 2.85 | | | | | | | | | | |
| | E | | | | 2.66 | 3.00 | | 2.87 | | | | | | | | | | |
| | F | | | | 2.63 | 3.10 | | 2.89 | | | | | | | | | | |
| | Avg | 2.84 | 3.04 | | 2.84 | 3.23 | 3.38 | 2.78 | | 1.16 | 3.70 | 2.94 | 3.14 | 0.66 | 20.9% | 11.56 | 1.16 | 19 |
| | SD | 0.05 | | | 0.23 | 0.26 | 0.06 | 0.10 | | 0.09 | 0.08 | 0.04 | | | | | | |
| Sample B: SRM 3300 Turmeric Extract (mg/g) | A | | 15.68 | | 19.9 | 11.95 | 19.90 | 19.91 | | 13.77 | 19.41 | 25.35 | | | | | | |
| | B | | | | 19.3 | 12.56 | 19.11 | 19.92 | | 13.42 | 19.37 | 23.96 | | | | | | |
| | C | | | | 18.2 | 13.56 | 18.93 | 20.10 | | 13.60 | 18.97 | 23.83 | | | | | | |
| | D | | | | 14.0 | 13.54 | | 15.41 | | | | | | | | | | |
| | E | | | | 14.7 | 15.04 | | 15.52 | | | | | | | | | | |
| | F | | | | 14.5 | 15.60 | | 15.48 | | | | | | | | | | |
| | Avg | 18.25 | 15.68 | | 16.77 | 13.71 | 19.31 | 17.72 | | 13.60 | 19.25 | 24.38 | 17.1 | 2.6 | 15.3% | 85.7 | 9.6 | 20 |
| | SD | 0.49 | | | 2.66 | 1.40 | 0.51 | 2.47 | | 0.18 | 0.24 | 0.84 | | | | | | |
| Sample C: Turmeric Root Powder (mg/g) | A | | 11.15 | | 9.94 | 9.92 | | 9.46 | | | | 10.78 | | | | | | |
| | B | | 11.22 | | 9.94 | 10.60 | | 9.26 | | | | 12.13 | | | | | | |
| | C | | 10.89 | | 9.85 | 9.77 | | 9.49 | | | | 11.47 | | | | | | |
| | Avg | | 11.09 | | 9.91 | 10.10 | | 9.40 | | | | 11.46 | 10.7 | 1.1 | 10.3% | 39.0 | 9.2 | 15 |
| | SD | | 0.17 | | 0.05 | 0.44 | | 0.12 | | | | 0.68 | | | | | | |
| Sample D: Turmeric Smoothie Additive (mg/g) | A | | | | 0.553 | 0.710 | 0.730 | 0.948 | | 0.460 | 0.901 | | | | | | | |
| | B | | | | 0.571 | 0.796 | 0.739 | 0.995 | | 0.500 | 0.888 | | | | | | | |
| | C | | | | 0.613 | 0.715 | 0.733 | 1.005 | | 0.550 | 0.940 | | | | | | | |
| | Avg | | | | 0.579 | 0.740 | 0.734 | 0.983 | | 0.503 | 0.910 | | 0.78 | 0.22 | 27.7% | 1.31 | 0.50 | 13 |
| | SD | | | | 0.031 | 0.048 | 0.005 | 0.030 | | 0.045 | 0.027 | | | | | | | |
| Sample E: Turmeric Root Capsule (mg/g) | A | | | | 3.98 | 4.12 | 4.43 | 4.60 | | 3.52 | 5.85 | | | | | | | |
| | B | | | | 3.98 | 4.12 | 4.54 | 4.35 | | 3.36 | 5.89 | | | | | | | |
| | C | | | | 3.90 | 4.22 | 4.44 | 4.34 | | 3.50 | 6.17 | | | | | | | |
| | Avg | | | | 3.95 | 4.15 | 4.47 | 4.43 | | 3.46 | 5.97 | | 4.49 | 0.84 | 18.8% | 5.97 | 1.48 | 15 |
| | SD | | | | 0.04 | 0.06 | 0.06 | 0.15 | | 0.09 | 0.18 | | | | | | | |
| Sample F: Turmeric Extract/Root Capsule with Black Pepper (mg/g) | A | | | | 2.553 | 2.881 | 3.236 | 3.143 | | 2.360 | 5.254 | | | | | | | |
| | B | | | | 2.555 | 2.709 | 3.208 | 3.302 | | 2.290 | 5.060 | | | | | | | |
| | C | | | | 2.558 | 2.713 | 3.247 | 3.204 | | 2.330 | 5.146 | | | | | | | |
| | Avg | | | | 2.555 | 2.768 | 3.230 | 3.216 | | 2.327 | 5.153 | | 3.18 | 0.72 | 22.7% | 5.15 | 0.96 | 15 |
| | SD | | | | 0.002 | 0.098 | 0.020 | 0.080 | | 0.035 | 0.097 | | | | | | | |
| Sample G: Turmeric Extract/Root Capsule with Black Pepper & Coconut Oil (mg/g) | A | | 10.72 | | 10.61 | 12.47 | | 8.81 | | | | 15.10 | | | | | | |
| | B | | 10.59 | | 10.59 | 13.12 | | 9.29 | | | | 15.96 | | | | | | |
| | C | | 10.99 | | 10.48 | 15.27 | | 8.97 | | | | 16.62 | | | | | | |
| | Avg | | 10.77 | | 10.56 | 13.62 | | 9.02 | | | | 15.89 | 11.7 | 2.5 | 21.3% | 42.9 | 8.1 | 14 |
| | SD | | 0.20 | | 0.07 | 1.47 | | 0.24 | | | | 0.76 | | | | | | |
| Sample H: Turmeric Tincture (mg/L) | A | | 8.56 | | 229.0 | 206.5 | | 132.8 | | | | 267.5 | | | | | | |
| | B | | 8.40 | | 225.9 | 212.4 | | 134.7 | | | | 245.9 | | | | | | |
| | C | | 8.35 | | 227.8 | 255.3 | | 131.1 | | | | 273.3 | | | | | | |
| | Avg | | 8.44 | | 227.6 | 224.7 | | 132.9 | | | | 262.2 | 156 | 133 | 85.7% | 3187 | 0.26 | 14 |
| | SD | | 0.11 | | 1.6 | 26.6 | | 1.8 | | | | 14.4 | | | | | | |
| Sample I: Turmeric Gelcap with Coconut (mg/g) | A | | 1.14 | | 1.24 | 0.60 | | 1.19 | | | | 1.20 | | | | | | |
| | B | | | | 1.16 | 0.56 | | 1.12 | | | | 1.02 | | | | | | |
| | C | | | | 1.14 | 0.63 | | 1.08 | | | | 1.02 | | | | | | |
| | Avg | | 1.14 | | 1.18 | 0.60 | | 1.13 | | | | 1.08 | 1.26 | 0.45 | 35.7% | 3.63 | 0.60 | 13 |
| | SD | | | | 0.05 | 0.04 | | 0.06 | | | | 0.10 | | | | | | |
| Sample J: Turmeric Gelcap, Liquid Curcumin (mg/g) | A | | | | 1.671 | 1.740 | 1.837 | 2.945 | | 1.430 | 2.260 | | | | | | | |
| | B | | | | 1.638 | 1.610 | 1.839 | 2.587 | | 1.330 | 2.349 | | | | | | | |
| | C | | | | 1.622 | 1.660 | 1.846 | 2.762 | | 1.330 | 2.301 | | | | | | | |
| | Avg | | | | 1.644 | 1.670 | 1.841 | 2.765 | | 1.363 | 2.303 | | 1.89 | 0.37 | 19.8% | 2.76 | 0.58 | 15 |
| | SD | | | | 0.025 | 0.066 | 0.005 | 0.179 | | 0.058 | 0.045 | | | | | | | |

**Table 2-5.** Data summary table for bisdemethoxycurcumin (BDMC) in turmeric commercial products. Individual results are displayed in this table for the laboratories that reported using AOAC 2016.16 for analysis. Data highlighted in red have been flagged as potential outliers (e.g., Grubb and/or Cochran) by the NIST software package. Data shown in italicized font were collected in the second part of the study. Data for laboratory O411 was not included in the collaborative study because only a single sample was analyzed.

| | | BDMC by AOAC 2016.16 | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Individual Results | | | | | | | | | | | Community Results | | | | | |
| | Lab | NIST | O404 | O407 | ~~O411~~ | O416 | O419 | O433 | O437 | O446 | O449 | O455 | Mean | SD | % RSD | Max | Min | N |
| Sample A: SRM 3299 Turmeric Rhizome (mg/g) | A | | 3.14 | 2.46 | | 3.75 | 2.91 | 3.14 | 2.35 | 3.09 | 3.44 | 2.69 | | | | | | |
| | B | | 3.14 | 2.47 | | 2.65 | 2.97 | 2.85 | 2.35 | 3.04 | 3.64 | 2.69 | | | | | | |
| | C | | 3.18 | 2.48 | | 3.30 | 2.99 | 3.05 | 2.40 | 3.04 | 3.22 | 2.67 | | | | | | |
| | D | | *2.42* | *3.35* | | *2.78* | | | | *2.62* | *3.00* | *2.85* | | | | | | |
| | E | | *2.55* | *3.34* | | *2.80* | | | | *2.66* | *3.00* | *2.87* | | | | | | |
| | F | | *2.45* | *3.23* | | *2.97* | | | | *2.63* | *3.10* | *2.89* | | | | | | |
| | Avg | 2.84 | 2.81 | 2.89 | | 3.04 | 2.96 | 3.01 | 2.37 | 2.84 | 3.23 | 2.78 | 2.91 | 0.23 | 7.8% | 3.23 | 2.37 | 9 |
| | SD | 0.05 | 0.38 | 0.46 | | 0.41 | 0.04 | 0.15 | 0.03 | 0.23 | 0.26 | 0.10 | | | | | | |
| Sample B: SRM 3300 Tumeric Extract (mg/g) | A | | 16.2 | 16.6 | | 19.1 | 15.4 | 17.5 | 15.1 | 19.9 | 11.9 | 19.9 | | | | | | |
| | B | | 16.5 | 15.1 | | 19.2 | 15.3 | 17.5 | 15.8 | 19.3 | 12.6 | 19.9 | | | | | | |
| | C | | 16.1 | 16.4 | | 17.6 | 15.2 | 17.2 | 15.4 | 18.2 | 13.6 | 20.1 | | | | | | |
| | D | | *14.0* | *18.0* | | *14.1* | *15.7* | | | *14.0* | *13.5* | *15.4* | | | | | | |
| | E | | *13.6* | *18.4* | | *14.4* | *15.7* | | | *14.7* | *15.0* | *15.5* | | | | | | |
| | F | | *14.3* | *18.2* | | *15.1* | *15.7* | | | *14.5* | *15.6* | *15.5* | | | | | | |
| | Avg | 18.3 | 15.1 | 17.1 | | 16.6 | 15.5 | 17.4 | 15.4 | 16.8 | 13.7 | 17.7 | 16.2 | 1.5 | 9.1% | 17.7 | 13.7 | 9 |
| | SD | 0.5 | 1.3 | 1.3 | | 2.3 | 0.3 | 0.2 | 0.4 | 2.7 | 1.4 | 2.5 | | | | | | |
| Sample C: Turmeric Root Powder (mg/g) | A | | *9.93* | 9.40 | ~~11.15~~ | 11.80 | *10.31* | *10.31* | 9.07 | 9.94 | 9.92 | *9.46* | | | | | | |
| | B | | *9.97* | 10.00 | ~~11.98~~ | 11.26 | *10.42* | *9.90* | 9.27 | 9.94 | 10.60 | *9.26* | | | | | | |
| | C | | *10.11* | 10.20 | ~~12.10~~ | 10.84 | *10.34* | *10.16* | 9.29 | 9.85 | 9.77 | *9.49* | | | | | | |
| | Avg | | *10.00* | 9.87 | ~~11.74~~ | 11.30 | *10.35* | *10.13* | 9.21 | 9.91 | 10.10 | *9.40* | 9.97 | 0.55 | 5.5% | 11.7 | 9.2 | 10 |
| | SD | | *0.10* | 0.42 | ~~0.52~~ | 0.48 | *0.05* | *0.21* | 0.12 | 0.05 | 0.44 | *0.12* | | | | | | |
| Sample D: Turmeric Smoothie Additive (mg/g) | A | | 1.39 | *0.61* | | *1.43* | 0.63 | 0.79 | *0.75* | *0.55* | 0.71 | 0.95 | | | | | | |
| | B | | 1.40 | *0.70* | | *1.33* | 0.63 | 0.77 | *0.74* | *0.57* | 0.80 | 1.00 | | | | | | |
| | C | | 1.37 | *0.68* | | *1.16* | 0.65 | 1.01 | *0.74* | *0.61* | 0.72 | 1.01 | | | | | | |
| | Avg | | 1.39 | *0.66* | | *1.31* | 0.64 | 0.86 | *0.74* | *0.58* | 0.74 | 0.98 | 0.87 | 0.33 | 37.4% | 1.39 | 0.58 | 9 |
| | SD | | 0.02 | *0.05* | | *0.14* | 0.01 | 0.13 | *0.01* | *0.03* | 0.05 | 0.03 | | | | | | |
| Sample E: Turmeric Root Capsule (mg/g) | A | | 4.74 | *5.42* | | *4.65* | 4.29 | 4.48 | *3.97* | *3.98* | 4.12 | 4.60 | | | | | | |
| | B | | 4.86 | *5.46* | | *4.93* | 4.34 | 4.76 | *4.04* | *3.98* | 4.12 | 4.35 | | | | | | |
| | C | | 4.78 | *5.21* | | *4.88* | 4.35 | 4.46 | *3.97* | *3.90* | 4.22 | 4.34 | | | | | | |
| | Avg | | 4.79 | *5.36* | | *4.82* | 4.33 | 4.57 | *3.99* | *3.95* | 4.15 | 4.43 | 4.47 | 0.47 | 10.4% | 5.36 | 3.95 | 9 |
| | SD | | 0.06 | *0.13* | | *0.15* | 0.03 | 0.17 | *0.04* | *0.04* | 0.06 | 0.15 | | | | | | |
| Sample F: Turmeric Extract/Root Capsule with Black Pepper (mg/g) | A | | 3.52 | *3.41* | | *3.22* | 2.94 | 3.71 | *2.98* | *2.55* | 2.88 | 3.14 | | | | | | |
| | B | | 3.45 | *3.25* | | *3.10* | 2.97 | 3.64 | *2.94* | *2.55* | 2.71 | 3.30 | | | | | | |
| | C | | 3.47 | *3.41* | | *2.92* | 2.99 | 3.48 | *2.88* | *2.56* | 2.71 | 3.20 | | | | | | |
| | Avg | | 3.48 | *3.36* | | *3.08* | 2.97 | 3.61 | *2.93* | *2.56* | 2.77 | 3.22 | 3.11 | 0.39 | 12.6% | 3.61 | 2.56 | 9 |
| | SD | | 0.04 | *0.09* | | *0.15* | 0.03 | 0.12 | *0.05* | *0.00* | 0.10 | 0.08 | | | | | | |
| Sample G: Turmeric Extract/Root Capsule with Black Pepper & Coconut Oil (mg/g) | A | | | 6.1 | | 12.9 | *10.8* | *11.7* | 10.3 | 10.6 | 12.5 | *8.8* | | | | | | |
| | B | | | 8.5 | | 12.5 | *10.9* | *9.7* | 10.1 | 10.6 | 13.1 | *9.3* | | | | | | |
| | C | | | 9.8 | | 12.6 | *11.0* | *10.7* | 10.0 | 10.5 | 15.3 | *9.0* | | | | | | |
| | Avg | | | 8.1 | | 12.6 | *10.9* | *10.7* | 10.1 | 10.6 | 13.6 | *9.0* | 10.7 | 2.1 | 19.2% | 13.6 | 8.1 | 8 |
| | SD | | | 1.9 | | 0.2 | *0.1* | *1.0* | 0.2 | 0.1 | 1.5 | *0.2* | | | | | | |
| Sample H: Turmeric Tincture (mg/L) | A | | *22.9* | 235 | | 243 | *198* | *210* | 212 | 229 | 207 | *133* | | | | | | |
| | B | | *30.2* | 236 | | 270 | *200* | *211* | 209 | 226 | 212 | *135* | | | | | | |
| | C | | *26.4* | 236 | | 213 | *198* | *210* | 205 | 228 | 255 | *131* | | | | | | |
| | Avg | | *26.5* | 236 | | 242 | *199* | *210* | 209 | 228 | 225 | *133* | 201 | 47 | 23.6% | 242 | 26.5 | 9 |
| | SD | | *3.6* | 1 | | 29 | *1* | *1* | 3 | 2 | 27 | *2* | | | | | | |
| Sample I: Turmeric Gelcap with Coconut (mg/g) | A | | | 0.98 | | *1.53* | *1.08* | *1.05* | 1.05 | 1.24 | *0.60* | *1.19* | | | | | | |
| | B | | | 1.02 | | *1.60* | *1.08* | *1.05* | 1.11 | 1.16 | *0.56* | *1.12* | | | | | | |
| | C | | | 1.06 | | *1.29* | *1.07* | *0.97* | 1.06 | 1.14 | *0.63* | *1.08* | | | | | | |
| | Avg | | | 1.02 | | *1.47* | *1.07* | *1.02* | 1.07 | 1.18 | *0.60* | *1.13* | 1.08 | 0.15 | 13.7% | 1.47 | 0.60 | 8 |
| | SD | | | 0.04 | | *0.16* | *0.01* | *0.05* | 0.03 | 0.05 | *0.04* | *0.06* | | | | | | |
| Sample J: Turmeric Gelcap, Liquid Curcumin (mg/g) | A | | 2.13 | *1.97* | | *2.34* | 1.75 | 1.77 | *2.22* | *1.67* | *1.74* | *2.95* | | | | | | |
| | B | | 2.16 | *1.94* | | *2.22* | 1.77 | 1.79 | *2.16* | *1.64* | *1.61* | *2.59* | | | | | | |
| | C | | 2.17 | *1.98* | | *2.27* | 1.74 | 1.80 | *2.25* | *1.62* | *1.66* | *2.76* | | | | | | |
| | Avg | | 2.16 | *1.96* | | *2.28* | 1.75 | 1.79 | *2.21* | *1.64* | *1.67* | *2.76* | 2.00 | 0.35 | 17.6% | 2.76 | 1.64 | 9 |
| | SD | | 0.02 | *0.02* | | *0.06* | 0.02 | 0.01 | *0.05* | *0.03* | *0.07* | *0.18* | | | | | | |

**Table 2-6.1.** Data summary table for desmethoxycurcumin (DMC) in turmeric commercial products. Individual results are displayed in this table for eleven of the laboratories that requested samples (O404 through O419), while community results are shown for all laboratories participating the study. Results for additional laboratories can be found in Tables 2-6.2 and 2-6.3. Data highlighted in red have been flagged as potential outliers (e.g., Grubb and/or Cochran) by the NIST software package.

| | | Desmethoxycurcumin | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Individual Results - Page 1 of 3 | | | | | | | | | | | | Community Results | | | | | |
| | Lab | NIST | O404 | O405 | O406 | O407 | O409 | O410 | O411 | O414 | O415 | O416 | O419 | Mean | SD | % RSD | Max | Min | N |
| Sample A: SRM 3299 Turmeric Rhizome (mg/g) | A | | 3.37 | | 3.06 | 2.95 | | 11.43 | | 4.00 | 5.11 | 4.73 | 3.17 | | | | | | |
| | B | | 3.37 | | 3.06 | 2.94 | | 11.41 | | 4.00 | 5.14 | 3.32 | 3.25 | | | | | | |
| | C | | 3.42 | | 3.09 | 2.96 | | 11.48 | | 4.00 | 5.15 | 4.00 | 3.22 | | | | | | |
| | D | | 3.29 | | | 3.23 | | | | | | 3.77 | 3.50 | | | | | | |
| | E | | 3.51 | | | 3.18 | | | | | | 3.35 | 3.51 | | | | | | |
| | F | | 3.37 | | | 3.03 | | | | | | 3.21 | 3.55 | | | | | | |
| | Avg | 3.14 | 3.39 | | 3.07 | 3.05 | | 11.44 | | 4.00 | 5.13 | 3.73 | 3.37 | 3.37 | 0.72 | 21.3% | 11.44 | 0.82 | 18 |
| | SD | 0.06 | 0.07 | | 0.02 | 0.13 | | 0.04 | | 0.00 | 0.02 | 0.58 | 0.17 | | | | | | |
| Sample B: SRM 3300 Turmeric Extract (mg/g) | A | | 3.4 | | 123.0 | 120.0 | | 323.8 | | 123.0 | 114.0 | 141.7 | 124.2 | | | | | | |
| | B | | 3.4 | | 123.0 | 110.0 | | 320.8 | | 123.0 | 113.0 | 148.7 | 123.2 | | | | | | |
| | C | | 3.4 | | 124.0 | 117.0 | | 317.6 | | 124.0 | 113.0 | 121.8 | 128.0 | | | | | | |
| | D | | 116.0 | | | 125.0 | | | | | | 108.9 | 124.2 | | | | | | |
| | E | | 115.0 | | | 127.0 | | | | | | 120.0 | 126.1 | | | | | | |
| | F | | 118.1 | | | 125.0 | | | | | | 113.9 | 126.9 | | | | | | |
| | Avg | 117 | 59.9 | | 123.3 | 120.7 | | 320.7 | | 123.3 | 113.3 | 125.8 | 125.4 | 117.2 | 9.5 | 8.1% | 320.7 | 11.9 | 19 |
| | SD | 1 | 61.9 | | 0.6 | 6.4 | | 3.1 | | 0.6 | 0.6 | 15.9 | 1.9 | | | | | | |
| Sample C: Turmeric Root Powder (mg/g) | A | | 7.65 | | 7.33 | 6.83 | | 22.18 | 10.10 | | 8.88 | 8.16 | 7.74 | | | | | | |
| | B | | 7.63 | | 7.03 | 7.33 | | 22.57 | 9.65 | | 9.20 | 7.90 | 7.83 | | | | | | |
| | C | | 7.74 | | 7.36 | 7.42 | | 22.20 | 9.00 | | 9.03 | 8.10 | 7.77 | | | | | | |
| | Avg | | 7.68 | | 7.24 | 7.19 | | 22.32 | 9.58 | | 9.04 | 8.05 | 7.78 | 7.9 | 1.3 | 16.2% | 22.3 | 6.0 | 14 |
| | SD | | 0.06 | | 0.18 | 0.32 | | 0.22 | 0.55 | | 0.16 | 0.14 | 0.05 | | | | | | |
| Sample D: Turmeric Smoothie Additive (mg/g) | A | | 2.378 | | | 1.640 | | | | 2.0 | | 2.012 | 1.690 | | | | | | |
| | B | | 2.309 | | | 1.740 | | | | 2.0 | | 2.720 | 1.720 | | | | | | |
| | C | | 2.294 | | | 1.740 | | | | 2.0 | | 2.590 | 1.740 | | | | | | |
| | Avg | | 2.327 | | | 1.707 | | | | 2.0 | | 2.441 | 1.717 | 1.90 | 0.28 | 14.6% | 2.44 | 1.49 | 13 |
| | SD | | 0.045 | | | 0.058 | | | | 0.0 | | 0.377 | 0.025 | | | | | | |
| Sample E: Turmeric Root Capsule (mg/g) | A | | 6.240 | | | 1.640 | | | | 6.0 | | 6.105 | 5.630 | | | | | | |
| | B | | 6.463 | | | 1.740 | | | | 6.0 | | 5.900 | 5.670 | | | | | | |
| | C | | 6.349 | | | 1.740 | | | | 6.0 | | 6.102 | 5.700 | | | | | | |
| | Avg | | 6.351 | | | 1.707 | | | | 6.0 | | 6.036 | 5.667 | 5.77 | 0.70 | 12.1% | 6.82 | 0.97 | 14 |
| | SD | | 0.112 | | | 0.058 | | | | 0.0 | | 0.118 | 0.035 | | | | | | |
| Sample F: Turmeric Extract/Root Capsule with Black Pepper (mg/g) | A | | 4.750 | | | 4.200 | | | | 5.0 | | 5.150 | 3.990 | | | | | | |
| | B | | 4.670 | | | 3.980 | | | | 5.0 | | 5.330 | 4.010 | | | | | | |
| | C | | 4.718 | | | 4.110 | | | | 5.0 | | 4.870 | 4.070 | | | | | | |
| | Avg | | 4.713 | | | 4.097 | | | | 5.0 | | 5.117 | 4.023 | 4.24 | 0.85 | 20.1% | 5.70 | 0.87 | 14 |
| | SD | | 0.040 | | | 0.111 | | | | 0.0 | | 0.232 | 0.042 | | | | | | |
| Sample G: Turmeric Extract/Root Capsule with Black Pepper & Coconut Oil (mg/g) | A | | 2.0 | | 64.5 | 31.2 | | 200.0 | | | 57.7 | 54.6 | 67.6 | | | | | | |
| | B | | 2.1 | | 64.3 | 46.6 | | 128.1 | | | 63.7 | 69.2 | 67.9 | | | | | | |
| | C | | 1.9 | | 62.5 | 54.5 | | 165.5 | | | 61.6 | 69.9 | 67.5 | | | | | | |
| | Avg | | 2.0 | | 63.8 | 44.1 | | 164.5 | | | 61.0 | 64.6 | 67.6 | 61 | 12 | 18.9% | 165 | 44 | 14 |
| | SD | | 0.1 | | 1.1 | 11.8 | | 36.0 | | | 3.0 | 8.7 | 0.2 | | | | | | |
| Sample H: Turmeric Tincture (mg/L) | A | | 138 | | 0.269 | 237 | | 0.743 | | | 3150 | 253.3 | 195 | | | | | | |
| | B | | 165 | | 0.272 | 236 | | 0.719 | | | 3070 | 282.0 | 196 | | | | | | |
| | C | | 146 | | 0.286 | 237 | | 0.718 | | | 2740 | 222.4 | 194 | | | | | | |
| | Avg | | 150 | | 0.276 | 237 | | 0.727 | | | 2987 | 252.5 | 195 | 159 | 123 | 77.3% | 2987 | 0.28 | 14 |
| | SD | | 14 | | 0.009 | 1 | | 0.014 | | | 217 | 29.8 | 1 | | | | | | |
| Sample I: Turmeric Gelcap with Coconut (mg/g) | A | | 6.17 | | 4.95 | 4.33 | | 14.37 | | | 7.02 | 5.13 | 5.06 | | | | | | |
| | B | | 6.34 | | 4.75 | 4.56 | | 14.86 | | | 6.57 | 4.78 | 5.06 | | | | | | |
| | C | | 6.10 | | 4.68 | 4.70 | | 13.65 | | | 7.03 | 5.93 | 4.98 | | | | | | |
| | Avg | | 6.20 | | 4.79 | 4.53 | | 14.29 | | | 6.87 | 5.28 | 5.03 | 4.82 | 0.73 | 15.2% | 14.29 | 3.36 | 13 |
| | SD | | 0.12 | | 0.14 | 0.19 | | 0.61 | | | 0.26 | 0.59 | 0.05 | | | | | | |
| Sample J: Turmeric Gelcap, Liquid Curcumin (mg/g) | A | | 11.14 | | | 10.00 | | | | 11.0 | | 11.02 | 10.10 | | | | | | |
| | B | | 11.17 | | | 10.30 | | | | 11.0 | | 11.77 | 10.30 | | | | | | |
| | C | | 10.89 | | | 10.50 | | | | 11.0 | | 11.34 | 10.08 | | | | | | |
| | Avg | | 11.07 | | | 10.27 | | | | 11.0 | | 11.38 | 10.16 | 10.13 | 0.85 | 8.4% | 11.38 | 2.21 | 14 |
| | SD | | 0.2 | | | 0.25 | | | | 0.0 | | 0.38 | 0.12 | | | | | | |

**Table 2-6.2.** Data summary table for desmethoxycurcumin (DMC) in turmeric commercial products. Individual results are displayed in this table for ten of the laboratories that requested samples (O420 through O437), while community results are shown for all laboratories participating the study. Results for additional laboratories can be found in Tables 2-6.1 and 2-6.3. Data highlighted in red have been flagged as potential outliers (e.g., Grubb and/or Cochran) by the NIST software package.

| | | Desmethoxycurcumin | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Individual Results - Page 2 of 3 | | | | | | | | | | | Community Results | | | | | |
| | Lab | NIST | O420 | O421 | O423 | O425 | O428 | O429 | O431 | O433 | O434 | O437 | Mean | SD | % RSD | Max | Min | N |
| Sample A: SRM 3299 Turmeric Rhizome (mg/g) | A | | 4.74 | 2.36 | | | | | 3.56 | 3.15 | | 2.98 | | | | | | |
| | B | | 4.74 | 2.33 | | | | | 3.59 | 2.95 | | 2.98 | | | | | | |
| | C | | 4.92 | 2.43 | | | | | 3.62 | 2.99 | | 3.04 | | | | | | |
| | D | | | | | | | | | | | | | | | | | |
| | E | | | | | | | | | | | | | | | | | |
| | F | | | | | | | | | | | | | | | | | |
| | Avg | 11.04 | 4.80 | 2.37 | | | | | 3.59 | 3.03 | | 3.00 | 3.37 | 0.72 | 21.3% | 11.44 | 0.82 | 19 |
| | SD | 0.21 | 0.11 | 0.05 | | | | | 0.03 | 0.11 | | 0.03 | | | | | | |
| Sample B: SRM 3300 Turmeric Extract (mg/g) | A | | 132.5 | 115.0 | | 24.2 | | | 135.5 | 111.9 | | 107.1 | | | | | | |
| | B | | 123.5 | 97.3 | | | | | 120.2 | 111.7 | | 111.2 | | | | | | |
| | C | | 123.5 | 114.7 | | 24.9 | | | 122.0 | 111.3 | | 110.1 | | | | | | |
| | D | | | | | | | | | | | | | | | | | |
| | E | | | | | | | | | | | | | | | | | |
| | F | | | | | | | | | | | | | | | | | |
| | Avg | 822 | 126.5 | 109.0 | | 24.5 | | | 125.9 | 111.6 | | 109.5 | 117.2 | 9.5 | 8.1% | 320.7 | 11.9 | 20 |
| | SD | 11 | 5.2 | 10.1 | | 0.5 | | | 8.4 | 0.3 | | 2.1 | | | | | | |
| Sample C: Turmeric Root Powder (mg/g) | A | | 9.96 | | | | | | | 7.33 | | 6.86 | | | | | | |
| | B | | 10.50 | | | | | | | 6.94 | | 6.92 | | | | | | |
| | C | | 10.39 | | | | | | | 7.06 | | 6.98 | | | | | | |
| | Avg | | 10.28 | | | | | | | 7.11 | | 6.92 | 7.9 | 1.3 | 16.2% | 22.3 | 6.0 | 14 |
| | SD | | 0.29 | | | | | | | 0.20 | | 0.06 | | | | | | |
| Sample D: Turmeric Smoothie Additive (mg/g) | A | | | 1.530 | | | | | 2.088 | 1.948 | | 1.960 | | | | | | |
| | B | | | 1.460 | | | | | 2.096 | 1.902 | | 1.840 | | | | | | |
| | C | | | 1.470 | | | | | 2.150 | 2.724 | | 1.860 | | | | | | |
| | Avg | | | 1.487 | | | | | 2.111 | 2.191 | | 1.887 | 1.90 | 0.28 | 14.6% | 2.19 | 1.49 | 14 |
| | SD | | | 0.038 | | | | | 0.034 | 0.462 | | 0.064 | | | | | | |
| Sample E: Turmeric Root Capsule (mg/g) | A | | | 4.920 | | 0.700 | | | 6.750 | 5.588 | | 5.570 | | | | | | |
| | B | | | 4.820 | | 1.090 | | | 6.640 | 5.761 | | 5.640 | | | | | | |
| | C | | | 4.810 | | 1.120 | | | 6.650 | 5.650 | | 5.550 | | | | | | |
| | Avg | | | 4.850 | | 0.970 | | | 6.680 | 5.666 | | 5.587 | 5.77 | 0.70 | 12.1% | 6.82 | 0.97 | 15 |
| | SD | | | 0.061 | | 0.234 | | | 0.061 | 0.088 | | 0.047 | | | | | | |
| Sample F: Turmeric Extract/Root Capsule with Black Pepper (mg/g) | A | | | 3.370 | | 0.870 | | | 5.600 | 4.399 | | 4.230 | | | | | | |
| | B | | | 3.200 | | 0.830 | | | 5.550 | 4.540 | | 4.190 | | | | | | |
| | C | | | 3.330 | | 0.900 | | | 5.420 | 4.579 | | 4.080 | | | | | | |
| | Avg | | | 3.300 | | 0.867 | | | 5.523 | 4.506 | | 4.167 | 4.24 | 0.85 | 20.1% | 5.70 | 0.87 | 15 |
| | SD | | | 0.089 | | 0.035 | | | 0.093 | 0.095 | | 0.078 | | | | | | |
| Sample G: Turmeric Extract/Root Capsule with Black Pepper & Coconut Oil (mg/g) | A | | 66.8 | | | | | | | 63.6 | | 58.8 | | | | | | |
| | B | | 66.7 | | | | | | | 51.7 | | 57.7 | | | | | | |
| | C | | 67.9 | | | | | | | 60.3 | | 57.1 | | | | | | |
| | Avg | | 67.1 | | | | | | | 58.5 | | 57.9 | 61 | 12 | 18.9% | 165 | 44 | 14 |
| | SD | | 0.6 | | | | | | | 6.1 | | 0.9 | | | | | | |
| Sample H: Turmeric Tincture (mg/L) | A | | 1.24 | | | | | | | 198 | | 218 | | | | | | |
| | B | | 1.20 | | | | | | | 200 | | 214 | | | | | | |
| | C | | 1.23 | | | | | | | 200 | | 211 | | | | | | |
| | Avg | | 1.22 | | | | | | | 199 | | 214 | 159 | 123 | 77.3% | 2987 | 0.28 | 14 |
| | SD | | 0.02 | | | | | | | 1 | | 4 | | | | | | |
| Sample I: Turmeric Gelcap with Coconut (mg/g) | A | | 5.54 | | | | | | | 4.65 | | 4.39 | | | | | | |
| | B | | 5.66 | | | | | | | 4.65 | | 4.57 | | | | | | |
| | C | | 5.51 | | | | | | | 4.28 | | 4.39 | | | | | | |
| | Avg | | 5.57 | | | | | | | 4.52 | | 4.45 | 4.82 | 0.73 | 15.2% | 14.29 | 3.36 | 13 |
| | SD | | 0.08 | | | | | | | 0.22 | | 0.10 | | | | | | |
| Sample J: Turmeric Gelcap, Liquid Curcumin (mg/g) | A | | | 8.98 | | 2.11 | | | 12.20 | 9.57 | | 9.98 | | | | | | |
| | B | | | 8.92 | | 2.15 | | | 10.90 | 9.75 | | 9.96 | | | | | | |
| | C | | | 9.27 | | 2.37 | | | 10.90 | 9.84 | | 10.30 | | | | | | |
| | Avg | | | 9.06 | | 2.21 | | | 11.33 | 9.72 | | 10.08 | 10.13 | 0.85 | 8.4% | 11.33 | 2.21 | 15 |
| | SD | | | 0.19 | | 0.14 | | | 0.75 | 0.14 | | 0.19 | | | | | | |

**Table 2-6.3.** Data summary table for desmethoxycurcumin (DMC) in turmeric commercial products. Individual results are displayed in this table for ten of the laboratories that requested samples (O433 through O458), while community results are shown for all laboratories participating the study. Results for additional laboratories can be found in Tables 2-6.1 and 2-6.2. Data highlighted in red have been flagged as potential outliers (e.g., difference from reference value, Grubb and/or Cochran) by the NIST software package.

| | | Desmethoxycurcumin | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Individual Results - Page 3 of 3 | | | | | | | | | | | Community Results | | | | | |
| | Lab | NIST | O440 | O443 | O446 | O449 | O452 | O455 | O458 | O459 | O460 | O461 | Mean | SD | % RSD | Max | Min | N |
| Sample A: SRM 3299 Turmeric Rhizome (mg/g) | A | | 3.21 | | 3.19 | 3.43 | 3.84 | 2.82 | | 0.95 | 3.62 | 2.46 | | | | | | |
| | B | | | | 3.13 | 3.55 | 3.84 | 2.73 | | 0.78 | 3.51 | 2.37 | | | | | | |
| | C | | | | 3.13 | 3.11 | 3.71 | 2.93 | | 0.72 | 3.66 | 2.42 | | | | | | |
| | D | | | | 3.09 | 2.99 | | 3.43 | | | | | | | | | | |
| | E | | | | 2.97 | 3.03 | | 3.46 | | | | | | | | | | |
| | F | | | | 2.85 | 3.33 | | 3.47 | | | | | | | | | | |
| | Avg | 11.04 | 3.21 | | 3.06 | 3.24 | 3.80 | 3.14 | | 0.82 | 3.60 | 2.42 | 3.37 | 0.72 | 21.3% | 11.44 | 0.82 | 19 |
| | SD | 0.21 | | | 0.13 | 0.23 | 0.07 | 0.35 | | 0.12 | 0.08 | 0.05 | | | | | | |
| Sample B: SRM 3300 Turmeric Extract (mg/g) | A | | 107.0 | | 104.5 | 139.5 | 124.0 | 117.4 | | 115.0 | 11.9 | 84.0 | | | | | | |
| | B | | | | 107.7 | 128.6 | 120.3 | 118.7 | | 112.9 | 11.8 | 85.4 | | | | | | |
| | C | | | | 104.1 | 135.0 | 121.3 | 120.0 | | 114.0 | 12.1 | 83.7 | | | | | | |
| | D | | | | 106.6 | 112.2 | | 115.7 | | | | | | | | | | |
| | E | | | | 111.2 | 114.8 | | 116.4 | | | | | | | | | | |
| | F | | | | 109.4 | 115.1 | | 116.0 | | | | | | | | | | |
| | Avg | 822 | 107.0 | | 107.3 | 124.2 | 121.9 | 117.4 | | 114.0 | 11.9 | 84.4 | 117.2 | 9.5 | 8.1% | 320.7 | 11.9 | 20 |
| | SD | 11 | | | 2.8 | 11.7 | 1.9 | 1.7 | | 1.1 | 0.1 | 0.9 | | | | | | |
| Sample C: Turmeric Root Powder (mg/g) | A | | 7.57 | | 6.92 | 7.84 | | 7.24 | | | | 5.72 | | | | | | |
| | B | | 7.53 | | 6.86 | 8.24 | | 7.15 | | | | 6.28 | | | | | | |
| | C | | 7.42 | | 6.83 | 7.67 | | 7.30 | | | | 6.06 | | | | | | |
| | Avg | | 7.51 | | 6.87 | 7.91 | | 7.23 | | | | 6.02 | 7.9 | 1.3 | 16.2% | 22.3 | 6.0 | 15 |
| | SD | | 0.08 | | 0.05 | 0.29 | | 0.08 | | | | 0.28 | | | | | | |
| Sample D: Turmeric Smoothie Additive (mg/g) | A | | | | 1.539 | 1.865 | 1.983 | 1.679 | | 1.670 | 2.076 | | | | | | | |
| | B | | | | 1.549 | 1.806 | 1.986 | 1.578 | | 1.740 | 2.172 | | | | | | | |
| | C | | | | 1.582 | 1.776 | 1.973 | 1.601 | | 1.840 | 2.067 | | | | | | | |
| | Avg | | | | 1.557 | 1.816 | 1.981 | 1.619 | | 1.750 | 2.105 | | 1.90 | 0.28 | 14.6% | 2.19 | 1.49 | 14 |
| | SD | | | | 0.022 | 0.045 | 0.007 | 0.053 | | 0.085 | 0.058 | | | | | | | |
| Sample E: Turmeric Root Capsule (mg/g) | A | | | | 5.300 | 5.775 | 6.090 | 5.998 | | 4.970 | 6.688 | | | | | | | |
| | B | | | | 5.289 | 5.696 | 6.215 | 5.563 | | 4.740 | 6.695 | | | | | | | |
| | C | | | | 5.213 | 5.772 | 6.105 | 5.607 | | 4.920 | 7.083 | | | | | | | |
| | Avg | | | | 5.267 | 5.748 | 6.137 | 5.723 | | 4.877 | 6.822 | | 5.77 | 0.70 | 12.1% | 6.82 | 0.97 | 15 |
| | SD | | | | 0.047 | 0.045 | 0.068 | 0.239 | | 0.121 | 0.226 | | | | | | | |
| Sample F: Turmeric Extract/Root Capsule with Black Pepper (mg/g) | A | | | | 3.546 | 3.985 | 4.894 | 3.873 | | 3.430 | 5.823 | | | | | | | |
| | B | | | | 3.524 | 4.006 | 4.869 | 4.019 | | 3.360 | 5.622 | | | | | | | |
| | C | | | | 3.542 | 3.984 | 4.911 | 4.027 | | 3.450 | 5.659 | | | | | | | |
| | Avg | | | | 3.537 | 3.992 | 4.891 | 3.973 | | 3.413 | 5.701 | | 4.24 | 0.85 | 20.1% | 5.70 | 0.87 | 15 |
| | SD | | | | 0.012 | 0.012 | 0.021 | 0.087 | | 0.047 | 0.107 | | | | | | | |
| Sample G: Turmeric Extract/Root Capsule with Black Pepper & Coconut Oil (mg/g) | A | | 57.3 | | 56.9 | 78.5 | | 54.1 | | | | 44.8 | | | | | | |
| | B | | 56.5 | | 56.6 | 77.8 | | 56.7 | | | | 45.1 | | | | | | |
| | C | | 59.4 | | 56.2 | 86.3 | | 54.9 | | | | 45.7 | | | | | | |
| | Avg | | 57.7 | | 56.6 | 80.9 | | 55.2 | | | | 45.2 | 61 | 12 | 18.9% | 165 | 44 | 14 |
| | SD | | 1.5 | | 0.4 | 4.8 | | 1.3 | | | | 0.5 | | | | | | |
| Sample H: Turmeric Tincture (mg/L) | A | | 8.20 | | 221.2 | 229 | | 128 | | | | 213 | | | | | | |
| | B | | 8.14 | | 217.3 | 217 | | 131 | | | | 201 | | | | | | |
| | C | | 8.09 | | 219.3 | 252 | | 127 | | | | 215 | | | | | | |
| | Avg | | 8.14 | | 219.3 | 233 | | 129 | | | | 210 | 159 | 123 | 77.3% | 2987 | 0.28 | 14 |
| | SD | | 0.06 | | 2.0 | 18 | | 2 | | | | 8 | | | | | | |
| Sample I: Turmeric Gelcap with Coconut (mg/g) | A | | 4.66 | | 4.62 | 4.64 | | 4.60 | | | | 3.40 | | | | | | |
| | B | | | | 4.31 | 4.58 | | 4.33 | | | | 3.31 | | | | | | |
| | C | | | | 4.23 | 4.03 | | 4.09 | | | | 3.38 | | | | | | |
| | Avg | | 4.66 | | 4.39 | 4.42 | | 4.34 | | | | 3.36 | 4.82 | 0.73 | 15.2% | 14.29 | 3.36 | 13 |
| | SD | | | | 0.21 | 0.34 | | 0.26 | | | | 0.05 | | | | | | |
| Sample J: Turmeric Gelcap, Liquid Curcumin (mg/g) | A | | | | 9.65 | 10.46 | 10.51 | 10.11 | | 10.12 | 10.89 | | | | | | | |
| | B | | | | 9.30 | 9.83 | 10.52 | 10.62 | | 9.84 | 11.08 | | | | | | | |
| | C | | | | 9.39 | 10.12 | 10.56 | 10.29 | | 9.62 | 10.92 | | | | | | | |
| | Avg | | | | 9.44 | 10.14 | 10.53 | 10.34 | | 9.86 | 10.96 | | 10.13 | 0.85 | 8.4% | 11.33 | 2.21 | 15 |
| | SD | | | | 0.18 | 0.32 | 0.03 | 0.26 | | 0.25 | 0.11 | | | | | | | |

**Table 2-7.** Data summary table for desmethoxycurcumin (DMC) in turmeric commercial products. Individual results are displayed in this table for the laboratories that reported using AOAC 2016.16 for analysis. Data highlighted in red have been flagged as potential outliers (e.g., Grubb and/or Cochran) by the NIST software package. Data shown in italicized font were collected in the second part of the study. Data for laboratory O411 was not included in the collaborative study because only a single sample was analyzed.

| | | DMC by AOAC 2016.16 | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Individual Results | | | | | | | | | | | Community Results | | | | | |
| | Lab | NIST | O404 | O407 | ~~O411~~ | O416 | O419 | O433 | O437 | O446 | O449 | O455 | Mean | SD | % RSD | Max | Min | N |
| Sample A: SRM 3299 Turmeric Rhizome (mg/g) | A | | 3.37 | 2.95 | | 4.73 | 3.28 | 3.15 | 2.98 | 3.19 | 3.43 | 2.82 | | | | | | |
| | B | | 3.37 | 2.94 | | 3.32 | 3.36 | 2.95 | 2.98 | 3.13 | 3.55 | 2.73 | | | | | | |
| | C | | 3.42 | 2.96 | | 4.00 | 3.33 | 2.99 | 3.04 | 3.13 | 3.11 | 2.93 | | | | | | |
| | D | | *3.29* | *3.23* | | *3.77* | *3.50* | | | *3.09* | *2.99* | *3.43* | | | | | | |
| | E | | *3.51* | *3.18* | | *3.35* | *3.51* | | | *2.97* | *3.03* | *3.46* | | | | | | |
| | F | | *3.37* | *3.03* | | *3.21* | *3.55* | | | *2.85* | *3.33* | *3.47* | | | | | | |
| | Avg | 3.14 | 3.39 | 3.05 | | 3.73 | 3.42 | 3.03 | 3.00 | 3.06 | 3.24 | 3.14 | 3.21 | 0.24 | 7.3% | 3.73 | 3.00 | 9 |
| | SD | 0.06 | 0.07 | 0.13 | | 0.58 | 0.11 | 0.11 | 0.03 | 0.13 | 0.23 | 0.35 | | | | | | |
| Sample B: SRM 3300 Tumeric Extract (mg/g) | A | | 122.2 | 120.0 | | 141.7 | 121.4 | 111.9 | 107.1 | 104.5 | 139.5 | 117.4 | | | | | | |
| | B | | 114.1 | 110.0 | | 148.7 | 120.4 | 111.7 | 111.2 | 107.7 | 128.6 | 118.7 | | | | | | |
| | C | | 112.0 | 117.0 | | 121.8 | 125.3 | 111.3 | 110.1 | 104.1 | 135.0 | 120.0 | | | | | | |
| | D | | *116.0* | *125.0* | | *108.9* | *124.2* | | | *106.6* | *112.2* | *115.7* | | | | | | |
| | E | | *115.0* | *127.0* | | *120.0* | *126.1* | | | *111.2* | *114.8* | *116.4* | | | | | | |
| | F | | *118.1* | *125.0* | | *113.9* | *126.9* | | | *109.4* | *115.1* | *116.0* | | | | | | |
| | Avg | 117.1 | 116.2 | 120.7 | | 125.8 | 124.0 | 111.6 | 109.5 | 107.3 | 124.2 | 117.4 | 117.4 | 7.9 | 6.7% | 125.8 | 107.3 | 9 |
| | SD | 1.1 | 3.5 | 6.4 | | 15.9 | 2.6 | 0.3 | 2.1 | 2.8 | 11.7 | 1.7 | | | | | | |
| Sample C: Turmeric Root Powder (mg/g) | A | | *7.65* | 6.83 | ~~10.10~~ | 8.16 | *7.74* | *7.33* | 6.86 | 6.92 | 7.84 | *7.24* | | | | | | |
| | B | | *7.63* | 7.33 | ~~9.65~~ | 7.90 | *7.83* | *6.94* | 6.92 | 6.86 | 8.24 | *7.15* | | | | | | |
| | C | | *7.74* | 7.42 | ~~9.00~~ | 8.10 | *7.77* | *7.06* | 6.98 | 6.83 | 7.67 | *7.30* | | | | | | |
| | Avg | | *7.68* | 7.19 | ~~9.58~~ | 8.05 | *7.78* | *7.11* | 6.92 | 6.87 | 7.91 | *7.23* | 7.42 | 0.51 | 6.9% | 9.58 | 6.87 | 10 |
| | SD | | *0.06* | 0.32 | ~~0.55~~ | 0.14 | *0.05* | *0.20* | 0.06 | 0.05 | 0.29 | *0.08* | | | | | | |
| Sample D: Turmeric Smoothie Additive (mg/g) | A | | 2.38 | *1.64* | | *2.31* | 1.81 | 1.95 | *1.96* | *1.54* | *1.87* | 1.68 | | | | | | |
| | B | | 2.31 | *1.74* | | *2.00* | 1.84 | 1.90 | *1.84* | *1.55* | *1.81* | 1.58 | | | | | | |
| | C | | 2.29 | *1.74* | | *1.92* | 1.87 | 2.72 | *1.86* | *1.58* | *1.78* | 1.60 | | | | | | |
| | Avg | | 2.33 | *1.71* | | *2.08* | 1.84 | 2.19 | *1.89* | *1.56* | *1.82* | 1.62 | 1.89 | 0.30 | 15.8% | 2.33 | 1.56 | 9 |
| | SD | | 0.04 | *0.06* | | *0.21* | 0.03 | 0.46 | *0.06* | *0.02* | *0.05* | 0.05 | | | | | | |
| Sample E: Turmeric Root Capsule (mg/g) | A | | 6.24 | *6.14* | | *6.08* | 5.73 | 5.59 | *5.57* | *5.30* | *5.78* | 6.00 | | | | | | |
| | B | | 6.46 | *6.16* | | *6.03* | 5.76 | 5.76 | *5.64* | *5.29* | *5.70* | 5.56 | | | | | | |
| | C | | 6.35 | *5.80* | | *6.14* | 5.79 | 5.65 | *5.55* | *5.21* | *5.77* | 5.61 | | | | | | |
| | Avg | | 6.35 | *6.03* | | *6.08* | 5.76 | 5.67 | *5.59* | *5.27* | *5.75* | 5.72 | 5.80 | 0.35 | 6.1% | 6.35 | 5.27 | 9 |
| | SD | | 0.11 | *0.20* | | *0.06* | 0.03 | 0.09 | *0.05* | *0.05* | *0.04* | 0.24 | | | | | | |
| Sample F: Turmeric Extract/Root Capsule with Black Pepper (mg/g) | A | | 4.75 | *4.20* | | *4.22* | 4.10 | 4.40 | *4.23* | *3.55* | *3.99* | 3.87 | | | | | | |
| | B | | 4.67 | *3.98* | | *3.73* | 4.12 | 4.54 | *4.19* | *3.52* | *4.01* | 4.02 | | | | | | |
| | C | | 4.72 | *4.11* | | *4.34* | 4.17 | 4.58 | *4.08* | *3.54* | *3.98* | 4.03 | | | | | | |
| | Avg | | 4.71 | *4.10* | | *4.10* | 4.13 | 4.51 | *4.17* | *3.54* | *3.99* | 3.97 | 4.14 | 0.33 | 8.1% | 4.71 | 3.54 | 9 |
| | SD | | 0.04 | *0.11* | | *0.32* | 0.04 | 0.09 | *0.08* | *0.01* | *0.01* | 0.09 | | | | | | |
| Sample G: Turmeric Extract/Root Capsule with Black Pepper & Coconut Oil (mg/g) | A | | | 31 | | 55 | *68* | *64* | 59 | 57 | 79 | *54* | | | | | | |
| | B | | | 47 | | 69 | *68* | *52* | 58 | 57 | 78 | *57* | | | | | | |
| | C | | | 55 | | 70 | *67* | *60* | 57 | 56 | 86 | *55* | | | | | | |
| | Avg | | | 44 | | 65 | *68* | *59* | 58 | 57 | 81 | *55* | 60 | 11 | 18.7% | 81 | 44 | 8 |
| | SD | | | 12 | | 9 | *0* | *6* | 1 | 0 | 5 | *1* | | | | | | |
| Sample H: Turmeric Tincture (mg/L) | A | | *138* | 237 | | 253 | *195* | *198* | 218 | 221 | 229 | *128* | | | | | | |
| | B | | *165* | 236 | | 282 | *196* | *200* | 214 | 217 | 217 | *131* | | | | | | |
| | C | | *146* | 237 | | 222 | *194* | *200* | 211 | 219 | 252 | *127* | | | | | | |
| | Avg | | *150* | 237 | | 253 | *195* | *199* | 214 | 219 | 233 | *129* | 204 | 44 | 21.4% | 253 | 129 | 9 |
| | SD | | *14* | 1 | | 30 | *1* | *1* | 4 | 2 | 18 | *2* | | | | | | |
| Sample I: Turmeric Gelcap with Coconut (mg/g) | A | | | 4.33 | | 5.13 | *5.06* | *4.65* | 4.39 | 4.62 | 4.64 | *4.60* | | | | | | |
| | B | | | 4.56 | | 4.78 | *5.06* | *4.65* | 4.57 | 4.31 | 4.58 | *4.33* | | | | | | |
| | C | | | 4.70 | | 5.93 | *4.98* | *4.28* | 4.39 | 4.23 | 4.03 | *4.09* | | | | | | |
| | Avg | | | 4.53 | | 5.28 | *5.03* | *4.52* | 4.45 | 4.39 | 4.42 | *4.34* | 4.60 | 0.37 | 8.0% | 5.28 | 4.34 | 8 |
| | SD | | | 0.19 | | 0.59 | *0.05* | *0.22* | 0.10 | 0.21 | 0.34 | *0.26* | | | | | | |
| Sample J: Turmeric Gelcap, Liquid Curcumin (mg/g) | A | | 11.14 | *10.00* | | *9.36* | 10.10 | 9.57 | *9.98* | *9.65* | *10.46* | 10.11 | | | | | | |
| | B | | 11.17 | *10.30* | | *9.12* | 10.29 | 9.75 | *9.96* | *9.30* | *9.83* | 10.62 | | | | | | |
| | C | | 10.89 | *10.50* | | *9.25* | 10.08 | 9.84 | *10.30* | *9.39* | *10.12* | 10.29 | | | | | | |
| | Avg | | 11.07 | *10.27* | | *9.24* | 10.16 | 9.72 | *10.08* | *9.44* | *10.14* | 10.34 | 10.03 | 0.57 | 5.7% | 11.07 | 9.24 | 8 |
| | SD | | 0.15 | *0.25* | | *0.12* | 0.12 | 0.14 | *0.19* | *0.18* | *0.32* | 0.26 | | | | | | |

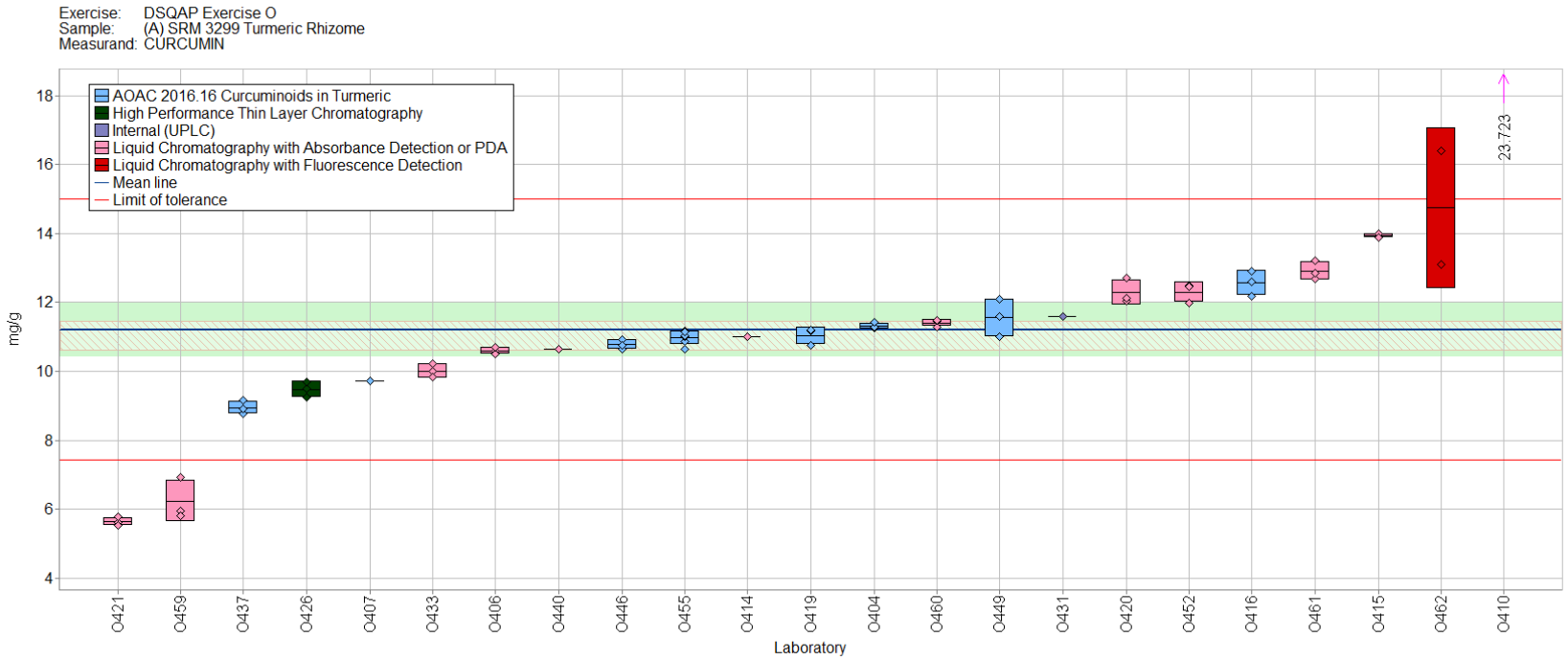


**Figure 2-1.** Curcumin in candidate SRM 3299 Turmeric Rhizome (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by its uncertainty ($U_{\text{NIST}}$) and represents the range that results in an acceptable $Z_{\text{NIST}}$ score, $|Z_{\text{NIST}}| \leq 2$.

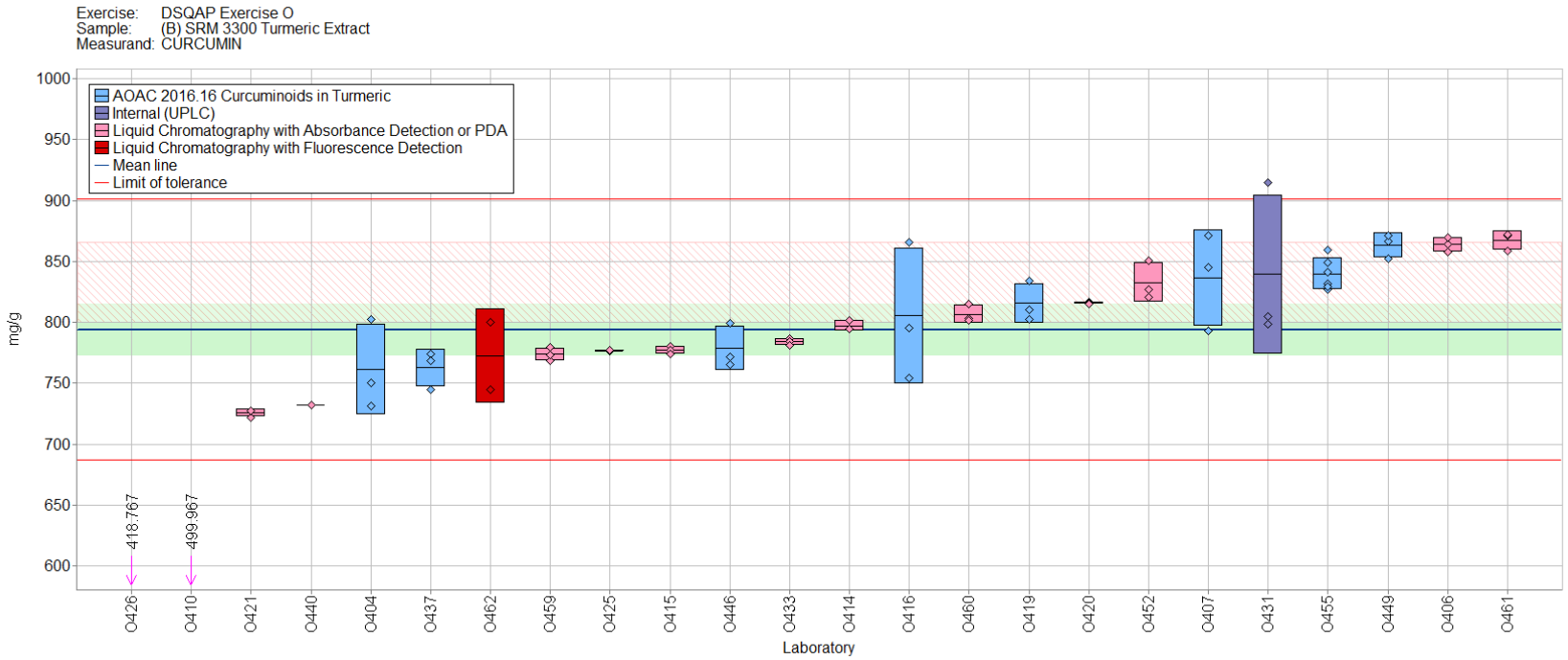


**Figure 2-2.** Curcumin in candidate SRM 3300 Turmeric Extract (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by its uncertainty ($U_{\text{NIST}}$) and represents the range that results in an acceptable $Z_{\text{NIST}}$ score, $|Z_{\text{NIST}}| \leq 2$.

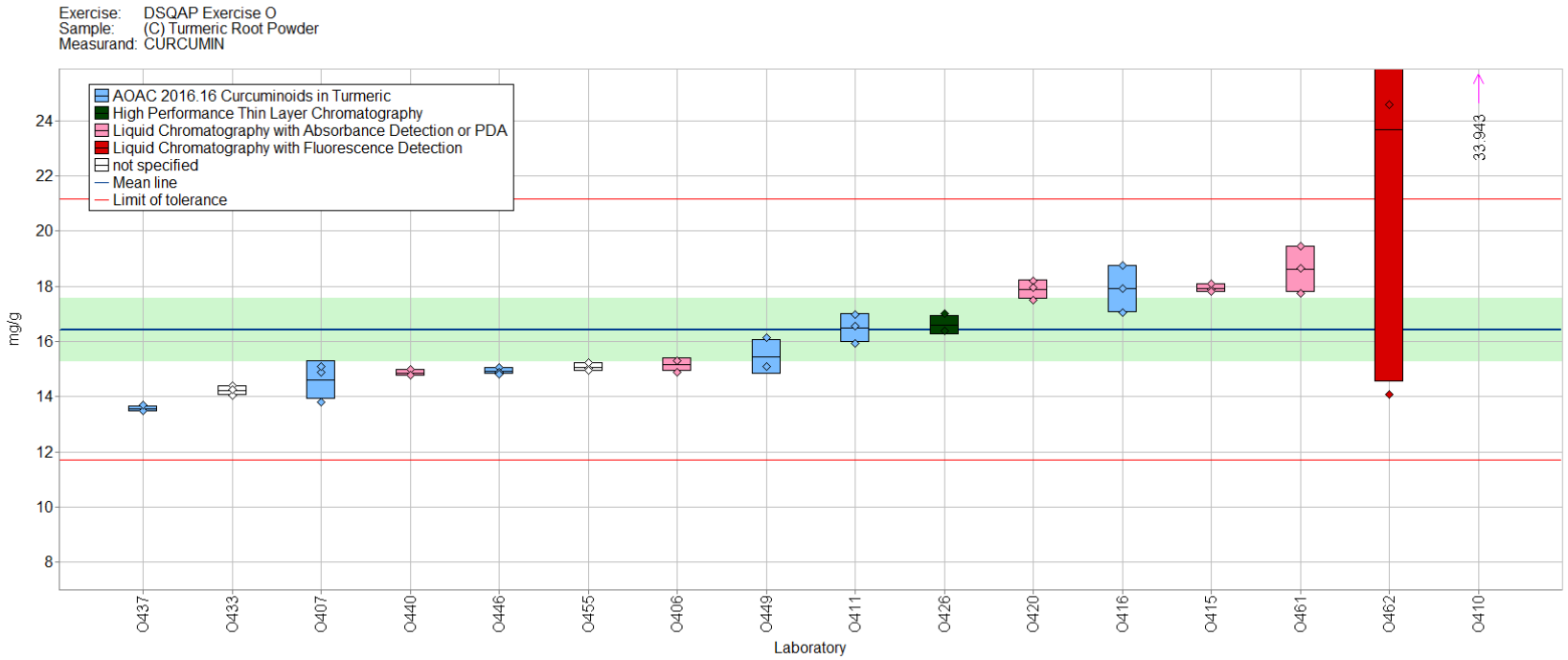


**Figure 2-3.** Curcumin in Turmeric Root Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the sample preparation method employed. The solid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

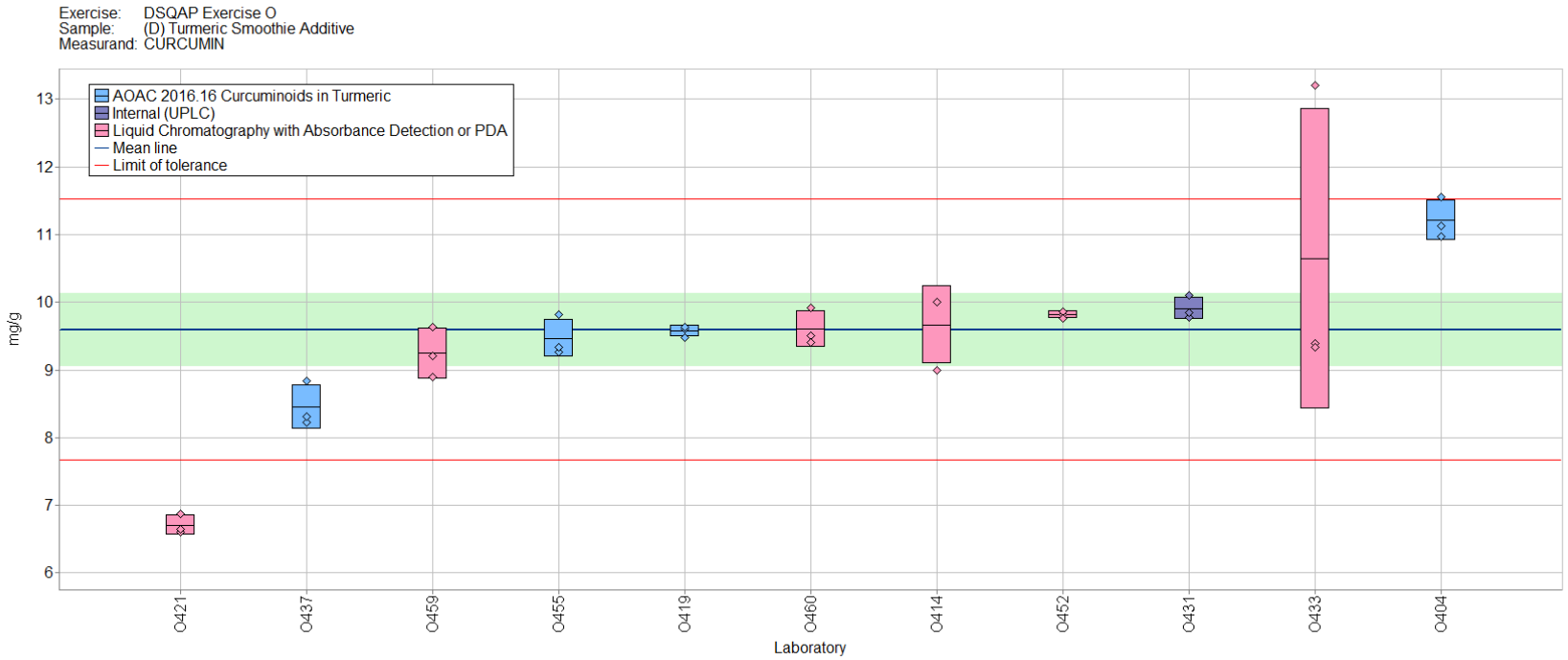


**Figure 2-4.** Curcumin in Turmeric Smoothie Additive (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the sample preparation method employed.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

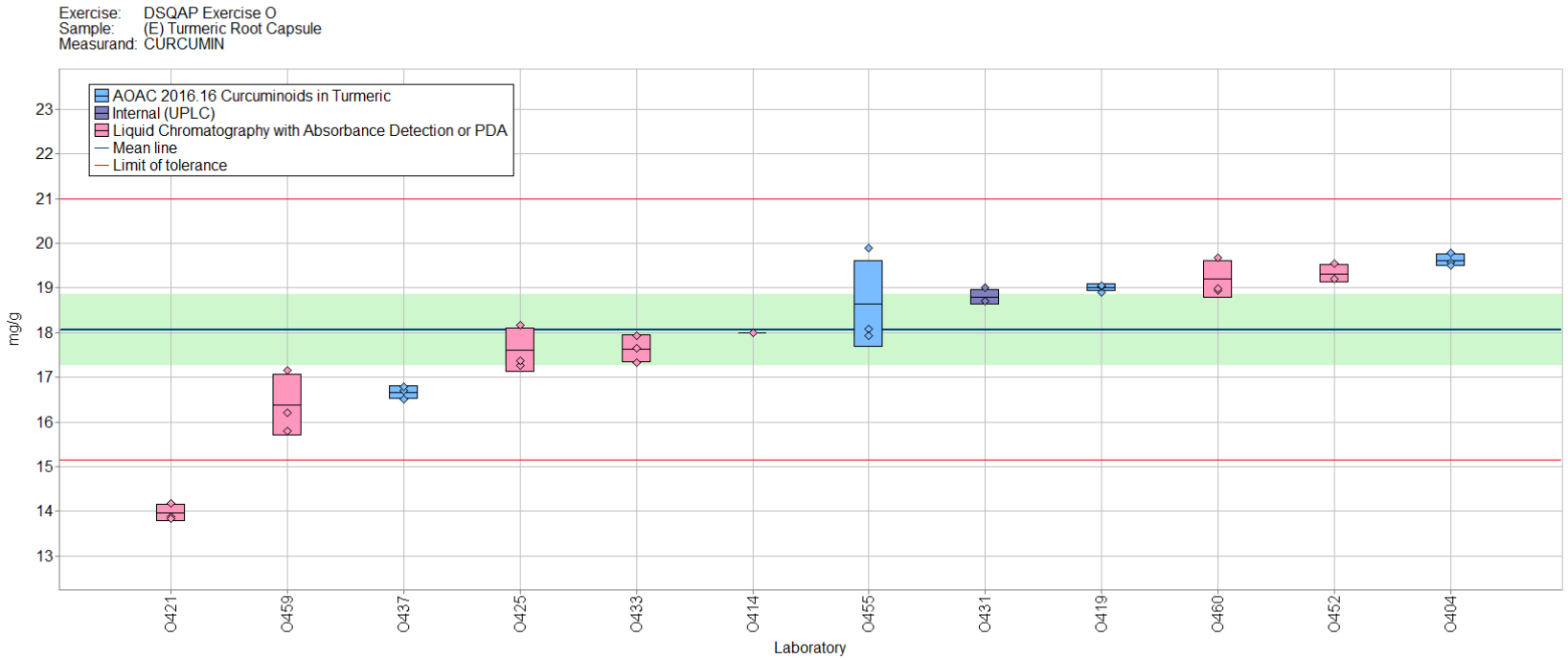


**Figure 2-5.** Curcumin in Turmeric Root Capsule (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the sample preparation method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$. A NIST value has not been determined in this material.

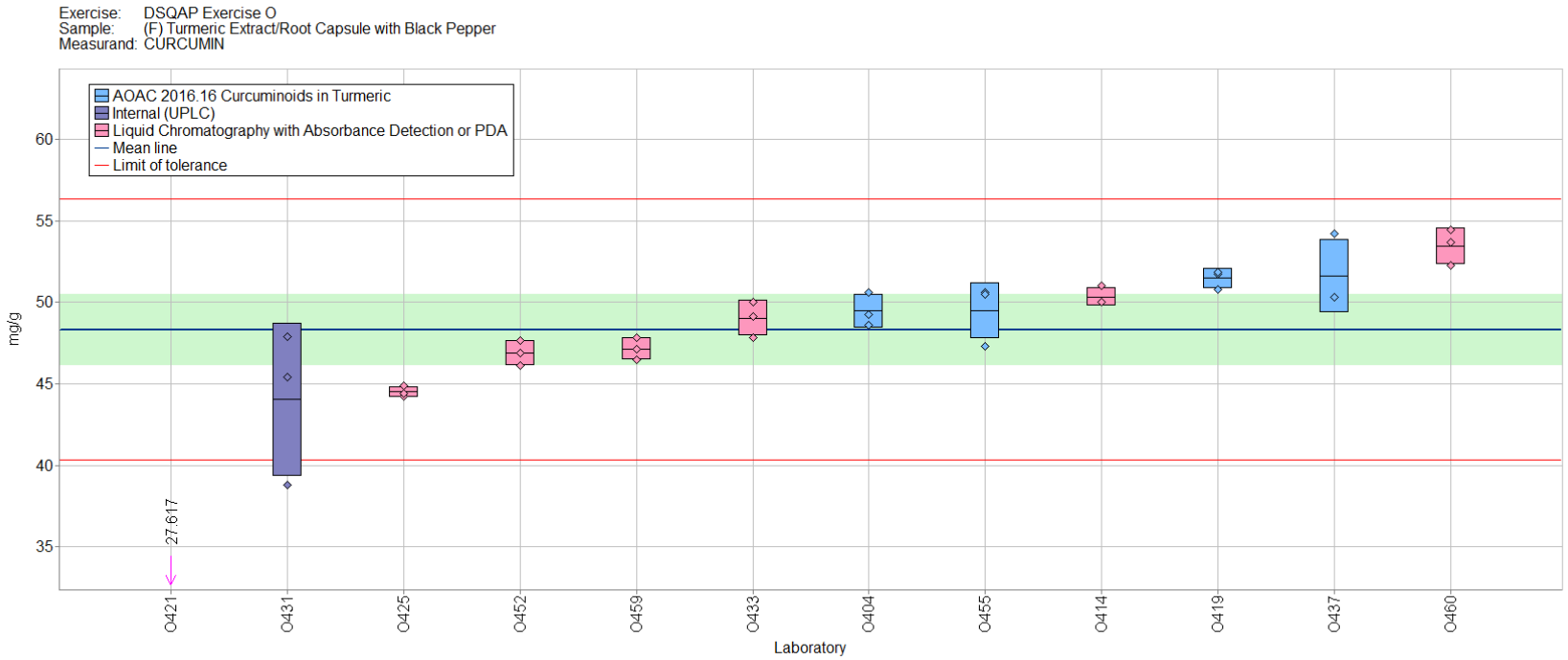


**Figure 2-6.** Curcumin in Turmeric Extract/Root Capsule with Black Pepper (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the sample preparation method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$. A NIST value has not been determined in this material.

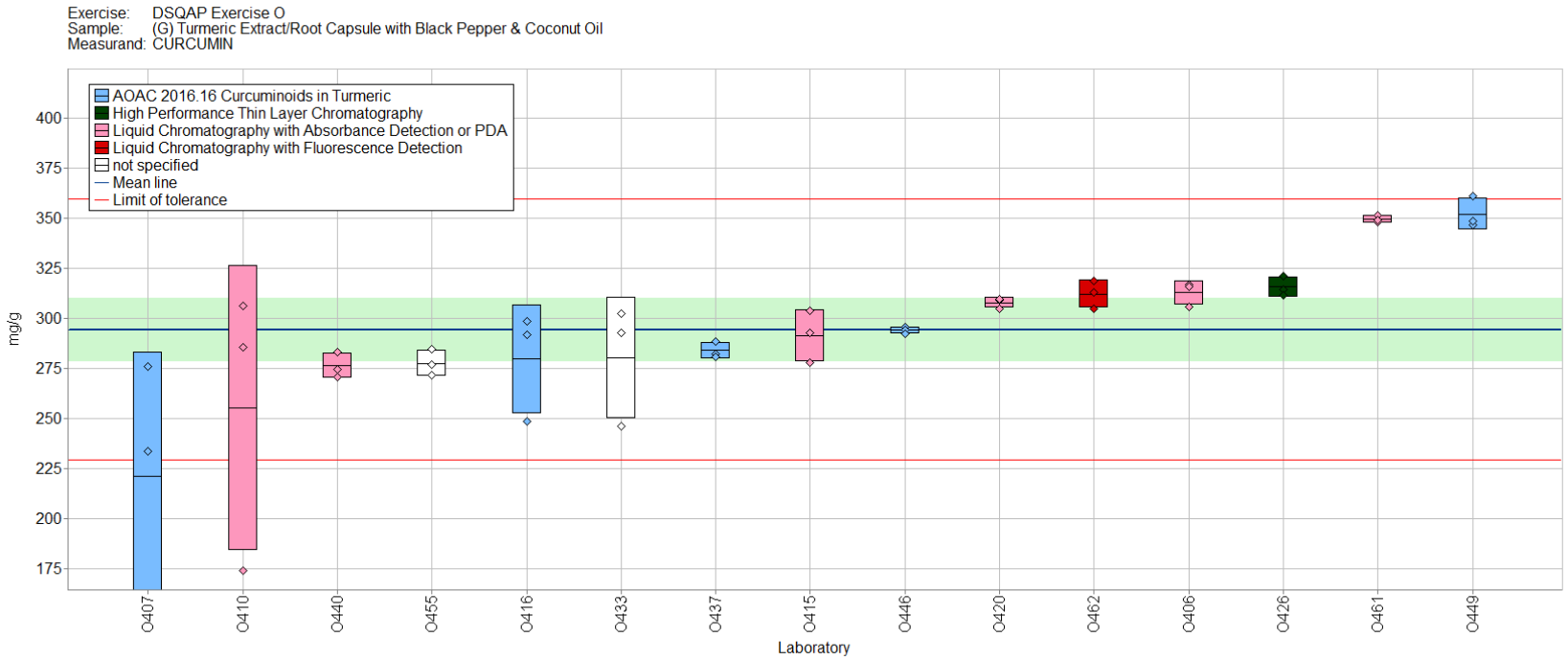


**Figure 2-7.** Curcumin in Turmeric Extract/Root Capsule with Black Pepper & Coconut Oil (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the sample preparation method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$. A NIST value has not been determined in this material.

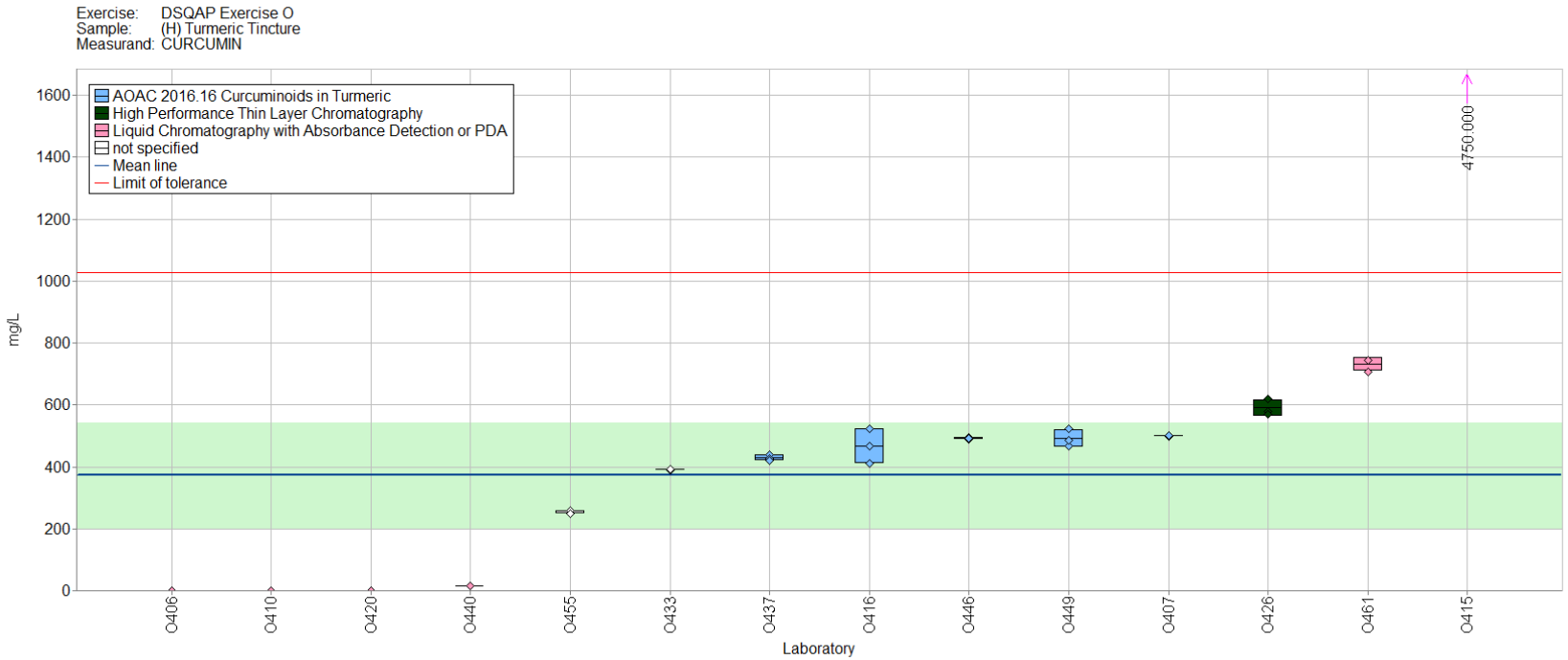


**Figure 2-8.** Curcumin in Turmeric Tincture (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the sample preparation method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the value above the consensus mean that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$, with the lower limit has been set to zero. A NIST value has not been determined in this material.

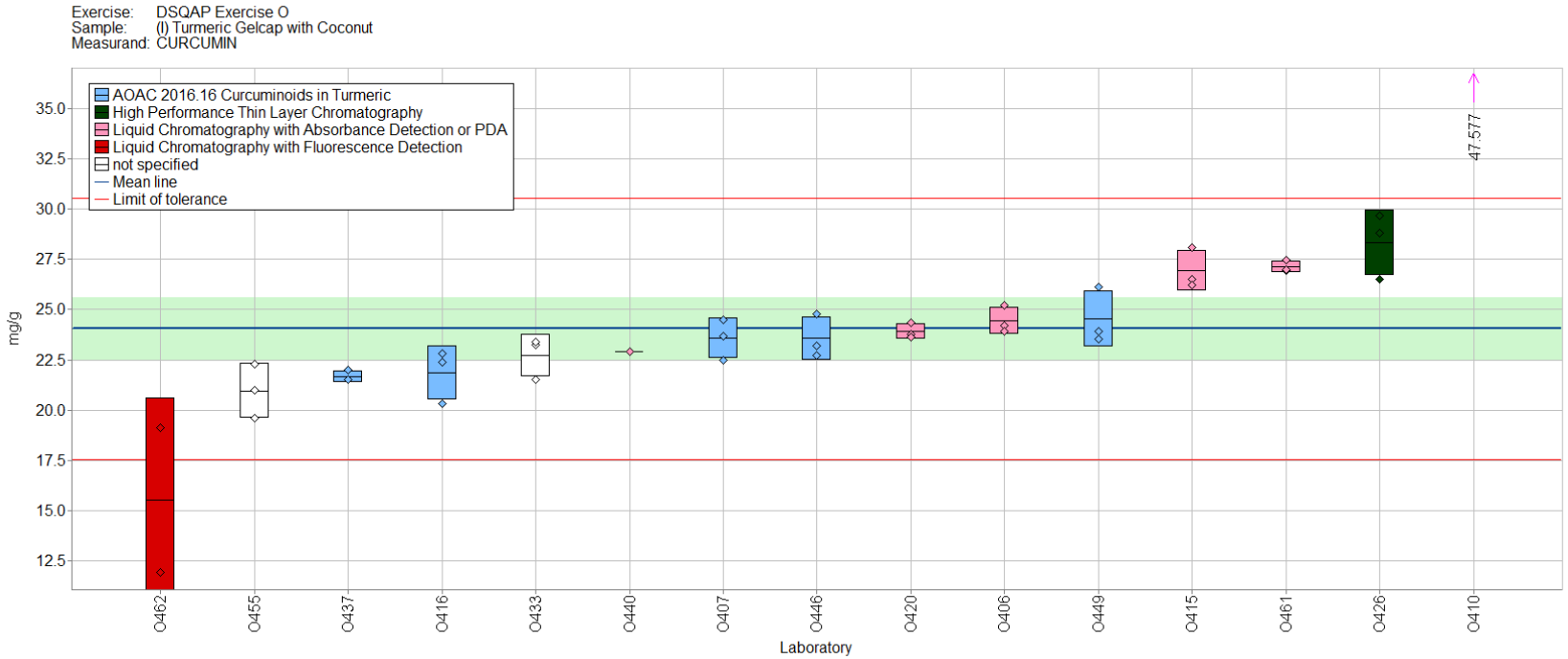


**Figure 2-9.** Curcumin in Turmeric Gelcap with Coconut (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the sample preparation method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$. A NIST value has not been determined in this material.

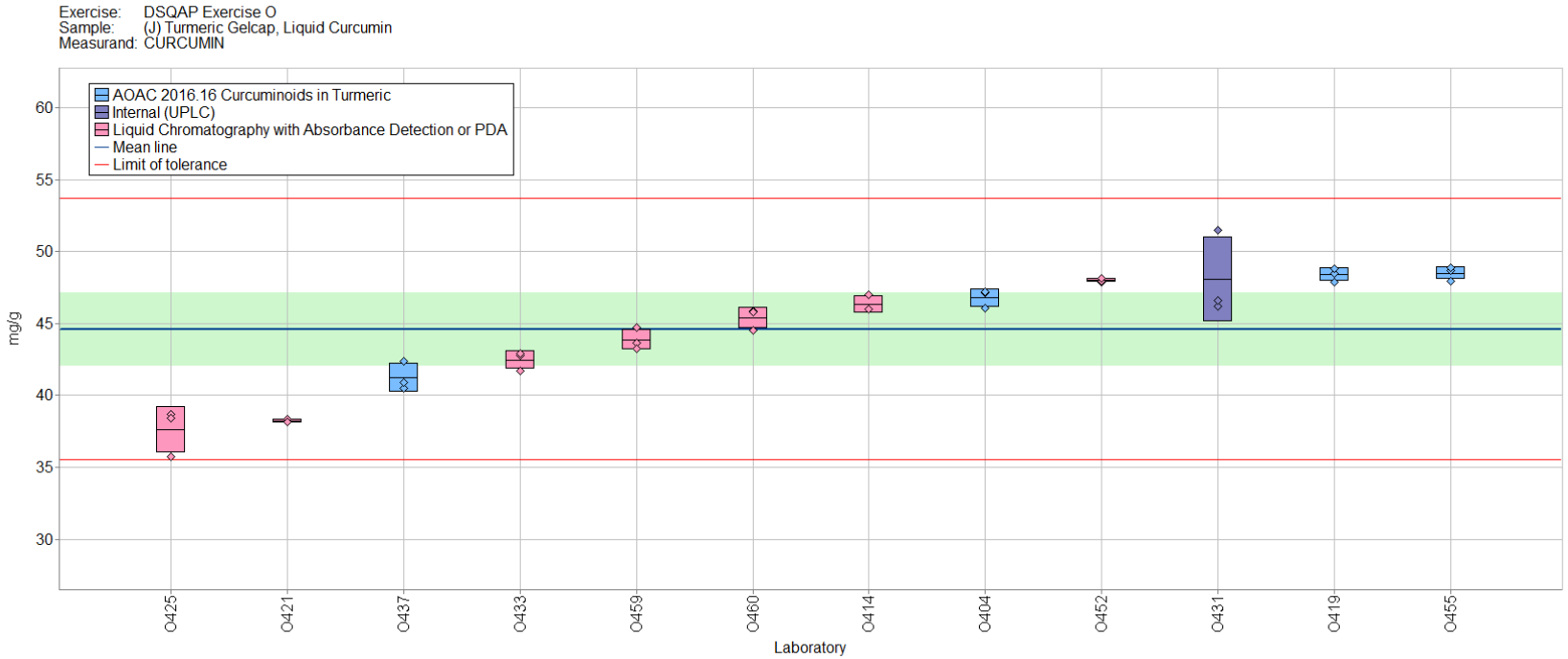


**Figure 2-10.** Curcumin in Turmeric Gelcap, Liquid Curcumin (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the sample preparation method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$. A NIST value has not been determined in this material.

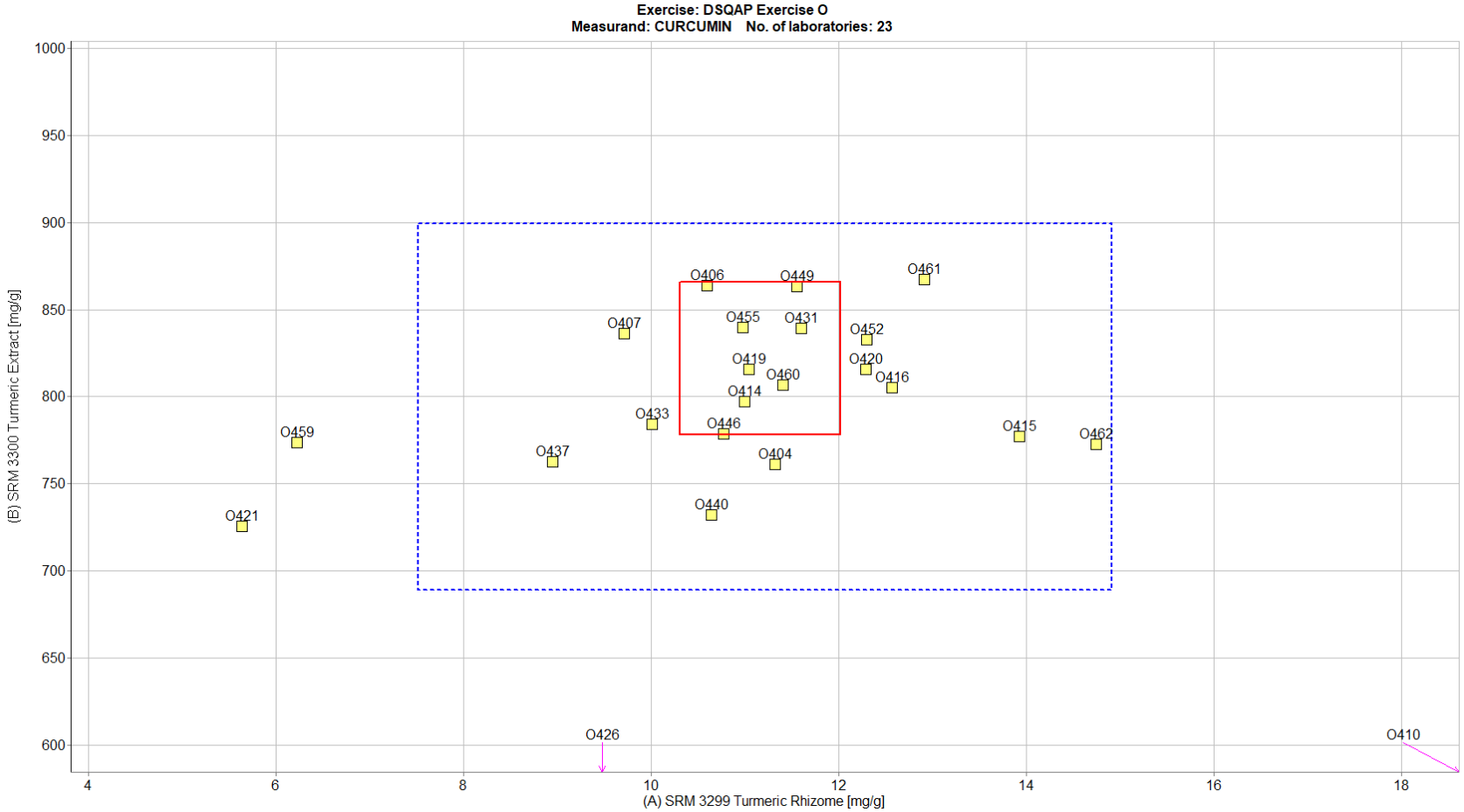


**Figure 2-11.** Laboratory means for curcumin in candidate SRM 3299 Turmeric Rhizome and candidate SRM 3300 Turmeric Extract (sample/sample comparison view). In this view, the individual laboratory mean for one sample (turmeric rhizome) is compared to the mean for a second sample (turmeric extract). The solid red box represents the NIST range of tolerance for the two samples, turmeric rhizome (x-axis) and turmeric extract (y-axis), which encompasses the NIST-determined values bounded by their uncertainties ($U_{\mathrm{NIST}}$) and represents the range that results in an acceptable $Z_{\mathrm{NIST}}$ score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for turmeric rhizome (x-axis) and turmeric extract (y-axis), calculated as the values above and below the consensus means that result in an acceptable $Z'_{\mathrm{comm}}$ score, $|Z'_{\mathrm{comm}}| \leq 2$.

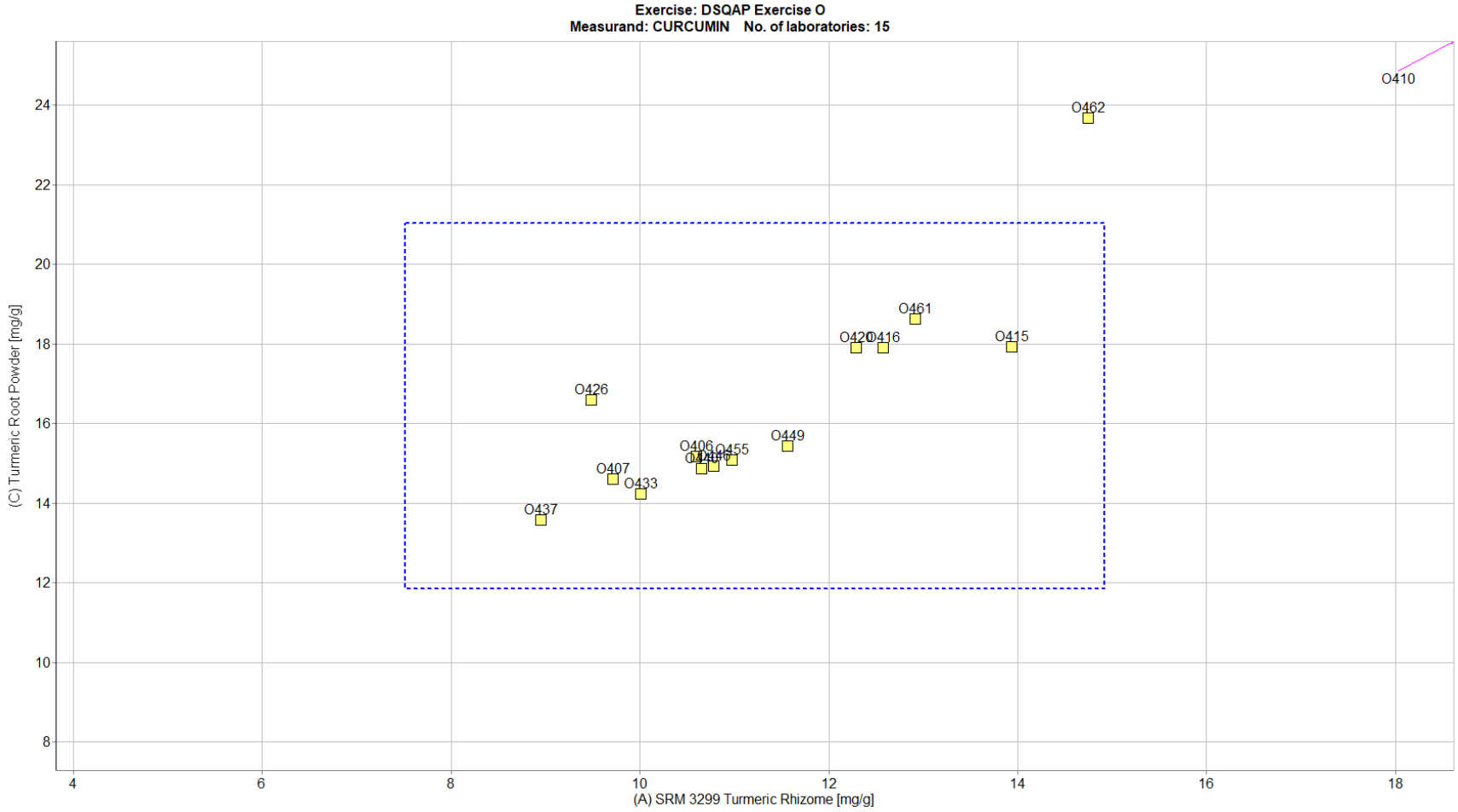


**Figure 2-12.** Laboratory means for curcumin in candidate SRM 3299 Turmeric Rhizome and Turmeric Root Powder (sample/sample comparison view). In this view, the individual laboratory mean for one sample (turmeric rhizome) is compared to the mean for a second sample (turmeric root powder). The dotted blue box represents the consensus range of tolerance for turmeric rhizome (x-axis) and turmeric root powder (y-axis), calculated as the values above and below the consensus means that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$.

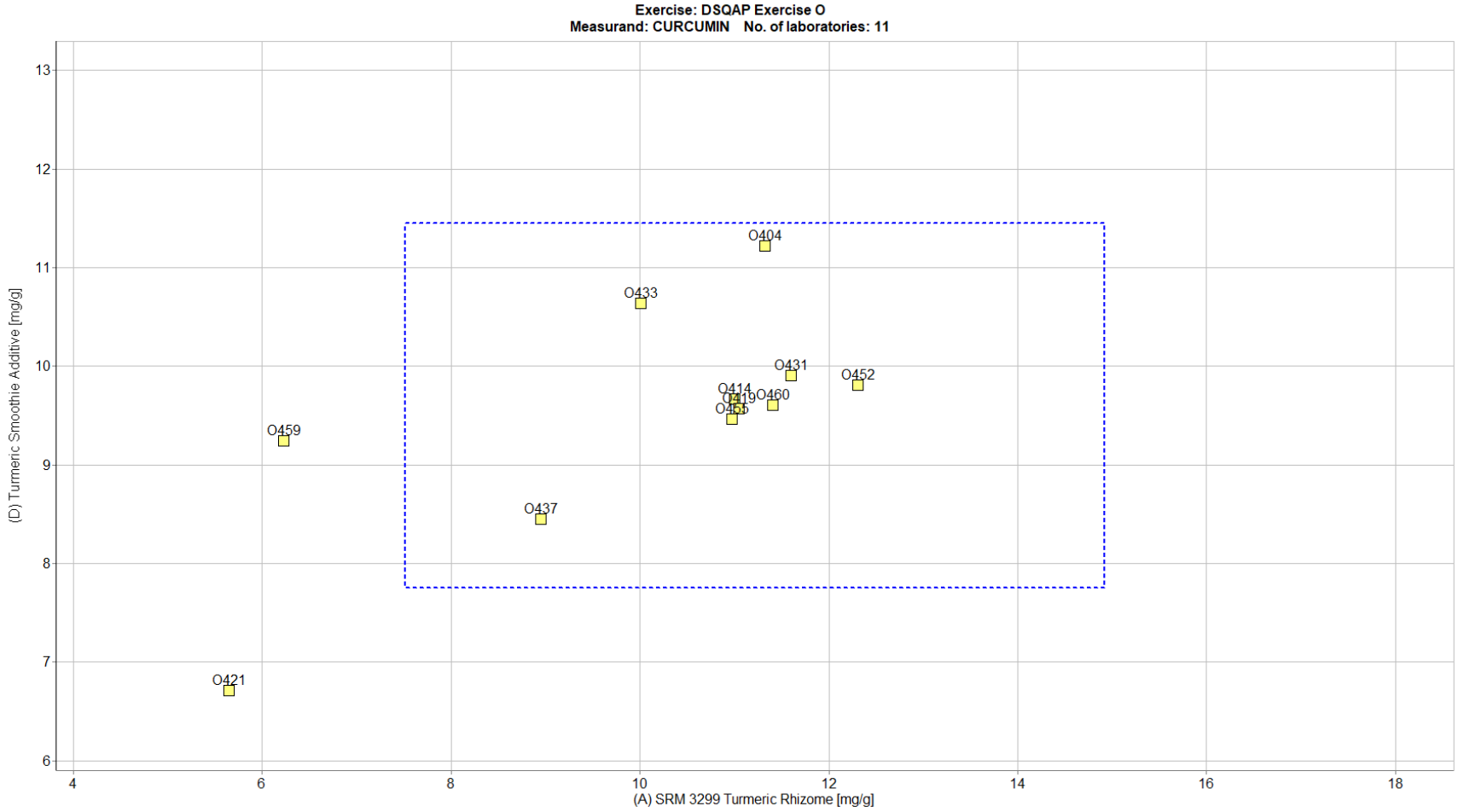


**Figure 2-13.** Laboratory means for curcumin in candidate SRM 3299 Turmeric Rhizome and Turmeric Smoothie Additive (sample/sample comparison view). In this view, the individual laboratory mean for one sample (turmeric rhizome) is compared to the mean for a second sample (turmeric smoothie additive). The dotted blue box represents the consensus range of tolerance for turmeric rhizome (x-axis) and turmeric smoothie additive (y-axis), calculated as the values above and below the consensus means that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$.

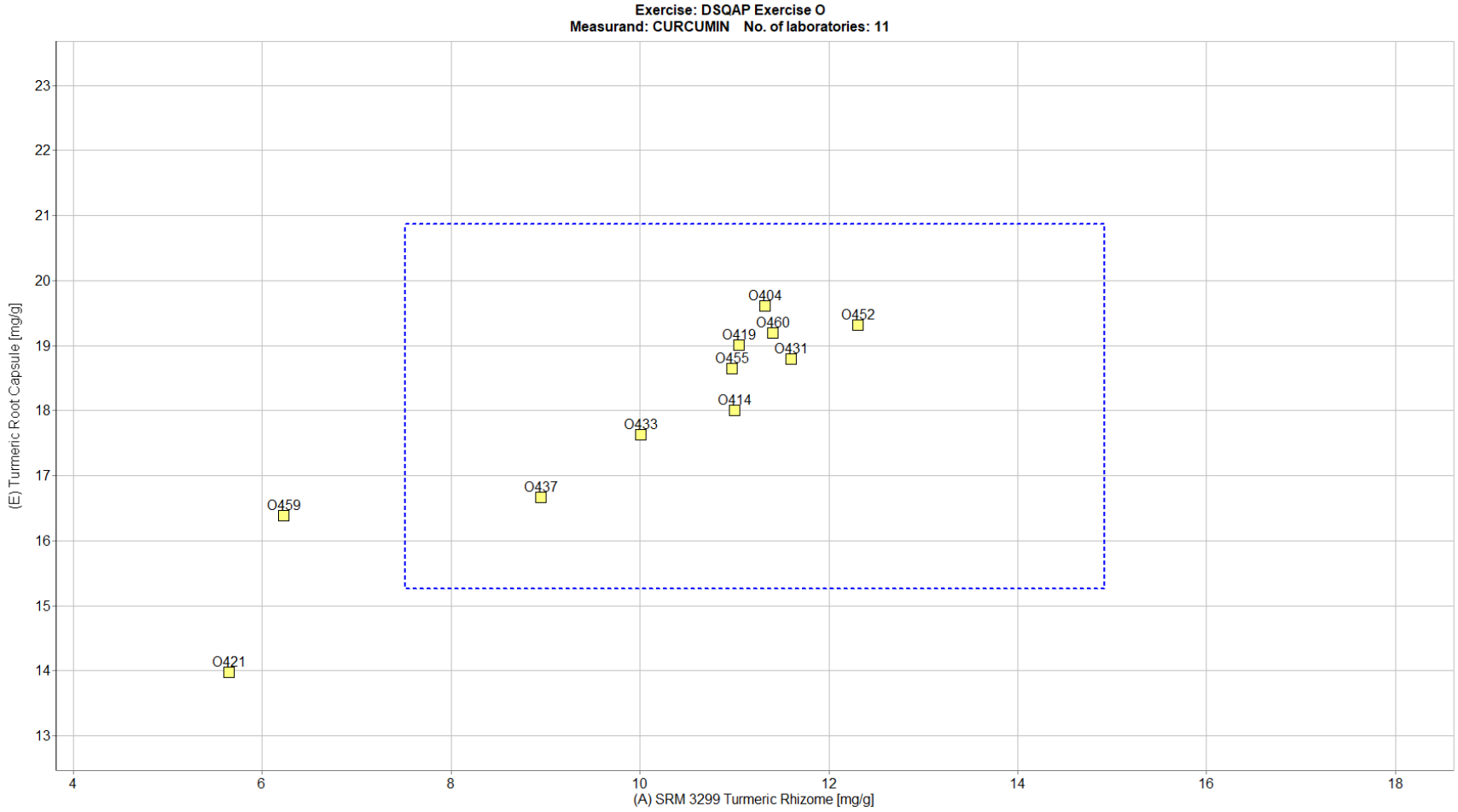


**Figure 2-14.** Laboratory means for curcumin in candidate SRM 3299 Turmeric Rhizome and Turmeric Root Capsule (sample/sample comparison view). In this view, the individual laboratory mean for one sample (turmeric rhizome) is compared to the mean for a second sample (turmeric root capsule). The dotted blue box represents the consensus range of tolerance for turmeric rhizome (x-axis) and turmeric root capsule (y-axis), calculated as the values above and below the consensus means that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$.

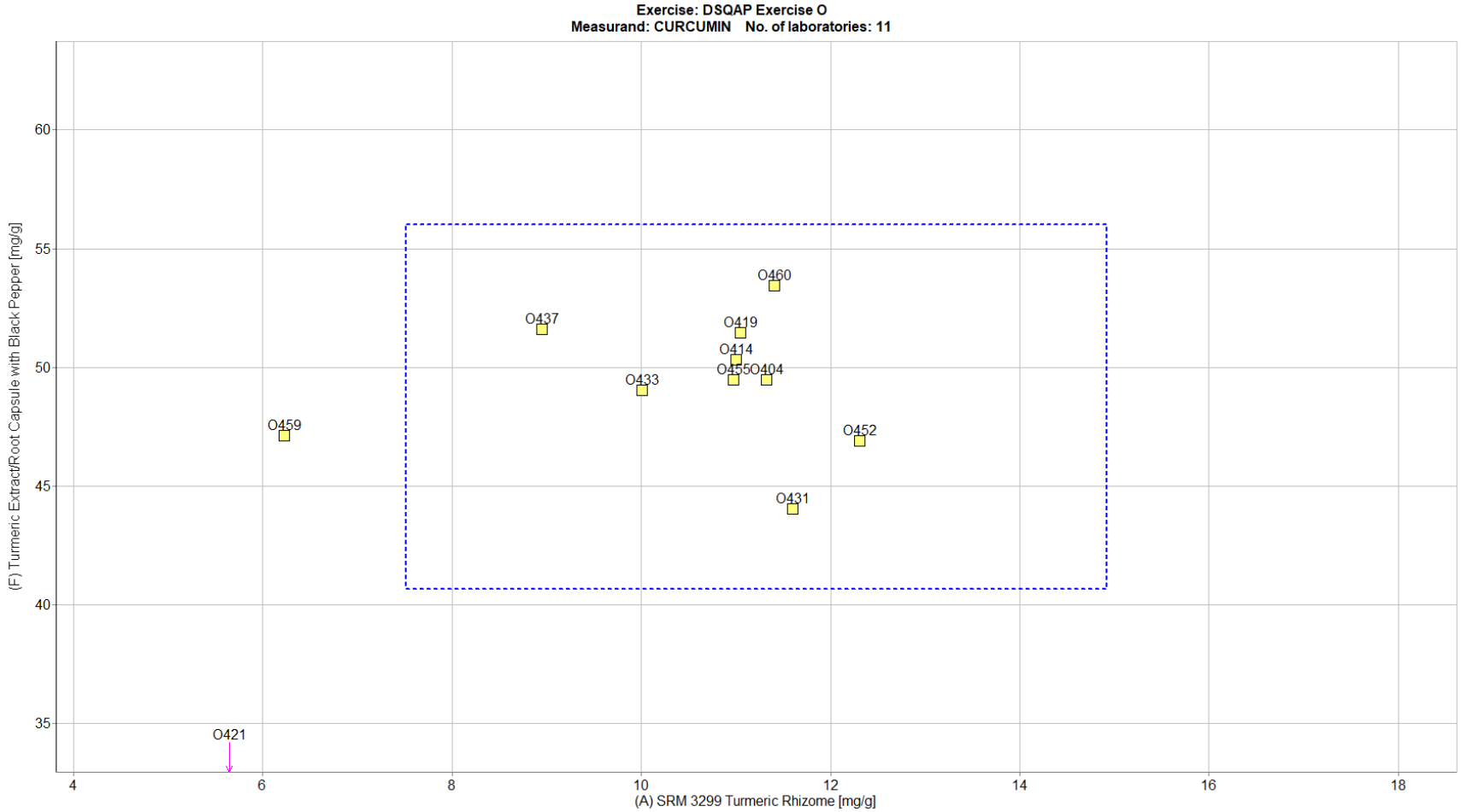


**Figure 2-15.** Laboratory means for curcumin in candidate SRM 3299 Turmeric Rhizome and Turmeric Extract/Root Capsule with Black Pepper (sample/sample comparison view). In this view, the individual laboratory mean for one sample (turmeric rhizome) is compared to the mean for a second sample (turmeric capsule). The dotted blue box represents the consensus range of tolerance for turmeric rhizome (x-axis) and turmeric capsule (y-axis), calculated as the values above and below the consensus means that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$.

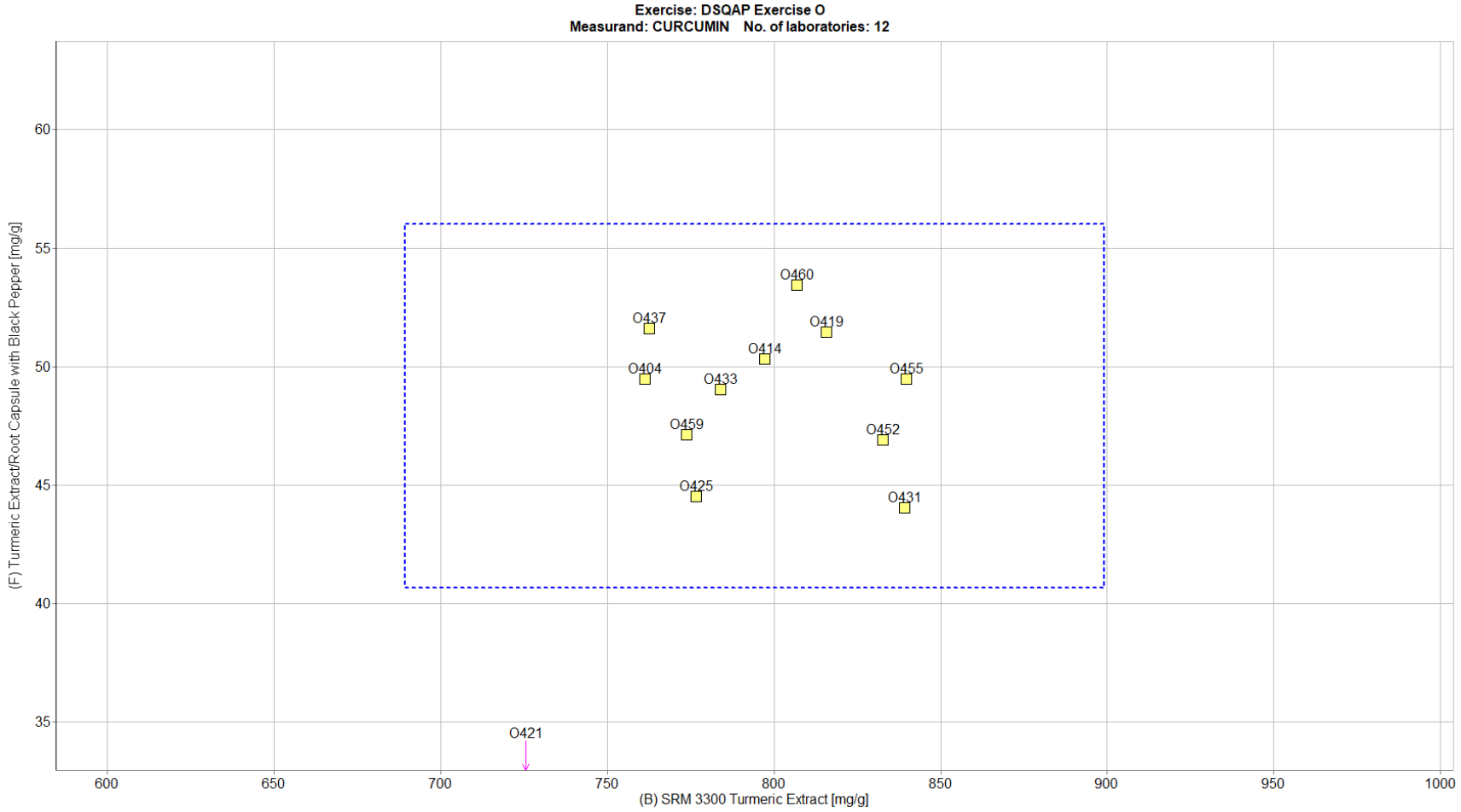


**Figure 2-16.** Laboratory means for curcumin in candidate SRM 3300 Turmeric Extract and Turmeric Extract/Root Capsule with Black Pepper (sample/sample comparison view). In this view, the individual laboratory mean for one sample (turmeric extract) is compared to the mean for a second sample (turmeric capsule). The dotted blue box represents the consensus range of tolerance for turmeric rhizome (x-axis) and turmeric capsule (y-axis), calculated as the values above and below the consensus means that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$.

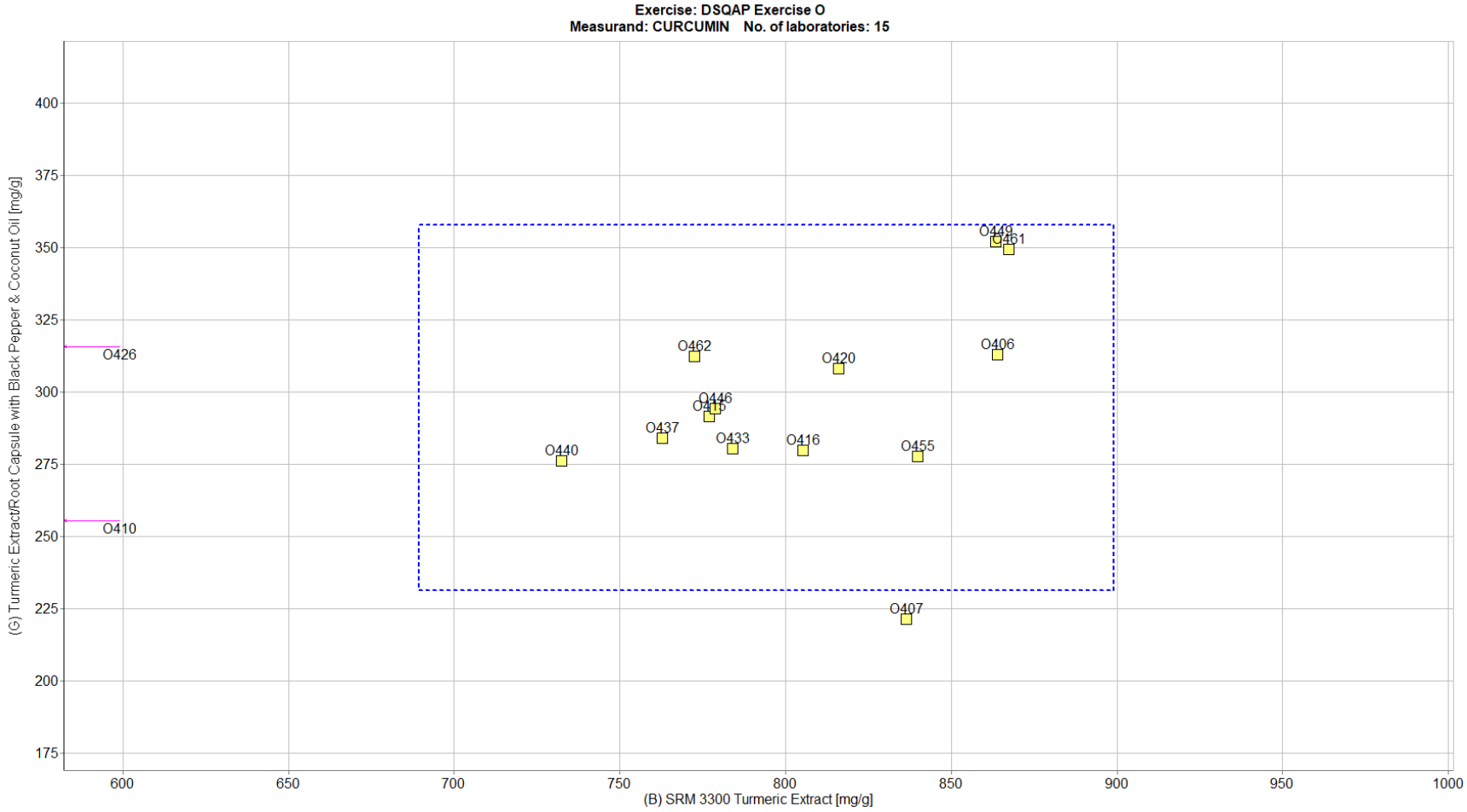


**Figure 2-17.** Laboratory means for curcumin in candidate SRM 3300 Turmeric Extract and Turmeric Extract/Root Capsule with Black Pepper & Coconut Oil (sample/sample comparison view). In this view, the individual laboratory mean for one sample (turmeric extract) is compared to the mean for a second sample (turmeric capsule). The dotted blue box represents the consensus range of tolerance for turmeric extract (x-axis) and turmeric capsule (y-axis), calculated as the values above and below the consensus means that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$.

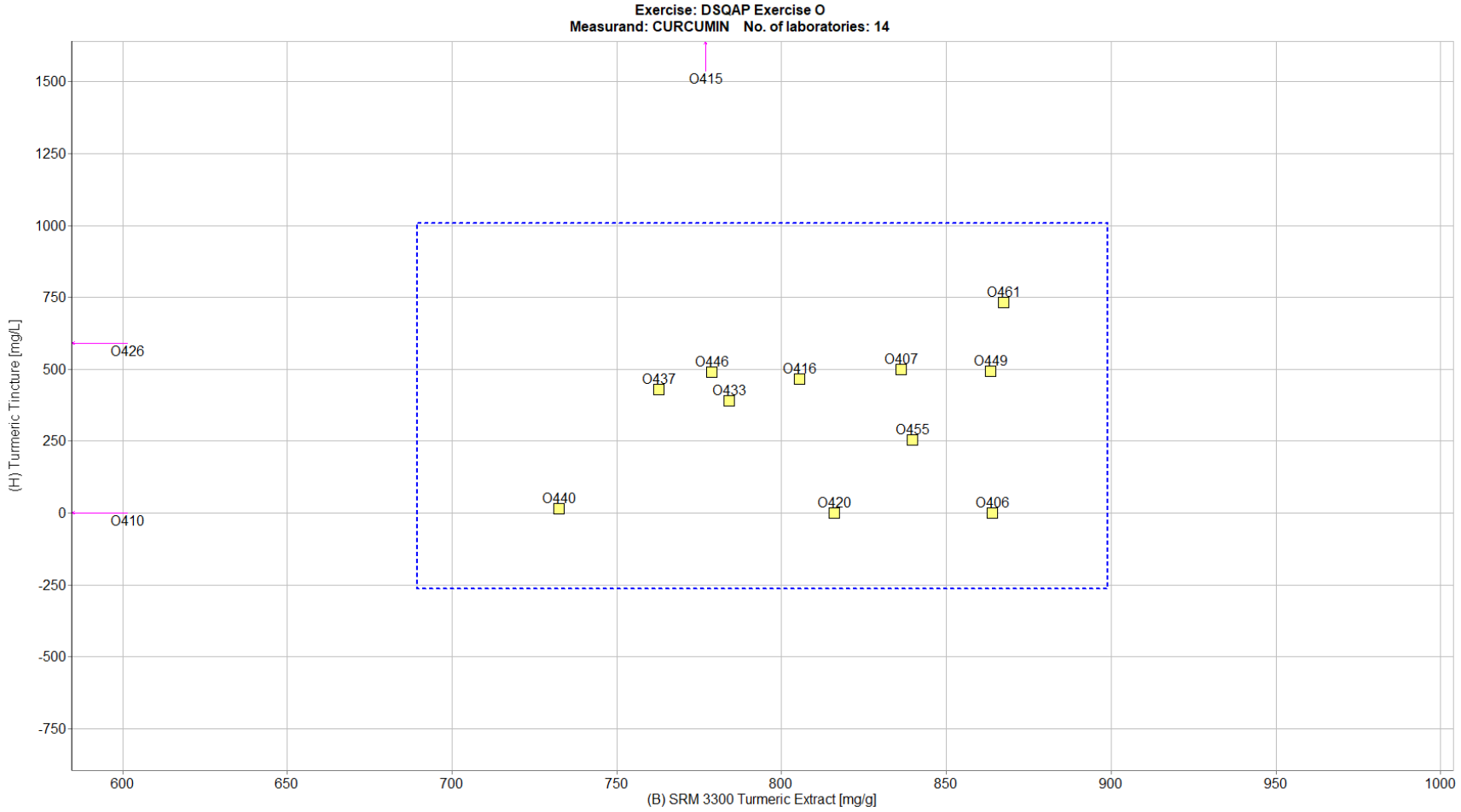


**Figure 2-18.** Laboratory means for curcumin in candidate SRM 3300 Turmeric Extract and Turmeric Tincture (sample/sample comparison view). In this view, the individual laboratory mean for one sample (turmeric extract) is compared to the mean for a second sample (turmeric tincture). The dotted blue box represents the consensus range of tolerance for turmeric extract (x-axis) and turmeric tincture (y-axis), calculated as the values above and below the consensus means that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$.

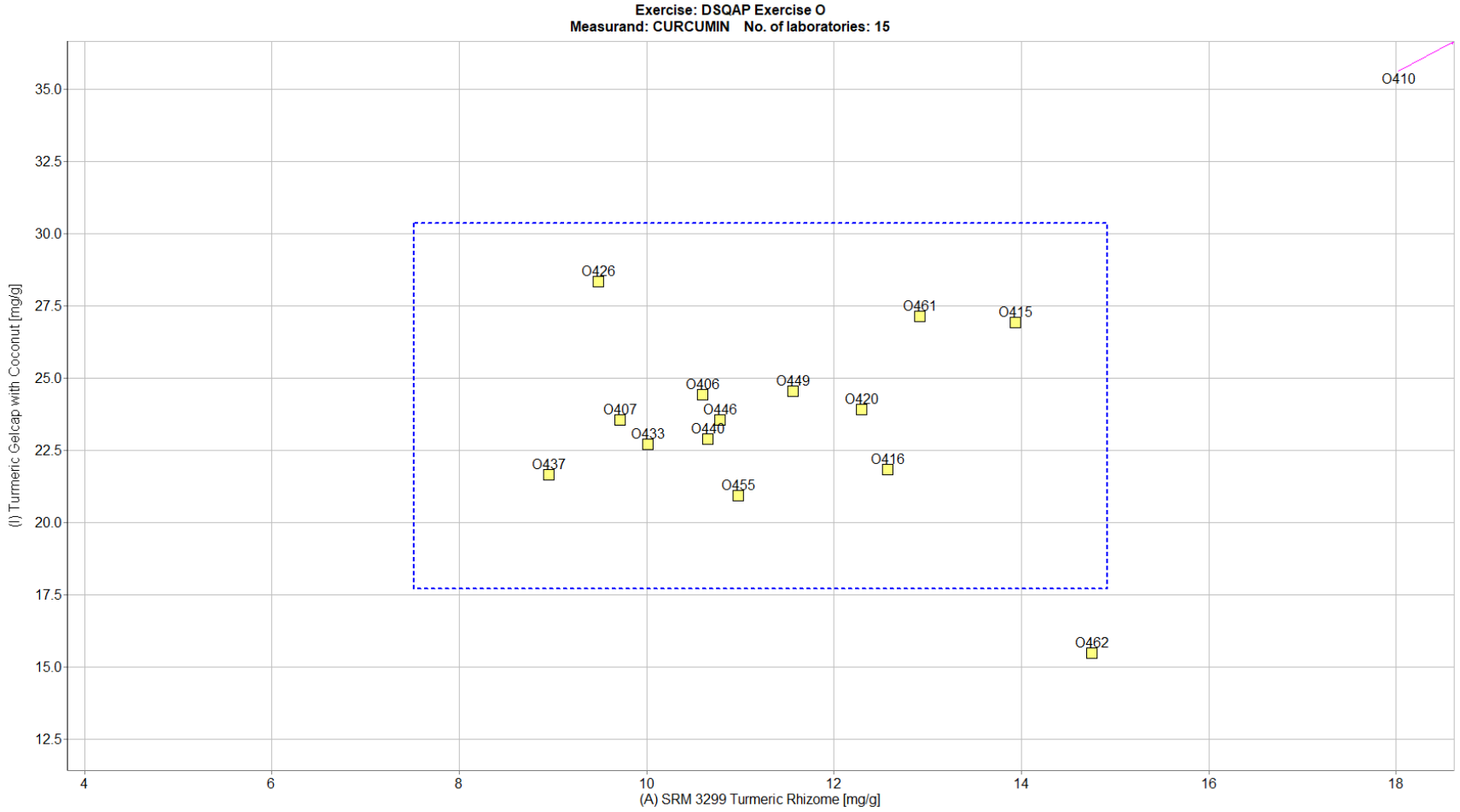


**Figure 2-19.** Laboratory means for curcumin in candidate SRM 3299 Turmeric Rhizome and Turmeric Gelcap with Coconut (sample/sample comparison view). In this view, the individual laboratory mean for one sample (turmeric rhizome) is compared to the mean for a second sample (turmeric gelcap). The dotted blue box represents the consensus range of tolerance for turmeric rhizome (x-axis) and turmeric gelcap (y-axis), calculated as the values above and below the consensus means that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$.

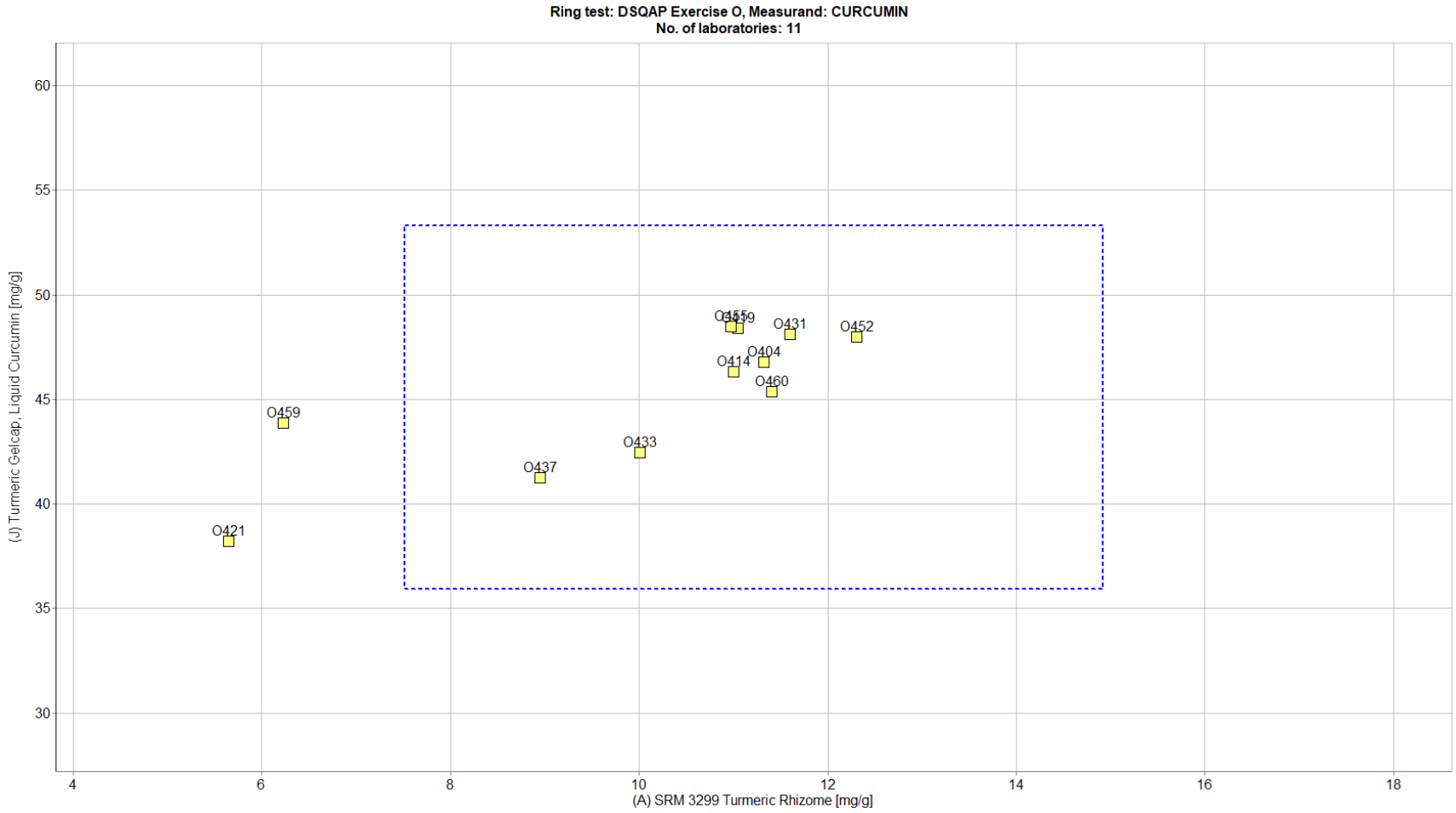


**Figure 2-20.** Laboratory means for curcumin in candidate SRM 3299 Turmeric Rhizome and Turmeric Gelcap, Liquid Curcumin (sample/sample comparison view). In this view, the individual laboratory mean for one sample (turmeric rhizome) is compared to the mean for a second sample (turmeric gelcap). The dotted blue box represents the consensus range of tolerance for turmeric rhizome (x-axis) and turmeric gelcap (y-axis), calculated as the values above and below the consensus means that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$.

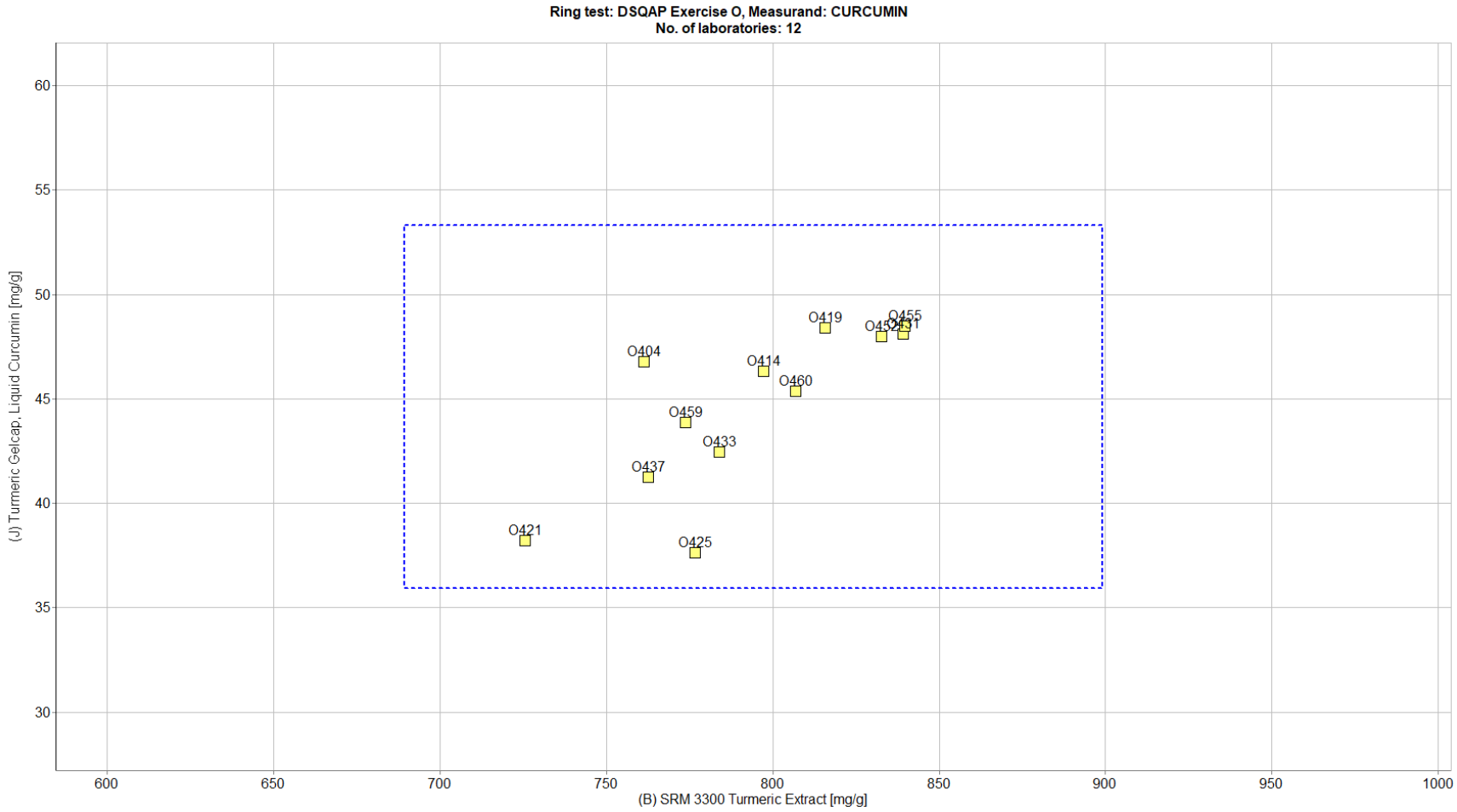


**Figure 2-21.** Laboratory means for curcumin in candidate SRM 3300 Turmeric Extract and Turmeric Gelcap, Liquid Curcumin (sample/sample comparison view). In this view, the individual laboratory mean for one sample (turmeric extract) is compared to the mean for a second sample (turmeric gelcap). The dotted blue box represents the consensus range of tolerance for turmeric extract (x-axis) and turmeric gelcap (y-axis), calculated as the values above and below the consensus means that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$.

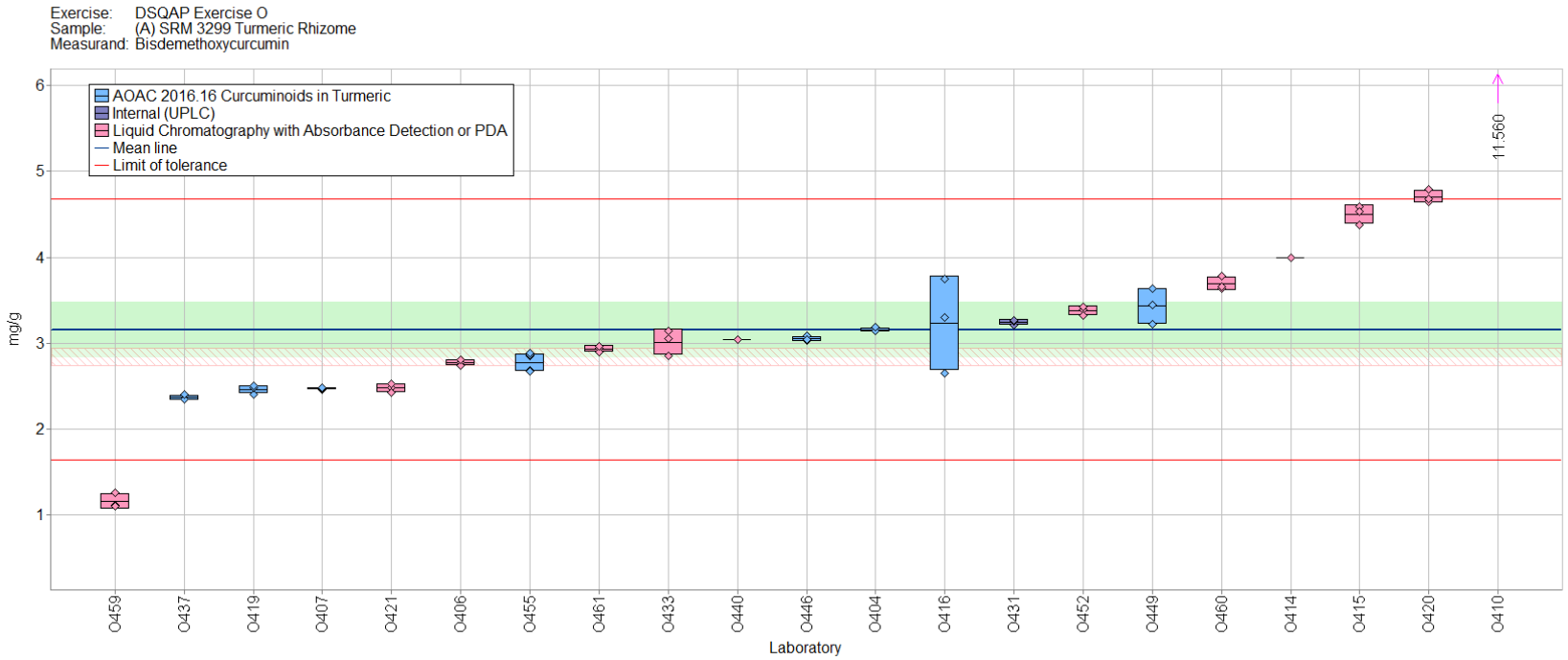


**Figure 2-22.** BDMC in candidate SRM 3299 Turmeric Rhizome (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by its uncertainty ($U_{\text{NIST}}$) and represents the range that results in an acceptable $Z_{\text{NIST}}$ score, $|Z_{\text{NIST}}| \leq 2$.

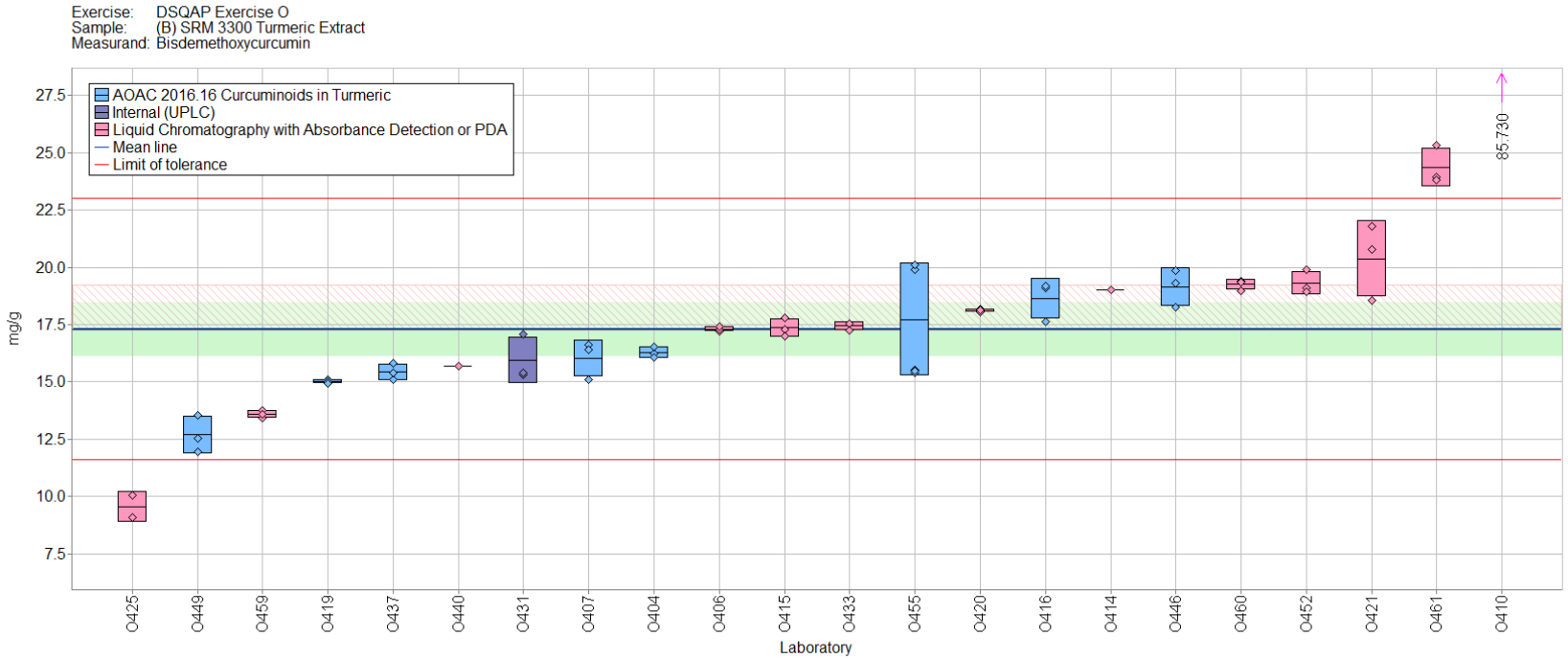


**Figure 2-23.** BDMC in candidate SRM 3300 Turmeric Extract (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by its uncertainty ($U_{\text{NIST}}$) and represents the range that results in an acceptable $Z_{\text{NIST}}$ score, $|Z_{\text{NIST}}| \leq 2$.

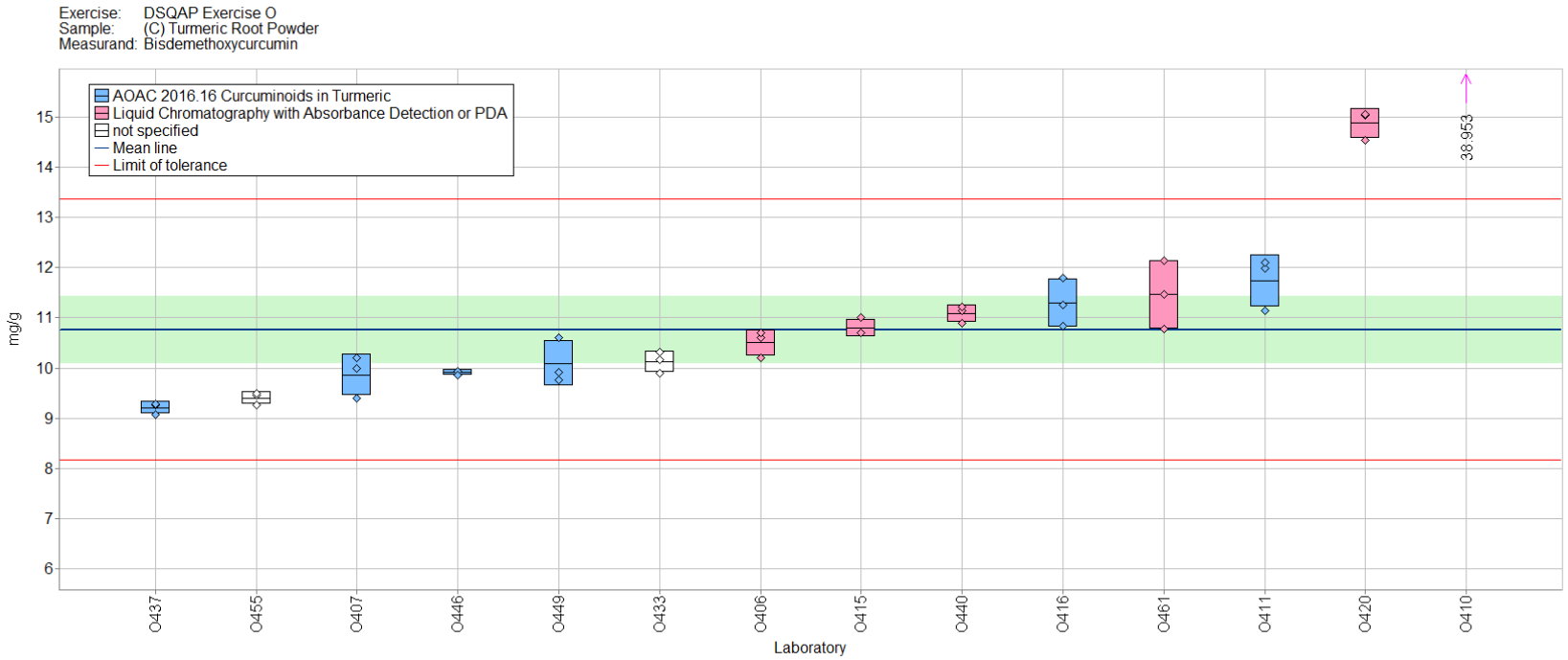


**Figure 2-24.** BDMC in Turmeric Root Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$. A NIST value has not been determined in this material.

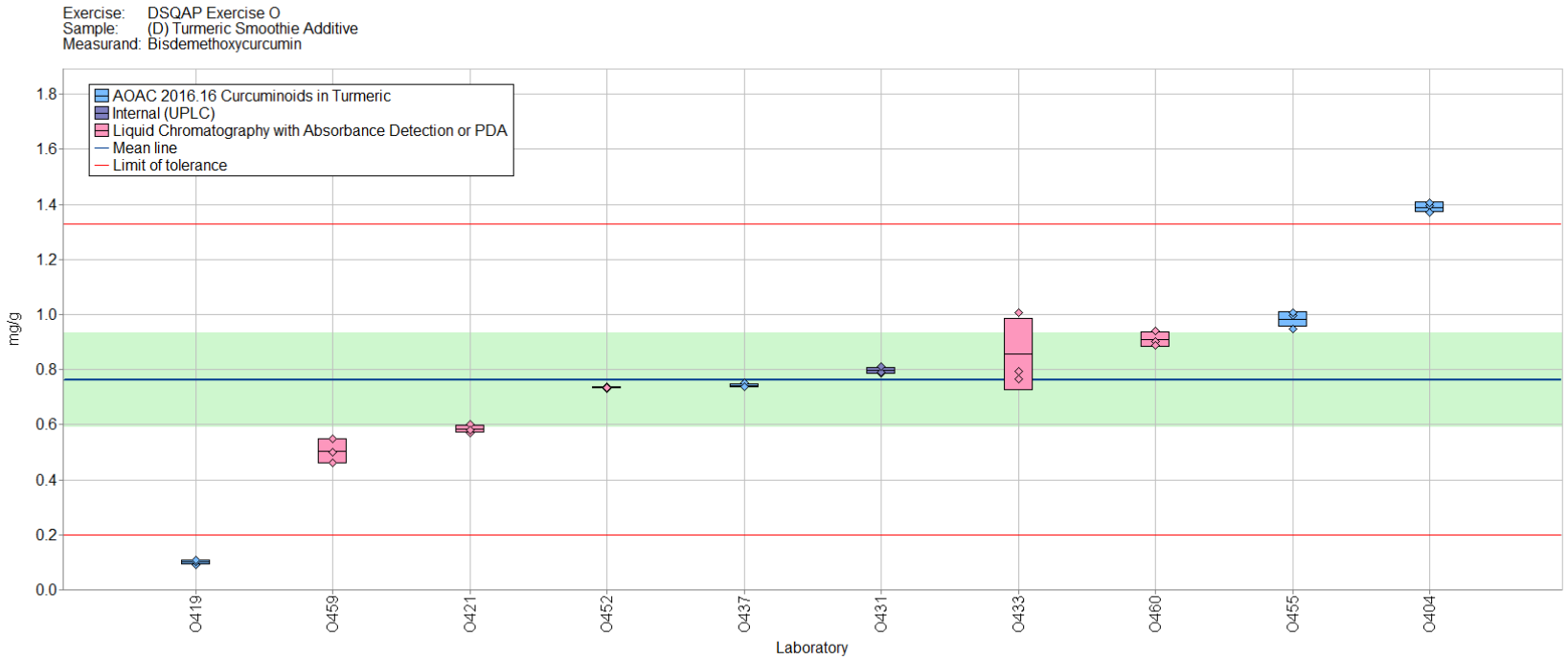


**Figure 2-25.** BDMC in Turmeric Smoothie Additive (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$. A NIST value has not been determined in this material.

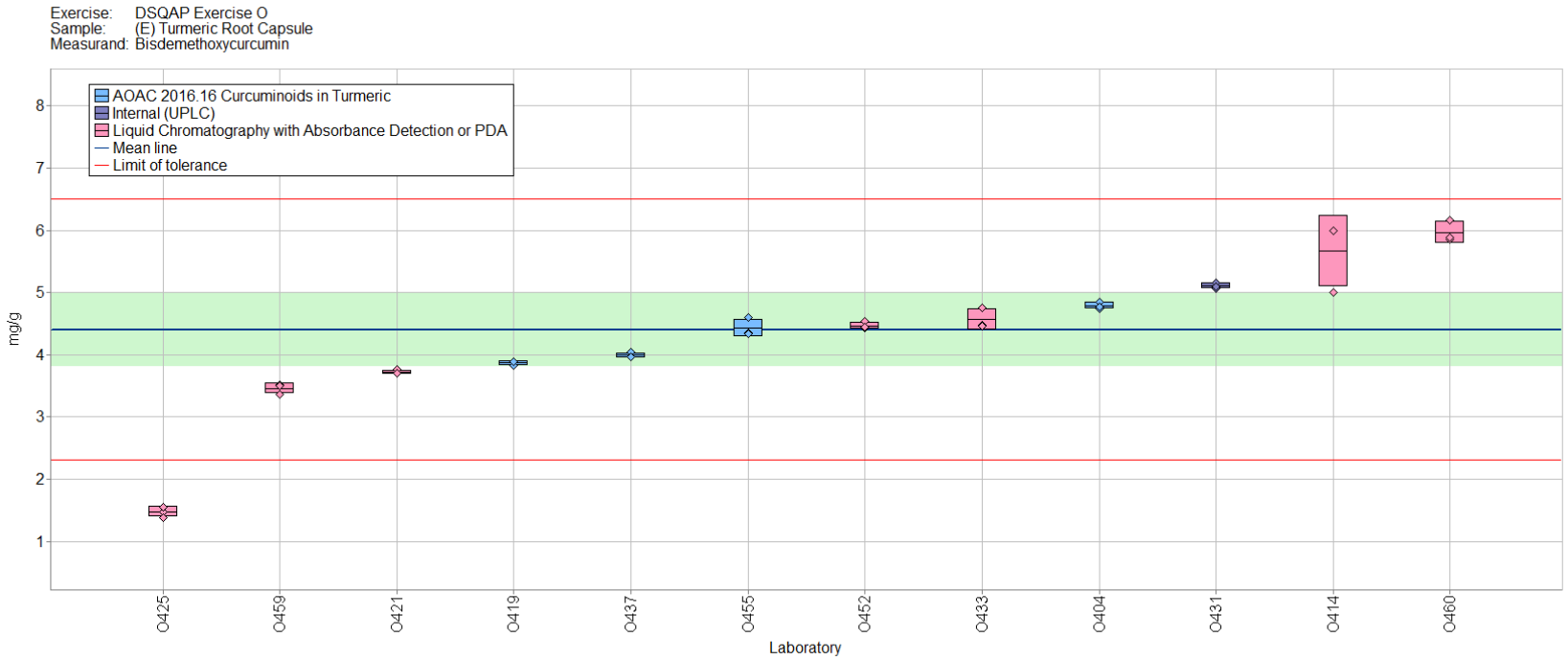


**Figure 2-26.** BDMC in Turmeric Root Capsule (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$. A NIST value has not been determined in this material.

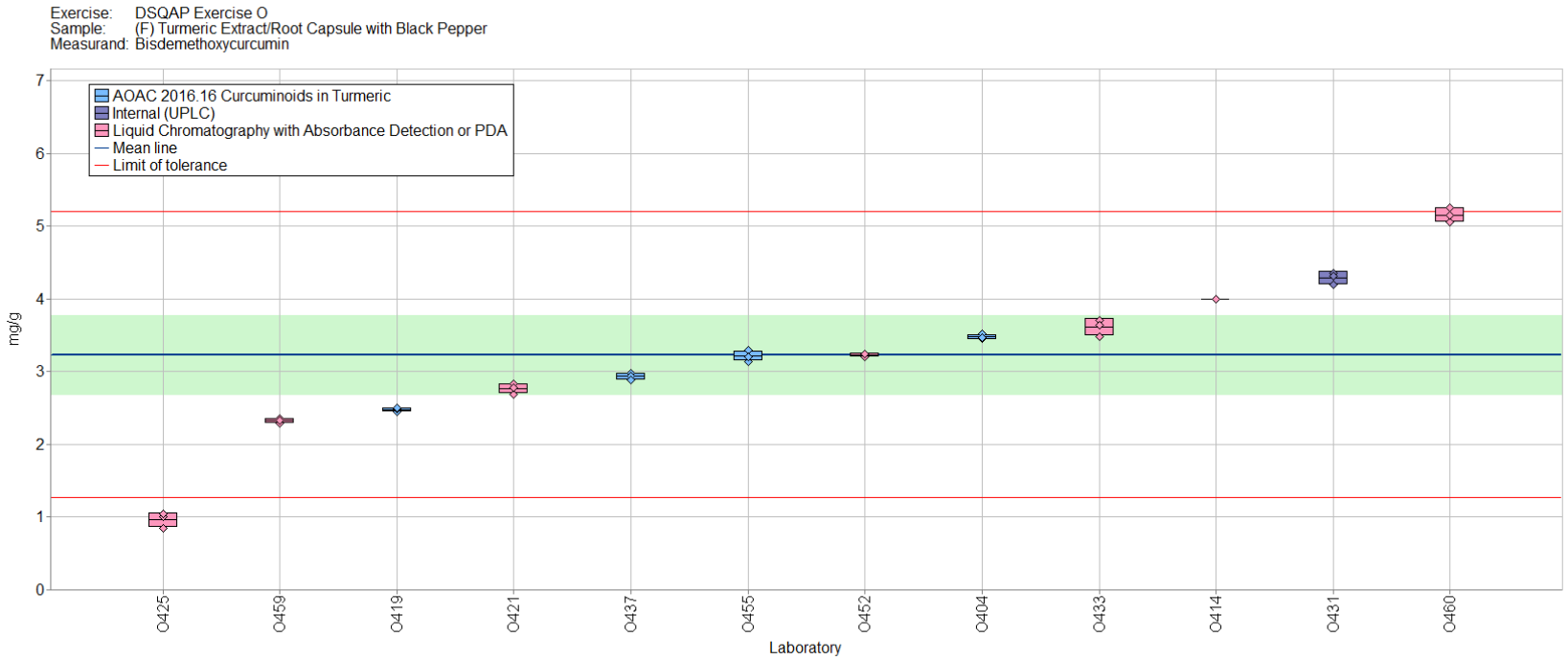


**Figure 2-27.** BDMC in Turmeric Extract/Root Capsule with Black Pepper (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$. A NIST value has not been determined in this material.

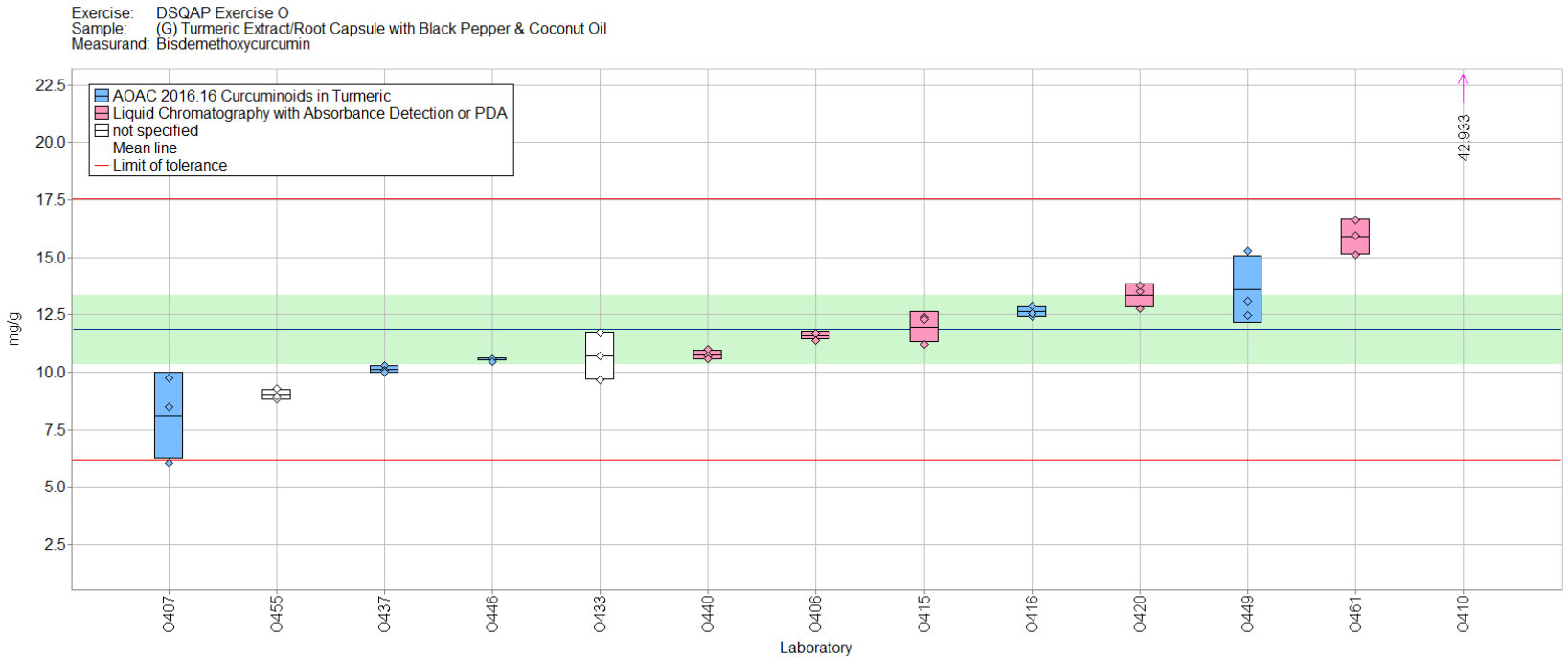


**Figure 2-28.** BDMC in Turmeric Extract/Root Capsule with Black Pepper & Coconut Oil (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$. A NIST value has not been determined in this material.

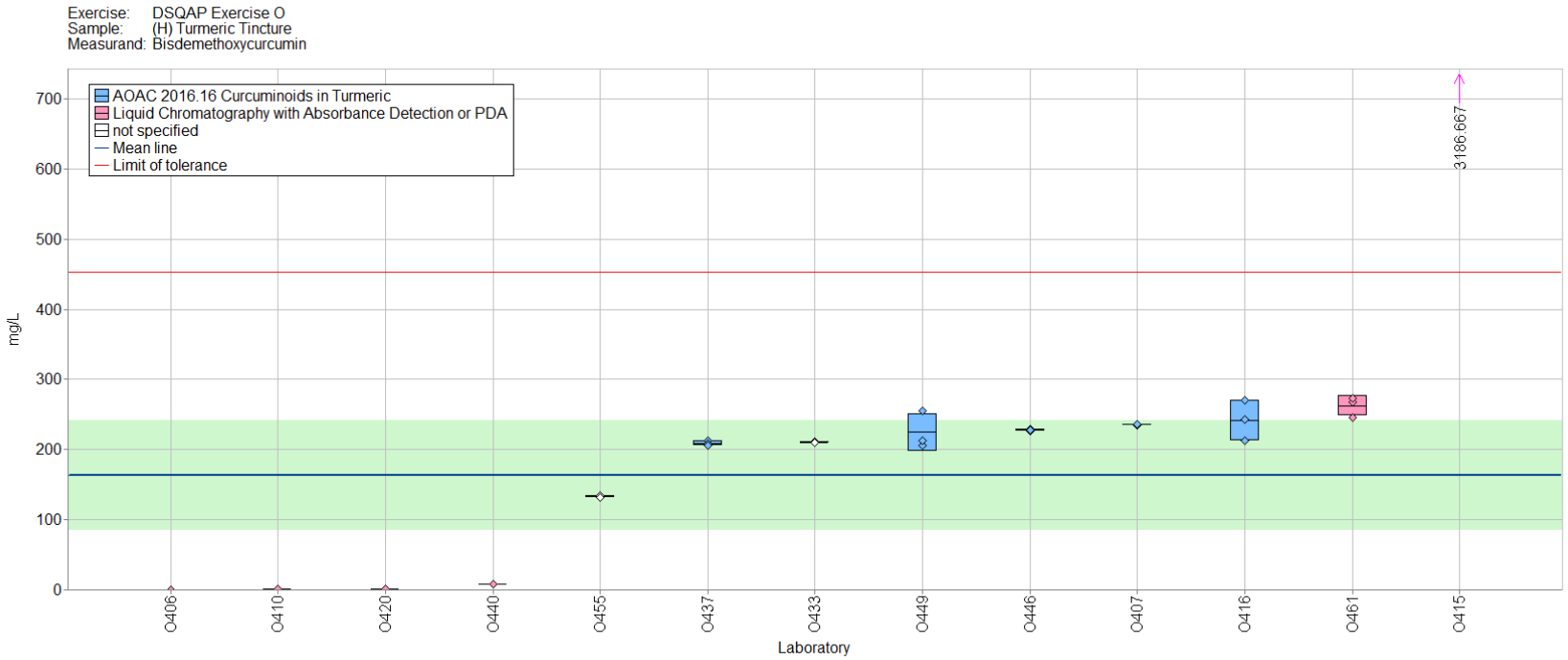


**Figure 2-29.** BDMC in Turmeric Tincture (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above the consensus mean that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$, with the lower value set to zero. A NIST value has not been determined in this material.

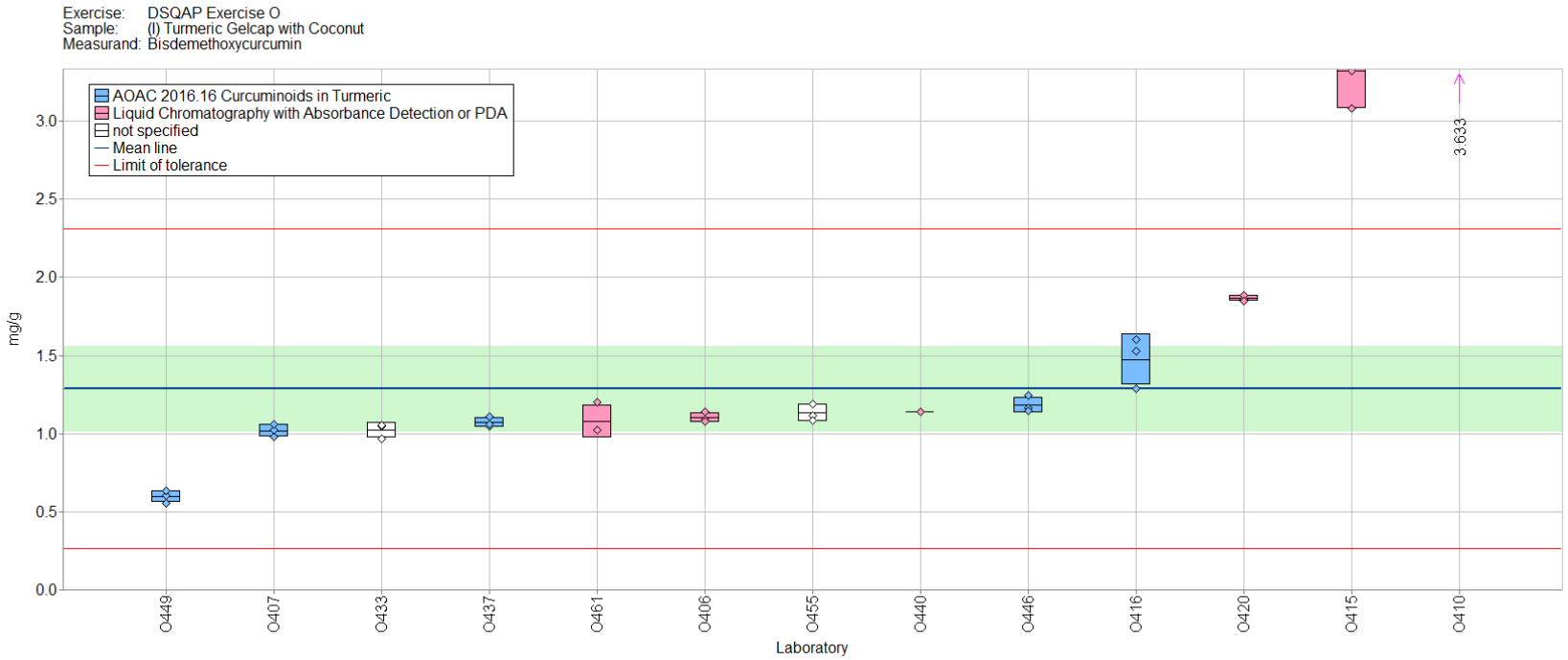


**Figure 2-30.** BDMC in Turmeric Gelcap with Coconut (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$. A NIST value has not been determined in this material.

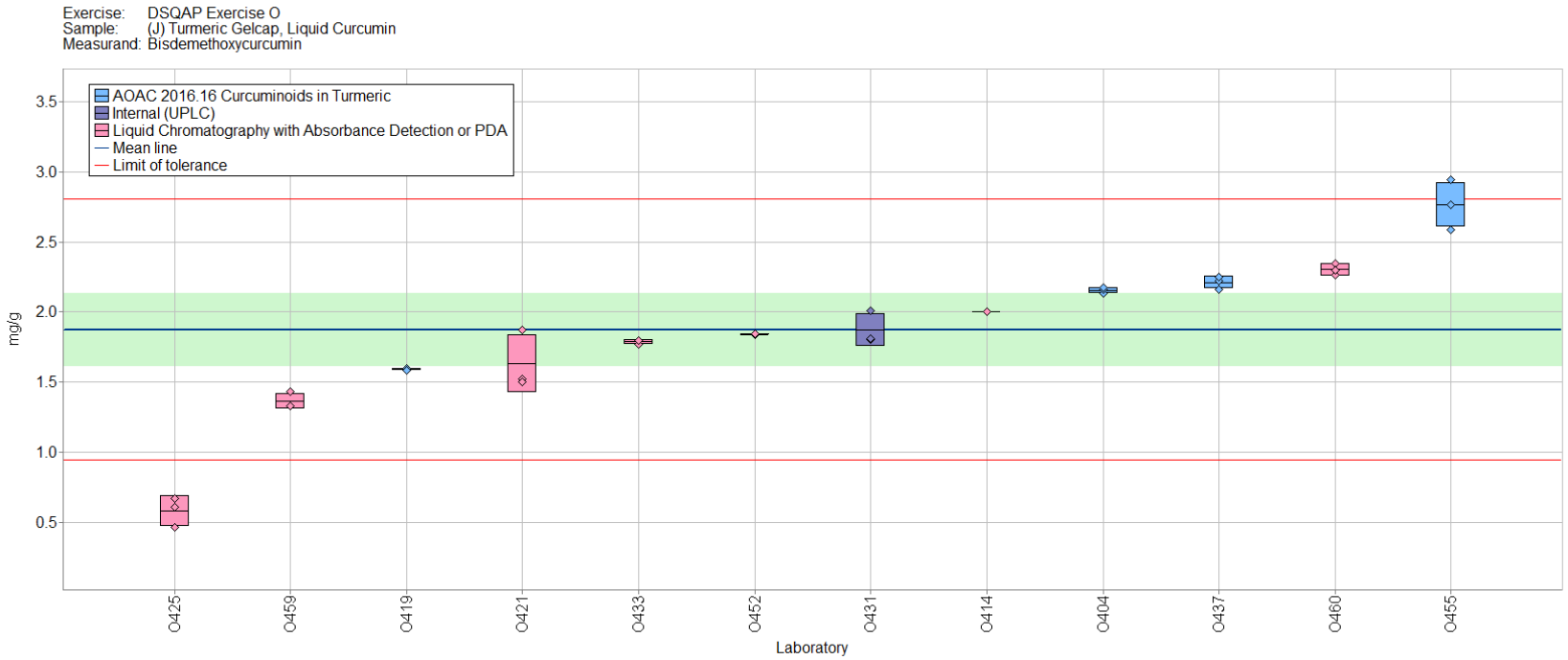


**Figure 2-31.** BDMC in Turmeric Gelcap, Liquid Curcumin (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$. A NIST value has not been determined in this material.

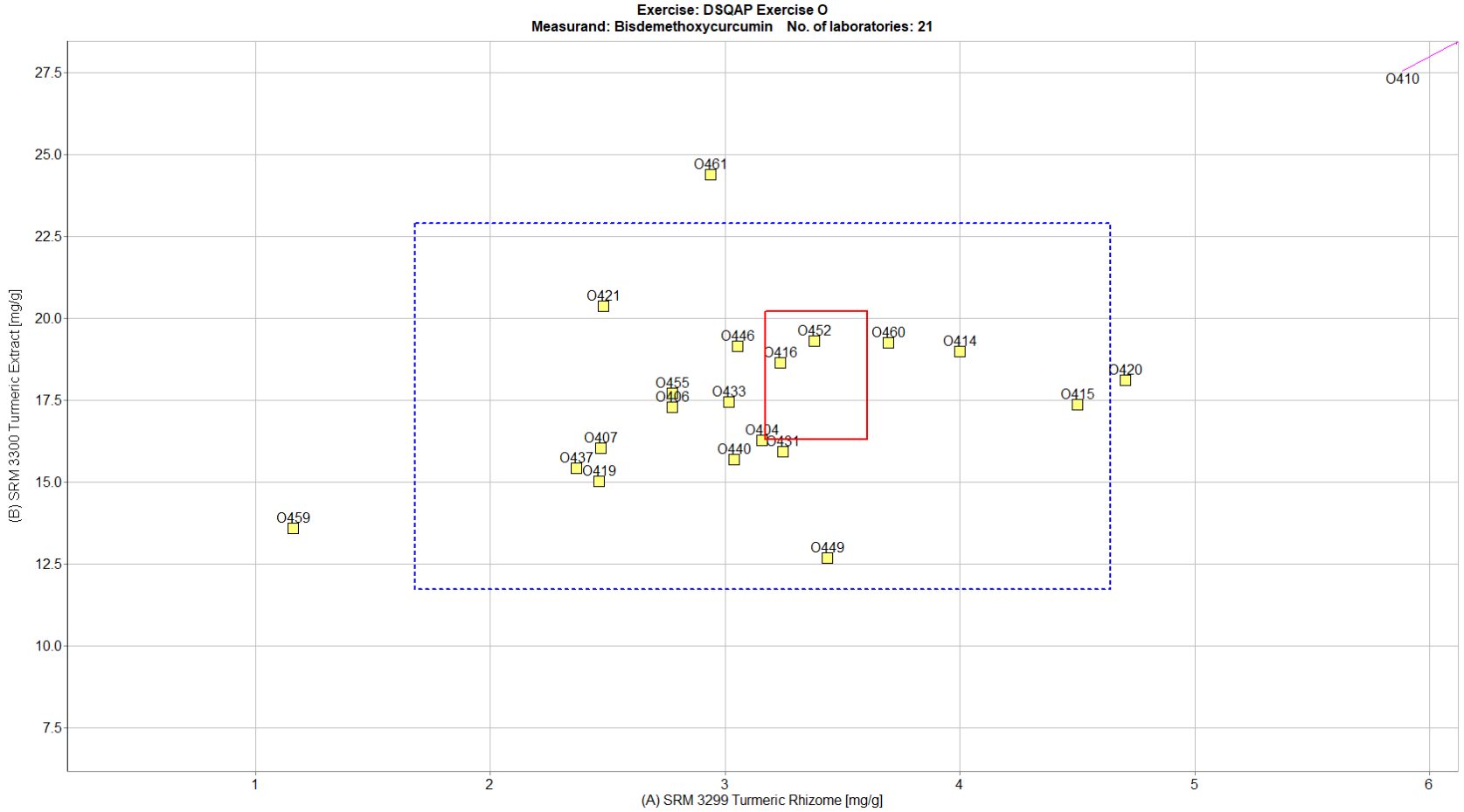


**Figure 2-32.** Laboratory means for BDMC in candidate SRM 3299 Turmeric Rhizome and candidate SRM 3300 Turmeric Extract (sample/sample comparison view). In this view, the individual laboratory mean for one sample (turmeric rhizome) is compared to the mean for a second sample (turmeric extract). The solid red box represents the NIST range of tolerance for the two samples, turmeric rhizome (x-axis) and turmeric extract (y-axis), which encompasses the NIST-determined values bounded by their uncertainties ($U_{\text{NIST}}$) and represents the range that results in an acceptable $Z_{\text{NIST}}$ score, $|Z_{\text{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for turmeric rhizome (x-axis) and turmeric extract (y-axis), calculated as the values above and below the consensus means that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$.

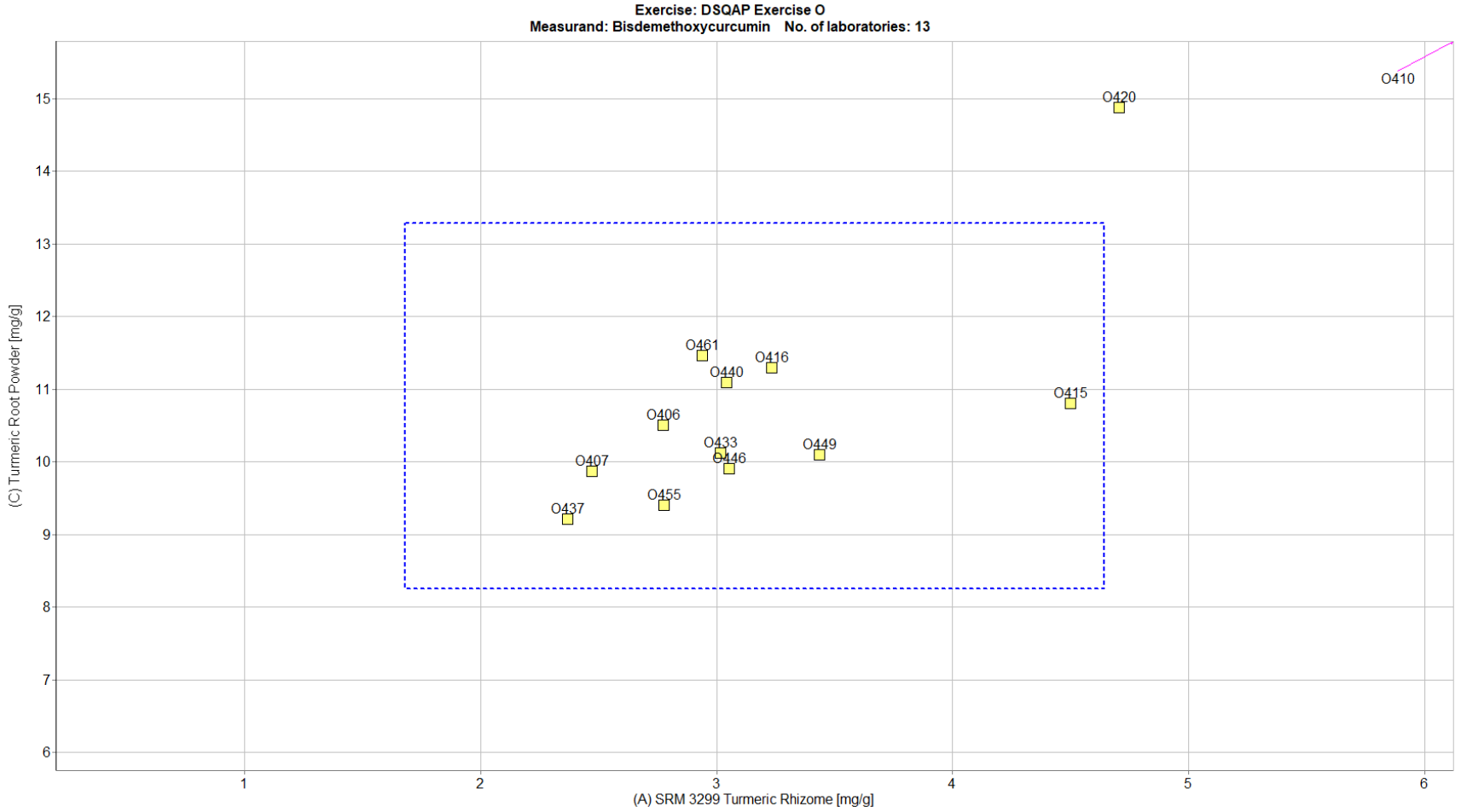


**Figure 2-33.** Laboratory means for BDMC in candidate SRM 3299 Turmeric Rhizome and Turmeric Root Powder (sample/sample comparison view). In this view, the individual laboratory mean for one sample (turmeric rhizome) is compared to the mean for a second sample (turmeric root powder). The dotted blue box represents the consensus range of tolerance for turmeric rhizome (x-axis) and turmeric root powder (y-axis), calculated as the values above and below the consensus means that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$.

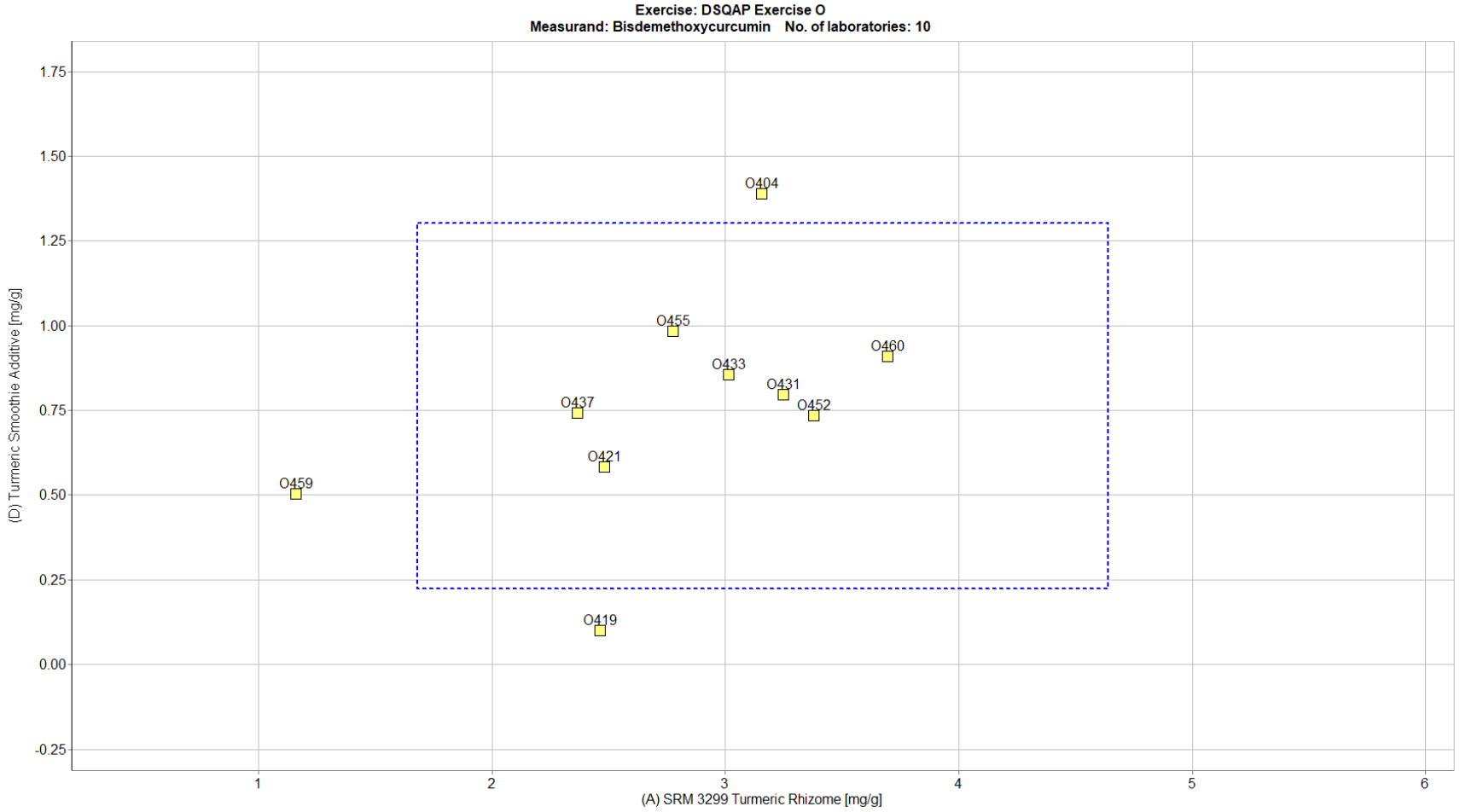


**Figure 2-34.** Laboratory means for BDMC in candidate SRM 3299 Turmeric Rhizome and Turmeric Smoothie Additive (sample/sample comparison view). In this view, the individual laboratory mean for one sample (turmeric rhizome) is compared to the mean for a second sample (turmeric smoothie additive). The dotted blue box represents the consensus range of tolerance for turmeric rhizome (x-axis) and turmeric smoothie additive (y-axis), calculated as the values above and below the consensus means that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$.

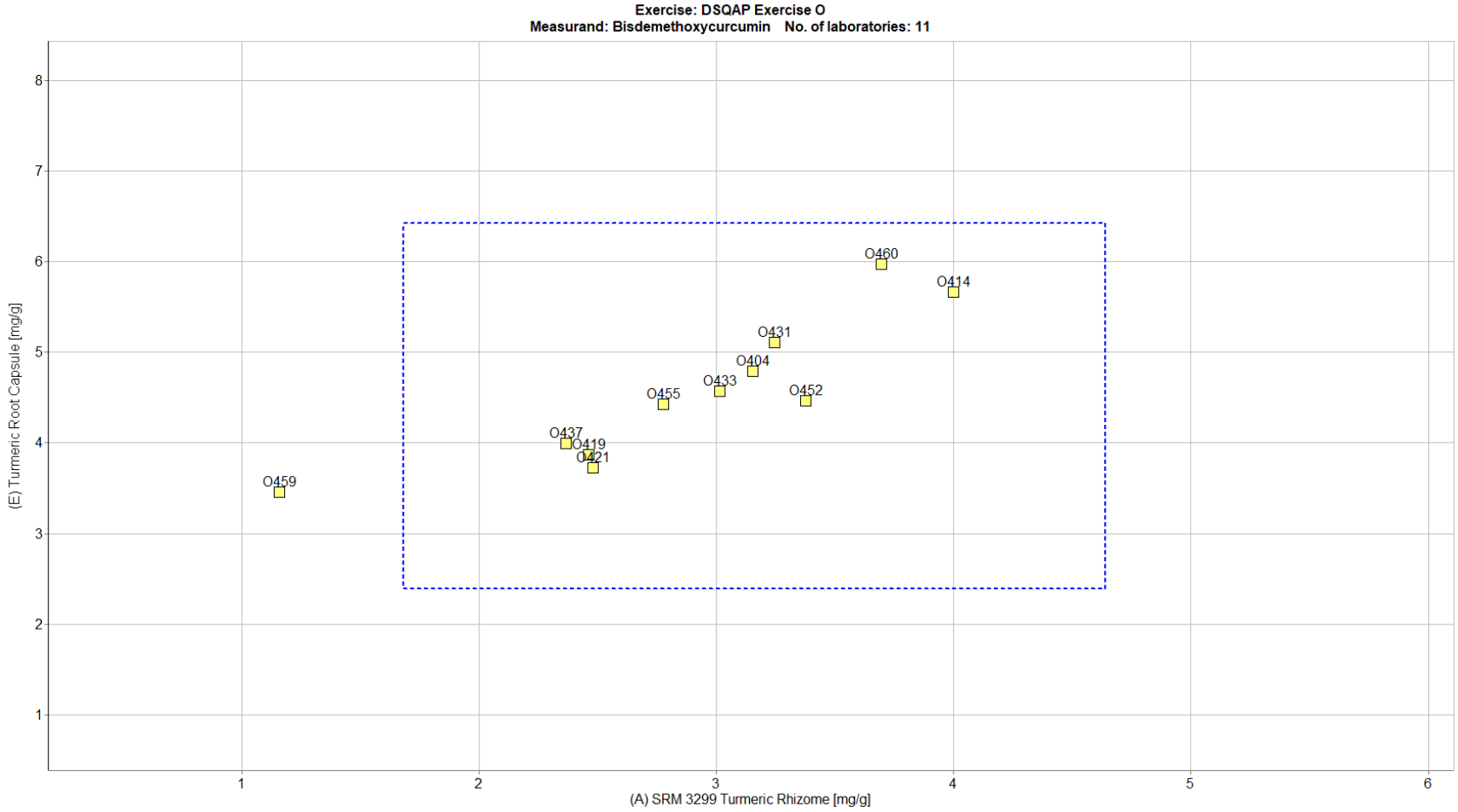


**Figure 2-35.** Laboratory means for BDMC in candidate SRM 3299 Turmeric Rhizome and Turmeric Root Capsule (sample/sample comparison view). In this view, the individual laboratory mean for one sample (turmeric rhizome) is compared to the mean for a second sample (turmeric capsule). The dotted blue box represents the consensus range of tolerance for turmeric rhizome (x-axis) and turmeric capsule (y-axis), calculated as the values above and below the consensus means that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$.

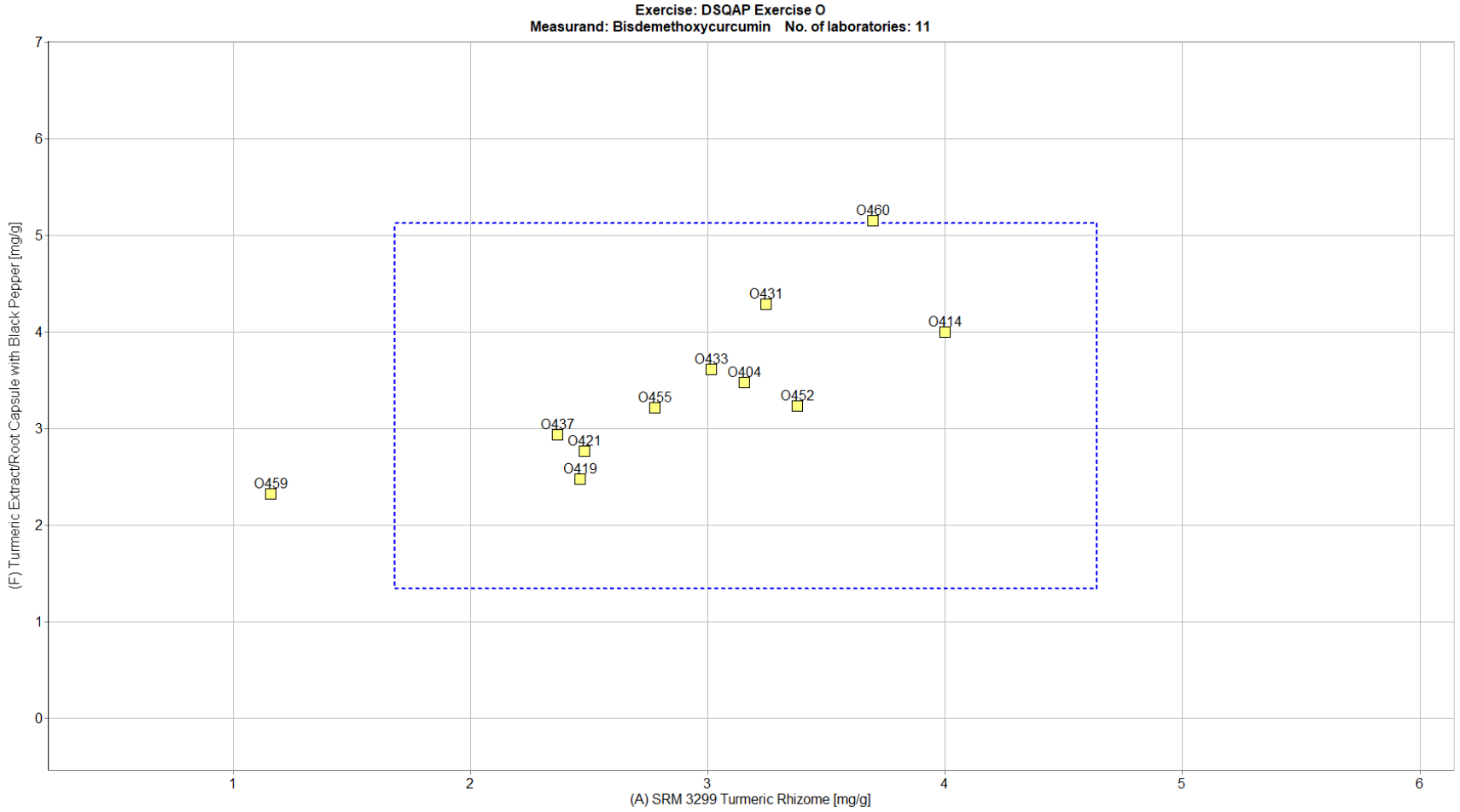


**Figure 2-36.** Laboratory means for BDMC in candidate SRM 3299 Turmeric Rhizome and Turmeric Extract/Root Capsule with Black Pepper (sample/sample comparison view). In this view, the individual laboratory mean for one sample (turmeric rhizome) is compared to the mean for a second sample (turmeric capsule). The dotted blue box represents the consensus range of tolerance for turmeric rhizome (x-axis) and turmeric capsule (y-axis), calculated as the values above and below the consensus means that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$.

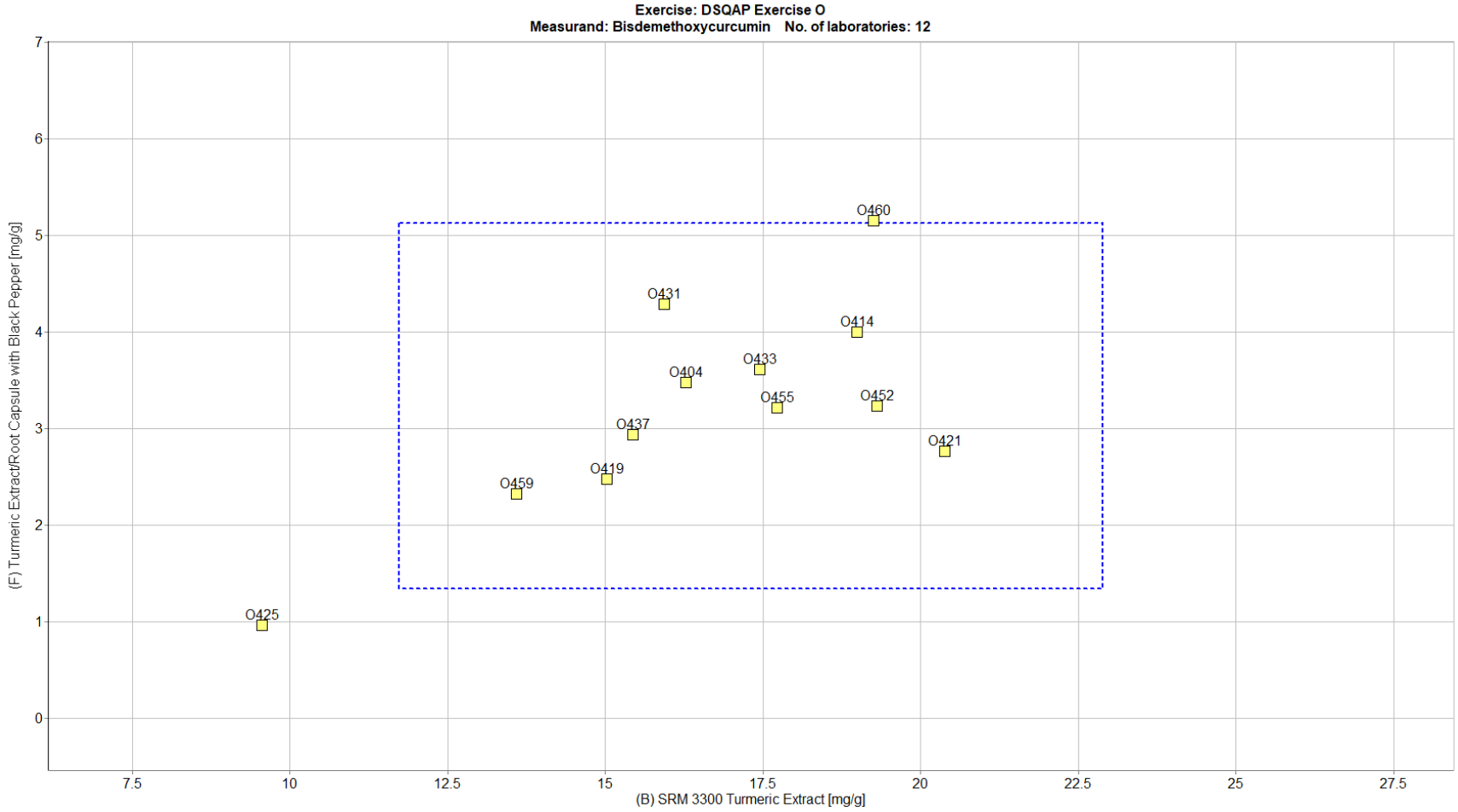


**Figure 2-37.** Laboratory means for BDMC in candidate SRM 3300 Turmeric Extract and Turmeric Extract/Root Capsule with Black Pepper (sample/sample comparison view). In this view, the individual laboratory mean for one sample (turmeric extract) is compared to the mean for a second sample (turmeric capsule). The dotted blue box represents the consensus range of tolerance for turmeric extract (x-axis) and turmeric capsule (y-axis), calculated as the values above and below the consensus means that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$.

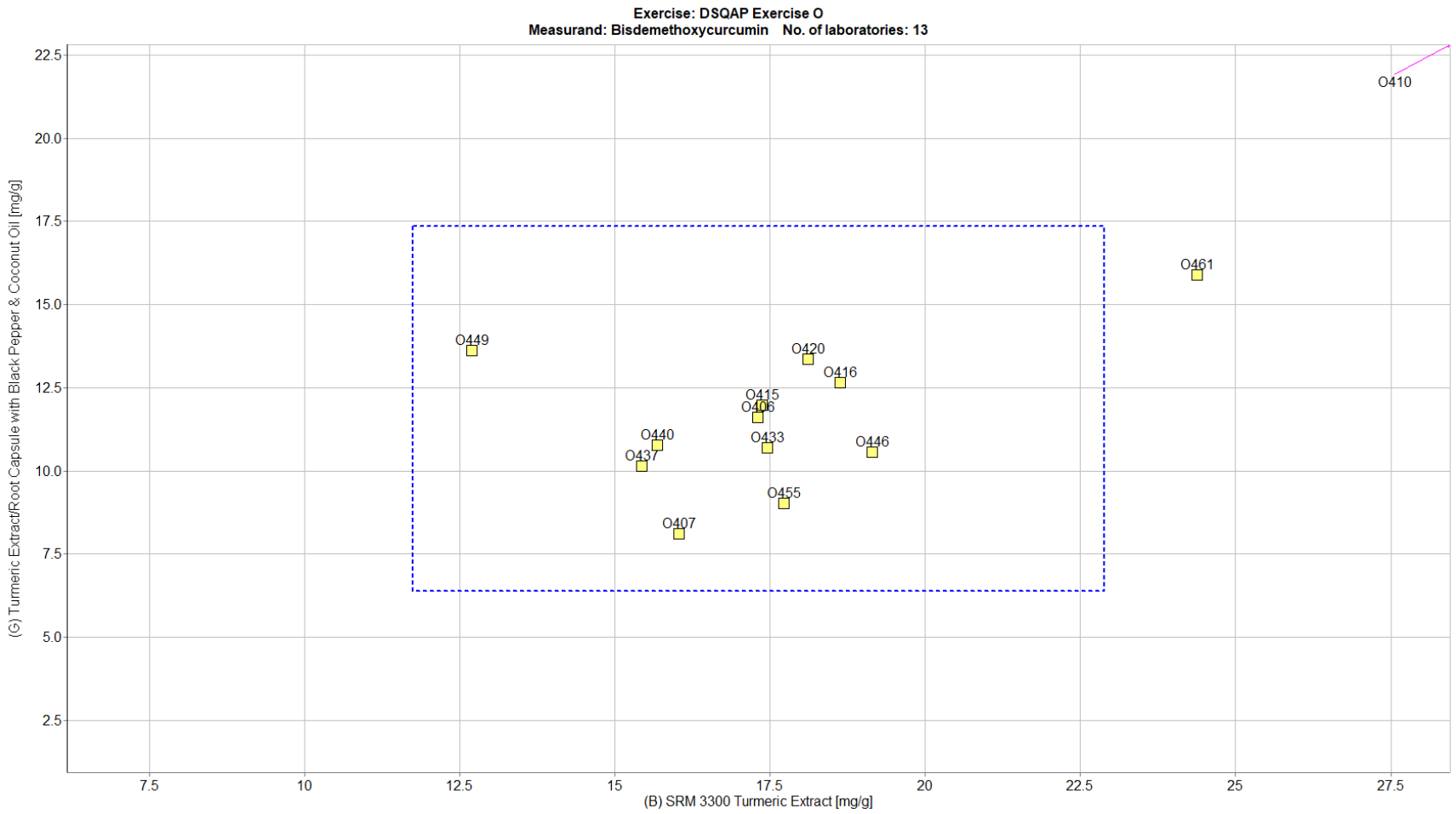


**Figure 2-38.** Laboratory means for BDMC in candidate SRM 3300 Turmeric Extract and Turmeric Extract/Root Capsule with Black Pepper & Coconut Oil (sample/sample comparison view). In this view, the individual laboratory mean for one sample (turmeric extract) is compared to the mean for a second sample (turmeric capsule). The dotted blue box represents the consensus range of tolerance for turmeric extract (x-axis) and turmeric capsule (y-axis), calculated as the values above and below the consensus means that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$.

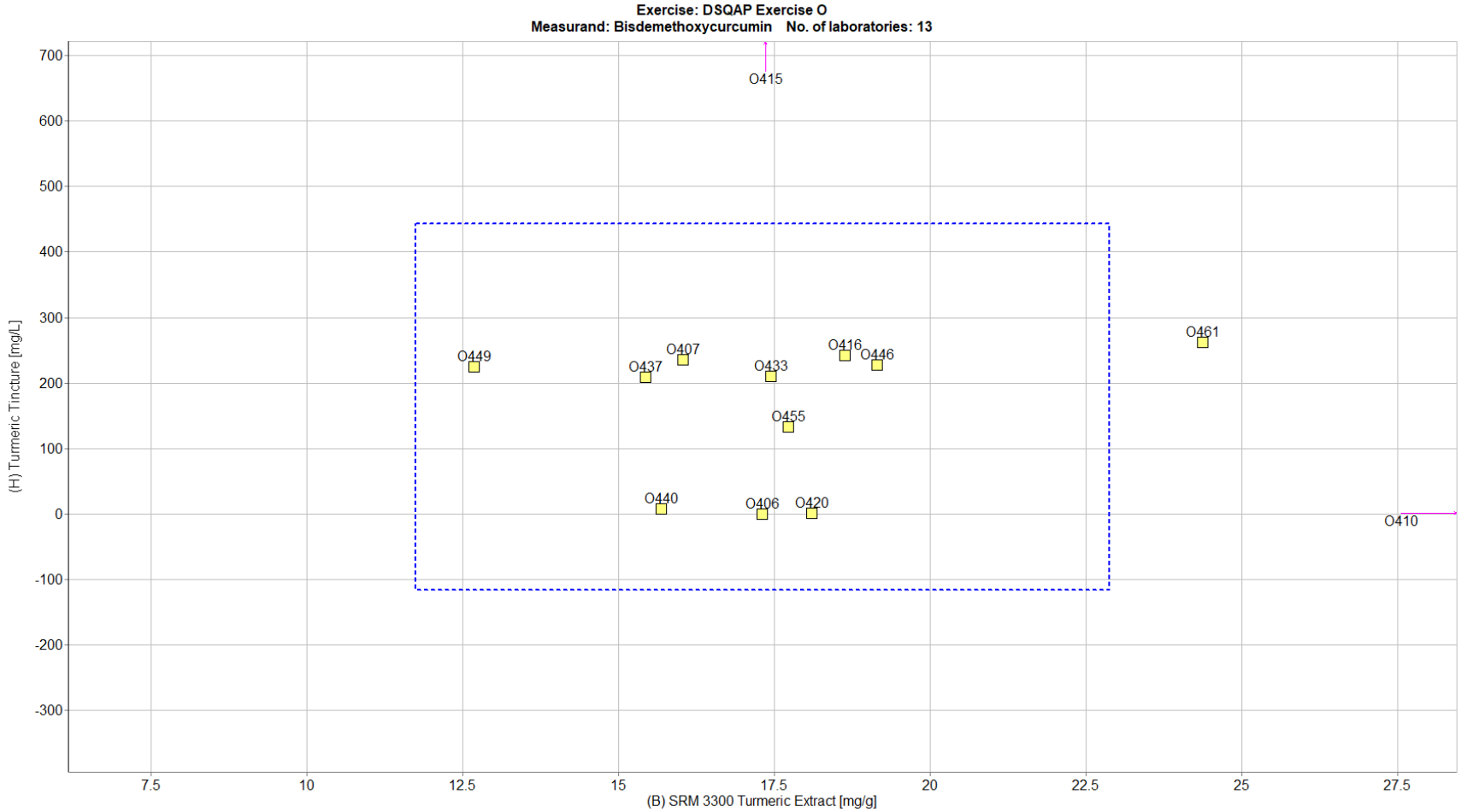


**Figure 2-39.** Laboratory means for BDMC in candidate SRM 3300 Turmeric Extract and Turmeric Tincture (sample/sample comparison view). In this view, the individual laboratory mean for one sample (turmeric extract) is compared to the mean for a second sample (turmeric tincture). The dotted blue box represents the consensus range of tolerance for turmeric extract (x-axis) and turmeric tincture (y-axis), calculated as the values above and below the consensus means that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$.

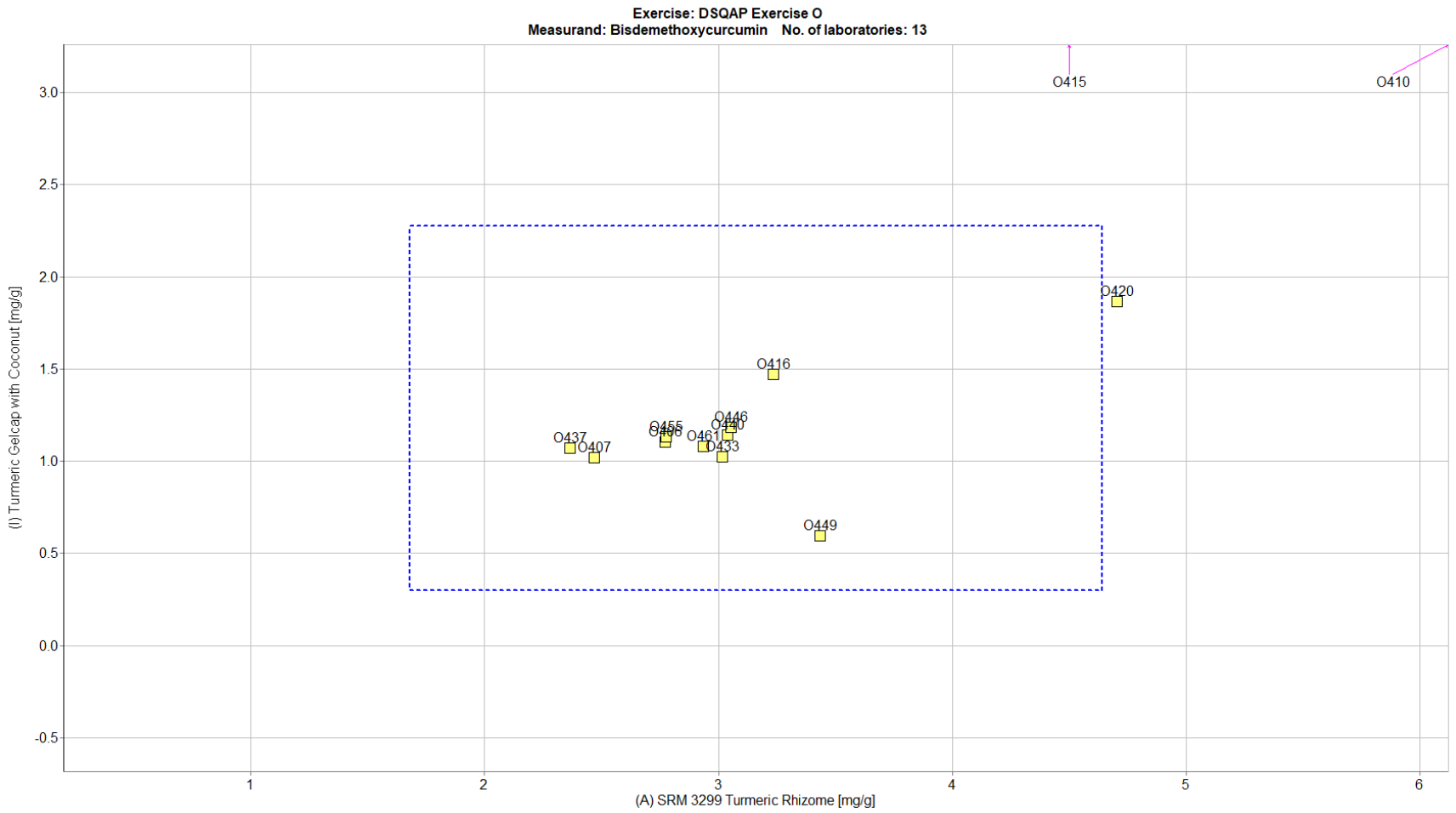


**Figure 2-40.** Laboratory means for BDMC in candidate SRM 3299 Turmeric Rhizome and Turmeric Gelcap with Coconut (sample/sample comparison view). In this view, the individual laboratory mean for one sample (turmeric rhizome) is compared to the mean for a second sample (turmeric gelcap). The dotted blue box represents the consensus range of tolerance for turmeric rhizome (x-axis) and turmeric gelcap (y-axis), calculated as the values above and below the consensus means that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$.

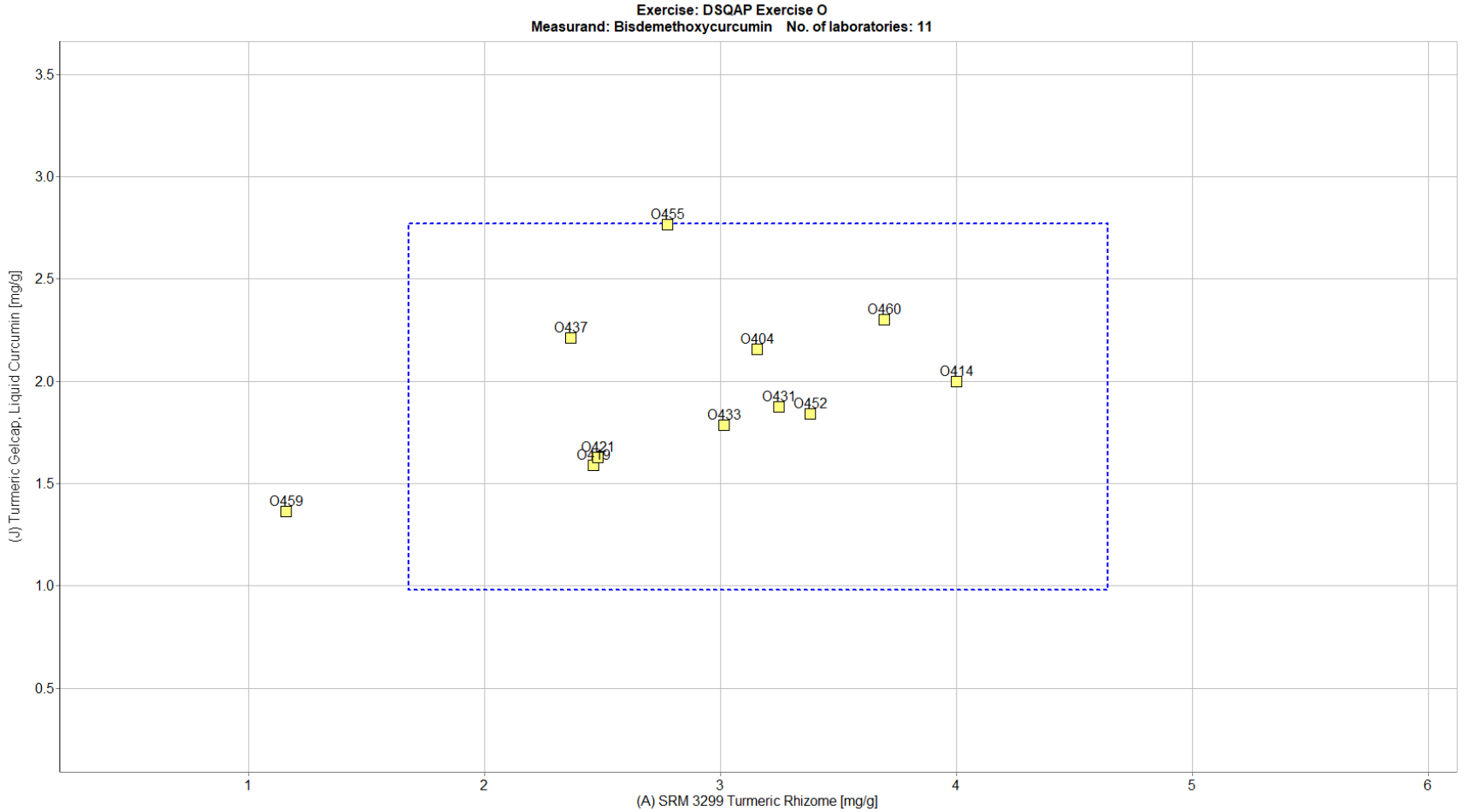


**Figure 2-41.** Laboratory means for BDMC in candidate SRM 3299 Turmeric Rhizome and Turmeric Gelcap, Liquid Curcumin (sample/sample comparison view). In this view, the individual laboratory mean for one sample (turmeric rhizome) is compared to the mean for a second sample (turmeric gelcap). The dotted blue box represents the consensus range of tolerance for turmeric rhizome (x-axis) and turmeric gelcap (y-axis), calculated as the values above and below the consensus means that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$.

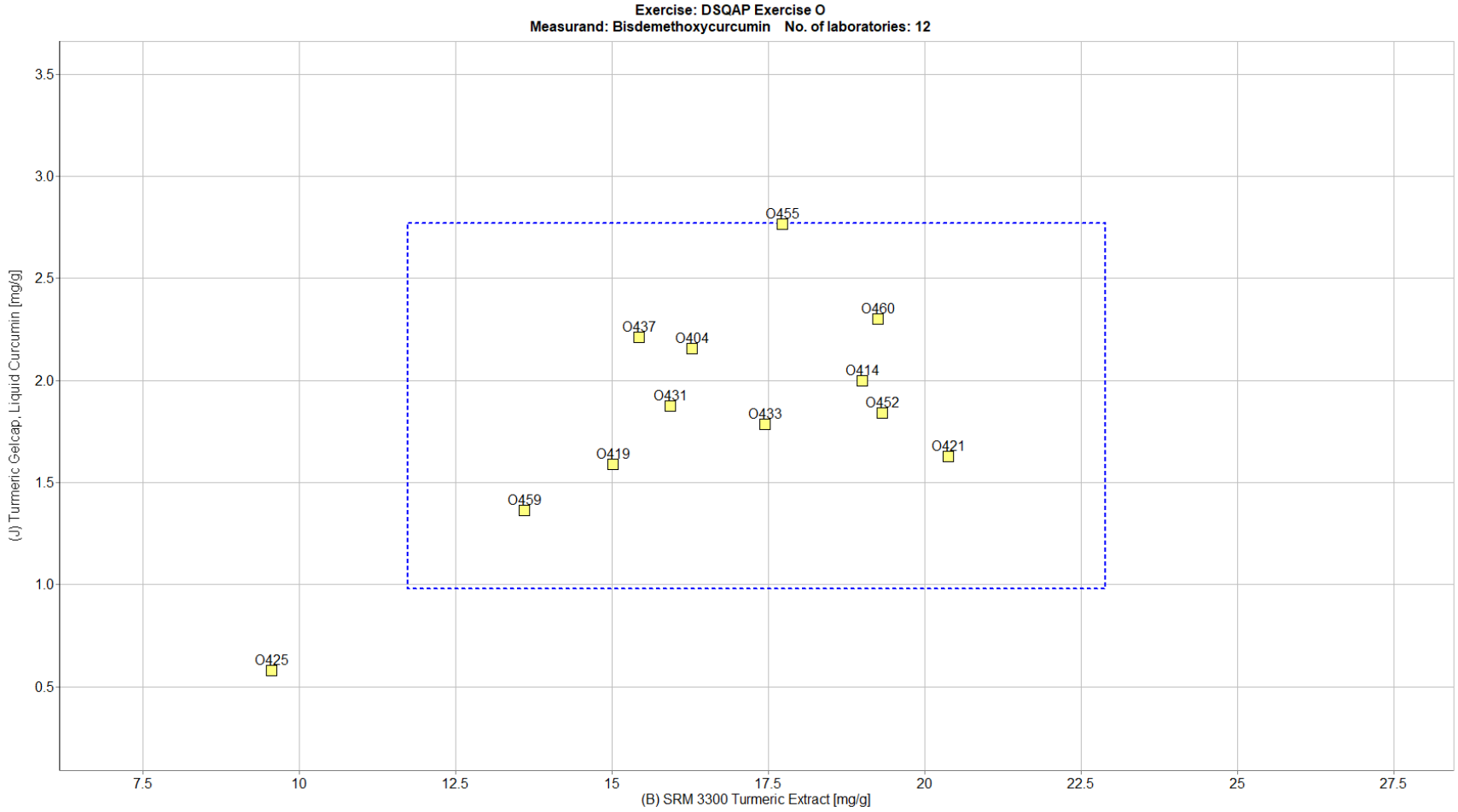


**Figure 2-42.** Laboratory means for BDMC in candidate SRM 3300 Turmeric Extract and Turmeric Gelcap with Coconut (sample/sample comparison view). In this view, the individual laboratory mean for one sample (turmeric extract) is compared to the mean for a second sample (turmeric gelcap). The dotted blue box represents the consensus range of tolerance for turmeric extract (x-axis) and turmeric gelcap (y-axis), calculated as the values above and below the consensus means that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$.

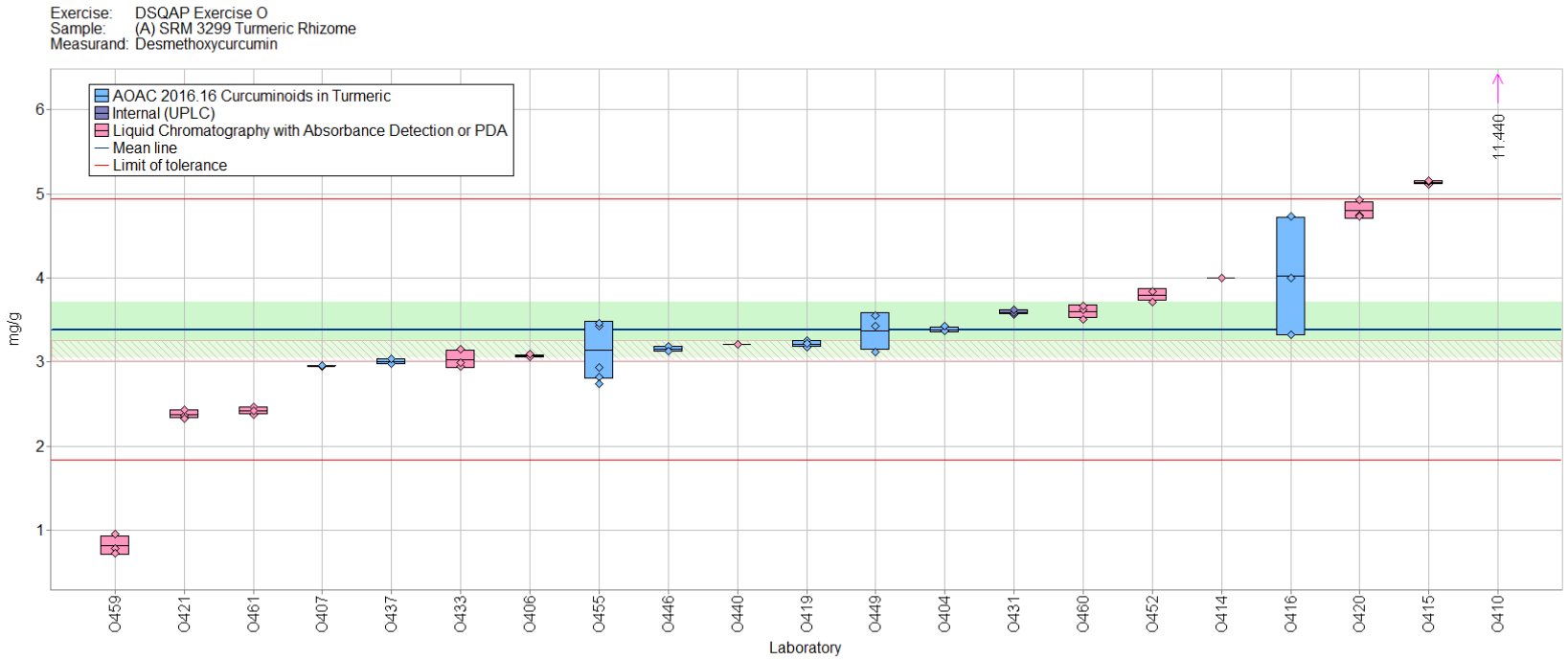


**Figure 2-43.** DMC in candidate SRM 3299 Turmeric Rhizome (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by its uncertainty ($U_{\text{NIST}}$) and represents the range that results in an acceptable $Z_{\text{NIST}}$ score, $|Z_{\text{NIST}}| \leq 2$.

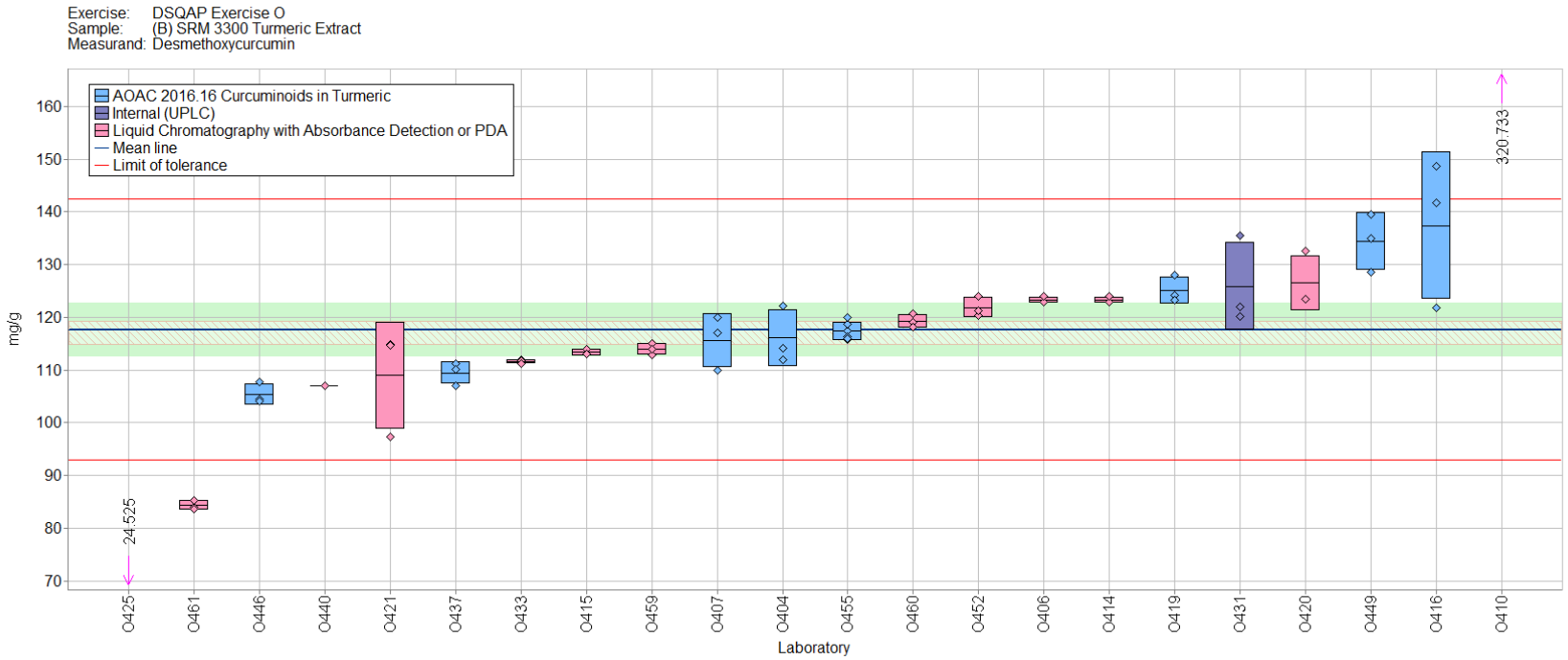


**Figure 2-44.** DMC in candidate SRM 3300 Turmeric Extract (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by its uncertainty ($U_{\text{NIST}}$) and represents the range that results in an acceptable $Z_{\text{NIST}}$ score, $|Z_{\text{NIST}}| \leq 2$.

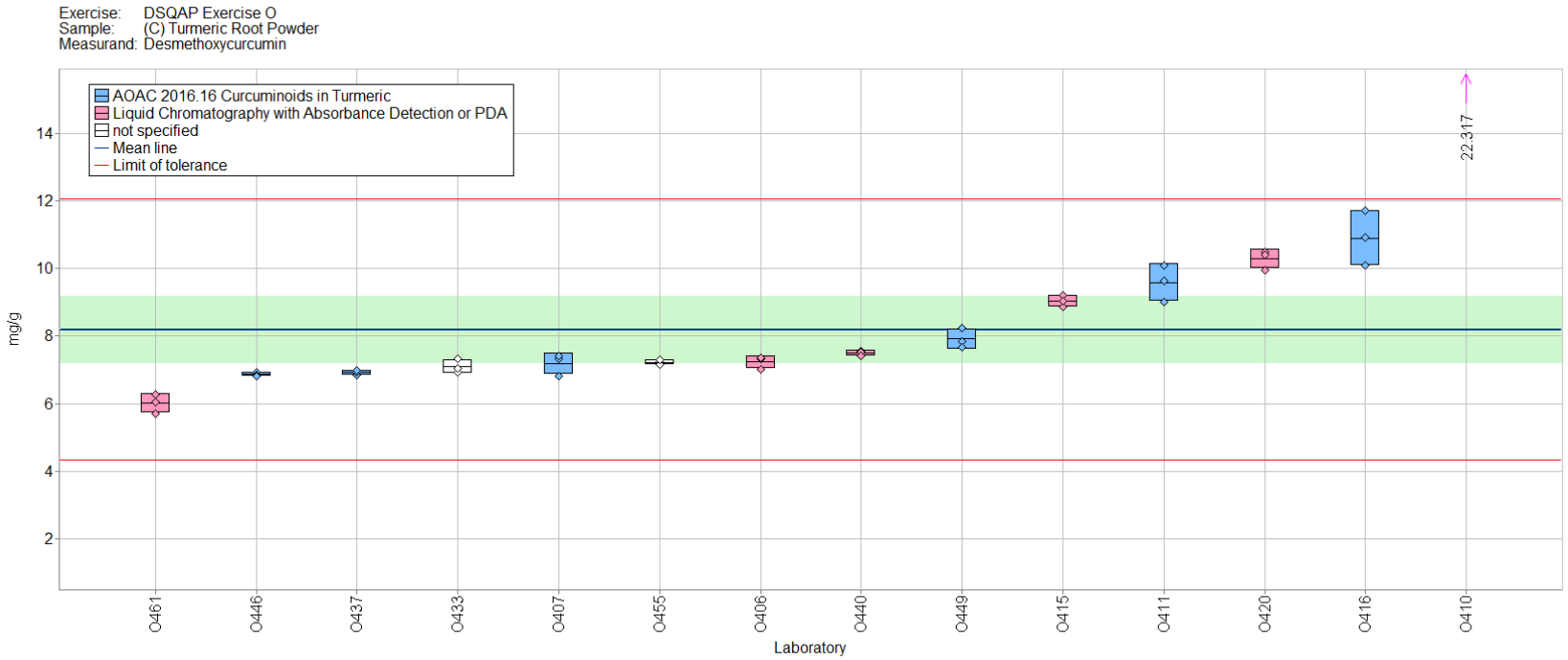


**Figure 2-45.** DMC in Turmeric Root Powder (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$. A NIST value has not been determined in this material.

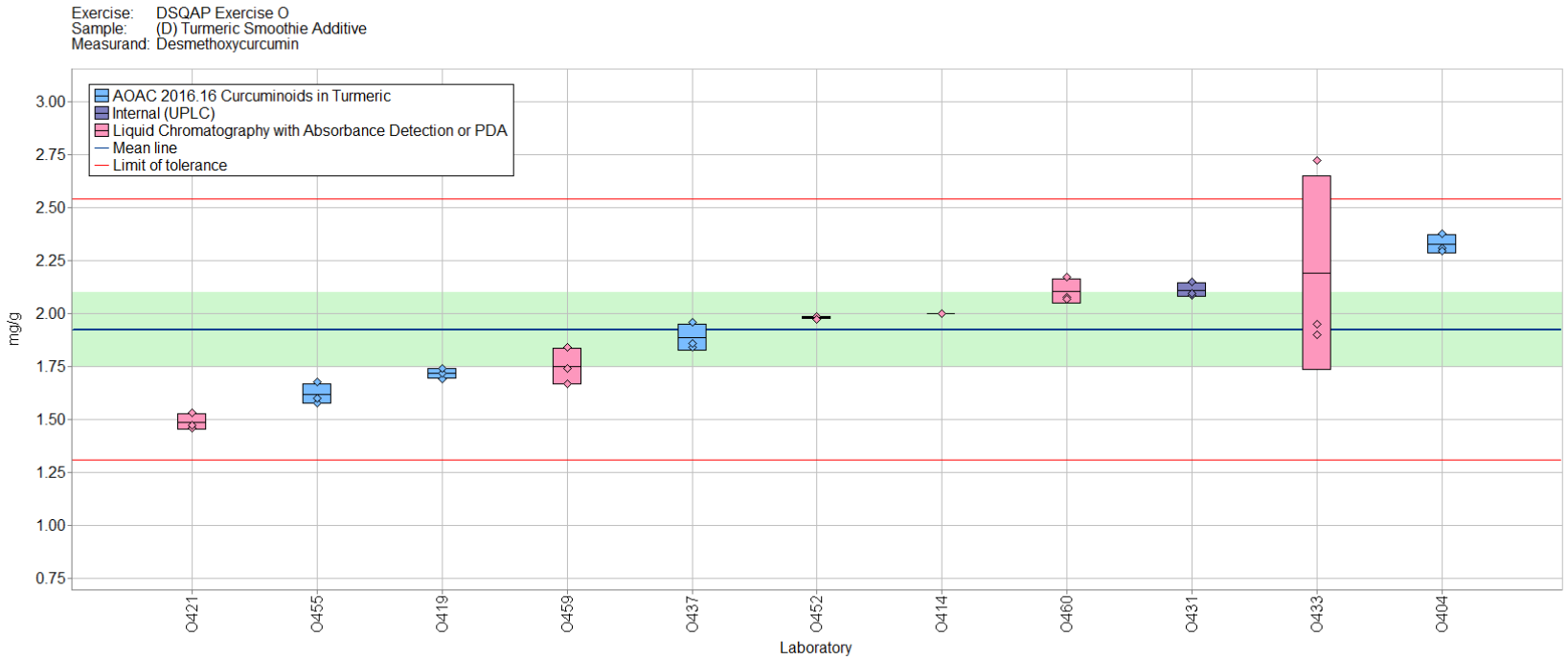


**Figure 2-46.** DMC in Turmeric Smoothie Additive (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$. A NIST value has not been determined in this material.

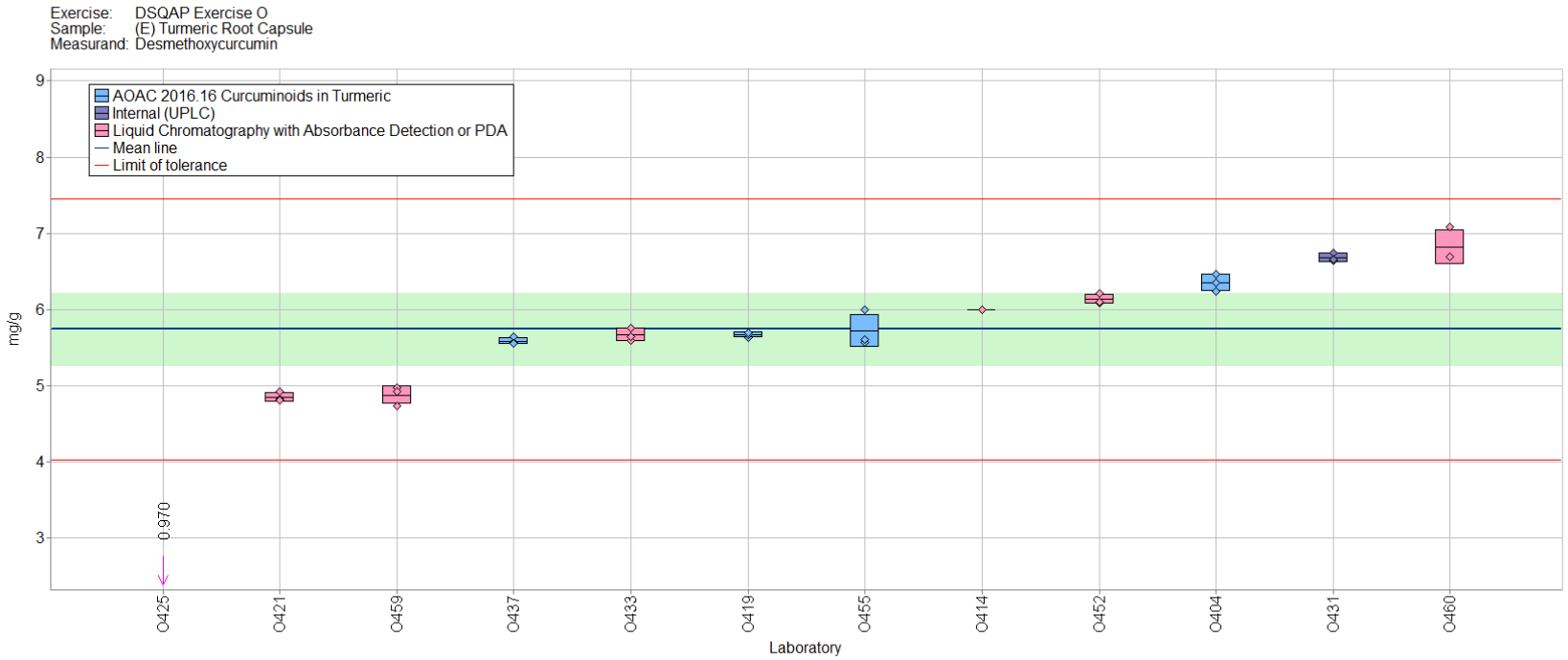


**Figure 2-47.** DMC in Turmeric Root Capsule (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$. A NIST value has not been determined in this material.

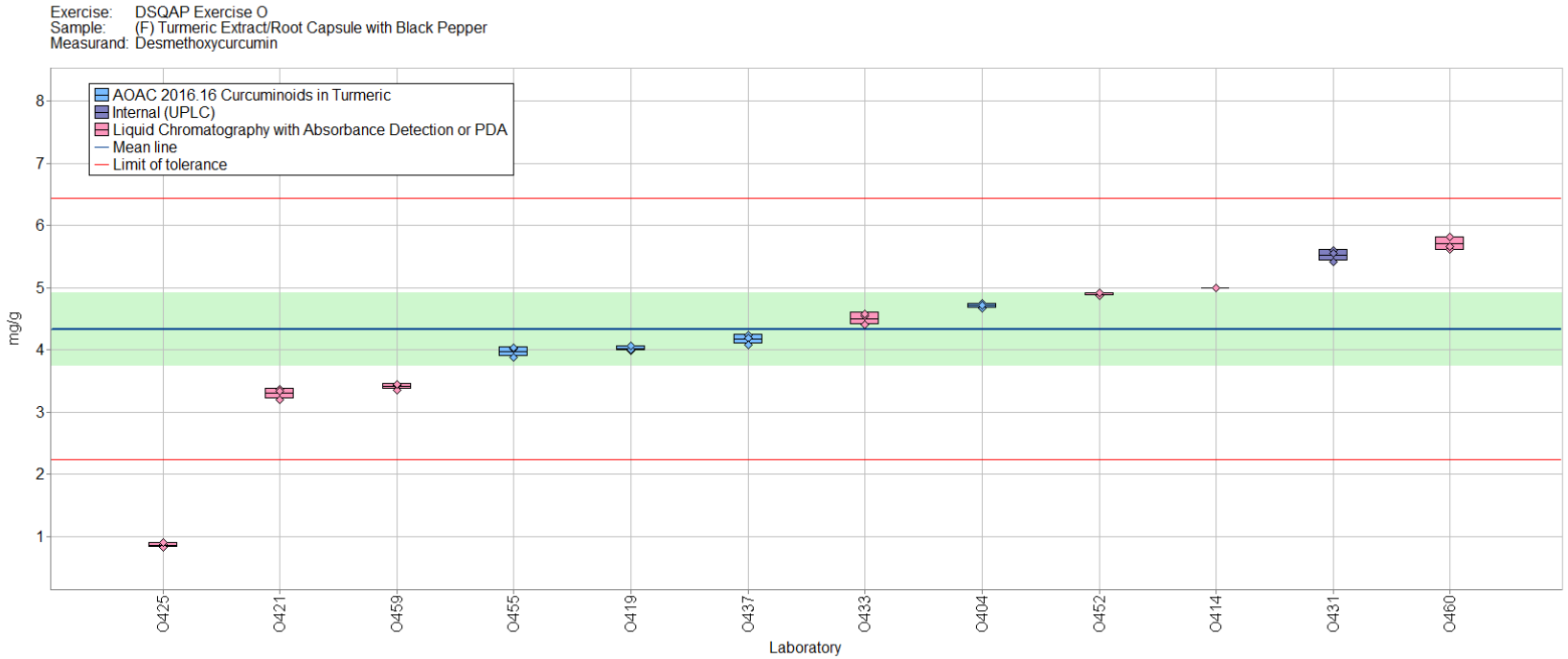


**Figure 2-48.** DMC in Turmeric Extract/Root Capsule with Black Pepper (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$. A NIST value has not been determined in this material.

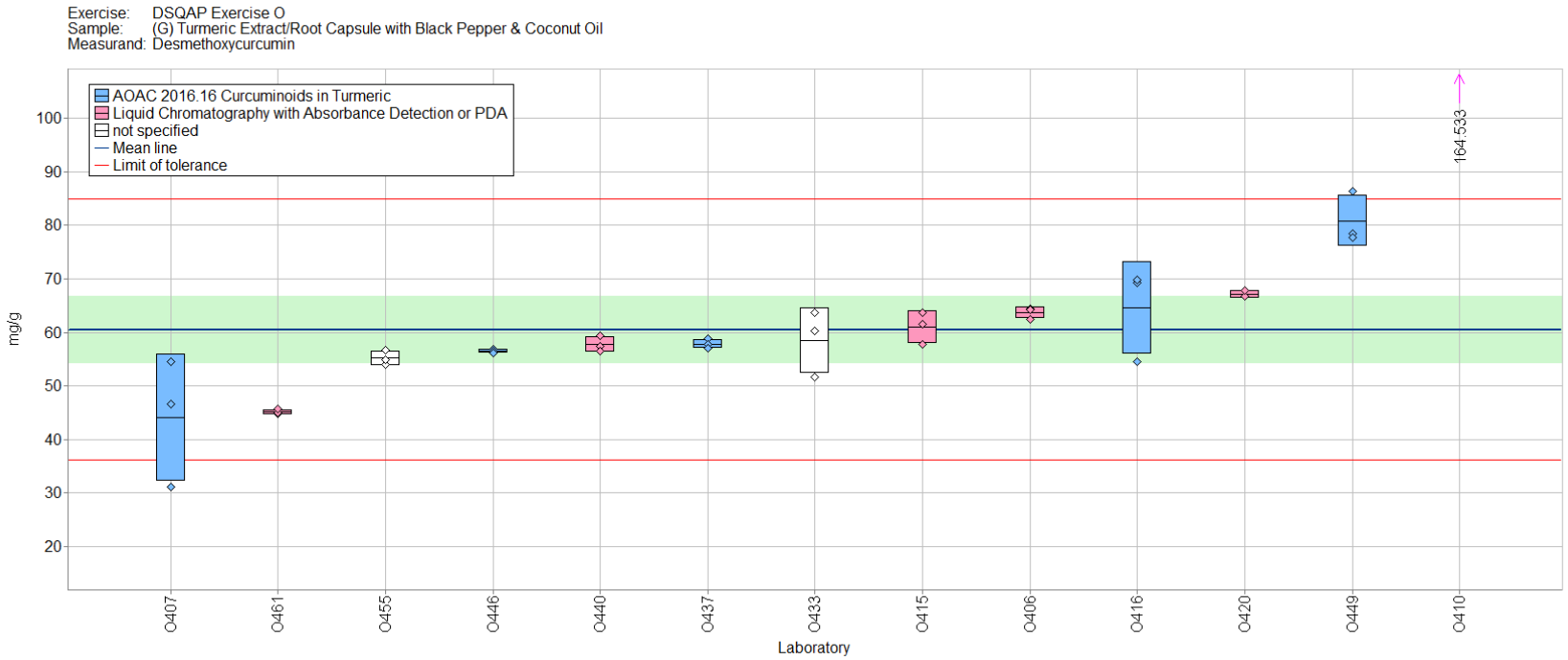


**Figure 2-49.** DMC in Turmeric Extract/Root Capsule with Black Pepper & Coconut Oil (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$. A NIST value has not been determined in this material.

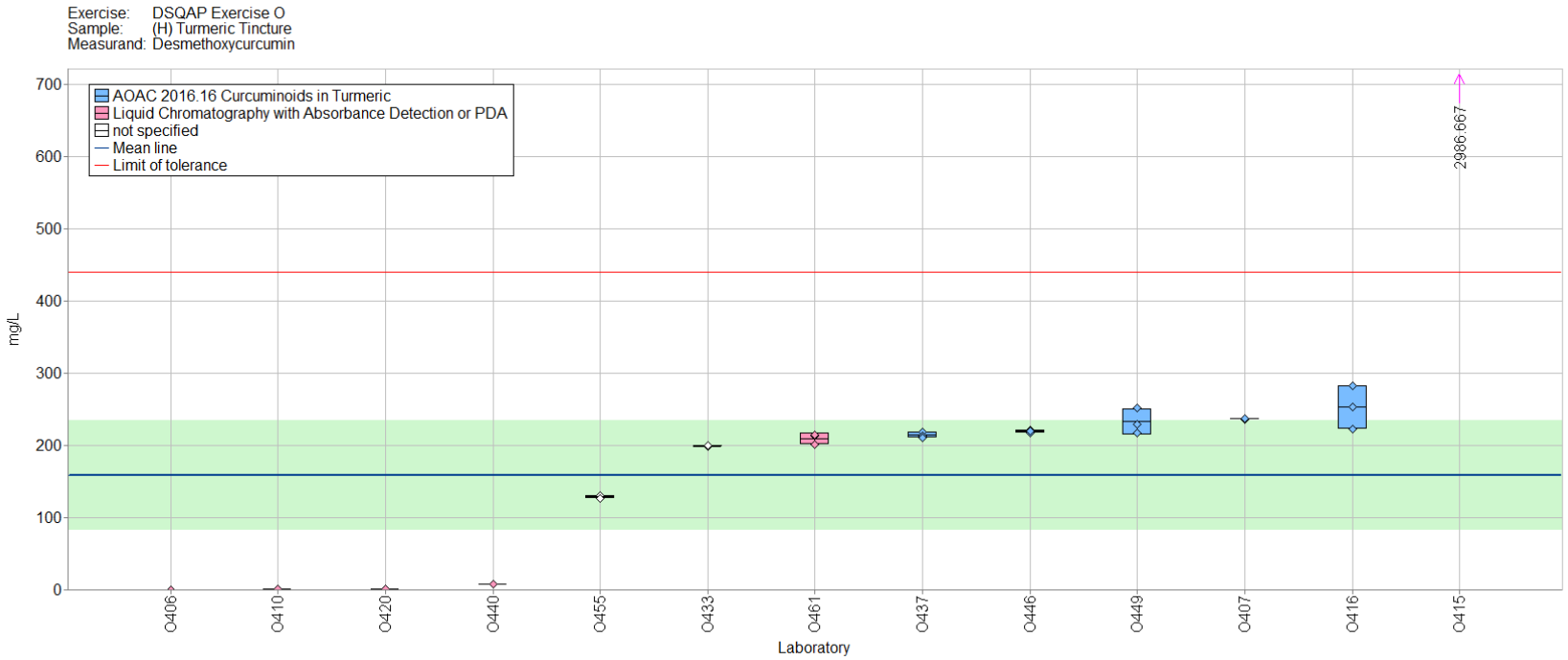


**Figure 2-50.** DMC in Turmeric Tincture (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$. A NIST value has not been determined in this material.

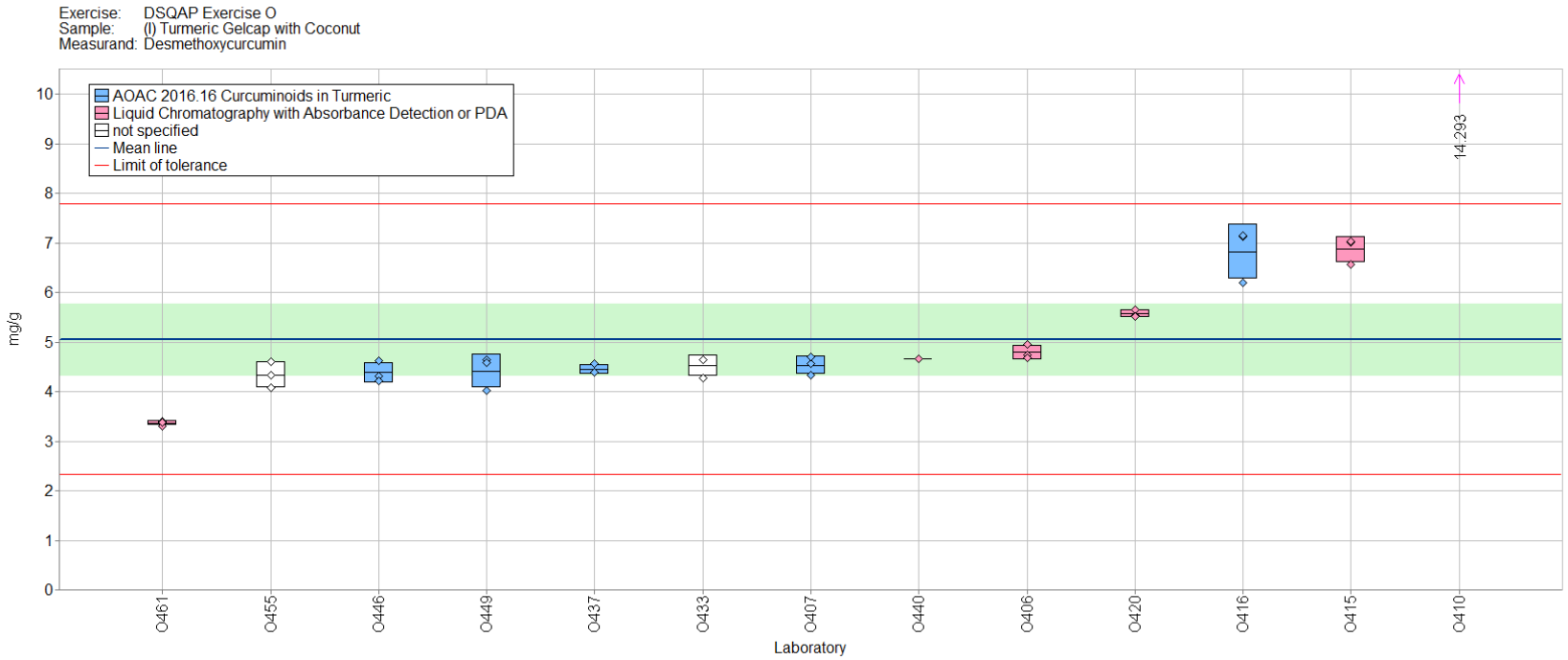


**Figure 2-51.** DMC in Turmeric Gelcap with Coconut (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$. A NIST value has not been determined in this material.

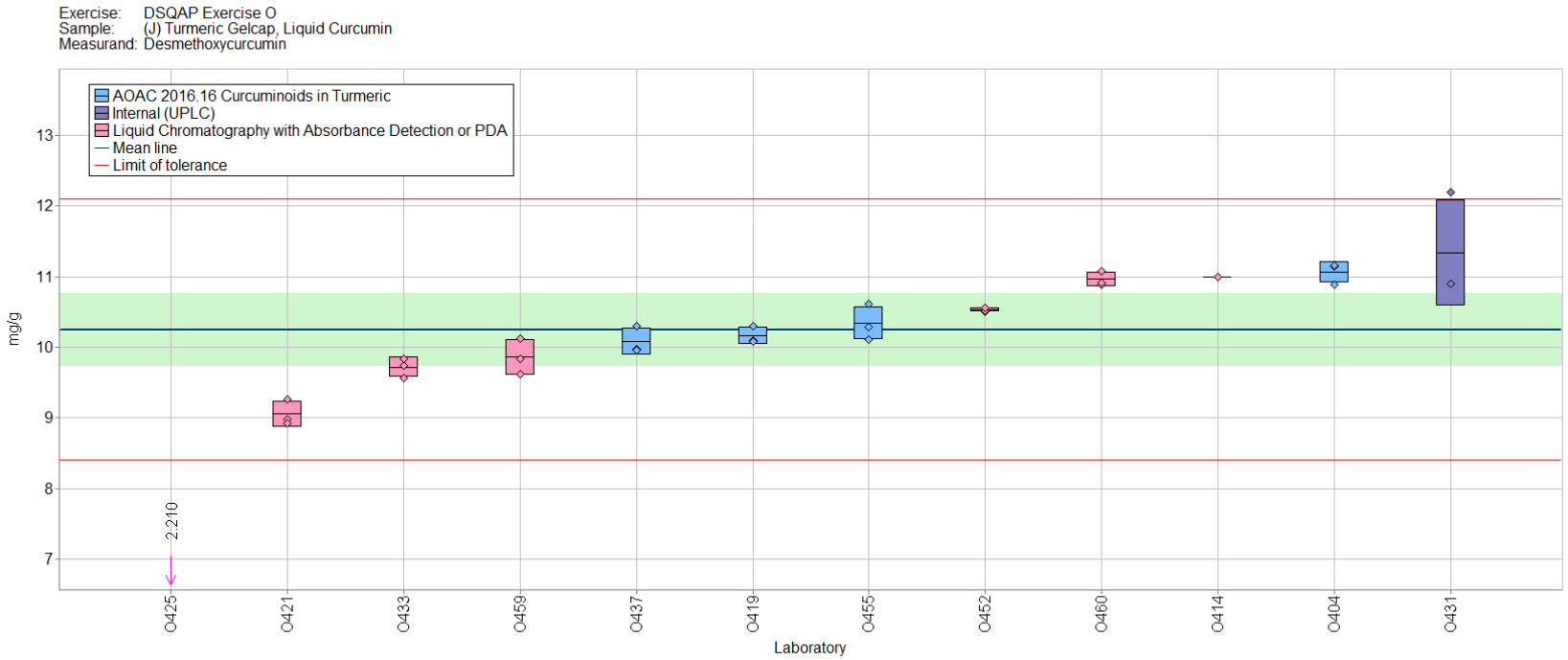


**Figure 2-52.** DMC in Turmeric Gelcap, Liquid Curcumin (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$. A NIST value has not been determined in this material.

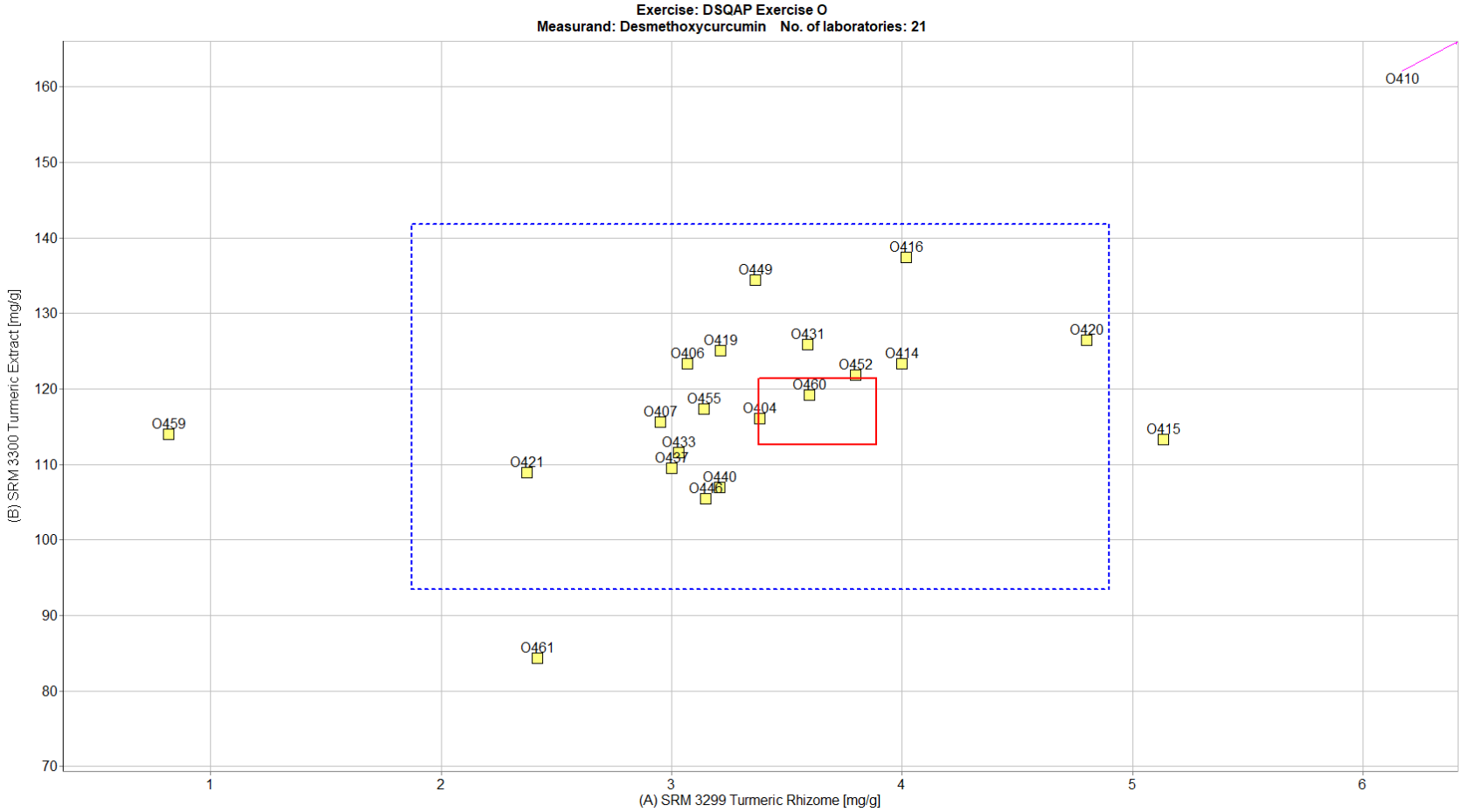


**Figure 2-53.** Laboratory means for DMC in candidate SRM 3299 Turmeric Rhizome and candidate SRM 3300 Turmeric Extract (sample/sample comparison view). In this view, the individual laboratory mean for one sample (turmeric rhizome) is compared to the mean for a second sample (turmeric extract). The solid red box represents the NIST range of tolerance for the two samples, turmeric rhizome (x-axis) and turmeric extract (y-axis), which encompasses the NIST-determined values bounded by their uncertainties ($U_{\mathrm{NIST}}$) and represents the range that results in an acceptable $Z_{\mathrm{NIST}}$ score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for turmeric rhizome (x-axis) and turmeric extract (y-axis), calculated as the values above and below the consensus means that result in an acceptable $Z'_{\mathrm{comm}}$ score, $|Z'_{\mathrm{comm}}| \leq 2$.

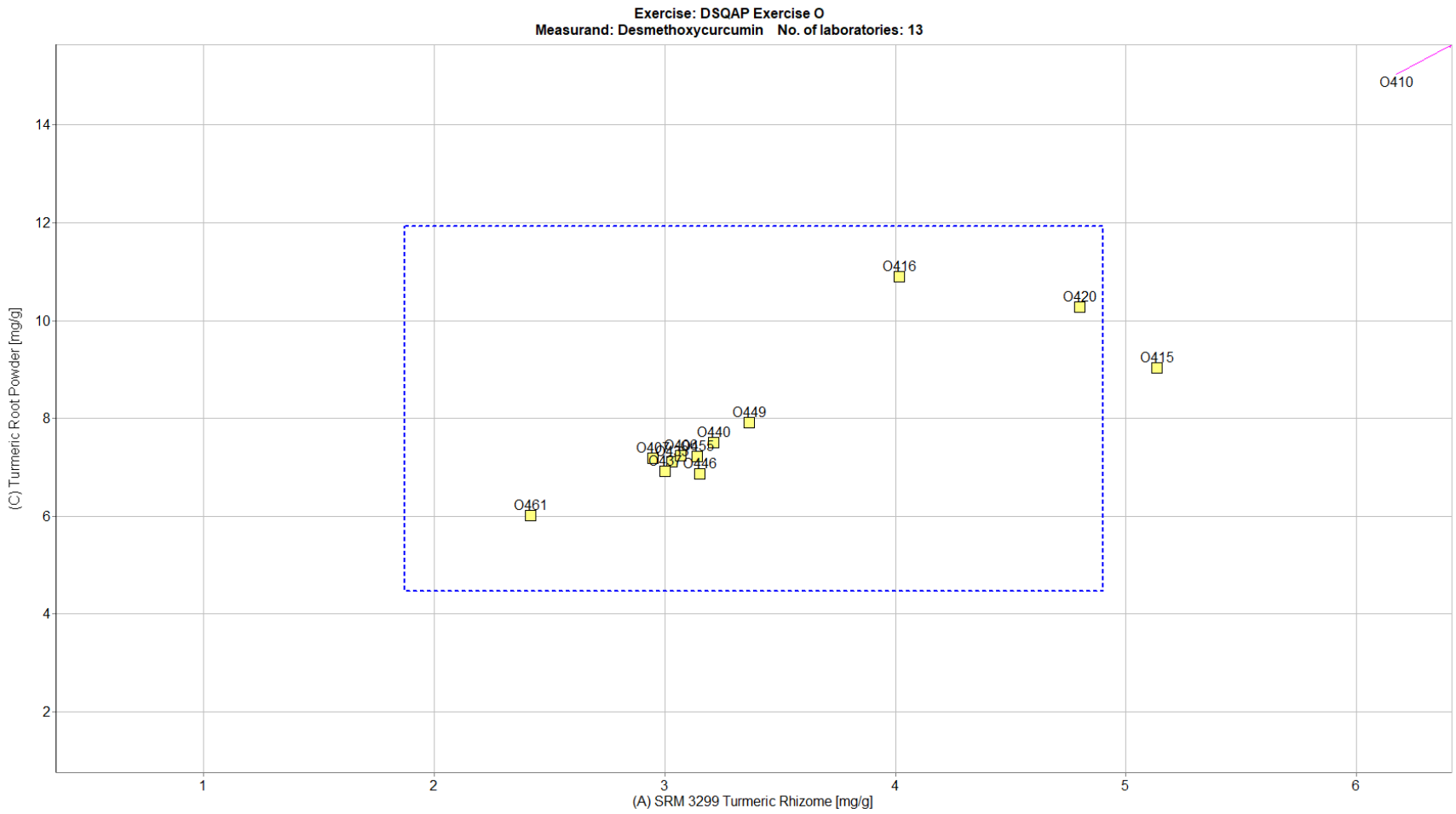


**Figure 2-54.** Laboratory means for DMC in candidate SRM 3299 Turmeric Rhizome and Turmeric Root Powder (sample/sample comparison view). In this view, the individual laboratory mean for one sample (turmeric rhizome) is compared to the mean for a second sample (turmeric root powder). The dotted blue box represents the consensus range of tolerance for turmeric rhizome (x-axis) and turmeric root powder (y-axis), calculated as the values above and below the consensus means that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$.

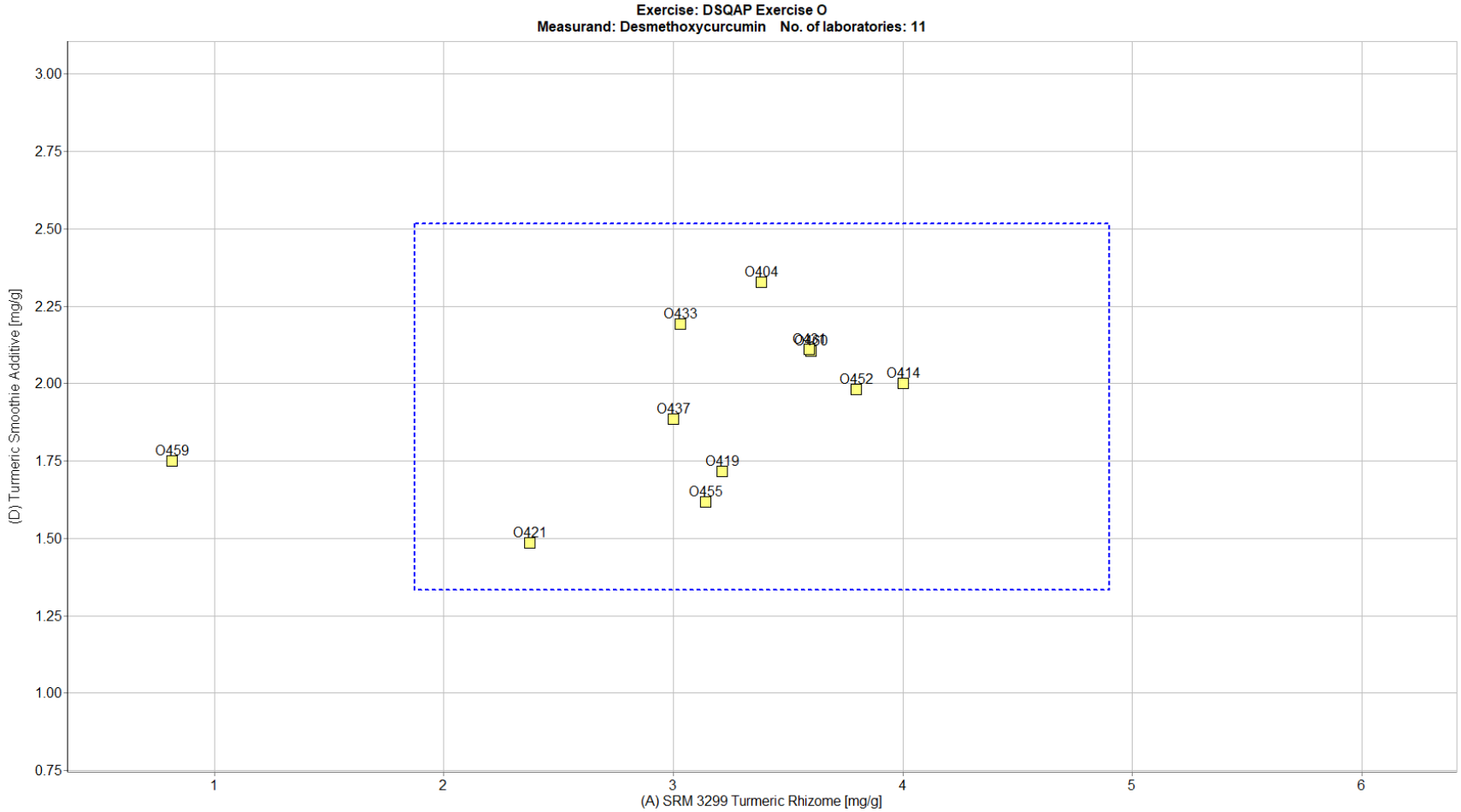


**Figure 2-55.** Laboratory means for DMC in candidate SRM 3299 Turmeric Rhizome and Turmeric Smoothie Additive (sample/sample comparison view). In this view, the individual laboratory mean for one sample (turmeric rhizome) is compared to the mean for a second sample (turmeric smoothie additive). The dotted blue box represents the consensus range of tolerance for turmeric rhizome (x-axis) and turmeric smoothie additive (y-axis), calculated as the values above and below the consensus means that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$.

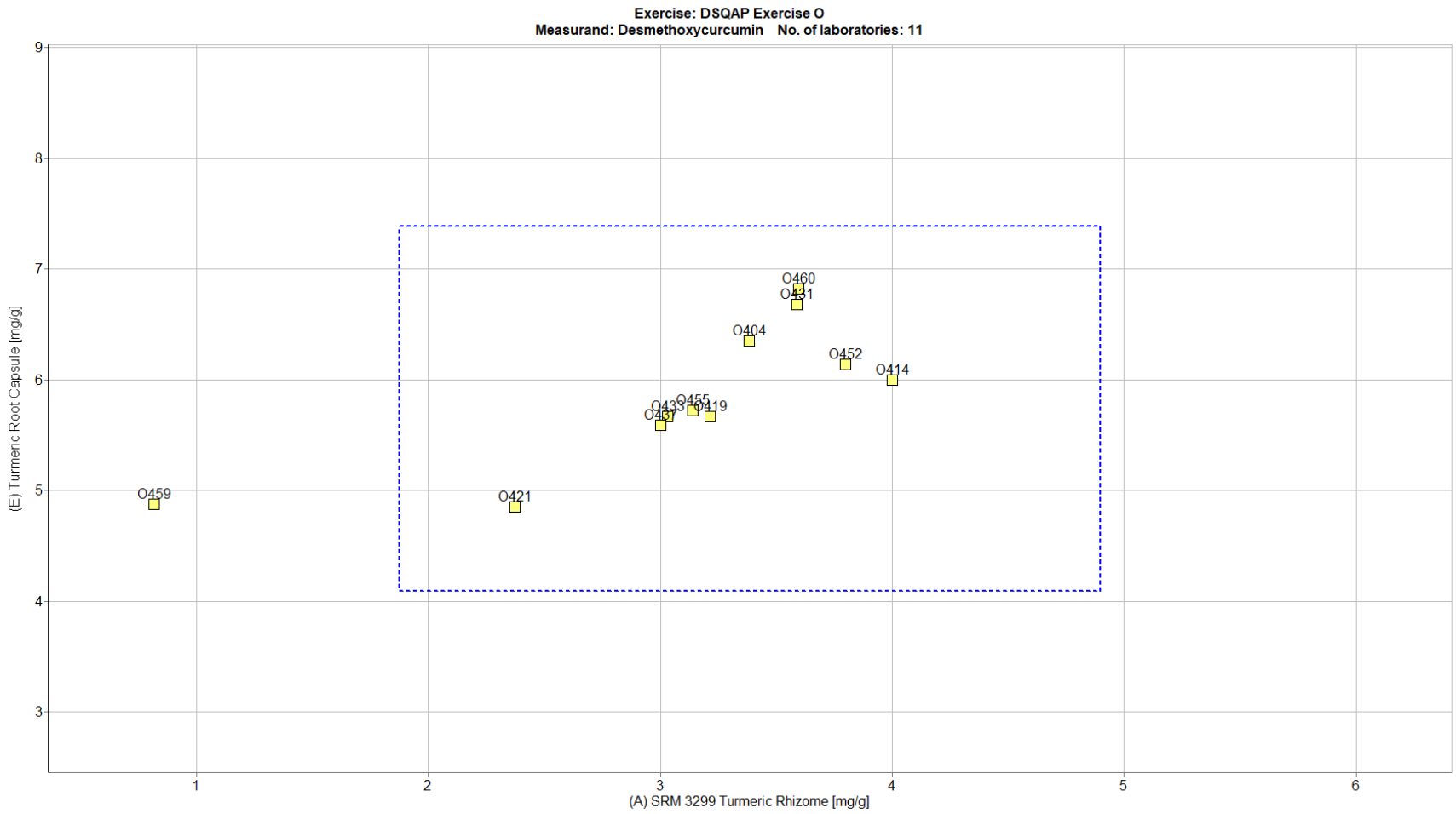


**Figure 2-56.** Laboratory means for DMC in candidate SRM 3299 Turmeric Rhizome and Turmeric Root Capsule (sample/sample comparison view). In this view, the individual laboratory mean for one sample (turmeric rhizome) is compared to the mean for a second sample (turmeric capsule). The dotted blue box represents the consensus range of tolerance for turmeric rhizome (x-axis) and turmeric capsule (y-axis), calculated as the values above and below the consensus means that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$.

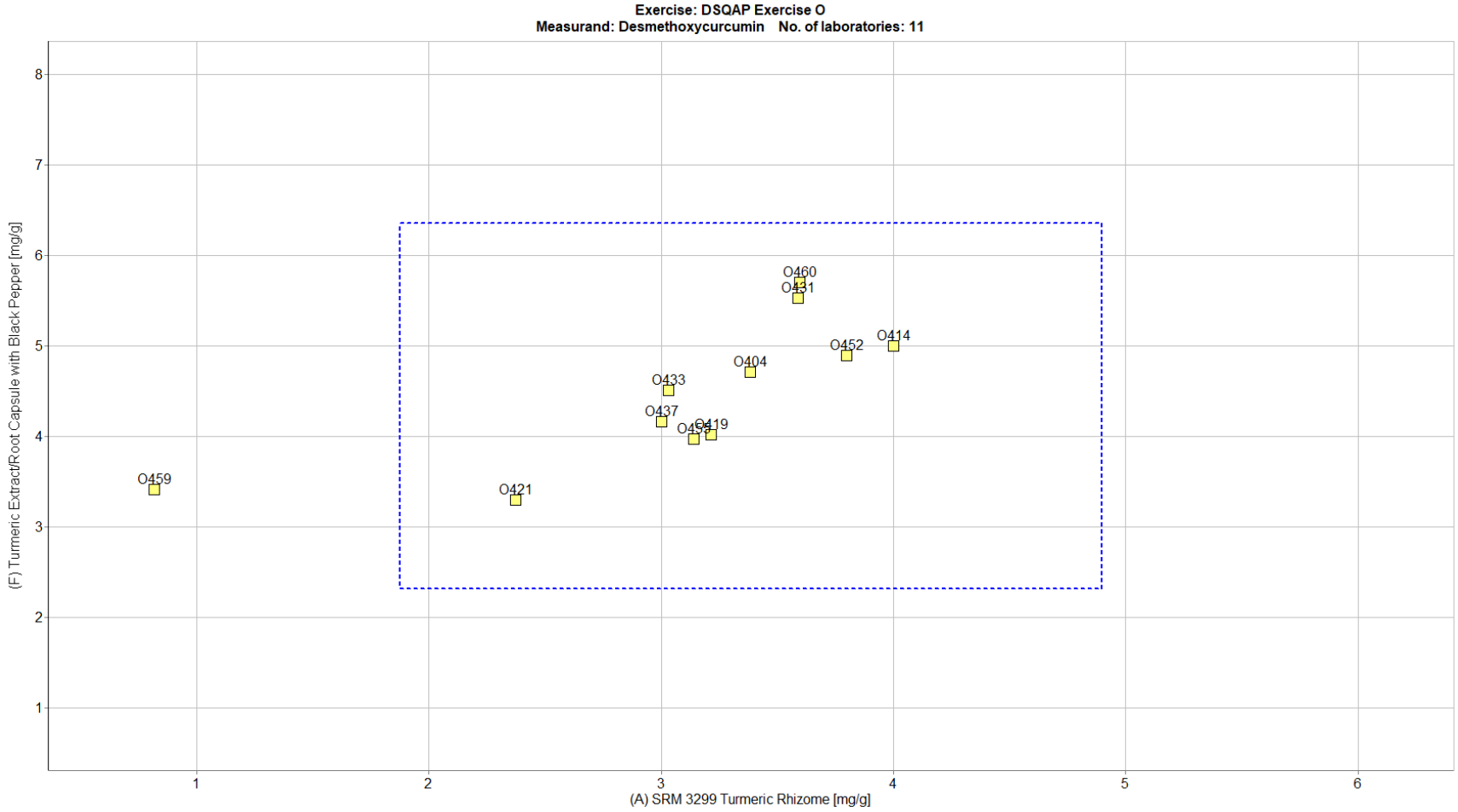


**Figure 2-57.** Laboratory means for DMC in candidate SRM 3299 Turmeric Rhizome and Turmeric Extract/Root Capsule with Black Pepper (sample/sample comparison view). In this view, the individual laboratory mean for one sample (turmeric rhizome) is compared to the mean for a second sample (turmeric capsule). The dotted blue box represents the consensus range of tolerance for turmeric rhizome (x-axis) and turmeric capsule (y-axis), calculated as the values above and below the consensus means that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$.

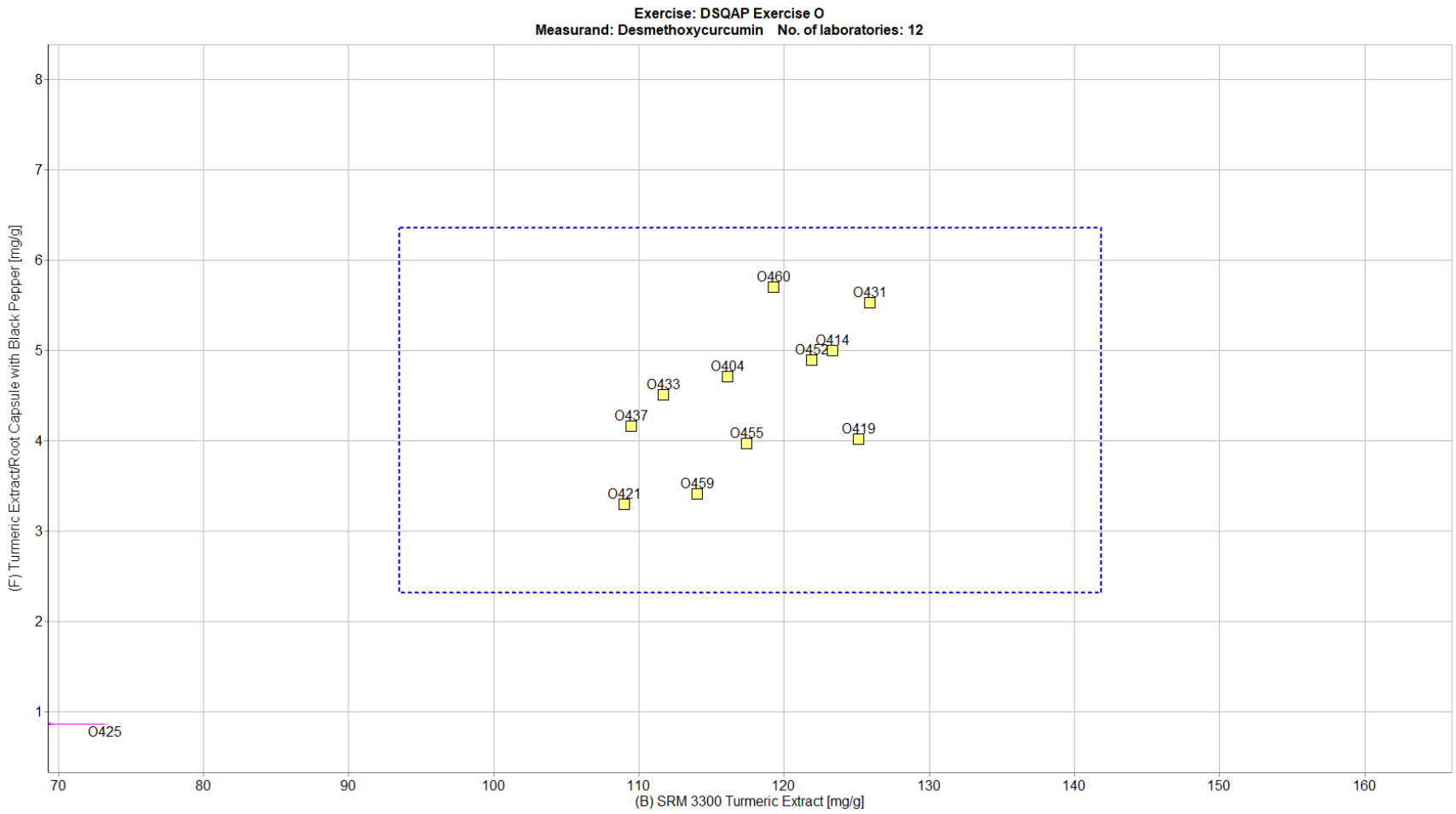


**Figure 2-58.** Laboratory means for DMC in candidate SRM 3300 Turmeric Extract and Turmeric Extract/Root Capsule with Black Pepper (sample/sample comparison view). In this view, the individual laboratory mean for one sample (turmeric extract) is compared to the mean for a second sample (turmeric capsule). The dotted blue box represents the consensus range of tolerance for turmeric extract (x-axis) and turmeric capsule (y-axis), calculated as the values above and below the consensus means that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$.

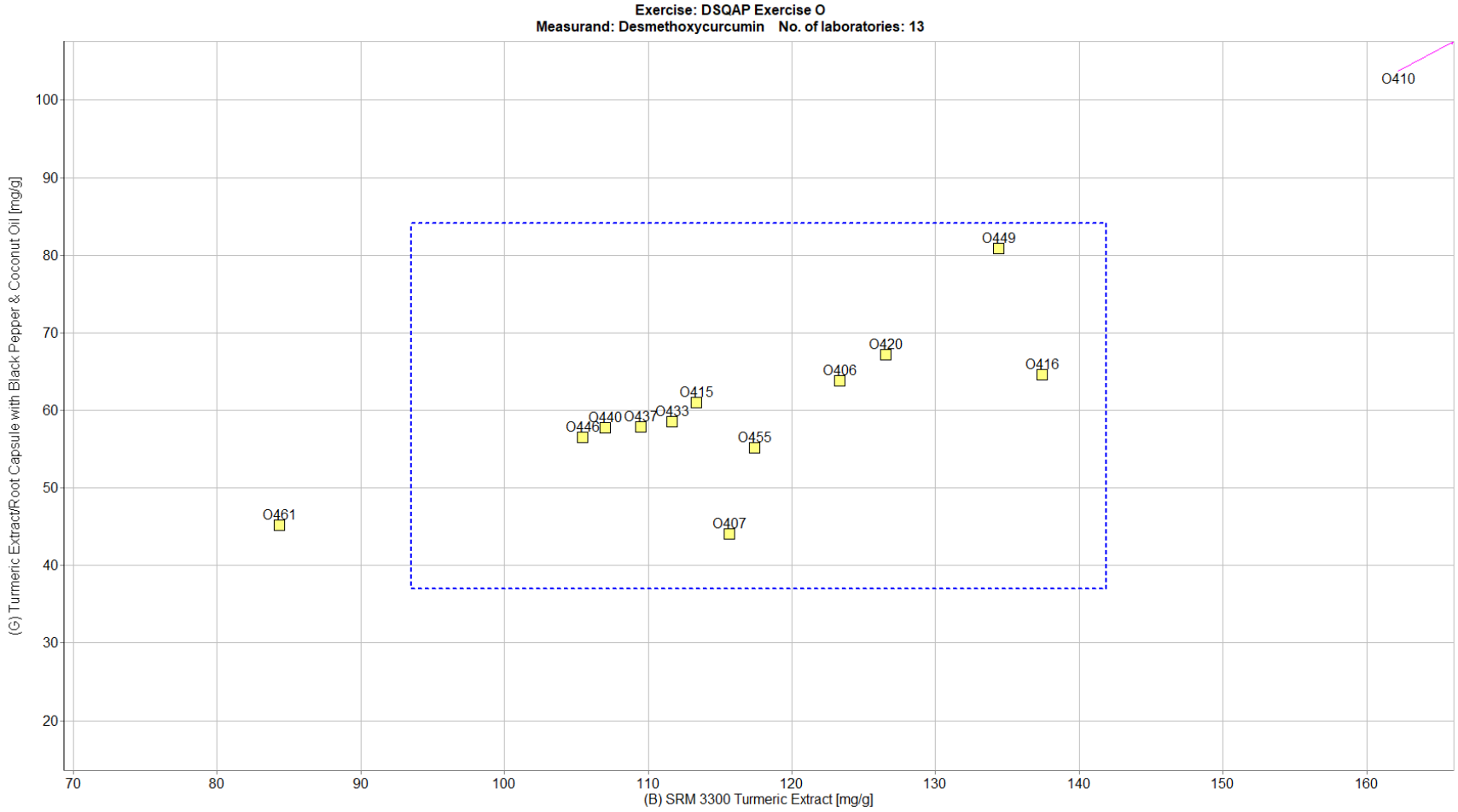


**Figure 2-59.** Laboratory means for DMC in candidate SRM 3300 Turmeric Extract and Turmeric Extract/Root Capsule with Black Pepper & Coconut Oil (sample/sample comparison view). In this view, the individual laboratory mean for one sample (turmeric extract) is compared to the mean for a second sample (turmeric capsule). The dotted blue box represents the consensus range of tolerance for turmeric extract (x-axis) and turmeric capsule (y-axis), calculated as the values above and below the consensus means that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$.

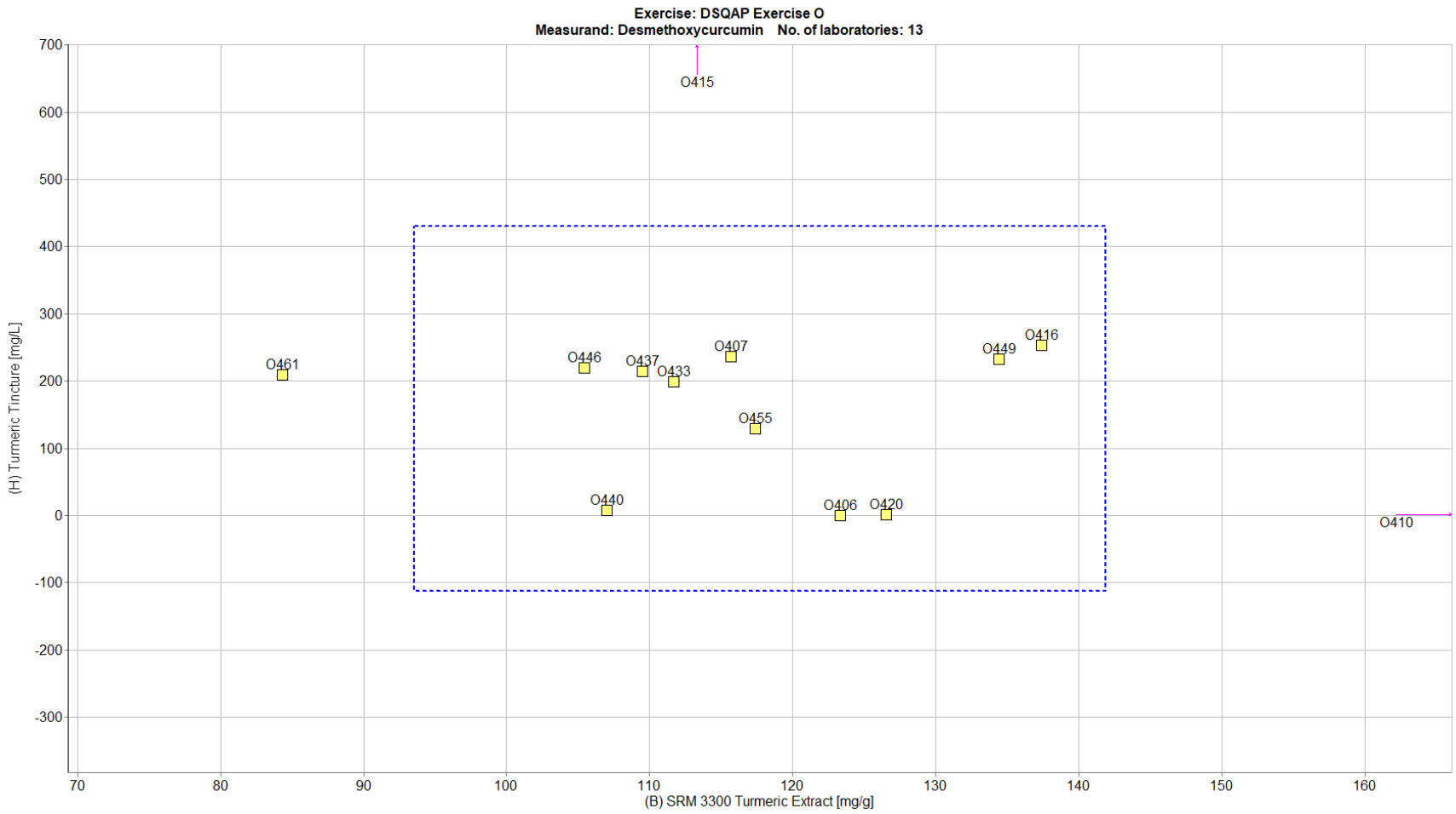


**Figure 2-60.** Laboratory means for DMC in candidate SRM 3300 Turmeric Extract and Turmeric Tincture (sample/sample comparison view). In this view, the individual laboratory mean for one sample (turmeric extract) is compared to the mean for a second sample (turmeric tincture). The dotted blue box represents the consensus range of tolerance for turmeric extract (x-axis) and turmeric tincture (y-axis), calculated as the values above and below the consensus means that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$.

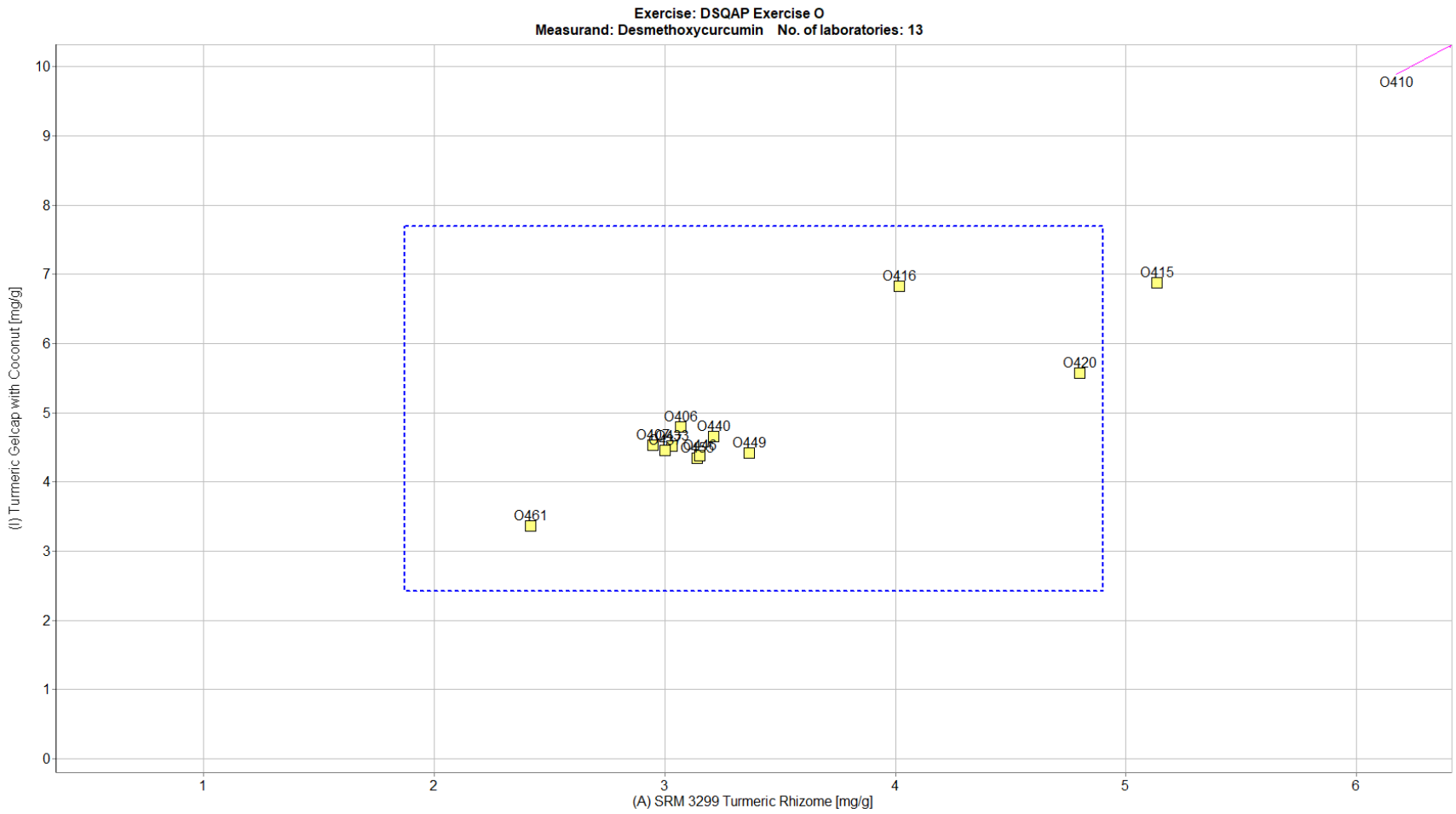


**Figure 2-61.** Laboratory means for DMC in candidate SRM 3299 Turmeric Rhizome and Turmeric Gelcap with Coconut (sample/sample comparison view). In this view, the individual laboratory mean for one sample (turmeric rhizome) is compared to the mean for a second sample (turmeric gelcap). The dotted blue box represents the consensus range of tolerance for turmeric rhizome (x-axis) and turmeric gelcap (y-axis), calculated as the values above and below the consensus means that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$.

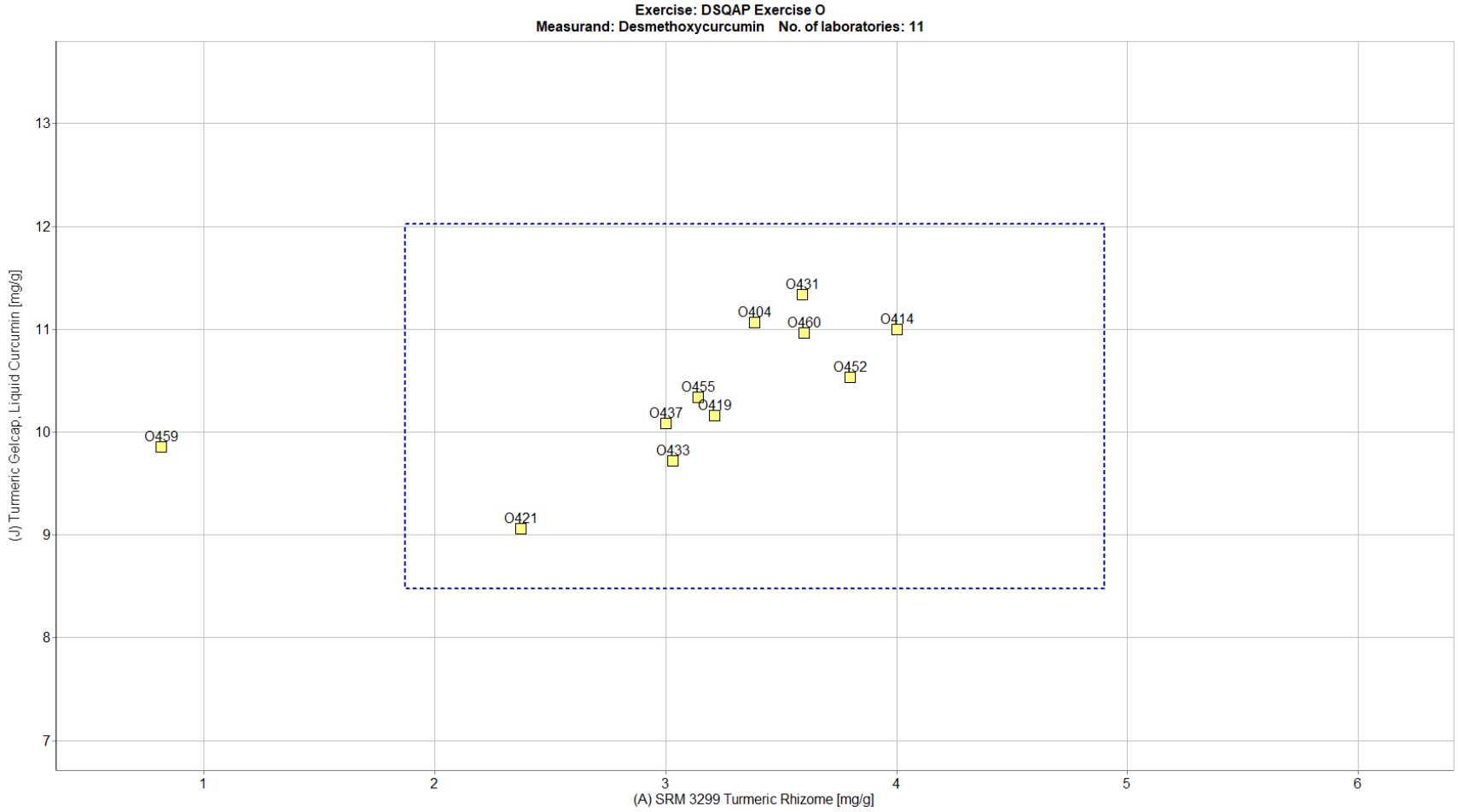


**Figure 2-62.** Laboratory means for DMC in candidate SRM 3299 Turmeric Rhizome and Turmeric Gelcap with Coconut (sample/sample comparison view). In this view, the individual laboratory mean for one sample (turmeric rhizome) is compared to the mean for a second sample (turmeric gelcap). The dotted blue box represents the consensus range of tolerance for turmeric rhizome (x-axis) and turmeric gelcap (y-axis), calculated as the values above and below the consensus means that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$.

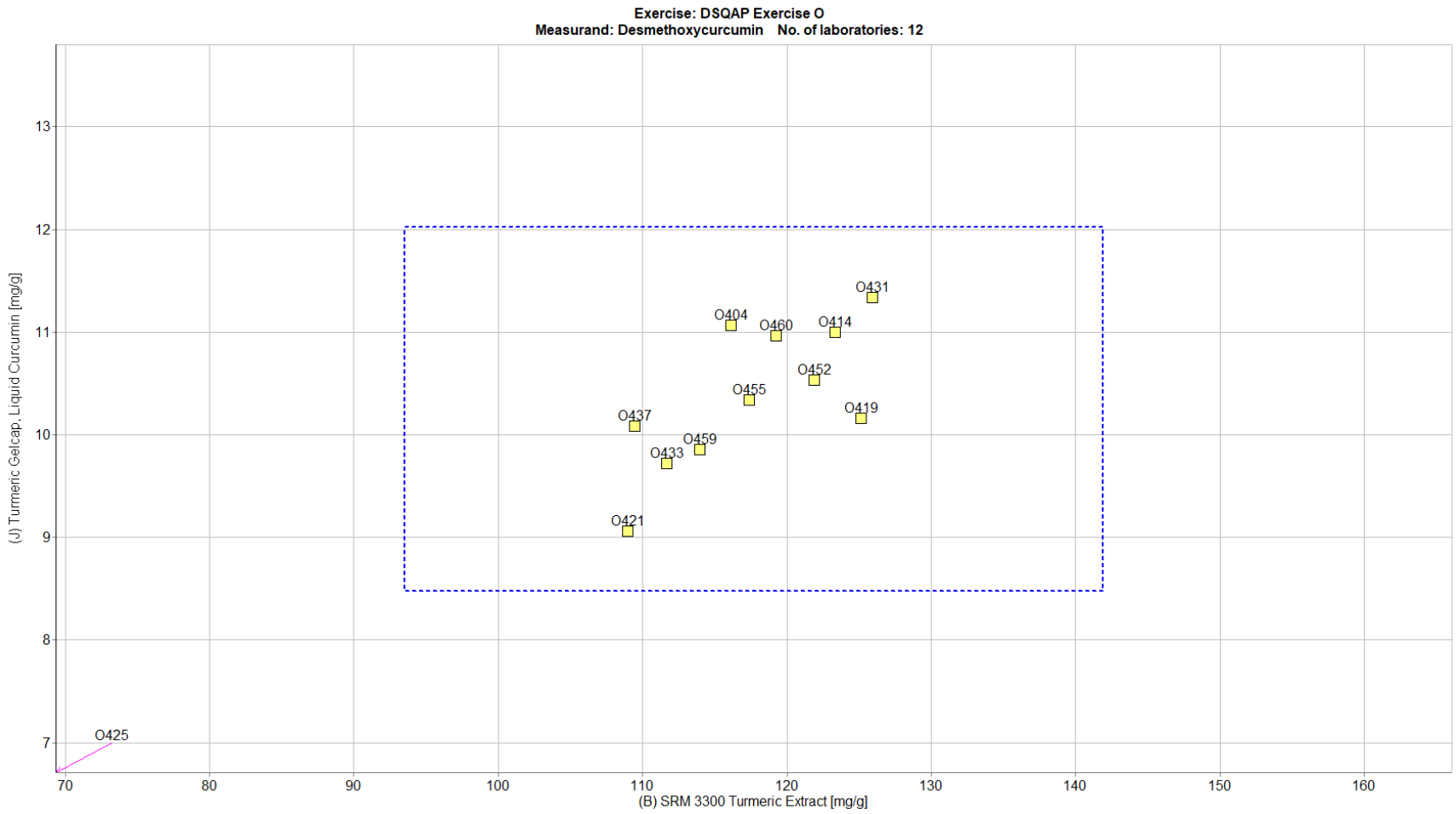


**Figure 2-63.** Laboratory means for DMC in candidate SRM 3300 Turmeric Extract and Turmeric Gelcap with Coconut (sample/sample comparison view). In this view, the individual laboratory mean for one sample (turmeric extract) is compared to the mean for a second sample (turmeric gelcap). The dotted blue box represents the consensus range of tolerance for turmeric extract (x-axis) and turmeric gelcap (y-axis), calculated as the values above and below the consensus means that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$.

## SECTION 3: CHONDROITIN IN DIETARY SUPPLEMENTS

Study Overview

In this study, participants were provided with seven different chondroitin dietary supplements. Participants were asked to use the AOAC First Action *Official Method of Analysis 2015.11 Chondroitin Sulfate Content in Raw Materials and Dietary Supplements* or in-house methods to determine the mass fraction (μg/g) of total chondroitin sulfate in each matrix. For those laboratories interested in using the AOAC method, a copy of the method was enclosed, and participants were advised to follow the method exactly. The data from laboratories using the AOAC method will be included in a collaborative study effort to evaluate the reproducibility of the method to support *Final Action* status. All data submitted by participants regardless of the method is reported in the community tables and graphs below.

Sample Information

Participants received each sample listed in the table below. Before use, participants were instructed to thoroughly mix the contents of each package of ground material. Instructions for preparation of samples from tablets, caplets, and capsules were given in AOAC 2015.11 along with a minimum sample size to use for analysis. The approximate analyte levels were not reported to participants prior to the study. Participants were asked to store the materials at controlled room temperature, 20 °C to 25 °C, and report all results as total chondroitin sulfate on a dry-mass basis in units of μg/g. Values for total chondroitin sulfate in these products were not determined by NIST prior to the study.

| Sample | Quantity and Packaging | Quantity per Package | How to report |
|---|---|---|---|
| Sample A: Chondroitin Caplets | 3 packets | 20 caplets | Prepare 1 sample and report 1 value per packet |
| Sample B: Chondroitin Tablets | 3 packets | 20 tablets | Prepare 1 sample and report 1 value per packet |
| Sample C: Chondroitin Chewables for Dogs | 3 packets | 20 tablets | Prepare 1 sample and report 1 value per packet |
| Sample D: Chondroitin Capsules | 3 packets | 20 caplets | Prepare 1 sample and report 1 value per packet |
| Sample E: Chondroitin Sulfate Sodium | 3 vials | 4 g of powder | Prepare 1 sample and report 1 value per vial |
| Sample F: Chondroitin Sulfate Sodium | 3 vials | 4 g of powder | Prepare 1 sample and report 1 value per vial |
| Sample G: Chondroitin Beverage | 1 bottle | 237 mL | Prepare 3 samples and report 3 values from the single bottle |

Study Results

- Fourteen laboratories enrolled in the exercise and received samples to measure total chondroitin sulfate in seven different dietary supplements. Five laboratories reported results for every sample (36 % participation). A sixth laboratory reported one result for three of the supplements.
- The between-laboratory variability was good for samples A through F (<18 % RSD) and poor for Sample G (82 % RSD).

| Sample ID | Between-Laboratory Variability (RSD) |
|---|---|
| Sample A: Chondroitin Caplets | 15.1 % |
| Sample B: Chondroitin Tablets | 11.7 % |
| Sample C: Chondroitin Chewables for Dogs | 13.6 % |
| Sample D: Chondroitin Capsules | 17.3 % |
| Sample E: Chondroitin Sulfate Sodium | 3.9 % |
| Sample F: Bovine Chondroitin Sulfate | 6.0 % |
| Sample G: Chondroitin Beverage | 82.0 % |

- Most laboratories reported enzymatic hydrolysis as their sample preparation method (83 %). One laboratory reported using acid hydrolysis (17 %) for sample preparation.
- Laboratories reported using AOAC 2015.11 (50 %), the USP Chondroitin Sulfate Sodium method (17 %), LC-absorbance (17 %), or in-house methods (17 %) for determination of total chondroitin sulfate.

Technical Recommendations

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

- The small number of laboratories reporting data does not allow meaningful conclusions to be drawn from performance of specific analytical methods or sample preparation approaches.
- Analysis of chondroitin sulfate can be challenging because of molecular weight variation of chondroitin sulfate polymers, poor UV absorbance, and strong ionic nature.
- Other glycosaminoglycans may be present as impurities or adulterants in chondroitin-containing products. Therefore, analytical methodology must be designed to quantify total chondroitin sulfate in the presence of these glycosaminoglycans.
- All results should be checked closely to avoid calculation errors and to be sure that results are reported in the requested units.
- The between-laboratory variability for most of the samples was very good. With more participating laboratories, AOAC 2015.11 may meet the performance requirements and become a fully validated approach for determination of total chondroitin sulfate in supplements.

**Table 3-1.** NIST data summary table for chondroitin in dietary supplements.

## *National Institute of Standards and Technology*

**DSQAP Exercise O - Natural Products**

| | | **Lab Code:** | **NIST** | **1. Your Results** | | | | **2. Community Results** | | | **3. Target** | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Analyte | Sample | | Units | $x_i$ | $s_i$ | $Z'_{comm}$ | $Z_{NIST}$ | N | x* | s* | $x_{NIST}$ | *U* |
| Total Chondroitin Sulfate | Chondroitin Sample A | | µg/g | | | | | 5 | 362000 | 24000 | | |
| Total Chondroitin Sulfate | Chondroitin Sample B | | µg/g | | | | | 6 | 324000 | 15000 | | |
| Total Chondroitin Sulfate | Chondroitin Sample C | | µg/g | | | | | 6 | 152000 | 8400 | | |
| Total Chondroitin Sulfate | Chondroitin Sample D | | µg/g | | | | | 6 | 299000 | 21000 | | |
| Total Chondroitin Sulfate | Chondroitin Sample E | | µg/g | | | | | 5 | 934000 | 16000 | | |
| Total Chondroitin Sulfate | Chondroitin Sample F | | µg/g | | | | | 5 | 963000 | 25000 | | |
| Total Chondroitin Sulfate | Chondroitin Sample G | | µg/g | | | | | 5 | 1040 | 380 | | |

$x_i$ Mean of reported values

$s_i$ Standard deviation of reported values

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

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

N Number of quantitative values reported

x* Robust mean of reported values

s* Robust standard deviation

$x_{NIST}$ NIST-assessed value

*U* expanded uncertainty about the NIST-assessed value

**Table 3-2.1.** Data summary table for chondroitin in dietary supplements. Individual results are displayed in this table for seven of the laboratories that requested samples (O403 through O423), while community results are shown for all laboratories participating the study. Results for additional laboratories can be found in Table 3-2.2. Data highlighted in red have been flagged as potential outliers (e.g., Grubb and/or Cochran) by the NIST software package.

| | | Chondroitin | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Individual Results - Page 1 of 2 | | | | | | | | Community Results | | | | | |
| | Lab | NIST | O403 | O406 | O409 | O412 | O419 | O420 | O423 | Mean | SD | % RSD | Max | Min | N |
| Sample A: Chondroitin Caplets (µg/g) | A | | | 362000 | | | 400222 | 399310 | | | | | | | |
| | B | | | 362000 | | | 417927 | 385040 | | | | | | | |
| | C | | | 365000 | | | 417316 | 374280 | | | | | | | |
| | Avg | | | 363000 | | | 411822 | 386210 | | 362467 | 54755 | 15.1% | 411822 | 363000 | 4 |
| | SD | | | 1732 | | | 10050 | 12556 | | | | | | | |
| Sample B: Chondroitin Tablets (µg/g) | A | | | 309000 | | | 356471 | 367420 | | | | | | | |
| | B | | | 299000 | | | 363469 | 329471 | | | | | | | |
| | C | | | 305000 | | | 364634 | 310140 | | | | | | | |
| | Avg | | | 304333 | | | 361525 | 335677 | | 324480 | 38015 | 11.7% | 361525 | 428 | 5 |
| | SD | | | 5033 | | | 4415 | 29140 | | | | | | | |
| Sample C: Chondroitin Chewables for Dogs (µg/g) | A | | | 149000 | | | 174313 | 160470 | | | | | | | |
| | B | | | 147000 | | | 179164 | 159530 | | | | | | | |
| | C | | | 154000 | | | 177758 | 159990 | | | | | | | |
| | Avg | | | 150000 | | | 177078 | 159997 | | 152211 | 20721 | 13.6% | 177078 | 219 | 5 |
| | SD | | | 3606 | | | 2496 | 470 | | | | | | | |
| Sample D: Chondroitin Capsules (µg/g) | A | | | 308000 | | | 325160 | 343690 | | | | | | | |
| | B | | | 296000 | | | 338664 | 370020 | | | | | | | |
| | C | | | 295000 | | | 329110 | 337670 | | | | | | | |
| | Avg | | | 299667 | | | 330978 | 350460 | | 299456 | 51861 | 17.3% | 350460 | 381 | 5 |
| | SD | | | 7234 | | | 6943 | 17205 | | | | | | | |
| Sample E: Chondroitin Sulfate Sodium (µg/g) | A | | | 918000 | | | 975519 | 923200 | | | | | | | |
| | B | | | 917000 | | | 989761 | 936030 | | | | | | | |
| | C | | | 915000 | | | 993918 | 944460 | | | | | | | |
| | Avg | | | 916667 | | | 986399 | 934563 | | 933749 | 36269 | 3.9% | 986399 | 916667 | 4 |
| | SD | | | 1528 | | | 9649 | 10706 | | | | | | | |
| Sample F: Bovine Chondroitin Sulfate (µg/g) | A | | | 933000 | | | 993174 | 1026010 | | | | | | | |
| | B | | | 900000 | | | 1079737 | 986190 | | | | | | | |
| | C | | | 929000 | | | 1010293 | 999780 | | | | | | | |
| | Avg | | | 920667 | | | 1027735 | 1003993 | | 962647 | 58222 | 6.0% | 1027735 | 920667 | 4 |
| | SD | | | 18009 | | | 45842 | 20242 | | | | | | | |
| Sample G: Chondroitin Beverage (µg/g) | A | | | 571 | | | 1029 | 980 | | | | | | | |
| | B | | | | | | 1012 | 880 | | | | | | | |
| | C | | | | | | 999 | 810 | | | | | | | |
| | Avg | | | 571 | | | 1013 | 890 | | 1037 | 850 | 82.0% | 1013 | 406 | 4 |
| | SD | | | | | | 15 | 85 | | | | | | | |

**Table 3-2.2.** Data summary table for chondroitin in dietary supplements. Individual results are displayed in this table for seven of the laboratories that requested samples (O425 through O462), while community results are shown for all laboratories participating the study. Results for additional laboratories can be found in Table 3-2.1. Data highlighted in red have been flagged as potential outliers (e.g., Grubb and/or Cochran) by the NIST software package.

| | | Chondroitin | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Individual Results - Page 2 of 2 | | | | | | | | Community Results | | | | | |
| | Lab | NIST | O425 | O431 | O434 | O440 | O449 | O452 | O462 | Mean | SD | % RSD | Max | Min | N |
| Sample A: Chondroitin Caplets (µg/g) | A | | | 283468 | | | 369500 | | | | | | | | |
| | B | | | | | | 370600 | | | | | | | | |
| | C | | | | | | 363400 | | | | | | | | |
| | Avg | | | 283468 | | | 367833 | | | 362467 | 54755 | 15.1% | 411822 | 363000 | 4 |
| | SD | | | | | | 3879 | | | | | | | | |
| Sample B: Chondroitin Tablets (µg/g) | A | | 428 | 343301 | | | 330600 | | | | | | | | |
| | B | | | | | | 335600 | | | | | | | | |
| | C | | | | | | 337200 | | | | | | | | |
| | Avg | | 428 | 343301 | | | 334467 | | | 324480 | 38015 | 11.7% | 361525 | 428 | 5 |
| | SD | | | | | | 3443 | | | | | | | | |
| Sample C: Chondroitin Chewables for Dogs (µg/g) | A | | 218.9 | 155315 | | | 155700 | | | | | | | | |
| | B | | | | | | 144700 | | | | | | | | |
| | C | | | | | | 148600 | | | | | | | | |
| | Avg | | 218.9 | 155315 | | | 149667 | | | 152211 | 20721 | 13.6% | 177078 | 219 | 5 |
| | SD | | | | | | 5577 | | | | | | | | |
| Sample D: Chondroitin Capsules (µg/g) | A | | 381.1 | 277299 | | | 316000 | | | | | | | | |
| | B | | | | | | 306900 | | | | | | | | |
| | C | | | | | | 326200 | | | | | | | | |
| | Avg | | 381.1 | 277299 | | | 316367 | | | 299456 | 51861 | 17.3% | 350460 | 381 | 5 |
| | SD | | | | | | 9655 | | | | | | | | |
| Sample E: Chondroitin Sulfate Sodium (µg/g) | A | | | 904018 | | | 901900 | | | | | | | | |
| | B | | | | | | 964200 | | | | | | | | |
| | C | | | | | | 915200 | | | | | | | | |
| | Avg | | | 904018 | | | 927100 | | | 933749 | 36269 | 3.9% | 986399 | 916667 | 4 |
| | SD | | | | | | 32811 | | | | | | | | |
| Sample F: Bovine Chondroitin Sulfate (µg/g) | A | | | 918740 | | | 961800 | | | | | | | | |
| | B | | | | | | 930100 | | | | | | | | |
| | C | | | | | | 934400 | | | | | | | | |
| | Avg | | | 918740 | | | 942100 | | | 962647 | 58222 | 6.0% | 1027735 | 920667 | 4 |
| | SD | | | | | | 17196 | | | | | | | | |
| Sample G: Chondroitin Beverage (µg/g) | A | | | 2306 | | | 415 | | | | | | | | |
| | B | | | | | | 397 | | | | | | | | |
| | C | | | | | | 405 | | | | | | | | |
| | Avg | | | 2306 | | | 406 | | | 1037 | 850 | 82.0% | 1013 | 406 | 4 |
| | SD | | | | | | 9 | | | | | | | | |

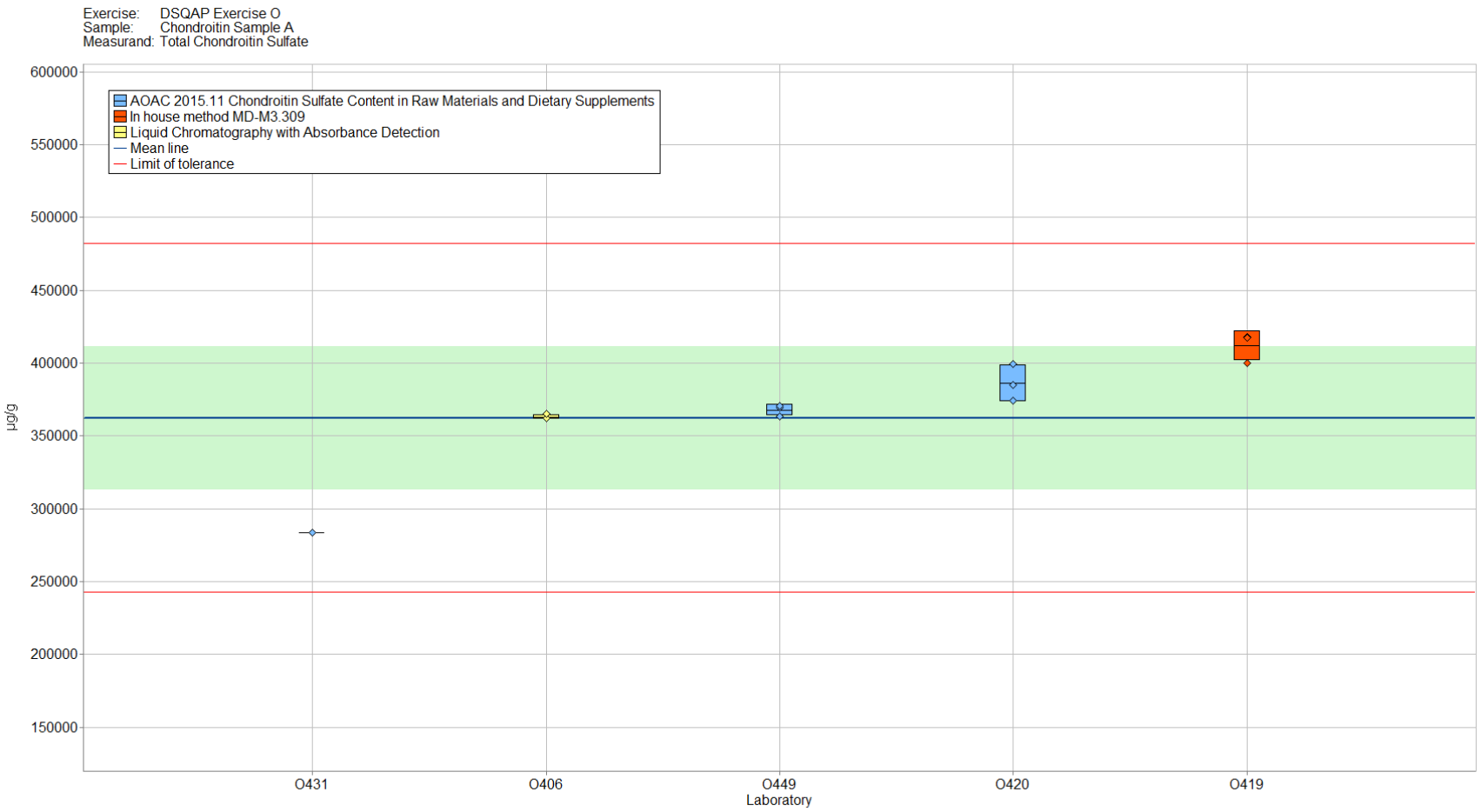


**Figure 3-1.** Total chondroitin sulfate in Chondroitin Caplets (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$. A NIST value has not been determined in this material.

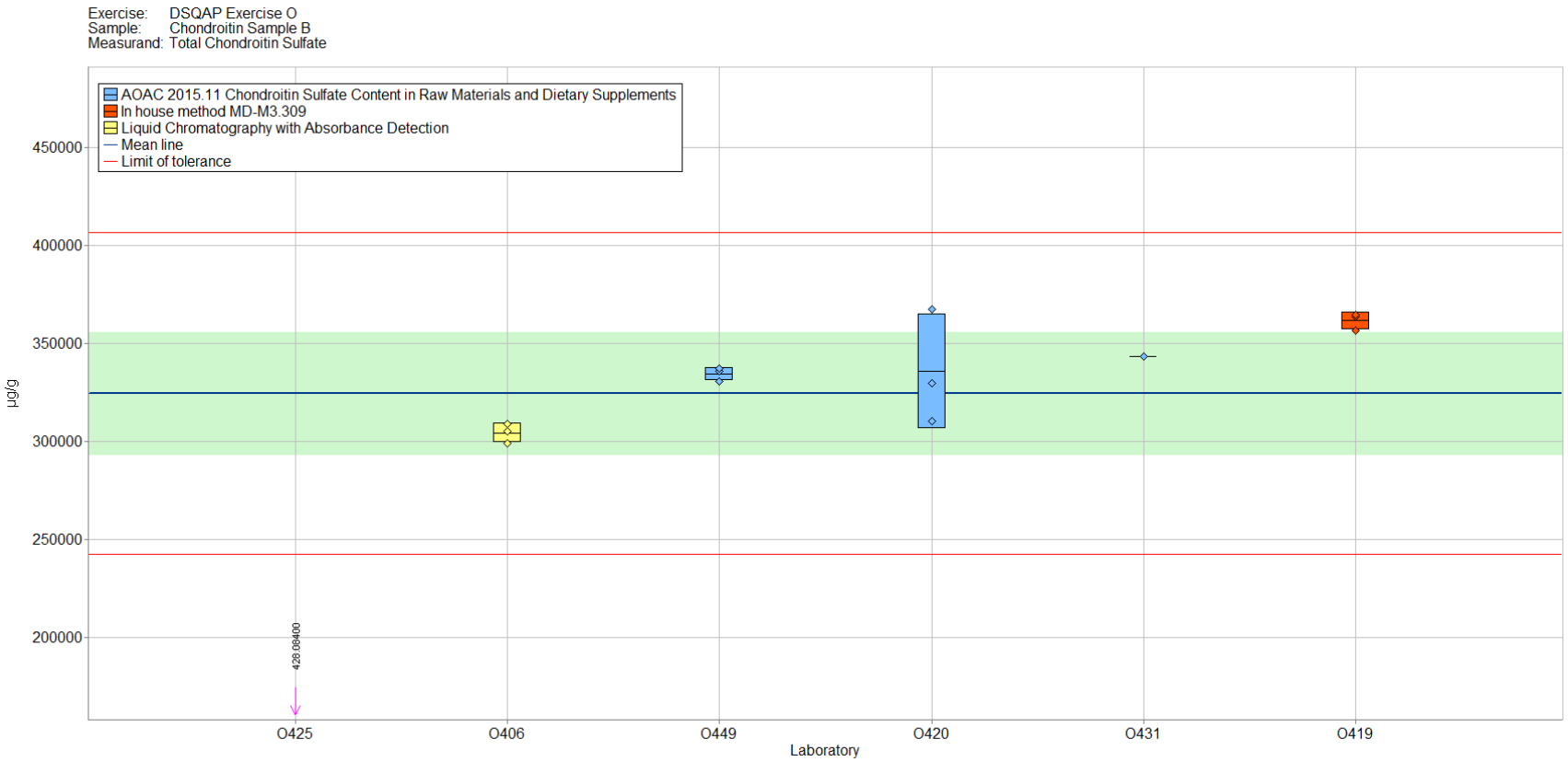


**Figure 3-2.** Total chondroitin sulfate in Chondroitin Tablets (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed.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

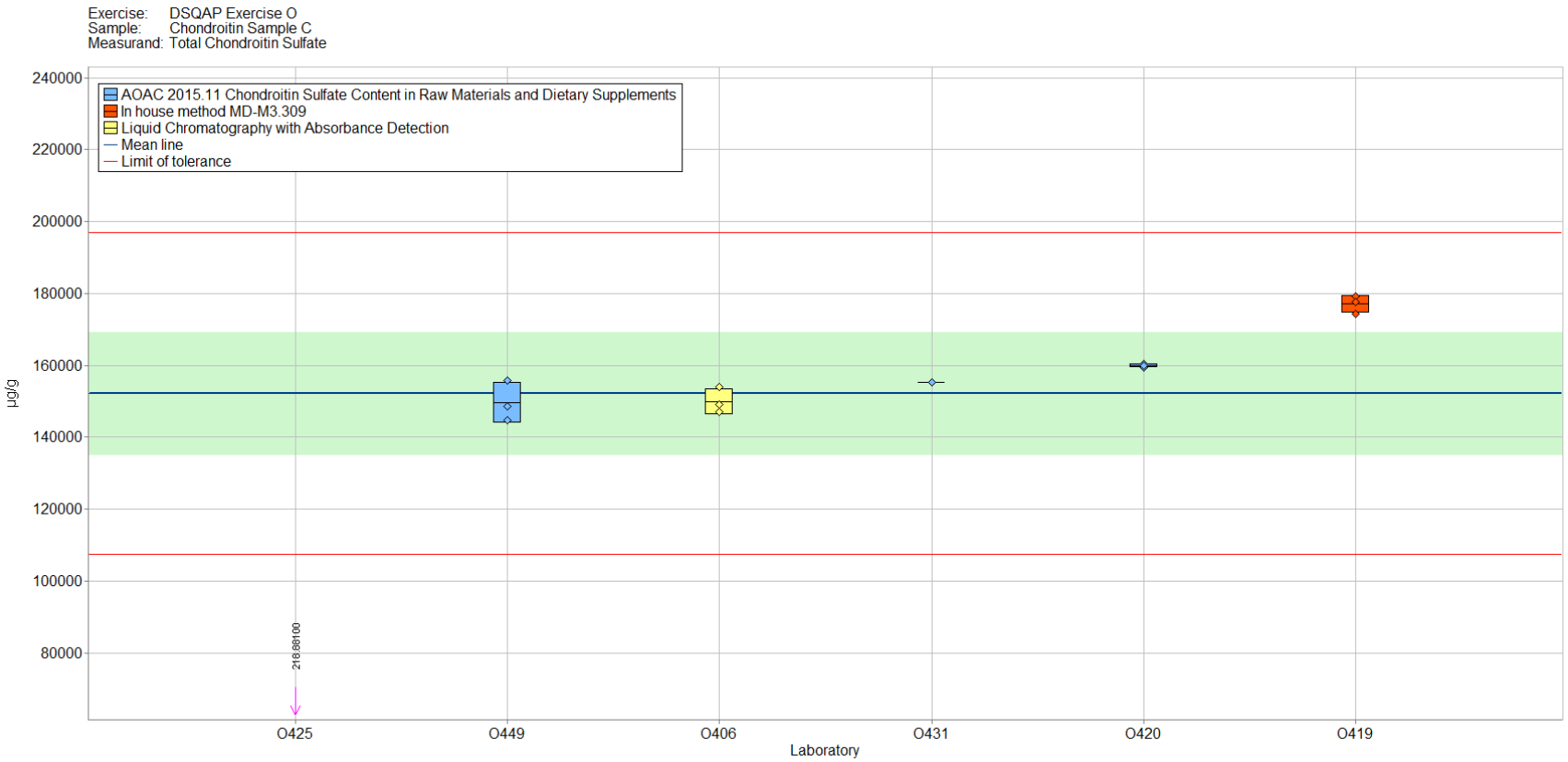


**Figure 3-3.** Total chondroitin sulfate in Chondroitin Chewables for Dogs (data summary view – analytical method In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$. A NIST value has not been determined in this material.

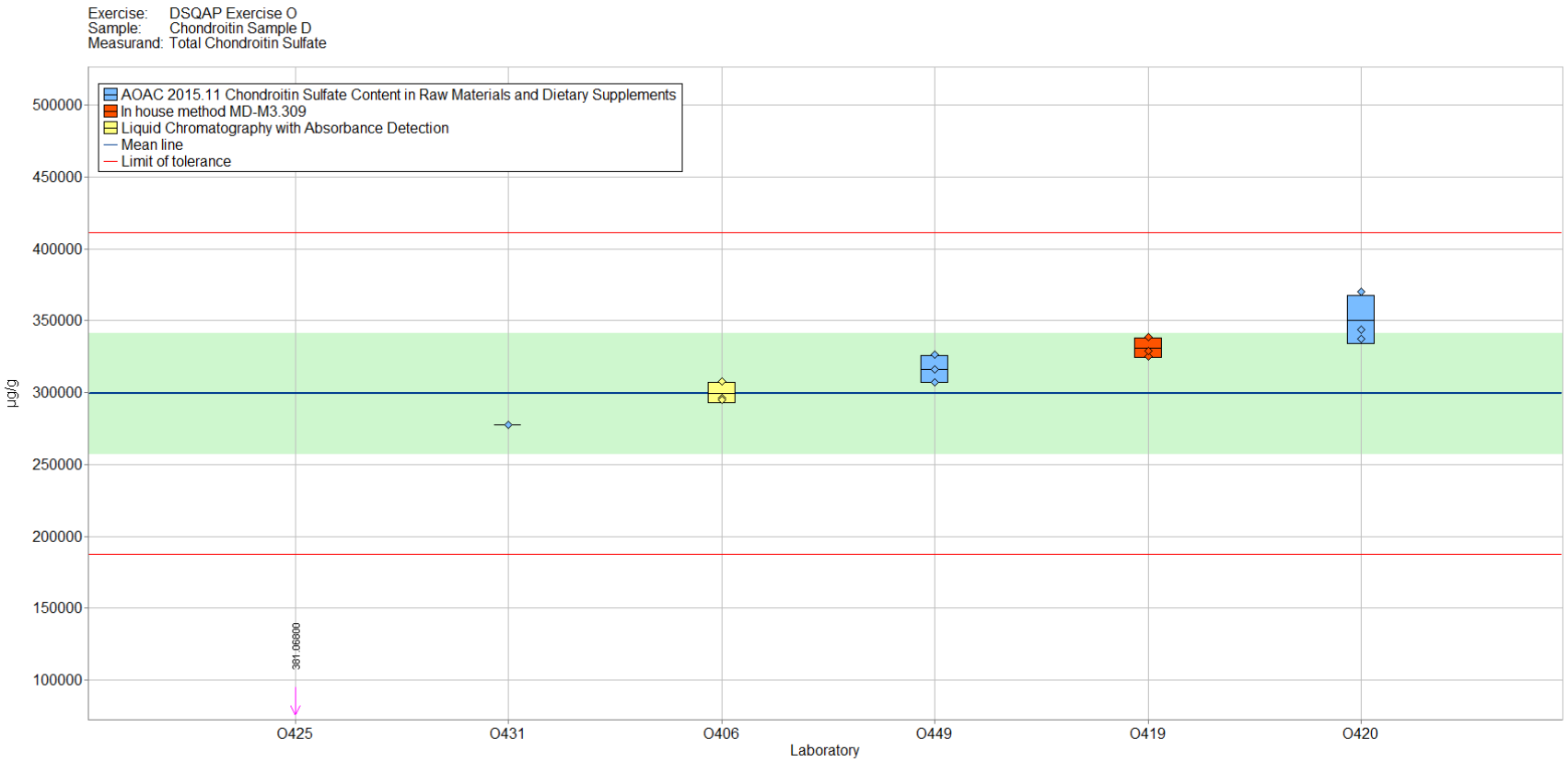


**Figure 3-4.** Total chondroitin sulfate in Chondroitin Capsules (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$. A NIST value has not been determined in this material.

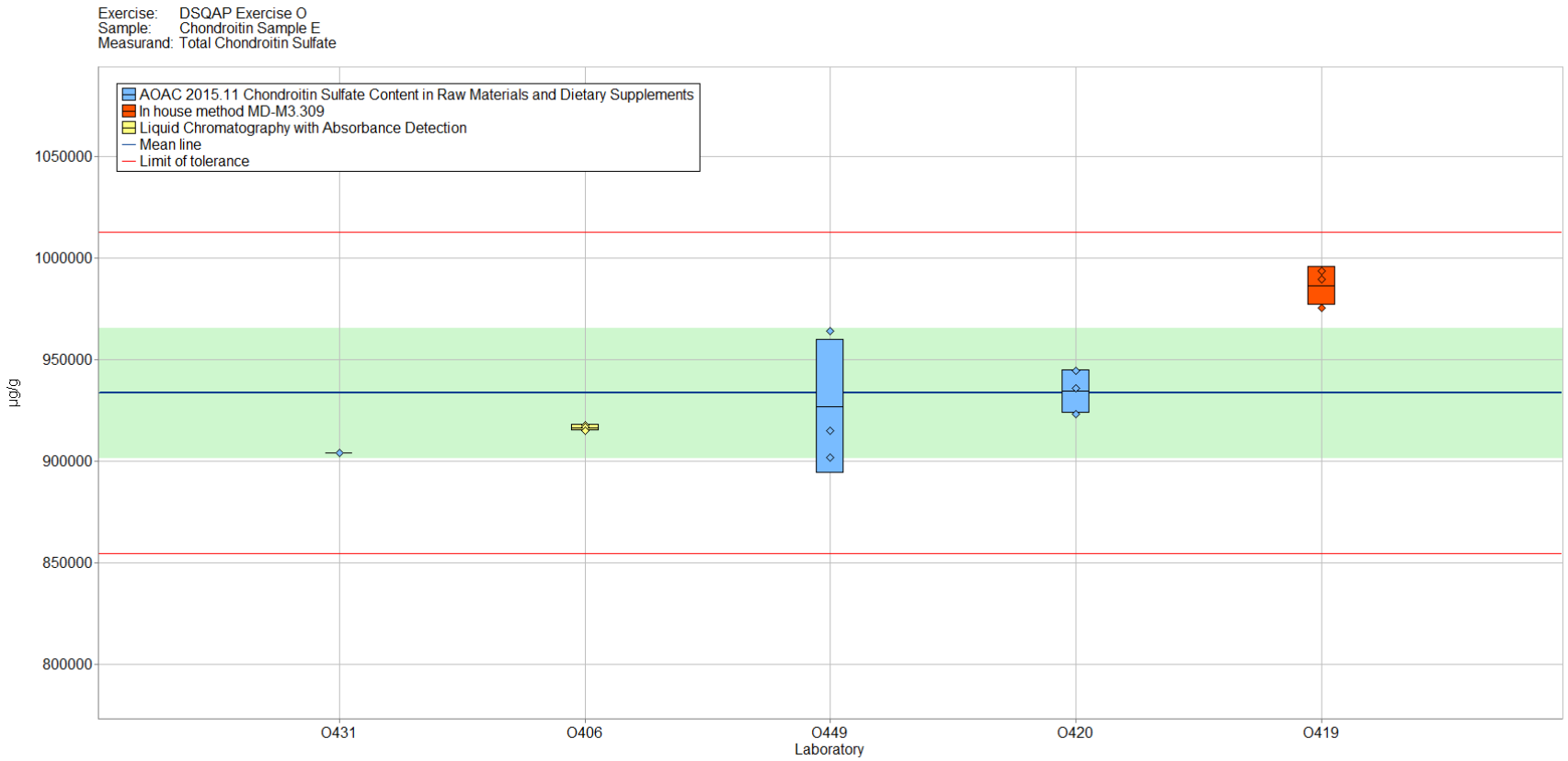


**Figure 3-5.** Total chondroitin sulfate in Chondroitin Sulfate Sodium (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$. A NIST value has not been determined in this material.

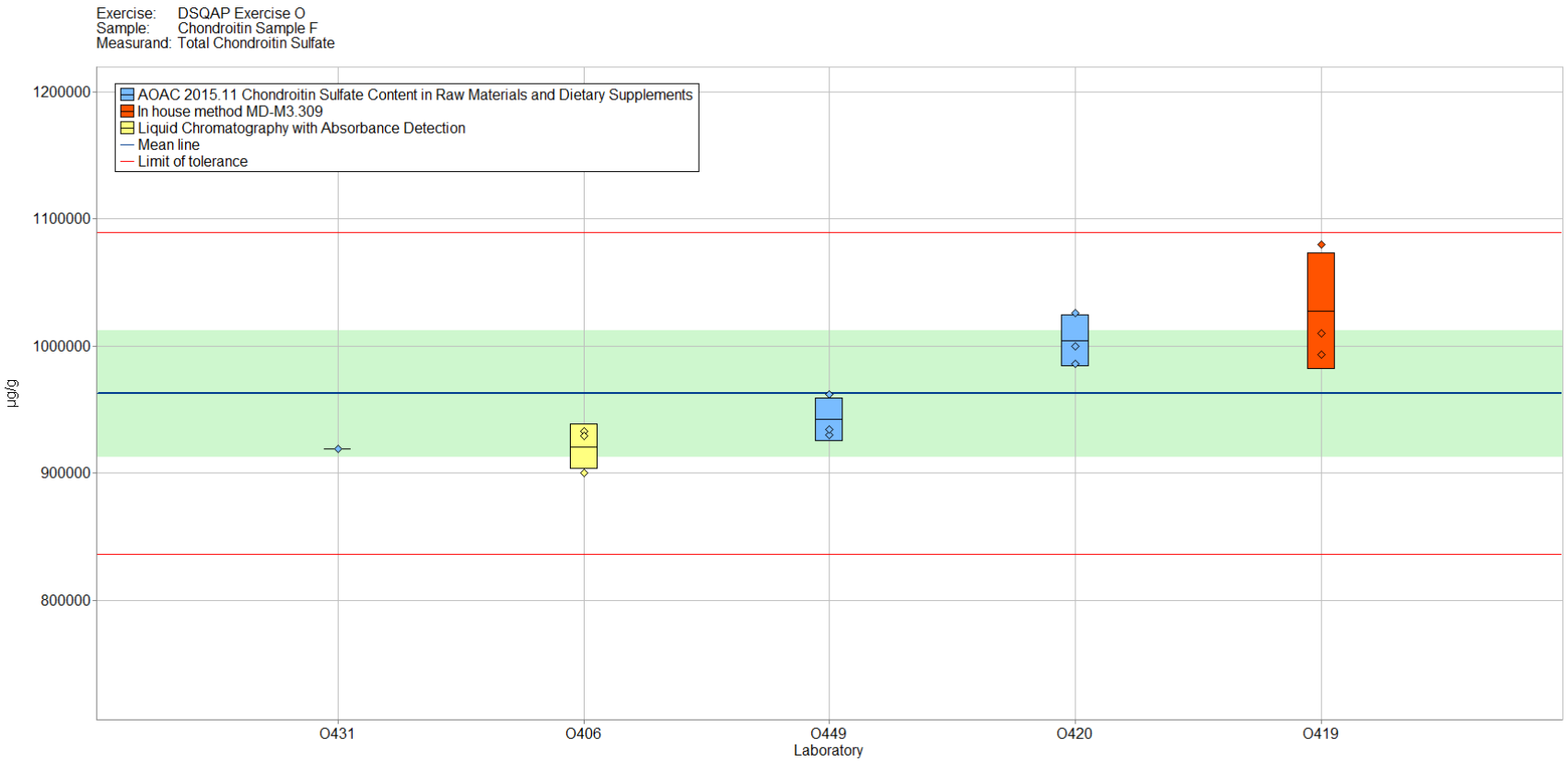


**Figure 3-6.** Total chondroitin sulfate in Bovine Chondroitin Sulfate (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$. A NIST value has not been determined in this material.

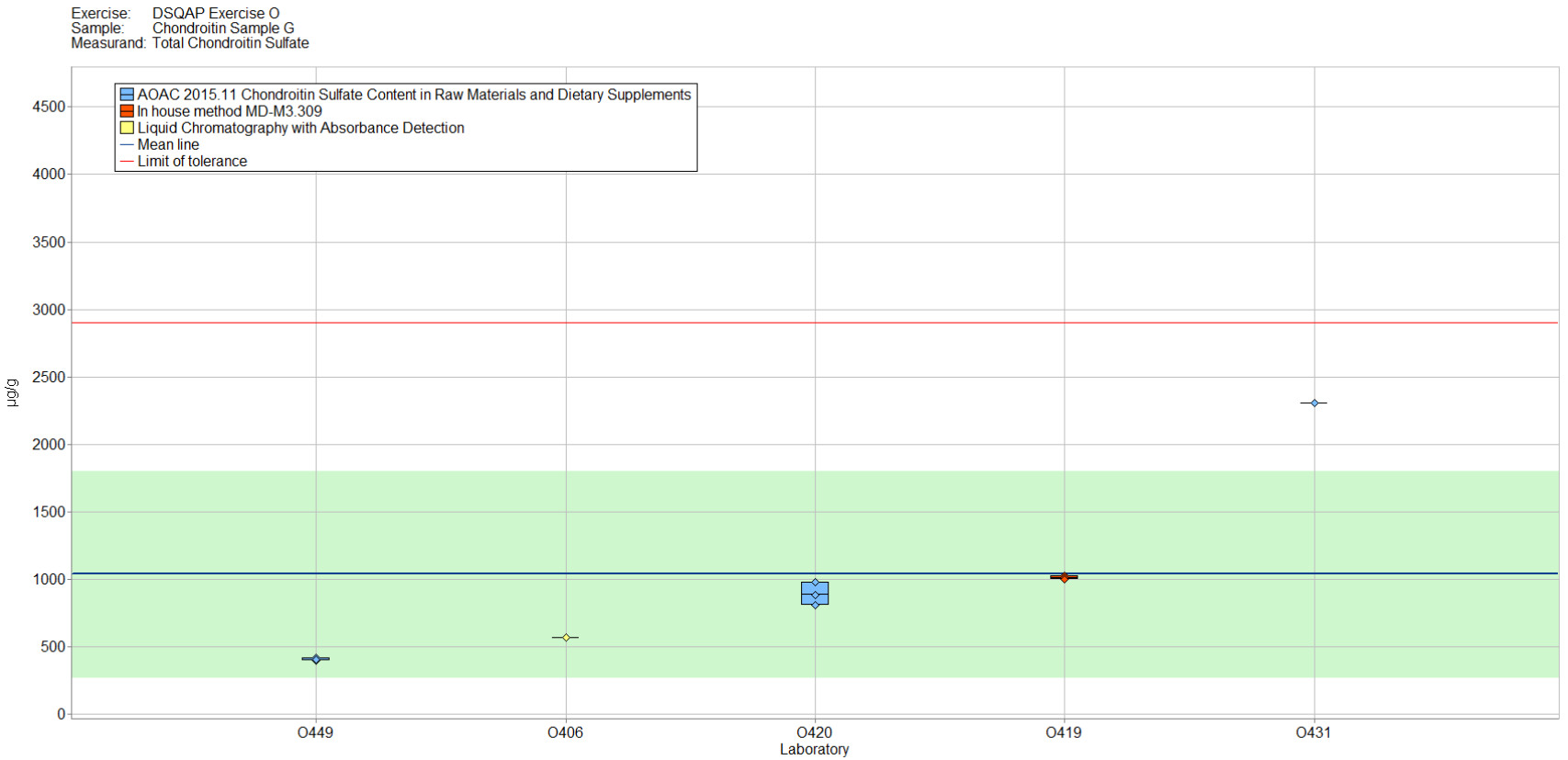


**Figure 3-7.** Total chondroitin sulfate in Chondroitin Beverage (data summary view – analytical method). In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The color of the data point represents the analytical method employed. The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The solid red line represents the upper consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable $Z'_{\text{comm}}$ score, $|Z'_{\text{comm}}| \leq 2$, with the lower range set at zero. A NIST value has not been determined in this material.

## SECTION 4: IDENTIFICATION OF *GINKGO BILOBA* IN BOTANICAL SUPPLEMENTS

Study Overview

In this study, participants were provided with ground *Ginkgo biloba* leaf and extract materials at various levels of adulteration. Participants were asked to use their usual in-house methods of analysis to determine authenticity of test samples in order to compare the performance of all reported methods. A secondary goal of this study was to help the community understand the effectiveness of DNA sequencing techniques for botanical ingredient identification. The data gathered from this exercise will be used in collaboration with the American Herbal Products Association (AHPA) to establish resources and provide recommendations to help effective development and advance this emerging technology.

Sample Information

Participants were provided with two sample sets, Samples A and Samples B, each containing 16 sample packets. Samples A contained *Ginkgo biloba* plant materials and Samples B contained *Ginkgo biloba* extract materials. Each packet contained a minimum of 3 g of powdered *Ginkgo biloba* material with up to 15 % (by weight) of *Sophora japonica* extract (see table below). The material was ground, homogenized, and heat-sealed inside 4 mil polyethylene bags, which were then sealed inside aluminized plastic bags. Before use, participants were instructed to thoroughly mix the contents of each packet. Participants were asked to store the material at controlled room temperature, 20 °C to 25 °C. The approximate levels of adulteration and material source were not reported to participants prior to the study.

| | | Percent *Sophora* Fruit Extract | | | |
|---|---|---|---|---|---|
| | *Ginkgo* Source | 0% | 3% | 7% | 15% |
| *Ginkgo* Samples A | *Ginkgo biloba* leaves | A9 | A3 | A16 | A4 & A12 |
| | *Ginkgo biloba* leaves (steam treated) | A5 | A14 | A7 & A13 | A8 |
| | *Ginkgo biloba* stem | A15 | A2 & A10 | A1 | A11 |
| | SRM 3246 *Ginkgo biloba leaves* | A6 | | | |
| *Ginkgo* Samples B | Aqueous *Ginkgo* extract | B10 | B5 | B13 | B7 |
| | Ethanol:Water *Ginkgo* extract 1 | B3 | | | |
| | Ethanol:Water *Ginkgo* extract 2 | B8 | B12 & B16 | B1 | B9 |
| | Acetone:Water *Ginkgo* extract 1 | B6 | B14 | B11 | B4 & B15 |
| | Acetone:Water *Ginkgo* extract 2 | B2 | | | |

Study Results

*Participation and Methods*

- Thirty-six laboratories enrolled in this exercise and received samples. Twenty-two laboratories reported results (61 % participation). Six laboratories reported results for multiple methods.
- Sixteen laboratories reported using chromatography as one of their analytical methods (57 % of total data sets). Eight laboratories reported using genomic methods (28 %), and four reported using microscopy (14 %).

*Ginkgo Samples A*

Correctly identifying the presence of *Gingko biloba* in plant materials **(Table 4-1)**:

- Of the eight laboratories reporting the use of genomic methods, seven (88 %) were able to correctly identify the presence of *Ginkgo biloba* in plant materials, including stems and one laboratory (12 %) reported inconclusive results for all plant materials. The laboratory reported inconclusive results stating that their method was not robust and could not be applied to *Ginkgo*.
- Of the sixteen laboratories reporting chromatography methods, 14 to 16 laboratories (88 % to 100 %) were able to correctly identify the presence of *Ginkgo biloba* in leaf materials. One laboratory reported inconclusive results. One laboratory reported that *Ginkgo biloba* was not present in a leaf sample.
- Of the sixteen laboratories reporting chromatography methods, four to five (25 % to 31 %) were able to correctly identify the presence of *Ginkgo biloba* in stem materials.
  - Six laboratories (38 %) reported that no *Ginkgo biloba* was present in any samples containing *Ginkgo* stem.
  - Five laboratories (31 %) reported inconclusive results or a combination of inconclusive results and that no *Ginkgo biloba* was present for samples containing *Ginkgo* stem.
  - For the sample containing no *Sophora*, more laboratories reported that no *Ginkgo* was present than when some *Sophora* had been added to the sample.
- No laboratory reporting the use of microscopy methods was able to identify the presence of *Ginkgo biloba* in all samples.
  - One of the four laboratories (25 %) identified the presence of *Ginkgo biloba* in all but one sample, which was reported as inconclusive.
  - The remaining three laboratories (75 %) reported a combination of positive and inconclusive results for the samples.

Correctly identifying *Gingko biloba* leaf or stem as the source in plant materials **(Table 4-2)**:

- All eight laboratories reporting the use of genomic methods reported inconclusive results or did not report results for plant part.
- Three laboratories (19 %) using chromatography methods correctly identified the plant part in all leaf and stem samples. Three laboratories (19 %) correctly identified the plant part in all leaf samples. Seven laboratories (44 %) reported a combination of correct and inconclusive results for the plant samples, while three laboratories (19 %) reported incorrect plant parts for some samples.
- One laboratory using microscopy (25 %) correctly identified the plant part in all leaf and stem samples, and one laboratory (25 %) correctly identified the plant part in a majority of the leaf

and stem samples. Two laboratories (50 %) were not able to consistently identify the plant part in the leaf and stem samples.

*Ginkgo Samples B*

Correctly identifying the presence of *Gingko biloba* in extract materials **(Table 4-1)**:

- Of the eight laboratories reporting the use of genomic methods, four (50 %) reported inconclusive results for all *Ginkgo* extract samples. Remaining laboratories reported a combination of positive identifications and inconclusive results for the extract samples.
- Of the sixteen laboratories reporting chromatography methods, nine (56 %) correctly identified *Ginkgo biloba* in all of the extract samples.
  - Four laboratories (25 %) reported inconclusive results for some of the *Ginkgo* extract samples.
  - Three laboratories (19 %) reported that no *Ginkgo biloba* was present in the extract samples.
  - No laboratories reported results for microscopy evaluation of extract samples.

Correctly identifying *Gingko biloba* leaf as the source in extract materials **(Table 4-2)**:

- All eight laboratories reporting the use of genomic methods reported inconclusive results or did not report results for plant part.
- Three laboratories (19 %) using chromatography methods correctly identified the plant part in all extract samples. Ten laboratories (63 %) reported a combination of correct and inconclusive results for the extract samples, while three laboratories (19 %) reported incorrect plant parts for one or more samples.
- All four laboratories using microscopy reported inconclusive or did not report results for extract samples.

*Ginkgo Adulterants*

Correctly identifying *Gingko biloba* adulterants **(Table 4-3)**:

- For genomic methods, two of the eight laboratories (25 %) reported the presence of other species.
  - One laboratory reported adulteration for nearly every sample, regardless of adulteration level.
  - One laboratory reported the presence of unexpected species primarily for the samples containing *Ginkgo* stem.
- For chromatographic methods, seven of the 16 laboratories (44 %) did not report adulteration for any of the samples. Remaining laboratories reported adulteration levels consistent with the in-house adulteration levels for most of the samples.
- For microscopy methods, two of the four laboratories (50 %) reported adulteration in all plant samples, and one laboratory also reported adulteration of all extract samples. Two laboratories (50 %) correctly identified the level of adulteration in most plant samples. Three laboratories (75 %) did not report adulteration in any extract samples.

Technical Recommendations

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

- No single method was able to correctly identify the presence of *Ginkgo biloba*, the plant part, and the level of adulteration in every sample. The laboratories that were most successful in this study utilized multiple fit-for-purpose methods.
  - A macroscopic investigation of samples can yield valuable information, such as an easily identifiable texture or color difference (**Figures 4-1 and 4-2**). Microscopic investigation can also be useful to identify plant parts or presence of unexpected substances.
  - Following macroscopic and microscopic evaluation, a combination of genomic and chromatographic methods is recommended.
    - Genomic methods can be used to confirm the presence of the proper species, provided that a sufficient quantity of DNA is available for testing.
    - Some of the genomic methods found species in addition to *Ginkgo biloba* and *Sophora japonica*, emphasizing the importance of reporting and introduces the question, if *Hypericum perforatum* DNA is reported, does that make the material adulterated?
    - Genomic methods could not be used to identify plant parts, and most could not identify the *Sophora japonica* extract.
    - Chromatographic methods can be used to confirm consistency of the chemical profile, which often corresponds to the plant part. The ratios of peaks or bands corresponding to marker compounds, as well as the relative intensity of unexpected peaks, can be used to identify and quantify the presence of adulteration.
- In future QAP authentication/identification studies, more specific questions will be asked about testing methods and the responses will be used to pinpoint strengths and weaknesses of each approach.
- In future studies, laboratories will be given specific instructions on whether to test for authenticity/identity or adulteration.

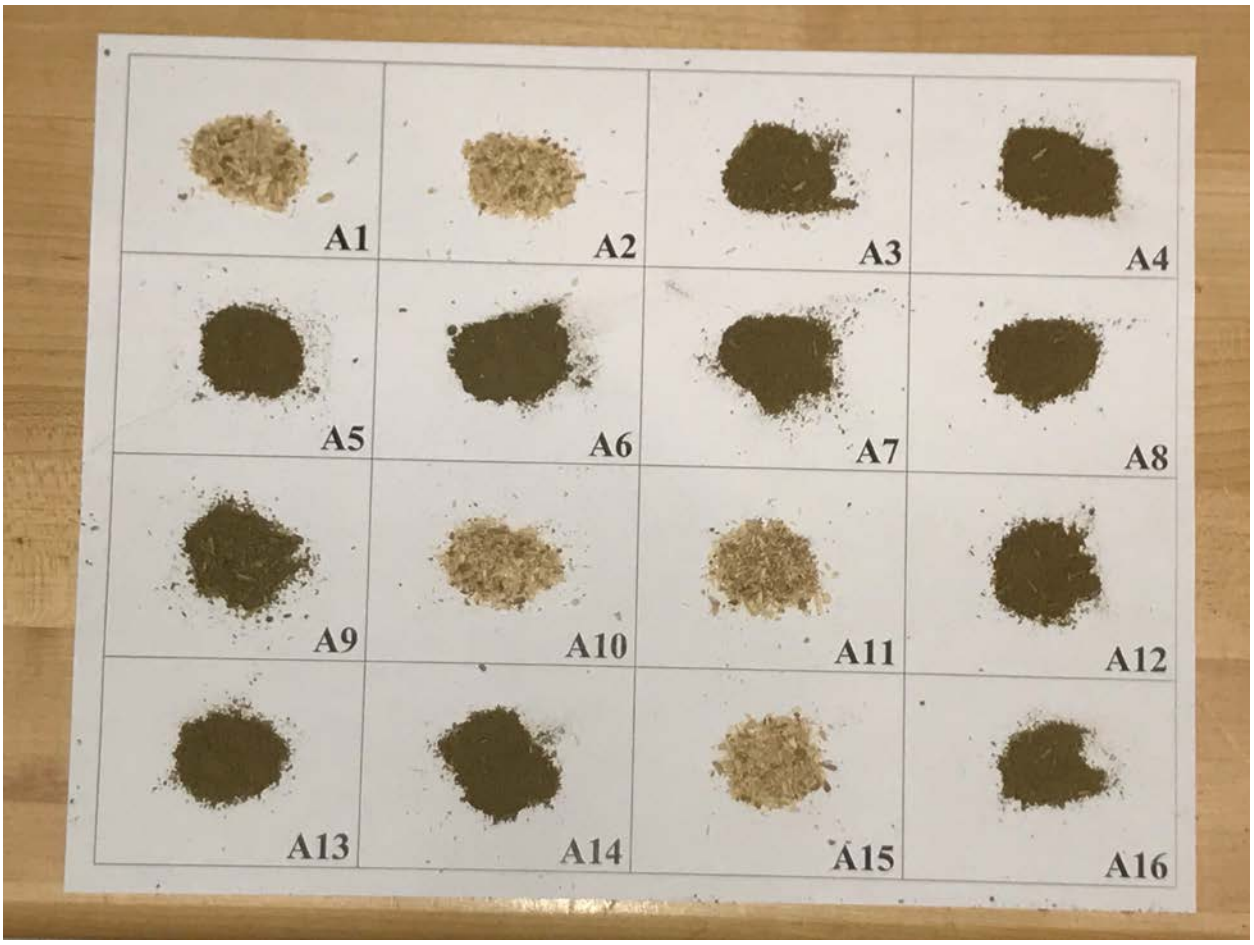


**Figure 4-1.** Macroscopic investigation of the *Ginkgo biloba* plant samples (Samples A).

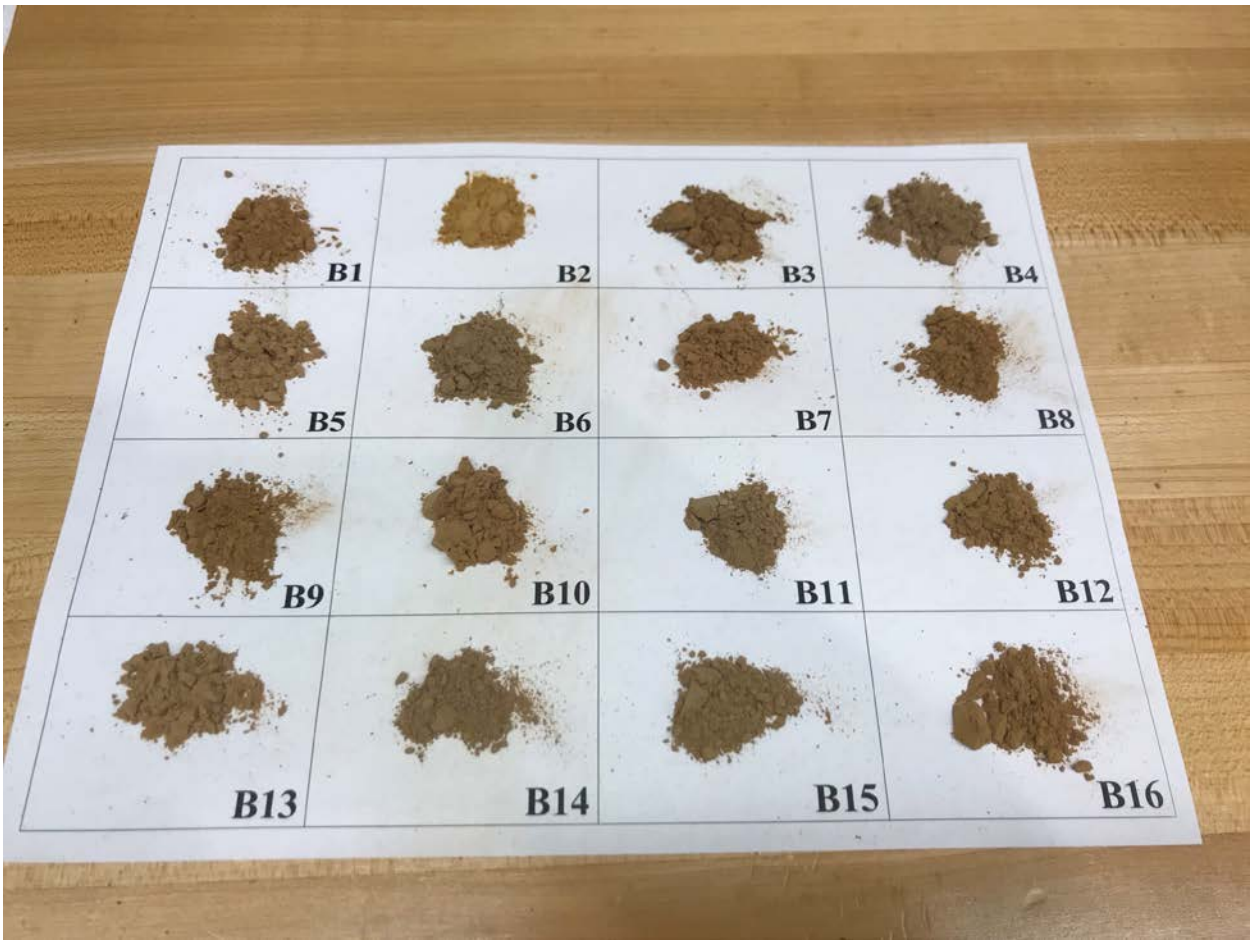


**Figure 4-2.** Macroscopic investigation of the *Ginkgo biloba* extract samples (Samples B).

**Table 4-1.1.** Data summary table for identifying presence of *Ginkgo biloba* in botanical supplements by lab code by answering whether *Ginkgo biloba* is present in this material.

**Is *Ginkgo biloba* present in this material?**
**(arranged by lab code)**

Y Yes | N No | I Inconclusive | NR Not Reported | C Chromatography | G Genomic | M Microscopy

| | | Percent Sophora | O401 | O402 | O404 | | O406 | O407 | | O416 | O419 | O420 | O429 | O425 | O432 | O433 | | O438 | | O439 | O444 | O449 | O450 | O451 | | O452 | | | O453 | O455 | O462 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | **Ginkgo Source** | **Fruit Extract** | **C** | **G** | **G** | **C** | **C** | **C** | **M** | **C** | **C** | **C** | **C** | **C** | **G** | **C** | **M** | **C** | **M** | **G** | **G** | **C** | **G** | **G** | **C** | **C** | **C** | **M** | **G** | **C** | **C** |
| A6 | SRM 3246 *Ginkgo biloba* leaves | 0 | Y | Y | Y | Y | Y | Y | I | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | I | Y | Y | NR | I | N | Y | Y | Y |
| A9 | *Ginkgo biloba* leaves untreated | 0 | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | I | Y | Y | Y | Y | Y | I | Y | Y | NR | Y | Y | Y | Y | Y |
| A3 | *Ginkgo biloba* leaves untreated | 3 | Y | Y | Y | Y | I | Y | Y | Y | Y | Y | Y | Y | Y | Y | I | Y | Y | Y | Y | Y | I | Y | Y | NR | Y | Y | Y | Y | Y |
| A16 | *Ginkgo biloba* leaves untreated | 7 | Y | Y | Y | Y | I | Y | Y | Y | Y | N | Y | I | Y | Y | I | Y | Y | Y | Y | Y | I | Y | Y | NR | Y | Y | Y | Y | Y |
| A4 | *Ginkgo biloba* leaves untreated | 15 | Y | Y | Y | Y | I | Y | Y | Y | Y | Y | Y | I | Y | Y | Y | Y | Y | Y | Y | Y | I | Y | Y | NR | Y | Y | Y | Y | Y |
| A12 | *Ginkgo biloba* leaves untreated | 15 | Y | Y | Y | Y | I | I | I | Y | Y | Y | Y | I | Y | Y | I | I | Y | Y | Y | Y | I | Y | Y | NR | Y | Y | Y | Y | Y |
| A5 | *Ginkgo biloba* leaves steam treated | 0 | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | I | Y | Y | NR | Y | Y | Y | Y | Y |
| A14 | *Ginkgo biloba* leaves steam treated | 3 | Y | Y | Y | Y | I | Y | Y | Y | Y | Y | Y | I | Y | Y | I | Y | Y | Y | Y | Y | I | Y | Y | NR | Y | Y | Y | Y | Y |
| A7 | *Ginkgo biloba* leaves steam treated | 7 | Y | Y | Y | Y | I | I | Y | Y | Y | Y | Y | I | Y | Y | I | Y | Y | Y | Y | Y | I | Y | Y | NR | Y | Y | Y | Y | Y |
| A13 | *Ginkgo biloba* leaves steam treated | 7 | Y | Y | Y | Y | I | I | I | Y | Y | Y | Y | I | Y | Y | I | Y | Y | Y | Y | Y | I | Y | Y | NR | Y | Y | Y | Y | Y |
| A11 | *Ginkgo biloba* stem | 15 | Y | Y | Y | Y | I | I | I | N | N | I | N | I | Y | Y | I | I | I | Y | Y | N | I | Y | Y | NR | N | I | Y | N | N |
| A15 | *Ginkgo biloba* stem | 0 | Y | Y | Y | Y | I | I | I | N | N | N | N | N | Y | I | I | Y | Y | Y | Y | N | I | Y | Y | NR | N | I | Y | N | N |
| A2 | *Ginkgo biloba* stem | 3 | Y | Y | Y | Y | I | I | I | N | Y | I | N | I | Y | Y | Y | I | Y | Y | Y | N | I | Y | Y | NR | N | I | Y | N | N |
| A10 | *Ginkgo biloba* stem | 3 | Y | Y | Y | Y | I | I | I | N | N | I | N | I | Y | Y | I | I | Y | Y | Y | N | I | Y | Y | NR | N | I | Y | N | N |
| A1 | *Ginkgo biloba* stem | 7 | Y | Y | Y | Y | I | I | I | N | Y | I | N | I | Y | Y | Y | I | Y | Y | Y | N | I | Y | Y | NR | N | I | Y | N | N |
| A8 | *Ginkgo biloba* leaves steam treated | 15 | Y | Y | Y | Y | I | I | Y | Y | Y | Y | Y | I | Y | Y | I | Y | Y | Y | Y | Y | I | Y | Y | NR | Y | Y | Y | Y | Y |
| B10 | Aqueous ginkgo extract | 0 | Y | I | I | Y | I | Y | NR | Y | Y | Y | Y | Y | Y | Y | I | Y | NR | Y | I | Y | I | I | Y | Y | NR | NR | I | Y | Y |
| B5 | Aqueous ginkgo extract | 3 | Y | I | I | Y | I | Y | NR | Y | Y | Y | Y | Y | Y | Y | I | Y | NR | Y | I | Y | I | I | Y | Y | NR | NR | Y | Y | Y |
| B13 | Aqueous ginkgo extract | 7 | Y | I | I | Y | I | I | NR | Y | Y | Y | Y | Y | Y | Y | I | I | NR | I | I | Y | I | I | Y | Y | NR | NR | Y | Y | Y |
| B7 | Aqueous ginkgo extract | 15 | Y | I | I | Y | I | I | NR | Y | Y | Y | Y | Y | Y | Y | I | Y | NR | Y | Y | Y | I | I | Y | Y | NR | NR | I | Y | Y |
| B3 | Ethanol:Water extract 1 | 0 | Y | I | I | Y | Y | Y | NR | Y | Y | Y | Y | I | Y | Y | I | I | NR | Y | Y | N | I | I | Y | Y | NR | NR | Y | N | Y |
| B8 | Ethanol:Water extract 2 | 0 | Y | I | I | Y | Y | Y | NR | Y | Y | Y | Y | I | I | Y | I | I | NR | I | Y | N | I | I | Y | Y | NR | NR | Y | N | Y |
| B12 | Ethanol:Water extract 2 | 3 | Y | I | I | Y | Y | Y | NR | Y | N | Y | Y | I | Y | Y | I | I | NR | Y | Y | N | I | I | Y | Y | NR | NR | I | N | Y |
| B16 | Ethanol:Water extract 2 | 3 | Y | I | I | Y | Y | Y | NR | Y | N | Y | Y | I | Y | Y | I | I | NR | Y | Y | N | I | I | Y | Y | NR | NR | Y | N | Y |
| B1 | Ethanol:Water extract 2 | 7 | Y | I | I | Y | Y | Y | NR | Y | Y | Y | Y | I | Y | Y | I | I | NR | Y | I | N | I | I | Y | Y | NR | NR | Y | N | Y |
| B9 | Ethanol:Water extract 2 | 15 | Y | I | I | Y | Y | Y | NR | Y | N | Y | Y | I | Y | Y | I | I | NR | Y | Y | N | I | I | Y | Y | NR | NR | Y | N | Y |
| B2 | Acetone:Water extract 2 | 0 | Y | I | I | Y | N | Y | NR | Y | Y | Y | Y | I | I | Y | I | I | NR | Y | I | Y | I | I | Y | Y | NR | NR | Y | Y | Y |
| B14 | Acetone:Water extract | 3 | Y | I | I | Y | Y | Y | NR | Y | N | Y | Y | I | Y | Y | I | I | NR | I | Y | Y | I | I | Y | Y | NR | NR | I | Y | Y |
| B6 | Acetone:Water extract | 7 | Y | I | I | Y | Y | Y | NR | Y | Y | Y | Y | I | Y | Y | I | I | NR | Y | I | Y | I | I | Y | Y | NR | NR | I | Y | Y |
| B11 | Acetone:Water extract | 7 | Y | I | I | Y | Y | Y | NR | Y | N | Y | Y | I | Y | Y | I | I | NR | Y | Y | Y | I | I | Y | Y | NR | NR | I | Y | Y |
| B4 | Acetone:Water extract | 15 | Y | I | I | Y | Y | Y | NR | Y | Y | Y | Y | I | Y | Y | I | I | NR | Y | Y | Y | I | I | Y | Y | NR | NR | I | Y | Y |
| B15 | Acetone:Water extract | 15 | Y | I | I | Y | Y | Y | NR | Y | N | Y | Y | I | Y | Y | I | I | NR | Y | Y | Y | I | I | Y | Y | NR | NR | Y | Y | Y |

**Table 4-1.2.** Data summary table for identifying presence of *Ginkgo biloba* in botanical supplements by technique by answering whether *Ginkgo biloba* is present in this material.

**Is *Ginkgo biloba* present in this material?**
**(arranged by technique)**

Y Yes | N No | I Inconclusive | NR Not Reported | C Chromatography | G Genomic | M Microscopy

| | Ginkgo Source | Percent Sophora Fruit Extract | O402 | O404 | O432 | O439 | O444 | O450 | O451 | O453 | O401 | O404 | O406 | O407 | O416 | O419 | O420 | O425 | O429 | O433 | O438 | O449 | O451 | O452 | O452 | O455 | O462 | O407 | O433 | O438 | O452 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | G | G | G | G | G | G | G | G | C | C | C | C | C | C | C | C | C | C | C | C | C | C | C | C | C | M | M | M | M |
| A6 | SRM 3246 *Ginkgo biloba* leaves | 0 | Y | Y | Y | Y | Y | I | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | NR | I | Y | Y | I | Y | Y | N |
| A9 | *Ginkgo biloba* leaves untreated | 0 | Y | Y | Y | Y | Y | I | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | NR | Y | Y | Y | Y | I | Y | Y |
| A3 | *Ginkgo biloba* leaves untreated | 3 | Y | Y | Y | Y | Y | I | Y | Y | Y | Y | I | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | NR | Y | Y | Y | Y | I | Y | Y |
| A16 | *Ginkgo biloba* leaves untreated | 7 | Y | Y | Y | Y | Y | I | Y | Y | Y | Y | I | Y | Y | Y | N | I | Y | Y | Y | Y | Y | NR | Y | Y | Y | Y | I | Y | Y |
| A4 | *Ginkgo biloba* leaves untreated | 15 | Y | Y | Y | Y | Y | I | Y | Y | Y | Y | I | Y | Y | Y | Y | I | Y | Y | Y | Y | Y | NR | Y | Y | Y | Y | Y | Y | Y |
| A12 | *Ginkgo biloba* leaves untreated | 15 | Y | Y | Y | Y | Y | I | Y | Y | Y | Y | I | I | Y | Y | Y | I | Y | Y | I | Y | Y | NR | Y | Y | Y | I | I | Y | Y |
| A5 | *Ginkgo biloba* leaves steam treated | 0 | Y | Y | Y | Y | Y | I | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | NR | Y | Y | Y | Y | Y | Y | Y |
| A14 | *Ginkgo biloba* leaves steam treated | 3 | Y | Y | Y | Y | Y | I | Y | Y | Y | Y | I | Y | Y | Y | Y | I | Y | Y | Y | Y | Y | NR | Y | Y | Y | Y | I | Y | Y |
| A7 | *Ginkgo biloba* leaves steam treated | 7 | Y | Y | Y | Y | Y | I | Y | Y | Y | Y | I | I | Y | Y | Y | I | Y | Y | Y | Y | Y | NR | Y | Y | Y | Y | I | Y | Y |
| A13 | *Ginkgo biloba* leaves steam treated | 7 | Y | Y | Y | Y | Y | I | Y | Y | Y | Y | I | I | Y | Y | Y | I | Y | Y | Y | Y | Y | NR | Y | Y | Y | I | I | Y | Y |
| A11 | *Ginkgo biloba* stem | 15 | Y | Y | Y | Y | Y | I | Y | Y | Y | Y | I | I | N | N | I | I | N | Y | I | N | Y | NR | N | N | N | I | I | I | I |
| A15 | *Ginkgo biloba* stem | 0 | Y | Y | Y | Y | Y | I | Y | Y | Y | Y | I | I | N | N | N | N | N | I | Y | N | Y | NR | N | N | N | I | I | Y | I |
| A2 | *Ginkgo biloba* stem | 3 | Y | Y | Y | Y | Y | I | Y | Y | Y | Y | I | I | N | Y | I | I | N | Y | I | N | Y | NR | N | N | N | I | Y | Y | I |
| A10 | *Ginkgo biloba* stem | 3 | Y | Y | Y | Y | Y | I | Y | Y | Y | Y | I | I | N | N | I | I | N | Y | I | N | Y | NR | N | N | N | I | I | Y | I |
| A1 | *Ginkgo biloba* stem | 7 | Y | Y | Y | Y | Y | I | Y | Y | Y | Y | I | I | N | Y | I | I | N | Y | I | N | Y | NR | N | N | N | I | Y | Y | I |
| A8 | *Ginkgo biloba* leaves steam treated | 15 | Y | Y | Y | Y | Y | I | Y | Y | Y | Y | I | I | Y | Y | Y | I | Y | Y | Y | Y | Y | NR | Y | Y | Y | Y | I | Y | Y |
| B10 | Aqueous ginkgo extract | 0 | I | I | Y | Y | I | I | I | I | Y | Y | I | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | NR | Y | Y | NR | I | NR | NR |
| B5 | Aqueous ginkgo extract | 3 | I | I | Y | Y | I | I | I | Y | Y | Y | I | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | NR | Y | Y | NR | I | NR | NR |
| B13 | Aqueous ginkgo extract | 7 | I | I | Y | I | I | I | I | Y | Y | Y | I | I | Y | Y | Y | Y | Y | Y | I | Y | Y | Y | NR | Y | Y | NR | I | NR | NR |
| B7 | Aqueous ginkgo extract | 15 | I | I | Y | Y | Y | I | I | I | Y | Y | I | I | Y | Y | Y | Y | Y | Y | Y | Y | Y | Y | NR | Y | Y | NR | I | NR | NR |
| B3 | Ethanol:Water extract 1 | 0 | I | I | Y | Y | Y | I | I | Y | Y | Y | Y | Y | Y | Y | Y | I | Y | Y | I | N | Y | Y | NR | N | Y | NR | I | NR | NR |
| B8 | Ethanol:Water extract 2 | 0 | I | I | I | I | Y | I | I | Y | Y | Y | Y | Y | Y | Y | Y | I | Y | Y | I | N | Y | Y | NR | N | Y | NR | I | NR | NR |
| B12 | Ethanol:Water extract 2 | 3 | I | I | Y | Y | Y | I | I | I | Y | Y | Y | Y | Y | N | Y | I | Y | Y | I | N | Y | Y | NR | N | Y | NR | I | NR | NR |
| B16 | Ethanol:Water extract 2 | 3 | I | I | Y | Y | Y | I | I | Y | Y | Y | Y | Y | Y | N | Y | I | Y | Y | I | N | Y | Y | NR | N | Y | NR | I | NR | NR |
| B1 | Ethanol:Water extract 2 | 7 | I | I | Y | Y | I | I | I | Y | Y | Y | Y | Y | Y | Y | Y | I | Y | Y | I | N | Y | Y | NR | N | Y | NR | I | NR | NR |
| B9 | Ethanol:Water extract 2 | 15 | I | I | Y | Y | Y | I | I | Y | Y | Y | Y | Y | Y | N | Y | I | Y | Y | I | N | Y | Y | NR | N | Y | NR | I | NR | NR |
| B2 | Acetone:Water extract 2 | 0 | I | I | I | Y | I | I | I | Y | Y | Y | N | Y | Y | Y | Y | I | Y | Y | I | Y | Y | Y | NR | Y | Y | NR | I | NR | NR |
| B14 | Acetone:Water extract | 3 | I | I | Y | I | Y | I | I | I | Y | Y | Y | Y | Y | N | Y | I | Y | Y | I | Y | Y | Y | NR | Y | Y | NR | I | NR | NR |
| B6 | Acetone:Water extract | 7 | I | I | Y | Y | I | I | I | I | Y | Y | Y | Y | Y | Y | Y | I | Y | Y | I | Y | Y | Y | NR | Y | Y | NR | I | NR | NR |
| B11 | Acetone:Water extract | 7 | I | I | Y | Y | Y | I | I | I | Y | Y | Y | Y | Y | N | Y | I | Y | Y | I | Y | Y | Y | NR | Y | Y | NR | I | NR | NR |
| B4 | Acetone:Water extract | 15 | I | I | Y | Y | Y | I | I | I | Y | Y | Y | Y | Y | Y | Y | I | Y | Y | I | Y | Y | Y | NR | Y | Y | NR | I | NR | NR |
| B15 | Acetone:Water extract | 15 | I | I | Y | Y | Y | I | I | Y | Y | Y | Y | Y | Y | N | Y | I | Y | Y | I | Y | Y | Y | NR | Y | Y | NR | I | NR | NR |

**Table 4-2.1.** Data summary table for identifying *Ginkgo biloba* plant part in botanical supplements by lab code by answering whether the source of the sample can be classified into one of the following groups.

**Can the source of the sample be classified into one of the following groups?** L Leaf; B Bark; S Stem; F Fruit; I Inconclusive; NR Not Reported; C Chromatography; M Microscopy; G Genomic

**(arranged by lab code)**

| | Ginkgo Source | Percent Sophora Fruit Extract | O401 | O402 | O404 | | O406 | O407 | | O416 | O419 | O420 | O425 | O429 | O432 | O433 | | O438 | | O439 | O444 | O449 | O450 | O451 | | O452 | | | O453 | O455 | O462 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | C | G | G | C | C | C | M | C | C | C | C | C | G | C | M | C | M | G | G | C | G | G | C | C | C | M | G | C | C |
| A6 | SRM 3246 *Ginkgo biloba* leaves | 0 | L | I | I | L | I | L | I | L | L | L | L | L | I | L | L | L | B | I | I | I | NR | I | L | NR | L | L | NR | L | I |
| A9 | *Ginkgo biloba* leaves untreated | 0 | L | I | I | L | I | L | L | L | L | L | L | L | I | L | I | L | L | I | I | L | NR | I | L | NR | L | L | NR | L | L |
| A3 | *Ginkgo biloba* leaves untreated | 3 | L | I | I | L | I | L | L | I | L | I | L | L | I | L | I | L | L | I | I | L | NR | I | L | NR | L | L | NR | S | L |
| A16 | *Ginkgo biloba* leaves untreated | 7 | L | I | I | L | I | L | L | I | L | I | I | L | I | L | I | L | L | I | I | I | NR | I | L | NR | L | L | NR | S | L |
| A4 | *Ginkgo biloba* leaves untreated | 15 | L | I | I | L | I | L | L | I | L | I | I | L | I | L | L | L | L | I | I | I | NR | I | L | NR | L | L | NR | S | L |
| A12 | *Ginkgo biloba* leaves untreated | 15 | L | I | I | L | I | I | I | I | L | I | I | L | I | L | I | I | L | I | I | B | NR | I | L | NR | L | L | NR | S | L |
| A5 | *Ginkgo biloba* leaves steam treated | 0 | L | I | I | L | I | L | L | L | L | L | L | L | I | L | L | L | L | I | I | I | NR | I | L | NR | L | L | NR | L | I |
| A14 | *Ginkgo biloba* leaves steam treated | 3 | L | I | I | L | I | L | L | I | L | I | I | L | I | L | I | L | L | I | I | L | NR | I | L | NR | L | L | NR | S | L |
| A7 | *Ginkgo biloba* leaves steam treated | 7 | L | I | I | L | I | I | I | I | L | I | I | L | I | L | I | L | L | I | I | I | NR | I | L | NR | L | L | NR | S | L |
| A13 | *Ginkgo biloba* leaves steam treated | 7 | L | I | I | L | I | I | I | I | L | I | I | L | I | L | I | L | L | I | I | I | NR | I | L | NR | L | L | NR | S | L |
| A11 | *Ginkgo biloba* stem | 15 | I | I | I | S | I | I | I | I | I | I | I | I | I | L | I | I | S | I | I | B | NR | I | S | NR | S | S | NR | S | I |
| A15 | *Ginkgo biloba* stem | 0 | I | I | I | S | I | I | I | I | I | I | I | I | I | I | I | B | B | I | I | I | NR | I | S | NR | S | S | NR | I | I |
| A2 | *Ginkgo biloba* stem | 3 | I | I | I | S | I | I | I | I | L | I | I | I | I | L | L | I | S | I | I | B | NR | I | S | NR | S | S | NR | I | I |
| A10 | *Ginkgo biloba* stem | 3 | I | I | I | S | I | I | I | I | I | I | I | I | I | L | I | I | B | I | I | B | NR | I | S | NR | S | S | NR | I | I |
| A1 | *Ginkgo biloba* stem | 7 | I | I | I | S | I | I | I | I | L | I | I | I | I | L | L | I | S | I | I | B | NR | I | S | NR | S | S | NR | I | I |
| A8 | *Ginkgo biloba* leaves steam treated | 15 | L | I | I | L | I | I | I | I | L | I | I | L | I | L | I | L | L | I | I | I | NR | I | L | NR | L | L | NR | S | L |
| B10 | Aqueous ginkgo extract | 0 | L | I | NR | L | I | L | NR | L | L | L | L | L | I | L | I | L | NR | I | I | S | NR | I | I | I | NR | NR | NR | L | I |
| B5 | Aqueous ginkgo extract | 3 | L | I | NR | L | I | L | NR | I | L | I | L | L | I | L | I | L | NR | I | I | S | NR | I | I | I | NR | NR | NR | L | I |
| B13 | Aqueous ginkgo extract | 7 | L | I | NR | L | I | I | NR | I | L | I | L | L | I | L | I | I | NR | I | I | S | NR | I | I | I | NR | NR | NR | S | I |
| B7 | Aqueous ginkgo extract | 15 | L | I | NR | L | I | I | NR | I | L | I | L | L | I | L | I | L | NR | I | I | S | NR | I | I | I | NR | NR | NR | S | I |
| B3 | Ethanol:Water extract 1 | 0 | L | I | NR | L | I | L | NR | I | L | L | I | L | I | L | I | I | NR | I | I | S | NR | I | L | I | NR | NR | NR | I | I |
| B8 | Ethanol:Water extract 2 | 0 | L | I | NR | L | I | L | NR | L | L | L | I | L | I | L | I | I | NR | I | I | S | NR | I | L | I | NR | NR | NR | I | I |
| B12 | Ethanol:Water extract 2 | 3 | L | I | NR | L | I | L | NR | I | I | I | I | L | I | L | I | I | NR | I | I | S | NR | I | L | I | NR | NR | NR | I | I |
| B16 | Ethanol:Water extract 2 | 3 | L | I | NR | L | I | L | NR | L | I | I | I | L | I | L | I | I | NR | I | I | S | NR | I | L | I | NR | NR | NR | I | I |
| B1 | Ethanol:Water extract 2 | 7 | L | I | NR | L | I | L | NR | I | L | I | I | L | I | L | I | I | NR | I | I | S | NR | I | L | I | NR | NR | NR | I | I |
| B9 | Ethanol:Water extract 2 | 15 | L | I | NR | L | I | L | NR | I | I | I | I | L | I | F | I | I | NR | I | I | S | NR | I | L | I | NR | NR | NR | I | I |
| B2 | Acetone:Water extract 2 | 0 | L | I | NR | L | I | L | NR | L | L | L | I | L | I | L | I | I | NR | I | I | S | NR | I | I | I | NR | NR | NR | S | I |
| B14 | Acetone:Water extract | 3 | L | I | NR | L | I | L | NR | I | I | I | I | L | I | L | I | I | NR | I | I | S | NR | I | L | I | NR | NR | NR | S | L |
| B6 | Acetone:Water extract | 7 | L | I | NR | L | I | L | NR | I | L | L | I | L | I | L | I | I | NR | I | I | S | NR | I | L | I | NR | NR | NR | L | L |
| B11 | Acetone:Water extract | 7 | L | I | NR | L | I | L | NR | I | I | I | I | L | I | L | I | I | NR | I | I | S | NR | I | L | I | NR | NR | NR | S | L |
| B4 | Acetone:Water extract | 15 | L | I | NR | L | I | L | NR | I | L | I | I | L | I | L | I | I | NR | I | I | S | NR | I | L | I | NR | NR | NR | S | L |
| B15 | Acetone:Water extract | 15 | L | I | NR | L | I | L | NR | I | I | I | I | L | I | L | I | I | NR | I | I | S | NR | I | L | I | NR | NR | NR | S | L |

**Table 4-2.2.** Data summary table for identifying *Ginkgo biloba* plant part in botanical supplements by technique by answering whether the source of the sample can be classified into one of the following groups.

**Can the source of the sample be classified into one of the following groups?** L Leaf; B Bark; S Stem; F Fruit; I Inconclusive; NR Not Reported; C Chromatography; M Microscopy; G Genomic

**(arranged by technique)**

| | Ginkgo Source | Percent Sophora Fruit Extract | O402 | O404 | O432 | O439 | O444 | O450 | O451 | O453 | O401 | O404 | O406 | O407 | O416 | O419 | O420 | O425 | O429 | O433 | O438 | O449 | O451 | O452 | O452 | O455 | O462 | O407 | O433 | O438 | O452 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | G | G | G | G | G | G | G | G | C | C | C | C | C | C | C | C | C | C | C | C | C | C | C | C | C | M | M | M | M |
| A6 | SRM 3246 *Ginkgo biloba* leaves | 0 | I | I | I | I | I | NR | I | NR | L | L | I | L | L | L | L | L | L | L | L | I | L | NR | L | L | I | I | L | B | L |
| A9 | *Ginkgo biloba* leaves untreated | 0 | I | I | I | I | I | NR | I | NR | L | L | I | L | L | L | L | L | L | L | L | L | L | NR | L | L | L | L | I | L | L |
| A3 | *Ginkgo biloba* leaves untreated | 3 | I | I | I | I | I | NR | I | NR | L | L | I | L | I | L | I | L | L | L | L | L | L | NR | L | S | L | L | I | L | L |
| A16 | *Ginkgo biloba* leaves untreated | 7 | I | I | I | I | I | NR | I | NR | L | L | I | L | I | L | I | I | L | L | L | I | L | NR | L | S | L | L | I | L | L |
| A4 | *Ginkgo biloba* leaves untreated | 15 | I | I | I | I | I | NR | I | NR | L | L | I | L | I | L | I | I | L | L | L | I | L | NR | L | S | L | L | L | L | L |
| A12 | *Ginkgo biloba* leaves untreated | 15 | I | I | I | I | I | NR | I | NR | L | L | I | I | I | L | I | I | L | L | I | B | L | NR | L | S | L | I | I | L | L |
| A5 | *Ginkgo biloba* leaves steam treated | 0 | I | I | I | I | I | NR | I | NR | L | L | I | L | L | L | L | L | L | L | L | I | L | NR | L | L | I | L | L | L | L |
| A14 | *Ginkgo biloba* leaves steam treated | 3 | I | I | I | I | I | NR | I | NR | L | L | I | L | I | L | I | I | L | L | L | L | L | NR | L | S | L | L | I | L | L |
| A7 | *Ginkgo biloba* leaves steam treated | 7 | I | I | I | I | I | NR | I | NR | L | L | I | I | I | L | I | I | L | L | L | I | L | NR | L | S | L | I | I | L | L |
| A13 | *Ginkgo biloba* leaves steam treated | 7 | I | I | I | I | I | NR | I | NR | L | L | I | I | I | L | I | I | L | L | L | I | L | NR | L | S | L | I | I | L | L |
| A11 | *Ginkgo biloba* stem | 15 | I | I | I | I | I | NR | I | NR | I | S | I | I | I | I | I | I | I | L | I | B | S | NR | S | S | I | I | I | S | S |
| A15 | *Ginkgo biloba* stem | 0 | I | I | I | I | I | NR | I | NR | I | S | I | I | I | I | I | I | I | I | B | I | S | NR | S | I | I | I | I | B | S |
| A2 | *Ginkgo biloba* stem | 3 | I | I | I | I | I | NR | I | NR | I | S | I | I | I | L | I | I | I | L | I | B | S | NR | S | I | I | I | L | S | S |
| A10 | *Ginkgo biloba* stem | 3 | I | I | I | I | I | NR | I | NR | I | S | I | I | I | I | I | I | I | L | I | B | S | NR | S | I | I | I | I | B | S |
| A1 | *Ginkgo biloba* stem | 7 | I | I | I | I | I | NR | I | NR | I | S | I | I | I | L | I | I | I | L | I | B | S | NR | S | I | I | I | L | S | S |
| A8 | *Ginkgo biloba* leaves steam treated | 15 | I | I | I | I | I | NR | I | NR | L | L | I | I | I | L | I | I | L | L | L | I | L | NR | L | S | L | I | I | L | L |
| B10 | Aqueous ginkgo extract | 0 | I | NR | I | I | I | NR | I | NR | L | L | I | L | L | L | L | L | L | L | L | S | I | I | NR | L | I | NR | I | NR | NR |
| B5 | Aqueous ginkgo extract | 3 | I | NR | I | I | I | NR | I | NR | L | L | I | L | I | L | I | L | L | L | L | S | I | I | NR | L | I | NR | I | NR | NR |
| B13 | Aqueous ginkgo extract | 7 | I | NR | I | I | I | NR | I | NR | L | L | I | I | I | L | I | L | L | L | I | S | I | I | NR | S | I | NR | I | NR | NR |
| B7 | Aqueous ginkgo extract | 15 | I | NR | I | I | I | NR | I | NR | L | L | I | I | I | L | I | L | L | L | L | S | I | I | NR | S | I | NR | I | NR | NR |
| B3 | Ethanol:Water extract 1 | 0 | I | NR | I | I | I | NR | I | NR | L | L | I | L | I | L | L | I | L | L | I | S | L | I | NR | I | I | NR | I | NR | NR |
| B8 | Ethanol:Water extract 2 | 0 | I | NR | I | I | I | NR | I | NR | L | L | I | L | L | L | L | I | L | L | I | S | L | I | NR | I | I | NR | I | NR | NR |
| B12 | Ethanol:Water extract 2 | 3 | I | NR | I | I | I | NR | I | NR | L | L | I | L | I | I | I | I | L | L | I | S | L | I | NR | I | I | NR | I | NR | NR |
| B16 | Ethanol:Water extract 2 | 3 | I | NR | I | I | I | NR | I | NR | L | L | I | L | L | I | I | I | L | L | I | S | L | I | NR | I | I | NR | I | NR | NR |
| B1 | Ethanol:Water extract 2 | 7 | I | NR | I | I | I | NR | I | NR | L | L | I | L | I | L | I | I | L | L | I | S | L | I | NR | I | I | NR | I | NR | NR |
| B9 | Ethanol:Water extract 2 | 15 | I | NR | I | I | I | NR | I | NR | L | L | I | L | I | I | I | I | L | F | I | S | L | I | NR | I | I | NR | I | NR | NR |
| B2 | Acetone:Water extract 2 | 0 | I | NR | I | I | I | NR | I | NR | L | L | I | L | L | L | L | I | L | L | I | S | I | I | NR | S | I | NR | I | NR | NR |
| B14 | Acetone:Water extract | 3 | I | NR | I | I | I | NR | I | NR | L | L | I | L | I | I | I | I | L | L | I | S | L | I | NR | S | L | NR | I | NR | NR |
| B6 | Acetone:Water extract | 7 | I | NR | I | I | I | NR | I | NR | L | L | I | L | I | L | L | I | L | L | I | S | L | I | NR | L | L | NR | I | NR | NR |
| B11 | Acetone:Water extract | 7 | I | NR | I | I | I | NR | I | NR | L | L | I | L | I | I | I | I | L | L | I | S | L | I | NR | S | L | NR | I | NR | NR |
| B4 | Acetone:Water extract | 15 | I | NR | I | I | I | NR | I | NR | L | L | I | L | I | L | I | I | L | L | I | S | L | I | NR | S | L | NR | I | NR | NR |
| B15 | Acetone:Water extract | 15 | I | NR | I | I | I | NR | I | NR | L | L | I | L | I | I | I | I | L | L | I | S | L | I | NR | S | L | NR | I | NR | NR |

**Table 4-3.1.** Data summary table for identifying *Ginkgo biloba* adulterants in botanical supplements by lab code.

**What else was found in the Sample?**
**(arranged by lab code)**

Not Reported | Description of sample/cells without definitive statement of adulteration | Identification of unexpected species | *Sophora japonica* was not reported as an adulterant | Results consistent with in-house adulteration | C Chromatography | G Genomic | M Microscopy

| | Ginkgo Source | Percent Sophora Fruit Extract | O401 | O402 | O404 | | O406 | O407 | O407 | O416 | O419 | O420 | O425 | O429 | O432 | O433 | O433 | O438 | O438 | O439 | O444 | O449 | O450 | O451 | O451 | O452 | O452 | O452 | O453 | O455 | O462 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | C | G | G | C | C | C | M | C | C | C | C | C | G | C | M | C | M | G | G | C | G | G | C | C | C | M | G | C | C |
| A6 | SRM 3246 *Ginkgo biloba* leaves | 0 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| A9 | *Ginkgo biloba* leaves untreated | 0 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| A3 | *Ginkgo biloba* leaves untreated | 3 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| A16 | *Ginkgo biloba* leaves untreated | 7 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| A4 | *Ginkgo biloba* leaves untreated | 15 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| A12 | *Ginkgo biloba* leaves untreated | 15 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| A5 | *Ginkgo biloba* leaves steam treated | 0 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| A14 | *Ginkgo biloba* leaves steam treated | 3 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| A7 | *Ginkgo biloba* leaves steam treated | 7 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| A13 | *Ginkgo biloba* leaves steam treated | 7 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| A11 | *Ginkgo biloba* stem | 15 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| A15 | *Ginkgo biloba* stem | 0 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| A2 | *Ginkgo biloba* stem | 3 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| A10 | *Ginkgo biloba* stem | 3 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| A1 | *Ginkgo biloba* stem | 7 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| A8 | *Ginkgo biloba* leaves steam treated | 15 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| B10 | Aqueous ginkgo extract | 0 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| B5 | Aqueous ginkgo extract | 3 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| B13 | Aqueous ginkgo extract | 7 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| B7 | Aqueous ginkgo extract | 15 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| B3 | Ethanol:Water extract 1 | 0 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| B8 | Ethanol:Water extract 2 | 0 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| B12 | Ethanol:Water extract 2 | 3 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| B16 | Ethanol:Water extract 2 | 3 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| B1 | Ethanol:Water extract 2 | 7 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| B9 | Ethanol:Water extract 2 | 15 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| B2 | Acetone:Water extract 2 | 0 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| B14 | Acetone:Water extract | 3 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| B6 | Acetone:Water extract | 7 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| B11 | Acetone:Water extract | 7 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| B4 | Acetone:Water extract | 15 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| B15 | Acetone:Water extract | 15 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |

**Table 4-3.2.** Data summary table for identifying *Ginkgo biloba* adulterants in botanical supplements by technique.

**What else was found in the Sample?**
**(arranged by technique)**

Not Reported

Description of sample/cells without definitive statement of adulteration

Identification of unexpected species

*Sophora japonica* was not reported as an adulterant.

Results consistent with in-house adulteration

**C** Chromatography **G** Genomic **M** Microscopy

| | Ginkgo Source | Percent Sophora Fruit Extract | O402 G | O404 G | O432 G | O439 G | O444 G | O450 G | O451 G | O453 G | O401 C | O404 C | O406 C | O407 C | O416 C | O419 C | O420 C | O425 C | O429 C | O433 C | O438 C | O449 C | O451 C | O452 C | O452 C | O455 C | O462 C | O407 M | O433 M | O438 M | O452 M |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| A6 | SRM 3246 *Ginkgo biloba* leaves | 0 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| A9 | *Ginkgo biloba* leaves untreated | 0 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| A3 | *Ginkgo biloba* leaves untreated | 3 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| A16 | *Ginkgo biloba* leaves untreated | 7 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| A4 | *Ginkgo biloba* leaves untreated | 15 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| A12 | *Ginkgo biloba* leaves untreated | 15 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| A5 | *Ginkgo biloba* leaves steam treated | 0 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| A14 | *Ginkgo biloba* leaves steam treated | 3 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| A7 | *Ginkgo biloba* leaves steam treated | 7 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| A13 | *Ginkgo biloba* leaves steam treated | 7 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| A11 | *Ginkgo biloba* stem | 15 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| A15 | *Ginkgo biloba* stem | 0 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| A2 | *Ginkgo biloba* stem | 3 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| A10 | *Ginkgo biloba* stem | 3 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| A1 | *Ginkgo biloba* stem | 7 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| A8 | *Ginkgo biloba* leaves steam treated | 15 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| B10 | Aqueous ginkgo extract | 0 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| B5 | Aqueous ginkgo extract | 3 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| B13 | Aqueous ginkgo extract | 7 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| B7 | Aqueous ginkgo extract | 15 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| B3 | Ethanol:Water extract 1 | 0 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| B8 | Ethanol:Water extract 2 | 0 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| B12 | Ethanol:Water extract 2 | 3 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| B16 | Ethanol:Water extract 2 | 3 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| B1 | Ethanol:Water extract 2 | 7 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| B9 | Ethanol:Water extract 2 | 15 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| B2 | Acetone:Water extract 2 | 0 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| B14 | Acetone:Water extract | 3 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| B6 | Acetone:Water extract | 7 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| B11 | Acetone:Water extract | 7 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| B4 | Acetone:Water extract | 15 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| B15 | Acetone:Water extract | 15 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |