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Dietary Supplement Laboratory Quality Assurance Program: Exercise 1 Final Report

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Abstract

The National Institute of Standards and Technology (NIST) Dietary Supplement Laboratory Quality Assurance Program (DSQAP) was launched in 2007 in part as a collaboration with the National Institutes of Health (NIH) Office of Dietary Supplements (ODS). The DSQAP enables laboratories to improve the accuracy of measurements in samples for nutrients, marker compounds, toxic elements, and/or contaminants in dietary supplement ingredients and finished products. Exercise 1 is the sixteenth DSQAP exercise (previously they were designated "A" through "O"). Exercise 1 was designed with 7 studies, offering the opportunity for laboratories to assess their in-house techniques on a variety measurements. Studies included determinations of select toxic and nutritional elements, vitamins, contaminants, and proximates in kelp, polyphenol content in kelp and green tea, water-soluble vitamins in meal replacement drink formulations, and botanical marker compounds in dietary supplement ingredient materials and finished products. This report summarizes the results, describes observations, and provides technical recommendations for measurement improvements.

Keywords

Botanicals; Consumer Safety; Dietary Supplements; Kelp; Quality Assurance; Reference Materials.

Abstract i
Keywords i
Acknowledgements 14
1. Introduction 1
1.1. Background 1
1.2. Overview of Data Treatment and Representation 2
1.2.1. Statistics 2
1.2.2. Individualized Data Tables 3
1.2.3. Summary Data Tables 4
1.2.4. Figures 5
2. Overall Technical Recommendations 7
3. Elements in Kelp 8
3.1. Executive Summary 8
3.2. Background 8
3.3. Study Information 8
3.4. Study Results and Technical Recommendations 10
3.4.1. Total Arsenic (tAs) 12
3.4.2. Inorganic Arsenic (iAs) 17
3.4.3. Cadmium (Cd) 22
3.4.4. Calcium (Ca) 27
3.4.5. Chromium (Cr) 35
3.4.6. Copper (Cu) 40
3.4.7. Iodine (I) 45
3.4.8. Lead (Pb) 50
3.4.9. Magnesium (Mg) 55
3.4.10. Mercury (Hg) 60
3.4.11. Potassium (K) 65
3.4.12. Selenium (Se) 70
3.4.13. Sodium (Na) 75
3.4.14. Sulfur (S) 80
3.4.15. Zinc (Zn) 85
4. Vitamins I (Vitamins B3 and K1 in kelp) 90
4.1. Executive Summary 90
4.2. Background 90
4.3. Study Information 90
4.4. Study Results and Technical Recommendations 91

4.4.1. Niacin and Niacinamide ... 91
4.4.2. Phylloquinone ... 93
5. Botanicals I (Phenolic Content) ... 96
5.1. Executive Summary ... 96
5.2. Background ... 96
5.3. Study Information ... 96
5.4. Study Results and Technical Recommendations ... 97
5.4.1. Gallic Acid ... 97
5.4.2. Gallic Acid Equivalents ... 101
6. Proximates ... 105
6.1. Executive Summary ... 105
6.2. Background ... 105
6.3. Study Information ... 105
6.4. Study Results and Technical Recommendations ... 106
6.4.1. Ash ... 106
6.4.2. Fat ... 108
6.4.3. Protein ... 110
6.4.4. Solids ... 112
6.4.5. Starch ... 114
6.4.6. Total Dietary Fiber (TDF) ... 116
6.4.7. Carbohydrates ... 119
6.4.8. Calories ... 121
7. Contaminants ... 123
7.1. Executive Summary ... 123
7.2. Background ... 123
7.3. Study Information ... 124
7.4. Study Results and Technical Recommendations ... 124
8. Vitamins II (B Vitamins in Meal Replacement Formulations) ... 125
8.1. Executive Summary ... 125
8.2. Background ... 125
8.3. Study Information ... 125
8.4. Study Results and Technical Recommendations ... 127
8.4.1. Thiamine (B_1) ... 130
8.4.2. Riboflavin (B_2) ... 135
8.4.3. Niacin (B_3) ... 139
8.4.4. Pantothenic Acid (B_5) ... 143

8.4.5. Pyridoxine (B_6) ... 148
8.4.6. Biotin (B_7) ... 152
8.4.7. Folic Acid (B_9) ... 157
8.4.8. Cobalamin (B_{12}) ... 161
9. Botanicals II (Withanolides) ... 166
9.1. Executive Summary ... 166
9.2. Study Background and Participation ... 166
9.3. Study Results and Technical Recommendations ... 167
9.3.1. Ashwagandha Root Powder ... 167
9.3.2. Ashwagandha Root Powder Extract ... 172
References ... 176
Appendix A. List of Acronyms ... 182
Appendix B. Elements Supplemental Tables and Figures ... 184
Appendix C. Vitamins I Supplemental Tables and Figures ... 245
Appendix D. Botanicals I Supplemental Tables and Figures ... 254
Appendix E. Proximates Supplemental Tables and Figures ... 261
Appendix F. Vitamins II Supplemental Tables and Figures ... 270
Appendix G. Botanicals II Supplemental Tables and Figures ... 293

List of Tables

Table 1-1. Summary of DSQAP Exercise 1 Studies ... 2
Table 1-2. Individualized Data Table Template. ... 3
Table 1-3. Summary Data Table Template ... 5
Table 3-1. Enrollment and Participation Statistics for Elements in Kelp. ... 9
Table 3-2. Description of the consensus confidence interval in relation to the NIST target range for elements in kelp. ... 10
Table 3-3. Summary of results and laboratory variabilities for tAs in kelp. ... 12
Table 3-4. Summary of sample preparation and analytical methods averaged across all samples for determination of tAs in kelp. ... 13
Table 3-5. Summary of results and laboratory variabilities for iAs in kelp. ... 17
Table 3-6. Summary of sample preparation and analytical methods averaged across all samples for determination of iAs in kelp ... 18
Table 3-7. Summary of results and variabilities for Cd in kelp. ... 22
Table 3-8. Summary of sample preparation and analytical methods averaged across all samples for determination of Cd in kelp. ... 22
Table 3-9. Summary of results and laboratory variabilities for Ca in kelp. ... 27
Table 3-10. Summary of sample preparation and analytical methods averaged across all samples for determination of Ca in kelp. ... 28
Table 3-11. Summary of results and laboratory variabilities for Cr in kelp. ... 35
Table 3-12. Summary of sample preparation and analytical methods averaged across all samples for determination of Cr in kelp. ... 36

Table 3-13. Summary of results and laboratory variabilities for Cu in kelp. 40
Table 3-14. Summary of sample preparation and analytical methods averaged across all samples for determination of Cu in kelp. 40
Table 3-15. Summary of results and laboratory variabilities for I in kelp. 45
Table 3-16. Summary of sample preparation and analytical methods averaged across all samples for determination of I in kelp. 46
Table 3-17. Summary of results and laboratory variabilities for Pb in kelp. 50
Table 3-18. Summary of sample preparation and analytical methods averaged across all samples for determination of Pb in kelp. 50
Table 3-19. Summary of results and laboratory variabilities for Mg in kelp. 55
Table 3-20. Summary of sample preparation and analytical methods averaged across all samples for determination of Mg in kelp. 56
Table 3-21. Summary of results and laboratory variabilities for Hg in kelp. 60
Table 3-22. Summary of sample preparation and analytical methods averaged across all samples for determination of Hg in kelp. 61
Table 3-23. Summary of results and laboratory variabilities for K in kelp. 65
Table 3-24. Summary of sample preparation and analytical methods averaged across all samples for determination of K in kelp. 66
Table 3-25. Summary of results and laboratory variabilities for Se in kelp. 70
Table 3-26. Summary of sample preparation and analytical methods averaged across all samples for determination of Se in kelp. 70
Table 3-27. Summary of results and laboratory variabilities for Na in kelp. 75
Table 3-28. Summary of sample preparation and analytical methods averaged across all samples for determination of Na in kelp. 76
Table 3-29. Summary of results and laboratory variabilities for S in kelp. 80
Table 3-30. Summary of sample preparation and analytical methods averaged across all samples for determination of S in kelp. 80
Table 3-31. Summary of results and laboratory variabilities for Zn in kelp. 85
Table 3-32. Summary of sample preparation and analytical methods averaged across all samples for determination of Zn in kelp. 86
Table 4-1. Summary of results and laboratory variabilities for phylloquinone in kelp. 93
Table 5-1. Target values, consensus values, and variabilities for gallic acid in green tea and kelp. 97
Table 5-2. Consensus values and summary of variabilities for gallic acid equivalents in green tea and kelp. 101
Table 7-1. PFAS compounds to be analyzed in kelp. 123
Table 7-2. Results for PFAS compounds in kelp. 124
Table 8-1. Target mass fractions of B vitamins for meal replacement drinks and SRM 3252.. 126
Table 8-2. Enrollment and reporting statistics of B vitamins in meal replacement drinks and SRM 3252. 126
Table 8-3.Description of the location of the consensus range in relation to the target range for B vitamins in meal replacement drinks and SRM 3252. 128
Table 8-4. Summary of sample preparation methods for determination of B vitamins in meal replacement drinks and SRM 3252. 128
Table 8-5. Summary of analytical methods for determination of B vitamins in meal replacement drinks and SRM 3252. 129
Table 8-6. Summary of laboratory variabilities for thiamine in meal replacement drinks and SRM 3252. 130
Table 8-7. Summary of laboratory variabilities for riboflavin in meal replacement drinks and SRM 3252. 135

Table 8-8. Summary of laboratory variabilities for niacin in meal replacement drinks and SRM 3252. 139
Table 8-9. Summary of laboratory variabilities for pantothenic acid in meal replacement drinks and SRM 3252. 143
Table 8-10. Summary of laboratory variabilities for pyridoxine in meal replacement drinks and SRM 3252. 148
Table 8-11. Summary of laboratory variabilities for biotin in meal replacement drinks and SRM 3252. 152
Table 8-12. Summary of laboratory variabilities for folic acid in meal replacement drinks and SRM 3252. 157
Table 8-13. Summary of laboratory variabilities for cobalamin in meal replacement drinks and SRM 3252. 161
Table 9-1. Enrollment and reporting statistics for withanolides in ashwagandha root powder and ashwagandha root powder extract. 167
Table 9-2. Summary of analytical methods for determination of withanolides in ashwagandha root powder. 168
Table 9-3. Summary of laboratory variabilities for withanolides in ashwagandha root powder. 168
Table 9-4. Summary of laboratory variabilities for withanolides in ashwagandha root powder extract. 172
Table B-1. Individualized data summary table (example) for elements in kelp. 184
Table B-2. Data summary table for total arsenic in kelp. 185
Table B-3. Data summary table for inorganic arsenic in kelp. 189
Table B-4. Data summary table for cadmium in kelp. 193
Table B-5. Data summary table for calcium in kelp. 197
Table B-6. Data summary table for chromium in kelp. 201
Table B-7. Data summary table for copper in kelp. 205
Table B-8. Data summary table for iodine in kelp. 209
Table B-9. Data summary table for lead in kelp. 213
Table B-10. Data summary table for magnesium in kelp. 217
Table B-11. Data summary table for mercury in kelp. 221
Table B-12. Data summary table for potassium in kelp. 225
Table B-13. Data summary table for selenium in kelp. 229
Table B-14. Data summary table for sodium in kelp. 233
Table B-15. Data summary table for sulfur in kelp. 237
Table B-16. Data summary table for zinc in kelp. 241
Table C-1. Individualized data summary table (example) for vitamins in kelp. 245
Table C-2. Data summary table for niacin in kelp. 246
Table C-3. Data summary table for niacinamide in kelp. 249
Table C-4. Data summary table for phylloquinone in kelp. 252
Table D-1. Individualized data summary table (example) for botanicals in kelp and green tea. 254
Table D-2. Data summary table for gallic acid in SRM 3254 Green Tea *(Camellia sinensis)* Leaves, SRM 3255 Green Tea *(Camellia sinensis)* Extract, and Kelp A. 255
Table D-3. Data summary table for gallic acid equivalents SRM 3254 Green Tea *(Camellia sinensis)* Leaves, SRM 3255 Green Tea *(Camellia sinensis)* Extract, and Kelp A. 257
Table E-1. Individualized data summary table (example) for proximates in kelp. 261
Table E-2. Data summary table for ash in kelp. 262
Table E-3. Data summary table for carbohydrates in kelp. 263
Table E-4. Data summary table for fat in kelp. 264
Table E-5. Data summary table for protein in kelp. 265
Table E-6. Data summary table for solids in kelp. 266

Table E-7. Data summary table for starch in kelp. 267
Table E-8. Data summary table for total dietary fiber in kelp. 268
Table E-9. Data summary table for calories in kelp. 269
Table F-1. Individualized data summary table (example) for vitamins in meal replacement formulations 270
Table F-2. Data summary table for thiamine in Meal Replacement Drink (Liquid), Meal Replacement Drink (Powder), and SRM 3252 Protein Drink Mix. 271
Table F-3. Data summary table for riboflavin in Meal Replacement Drink (Liquid), Meal Replacement Drink (Powder), and SRM 3252 Protein Drink Mix. 274
Table F-4. Data summary table for niacin in Meal Replacement Drink (Liquid), Meal Replacement Drink (Powder), and SRM 3252 Protein Drink Mix. 277
Table F-5. Data summary table for pantothenic acid in Meal Replacement Drink (Liquid), Meal Replacement Drink (Powder), and SRM 3252 Protein Drink Mix. 280
Table F-6. Data summary table for pyridoxine in Meal Replacement Drink (Liquid), Meal Replacement Drink (Powder), and SRM 3252 Protein Drink Mix. 283
Table F-7. Data summary table for biotin in Meal Replacement Drink (Liquid), Meal Replacement Drink (Powder), and SRM 3252 Protein Drink Mix. 286
Table F-8. Data summary table for folic acid in Meal Replacement Drink (Liquid), Meal Replacement Drink (Powder), and SRM 3252 Protein Drink Mix. 289
Table F-9. Data summary table for cobalamin in Meal Replacement Drink (Liquid), Meal Replacement Drink (Powder), and SRM 3252 Protein Drink Mix. 292
Table G-1. Individualized data summary table (example) for botanicals in ashwagandha root powder and root powder extract. 293
Table G-2. Data summary table for 12-deoxywithastromonolide in ashwagandha root powder and ashwagandha root powder extract. 294
Table G-3. Data summary table for withaferin A in ashwagandha root powder and ashwagandha root powder extract. 297
Table G-4. Data summary table for withanolide A in ashwagandha root powder and ashwagandha root powder extract. 301
Table G-5. Data summary table for withanolide B in ashwagandha root powder and ashwagandha root powder extract. 305
Table G-6. Data summary table for withanoside IV in ashwagandha root powder and ashwagandha root powder extract. 308
Table G-7. Data summary table for withanoside V in ashwagandha root powder and ashwagandha root powder extract. 310

List of Figures

Fig. 3-1. Total arsenic in Kelp A (data summary view – analytical method). 14
Fig. 3-2. Total arsenic in Kelp B (data summary view – analytical method). 15
Fig. 3-3. Total arsenic in SRM 3232 Kelp Powder (*Thallus laminariae*) (data summary view – analytical method). 16
Fig. 3-4. Inorganic arsenic in Kelp A (data summary view – analytical method). 19
Fig. 3-5. Inorganic arsenic in Kelp B (data summary view – analytical method). 20
Fig. 3-6. Inorganic arsenic in SRM 3232 Kelp Powder (*Thallus laminariae*) (data summary view – analytical method). 21
Fig. 3-7. Cadmium in Kelp A (data summary view – analytical method). 24
Fig. 3-8. Cadmium in Kelp B (data summary view – analytical method). 25
Fig. 3-9. Cadmium in SRM 3232 Kelp Powder (*Thallus laminariae*) (data summary view – analytical method). 26

Fig. 3-10. Calcium in Kelp A (data summary view – analytical method). 29
Fig. 3-11. Kernel density estimation for calcium in Kelp A. 30
Fig. 3-12. Calcium in Kelp B (data summary view – analytical method). 31
Fig. 3-13. Kernel density estimation for calcium in Kelp B. 32
Fig. 3-14. Calcium in SRM 3232 Kelp Powder (*Thallus laminariae*) (data summary view – analytical method). 33
Fig. 3-15. Kernel density estimation for calcium in SRM 3232. 34
Fig. 3-16. Chromium in Kelp A (data summary view – analytical method). 37
Fig. 3-17. Chromium in Kelp B (data summary view – analytical method). 38
Fig. 3-18. Chromium in SRM 3232 Kelp Powder (*Thallus laminariae*) (data summary view – analytical method). 39
Fig. 3-19. Copper in Kelp A (data summary view – analytical method). 42
Fig. 3-20. Copper in Kelp B (data summary view – analytical method). 43
Fig. 3-21. Copper in SRM 3232 Kelp Powder (*Thallus laminariae*) (data summary view – analytical method). 44
Fig. 3-22. Iodine in Kelp A (data summary view – analytical method). 47
Fig. 3-23. Iodine in Kelp B (data summary view – analytical method). 48
Fig. 3-24. Iodine in SRM 3232 Kelp Powder (*Thallus laminariae*) (data summary view – analytical method). 49
Fig. 3-25. Lead in Kelp A (data summary view – analytical method). 52
Fig. 3-26. Lead in Kelp B (data summary view – analytical method). 53
Fig. 3-27. Lead in SRM 3232 Kelp Powder (*Thallus laminariae*) (data summary view – analytical method). 54
Fig. 3-28. Magnesium in Kelp A (data summary view – analytical method). 57
Fig. 3-29. Magnesium in Kelp B (data summary view – analytical method). 58
Fig. 3-30. Magnesium in SRM 3232 Kelp Powder (*Thallus laminariae*) (data summary view – analytical method). 59
Fig. 3-31. Mercury in Kelp A (data summary view – analytical method). 62
Fig. 3-32. Mercury in Kelp B (data summary view – analytical method). 63
Fig. 3-33. Mercury in SRM 3232 Kelp Powder (*Thallus laminariae*) (data summary view – analytical method). 64
Fig. 3-34. Potassium in Kelp A (data summary view – analytical method). 67
Fig. 3-35. Potassium in Kelp B (data summary view – analytical method). 68
Fig. 3-36. Potassium in SRM 3232 Kelp Powder (*Thallus laminariae*) (data summary view – analytical method). 69
Fig. 3-37. Selenium in Kelp A (data summary view – analytical method). 72
Fig. 3-38. Selenium in Kelp B (data summary view – analytical method). 73
Fig. 3-39. Selenium in SRM 3232 Kelp Powder (*Thallus laminariae*) (data summary view – analytical method). 74
Fig. 3-40. Sodium in Kelp A (data summary view – analytical method). 77
Fig. 3-41. Sodium in Kelp B (data summary view – analytical method). 78
Fig. 3-42. Sodium in SRM 3232 Kelp Powder (*Thallus laminariae*) (data summary view – analytical method). 79
Fig. 3-43. Sulfur in Kelp A (data summary view – analytical method). 82
Fig. 3-44. Sulfur in Kelp B (data summary view – analytical method). 83
Fig. 3-45. Sulfur in SRM 3232 Kelp Powder (*Thallus laminariae*) (data summary view – analytical method). 84
Fig. 3-46. Zinc in Kelp A (data summary view – analytical method). 87
Fig. 3-47. Zinc in Kelp B (data summary view – analytical method). 88
Fig. 3-48. Zinc in SRM 3232 Kelp Powder (*Thallus laminariae*) (data summary view – analytical method). 89

Fig. 4-1. Vitamin B_3 (niacin) in Kelp A (data summary view – analytical method). 92
Fig. 4-2. Vitamin B_3 (niacinamide) in Kelp A (data summary view – analytical method). 92
Fig. 4-3. Vitamin K_1 (phylloquinone) in Kelp B (data summary view – analytical method). 94
Fig. 4-4. Vitamin K_1 (phylloquinone) in SRM 3232 Kelp Powder (*Thallus laminariae*) (data summary view – analytical method). 95
Fig. 5-1. Gallic acid in SRM 3254 Green Tea *(Camellia sinensis)* Leaves (data summary view – analytical method)..... 98
Fig. 5-2. Gallic acid in SRM 3255 Green Tea *(Camellia sinensis)* Extract (data summary view – analytical method)..... 99
Fig. 5-3. Laboratory means for gallic acid in SRM 3255 Green Tea *(Camellia sinensis)* Extract and SRM 3254 Green Tea *(Camellia sinensis)* Leaves (sample/sample comparison view). ... 100
Fig. 5-4. Gallic acid equivalents in SRM 3254 Green Tea *(Camellia sinensis)* Leaves (data summary view – analytical method). 102
Fig. 5-5. Gallic acid equivalents in SRM 3255 Green Tea *(Camellia sinensis)* Extract (data summary view – analytical method). 103
Fig. 5-6. Gallic acid equivalents in Kelp A (data summary view – analytical method)..... 104
Fig. 6-1. Ash in Kelp A (data summary view – analytical method)..... 107
Fig. 6-2. Fat in Kelp A (data summary view – analytical method)..... 109
Fig. 6-3. Protein in Kelp A (data summary view – analytical method)..... 111
Fig. 6-4. Solids in Kelp A (data summary view – analytical method). 113
Fig. 6-5. Starch in Kelp A (data summary view – analytical method)..... 115
Fig. 6-6. Total dietary fiber in Kelp A (data summary view – analytical method)..... 117
Fig. 6-7. Kernel density estimation for TDF in Kelp A..... 118
Fig. 6-8. Carbohydrates in Kelp A (data summary view – analytical method). 120
Fig. 6-9. Calories in Kelp A (data summary view – analytical method)..... 122
Fig. 8-1. Vitamin B_1 (thiamine) in Meal Replacement Drink (Liquid) (data summary view – analytical method)..... 132
Fig. 8-2. Vitamin B_1 (thiamine) in Meal Replacement Drink (Powder) (data summary view – analytical method)..... 133
Fig. 8-3. Vitamin B_1 (thiamine) in SRM 3252 Protein Drink Mix (data summary view – analytical method)..... 134
Fig. 8-4. Vitamin B_2 (riboflavin) in Meal Replacement Drink (Liquid) (data summary view – analytical method)..... 136
Fig. 8-5. Vitamin B_2 (riboflavin) in Meal Replacement Drink (Powder) (data summary view – analytical method)..... 137
Fig. 8-6. Vitamin B_2 (riboflavin) in SRM 3252 Protein Drink Mix (data summary view – analytical method)..... 138
Fig. 8-7. Vitamin B_3 (niacin) in Meal Replacement Drink (Liquid) (data summary view – analytical method)..... 140
Fig. 8-8. Vitamin B_3 (niacin) in Meal Replacement Drink (Powder) (data summary view – analytical method)..... 141
Fig. 8-9. Vitamin B_3 (niacin) in SRM 3252 Protein Drink Mix (data summary view – analytical method)..... 142
Fig. 8-10. Vitamin B_5 (pantothenic acid) in Meal Replacement Drink (Liquid) (data summary view – analytical method). 145
Fig. 8-11. Vitamin B_5 (pantothenic acid) in Meal Replacement Drink (Powder) (data summary view – analytical method). 146
Fig. 8-12. Vitamin B_5 (pantothenic acid) in SRM 3252 Protein Drink Mix (data summary view – analytical method)..... 147
Fig. 8-13. Vitamin B_6 (pyridoxine) in Meal Replacement Drink (Liquid) (data summary view – analytical method)..... 149

Fig. 8-14. Vitamin B_6 (pyridoxine) in Meal Replacement Drink (Powder) (data summary view – analytical method). 150
Fig. 8-15. Vitamin B_6 (pyridoxine) in SRM 3252 Protein Drink Mix (data summary view – analytical method). 151
Fig. 8-16. Vitamin B_7 (biotin) in Meal Replacement Drink (Liquid) (data summary view – analytical method). 154
Fig. 8-17. Vitamin B_7 (biotin) in Meal Replacement Drink (Powder) (data summary view – analytical method). 155
Fig. 8-18. Vitamin B_7 (biotin) in SRM 3253 Protein Drink Mix (data summary view – analytical method). 156
Fig. 8-19. Vitamin B_9 (folic acid) in Meal Replacement Drink (Liquid) (data summary view – analytical method). 158
Fig. 8-20. Vitamin B_9 (folic acid) in Meal Replacement Drink (Powder) (data summary view – analytical method). 159
Fig. 8-21. Vitamin B_9 (folic acid) in SRM 3252 Protein Drink Mix (data summary view – analytical method). 160
Fig. 8-22. Vitamin B_{12} (cobalamin) in Meal Replacement Drink (Liquid) (data summary view – analytical method). 163
Fig. 8-23. Vitamin B_{12} (cobalamin) in Meal Replacement Drink (Powder) (data summary view – analytical method). 164
Fig. 8-24. Vitamin B_{12} (cobalamin) in SRM 3252 Protein Drink Mix (data summary view – analytical method). 165
Fig. 9-1. Withanoside IV in ashwagandha root powder (data summary view – analytical method). 170
Fig. 9-2. 12-deoxywithastromonolide in ashwagandha root powder (data summary view – analytical method). 171
Fig. 9-3. Withanoside IV in ashwagandha root powder extract (data summary view – analytical method). 174
Fig. 9-4. Withanolide B in ashwagandha root powder extract (data summary view – analytical method). 175
Fig. B-1. Laboratory means for total arsenic in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp A (sample/sample comparison view). 187
Fig. B-2. Laboratory means for total arsenic in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp B (sample/sample comparison view). 188
Fig. B-3. Laboratory means for inorganic arsenic in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp A (sample/sample comparison view). 191
Fig. B-4. Laboratory means for inorganic arsenic in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp B (sample/sample comparison view). 192
Fig. B-5. Laboratory means for cadmium in SRM 3232 Kelp Powder (Thallus laminariae) and Kelp A (sample/sample comparison view). 195
Fig. B-6. Laboratory means for cadmium in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp B (sample/sample comparison view). 196
Fig. B-7. Laboratory means for calcium in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp A (sample/sample comparison view). 199
Fig. B-8. Laboratory means for calcium in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp B (sample/sample comparison view). 200
Fig. B-9. Laboratory means for chromium in SRM 3232 Kelp Powder (Thallus laminariae) and Kelp A (sample/sample comparison view). 203
Fig. B-10. Laboratory means for chromium in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp B (sample/sample comparison view). 204

Fig. B-11. Laboratory means for copper in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp A (sample/sample comparison view). 207
Fig. B-12. Laboratory means for copper in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp B (sample/sample comparison view). 208
Fig. B-13. Laboratory means for iodine in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp A (sample/sample comparison view)..... 211
Fig. B-14. Laboratory means for iodine in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp B (sample/sample comparison view)..... 212
Fig. B-15. Laboratory means for lead in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp A (sample/sample comparison view)..... 215
Fig. B-16. Laboratory means for lead in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp B (sample/sample comparison view)..... 216
Fig. B-17. Laboratory means for magnesium in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp A (sample/sample comparison view). 219
Fig. B-18. Laboratory means for magnesium in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp B (sample/sample comparison view). 220
Fig. B-19. Laboratory means for mercury in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp A (sample/sample comparison view). 223
Fig. B-20. Laboratory means for mercury in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp B (sample/sample comparison view). 224
Fig. B-21. Laboratory means for potassium in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp A (sample/sample comparison view). 227
Fig. B-22. Laboratory means for potassium in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp B (sample/sample comparison view). 228
Fig. B-23. Laboratory means for selenium in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp A (sample/sample comparison view). 231
Fig. B-24. Laboratory means for selenium in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp B (sample/sample comparison view). 232
Fig. B-25. Laboratory means for sodium in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp A (sample/sample comparison view). 235
Fig. B-26. Laboratory means for sodium in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp B (sample/sample comparison view). 236
Fig. B-27. Laboratory means for sulfur in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp A (sample/sample comparison view)..... 239
Fig. B-28. Laboratory means for sulfur in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp B (sample/sample comparison view)..... 240
Fig. B-29. Laboratory means for zinc in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp A (sample/sample comparison view)..... 243
Fig. B-30. Laboratory means for zinc in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp B (sample/sample comparison view)..... 244
Fig. C-1. Vitamin B_3 (niacin) in Kelp B (data summary view – analytical method)..... 247
Fig. C-2. Vitamin B_3 (niacin) in SRM 3232 Kelp Powder (*Thallus laminariae*) (data summary view – analytical method). 248
Fig. C-3. Vitamin B_3 (niacinamide) in Kelp B (data summary view – analytical method)..... 250
Fig. C-4. Vitamin B_3 (niacinamide) in SRM 3232 Kelp Powder (*Thallus laminariae*) (data summary view – analytical method). 251
Fig. C-5. Vitamin K (phylloquinone) in Kelp A (data summary view – analytical method)..... 253
Fig. D-1. Gallic acid in Kelp A (data summary view – analytical method)..... 256
Fig. D-2. Laboratory means for gallic acid equivalents in SRM 3255 Green Tea *(Camellia sinensis)* Extract and SRM 3254 Green Tea *(Camellia sinensis)* Leaves (sample/sample comparison view). 258

Fig. D-3. Laboratory means for gallic acid equivalents in SRM 3254 Green Tea *(Camellia sinensis)* Leaves and Kelp A (sample/sample comparison view). 259
Fig. D-4. Laboratory means for gallic acid equivalents in SRM 3255 Green Tea *(Camellia sinensis)* Extract and Kelp A (sample/sample comparison view). 260
Fig. F-1. Laboratory means for thiamine in SRM 3252 Protein Drink Mix and Meal Replacement Drink (Liquid) (sample/sample comparison view). 272
Fig. F-2. Laboratory means for thiamine in SRM 3252 Protein Drink Mix and Meal Replacement Drink (Powder) (sample/sample comparison view). 273
Fig. F-3. Laboratory means for riboflavin in SRM 3252 Protein Drink Mix and Meal Replacement Drink (Liquid) (sample/sample comparison view). 275
Fig. F-4. Laboratory means for riboflavin in SRM 3252 Protein Drink Mix and Meal Replacement Drink (Powder) (sample/sample comparison view). 276
Fig. F-5. Laboratory means for niacin in SRM 3252 Protein Drink Mix and Meal Replacement Drink (Liquid) (sample/sample comparison view). 278
Fig. F-6. Laboratory means for niacin in SRM 3252 Protein Drink Mix and Meal Replacement Drink (Powder) (sample/sample comparison view). 279
Fig. F-7. Laboratory means for pantothenic acid in SRM 3252 Protein Drink Mix and Meal Replacement Drink (Liquid) (sample/sample comparison view). 281
Fig. F-8. Laboratory means for pantothenic acid in SRM 3252 Protein Drink Mix and Meal Replacement Drink (Powder) (sample/sample comparison view). 282
Fig. F-9. Laboratory means for pyridoxine in SRM 3252 Protein Drink Mix and Meal Replacement Drink (Liquid) (sample/sample comparison view). 284
Fig. F-10. Laboratory means for pyridoxine in SRM 3252 Protein Drink Mix and Meal Replacement Drink (Powder) (sample/sample comparison view). 285
Fig. F-11. Laboratory means for biotin in SRM 3252 Protein Drink Mix and Meal Replacement Drink (Liquid) (sample/sample comparison view). 287
Fig. F-12. Laboratory means for biotin in SRM 3252 Protein Drink Mix and Meal Replacement Drink (Powder) (sample/sample comparison view). 288
Fig. F-13. Laboratory means for folic acid in SRM 3252 Protein Drink Mix and Meal Replacement Drink (Liquid) (sample/sample comparison view). 290
Fig. F-14. Laboratory means for folic acid in SRM 3252 Protein Drink Mix and Meal Replacement Drink (Powder) (sample/sample comparison view). 291
Fig. G-1. 12-deoxywithastromonolide in ashwagandha root powder extract (data summary view – analytical method). 295
Fig. G-2. Laboratory means for 12-deoxywithastromonolide in ashwagandha root powder and ashwagandha root powder extract (sample/sample comparison view). 296
Fig. G-3. Withaferin A in ashwagandha root powder (data summary view – analytical method). 298
Fig. G-4. Withaferin A in ashwagandha root powder extract (data summary view – analytical method). 299
Fig. G-5. Laboratory means for withaferin A in ashwagandha root powder and ashwagandha root powder extract (sample/sample comparison view). 300
Fig. G-6. Withanolide A in ashwagandha root powder (data summary view – analytical method). 302
Fig. G-7. Withanolide A in ashwagandha root powder extract (data summary view – analytical method). 303
Fig. G-8. Laboratory means for withanolide A in ashwagandha root powder and ashwagandha root powder extract (sample/sample comparison view). 304
Fig. G-9. Withanolide B in ashwagandha root powder (data summary view – analytical method). 306

Fig. G-10. Laboratory means for withanolide B in ashwagandha root powder and ashwagandha root powder extract (sample/sample comparison view). 307
Fig. G-11. Laboratory means for withanoside IV in ashwagandha root powder and ashwagandha root powder extract (sample/sample comparison view). 309
Fig. G-12. Withanoside V in ashwagandha root powder (data summary view – analytical method). 311
Fig. G-13. Withanoside V in ashwagandha root powder extract (data summary view – analytical method). 312
Fig. G-14. Laboratory means for withanoside V in ashwagandha root powder and ashwagandha root powder extract (sample/sample comparison view). 313

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1. Introduction

1.1. Background

The National Institute of Standards and Technology (NIST) Dietary Supplement Laboratory Quality Assurance Program (DSQAP) was first established in 2007 in part as a collaboration with the National Institutes of Health (NIH) Office of Dietary Supplements (ODS). The program was integrated into the Health Assessment Measurements Quality Assurance Program (HAMQAP) in 2017 but was revived as the DSQAP in 2022. The current DSQAP continues the ongoing collaborative efforts between NIST and the NIH ODS.

NIST has more than 30 years of experience in the administration of QAPs, including historical programs [i.e., Micronutrients Measurement QAP (MMQAP), Vitamin D Metabolites QAP (VitDQAP), and HAMQAP] and currently active programs [i.e., Cannabis Laboratory QAP (CannaQAP), Food Nutrition and Safety Measurements QAP (FNSQAP)]. The DSQAP focuses on improving the measurement capabilities of the dietary supplement and natural product measurement communities. Participating laboratories are interested in evaluating in-house methods on a wide variety of challenging, real-world matrices to demonstrate that their performance is comparable to that of the broader community and that their methods provide accurate results. In areas where few consensus or official methods have been recognized, DSQAP is a unique tool for assessment of the quality of measurements and provides feedback about performance that can assist participants in improving laboratory operations.

DSQAP offers the opportunity for laboratories to assess their in-house measurements of nutritional and toxic elements, water- and fat -soluble vitamins, marker compounds, and organic contaminants in samples distributed by NIST. Reports and certificates of participation are provided and may be used to demonstrate compliance with current Good Manufacturing Practices (cGMPs) or to fulfill proficiency requirements established by accreditation bodies. In addition, NIST and DSQAP assist the NIH ODS Analytical Methods and Reference Materials (AMRM) program in supporting the development and dissemination of analytical tools and reference materials. Results from DSQAP exercises could be used by ODS and NIST to identify problematic matrices and analytes for which consensus-based methods of analysis would benefit the dietary supplements measurement communities.

DSQAP Exercise 1 was leveraged to help identify measurement discrepancies that impact the seaweed farming industry and pinpoint methodologies that could benefit from standardization to improve the compositional testing of kelp materials. These studies were sponsored through a collaboration between NIST and World Wildlife Fund (WWF).

While NIST QAP exercises are not proficiency tests (PT) and are not intended to pass strict evaluation of laboratory performance, they are conducted according to International Organization for Standardization (ISO)/ International Electrotechnical Commission (IEC) 17043 and are designed to assist participants in evaluation and improvement of their measurement capabilities. Additionally, industry stakeholders can observe measurement challenges and NIST gains knowledge to guide the production and maintenance of reference materials.

This report summarizes the results from Exercise 1 of the DSQAP (fifteen previous DSQAP exercises were named Exercise A through Exercise O). Seventy-nine laboratories responded to the January 2022 call for study participation for DSQAP Exercise 1 as seen in **Table 1-1**. Samples were shipped to participants in May 2022 and results were returned to NIST by June 17, 2022. This report contains the final data and information that was disseminated to the participants in October 2023.

Table 1-1. Summary of DSQAP Exercise 1 Studies.

Study Group	Analytes	Samples
Elements	tAs, iAs, Cd, Ca, Cr, Cu, I, Pb, Mg, Hg, K, Se, Na, S, Zn	Kelp
Vitamins I	Vitamin B_3, Vitamin K_1	Kelp
Botanicals I	Gallic acid, Gallic acid equivalents	Green Tea Leaves and Extract, Kelp
Proximates	Ash, Carbohydrates, Fat, Protein, Solids, Starch, Total Dietary Fiber, Calories	Kelp
Contaminants	PFBS, PFBA, PFDOA, PFHPA, PFHXDA, PFHXS, PFHXA, PFNA, PFOS, PFOA, PFPEA, PFODA, PFTEDA, PFTRDA, PFUDA	Kelp
Vitamins II	Vitamins B_1, B_2, B_3, B_5, B_6, B_7, B_9, B_{12}	Meal Replacement Drink formulations, Protein Powder
Botanicals II	12-deoxywithastromonolide, Withaferin A, Withanolide A, Withanolide B, Withanoside IV, Withanoside V	Ashwagandha Root Powder and Extract

Each study is summarized individually with appropriate tables, figures, and text, and is reported by section. Additional tables and figures can be found in the Appendices. Conclusions and technical recommendations are drawn for the entire exercise when possible and reported in the **Overall Technical Recommendations** section.

1.2. 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 are also included in each section of this report. Community tables and figures are provided using randomized laboratory codes. Laboratories only know their own participation code. The statistical approaches are outlined below for each type of data representation.

1.2.1. Statistics

Data tables and figures throughout this report contain information about the performance of each laboratory relative to that of the other participants in this study and relative to a target around the expected result, if available. All calculations are performed in PROLab Plus (QuoData GmbH, Dresden, Germany). The consensus means and standard deviations are calculated according to the robust Q/Hampel method outlined in ISO 13528:2022 Annex C [1].

1.2.2. Individualized Data Tables

The data in **Table 1-2** is individualized to each participating laboratory and is provided to allow participants to directly compare their data to the summary statistics (consensus or community data as well as NIST certified, non-certified, or estimated values, when available). Participating laboratories receive uniquely coded individualized data tables in a separate distribution, with the randomized laboratory code in the upper left of the data table. Example individualized data tables included in this report are made with this section blank to protect the identity and performance of participants.

Table 1-2. Individualized Data Table Template.

(Laboratory Name)

Exercise 1 - (Study Name)

	Lab Code:	**(code)**	**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*
Analyte 1	Sample Name A	unit	*Individual laboratory results will appear in this section; Laboratory-specific results were provided to each participant separately from this report*				*Community results will appear in this section*			*Target values will appear in this section*	
Analyte 1	Sample Name B	unit									
Analyte 2	Sample Name A	unit									
Analyte 2	Sample Name B	unit									
Analyte 3	Sample Name A	unit									
Analyte 3	Sample Name B	unit									

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

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 from that laboratory for the corresponding analyte or matrix. An empty box for standard deviation indicates that the participant reported a single value or a value below the limit of quantification (LOQ) and therefore that value was not included in the calculation of the consensus data.

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

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

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

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

or

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

Significance of the *Z*-scores:

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

Section 2 of the data table (*Community Results*) contains the consensus results, including the number of laboratories reporting more than a single quantitative value for each analyte, the mean value determined for each analyte, and a robust estimate of the standard deviation of the reported values [1]. Consensus means and standard deviations are calculated using the laboratory means; if a laboratory reported a single value, the reported value is not included in determination of the consensus values [1]. Additional information on calculation of the consensus mean and standard deviation can be found in the previous section.

Section 3 of the data table (*Target*) contains the target values for each analyte, when available. When a NIST Standard Reference Material (SRM) or Reference Material (RM) is used as a sample in the study, the NIST certified or non-certified values and associated uncertainties (U_{95}) are used as target values. The criteria used by NIST to assign certified and non-certified values is described elsewhere [2]. Target values for other study samples may be determined at NIST or by a collaborating laboratory as the mean of at least three replicates. Target values may also be based on information provided by the material manufacturer or determined from another interlaboratory study or proficiency testing program, where the consensus value and uncertainty from the completed round is used as the target range. The exact methods for determination of the study target values are outlined in detail within each section of this report.

1.2.3. Summary Data Tables

This data table includes a summary of all reported data for a particular analyte in a particular study. Participants can compare the raw data from their laboratory to data reported by the other participating laboratories and to the consensus data. A blank indicates that the laboratory signed up and received samples for that analyte and matrix, but NIST does not have data on file from that laboratory. An empty box for standard deviation indicates that the participant reported a single value or a value below the LOQ and therefore that value was not included in the calculation of the consensus data. Data highlighted in red have been flagged as a data entry of zero or results that include text (e.g., "< LOQ" or "present"). Data highlighted in blue have been identified as outside the consensus tolerance limits and would be estimated to yield $|Z'_{\mathrm{comm}}| > 2$ by the PROLab software package. A summary data table example is shown in **Table 1-3** and the following are some laboratory data reporting examples. Laboratory code 4 only reported one value for one sample and therefore no standard deviation is shown. Laboratory code 6 data

resulted in values outside the consensus tolerance limits and is highlighted with blue text. Laboratory code 10 reported zero, which is not an appropriate result, and is highlighted in red text.

Table 1-3. Summary Data Table Template.

		Analyte 1									
		Sample Name A (unit)					Sample Name B (unit)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
Individual Results	Target				Target	U				Target	U
	(lab code 1)	Value 1	Value 2	Value 3	Avg	SD	Value 1	Value 2	Value 3	Avg	SD
	(lab code 2)	Value 1	Value 2	Value 3	Avg	SD	Value 1	Value 2	Value 3	Avg	SD
	(lab code 3)	Value 1	Value 2	Value 3	Avg	SD	Value 1	Value 2	Value 3	Avg	SD
	(lab code 4)	Value 1			Value 1						
	(lab code 5)	Value 1	Value 2	Value 3	Avg	SD	Value 1	Value 2	Value 3	Avg	SD
	(lab code 6)	Value 1	Value 2	Value 3	Avg	SD	Value 1	Value 2	Value 3	Avg	SD
	(lab code 7)	Value 1	Value 2	Value 3	Avg	SD	Value 1	Value 2	Value 3	Avg	SD
	(lab code 8)	Value 1	Value 2	Value 3	Avg	SD	Value 1	Value 2	Value 3	Avg	SD
	(lab code 9)	Value 1	Value 2	Value 3	Avg	SD	Value 1	Value 2	Value 3	Avg	SD
	(lab code 10)	0	0	0	0	0	0	0	0	0	0
Community Results		Consensus Mean			(Avg)		Consensus Mean			(Avg)	
		Consensus Standard Deviation			(SD)		Consensus Standard Deviation			(SD)	
		Maximum			(Max)		Maximum			(Max)	
		Minimum			(Min)		Minimum			(Min)	
		N			(N)		N			(N)	

1.2.4. Figures

1.2.4.1. Data Summary View (Method Comparison Data Summary View)

In this view, individual laboratory data (diamonds) are plotted with the individual laboratory standard deviation (rectangle). Laboratories reporting values below their LOQ are shown in this view as downward triangles beginning at the LOQ, reported as quantification 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 uncertainty of the consensus mean. The uncertainty in the consensus mean is calculated using the equation below, based on the repeatability standard deviation (s_{r}), the reproducibility standard deviation (s_{R}), the number of participants reporting data, and the average number of replicates reported by each participant. The uncertainty about the consensus mean is independent of the range of tolerance.

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

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

1.2.4.2. Sample/Sample Comparison View

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

2. Overall Technical Recommendations

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

The *use of quality assurance or quality control materials* (commercially available reference materials or appropriately characterized in-house materials) helps to establish that sample preparation methods and analytical methods are appropriate and performing as expected. The analysis of blanks can provide information about sources of analytical variability, such as from the sample preparation procedure or the material itself. Analysis of a statistically sufficient number of procedural blanks is important, especially when determining an LOQ or when trying to reduce sample-to-sample variability.

Proper calibration is critical to successful measurements. When using a calibration curve, linearity must be ensured at the mass fractions of the sample solutions being measured and the range of calibrant mass fractions should encompass the as-measured sample mass fractions. No as-measured sample mass fractions should be outside of the linear range. Materials used in calibrant preparation should be assessed for purity, and the measured purity should be used to correct the gravimetric or volumetric concentrations of the solutions used for calibration. Calibrant materials should also be assessed for the presence of residual solvents prior to use. Purity evaluation is especially critical for vitamins and botanical marker compounds. Calibrants should be prepared in a manner to match the final sample preparation solution (i.e., similar mass fractions and similar solvent) whenever possible to avoid potential biases that may arise during sample preparation or from differences in chromatographic retention time or detector sensitivity. The addition of an internal standard is recommended to help improve the precision of the instrumental measurements. Selecting the appropriate internal standard will help to correct measurement variability between the calibration standards and the samples.

Specifically to the QAP, *calculations and reporting units should be verified* prior to submission of results. Laboratories often report results in the wrong units or forget a dilution factor during the calculation of the final results, resulting in poor performance on the study. Laboratories reporting results which have been flagged as outside of consensus tolerance limits on preliminary data sheets should check for these types of errors and provide corrected results. Results should also be recorded appropriately in the online data entry system. For example, zero is not a quantity that can be measured and should not be reported; if results are below a method LOQ, values should be reported as such (e.g., “< 0.02 %”). Blank data entry fields are only appropriate when no measurements were made.

3. Elements in Kelp

3.1. Executive Summary

Elemental analysis of foods and dietary supplements is critical to consumer health and safety. The goal of this study was to understand how the measurement community is performing for the determination of toxic and nutritional elements in powdered kelp materials. Between 39 and 53 laboratories registered for individual elements, as seen in **Table 3-1**, with data return rates between 17 % and 67 %. Overall, laboratories performed well, though a few elements challenged the community, including mercury and selenium.

3.2. Background

Consumers expect labeling information to be accurate on the food and dietary supplement products they purchase. In the United States (U.S.), accurate measurements of nutrients are needed to ensure compliance with the U.S. Food and Drug Administration (FDA) regulations on the levels claimed on Nutrition Facts and Supplement Facts labels. Seaweeds are used internationally in food and dietary supplement products, both as a standalone source of nutrients or as a functional ingredient. Laboratories must establish scientifically valid methods for the determination of toxic and nutritional elements to demonstrate the products are safe and meet their specifications. Monitoring toxic substances in foods and dietary supplements helps prevent hazardous exposures for consumers and reduces the risk of related negative health outcomes. A challenge in the kelp space is the variation and uncertainty of regulations for both toxic and nutritional elements within and among countries. Understanding the amounts and forms of elements in kelp will help underpin research and support evidence-based regulations for the many different end-uses of seaweed materials.

In this study, participants were asked to use in-house analytical methods to determine the mass fractions of arsenic (total, tAs, and inorganic, iAs), cadmium (Cd), calcium (Ca), chromium (Cr), copper (Cu), iodine (I), lead (Pb), magnesium (Mg), mercury (Hg), potassium (K), selenium (Se), sodium (Na), sulfur (S), and zinc (Zn) in the kelp samples. These elements were selected to encompass both nutritionally important and known toxic elements with varying mass fractions in samples of three different powdered kelp species.

3.3. Study Information

Participants were provided with samples of Kelp A (three 10 g packets, *Saccharina latissima f. angustissima* from the coast of Maine, U.S.), Kelp B (three 5 g packets, *Ascophyllum nodosum* from the Northern Atlantic Ocean), and Kelp C (three 5 g packets, *Thallus laminariae* from the East China Sea). SRM 3232 Kelp Powder (*Thallus laminariae*) was labelled as Kelp C for this DSQAP exercise to conceal the identity of the material to participants and will be referred to as SRM 3232 for the remainder of the report. Participants were asked to store the materials at controlled room temperature, 20 °C to 25 °C, in the original unopened packets until analysis and to prepare one sample and report one value from each packet provided. Before use, participants were instructed to thoroughly mix the contents of the packet and to allow contents to settle for one minute prior to opening to minimize the loss of fine particles prior to removal of a test

portion for analysis, and to use a sample size of at least 0.5 g for elemental analyses. Approximate analyte levels were not reported to participants prior to the study.

Target values, associated uncertainties, and details on analytical methods are listed in the following individual analyte sections. For most elements in SRM 3232, target values were taken from the Certificate of Analysis (COA) at the time of this report [3]. These values were transformed to as-measured by moisture content by using the moisture correction in the COA (0.9368 g dry mass/g as-received mass) in order for the values to be comparable to the as-received units requested for reporting by participants. Target values for Kelp A and B were determined at NIST with at least triplicate sample preparations.

Enrollment and participation rates, averaged for the three kelp materials, for this study are detailed in **Table 3-1.** Some of the reported values were non-quantitative (zero or below LOQ) but are included in the participation and reporting statistics.

Table 3-1. Enrollment and Participation Statistics for Elements in Kelp.

Analyte	Number of Laboratories Requesting Samples	Number of Laboratories Reporting Results (Percent Participation) Averaged for all Samples
Total Arsenic (tAs)	53	32 (60 %)
Inorganic Arsenic (iAs)	40	7 (17 %)
Cadmium (Cd)	53	34 (64 %)
Calcium (Ca)	51	34 (67 %)
Chromium (Cr)	50	31 (61 %)
Copper (Cu)	52	28 (54 %)
Iodine (I)	39	12 (31 %)
Lead (Pb)	53	33 (62 %)
Magnesium (Mg)	51	33 (65 %)
Mercury (Hg)	51	30 (59 %)
Potassium (K)	51	33 (65 %)
Selenium (Se)	47	27 (57 %)
Sodium (Na)	49	29 (59 %)
Sulfur (S)	42	14 (33 %)
Zinc (Zn)	51	28 (56 %)

3.4. Study Results and Technical Recommendations

The consensus confidence interval was compared to the NIST target range for each analyte to assess the performance of the participants and is summarized in **Table 3-2**. A consensus mean within the target range is an indication that the community is performing well.

Table 3-2. Description of the consensus confidence interval in relation to the NIST target range for elements in kelp.

	Consensus Confidence Interval in relation to NIST Target Range		
Analyte	Kelp A	Kelp B	SRM 3232
Total Arsenic (tAs)	Overlapping Above (mean at top of range)	Within (mean above target)	Within (mean = target)
Inorganic Arsenic (iAs)	Overlapping (mean = target)	Within (mean below target)	Overlapping Above (mean above range)
Cadmium (Cd)	Overlapping Below (mean below range)	Below (mean below range)	Overlapping Below (mean below range)
Calcium (Ca)	Overlapping Above (mean above target)	Above (mean above range)	Overlapping Above (mean above target)
Chromium (Cr)	Overlapping Below (mean below range)	Overlapping Below (mean within range)	Within (mean below target)
Copper (Cu)	Within (mean = target)	Overlapping (mean at top of range)	Overlapping Below (mean at bottom of range)
Iodine (I)	(no target)	(no target)	Within (mean below target)
Lead (Pb)	Overlapping Below (mean below target)	Overlapping (mean = target)	Overlapping Below (mean below range)
Magnesium (Mg)	Within (mean above target)	Within (mean above target)	Within (mean = target)
Mercury (Hg)	Overlapping (mean above range)	Above (mean above range)	Overlapping Below (mean below range)
Potassium (K)	Below (mean below range)	Within (mean below target)	Overlapping Below (mean below range)
Selenium (Se)	Above (mean above range)	Above (mean above range)	Above (mean above range)
Sodium (Na)	Overlapping (mean within range)	Overlapping Below (mean at bottom of range)	Overlapping Below (mean below target)
Sulfur (S)	(no target)	(no target)	(no target)
Zinc (Zn)	Within (mean below target)	Within (mean = target)	Within (mean = target)

In order to assess performance of methods run by individual participants and the community as a whole, repeatability and reproducibility were compared to AOAC Standard Method Performance Requirements (SMPRs). At the time of this report, no SMPRs had been published specific to kelp or seaweed matrices, nor any that include all of the elements measured in this study. Several SMPRs were identified as acceptable proxies, including AOAC SMPR 2020.001 Determination of Heavy Metals in a Variety of Cannabis and Cannabis-Derived Products for tAs, Cd, Cr, Cu, Hg, Pb, Se, and Zn [4]; AOAC SMPR 2015.006 Quantitation of Arsenic Species in Selected Foods and Beverages for iAs [5]; AOAC SMPR 2014.004 Minerals and Trace Elements in Infant Formula and Adult/Pediatric Nutritional Formula for Ca, K, Mg, and Na [6]; and AOAC SMPR 2012.008 Iodine in Infant Formula and Adult/Pediatric Nutritional Formula for I [7]. A suitable SMPR for evaluation of performance for methods determining S was not identified. The method performance requirements for cannabis were used when available for an element as the matrix was deemed a more appropriate proxy versus beverages and infant formula. Repeatability, demonstrated by within-laboratory variability, and reproducibility, demonstrated by between-laboratory variability, are discussed in the individual sections.

Most laboratories indicated use of microwave digestion as the sample preparation approach and inductively coupled plasma mass spectrometry (ICP-MS) as the analytical technique for determination of elements in kelp samples. Sulfur was an exception, with most laboratories indicating use of inductively coupled plasma optical emission spectrometry (ICP-OES) as the analytical technique. Individual sample preparation and analytical method statistics for each measurand are described in the individual element results sections. The tables summarize the reported sample preparation and analytical methods averaged for all samples, simplifying the methods. For example, microwave digestion includes aqueous, acid-assisted, and base-assisted techniques while ICP-MS includes liquid chromatography ICP-MS (LC ICP-MS), isotope dilution ICP-MS (ID-ICP-MS), ICP-MS in kinetic energy discrimination (KED) mode, etc.

In addition to the overall recommendations made in Section 2, a few key recommendations should be highlighted for determination of elements in kelp. Sample preparation methods should be well validated prior to analyzing unknown samples. Established quality control materials (SRMs, CRMs, RMs, and in-house materials when not commercially available) and established methods of analysis should be used whenever possible. Larger than expected within-laboratory variability may be due to challenges in sample preparation, sample processing errors, or the use of smaller than recommended sample sizes for analysis. Also, laboratories reporting low values for all three samples should look for calibration issues or incomplete sample digestion.

When using ICP-MS, laboratories should ensure proper use of the instrumental parameters and features. Many ICP-MS instruments run in pulse counting mode, which is more sensitive than analog mode. Instruments typically switch automatically between pulse counting and analog modes depending on the dynamic range and instrument sensitivity for the analyte, and therefore the instrument must be calibrated for both modes. To ensure that the calibration curve is linear in the pulse mode, a narrower range of calibration points should be used and all solutions should be diluted to fall within this lower range. When using ICP-OES, monitoring more than one wavelength for each analyte helps identify interferences or background shifts due to matrix effects at a given wavelength but also helps identify and prevent bias. Collision cell or reaction cell mode can be used to reduce or eliminate the interferences caused by molecular ions that have the same mass-to-charge ratio as the element of interest isotope. Laboratories should also be aware of carryover issues and use longer washout times between samples if required (i.e., Hg).

3.4.1. Total Arsenic (tAs)

Target values for tAs are summarized in **Table 3-3**. The target value for tAs in Kelp A was determined at NIST using nitric and hydrofluoric acid assisted microwave digestion and ICP-MS. The target value for tAs in Kelp B was determined by combining results from HAMQAP Exercise 1 [8] and values determined in the same manner as those for Kelp A. The target value for tAs in SRM 3232 was determined at NIST using (1) nitric acid assisted microwave digestion and ICP-MS measurements and (2) a methanol extraction and liquid chromatography followed by offline Instrumental Neutron Activation Analysis (INAA) determination.

Table 3-3. Summary of results and laboratory variabilities for tAs in kelp.

Total Arsenic (tAs)			
Within-Laboratory Variability (% RSD)	Averaged for all Samples		Maximum
	3.6 %		29 %
	Kelp A	Kelp B	SRM 3232
Between-Laboratory Variability (% RSD)	9 %	12 %	11 %
Target Value ± U_{95} Mass Fraction (µg/g)	62.78 ± 0.69	27.5 ± 1.0	35.9 ± 1.2
Consensus Mean ± SD Mass Fraction (µg/g)	65.3 ± 6.1	28.2 ± 3.3	35.5 ± 4.0

For the determination of tAs, 32 of the 53 laboratories requesting samples reported results, for a participation rate of 60 %. Within-laboratory variabilities for most laboratories were at or below 3.6 %, with only six of the 32 laboratories greater than the published requirement of 7.3 % which demonstrates that most participants' in-house methods achieve successful repeatability[4]. The between-laboratory variability was at or below 12 % for all samples, as seen in **Table 3-3**, which is slightly outside the published recommendation (8 %, [4]). The levels of tAs targeted in this study were higher than those in the SMPR (at or below 10 µg/g), which should make the recommended reproducibility more achievable. However, SMPRs are designed to evaluate the reproducibility of a single method being used in multiple laboratories, and in this study, laboratories were not using identical protocols. Considering all of these factors, the overall between-laboratory variability of 12 % or less for tAs is reasonable and acceptable for this study. As noted previously, the higher levels of tAs in the kelp materials may result in calibration bias if appropriate sample preparation steps (i.e., dilution) are not taken to ensure the as-measured sample concentration falls within the calibration range.

More than half of laboratories reported using microwave digestion as their sample preparation method for tAs as shown in **Table 3-4**. Most laboratories reported using ICP-MS as their analytical method, with one laboratory using Triple Quadrapole ICP-MS (QQQ-ICP-MS). No definitive method bias was observed although 31 % of laboratories did not report a sample preparation method. Two laboratories used ICP-OES, including A024 (off scale in **Fig. 3-1**, **3-2**, and **3-3**), and these results were on the lower half of the consensus for all three materials. One laboratory used radiochemical neutron activation analysis (RNAA) and the results were on the upper half of the consensus for all three materials.

Table 3-4. Summary of sample preparation and analytical methods averaged across all samples for determination of tAs in kelp.

Sample Preparation		Analytical Method	
Acid Digestion (no heat or microwave)	10 %	ICP-MS	82 %
Hot Block Digestion	3 %	ICP-OES	6 %
Microwave Digestion	56 %	QQQ-ICP-MS	3 %
None Reported	31 %	RNAA	3 %
		Other/None Reported	6 %

The consensus mean was above the target value for Kelp A, as shown in **Fig. 3-1**. The consensus mean was close to the target value, as seen in **Fig. 3-2** and **3-3** for Kelp B and SRM 3232, respectively. The confidence interval for the consensus mean was within the target range for Kelp B and SRM 3232 and overlapped on the upper range of the target range for Kelp A. The high bias observed for the community in measurement of tAs in Kelp A may have resulted from a unique interference found in the precise species or growing area of this material or the specific manner in which this material was treated during harvest, processing, and packaging prior to the study.

Approximately one quarter of laboratories reported values below both the consensus and target ranges for one or more samples. These laboratories should consider that arsenic is volatile and can be lost during sample preparation. In general, high temperatures in a closed system are required to ensure complete digestion of the materials prior to analysis for tAs. Laboratories that use open systems should consider instead a vigorous microwave digestion that should convert all volatile organoarsenic species to arsenic acid (AsV), after which point subsequent heating will not result in loss of As. Open beaker digestion may lead to low results due to loss of volatile As species. Closed-vessel digestions should be opened with care ensuring that no arsenic is lost because of inadvertent venting.

Additionally, approximately one third of laboratories reported values above both the consensus and target ranges for one or more samples. Collision cell technology can be used to minimize the molecular ion interferences that may be found when analyzing tAs in these materials. Dilution of samples can also assist in reducing matrix interferences while ensuring that the as-measured analyte concentrations are within the calibration range. Use of standard additions or matrix-matched calibration as well as analysis of appropriate blank samples will prevent misidentification of interferences as analyte signal.

Additional tables and figures for tAs in kelp are located in **Appendix B**.

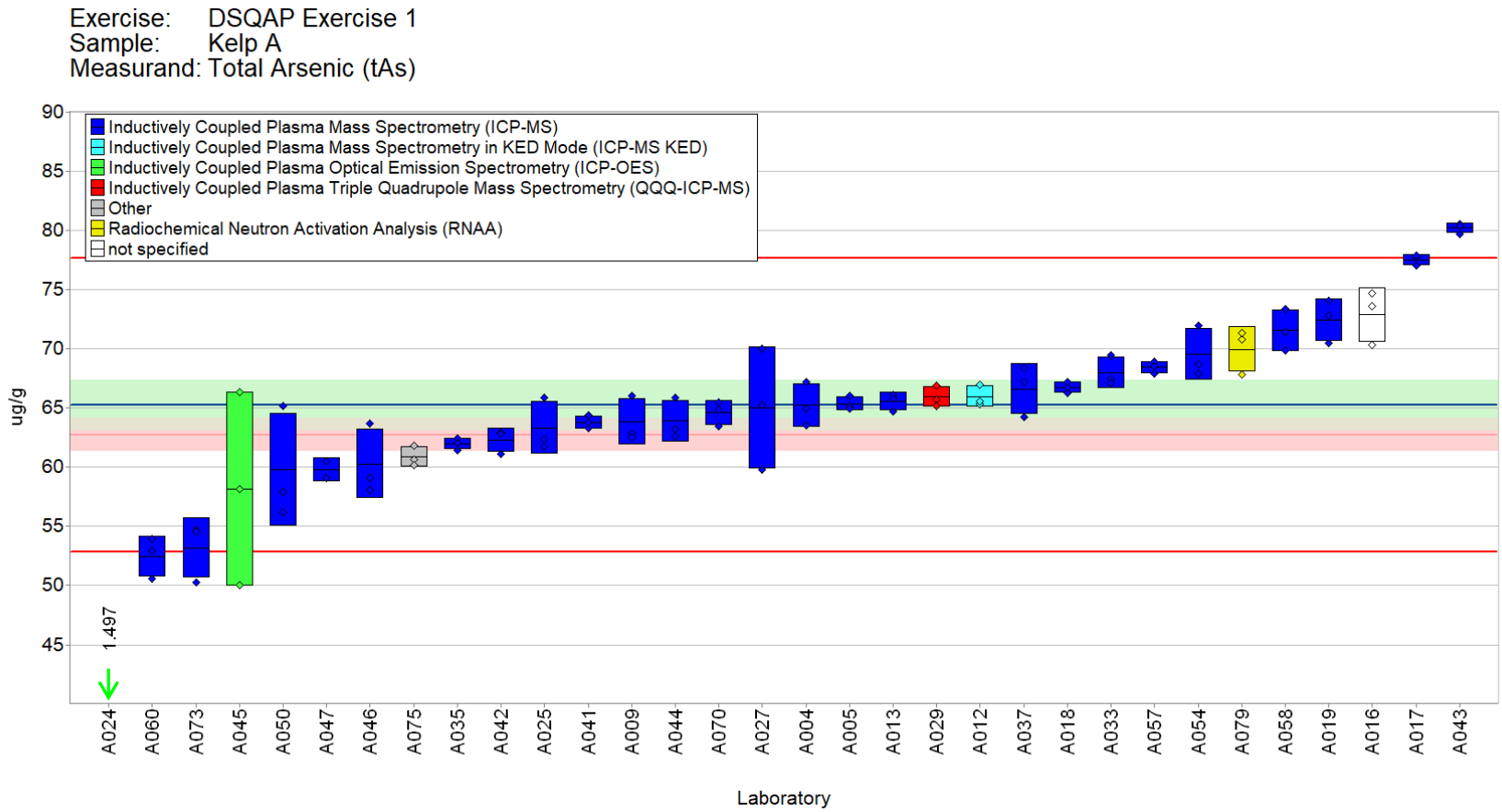

Fig. 3-1. Total arsenic in Kelp A (data summary view – analytical method).

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'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

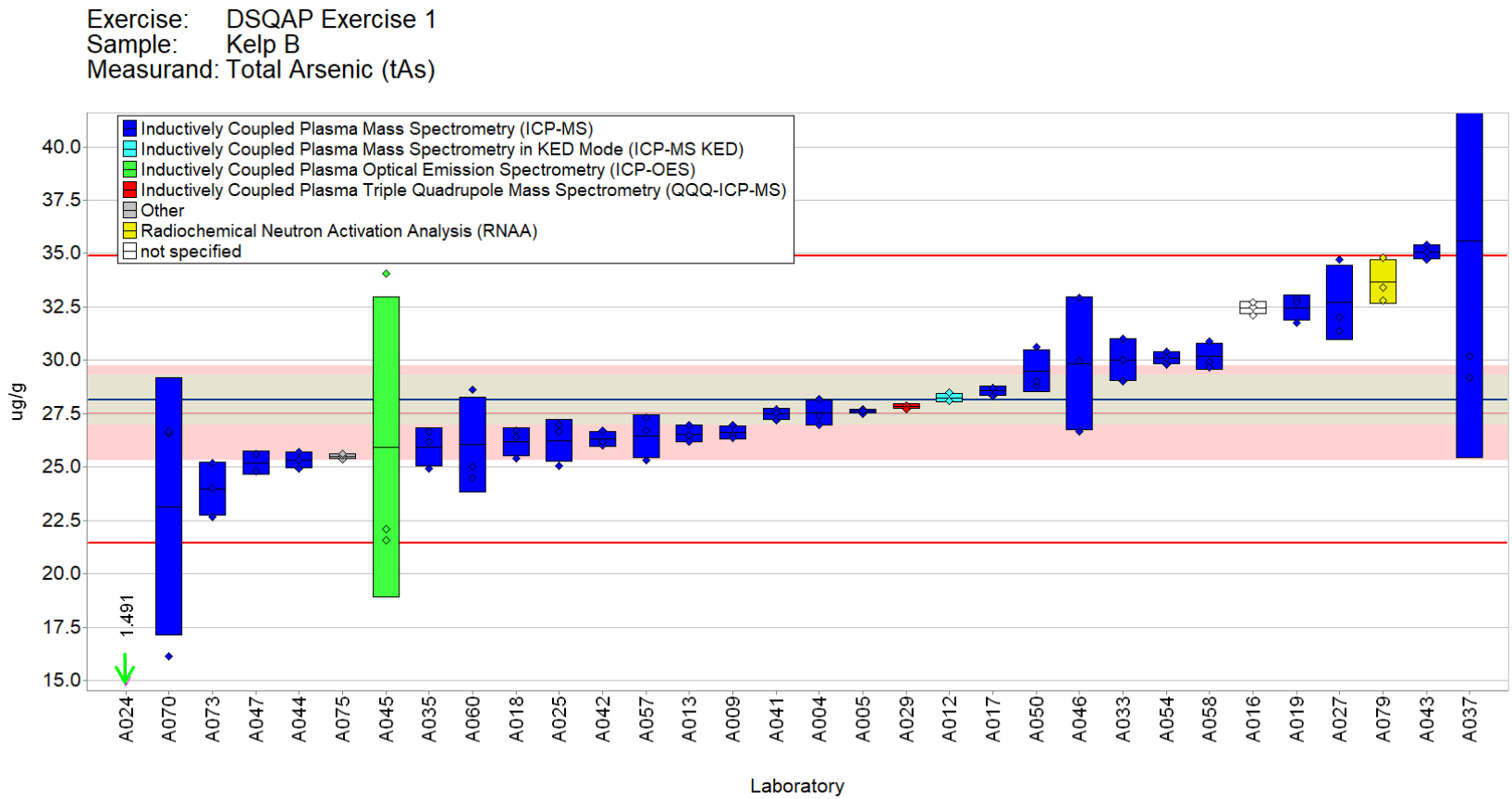

Fig. 3-2. Total arsenic in Kelp B (data summary view – analytical method).

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'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

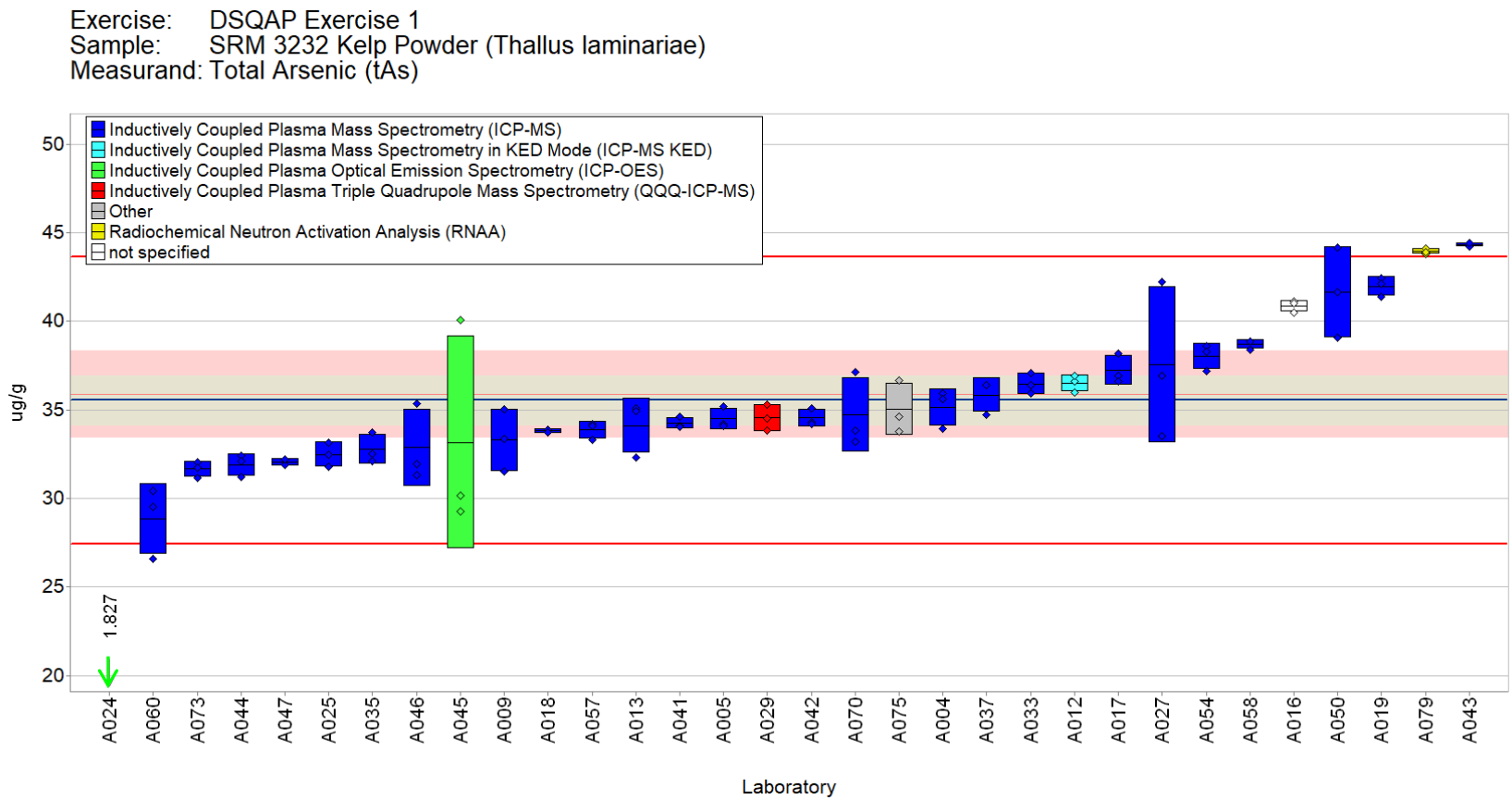

Fig. 3-3. Total arsenic in SRM 3232 Kelp Powder (*Thallus laminariae*) (data summary view – analytical method).

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'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

3.4.2. Inorganic Arsenic (iAs)

Target values for iAs are summarized in **Table 3-5**. The target value for iAs in Kelp A was determined at NIST using extraction followed by liquid chromatography ICP-MS (LC-ICP-MS) for analysis [9]. The target value for iAs in Kelp B was determined from HAMQAP Exercise 1 results [8]. The target value for iAs in SRM 3232 was determined at NIST using a methanol extraction procedure and liquid chromatography followed by offline INAA determination and online ICP-MS determination (LC-ICP-MS).

Table 3-5. Summary of results and laboratory variabilities for iAs in kelp.

Inorganic Arsenic (iAs)			
Within-Laboratory Variability (% RSD)	Averaged for all Samples		Maximum
	5.6 %		12 %
	Kelp A	Kelp B	SRM 3232
Between-Laboratory Variability (% RSD)	>100 %	>100 %	52 %
Target Value ± U_{95} Mass Fraction (µg/g)	0.168 ± 0.035	0.16 ± 0.19	0.092 ± 0.015
Consensus Mean ± SD Mass Fraction (µg/g)	0.19 ± 0.30	0.08 ± 0.15	0.125 ± 0.065

For the determination of iAs, only 7 of 40 participating laboratories returned results (17 %). All laboratories achieved within-laboratory variability within the published repeatability requirement of 13 % (**Table 3-5**) [5]. The 6 laboratories reporting quantitative results for iAs were in poor agreement, with between-laboratory variabilities over 100 % for two of the kelp samples. The SMPR specifies desirable reproducibility to be at or below 20 % for foods and beverages in this concentration range [5].

Three laboratories reported using hot block digestion as their sample preparation method while one laboratory reported using acid digestion (no heat or microwave) as seen in **Table 3-6**. Five laboratories reported using ICP-MS as their analytical method. **Figures 3-4**, **3-5**, and **3-6** show the spread of results with analytical method indicated by the color of the points. Due to the small data set no conclusions could be made regarding method bias, although the values obtained using LC-ICP-MS were consistently greater than the consensus mean and the values obtained using ICP-OES were consistently less than the consensus mean. The laboratories using these analytical methods should investigate whether their overall methods have biases not seen in other methods. The results off-scale in **Fig. 3-5** and **3-6** were obtained using LC-ICP-MS and are likely influenced by a calculation or reporting error.

Table 3-6. Summary of sample preparation and analytical methods averaged across all samples for determination of iAs in kelp.

Sample Preparation		Analytical Method	
Acid Digestion (no heat or microwave)	15 %	ICP-MS	70 %
Hot Block Digestion	45 %	ICP-OES	15 %
None Reported	40 %	AAS	15 %

The consensus mean for Kelp A was close to the target value and the confidence interval was overlapping the target range, as seen in **Fig. 3-4**. The consensus mean for Kelp B was below the target value and the confidence interval was within the target range, as seen in **Fig. 3-5**. The consensus mean for SRM 3232 was above the target value and the confidence interval was overlapping the upper range of the target range, as seen in **Fig. 3-6**.

In order to make useful interpretation of the data, more information regarding the sample preparation methods used by the participants is required, including extraction solvent type and duration. A mild extraction procedure should be used for iAs quantification, as harsh chemicals and extraction conditions promote conversion of As species which can lead to biases [9, 10]. Measurement methods should be reported correctly and completely. Laboratories might have incorrectly reported using ICP-MS or ICP-OES to determine iAs, as these are elemental detectors that cannot distinguish the species of an element without use of a separation technique.

Additional tables and figures for iAs in kelp are located in **Appendix B**.

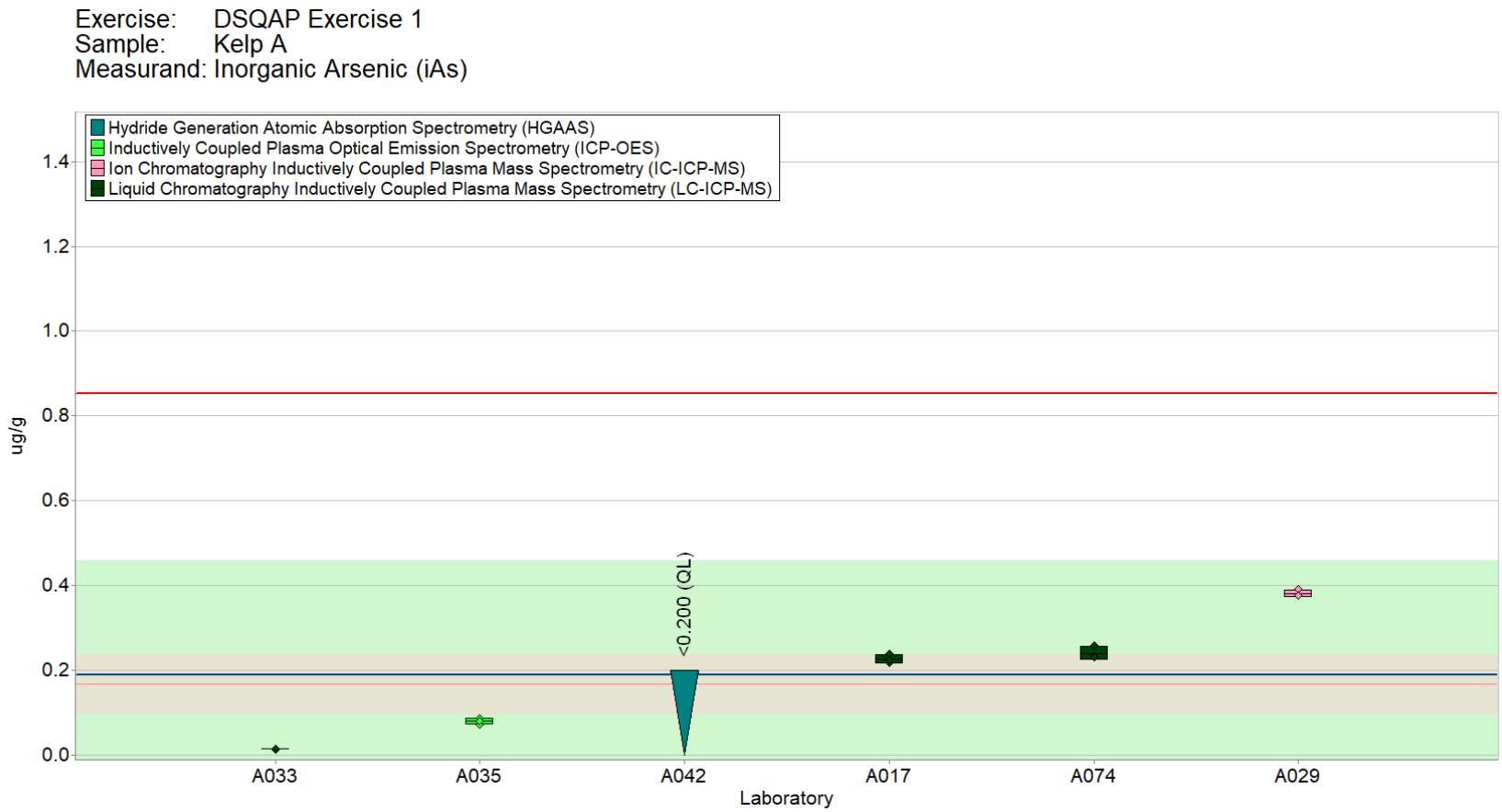

Fig. 3-4. Inorganic arsenic in Kelp A (data summary view – analytical method).

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 bound of the consensus range of tolerance, calculated as the value above the consensus mean that results in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$, with the lower bound set to zero. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

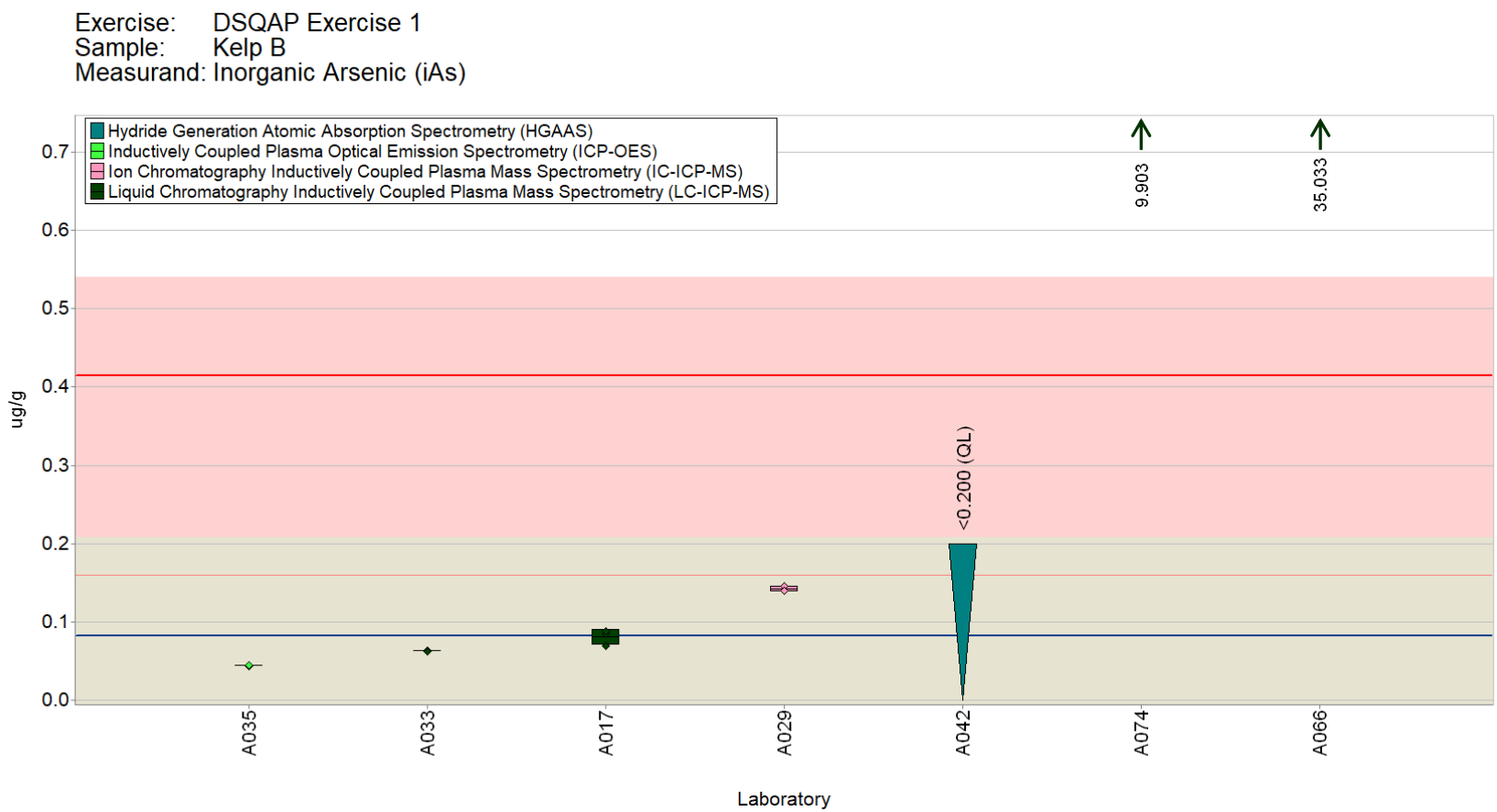

Fig. 3-5. Inorganic arsenic in Kelp B (data summary view – analytical method).

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 bound of the consensus range of tolerance, calculated as the value above the consensus mean that results in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$, with the lower bound set to zero. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

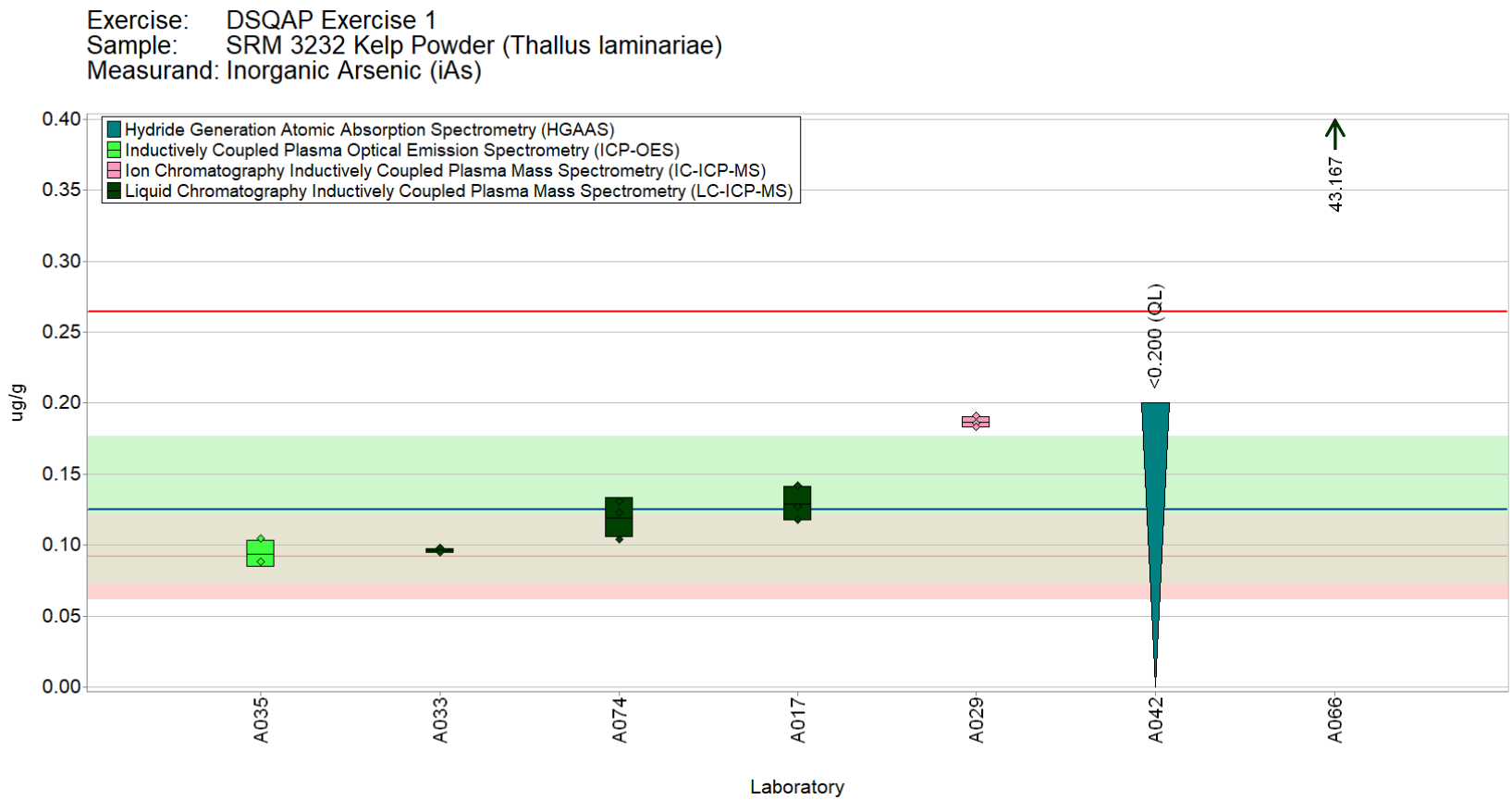

Fig. 3-6. Inorganic arsenic in SRM 3232 Kelp Powder (*Thallus laminariae*) (data summary view – analytical method).

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 bound of the consensus range of tolerance, calculated as the value above the consensus mean that results in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$, with the lower bound set to zero. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

3.4.3. Cadmium (Cd)

Target values for Cd are summarized in **Table 3-7**. The target values for Cd in Kelp A and Kelp B were determined at NIST using nitric acid assisted microwave digestion and ICP-MS. The target value for Cd in SRM 3232 was determined at NIST using nitric and hydrofluoric acid assisted microwave digestion and isotope dilution ICP-MS (ID-ICP-MS).

Table 3-7. Summary of results and variabilities for Cd in kelp.

Cadmium (Cd)			
Within-Laboratory Variability (% RSD)	Averaged for all Samples		Maximum
	5.1 %		59 %
	Kelp A	Kelp B	SRM 3232
Between-Laboratory Variability (% RSD)	9 %	8 %	11 %
Target Value ± U_{95} Mass Fraction (µg/g)	1.16 ± 0.02	0.38 ± 0.006	0.40 ± 0.008
Consensus Mean ± SD Mass Fraction (µg/g)	1.10 ± 0.10	0.36 ± 0.03	0.37 ± 0.04

For the determination of Cd, 34 of 53 laboratories reported results (64 %). Of the 33 laboratories reporting quantitative results, 28 laboratories reported within-laboratory variabilities within the published requirements of 7.3 % for mass fractions above 1 µg/g and 11 % for mass fractions below 1 µg/g [4] in all kelp samples (**Table 3-7**). The average within-laboratory variability was 5.1 %, which demonstrates that most participants achieved acceptable repeatability for determination of Cd using their in-house methods. The between-laboratory variabilities were at or below 11 % for all samples, generally consistent with the published reproducibility recommendations of at or below 8 % for mass fractions above 1 µg/g and at or below 16 % for mass fraction below 1 µg/g [4].

More than half of the laboratories that reported a sample preparation method indicated using microwave digestion (**Table 3-8**). Most of the laboratories reported using ICP-MS as their analytical method. No method bias was observed, although 32 % of laboratories did not report a sample preparation method.

Table 3-8. Summary of sample preparation and analytical methods averaged across all samples for determination of Cd in kelp.

Sample Preparation		Analytical Method	
Acid Digestion (no heat or microwave)	9 %	ICP-MS	82 %
Hot Block Digestion	3 %	ICP-OES	6 %
Microwave Digestion	56 %	QQQ-ICP-MS	3 %
None Reported	32 %	RNAA	3 %
		Other/None Reported	6 %

The consensus mean for Cd was outside of the target range and the confidence interval was overlapping the lower range of the target range for all three kelp samples, as seen in **Fig. 3-7**, **3-8**, and **3-9** for Kelp A, Kelp B, and SRM 3232, respectively.

Laboratories that consistently report low values should evaluate their sample preparation method to determine whether complete extraction of Cd from the materials is being achieved. A high temperature in a closed vessel system is suggested to ensure a complete digestion of the sample.

Laboratories that consistently report values above the target range should consider potential interferences. Spectral/isobaric interferences can make Cd difficult to measure accurately by ICP-MS. Presence of certain elements (e.g., Mo, Sn, or Zr) in samples is known to cause interferences in the analysis of Cd by ICP-MS. Isobaric spectral interferences such as $^{95}Mo^{16}O^{+}$ and $^{97}Mo^{16}O^{+}$ can affect the accuracy of Cd determination at *m*/*z* 111 and *m*/*z* 113 by ICP-MS and usually result in biasing the results above the true value. Most ICP-MS instruments allow an elemental survey of the sample prior to the measurement of analytes of interest without the need for calibration standards. Such a scan of the sample before analysis will help to identify any potential interferences in the sample that will need to be addressed.

Laboratory A079 reported LOQ values above the consensus range of tolerance for all three materials when using RNAA. SMPR 2020.001 recommends that methods for Cd should achieve an LOQ of 0.1 ug/kg [4], which is sufficiently low to measure Cd in the kelp materials in this study.

Additional tables and figures for Cd in kelp are located in **Appendix B**.

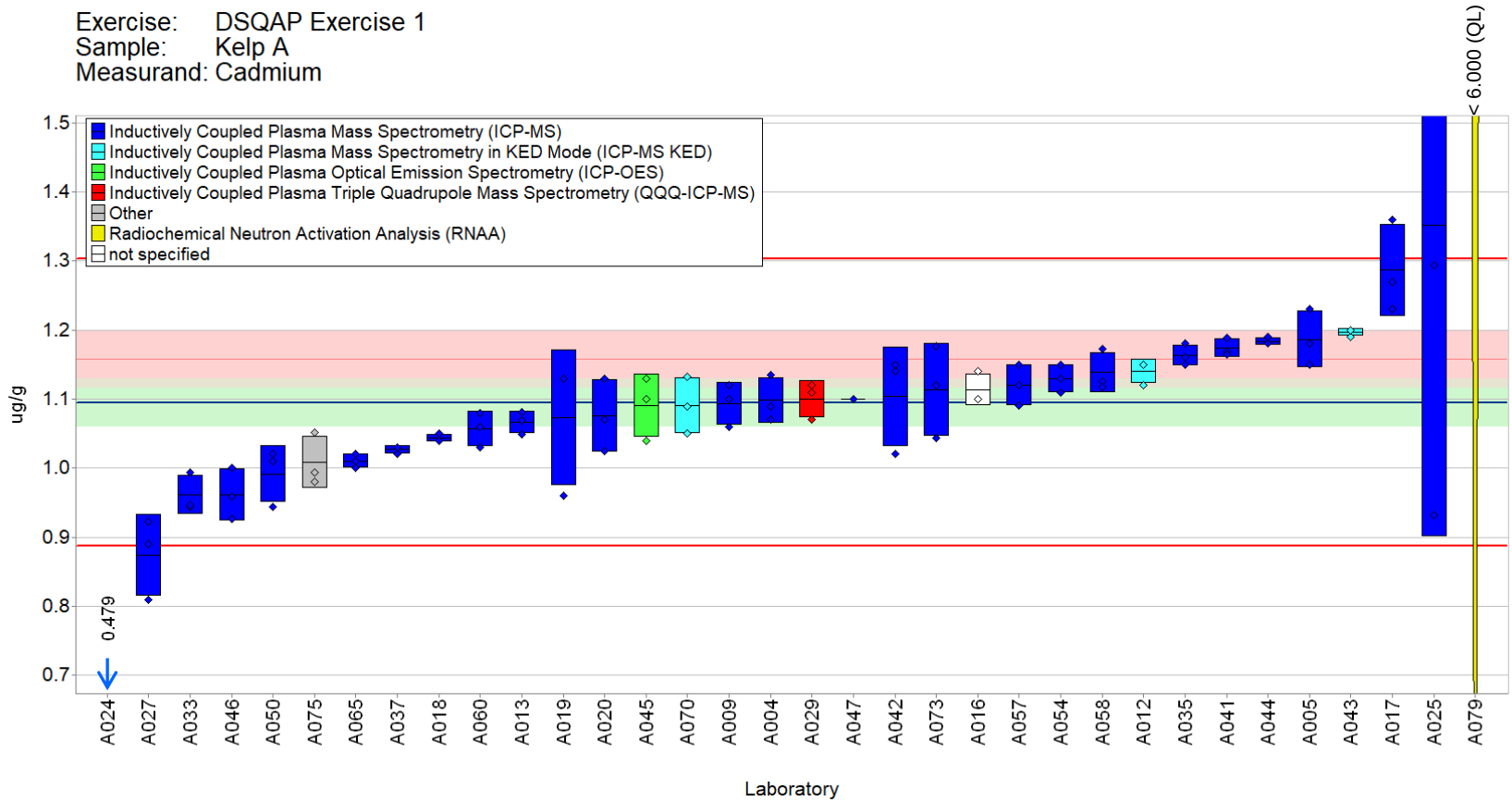

Fig. 3-7. Cadmium in Kelp A (data summary view – analytical method).

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'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

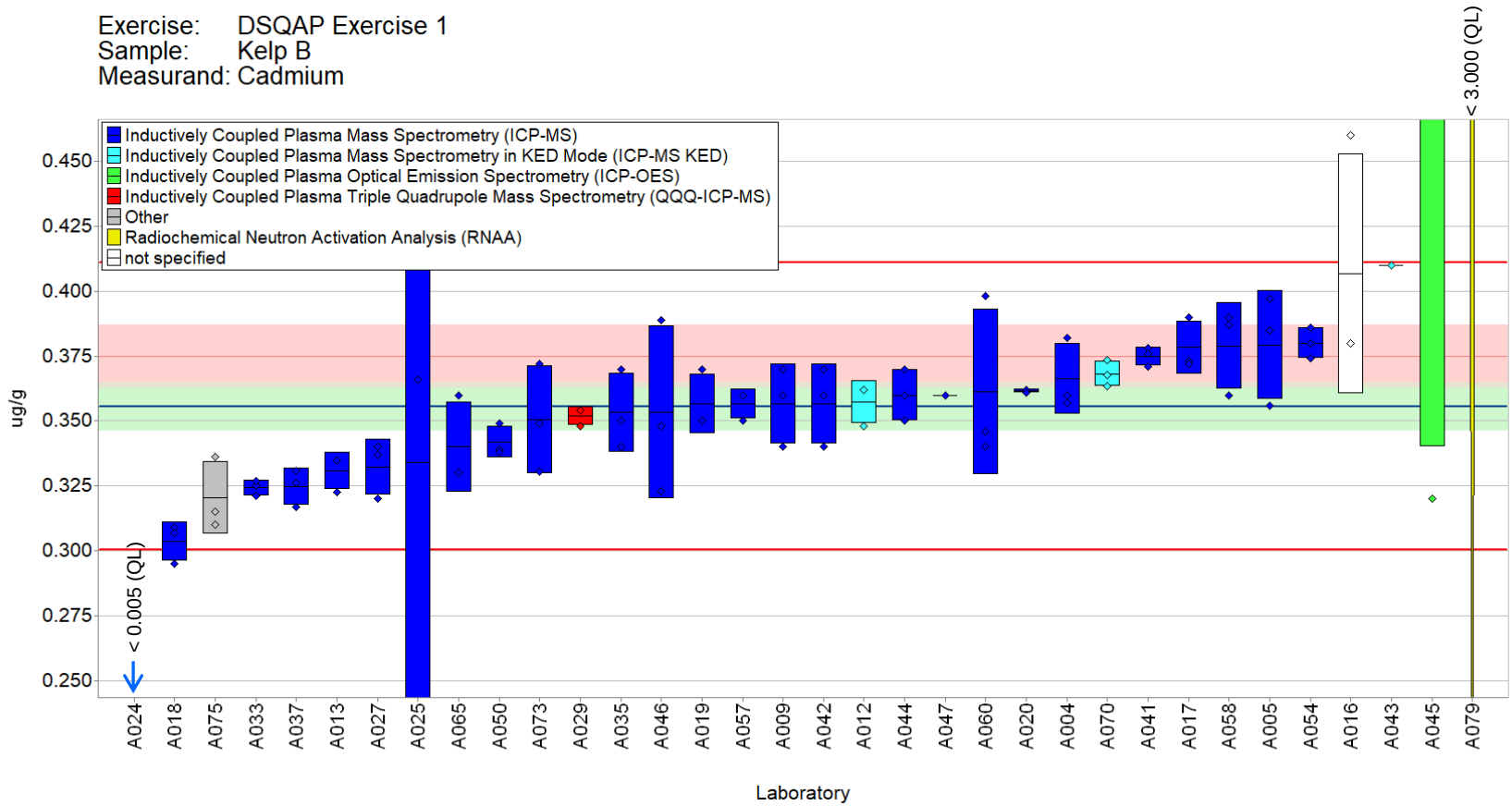

Fig. 3-8. Cadmium in Kelp B (data summary view – analytical method).

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'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

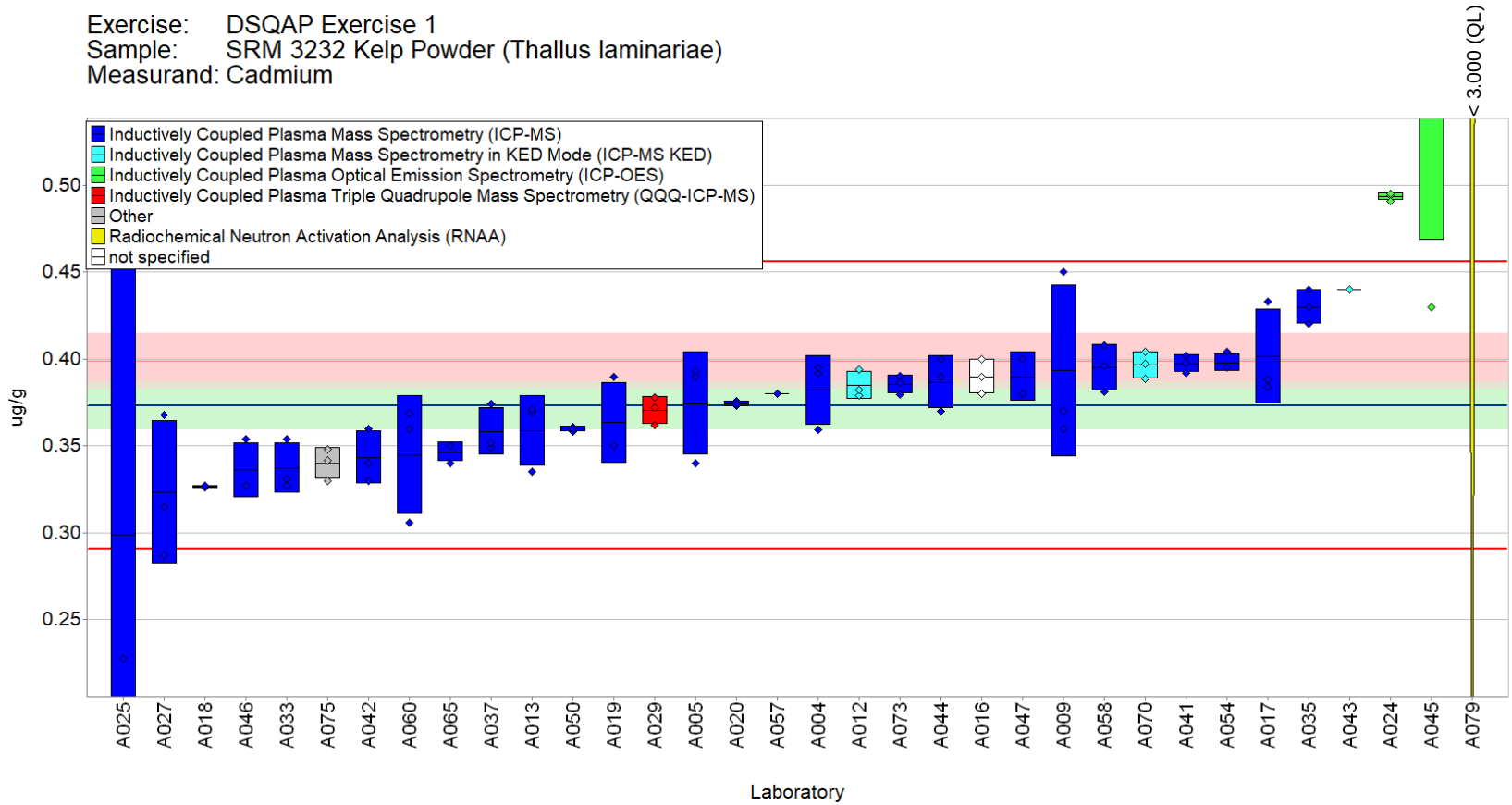

Fig. 3-9. Cadmium in SRM 3232 Kelp Powder (*Thallus laminariae*) (data summary view – analytical method).

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'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

3.4.4. Calcium (Ca)

Target values for Ca are summarized in **Table 3-9**. The target values for Ca in Kelp A and Kelp B were determined at NIST using nitric and hydrofluoric acid assisted microwave digestion and ICP-OES. The target value for Ca in SRM 3232 was determined at NIST using (1) INAA after the material was pressed into pellets using a stainless-steel die and (2) nitric acid assisted microwave digestion and ICP-OES.

Table 3-9. Summary of results and laboratory variabilities for Ca in kelp.

Calcium (Ca)			
Within-Laboratory Variability (% RSD)	Averaged for all Samples		Maximum
	4.1 %		74 %
	Kelp A	Kelp B	SRM 3232
Between-Laboratory Variability (% RSD)	18 %	14 %	14 %
Target Value ± U_{95} Mass Fraction (mg/g)	9.22 ± 0.57	12.20 ± 0.19	11.49 ± 0.64
Consensus Mean ± SD Mass Fraction (mg/g)	10.0 ± 1.8	13.2 ± 1.9	12.4 ± 1.7

For the determination of Ca, 34 of 51 laboratories reported results (67 %). The average within-laboratory variability was 4.1 % (**Table 3-9**) with only seven laboratories above 5 %, which indicates that most participants' in-house methods achieve repeatability consistent with the published requirement (5 % [6]). At least one laboratory reported variability greater than 50 %, and these results should be assessed for any calculation and reporting errors. The between-laboratory variabilities ranged from 14 % to 18 % for the kelp samples, which is greater than the published requirement of 10 % for multiple laboratories using the same method [6]. Notably, the Ca levels in the kelp materials were greater than the upper analytical range of the SMPR (0.0016 mg/g), and higher levels generally result in community results with greater agreement. To ensure samples are within the calibration range, however, appropriate sample preparation steps (i.e., dilution) should be taken for higher concentrations of Ca.

Nearly half of the laboratories that reported their sample preparation method indicated using microwave digestion as seen in **Table 3-10**. Most laboratories reported using either ICP-MS or ICP-OES as their analytical method. A positive bias for ICP-MS was observed in the Ca data from several laboratories. No sample preparation method bias was observed although 32 % of laboratories did not report a method.

Table 3-10. Summary of sample preparation and analytical methods averaged across all samples for determination of Ca in kelp.

Sample Preparation		Analytical Method	
Acid Digestion (no heat or microwave)	9 %	ICP-MS	47 %
Hot Block Digestion	9 %	ICP-OES	39 %
Microwave Digestion	45 %	QQQ-ICP-MS	3 %
Open Beaker Digestion	3 %	ICP	3 %
Thermal Decomposition	2 %	RNAA	3 %
None Reported	32 %	Other/None Reported	6 %

The consensus mean was above the target value for all three kelp samples, as seen in **Fig. 3-10**, **3-12**, and **3-14** for Kelp A, Kelp B, and SRM 3232, respectively. All results off-scale in these figures were obtained using ICP-MS. The consensus confidence interval was overlapping the upper range of the target range for both Kelp A and SRM 3232 and was above the target range for Kelp B. Most laboratories were able to successfully measure Ca in SRM 3232 but had more difficulties measuring it in Kelp A and Kelp B.

In all materials, more than 40 % of the ICP-MS reported values were above the target range, as seen in **Fig. 3-10**, **3-12**, and **3-14**, suggesting a potential positive bias for many users of this analytical method. The kernel density estimation (KDE) plots shown in **Fig. 3-11**, **3-13**, and **3-15** for Kelp A, Kelp B, and SRM 3232, respectively, further demonstrate this high bias for Ca results measured using ICP-MS. In the KDE plot, the blue line represents the distribution of ICP-MS results as compared to results from all other reported analytical methods (black line). Although a high bias is indicated, the same two laboratories with results that are off-scale and significantly lower than the consensus mean in **Fig. 3-10**, **3-12**, and **3-14** also reported using ICP-MS. These laboratories should take care to ensure appropriate units and calculations are used, as well as check for any calibration and sample preparation issues.

Spectral/isobaric interferences can make Ca difficult to measure accurately by ICP-MS. High mass fractions of certain elements (e.g., Ar) are known to cause interferences in the analysis of Ca by ICP-MS. Isobaric spectral interferences such as $^{40}Ar^{+}$, $^{12}C^{16}O_2$, and $^{14}N_2{}^{16}O$ are common, with $^{40}Ar^{+}$ being the largest potential interference. Collision cell technology can be used to minimize molecular interferences that may be found in these three materials. If ^{44}Ca is the atomic mass measured, He should be used as the collision gas. If ^{40}Ca is the atomic mass measured, H_2 should be used as the collision gas.

Additional tables and figures for Ca in kelp are located in **Appendix B**.

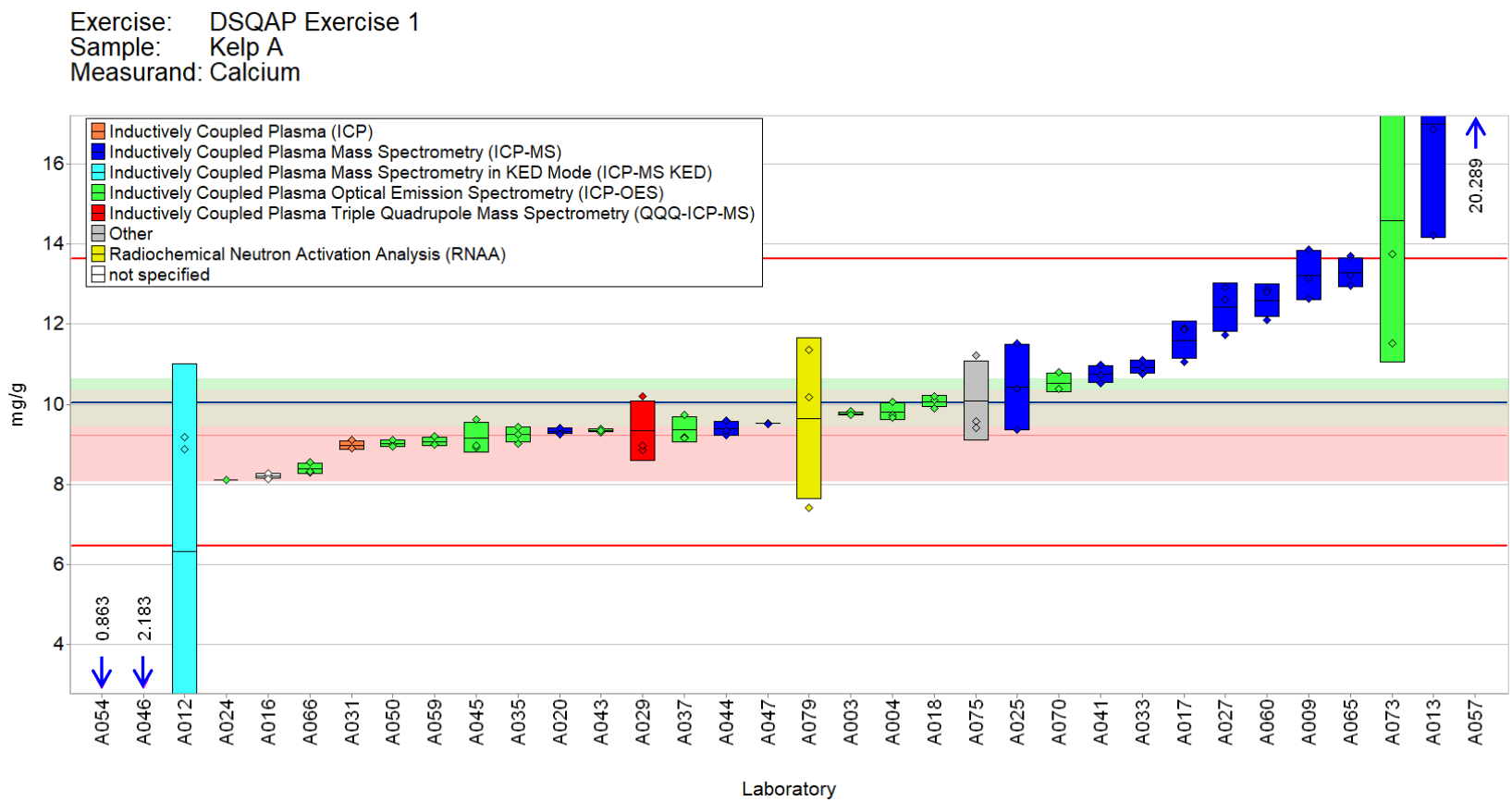

Fig. 3-10. Calcium in Kelp A (data summary view – analytical method).

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'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

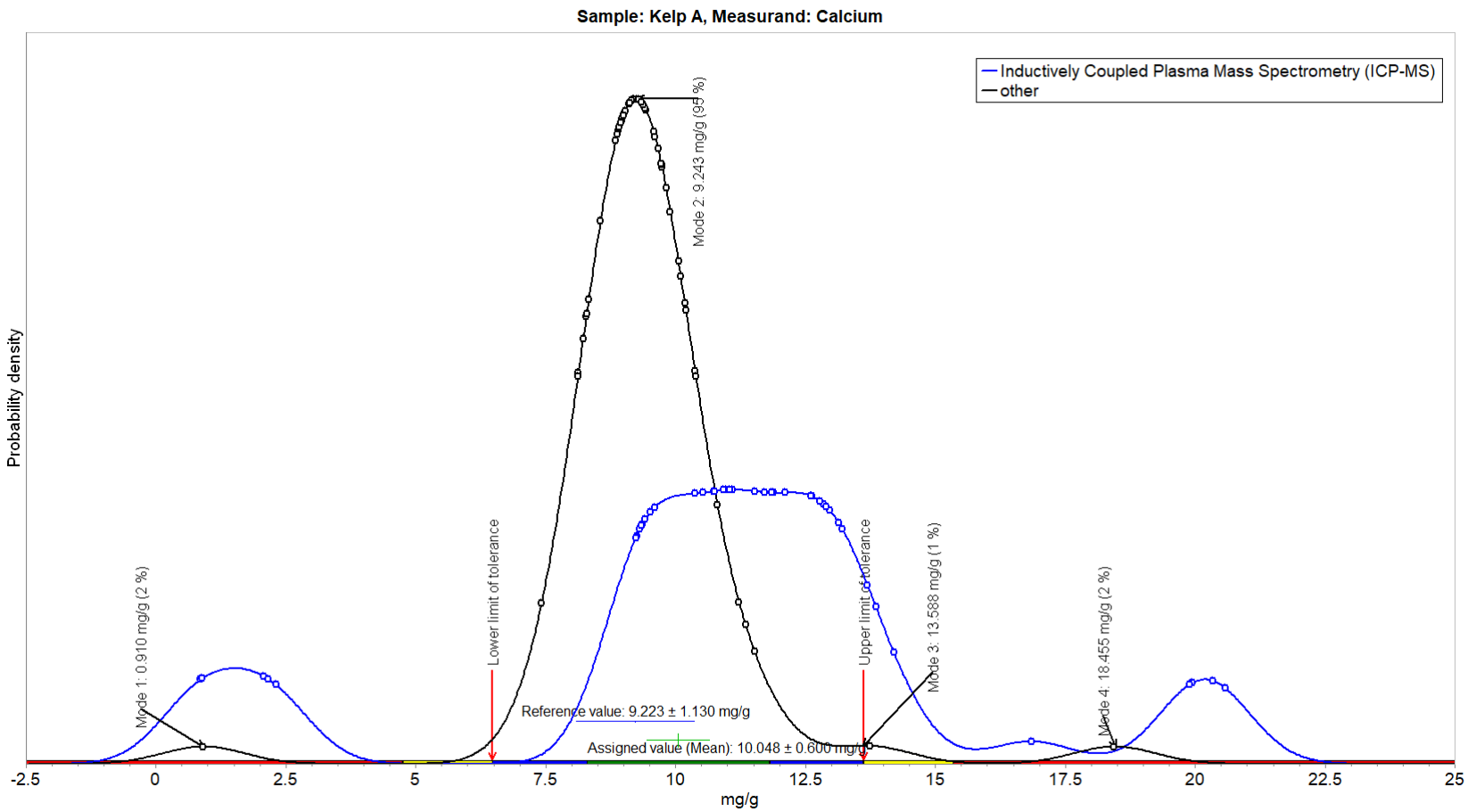

Fig. 3-11. Kernel density estimation for calcium in Kelp A.

In this view, the kernel density of the distribution is estimated as a function of a single method selection (ICP-MS, solid blue line) compared to the estimated distribution from other reported results (solid black line). The target values are shown as the upper blue horizontal bars, and the consensus means are indicated by the lower green horizontal bars. Upper and lower limits of tolerance are indicated by red arrows.

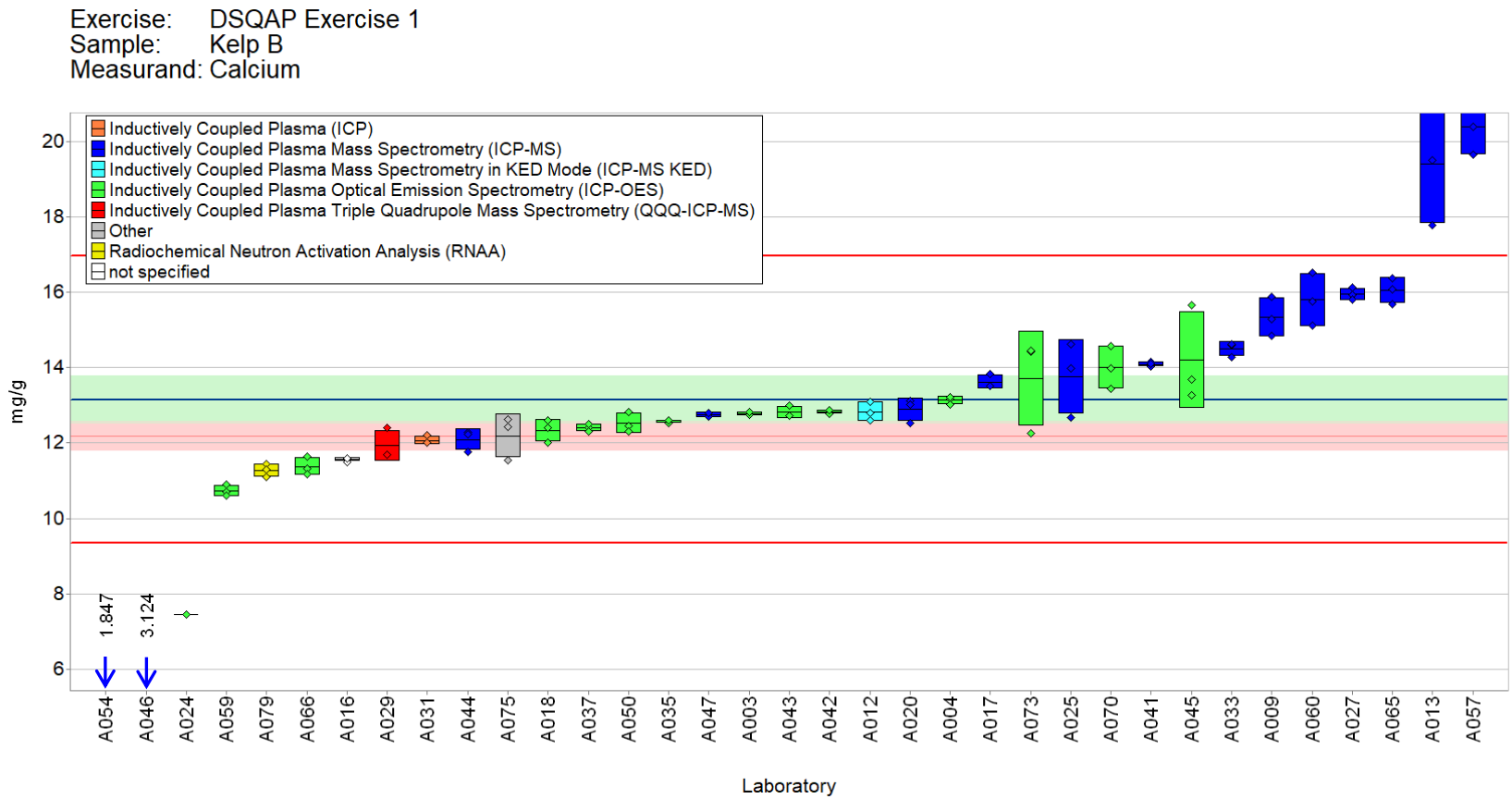

Fig. 3-12. Calcium in Kelp B (data summary view – analytical method).

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

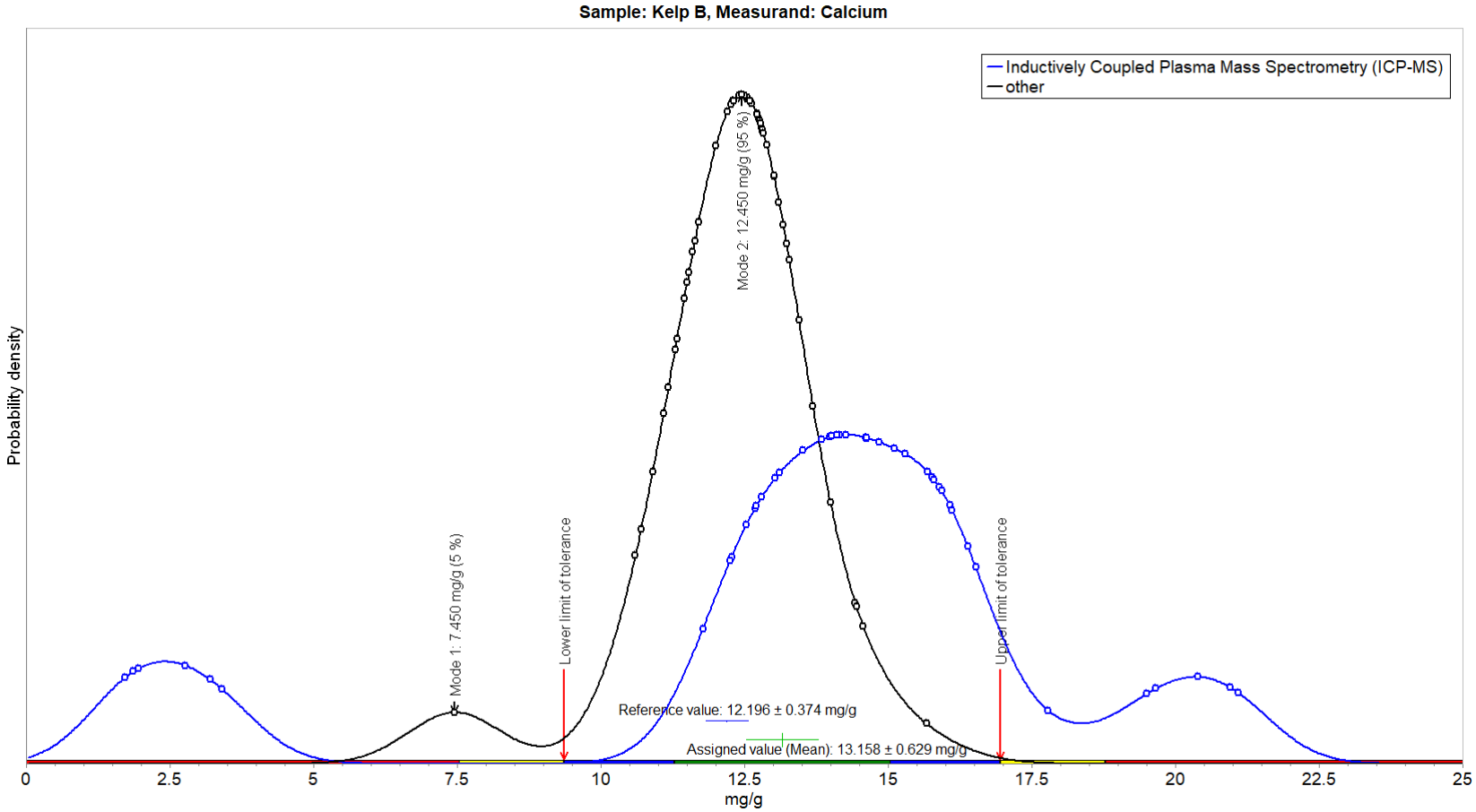

Fig. 3-13. Kernel density estimation for calcium in Kelp B.

In this view, the kernel density of the distribution is estimated as a function of a single method selection (ICP-MS, solid blue line) compared to the estimated distribution from other reported results (solid black line). The target values are shown as the upper blue horizontal bars, and the consensus means are indicated by the lower green horizontal bars. Upper and lower limits of tolerance are indicated by red arrows.

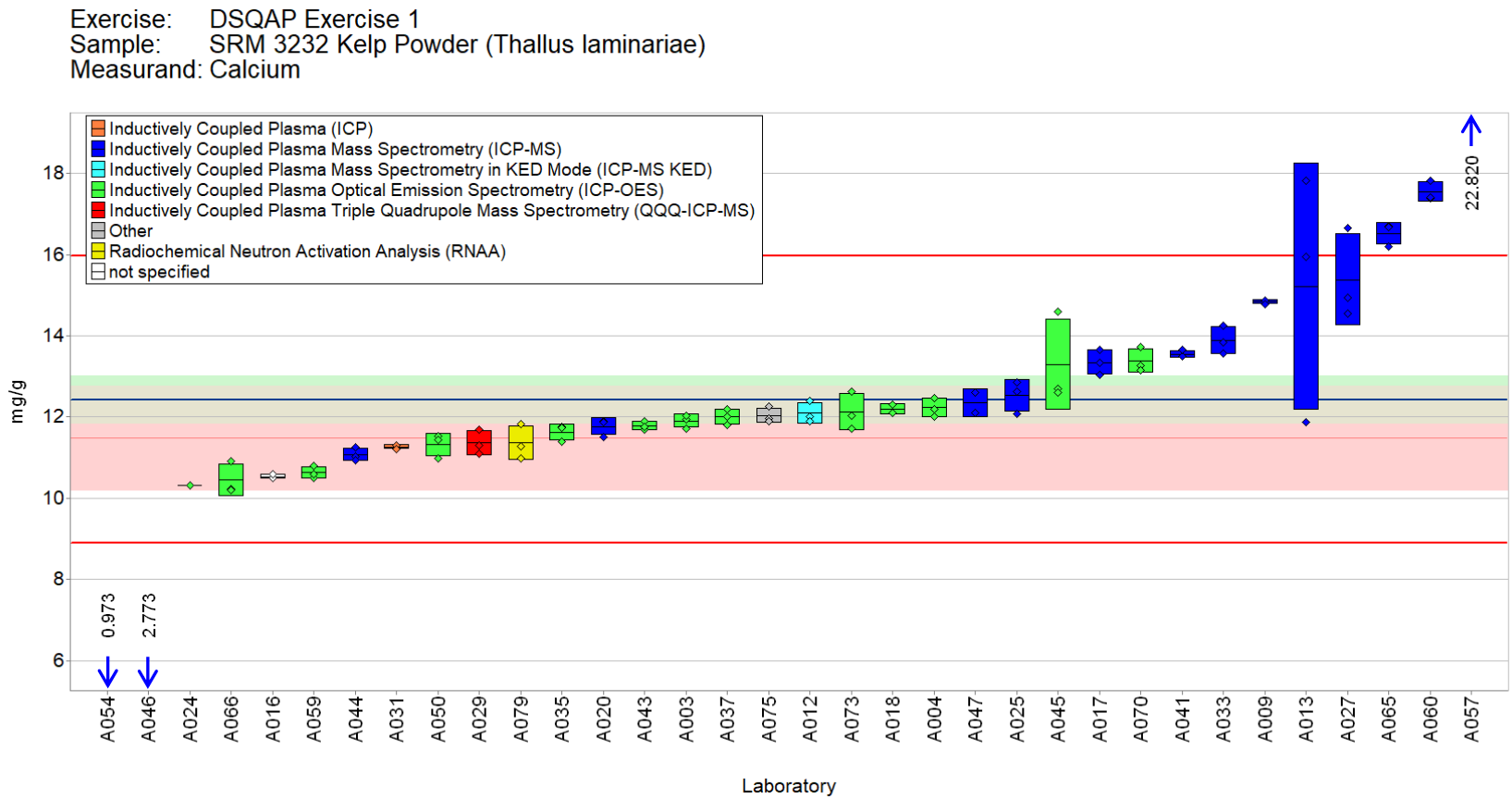

Fig. 3-14. Calcium in SRM 3232 Kelp Powder (*Thallus laminariae*) (data summary view – analytical method).

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'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

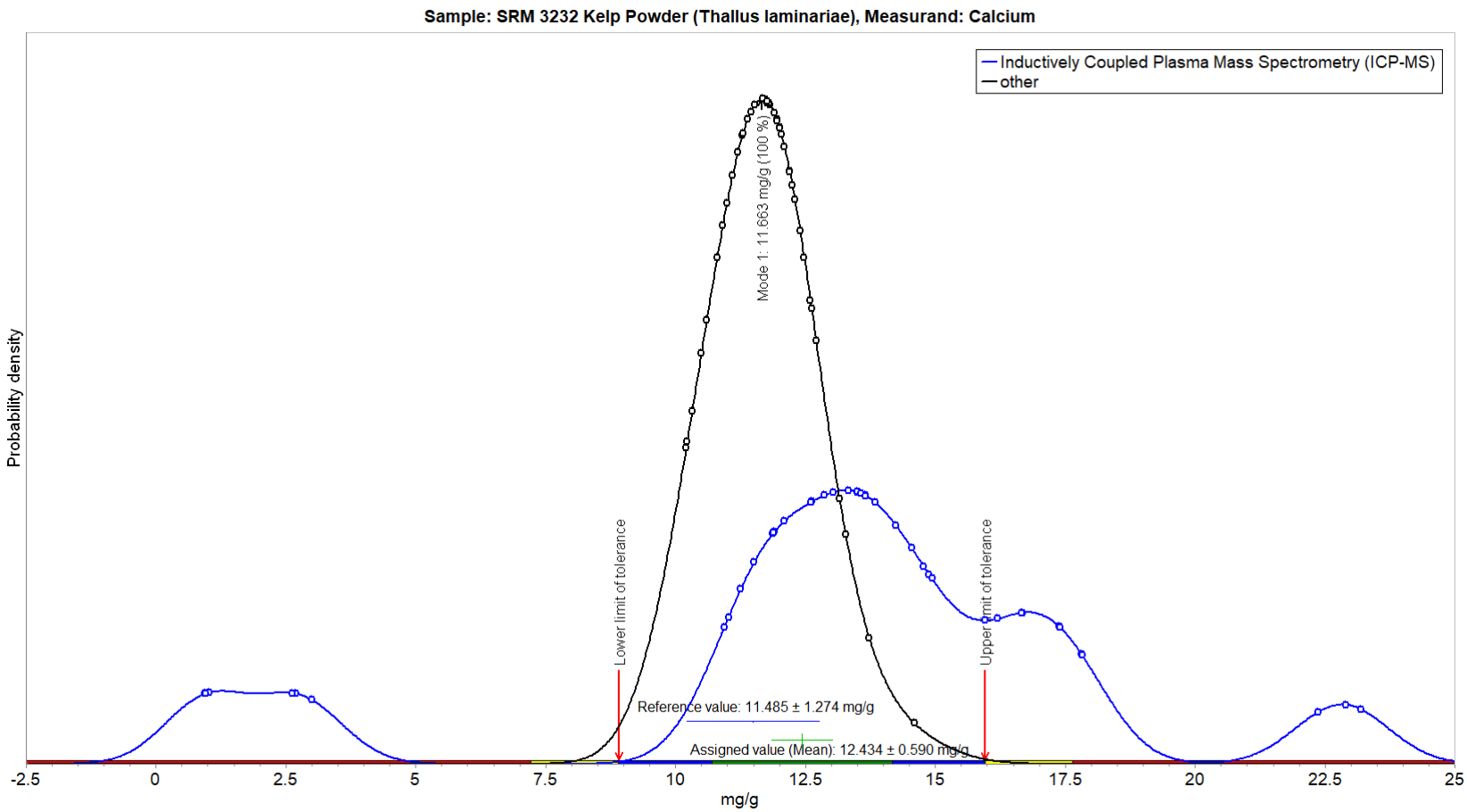

Fig. 3-15. Kernel density estimation for calcium in SRM 3232.

In this view, the kernel density of the distribution is estimated as a function of a single method selection (ICP-MS, solid blue line) compared to the estimated distribution from other reported results (solid black line). The target values are shown as the upper blue horizontal bars, and the consensus means are indicated by the lower green horizontal bars. Upper and lower limits of tolerance are indicated by red arrows.

3.4.5. Chromium (Cr)

Target values for Cr are provided in **Table 3-11**. The target values for Cr in Kelp A and Kelp B were determined at NIST using nitric acid assisted microwave digestion and ICP-MS. The target value for Cr in SRM 3232 was determined at NIST using nitric and hydrofluoric acid assisted microwave digestion and ID-ICP-MS.

Table 3-11. Summary of results and laboratory variabilities for Cr in kelp.

Chromium (Cr)			
Within-Laboratory Variability (% RSD)	Averaged for all Samples		Maximum
	11 %		>100 %
	Kelp A	Kelp B	SRM 3232
Between-Laboratory Variability (% RSD)	32 %	35 %	32 %
Target Value ± U_{95} Mass Fraction (µg/g)	4.36 ± 0.13	0.546 ± 0.030	5.55 ± 0.48
Consensus Mean ± SD Mass Fraction (µg/g)	3.9 ± 1.2	0.51 ± 0.16	5.0 ± 1.5

For the determination of Cr, 31 of 50 laboratories reported results (61 %). Of the 31 laboratories reporting quantitative results, only 15 laboratories reported within-laboratory variabilities within the published requirements of 7.3 % for mass fractions above 1 µg/g [4] which is represented by Kelp A and SRM 3232 (**Table 3-11**); however, the average within-laboratory variability between these two materials was 11 %. Only five laboratories reported within-laboratory variabilities for Kelp B above the published requirements of 11 % for mass fractions below 1 µg/g [4]. At least one laboratory reported variability greater than 100 %, and these results should be assessed for any calculation and reporting errors. The between-laboratory variabilities ranged from 32 % to 35 % for the analysis of Cr, which were more than twice the published requirements of at or below 8 % for mass fractions above 1 µg/g and at or below 16 % for mass fractions below 1 µg/g [4].

Half of the laboratories that reported a sample preparation method indicated using microwave digestion as shown in **Table 3-12**. Most laboratories reported using ICP-MS as their analytical method, including the results that are off scale in **Fig. 3-17**. No method bias was observed although 35 % of laboratories did not report a sample preparation method.

Table 3-12. Summary of sample preparation and analytical methods averaged across all samples for determination of Cr in kelp.

Sample Preparation		Analytical Method	
Acid Digestion (no heat or microwave)	7 %	ICP-MS	74 %
Hot Block Digestion	7 %	ICP-OES	13 %
Microwave Digestion	50 %	QQQ-ICP-MS	3 %
Thermal Decomposition	1 %	RNAA	3 %
None Reported	35 %	Other/None Reported	7 %

The consensus mean was below the target values for all three kelp samples, as seen in **Fig. 3-16**, **3-17**, and **3-18** for Kelp A, Kelp B, and SRM 3232, respectively. The consensus confidence interval is slightly overlapping the lower range of the target range for Kelp A and is overlapping most of the target range for Kelp B and SRM 3232. Most laboratories were able to successfully measure Cr in SRM 3232, but about half of laboratories were outside the target range for both Kelp A and Kelp B.

A group of laboratories consistently reported values higher than the target (**Fig. 3-16**, **3-17**, and **3-18**). Laboratories reporting high values for Cr when using ICP-MS should be aware of spectral/isobaric interferences that can make Cr difficult to measure accurately. High mass fraction of certain elements (e.g., Ar, C, Cl, S) are known to cause interferences in the analysis of Cr by ICP-MS, including Cl^- or S^- compounds. Most ICP-MS instruments allow an elemental survey of the sample prior to the measurement of analytes of interest without the need for calibration standards. Such a scan of the sample before analysis will help to identify any potential interferences in the sample that will need to be addressed so that the measurement data is not biased high. These types of inferences usually result in biasing the results above the true value. Additionally, the Cr mass fraction level in Kelp B was an order of magnitude lower than in Kelp A and SRM 3232, making calibration and sample preparation critical details, and a single calibration and sample preparation scheme may not be appropriate for all samples being measured.

Additional tables and figures for Cr in kelp are located in **Appendix B**.

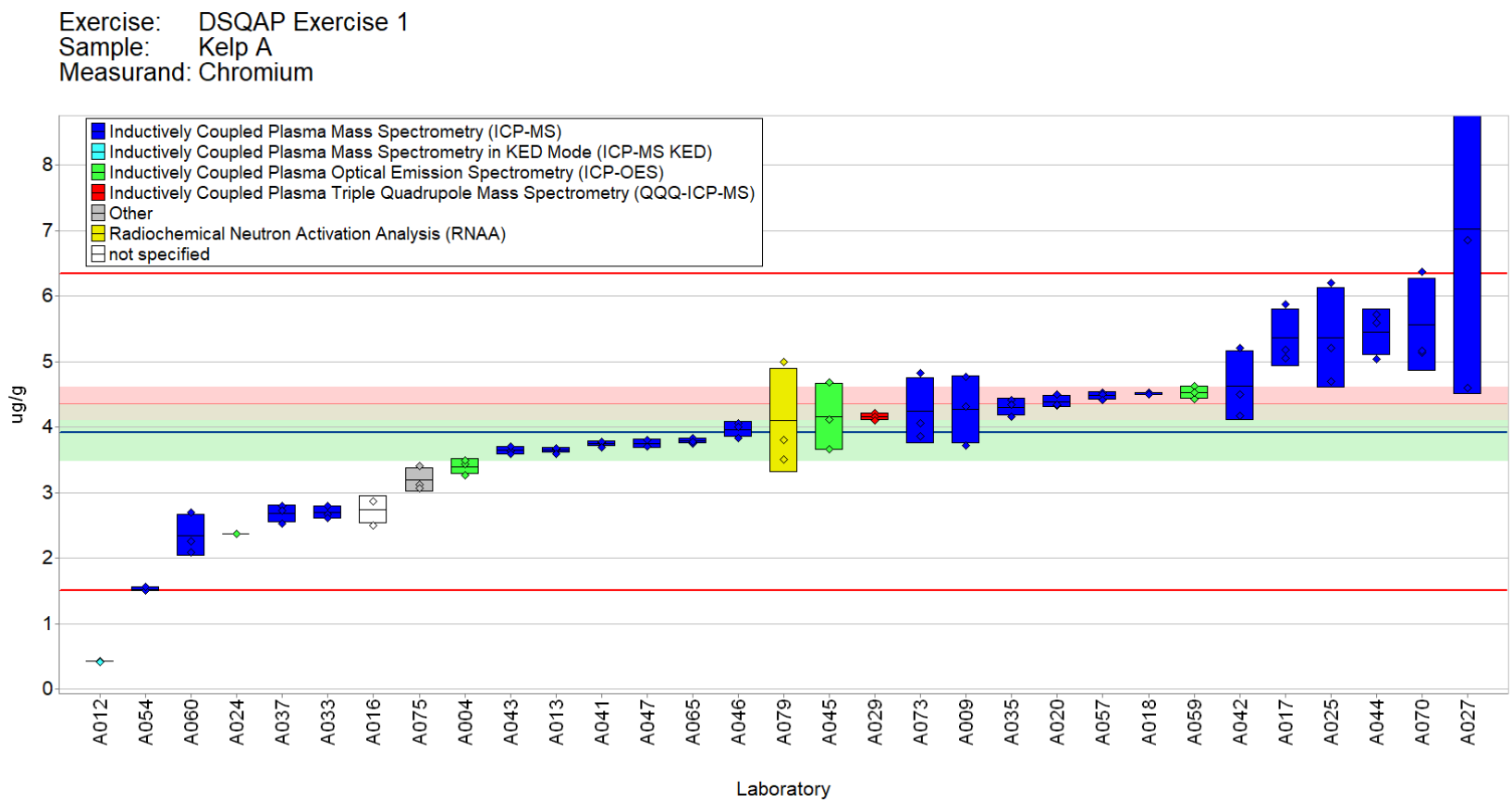

Fig. 3-16. Chromium in Kelp A (data summary view – analytical method).

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'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

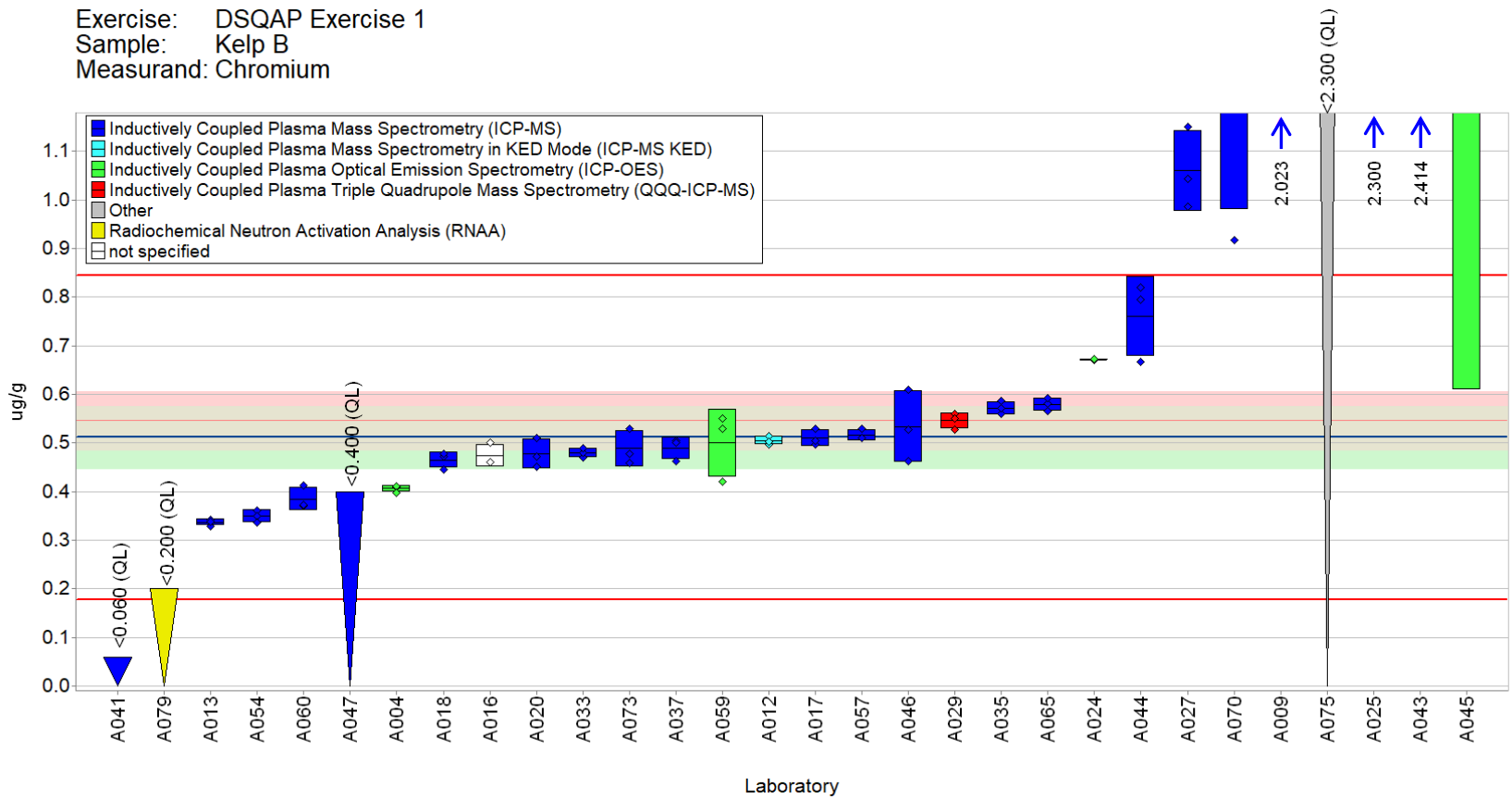

Fig. 3-17. Chromium in Kelp B (data summary view – analytical method).

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'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

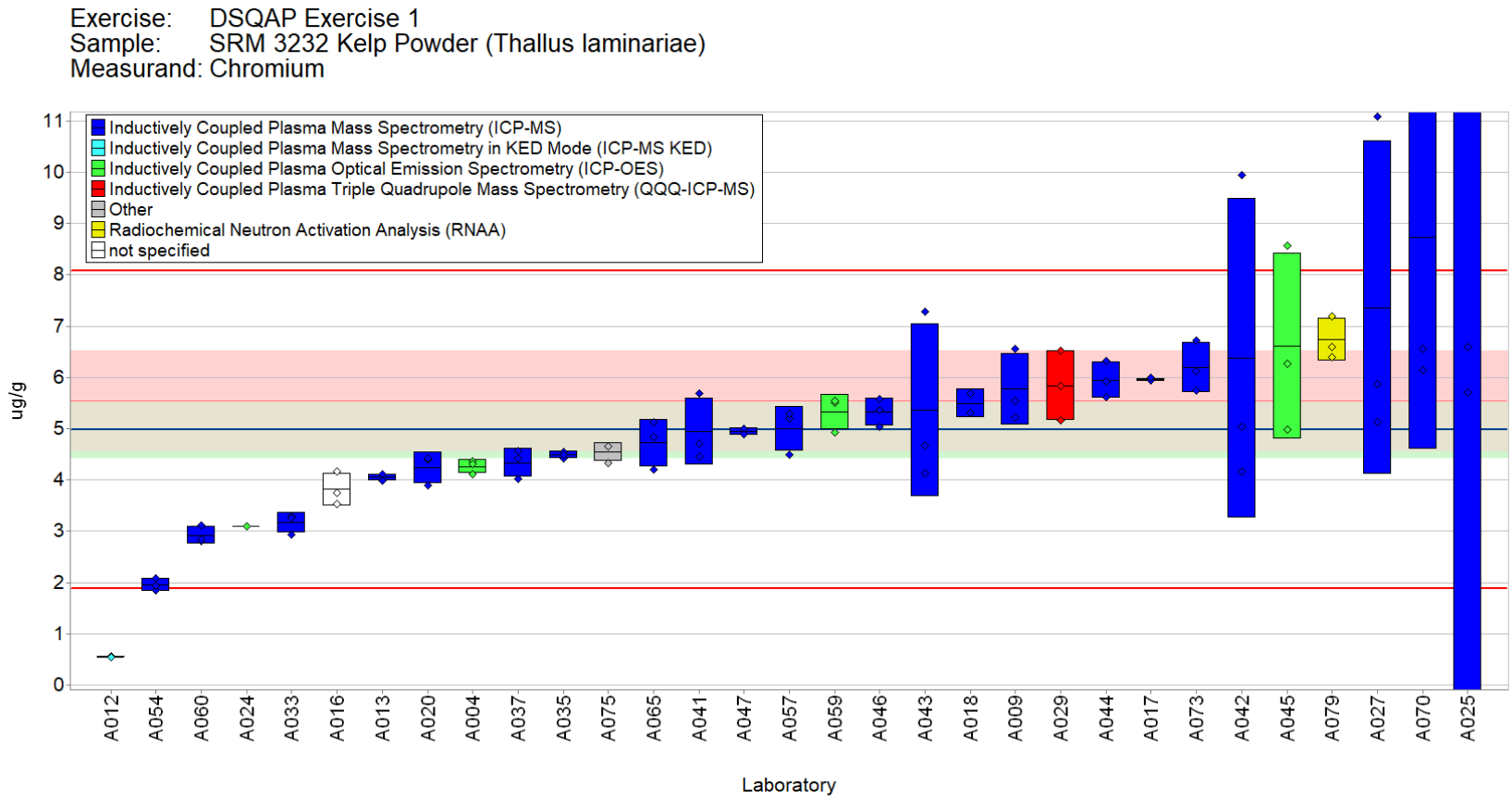

Fig. 3-18. Chromium in SRM 3232 Kelp Powder (*Thallus laminariae*) (data summary view – analytical method).

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'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

3.4.6. Copper (Cu)

Target values for Cu are provided in **Table 3-13**. The target values for Cu in Kelp A and Kelp B were determined at NIST using nitric acid assisted microwave digestion and ICP-MS. The target value for Cu in SRM 3232 was determined at NIST using nitric and hydrofluoric acid assisted microwave digestion and ID-ICP-MS.

Table 3-13. Summary of results and laboratory variabilities for Cu in kelp.

Copper (Cu)			
Within-Laboratory Variability (% RSD)	Averaged for all Samples		Maximum
	9.1 %		>100 %
	Kelp A	Kelp B	SRM 3232
Between-Laboratory Variability (% RSD)	36 %	28 %	22 %
Target Value ± U_{95} Mass Fraction (µg/g)	1.34 ± 0.06	1.124 ± 0.045	3.630 ± 0.082
Consensus Mean ± SD Mass Fraction (µg/g)	1.37 ± 0.49	1.18 ± 0.33	3.45 ± 0.77

For the determination of Cu, 28 of 52 laboratories reported results (54 %). Of the 24 laboratories reporting quantitative results, 15 laboratories reported within-laboratory variabilities within the published requirements of 7.3 % for mass fractions above 1 µg/g [4]. The average within-laboratory variability was 9.1 %, which indicates that some participants' in-house methods do not achieve adequate repeatability (**Table 3-13**). At least one laboratory reported variability greater than 100 %, and these results should be assessed for any calculation and reporting errors. The between-laboratory variabilities ranging from 28 % to 36 % were significantly greater than the published recommendation of at or below 8 % [4].

Almost half of the laboratories that reported a sample preparation method indicated using microwave digestion as seen in **Table 3-14**. Two-thirds of the laboratories reported using ICP-MS as their analytical method, including the results off-scale in **Fig. 3-20** and **3-21**. No method bias was observed although 39 % of laboratories did not report a sample preparation method.

Table 3-14. Summary of sample preparation and analytical methods averaged across all samples for determination of Cu in kelp.

Sample Preparation		Analytical Method	
Acid Digestion (no heat or microwave)	11 %	ICP-MS	65 %
Hot Block Digestion	3 %	ICP-OES	18 %
Microwave Digestion	47 %	QQQ-ICP-MS	4 %
None Reported	39 %	ICP	4 %
		RNAA	4 %
		Other/None Reported	7 %

The consensus means were within the target range for all materials. The consensus was above the target value for both Kelp A and Kelp B, **Fig. 3-19** and **3-20**, and below the target value for SRM 3232, **Fig 3-21**. The target ranges were all within the consensus confidence intervals, where the target range for Kelp A was in the middle of the confidence interval, the target range for Kelp B was in the lower half of the confidence interval, and the target range for SRM 3232 was in the upper half of the confidence interval.

The between-laboratory variabilities were greater than 22 %, and some of the within-laboratory variabilities were greater than 100 %, indicating global analytical challenges. The mass fractions of Cu in the kelp samples should not have challenged method LOQs. Laboratories reporting outside of the consensus confidence interval should review the general technical recommendations listed in Section 2 and in the elements study executive summary section. Some key points to highlight for consideration include ensuring that calibration curves are within the dynamic range for pulse mode and that both pulse mode and analog mode are calibrated when necessary. Monitoring multiple masses (for MS) or multiple wavelengths through axial and radial views of the plasma (for OES) can help identify and avoid interferences and measurement biases.

Laboratory A079 reported an LOQ significantly higher than the target range for all materials. This laboratory should check for calculation or reporting errors, as analytical techniques used for determining Cu should be optimized to achieve a LOQ around 0.01 ug/kg [4].

Additional tables and figures for Cu in kelp are located in **Appendix B**.

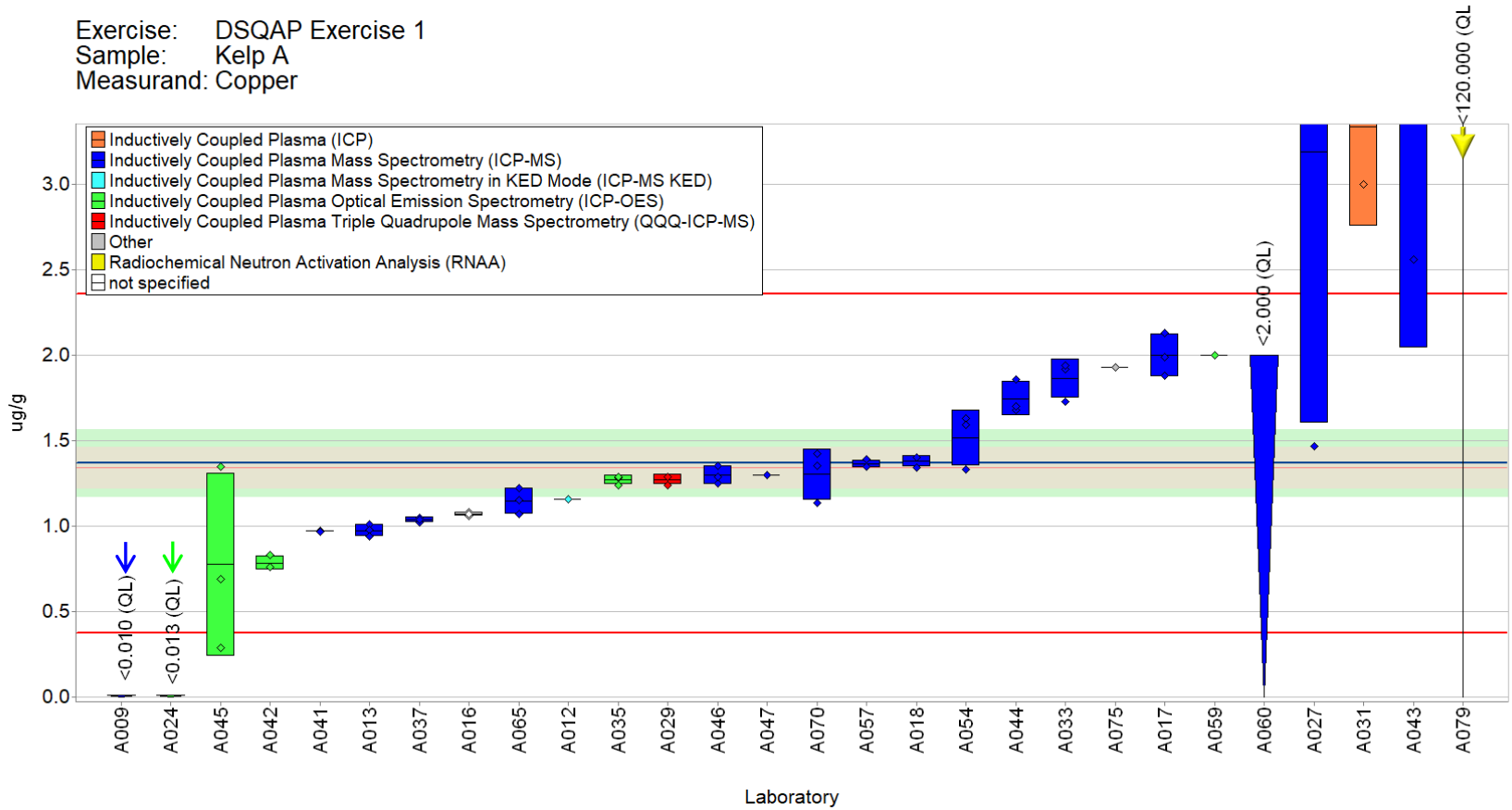

Fig. 3-19. Copper in Kelp A (data summary view – analytical method).

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'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

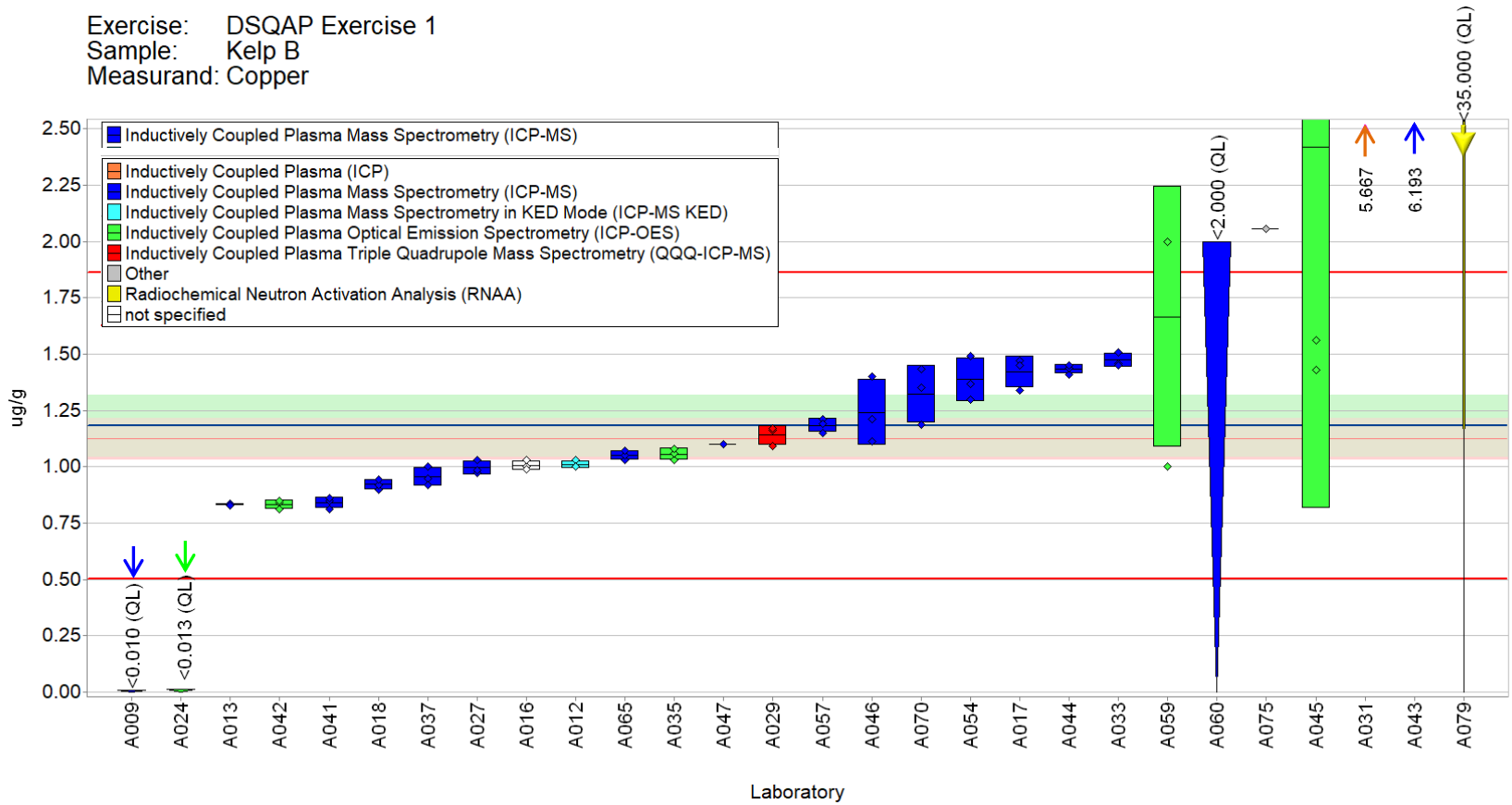

Fig. 3-20. Copper in Kelp B (data summary view – analytical method).

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'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region (beige here due to overlap with the green consensus confidence interval) represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

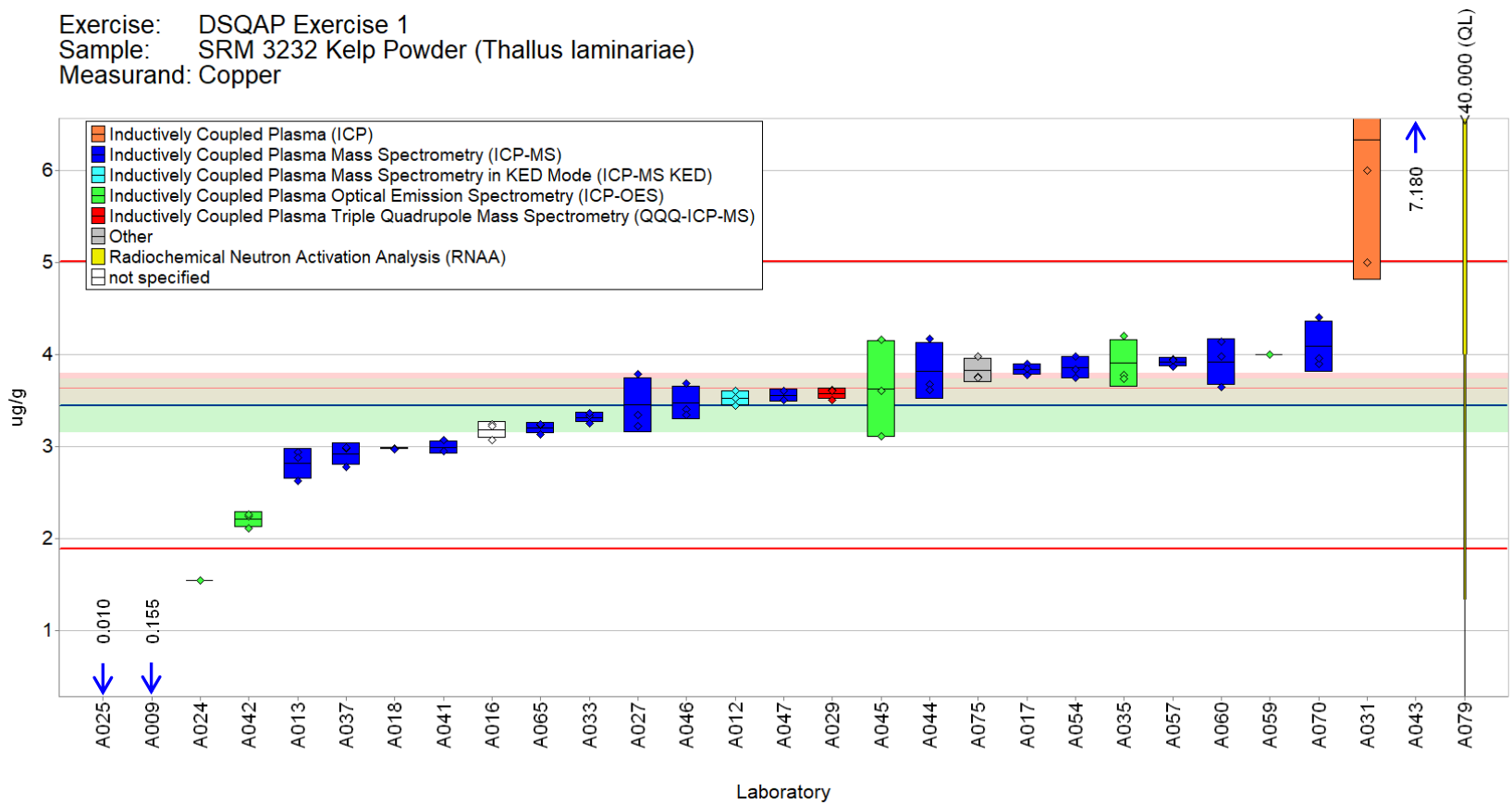

Fig. 3-21. Copper in SRM 3232 Kelp Powder (*Thallus laminariae*) (data summary view – analytical method).

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'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

3.4.7. Iodine (I)

The target value for I in SRM 3232 is provided in **Table 3-15** and was determined at NIST using (1) nitric acid assisted microwave digestion and (2) INAA after the material was pressed into pellets using a stainless-steel die. At the time of this report, target values were not available for I in Kelp A and Kelp B.

Table 3-15. Summary of results and laboratory variabilities for I in kelp.

Iodine (I)			
Within-Laboratory Variability (% RSD)	Averaged for all Samples		Maximum
	4.4 %		26 %
	Kelp A	Kelp B	SRM 3232
Between-Laboratory Variability (% RSD)	13 %	15 %	16 %
Target Value ± U_{95} Mass Fraction (µg/g)	-	-	884 ± 82
Consensus Mean ± SD Mass Fraction (µg/g)	2060 ± 260	880 ± 130	780 ± 130

For the determination of I, 12 of 39 laboratories reported results (31 %). Nine of the laboratories reported within-laboratory variabilities consistent with the published recommendation of 8 % [7], and the average within-laboratory variability across all laboratories and samples was 4.4 % (**Table 3-15**). The between-laboratory variabilities ranged from 13 % to 16 %, which is excellent performance compared to the published standard of 15 % for a single method across multiple laboratories. The I levels in the kelp materials is greater than the upper analytical range in the SMPR (10 µg/g), so laboratories with high variability should ensure that appropriate sample preparation steps (i.e., dilution) are taken.

Most laboratories that reported sample preparation methods indicated using either microwave digestion or base extraction (no heat or microwave) as shown in **Table 3-16**, though 33 % of laboratories did not provide sample preparation information. Nearly all laboratories reported using ICP-MS as their analytical method with only one laboratory using RNAA. Results from the RNAA analysis consistently fell in the lower half of the measurement results for all three samples but is not necessarily a direct correlation to RNAA and could be contributed to another factor such as calibration issues. No other analytical method bias was observed.

Table 3-16. Summary of sample preparation and analytical methods averaged across all samples for determination of I in kelp.

Sample Preparation		Analytical Method	
Acid-Assisted Microwave Digestion	17 %	ICP-MS	84 %
Acid-Assisted Microwave Digestion converted to Base	8 %	ICP-MS KED	8 %
Base-Assisted Hot Block Digestion	8 %	RNAA	8 %
Base-Assisted Microwave Digestion	8 %		
Base Extraction (no heat or microwave)	8 %		
TMAH Base Digestion	17 %		
None Reported	33 %		

The consensus mean was below the target value for SRM 3232 and the confidence interval was almost completely within the lower half of target range, as seen in **Fig. 3-24**. Over half of the participants reported results within the target range. Target values for Kelp A and Kelp B were not available at the time of this report.

Sample preparation methods must be able to fully extract I from the samples while also being mindful of any potential degradation and/or conversion. Samples that were not properly digested could result in a low bias as seen in the data for SRM 3232 (**Fig. 3-24**). Iodine is a volatile element and can form hydrogen iodide (HI) during acid digestion. Iodine is also light sensitive and at some stages of sample preparation solutions may need to be kept in amber or covered sample vessels.

While the participants of this study did not report levels greater than the tolerance limit, it is still important to consider potential issues with sample preparation solution choices. When using ICP-MS, an acidic sample solution can result in sample carryover leading to variable and biased measurements. Using a basic solution or a surfactant such as Triton X-100 will improve washout of I. Some protocols use an alkaline digestion with tetramethylammonium hydroxide (TMAH), but extreme caution must be taken when using TMAH, which is a very strong base with high toxicity. A safer alternative may be to use an acid digestion followed by immediate neutralization of solutions with a base such as ammonium hydroxide.

Additional tables and figures for I in kelp are located in **Appendix B**.

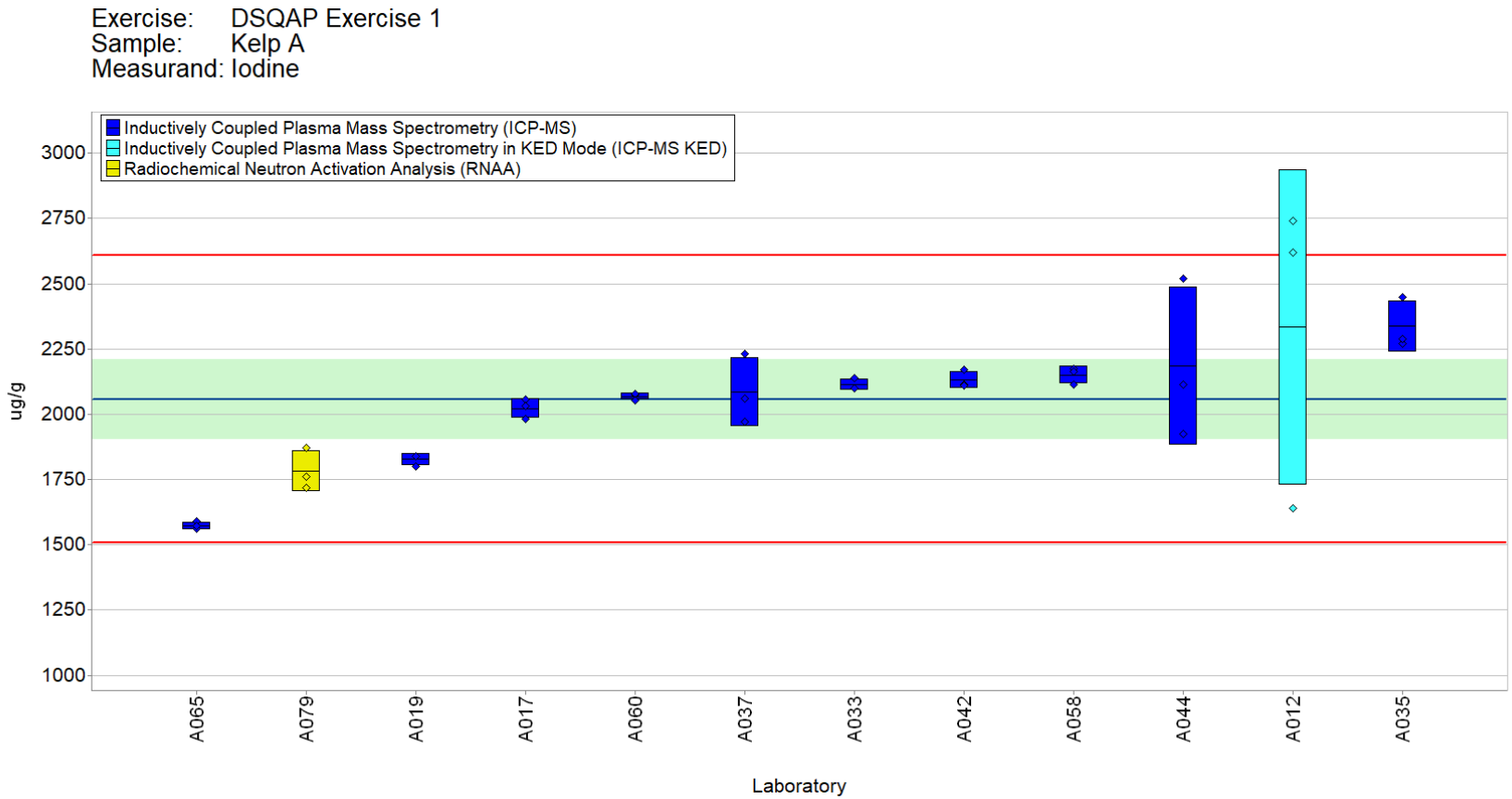

Fig. 3-22. Iodine in Kelp A (data summary view – analytical method).

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'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

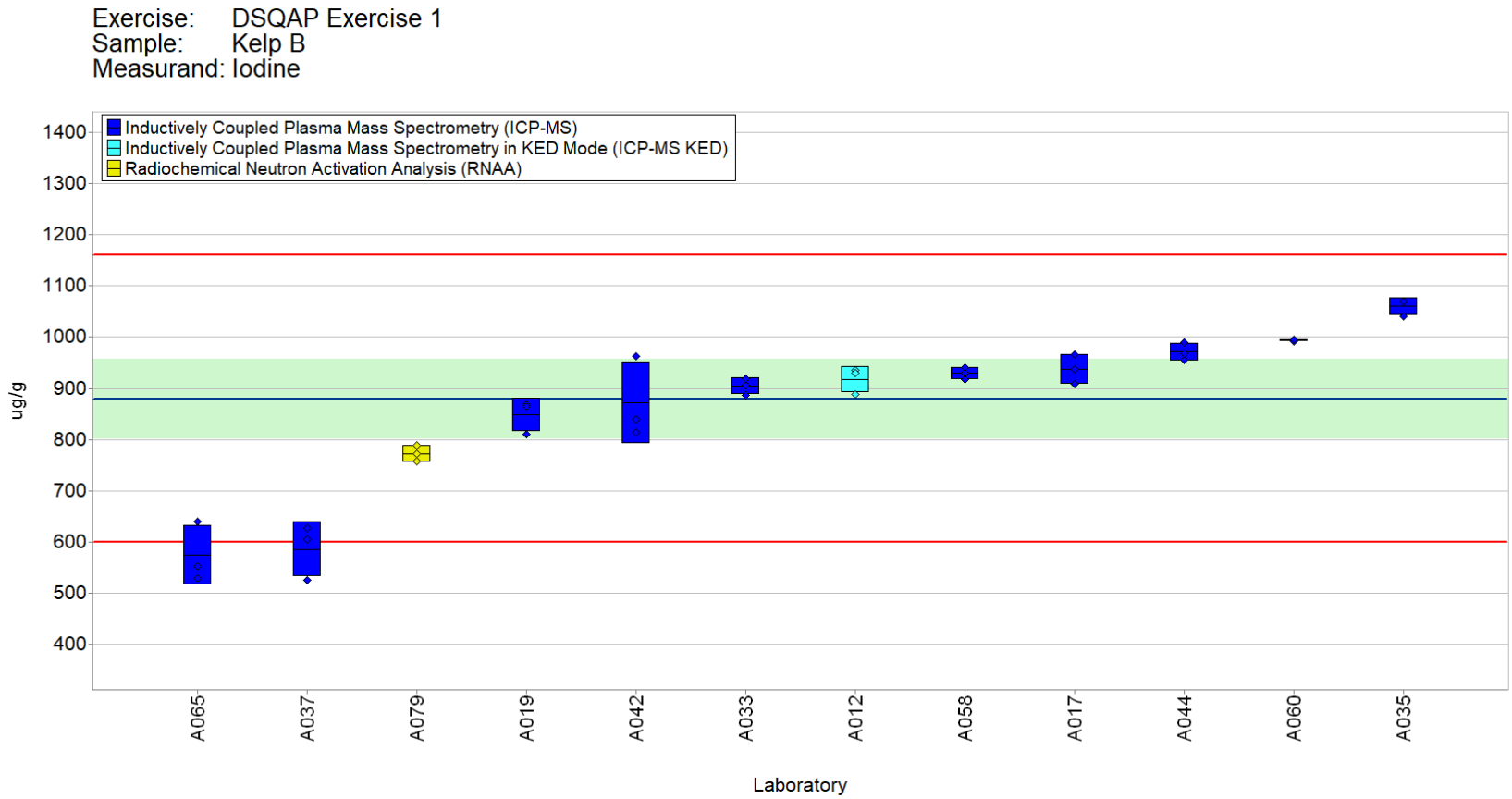

Fig. 3-23. Iodine in Kelp B (data summary view – analytical method).

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'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

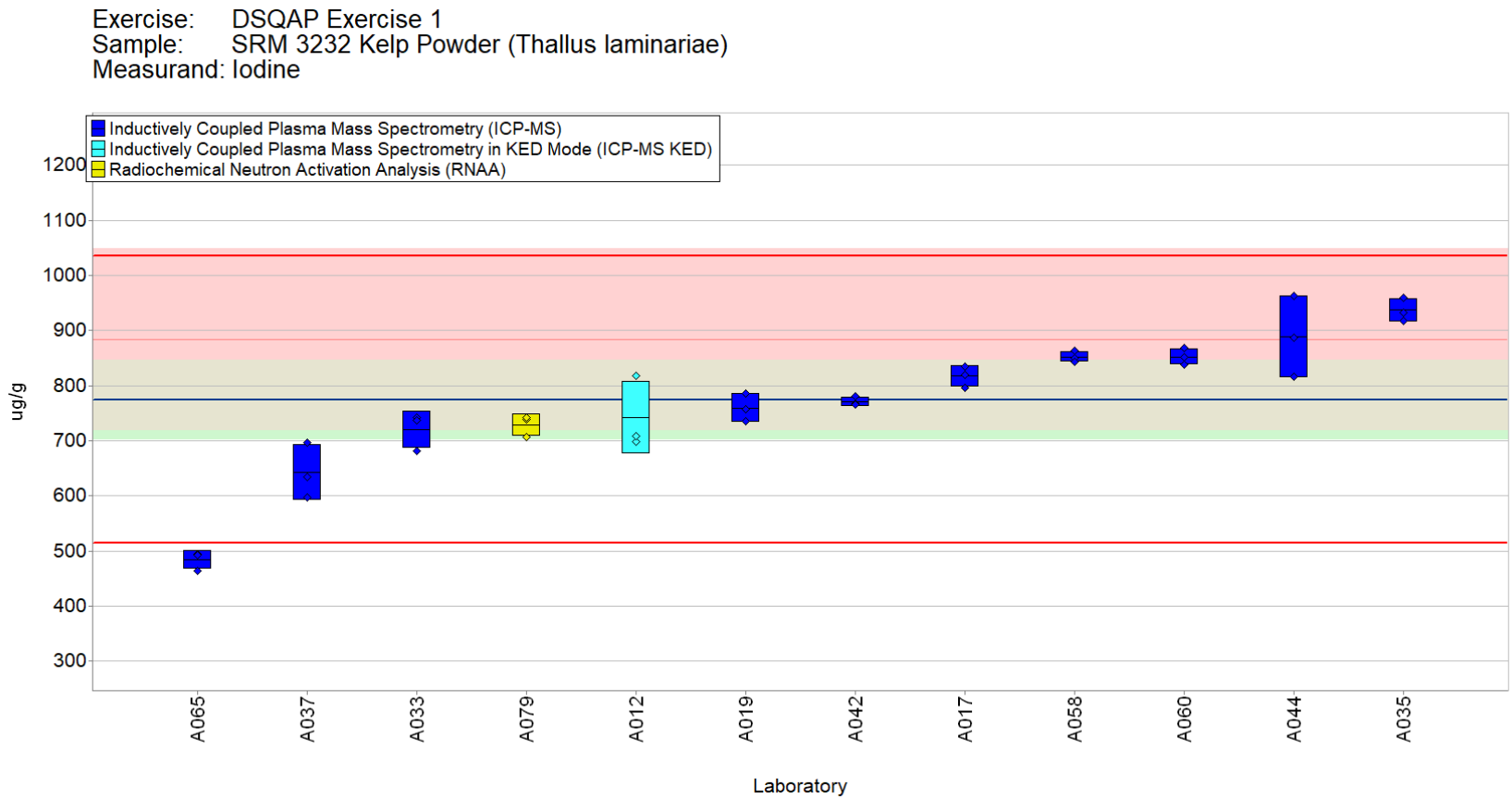

Fig. 3-24. Iodine in SRM 3232 Kelp Powder (*Thallus laminariae*) (data summary view – analytical method).

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 (seen here as beige) represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

3.4.8. Lead (Pb)

Target values for Pb are summarized in **Table 3-17**. The target values for Pb in Kelp A and Kelp B were determined at NIST using nitric acid assisted microwave digestion and ICP-MS. The target value for Pb in SRM 3232 was determined at NIST using nitric and hydrofluoric acid assisted microwave digestion and ID-ICP-MS.

Table 3-17. Summary of results and laboratory variabilities for Pb in kelp.

Lead (Pb)								
Within-Laboratory Variability (% RSD)	Averaged for all Samples					Maximum		
	5.0 %					48 %		
	Kelp A			Kelp B		SRM 3232		
Between-Laboratory Variability (% RSD)	15 %			11 %		14 %		
Target Value ± U_{95} Mass Fraction (µg/g)	0.594	±	0.017	0.406	±	0.005	0.967	±	0.037
Consensus Mean ± SD Mass Fraction (µg/g)	0.571	±	0.088	0.403	±	0.045	0.88	±	0.12

For the determination of Pb, 33 of 53 laboratories reported results (62 %). Of the 30 laboratories that reported quantitative results, 22 laboratories reported within-laboratory variabilities within the published requirements of 11 % for mass fractions below 1 µg/g [4] for all three materials. The average within-laboratory variability was 5.0 % (**Table 3-17**), which indicates that most participants' in-house methods achieve acceptable repeatability. The between-laboratory variabilities ranged from 11 % to 15 %, indicating that the community demonstrated acceptable agreement for all materials with respect to the published requirement of 16 % [4].

More than half of laboratories that reported sample preparation method indicated using microwave digestion as seen in **Table 3-18**. Most laboratories reported using ICP-MS as their analytical method. No method bias was observed although 27 % of laboratories did not report a sample preparation method.

Table 3-18. Summary of sample preparation and analytical methods averaged across all samples for determination of Pb in kelp.

Sample Preparation		Analytical Method	
Acid Digestion (no heat or microwave)	9 %	ICP-MS	85 %
Hot Block Digestion	3 %	ICP-OES	6 %
Microwave Digestion	58 %	QQQ-ICP-MS	3 %
Thermal Decomposition	3 %	Other/None Reported	6 %
None Reported	27 %		

The consensus mean was below the target value for both Kelp A and SRM 3232, as seen in **Fig. 3-25** and **3-27**, while the consensus mean was equal to the target value for Kelp B, as seen in **Fig. 3-26**. The consensus confidence interval was overlapping the lower range of the target range for Kelp A and SRM 3232, while it was completely overlapping the target range for Kelp B.

When using concentrated acid and microwave digestion sample preparation methods, high temperature and pressure ensure complete digestion for Pb from the sample into solution. Laboratories reporting results consistently below the target value (A009, A024, and A025) may have been performing digestion with HCl, which can form insoluble $PbCl_2$ precipitate and result in undissolved Pb going undetected. As Pb mass fractions increase, $PbCl_2$ precipitation due to HCl could become more problematic, and may explain why the consensus means for Kelp A and SRM 3232 were lower than the target, since the Pb mass fractions were about twice that of Kelp B. To prevent this bias, digestion with HNO_3 is recommended for analysis of Pb. If HCl is used in digestion, dilute HNO_3 should be used to repeatedly wash the side of the digestion vessels to redissolve any $PbCl_2$ that may have formed. When using ICP-MS, the three most abundant Pb isotopes (^{206}Pb, ^{207}Pb, ^{208}Pb) should be monitored and their signals averaged to account for natural differences in Pb isotopic composition between standards and sample types.

Additional tables and figures for Pb in kelp are located in **Appendix B**.

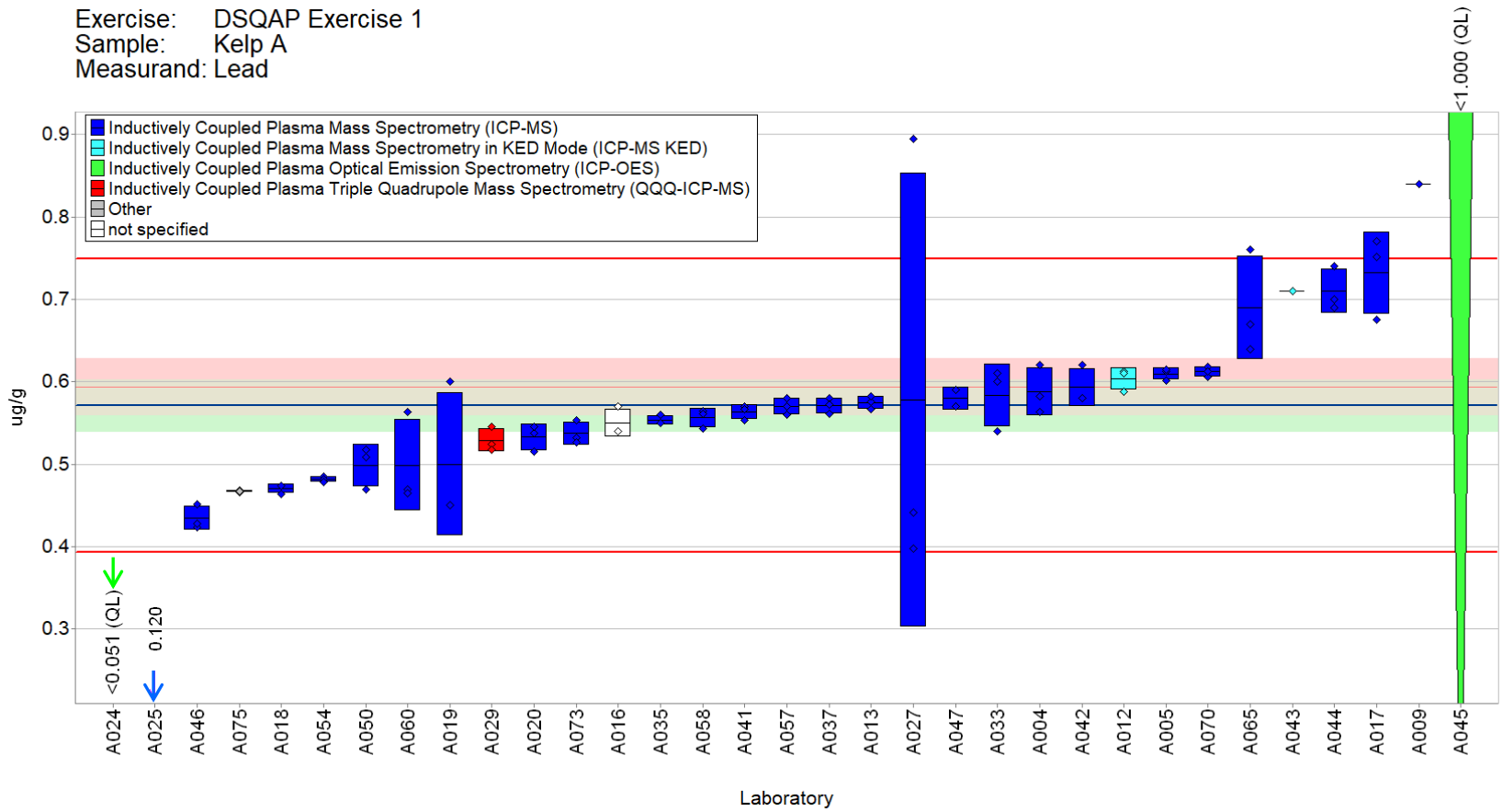

Fig. 3-25. Lead in Kelp A (data summary view – analytical method).

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'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

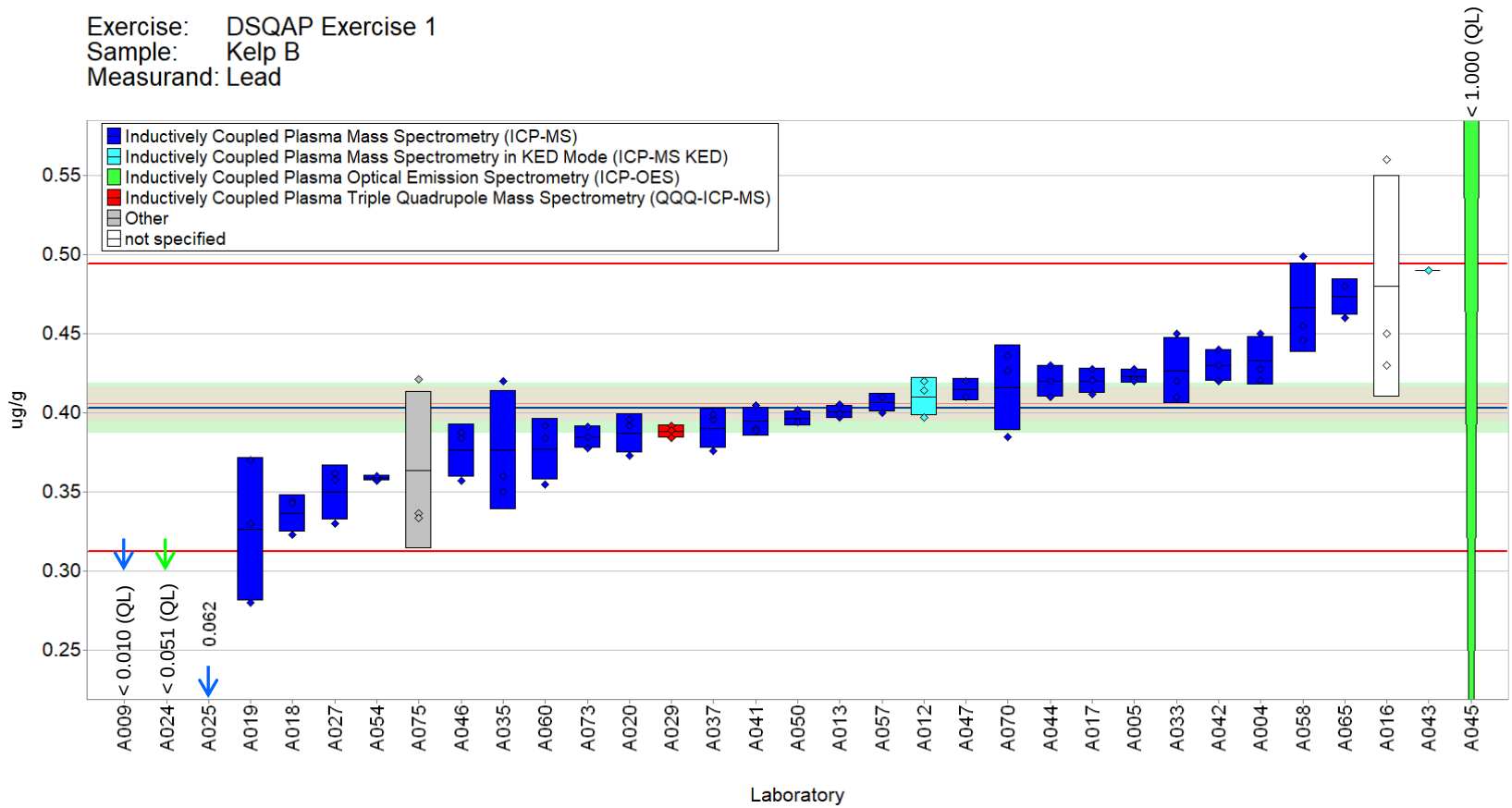

Fig. 3-26. Lead in Kelp B (data summary view – analytical method).

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'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region (beige here due to overlap with the green consensus confidence interval) represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

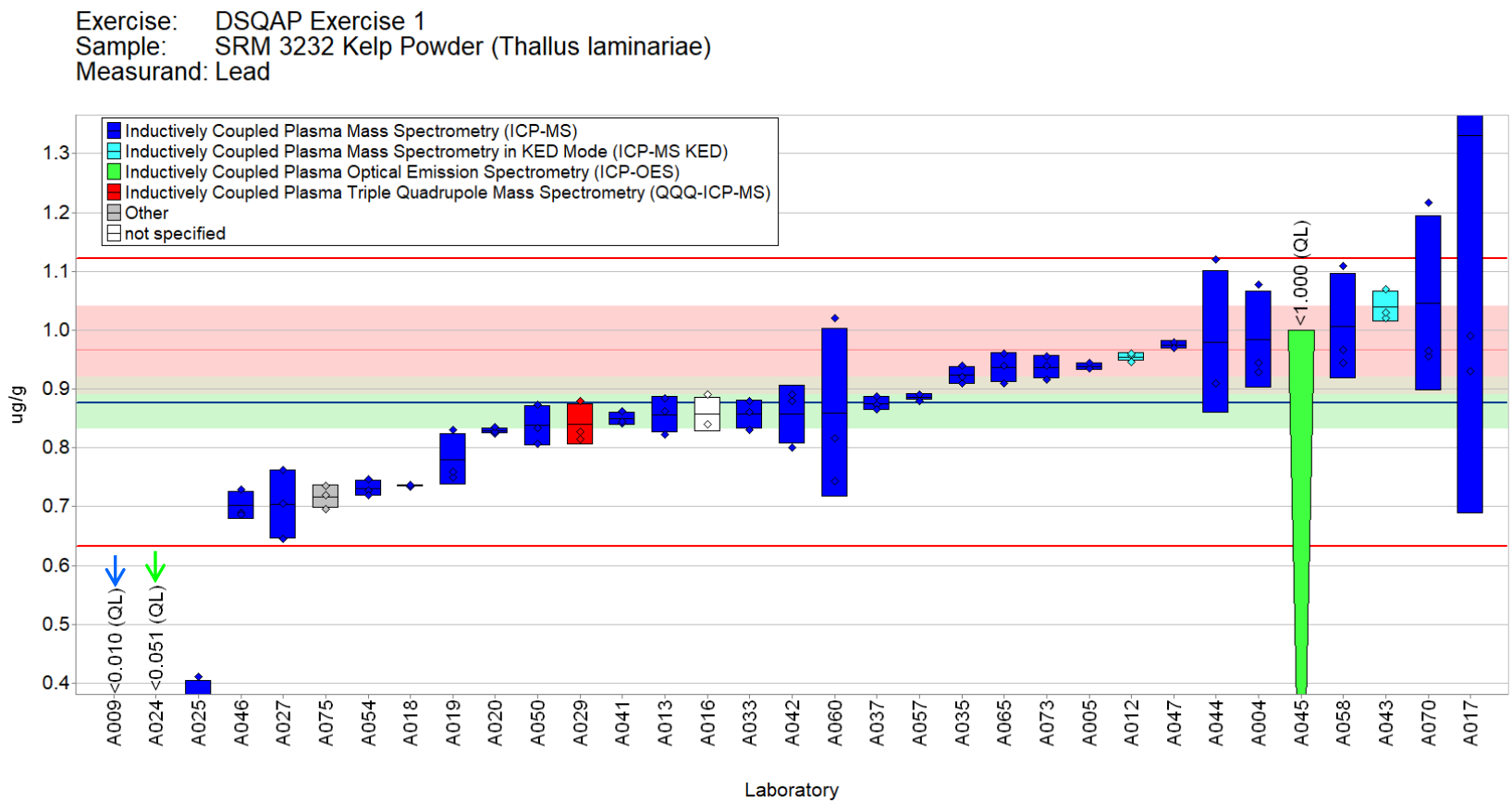

Fig. 3-27. Lead in SRM 3232 Kelp Powder (*Thallus laminariae*) (data summary view – analytical method).

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'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

3.4.9. Magnesium (Mg)

Target values for Mg are provided in **Table 3-19**. The target values for Mg in Kelp A and Kelp B were determined at NIST using nitric and hydrofluoric acid assisted microwave digestion and ICP-OES. The target value for Mg in SRM 3232 was determined at NIST using (1) INAA after the material was pressed into pellets using a stainless-steel die and (2) nitric acid assisted microwave digestion and ICP-OES.

Table 3-19. Summary of results and laboratory variabilities for Mg in kelp.

Magnesium (Mg)			
Within-Laboratory Variability (% RSD)	Averaged for all Samples		Maximum
	2.4 %		13 %
	Kelp A	Kelp B	SRM 3232
Between-Laboratory Variability (% RSD)	7 %	10 %	9 %
Target Value ± U_{95} Mass Fraction (µg/g)	6880 ± 920	5300 ± 2300	5740 ± 170
Consensus Mean ± SD Mass Fraction (µg/g)	7200 ± 450	8310 ± 710	5790 ± 470

For the determination of Mg, 33 of 51 laboratories reported results (65 %), and 26 laboratories reported within-laboratory variabilities within the published requirement of 5 % [6]. The average within-laboratory variability was 2.4 %, with all laboratories at or below 13 %, which demonstrates that that most participants' in-house methods achieve successful repeatability. The between-laboratory variabilities were at or below 10 % for all three samples (**Table 3-19**), which is at or below the published requirement of 10 % for multiple laboratories using the same method [6]. Notably, the Mg levels in the kelp samples were significantly greater than the upper analytical range of the SMPR (0.1375 µg/g). Laboratories reporting variabilities outside of the recommended ranges should ensure that appropriate sample preparation steps (i.e., dilution) are taken for higher concentrations of Mg.

Nearly half of the laboratories that reported a sample preparation method indicated using microwave digestion as shown in **Table 3-20**. Almost half of the laboratories reported using ICP-MS as their analytical method, including results off-scale in **Figs. 3-28** and **3-30**, along with more than one-third of the laboratories using ICP-OES. No method bias was observed although 30 % of laboratories did not report a sample preparation method.

Table 3-20. Summary of sample preparation and analytical methods averaged across all samples for determination of Mg in kelp.

Sample Preparation		Analytical Method	
Acid Digestion (no heat or microwave)	9 %	ICP-MS	49 %
Hot Block Digestion	9 %	ICP-OES	38 %
Microwave Digestion	49 %	QQQ-ICP-MS	3 %
Open Beaker Digestion	3 %	ICP	3 %
None Reported	30 %	Other/None Reported	6 %

The consensus mean was above the target value and the confidence interval was within the target range for all three kelp samples, as seen in **Fig. 3-28**, **3-29**, and **3-30** for Kelp A, Kelp B, and SRM 3232, respectively.

Laboratories reporting high values for Mg when using ICP-MS should be aware of $^{12}C_2^+$ interferences. These interferences can be minimized by using He as a collision cell gas with KED mode. Laboratories should also consider the general ICP-MS recommendations for Mg when evaluating their methods.

Additional tables and figures for Mg in kelp are located in **Appendix B**.

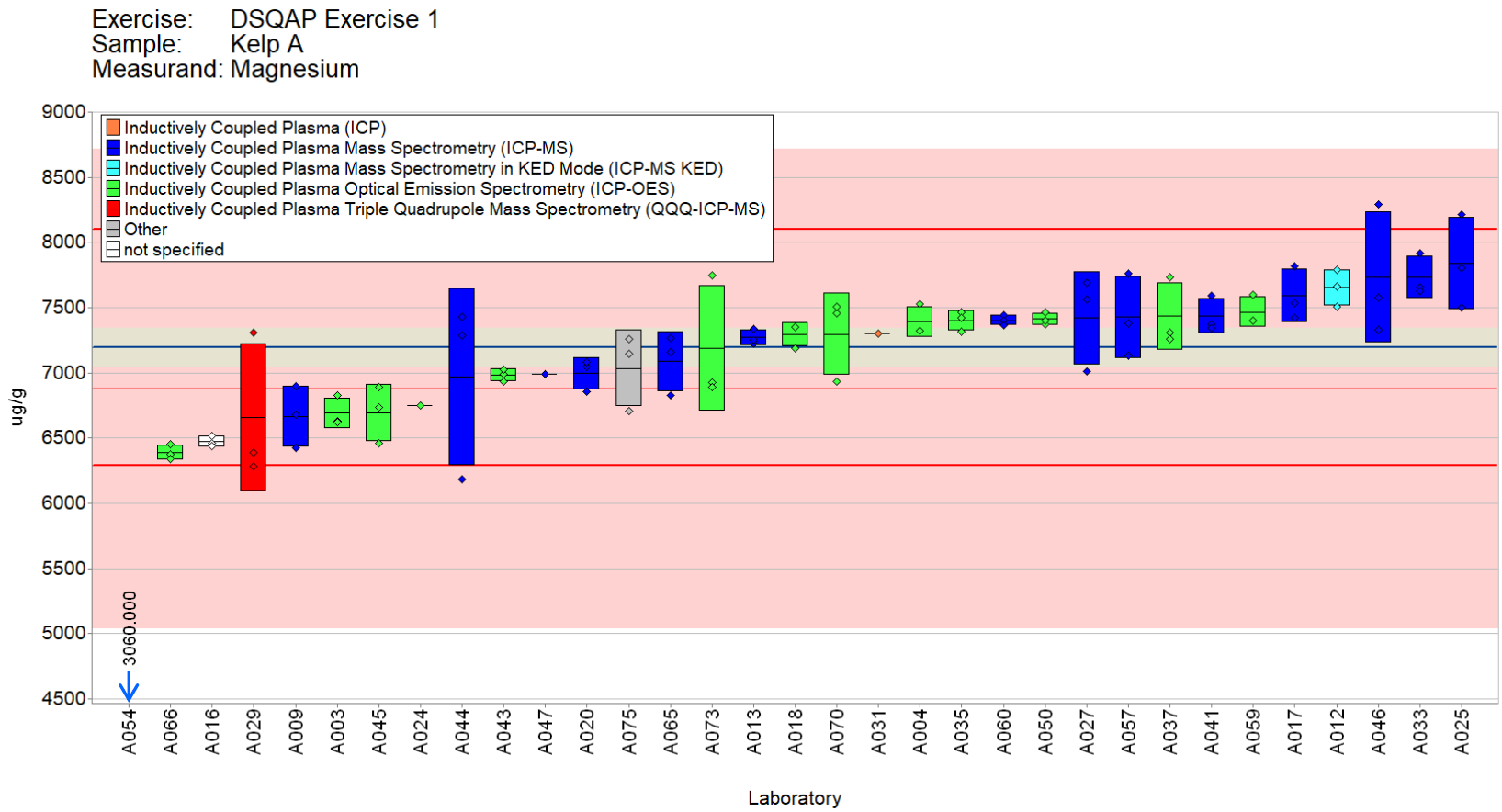

Fig. 3-28. Magnesium in Kelp A (data summary view – analytical method).

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 (beige here due to overlap with the red NIST target range) region represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$.

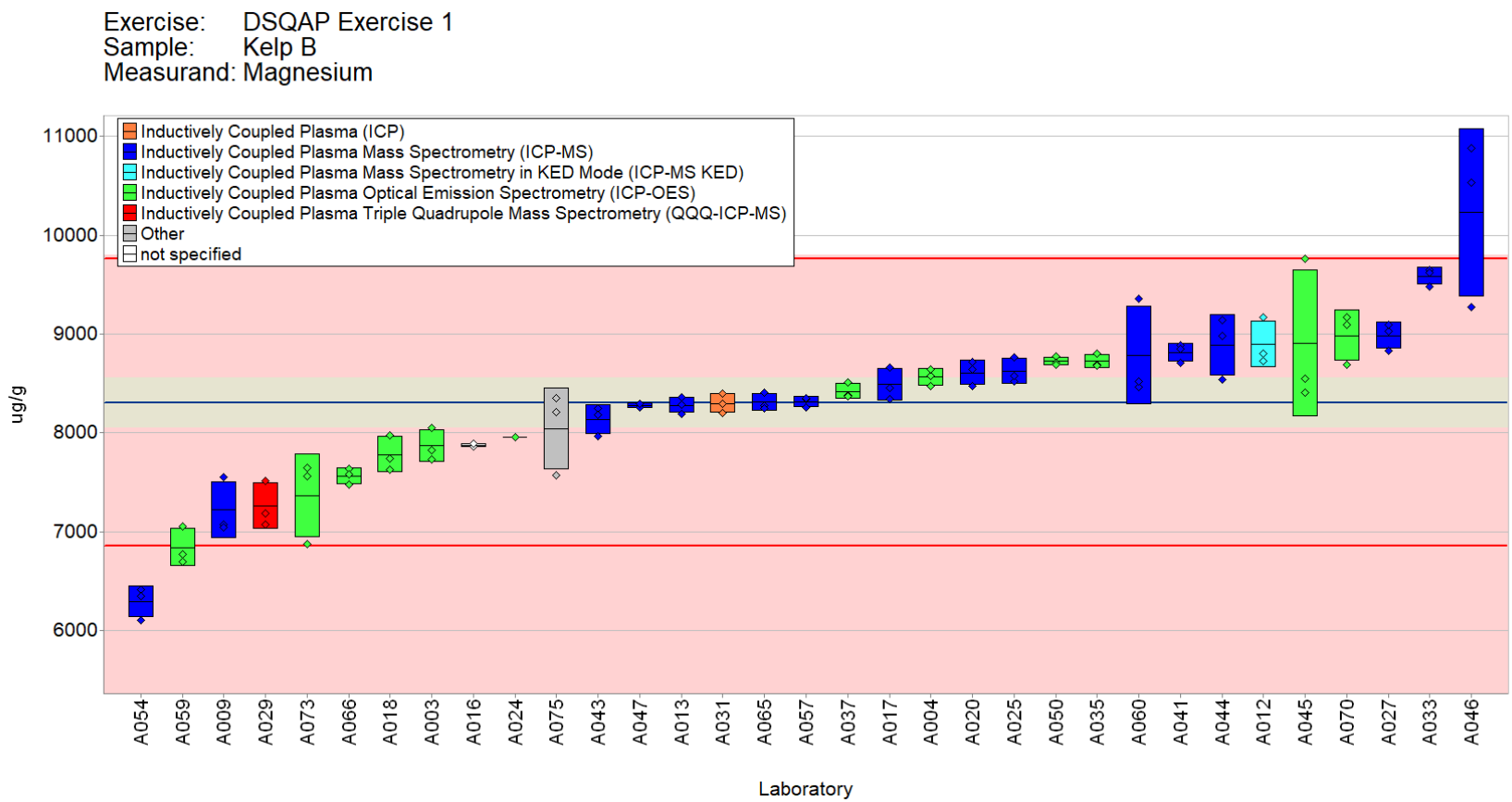

Fig. 3-29. Magnesium in Kelp B (data summary view – analytical method).

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 (beige here due to overlap with the red NIST target range) represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$.

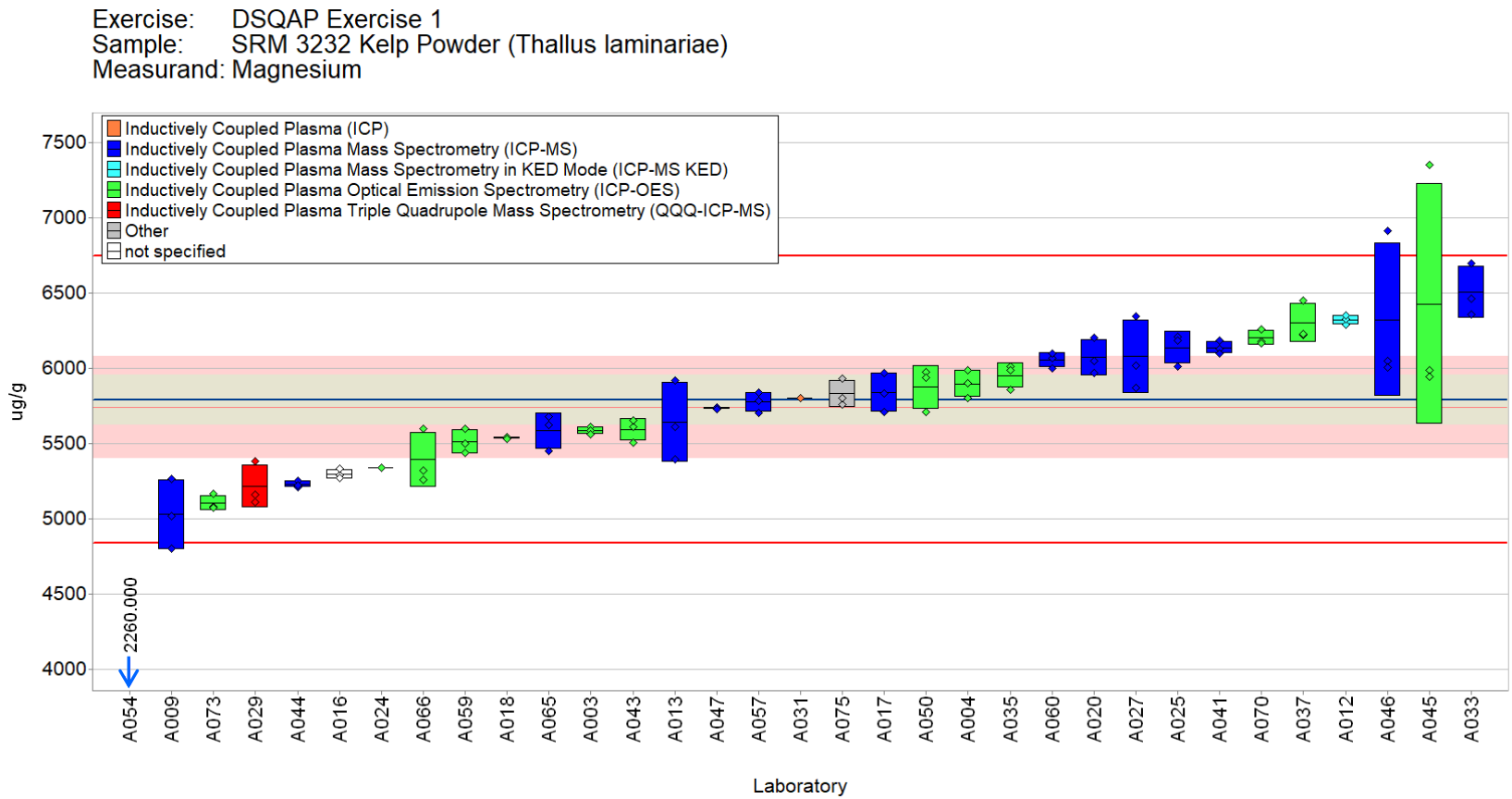

Fig. 3-30. Magnesium in SRM 3232 Kelp Powder (*Thallus laminariae*) (data summary view – analytical method).

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 (beige here due to overlap with the red NIST target range) represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$.

3.4.10. Mercury (Hg)

The target values for Hg are listed in **Table 3-21**. The target values for Hg in Kelp A and Kelp B were determined at NIST using direct combustion atomic absorption spectrometry (DC AAS). The target value for Hg in SRM 3232 was determined at NIST using hydrochloric and nitric acid assisted microwave digestion and isotope dilution cold-vapor generation ICP-MS (ID-CV-ICP-MS).

Table 3-21. Summary of results and laboratory variabilities for Hg in kelp.

Mercury (Hg)			
Within-Laboratory Variability (% RSD)	Averaged for all Samples		Maximum
	9.9 %		>100 %
	Kelp A	Kelp B	SRM 3232
Between-Laboratory Variability (% RSD)	37 %	30 %	18 %
Target Value ± U_{95} Mass Fraction (ng/g)	31.29 ± 0.46	23.09 ± 0.42	105.8 ± 3.0
Consensus Mean ± SD Mass Fraction (ng/g)	34 ± 12	27.0 ± 8.2	96 ± 17

For the determination of Hg, 30 of 51 laboratories reported results (59 %). The average within-laboratory variability was 9.9 % with 15 laboratories below 15 % for Kelp A and Kelp B and 25 laboratories below 11 % for SRM 3232, which indicates that some participants' in-house methods achieve successful repeatability while others did not demonstrate acceptable repeatability The SMPR specifies successful repeatability to be at or below 11 % for mass fractions above 100 ng/g (SRM 3232) and at or below 15 % for mass fractions between 10 ng/g and 100 ng/g [4] (Kelp A and Kelp B). At least one laboratory reported variability greater than 100 %, and these results should be assessed for any calculation and reporting errors. The overall performance for the analysis of Hg, indicated by between-laboratory variabilities generally aligned with the published expectations of at or below 16 % for mass fractions above 100 ng/g and at or below 32 % for mass fractions above 10 ng/g [4] (**Table 3-21**). Target reproducibilities are recommended for multiple laboratories using the same method; however, the reproducibilities in this study represent multiple laboratories using different methods.

Approximately half of the laboratories that reported a sample preparation method indicated using microwave digestion as seen in **Table 3-22**. Most of the laboratories reported using ICP-MS as their analytical method. No method bias was observed although 36 % of laboratories did not report a sample preparation method.

Table 3-22. Summary of sample preparation and analytical methods averaged across all samples for determination of Hg in kelp.

Sample Preparation		Analytical Method	
Acid Digestion (no heat or microwave)	7 %	ICP-MS	73 %
Hot Block Digestion	3 %	ICP-OES	8 %
Microwave Digestion	53 %	QQQ-ICP-MS	3 %
Thermal Decomposition	1 %	AAS	3 %
None Reported	36 %	RNAA	3 %
		DMA	3 %
		Other/None Reported	7 %

The consensus mean was above the target value for both Kelp A and Kelp B, as seen in **Fig. 3-31** and **3-32**, while it was below the target value for SRM 3232, as seen in **Fig. 3-33**. The consensus confidence interval was completely overlapping the target range for Kelp A, above the target range for Kelp B, and slightly overlapping the lower range of the target range for SRM 3232.

The volatility of Hg can lead to high variability in some analytical approaches. High temperature microwave digestion using closed quartz or borosilicate glass vessels is recommended for sample preparation prior to Hg analysis. The high temperatures will ensure a complete digestion with little volatile loss of Hg from the closed vessels. Quartz and borosilicate glass vessels are more suitable for Hg sample digestion than polytetrafluoroethylene (PTFE) as they can be single use or easily cleaned to prevent retention of Hg. Nearly half of the laboratories reported using a sample preparation method that was not microwave digestion or did not report their method. Laboratories that did not use microwave digestion should evaluate their sample preparation method to determine whether complete extraction of Hg from the materials, without volatile loss, is being achieved. Samples that were not properly digested could have resulted in a low bias, such as those observed for SRM 3232. Laboratories using microwave digestion should ensure that values are not being biased high due to a memory effect from digestion vessels, such as observed for Kelp A and Kelp B.

The Hg levels in these materials should be well above method LOQs, but depending on the sample preparation and dilution steps, the as-measured mass fractions may be pushing the method detection limits (MDL). Cold vapor Hg generation can be used to increase the sensitivity of ICP-MS for Hg, which allows lower levels of Hg to be measured. Laboratories need to ensure that a sufficient number of blanks are analyzed to determine an accurate MDL and LOQ for any method. Mercury blanks and backgrounds may be large, making determination of Hg values in samples containing low levels of Hg difficult, such as those used for MDL or LOQ determinations. The laboratory using RNAA reported varying LOQs for the three kelp materials with Kelp A and Kelp C having LOQs below the target range. The LOQs should be similar, even if measured in separate analytical sets.

Erratic results can occur due to Hg carryover between samples resulting in high within-laboratory variability as seen in Kelp A. Adequate washout time is needed after each measurement and the addition of dilute HCl to the washout solution can help reduce the length of washout time needed.

Additional tables and figures for Hg in kelp are located in **Appendix B**.

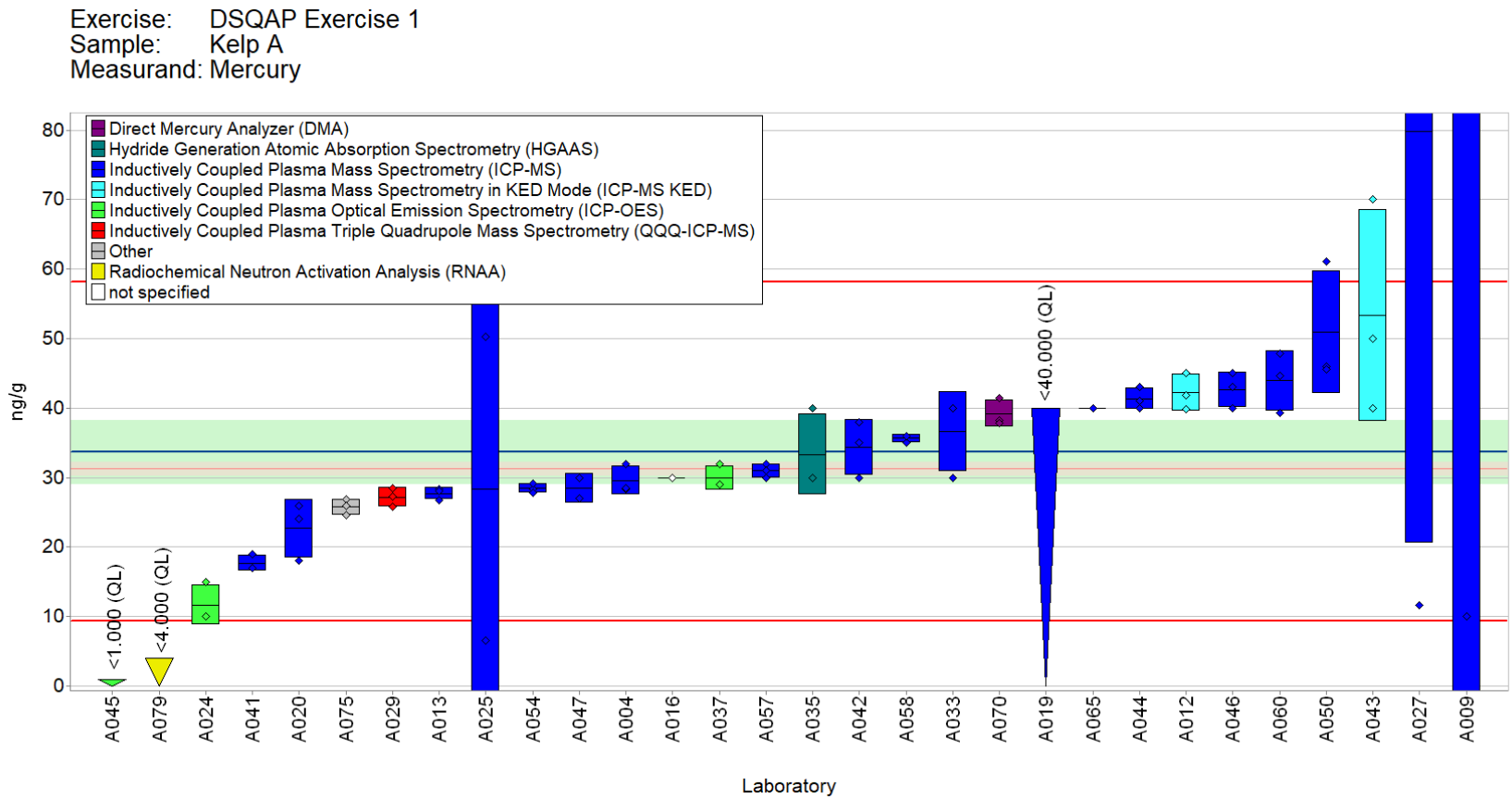

Fig. 3-31. Mercury in Kelp A (data summary view – analytical method).

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'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

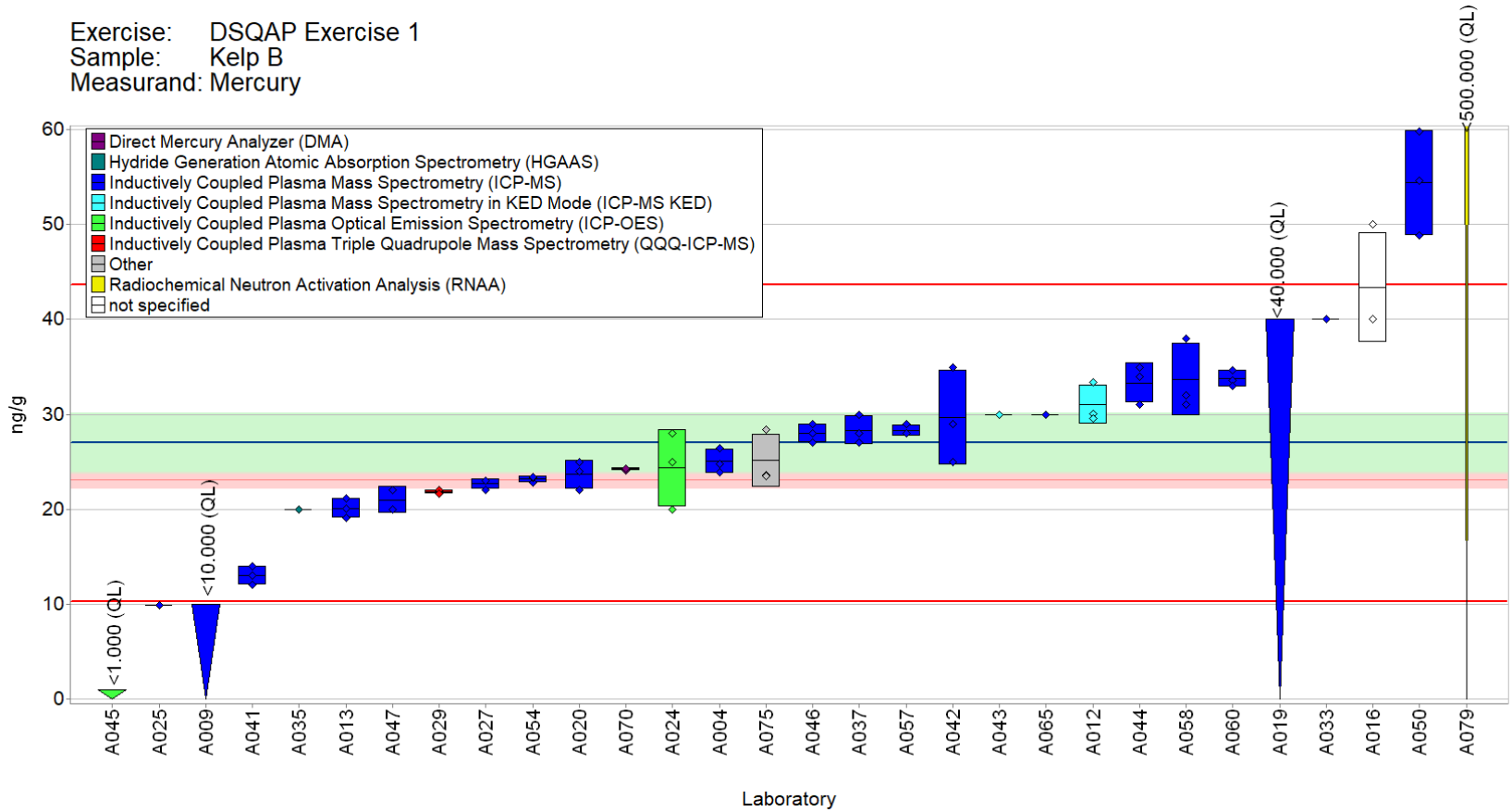

Fig. 3-32. Mercury in Kelp B (data summary view – analytical method).

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

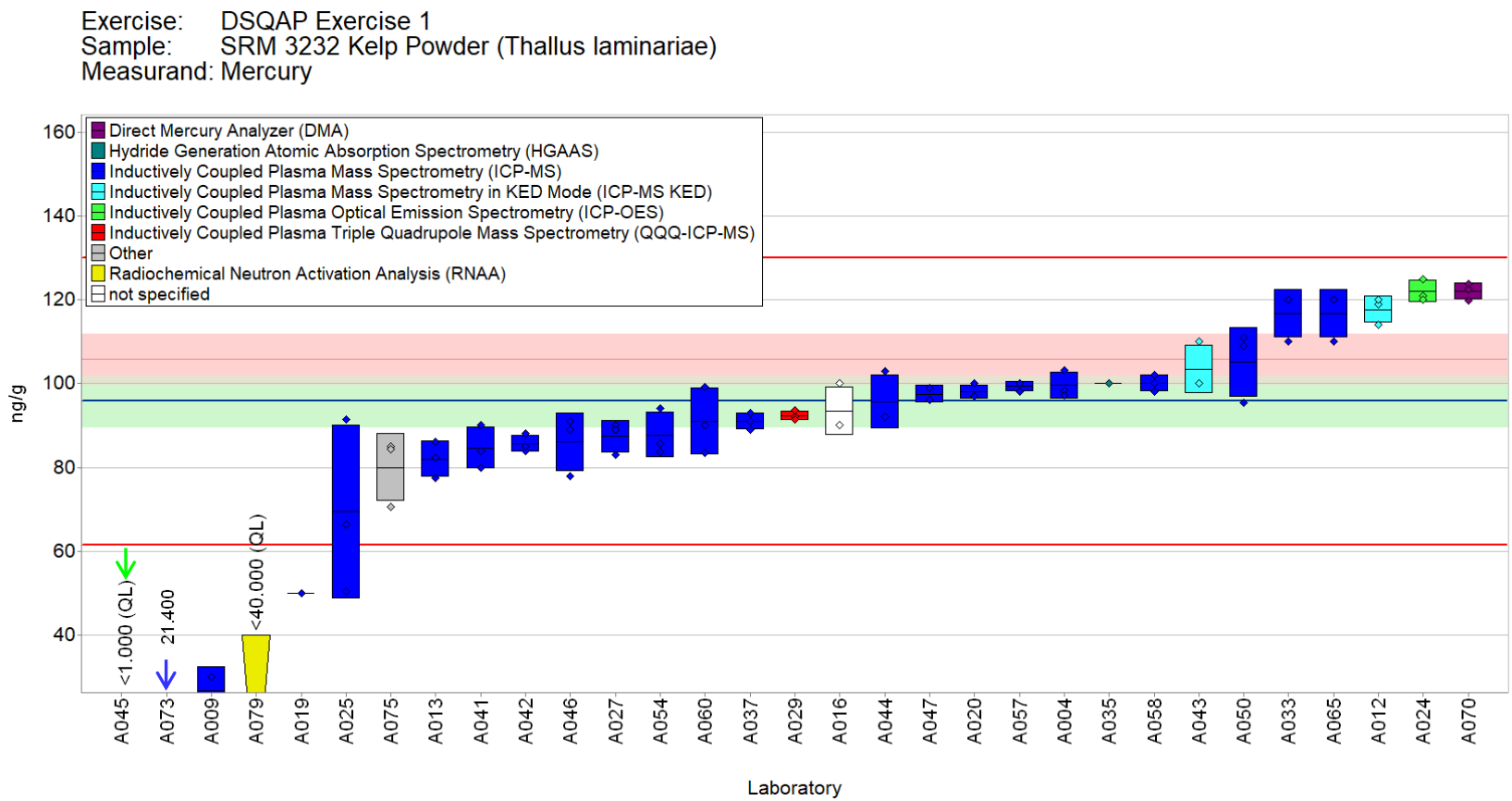

Fig. 3-33. Mercury in SRM 3232 Kelp Powder (*Thallus laminariae*) (data summary view – analytical method).

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'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

3.4.11. Potassium (K)

Target values for K are summarized in **Table 3-23**. The target values for K in Kelp A and Kelp B were determined at NIST using nitric and hydrofluoric acid assisted microwave digestion and ICP-OES. The target value for K in SRM 3232 was determined at NIST using (1) INAA after the material was pressed into pellets using a stainless-steel die and (2) nitric acid assisted microwave digestion and ICP-OES.

Table 3-23. Summary of results and laboratory variabilities for K in kelp.

Potassium (K)			
Within-Laboratory Variability (% RSD)	Averaged for all Samples		Maximum
	4.7 %		>100 %
	Kelp A	Kelp B	SRM 3232
Between-Laboratory Variability (% RSD)	11 %	9 %	9 %
Target Value ± U_{95} Mass Fraction (mg/g)	126.3 ± 2.5	21.8 ± 0.77	71.2 ± 1.0
Consensus Mean ± SD Mass Fraction (mg/g)	118 ± 12	20.7 ± 1.6	67.7 ± 5.6

For the determination of K, 33 of 51 laboratories reported results (65 %). The average within-laboratory variability was 4.7 %, with only six laboratories above 5 %, which indicates that most participants' in-house methods achieve repeatability consistent with published requirements (5 %, [6]). At least one laboratory reported variability greater than 100 %, and these results should be assessed for any calculation and reporting errors. The between-laboratory variabilities ranged from 9 % to 11 % in the three materials (**Table 3-23**), consistent with the published expectation of 10 % for laboratories using the same method [6]). Notably, the upper analytical range in the SMPR is 0.02 mg/g, which is lower than the K levels in the kelp materials and appropriate sample preparation steps (i.e., dilution) should be taken to ensure that the concentration of K in the extracted sample is within the calibration range.

Half of the laboratories that reported a sample preparation method indicated using microwave digestion as shown in **Table 3-24**. The majority of laboratories reported using either ICP-MS or ICP-OES as their analytical method. No method bias was observed although 30 % of laboratories did not report a sample preparation method.

Table 3-24. Summary of sample preparation and analytical methods averaged across all samples for determination of K in kelp.

Sample Preparation		Analytical Method	
Acid Digestion (no heat or microwave)	9 %	ICP-MS	44 %
Hot Block Digestion	9 %	ICP-OES	38 %
Microwave Digestion	50 %	QQQ-ICP-MS	3 %
Open Beaker Digestion	2 %	ICP	3 %
None Reported	30 %	AAS	3 %
		RNAA	3 %
		Other/None Reported	6 %

The consensus mean for K was below the target value in all three kelp samples, as seen in **Fig. 3-34**, **3-35**, and **3-36** for Kelp A, Kelp B, and SRM 3232, respectively. The consensus confidence interval was below the target range for Kelp A and SRM 3232 and was overlapping the lower range of the target range for Kelp B. Most laboratories were able to successfully measure K in Kelp B but about half of the laboratories measured outside of the target ranges for Kelp A and SRM 3232. Since Kelp A and SRM 3232 contained approximately three to six times more K than Kelp B, dilution factors should be evaluated that can impact as-measured mass fractions relative to the calibration scheme. Potassium is stable in an aqueous solution. Therefore, the primary consideration in the sample preparation for K measurements should be a complete digestion and minimization of contamination and sample loss, which could result in laboratories reporting low values.

Numerous analytical methods can accurately determine potassium. Potassium is easily atomized and ionized, which makes the element a good candidate for flame atomic absorption spectroscopy (FAAS) and flame atomic emission spectroscopy (FAES) measurements. Potassium has three stable isotopes at *m*/*z* 39, 40, and 41 that can be used in measurements by ICP-MS, but interferences from $^{38}Ar^{1}H^{+}$, $^{40}Ar^{+}$, and $^{40}Ar^{1}H^{+}$, respectively, must be avoided. A cool plasma condition should be used to minimize the isobaric interference from $40Ar^{1}H^{+}$ at *m*/*z* 39 [11]. Alternatively, the isobaric interferences at *m*/*z* 39 can be resolved by measuring $^{39}K^{+}$ at mass resolutions greater than 8000 using HR-ICP-MS [12]. Use of ICP-OES is not recommended for potassium measurement, as the difficulty of exciting the electron shell of potassium ions results in low sensitivity.

Additional tables and figures for K in kelp are located in **Appendix B**.

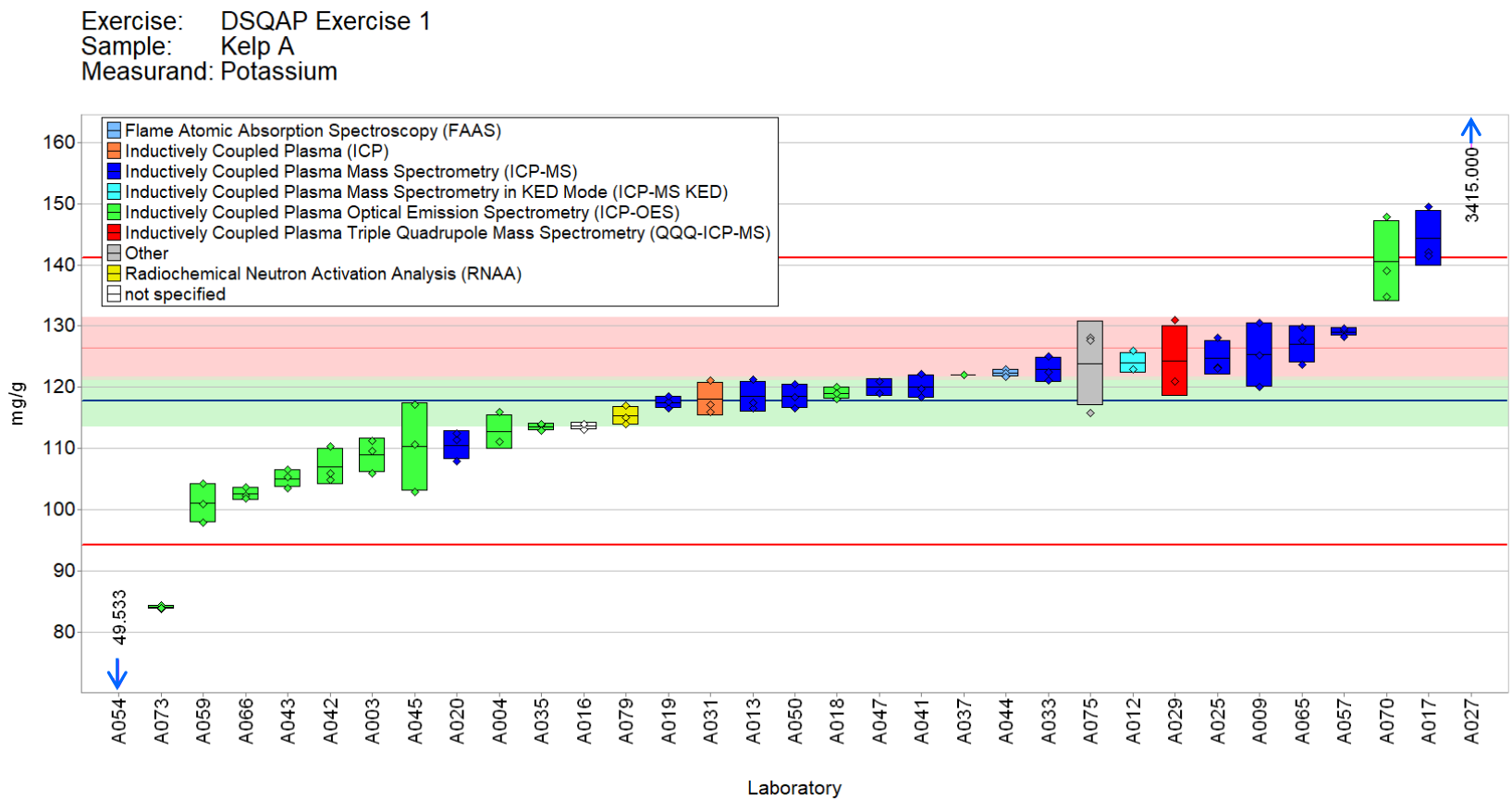

Fig. 3-34. Potassium in Kelp A (data summary view – analytical method).

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

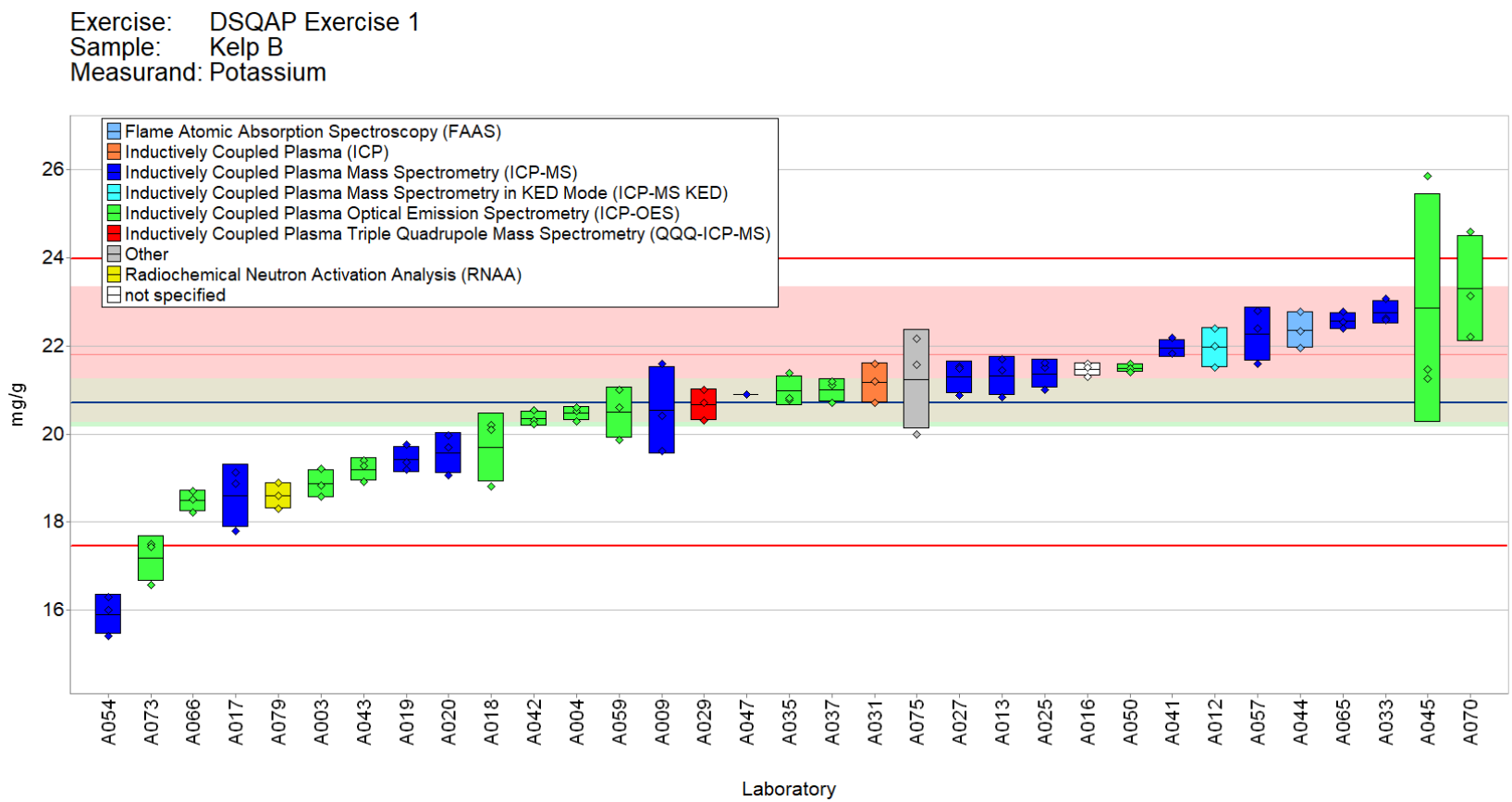

Fig. 3-35. Potassium in Kelp B (data summary view – analytical method).

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 (beige here due to overlap with the red NIST target range) represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$.

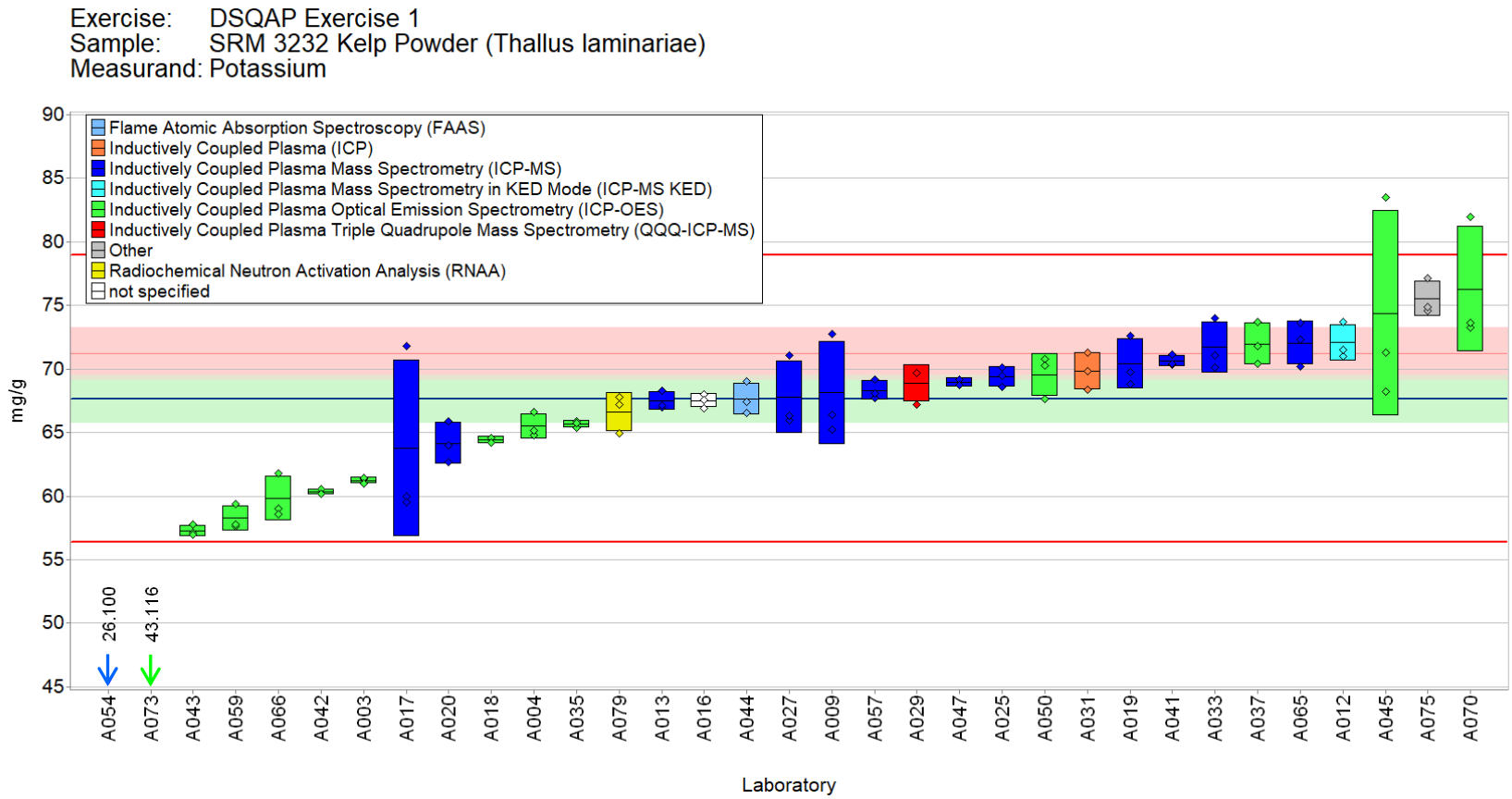

Fig. 3-36. Potassium in SRM 3232 Kelp Powder (*Thallus laminariae*) (data summary view – analytical method).

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

3.4.12. Selenium (Se)

Target values for Se are summarized in **Table 3-25**. The target values for Se in all samples were determined at NIST using nitric acid assisted microwave digestion and ICP-MS.

Table 3-25. Summary of results and laboratory variabilities for Se in kelp.

Selenium (Se)			
Within-Laboratory Variability (% RSD)	Averaged for all Samples		Maximum
	14 %		>100 %
	Kelp A	Kelp B	SRM 3232
Between-Laboratory Variability (% RSD)	93 %	95 %	99 %
Target Value ± U_{95} Mass Fraction (ng/g)	89.2 ± 4.7	38.3 ± 6.9	53 ± 14
Consensus Mean ± SD Mass Fraction (ng/g)	170 ± 150	110 ± 95	120 ± 110

For the determination of Se, 27 of 47 laboratories reported results (57 %). Of the 22 laboratories reporting quantitative results, 10 laboratories reported within-laboratory variabilities outside the published requirement of 15 %, as seen in **Table 3-25** [4]. The average within-laboratory variability was 14 %, indicating that many participants' in-house methods achieve successful repeatability. At least one laboratory reported variability greater than 100 %, and these results should be assessed for any calculation and reporting errors. The community struggled with the analysis of Se in kelp, indicated by the between-laboratory variabilities between 93 % and 99 %. The SMPR specifies successful reproducibility to be at or below 32 % for laboratories using the same method [4].

Most of the laboratories that reported a sample preparation method indicated using microwave digestion as seen in **Table 3-26**. The majority of laboratories reported using ICP-MS as their analytical method, including results off scale in **Fig. 3-37** and **3-38**. No method bias was observed although 39 % of laboratories did not report a sample preparation method.

Table 3-26. Summary of sample preparation and analytical methods averaged across all samples for determination of Se in kelp.

Sample Preparation		Analytical Method	
Acid Digestion (no heat or microwave)	4 %	ICP-MS	73 %
Hot Block Digestion	15 %	ICP-OES	8 %
Microwave Digestion	40 %	QQQ-ICP-MS	4 %
Open Beaker Digestion	1 %	AAS	4 %
None Reported	39 %	RNAA	4 %
		Other/None Reported	7 %

The consensus mean was above the target value for all three kelp samples, as seen in **Fig. 3-37**, **3-38**, and **3-39** for Kelp A, Kelp B, and SRM 3232, respectively. The consensus confidence interval was outside of the target range for Kelp A and Kelp B while it slightly overlapped the upper portion of the target range for SRM 3232.

The Se mass fractions in the study samples were relatively low and could have challenged participants' method LOQs. Laboratories should ensure that LOQs have been appropriately determined and consider improving sensitivity through use of appropriate blanks, implementing system cleaning techniques, and minimizing interferences. The sample preparation approach used for Se determination should ensure appropriate dilution factors are used based on the level of Se expected in the sample, to prevent dilution of the prepared sample beyond the LOQ. Additionally, Se is highly volatile and may be lost during open beaker digestion resulting in a low bias. Laboratories that did not use microwave digestion and are consistently reporting low values should ensure their sample preparation method is appropriate. Closed vessel digestions should be opened with care to ensure no Se is lost during venting.

Spectral/isobaric interferences can make Se difficult to measure accurately by ICP-MS. Elevated mass fractions of certain elements (e.g., Ar) are known to cause interferences in the analysis of Se by ICP-MS, including Ar_2^*. Most Se isotopes also suffer isobaric overlap. Collision cell technology can be used to minimize molecular interferences that may be found in these three materials, and specifically use of He as the collision cell gas can reduce Ar_2^* inference. If using QQQ-ICP-MS, O_2 gas can be added to mass shift Se isotopes 16 *m*/*z* units higher than their native *m*/*z* state for measurement with reduced interference.

Additional tables and figures for Se in kelp are located in **Appendix B**.

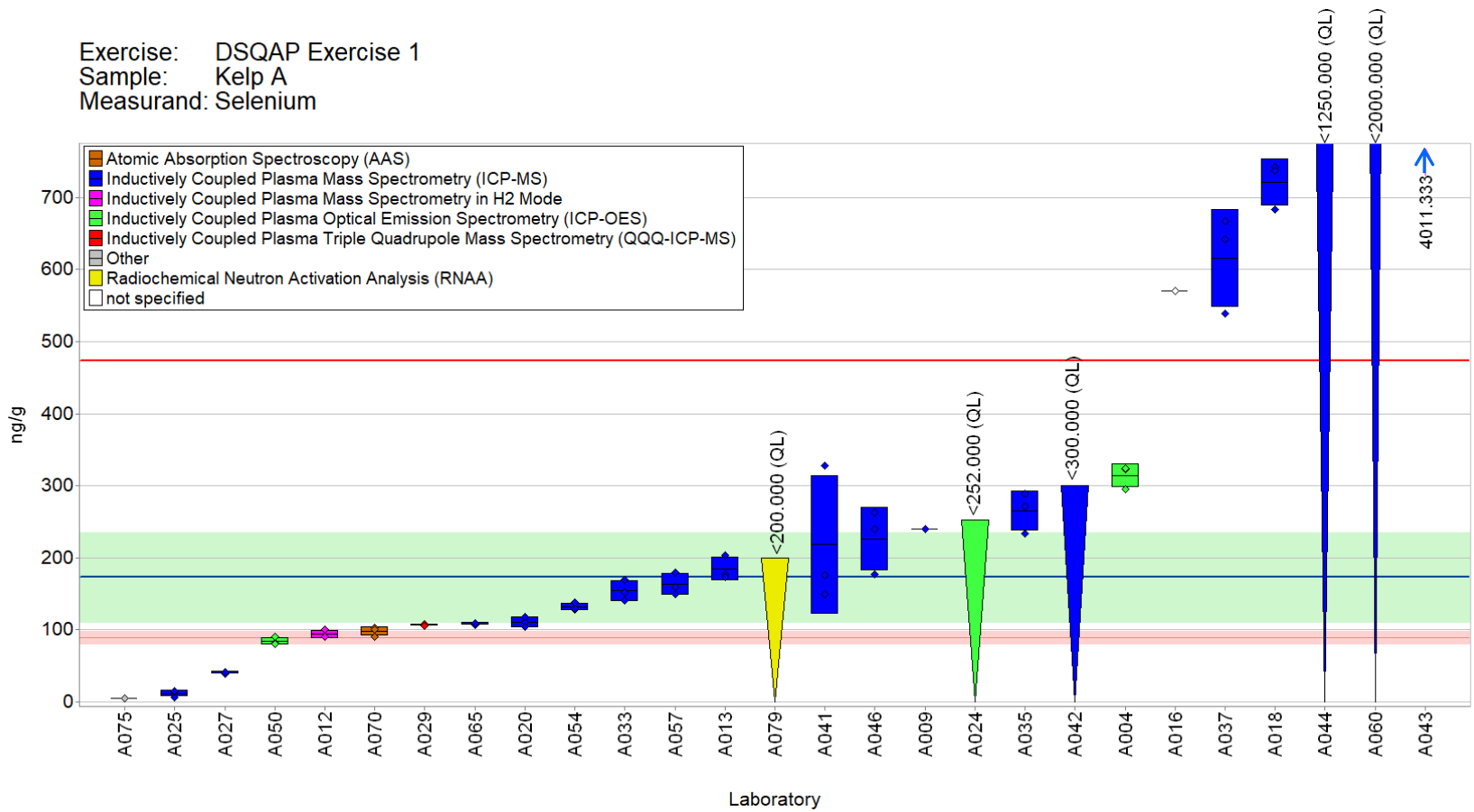

Fig. 3-37. Selenium in Kelp A (data summary view – analytical method).

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 bound of the consensus range of tolerance, calculated as the value above the consensus mean that results in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$, with the lower bound set to zero. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

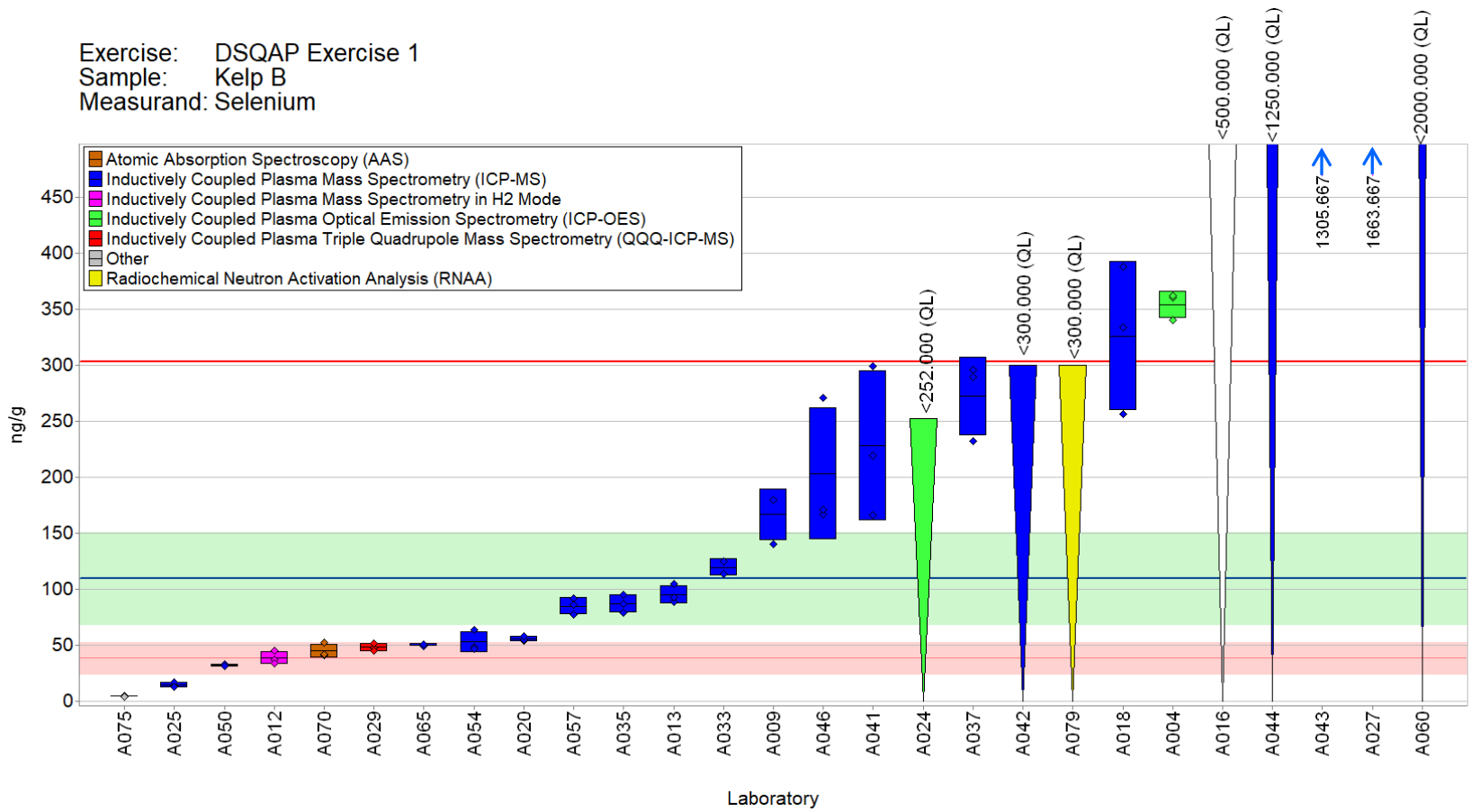

Fig. 3-38. Selenium in Kelp B (data summary view – analytical method).

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 bound of the consensus range of tolerance, calculated as the value above the consensus mean that results in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$, with the lower bound set to zero. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$.

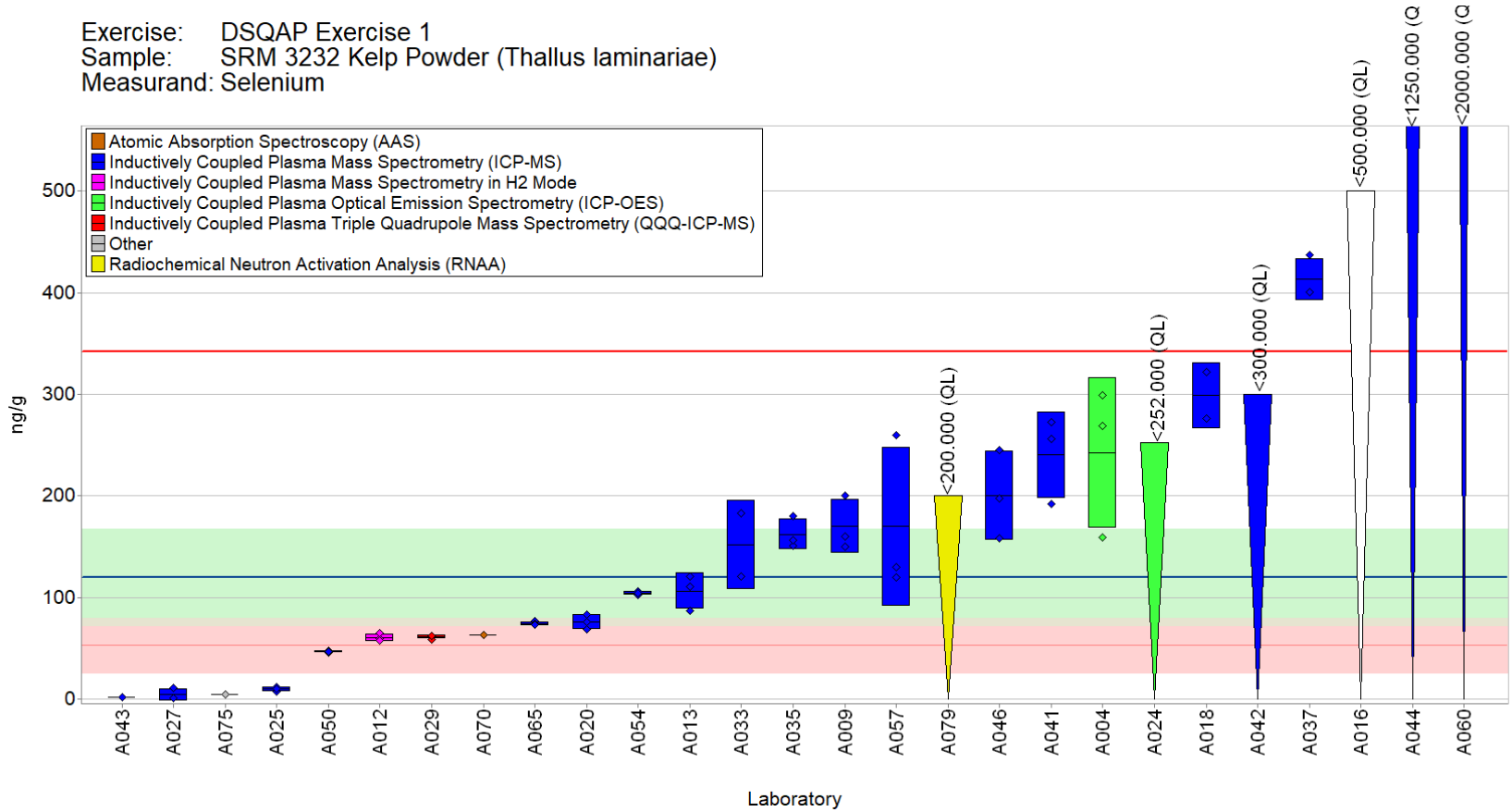

Fig. 3-39. Selenium in SRM 3232 Kelp Powder (*Thallus laminariae*) (data summary view – analytical method).

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 bound of the consensus range of tolerance, calculated as the value above the consensus mean that results in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$, with the lower bound set to zero. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

3.4.13. Sodium (Na)

Target values for Na are summarized in **Table 3-27**. The target values for Na in Kelp A and Kelp B were determined at NIST using nitric and hydrofluoric acid assisted microwave digestion and ICP-OES. The target value for Na in SRM 3232 was determined at NIST using (1) INAA after the material was pressed into pellets using a stainless-steel die and (2) nitric acid assisted microwave digestion and ICP-OES.

Table 3-27. Summary of results and laboratory variabilities for Na in kelp.

Sodium (Na)			
Within-Laboratory Variability (% RSD)	Averaged for all Samples		Maximum
	2.6 %		17 %
	Kelp A	Kelp B	SRM 3232
Between-Laboratory Variability (% RSD)	10 %	11 %	9 %
Target Value ± U_{95} Mass Fraction (µg/g)	39460 ± 130	31740 ± 430	15300 ± 360
Consensus Mean ± SD Mass Fraction (µg/g)	39300 ± 3400	30900 ± 3100	14700 ± 1300

For the determination of Na, 29 of 49 laboratories returned data (59 %). All laboratories reported quantitative values, and only 3 laboratories reported within-laboratory variabilities above the published 5 % recommendation [6]. The average within-laboratory variability was 2.6 % (**Table 3-27**), which indicates that most participants' in-house methods achieve acceptable repeatability. The average between-laboratory variability was at or below 11 % for all samples, as seen in **Table 3-27**, indicating that the community is performing well with respect to the 10 % published recommendation for laboratories using the same method [6]. Notably, the upper analytical range in the SMPR (8.5 µg/g) is lower than the Na levels in the kelp samples, so appropriate sample preparation steps (i.e., dilution) should be taken to ensure that the level of Na in prepared samples is within the calibration range.

Nearly half of the laboratories that reported a sample preparation method indicated using microwave digestion as seen in **Table 3-28**. Almost half of the laboratories reported using ICP-MS as their analytical method, including results off scale in **Fig. 3-40** and **3-42**, along with one-third of the laboratories using ICP-OES. No method bias was observed although 35 % of laboratories did not report a sample preparation method. While the laboratory using QQQ-ICP-MS consistently reported lower values, QQQ cannot be correlated with low bias based on the results from a single laboratory.

Table 3-28. Summary of sample preparation and analytical methods averaged across all samples for determination of Na in kelp.

Sample Preparation		Analytical Method	
Acid Digestion (no heat or microwave)	10 %	ICP-MS	49 %
Hot Block Digestion	10 %	ICP-OES	36 %
Microwave Digestion	45 %	QQQ-ICP-MS	3 %
None Reported	35 %	ICP	3 %
		AAS	3 %
		RNAA	3 %
		Other/None Reported	3 %

The consensus mean was below the target value for all three kelp samples, as seen in **Fig. 3-40**, **3-41**, and **3-42** for Kelp A, Kelp B, and SRM 3232, respectively. The consensus confidence interval was completely overlapping the target range for Kelp A and overlapping the lower range of the target ranges for Kelp B and SRM 3232.

No trends based on analytical method were observed, but appropriate sample preparation could play a large role in accurate determination of Na. Laboratories that consistently reported low values should ensure their approach achieves complete digestion of Na, as open beaker digestions are more likely to be incomplete and result in a low bias compared to microwave digestion techniques. Laboratories that consistently report high values likely have issues with contamination of samples with Na from the environment during sample preparation. Na is present in the matrix of most samples and most instruments can be easily contaminated by Na. The sample introduction system should be cleaned and the baseline counts checked to ensure the instrument is suitable for the measurement. Similarly, work areas, glassware and plasticware, etc. should be acid cleaned prior to use to prevent Na contamination from common soaps. If a soap solution is used for cleaning, Na levels in blank solutions must be rigorously checked. When performing measurements, the concentration of Na in the sample should be prepared at the high end of the linear range to minimize the Na baseline intensity relative to the Na signal from the sample. Extra procedural reagent blanks should be prepared along long with samples to understand the extent of Na contamination from the analysis.

Additional tables and figures for Na in kelp are located in **Appendix B**.

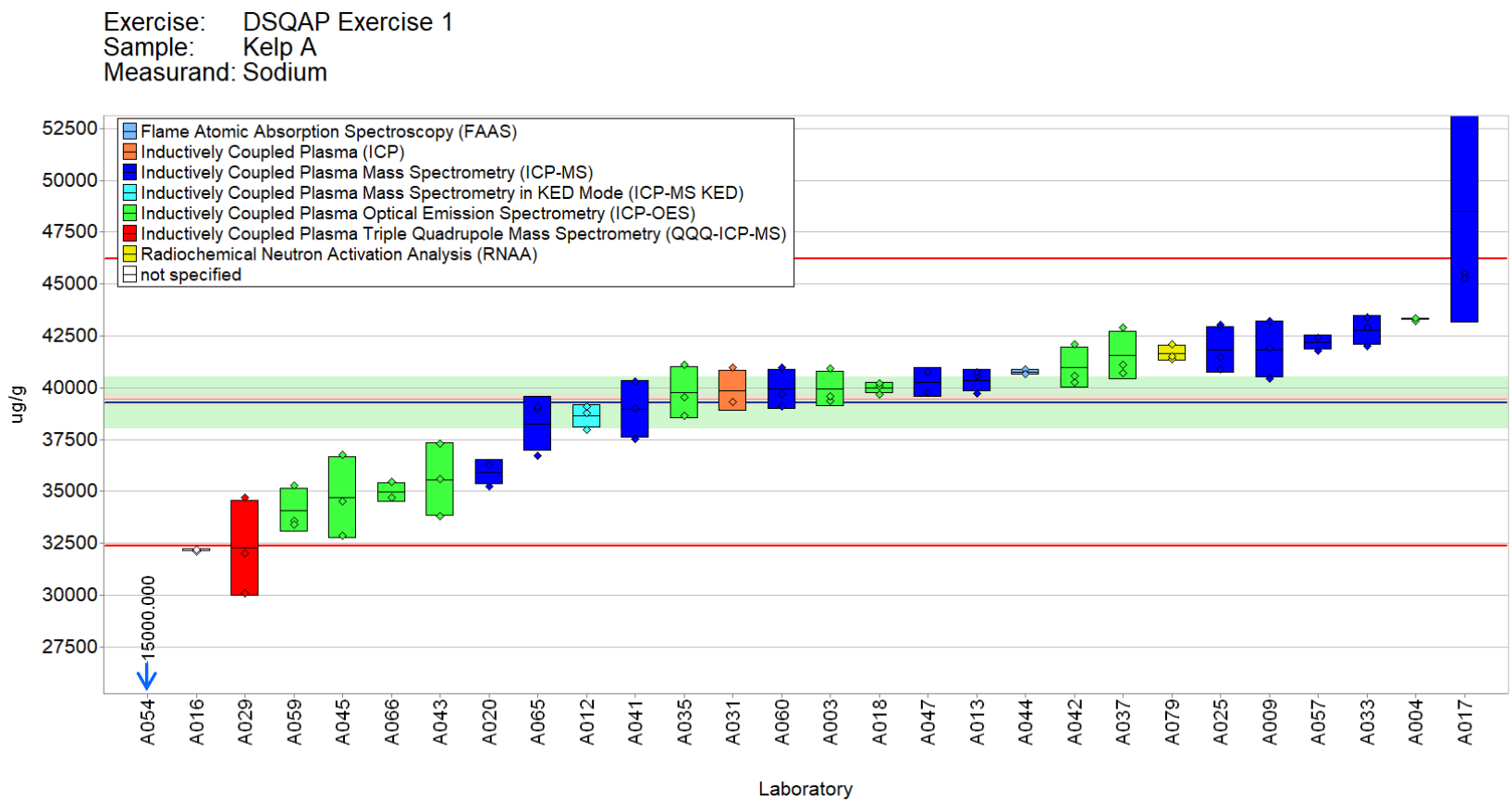

Fig. 3-40. Sodium in Kelp A (data summary view – analytical method).

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'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$. The red shaded region (beige here due to overlap with the green consensus confidence interval) represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$.

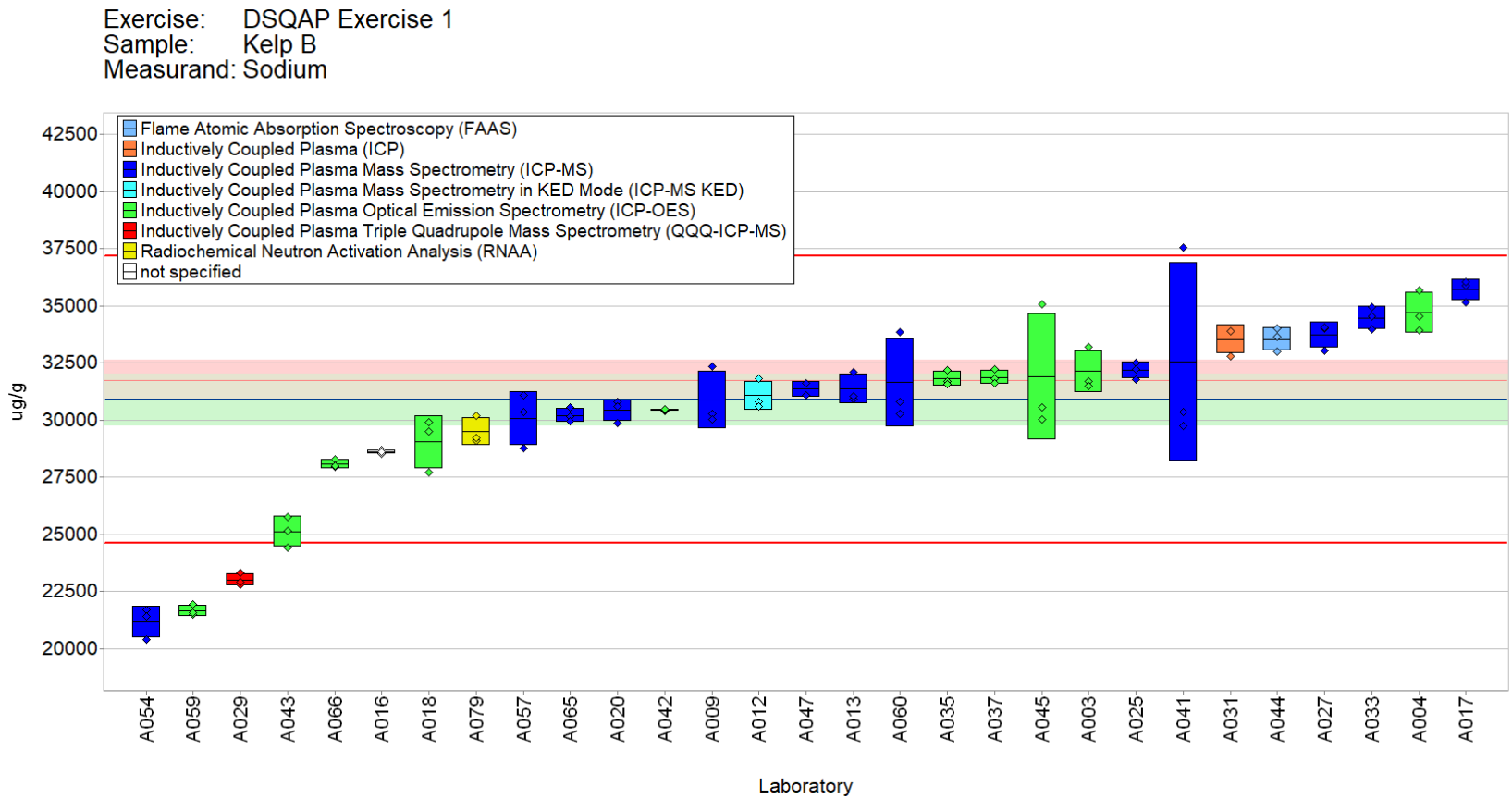

Fig. 3-41. Sodium in Kelp B (data summary view – analytical method).

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'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

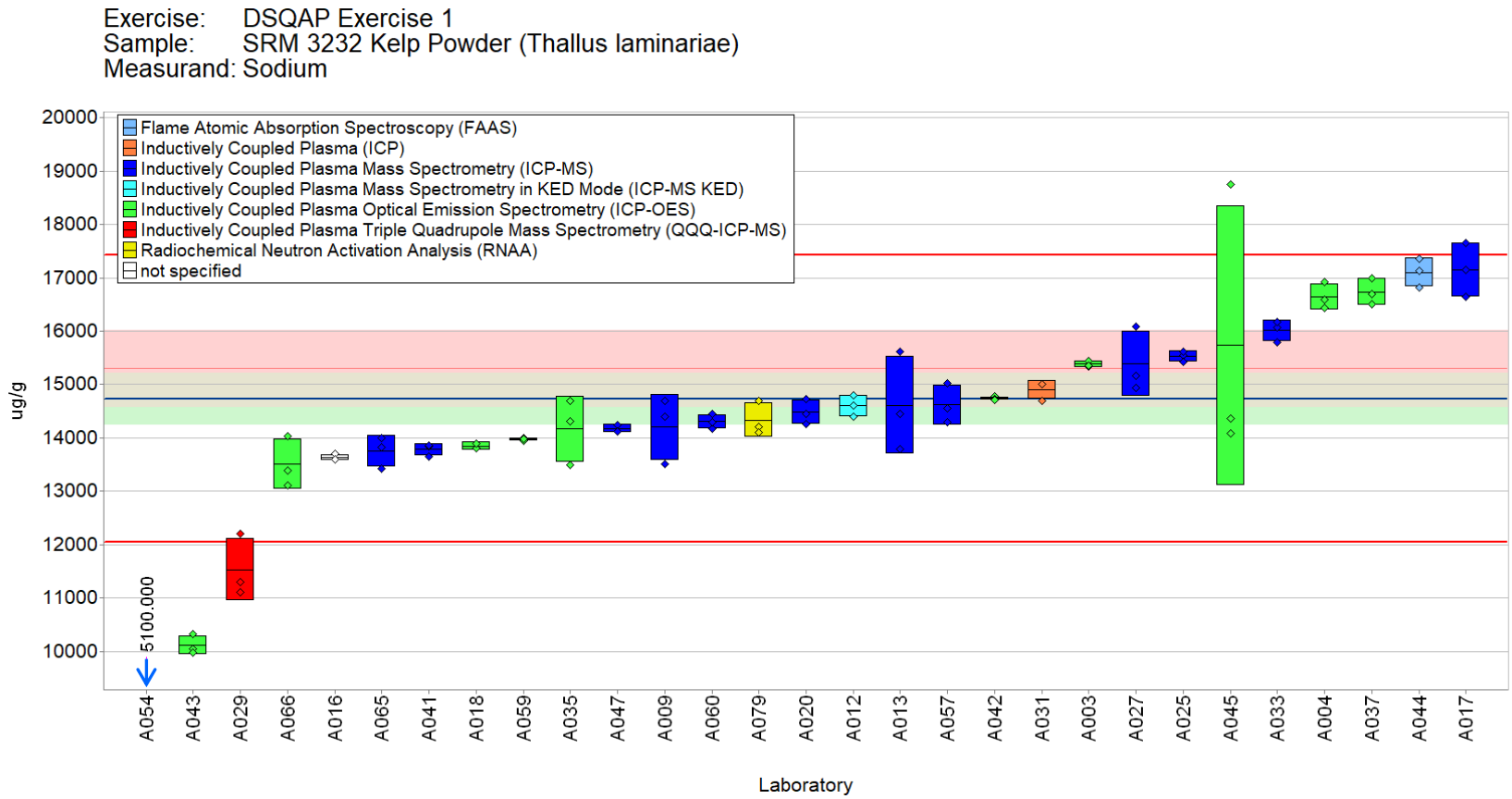

Fig. 3-42. Sodium in SRM 3232 Kelp Powder (*Thallus laminariae*) (data summary view – analytical method).

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'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

3.4.14. Sulfur (S)

For the determination of S, 14 of 42 laboratories reported results (33 %). The average within-laboratory variability for the 13 laboratories reporting quantitative values was 2.4 %, with all laboratories at or below 11 % repeatability, as seen in **Table 3-29**. The between-laboratory variabilities were between 18 % and 21 %. At the time of this report, target values for S were not available in any of the kelp samples and a suitable SMPR for method performance assessment was not identified.

Table 3-29. Summary of results and laboratory variabilities for S in kelp.

Sulfur (S)			
Within-Laboratory Variability (% RSD)	Averaged for all Samples		Maximum
	2.4 %		11 %
	Kelp A	Kelp B	SRM 3232
Between-Laboratory Variability (% RSD)	21 %	21 %	18 %
Consensus Mean ± SD Mass Fraction (µg/g)	6600 ± 1400	22100 ± 3300	10600 ± 1900

Half of the laboratories that reported a sample preparation method indicated using microwave digestion as shown in **Table 3-30**. Many different analytical methods were reported, as seen in **Fig. 3-43**, **3-44**, and **3-45** for Kelp A, Kelp B, and SRM 3232, respectively, making identification of trends in the data regarding method bias difficult. Five laboratories reported ICP-OES and three laboratories reported using ICP-MS (including the results off scale in **Fig. 3-43** and **3-35**). The results using the C/S analyzer and QQQ-ICP-MS were consistently lower than the consensus mean in all samples, but a low bias cannot be directly correlated to these methods since only one laboratory reported each.

Table 3-30. Summary of sample preparation and analytical methods averaged across all samples for determination of S in kelp.

Sample Preparation		Analytical Method	
Hot Block Digestion	14 %	ICP-MS	29 %
Microwave Digestion	50 %	ICP-OES	36 %
Thermal Decomposition	7 %	QQQ-ICP-MS	7 %
None Reported	29 %	ICP	7 %
		RNAA	7 %
		C/S Analyzer	7 %
		Other/None Reported	7 %

Since no target values for S were available at the time of this report, no specific recommendations related to bias can be made. In general, unmitigated spectral interferences can lead to a high bias in S results obtained using ICP-MS techniques. Traditionally, collision cell technology or a C/S analyzer can be used to minimize interferences. Very few laboratories reported using an analytical technique that mitigated interferences and consequently the consensus data could be biased high relative to a true value.

Additional tables and figures for S in kelp are located in **Appendix B**.

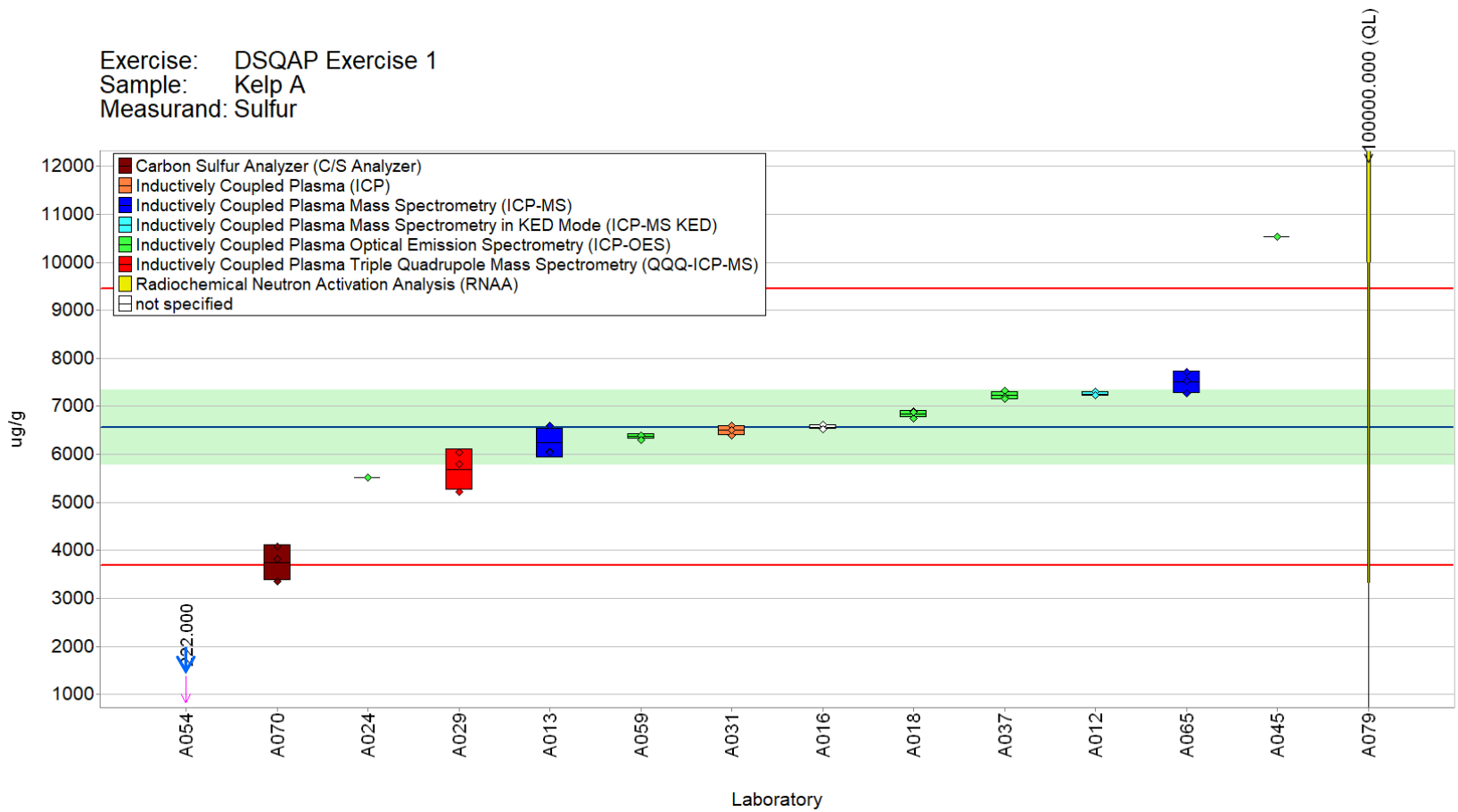

Fig. 3-43. Sulfur in Kelp A (data summary view – analytical method).

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'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

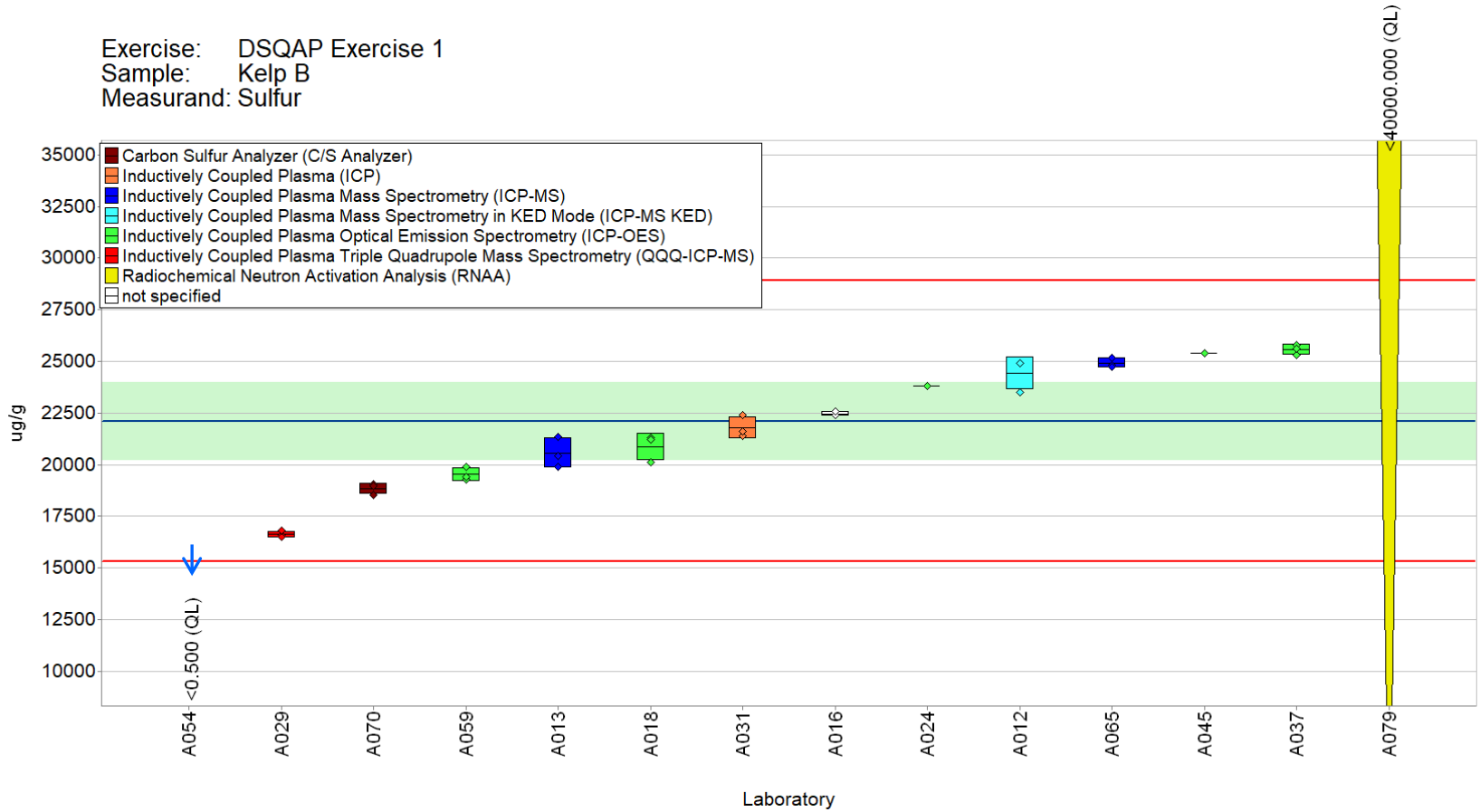

Fig. 3-44. Sulfur in Kelp B (data summary view – analytical method).

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'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

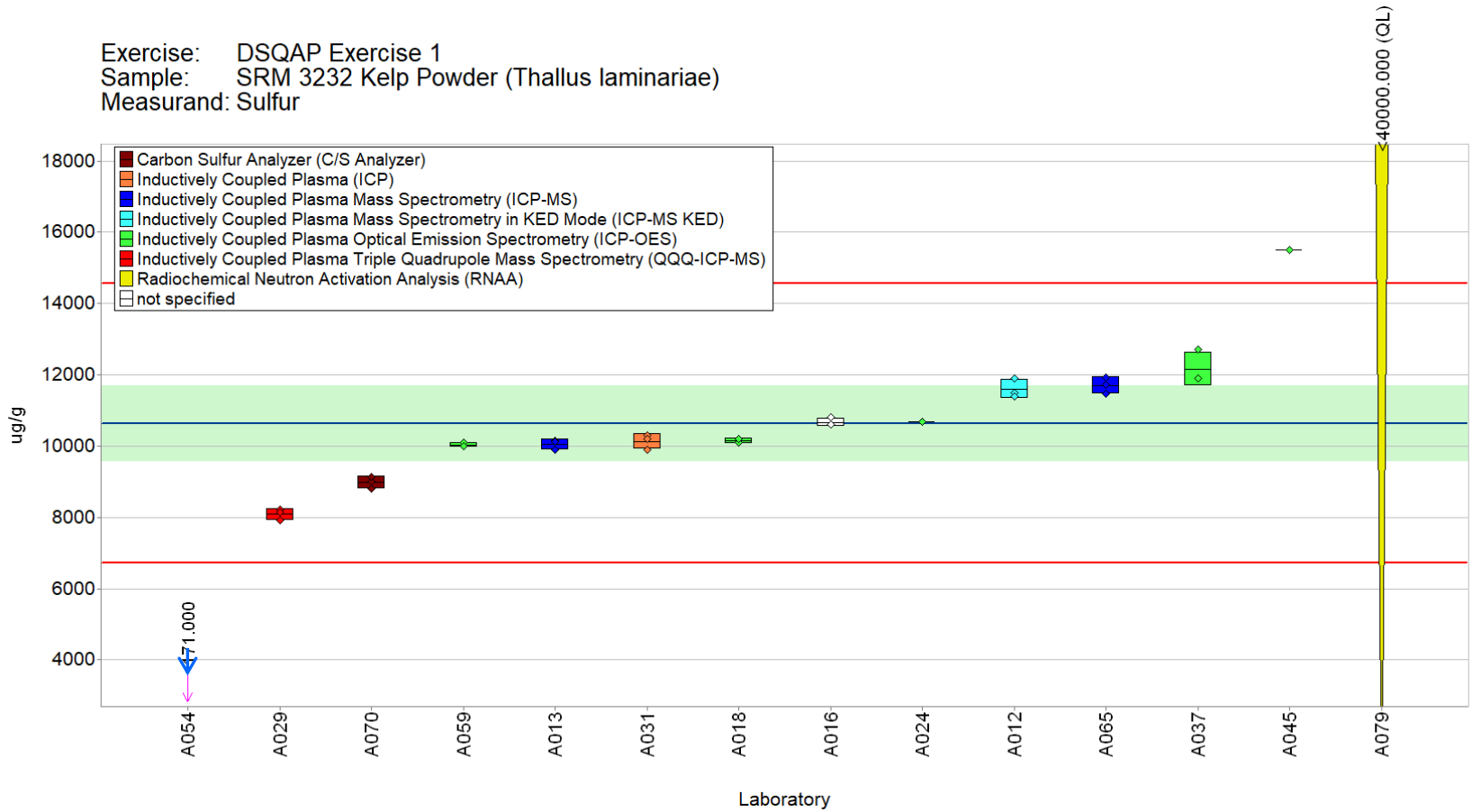

Fig. 3-45. Sulfur in SRM 3232 Kelp Powder (*Thallus laminariae*) (data summary view – analytical method).

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'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

3.4.15. Zinc (Zn)

Target values for Zn are summarized in **Table 3-31**. The target values for Zn in Kelp A and Kelp B were determined at NIST using nitric and hydrofluoric acid assisted microwave digestion and ICP-OES. The target value for Zn in SRM 3232 was determined at NIST using (1) INAA after the material was pressed into pellets using a stainless-steel die and (2) nitric acid assisted microwave digestion and ICP-OES.

Table 3-31. Summary of results and laboratory variabilities for Zn in kelp.

Zinc (Zn)			
Within-Laboratory Variability (% RSD)	Averaged for all Samples		Maximum
	6.0 %		83 %
	Kelp A	Kelp B	SRM 3232
Between-Laboratory Variability (% RSD)	17 %	14 %	16 %
Target Value ± U_{95} Mass Fraction (µg/g)	18.6 ± 1.7	32.0 ± 3.2	25.7 ± 1.0
Consensus Mean ± SD Mass Fraction (µg/g)	18.0 ± 3.1	31.5 ± 4.4	25.6 ± 4.0

For the determination of Zn, 28 of 51 laboratories reported results (56 %). Of those 28 laboratories, 3 laboratories reported within-laboratory variabilities above the published recommendation of 7.3 %, as seen in **Table 3-31** [4]. The average within-laboratory variability was 6.0 %, indicating that much of the community is achieving acceptable repeatability for determination of Zn. One laboratory reported variability above 80 %, and these results should be assessed for any calculation or reporting errors. Between-laboratory variabilities were between 14 % and 17 %, which is significantly higher than the published expectation of below 8 % for laboratories using the same method [4]. Notably, the upper analytical range in the SMPR (10 µg/g) is lower than the Zn levels in the kelp materials and appropriate sample preparation steps (i.e., dilution) should be taken to ensure that the samples as measured are within the calibration range.

Most of the laboratories that reported a sample preparation method indicated using microwave digestion as shown in **Table 3-32**. The majority of laboratories used ICP-MS as their analytical method followed by ICP-OES as the second most reported. No method bias was observed although 39 % of laboratories did not report a sample preparation method.

Table 3-32. Summary of sample preparation and analytical methods averaged across all samples for determination of Zn in kelp.

Sample Preparation		Analytical Method	
Acid Digestion (no heat or microwave)	7 %	ICP-MS	56 %
Hot Block Digestion	4 %	ICP-OES	26 %
Microwave Digestion	49 %	QQQ-ICP-MS	4 %
Open Beaker Digestion	1 %	ICP	4 %
None Reported	39 %	RNAA	4 %
		Other/None Reported	7 %

The consensus mean was slightly below the target value for Zn in Kelp A and Kelp B, as seen in **Fig. 3-46** and **3-47** and was equal to the target value for SRM 3232, as seen in **Fig. 3-48**. The consensus confidence interval was within the target range for all three kelp samples. Most laboratories were able to successfully measure Zn in the three kelp samples.

Bias in Zn results may have resulted from sample preparation method or calibration curve. Laboratories reporting results outside of the consensus range of tolerance should review the recommendations discussed in Section 2 and Section 3.1. When using ICP-MS, KED mode can work well for the reduction of PO_2^+ and SO_2^+ interferences on measurement of Zn.

Additional tables and figures for Zn in kelp are located in **Appendix B**.

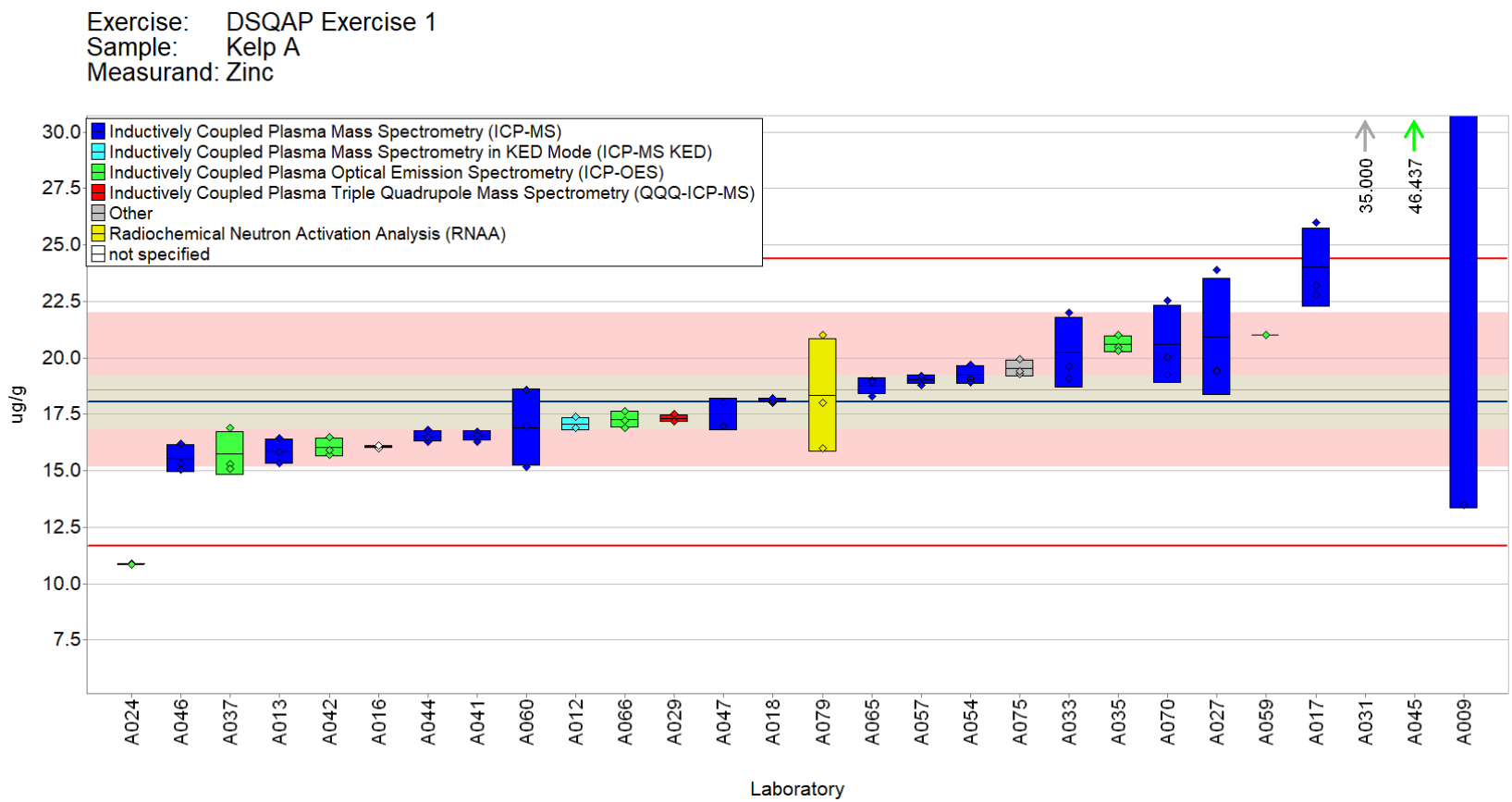

Fig. 3-46. Zinc in Kelp A (data summary view – analytical method).

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 (beige here due to overlap with the red NIST target range) represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$.

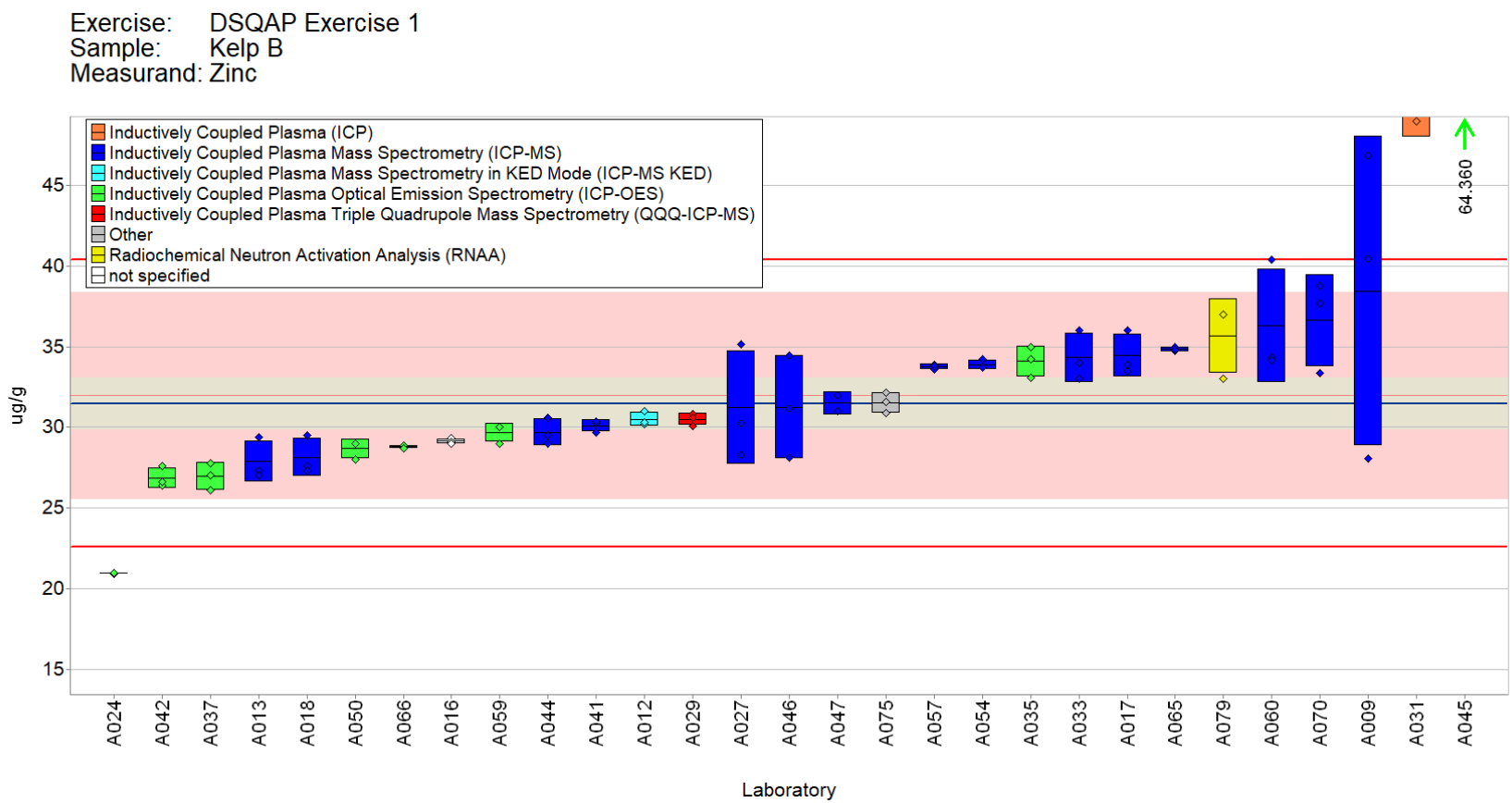

Fig. 3-47. Zinc in Kelp B (data summary view – analytical method).

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 (beige here due to overlap with the red NIST target range) represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$.

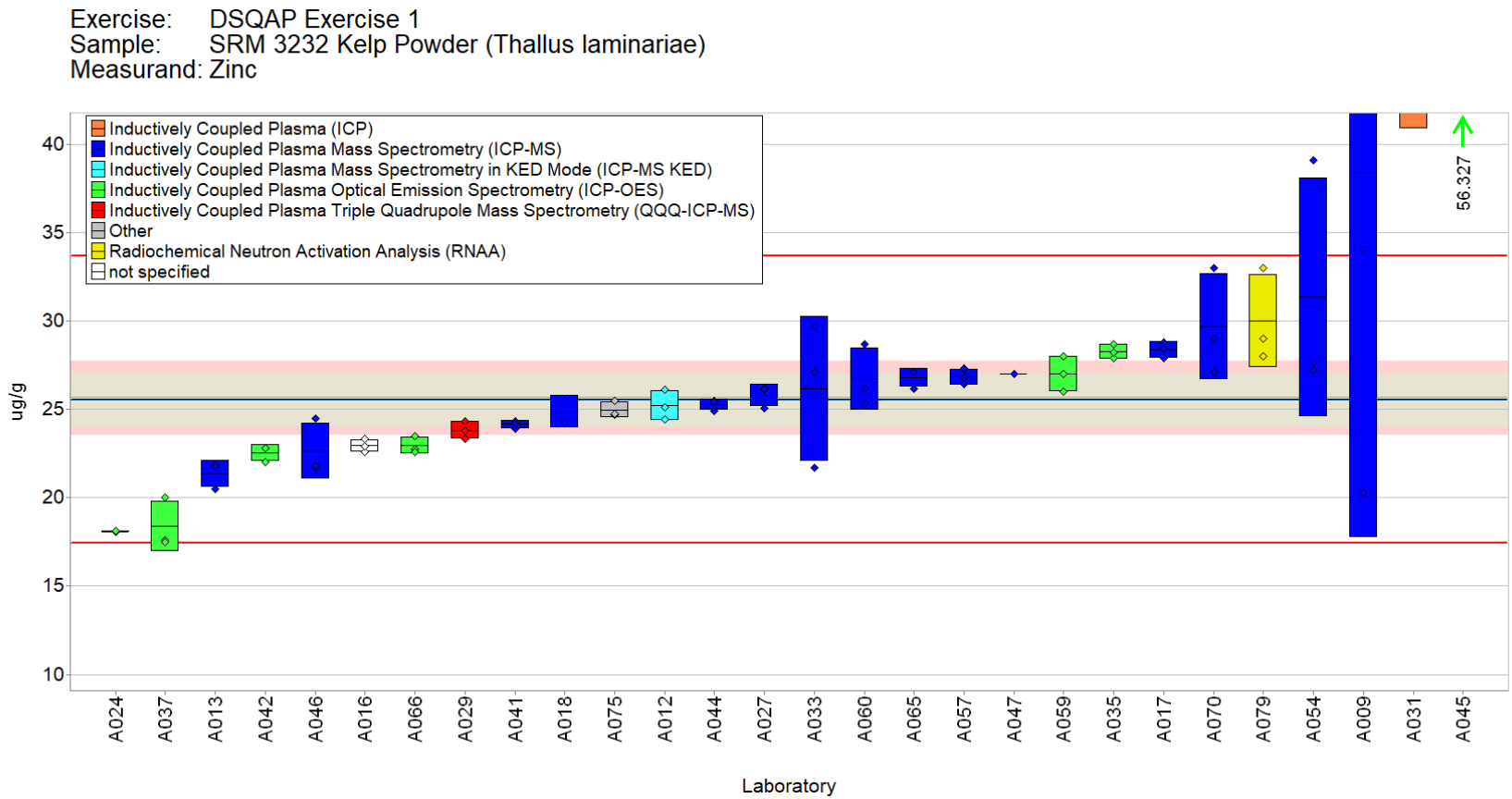

Fig. 3-48. Zinc in SRM 3232 Kelp Powder (*Thallus laminariae*) (data summary view – analytical method).

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 (beige here due to overlap with the red NIST target range) represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$.

4. Vitamins I (Vitamins B3 and K1 in kelp)

4.1. Executive Summary

Niacin and niacinamide (vitamin B_3) and phylloquinone (vitamin K_1) are important water- and fat-soluble vitamins known to be present in kelp. The participation rate in these studies was extremely low and prevented meaningful conclusions from being drawn about the methods used to determine these nutrients.

4.2. Background

Niacin and niacinamide (vitamin B_3) are water-soluble vitamins that are an essential part of many reactions in the body that require the coenzyme nicotinamide adenine dinucleotide (NAD). Vitamin B_3 is important for the conversion of food into energy and for the development and function of many cell types in the body [13, 14]. Vitamin K is the general name for a group of nutrient compounds that are important for blood clotting and healthy bones. Phylloquinone (vitamin K_1) one of the major forms found naturally in plants, is present in *cis*- and *trans*- forms, with *trans*-vitamin K_1 generally being the more dominant form. *cis*-vitamin K_1 is also thought to have lower bioactivity [15]. Kelp is often advertised for its health benefits, including vitamin and mineral content [16, 17]. According to USDA FoodData Central, kelp contains measurable levels of both niacin and phylloquinone [18]. The vitamin levels are likely dependent on species and growing conditions. Standardized methods for these vitamins in foods and dietary supplements, including seaweeds, can help ensure accurate dietary intake estimates and product labeling. Proper measurements and values can then help inform farming communities on the best species and growing seasons to achieve desired vitamin levels. In this study, participants were asked to use in-house analytical methods to determine the mass fraction (mg/kg) of niacin, niacinamide, and phylloquinone in three different species of powdered seaweeds.

4.3. Study Information

Participants were provided with samples of Kelp A (three 10 g packets, *Saccharina latissima f. angustissima*, Maine, USA), Kelp B (three 5 g packets, *Ascophyllum nodosum*, Northern Atlantic Ocean), and SRM 3232 (three 5 g packets, *Thallus laminariae*, East China Sea). Participants were asked to store the materials at controlled room temperature, 20 °C to 25 °C, in the original unopened packets and to prepare one sample and report one value from each packet provided. Before use, participants were instructed to thoroughly mix the contents of the packet and to allow contents to settle for one minute prior to opening to minimize the loss of fine particles prior to removal of a test portion for analysis, and to use sample sizes appropriate for their in-house analytical methods. Approximate analyte levels were not reported to participants prior to the study.

4.4. Study Results and Technical Recommendations

4.4.1. Niacin and Niacinamide

Of the 24 laboratories requesting samples for niacin and 20 laboratories requesting samples for niacinamide, only 3 to 6 laboratories reported results for each sample (15 % to 25 % participation). The low participation rate and lack of method information reported makes interpretation of the results difficult. Also, target values were not available at the time of this report. The within-laboratory variabilities were less than 4 % for niacin in all samples, and below 14 % for niacinamide in all samples. The between-laboratory variabilities indicate challenges across the community for these measurements, as % RSDs were between 36 % and 81 % for niacin and between 46 % and above 100 % for niacinamide.

Vitamin B_3 is relatively stable, making a variety of sample extraction techniques viable. Understanding the form(s) of vitamin B_3 being measured and reported is important, as vitamin B_3 can be reported as a total (as either niacin or niacinamide) or in the individual forms. Laboratories that provided sample preparation information reported using enzymatic digestion, solvent extraction, or solvent extraction and solid phase extraction for the determination of niacin. One laboratory reported using enzymatic digestion for the determination of niacinamide. While an aqueous extraction can be used for free and fortified niacin, bound niacin needs to be released from any complex forms as well as from the sample matrix. Additionally, base hydrolysis will convert all forms to niacin [19]. Enzymatic hydrolysis can be used, and the measured vitamin form will depend on the enzyme(s) used. It is difficult to determine if laboratories reported total B_3 or specific vitamers due to low participation in this study. In the Water-Soluble Vitamin study, it is evident that some participants reported total B_3 when asked to report niacin. Participants should be aware of the specific vitamin forms their methods are able to determine to better understand total vitamin composition in the material. For additional vitamin B_3 related discussion, please see **Section 8** of this report.

Laboratories that provided analytical method information reported using liquid chromatography mass spectrometry (LC-MS), liquid chromatography with tandem mass spectrometry (LC-MS/MS), microbiological assay, or spectrophotometry for the determination of niacin. Laboratories reported using LC-MS, LC-MS/MS, or other for the determination of niacinamide. **Figures 4-1** and **4-2** show the reported results with analytical methods indicated by different colors. No method bias was observed.

Laboratories reporting LOQs of above 100 mg/kg for both niacin and niacinamide should assess their LOQ determination process and their analytical methods.

Additional tables and figures for vitamin B_3 in kelp are located in **Appendix C**.

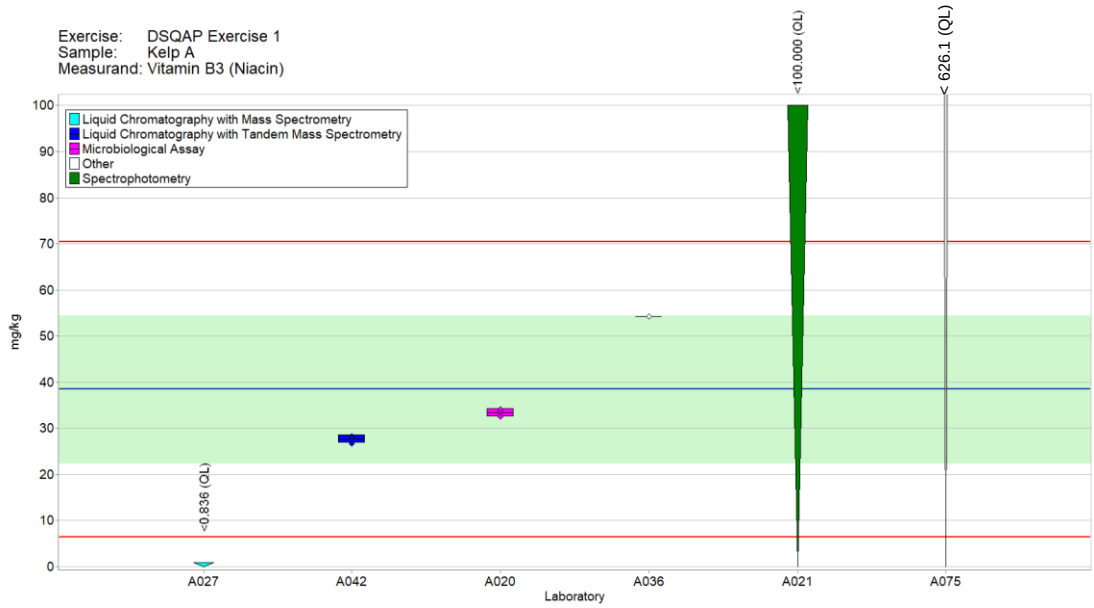

Fig. 4-1. Vitamin B_3 (niacin) in Kelp A (data summary view – analytical method).

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'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

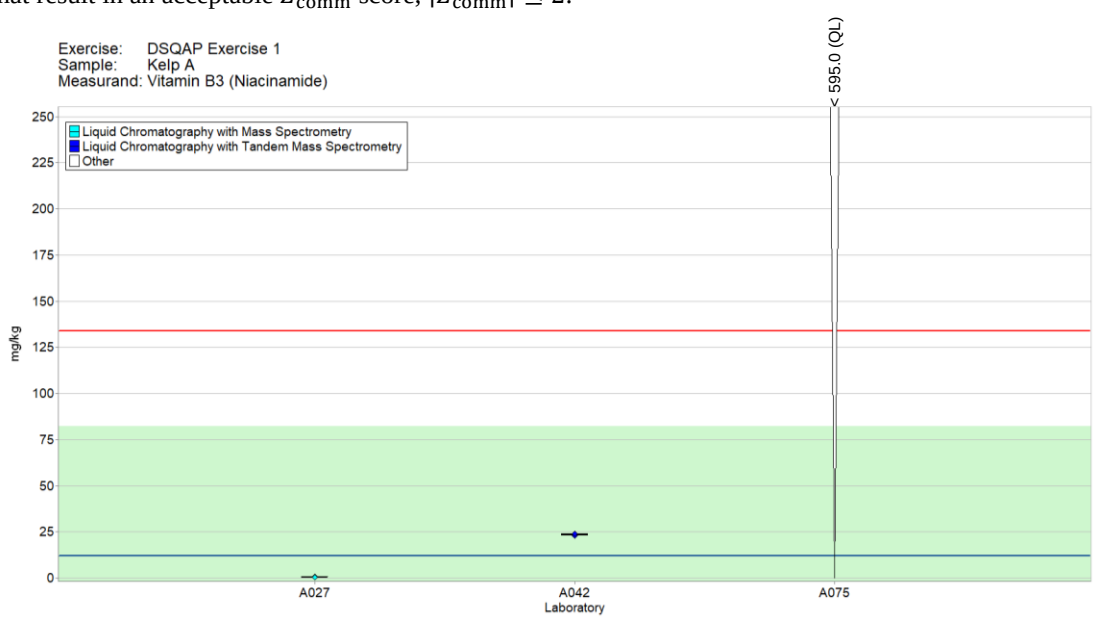

Fig. 4-2. Vitamin B_3 (niacinamide) in Kelp A (data summary view – analytical method).

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 bound of the consensus range of tolerance, calculated as the value above the consensus mean that results in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$ with the lower bound set to zero.

4.4.2. Phylloquinone

Of the 19 laboratories requesting samples for phylloquinone, only 4 laboratories reported results for each sample (21 % participation). The low participation rate and lack of method information reported makes interpretation of the results difficult. The within-laboratory variabilities were between 18 % and 22 %, and the between-laboratory variabilities indicate challenges across the community for these measurements, as % RSDs were between 29 % and above 100 %. Phylloquinone target values and consensus values are listed in **Table 4-1**. A target value for phylloquinone in Kelp A was not available at the time of this report. The target value for phylloquinone in Kelp B was determined by combining individual laboratory results from DSQAP Exercise M [20] and HAMQAP Exercise 7 [21]. The moisture corrected target value for phylloquinone in SRM 3232 was determined at NIST by ID-LC-MS/MS.

Table 4-1. Summary of results and laboratory variabilities for phylloquinone in kelp.

Phylloquinone

Sample	Target Value ± U_{95} Mass Fraction (mg/kg)	Consensus Mean ± SD Mass Fraction (mg/kg)
Kelp A	-	3.7 ± 4.4
Kelp B	1.94 ± 0.50	1.70 ± 0.49
SRM 3232	0.404 ± 0.075	0.47 ± 0.91

Sample	Within-Laboratory Variability (Max % RSD)	Between-Laboratory Variability (% RSD)
Kelp A	19 %	> 100 %
Kelp B	22 %	29 %
SRM 3232	18 %	> 100 %

Three of the four laboratories provided method details. Solvent extraction was reported as the sample preparation technique, and LC-MS/MS, liquid chromatography with absorbance or photodiode array detection (LC-Abs, -PDA), and liquid chromatography with fluorescence detection (LC-FLD) were all reported as analytical methods for determination of phylloquinone. The consensus means were close to the target values for Kelp B and SRM 3232, **Fig. 4-3** and **Fig. 4-4**; however, the large between-laboratory variability due to two sets of data points being low and two sets being high, in comparison to the target value, indicates a need for improved measurement capabilities.

Naturally occurring fat-soluble vitamins need to be released from the matrix and isolated using a hydrophobic solvent. Analyte degradation and hydrolysis during sample preparation, due to exposure to light or strong alkaline conditions, are major concerns and can lead to low reported values. All of the reported analytical methods techniques use LC, which can remove interfering matrix compounds and also potentially physically separate the *cis*- and *trans*- forms of phylloquinone. Well characterized, quality reference standards are essential for accurate determinations and measurements. Standards should be selected to match the forms being measured in the sample.

Additional tables and figures for vitamin K_1 in kelp are located in **Appendix C**.

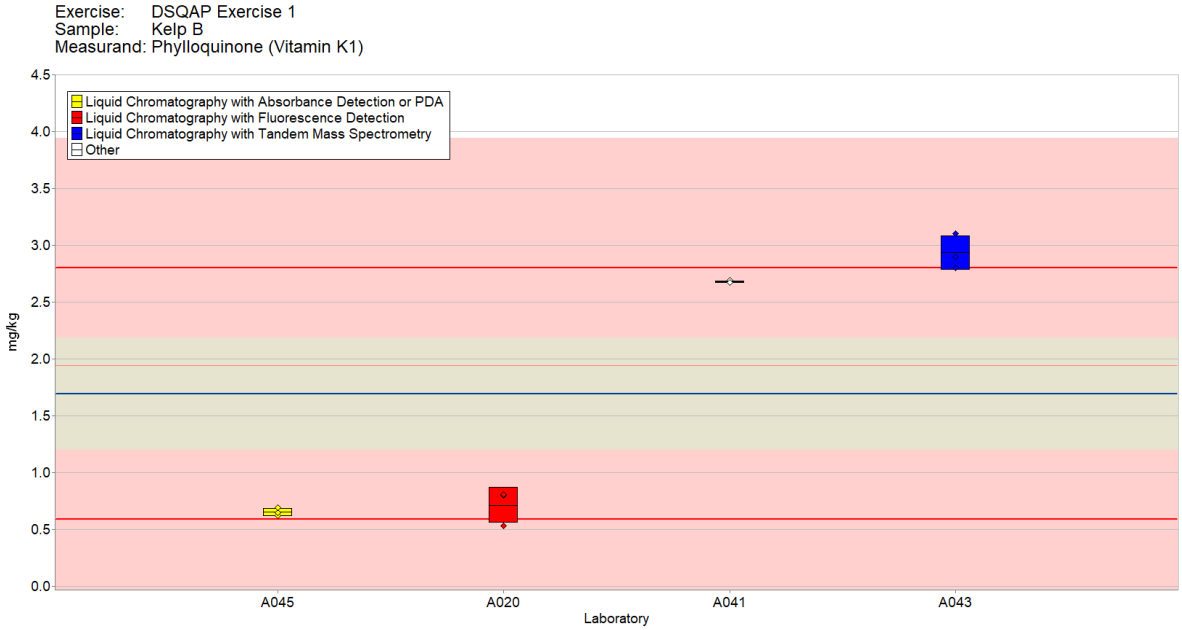

Fig. 4-3. Vitamin K_1 (phylloquinone) in Kelp B (data summary view – analytical method).

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 (beige here due to overlap with the red NIST target range) represents the 95 % confidence interval for the consensus mean. The solid red lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$.

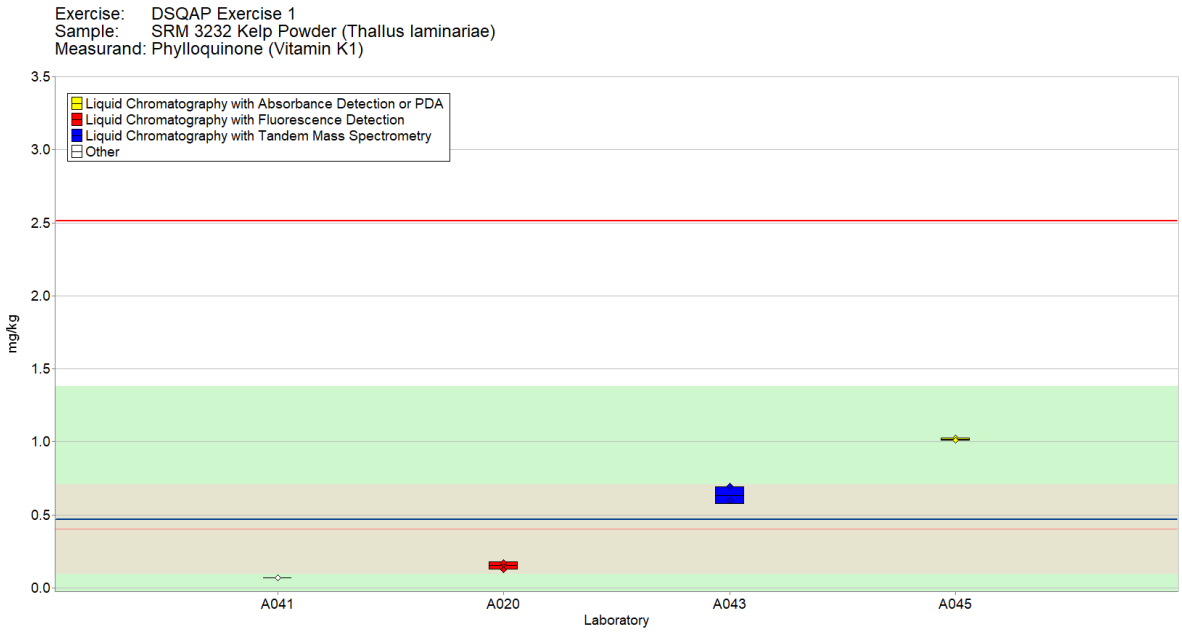

Fig. 4-4. Vitamin K_1 (phylloquinone) in SRM 3232 Kelp Powder (*Thallus laminariae*) (data summary view – analytical method).

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 bound of the consensus range of tolerance, calculated as the value above the consensus mean that results in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$, with the lower bound set to zero. The red shaded region (beige here due to overlap with the green consensus confidence interval) represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$.

5. Botanicals I (Phenolic Content)

5.1. Executive Summary

Polyphenol-rich diets have been correlated with many health benefits, and accurate determination of these compounds in foods or supplements is important to facilitate standardization for clinical investigations of these health effects. This study focused on measurement of gallic acid and total phenolic content as gallic acid equivalents (GAE) in green tea and kelp materials. Most laboratories were able to accurately measure gallic acid in the samples, and the within-laboratory and between-laboratory variabilities for GAE determinations were consistent with industry expectations [22].

5.2. Background

Polyphenol-rich diets have been correlated with many health benefits. Polyphenols are a class of bioactive compounds found in kelp, which may contribute bioactive profiles. Although many approaches exist for determination of total phenolic content, this study was focused on the measurement of a specific polyphenol, gallic acid, as well as total polyphenols as GAE in green tea and kelp materials. Accurate determination of these compounds in foods or supplements is important to ensure product quality and to facilitate standardization for clinical investigations of health effects. In this study, participants were asked to use either their in-house analytical methods or AOAC First Action *Official Method* 2017.13 [23] for the measurement of total phenolic content reported in the mass percent (% w/w) of gallic acid and/or GAE in powdered green tea and kelp materials.

5.3. Study Information

Participants were provided samples of SRM 3254 Green Tea *(Camellia sinensis)* Leaves (three 3 g packets), SRM 3255 Green Tea *(Camellia sinensis)* Extract (three 1 g packets), and Kelp A (three 10 g packets, *Saccharina latissima f. angustissima*, origin ME, USA). Participants were asked to store the materials at controlled room temperature, 20 °C to 25 °C, and to prepare one sample and report one value from each packet provided. Before use, participants were instructed to mix the contents of each packet thoroughly, allow contents to settle for one minute prior to opening to minimize the loss of fine particles, and to use a sample size at least 0.1 g to determine the mass percent (% w/w) of gallic acid and GAE. The approximate analyte levels were not reported to participants prior to the study.

5.4. Study Results and Technical Recommendations

5.4.1. Gallic Acid

Of the 23 laboratories requesting samples for gallic acid, 7 to 8 laboratories reported results for each sample (30 % to 35 % participation). As seen in **Table 5-1**, the within-laboratory variabilities were between 3 % and 7 % for the green tea materials (SRMs 3254 and 3255), and 34 % for Kelp A. The between-laboratory variabilities indicate challenges across the community for these measurements.

Table 5-1. Target values, consensus values, and variabilities for gallic acid in green tea and kelp.

Gallic Acid			
	SRM 3254	SRM 3255	Kelp A
Target Mass Percent ± U_{95} (%)	0.106 ± 0.058	0.313 ± 0.083	-
COA Mass Fraction ± U_{95} (mg/g)	1.12 ± 0.61	3.23 ± 0.86	-
Consensus Mean ± SD (%)	0.081 ± 0.048	0.334 ± 0.062	1.27 ± 3.88
Within-Laboratory Variability (Median % RSD)	7 %	3 %	34 %
Between-Laboratory Variability (% RSD)	59 %	19 %	> 100 %

The target values for gallic acid in SRM 3254 and SRM 3255 are shown in **Table 5-1**, in both % w/w and mg/g. The values for gallic acid on the COAs are based on NIST measurements using LC-Abs and LC-MS, in mg/g on a dry mass basis. The % w/w values were calculated through moisture correction (0.9481 g dry mass/g as-received mass) and unit conversion. Target values for Kelp A were not available at the time of this report.

Most laboratories that returned results were able to successfully measure gallic acid in the green tea samples ($|Z_{\mathrm{NIST}}| \leq 2$), as seen in **Fig. 5-1** and **5-2**. Some laboratories reported values with high biases in more than one more sample (**Fig. 5-3**) and should review their calibration, calculations, and units reported to identify any systematic errors. Improved separation techniques and more specific detectors could also be used to help reduce interferences.

The between-laboratory variability was lowest for the green tea extract. The green tea extract was expected to pose less of an analytical challenge for laboratories, as the gallic acid level in the green tea extract was higher than in the green tea leaves, and the gallic acid content in the green tea extract was already isolated from the plant material and could be more readily available through dilution or solvent extraction.

The level of gallic acid in the kelp material was below the LOQ of most laboratories. An overall phenolic content method may be more appropriate for characterization of this particular species of kelp.

Additional tables and figures for gallic acid in green tea and kelp are located in **Appendix D**.

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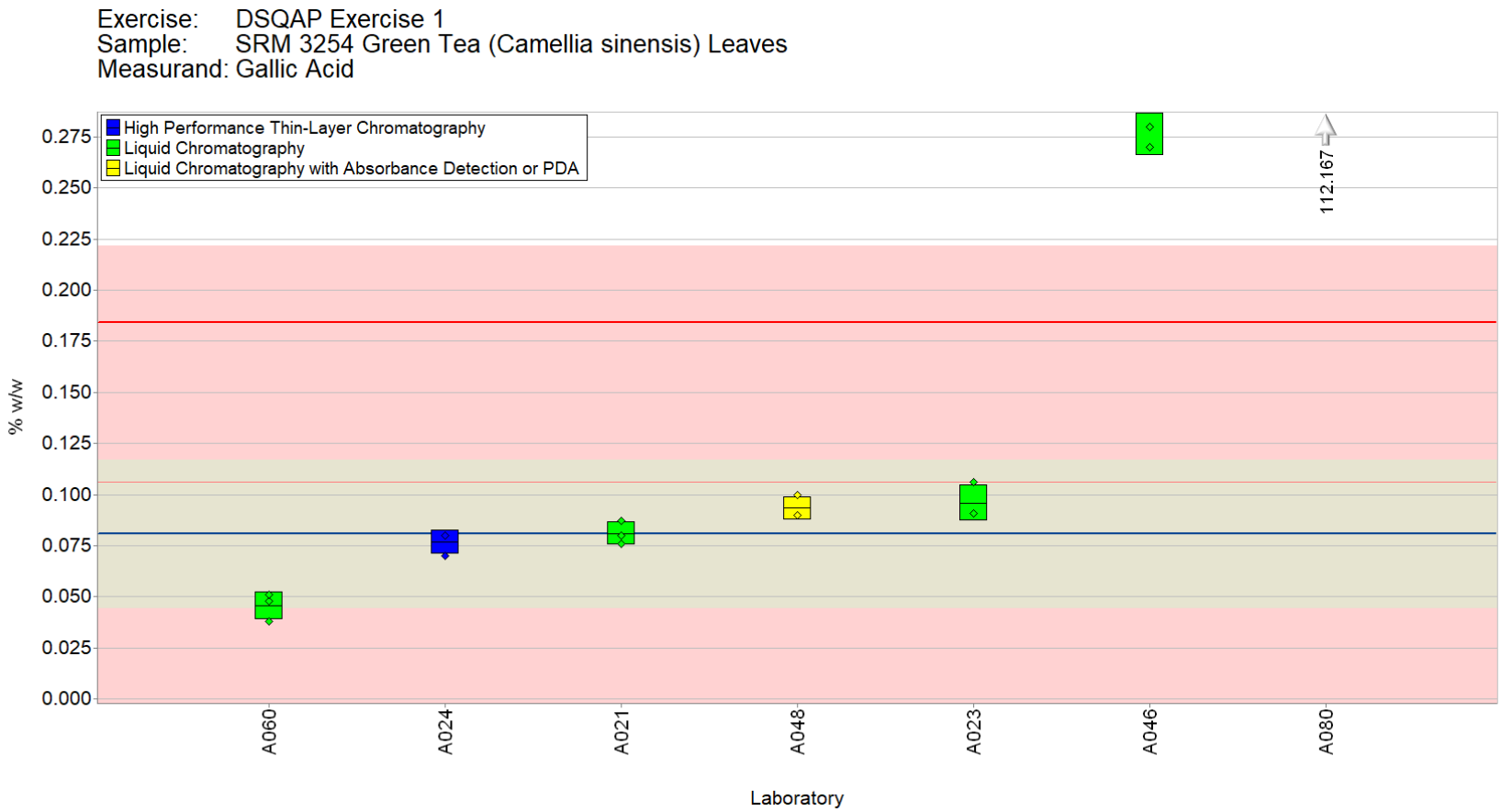

Fig. 5-1. Gallic acid in SRM 3254 Green Tea *(Camellia sinensis)* Leaves (data summary view – analytical method).

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 (beige here due to overlap with the red NIST target range) represents the 95 % confidence interval for the consensus mean. The red solid line represents the upper bound of the consensus range of tolerance, calculated as the value above the consensus mean that results in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$, with the lower bound set to zero. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$.

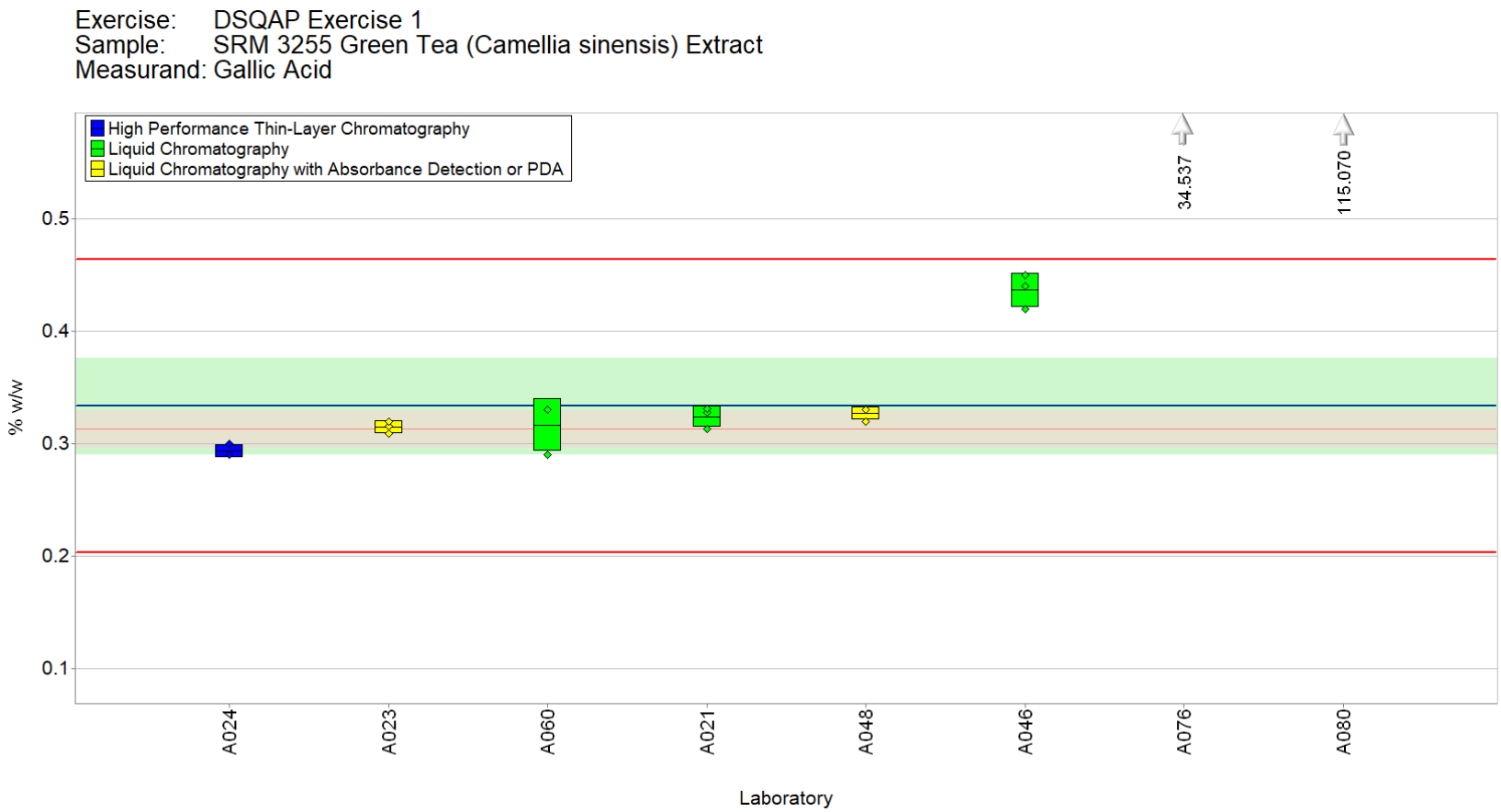

Fig. 5-2. Gallic acid in SRM 3255 Green Tea *(Camellia sinensis)* Extract (data summary view – analytical method).

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 red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region (beige here due to overlap with the green consensus confidence interval) represents the NIST range of tolerance, which encompasses the target value bounded by twice its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

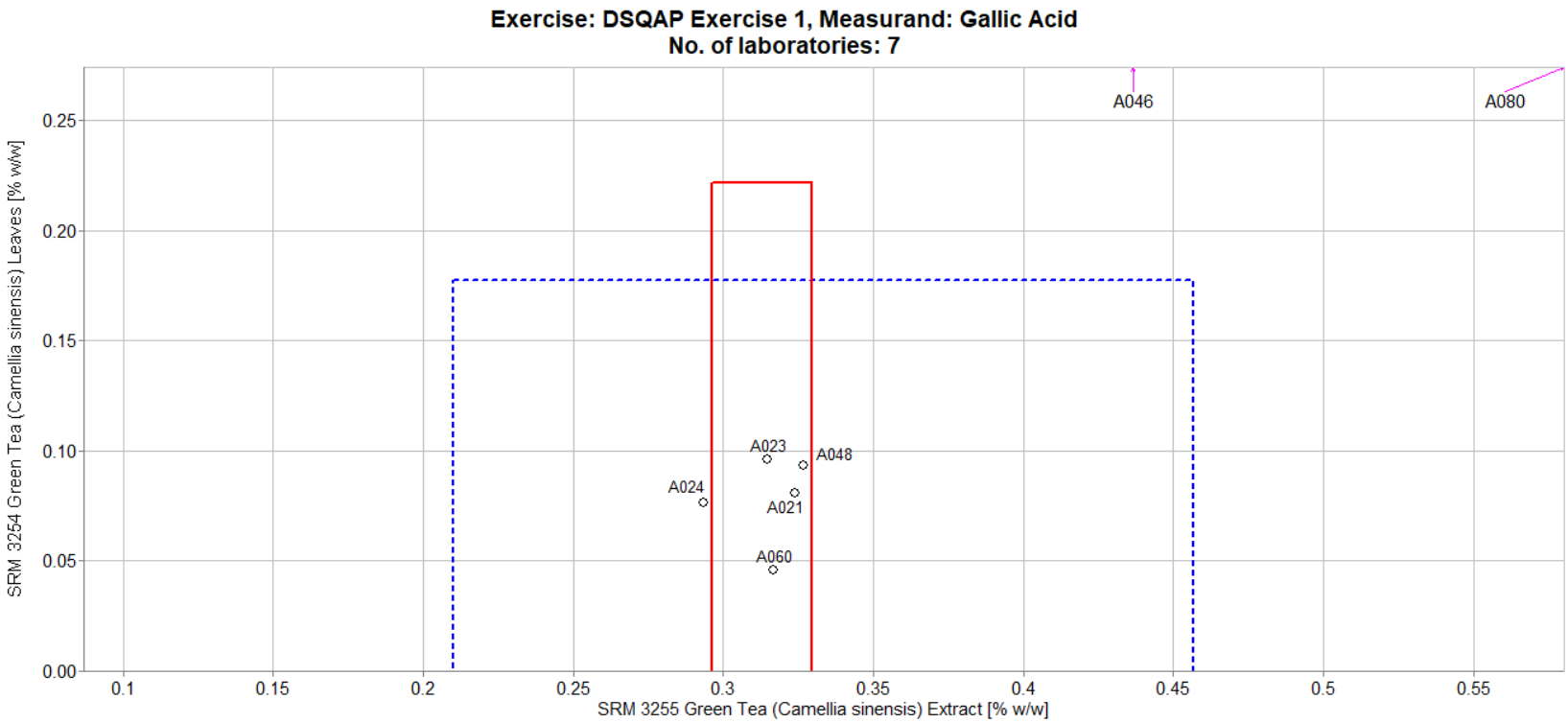

Fig. 5-3. Laboratory means for gallic acid in SRM 3255 Green Tea *(Camellia sinensis)* Extract and SRM 3254 Green Tea *(Camellia sinensis)* Leaves (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3255, is compared to the individual laboratory mean for a second sample, SRM 3254. The solid red box represents the NIST range of tolerance for the two samples, SRM 3255 (x-axis) and SRM 3254 (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3255 (x-axis) and SRM 3254 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

5.4.2. Gallic Acid Equivalents

Of the 26 laboratories requesting samples for GAE, 10 to 11 laboratories reported results for each sample (38 % to 41 % participation). As seen in **Table 5-2**, the within-laboratory variabilities were at or below 5 % for all samples, which is acceptable when compared to the AOAC SMPR [22] for repeatability (within-laboratory variability) at or below 7 %. When compared to the AOAC SMPR criteria for reproducibility (between-laboratory variability) at or below 10 %, the between-laboratory variabilities indicate some analytical challenges across the community for these measurements, especially in the green tea leaves (30 % RSD) and kelp (63 % RSD) [22]. Raw botanical materials are generally more analytically challenging than extracts due to matrix complexity which is evident from the tighter reproducibility (11 %) for the green tea extract measurements. Target values for gallic acid in Kelp A and GAE in all materials were not available at the time of this report.

Table 5-2. Consensus values and summary of variabilities for gallic acid equivalents in green tea and kelp.

Gallic Acid Equivalents

	SRM 3254	SRM 3255	Kelp A
Consensus Mean ± SD (% w/w)	15.06 ± 4.57	80.94 ± 9.25	0.26 ± 0.17
Between-Laboratory Variability (% RSD)	30 %	11 %	63 %
Within-Laboratory Variability (Median % RSD)	2 %	2 %	5 %

Most laboratories reported using a Folin-C assay or AOAC 2017.13 (a Folin-C assay-based method) for the analytical technique which is expected for GAE determination. One laboratory reported using spectroscopy, which is based on a similar detection concept to the Folin-C assay. The distribution of results and methods reported are shown in **Fig. 5-4**, **5-5**, and **5-6** for SRM 3254, SRM 3255, and Kelp A, respectively. For overall phenolic content, other methods focusing on total anthocyanins, total proanthocyanidins, free radical scavenging, etc. can also be used.

The methods used by the participants of this study respond to any reducing capacity in the sample matrix, not just the content of phenols [24]. With an understanding of the quantity being reported and its fitness-for-purpose, these methods can be a suitable way to assess the antioxidant strength of botanical materials, especially when the specific compounds are unknown or can change with species and growing season.

Overall, ensuring that high quality reagents and standards are used is essential for proper calibration and accurate measurements. Sample size and homogeneity of sampling is also important for reducing measurement variabilities. Additional tables and figures for GAE in green tea and kelp are located in **Appendix D**.

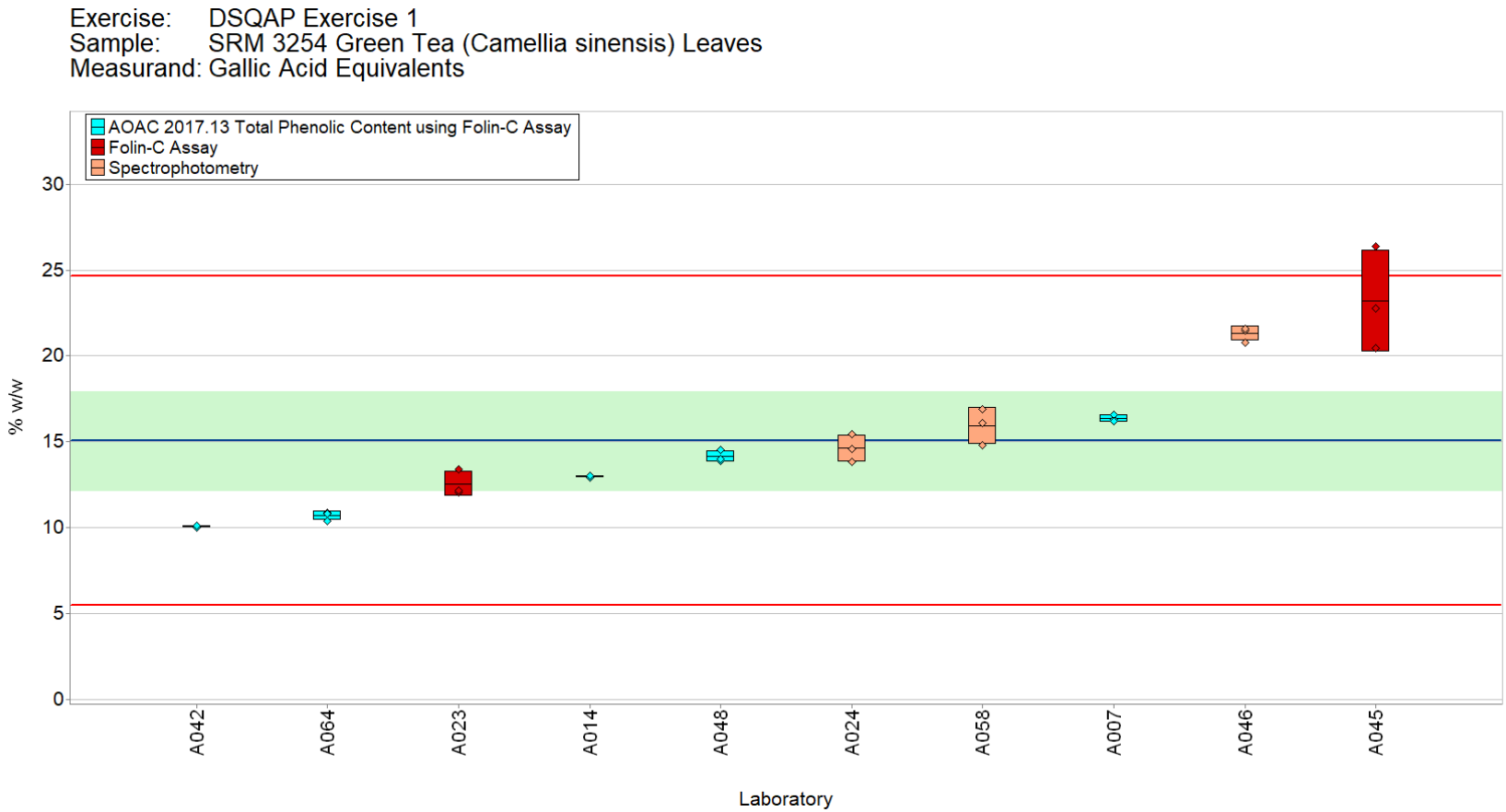

Fig. 5-4. Gallic acid equivalents in SRM 3254 Green Tea *(Camellia sinensis)* Leaves (data summary view – analytical method).

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 red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

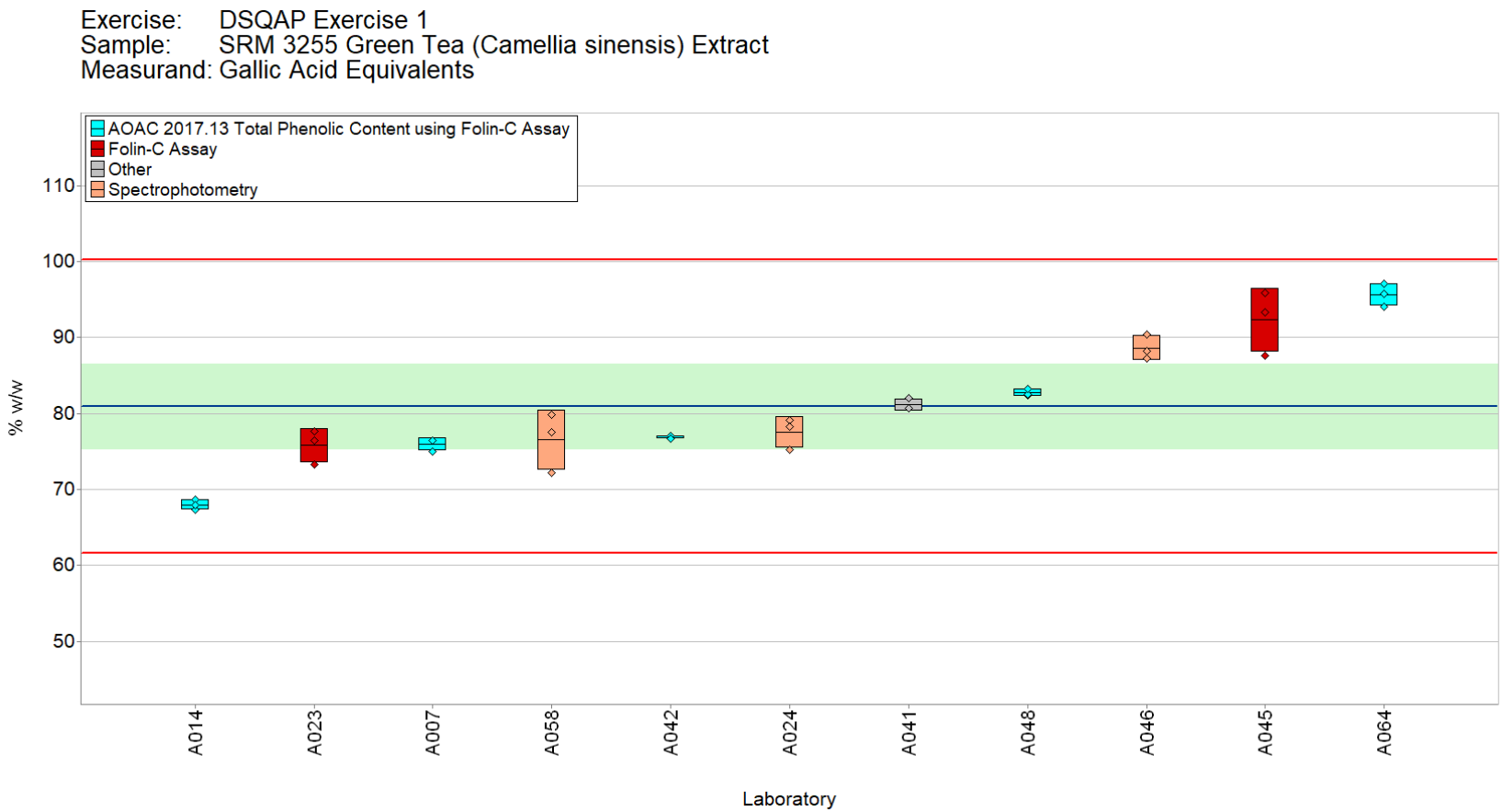

Fig. 5-5. Gallic acid equivalents in SRM 3255 Green Tea *(Camellia sinensis)* Extract (data summary view – analytical method).

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 red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

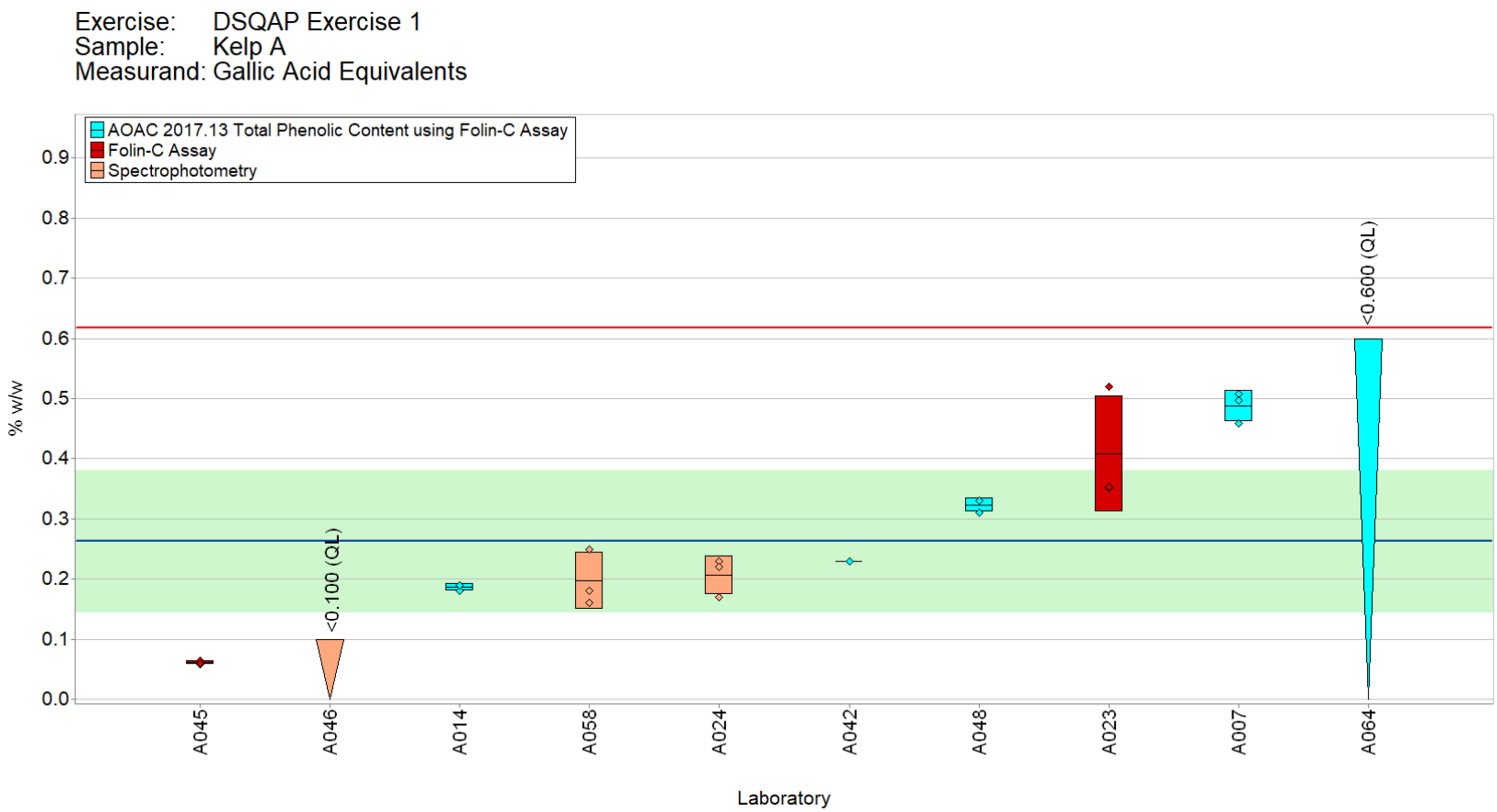

Fig. 5-6. Gallic acid equivalents in Kelp A (data summary view – analytical method).

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 red solid line represents the upper bound of the consensus range of tolerance, calculated as the value above the consensus mean that results in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$, with the lower bound set to zero.

6. Proximates

6.1. Executive Summary

Accurate measurement of proximates and calories in foods is necessary to support reliable food labeling and inform population studies that impact dietary guidelines. Overall, the participating laboratories were able to successfully measure calories, protein, ash, and solids in the kelp sample, but results were more variable for fat, carbohydrates, fiber, and starch. Because determinations for one proximate may be used in calculation of another proximate, participants should check all results closely to avoid calculation or other errors and to be sure that results are reported in the requested units.

6.2. Background

Proximates make up the macronutrient profile of foods and their measurement is critical for crop designation and health, as well as understanding the impact of macronutrient consumption on human health. Variations between species, growing seasons, or growth stages may affect the nutritional makeup of kelp. Proximates are also important from an analytical perspective, as the relative fat/protein/carbohydrate ratios of a food are critical factors for predicting measurement challenges and selecting appropriate control materials. Accurate measurement of proximates and calories in foods is necessary to support reliable food labeling and to design and interpret population studies that impact dietary guidelines and for kelp, to understand differences in seaweed species and growing environments and determine the best product for different end-uses (e.g., human and animal foods, pharmaceuticals, biofuels). This study offered enrollment for various proximates and participants were asked to use their in-house analytical methods to determine ash, carbohydrates, fat, protein, solids, starch, and total dietary fiber (TDF) as mass fraction (percent), and energy as calories (kcal/100 g) in a powdered kelp material.

6.3. Study Information

Participants were provided with packets of Kelp A, which was a dried powdered kelp (*Saccharina latissimi f. angustissima*, origin ME, USA). The number of 50 g packets provided was determined by the specific measurands for which each participating laboratory registered. Participants received three total packets to analyze ash, fat, protein, carbohydrates, and calories. Participants received additional samples if enrolled in solids, starch, and total dietary fiber (1, 1, and 3 packets respectively). Participants were asked to store the material at controlled room temperature, 20 °C to 25 °C, in the original unopened packets and to prepare one sample and report one value from each packet provided. Before use, participants were instructed to thoroughly mix the contents of the packet and to allow contents to settle for one minute prior to opening to minimize the loss of fine particles prior to removal of a test portion for analysis. The approximate analyte levels were not reported to participants prior to the study and not available at the time of this report.

6.4. Study Results and Technical Recommendations

6.4.1. Ash

Ash content refers to the mineral and inorganic matter remaining after a product has been heated to a very high temperature to remove any moisture, organics, and volatiles. The determination of ash is important for understanding the full nutritional value and quality of a food, as well as the biomass energy for other potential industrial applications.

For the determination of ash, 22 of the 35 laboratories that enrolled reported results (63 % participation). Overall, participants were able to measure ash in powdered kelp with good within-laboratory precision (average 0.7 %, all at or below 4 %) and between-laboratory variability (below 2 %). Three laboratories reported results below the limit of tolerance of the consensus mean, and these laboratories should assess their method procedure, check for calculation any errors, and ensure correct reporting units.

All of the analytical methods reported were similar, using oven drying at temperatures between 500 °C and 600 °C. Several laboratories indicated use of AOAC methods including AOAC 923.03 Ash of Flour (550 °C) [25], AOAC 942.05 Ash in Animal Feed (600 °C) [26], and AOAC 945.46 Ash of Milk (550 °C) [27]. Comparing these methods, the main difference is the oven temperature, though recommended modifications to AOAC 942.05 have been published, including a recommendation to decrease the temperature to 550 °C [28]. Additional modifications, which may be relevant for laboratories reporting outlying results for this study, include running two 3-hour cycles to ensure complete carbon release by adding fresh air supply between ignitions as well as starting the sample in a cold furnace and ramping up to final temperature to alleviate potential issues from rapid sample ignition. Kelp samples would likely be a similar matrix to flour, and this would be a good method to follow. Three laboratories reported “other” and two laboratories did not report a method. The results are summarized in **Fig. 6-1**, with the different colors indicating methods with similar oven temperatures. Methods using temperatures below 550 °C could have a high bias, although this cannot be confirmed since only two laboratories reported these methods.

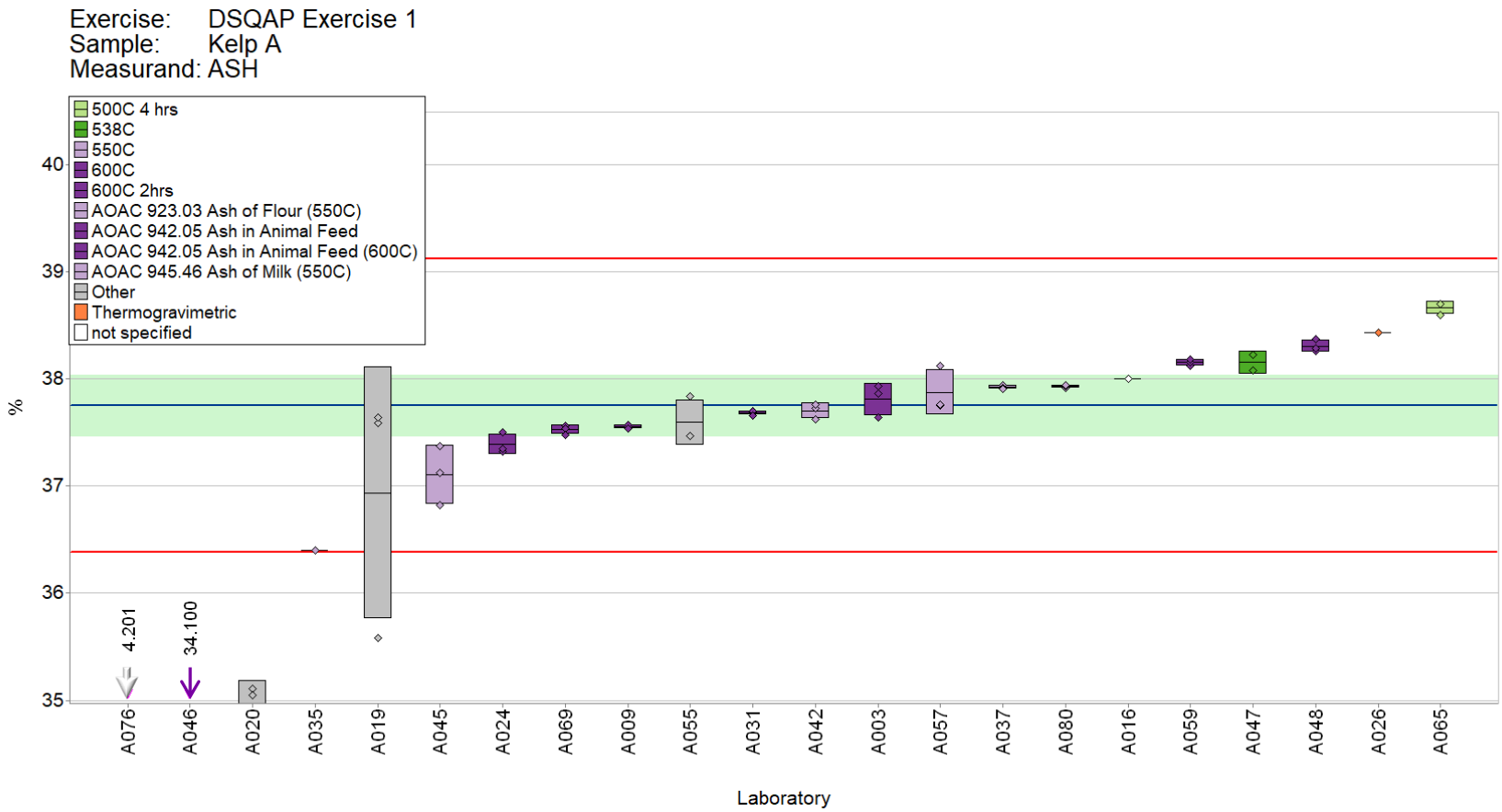

Fig. 6-1. Ash in Kelp A (data summary view – analytical method).

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 red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

6.4.2. Fat

Fat, in moderation, is an important part of a healthy diet. Fat helps the body absorb vitamins A, D, and E and is also needed for cell membrane integrity and hormone production. Seaweeds are typically low in fat content (between 0.61 % and 4.15 % dry weight). However, some seaweed species may have higher content, making them a potential source of healthy, essential fatty acids [29].

For the determination of fat, 19 of the 33 laboratories that enrolled reported results (58 % participation). Most laboratories showed excellent precision for their measurements (at or below 5 %), and five laboratories had good precision (at or below 10 %), which is on par with the AOAC Peer-Verified Method for determining total fat in foods and feeds. Two laboratories had variabilities at or below 17 %. Two laboratories reported results above the limit of tolerance of the consensus mean, and should assess their method procedure, check for calculation any errors, and ensure correct reporting units.

The fat content in this kelp sample was low, and potentially challenging to measure. Laboratories that found the fat content to be below their LOQ should have increased their sample size to obtain a measurable amount of fat in the kelp samples. In **Fig. 6-2**, reported analytical methods are displayed and similar methods are grouped using similar colors. For blue hued methods, the amount of fat is determined by weighing the fat following solvent extraction. The different blue colors indicate solvent (i.e., ether, chloroform/methanol, hexane). For red hued methods, a digestion or hydrolysis of the sample and fat is conducted followed by a direct measurement of the fats either through absorbance or other signal measurement. While no method bias was observed, more between-method variability was noted for solvent extraction-based methods. Laboratories should ensure their method appropriateness with the use of similar matrix-matched reference or QC material, and in this case a low fat, plant or algae-based material.

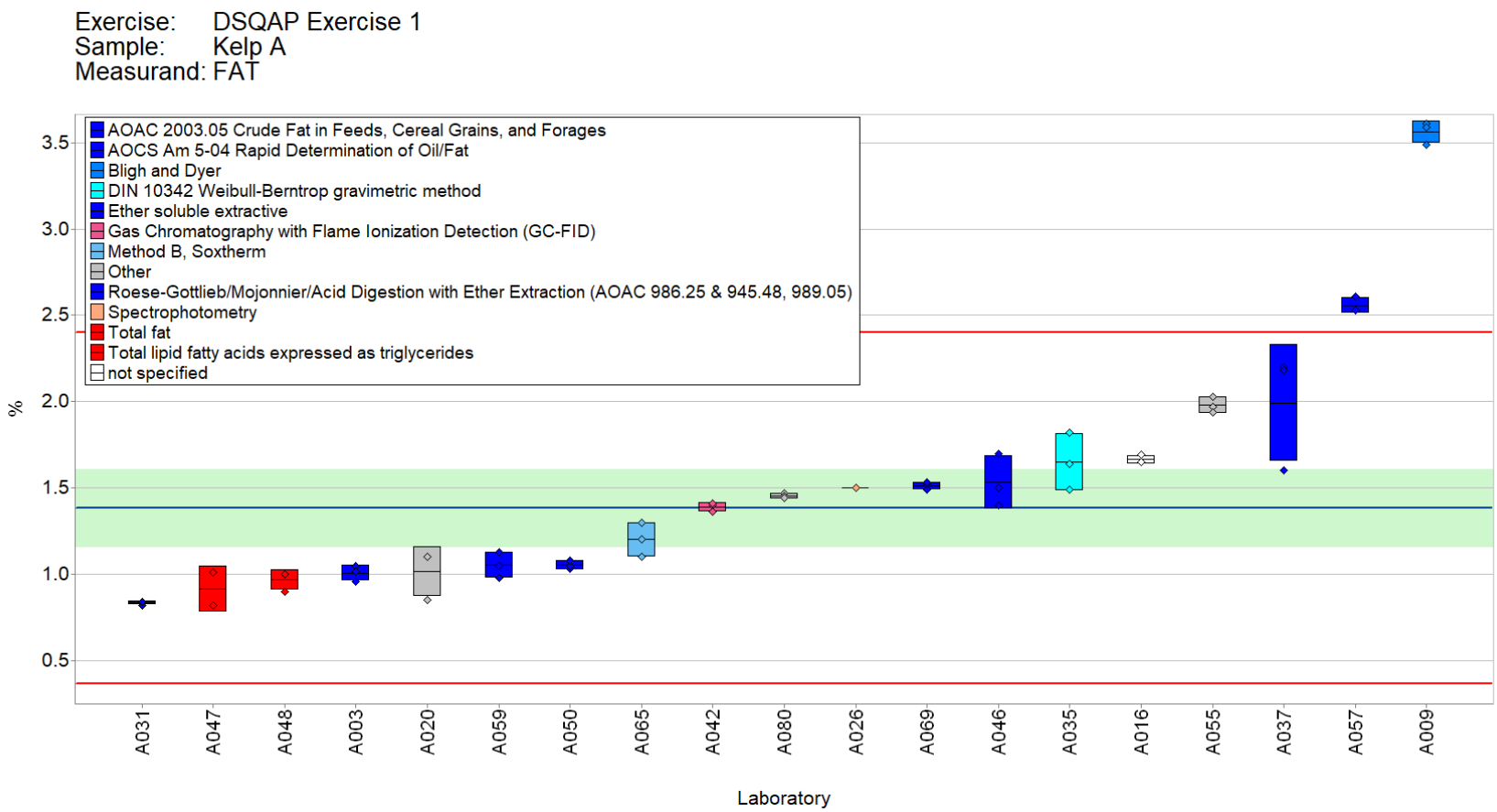

Fig. 6-2. Fat in Kelp A (data summary view – analytical method).

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 the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$, with the lower limit set at zero.

6.4.3. Protein

Protein analysis can be complex, as proteins can be classified by composition, structure, or solubility. Determination of the type and quality of protein is important, as protein has many roles in the body. Many methods for determining protein levels, as described below, require quantifying total nitrogen content to understand nutritional and other kelp end-uses, such as fertilizers.

The most common methods for quantifying protein in food are Kjeldahl, Dumas, absorbance, and refractive index [30]. The Kjeldahl method uses acid digestion and titration to determine total nitrogen content in a sample. A nitrogen-to-protein (NP) conversion factor is then applied to the measured nitrogen to estimate the protein content. The Dumas method also measures nitrogen content following combustion but in a faster, automated technique. Standard conversion factors may not be suitable for all materials and protein types, and so care should be taken when choosing the correct factor. The major recommendation for protein determination in kelp is to determine the best seaweed nitrogen to protein (SNP) conversion factors for different species of kelp. The general assumption is that the total nitrogen content in plant and animal proteins is roughly 16 % which leads to a conversion factor of 6.25 (100 ÷ 16 = 6.25) [31]. Several conversion factors have been published, with 6.25 as the standard for food stuffs which may overestimate protein content in seaweeds. Species specific SNP conversion factors range from 3.53 to 5.13 [31, 32]. Direct amino acid determination (e.g., LC) can be very accurate but time consuming as the initial investment in equipment can be costly.

For the determination of protein, 20 of the 33 laboratories that enrolled reported results (61 % participation). Within-laboratory variabilities were all less than 5 %, and the between-laboratory variability was 5 %. One laboratory had a variability of 40 %. Five laboratories reported results below the limit of tolerance of the consensus mean. Laboratory A057 should double check that triplicate values were reported correctly.

Most laboratories reported using a combustion method or nitrogen by Kjedahl method. Several laboratories indicated use of AOAC methods including AOAC 2001.11 Protein (crude) [33, 34], AOAC 990.03 Protein (Crude) in Animal Feed [35], AOAC 992.15 Crude protein in meat and meat products [36], and AOAC Method 968.06 Protein (Crude) in animal feed [37]. Two laboratories reported “other” and one laboratory did not report a method. In **Fig. 6-3**, the different reported methods are assigned colors based on similarities (e.g., all methods using combustion are colored red). No clear method bias was observed, although any potential bias can be likely contributed to the choice of conversion factor.

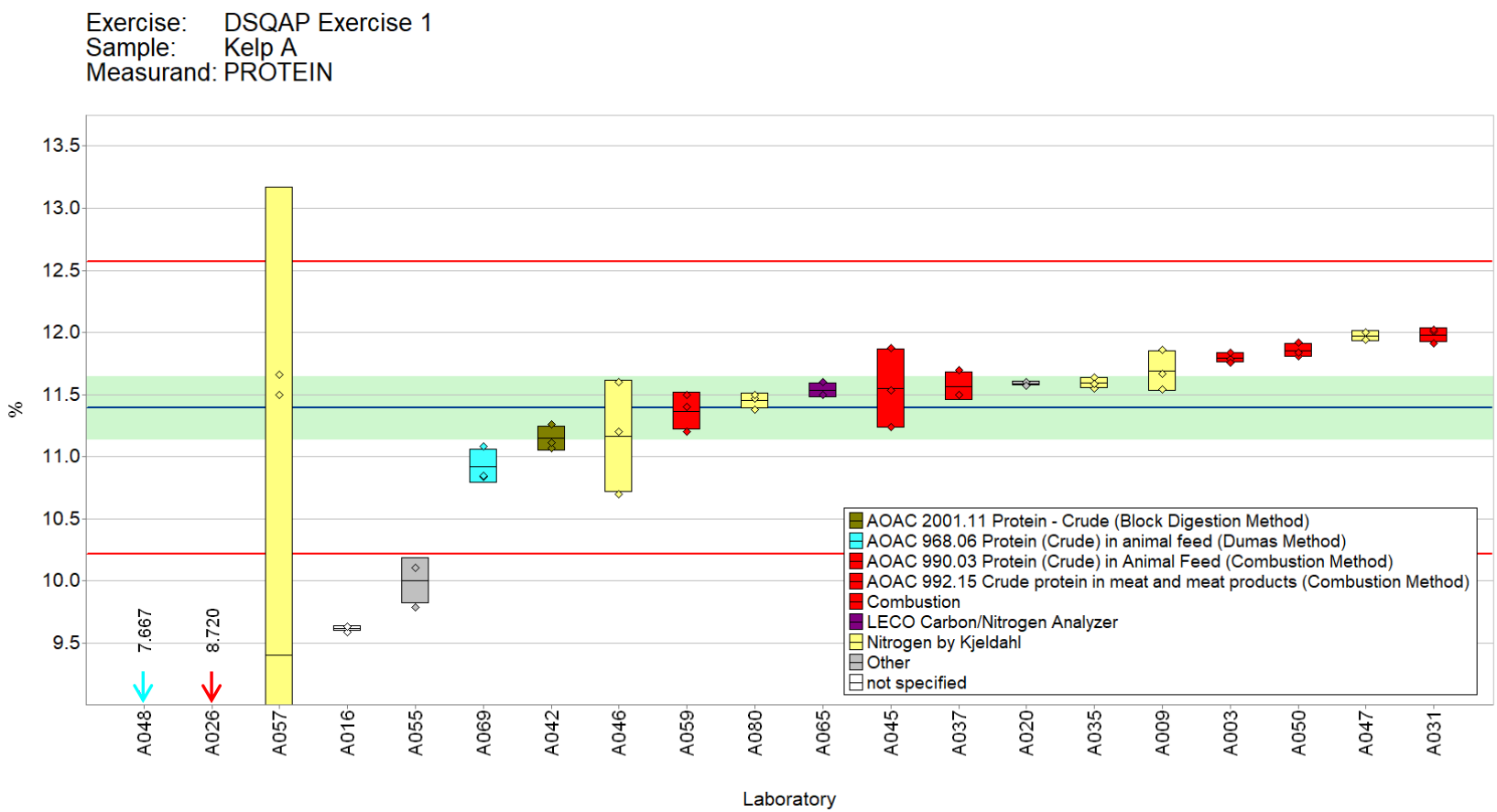

Fig. 6-3. Protein in Kelp A (data summary view – analytical method).

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 red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

6.4.4. Solids

The measurement of solids, and the closely related moisture content, is important for food materials. Regulations establish the amount of water that can, or must, be present in certain types of food, which is directly related to costs to consumers, microbial growth, and food quality. An understanding of solids and moisture content can also be used to predict behavior of materials during processing. While solids and moisture content determinations are important and common, achieving accurate and precise data depend on the choice of analysis method with respect to the expected moisture content, other matrix constituents present, accuracy and precision required, and intended purpose.

For the determination of solids, 13 of the 29 laboratories that enrolled reported results (45 % participation). Within-laboratory variabilities were all less than 0.1 %, and the between-laboratory variability was 1 %. Two laboratories reported results above the limit of tolerance of the consensus mean, and these laboratories should assess their method procedure, check for calculation any errors, and ensure correct reporting units.

The results here can be used to assess measurement capabilities for dried, finished product materials, similar to the dried, powdered materials provided in the study, but not necessarily on raw seaweed samples. The reported analytical methods for the determination of solids were varied, as seen in **Fig. 6-4**. Several laboratories indicated use of AOAC methods including AOAC 925.09 Solids (Total) and Moisture in Flour [38], AOAC 934.01 Dry Matter for Feeds [39], and AOAC 990.20 Solids in Milk [40]. Two laboratories reported “other”. No method bias was observed, though laboratories above the tolerance limit should determine if all the moisture and volatile compounds were removed for the solids determination.

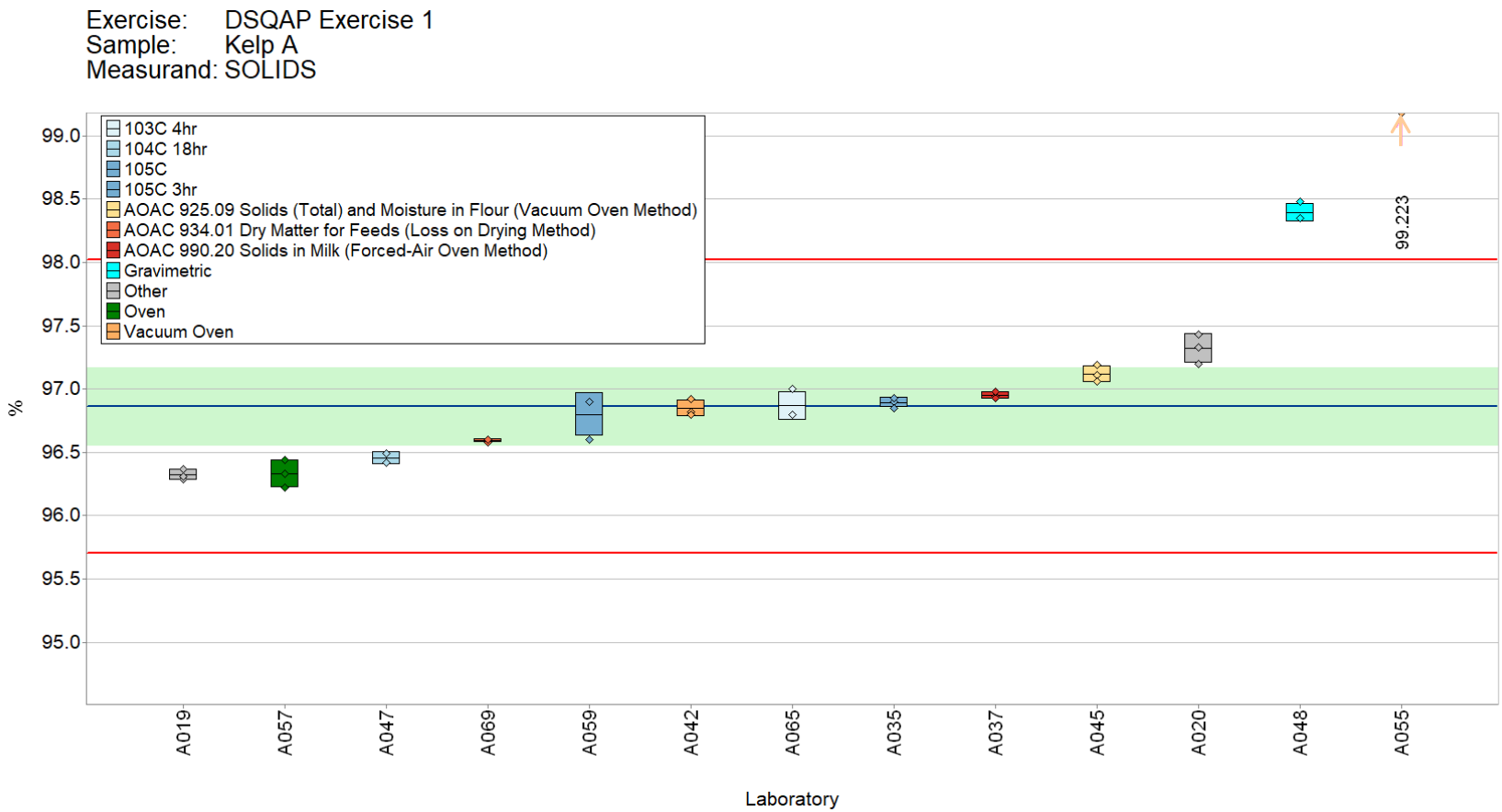

Fig. 6-4. Solids in Kelp A (data summary view – analytical method).

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 red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

6.4.5. Starch

Starch is a carbohydrate produced by most green plants for energy storage. Starch is a component of human diets in many foods (e.g., wheat, potatoes, and rice), converted into sugars and then fermented for the manufacture of alcoholic beverages and biofuel, as well as used as processed sugars or as a thickening agent. Starch is used industrially as an adhesive in the papermaking process and as clothing starch. Studies are underway to assess and develop sustainable starch production from seaweeds [41].

For the determination of starch, only 6 of the 28 laboratories that enrolled reported results (21 % participation). Within-laboratory variabilities were all below 14 %, and the between-laboratory variability was high (above 100 %). Three laboratories reported that the starch content in the samples was below their LOQ.

The starch content of the kelp material was very low, below 1 %. As a result, a larger sample size may have been needed for test methods to arrive at the correct result. Certain kelp species have been reported to contain little to no starch, which is important when considering kelp materials for starch-based end-use applications. The reported analytical methods for the determination of starch were varied. No method was bias observed, as seen in **Fig. 6-5**.

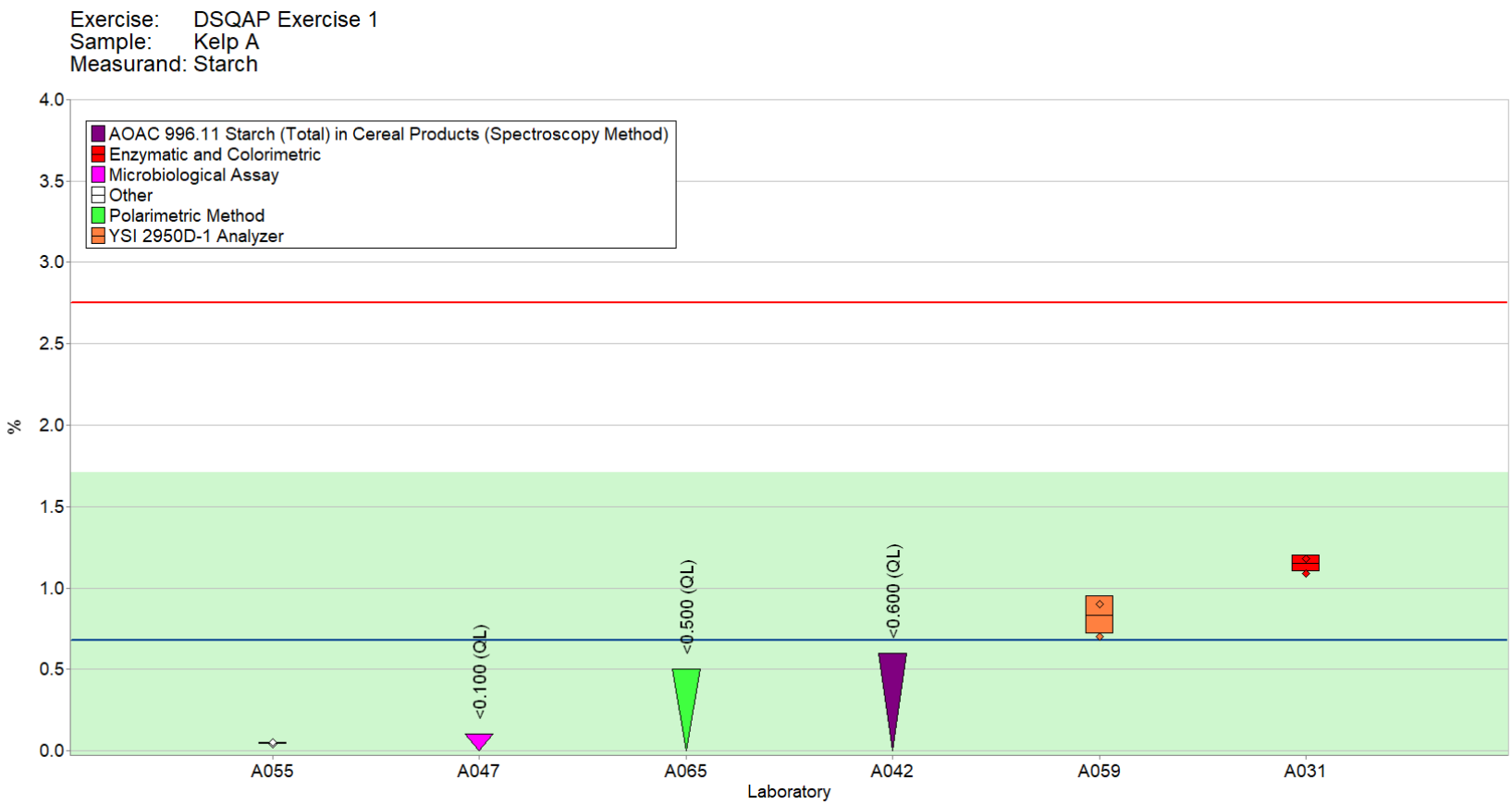

Fig. 6-5. Starch in Kelp A (data summary view – analytical method).

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 red solid line represents the upper bound of the consensus range of tolerance, calculated as the value above the consensus mean that results in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$, with the lower bound set to zero.

6.4.6. Total Dietary Fiber (TDF)

Dietary fiber is a complex group of plant components described analytically as non-starch polysaccharides and lignin from plants. Reported as either soluble, insoluble, or total dietary fiber (TDF) on food labels, fiber is a major component of a balanced diet and both insoluble and soluble forms have important health benefits. Seaweeds have been reported to contain a high proportion of soluble fiber with soluble/insoluble fiber ratios greater than that of many non-marine based vegetables [42].

For the determination of TDF, 9 of the 29 laboratories that enrolled reported results (31 % participation). While most laboratories reported acceptable precision for their measurements (at or below 5 %), the agreement between laboratories was higher at 24 %. All laboratories should assess their method procedure, check for calculation any errors, and ensure correct reporting units.

Laboratories reported using Ankom Fiber, AOAC 2017.16 Total Dietary Fiber in Foods [43, 44], AOAC 985.29/991.43 Total, Soluble, and Insoluble Dietary Fiber in Foods [45, 46], AOAC 991.43/AACC 32.07 Lee Method [46, 47], or crude fiber methods. Two laboratories reported "other" and one laboratory did not report a method. Despite the low reporting numbers, a multimodal distribution was observed across the analytical methods (**Fig. 6-6** and **Fig. 6-7**), indicating a potential for low bias for crude fiber methods compared to other methods. Mode 1 and 2 include crude fiber methods and "other" (on the left side of the plot), and mode 3 (on the right side of the plot) includes the remaining methods. Methods reporting lower values may be measuring different forms of fiber and may not be inclusive all forms. Brown seaweeds similar to the sample used in this study have been reported to contain approximately 17 % soluble fiber and 13 % insoluble fiber. Also, seaweeds and algae contain alginate forms of fiber which are known to be highly soluble. Crude fiber methods do not account for soluble forms and should not be used as a stand-alone method when determining TDF.

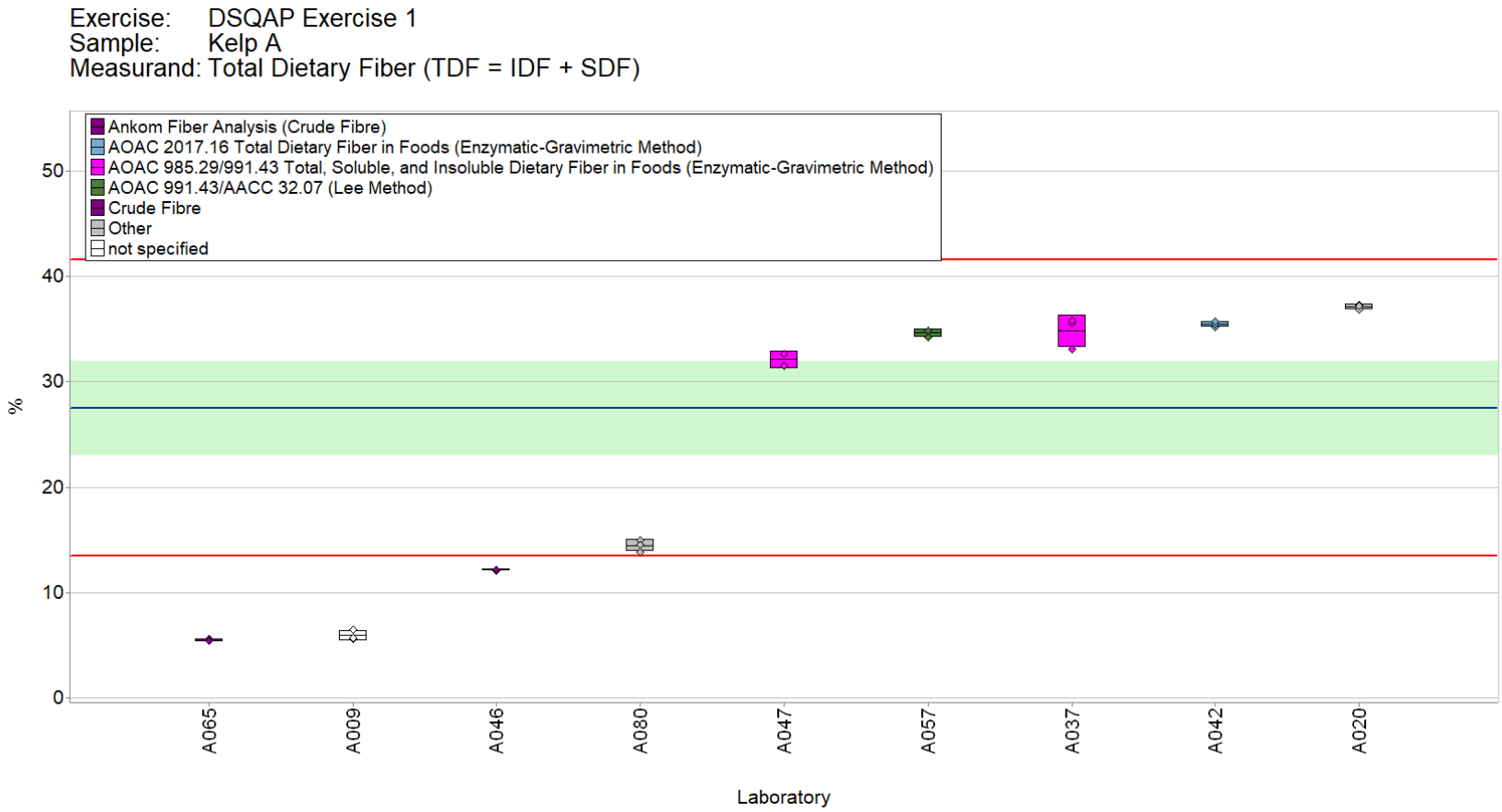

Fig. 6-6. Total dietary fiber in Kelp A (data summary view – analytical method).

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 red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

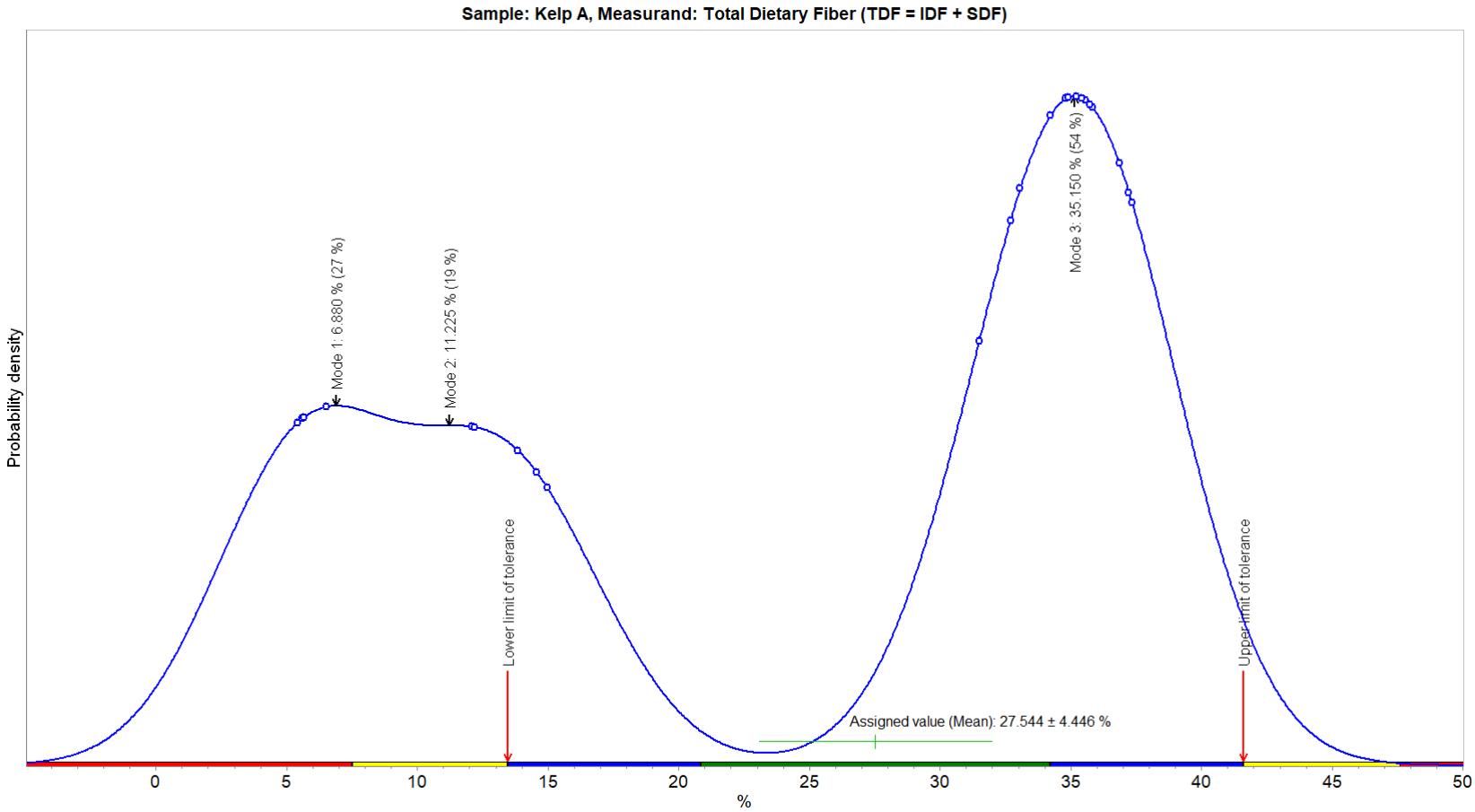

Fig. 6-7. Kernel density estimation for TDF in Kelp A.

In this view, the kernel density of the distribution is estimated as a function of all reported results (solid blue) with the different modes indicated on the line. The consensus mean is indicated by the lower green horizontal bar. Upper and lower limits of tolerance are indicated by red arrows.

6.4.7. Carbohydrates

Carbohydrates are important biomolecules, most often polysaccharides, that are involved in energy storage, immune system functions, blood clotting, and cell development. Seaweeds are promoted as a healthy source of carbohydrates, although the bioavailability of the forms present has not been established [48]. Polysaccharides found in seaweeds are also used in foods as thickeners, gelling agents, and emulsion stabilizers.

For the determination of carbohydrates, 11 of the 29 laboratories that enrolled reported results (38 % participation). While most laboratories reported acceptable precision for their measurements (at or below 5 %), the agreement between laboratories was higher at 27 %. Three laboratories reported results below the limit of tolerance of the consensus mean, and these laboratories should assess their method procedure, check for calculation any errors, and ensure correct reporting units.

The greatest source of error in carbohydrate calculations is the error in contributing values (e.g., fat, protein, solids, and ash). Most laboratories reported using a calculation method (45 %) or a spectrophotometric method (9 %) for the determination of carbohydrates, as seen in **Fig. 6-8**. Three laboratories reported “other” and one laboratory did not report a method. One potential bias noted from the limited data set was the low bias of direct measurement compared to calculation-based measurement of carbohydrates.

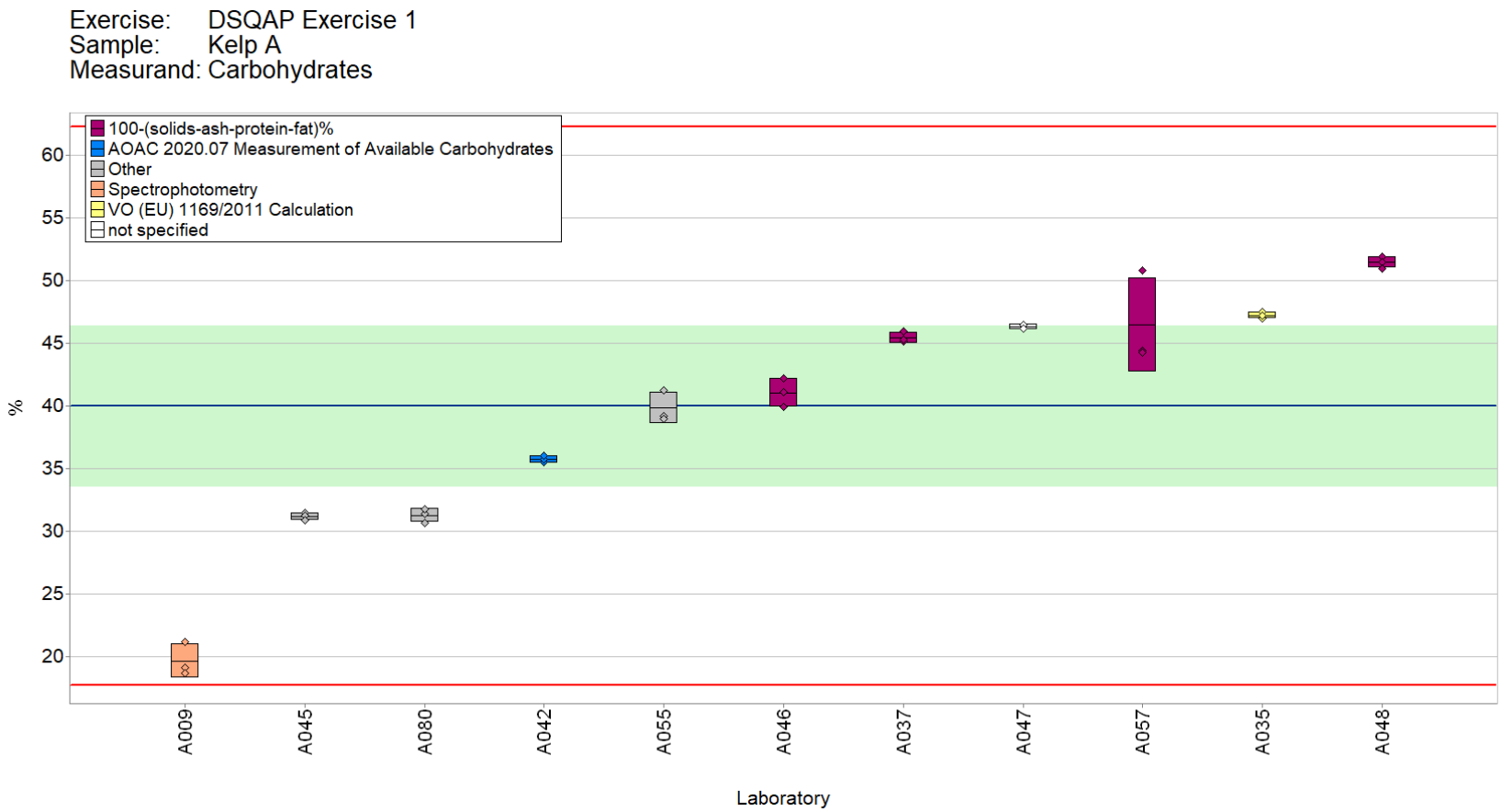

Fig. 6-8. Carbohydrates in Kelp A (data summary view – analytical method).

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 red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

6.4.8. Calories

Calories are the measure of energy in foods that our bodies use in all cellular functions, and caloric intake has significant influences on human health [49]. Accurate determination of calories in food is important to ensure that Nutrition Facts and Supplement Facts labels are correct, and consumers are well informed.

For the determination of calories, only 8 of the 31 laboratories that enrolled reported results (26 % participation). While most laboratories reported acceptable precision for their measurements (average across the participants was 0.5 %), the agreement between laboratories was higher at 10 %.

Most laboratories reported using a calculation method or a calorimetry method for the determination of calories. As seen with to carbohydrate calculation methods, the greatest source of error when calculating calories versus direct determination comes from error in contributing values. One laboratory reported “other” and one laboratory did not report a method. Calorimetry is a direct measurement of energy, whereas calculation methods are based on measurements of fat, protein, solids, and ash. While difficult to asses with the number of values reported, higher variability and potentially a low bias was observed for the calculation methods in comparison to the calorimetry methods (**Fig. 6-9**).

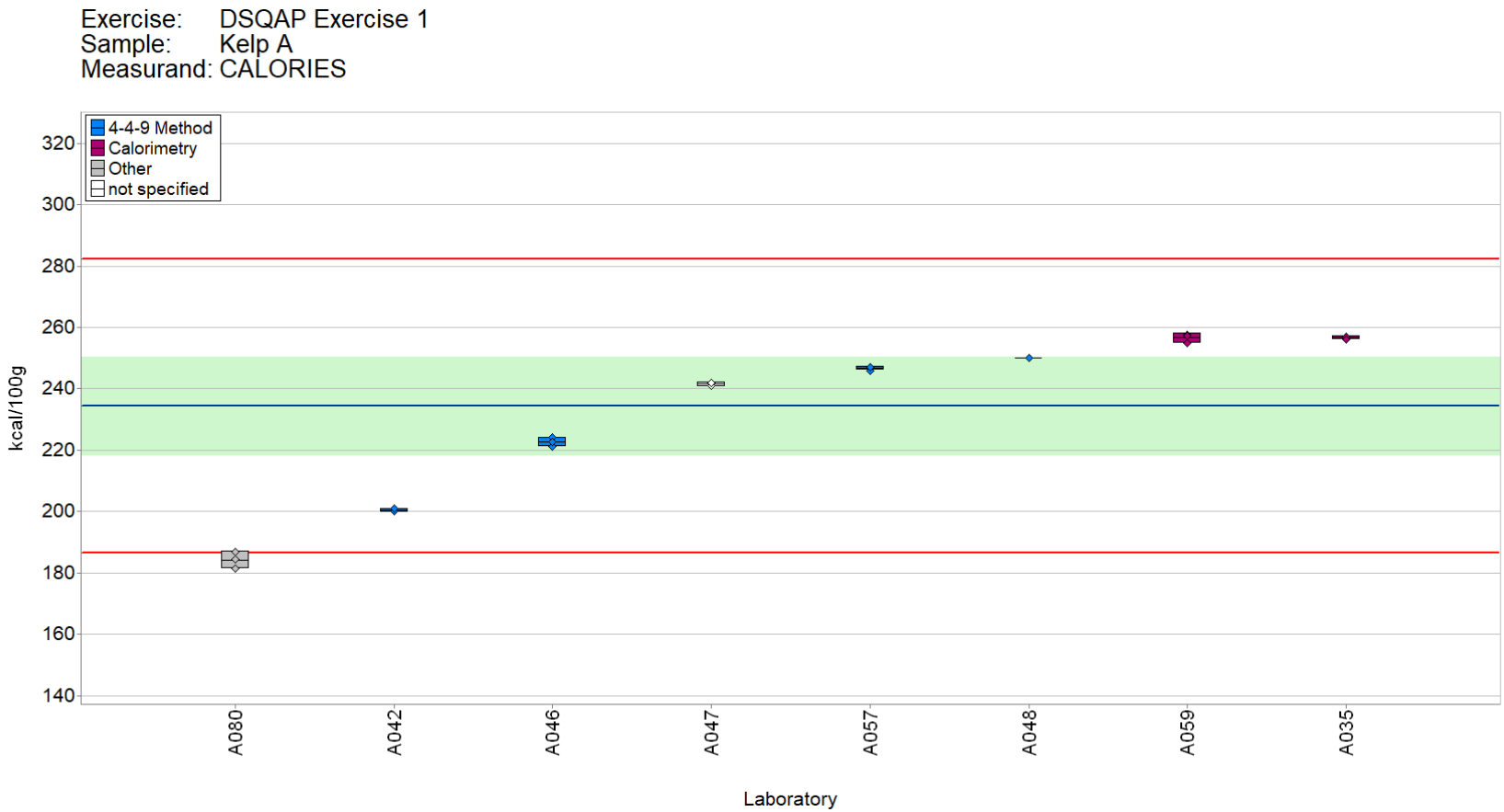

Fig. 6-9. Calories in Kelp A (data summary view – analytical method).

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 red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

7. Contaminants

7.1. Executive Summary

Per- and polyfluoroalkyl substances (PFAS) are a class of industrial compounds used in a variety of processes and consumer products and exposure has become a major public health concern due to potential adverse health effects [50, 51]. Participation in this study was extremely low, with only one laboratory reporting results for PFAS in the kelp sample.

7.2. Background

Algae are known to take up chemical compounds in their environment, and consumer products must be free from high levels of harmful contaminants. Per- and polyfluoroalkyl substances (PFAS) are a class of industrial compounds used in a variety of manufacturing processes for consumer products and exposure has become a major public health concern due to potential adverse health effects. PFAS are highly resistant to degradation and can persist in the environment and the body for years. Standardization and harmonization of analytical measurement techniques are essential for the detection of PFAS for consumer safety and for the association of PFAS concentrations with exposure sources and health outcomes. In this study participants were asked to use their in-house analytical method to detect the presence of 15 select PFAS as shown in **Table 7-1**.

Table 7-1. PFAS compounds to be analyzed in kelp.

Abbreviation	Compound Name
PFBS	perfluorobutane sulfonic acid
PFBA	perfluorobutanoic acid
PFDOA	perfluorododecanoic acid
PFHPA	perfluoroheptanoic acid
PFHXDA	perfluorohexadecanoic acid
PFHXS	perfluorohexane sulfonic acid
PFHXA	perfluorohexanoic acid
PFNA	perfluorononanoic acid
PFOS	perfluorooctane sulfonic acid
PFOA	perfluorooctanoic acid
PFPEA	perfluoropentanoic acid
PFODA	perfluorooctadecanoic acid
PFTEDA	perfluorotetradecanoic acid
PFTRDA	perfluorotridecanoic acid
PFUDA	perfluoroundecanoic acid

7.3. Study Information

Participants were provided with three packets each containing 50 g of Kelp A (*Saccharina latissima f. angustissima*, origin ME, USA). Participants were asked to store the material at controlled room temperature, 20 °C to 25 °C, in the original unopened packets and to prepare one sample and report one value from each packet provided. Before use, participants were instructed to thoroughly mix the contents of the packet and to allow contents to settle for one minute prior to opening to minimize the loss of fine particles prior to removal of a test portion for analysis. The approximate analyte levels were not reported to participants prior to the study.

7.4. Study Results and Technical Recommendations

The enrollment for the contaminants study was low, with only six laboratories requesting samples. One laboratory returned results for PFBS, PFOS, and PFNA. The results returned from this laboratory are presented in **Table 7-2**.

Table 7-2. Results for PFAS compounds in kelp.

Analyte	Reported Value from Laboratory A011 Mass Fraction ± SD (ng/g)		
PFBS	0.009	±	0*
PFOS	0.027	±	0.004
PFNA	0.04	±	0*

* Standard deviation is zero because all three values reported were equivalent

Because only one laboratory provided results, no technical recommendations can be made at this time. Based on this data, the levels are very low in the material and are similar to those found in the FDA's Total Diet Study analysis, which are near their method detection limits [52]. Contaminant uptake into algae is still of concern and other environmental organic contaminants could be a focus of a future study.

8. Vitamins II (B Vitamins in Meal Replacement Formulations)

8.1. Executive Summary

This study was designed to identify discrepancies and/or analytical challenges for the measurements of B vitamins across the testing community, using similarly promoted consumer products with different formulations that would result in different sample preparation requirements. Participants measured eight B vitamins in powdered and ready-to-drink materials, and participation rates ranged from 23 % to 48 %. Overall, laboratories had more difficulty with water-soluble vitamin measurements in the liquid sample. Participants also struggled to achieve results within the target range of tolerance for all measurands in SRM 3252, however, laboratories using LC-MS/MS performed well because of the increased sensitivity and selectivity of multiple reaction monitoring. Several participants using LC-Abs were unable to quantify multiple vitamins in each material.

8.2. Background

B vitamins are a group of water-soluble compounds important for maintaining good health and well-being. These vitamins impact energy levels, brain function, and cell metabolism as they are involved in converting food into energy, creating new blood cells, and maintaining healthy cells throughout the body [53]. B Vitamin deficiencies have been linked to anemia, depression, fatigue, muscle weakness, and poor memory [53]. Meal replacement supplements contain many nutritional compounds, including water-soluble vitamins. Products in both powdered and ready-to-consume forms are available on the market and have identical labeling requirements, but the matrix complexities and differences can pose measurement challenges for analytical testing laboratories.

8.3. Study Information

Participants were provided with one bottle containing 325 mL of liquid meal replacement drink formulation, three packets each containing 10 g of a meal replacement drink powder formulation, and three packets each containing 10 g of SRM 3252 Protein Drink Mix. Participants were asked to store all materials at controlled room temperature, 20 °C to 25 °C, in the original unopened packaging. Before use, participants were instructed to mix the contents of the liquid meal replacement drink formulation bottle thoroughly and to prepare three samples of at least 5 g before using their in-house method of analysis to report three values for the determination of B vitamins. For the meal replacement drink powder formulation and SRM 3252, participants were instructed to thoroughly mix the contents of each packet individually prior to removal of a test portion. For analysis, the participants were instructed to use a sample size of at least 5 g for the determination of vitamins B_1, B_2, B_3 (niacin), B_5, B_6, at least 1 g for the determination of vitamin B_7 (biotin), and to use an appropriate sample size for their in-house analytical methods for the determination of vitamins B_9 and B_{12}.

The target values and standard uncertainties for water-soluble vitamins used in this study are provided in **Table 8-1** on an as-received basis. The target values and uncertainties (set at 10 % of target value) for water-soluble vitamins in the two meal replacement materials were determined based on product labels. The certified values for thiamine, riboflavin, niacin, pantothenic acid, and pyridoxine in SRM 3252 were determined at NIST by LC-MS/MS. The certified value for biotin in SRM 3252 was determined at NIST by LC-MS. The target values for folic acid and cyanocobalamin were determined by collaborating laboratories using methods referenced in the COA [54]. Target analyte levels were not reported to participants prior to the study. The enrollment and reporting statistics for this study are described in **Table 8-2**.

Table 8-1. Target mass fractions of B vitamins for meal replacement drinks and SRM 3252.

Analyte	Target Mass Fraction (mg/kg)								
	Meal Replacement Drink (Liquid)[a]			Meal Replacement Drink (Powder)[a]			SRM 3252[b]		
Thiamine (B_1)	1.23	±	0.12	11.5	±	1.2	11.7	±	1.5
Riboflavin (B_2)	1.32	±	0.13	6.92	±	0.69	27.3	±	2.6
Niacin (B_3)	16.3	±	1.6	203.8	±	20.4	6.96	±	0.25
Pantothenic acid (B_5)	5.23	±	0.52	38.5	±	3.9	142	±	11
Pyridoxine (B_6)	1.75	±	0.18	19.23	±	1.92	27.7	±	1.5
Biotin (B_7)	0.0310	±	0.0031	0.385	±	0.039	4.21	±	0.18
Folic acid (B_9)	0.246	±	0.025	2.31	±	0.23	7.2	±	1.8
Cobalamin (B_{12})	0.00740	±	0.00074	0.0385	±	0.0039	0.103	±	0.025

[a] target values and standard uncertainties (10 % of value) determined from the manufacturer product labels.
[b] target value ± U_{95}

Table 8-2. Enrollment and reporting statistics of B vitamins in meal replacement drinks and SRM 3252.

Analyte	Number of Laboratories Requesting Samples	Number of Laboratories Reporting Results (Percent Participation)		
		Meal Replacement Drink (Liquid)	Meal Replacement Drink (Powder)	SRM 3252
Thiamine (B_1)	27	10 (37 %)	11 (41 %)	8 (30 %)
Riboflavin (B_2)	26	11 (42 %)	10 (38 %)	9 (35 %)
Niacin (B_3)	25	8 (32 %)	10 (40 %)	9 (36 %)
Pantothenic acid (B_5)	25	11 (44 %)	12 (48 %)	9 (36 %)
Pyridoxine (B_6)	26	9 (35 %)	10 (38 %)	8 (31 %)
Biotin (B_7)	25	8 (32 %)	8 (32 %)	7 (28 %)
Folic acid (B_9)	25	11 (44 %)	9 (36 %)	8 (32 %)
Cobalamin (B_{12})	26	8 (31 %)	7 (27 %)	6 (23 %)

8.4. Study Results and Technical Recommendations

The goal for this study was to determine any discrepancies and/or analytical challenges for the measurements of B vitamins across the testing community, using similarly promoted consumer products with different sample preparation requirements. Participants were asked to measure eight B vitamins in powdered and ready-to-drink materials. Participation rates ranged from 23 % to 48 % based on the specific vitamin and material and a detailed breakdown can be found in the sections below. The relatively low participation rates affect overall technical recommendations. Most of the reported results for the Meal Replacement Drink (Powder) and SRM 3252 Protein Drink Mix (Powder) were within or overlapping with the consensus range of tolerance (**Table 8-3**). Sample preparation techniques used for powdered materials may be more selective to B vitamins and other similar analytes which can help simplify the final sample for analysis. However, sample preparation techniques used for liquid materials generally involve dilution without removing other matrix components, which may lead to interferences in the instrumental analysis.

The target values associated with the meal replacement materials (liquid and powder) were based on their respective product label claims. Regulations permit manufacturers to add higher levels of nutrients to a product with respect to the product label claims to compensate for changes over the shelf life [55]. Consistent with this practice, most participants reported values for B vitamins in both commercial meal replacement materials above the target range of tolerance. Participants also struggled to achieve results within the target range of tolerance for SRM 3252 for all measurands. Laboratories using LC-MS/MS obtained values within the target range of tolerance for most of the B vitamins in SRM 3252, likely due to the ability to increase sensitivity and selectivity through multiple reaction monitoring. B vitamins tend to have multiple stable fragmentation patterns which is a desirable property for LC-MS/MS analysis. Several participants using LC-Abs reported values below their LOQ for multiple vitamins in each material.

Table 8-3.Description of the location of the consensus range in relation to the target range for B vitamins in meal replacement drinks and SRM 3252.

Analyte	Consensus Range in relation to Target Range		
	Meal Replacement Drink (Liquid)	Meal Replacement Drink (Powder)	SRM 3252
Thiamine (B_1)	Overlapping Above (mean within range)	Overlapping Above (mean above range)	Overlapping Below (mean slightly below range)
Riboflavin (B_2)	Slightly Above	Overlapping (mean within range)	Overlapping Below (mean within range)
Niacin (B_3)	Slightly Above	Within (mean = target)	Significantly Above
Pantothenic acid (B_5)	Overlapping (mean above range)	Slightly Above	Overlapping Above (mean within range)
Pyridoxine (B_6)	Above	Overlapping Above (mean within range)	Overlapping (mean above range)
Biotin (B_7)	Above	Within	Overlapping Below (mean within range)
Folic acid (B_9)	Above	Within	Within
Cobalamin (B_{12})	Overlapping Below (mean slightly below range)	Overlapping Above (mean within range)	Within

Most laboratories that reported sample preparation methods reported using solvent extraction to determine thiamine, pyridoxine, cobalamin, niacin, pantothenic acid, and folic acid in the samples as seen in **Table 8-4**.

Table 8-4. Summary of sample preparation methods for determination of B vitamins in meal replacement drinks and SRM 3252.

Reported Sample Preparation	Percent Reporting %							
	B_1	B_2	B_3	B_5	B_6	B_7	B_9	B_{12}
Solvent extraction	31%	21%	25%	43%	30%	36%	44%	58%
Solvent extraction & solid phase extraction	23%	14%	25%	29%	10%	45%	22%	25%
Enzymatic digestion	23%	21%	19%	-	30%	-	-	-
Enzymatic hydrolysis	8%	7%	19%	14%	10%	18%	33%	17%
Dilution	15%	14%	13%	14%	20%	-	-	-
Acid digestion	-	21%	-	-	-	-	-	-

Most laboratories reported using LC-Abs to determine thiamine, riboflavin, pyridoxine, biotin, and folic acid. Most laboratories reported other as the analysis methods for determining niacin and pantothenic acid. Most laboratories reported using either LC-MS or microbial assay to determine cobalamin as shown in **Table 8-5**.

Table 8-5. Summary of analytical methods for determination of B vitamins in meal replacement drinks and SRM 3252.

Reported Analytical Method	Percent Reporting %							
	B_1	B_2	B_3	B_5	B_6	B_7	B_9	B_{12}
LC	-	-	7%	7%	-	-	12%	-
LC-MS	-	11%	-	7%	-	25%	12%	22%
LC-MS/MS	12%	11%	11%	14%	13%	20%	12%	17%
LC-Abs	46%	26%	19%	21%	33%	30%	40%	17%
LC-FLD	12%	11%	19%	3%	13%	10%	4%	6%
Microbial Assay	12%	11%	11%	10%	13%	15%	20%	22%
Spectrophotometry	-	11%	11%	10%	-	-	-	17%
Other	19%	19%	22%	28%	29%	-	-	-

Several laboratories did not report sample preparation techniques, so conclusions about performance based on approach cannot be drawn. However, improper sample preparation may lead to biased results and laboratories reporting results outside of the target range of tolerance should ensure the utilized sample preparation protocols are appropriate for the specific sample matrix. Sample preparation techniques must be able to fully extract the analytes from the sample matrix, while also being mindful of analyte degradation and/or conversion. The use of reduced lighting/yellow lighting when conducting preparation techniques and storing samples in the dark or in amber colored vials can significantly reduce UV induced analyte degradation.

For water-soluble vitamins, especially those with different chemical forms, an understanding of what analyte is being measured and reported, and use of appropriate, high quality, and well characterized calibrants, are critical. Calculations and reporting units must be verified prior to submission of results. Laboratories often report results in the wrong units or forget a dilution factor during the calculation of the results, resulting in poor performance for the study. Quality assurance samples are important for ensuring the measurement system is in control. QAs can be commercially available reference materials (CRMs, SRMs, or RMs) or materials prepared and characterized in-house.

8.4.1. Thiamine (B_1)

The average within-laboratory RSD for thiamine in meal replacement drink (powder) of 3.8 % was acceptable compared to the published standard of at or below 5 % (**Table 8-6**) [56]. Repeatabilities for the liquid sample and SRM 3252, and reproducibilities for all samples, were outside of the established performance requirements for laboratories using the same method [56]. The between-laboratory RSDs for all three materials are significantly higher indicating that the participants and the methods being used are not in agreement with each other.

Table 8-6. Summary of laboratory variabilities for thiamine in meal replacement drinks and SRM 3252.

	Thiamine (B_1)	
Material	Within-Laboratory Variability (% RSD)	Between-Laboratory Variability (% RSD)
Meal Replacement Drink (Liquid)	7.5 %	34 %
Meal Replacement Drink (Powder)	3.8 %	67 %
SRM 3252	11.1 %	40 %

Overall, five participants reported values for B_1 in the meal replacement drink (liquid) that were within the 95 % confidence interval for the consensus mean which overlaps with the NIST range of tolerance (**Fig. 8-1**). The consensus mean (1.51 ± 0.51 mg/kg) was just within the target range. While the between-laboratory variability (34 %) was outside of the established performance requirements, the RSD is skewed by the small data set and laboratory A018 which reported results about ten times higher than the target value. This participant should check for potential unit errors and dilution factor miscalculations.

Similarly for the meal replacement drink (powder), the 95 % confidence interval for the consensus mean overlapped with the NIST range of tolerance (**Fig. 8-2**). The consensus mean was above the target range and nearly two times the target value. Laboratory A036 reported results for B_1 in the meal replacement drink (powder) about ten times higher than the target value indicating potential unit and calculation errors.

The results for B_1 in SRM 3252 showed the opposite trend when compared to the two meal replacement materials. Three laboratories reported results below the NIST range of tolerance (**Fig. 8-3**) with zero overlap between the 95 % confidence interval for the consensus mean and the target range. This is likely due to the combination of target values in the SRM being well characterized and laboratories struggling to reach similar levels of extraction completeness. Whereas the target values associated with the commercial products are based on label claims which are most likely underestimating true vitamin levels. Laboratory A018 reported values roughly nine times the consensus mean and should check for unit and calculation errors. Since only four participants reported results without LOQ limitations for B_1 in SRM 3252, specific technical recommendations are limited.

Vitamin B_1 is known to be chromatographically unretained and therefore can be challenging to measure by traditional LC methods. Unstable retention times and peak areas due to unretained components would produce higher observed repeatability and increase the potential susceptibility to interferences from other unretained components; however, this common analytical challenge for B_1 is not evident in the within-laboratory variabilities. In general, it is good practice to understand in-house methods recovery capabilities for specific analytes to ensure accurate measurements. Another general point to consider is the reporting form of thiamine. Analytical standards for B_1 are typically an HCl or mononitrate salts and should be factored into the mass fraction calculations.

Four laboratories did not report quantitative values for thiamine in all three materials due to mass fractions being below the LOQ of their LC methods with absorbance or fluorescence detection. Since these materials are representative of commercial products, participants should strive to improve their LOQs for B_1 measurements. The LOQ recommendation for vitamin B_1 determination in AOAC SMPR 2015.002 is 0.025 mg/kg, at which level methods would be able to measure vitamin B_1 in the study samples [56]. Notably, laboratories that used mass spectrometry or microbiological assay were within or just outside of the target range of tolerance.

Additional tables and figures for the analysis of thiamine can be found in **Appendix F.**

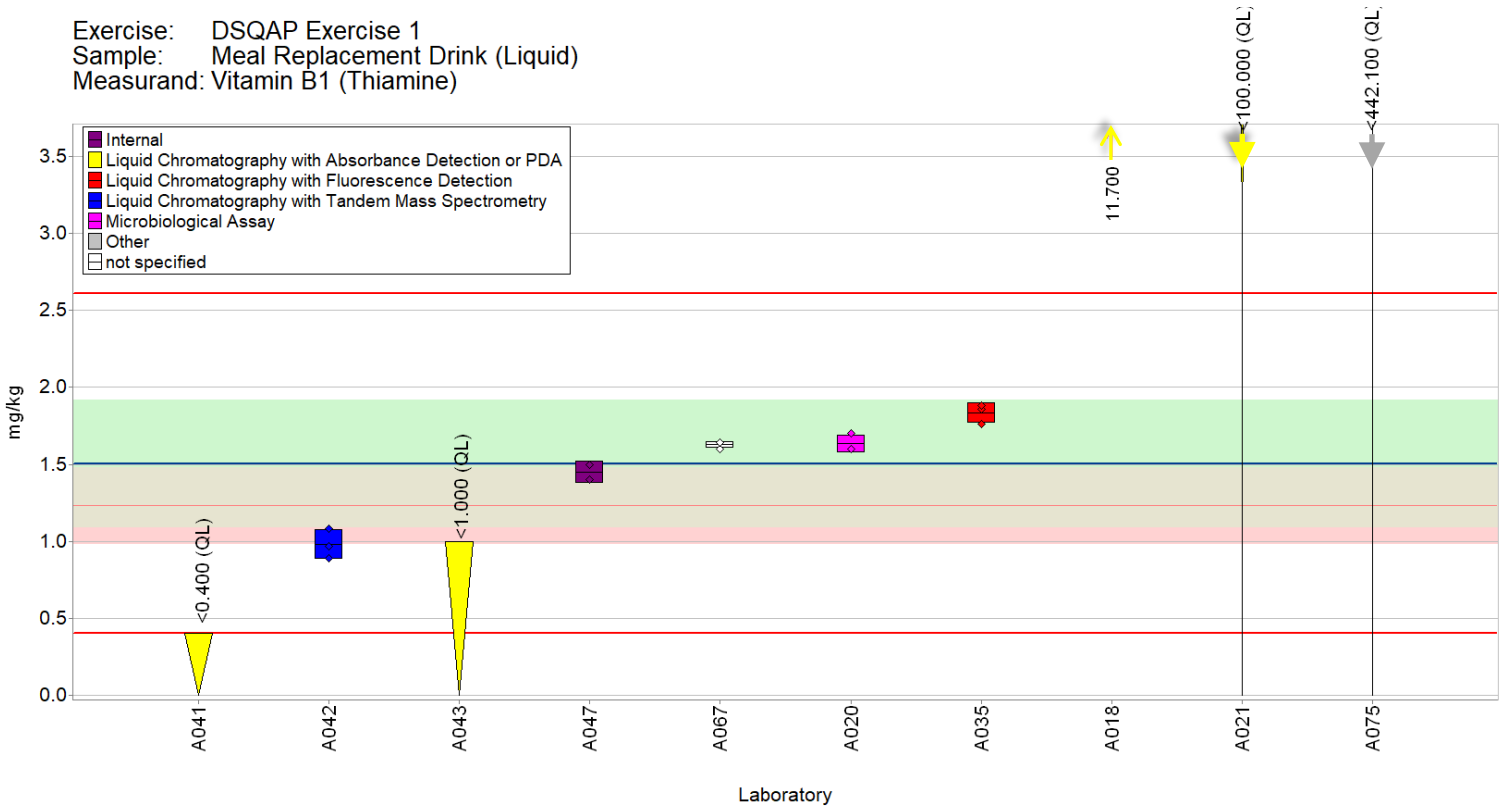

Fig. 8-1. Vitamin B_1 (thiamine) in Meal Replacement Drink (Liquid) (data summary view – analytical method).

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 red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

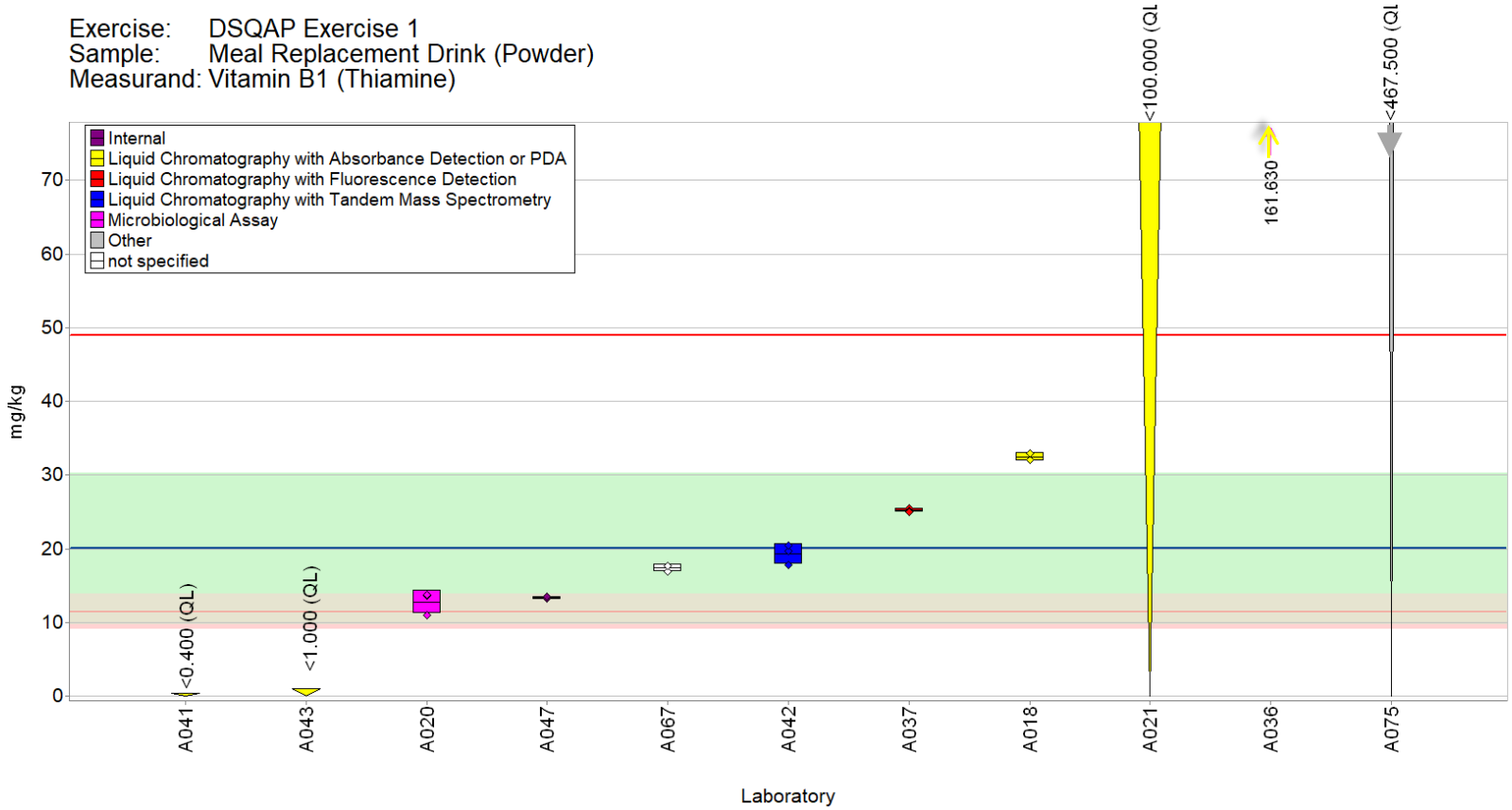

Fig. 8-2. Vitamin B_1 (thiamine) in Meal Replacement Drink (Powder) (data summary view – analytical method).

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 red solid line represents the upper bound of the consensus range of tolerance, calculated as the value above the consensus mean that results in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$, with the lower bound set to zero. The red shaded region (beige here due to overlap with the green consensus confidence interval) represents the NIST range of tolerance, which encompasses the target value bounded by its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$.

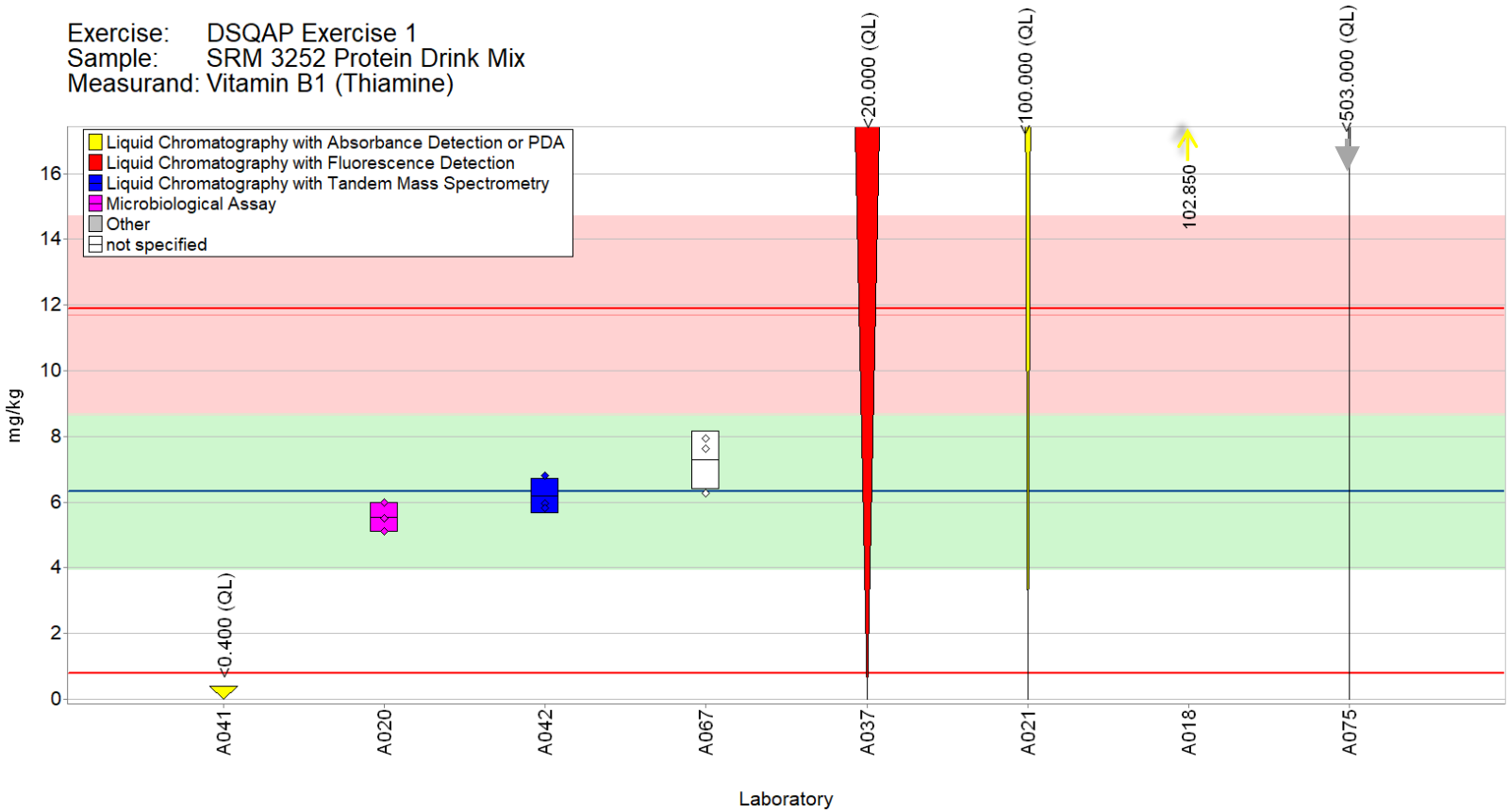

Fig. 8-3. Vitamin B_1 (thiamine) in SRM 3252 Protein Drink Mix (data summary view – analytical method).

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 red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

8.4.2. Riboflavin (B_2)

The within-laboratory RSDs for 3 of the 7 laboratories and 4 of the 6 laboratories reporting quantitative results for riboflavin in the two commercial meal replacement products were within the published repeatability recommendation of at or below 5 % [57]. Overall, the average within-laboratory RSDs were below 10 % for riboflavin in the two commercial meal replacement products, indicating that participants should identify and minimize sources of variability in their in-house methods (**Table 8-7**). The average within-laboratory RSD for SRM 3252 was slightly higher at 15.4 %, with only 1 of 6 laboratories reporting repeatabilities within the recommended limit of 5 %. The between-laboratory RSDs for all three materials were outside of the established performance requirements of 10 % [57] indicating that existing methods have unidentified biases or that laboratories are not successfully implementing unbiased methods.

Table 8-7. Summary of laboratory variabilities for riboflavin in meal replacement drinks and SRM 3252.

	Riboflavin (B_2)	
Analyte	Within-Laboratory Variability (% RSD)	Between-Laboratory Variability (% RSD)
Meal Replacement Drink (Liquid)	9.6 %	22 %
Meal Replacement Drink (Powder)	3.7 %	83 %
SRM 3252	15.4 %	40 %

The 95 % confidence interval for the consensus mean for the meal replacement drink (liquid) was above the NIST range of tolerance; however, the NIST range of tolerance overlapped with the lower limit of consensus range of tolerance (**Fig. 8-4**). The consensus mean is slightly above the target value (product label) which is consistent with the allowed overage regulations.

Although the between-laboratory variability for the meal replacement drink (powder) was well outside of the established performance requirements, the 95 % confidence interval for the consensus mean overlaps with the NIST range of tolerance (**Fig. 8-5**). Similarly, for SRM 3252, the 95 % confidence interval for the consensus mean overlapped with the NIST range of tolerance (**Fig. 8-6**). The consensus means for both the meal replacement drink (powder) and SRM 3252 were below the target value. Although vitamin B_2 is considered a water-soluble vitamin, its aqueous solubility is lower than other B vitamins [58]. Adding modifiers to the solvent and using lower concentrations for stock solution preparations can aid in solubility for vitamin B_2. Laboratories should consider the solubility properties of these analytes when optimizing sample preparation.

For each sample, three to four laboratories reported that values for riboflavin were below their LOQ. The levels of riboflavin in the study samples were about an order of magnitude higher than the recommended LOQ for riboflavin methods (0.0375 mg/kg) [57]. Laboratories should evaluate their process for determining their method LOQ and consider modifications to allow detection of riboflavin at levels commonly found in commercial products.

Additional tables and figures for the analysis of riboflavin can be found in **Appendix F**.

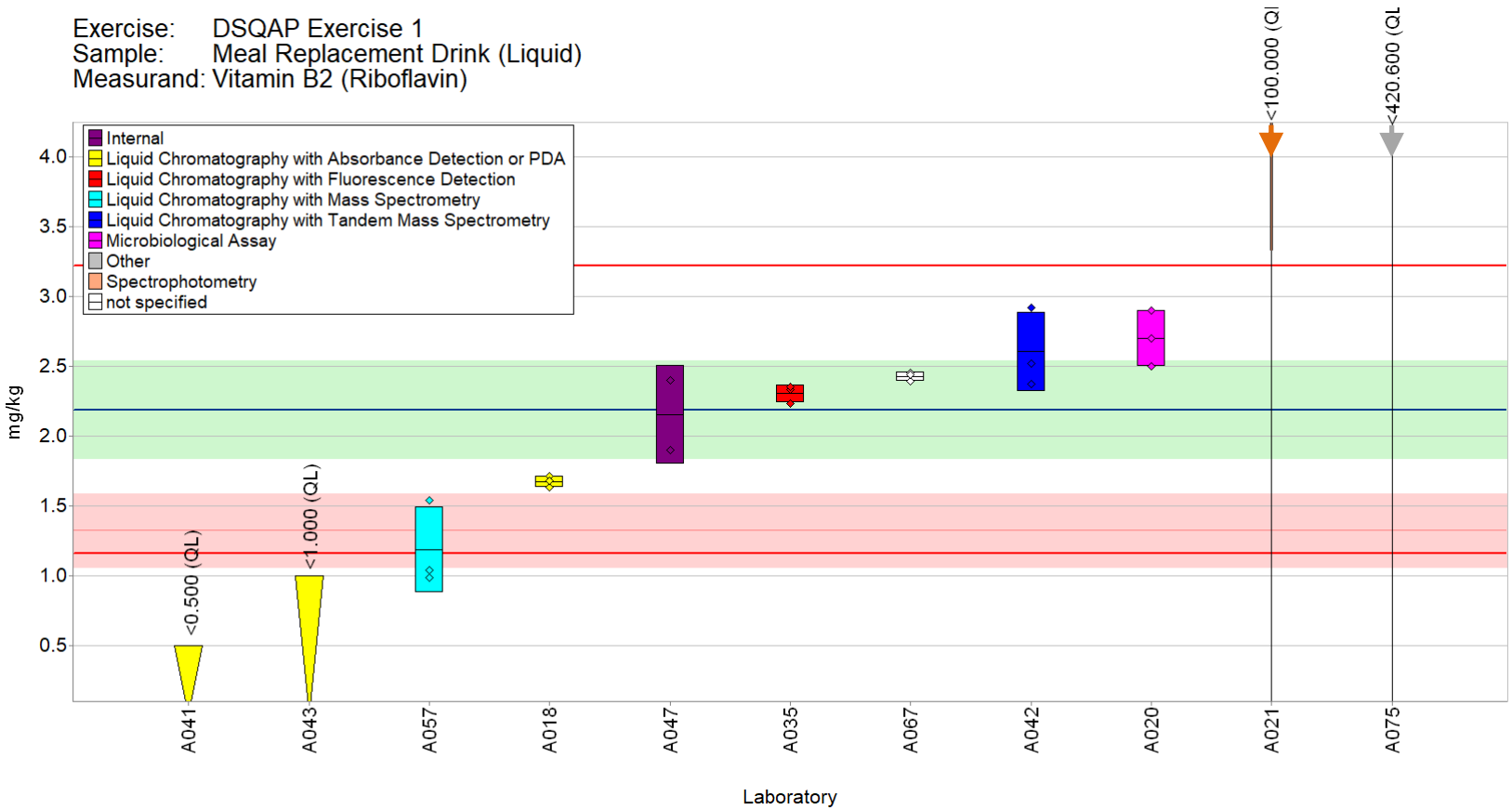

Fig. 8-4. Vitamin B_2 (riboflavin) in Meal Replacement Drink (Liquid) (data summary view – analytical method).

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 red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

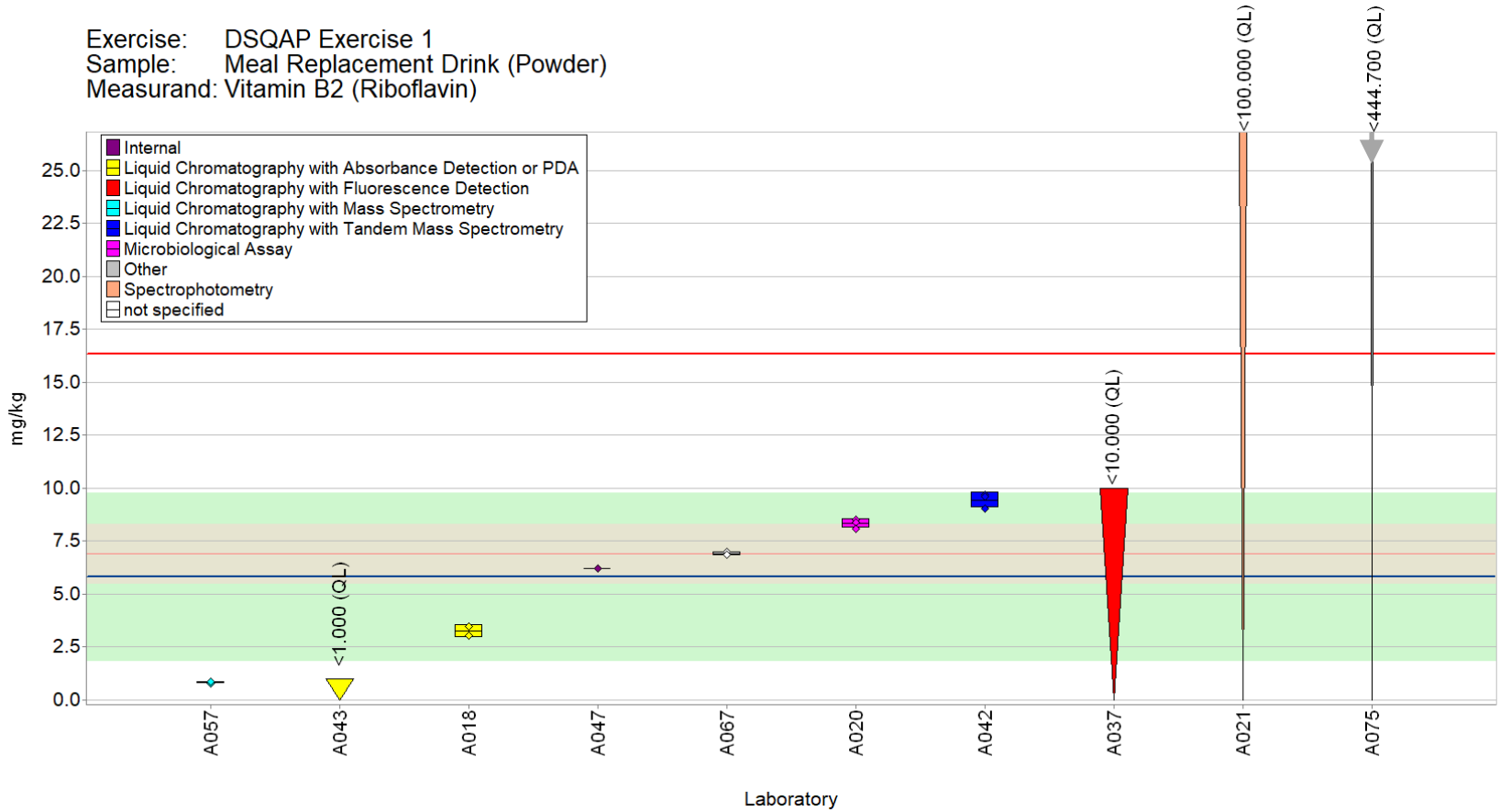

Fig. 8-5. Vitamin B_2 (riboflavin) in Meal Replacement Drink (Powder) (data summary view – analytical method).

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 red solid line represents the upper bound of the consensus range of tolerance, calculated as the value above the consensus mean that results in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$, with the lower bound set to zero. The red shaded region (beige here due to overlap with the green consensus confidence interval) represents the NIST range of tolerance, which encompasses the target value bounded by its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$.

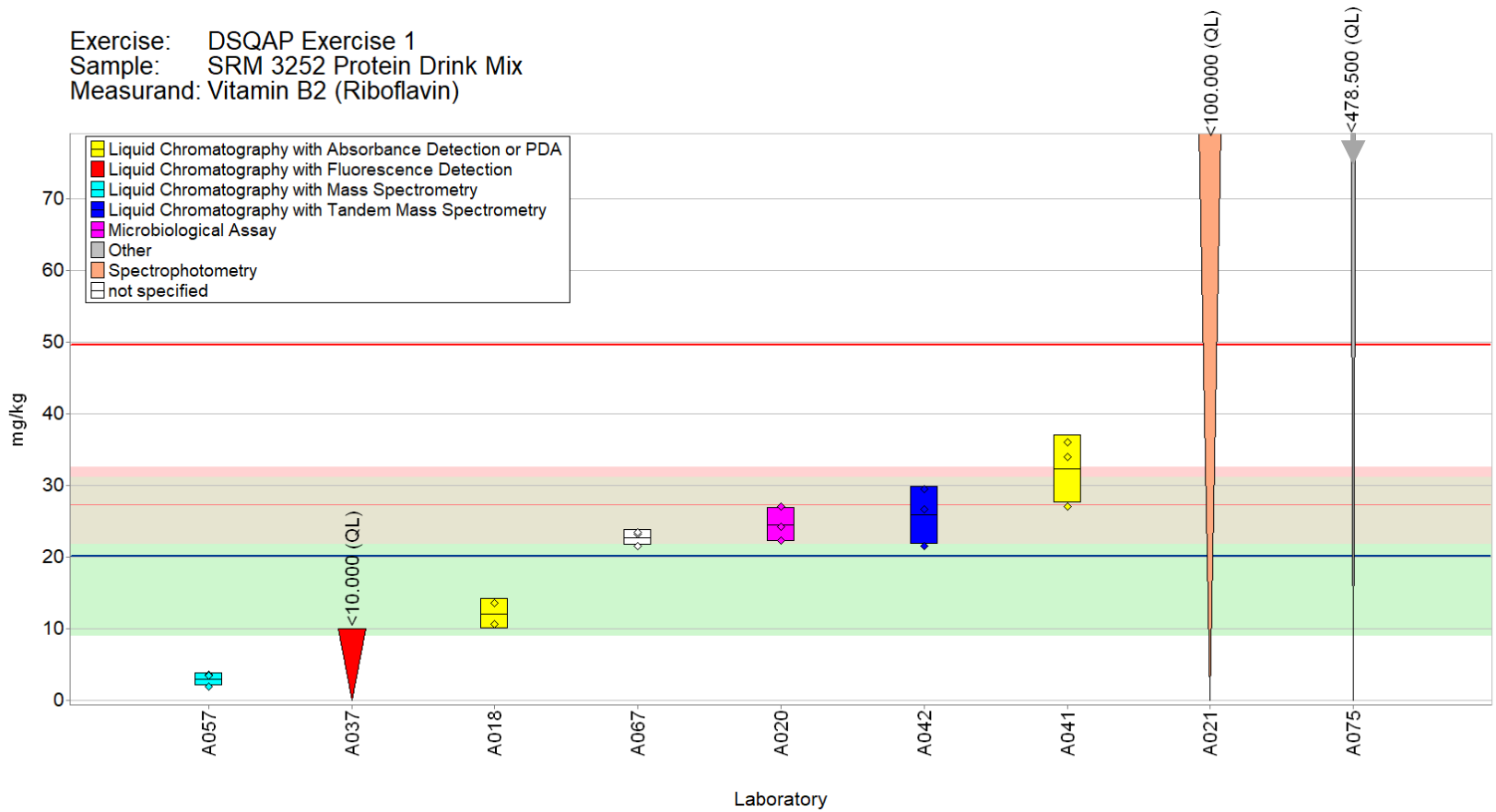

Fig. 8-6. Vitamin B_2 (riboflavin) in SRM 3252 Protein Drink Mix (data summary view – analytical method).

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 red solid line represents the upper bound of the consensus range of tolerance, calculated as the value above the consensus mean that results in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$, with the lower bound set to zero. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

8.4.3. Niacin (B_3)

The within-laboratory RSDs for 5 of the 6 laboratories reporting quantitative results for niacin in the meal replacement drink liquid were within the published repeatability recommendation of at or below 5 % [59], and the average repeatability across all laboratories was 2.2 % (**Table 8-8**). For the meal replacement drink powder, 3 of the 7 laboratories reporting quantitative results for niacin were within the published repeatability recommendation and the average repeatability across all laboratories was higher at 7.4 %. The repeatabilities for 3 of the 7 laboratories reporting quantitative results for niacin in SRM 3252 were within the published recommendation, with an average repeatability across all laboratories of 5.9 %. Laboratories reporting repeatabilities greater than 5 % for determination of niacin in these samples should identify and minimize sources of variability in their in-house methods. The between-laboratory RSDs for all three materials were outside of the established performance requirements of at or below 10 %; however, the reproducibility is much better for niacin than other B vitamins in the study at below 20 % for all samples.

Table 8-8. Summary of laboratory variabilities for niacin in meal replacement drinks and SRM 3252.

	Niacin (B_3)	
Analyte	Within-Laboratory Variability (% RSD)	Between-Laboratory Variability (% RSD)
Meal Replacement Drink (Liquid)	2.2 %	20 %
Meal Replacement Drink (Powder)	7.4 %	14 %
SRM 3252	5.9 %	15 %

Overall, the target value (product label) for both commercial materials were within the respective consensus range of tolerance. The 95 % confidence interval for the consensus mean for the meal replacement drink (liquid) was above the NIST range of tolerance (**Fig. 8-7**) while both statistical parameters overlapped for the meal replacement drink (powder) (**Fig. 8-8**).

For SRM 3252, the consensus range of tolerance for niacin was significantly above the NIST range of tolerance (**Fig. 8-9**). The target value for niacin in SRM 3252 is 7.33 mg/kg, which is a minor contribution to the total B_3 (as niacin and niacinamide) in the material. Reported values are closely aligned with the total B_3 target value in SRM 3252 (287 mg/kg), indicating that participants are measuring and reporting total B_3. Participants should be aware of their method's capabilities to measure total B_3 and/or the specific vitamers and confirm the requested form prior to reporting results.

Participants indicating levels of niacin in these materials below their LOQs should evaluate their analytical techniques as these levels should not be challenging to measure. The recommended LOQ for total B_3 is 0.25 mg/kg [59]. Additional tables and figures with raw data for the analysis of niacin can be found in **Appendix F**.

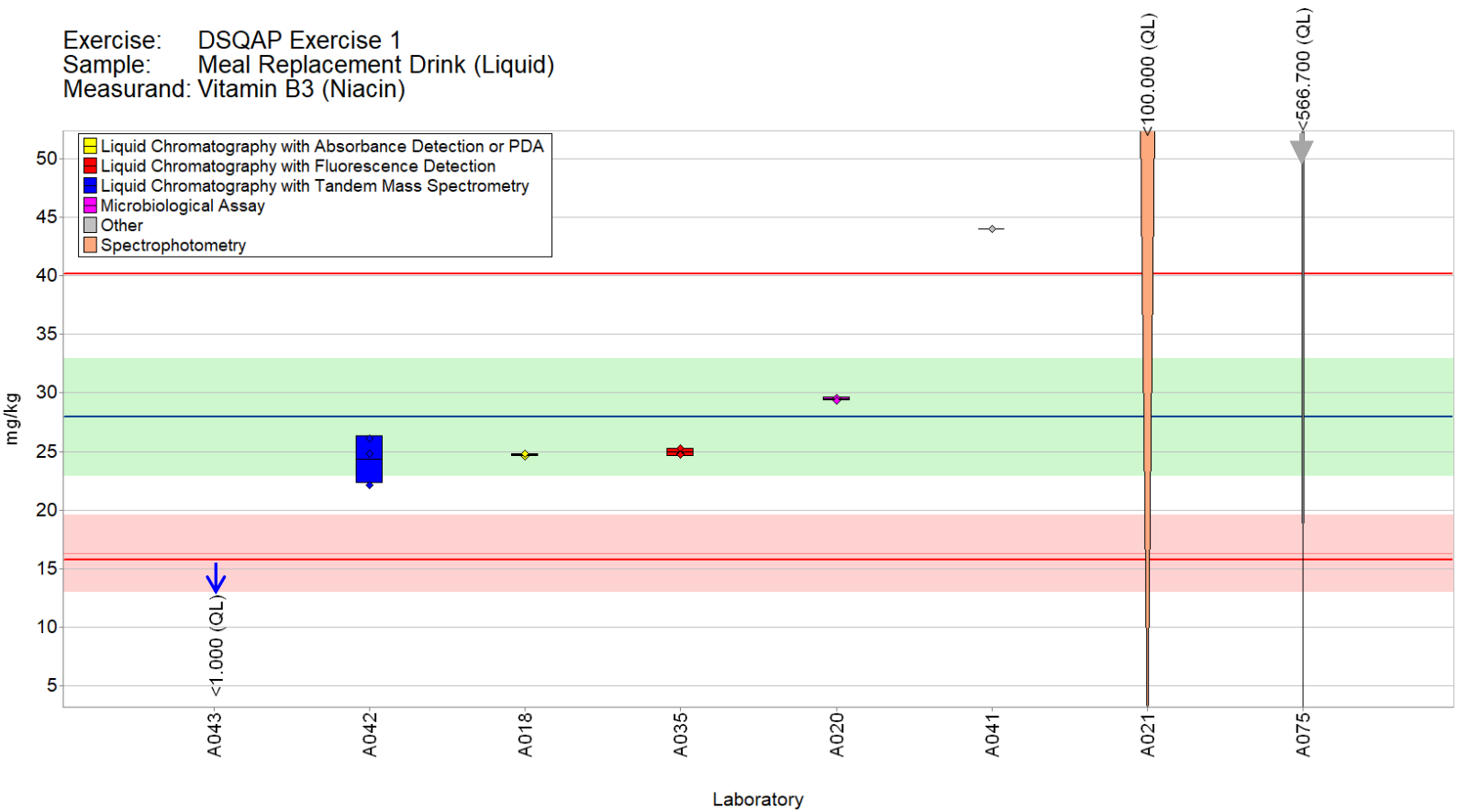

Fig. 8-7. Vitamin B_3 (niacin) in Meal Replacement Drink (Liquid) (data summary view – analytical method).

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 red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$.

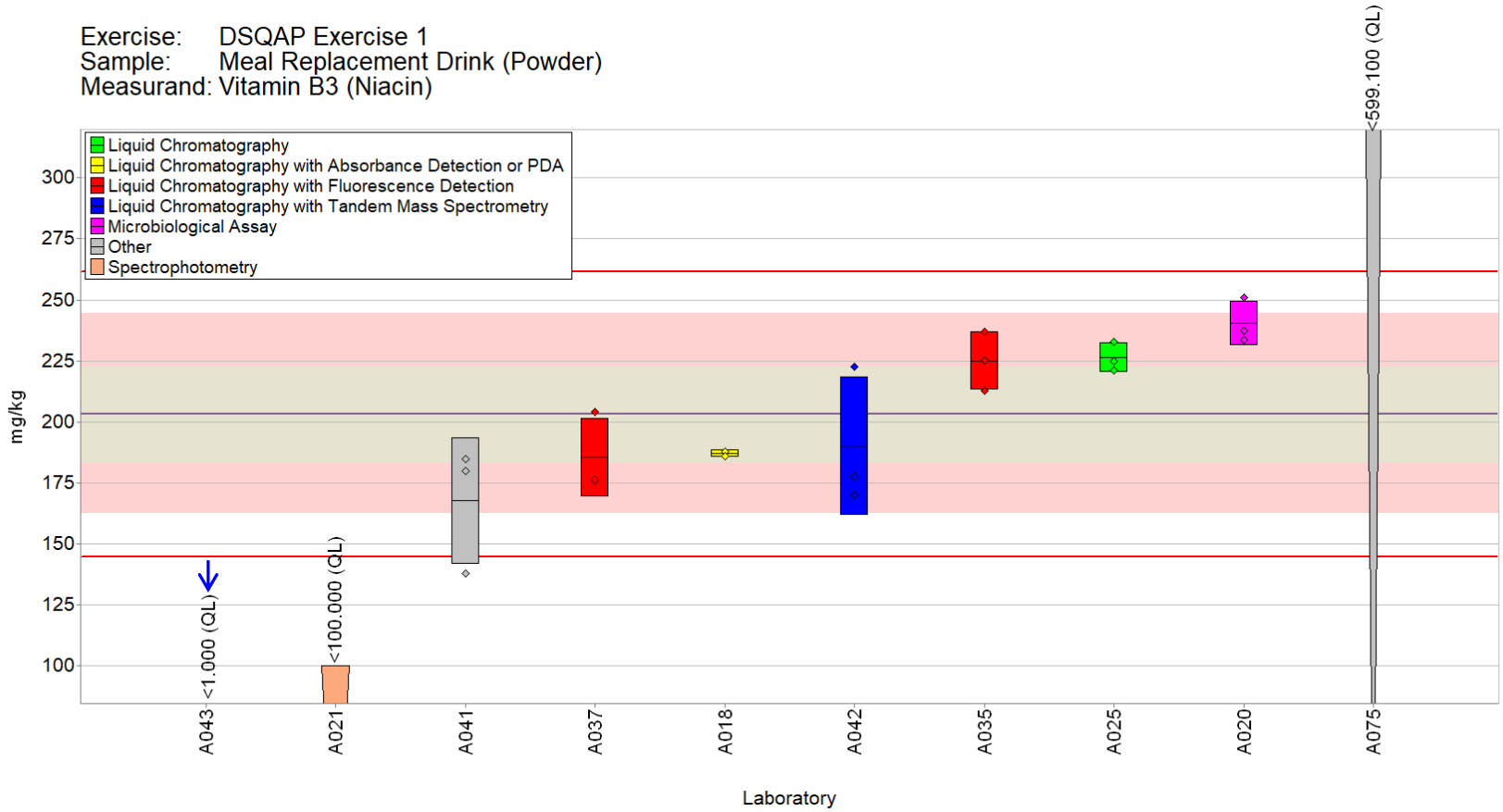

Fig. 8-8. Vitamin B_3 (niacin) in Meal Replacement Drink (Powder) (data summary view – analytical method).

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 (beige here due to overlap with the red NIST target range) represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

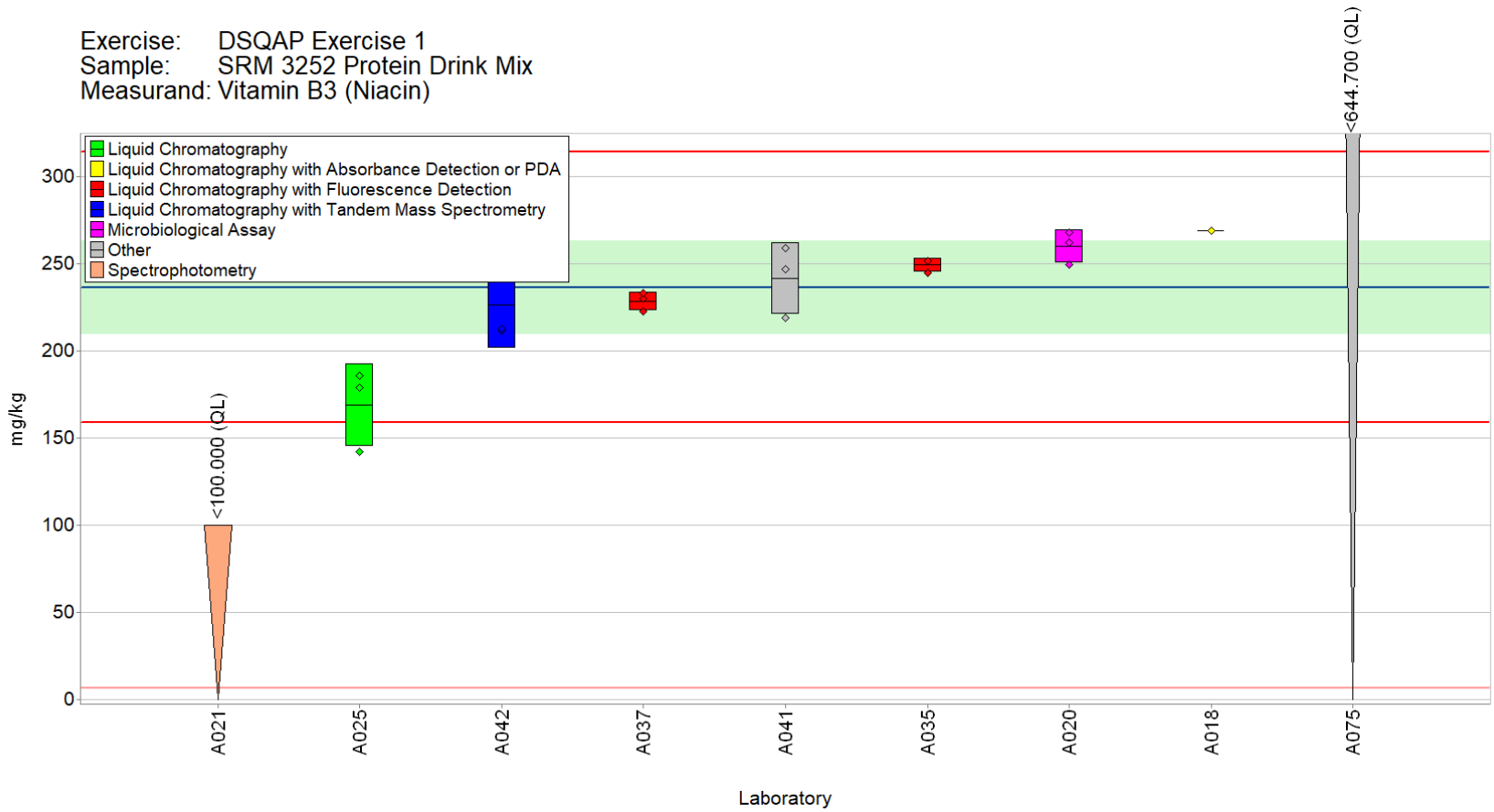

Fig. 8-9. Vitamin B_3 (niacin) in SRM 3252 Protein Drink Mix (data summary view – analytical method).

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 red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$. The red shaded region (at 7 mg/kg) represents the NIST range of tolerance, which encompasses the target value bounded by its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$.

8.4.4. Pantothenic Acid (B_5)

Compared to established analytical method performance requirements [60], the average repeatability across all laboratories for pantothenic acid the meal replacement drink (liquid) is within the 5 % recommendation (**Table 8-9**), and 5 of the 7 laboratories reporting quantitative results also demonstrating repeatabilities of at or below 5 %. For the meal replacement drink (powder), 2 of the 9 laboratories reporting quantitative results for pantothenic acid demonstrated repeatabilities of at or below 5 % with an average of 9.2 % across all laboratories. For SRM 3252, 1 of the 8 laboratories reporting quantitative results for pantothenic acid demonstrated repeatabilities of at or below 5 % with an average of 10.8 % across all laboratories. Laboratories reporting repeatabilities greater than 5 % for determination of pantothenic acid in these samples should identify and minimize sources of variability in their in-house methods. The between-laboratory RSDs for all three materials were significantly higher and outside of the recommended 15 %, indicating that existing methods may have unidentified biases or that laboratories are not successfully implementing unbiased methods.

Table 8-9. Summary of laboratory variabilities for pantothenic acid in meal replacement drinks and SRM 3252.

	Pantothenic Acid (B_5)	
Material	Within-Laboratory Variability (% RSD)	Between-Laboratory Variability (% RSD)
Meal Replacement Drink (Liquid)	3.1 %	> 100 %
Meal Replacement Drink (Powder)	9.2 %	59 %
SRM 3252	10.8 %	44 %

In the meal replacement drink (liquid), the 95 % confidence interval for the consensus mean overlapped with the NIST range of tolerance (**Fig. 8-10**). Laboratories A036 and A077 reported values significantly above the consensus range of tolerance indicating potential calculation and/or unit errors. Vitamin B_5 standards are typically in the form of calcium pantothenate which requires a conversion factor to obtain accurate mass fractions for pantothenic acid. While the meal replacement drink (liquid) contains calcium D-pantothenate, the value on the product label is converted to pantothenic acid. Laboratories should be aware of the form of vitamin B_5 being measured and reported. The high between-laboratory variability for this material is significantly skewed due to the two laboratories of seven with potential calculation errors.

Laboratories were in closer agreement with each other for the meal replacement drink (powder) and SRM 3252 compared to the liquid sample. The high between-laboratory variabilities for these two materials were less affected by laboratories with potential calculation and/or unit errors because more laboratories reported quantitative values for these samples. While the 95 % confidence interval for the consensus mean for the meal replacement drink (powder) was above the NIST range of tolerance, the NIST targe value fell within the consensus range of tolerance (**Fig. 8-11**). The NIST target value for SRM 3252 overlapped with the 95 % confidence interval for the consensus mean (**Fig. 8-12**).

Similar to other B vitamins, one to four laboratories report values for pantothenic acid that were below their method LOQ. These levels of B_5 should not be challenging to measure in commercial products as the recommended LOQ is 0.0625 mg/kg [60], so laboratories reporting LOQ limitations should aim to achieve quantitation abilities at lower levels.

Additional tables and figures for the analysis of pantothenic acid can be found in **Appendix F**.

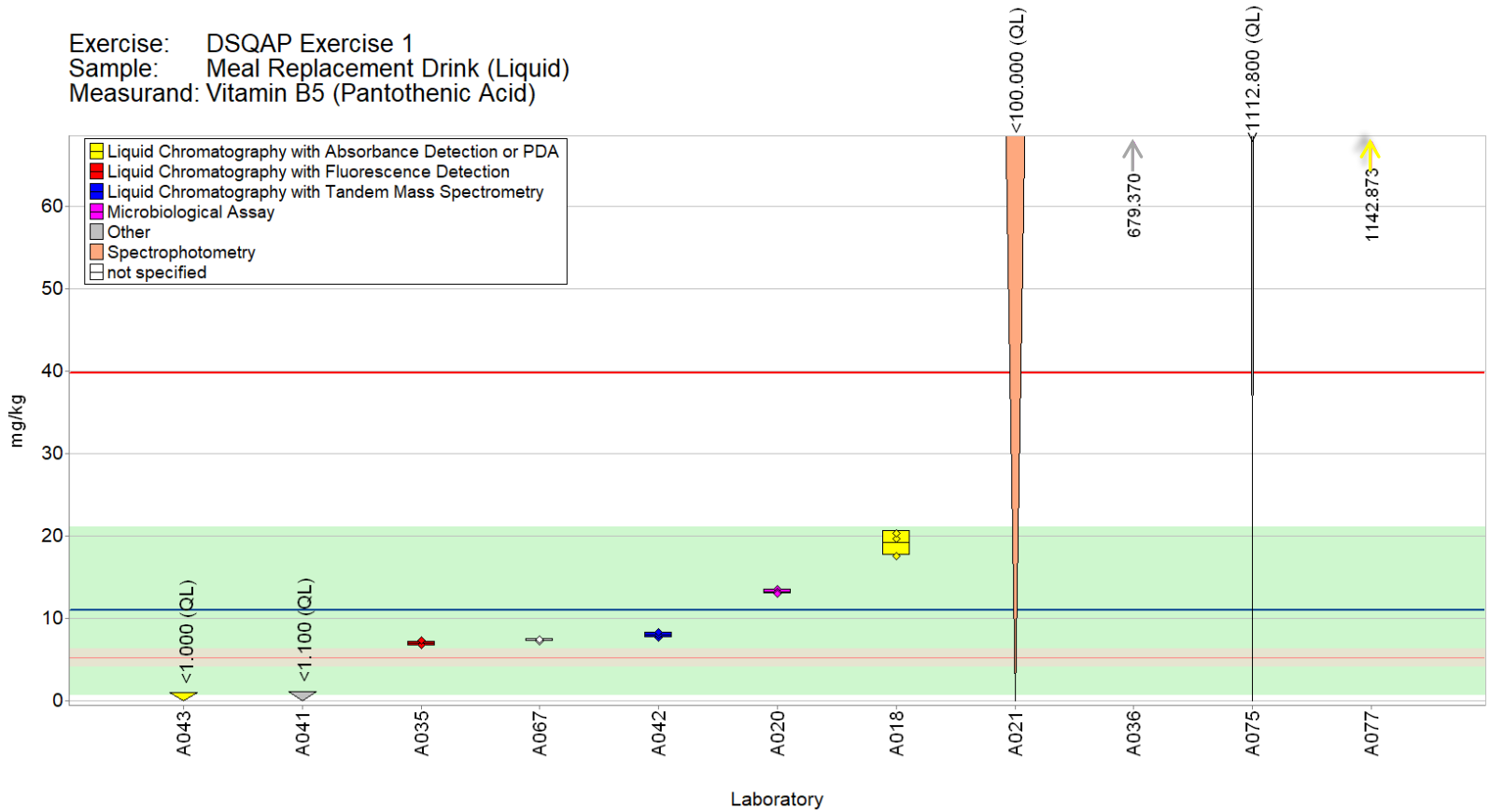

Fig. 8-10. Vitamin B_5 (pantothenic acid) in Meal Replacement Drink (Liquid) (data summary view – analytical method).

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 red solid line represents the upper bound of the consensus range of tolerance, calculated as the value above the consensus mean that results in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$, with the lower bound set to zero. The red shaded region (beige here due to overlap with the green consensus confidence interval) represents the NIST range of tolerance, which encompasses the target value bounded by its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$.

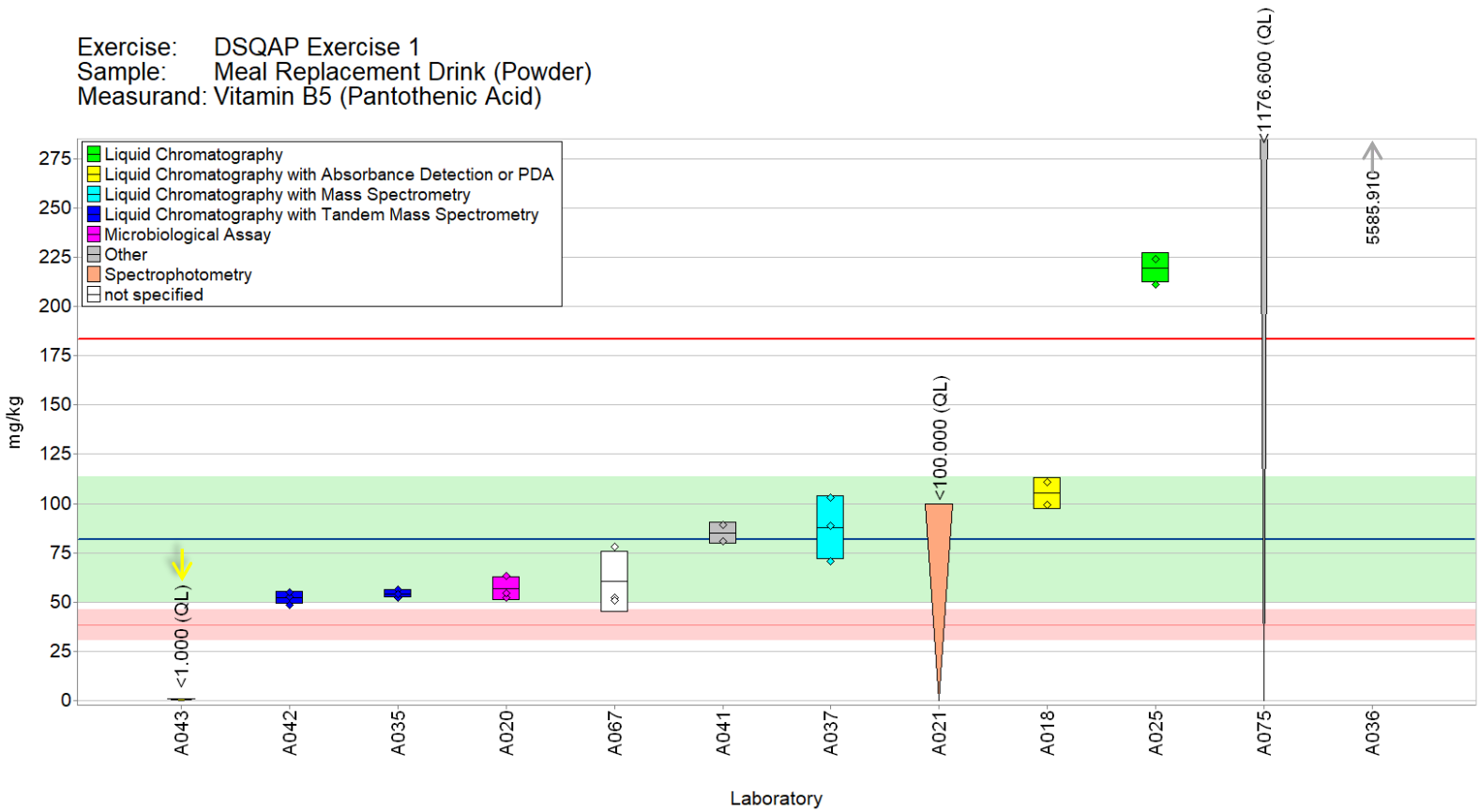

Fig. 8-11. Vitamin B_5 (pantothenic acid) in Meal Replacement Drink (Powder) (data summary view – analytical method).

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 red solid line represents the upper bound of the consensus range of tolerance, calculated as the value above the consensus mean that results in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$, with the lower bound set to zero. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$.

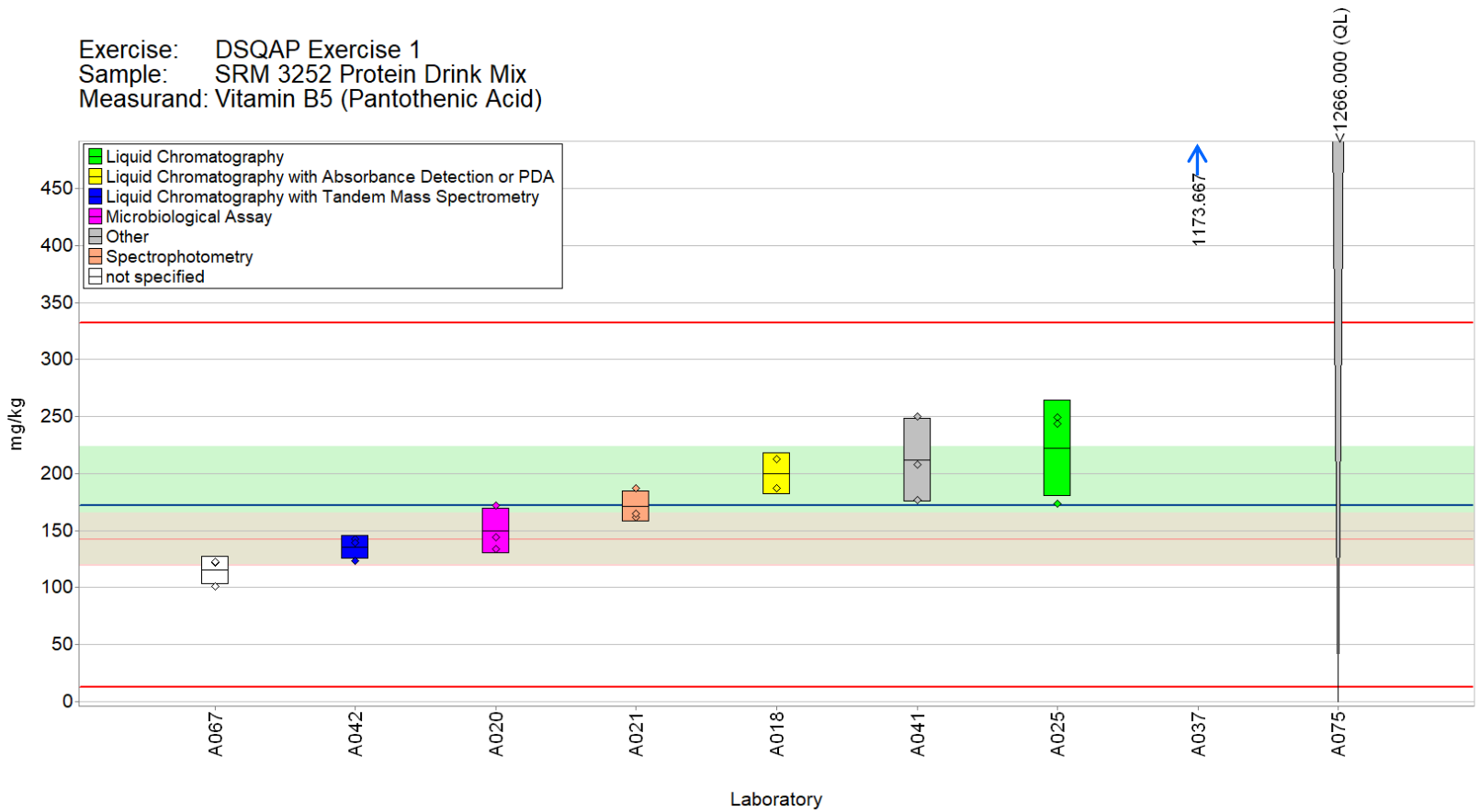

Fig. 8-12. Vitamin B_5 (pantothenic acid) in SRM 3252 Protein Drink Mix (data summary view – analytical method).

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 red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

8.4.5. Pyridoxine (B_6)

For the determination of pyridoxine, 2 of the 5 laboratories reporting quantitative values for the meal replacement drink (liquid) demonstrated repeatability within the published recommendation of at or below 5 % [61]. The average within-laboratory RSD for pyridoxine across all laboratories in the meal replacement drink (liquid) was 5.9 % (**Table 8-10**). For the meal replacement drink (powder), 2 of the 6 laboratories reporting quantitative values for pyridoxine demonstrated repeatability within the published recommendation and the average across all laboratories was 13 %. For SRM 3252, 0 of the 5 laboratories reporting quantitative values for pyridoxine demonstrated repeatability within the published recommendation and the average across all laboratories was 6.4 %. Laboratories with repeatability greater than 5 % should identify and minimize sources of variability in their in-house methods. The between-laboratory variability for the meal replacement drink (liquid) was slightly outside the published 10 % RSD recommendation for laboratories using the same method, while the reproducibilities observed for the meal replacement drink (powder) and SRM 3252 were significantly above 10 %.

Table 8-10. Summary of laboratory variabilities for pyridoxine in meal replacement drinks and SRM 3252.

	Pyridoxine (B_6)	
Material	Within-Laboratory Variability (% RSD)	Between-Laboratory Variability (% RSD)
Meal Replacement Drink (Liquid)	5.9 %	14 %
Meal Replacement Drink (Powder)	13.0 %	53 %
SRM 3252	6.4 %	79 %

Participants reporting quantitative values were in close agreement with each other for the analysis of pyridoxine in the meal replacement drink (liquid). The 95 % confidence interval for the consensus mean was above the NIST target value (product label) with the lower limit of the consensus range of tolerance overlapping with the NIST range of tolerance (**Fig. 8-13**). The between-laboratory RSD for the meal replacement drink (powder) (**Fig. 8-14**) and SRM 3252 (**Fig. 8-15**) were high compared to industry standards.

Although no trends were observed, participants should be cognizant of their method capabilities to measure pyridoxine and/or total B_6 which includes several vitamers and take appropriate measures to minimize pyridoxine degradation. SRM 3252 contains pyridoxine and pyridoxamine; however, there are no indications that participants measured total B_6. Additionally, pyridoxine is typically calibrated using pyridoxine HCl which is often the form added to formulations and is present in both commercial meal replacement materials. Participants calibrating with the pyridoxine HCl should ensure molecular weight differences are accounted for when calculating and reporting mass fractions for pyridoxine.

Three to four laboratories reported that pyridoxine values in these samples were below their method LOQ. The levels in these samples were significantly higher than the LOQ (0.0125 mg/kg) established in method performance requirements [61]. Participants with LOQs above this recommendation should consider additional method development to achieve lower LOQs.

Additional tables and figures for the analysis of pyridoxine can be found in **Appendix F**.

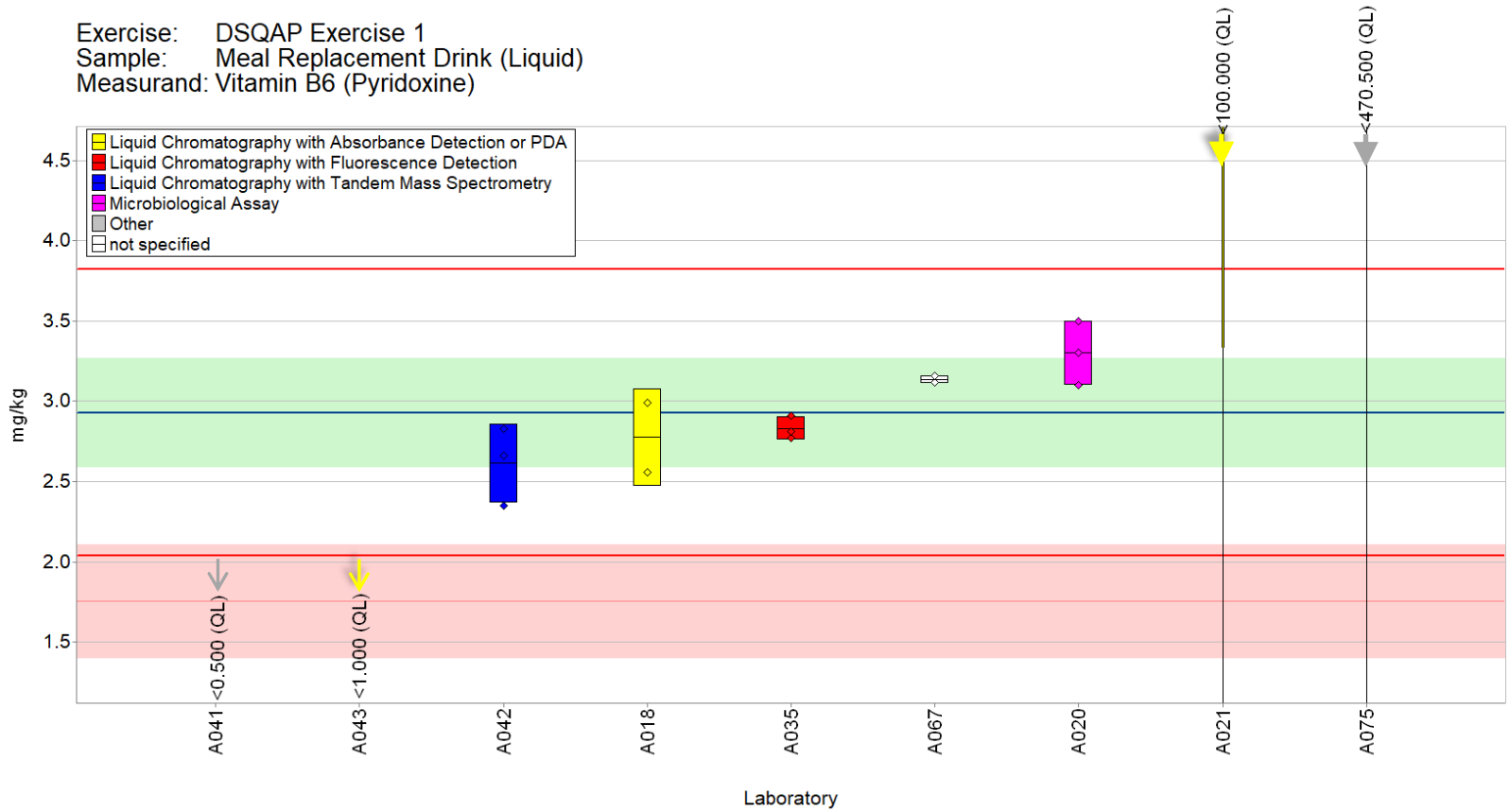

Fig. 8-13. Vitamin B_6 (pyridoxine) in Meal Replacement Drink (Liquid) (data summary view – analytical method).

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 red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$.

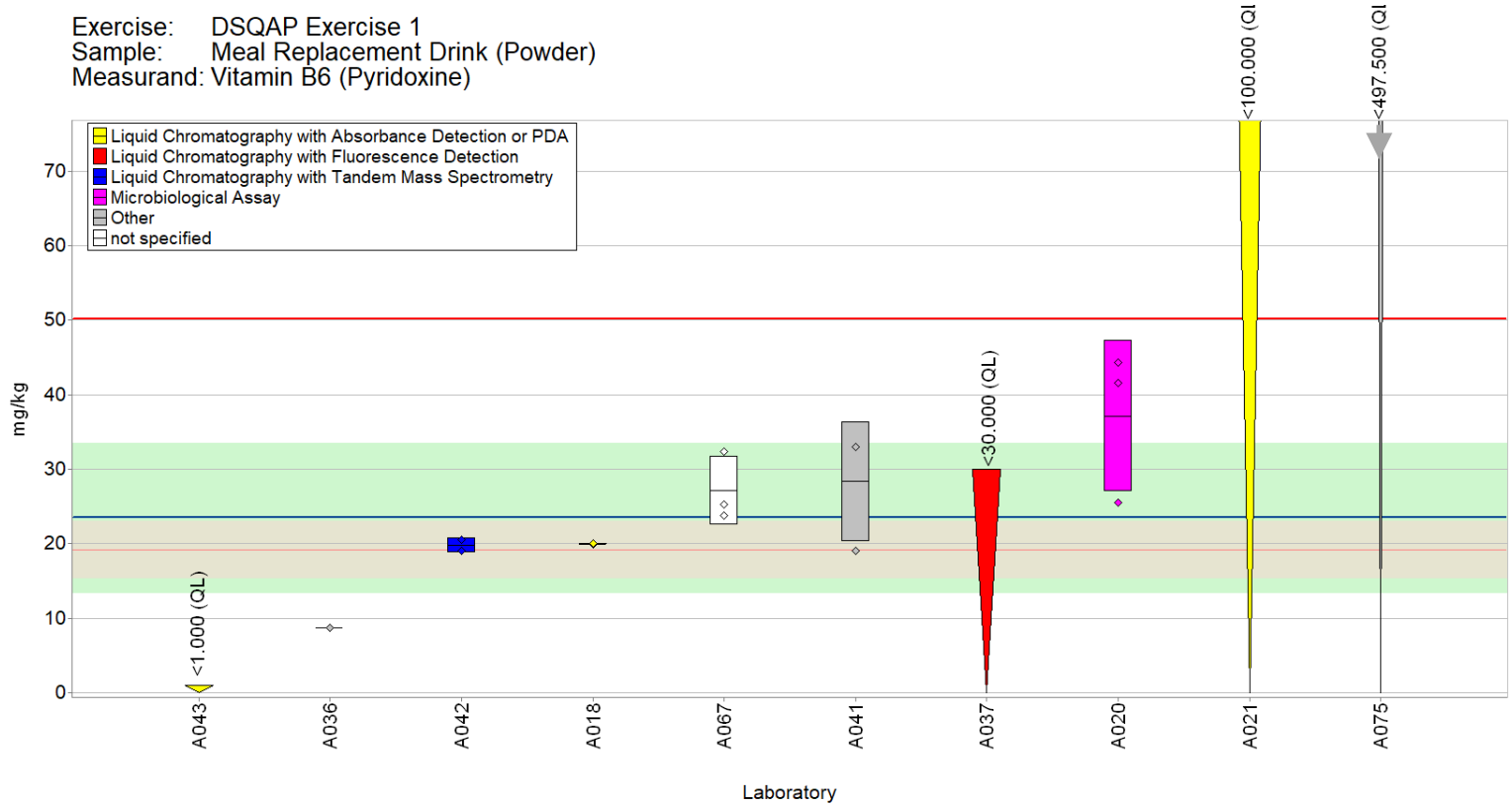

Fig. 8-14. Vitamin B_6 (pyridoxine) in Meal Replacement Drink (Powder) (data summary view – analytical method).

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 red solid line represents the upper bound of the consensus range of tolerance, calculated as the value above the consensus mean that results in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$, with the lower bound set to zero. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

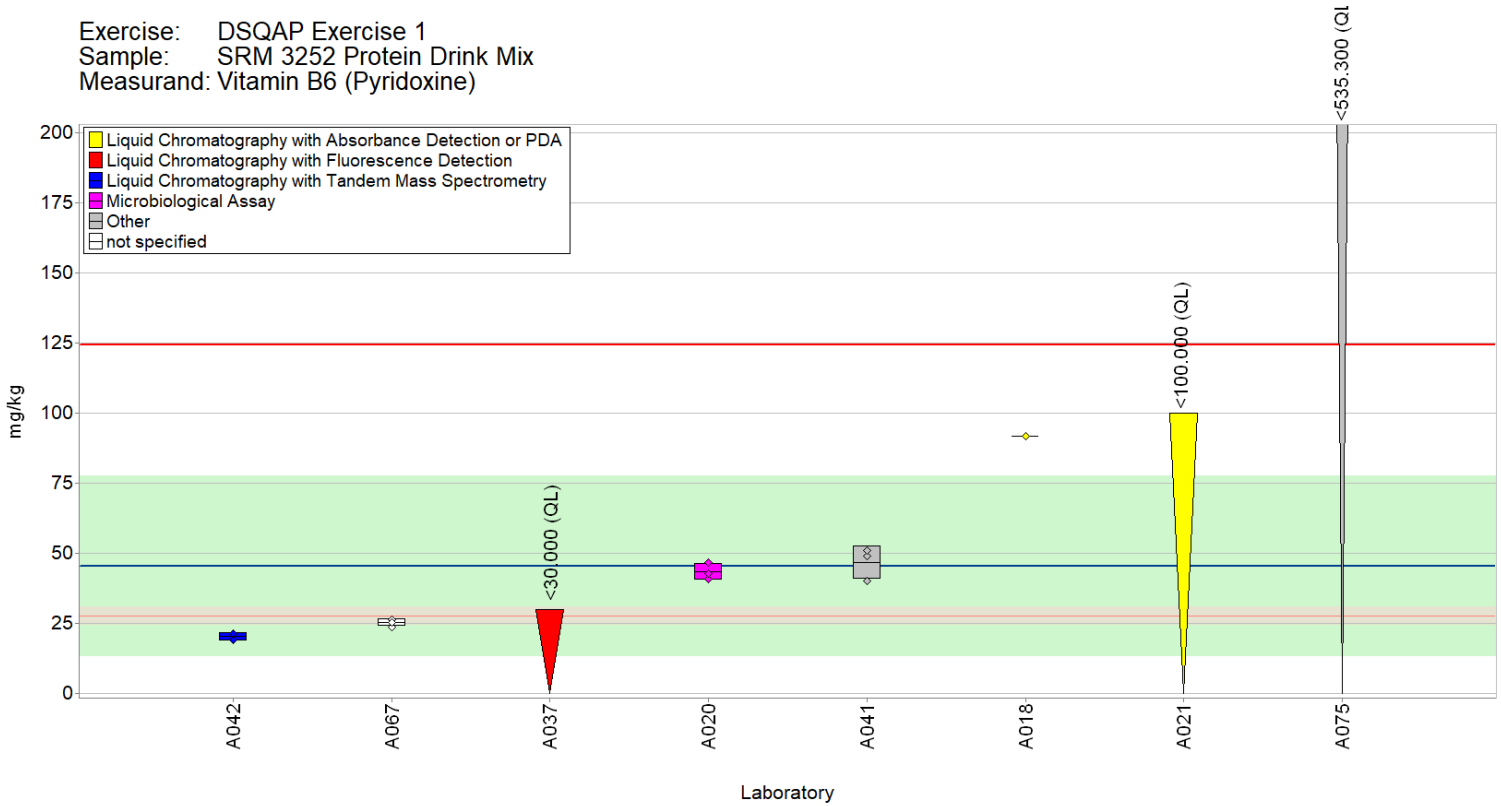

Fig. 8-15. Vitamin B_6 (pyridoxine) in SRM 3252 Protein Drink Mix (data summary view – analytical method).

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 red solid line represents the upper bound of the consensus range of tolerance, calculated as the value above the consensus mean that results in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$, with the lower bound set to zero. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

8.4.6. Biotin (B_7)

Of the 6 laboratories reporting quantitative results for biotin in the commercial meal replacement drinks, 6 laboratories for the liquid sample and 0 laboratories for the powder sample reported within-laboratory variabilities within the published requirement of 8 % [62]. The average within-laboratory RSD across all laboratories for the liquid sample (4.7 %, **Table 8-11**) was also within the published requirement. For SRM 3252, 2 of the 7 laboratories reporting quantitative results for biotin reported repeatabilities within the published requirement, and the average within-laboratory RSDs for the meal replacement drink (powder) and SRM 3252 were both around 10 %. The between-laboratory variabilities for the two commercial meal replacement products are outside of the established performance requirement of 16 % while the reproducibility for SRM 3252 meets published guidelines.

Table 8-11. Summary of laboratory variabilities for biotin in meal replacement drinks and SRM 3252.

	Biotin (B_7)	
Material	Within-Laboratory Variability (% RSD)	Between-Laboratory Variability (% RSD)
Meal Replacement Drink (Liquid)	4.7 %	67 %
Meal Replacement Drink (Powder)	10.6 %	25 %
SRM 3252	10.2 %	15 %

The consensus range of tolerance was within the NIST range of tolerance for the meal replacement (liquid), but the 95 % confidence interval for the consensus mean was below the NIST range of tolerance (**Fig. 8-16**). Although the repeatability for each laboratory is within the recommended 8 % RSD, the large between-laboratory variability indicates potential challenges and inconsistencies with sample preparation within the community for biotin measurements in ready-to-drink products. Low levels of biotin can be challenging to accurately quantify due to interferences from matrix components present in higher concentrations. Sample preparation for liquid matrices typically involves dilution as a primary technique which can aid in eliminating matrix interferences. Participants should dilute samples to levels that are appropriate for their LOQs and calibration to ensure minimal interferences and detector overload.

Overall, participants were in better agreement with each other for biotin in both powder materials. The 95 % confidence interval for the consensus mean for the meal replacement drink (powder) overlapped with the NIST target value (**Fig. 8-17**). The reproducibility for this material is skewed high due to the small number of laboratories reporting quantitative results and laboratory A043 reporting values significantly above the consensus range of tolerance. This laboratory reported high values for biotin for all three materials indicating potential calibration issues and/or interferences, but as the only laboratory that reported using LC-MS/MS, a specific analytical method bias cannot be determined. The 95 % confidence interval for the consensus mean for SRM 3252 overlapped on the lower end of the NIST range of tolerance (**Fig. 8-18**). While no trends are observed for analytical techniques, the lower reported values may be due to calibration issues. Calibration of biotin can be challenging when preparing stock solutions and working calibration solutions at low concentrations. Participants should consider preparing calibrants at higher concentrations and diluting to reach levels within the materials.

Two laboratories reported values for biotin below their LOQ for the two meal replacement drink materials, but were able to report biotin mass fractions within the target range of tolerance for SRM 3252 since this material has significantly higher levels of biotin.

Additional tables and figures for the analysis of biotin can be found in **Appendix F**.

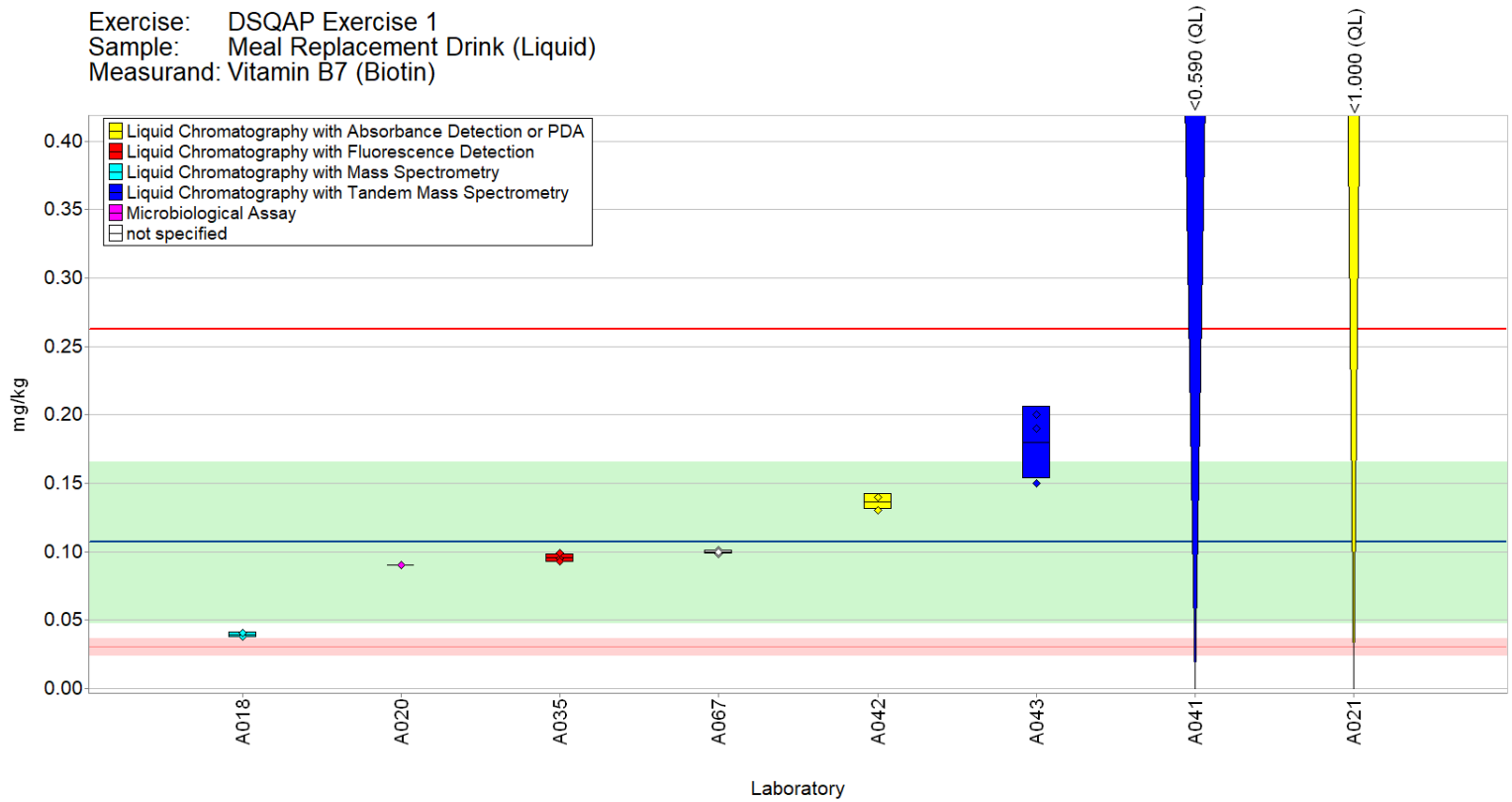

Fig. 8-16. Vitamin B_7 (biotin) in Meal Replacement Drink (Liquid) (data summary view – analytical method).

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 red solid line represents the upper bound of the consensus range of tolerance, calculated as the value above the consensus mean that results in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$, with the lower bound set to zero. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$.

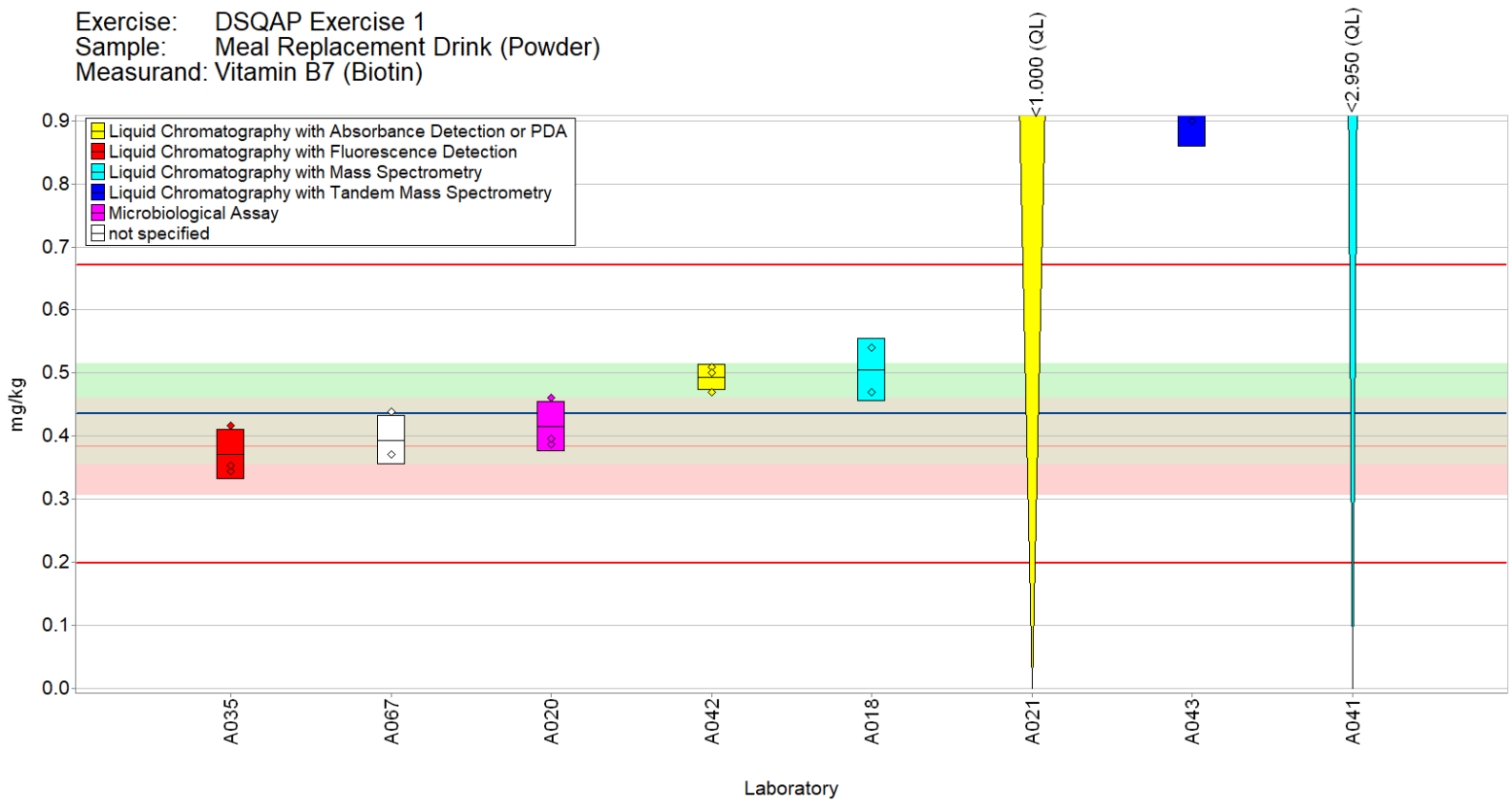

Fig. 8-17. Vitamin B_7 (biotin) in Meal Replacement Drink (Powder) (data summary view – analytical method).

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 red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

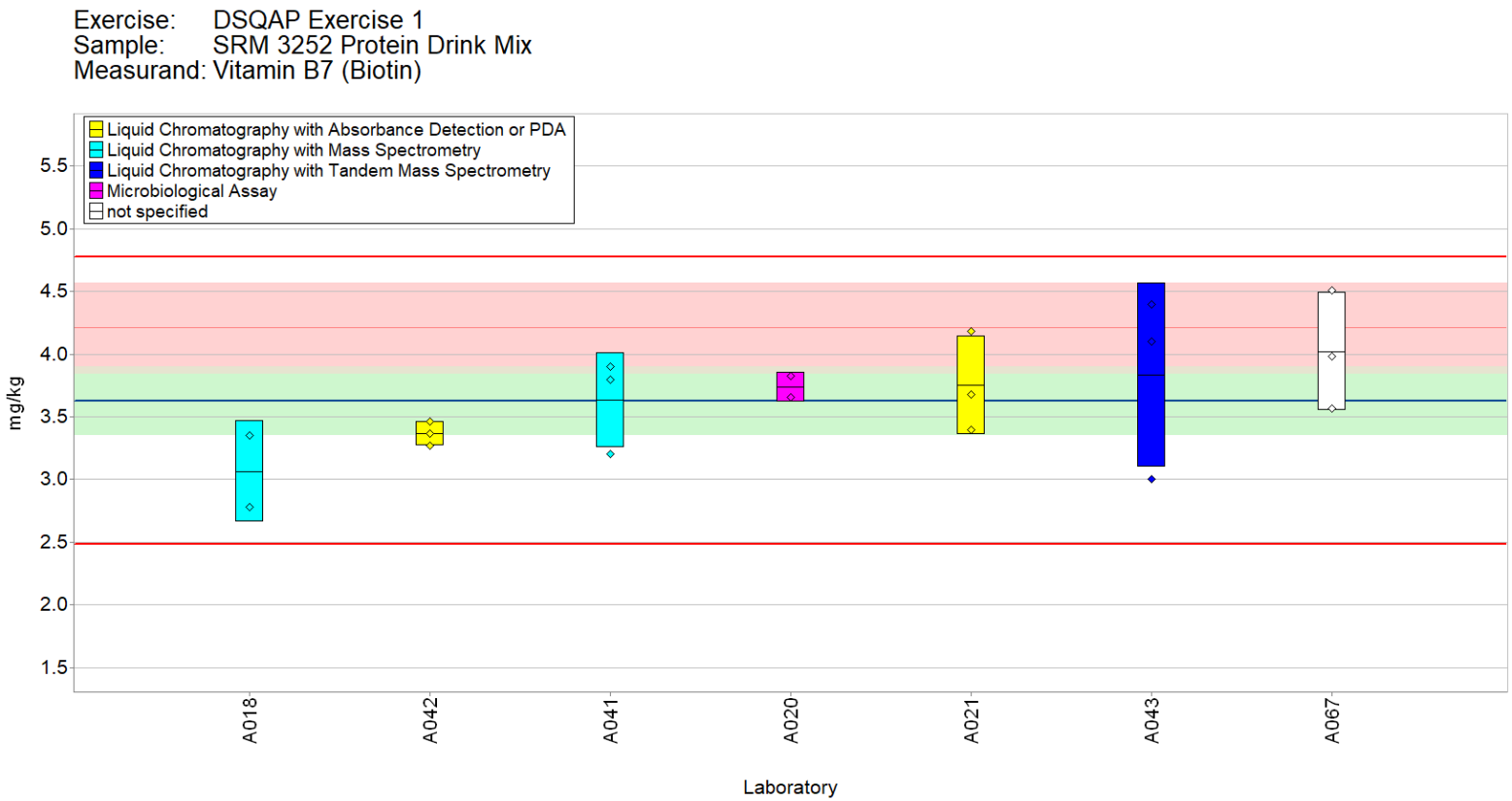

Fig. 8-18. Vitamin B_7 (biotin) in SRM 3253 Protein Drink Mix (data summary view – analytical method).

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 red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

8.4.7. Folic Acid (B_9)

The average within-laboratory variability for folic acid was less than 7.5 % in all three samples (**Table 8-12**) with only 2 laboratories above 10 %, which indicates that most participants' in-house methods achieve repeatability consistent with the published requirement of at or below 10 % [63]. The between-laboratory variabilities ranged from 20 % and 24 % for the meal replacement drinks to 96 % for SRM 3252. The performance for the meal replacement drinks was reasonable with respect to the published recommendation for B_9 in dietary supplements of 15 % RSD for laboratories using the same method, given that participants in this study were using different methods.

Table 8-12. Summary of laboratory variabilities for folic acid in meal replacement drinks and SRM 3252.

	Folic Acid (B_9)	
Material	Within-Laboratory Variability (% RSD)	Between-Laboratory Variability (% RSD)
Meal Replacement Drink (Liquid)	5.9 %	24 %
Meal Replacement Drink (Powder)	6.7 %	20 %
SRM 3252	7.5 %	96 %

The lower limit of the consensus range of tolerance for folic acid barely overlapped with the NIST range of tolerance for the meal replacement drink (liquid) (**Fig. 8-19**). The 95 % confidence interval for the consensus mean is above the NIST range of tolerance which is consistent with other B vitamins in the meal replacement drink (liquid). As mentioned previously, the higher vitamin levels in the liquid matrix compared to the target value (product label) could be a result of manufacturers adding over label claims to account for stability throughout shelf life.

The 95 % confidence interval for the consensus mean for the meal replacement drink (powder) (**Fig. 8-20**) and SRM 3252 (**Fig. 8-21**) both overlapped with the NIST range of tolerance for each material. Laboratory A018 reported mass fractions for folic acid significantly above the consensus range of tolerance for all three materials, which because of the small data set is a main factor for the high between-laboratory RSDs. Laboratory A057 was consistently low compared to other laboratories for all three materials. These laboratories should evaluate their calibration techniques for measuring folic acid at low levels by ensuring calibration levels are representative of what is in the sample.

Two laboratories reported values for folic acid that were below their LC-Abs LOQ for all three materials. AOAC SMPR 2011.006 details LOQ requirements of 0.625 µg/kg for folate levels between 0.005 – 3 mg/kg [63].

Additional tables and figures for the analysis of folic acid can be found in **Appendix F**.

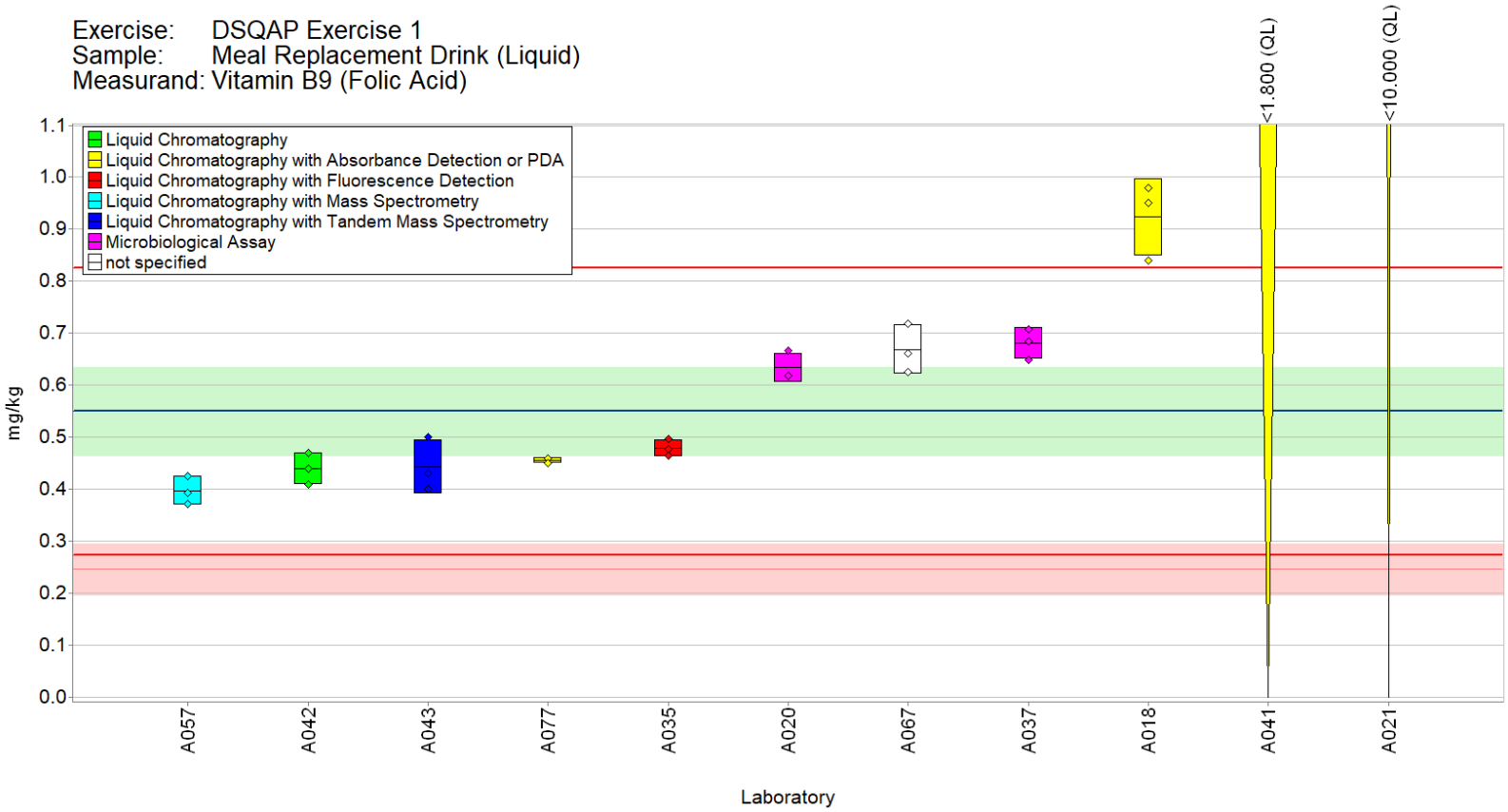

Fig. 8-19. Vitamin B_9 (folic acid) in Meal Replacement Drink (Liquid) (data summary view – analytical method).

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 red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$.

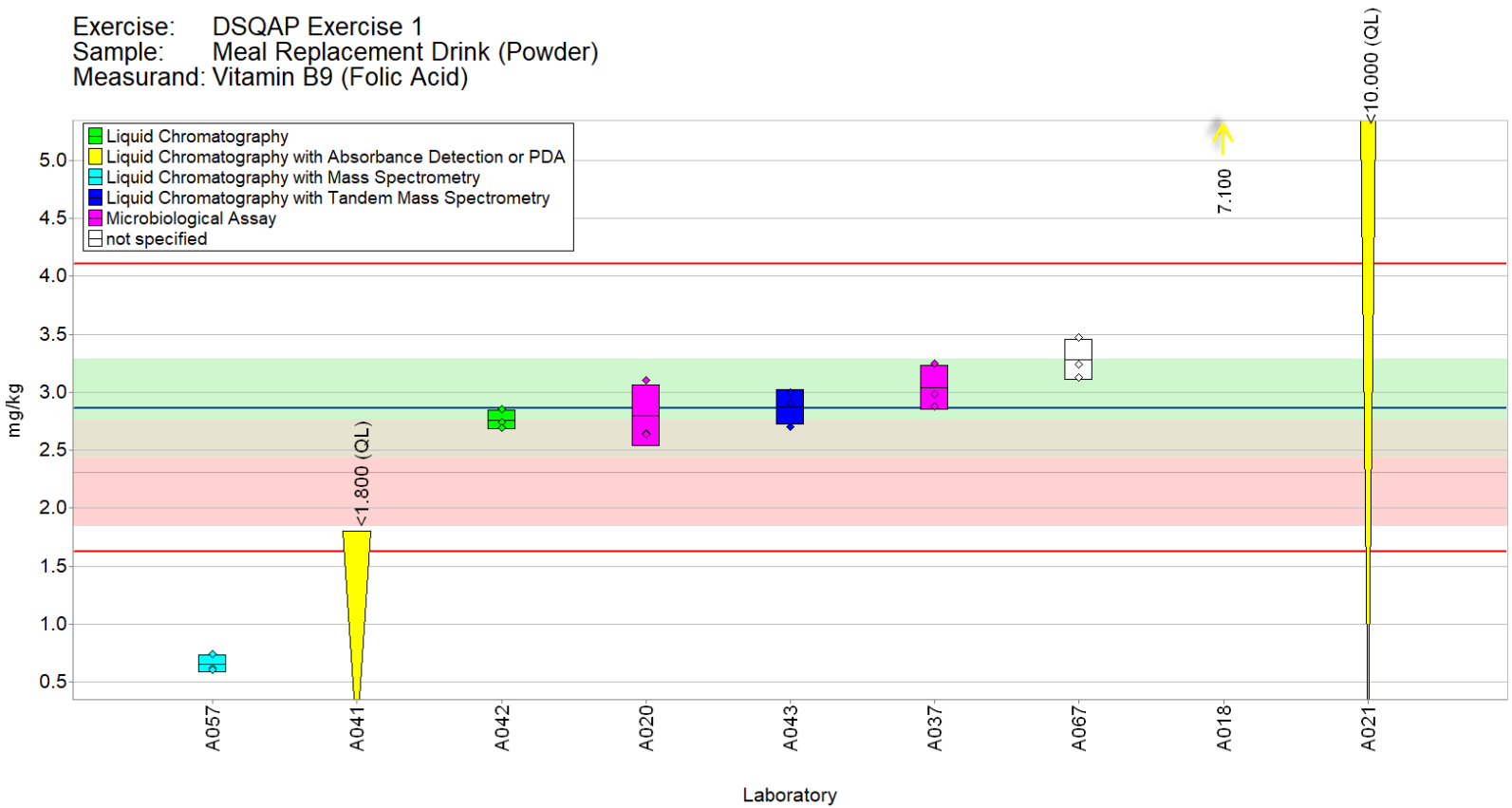

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Fig. 8-20. Vitamin B_9 (folic acid) in Meal Replacement Drink (Powder) (data summary view – analytical method).

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 red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

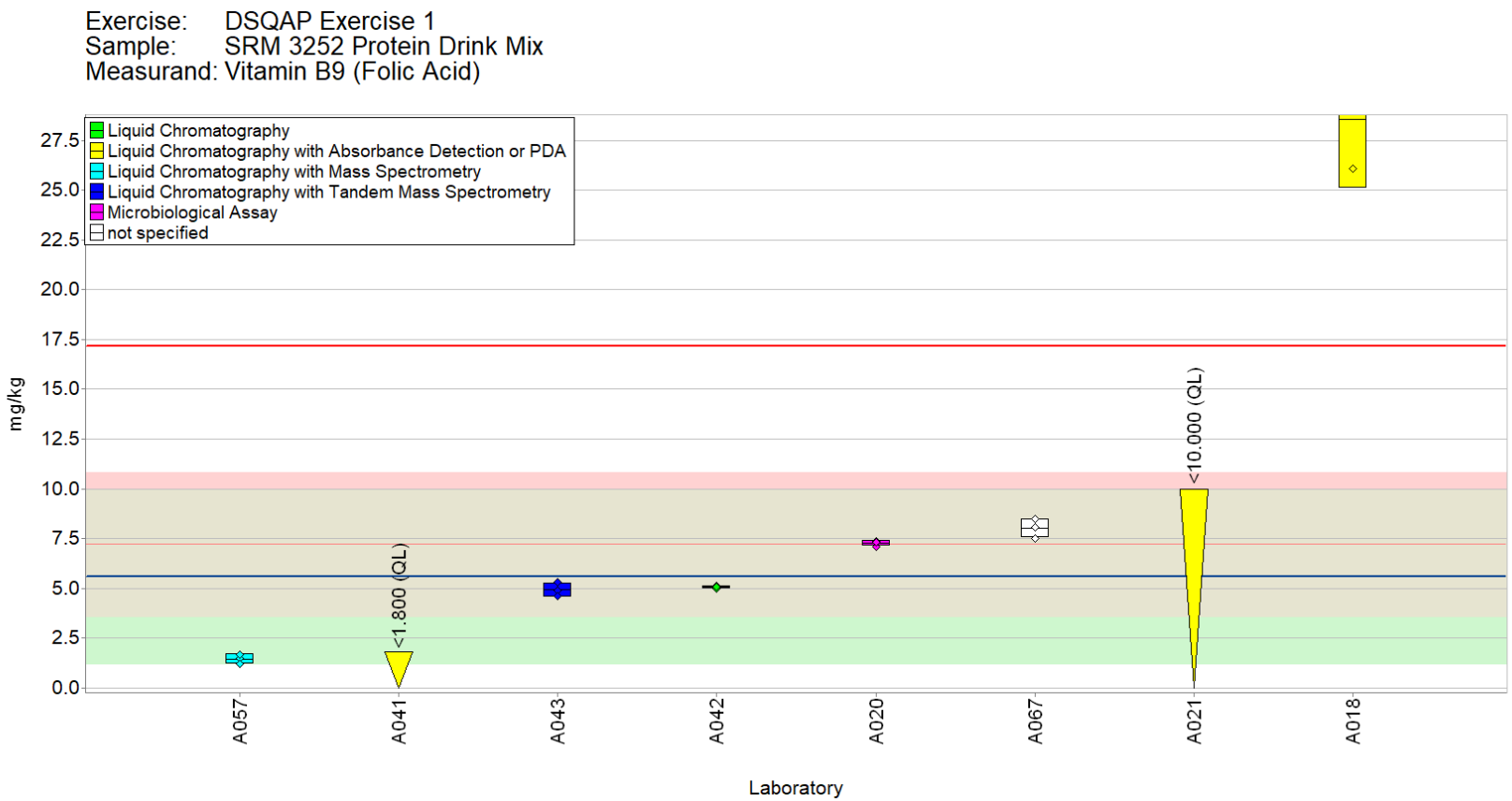

Fig. 8-21. Vitamin B_9 (folic acid) in SRM 3252 Protein Drink Mix (data summary view – analytical method).

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 red solid line represents the upper bound of the consensus range of tolerance, calculated as the value above the consensus mean that results in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$ with the lower bound set zero. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\text{NIST}}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

8.4.8. Cobalamin (B_{12})

The small number of quantitative results reported for vitamin B_{12} (only 3 to 5 laboratories per sample) significantly limits the meaning of statistical figures of merit for this study. The average within-laboratory variabilities for vitamin B_{12} in the meal replacement drink (powder) and SRM 3252 were within the recommended 7 % RSD for the 4 and 3 laboratories reporting quantitative results, respectively, as shown in **Table 8-13** [64]. The average within-laboratory variability for the meal replacement drink (liquid) was outside of the established recommendation due to the small number of laboratories reporting quantitative results (5) and one laboratory with extremely large repeatability (127 %). The within-laboratory variabilities for the remaining 3 of the 4 laboratories reporting quantitative results for the meal replacement drink (liquid) were within the recommended 7 % RSD. The between-laboratory variabilities were outside of the recommended 11 % RSD for all three materials.

Table 8-13. Summary of laboratory variabilities for cobalamin in meal replacement drinks and SRM 3252.

	Cobalamin (B_{12})	
Material	Within-Laboratory Variability (% RSD)	Between-Laboratory Variability (% RSD)
Meal Replacement Drink (Liquid)	25.6 %	75 %
Meal Replacement Drink (Powder)	5.1 %	28 %
SRM 3252	4.4 %	18 %

The 95 % confidence interval for the consensus mean for the meal replacement drink (liquid) overlaps with the low end of the NIST range of tolerance (**Fig. 8-22**). The reported values being below the NIST range of tolerance for the liquid matrix does not follow the typical trend observed with other B vitamins, perhaps due to sample preparation challenges for low levels of vitamin B_{12} in this liquid matrix. The extraction efficiency of B_{12} must be evaluated to ensure complete extraction of the analyte from the sample matrix. As mentioned above, the high repeatability and reproducibility for B_{12} in the meal replacement drink (liquid) is skewed due to the values reported by laboratory A020. This laboratory likely reported one of their triplicate measurements in error by omitting a zero, resulting in an order of magnitude difference between this value and the other two reported values, which affected this laboratory's mean and standard deviation, as well as the consensus mean and variability due to the small data set.

The 95 % confidence interval for the consensus mean for the meal replacement drink (powder) overlapped with the NIST target value (product label) which indicates laboratories performed well with this material (**Fig. 8-23**). The consensus mean for SRM 3252 fell within the lower end of the NIST range of tolerance (**Fig. 8-24**); however, only three laboratories reported results for vitamin B_{12} in SRM 3252 without LOQ limitations.

Three participants reported values for vitamin B_{12} that were below their LOQ for all three materials. According to the AOAC SMPR 2011.005 for vitamin B_{12} in similar matrices, the recommended LOQ is at or below 0.01 mg/kg [64]. The meal replacement drink (liquid) has a vitamin B_{12} target value below the recommended LOQ while the meal replacement drink (powder) and SRM 3252 are both above. Participants indicating LOQ limits for all three materials should consider further optimization of their analytical techniques to ensure accurate measurements for vitamin B_{12} at lower concentrations.

Additional tables and figures for the analysis of cobalamin can be found in **Appendix F**.

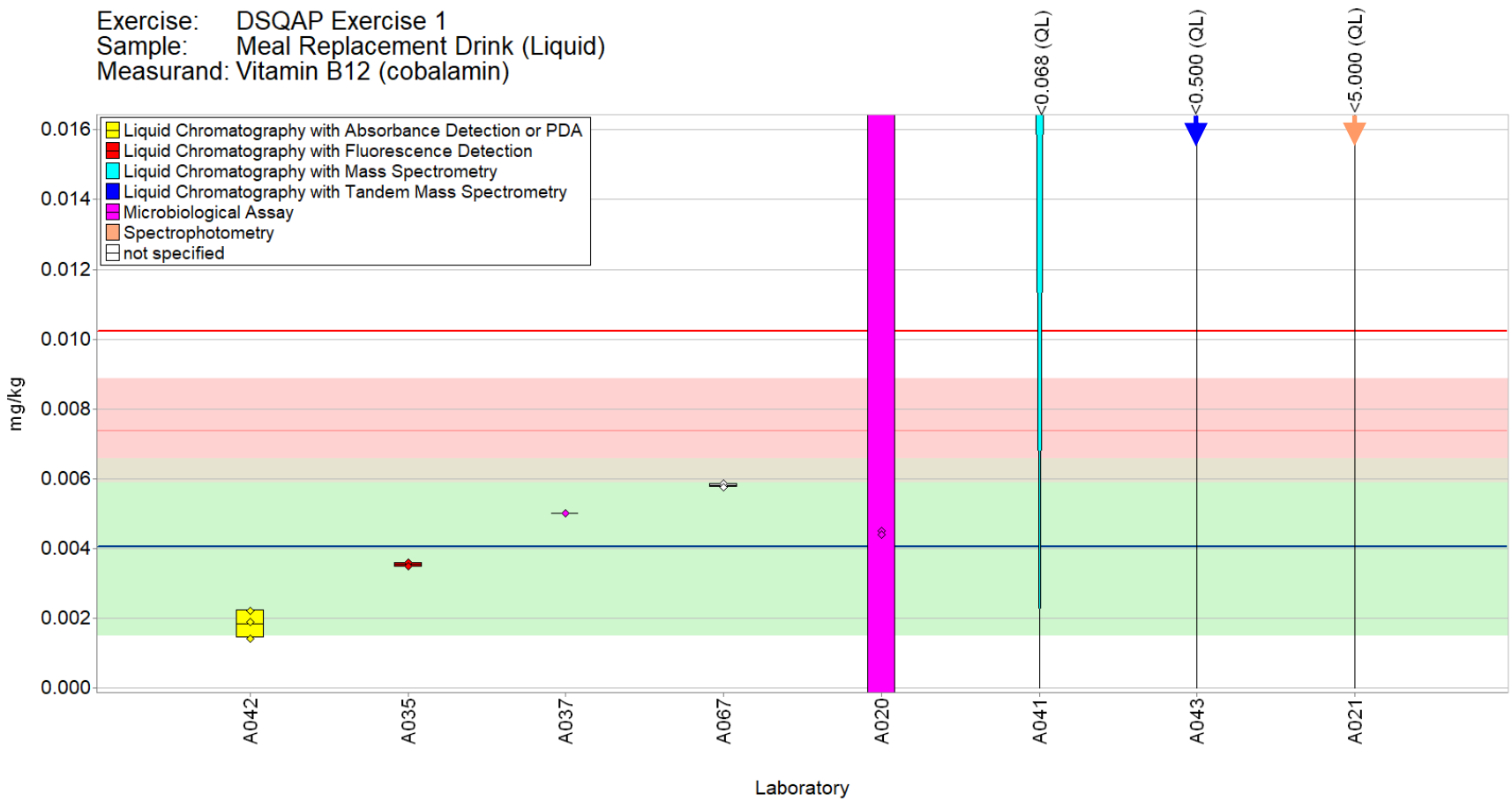

Fig. 8-22. Vitamin B_{12} (cobalamin) in Meal Replacement Drink (Liquid) (data summary view – analytical method).

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 red solid line represents the upper bound of the consensus range of tolerance, calculated as the value above the consensus mean that results in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$, with the lower bound set to zero. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

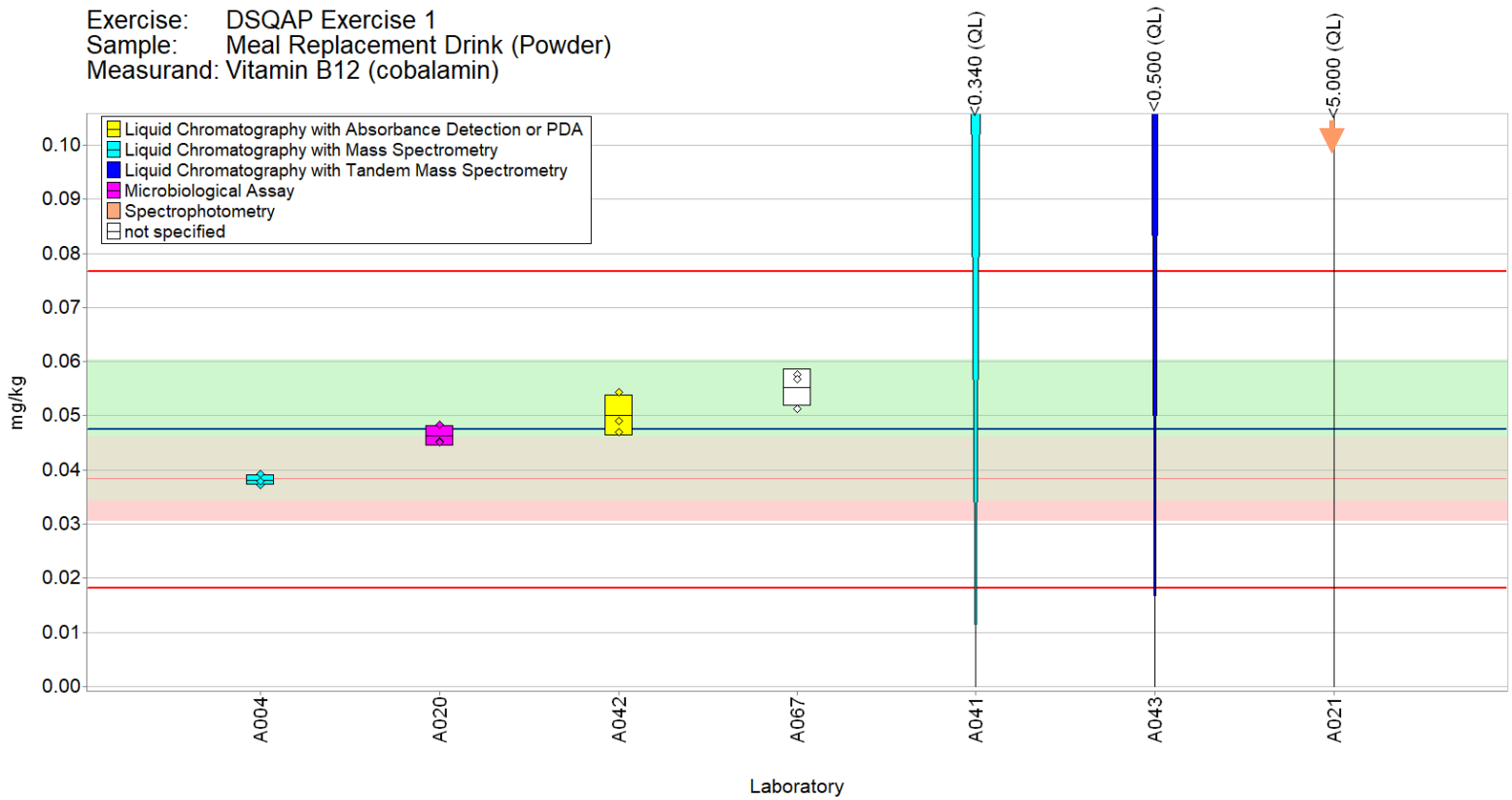

Fig. 8-23. Vitamin B_{12} (cobalamin) in Meal Replacement Drink (Powder) (data summary view – analytical method).

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 red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

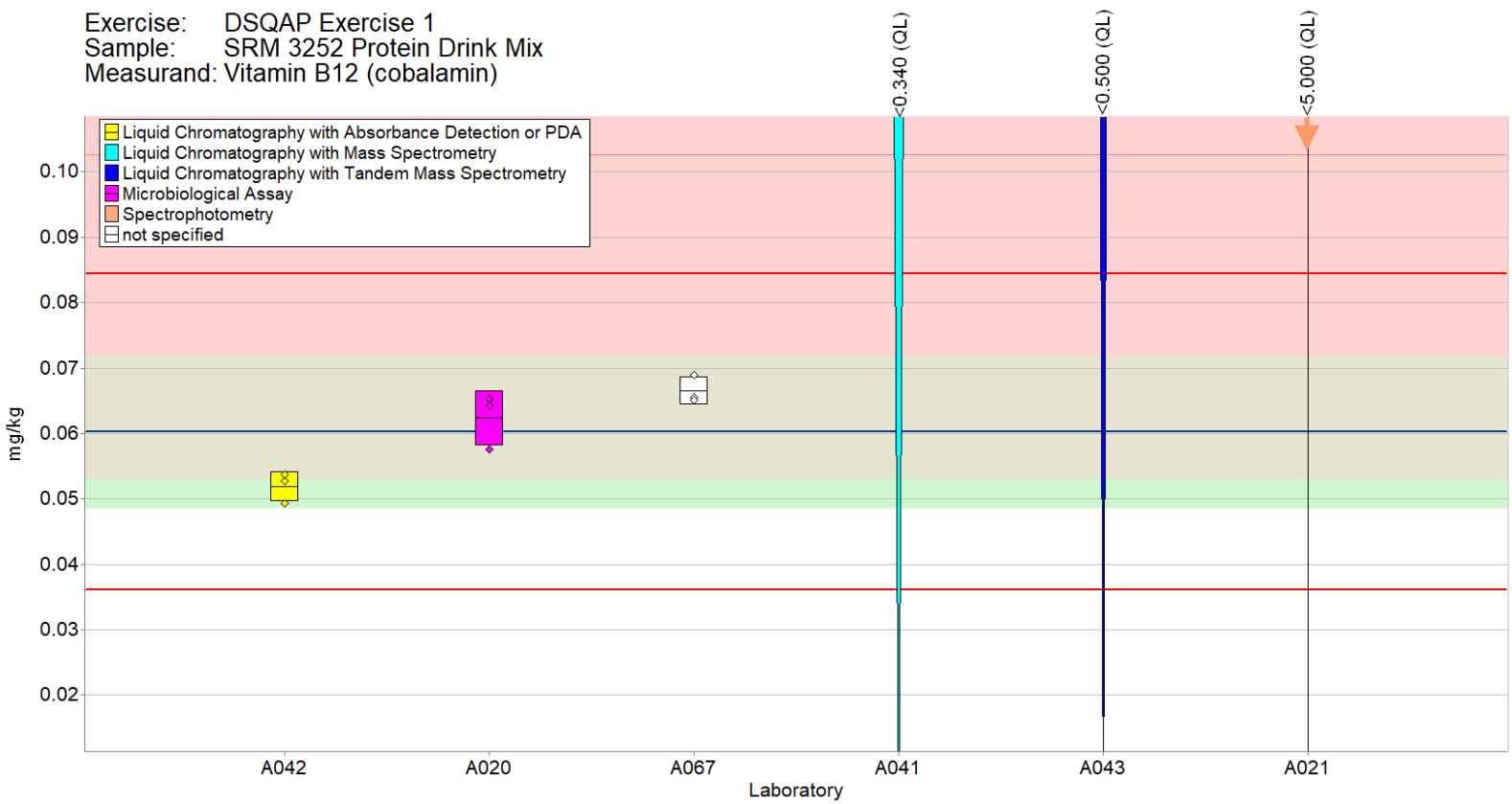

Fig. 8-24. Vitamin B_{12} (cobalamin) in SRM 3252 Protein Drink Mix (data summary view – analytical method).

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 red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$. The red shaded region represents the NIST range of tolerance, which encompasses the target value bounded by its uncertainty (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The shaded beige region represents the overlapping of the 95 % confidence interval for the consensus mean (green region) and the NIST range of tolerance (red region).

9. Botanicals II (Withanolides)

9.1. Executive Summary

Fourteen to sixteen laboratories enrolled in this study for withanolides in ashwagandha root and extract materials to identify analytical challenges associated with ashwagandha measurements within the community. Overall, the participants demonstrated excellent in-house precision in both materials with the average within-laboratory RSDs being below 6 % for the five targeted withanolides and 10 % for 12-deoxywithastromonolide. However, the between-laboratory RSDs were much higher ranging from 38 % to 70 % and 7 % to 47 % for the ashwagandha root powder and extract powder, respectively. The overall larger between-laboratory variability for the root powder material may be due to differences in extraction approaches and/or potential matrix interferences. The results of this study highlight the challenges associated with developing well-established analytical methods for measurands of interest in minimally processed botanical materials used for dietary supplement production as well as the need for ashwagandha reference materials to aid in the standardization of withanolide measurements within the dietary supplement community.

9.2. Study Background and Participation

Withanolides and withanosides (withanolide glycosides) are steroidal lactones found in the botanical ashwagandha (*Withania somnifera*), which is used in Ayurvedic medicine and is a popular dietary supplement ingredient due to the associated adaptogenic properties [65]. These marker compounds have been reported to be a major source of the botanical's bioactive properties; however, difficulties in developing accurate analytical methodologies for the extraction and detection of withanolides and withanosides can be attributed in part to a lack of authenticated certified reference materials for method development validation.

The goal of this study was to determine any community discrepancies and/or analytical challenges in the analysis of ashwagandha measurands of interest. This study also focused on evaluation of AOAC First Action Official Method 2015.17 and development of ashwagandha reference materials which will support the dietary supplement industry to substantiate product labels and/or health claims and to meet the requirements of regulatory agencies. Participants were asked to use their in-house analytical methods and/or AOAC First Action Official Method 2015.17 [66] to determine the mass fraction of withanolides in both materials.

Registration numbers for this study were dependent on the measurand of interest and ranged between 14 and 16 laboratories. The participation rates per measurand ranged from 43 % to 63 %. Some of the reported values were non-quantitative (zero or below LOQ) but are included in the participation and reporting statistics shown in **Table 9-1**.

Table 9-1. Enrollment and reporting statistics for withanolides in ashwagandha root powder and ashwagandha root powder extract.

Analyte	Number of Laboratories Requesting Samples	Number of Laboratories Reporting Results (Percent Participation)	
		Ashwagandha Root Powder	Ashwagandha Root Powder extract
12-deoxywithastromonolide	14	6 (43 %)	6 (43 %)
withaferin A	15	9 (60 %)	9 (60 %)
withanolide A	16	10 (63 %)	10 (63 %)
withanolide B	16	9 (56 %)	9 (56 %)
withanoside IV	15	7 (47 %)	7 (47 %)
withanoside V	15	7 (47 %)	7 (47 %)

9.3. Study Results and Technical Recommendations

9.3.1. Ashwagandha Root Powder

Participants were provided with three packets, each containing 5 g of ashwagandha (*Withania somnifera*) root powder. Participants were asked to store the material at controlled room temperature, 20 °C to 25 °C, and to prepare one sample and report one value from each packet provided. Before use, participants were instructed to mix the contents of the packets thoroughly, and to allow contents to settle for one minute prior to opening to minimize the loss of fine particles. Participants were instructed to use an appropriate sample size for their in-house analytical methods, or at least 2.5 g if using AOAC 2015.17, to determine the mass percent (% w/w) of select withanolides and withanosides. The approximate analyte levels were not reported to participants prior to the study and target values were not available at the time of this report.

All laboratories that reported sample preparation methods (44 %) indicated using solvent extraction for determination of all measurands of interest. Most laboratories indicated using LC-Abs as the analytical technique as seen in **Table 9-2**. The AOAC 2015.17 method is also based on LC-Abs. Laboratories reporting only LC should indicate the mode of detection to help identify potential sources of bias in their results.

Table 9-2. Summary of analytical methods for determination of withanolides in ashwagandha root powder.

	Analytical Method Percent Reporting Averaged for both Samples				
Analyte	AOAC 2015.17	HPTLC	LC	LC-Abs	Other/None Reported
12-deoxywithastromonolide	14 %	-	29 %	57 %	-
withaferin A	10 %	-	20 %	50 %	20 %
withanolide A	9 %	9 %	18 %	46 %	18 %
withanolide B	10 %	10 %	20 %	50 %	10 %
withanoside IV	12.5 %	-	25 %	50 %	12.5 %
withanoside V	12.5 %	-	25 %	50 %	12.5 %

The AOAC SMPR 2015.007 recommends repeatability and reproducibility for withanolides in ashwagandha to be at or below 7 % and 10 %, respectively [67]. The excellent average within-laboratory RSDs (at or below 10 %) for each measurand in the ashwagandha root powder material indicates each participants' in-house methods achieves successful repeatability as seen in **Table 9-3**. 12-deoxywithastromonolide had the highest within-laboratory variability at 10 % with only two laboratories reporting repeatabilities above 10 %. Participants utilizing the AOAC Official First Action Method 2015.17 reported repeatabilities consistent with what is published in the method [66]. When comparing between-laboratory variability, RSDs range from 38 % (withanoside IV) to 70 % (12-deoxywithastromonolide) and are also shown in **Fig. 9-1** and **Fig. 9-2**, respectively. The reproducibility is significantly higher than the recommended 10 %. Figures for the remaining measurands and individual laboratory results for ashwagandha root powder can be found in **Appendix G**.

Table 9-3. Summary of laboratory variabilities for withanolides in ashwagandha root powder.

	Ashwagandha Root Powder	
Analyte	Within-Laboratory Variability (% RSD)	Between-Laboratory Variability (% RSD)
12-deoxywithastromonolide	10.0 %	70 %
withaferin A	2.1 %	41 %
withanolide A	5.9 %	58 %
withanolide B	3.3 %	58 %
withanoside IV	2.6 %	38 %
withanoside V	4.0 %	40 %

Larger between-laboratory variability indicates that the community is not in complete agreement for the analysis of these measurands. Since most laboratories reported using LC-Abs, no trends were observed for analytical technique. The large between-laboratory variability may be due to inadequate sample preparation techniques used for the specific phytochemicals in ashwagandha root powder. Extractions from raw materials can be difficult due to various analytical challenges associated with the nature of botanical matrices. With only 44 % of participants reporting sample

preparation techniques (solvent extraction), technical recommendations are limited. Sample preparation techniques must be able to fully extract analytes from the sample matrix, while also being mindful of analyte degradation and/or conversion. Any extraction procedure should be optimized to determine the most effective extraction solvent and to ensure exhaustive extraction of the analyte from the matrix. Phytochemicals range in solubility and universal solvents might not be appropriate depending on the material. The optimum number of extraction cycles must be determined by sequential re-extraction of the sample matrix until no further increase in yield is observed. Sequential extractions may be needed if the extraction solvent becomes saturated during the first (or only) extraction cycle. AOAC Official Method 2015.17 utilizes three to four sequential extractions of 100 mL of methanol.

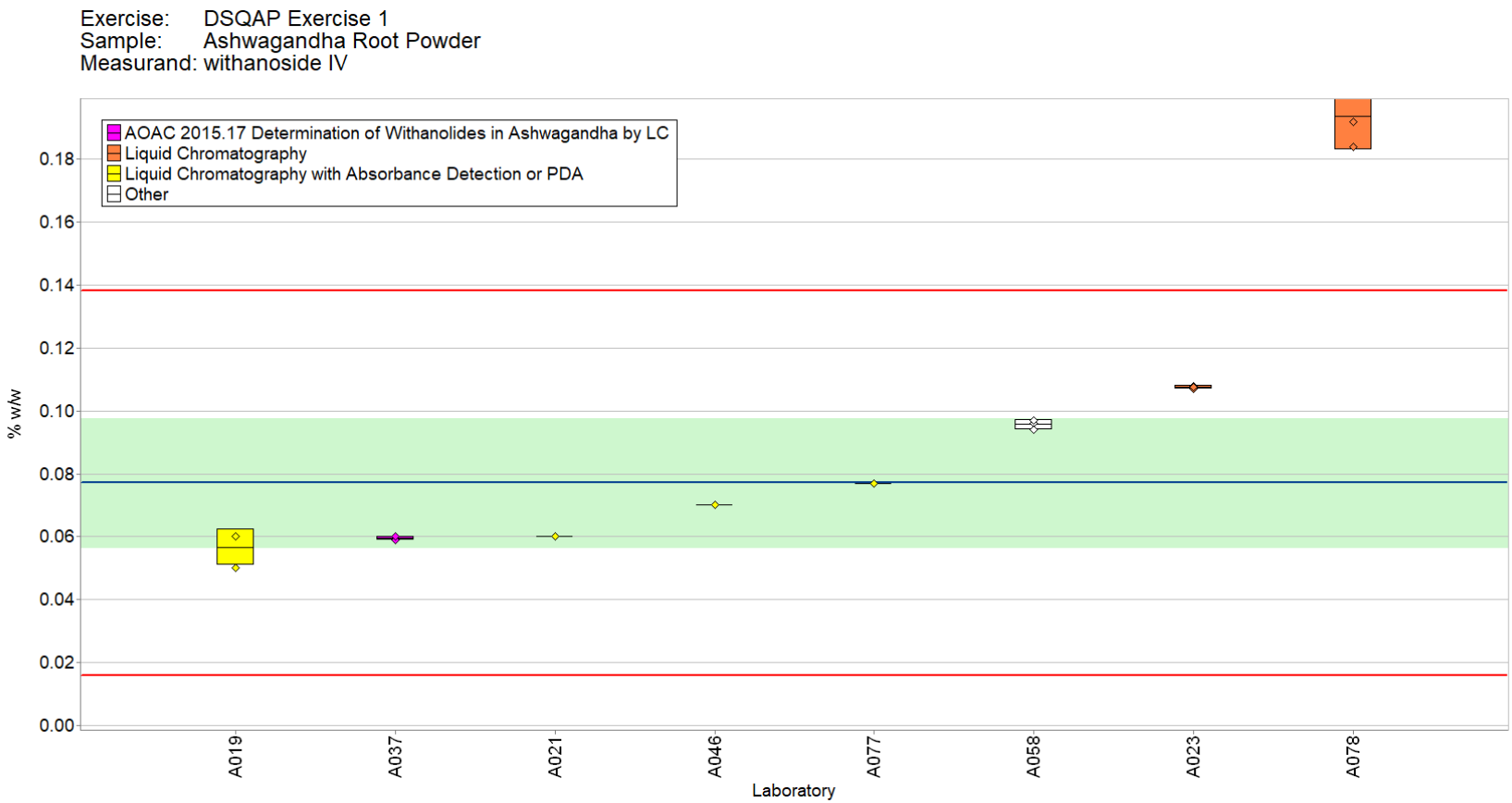

Fig. 9-1. Withanoside IV in ashwagandha root powder (data summary view – analytical method).

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 red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

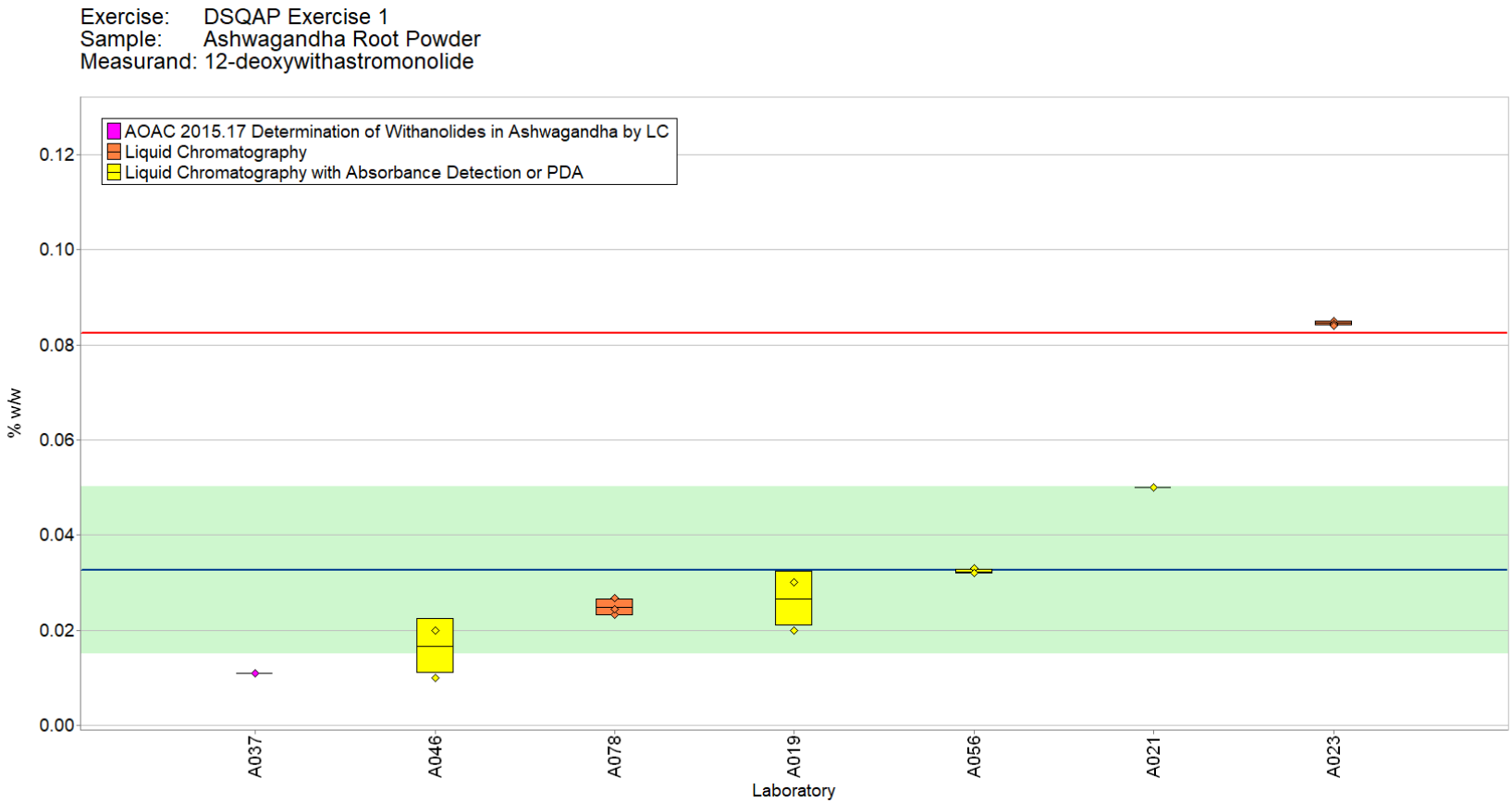

Fig. 9-2. 12-deoxywithastromonolide in ashwagandha root powder (data summary view – analytical method).

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 red solid line represents the upper bound of the consensus range of tolerance, calculated as the value above the consensus mean that results in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$, with the lower bound set to zero.

9.3.2. Ashwagandha Root Powder Extract

Participants were provided with three packets, each containing 1.5 g of ashwagandha (*Withania somnifera*) root powder extract. Participants were asked to store the material at controlled room temperature, 20 °C to 25 °C, and to prepare one sample and report one value from each packet provided. Before use, participants were instructed to mix the contents of the packets thoroughly, and to allow contents to settle for one minute prior to opening to minimize the loss of fine particles. Participants were instructed to use an appropriate sample size for their in-house analytical methods, or at least 0.5 g if using AOAC 2015.17, to determine the mass percent (% w/w) of select withanolides and withanosides. The approximate analyte levels were not reported to participants prior to the study and target values were not available at the time of this report.

As mentioned previously, all laboratories that reported sample preparation methods (44 %) indicated using solvent extraction. Instrumental techniques reported for the ashwagandha root powder extract were also the same for ashwagandha root powder (see previous section and **Table 9-3).**

The excellent average within-laboratory RSDs (at or below 5.4 %) for each measurand in the ashwagandha root powder extract material indicates each participants' in-house methods and/or AOAC 2015.17 are consistent with published expectations [67] as shown in **Table 9-4**. These within-laboratory RSDs are slightly lower than what was achieved for the ashwagandha root powder. Analyzing phytochemicals from extracts tend to be less challenging than root powders since the measurands are already extracted from the raw material. When comparing between-laboratory variability, RSDs range from 7 % (withanoside IV) to 47 % (withanolide B) and are also shown in **Fig. 9-3** and **9-4**, respectively. Figures for the remaining measurands and individual laboratory results for ashwagandha root powder extract can be found in **Appendix G**.

Table 9-4. Summary of laboratory variabilities for withanolides in ashwagandha root powder extract.

	Ashwagandha Root Powder Extract	
Analyte	Within-Laboratory Variability (% RSD)	Between-Laboratory Variability (% RSD)
12-deoxywithastromonolide	0.9 %	30 %
withaferin A	3.3 %	37 %
withanolide A	2.4 %	36 %
withanolide B	5.4 %	47 %
withanoside IV	2.0 %	7 %
withanoside V	3.9 %	16 %

Two laboratories reported values that were roughly double the consensus mean for several analytes (**Fig. 9-3** and **9-4**). Improper calibration is a frequent source of measurement error. Calibrant purity is also an important consideration in analytical measurements. Where possible, calibrants should be evaluated for purity and presence of residual solvents prior to use. The measured purity should be used to correct the concentrations of the solutions used for calibration. If a calibration curve is used, the calibrant concentrations should encompass the sample concentrations. No sample concentrations should be outside of the linear range. Individual matched calibrants should be used for quantitation whenever possible.

Similar to the ashwagandha root powder results, larger between-laboratory variability indicates that the community is not in complete agreement for the analysis of these measurands. However, the participants are in better agreement regarding withanoside IV and withanoside V in the root powder extract. Differences in RSDs between measurands is likely due to analyte accessibility and levels in different matrices. The overall discrepancy may be due to inadequate sample preparation techniques used for the specific phytochemicals in ashwagandha root powder extract. Technical recommendations for ashwagandha root powder extract are the same as the root powder and can be found in the previous section.

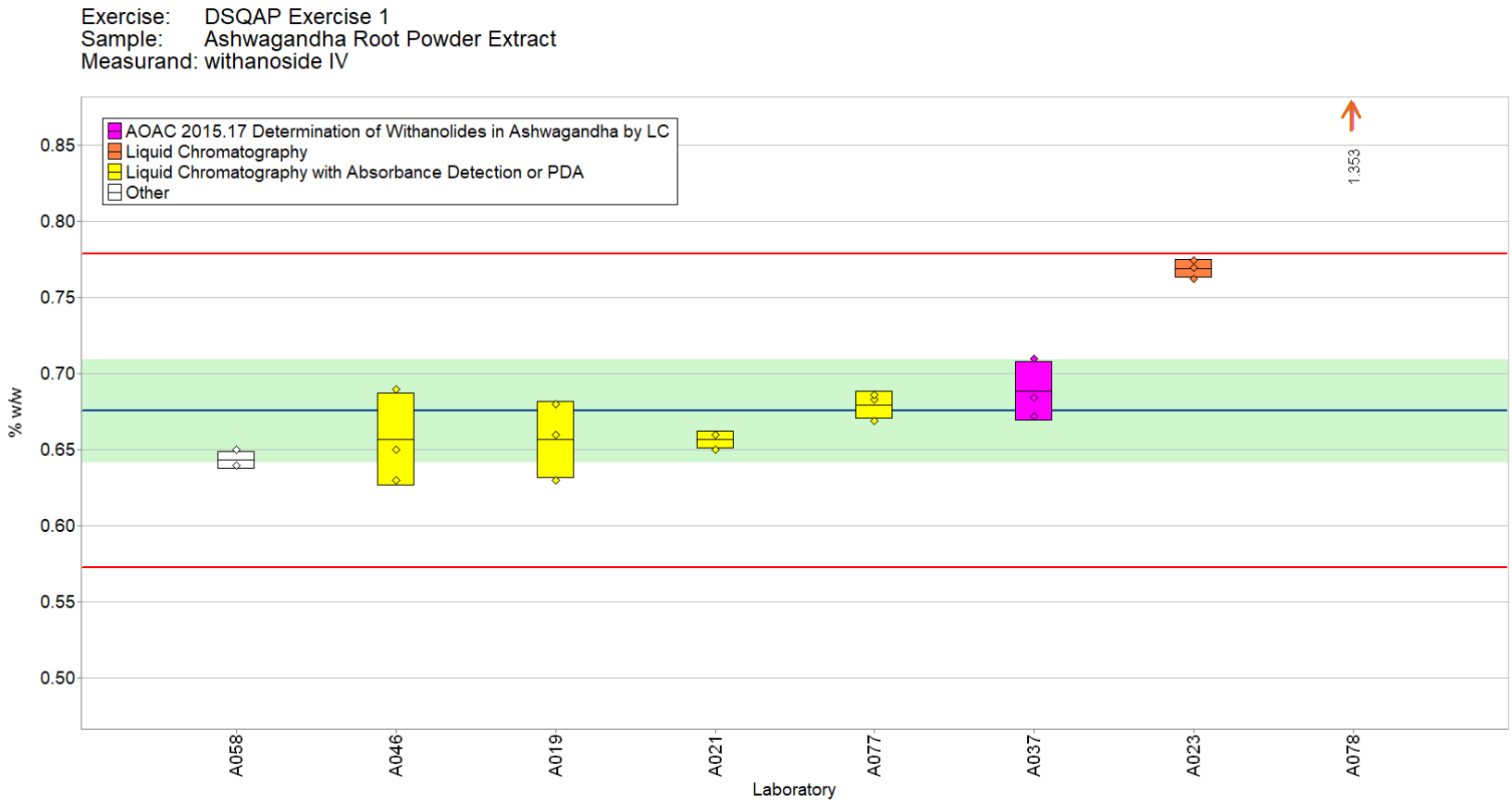

Fig. 9-3. Withanoside IV in ashwagandha root powder extract (data summary view – analytical method).

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 red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

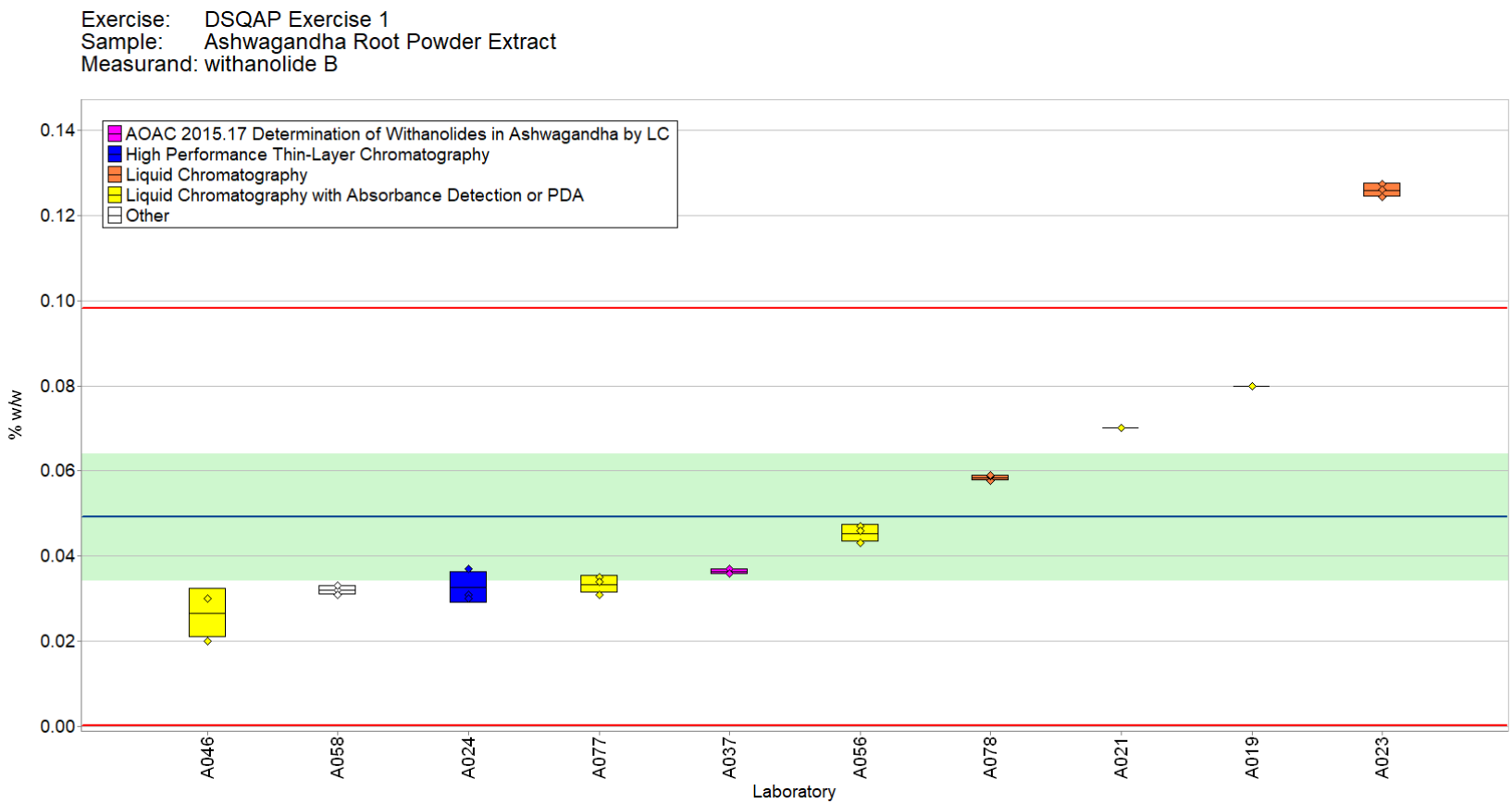

Fig. 9-4. Withanolide B in ashwagandha root powder extract (data summary view – analytical method).

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 red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

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Appendix A. List of Acronyms

AAS	Atomic Absorption Spectroscopy
AMRM	Analytical Methods and Reference Materials
CannaQAP	Cannabis Laboratory Quality Assurance Program
cGMP	Current Good Manufacturing Practice
COA	Certificate of Analysis
CRM	Certified Reference Material
DC AAS	Direct Combustion Atomic Absorption Spectrometry
DSQAP	Dietary Supplement Laboratory Quality Assurance Program
FAAS	Flame Atomic Absorption Spectroscopy
FAES	Flame Atomic Emission Spectroscopy
FDA	US Food and Drug Administration
FNSQAP	Food Nutrition and Safety Measurements Quality Assurance Program
GAE	Gallic Acid Equivalents
HAMQAP	Health Assessment Measurements Quality Assurance Program
HPTLC	High Performance Thin Layer Chromatography
ICP MS	Inductively Coupled Plasma Mass Spectrometry
ICP OES	Inductively Coupled Plasma Optical Emission Spectrometry
ID CV-ICP-MS	Isotope Dilution Cold-Vapor Generation Inductively Coupled Plasma Mass Spectrometry
ID-ICP MS	Isotope Dilution Inductively Coupled Plasma Mass Spectrometry
IEC	International Electrotechnical Commission
INAA	Instrumental Neutron Activation Analysis
ISO	International Organization for Standardization
KED	Kinetic Energy Discrimination
LC Abs	Liquid Chromatography with Absorbance Detection
LC FLD	Liquid Chromatography with Fluorescence Detection
LC MS	Liquid Chromatography Mass Spectrometry
LC MS/MS	Liquid Chromatography with Tandem Mass Spectrometry
LC-ICP-MS	Liquid Chromatography with Inductively Coupled Plasma Mass Spectrometry
LOQ	Limit of Quantification
MDL	Method Detection Limit
MMQAP	Micronutrients Measurement Quality Assurance Program
NIST	National Institute of Standards and Technology
NIH	National Institutes of Health
ODS	Office of Dietary Supplements
PDA	Photodiode Array Detection
PTFE	polytetrafluoroethylene
QA	Quality Assurance
QAP	Quality Assurance Program
QL	Quantification Limit
QQQ-ICP-MS	Inductively Coupled Triple Quadrupole Plasma Mass Spectrometry
RM	Reference Material
RNAA	Radiochemical Neutron Activation Analysis
RSD	Relative Standard Deviation
SD	Standard Deviation

SMPR	Standard Method Performance Requirements
SNP	Seaweed Nitrogen to Protein
SRM	Standard Reference Material
TDF	Total Dietary Fiber
TMAH	Tetramethylammonium Hydroxide
VitDQAP	Vitamin D Metabolites Quality Assurance Program

Appendix B. Elements Supplemental Tables and Figures

Table B-1. Individualized data summary table (example) for elements in kelp.

(Laboratory Name)

Exercise 1 - Elements in Kelp

	Lab Code:	**(code)**	**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 (tAs)	Kelp A	ug/g					32	65.30	6.10	62.80	0.69
Total Arsenic (tAs)	Kelp B	ug/g					32	28.20	3.30	27.50	1.03
Total Arsenic (tAs)	SRM 3232	ug/g					32	35.5	4.00	35.90	1.22
Inorganic Arsenic (iAs)	Kelp A	ug/g					5	0.20	0.30	0.17	0.04
Inorganic Arsenic (iAs)	Kelp B	ug/g					6	0.08	0.15	0.16	0.19
Inorganic Arsenic (iAs)	SRM 3232	ug/g					6	0.13	0.07	0.09	0.02
Cadmium	Kelp A	ug/g					33	1.10	0.10	1.16	0.02
Cadmium	Kelp B	ug/g					32	0.36	0.03	0.38	0.01
Cadmium	SRM 3232	ug/g					33	0.37	0.04	0.40	0.01
Calcium	Kelp A	mg/g					34	10.00	1.76	9.22	0.57
Calcium	Kelp B	mg/g					35	13.10	1.88	12.20	0.19
Calcium	SRM 3232	mg/g					34	12.40	1.74	11.50	0.64
Chromium	Kelp A	ug/g					31	3.92	1.19	4.36	0.13
Chromium	Kelp B	ug/g					26	0.51	0.16	0.55	0.03
Chromium	SRM 3232	ug/g					31	4.98	1.53	5.55	0.48
Copper	Kelp A	ug/g					24	1.37	0.49	1.34	0.06
Copper	Kelp B	ug/g					24	1.18	0.33	1.12	0.05
Copper	SRM 3232	ug/g					28	3.45	0.77	3.63	0.08
Iodine	Kelp A	ug/g					12	2060	264		
Iodine	Kelp B	ug/g					12	880	134		
Iodine	SRM 3232	ug/g					12	775	125	884	82
Lead	Kelp A	ug/g					31	0.57	0.09	0.59	0.02
Lead	Kelp B	ug/g					30	0.40	0.05	0.41	0.01
Lead	SRM 3232	ug/g					30	0.88	0.12	0.97	0.04
Magnesium	Kelp A	ug/g					33	7196	445	6881	917
Magnesium	Kelp B	ug/g					33	8310	714	5287	2254
Magnesium	SRM 3232	ug/g					33	5793	470	5743	169
Mercury	Kelp A	ng/g					27	33.7	12.00	31.30	0.46
Mercury	Kelp B	ng/g					26	27.10	8.20	23.10	0.42
Mercury	SRM 3232	ng/g					29	95.8	16.8	105.8	2.99
Potassium	Kelp A	mg/g					33	118	12	126.3	2.5
Potassium	Kelp B	mg/g					33	20.7	1.6	21.8	0.77
Potassium	SRM 3232	mg/g					33	67.7	5.6	71.2	1.0
Selenium	Kelp A	ng/g					21	173	147	89.2	4.7
Selenium	Kelp B	ng/g					20	110	94.8	38.3	6.9
Selenium	SRM 3232	ng/g					20	120	109	52.6	13.7
Sodium	Kelp A	ug/g					28	39300	3399	39457	129
Sodium	Kelp B	ug/g					29	30897	3084	31741	432
Sodium	SRM 3232	ug/g					29	14739	1321	15298	356
Sulfur	Kelp A	ug/g					13	6570	1386		
Sulfur	Kelp B	ug/g					12	22119	3262		
Sulfur	SRM 3232	ug/g					13	10645	1886		
Zinc	Kelp A	ug/g					28	18.00	3.12	18.60	1.70
Zinc	Kelp B	ug/g					29	31.50	4.37	32.00	3.20
Zinc	SRM 3232	ug/g					28	25.6	3.98	25.70	1.03

Individual laboratory results will appear in this section; Laboratory-specific results were provided to each participant separately from this report

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 value
U expanded uncertainty about the NIST value

Table B-2. Data summary table for total arsenic in kelp.

Data highlighted in blue have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable Z'_{comm} score, $|Z'_{comm}| > 2$. (Table continues to next page.)

		Total Arsenic (tAs)														
		Kelp A (ug/g)					Kelp B (ug/g)					SRM 3232 Kelp Powder (*Thallus laminariae*) (ug/g)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
Individual Results	Target				62.8	0.69				27.5	1.03				35.9	1.22
	A001															
	A002															
	A003															
	A004	67.196	63.538	64.95	65.2	1.84	26.988	28.182	27.38	27.5	0.61	35.905	33.952	35.587	35.1	1.05
	A005	66	65.2	64.9	65.4	0.57	27.5	27.7	27.6	27.6	0.10	35.2	34.2	34.1	34.5	0.61
	A008															
	A009	62.85	62.53	66.06	63.8	1.95	26.48	26.35	26.98	26.6	0.33	33.33	35.03	31.51	33.3	1.76
	A012	65.3	67	65.6	66.0	0.91	28.5	28.1	28.1	28.2	0.23	36.6	36	36.9	36.5	0.46
	A013	64.687	66.127	65.829	65.5	0.76	26.182	26.98	26.47	26.5	0.40	32.3231	35.0883	34.9095	34.1	1.55
	A015															
	A016	73.6	74.7	70.3	72.9	2.29	32.1	32.5	32.7	32.4	0.31	41.1	41	40.5	40.9	0.32
	A017	77.7	77.9	77	77.5	0.47	28.7	28.7	28.3	28.6	0.23	36.9	38.2	36.6	37.2	0.85
	A018	66.3	66.7	67.2	66.7	0.45	25.4	26.7	26.4	26.2	0.68	33.9	33.7		33.8	0.14
	A019	70.49	72.82	74.03	72.4	1.80	31.74	32.89	32.71	32.4	0.62	42.43	42.1	41.37	42.0	0.54
	A020															
	A022															
	A024	1.4971	1.4968	1.4975	1.50	0.00	1.4906	1.489	1.4925	1.49	0.00	1.827	1.8268	1.8273	1.83	0.00
	A025	65.857	61.766	62.338	63.3	2.22	25.073	26.662	26.9556	26.2	1.01	32.481	33.165	31.759	32.5	0.70
	A027	70.019	65.27	59.776	65.0	5.13	31.352	34.705	32.036	32.7	1.77	36.935	42.24	33.488	37.6	4.41
	A028															
	A029	66.9	65.7	65.2	65.9	0.87	27.9	27.9	27.7	27.8	0.12	34.5	33.8	35.3	34.5	0.75
	A031															
	A032															
	A033	67.4	67.1	69.5	68.0	1.31	31	30	29	30.0	1.00	36.4	35.9	37.1	36.5	0.60
	A035	62.4105	62.045	61.453	62.0	0.48	26.674	26.195	24.918	25.9	0.91	33.7215	32.538	32.0875	32.8	0.84
	A037	67.2	68.4	64.2	66.6	2.16	47.3	30.2	29.2	35.6	10.17	36.4	36.4	34.7	35.8	0.98
	A039															

		Total Arsenic (tAs)														
		Kelp A (ug/g)					Kelp B (ug/g)					SRM 3232 Kelp Powder (*Thallus laminariae*) (ug/g)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
	A041	64.354	63.263	63.782	63.8	0.55	27.704	27.163	27.542	27.5	0.28	34.608	34.093	34.056	34.3	0.31
	A042	62.9	61.1	62.8	62.3	1.01	26	26.2	26.7	26.3	0.36	35.1	34.3	34.2	34.5	0.49
	A043	79.71	80.55	80.37	80.2	0.44	35.42	35.06	34.71	35.1	0.36	44.33	44.21	44.43	44.3	0.11
	A044	65.9	62.6	63.2	63.9	1.76	25.3	25.7	24.9	25.3	0.40	32.4	32.1	31.2	31.9	0.62
	A045	50.01	58.13	66.34	58.2	8.17	22.11	21.58	34.07	25.9	7.06	29.24	40.07	30.15	33.2	6.01
	A046	63.667	58.102	59.118	60.3	2.96	32.909	29.948	26.669	29.8	3.12	35.35	31.951	31.292	32.9	2.18
	A047	59.1	60.5		59.8	0.99	24.8	25.6		25.2	0.57	31.9	32.2		32.1	0.21
	A050	65.17	57.95	56.18	59.8	4.76	30.64	28.8	29	29.5	1.01	39.08	41.64	44.18	41.6	2.55
	A051															
	A053															
	A054	68.7	67.9	72	69.5	2.17	30.4	30.1	29.8	30.1	0.30	37.2	38.6	38.3	38.0	0.74
	A057	68.9	67.9	68.5	68.4	0.50	25.3	26.7	27.3	26.4	1.03	34.2	34.1	33.3	33.9	0.49
	A058	69.887	73.357	71.418	71.6	1.74	29.92	29.682	30.897	30.2	0.64	38.877	38.377	38.847	38.7	0.28
	A060	52.9	50.6	53.9	52.5	1.69	28.6	24.5	25	26.0	2.24	26.6	30.4	29.5	28.8	1.99
	A061															
	A062															
	A065															
	A066															
	A068															
	A070	64.85	65.48	63.46	64.6	1.03	26.56	16.14	26.66	23.1	6.05	37.12	33.83	33.22	34.7	2.10
	A071															
	A073	54.7277	50.2611	54.5611	53.2	2.53	23.9944	25.1944	22.6611	23.9	1.27	31.1611	32.0611	31.7277	31.6	0.46
	A074															
	A075	61.8221	60.6564	60.1992	60.9	0.84	25.3473	25.5442	25.5968	25.5	0.13	36.6412	34.6229	33.7802	35.0	1.47
	A079	70.8	71.3	67.8	70.0	1.89	33.4	34.8	32.8	33.7	1.03	43.8	44.1	43.9	43.9	0.15
Community Results		Consensus Mean			65.3		Consensus Mean			28.2		Consensus Mean			35.5	
		Consensus Standard Deviation			6.1		Consensus Standard Deviation			3.3		Consensus Standard Deviation			3.9	
		Maximum			80.2		Maximum			35.6		Maximum			44.3	
		Minimum			1.50		Minimum			1.49		Minimum			1.83	
		N			32		N			32		N			32	

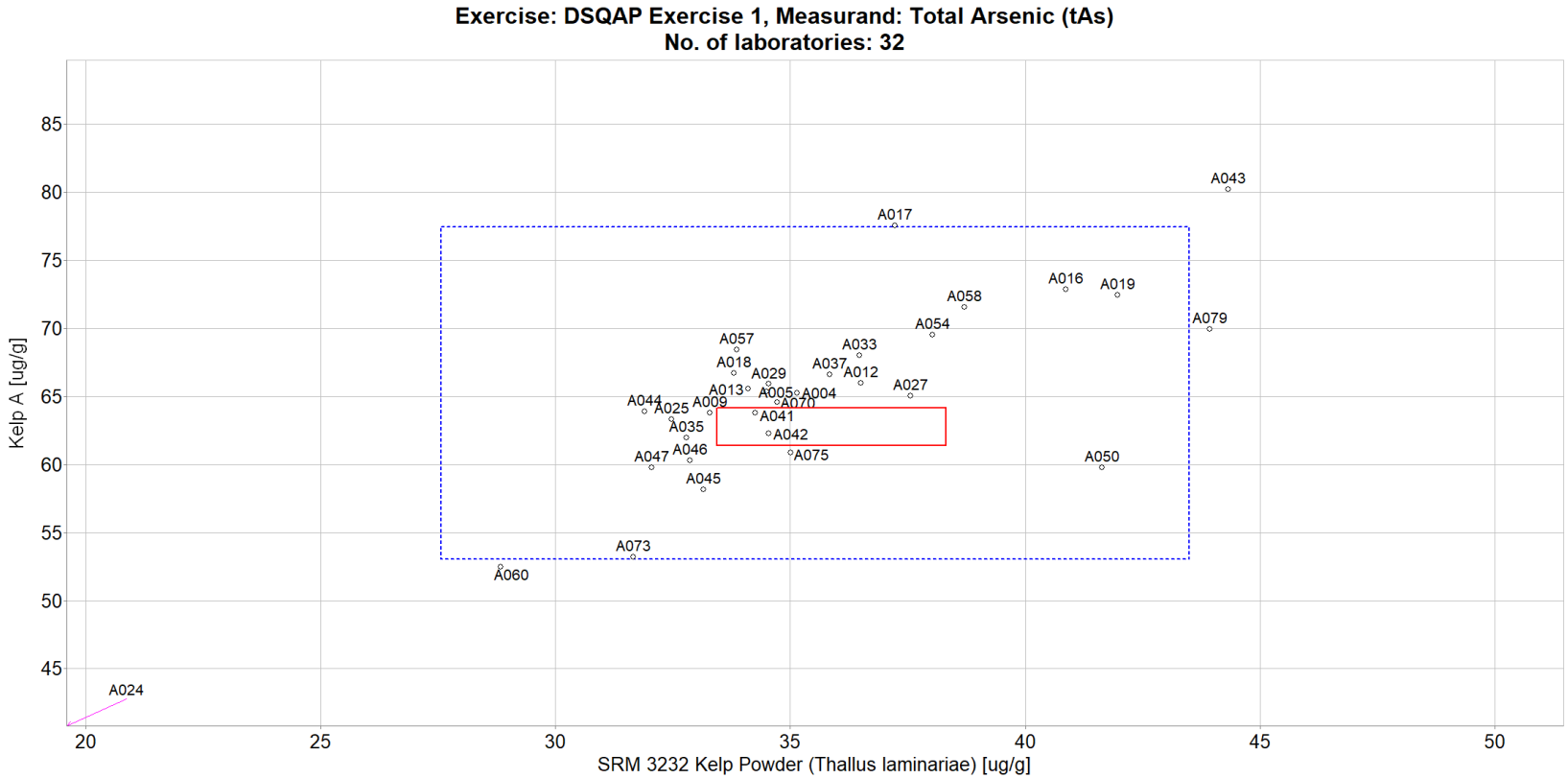

Fig. B-1. Laboratory means for total arsenic in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp A (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3232, is compared to the individual laboratory mean for a second sample, Kelp A. The solid red box represents the NIST range of tolerance for the two samples, SRM 3232 (x-axis) and Kelp A (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3232 (x-axis) and Kelp A (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

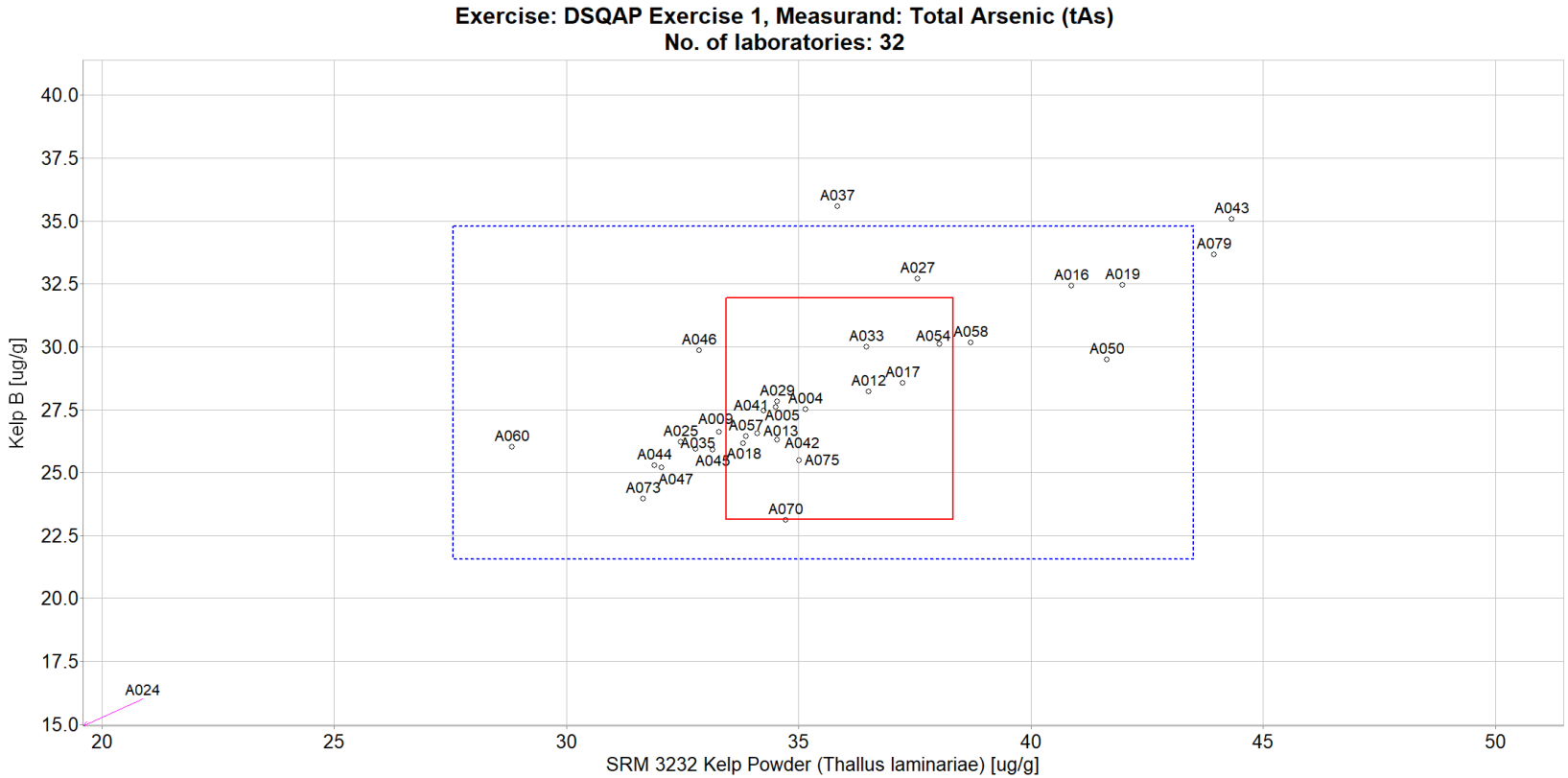

Fig. B-2. Laboratory means for total arsenic in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp B (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3232, is compared to the individual laboratory mean for a second sample, Kelp B. The solid red box represents the NIST range of tolerance for the two samples, SRM 3232 (x-axis) and Kelp B (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$.The dotted blue box represents the consensus range of tolerance for SRM 3232 (x-axis) and Kelp B (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

Table B-3. Data summary table for inorganic arsenic in kelp.

Data highlighted in blue have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable Z'_{comm} score, $|Z'_{comm}| > 2$. (Table continues to next page.)

		Inorganic Arsenic (iAs)														
		Kelp A (ug/g)					Kelp B (ug/g)					SRM 3232 Kelp Powder (*Thallus laminariae*) (ug/g)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
Individual Results	Target				0.17	0.04				0.16	0.19				0.09	0.02
	A001															
	A002															
	A004															
	A008															
	A012															
	A013															
	A017	0.217	0.225	0.239	0.23	0.01	0.085	0.088	0.069	0.08	0.01	0.118	0.142	0.127	0.13	0.01
	A018															
	A020															
	A022															
	A025															
	A027															
	A028															
	A029	0.391	0.376	0.376	0.38	0.01	0.14	0.146	0.14	0.14	0.003	0.186	0.183	0.191	0.19	0.004
	A031															
	A032															
	A033	0.014	0.014	0.015	0.01	0.008	0.063	0.064	0.062	0.06	0.001	0.098	0.095	0.095	0.10	0.002
	A035	0.0877	0.0721	0.0804	0.08	0.01	0.0447	0.043	0.0445	0.04	0.001	0.1047	0.0883	0.0884	0.09	0.01
	A037															
	A041															
	A042	< 0.200	< 0.200	< 0.200			< 0.200	< 0.200	< 0.200			< 0.200	< 0.200	< 0.200		
	A043															
	A044															
	A045															
	A046															
	A051															
	A052															

		Inorganic Arsenic (iAs)														
		Kelp A (ug/g)					Kelp B (ug/g)					SRM 3232 Kelp Powder (*Thallus laminariae*) (ug/g)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
	A053															
	A054															
	A057															
	A061															
	A062															
	A065															
	A066						35.3	34.6	35.2	35.03	0.38	41.5	43.5	44.5	43.17	1.53
	A068															
	A071															
	A073															
	A074	0.258	0.233	0.23	0.24	0.02	10.122	9.074	10.514	9.90	0.74	0.104	0.123	0.131	0.12	0.01
	A075															
	A079															
Community Results		Consensus Mean			0.19		Consensus Mean			0.08		Consensus Mean			0.13	
		Consensus Standard Deviation			0.30		Consensus Standard Deviation			0.15		Consensus Standard Deviation			0.07	
		Maximum			0.38		Maximum			35.03		Maximum			43.17	
		Minimum			0.01		Minimum			0.04		Minimum			0.09	
		N			5		N			6		N			6	

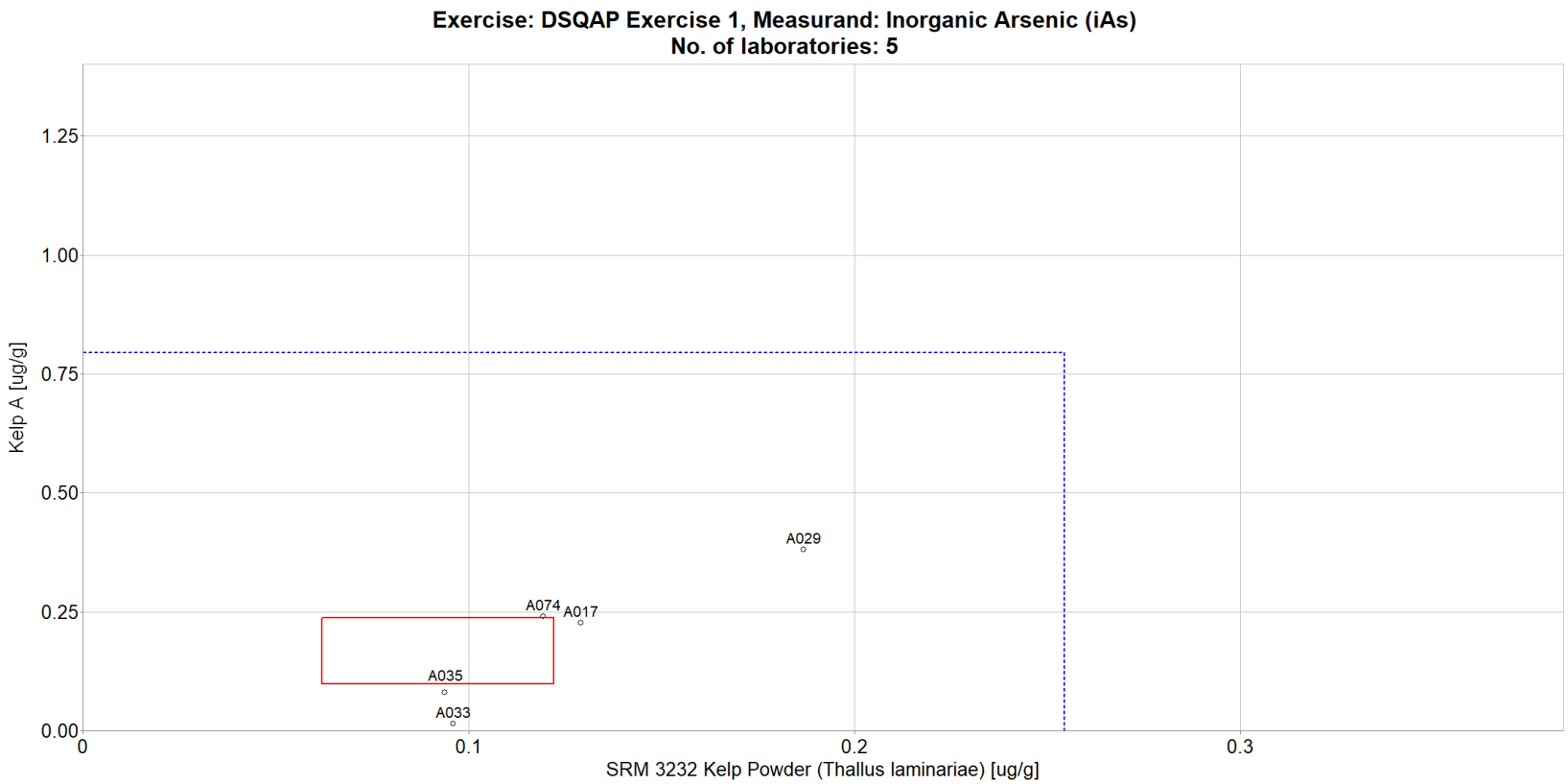

Fig. B-3. Laboratory means for inorganic arsenic in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp A (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3232, is compared to the individual laboratory mean for a second sample, Kelp A. The solid red box represents the NIST range of tolerance for the two samples, SRM 3232 (x-axis) and Kelp A (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3232 (x-axis) and Kelp A (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

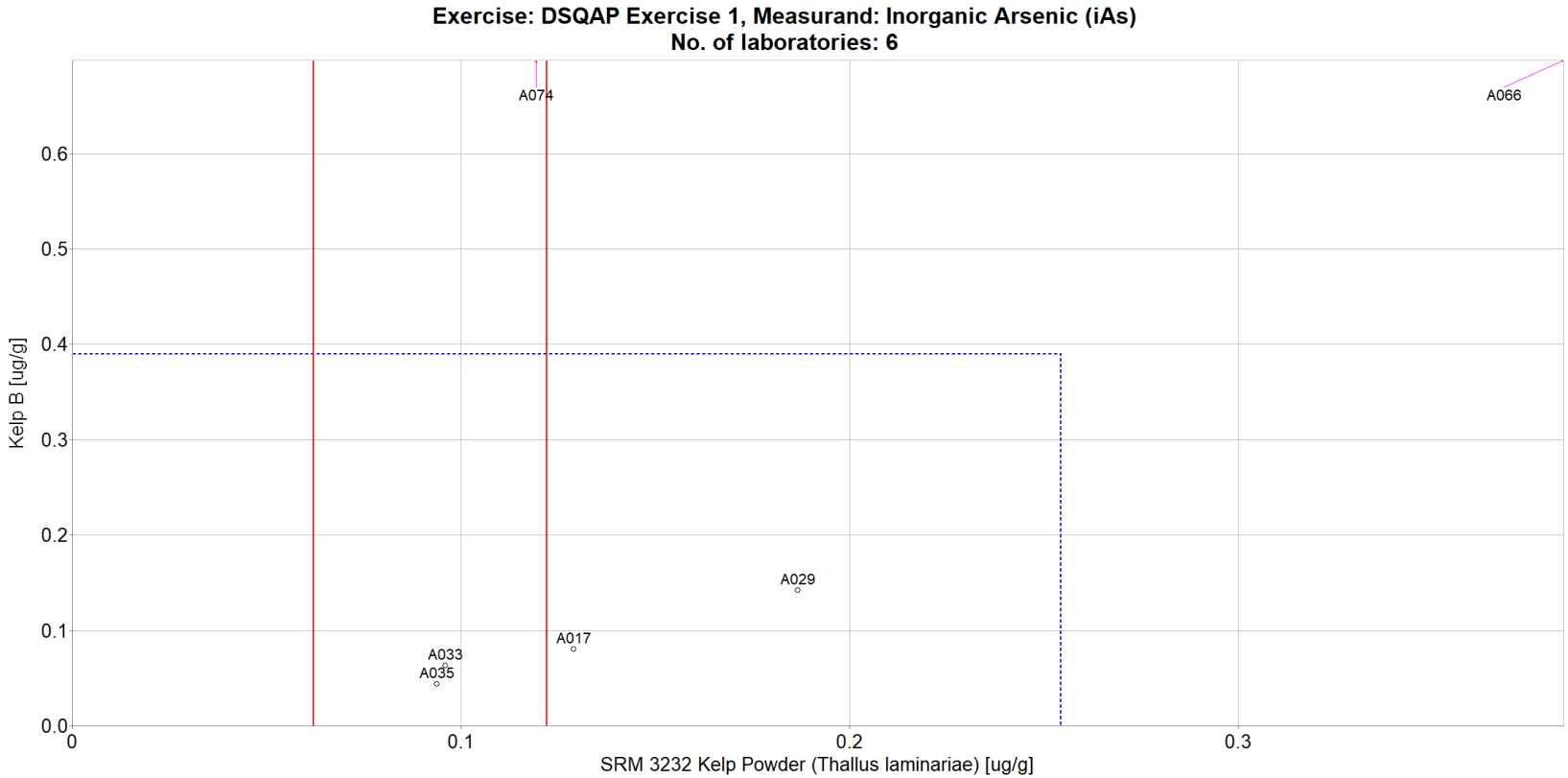

Fig. B-4. Laboratory means for inorganic arsenic in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp B (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3232, is compared to the individual laboratory mean for a second sample, Kelp B. The solid red box represents the NIST range of tolerance for the two samples, SRM 3232 (x-axis) and Kelp B (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$.The dotted blue box represents the consensus range of tolerance for SRM 3232 (x-axis) and Kelp B (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

Table B-4. Data summary table for cadmium in kelp.

Data highlighted in blue have been identified as outside the consensus range of tolerance and would be estimated to result in an unacceptable Z'_{comm} score, $|Z'_{comm}| > 2$. (Table continues to next page.)

		Cadmium														
		Kelp A (ug/g)					Kelp B (ug/g)					SRM 3232 Kelp Powder (*Thallus laminariae*) (ug/g)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
Individual Results	Target				1.16	0.02				0.38	0.01				0.40	0.01
	A001															
	A002															
	A003															
	A004	1.071	1.135	1.089	1.10	0.03	0.357	0.36	0.382	0.37	0.01	0.359	0.392	0.395	0.38	0.02
	A005	1.15	1.18	1.23	1.19	0.04	0.397	0.356	0.385	0.38	0.02	0.393	0.39	0.34	0.37	0.03
	A008															
	A009	1.06	1.1	1.12	1.09	0.03	0.34	0.37	0.36	0.36	0.02	0.36	0.37	0.45	0.39	0.05
	A012	1.12	1.15	1.15	1.14	0.02	0.362	0.362	0.348	0.36	0.01	0.379	0.382	0.394	0.39	0.01
	A013	1.0805	1.049	1.0689	1.07	0.02	0.33479	0.3349	0.32247	0.33	0.01	0.335	0.3695	0.3709	0.36	0.02
	A015															
	A016	1.1	1.14	1.1	1.11	0.02	0.38	0.46	0.38	0.41	0.05	0.39	0.38	0.4	0.39	0.01
	A017	1.27	1.23	1.36	1.29	0.07	0.373	0.39	0.372	0.38	0.01	0.388	0.433	0.384	0.40	0.03
	A018	1.04	1.05	1.04	1.04	0.01	0.295	0.309	0.307	0.30	0.01	0.326	0.327		0.33	0.001
	A019	0.96	1.13	1.13	1.07	0.10	0.35	0.37	0.35	0.36	0.01	0.39	0.35	0.35	0.36	0.02
	A020	1.071	1.13	1.025	1.08	0.05	0.362	0.362	0.361	0.36	0.001	0.373	0.376	0.374	0.37	0.002
	A022															
	A024	0.4786	0.4788	0.4781	0.48	0.0004	< 0.005	< 0.005	< 0.005			0.494	0.491	0.495	0.49	0.002
	A025	1.2933	0.9323	1.8288	1.35	0.45	0.3659	0.2154	0.4208	0.33	0.11	0.1679	0.5006	0.2275	0.30	0.18
	A027	0.923	0.89	0.809	0.87	0.06	0.32	0.337	0.34	0.33	0.01	0.315	0.368	0.287	0.32	0.04
	A028															
	A029	1.12	1.07	1.11	1.10	0.03	0.354	0.354	0.348	0.35	0.003	0.372	0.362	0.378	0.37	0.01
	A031															
	A032															
	A033	0.994	0.944	0.946	0.96	0.03	0.321	0.325	0.327	0.32	0.003	0.354	0.327	0.331	0.34	0.01
	A035	1.15	1.16	1.18	1.16	0.02	0.34	0.35	0.37	0.35	0.02	0.42	0.43	0.44	0.43	0.01
	A037	1.03	1.02	1.03	1.03	0.01	0.317	0.326	0.331	0.32	0.01	0.349	0.374	0.352	0.36	0.01
	A039															

		Cadmium														
		Kelp A (ug/g)					Kelp B (ug/g)					SRM 3232 Kelp Powder (*Thallus laminariae*) (ug/g)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
	A041	1.168	1.165	1.189	1.17	0.01	0.378	0.376	0.371	0.38	0.004	0.392	0.402	0.398	0.40	0.01
	A042	1.14	1.02	1.15	1.10	0.07	0.34	0.36	0.37	0.36	0.02	0.36	0.33	0.34	0.34	0.02
	A043	1.19	1.2	1.2	1.20	0.01	0.41	0.41	0.41	0.41	0.00	0.44	0.44	0.44	0.44	0
	A044	1.18	1.18	1.19	1.18	0.01	0.35	0.36	0.37	0.36	0.01	0.4	0.39	0.37	0.39	0.02
	A045	1.1	1.13	1.04	1.09	0.05	0.55	0.54	0.32	0.47	0.13	0.43	0.86	0.96	0.75	0.28
	A046	1.001	0.926	0.959	0.96	0.04	0.389	0.348	0.323	0.35	0.03	0.354	0.327	0.327	0.34	0.02
	A047	1.1	1.1		1.10	0.00	0.36	0.36		0.36	0	0.38	0.4		0.39	0.01
	A050	0.944	1.01	1.02	0.99	0.04	0.339	0.338	0.349	0.34	0.01	0.358	0.361	0.36	0.36	0.002
	A051															
	A052															
	A053															
	A054	1.13	1.11	1.15	1.13	0.02	0.38	0.374	0.386	0.38	0.01	0.395	0.404	0.395	0.40	0.01
	A057	1.15	1.09	1.12	1.12	0.03	0.36	0.35	0.36	0.36	0.01	0.38	0.38	0.38	0.38	0
	A058	1.125	1.118	1.172	1.14	0.03	0.387	0.39	0.36	0.38	0.02	0.396	0.408	0.381	0.40	0.01
	A060	1.06	1.03	1.08	1.06	0.03	0.398	0.34	0.346	0.36	0.03	0.306	0.36	0.369	0.35	0.03
	A061															
	A062															
	A065	1.01	1.02	1	1.01	0.01	0.33	0.33	0.36	0.34	0.02	0.35	0.35	0.34	0.35	0.01
	A066															
	A068															
	A070	1.0504	1.0889	1.1318	1.09	0.04	0.3636	0.3677	0.3734	0.37	0.005	0.4041	0.3972	0.3885	0.40	0.01
	A071															
	A073	1.12	1.0433	1.1766	1.11	0.07	0.349	0.372	0.3306	0.35	0.02	0.3903	0.3866	0.3796	0.39	0.01
	A075	0.97993	0.99319	1.05094	1.01	0.04	0.31019	0.33628	0.31502	0.32	0.01	0.32989	0.34159	0.34794	0.34	0.01
	A079	< 6.000	< 6.000	< 6.000			< 3.000	< 3.000	< 3.000			< 3.000	< 3.000	< 3.000		
Community Results		Consensus Mean			1.10		Consensus Mean			0.36		Consensus Mean			0.37	
		Consensus Standard Deviation			0.10		Consensus Standard Deviation			0.03		Consensus Standard Deviation			0.04	
		Maximum			1.35		Maximum			0.47		Maximum			0.75	
		Minimum			0.48		Minimum			0.30		Minimum			0.30	
		N			33		N			32		N			33	

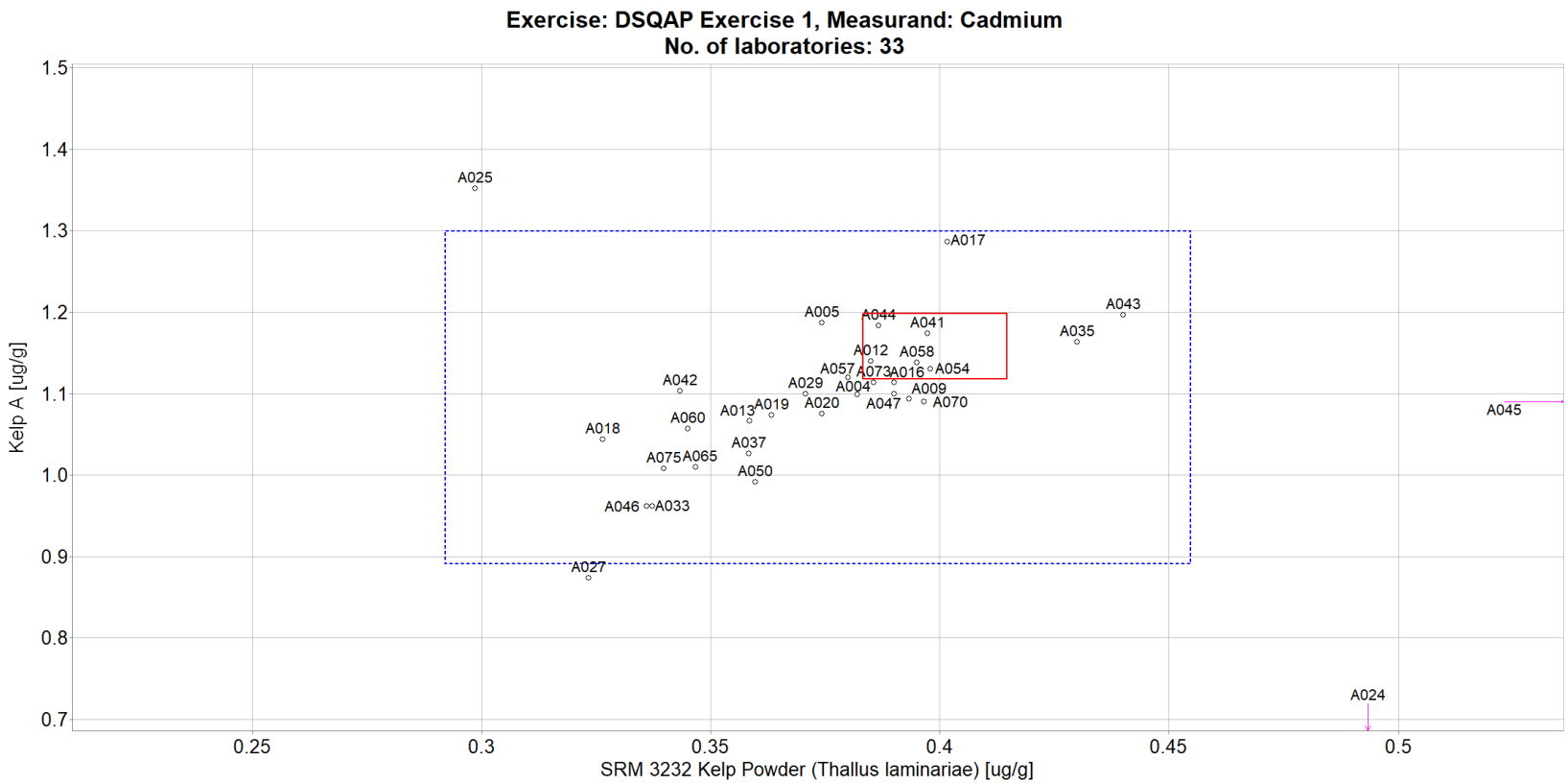

Fig. B-5. Laboratory means for cadmium in SRM 3232 Kelp Powder (Thallus laminariae) and Kelp A (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3232, is compared to the individual laboratory mean for a second sample, Kelp A. The solid red box represents the NIST range of tolerance for the two samples, SRM 3232 (x-axis) and Kelp A (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3232 (x-axis) and Kelp A (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

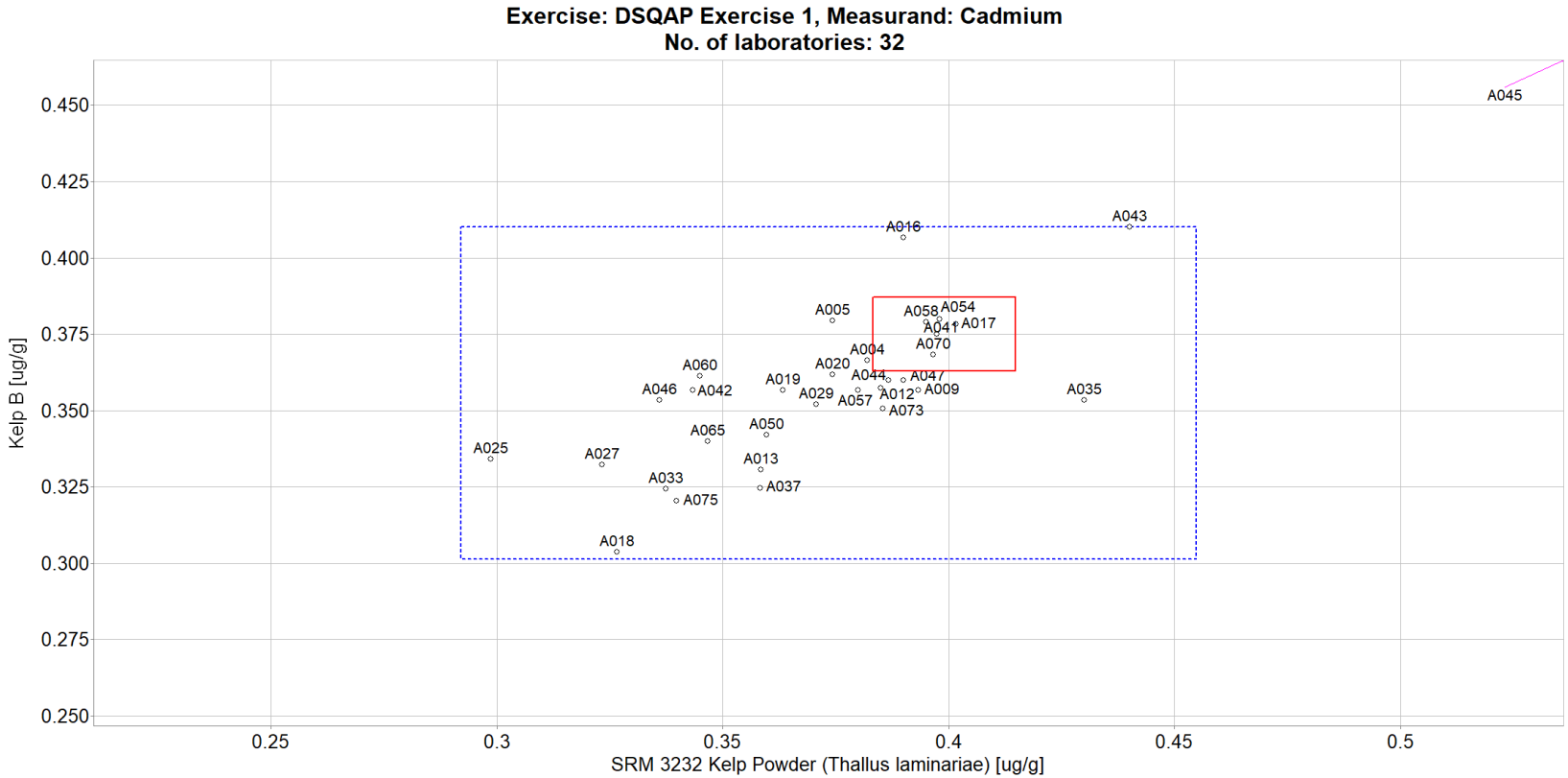

Fig. B-6. Laboratory means for cadmium in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp B (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3232, is compared to the individual laboratory mean for a second sample, Kelp B. The solid red box represents the NIST range of tolerance for the two samples, SRM 3232 (x-axis) and Kelp B (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3232 (x-axis) and Kelp B (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

Table B-5. Data summary table for calcium in kelp.

Data highlighted in blue have been identified as outside the consensus range of tolerance and would be estimated to result in an unacceptable Z'_{comm} score, $|Z'_{comm}| > 2$. (Table continues to next page.)

		Calcium														
		Kelp A (mg/g)					Kelp B (mg/g)					SRM 3232 Kelp Powder (*Thallus laminariae*) (mg/g)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
Individual Results	Target				9.22	0.57				12.2	0.19				11.5	0.64
	A001															
	A002															
	A003	9.819	9.742	9.73	9.76	0.05	12.75	12.76	12.82	12.8	0.04	11.95	12.04	11.71	11.9	0.17
	A004	10.056	9.727	9.661	9.81	0.21	13.171	13.012	13.23	13.1	0.11	12.479	12.006	12.204	12.2	0.24
	A005															
	A009	12.63	13.14	13.86	13.2	0.62	15.89	15.3	14.84	15.3	0.53	14.88	14.88	14.78	14.8	0.06
	A012	9.18	8.88	0.908	6.32	4.69	12.6	13.1	12.8	12.8	0.25	12	11.9	12.4	12.1	0.26
	A013	16.84648	14.20413	19.89288	17.0	2.85	17.7825	19.50319	20.94799	19.4	1.58	17.81739	11.87674	15.95609	15.2	3.04
	A015															
	A016	8.22	8.28	8.13	8.21	0.08	11.6	11.5	11.6	11.6	0.06	10.5	10.5	10.6	10.5	0.06
	A017	11.885	11.858	11.051	11.6	0.47	13.52	13.52	13.84	13.6	0.18	13.327	13.662	13.043	13.3	0.31
	A018	9.9	10.1	10.2	10.1	0.15	12	12.6	12.4	12.3	0.31	12.3	12.1		12.2	0.14
	A020	9.314	9.414	9.25	9.33	0.08	12.531	13.111	13.031	12.9	0.31	11.516	11.895	11.882	11.8	0.22
	A022															
	A024	8.12032	8.120319	8.120321	8.12	0.00	7.457758	7.457757	7.45776	7.46	0.00	10.32067	10.32067	10.32067	10.3	0.00
	A025	11.527	10.378	9.357	10.4	1.09	14.632	12.681	13.981	13.8	0.99	12.089	12.867	12.633	12.5	0.40
	A027	12.90722	11.71963	12.61513	12.4	0.62	15.93014	15.80081	16.11707	15.9	0.16	14.54879	16.66076	14.93781	15.4	1.12
	A028															
	A029	8.85	10.2	8.96	9.34	0.75	11.7	11.7	12.4	11.9	0.40	11.1	11.3	11.7	11.4	0.31
	A031	8.9	8.9	9.1	8.97	0.12	12	12	12.2	12.1	0.12	11.3	11.3	11.2	11.3	0.06
	A032															
	A033	10.925	10.75	11.099	10.9	0.17	14.61	14.272	14.627	14.5	0.20	13.844	13.572	14.244	13.9	0.34
	A035	9.252	9.436	9.027	9.24	0.20	12.55	12.54	12.61	12.6	0.04	11.77	11.39	11.73	11.6	0.21
	A037	9.19	9.73	9.16	9.36	0.32	12.4	12.3	12.5	12.4	0.10	12.2	11.8	12	12.0	0.20
	A039															
	A041	10.979	10.735	10.521	10.7	0.23	14.023	14.157	14.105	14.1	0.07	13.502	13.494	13.652	13.5	0.09
	A042						12.813	12.779	12.885	12.8	0.05					
	A043	9.284607	9.383883	9.341232	9.34	0.05	12.71704	13.00835	12.72034	12.8	0.17	11.90046	11.6857	11.76768	11.8	0.11

		Calcium														
		Kelp A (mg/g)					Kelp B (mg/g)					SRM 3232 Kelp Powder (*Thallus laminariae*) (mg/g)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
	A044	9.594661	9.233722	9.350044	9.39	0.18	12.27362	12.24594	11.77454	12.1	0.28	11.25841	11.0182	10.94081	11.1	0.17
	A045	8.92097	8.96955	9.608	9.17	0.38	13.69012	13.27732	15.66452	14.2	1.28	12.7072	12.59874	14.59593	13.3	1.12
	A046	2.311813	2.079786	2.156394	2.18	0.12	3.407991	3.198769	2.764655	3.12	0.33	3.003849	2.691318	2.622708	2.77	0.20
	A047	9.52	9.51		9.52	0.01	12.7	12.8		12.8	0.07	12.1	12.6		12.4	0.35
	A050	8.997	9.119	8.946	9.02	0.09	12.83	12.46	12.31	12.5	0.27	10.99	11.52	11.45	11.3	0.29
	A051															
	A052															
	A053															
	A054	0.852	0.852	0.885	0.86	0.02	1.96	1.72	1.86	1.85	0.12	0.94	1.02	0.959	0.97	0.04
	A057	20.577	19.944	20.346	20.3	0.32	19.662	20.391	21.098	20.4	0.72	22.899	23.192	22.368	22.8	0.42
	A059	9	9	9.2	9.07	0.12	10.7	10.6	10.9	10.7	0.15	10.8	10.5	10.6	10.6	0.15
	A060	12.856	12.785	12.102	12.6	0.42	16.525	15.116	15.768	15.8	0.71	17.38	17.41	17.829	17.5	0.25
	A061															
	A062															
	A065	13.20983	13.69506	12.96671	13.3	0.37	16.07201	15.68874	16.38411	16.0	0.35	16.68984	16.66612	16.20136	16.5	0.28
	A066	8.289	8.554	8.328	8.39	0.14	11.323	11.636	11.173	11.4	0.24	10.225	10.206	10.91	10.4	0.40
	A068															
	A070	10.376	10.805	10.392	10.5	0.24	14.57	13.447	13.994	14.0	0.56	13.731	13.276	13.157	13.4	0.30
	A071															
	A073	18.44234	13.75037	11.52192	14.6	3.53	14.42696	14.44845	12.2593	13.7	1.26	12.62068	11.71754	12.03952	12.1	0.46
	A075	9.579482	11.22349	9.418129	10.1	1.00	11.53174	12.62802	12.42536	12.2	0.58	12.25296	11.96229	11.90118	12.0	0.19
	A079	10.18	11.35	7.42	9.65	2.02	11.09	11.45	11.29	11.3	0.18	11.29	10.99	11.82	11.4	0.42
Community Results		Consensus Mean			10.0		Consensus Mean			13.2		Consensus Mean			12.4	
		Consensus Standard Deviation			1.76		Consensus Standard Deviation			1.88		Consensus Standard Deviation			1.74	
		Maximum			20.3		Maximum			20.4		Maximum			22.8	
		Minimum			0.86		Minimum			1.85		Minimum			0.97	
		N			34		N			35		N			34	

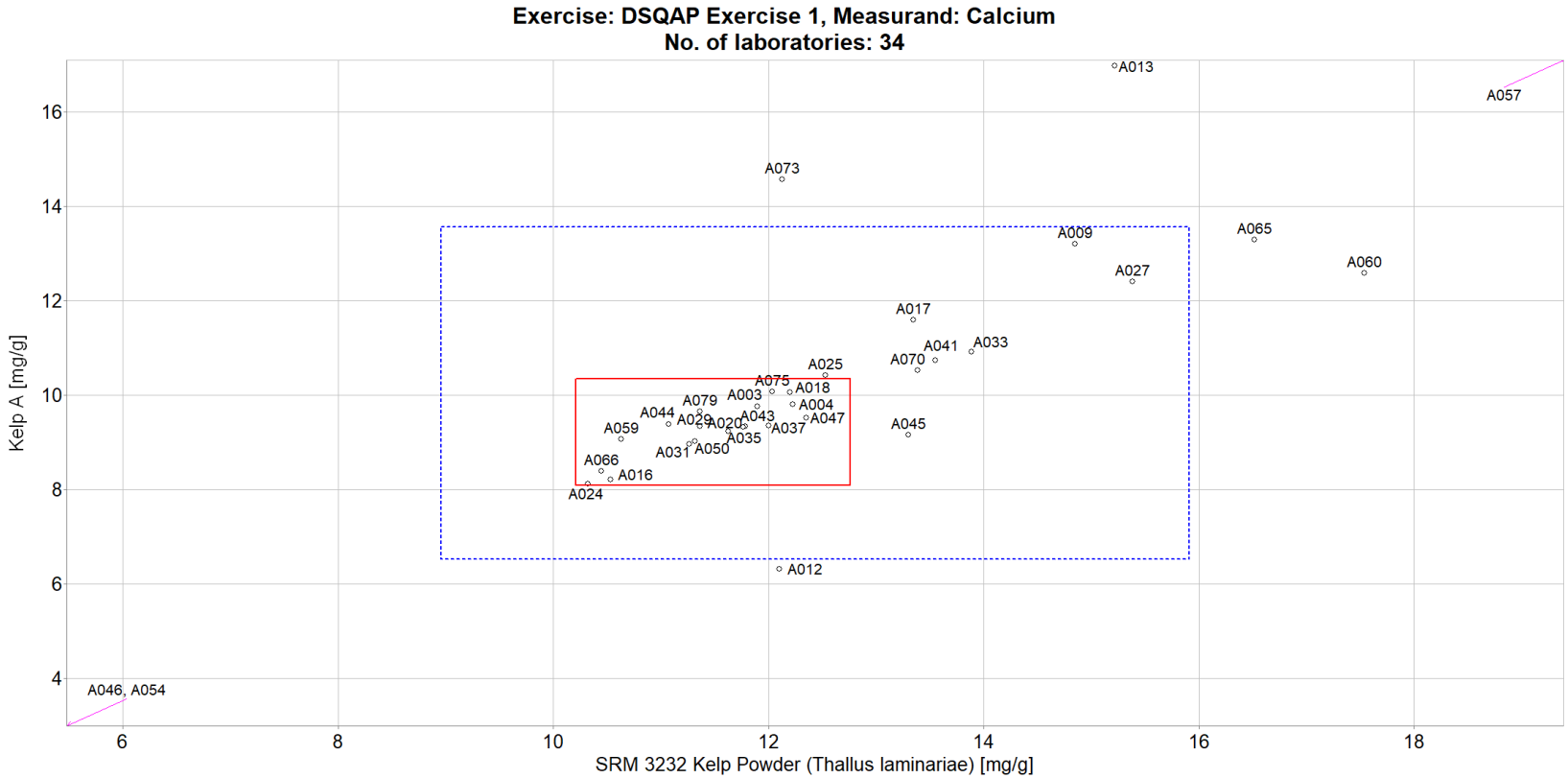

Fig. B-7. Laboratory means for calcium in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp A (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3232, is compared to the individual laboratory mean for a second sample, Kelp A. The solid red box represents the NIST range of tolerance for the two samples, SRM 3232 (x-axis) and Kelp A (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3232 (x-axis) and Kelp A (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

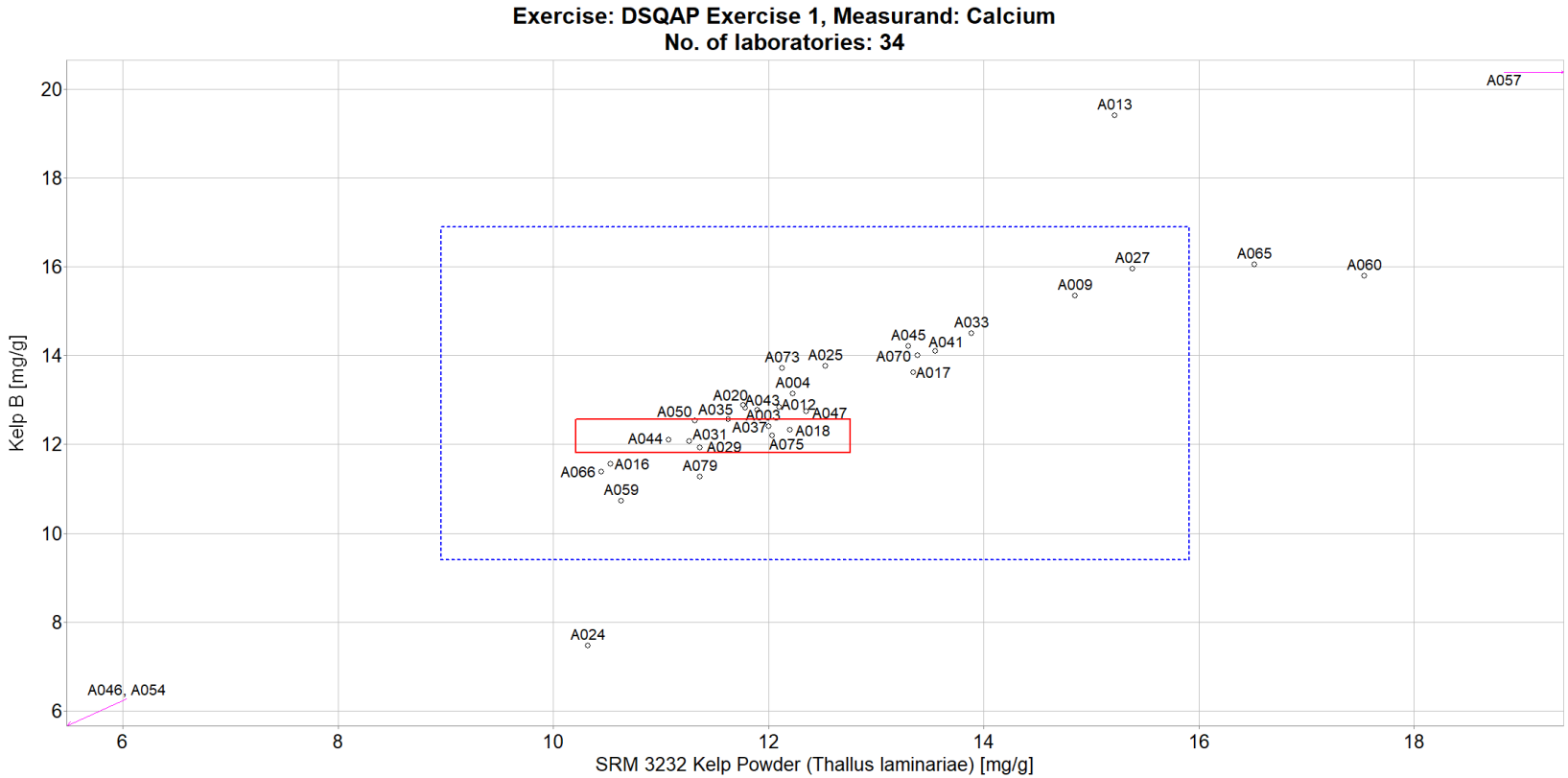

Fig. B-8. Laboratory means for calcium in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp B (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3232, is compared to the individual laboratory mean for a second sample, Kelp B. The solid red box represents the NIST range of tolerance for the two samples, SRM 3232 (x-axis) and Kelp B (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3232 (x-axis) and Kelp B (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

Table B-6. Data summary table for chromium in kelp.

Data highlighted in blue have been identified as outside the consensus range of tolerance and would be estimated to result in an unacceptable Z'_{comm} score, $|Z'_{comm}| > 2$. (Table continues to next page.)

		Chromium														
		Kelp A (ug/g)					Kelp B (ug/g)					SRM 3232 Kelp Powder (*Thallus laminariae*) (ug/g)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
Individual Results	Target				4.36	0.13				0.55	0.03				5.55	0.49
	A001															
	A002															
	A004	3.428	3.49	3.26	3.39	0.12	0.409	0.411	0.398	0.41	0.01	4.117	4.368	4.305	4.26	0.13
	A005															
	A008															
	A009	3.72	4.31	4.76	4.26	0.52	1.74	2.4	1.93	2.02	0.34	5.22	6.56	5.54	5.77	0.70
	A012	0.423	0.424	0.414	0.42	0.01	0.497	0.514	0.503	0.50	0.01	0.567	0.537	0.547	0.55	0.02
	A013	3.65878	3.67908	3.59219	3.64	0.05	0.32923	0.34263	0.33883	0.34	0.01	4.05395	4.11643	3.98852	4.05	0.06
	A015															
	A016	2.86	2.86	2.5	2.74	0.21	0.46	0.5	0.46	0.47	0.02	3.54	3.75	4.16	3.82	0.32
	A017	5.178	5.046	5.867	5.36	0.44	0.529	0.497	0.504	0.51	0.02	5.935	5.939	5.989	5.95	0.03
	A018	4.52	4.49	4.5	4.50	0.02	0.446	0.472	0.477	0.47	0.02	5.69	5.3		5.50	0.28
	A020	4.328	4.492	4.341	4.39	0.09	0.511	0.451	0.471	0.48	0.03	4.409	4.416	3.892	4.24	0.30
	A022															
	A024	2.3719	2.3697	2.3705	2.37	0.001	0.67038	0.67219	0.67167	0.67	0.001	3.0959	3.0911	3.0936	3.09	0.002
	A025	4.7	5.2	6.2	5.37	0.76	2.2	2.46	2.24	2.30	0.14	6.6	197.7	5.7	70.0	111
	A027	6.85	4.593	9.638	7.03	2.53	1.15	1.044	0.985	1.06	0.08	11.091	5.121	5.868	7.36	3.25
	A028															
	A029	4.22	4.15	4.1	4.16	0.06	0.56	0.55	0.527	0.55	0.02	6.53	5.16	5.83	5.84	0.69
	A031															
	A032															
	A033	2.67	2.61	2.8	2.69	0.10	0.47	0.48	0.49	0.48	0.01	2.94	3.3	3.26	3.17	0.20
	A035	4.152	4.41	4.346	4.30	0.13	0.559	0.586	0.571	0.57	0.01	4.419	4.554	4.502	4.49	0.07
	A037	2.53	2.79	2.72	2.68	0.13	0.463	0.504	0.501	0.49	0.02	4.57	4.02	4.42	4.34	0.28
	A039															
	A041	3.757	3.776	3.693	3.74	0.04	< 0.060	< 0.060	< 0.060			4.45	5.69	4.704	4.95	0.66
	A042	5.21	4.5	4.17	4.63	0.53						4.17	5.03	9.95	6.38	3.12
	A043	3.631	3.584	3.702	3.64	0.06	2.416	2.389	2.436	2.41	0.02	7.277	4.666	4.138	5.36	1.68

		Chromium														
		Kelp A (ug/g)					Kelp B (ug/g)					SRM 3232 Kelp Powder (*Thallus laminariae*) (ug/g)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
	A044	5.039	5.585	5.715	5.45	0.36	0.794	0.819	0.666	0.76	0.08	5.61	6.322	5.919	5.95	0.36
	A045	3.66	4.68	4.12	4.15	0.51	7.92	2.15	1.94	4.00	3.39	4.99	8.57	6.27	6.61	1.81
	A046	4.058	4	3.828	3.96	0.12	0.61	0.528	0.463	0.53	0.07	5.58	5.371	5.043	5.33	0.27
	A047	3.7	3.8		3.75	0.07	< 0.400	< 0.400				5	4.9		4.95	0.07
	A051															
	A052															
	A053															
	A054	1.5	1.5	1.56	1.52	0.03	0.362	0.35	0.337	0.35	0.01	1.85	1.94	2.09	1.96	0.12
	A057	4.53	4.5	4.41	4.48	0.06	0.53	0.51	0.51	0.52	0.01	5.2	5.29	4.5	5.00	0.43
	A059	4.43	4.63	4.53	4.53	0.10	0.42	0.53	0.55	0.50	0.07	5.5	4.93	5.55	5.33	0.34
	A060	2.2518	2.0846	2.7007	2.35	0.32	0.4129	0.3698	0.3717	0.38	0.02	2.806	2.8371	3.1221	2.92	0.17
	A061															
	A062															
	A065	3.83461	3.74903	3.77256	3.79	0.04	0.56489	0.58017	0.59155	0.58	0.01	4.8411	5.12007	4.21055	4.72	0.47
	A066															
	A068															
	A070	5.1407	5.1704	6.3754	5.56	0.70	0.9166	1.6535	1.8818	1.48	0.50	6.5607	13.4856	6.1386	8.73	4.13
	A071															
	A073	4.062	3.8587	4.822	4.25	0.51	0.4774	0.5297	0.4577	0.49	0.04	6.7187	5.7387	6.1287	6.20	0.49
	A075	3.1176	3.0636	3.4016	3.19	0.18	< 2.300	< 2.300	< 2.300			4.6542	4.3312	4.659	4.55	0.19
	A079	3.5	5	3.8	4.10	0.79	< 0.200	< 0.200	< 0.200			6.4	6.6	7.2	6.73	0.42
Community Results		Consensus Mean			3.92		Consensus Mean			0.51		Consensus Mean			4.98	
		Consensus Standard Deviation			1.19		Consensus Standard Deviation			0.16		Consensus Standard Deviation			1.53	
		Maximum			7.03		Maximum			4.00		Maximum			70.00	
		Minimum			0.42		Minimum			0.34		Minimum			0.55	
		N			31		N			26		N			31	

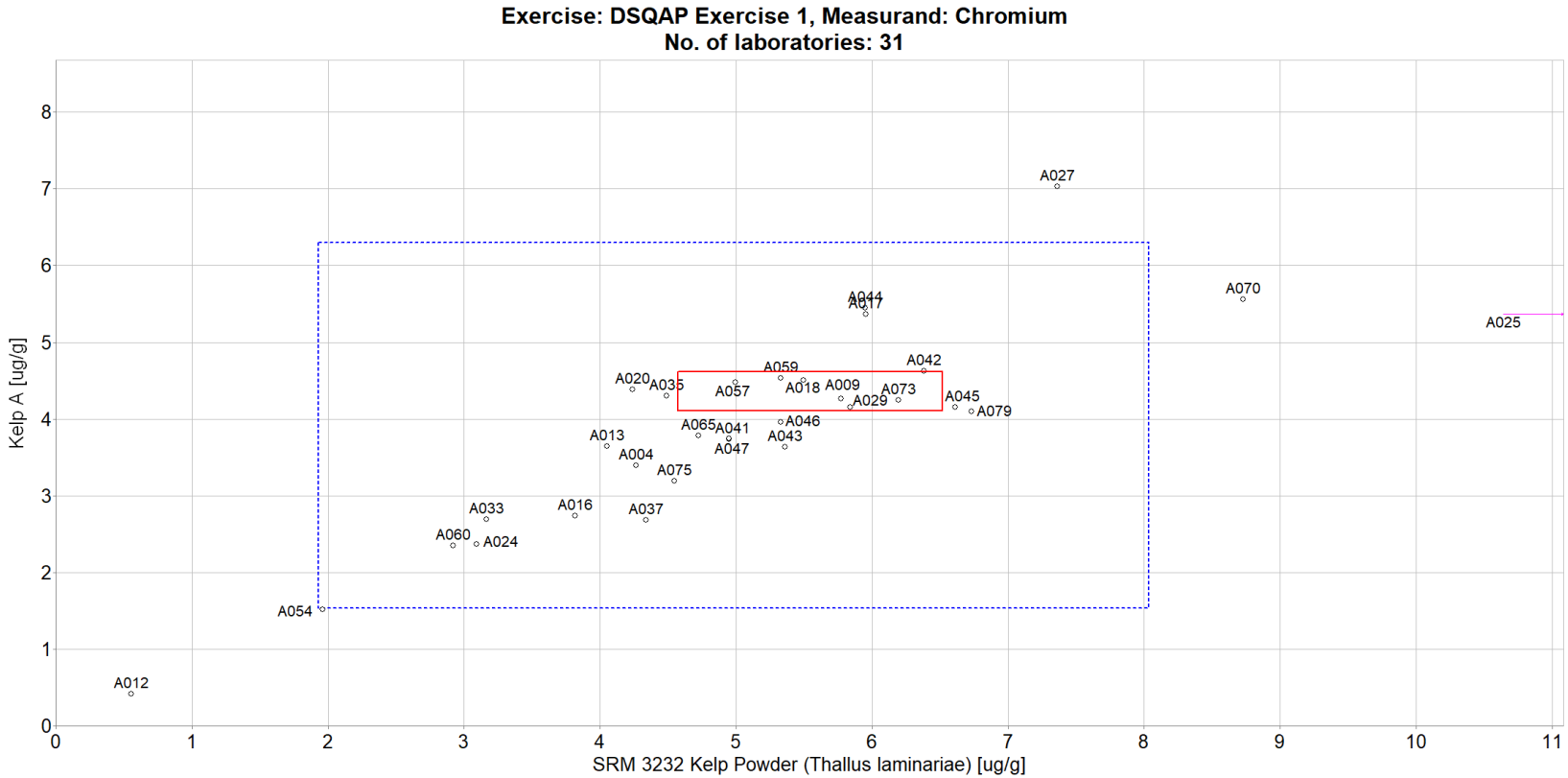

Fig. B-9. Laboratory means for chromium in SRM 3232 Kelp Powder (Thallus laminariae) and Kelp A (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3232, is compared to the individual laboratory mean for a second sample, Kelp A. The solid red box represents the NIST range of tolerance for the two samples, SRM 3232 (x-axis) and Kelp A (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3232 (x-axis) and Kelp A (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

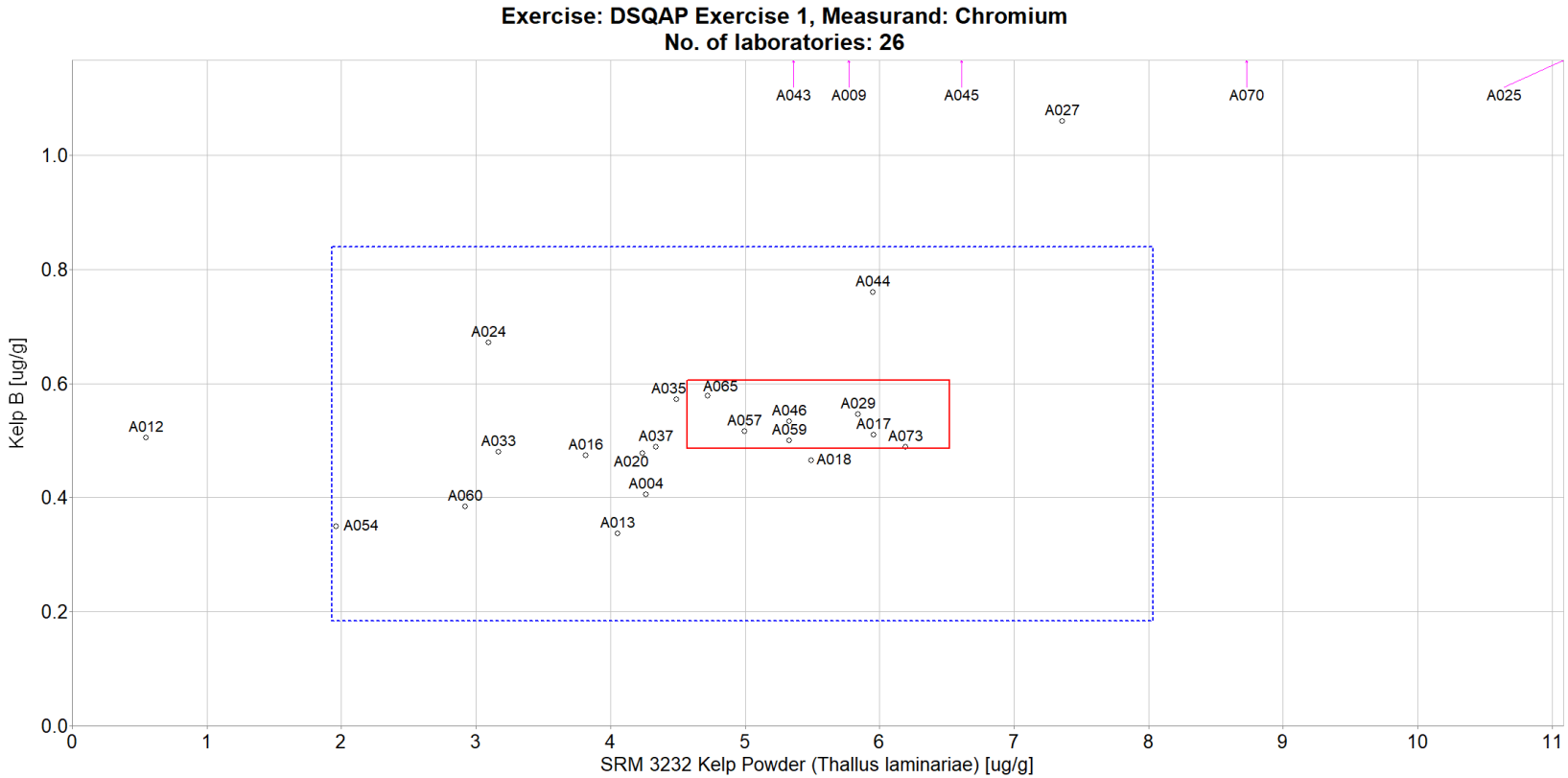

Fig. B-10. Laboratory means for chromium in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp B (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3232, is compared to the individual laboratory mean for a second sample, Kelp B. The solid red box represents the NIST range of tolerance for the two samples, SRM 3232 (x-axis) and Kelp B (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3232 (x-axis) and Kelp B (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

Table B-7. Data summary table for copper in kelp.

Data highlighted in blue have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable Z'_{comm} score, $|Z'_{comm}| > 2$. (Table continues to next page.)

		Copper														
		Kelp A (ug/g)					Kelp B (ug/g)					SRM 3232 Kelp Powder (*Thallus laminariae*) (ug/g)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
Individual Results	Target				1.34	0.06				1.12	0.05				3.63	0.08
	A001															
	A002															
	A003															
	A004															
	A005															
	A008															
	A009	< 0.010	< 0.010	< 0.010			< 0.010	< 0.010	< 0.010			< 0.010	0.27	0.04	0.16	0.16
	A012	1.16	1.16	1.16	1.16	0	1.03	1	1	1.01	0.02	3.52	3.44	3.6	3.52	0.08
	A013	1.0098	0.9763	0.9379	0.97	0.04	0.8343	0.83677	0.8276	0.83	0.005	2.6241	2.9353	2.8723	2.81	0.16
	A015															
	A016	1.08	1.06	1.07	1.07	0.01	1	1.03	0.99	1.01	0.02	3.07	3.24	3.22	3.18	0.09
	A017	1.99	1.88	2.13	2.00	0.13	1.47	1.45	1.34	1.42	0.07	3.77	3.89	3.84	3.83	0.06
	A018	1.34	1.4	1.4	1.38	0.03	0.9	0.918	0.945	0.92	0.02	2.98	2.97		2.98	0.01
	A020															
	A022															
	A024	< 0.013	< 0.013	< 0.013			< 0.013	< 0.013	< 0.013			1.5436	1.5439	1.543	1.54	0.00
	A025											< 0.001	0.0098	< 0.001	0.01	
	A027	3.507	4.593	1.469	3.19	1.59	0.974	1.032	0.985	1.00	0.03	3.339	3.219	3.787	3.45	0.30
	A028															
	A029	1.29	1.24	1.29	1.27	0.03	1.16	1.17	1.09	1.14	0.04	3.61	3.5	3.6	3.57	0.06
	A031	4	3	3	3.33	0.58	6	4	7	5.67	1.53	5	8	6	6.33	1.53
	A032															
	A033	1.73	1.92	1.94	1.86	0.12	1.51	1.46	1.45	1.47	0.03	3.33	3.36	3.25	3.31	0.06
	A035	1.24	1.28	1.29	1.27	0.03	1.03	1.06	1.08	1.06	0.03	4.2	3.77	3.73	3.90	0.26
	A037	1.05	1.02	1.04	1.04	0.02	1	0.948	0.917	0.96	0.04	2.98	2.99	2.77	2.91	0.12
	A039															
	A041	0.972	0.972	0.969	0.97	0.002	0.848	0.812	0.861	0.84	0.03	3.068	2.949	2.944	2.99	0.07
	A042	0.76	0.76	0.83	0.78	0.04	0.84	0.85	0.81	0.83	0.02	2.24	2.26	2.11	2.20	0.08

		Copper														
		Kelp A (ug/g)					Kelp B (ug/g)					SRM 3232 Kelp Powder (*Thallus laminariae*) (ug/g)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
	A043	2.56	3.71	7.34	4.54	2.49	6.3	5.95	6.33	6.19	0.21	7.48	7.08	6.98	7.18	0.26
	A044	1.86	1.68	1.7	1.75	0.10	1.41	1.44	1.45	1.43	0.02	3.67	4.17	3.61	3.82	0.31
	A045	0.29	0.69	1.35	0.78	0.54	1.56	4.27	1.43	2.42	1.60	3.11	4.16	3.6	3.62	0.53
	A046	1.355	1.248	1.286	1.30	0.05	1.401	1.212	1.111	1.24	0.15	3.678	3.401	3.335	3.47	0.18
	A047	1.3	1.3		1.3	0	1.1	1.1		1.1	0	3.6	3.5		3.55	0.07
	A050															
	A051															
	A052															
	A053															
	A054	1.33	1.63	1.59	1.52	0.16	1.49	1.37	1.3	1.39	0.10	3.74	3.98	3.83	3.85	0.12
	A057	1.39	1.35	1.35	1.36	0.02	1.15	1.21	1.19	1.18	0.03	3.95	3.86	3.94	3.92	0.05
	A059	2	2	2	2	0	2	2	1	1.67	0.58	4	4	4	4	0
	A060	< 2.000	< 2.000	< 2.000			< 2.000	< 2.000	< 2.000			3.6416	4.1353	3.9762	3.92	0.25
	A061															
	A062															
	A065	1.15	1.22	1.07	1.15	0.08	1.03	1.05	1.07	1.05	0.02	3.23	3.24	3.13	3.20	0.06
	A066															
	A068															
	A070	1.352	1.1355	1.421	1.30	0.15	1.185	1.4323	1.3514	1.32	0.13	3.893	3.9504	4.403	4.08	0.28
	A071															
	A073															
	A075	1.9304	< 1.949	< 1.949	1.93		< 1.756	2.058	< 1.756	2.06		3.7549	3.7419	3.9733	3.82	0.13
	A079	< 120.0	< 120.0	< 120.0			< 35.0	< 35.0	< 35.0			< 40.0	< 40.0	< 40.0		
Community Results		Consensus Mean			1.37		Consensus Mean			1.18		Consensus Mean			3.45	
		Consensus Standard Deviation			0.49		Consensus Standard Deviation			0.33		Consensus Standard Deviation			0.77	
		Maximum			4.54		Maximum			6.19		Maximum			7.18	
		Minimum			0.78		Minimum			0.83		Minimum			0.01	
		N			23		N			23		N			27	

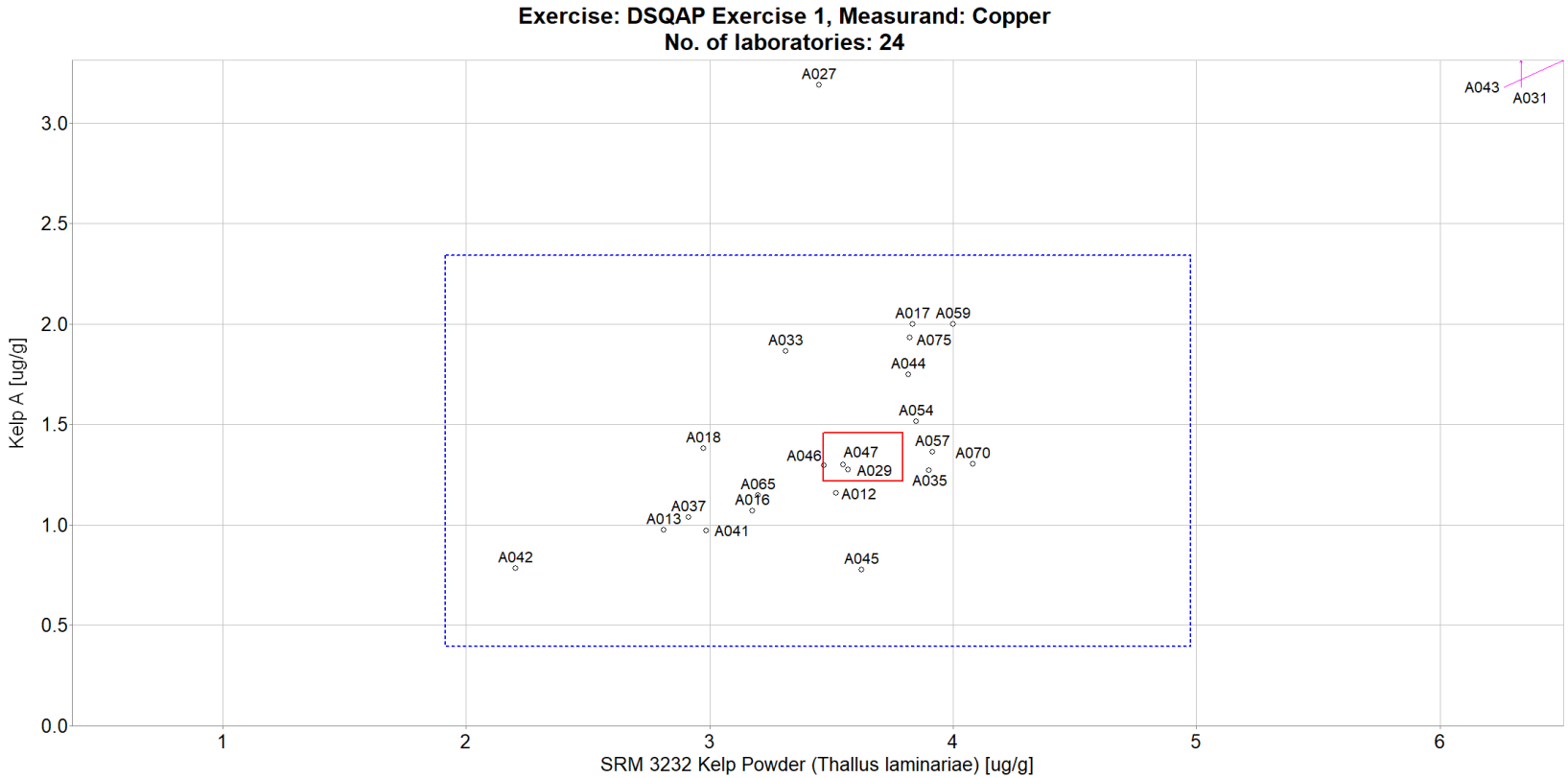

Fig. B-11. Laboratory means for copper in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp A (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3232, is compared to the individual laboratory mean for a second sample, Kelp A. The solid red box represents the NIST range of tolerance for the two samples, SRM 3232 (x-axis) and Kelp A (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3232 (x-axis) and Kelp A (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

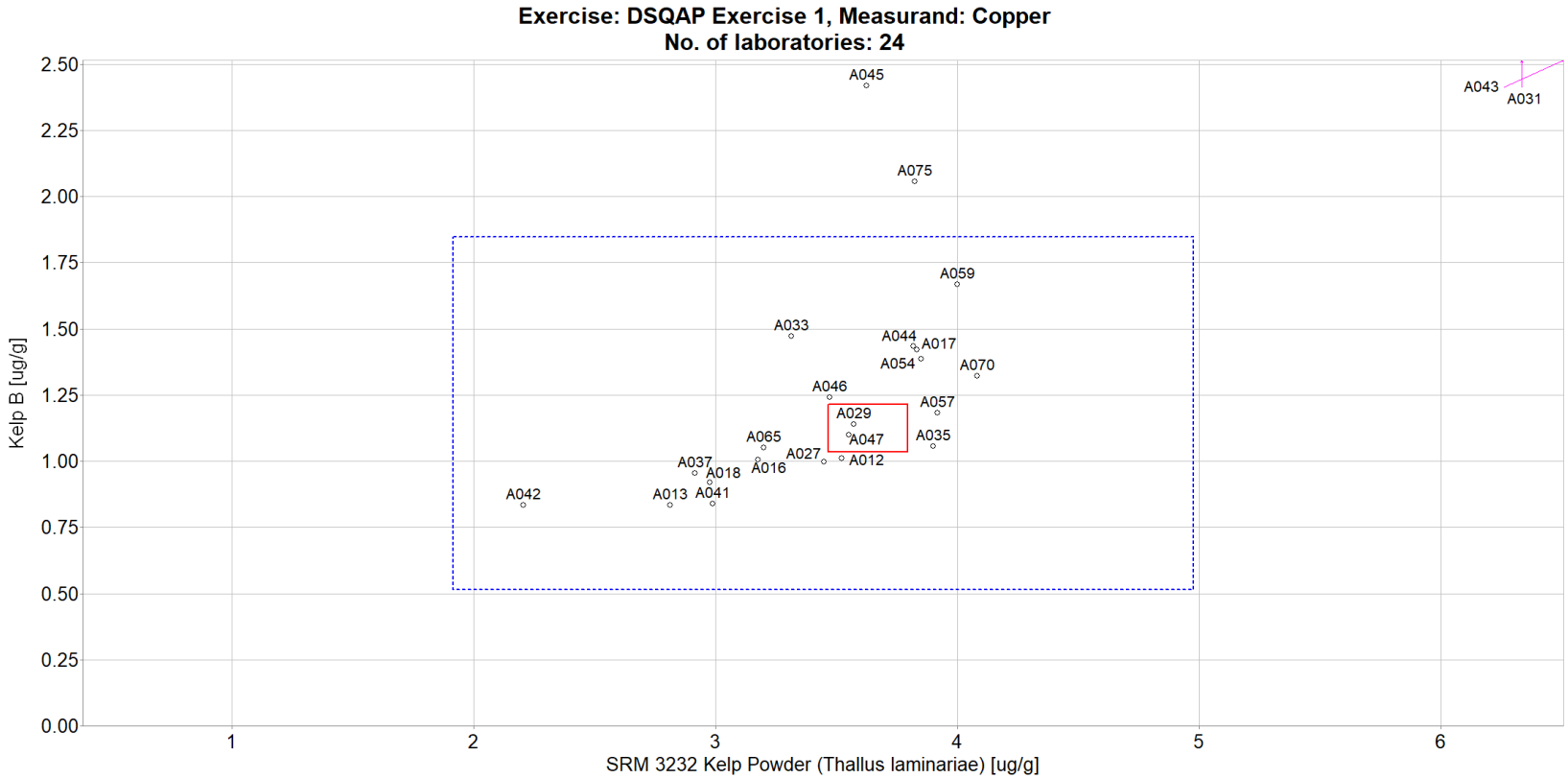

Fig. B-12. Laboratory means for copper in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp B (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3232, is compared to the individual laboratory mean for a second sample, Kelp B. The solid red box represents the NIST range of tolerance for the two samples, SRM 3232 (x-axis) and Kelp B (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3232 (x-axis) and Kelp B (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

Table B-8. Data summary table for iodine in kelp.

Data highlighted in blue have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable Z'_{comm} score, $|Z'_{comm}| > 2$. (Table continues to next page.)

		Iodine														
		Kelp A (ug/g)					Kelp B (ug/g)					SRM 3232 Kelp Powder (*Thallus laminariae*) (ug/g)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
Individual Results	Target														884	82.4
	A001															
	A002															
	A005															
	A012	1640	2620	2740	2333	603	935	889	930	918	25.2	699	817	708	741	65.7
	A013															
	A015															
	A017	2056	1982	2031	2023	37.6	966.8	936.4	908.1	937	29.4	820	796	835	817	19.7
	A018															
	A019	1800	1841	1841	1827	23.7	810	868	865	848	32.7	786	735	758	760	25.5
	A020															
	A022															
	A025															
	A027															
	A028															
	A029															
	A031															
	A032															
	A033	2139.9	2100.4	2101.1	2114	22.6	906	919	886.7	904	16.3	741.8	737.8	682	721	33.4
	A035	2450	2270	2290	2337	98.7	1070	1070	1040	1060	17.3	933	918	960	937	21.3
	A037	1970	2230	2060	2087	132	606	627	526	586	53.3	597	697	634	643	50.6
	A041															
	A042	2170	2115	2112	2132	32.7	839	963	814	872	79.8	767	765	780	771	8.1
	A043															
	A044	2518.69	2113.941	1923.534	2185	304	955.345	968.677	988.894	971	16.9	886.87	815.796	963.348	889	73.8
	A051															
	A053															
	A054															

		Iodine														
		Kelp A (ug/g)					Kelp B (ug/g)					SRM 3232 Kelp Powder (*Thallus laminariae*) (ug/g)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
	A057															
	A058	2175.337	2165.193	2112.904	2151	33.5	916.682	941.12	930.501	929	12.3	849.88	863.221	843.898	852	9.9
	A060	2076	2077	2054	2069	13.0	993	991	996	993	2.52	838	868	851	852	15.0
	A061															
	A062															
	A065	1588.841	1559.961	1570.971	1573	14.6	529.6211	553.92	639.8242	574	57.9	495.2	492.4294	464.7418	484	16.8
	A066															
	A068															
	A071															
	A073															
	A075															
	A079	1870	1720	1760	1783	77.7	788	772	757	772	15.5	739	742	706	729	20.0
Community Results		Consensus Mean			2059		Consensus Mean			880		Consensus Mean			775	
		Consensus Standard Deviation			264		Consensus Standard Deviation			134		Consensus Standard Deviation			125	
		Maximum			2337		Maximum			1060		Maximum			937	
		Minimum			1573		Minimum			574		Minimum			484	
		N			12		N			12		N			12	

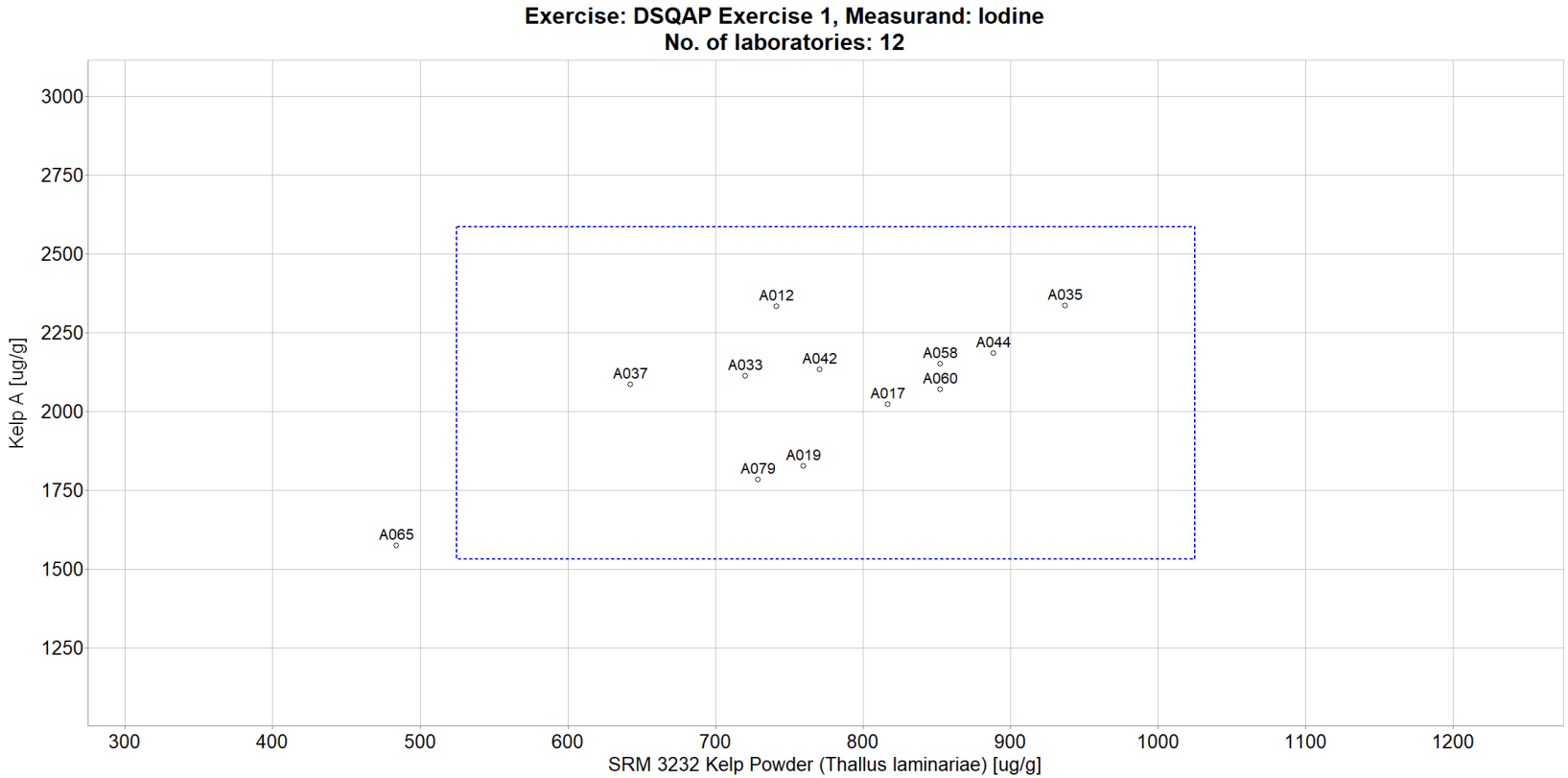

Fig. B-13. Laboratory means for iodine in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp A (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3232, is compared to the individual laboratory mean for a second sample, Kelp A. The dotted blue box represents the consensus range of tolerance for SRM 3232 (x-axis) and Kelp A (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

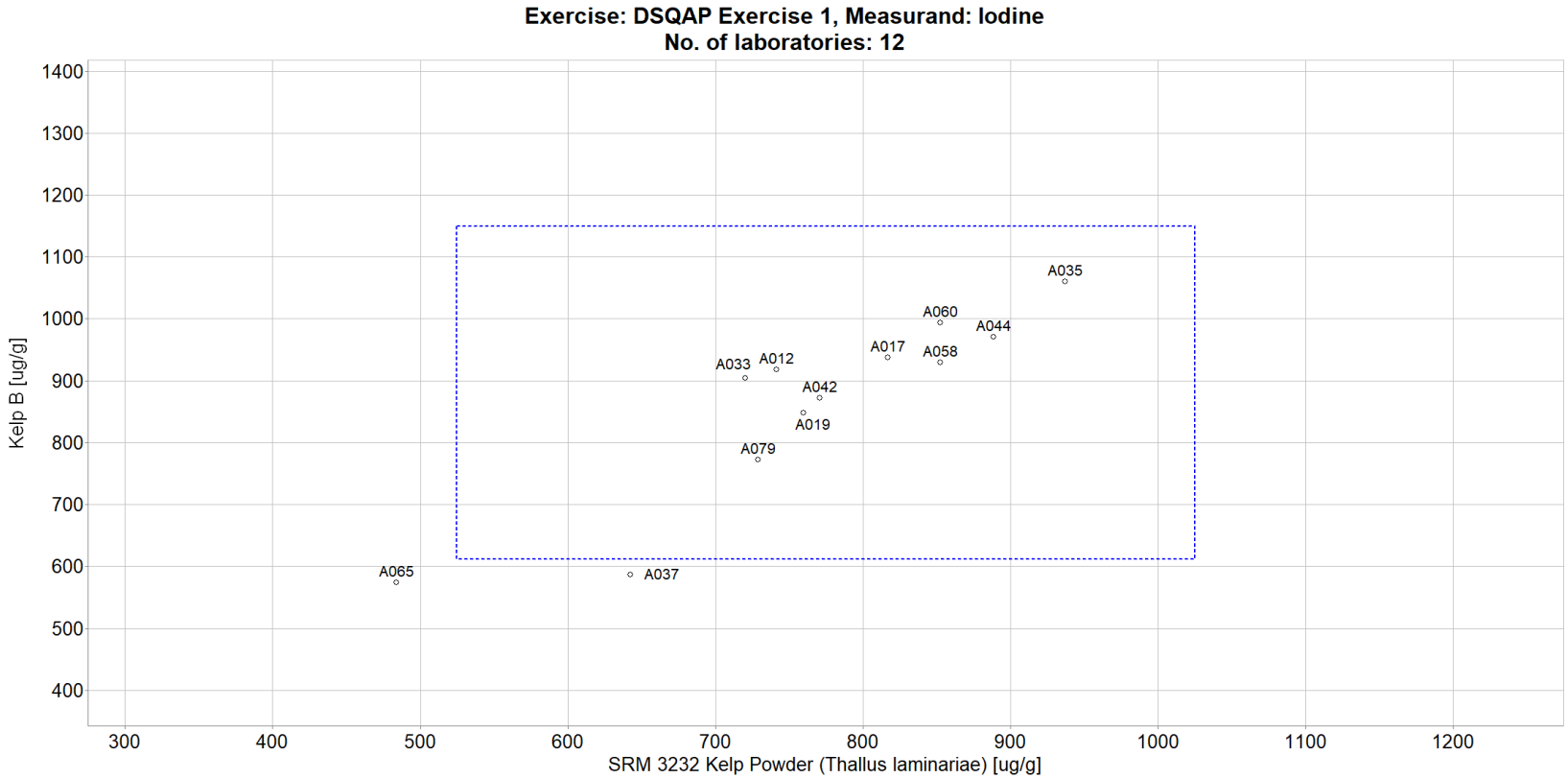

Fig. B-14. Laboratory means for iodine in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp B (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3232, is compared to the individual laboratory mean for a second sample, Kelp B. The dotted blue box represents the consensus range of tolerance for SRM 3232 (x-axis) and Kelp B (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

Table B-9. Data summary table for lead in kelp.

Data highlighted in blue have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable Z'_{comm} score, $|Z'_{comm}| > 2$. (Table continues to next page.)

		Lead														
		Kelp A (ug/g)					Kelp B (ug/g)					SRM 3232 Kelp Powder (*Thallus laminariae*) (ug/g)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
Individual Results	Target				0.59	0.02				0.41	0.01				0.97	0.04
	A001															
	A002															
	A003															
	A004	0.582	0.563	0.62	0.59	0.03	0.421	0.428	0.45	0.43	0.02	0.929	1.078	0.945	0.98	0.08
	A005	0.613	0.601	0.615	0.61	0.01	0.422	0.428	0.42	0.42	0.004	0.935	0.935	0.945	0.94	0.01
	A008															
	A009	0.84	< 0.010	< 0.010	0.84		< 0.010	< 0.010	< 0.010			< 0.010	< 0.010	< 0.010		
	A012	0.588	0.613	0.61	0.60	0.01	0.42	0.414	0.397	0.41	0.01	0.957	0.961	0.946	0.95	0.01
	A013	0.5759	0.582	0.5669	0.57	0.01	0.3974	0.4056	0.3993	0.40	0.004	0.8229	0.8615	0.8846	0.86	0.03
	A015															
	A016	0.54	0.57	0.54	0.55	0.02	0.45	0.43	0.56	0.48	0.07	0.84	0.89	0.84	0.86	0.03
	A017	0.751	0.675	0.77	0.73	0.05	0.412	0.428	0.421	0.42	0.01	2.07	0.99	0.93	1.33	0.64
	A018	0.474	0.464	0.474	0.47	0.01	0.323	0.344	0.343	0.34	0.01	0.734	0.737		0.74	0.002
	A019	0.6	0.45	0.45	0.50	0.09	0.37	0.28	0.33	0.33	0.05	0.83	0.75	0.76	0.78	0.04
	A020	0.538	0.546	0.515	0.53	0.02	0.373	0.392	0.396	0.39	0.01	0.825	0.827	0.835	0.83	0.01
	A022															
	A024	< 0.051	< 0.051	< 0.051			< 0.051	< 0.051	< 0.051			< 0.051	< 0.051	< 0.051		
	A025	< 0.001	0.12	< 0.001	0.12		< 0.001	< 0.001	0.0619	0.06		0.3322	0.3291	0.4113	0.36	0.05
	A027	0.895	0.441	0.398	0.58	0.28	0.33	0.362	0.358	0.35	0.02	0.705	0.762	0.645	0.70	0.06
	A028															
	A029	0.545	0.524	0.518	0.53	0.01	0.392	0.389	0.384	0.39	0.004	0.814	0.827	0.879	0.84	0.03
	A031															
	A032															
	A033	0.54	0.61	0.6	0.58	0.04	0.42	0.45	0.41	0.43	0.02	0.86	0.83	0.88	0.86	0.03
	A035	0.55	0.55	0.56	0.55	0.01	0.35	0.36	0.42	0.38	0.04	0.91	0.92	0.94	0.92	0.02
	A037	0.58	0.561	0.572	0.57	0.01	0.376	0.396	0.399	0.39	0.01	0.873	0.865	0.888	0.88	0.01
	A039															

		Lead														
		Kelp A (ug/g)					Kelp B (ug/g)					SRM 3232 Kelp Powder (*Thallus laminariae*) (ug/g)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
	A041	0.57	0.553	0.567	0.56	0.01	0.39	0.389	0.405	0.39	0.01	0.862	0.842	0.844	0.85	0.01
	A042	0.58	0.58	0.62	0.59	0.02	0.43	0.44	0.42	0.43	0.01	0.88	0.89	0.8	0.86	0.05
	A043	0.71	0.71	0.71	0.71	0.00	0.49	0.49	0.49	0.49	0	1.02	1.03	1.07	1.04	0.03
	A044	0.74	0.69	0.7	0.71	0.03	0.41	0.42	0.43	0.42	0.01	0.91	0.91	1.12	0.98	0.12
	A045	< 1.000	< 1.000	< 1.000			< 1.000	< 1.000	< 1.000			< 1.000	< 1.000	< 1.000		
	A046	0.451	0.424	0.428	0.43	0.01	0.384	0.388	0.357	0.38	0.02	0.729	0.69	0.686	0.70	0.02
	A047	0.57	0.59		0.58	0.01	0.41	0.42		0.42	0.01	0.98	0.97		0.98	0.01
	A050	0.469	0.517	0.509	0.50	0.03	0.394	0.402	0.394	0.40	0.005	0.807	0.873	0.833	0.84	0.03
	A051															
	A052															
	A053															
	A054	0.485	0.482	0.478	0.48	0.004	0.36	0.357	0.36	0.36	0.002	0.727	0.72	0.746	0.73	0.01
	A057	0.58	0.56	0.57	0.57	0.01	0.41	0.4	0.41	0.41	0.01	0.89	0.89	0.88	0.89	0.01
	A058	0.565	0.543	0.561	0.56	0.01	0.455	0.446	0.499	0.47	0.03	0.945	1.109	0.967	1.01	0.09
	A060	0.469	0.563	0.465	0.50	0.06	0.392	0.384	0.355	0.38	0.02	0.743	0.816	1.02	0.86	0.14
	A061															
	A062															
	A065	0.64	0.67	0.76	0.69	0.06	0.48	0.48	0.46	0.47	0.01	0.94	0.96	0.91	0.94	0.03
	A066															
	A068															
	A070	0.606	0.618	0.613	0.61	0.01	0.3851	0.4361	0.4268	0.42	0.03	1.217	0.9555	0.9652	1.05	0.15
	A071															
	A073	0.553	0.5323	0.5263	0.54	0.01	0.385	0.3916	0.378	0.38	0.01	0.939	0.9563	0.916	0.94	0.02
	A075	0.46836	0.46631	0.4672	0.47	0.001	0.33692	0.42138	0.33348	0.36	0.05	0.73582	0.71961	0.69651	0.72	0.02
	A079															
Community Results		Consensus Mean			0.57		Consensus Mean			0.40		Consensus Mean			0.88	
		Consensus Standard Deviation			0.09		Consensus Standard Deviation			0.05		Consensus Standard Deviation			0.12	
		Maximum			0.84		Maximum			0.49		Maximum			1.33	
		Minimum			0.12		Minimum			0.06		Minimum			0.36	
		N			29		N			29		N			30	

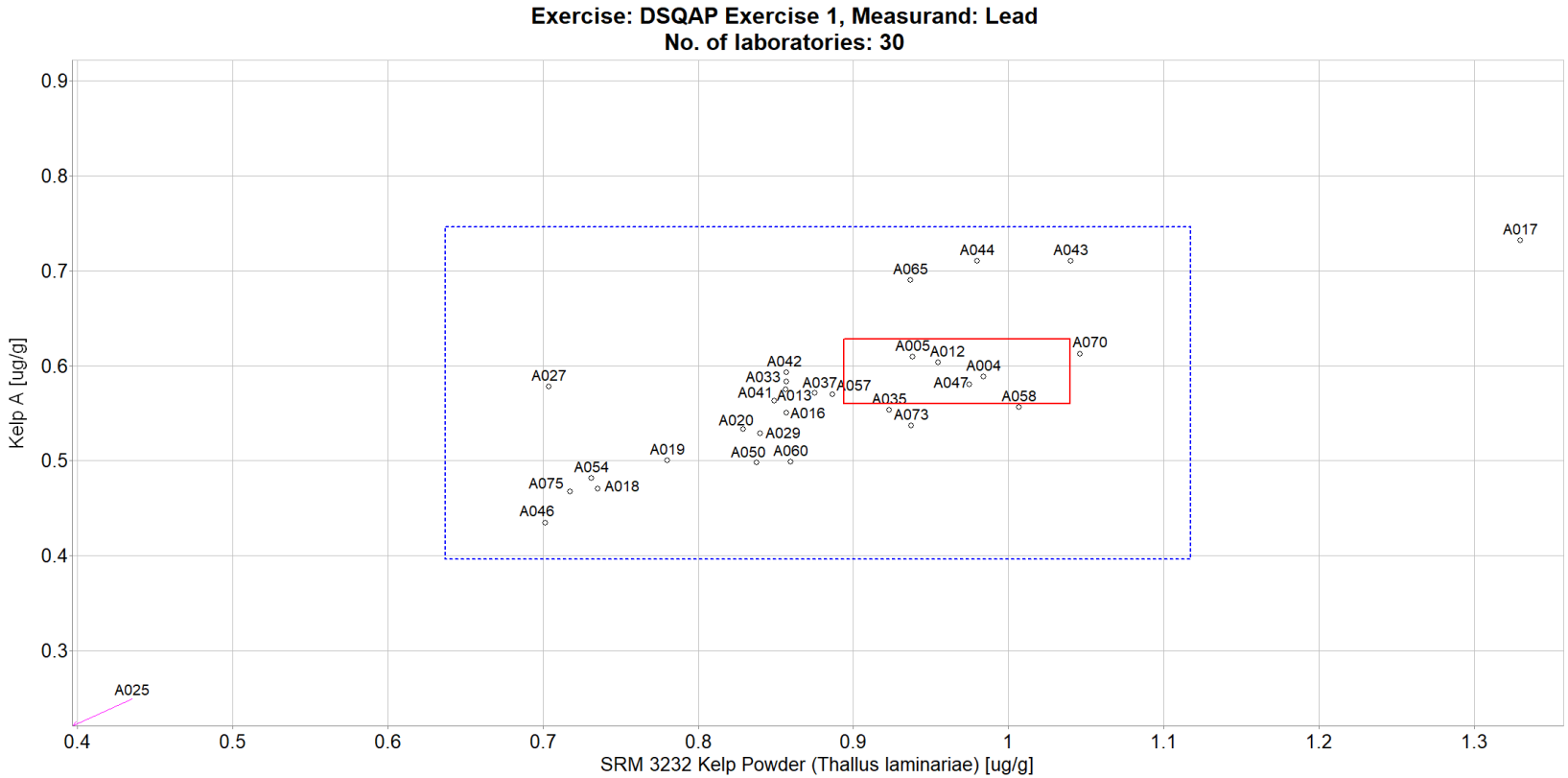

Fig. B-15. Laboratory means for lead in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp A (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3232, is compared to the individual laboratory mean for a second sample, Kelp A. The solid red box represents the NIST range of tolerance for the two samples, SRM 3232 (x-axis) and Kelp A (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3232 (x-axis) and Kelp A (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

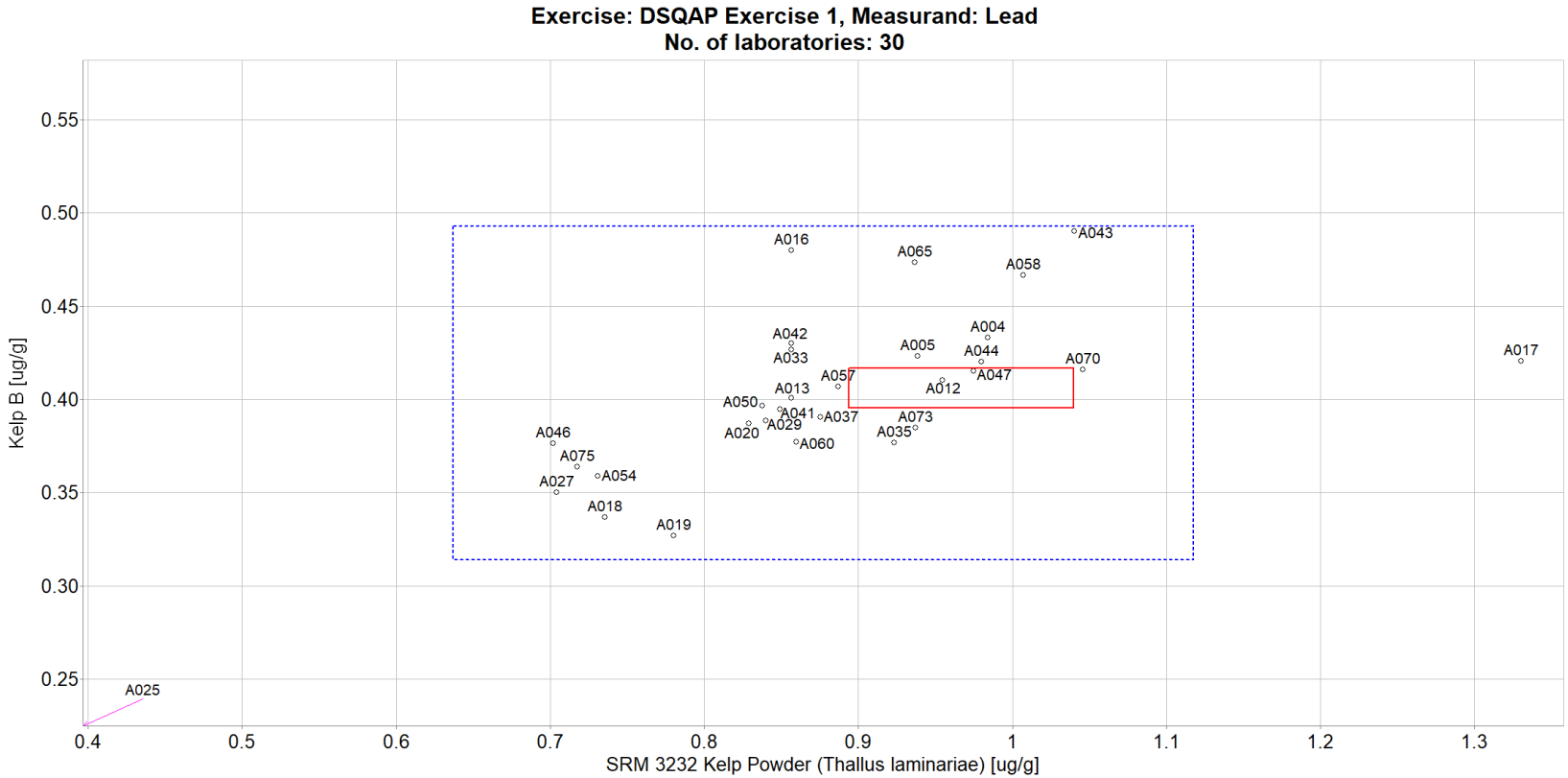

Fig. B-16. Laboratory means for lead in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp B (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3232, is compared to the individual laboratory mean for a second sample, Kelp B. The solid red box represents the NIST range of tolerance for the two samples, SRM 3232 (x-axis) and Kelp B (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3232 (x-axis) and Kelp B (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

Table B-10. Data summary table for magnesium in kelp.

Data highlighted in blue have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable Z'_{comm} score, $|Z'_{comm}| > 2$. (Table continues to next page.)

		Magnesium														
		Kelp A (ug/g)					Kelp B (ug/g)					SRM 3232 Kelp Powder (*Thallus laminariae*) (ug/g)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
Individual Results	Target				6881	917				5287	2254				5743	169
	A001															
	A002															
	A003	6629	6828	6622	6693	117	8053	7827	7729	7870	166	5612	5588	5561	5587	26
	A004	7528	7323	7321	7391	119	8580	8471	8642	8564	87	5986	5804	5902	5897	91
	A005															
	A009	6423.92	6679.51	6895.15	6666	236	7550.85	7075.24	7041.76	7223	285	5018.72	5264.74	4798.93	5027	233
	A012	7510	7790	7660	7653	140	9170	8800	8730	8900	236	6290	6330	6350	6323	31
	A013	7336.491	7225.539	7249.236	7270	58	8196.924	8282.988	8364.787	8282	84	5393.473	5920.027	5609.256	5641	265
	A015															
	A016	6460	6520	6440	6473	42	7860	7860	7890	7870	17	5290	5270	5330	5297	31
	A017	7537	7819	7424	7593	203	8460	8341	8665	8489	164	5835	5967	5709	5837	129
	A018	7190	7350	7350	7297	92	7630	7980	7740	7783	179	5540	5530		5535	7
	A020	7047	7083	6854	6995	123	8470	8717	8641	8609	127	5967	6203	6047	6072	120
	A022															
	A024	6750.68	6750.677	6750.681	6751	0.002	7954.63	7954.627	7954.629	7955	0.002	5336.512	5336.513	5336.512	5337	0.001
	A025	8212	7503	7803	7839	356	8770	8522	8577	8623	130	6211	6188	6014	6138	108
	A027	7560.314	7015.268	7688.206	7421	357	9031.768	8831.917	9090.913	8985	136	5868.531	6347.173	6016.061	6077	245
	A028															
	A029	6280	7310	6390	6660	566	7070	7190	7520	7260	233	5160	5110	5380	5217	144
	A031	7300	7300	7300	7300	0	8200	8300	8400	8300	100	5800	5800	5800	5800	0
	A032															
	A033	7918.9	7654.6	7628.4	7734	161	9482	9651.1	9624.3	9586	91	6697.9	6465.7	6356.2	6507	174
	A035	7467	7421	7314	7401	79	8802	8693	8677	8724	68	6015	5859	5986	5953	83
	A037	7310	7730	7260	7433	258	8380	8370	8510	8420	78	6450	6220	6230	6300	130
	A039															
	A041	7590.067	7373.93	7342.246	7435	135	8710.899	8887.673	8847.87	8815	93	6131.284	6100.686	6184.443	6139	42
	A042															
	A043	6990.678	7026.246	6933.327	6983	47	8249.442	8185.219	7965.576	8133	149	5613.079	5506.324	5650.904	5590	75
	A044	6186.98	7426.958	7287.762	6967	679	8543.072	8984.942	9141.968	8890	311	5209.877	5254.416	5224.067	5229	23
	A045	6456.41	6732.37	6891.46	6693	220	8545.02	8409.61	9759.65	8905	743	5987.56	5944.48	7353.36	6428	801

		Magnesium														
		Kelp A (ug/g)					Kelp B (ug/g)					SRM 3232 Kelp Powder (*Thallus laminariae*) (ug/g)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
	A046	8293.064	7329.045	7576.322	7733	501	10881.67	10533.84	9270.316	10229	848	6913.498	6050.507	6007.974	6324	511
	A047	6990	6990		6990	0	8260	8300		8280	28	5740	5730		5735	7
	A050	7371	7464	7402	7412	47	8773	8705	8692	8723	44	5709	5974	5939	5874	144
	A051															
	A052															
	A053															
	A054	3060	3020	3100	3060	40	6420	6350	6110	6293	163	2170	2380	2230	2260	108
	A057	7760	7135	7383	7426	315	8346	8254	8350	8317	54	5837	5781	5706	5775	66
	A059	7600	7400	7400	7467	115	6770	7060	6700	6843	191	5440	5500	5600	5513	81
	A060	7441	7402	7366	7403	38	9362	8465	8524	8784	502	6000	6067	6099	6055	51
	A061															
	A062															
	A065	7159.803	7269.812	6828.867	7086	230	8272.589	8246.784	8410.526	8310	88	5621.809	5676.195	5450.999	5583	118
	A066	6372	6342	6452	6389	57	7638	7583	7475	7565	83	5320	5258	5598	5392	181
	A068															
	A070	6932	7456	7507	7298	318	9173	8687	9092	8984	260	6179	6260	6167	6202	51
	A071															
	A073	7745.27	6929.98	6888.96	7188	483	7561.26	7650.62	6877.88	7363	423	5163.42	5080.03	5074.1	5106	50
	A075	7256.661	6707.581	7145.224	7036	290	7572.316	8347.826	8209.519	8043	414	5930.83	5761.147	5803.119	5832	88
	A079															
Community Results		Consensus Mean			7196		Consensus Mean			8310		Consensus Mean			5793	
		Consensus Standard Deviation			445		Consensus Standard Deviation			714		Consensus Standard Deviation			470	
		Maximum			7839		Maximum			10229		Maximum			6507	
		Minimum			3060		Minimum			6293		Minimum			2260	
		N			33		N			33		N			33	

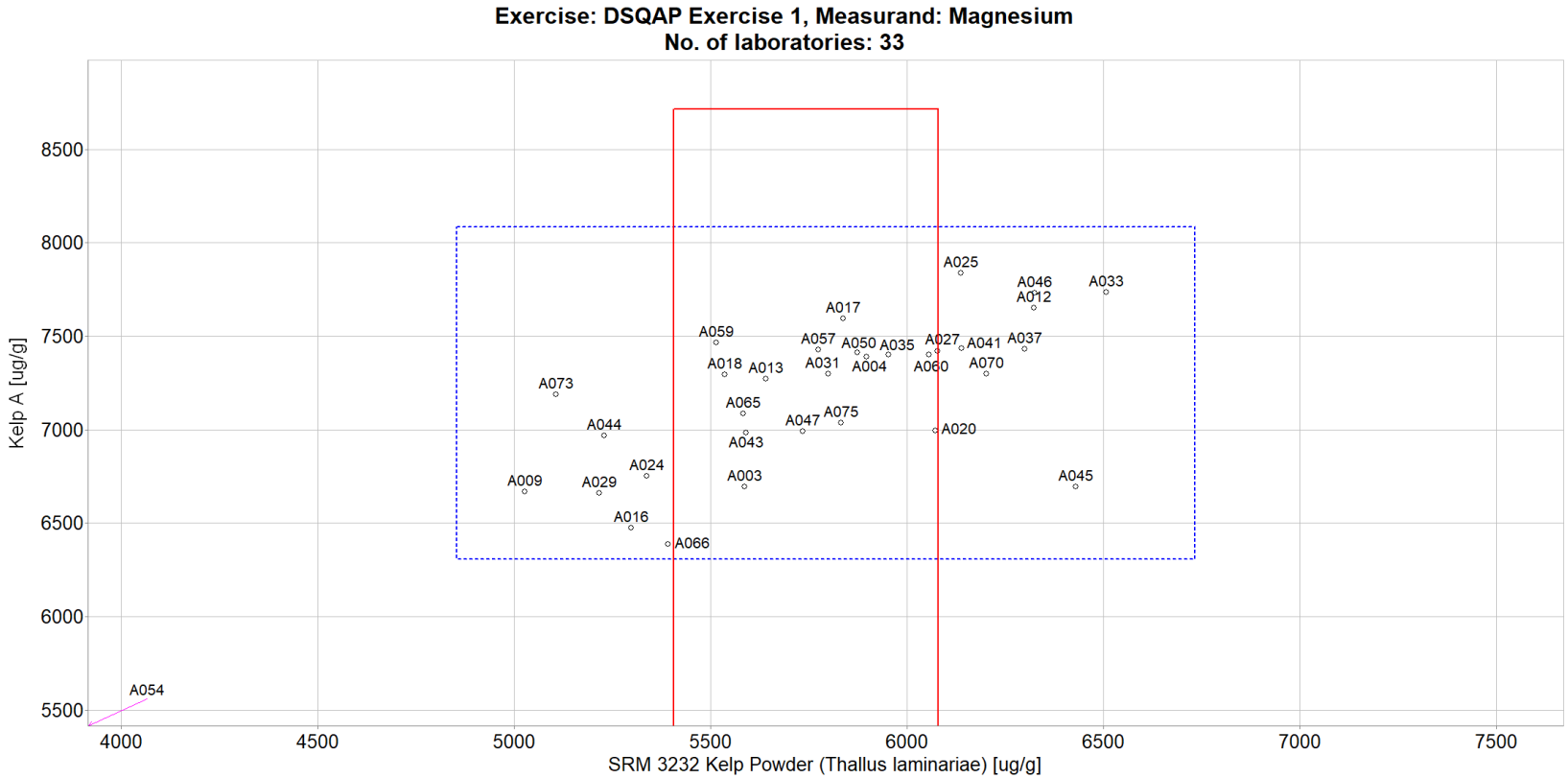

Fig. B-17. Laboratory means for magnesium in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp A (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3232, is compared to the individual laboratory mean for a second sample, Kelp A. The solid red box represents the NIST range of tolerance for the two samples, SRM 3232 (x-axis) and Kelp A (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3232 (x-axis) and Kelp A (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

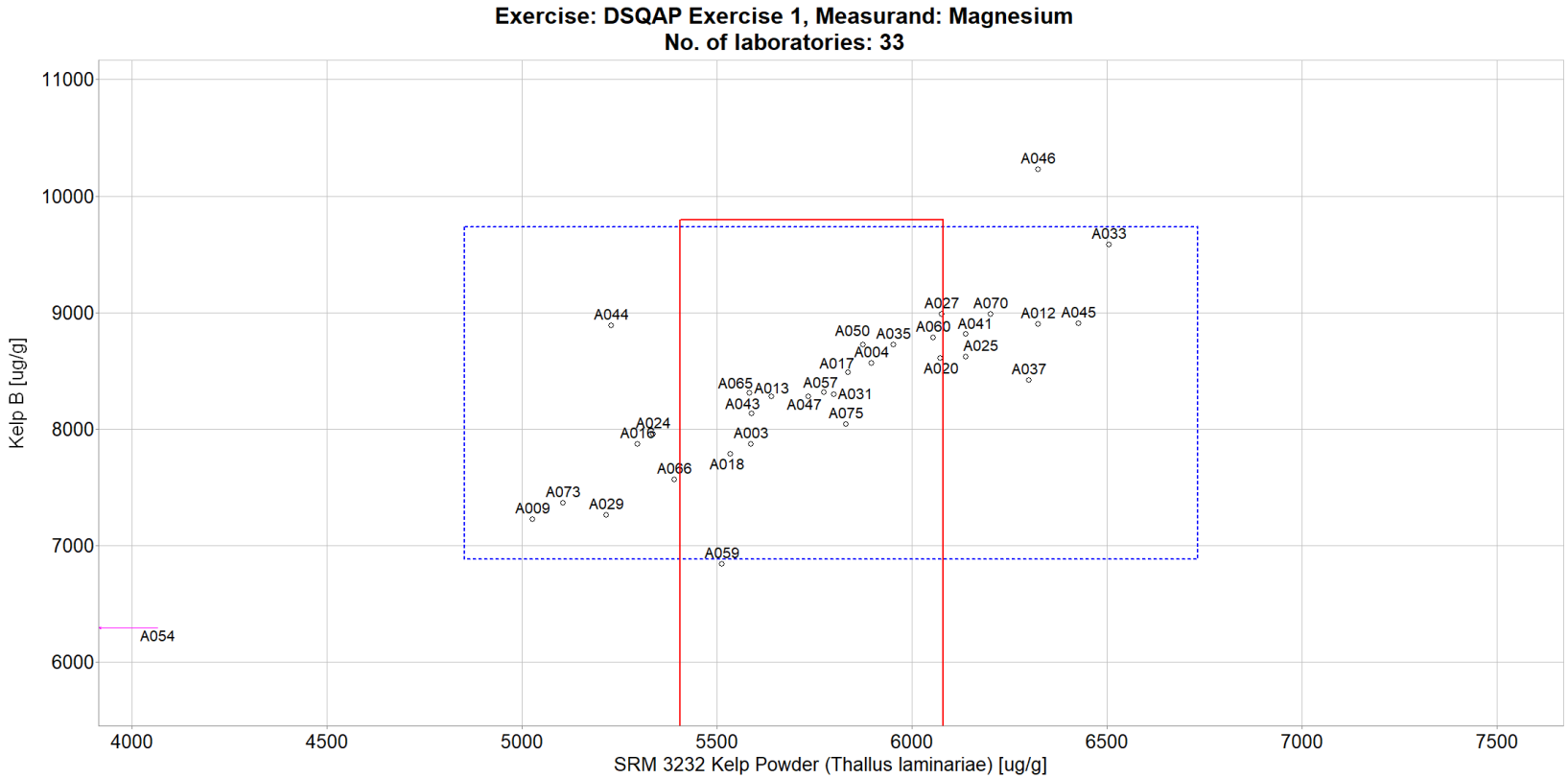

Fig. B-18. Laboratory means for magnesium in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp B (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3232, is compared to the individual laboratory mean for a second sample, Kelp B. The solid red box represents the NIST range of tolerance for the two samples, SRM 3232 (x-axis) and Kelp B (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3232 (x-axis) and Kelp B (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

Table B-11. Data summary table for mercury in kelp.

Data highlighted in blue have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable Z'_{comm} score, $|Z'_{comm}| > 2$. (Table continues to next page.)

		Mercury														
		Kelp A (ng/g)					Kelp B (ng/g)					SRM 3232 Kelp Powder (*Thallus laminariae*) (ng/g)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
Individual Results	Target				31.3	0.46				23.1	0.42				106	3.00
	A001															
	A002															
	A004	28.3	28.5	32	29.6	2.08	23.9	24.8	26.4	25.0	1.27	98.4	97.1	103.2	99.6	3.21
	A005															
	A008															
	A009	270	90	10	123.3	133.17	< 10.0	< 10.0	< 10.0			30	30	20	26.7	5.77
	A012	39.8	45.1	41.9	42.3	2.67	33.4	30.1	29.6	31.0	2.06	114	119	120	118	3.21
	A013	28.4	28.1	26.7	27.7	0.91	19.1	21.1	20.1	20.1	1.00	77.4	86.1	82.3	81.9	4.36
	A015															
	A016	30	30	30	30	0	50	40	40	43.3	5.77	90	100	90	93.3	5.77
	A017															
	A018															
	A019	< 40.0	< 40.0	< 40.0			< 40.0	< 40.0	< 40.0			50	50	50	50	0
	A020	26	24	18	22.7	4.16	24	25	22	23.7	1.53	97	97	100	98.0	1.73
	A022															
	A024	15	10	10	11.7	2.89	28	20	25	24.3	4.04	121	120	125	122	2.65
	A025	6.5	50.3	< 1.000	28.4	31.0	< 1.000	< 1.000	9.9	9.90		66.4	91.4	50.3	69.4	20.71
	A027	11.6281	109.342	118.357	79.8	59.2	23	23	22	22.7	0.58	90	89	83	87.3	3.79
	A028															
	A029	28.5	25.8	27.3	27.2	1.35	21.9	22	21.6	21.8	0.21	92.1	93.6	91.4	92.4	1.12
	A031															
	A032															
	A033	40	40	30	36.7	5.77	40	40	40	40	0	110	120	120	117	5.77
	A035	30	30	40	33.3	5.77	20	20	20	20	0	100	100	100	100	0
	A037	32	29	29	30.0	1.73	28	27	30	28.3	1.53	91	93	89	91.0	2.00
	A039															
	A041	17	17	19	17.7	1.15	12	13	14	13.0	1.00	80	84	90	84.7	5.03

		Mercury														
		Kelp A (ng/g)					Kelp B (ng/g)					SRM 3232 Kelp Powder (*Thallus laminariae*) (ng/g)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
	A042	30	38	35	34.3	4.04	35	29	25	29.7	5.03	84	88	85	85.7	2.08
	A043	70	50	40	53.3	15.28	30	30	30	30	0	100	100	110	103	5.77
	A044	40	43	41	41.3	1.53	31	34	35	33.3	2.08	92	92	103	95.7	6.35
	A045	< 1.000	< 1.000	< 1.000			< 1.000	< 1.000	< 1.000			< 1.000	< 1.000	< 1.000		
	A046	40	45	43	42.7	2.52	27	29	28	28.0	1.00	78	89	91	86.0	7.00
	A047	27	30		28.5	2.12	22	20		21.0	1.41	96	99		97.5	2.12
	A050	61.06	46.03	45.61	50.9	8.80	54.59	59.85	48.82	54.4	5.52	95.52	110.9	108.9	105	8.36
	A051															
	A053															
	A054	28.4	27.8	29.2	28.5	0.70	22.8	23.3	23.4	23.2	0.32	83.7	94	85.6	87.8	5.48
	A057	32	30	31	31.0	1.00	28	28	29	28.3	0.58	100	100	98	99.3	1.15
	A058	36	35	36	35.7	0.58	32	31	38	33.7	3.79	98	100	102	100	2.00
	A060	47.8	39.3	44.7	43.9	4.30	34.7	33.6	33	33.8	0.86	83.4	99.3	90.2	91.0	7.98
	A061															
	A062															
	A065	40	40	40	40	0	30	30	30	30	0	120	120	110	117	5.77
	A066															
	A068															
	A070	38.3	41.5	37.9	39.2	1.97	24.1	24.3	24.3	24.2	0.12	123.9	122.4	119.9	122	2.02
	A071															
	A073											20.5	21.6	22.1	21.4	0.82
	A075	24.6	26.85	25.89	25.8	1.13	23.6	28.37	23.49	25.2	2.79	70.56	85.06	84.39	80.0	8.19
	A079	< 4.000	< 4.000	< 4.000			< 500.0	< 500.0	< 500.0			< 40.0	< 40.0	< 40.0		
Community Results		Consensus Mean			33.7		Consensus Mean			27.0		Consensus Mean			95.8	
		Consensus Standard Deviation			12.0		Consensus Standard Deviation			8.20		Consensus Standard Deviation			16.8	
		Maximum			123		Maximum			54.4		Maximum			122	
		Minimum			11.7		Minimum			9.90		Minimum			21.4	
		N			27		N			25		N			29	

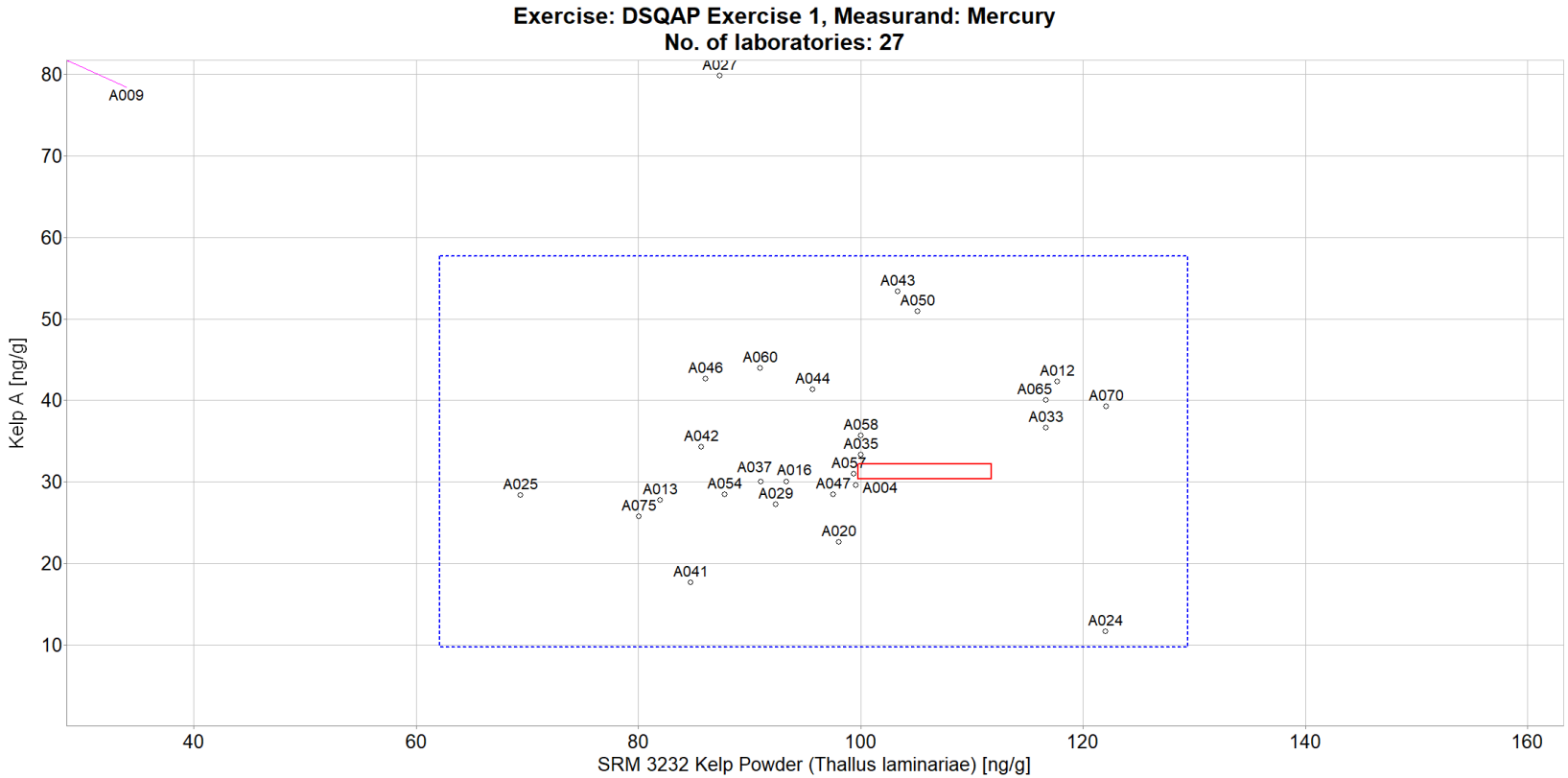

Fig. B-19. Laboratory means for mercury in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp A (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3232, is compared to the individual laboratory mean for a second sample, Kelp A. The solid red box represents the NIST range of tolerance for the two samples, SRM 3232 (x-axis) and Kelp A (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3232 (x-axis) and Kelp A (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

Fig. B-20. Laboratory means for mercury in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp B (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3232, is compared to the individual laboratory mean for a second sample, Kelp B. The solid red box represents the NIST range of tolerance for the two samples, SRM 3232 (x-axis) and Kelp B (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3232 (x-axis) and Kelp B (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

Table B-12. Data summary table for potassium in kelp.

Data highlighted in blue have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable Z'_{comm} score, $|Z'_{comm}| > 2$. (Table continues to next page.)

		Potassium														
		Kelp A (mg/g)					Kelp B (mg/g)					SRM 3232 Kelp Powder (*Thallus laminariae*) (mg/g)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
Individual Results	Target				126	2.53				21.8	0.77				71.2	1.03
	A001															
	A002															
	A003	109.5	111.3	105.9	109	2.75	19.2	18.83	18.58	18.9	0.31	61.37	61.39	60.96	61.2	0.24
	A004	111.067	111.055	116.003	113	2.85	20.519	20.291	20.592	20.5	0.16	66.637	64.753	65.128	65.5	1.00
	A005															
	A009	120.101	125.238	130.515	125	5.21	21.5929	20.4187	19.6179	20.5	0.99	66.4178	72.7299	65.2039	68.1	4.04
	A012	123	126	123	124	1.73	22.4	22	21.5	22.0	0.45	71	71.5	73.7	72.1	1.44
	A013	116.59	117.453	121.325	118	2.52	21.4387	21.7058	20.8393	21.3	0.44	68.2962	66.9485	67.1401	67.5	0.73
	A015															
	A016	114	113	114	114	0.58	21.6	21.5	21.3	21.5	0.15	66.9	68	67.6	67.5	0.56
	A017	149.5	142.1	141.4	144	4.49	19.127	17.785	18.876	18.6	0.71	59.992	71.77	59.523	63.8	6.94
	A018	118	120	119	119	1.00	18.8	20.2	20.1	19.7	0.78	64.6	64.2		64.4	0.28
	A019	116.5	117.6	118.5	118	1.00	19.18	19.75	19.35	19.4	0.29	69.73	72.61	68.84	70.4	1.97
	A020	111.378	112.415	107.853	111	2.39	19.962	19.058	19.699	19.6	0.46	62.648	65.881	63.961	64.2	1.63
	A022															
	A025	128.047	123.204	123.152	125	2.81	21.5	21	21.613	21.4	0.33	70.157	68.567	69.452	69.4	0.80
	A027	5944	1851	2450	3415	2211	21.5296	20.8713	21.4888	21.3	0.37	65.962	71.0654	66.2908	67.8	2.86
	A028															
	A029	131	121	121	124	5.77	20.3	20.7	21	20.7	0.35	69.7	67.2	69.7	68.9	1.44
	A031	115.9	117.2	121.1	118	2.71	21.2	20.7	21.6	21.2	0.45	71.3	69.8	68.4	69.8	1.45
	A032															
	A033	122.498	121.043	125.079	123	2.04	23.061	22.625	22.593	22.8	0.26	71.074	70.087	73.967	71.7	2.02
	A035	113.8	113.9	112.9	114	0.55	21.38	20.77	20.81	21.0	0.34	65.89	65.37	65.76	65.7	0.27
	A037	122	122	122	122	0	21.1	20.7	21.2	21.0	0.26	73.7	71.8	70.4	72.0	1.66
	A039															
	A041	122.236	119.708	118.4	120	1.95	21.8255	22.184	21.8321	21.9	0.21	70.305	70.4336	71.1716	70.6	0.47
	A042	110.402	104.868	105.92	107	2.94	20.297	20.539	20.214	20.4	0.17	60.564	60.191	60.196	60.3	0.21
	A043	106.485	105.273	103.513	105	1.49	19.3969	19.2824	18.9033	19.2	0.26	57.0835	56.9682	57.7432	57.3	0.42

		Potassium														
		Kelp A (mg/g)					Kelp B (mg/g)					SRM 3232 Kelp Powder (*Thallus laminariae*) (mg/g)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
	A044	122.969	122.352	121.701	122	0.63	22.3426	22.7718	21.9489	22.4	0.41	67.3892	69.0205	66.5617	67.7	1.25
	A045	102.913	110.707	117.204	110	7.16	21.4765	21.2571	25.8625	22.9	2.60	71.3199	68.2506	83.5229	74.4	8.08
	A046															
	A047	119	121		120	1.41	20.9	20.9		20.9	0	69.2	68.7		69.0	0.35
	A050	116.6	118.4	120.5	119	1.95	21.59	21.48	21.41	21.5	0.09	67.61	70.75	70.3	69.6	1.70
	A051															
	A052															
	A053															
	A054	48.9	49.2	50.5	50	0.85	16.3	16	15.4	15.9	0.46	25	27.4	25.9	26.1	1.21
	A057	129.27	128.287	129.634	129	0.70	21.603	22.401	22.797	22.3	0.61	69.155	67.691	68.09	68.3	0.76
	A059	97.9	100.94	104.3	101	3.20	19.86	21	20.6	20.5	0.58	59.4	57.6	57.8	58.3	0.99
	A060															
	A061															
	A062															
	A065	127.674	129.714	123.758	127	3.03	22.5456	22.3977	22.7674	22.6	0.19	72.2961	73.6585	70.2043	72.1	1.74
	A066	102.257	103.715	101.763	103	1.01	18.712	18.513	18.223	18.5	0.25	59.031	58.567	61.799	59.8	1.75
	A068															
	A070	139.048	147.819	134.839	141	6.62	24.59	22.202	23.124	23.3	1.20	81.984	73.25	73.63	76.3	4.94
	A071															
	A073	83.8416	84.376	83.8446	84	0.31	17.5069	17.438	16.5769	17.2	0.52	43.0901	42.7791	43.4785	43.1	0.35
	A075	128.149	115.868	127.68	124	6.96	19.9961	22.1739	21.5666	21.2	1.12	77.164	74.5826	74.8732	75.5	1.41
	A079	117	115	114	115	1.53	18.6	18.3	18.9	18.6	0.30	67.2	64.9	67.8	66.6	1.53
Community Results		Consensus Mean			118		Consensus Mean			20.7		Consensus Mean			67.7	
		Consensus Standard Deviation			11.5		Consensus Standard Deviation			1.61		Consensus Standard Deviation			5.56	
		Maximum			3415		Maximum			23.3		Maximum			76.3	
		Minimum			49.5		Minimum			15.9		Minimum			26.1	
		N			33		N			33		N			33	

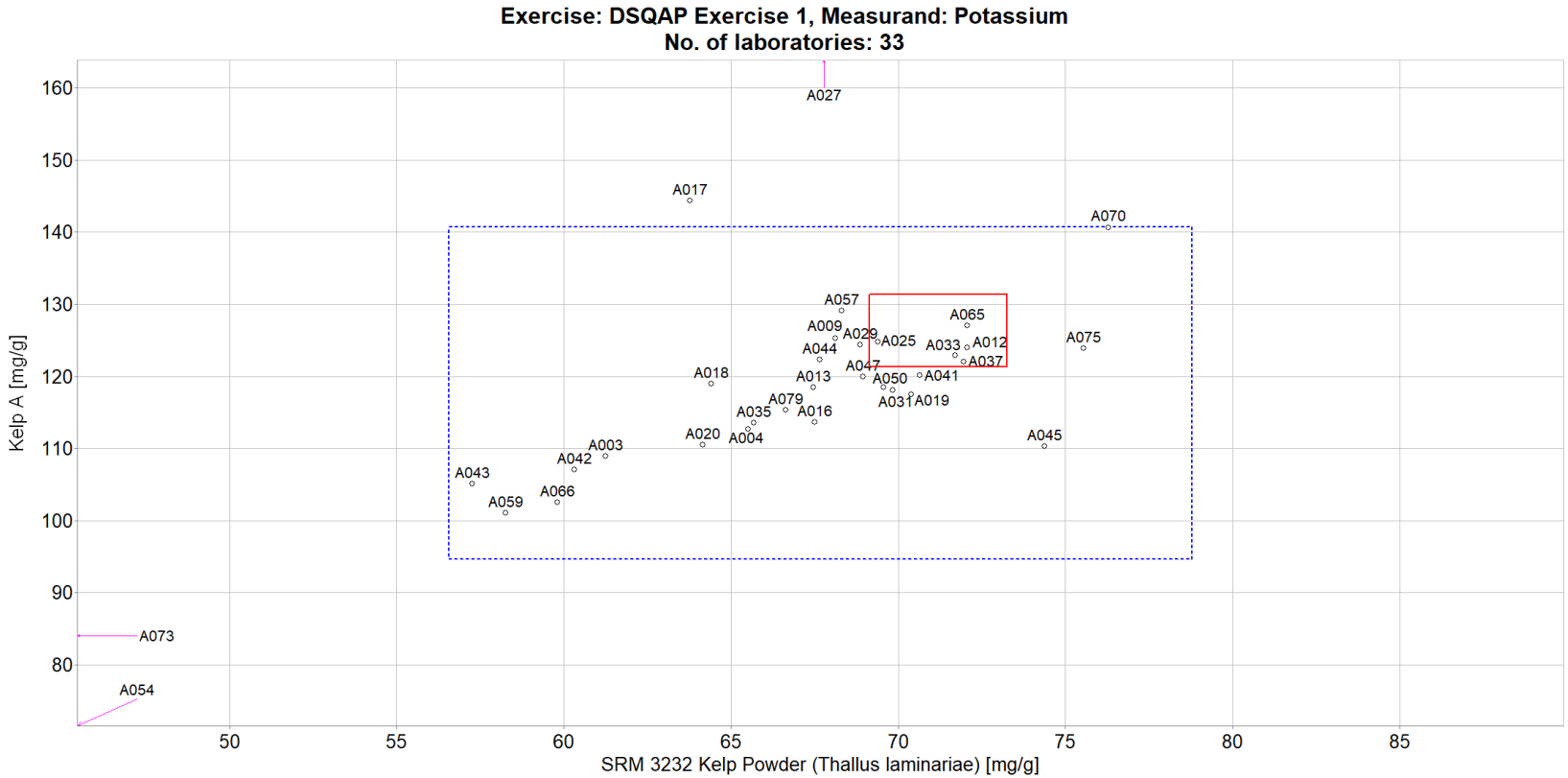

Fig. B-21. Laboratory means for potassium in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp A (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3232, is compared to the individual laboratory mean for a second sample, Kelp A. The solid red box represents the NIST range of tolerance for the two samples, SRM 3232 (x-axis) and Kelp A (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3232 (x-axis) and Kelp A (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

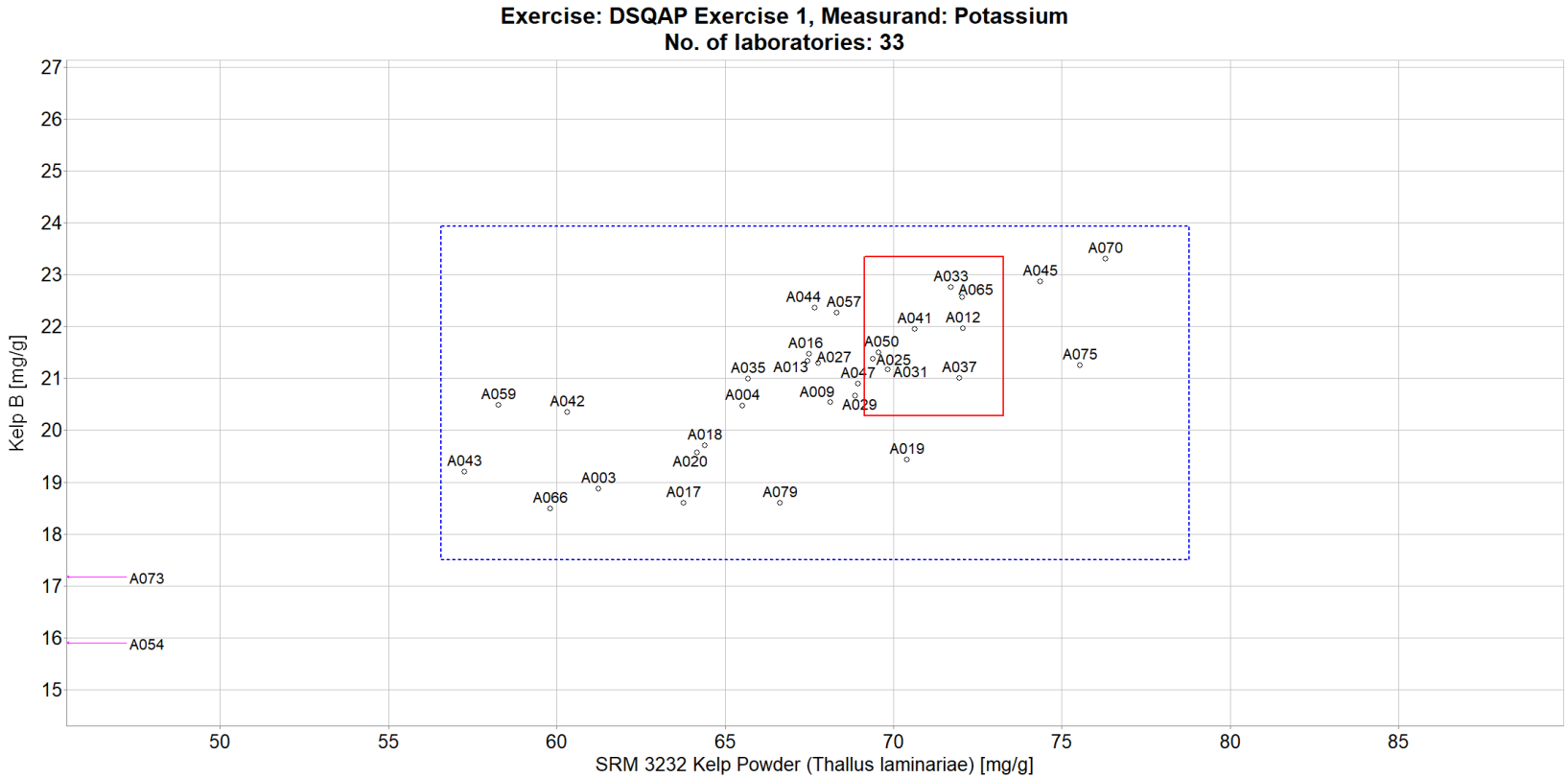

Fig. B-22. Laboratory means for potassium in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp B (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3232, is compared to the individual laboratory mean for a second sample, Kelp B. The solid red box represents the NIST range of tolerance for the two samples, SRM 3232 (x-axis) and Kelp B (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3232 (x-axis) and Kelp B (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

Table B-13. Data summary table for selenium in kelp.

Data highlighted in blue have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable Z'_{comm} score, $|Z'_{comm}| > 2$. (Table continues to next page.)

		Selenium														
		Kelp A (ng/g)					Kelp B (ng/g)					SRM 3232 Kelp Powder (*Thallus laminariae*) (ng/g)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
Individual Results	Target				89.2	4.70				38.3	6.90				52.6	13.7
	A001															
	A002															
	A003															
	A004	323	324	295	314.0	16.5	340	360	362	354	12.2	299	269	159	242	73.7
	A005															
	A009	240	240	240	240	0	140	180	180	167	23.1	160	200	150	170	26.5
	A012	91	100	90	93.7	5.51	37	34	45	38.7	5.69	59	58	65	60.7	3.79
	A013	177.117	173.08	203.862	185	16.7	88.981	92.721	104.483	95.4	8.09	111.1	86.948	121.193	106	17.6
	A015															
	A016	< 500.0	< 500.0	570	570		< 500.0	< 500.0	< 500.0			< 500.0	< 500.0	< 500.0		
	A017															
	A018	742	683	738	721	33.0	334	388	256	326	66.4	276	322		299	32.5
	A020	110	118	104	111	7.02	54	55	58	55.7	2.08	69	83	76	76.0	7.00
	A022															
	A024	< 252.000	< 252.000	< 252.000			< 252.000	< 252.000	< 252.000			< 252.000	< 252.000	< 252.000		
	A025	13.8	6.7	14.8	11.8	4.42	13.4	17.3	12.6	14.4	2.51	7.5	11.5	9.9	9.63	2.01
	A027	41.3013	38.705	41.629	40.5	1.60	1951	1802	1238	1664	376	11.091	0.945	0.862	4.30	5.88
	A028															
	A029	107	108	106	107	1.00	47.5	51.7	44.8	48.0	3.48	58.9	62.3	62.5	61.2	2.02
	A031															
	A032															
	A033	152	141	169	154	14.1	125	114	< 100.0	119.5	7.78	183	121	< 100.000	152	43.8
	A035	272	289	234	265	28.2	79	87	95	87.0	8.00	180	151	156	162	15.5
	A037	642	667	539	616	67.8	296	289	232	272	35.1	401	437	401	413	20.8
	A039															
	A041	176	150	328	218	96.1	299	219	166	228	67.0	192	273	256	240	42.7
	A042	< 300.0	< 300.0	< 300.0			< 300.0	< 300.0	< 300.0			< 300.0	< 300.0	< 300.0		
	A043	3939	4096	3999	4011	79.2	1336	1309	1272	1306	32.1	2.065	2.102	2.136	2.10	0.04

		Selenium														
		Kelp A (ng/g)					Kelp B (ng/g)					SRM 3232 Kelp Powder (*Thallus laminariae*) (ng/g)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
	A044	< 1250.0	< 1250.0	< 1250.0			< 1250.0	< 1250.0	< 1250.0			< 1250.0	< 1250.0	< 1250.0		
	A046	262	177	240	226	44.1	167	171	271	203	58.9	158	245	198	200	43.5
	A050	81.9	80.2	90.1	84.1	5.29	33.1	31.2	31.7	32.0	0.98	45.8	47.5	46.8	46.7	0.85
	A051															
	A053															
	A054	128	130	138	132	5.29	63.4	48.4	47	52.9	9.09	102	106	104	104	2.00
	A057	160	180	150	163	15.3	92	77	86	85.0	7.55	260	120	130	170	78.1
	A060	< 2000.0	< 2000.0	< 2000.0			< 2000.0	< 2000.0	< 2000.0			< 2000.0	< 2000.0	< 2000.0		
	A061															
	A062															
	A065	106.44	109.55	109.08	108	1.68	50.62	49.2	50.98	50.3	0.94	76.42	73.78	72.89	74.4	1.84
	A066															
	A068															
	A070	91	103	100	98.0	6.24	41	52	42	45.0	6.08	63	63	63	63	0
	A071															
	A073															
	A075	4.4408	4.8115	4.7758	4.68	0.20	4.7564	4.7343	4.3028	4.60	0.26	4.8419	4.8124	4.6027	4.75	0.13
	A079	< 200.0	< 200.0	< 200.0			< 300.0	< 300.0	< 300.0			< 200.0	< 200.0	< 200.0		
Community Results		Consensus Mean			173		Consensus Mean			110		Consensus Mean			120	
		Consensus Standard Deviation			147		Consensus Standard Deviation			94.8		Consensus Standard Deviation			109	
		Maximum			4011		Maximum			1664		Maximum			413	
		Minimum			4.68		Minimum			4.60		Minimum			2.10	
		N			21		N			21		N			21	

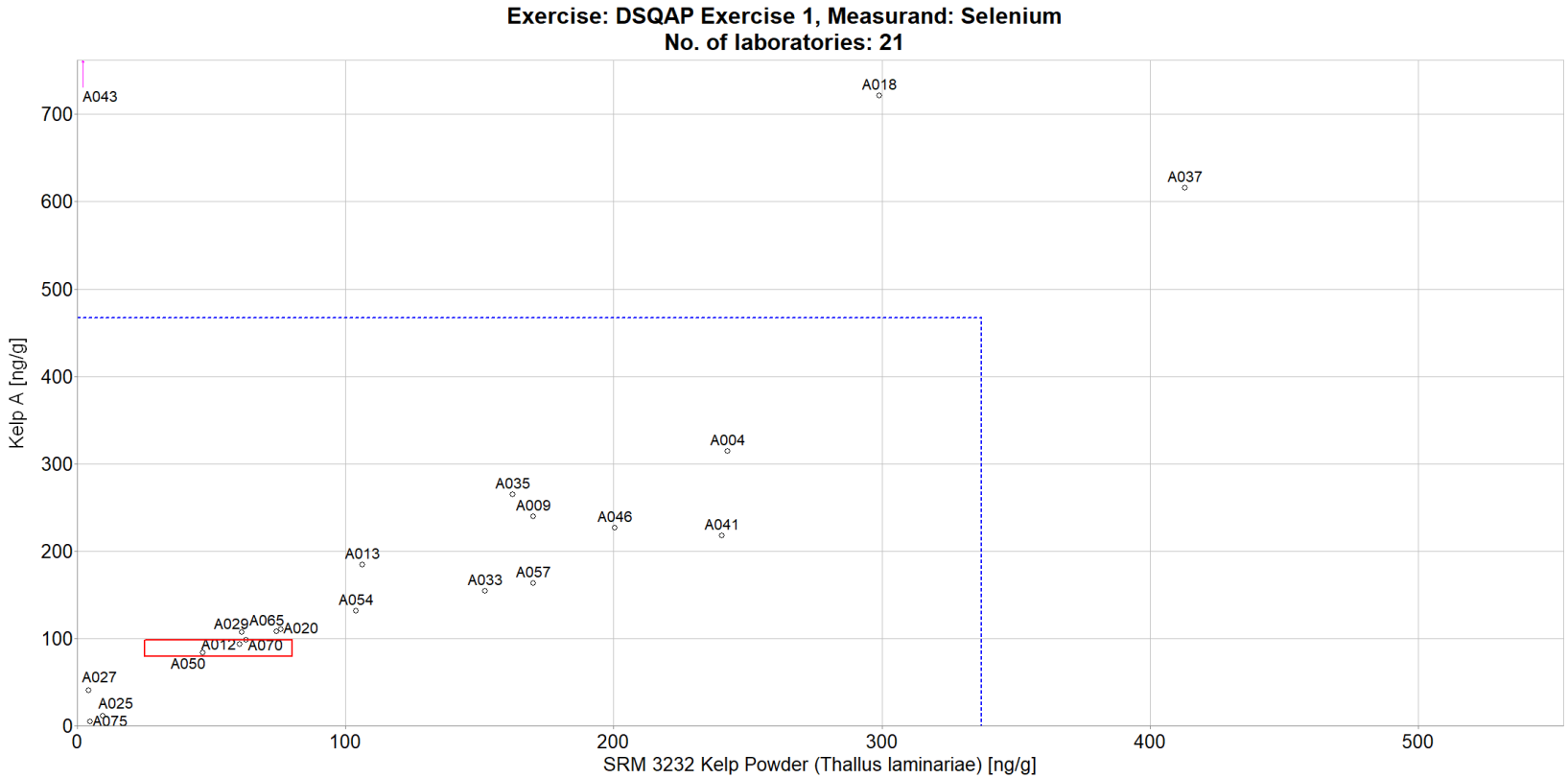

Fig. B-23. Laboratory means for selenium in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp A (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3232, is compared to the individual laboratory mean for a second sample, Kelp A. The solid red box represents the NIST range of tolerance for the two samples, SRM 3232 (x-axis) and Kelp A (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3232 (x-axis) and Kelp A (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

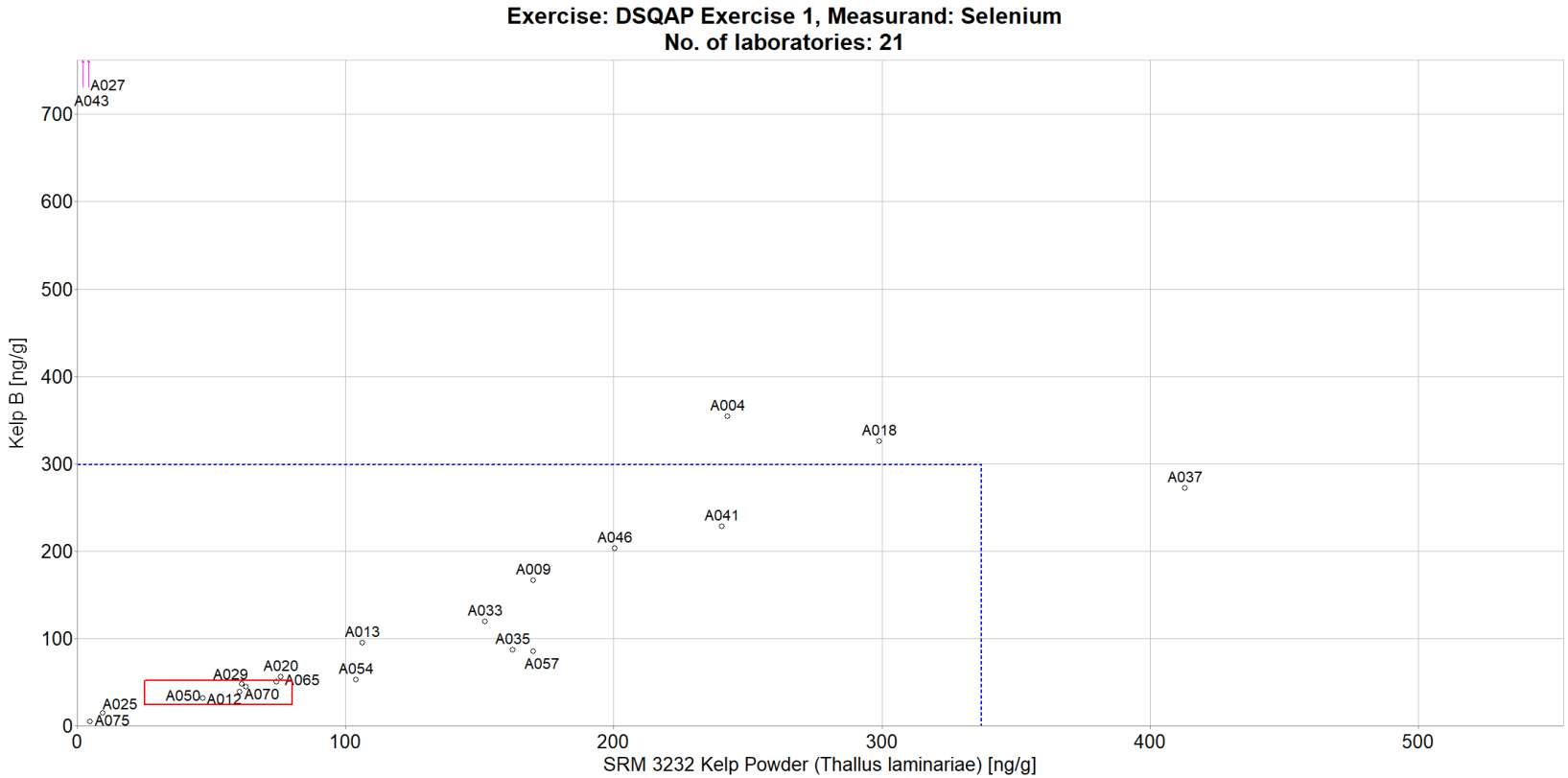

Fig. B-24. Laboratory means for selenium in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp B (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3232, is compared to the individual laboratory mean for a second sample, Kelp B. The solid red box represents the NIST range of tolerance for the two samples, SRM 3232 (x-axis) and Kelp B (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3232 (x-axis) and Kelp B (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

Table B-14. Data summary table for sodium in kelp.

Data highlighted in blue have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable Z'_{comm} score, $|Z'_{comm}| > 2$. (Table continues to next page.)

		Sodium														
		Kelp A (ug/g)					Kelp B (ug/g)					SRM 3232 Kelp Powder (*Thallus laminariae*) (ug/g)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
Individual Results	Target				39457	129				31741	432				15298	356
	A001															
	A002															
	A003	39350	40930	39590	39957	851	33190	31670	31500	32120	931	15450	15340	15360	15383	59
	A004	43311	43240	43355	43302	58	34536	33914	35669	34706	890	16918	16432	16597	16649	247
	A005															
	A009	40443.33	41904.21	43200.71	41849	1380	32347.35	30274.07	30034.59	30885	1272	14403.19	14694.99	13504.07	14201	621
	A012	38000	38800	39100	38633	569	31800	30800	30600	31067	643	14600	14400	14800	14600	200
	A013	39737.83	40559.69	40745.17	40348	536	32094.54	30910.33	31051.57	31352	647	13792.05	15609.35	14446.42	14616	920
	A015															
	A016	32200	32100	32200	32167	58	28700	28500	28600	28600	100	13700	13600	13600	13633	58
	A017	45525	45276	54686	48496	5362	35151	35888	36054	35698	481	17146	17657	16641	17148	508
	A018	39700	40100	40200	40000	265	27700	29900	29500	29033	1172	13800	13900		13850	71
	A020	36277	36287	35250	35938	596	29880	30778	30595	30418	475	14267	14732	14456	14485	234
	A022															
	A025	43060	41490	40910	41820	1112	32500	31788	32237	32175	360	15552	15423	15615	15530	98
	A027						34011	33047.65	34059.21	33706	571	14934.23	16084.97	15173.9	15398	607
	A028															
	A029	32000	34700	30100	32267	2312	22800	22900	23300	23000	265	12200	11100	11300	11533	586
	A031	39300	39300	41000	39867	981	32800	33900	33900	33533	635	15000	15000	14700	14900	173
	A032															
	A033	42922	42005	43389	42772	704	34946	33953	34522	34474	498	16074	15785	16174	16011	202
	A035	39530	41110	38660	39767	1242	32190	31680	31580	31817	327	13490	14310	14700	14167	618
	A037	41100	42900	40700	41567	1172	31800	31600	32200	31867	306	17000	16500	16700	16733	252
	A039															
	A041	40328.54	39009.58	37544.5	38961	1393	30329.78	29751.12	37544.5	32542	4342	13848.79	13851.22	13645.77	13782	118
	A042	42100	40256	40594	40983	982	30375	30417	30478	30423	52	14776	14730	14715	14740	32
	A043	37320.09	35588.65	33796.19	35568	1762	25755.93	25162.51	24429.99	25116	664	10317.85	10040.91	9983.006	10114	179
	A044	40674.04	40904.7	40663.19	40747	136	33996.98	32985.5	33630.03	33538	512	16819.53	17360.45	17132	17104	272
	A045	32853.2	34534.6	36779.56	34722	1970	30537.98	30038.9	35069.93	31882	2772	14362.56	14086.85	18750.01	15733	2616

		Sodium														
		Kelp A (ug/g)					Kelp B (ug/g)					SRM 3232 Kelp Powder (*Thallus laminariae*) (ug/g)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
	A046															
	A047	40760	39770		40265	700	31100	31590		31345	346	14240	14120		14180	85
	A051															
	A052															
	A053															
	A054	15100	14700	15200	15000	265	21700	21400	20400	21167	681	4850	5400	5050	5100	278
	A057	42421	41779	42398	42199	364	28758	30330	31073	30054	1182	15018	14291	14549	14619	369
	A059	33600	33400	35300	34100	1044	21490	21930	21560	21660	236	13990	13950	13980	13973	21
	A060	40985	39695	39117	39932	956	33838	30254	30796	31629	1932	14444	14171	14303	14306	137
	A061															
	A062															
	A065	38956.1	39079.98	36745.46	38261	1314	30124.75	29938.66	30532.12	30199	304	13832.12	13998.98	13419.3	13750	298
	A066	34698	34699	35491	34963	458	28285	27945	27978	28069	188	13392	13103	14025	13507	472
	A068															
	A070															
	A071															
	A073															
	A075															
	A079	41400	42100	41500	41667	379	29100	30200	29200	29500	608	14200	14100	14700	14333	321
Community Results		Consensus Mean			39300		Consensus Mean			30897		Consensus Mean			14739	
		Consensus Standard Deviation			3399		Consensus Standard Deviation			3084		Consensus Standard Deviation			1321	
		Maximum			48496		Maximum			35698		Maximum			17148	
		Minimum			15000		Minimum			21167		Minimum			5100	
		N			28		N			29		N			29	

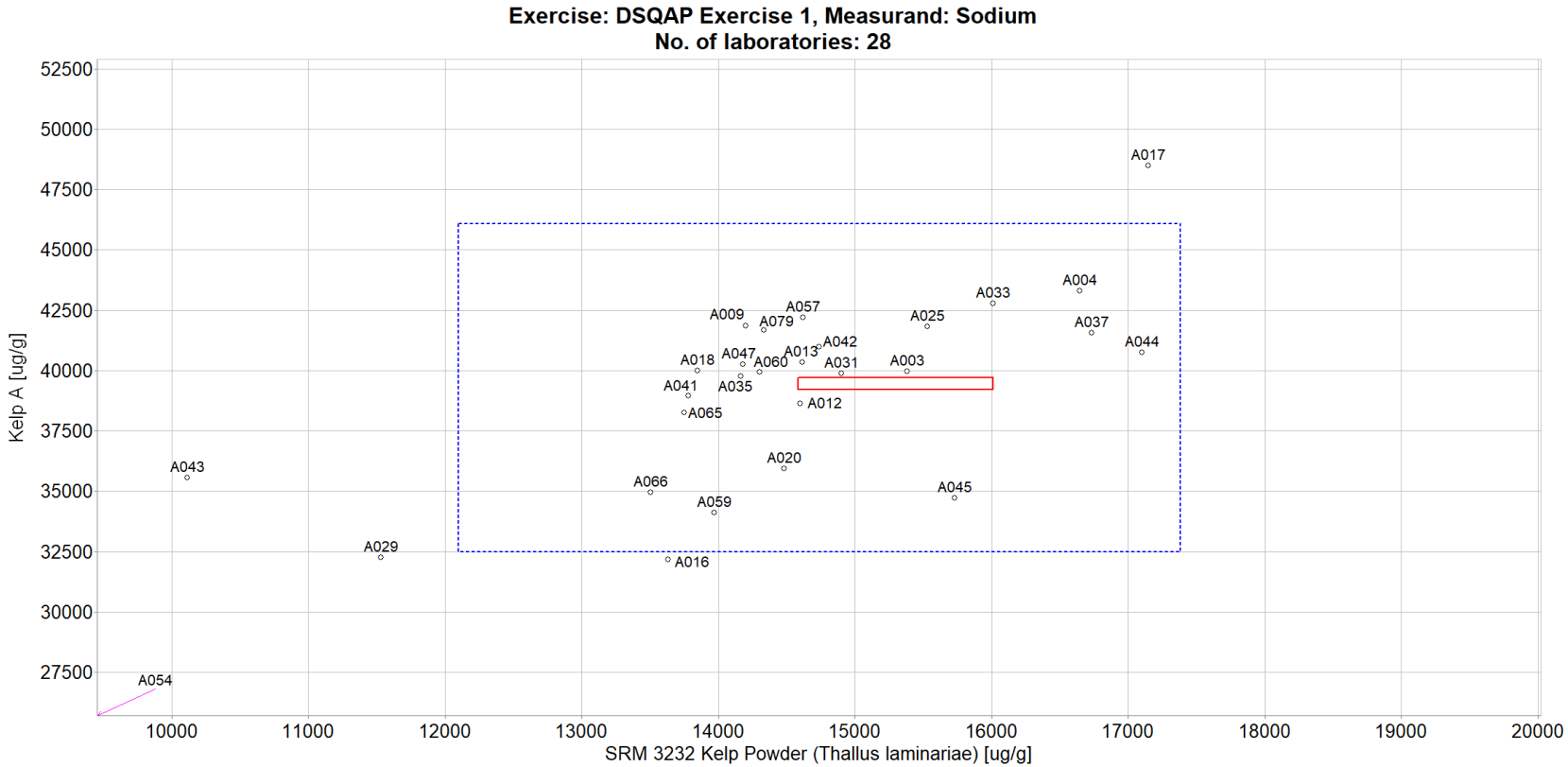

Fig. B-25. Laboratory means for sodium in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp A (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3232, is compared to the individual laboratory mean for a second sample, Kelp A. The solid red box represents the NIST range of tolerance for the two samples, SRM 3232 (x-axis) and Kelp A (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3232 (x-axis) and Kelp A (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

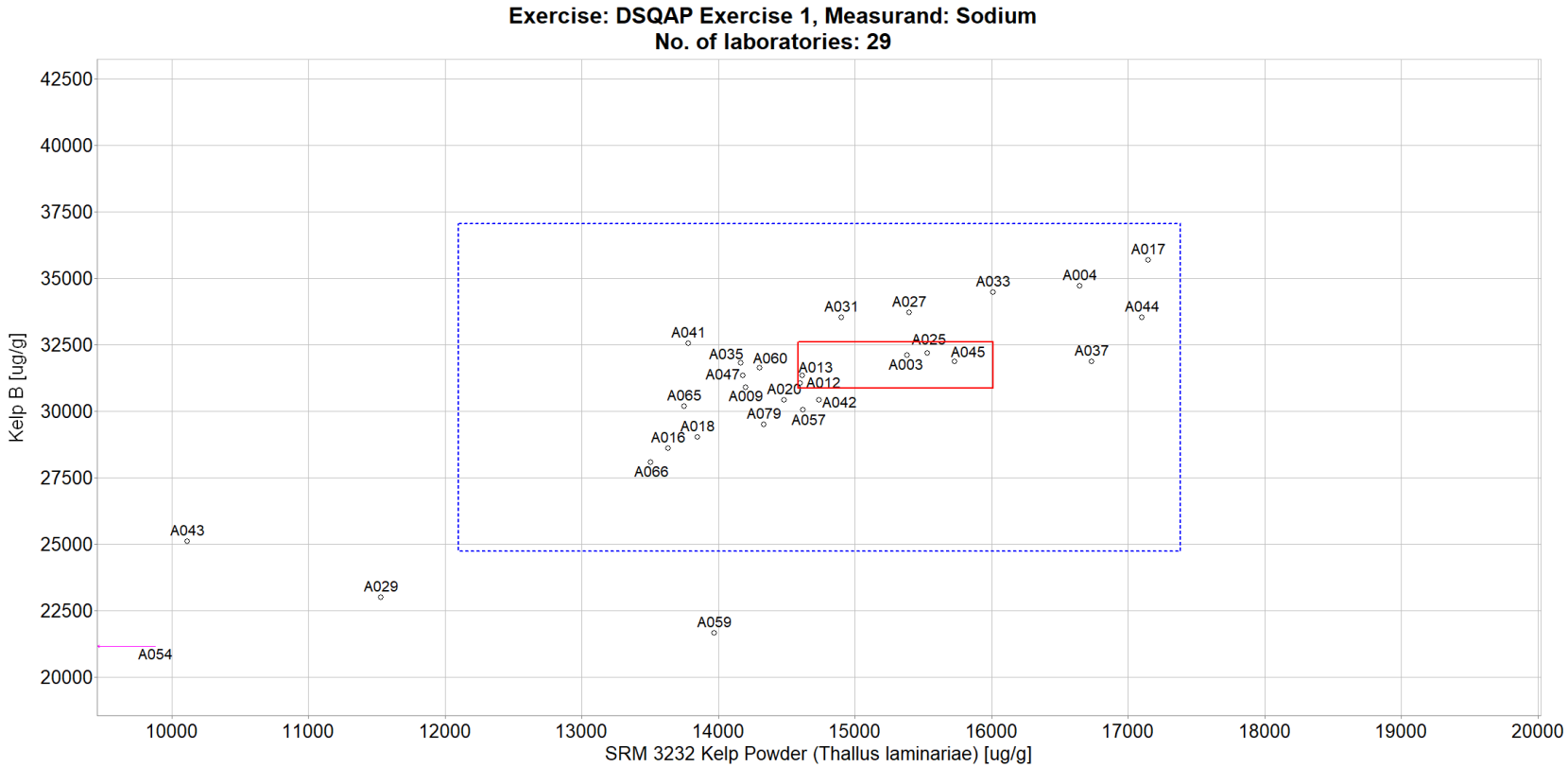

Fig. B-26. Laboratory means for sodium in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp B (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3232, is compared to the individual laboratory mean for a second sample, Kelp B. The solid red box represents the NIST range of tolerance for the two samples, SRM 3232 (x-axis) and Kelp B (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3232 (x-axis) and Kelp B (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

Table B-15. Data summary table for sulfur in kelp.

Data highlighted in blue have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable Z'_{comm} score, $|Z'_{comm}| > 2$. (Table continues to next page.)

		Sulfur														
		Kelp A (ug/g)					Kelp B (ug/g)					SRM 3232 Kelp Powder (*Thallus laminariae*) (ug/g)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
Individual Results	Target															
	A001															
	A002															
	A012	7230	7310	7230	7257	46.2	23500	24900	24900	24433	808	11500	11400	11900	11600	265
	A013	6061.807	6046.451	6595.828	6235	313	20433.68	19917.91	21352.45	20568	727	9884.621	10147.98	10137.17	10057	149
	A015															
	A016	6560	6610	6530	6567	40.4	22400	22400	22600	22467	115	10600	10600	10800	10667	115
	A017															
	A018	6750	6900	6870	6840	79.4	20100	21300	21200	20867	666	10100	10200		10150	71
	A020															
	A022															
	A024	5521.88	5521.886	5521.876	5522	0.01	23814.53	23814.53	23814.52	23815	0.004	10673.58	10673.59	10673.58	10674	0.003
	A025															
	A027															
	A028															
	A029	5800	6040	5210	5683	427	16600	16500	16800	16633	153	8220	7910	8140	8090	161
	A031	6400	6600	6500	6500	100	21400	21600	22400	21800	529	9900	10300	10200	10133	208
	A032															
	A033															
	A035															
	A037	7320	7190	7160	7223	85.0	25800	25300	25600	25567	252	11900	12700	11900	12167	462
	A041															
	A042															
	A043															
	A044															
	A045	10534.43			10534		25380.89			25381		15494.27			15494	
	A046															
	A051															
	A053															
	A054	240	232	194	222	24.6	< 0.500	< 0.500	< 0.500			185	151	177	171	17.8

		Sulfur														
		Kelp A (ug/g)					**Kelp B (ug/g)**					**SRM 3232 Kelp Powder (*Thallus laminariae*) (ug/g)**				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
	A057															
	A059	6400	6400	6300	6367	57.7	19300	19400	19900	19533	321	10100	10000	10000	10033	57.7
	A061															
	A062															
	A065	7525.988	7718.586	7265.308	7503	227	24817.28	24747.24	25199.87	24921	244	11731.85	11926.3	11466.38	11708	231
	A066															
	A068															
	A070	3347	4077	3817	3747	370	18547	19042	18970	18853	267	9009	8800	9147	8985	175
	A071															
	A073															
	A075															
	A079	< 100000	< 100000	< 100000			< 40000	< 40000	< 40000			< 40000	< 40000	< 40000		
Community Results		Consensus Mean			6570		Consensus Mean			22119		Consensus Mean			10645	
		Consensus Standard Deviation			1386		Consensus Standard Deviation			3262		Consensus Standard Deviation			1886	
		Maximum			10534		Maximum			25567		Maximum			15494	
		Minimum			222		Minimum			16633		Minimum			171	
		N			12		N			11		N			12	

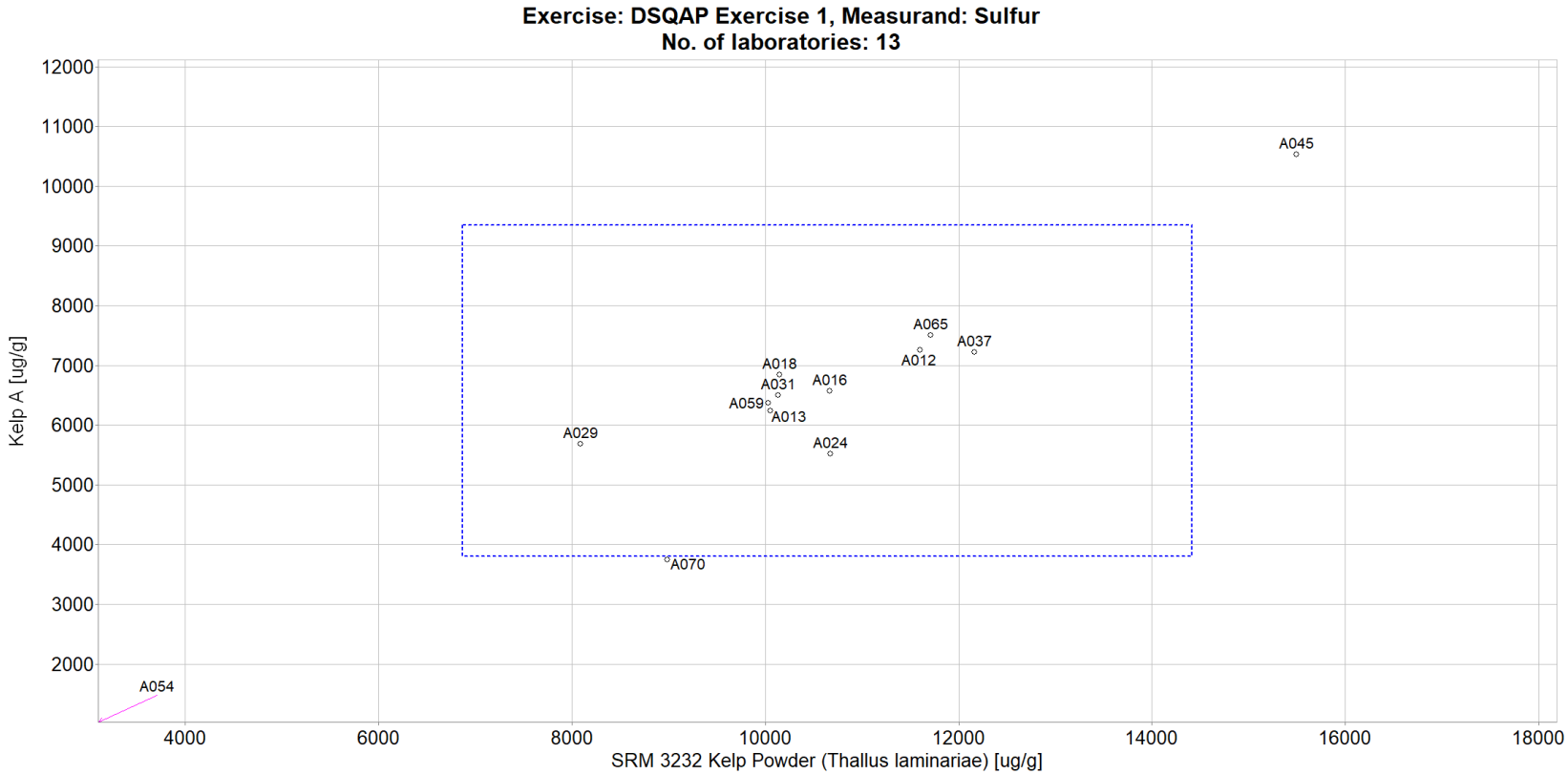

Fig. B-27. Laboratory means for sulfur in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp A (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3232, is compared to the individual laboratory mean for a second sample, Kelp A. The dotted blue box represents the consensus range of tolerance for SRM 3232 (x-axis) and Kelp A (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

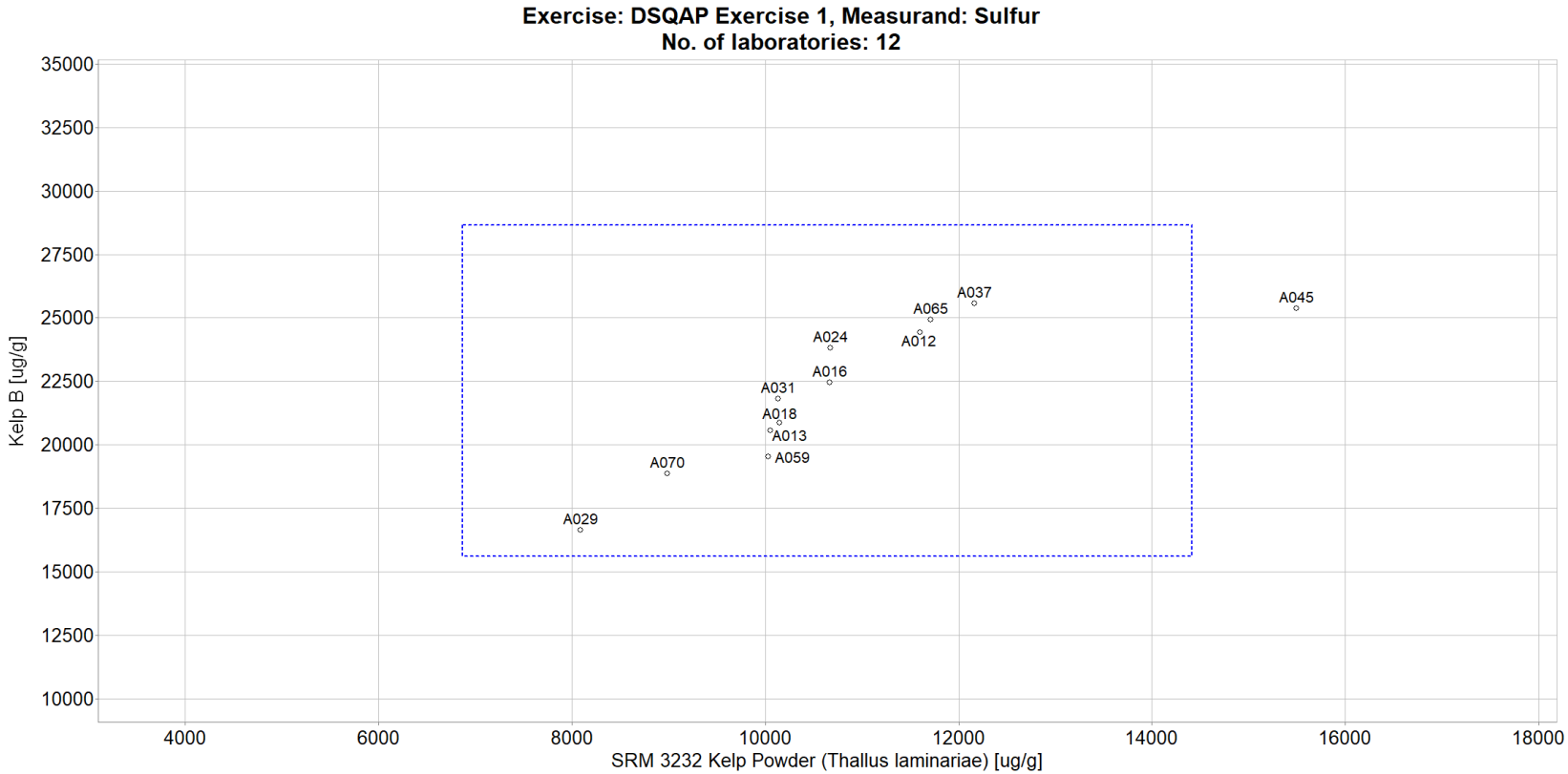

Fig. B-28. Laboratory means for sulfur in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp B (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3232, is compared to the individual laboratory mean for a second sample, Kelp B. The dotted blue box represents the consensus range of tolerance for SRM 3232 (x-axis) and Kelp B (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

Table B-16. Data summary table for zinc in kelp.

Data highlighted in blue have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable Z'_{comm} score, $|Z'_{comm}| > 2$. (Table continues to next page.)

		Zinc														
		Kelp A (ug/g)					Kelp B (ug/g)					SRM 3232 Kelp Powder (*Thallus laminariae*) (ug/g)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
Individual Results	Target				18.6	1.70				32.0	3.20				25.7	1.03
	A001															
	A002															
	A003															
	A004															
	A005															
	A009	13.48	142.72	77.57	77.9	64.6	28.04	46.87	40.47	38.5	9.57	20.25	34.03	60.84	38.4	20.6
	A012	16.9	17.4	16.9	17.1	0.29	31	30.2	30.3	30.5	0.44	24.4	25.1	26.1	25.2	0.85
	A013	16.433	15.83	15.3287	15.9	0.55	27.3297	29.3591	27.0224	27.9	1.27	20.474	21.8525	21.7446	21.4	0.77
	A015															
	A016	16.1	16	16.1	16.1	0.06	29.3	29.1	29	29.1	0.15	22.6	22.9	23.3	22.9	0.35
	A017	22.8	23.2	26	24.0	1.74	33.5	36	33.9	34.5	1.34	28.4	28.8	27.9	28.4	0.45
	A018	18	18.1	18.2	18.1	0.10	29.5	27.6	27.3	28.1	1.19	25.5	24.2		24.9	0.92
	A020															
	A022															
	A024	10.869	10.871	10.86	10.9	0.01	20.9425	20.9431	20.9516	20.9	0.01	18.06	18.101	18.094	18.1	0.02
	A025															
	A027	19.459	23.915	19.406	20.9	2.59	35.13	30.258	28.276	31.2	3.53	26.13	25.047	26.133	25.8	0.63
	A028															
	A029	17.5	17.2	17.2	17.3	0.17	30.8	30.6	30.1	30.5	0.36	23.3	23.8	24.3	23.8	0.50
	A031	36	35	34	35.0	1.00	53	49	49	50.3	2.31	43	49	42	44.7	3.79
	A032															
	A033	22	19.6	19.1	20.2	1.55	34	33	36	34.3	1.53	27.1	29.7	21.7	26.2	4.08
	A035	20.5	21	20.3	20.6	0.36	33.1	35	34.2	34.1	0.95	27.9	28.2	28.7	28.3	0.40
	A037	16.9	15.3	15.1	15.8	0.99	27.8	27	26.1	27.0	0.85	20	17.6	17.5	18.4	1.42
	A039															
	A041	16.738	16.298	16.574	16.5	0.22	30.234	29.671	30.386	30.1	0.38	24.328	24.206	23.893	24.1	0.22
	A042	16.5	15.7	15.9	16.0	0.42	26.4	26.6	27.6	26.9	0.64	22	22.8	22.8	22.5	0.46
	A043															

		Zinc														
		Kelp A (ug/g)					Kelp B (ug/g)					SRM 3232 Kelp Powder (*Thallus laminariae*) (ug/g)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
	A044	16.8	16.3	16.5	16.5	0.25	29	29.5	30.6	29.7	0.82	25.5	25.4	24.9	25.3	0.32
	A045	43.29	48.79	47.23	46.4	2.83	50.56	74.59	67.93	64.4	12.4	52.22	59.23	57.53	56.3	3.66
	A046	16.216	15.062	15.321	15.5	0.61	34.469	31.189	28.09	31.2	3.19	24.461	21.698	21.793	22.7	1.57
	A047	18	17		17.5	0.71	31	32		31.5	0.71	27	27		27	0
	A050						29	29	28	28.7	0.58					
	A051															
	A052															
	A053															
	A054	19.1	18.9	19.7	19.2	0.42	34.2	33.7	33.7	33.9	0.29	39.1	27.2	27.7	31.3	6.73
	A057	19.1	18.8	19.2	19.0	0.21	33.9	33.6	33.8	33.8	0.15	26.8	26.4	27.3	26.8	0.45
	A059	21	21	21	21	0	29	30	30	29.7	0.58	27	28	26	27.0	1.00
	A060	15.1587	16.9876	18.5687	16.9	1.71	40.3686	34.3637	34.1786	36.3	3.52	25.3345	26.1369	28.6977	26.7	1.76
	A061															
	A062															
	A065	18.98	18.96	18.31	18.8	0.38	34.99	34.74	34.8	34.8	0.13	27.11	27.08	26.16	26.8	0.54
	A066	17.23	17.65	16.9	17.3	0.38	28.84	28.86	28.7	28.8	0.09	22.75	22.59	23.49	22.9	0.48
	A068															
	A070	19.24	20.02	22.55	20.6	1.73	33.39	37.7	38.78	36.6	2.85	27.11	29.02	32.98	29.7	2.99
	A071															
	A073															
	A075	19.9384	19.2753	19.3957	19.5	0.35	32.1297	31.5749	30.857	31.5	0.64	25.4644	24.6612	24.7417	25.0	0.44
	A079	16	18	21	18.3	2.52	37	37	33	35.7	2.31	28	29	33	30.0	2.65
Community Results		Consensus Mean			18.0		Consensus Mean			31.5		Consensus Mean			25.6	
		Consensus Standard Deviation			3.12		Consensus Standard Deviation			4.37		Consensus Standard Deviation			3.98	
		Maximum			77.9		Maximum			64.4		Maximum			56.3	
		Minimum			10.9		Minimum			20.9		Minimum			18.1	
		N			28		N			29		N			28	

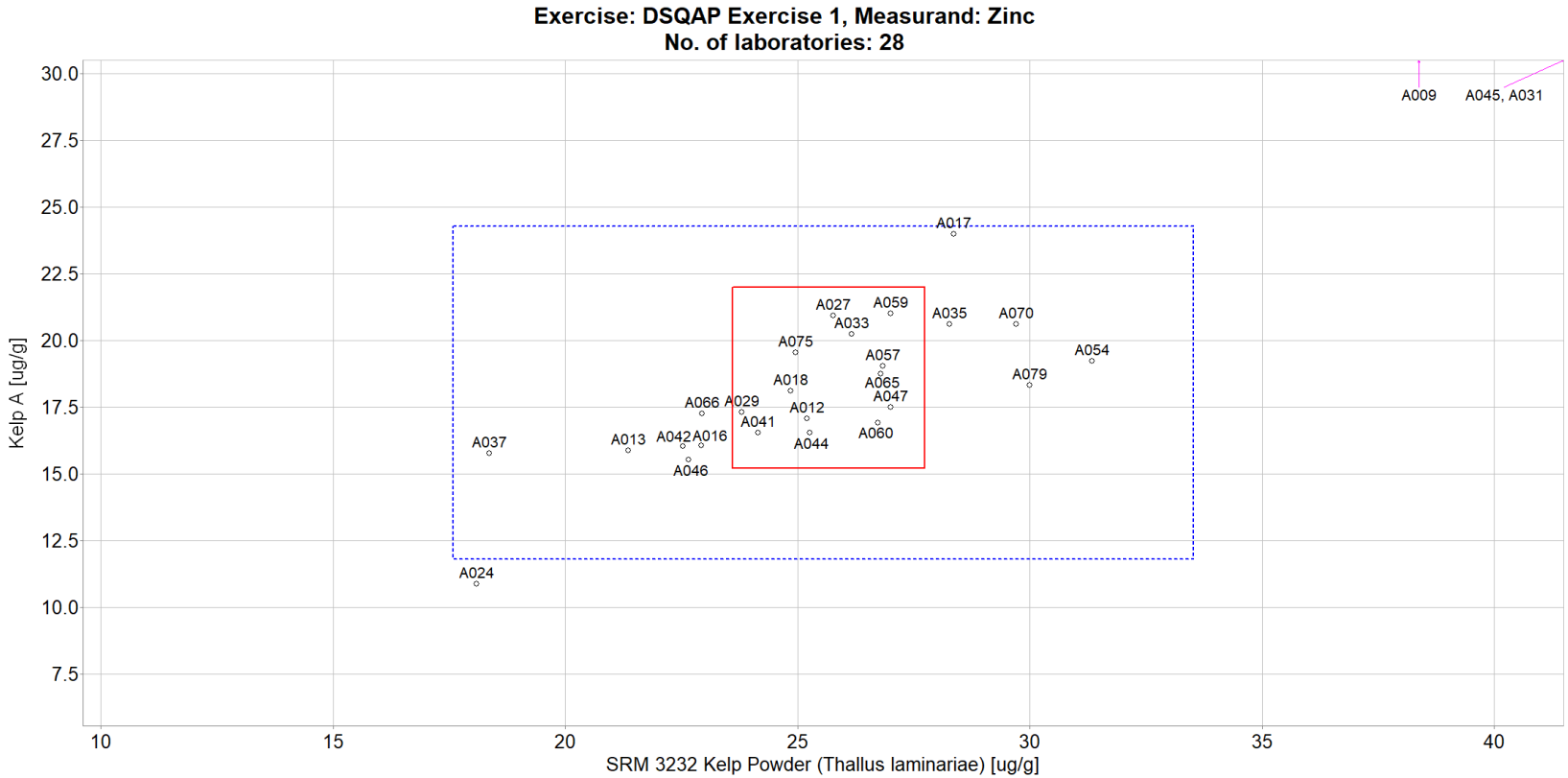

Fig. B-29. Laboratory means for zinc in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp A (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3232, is compared to the individual laboratory mean for a second sample, Kelp A. The solid red box represents the NIST range of tolerance for the two samples, SRM 3232 (x-axis) and Kelp A (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3232 (x-axis) and Kelp A (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

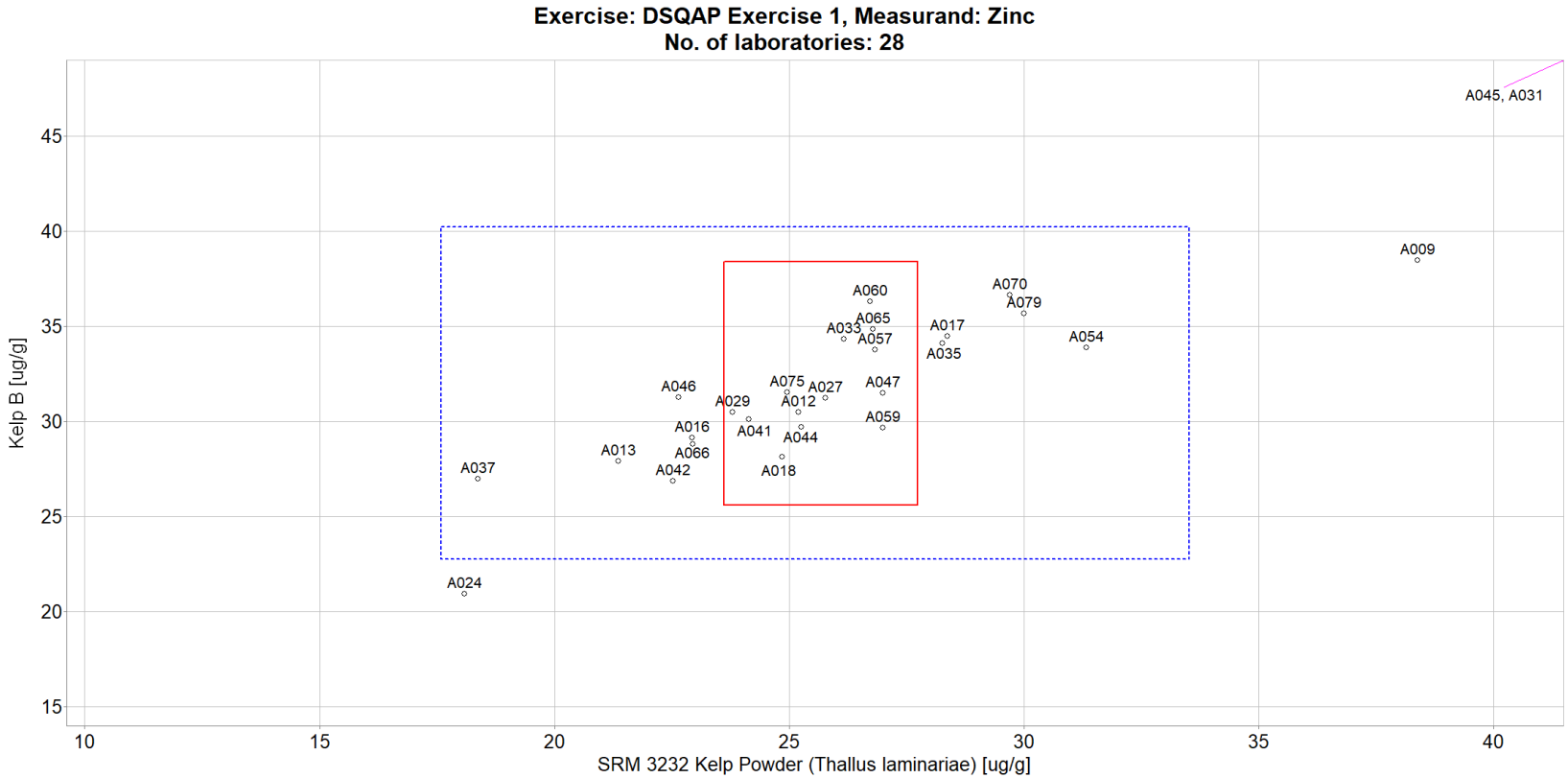

Fig. B-30. Laboratory means for zinc in SRM 3232 Kelp Powder (*Thallus laminariae*) and Kelp B (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3232, is compared to the individual laboratory mean for a second sample, Kelp B. The solid red box represents the NIST range of tolerance for the two samples, SRM 3232 (x-axis) and Kelp B (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3232 (x-axis) and Kelp B (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

Appendix C. Vitamins I Supplemental Tables and Figures

Table C-1. Individualized data summary table (example) for vitamins in kelp.

(Laboratory Name)

Exercise 1 - Vitamins I - Kelp

	Lab Code:	**(code)**	**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*
Niacin	Kelp A	mg/kg	*Individual laboratory results will appear in this section; Laboratory-specific results were provided to each participant separately from this report*				3	38.46	13.85		
Niacin	Kelp B	mg/kg					3	8.30	6.34		
Niacin	SRM 3232 Kelp	mg/kg					3	13.01	10.48		
Niacinamide	Kelp A	mg/kg					2	12.03	49.66		
Niacinamide	Kelp B	mg/kg					3	9.55	4.39		
Niacinamide	SRM 3232 Kelp	mg/kg					2	8.77	36.49		
Phylloquinone	Kelp A	mg/kg					4	3.75	4.48		
Phylloquinone	Kelp B	mg/kg					4	1.70	0.50	1.94	0.50
Phylloquinone	SRM 3232 Kelp	mg/kg					4	0.47	0.91	0.40	0.08

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

Table C-2. Data summary table for niacin in kelp.

		Vitamin B3 (Niacin)														
		Kelp A (mg/kg)					Kelp B (mg/kg)					SRM 3232 Kelp Powder (*Thallus laminariae*) (mg/kg)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
Individual Results	Target															
	A001															
	A002															
	A006															
	A018															
	A020	33.6	32.5	34.1	33.40	0.82	12.6	12.5	12.3	12.47	0.15	22.3	21.5	20.6	21.47	0.85
	A021	< 100.0	< 100.0	< 100.0			< 100.0	< 100.0	< 100.0			< 100.0	< 100.0	< 100.0		
	A022															
	A025															
	A027	< 0.836	< 0.836	< 0.836			< 0.836	< 0.836	< 0.836			< 0.836	< 0.836	< 0.836		
	A032															
	A034															
	A035															
	A036	54.28			54.28		2.69			2.69		0.306			0.306	
	A037															
	A039															
	A041															
	A042	28.29	26.69	28.08	27.69	0.87	9.93	9.5	9.79	9.74	0.22	17.72	16.94	17.15	17.27	0.40
	A043															
	A046															
	A057															
	A062															
	A063															
	A068															
	A075	< 626.1	< 626.1	< 626.1			< 683.3	< 683.3	< 683.3			< 738.5	< 738.5	< 738.5		
	A077															
Community Results		Consensus Mean			38.46		Consensus Mean			8.30		Consensus Mean			13.01	
		Consensus Standard Deviation			13.85		Consensus Standard Deviation			6.34		Consensus Standard Deviation			10.48	
		Maximum			54.28		Maximum			12.47		Maximum			21.47	
		Minimum			27.69		Minimum			2.69		Minimum			0.31	
		N			2		N			2		N			2	

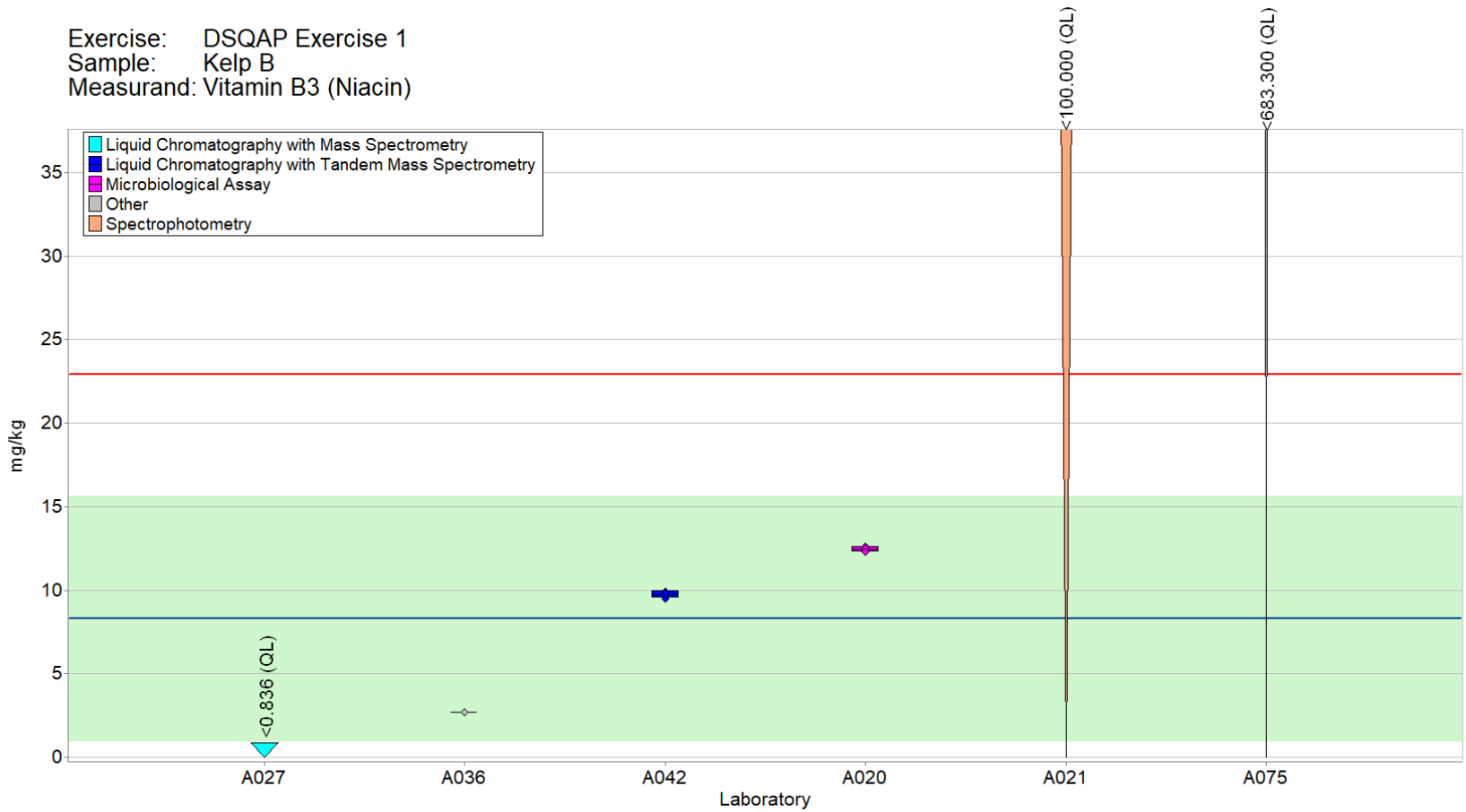

Fig. C-1. Vitamin B_3 (niacin) in Kelp B (data summary view – analytical method).

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 bound of the consensus range of tolerance, calculated as the value above the consensus mean that results in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$, with the lower bound set to zero.

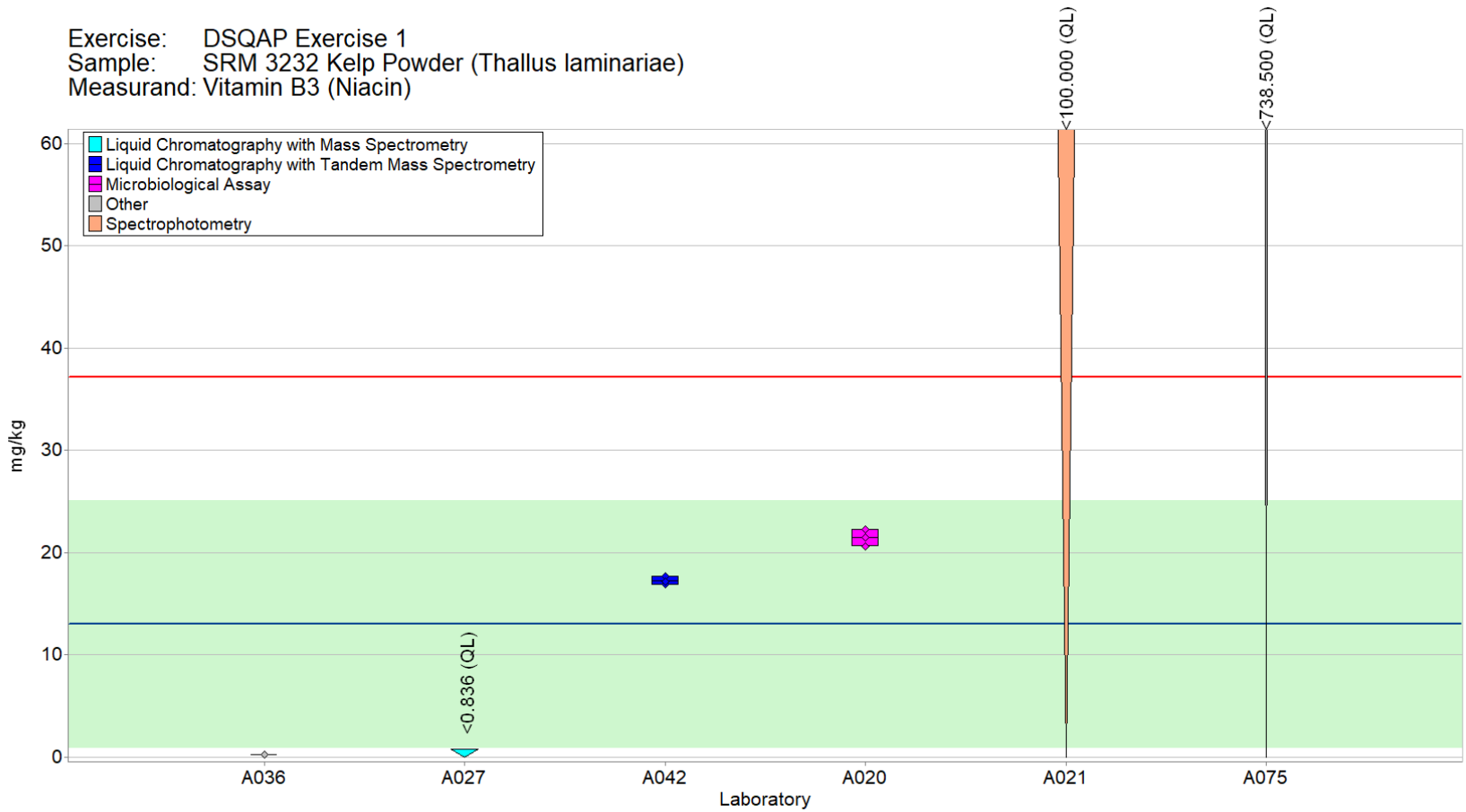

Fig. C-2. Vitamin B_3 (niacin) in SRM 3232 Kelp Powder (*Thallus laminariae*) (data summary view – analytical method).

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 bound of the consensus range of tolerance, calculated as the value above the consensus mean that results in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$, with the lower bound set to zero.

Table C-3. Data summary table for niacinamide in kelp.

Data highlighted in blue have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable Z'_{comm} score, $|Z'_{comm}| > 2$.

		Vitamin B3 (Niacinamide)														
		Kelp A (mg/kg)					Kelp B (mg/kg)					SRM 3232 Kelp Powder (*Thallus laminariae*) (mg/kg)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
Individual Results	Target															
	A001															
	A002															
	A006															
	A018															
	A020															
	A022															
	A027	0.583	0.525	0.523	0.54	0.03	31	26	27	28.00	2.65	0.35	0.467	0.418	0.41	0.06
	A032															
	A034															
	A037															
	A039															
	A041															
	A042	23.74	22.9	23.91	23.52	0.54	9.85	9.42	9.71	9.66	0.22	17.58	16.81	17.01	17.13	0.40
	A043															
	A045						7.5142	8.64	8.234	8.13	0.57					
	A046															
	A057															
	A063															
	A068															
	A075	< 595.0	< 595.0	< 595.0			< 649.4	< 649.4	< 649.4			< 701.8	< 701.8	< 701.8		
Community Results		Consensus Mean			12.03		Consensus Mean			9.55		Consensus Mean			8.77	
		Consensus Standard Deviation			49.66		Consensus Standard Deviation			4.39		Consensus Standard Deviation			36.49	
		Maximum			23.52		Maximum			28.00		Maximum			17.13	
		Minimum			0.54		Minimum			8.13		Minimum			0.41	
		N			2		N			3		N			2	

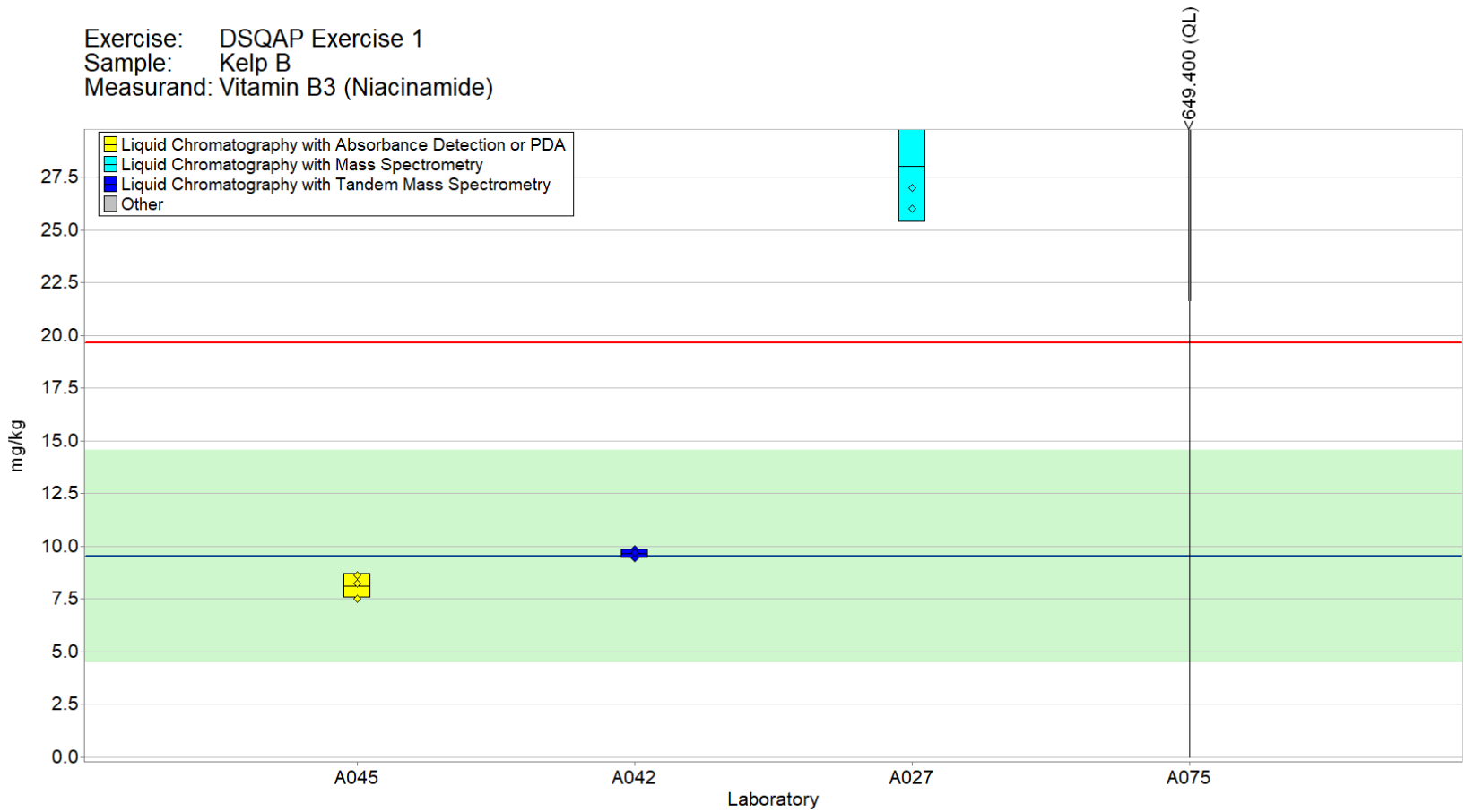

Fig. C-3. Vitamin B_3 (niacinamide) in Kelp B (data summary view – analytical method).

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 bound of the consensus range of tolerance, calculated as the value above the consensus mean that results in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$, with the lower bound set to zero.

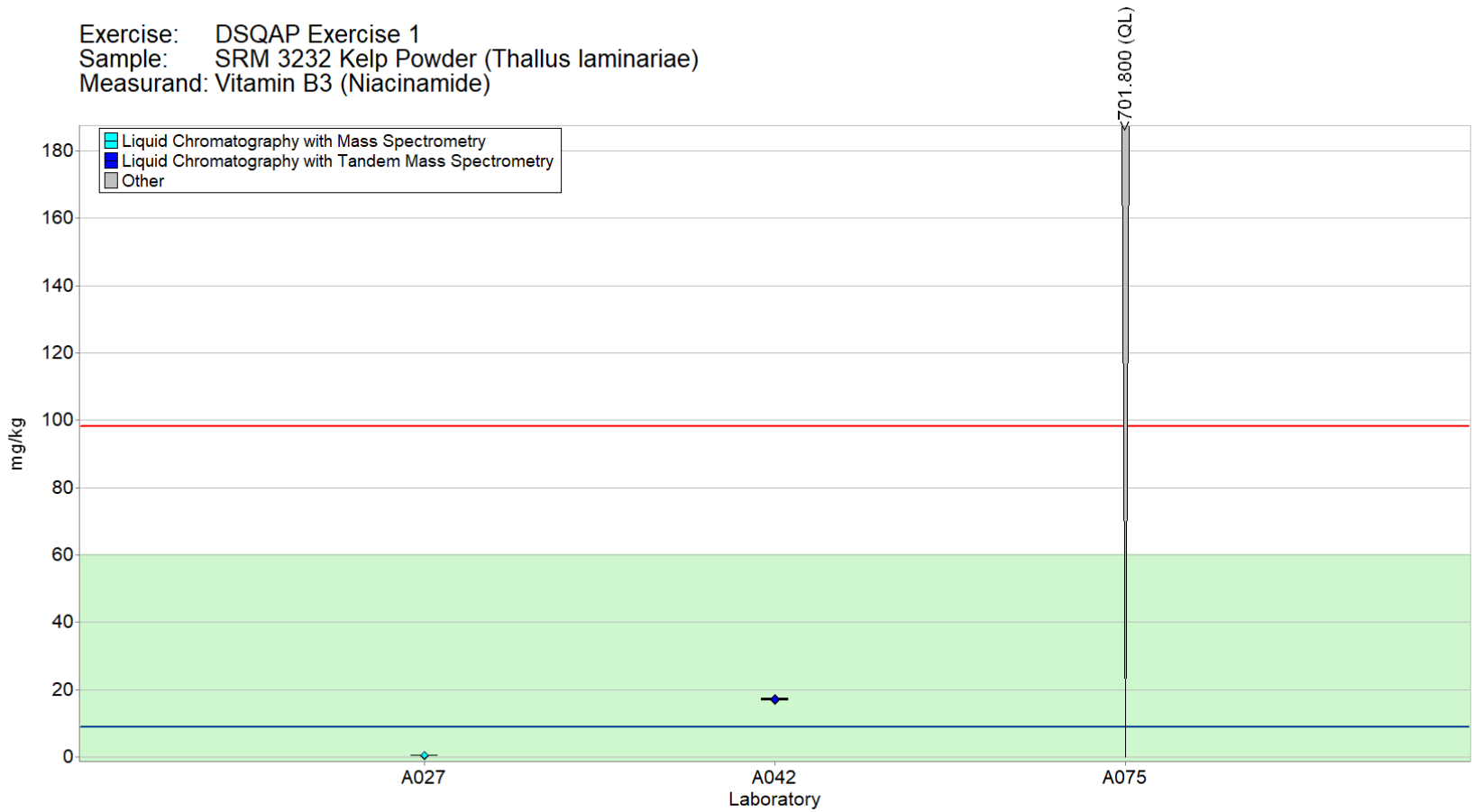

Fig. C-4. Vitamin B_3 (niacinamide) in SRM 3232 Kelp Powder (*Thallus laminariae*) (data summary view – analytical method).

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 bound of the consensus range of tolerance, calculated as the value above the consensus mean that results in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$, with the lower bound set to zero.

Table C-4. Data summary table for phylloquinone in kelp.

Data highlighted in blue have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable Z'_{comm} score, $|Z'_{comm}| > 2$.

		Phylloquinone (Vitamin K1)														
		Kelp A (mg/kg)					Kelp B (mg/kg)					SRM 3232 Kelp Powder (*Thallus laminariae*) (mg/kg)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
Individual Results	Target									1.94	1.00				0.40	0.08
	A001															
	A002															
	A006															
	A007															
	A018															
	A020	1.486	2.042	1.492	1.67	0.32	0.799	0.81	0.532	0.71	0.16	0.162	0.123	0.174	0.15	0.03
	A022															
	A027															
	A032															
	A034															
	A037															
	A041	7.2	7.35	7.32	7.29	0.08	2.69	2.67	2.67	2.68	0.01	0.072	0.071	0.07	0.07	0.001
	A042															
	A043	5.1	5.3	6.2	5.53	0.59	2.8	2.9	3.1	2.93	0.15	0.6	0.6	0.7	0.63	0.06
	A045	0.4844	0.5001	0.4792	0.49	0.01	0.6203	0.6452	0.6899	0.65	0.04	1.0112	1.0283	1.0124	1.02	0.01
	A057															
	A063															
	A068															
	A075															
Community Results		Consensus Mean			3.75		Consensus Mean			1.70		Consensus Mean			0.47	
		Consensus Standard Deviation			4.48		Consensus Standard Deviation			0.50		Consensus Standard Deviation			0.91	
		Maximum			7.29		Maximum			2.93		Maximum			1.02	
		Minimum			0.49		Minimum			0.65		Minimum			0.07	
		N			4		N			4		N			4	

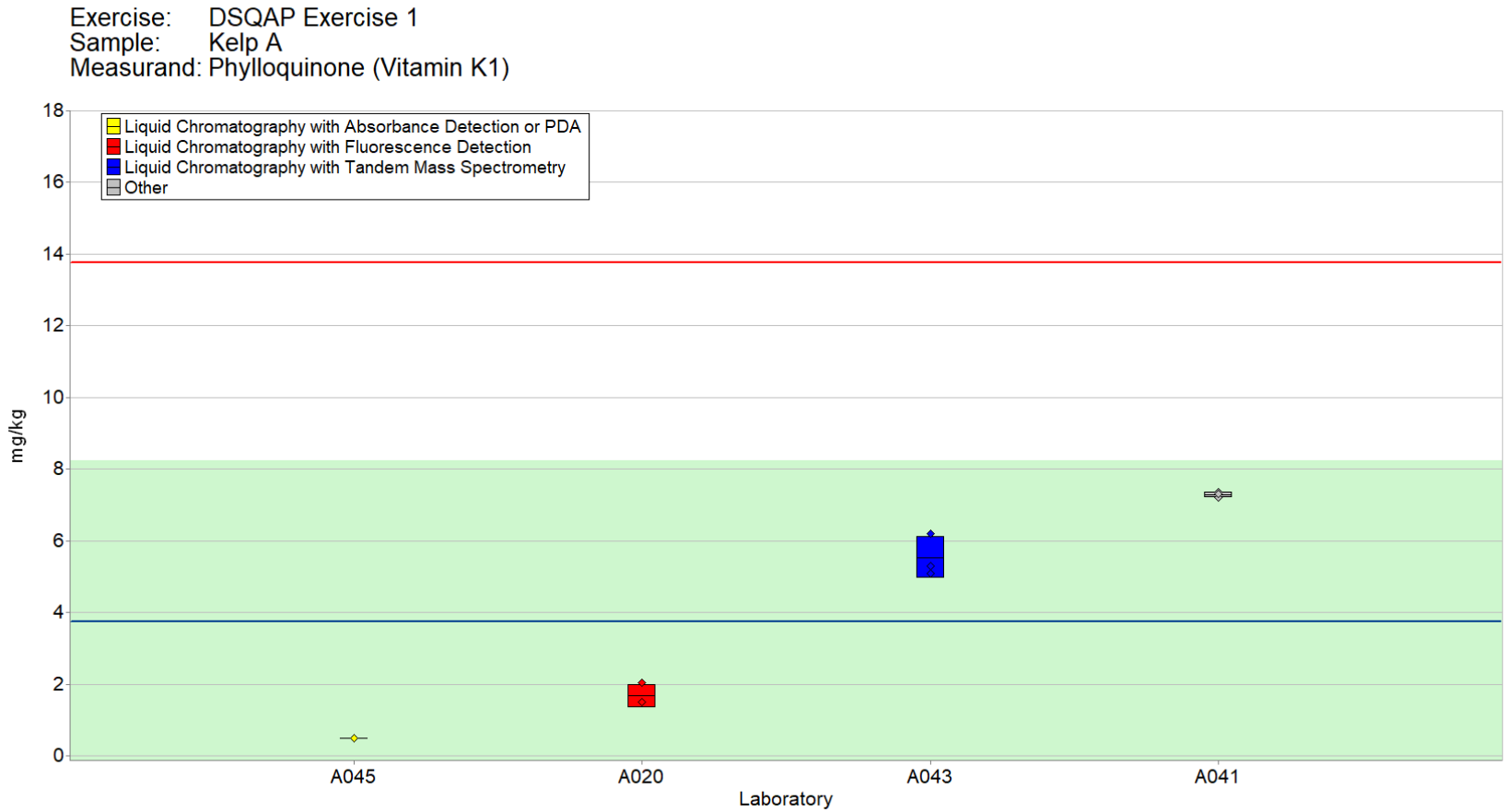

Fig. C-5. Vitamin K (phylloquinone) in Kelp A (data summary view – analytical method).

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 bound of the consensus range of tolerance, calculated as the value above the consensus mean that results in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$, with the lower bound set to zero.

Appendix D. Botanicals I Supplemental Tables and Figures

Table D-1. Individualized data summary table (example) for botanicals in kelp and green tea.

(Laboratory Name)

Exercise 1 - Botanicals I - Phenolic Content

		Lab Code:	**(code)**	**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*
Gallic Acid	SRM 3254 Green Tea Leaves	% w/w		*Individual laboratory results will appear in this section; Laboratory-specific results were provided to each participant separately from this report*				7	0.08	0.05	0.11	0.06
Gallic Acid	SRM 3255 Green Tea Extract	% w/w						8	0.33	0.06	0.31	0.01
Gallic Acid	Kelp A	% w/w						2	1.27	3.88		
Gallic Acid Equivalents	SRM 3254 Green Tea Leaves	% w/w						10	15.06	4.57		
Gallic Acid Equivalents	SRM 3255 Green Tea Extract	% w/w						11	80.94	9.25		
Gallic Acid Equivalents	Kelp A	% w/w						8	0.26	0.17		

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

Table D-2. Data summary table for gallic acid in SRM 3254 Green Tea *(Camellia sinensis)* Leaves, SRM 3255 Green Tea *(Camellia sinensis)* Extract, and Kelp A.

Data points highlighted in blue have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable Z'_{comm} score, $|Z'_{\text{comm}}| \geq 2$.

		Gallic Acid														
		SRM 3254 Green Tea (*Camellia sinensis*) Leaves (% w/w)					SRM 3255 Green Tea (*Camellia sinensis*) Extract (% w/w)					Kelp A (% w/w)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
Individual Results	Target				0.11	0.06				0.31	0.01					
	A001															
	A002															
	A007															
	A014															
	A021	0.087	0.08	0.076	0.08	0.01	0.313	0.328	0.331	0.32	0.01	< 0.010	< 0.010	< 0.010		
	A023	0.0909	0.1061	0.0908	0.10	0.01	0.3155	0.3088	0.3196	0.31	0.01	< 0.000	< 0.000	< 0.000		
	A024	0.07	0.08	0.08	0.08	0.01	0.3	0.29	0.29	0.29	0.01	< 0.003	< 0.003	< 0.003		
	A028															
	A032															
	A034															
	A038															
	A041															
	A042															
	A045															
	A046	0.31	0.27	0.28	0.29	0.02	0.45	0.44	0.42	0.44	0.02	0.003	0.004	0.003	0.003	0.001
	A048	0.09	0.09	0.1	0.09	0.01	0.33	0.33	0.32	0.33	0.01	< 0.001	< 0.001	< 0.001		
	A049															
	A057															
	A060	0.038	0.048	0.051	0.05	0.01	0.33	0.33	0.29	0.32	0.02	< 0.010	< 0.010	< 0.010		
	A063															
	A076						32.82	35.38	35.41	34.54	1.49					
	A077															
	A080	114.97	107.22	114.31	112.17	4.30	114.64	114.46	116.11	115.07	0.91	1.51	3.12	2.95	2.53	0.88
Community Results		Consensus Mean			0.08		Consensus Mean			0.33		Consensus Mean			1.27	
		Consensus Standard Deviation			0.05		Consensus Standard Deviation			0.06		Consensus Standard Deviation			3.88	
		Maximum			112.17		Maximum			115.07		Maximum			2.53	
		Minimum			0.05		Minimum			0.29		Minimum			0.00	
		N			7		N			8		N			2	

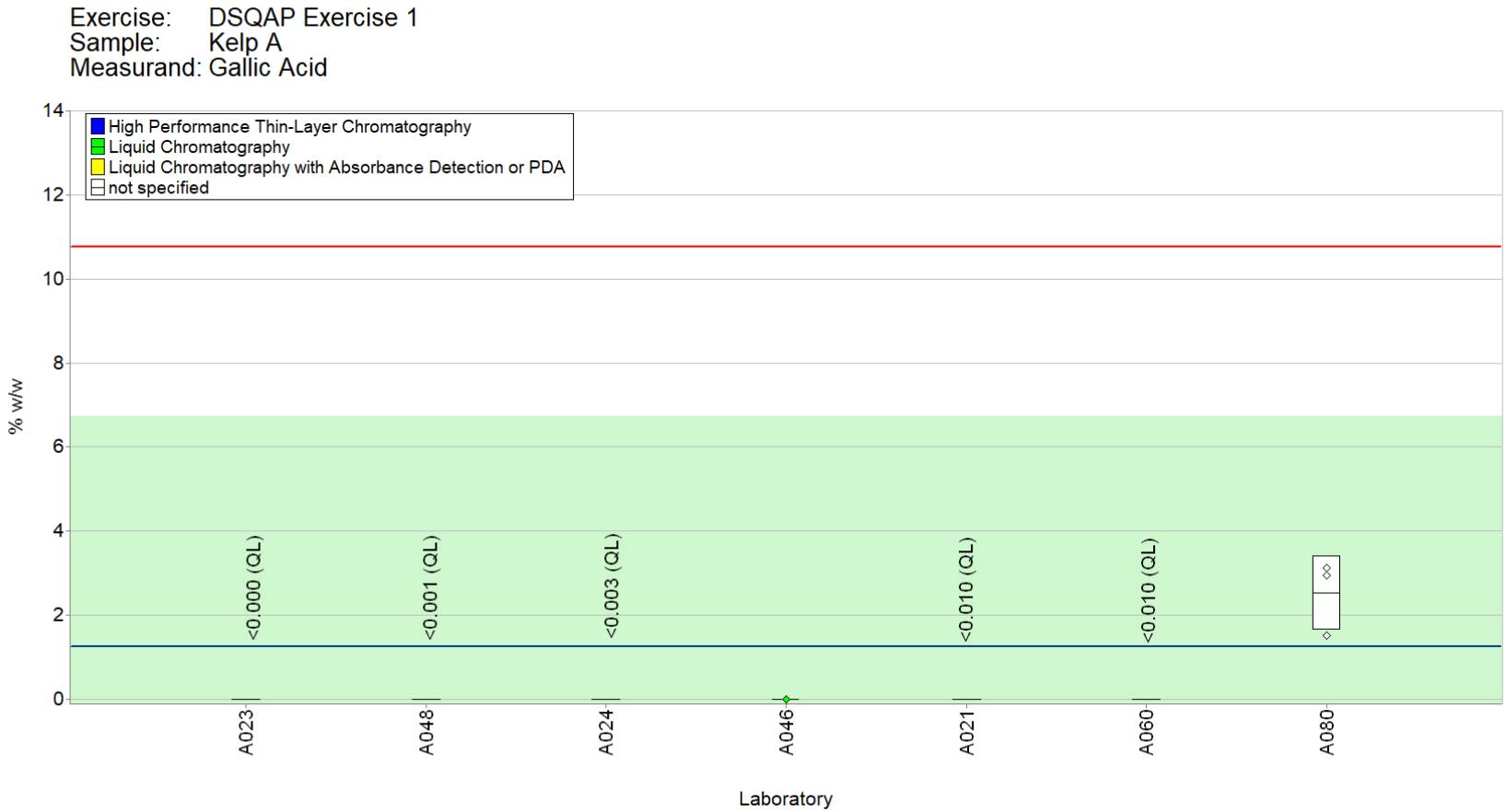

Fig. D-1. Gallic acid in Kelp A (data summary view – analytical method).

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 red solid line represents the upper bound of the consensus range of tolerance, calculated as the value above the consensus mean that results in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$, with the lower bound set to zero.

Table D-3. Data summary table for gallic acid equivalents SRM 3254 Green Tea *(Camellia sinensis)* Leaves, SRM 3255 Green Tea *(Camellia sinensis)* Extract, and Kelp A.

		Gallic Acid Equivalents														
		SRM 3254 Green Tea (*Camellia sinensis*) Leaves (% w/w)					**SRM 3255 Green Tea (*Camellia sinensis*) Extract (% w/w)**					**Kelp A (% w/w)**				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
Individual Results	Target															
	A001															
	A002															
	A007	16.3	16.6	16.2	16.37	0.21	75	76.5	76.5	76.00	0.87	0.508	0.497	0.459	0.49	0.03
	A014	12.9	13	13	12.97	0.06	68.7	67.3	68	68.00	0.70	0.18	0.19	0.19	0.19	0.01
	A021															
	A023	13.3883	12.0766	12.1824	12.55	0.73	73.2408	76.4367	77.6845	75.79	2.29	0.5197	0.3523	0.3528	0.41	0.10
	A024	13.86	14.59	15.43	14.63	0.79	79.15	75.23	78.29	77.56	2.06	0.23	0.17	0.22	0.21	0.03
	A028															
	A032															
	A034															
	A038															
	A039															
	A041						80.73	82.07	80.67	81.16	0.79					
	A042	10.01	10.02	10.12	10.05	0.06	76.79	77.04	76.74	76.86	0.16	0.23	0.23	0.23	0.23	0
	A045	26.35	20.47	22.75	23.19	2.96	93.367	95.89	87.65	92.30	4.22	0.064	0.0587	0.0592	0.06	0.003
	A046	21.5	21.6	20.8	21.30	0.44	88.2	90.4	87.2	88.60	1.64	< 0.100	< 0.100	< 0.100		
	A048	14.55	13.91	14.01	14.16	0.34	82.38	82.52	83.25	82.72	0.47	0.31	0.33	0.33	0.32	0.01
	A049															
	A057															
	A058	14.8	16.1	16.9	15.93	1.06	72.2	79.9	77.5	76.53	3.94	0.25	0.16	0.18	0.20	0.05
	A060															
	A063															
	A064	10.9	10.8	10.4	10.70	0.26	94.1	95.7	97.1	95.63	1.50	< 0.600	< 0.600	< 0.600		
	A076															
	A077															
	A080															
Community Results		Consensus Mean			15.06		Consensus Mean			80.94		Consensus Mean			0.26	
		Consensus Standard Deviation			4.57		Consensus Standard Deviation			9.25		Consensus Standard Deviation			0.17	
		Maximum			23.19		Maximum			95.63		Maximum			0.49	
		Minimum			10.05		Minimum			68.00		Minimum			0.06	
		N			10		N			11		N			8	

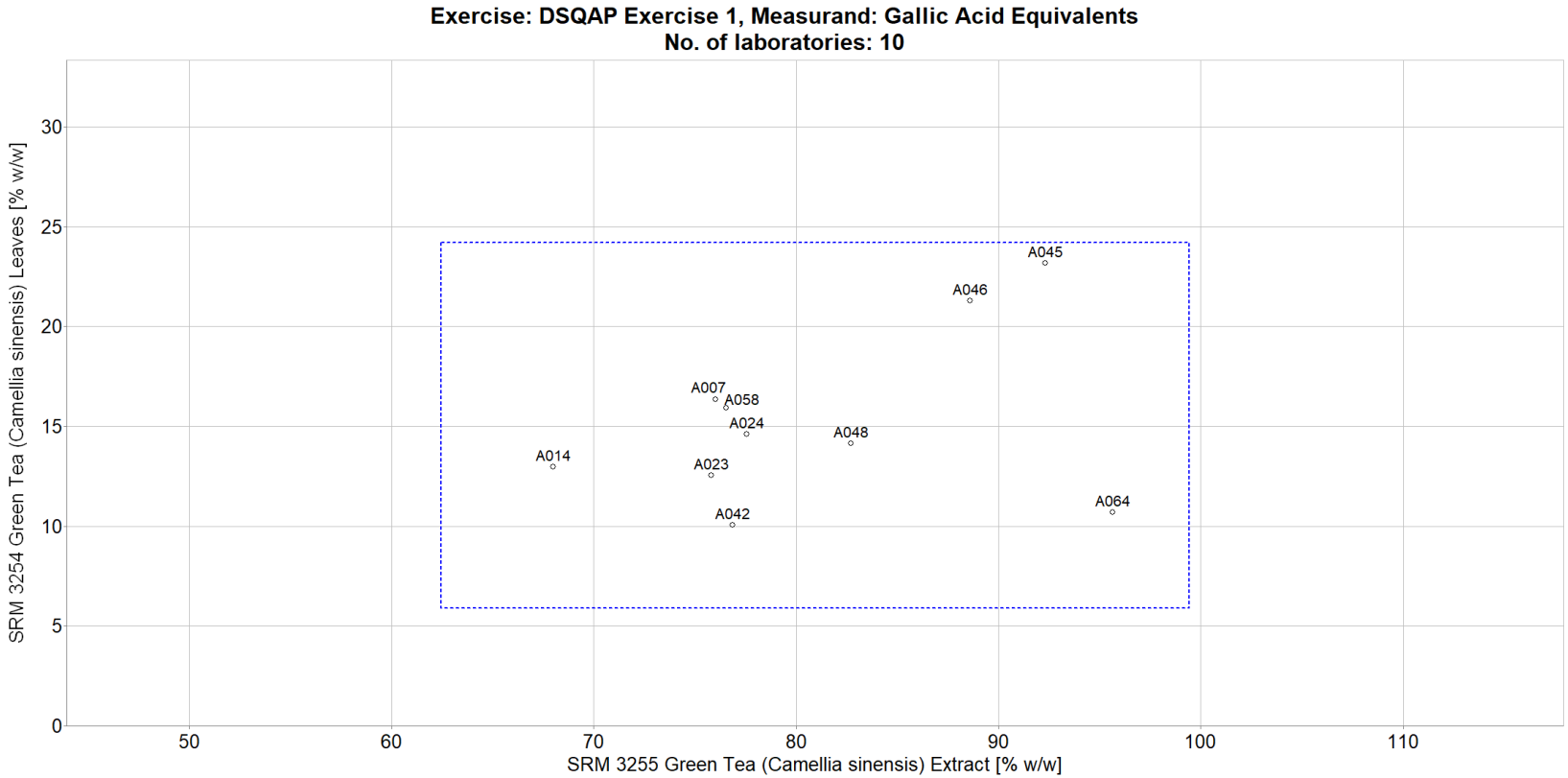

Fig. D-2. Laboratory means for gallic acid equivalents in SRM 3255 Green Tea *(Camellia sinensis)* Extract and SRM 3254 Green Tea *(Camellia sinensis)* Leaves (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3255, is compared to the individual laboratory mean for a second sample, SRM 3254. The dotted blue box represents the consensus range of tolerance for SRM 3255 (x-axis) and SRM 3254 (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

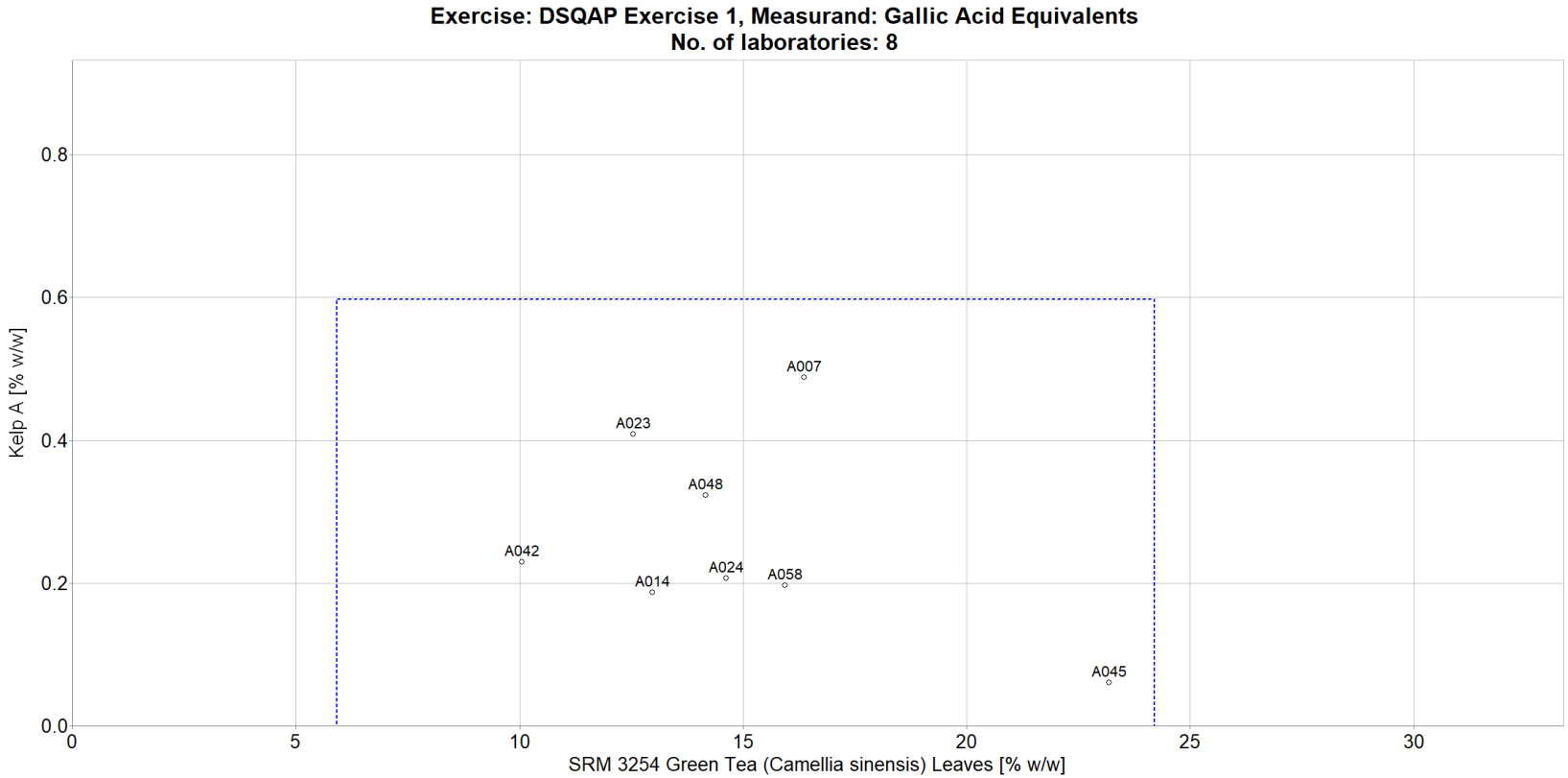

Fig. D-3. Laboratory means for gallic acid equivalents in SRM 3254 Green Tea *(Camellia sinensis)* Leaves and Kelp A (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3254, is compared to the individual laboratory mean for a second sample, Kelp A. The dotted blue box represents the consensus range of tolerance for SRM 3254 (x-axis) and Kelp A (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

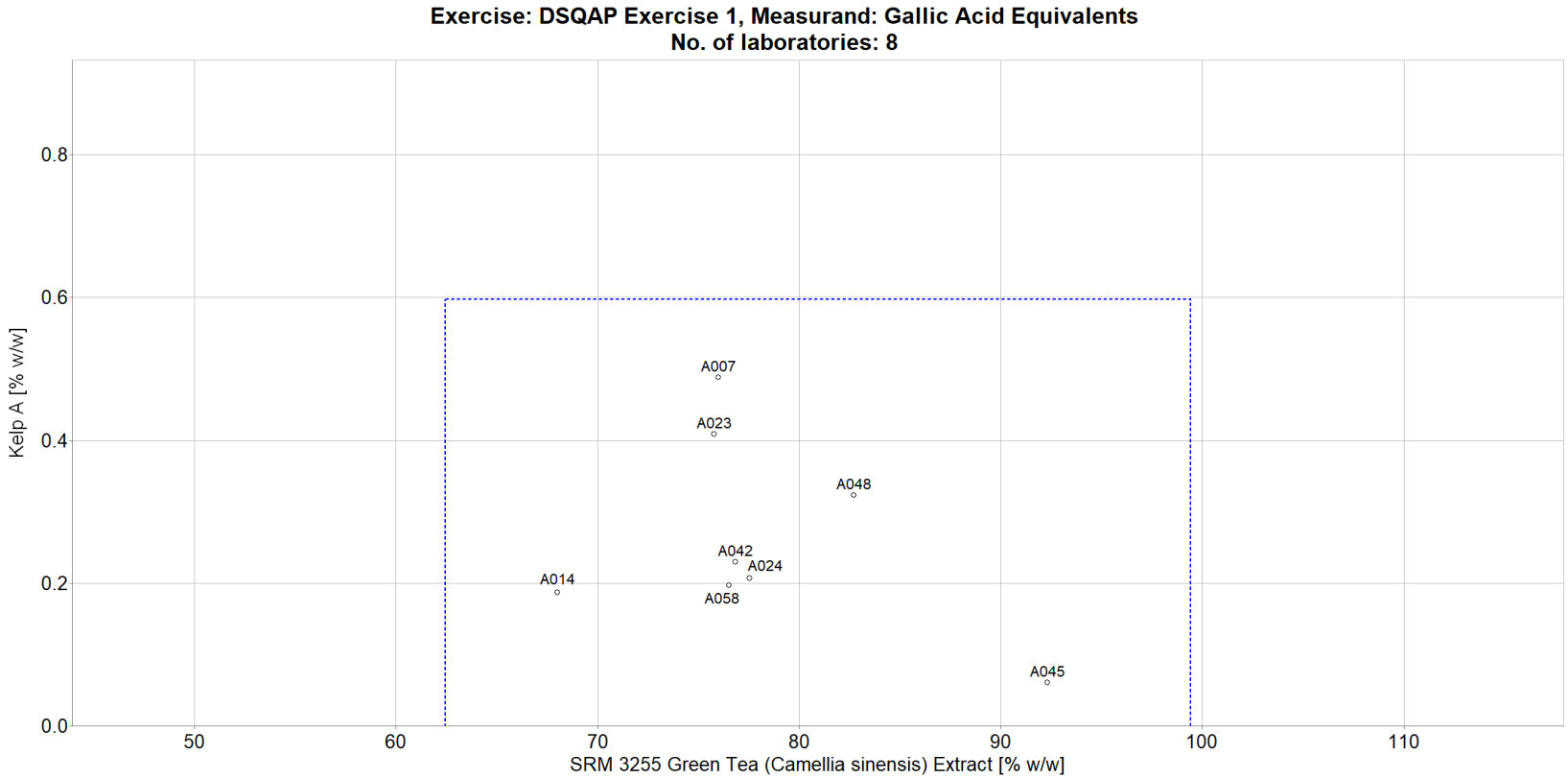

Fig. D-4. Laboratory means for gallic acid equivalents in SRM 3255 Green Tea *(Camellia sinensis)* Extract and Kelp A (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3255, is compared to the individual laboratory mean for a second sample, Kelp A. The dotted blue box represents the consensus range of tolerance for SRM 3255 (x-axis) and Kelp A (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

Appendix E. Proximates Supplemental Tables and Figures

Table E-1. Individualized data summary table (example) for proximates in kelp.

(Laboratory Name)

Exercise 1 - Proximates in Kelp

	Lab Code:	**(code)**	**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*
Ash	Kelp A	%	*Individual laboratory results will appear in this section; Laboratory-specific results were provided to each participant separately from this report*				22	37.80	0.67		
Carbohydrates	Kelp A	%					11	40.00	10.7		
Fat	Kelp A	%					19	1.38	0.50		
Protein	Kelp A	%					20	11.40	0.58		
Solids	Kelp A	%					13	96.90	0.56		
Starch	Kelp A	%					3	0.68	0.90		
TDF	Kelp A	%					9	27.50	6.68		
Calories	Kelp A	kcal/100g					8	234	22.6		

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

Table E-2. Data summary table for ash in kelp.

Data points highlighted in blue have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable Z'_{comm} score, $|Z'_{comm}| \geq 2$.

		Ash				
		Kelp A (%)				
	Lab	A	B	C	Avg	SD
Individual Results	Target					
	A001					
	A003	37.93	37.64	37.86	37.8	0.15
	A009	37.55	37.57	37.54	37.6	0.02
	A015					
	A016	38	38	38	38	0
	A019	37.59	37.64	35.58	36.9	1.18
	A020	35.11	34.65	35.05	34.9	0.25
	A022					
	A024	37.5	37.32	37.35	37.4	0.10
	A026	38.43			38.4	
	A028					
	A031	37.69	37.7	37.66	37.7	0.02
	A032					
	A035	36.4	36.4	36.4	36.4	0
	A037	37.94	37.92	37.91	37.9	0.02
	A038					
	A039					
	A040					
	A042	37.73	37.76	37.62	37.7	0.07
	A045	36.82	37.37	37.12	37.1	0.28
	A046	33.5	34.7	34.1	34.1	0.60
	A047	38.08	38.23		38.2	0.11
	A048	38.26	38.29	38.37	38.3	0.06
	A050					
	A052					
	A053					
	A055	37.84	37.47	37.47	37.6	0.21
	A057	37.75	38.12	37.76	37.9	0.21
	A059	38.12	38.16	38.18	38.2	0.03
	A065	38.7	38.6	38.7	38.7	0.06
	A068					
	A069	37.56	37.54	37.48	37.5	0.04
	A075					
	A076	4.037	4.368	4.197	4.20	0.17
	A080	37.92	37.93	37.94	37.9	0.01
Community Results		Consensus Mean			37.8	
		Consensus Standard Deviation			0.67	
		Maximum			38.7	
		Minimum			4.20	
		N			21	

Table E-3. Data summary table for carbohydrates in kelp.

		Carbohydrates				
		Kelp A (%)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**
Individual Results	Target					
	A001					
	A009	19.1	18.65	21.2	19.7	1.36
	A015					
	A020					
	A022					
	A028					
	A031					
	A032					
	A035	47	47.5	47.2	47.2	0.25
	A037	45.13	45.28	45.94	45.5	0.43
	A038					
	A039					
	A040					
	A042	35.5	35.71	36.05	35.8	0.28
	A045	31.45	31.25	30.89	31.2	0.28
	A046	42.2	39.9	41.1	41.1	1.15
	A047	46.5	46.2		46.4	0.21
	A048	51.9	51.5	51	51.5	0.45
	A050					
	A052					
	A053					
	A055	41.29	39.21	39.01	39.8	1.26
	A057	44.39	44.29	50.8	46.5	3.73
	A065					
	A068					
	A069					
	A075					
	A076					
	A080	30.68	31.37	31.8	31.3	0.57
Community Results		Consensus Mean			40.0	
		Consensus Standard Deviation			10.7	
		Maximum			51.5	
		Minimum			19.7	
		N			11	

Table E-4. Data summary table for fat in kelp.

Data points highlighted in blue have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable Z'_{comm} score, $|Z'_{comm}| \geq 2$.

		Fat				
		Kelp A (%)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**
Individual Results	Target					
	A001					
	A003	1.047	0.9592	1.016	1.01	0.04
	A005					
	A009	3.61	3.59	3.49	3.56	0.06
	A016	1.65	1.65	1.69	1.66	0.02
	A020	1.1	0.85	1.1	1.02	0.14
	A022					
	A026	1.5			1.5	
	A028					
	A031	0.82	0.84	0.84	0.83	0.01
	A032					
	A035	1.82	1.49	1.64	1.65	0.17
	A037	2.2	2.18	1.6	1.99	0.34
	A038					
	A039					
	A040					
	A042	1.4	1.41	1.36	1.39	0.03
	A045					
	A046	1.4	1.7	1.5	1.53	0.15
	A047	0.82	1.01		0.92	0.13
	A048	1	0.9	1	0.97	0.06
	A050	1.03	1.05	1.08	1.05	0.03
	A052					
	A053					
	A055	1.97	2.03	1.94	1.98	0.05
	A057	2.53	2.53	2.61	2.56	0.05
	A059	0.98	1.05	1.13	1.05	0.08
	A065	1.1	1.2	1.3	1.20	0.10
	A068					
	A069	1.49	1.52	1.53	1.51	0.02
	A075					
	A076					
	A080	1.47	1.45	1.44	1.45	0.02
Community Results		Consensus Mean			1.38	
		Consensus Standard Deviation			0.50	
		Maximum			3.56	
		Minimum			0.83	
		N			18	

Table E-5. Data summary table for protein in kelp.

Data points highlighted in blue have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable Z'_{comm} score, $|Z'_{comm}| \geq 2$.

		Protein				
		Kelp A (%)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**
Individual Results	Target					
	A001					
	A003	11.79	11.84	11.76	11.8	0.04
	A009	11.86	11.54	11.67	11.7	0.16
	A015					
	A016	9.59	9.63	9.63	9.62	0.02
	A020	11.6	11.6	11.57	11.6	0.02
	A022					
	A026	8.72			8.72	
	A028					
	A031	12.01	11.91	12.02	12.0	0.06
	A032					
	A035	11.64	11.55	11.59	11.6	0.05
	A037	11.7	11.5	11.5	11.6	0.12
	A038					
	A039					
	A040					
	A042	11.26	11.07	11.11	11.1	0.10
	A045	11.8735	11.2356	11.5365	11.5	0.32
	A046	10.7	11.6	11.2	11.2	0.45
	A047	11.94	12		12.0	0.04
	A048	7.3	7.7	8	7.67	0.35
	A050	11.81	11.92	11.84	11.9	0.06
	A052					
	A053					
	A055	10.11	10.11	9.79	10.0	0.18
	A057	11.66	11.5	5.05	9.40	3.77
	A059	11.5	11.4	11.2	11.4	0.15
	A065	11.5	11.5	11.6	11.5	0.06
	A068					
	A069	11.08	10.84	10.85	10.9	0.14
	A075					
	A076					
	A080	11.38	11.47	11.5	11.5	0.06
Community Results		Consensus Mean			11.4	
		Consensus Standard Deviation			0.58	
		Maximum			12.0	
		Minimum			7.67	
		N			19	

Table E-6. Data summary table for solids in kelp.

Data points highlighted in blue have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable Z'_{comm} score, $|Z'_{comm}| \geq 2$.

		Solids				
		Kelp A (%)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**
Individual Results	Target					
	A001					
	A019	96.29	96.37	96.31	96.3	0.04
	A020	97.33	97.2	97.43	97.3	0.12
	A022					
	A028					
	A031					
	A032					
	A035	96.9	96.93	96.85	96.9	0.04
	A037	96.94	96.93	96.98	97.0	0.03
	A038					
	A039					
	A040					
	A042	96.92	96.82	96.8	96.8	0.06
	A045	97.11	97.06	97.19	97.1	0.07
	A046					
	A047	96.49	96.42		96.5	0.05
	A048	98.48	98.35	98.35	98.4	0.08
	A050					
	A052					
	A053					
	A055	99.18	99.23	99.26	99.2	0.04
	A057	96.33	96.44	96.22	96.3	0.11
	A059	96.6	96.9	96.9	96.8	0.17
	A065	96.8	96.8	97	96.9	0.12
	A068					
	A069	96.58	96.6	96.6	96.6	0.01
	A075					
	A076					
	A080					
Community Results		Consensus Mean			96.9	
		Consensus Standard Deviation			0.56	
		Maximum			99.2	
		Minimum			96.3	
		N			13	

Table E-7. Data summary table for starch in kelp.

		Starch				
		Kelp A (%)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**
Individual Results	Target					
	A001					
	A003					
	A020					
	A022					
	A028					
	A031	1.18	1.18	1.09	1.15	0.05
	A032					
	A035					
	A037					
	A038					
	A040					
	A042	< 0.600	< 0.600	< 0.600		
	A045					
	A046					
	A047	< 0.100	< 0.100			
	A048					
	A050					
	A052					
	A053					
	A055	0.04	0.04	0.05	0.04	0.01
	A057					
	A059	0.7	0.9	0.9	0.83	0.12
	A065	< 0.500	< 0.500	< 0.500		
	A068					
	A069					
	A075					
	A076					
	A080					
Community Results		Consensus Mean			0.68	
		Consensus Standard Deviation			0.90	
		Maximum			1.15	
		Minimum			0.04	
		N			3	

Table E-8. Data summary table for total dietary fiber in kelp.

Data points highlighted in blue have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable Z'_{comm} score, $|Z'_{comm}| \geq 2$.

		Total Dietary Fiber				
		Kelp A (%)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**
Individual Results	Target					
	A001					
	A005					
	A009	5.64	6.5	5.65	5.93	0.49
	A020	36.87	37.35	37.23	37.2	0.25
	A022					
	A028					
	A031					
	A032					
	A035					
	A037	33.07	35.56	35.84	34.8	1.52
	A038					
	A039					
	A040					
	A042	35.22	35.43	35.74	35.5	0.26
	A045					
	A046	12.2	12.1	12.2	12.2	0.06
	A047	32.7	31.5		32.1	0.85
	A048					
	A050					
	A052					
	A053					
	A055					
	A057	34.82	34.9	34.22	34.6	0.37
	A065	5.4	5.6	5.4	5.47	0.12
	A068					
	A069					
	A075					
	A076					
	A080	14.97	14.57	13.85	14.5	0.57
Community Results		Consensus Mean			27.5	
		Consensus Standard Deviation			6.68	
		Maximum			37.2	
		Minimum			5.47	
		N			9	

Table E-9. Data summary table for calories in kelp.

Data points highlighted in blue have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable Z'_{comm} score, $|Z'_{comm}| \geq 2$.

		Calories				
		Kelp A (kcal/100g)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**
Individual Results	Target					
	A001					
	A009					
	A015					
	A020					
	A022					
	A026					
	A028					
	A031					
	A032					
	A035	257	257	256	257	0.58
	A037					
	A038					
	A039					
	A040					
	A042	200	200	201	200	0.58
	A045					
	A046	224.2	221.3	222.7	223	1.45
	A047	241	242		242	0.71
	A048	250	250	250	250	0
	A050					
	A052					
	A053					
	A055					
	A057	247	246	247	247	0.58
	A059	257.7	257.4	254.8	257	1.59
	A065					
	A068					
	A069					
	A075					
	A076					
	A080	181.47	184.41	186.94	184	2.74
Community Results		Consensus Mean			234	
		Consensus Standard Deviation			22.6	
		Maximum			257	
		Minimum			184	
		N			8	

Appendix F. Vitamins II Supplemental Tables and Figures

Table F-1. Individualized data summary table (example) for vitamins in meal replacement formulations.

(Laboratory Name)

Exercise 1 - Vitamins II - Meal Replacements

Lab Code: (code)

			1. Your Results				2. Community Results			3. Target	
Analyte	Sample	Units	x_i	s_i	Z'_{comm}	Z_{NIST}	N	x*	s*	x_{NIST}	*U*
Vitamin B1 (Thiamine)	Meal Replacement Drink (Liquid)	mg/kg					6	1.50	0.51	1.23	0.12
Vitamin B1 (Thiamine)	Meal Replacement Drink (Powder)	mg/kg					7	20.0	13.5	11.50	1.15
Vitamin B1 (Thiamine)	SRM 3252 Protein Drink Mix	mg/kg					4	6.30	2.50	11.70	1.52
Vitamin B2 (Riboflavin)	Meal Replacement Drink (Liquid)	mg/kg					7	2.19	0.48	1.32	0.13
Vitamin B2 (Riboflavin)	Meal Replacement Drink (Powder)	mg/kg					6	5.80	4.86	6.92	0.69
Vitamin B2 (Riboflavin)	SRM 3252 Protein Drink Mix	mg/kg					6	20.1	13.7	27.30	2.66
Vitamin B3 (Niacin)	Meal Replacement Drink (Liquid)	mg/kg					5	28.0	5.57	16.30	1.63
Vitamin B3 (Niacin)	Meal Replacement Drink (Powder)	mg/kg					7	203	27.5	203.8	20.38
Vitamin B3 (Niacin)	SRM 3252 Protein Drink Mix	mg/kg					7	237	36.5	6.96	0.25
Vitamin B5 (Pantothenic Acid)	Meal Replacement Drink (Liquid)	mg/kg					7	11.0	13.5	5.23	0.52
Vitamin B5 (Pantothenic Acid)	Meal Replacement Drink (Powder)	mg/kg					9	81.9	48.2	38.50	3.85
Vitamin B5 (Pantothenic Acid)	SRM 3252 Protein Drink Mix	mg/kg					8	172	75.5	142	11.40
Vitamin B6 (Pyridoxine)	Meal Replacement Drink (Liquid)	mg/kg					5	2.93	0.41	1.75	0.18
Vitamin B6 (Pyridoxine)	Meal Replacement Drink (Powder)	mg/kg					6	23.5	12.4	19.23	1.92
Vitamin B6 (Pyridoxine)	SRM 3252 Protein Drink Mix	mg/kg					5	45.5	36.0	27.70	1.52
Vitamin B7 (Biotin)	Meal Replacement Drink (Liquid)	mg/kg					6	0.11	0.07	0.03	0.003
Vitamin B7 (Biotin)	Meal Replacement Drink (Powder)	mg/kg					6	0.44	0.11	0.39	0.04
Vitamin B7 (Biotin)	SRM 3252 Protein Drink Mix	mg/kg					7	3.63	0.56	4.21	0.18
Vitamin B9 (Folic Acid)	Meal Replacement Drink (Liquid)	mg/kg					9	0.55	0.13	0.25	0.03
Vitamin B9 (Folic Acid)	Meal Replacement Drink (Powder)	mg/kg					7	2.87	0.58	2.31	0.23
Vitamin B9 (Folic Acid)	SRM 3252 Protein Drink Mix	mg/kg					6	5.59	5.37	7.22	1.80
Vitamin B12 (cobalamin)	Meal Replacement Drink (Liquid)	mg/kg					5	0.004	0.003	0.007	0.0007
Vitamin B12 (cobalamin)	Meal Replacement Drink (Powder)	mg/kg					4	0.047	0.013	0.039	0.004
Vitamin B12 (cobalamin)	SRM 3252 Protein Drink Mix	mg/kg					3	0.060	0.011	0.10	0.03

Individual laboratory results will appear in this section; Laboratory-specific results were provided to each participant separately from this report

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

Table F-2. Data summary table for thiamine in Meal Replacement Drink (Liquid), Meal Replacement Drink (Powder), and SRM 3252 Protein Drink Mix.

Data points highlighted in blue have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable Z'_{comm} score, $|Z'_{\text{comm}}| \geq 2$.

		Vitamin B1 (Thiamine)														
		Meal Replacement Drink (Liquid) (mg/kg)					Meal Replacement Drink (Powder) (mg/kg)					SRM 3252 Protein Drink Mix (mg/kg)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				1.23	0.12				11.5	1.15				11.7	1.52
	A001															
	A002															
	A016															
	A018	13.9	12.3	8.9	11.70	2.55		32.1	32.9	32.5	0.57		91.7	114	103	15.77
	A020	1.7	1.6	1.6	1.63	0.06	11	13.6	13.8	12.8	1.56	5.5	5.1	6	5.53	0.45
	A021	< 100.0	< 100.0	< 100.0			< 100.0	< 100.0	< 100.0			< 100.0	< 100.0	< 100.0		
	A022															
	A025															
	A027															
	A032															
	A034															
	A035	1.86	1.88	1.76	1.83	0.06										
	A036						161.63			161.63						
	A037						25.5	25.1	25	25.2	0.26	< 20.0	< 20.0	< 20.0		
	A039															
	A041	< 0.400					< 0.400	< 0.400	< 0.400			< 0.400	< 0.400	< 0.400		
	A042	1.08	0.97	0.89	0.98	0.10	19.63	20.47	17.81	19.3	1.36	5.97	6.8	5.81	6.19	0.53
	A043	< 1.0	< 1.0	< 1.0			< 1.0	< 1.0	< 1.0							
	A046															
	A047	1.4	1.5		1.45	0.07	13.2	13.5		13.4	0.21					
	A057															
	A062															
	A063															
	A067	1.6	1.64	1.64	1.63	0.02	17.7	16.9	17.7	17.4	0.46	7.93	6.27	7.63	7.28	0.88
	A068															
	A075	< 442.1	< 442.1	< 442.1			< 467.5	< 467.5	< 467.5			< 503.0	< 503.0	< 503.0		
	A077															
Community Results		Consensus Mean			1.51		Consensus Mean			20.1		Consensus Mean			6.33	
		Consensus Standard Deviation			0.51		Consensus Standard Deviation			13.5		Consensus Standard Deviation			2.51	
		Maximum			11.70		Maximum			162		Maximum			103	
		Minimum			0.98		Minimum			12.8		Minimum			5.53	
		N			6		N			6		N			4	

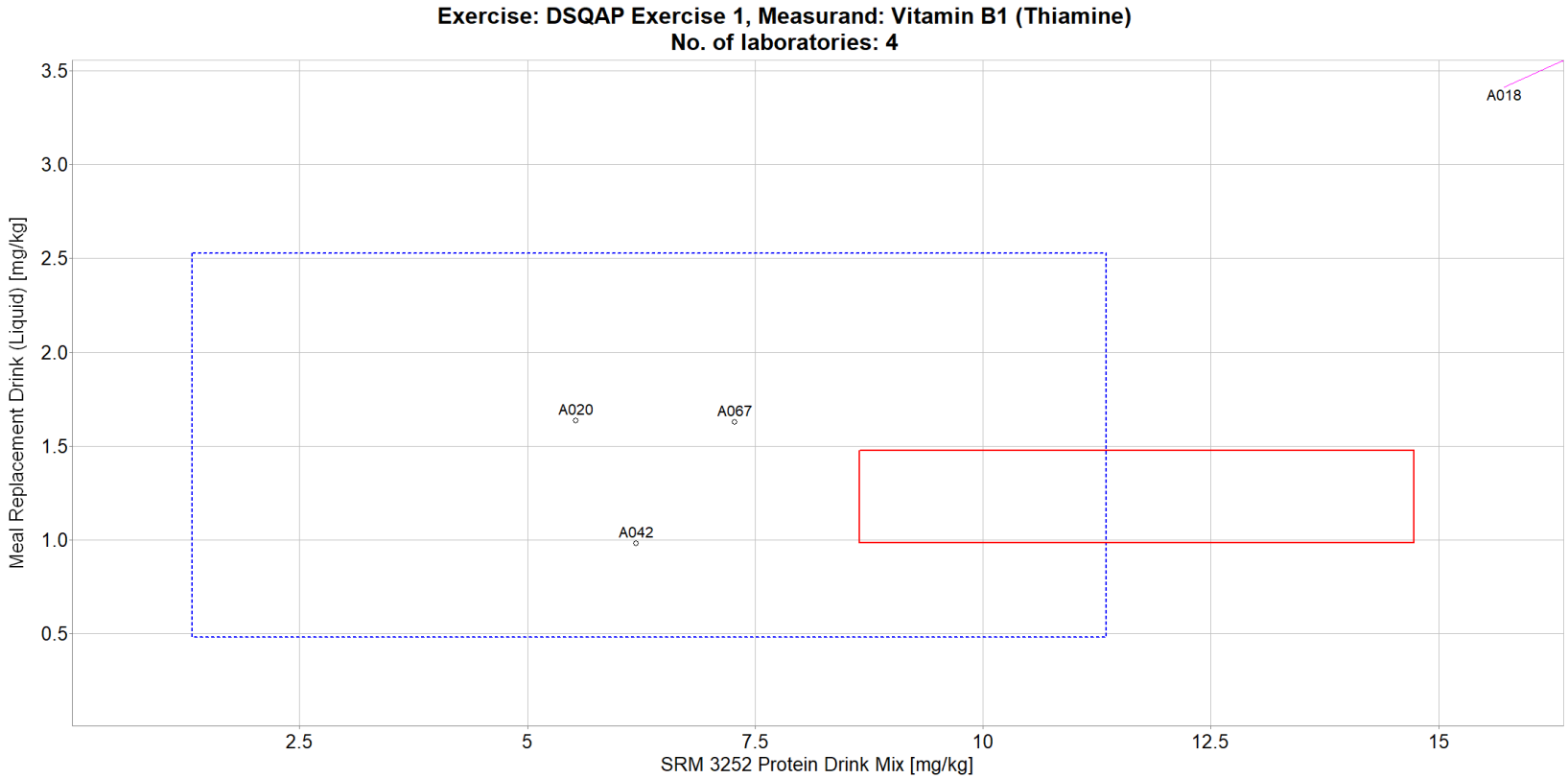

Fig. F-1. Laboratory means for thiamine in SRM 3252 Protein Drink Mix and Meal Replacement Drink (Liquid) (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3252, is compared to the individual laboratory mean for a second sample, Meal Replacement Drink (Liquid). The solid red box represents the NIST range of tolerance for the two samples, SRM 3252 (x-axis) and Meal Replacement Drink (Liquid) (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3252 (x-axis) and Meal Replacement Drink (Liquid) (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

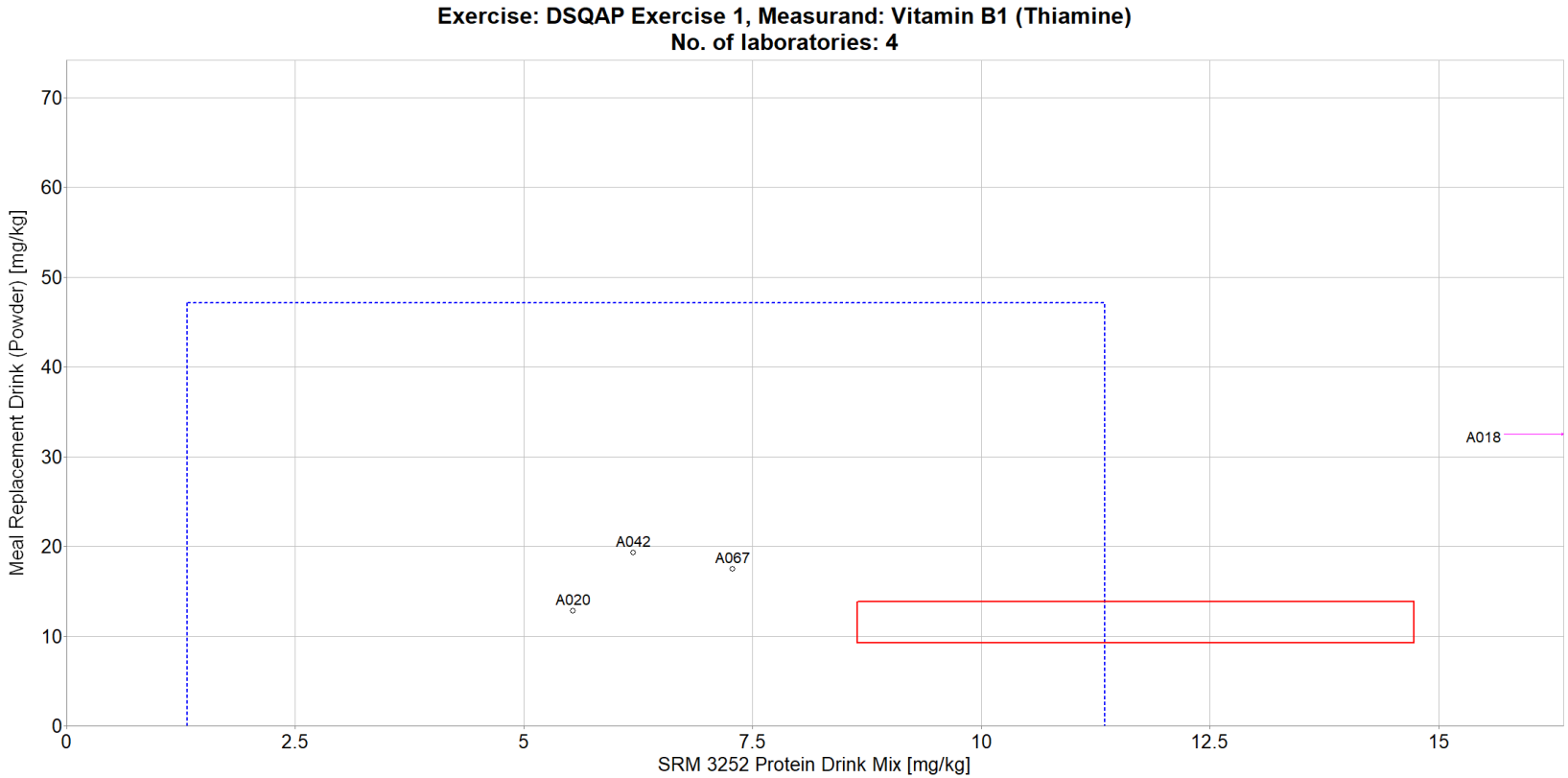

Fig. F-2. Laboratory means for thiamine in SRM 3252 Protein Drink Mix and Meal Replacement Drink (Powder) (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3252, is compared to the individual laboratory mean for a second sample, Meal Replacement Drink (Powder). The solid red box represents the NIST range of tolerance for the two samples, SRM 3252 (x-axis) and Meal Replacement Drink (Powder) (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3252 (x-axis) and Meal Replacement Drink (Powder) (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

Table F-3. Data summary table for riboflavin in Meal Replacement Drink (Liquid), Meal Replacement Drink (Powder), and SRM 3252 Protein Drink Mix.

		Vitamin B2 (Riboflavin)														
		Meal Replacement Drink (Liquid) (mg/kg)					Meal Replacement Drink (Powder) (mg/kg)					SRM 3252 Protein Drink Mix (mg/kg)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
Individual Results	Target				1.32	0.13				6.92	0.69				27.3	2.66
	A001															
	A002															
	A018	1.71	1.63	1.68	1.67	0.04		3.05	3.46	3.26	0.29		10.6	13.6	12.1	2.12
	A020	2.7	2.9	2.5	2.70	0.20	8.5	8.4	8.1	8.33	0.21	22.3	27	24.2	24.5	2.36
	A021	< 100.0	< 100.0	< 100.0			< 100.0	< 100.0	< 100.0			< 100.0	< 100.0	< 100.0		
	A022															
	A025															
	A027															
	A032															
	A034															
	A035	2.23	2.33	2.35	2.30	0.06										
	A036															
	A037						< 10.0	< 10.0	< 10.0			< 10.0	< 10.0	< 10.0		
	A039															
	A041	< 0.500										34	36	27	32.3	4.73
	A042	2.92	2.52	2.37	2.60	0.28	9.7	9.04	9.61	9.45	0.36	26.6	29.5	21.5	25.9	4.05
	A043	< 1.0	< 1.0	< 1.0			< 1.0	< 1.0	< 1.0							
	A046															
	A047	2.4	1.9		2.15	0.35	6.2	6.2		6.2	0					
	A057	1.54	0.982	1.04	1.19	0.31	0.817	0.767	0.865	0.82	0.05	3.54	3.48	1.96	2.99	0.90
	A062															
	A063															
	A067	2.39	2.45	2.44	2.43	0.03	6.99	6.85	6.86	6.90	0.08	21.5	23.2	23.5	22.7	1.08
	A068															
	A075	< 420.6	< 420.6	< 420.6			< 444.7	< 444.7	< 444.7			< 478.5	< 478.5	< 478.5		
	A077															
Community Results		Consensus Mean			2.19		Consensus Mean			5.83		Consensus Mean			20.1	
		Consensus Standard Deviation			0.48		Consensus Standard Deviation			4.86		Consensus Standard Deviation			13.7	
		Maximum			2.70		Maximum			9.45		Maximum			32.3	
		Minimum			1.19		Minimum			0.82		Minimum			2.99	
		N			7		N			6		N			6	

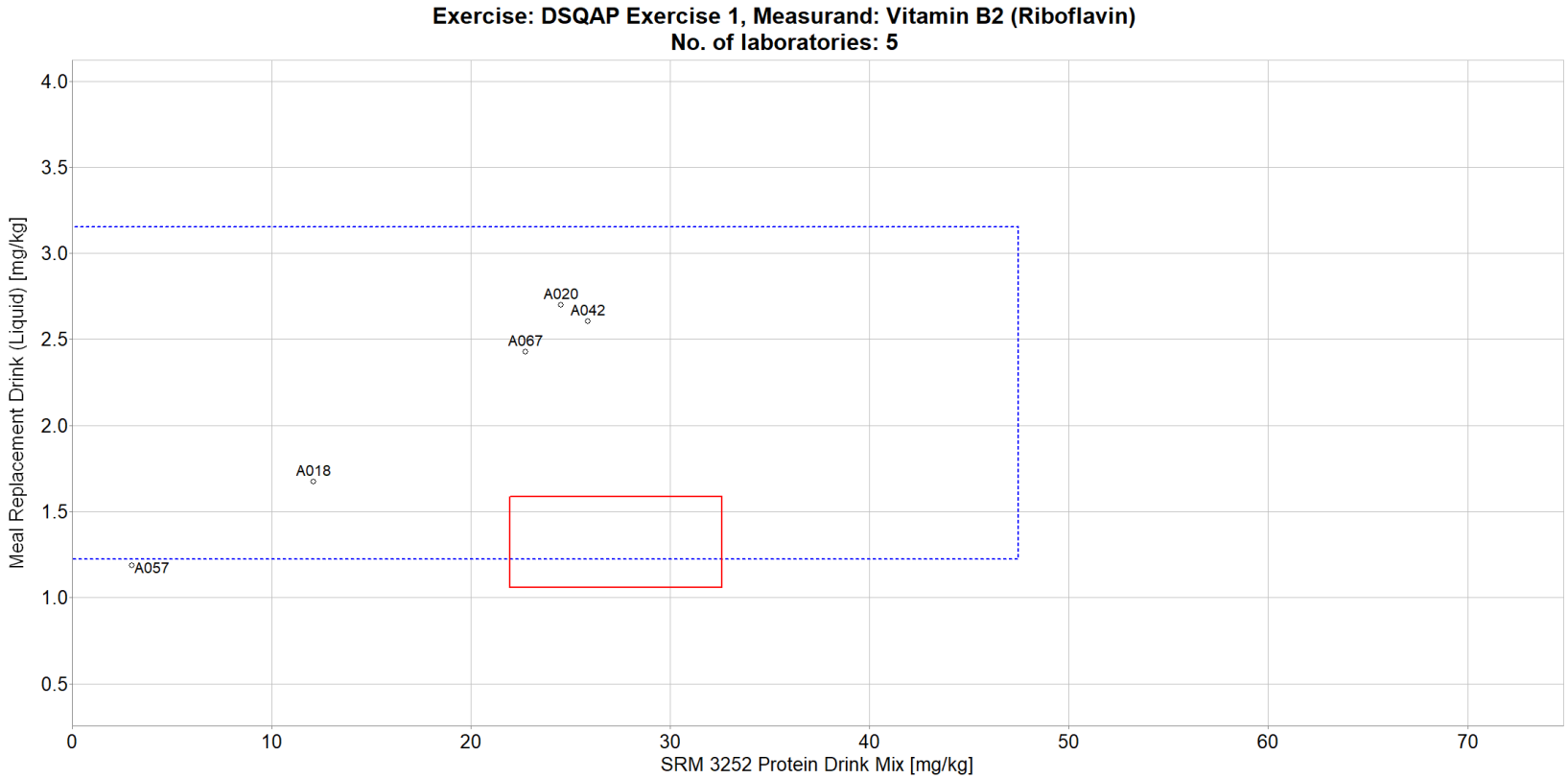

Fig. F-3. Laboratory means for riboflavin in SRM 3252 Protein Drink Mix and Meal Replacement Drink (Liquid) (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3252, is compared to the individual laboratory mean for a second sample, Meal Replacement Drink (Liquid). The solid red box represents the NIST range of tolerance for the two samples, SRM 3252 (x-axis) and Meal Replacement Drink (Liquid) (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3252 (x-axis) and Meal Replacement Drink (Liquid) (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

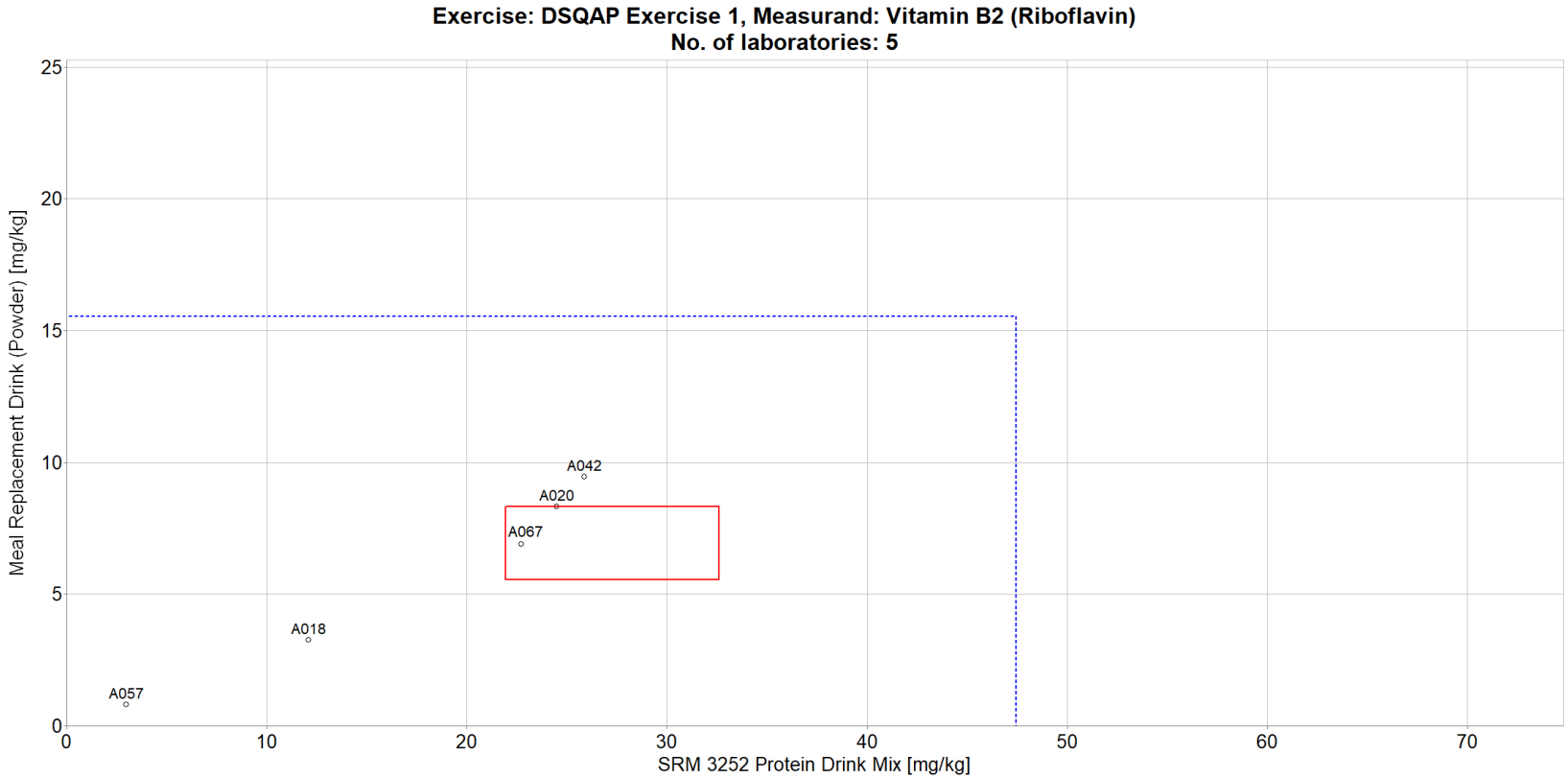

Fig. F-4. Laboratory means for riboflavin in SRM 3252 Protein Drink Mix and Meal Replacement Drink (Powder) (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3252, is compared to the individual laboratory mean for a second sample, Meal Replacement Drink (Powder). The solid red box represents the NIST range of tolerance for the two samples, SRM 3252 (x-axis) and Meal Replacement Drink (Powder) (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3252 (x-axis) and Meal Replacement Drink (Powder) (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

Table F-4. Data summary table for niacin in Meal Replacement Drink (Liquid), Meal Replacement Drink (Powder), and SRM 3252 Protein Drink Mix.

Data points highlighted in blue have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable Z'_{comm} score, $|Z'_{\text{comm}}| \geq 2$.

		Vitamin B3 (Niacin)														
		Meal Replacement Drink (Liquid) (mg/kg)					Meal Replacement Drink (Powder) (mg/kg)					SRM 3252 Protein Drink Mix (mg/kg)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
Individual Results	Target				16.3	1.63				204	20.4				6.97	0.25
	A001															
	A002															
	A006															
	A018	24.6	24.8		24.7	0.14		188	186	187	1.41			269	269	
	A020	29.6	29.6	29.3	29.5	0.17	250.8	237.3	233.5	241	9.09	249.5	262.5	268.3	260	9.63
	A021	< 100.0	< 100.0	< 100.0			< 100.0	< 100.0	< 100.0			< 100.0	< 100.0	< 100.0		
	A022															
	A025						225	221	233	226	6.11	179	186	142	169	23.64
	A027															
	A032															
	A034															
	A035	24.8	25.3	24.7	24.9	0.32	236.9	225.4	212.9	225	12.0	251.9	244.8	251.6	249	4.02
	A036															
	A037						175.6	204.1	176.6	185	16.2	230.1	233.1	222.6	229	5.41
	A039															
	A041	44			44.0		180	138	185	168	25.8	247	259	219	242	20.53
	A042	22.11	24.8	26.11	24.3	2.04	170.1	222.5	177.6	190	28.3	211.6	254.6	212.6	226	24.54
	A043	< 1.0	< 1.0	< 1.0			< 1.0	< 1.0	< 1.0							
	A046															
	A057															
	A062															
	A063															
	A068															
	A075	< 566.7	< 566.7	< 566.7			< 599.1	< 599.1	< 599.1			< 644.7	< 644.7	< 644.7		
	A077															
Community Results		Consensus Mean			28.0		Consensus Mean			203		Consensus Mean			237	
		Consensus Standard Deviation			5.57		Consensus Standard Deviation			27.5		Consensus Standard Deviation			36.5	
		Maximum			44.0		Maximum			241		Maximum			269	
		Minimum			24.3		Minimum			168		Minimum			169	
		N			4		N			7		N			6	

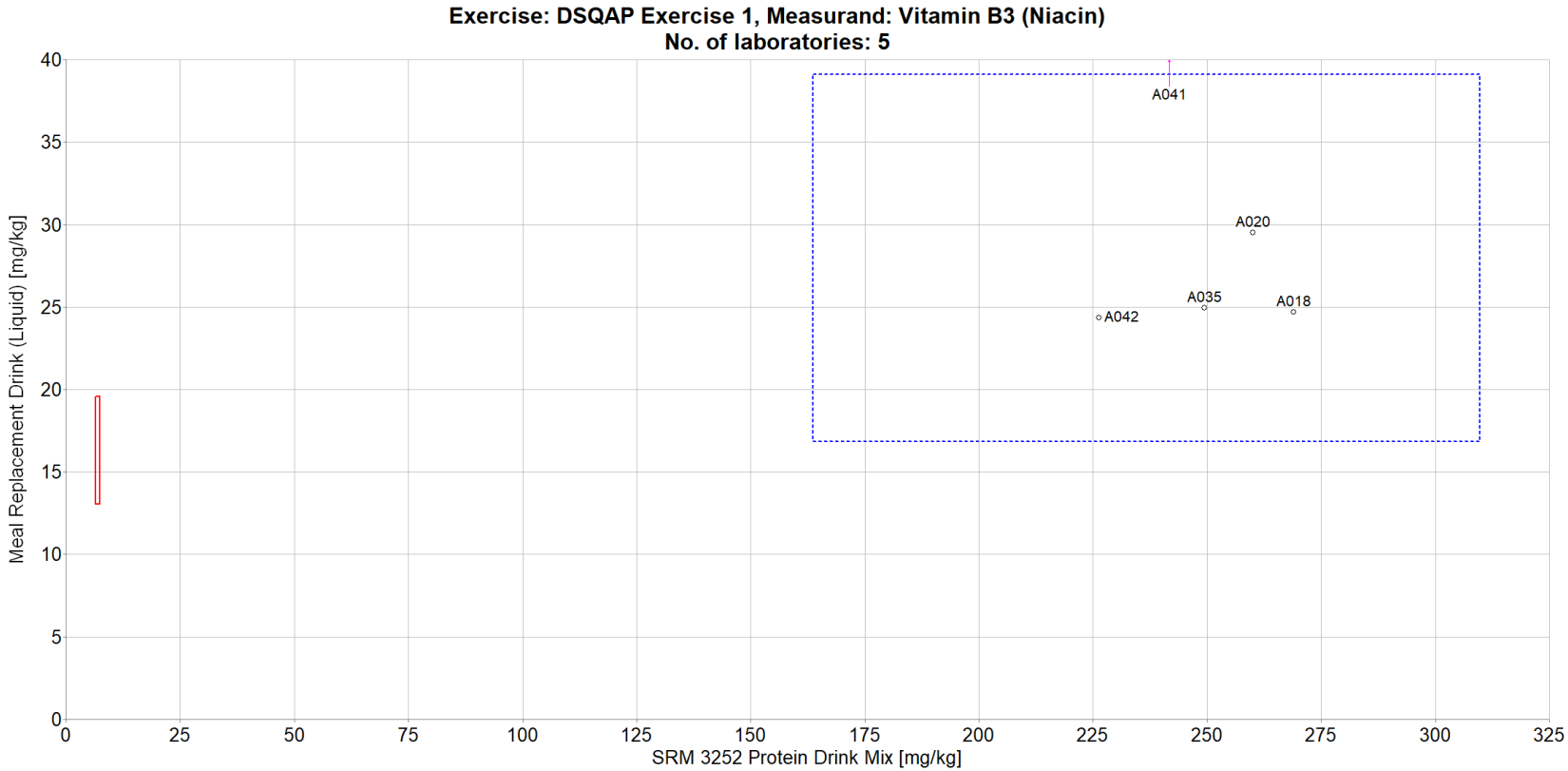

Fig. F-5. Laboratory means for niacin in SRM 3252 Protein Drink Mix and Meal Replacement Drink (Liquid) (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3252, is compared to the individual laboratory mean for a second sample, Meal Replacement Drink (Liquid). The solid red box represents the NIST range of tolerance for the two samples, SRM 3252 (x-axis) and Meal Replacement Drink (Liquid) (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$.The dotted blue box represents the consensus range of tolerance for SRM 3252 (x-axis) and Meal Replacement Drink (Liquid) (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

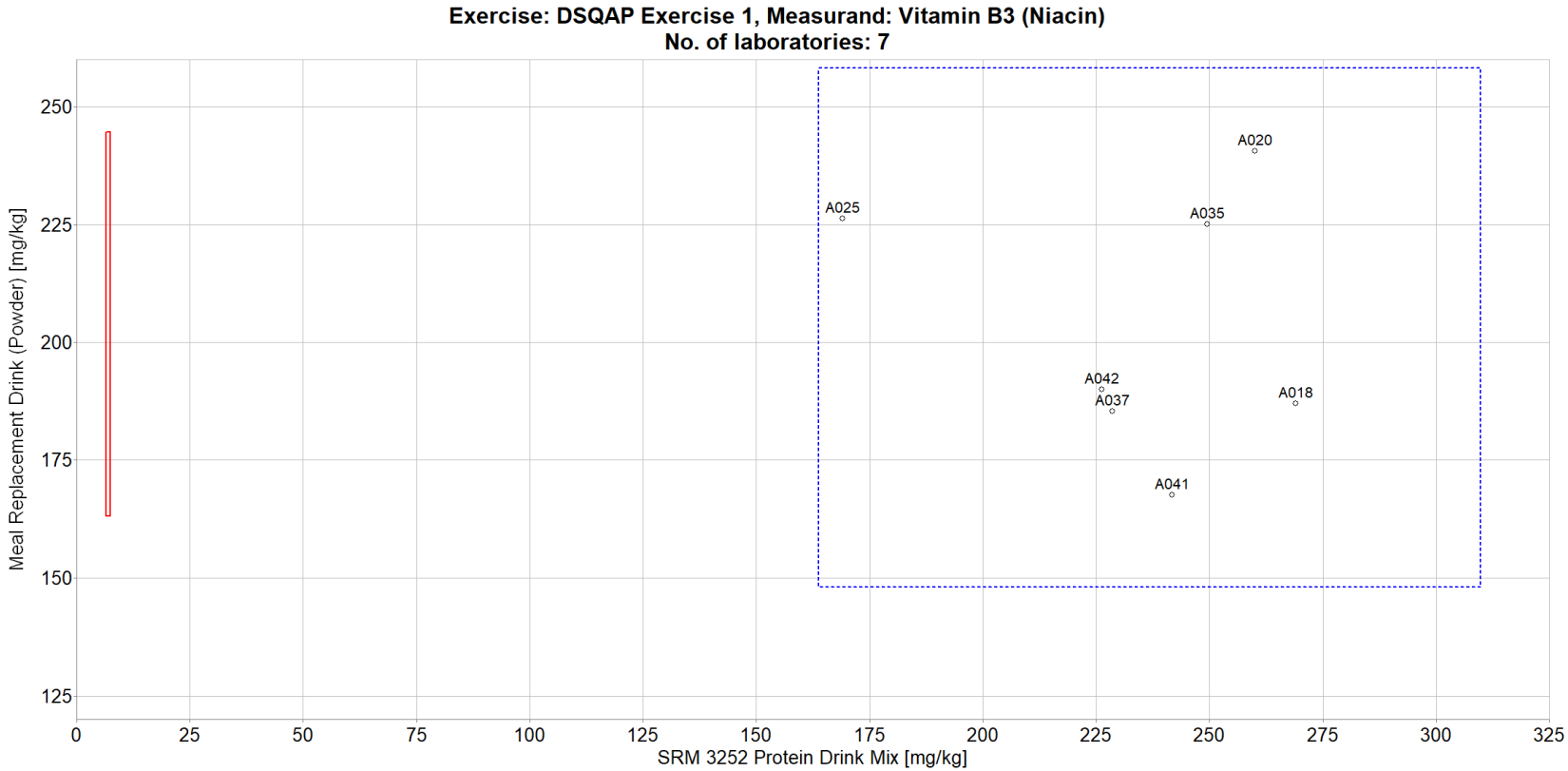

Fig. F-6. Laboratory means for niacin in SRM 3252 Protein Drink Mix and Meal Replacement Drink (Powder) (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3252, is compared to the individual laboratory mean for a second sample, Meal Replacement Drink (Powder). The solid red box represents the NIST range of tolerance for the two samples, SRM 3252 (x-axis) and Meal Replacement Drink (Powder) (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3252 (x-axis) and Meal Replacement Drink (Powder) (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

Table F-5. Data summary table for pantothenic acid in Meal Replacement Drink (Liquid), Meal Replacement Drink (Powder), and SRM 3252 Protein Drink Mix.

Data points highlighted in blue have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable Z'_{comm} score, $|Z'_{\text{comm}}| \geq 2$.

		Vitamin B5 (Pantothenic Acid)														
		Meal Replacement Drink (Liquid) (mg/kg)					Meal Replacement Drink (Powder) (mg/kg)					SRM 3252 Protein Drink Mix (mg/kg)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
Individual Results	Target				5.23	0.52				38.5	3.85				143	11.4
	A001															
	A002															
	A018	19.7	17.5	20.3	19.2	1.47		111	99.3	105	8.27		213	187	200	18.4
	A020	13.6	13.2	13	13.3	0.31	52	54.5	63.5	56.7	6.05	143.9	133.6	172	150	19.9
	A021	< 100.0	< 100.0	< 100.0			< 100.0	< 100.0	< 100.0			162	187	165	171	13.7
	A022															
	A025						224	211	224	220	7.51	249	244	174	222	41.9
	A027															
	A032															
	A034															
	A035	6.8	6.8	7.3	6.97	0.29	56.3	52.3	53.9	54.2	2.01					
	A036	679.37			679		5585.91			5586						
	A037						70.8	88.9	103.2	87.6	16.24	1182	1179	1160	1174	11.9
	A039															
	A041	< 1.1					81	< 1.1	89	85.0	5.66	177	250	208	212	36.6
	A042	7.71	7.99	8.37	8.02	0.33	48.5	52.9	55.2	52.2	3.40	123.6	142.9	139.7	135	10.3
	A043	< 1.0	< 1.0	< 1.0			< 1.0	< 1.0	< 1.0							
	A046															
	A057															
	A062															
	A063															
	A067	7.49	7.23	7.39	7.37	0.13	78.2	52	51	60.4	15.42	122	123	101	115	12.4
	A068															
	A075	< 1112.8	< 1112.8	< 1112.8			< 1176.6	< 1176.6	< 1176.6			< 1266.0	< 1266.0	< 1266.0		
	A077	1164.82	1124.95	1138.85	1143	20.24										
Community Results		Consensus Mean			11.0		Consensus Mean			81.9		Consensus Mean			172	
		Consensus Standard Deviation			13.5		Consensus Standard Deviation			48.2		Consensus Standard Deviation			75.5	
		Maximum			1143		Maximum			5586		Maximum			1174	
		Minimum			6.97		Minimum			52.2		Minimum			115	
		N			6		N			8		N			8	

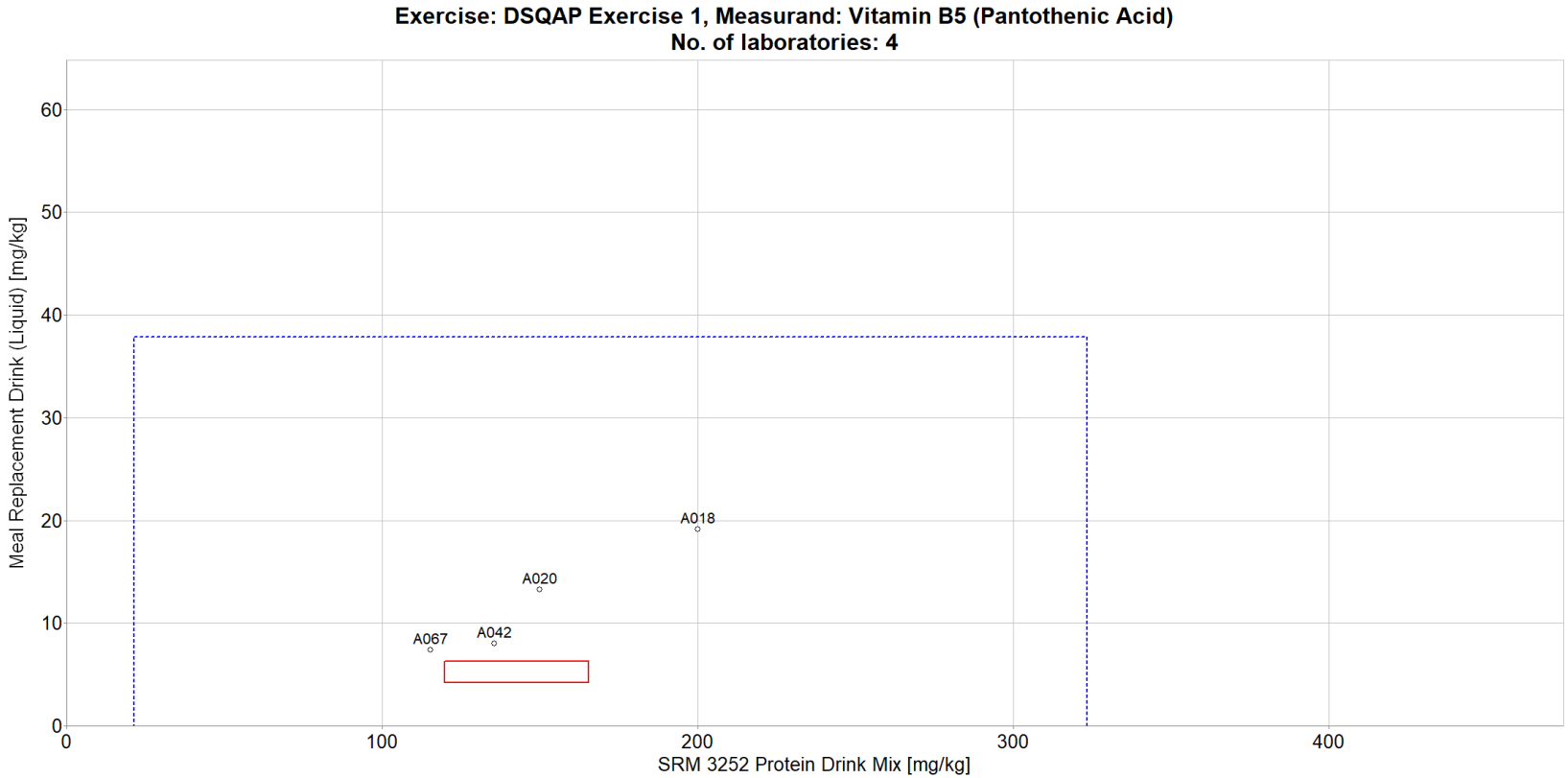

Fig. F-7. Laboratory means for pantothenic acid in SRM 3252 Protein Drink Mix and Meal Replacement Drink (Liquid) (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3252, is compared to the individual laboratory mean for a second sample, Meal Replacement Drink (Liquid). The solid red box represents the NIST range of tolerance for the two samples, SRM 3252 (x-axis) and Meal Replacement Drink (Liquid) (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3252 (x-axis) and Meal Replacement Drink (Liquid) (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

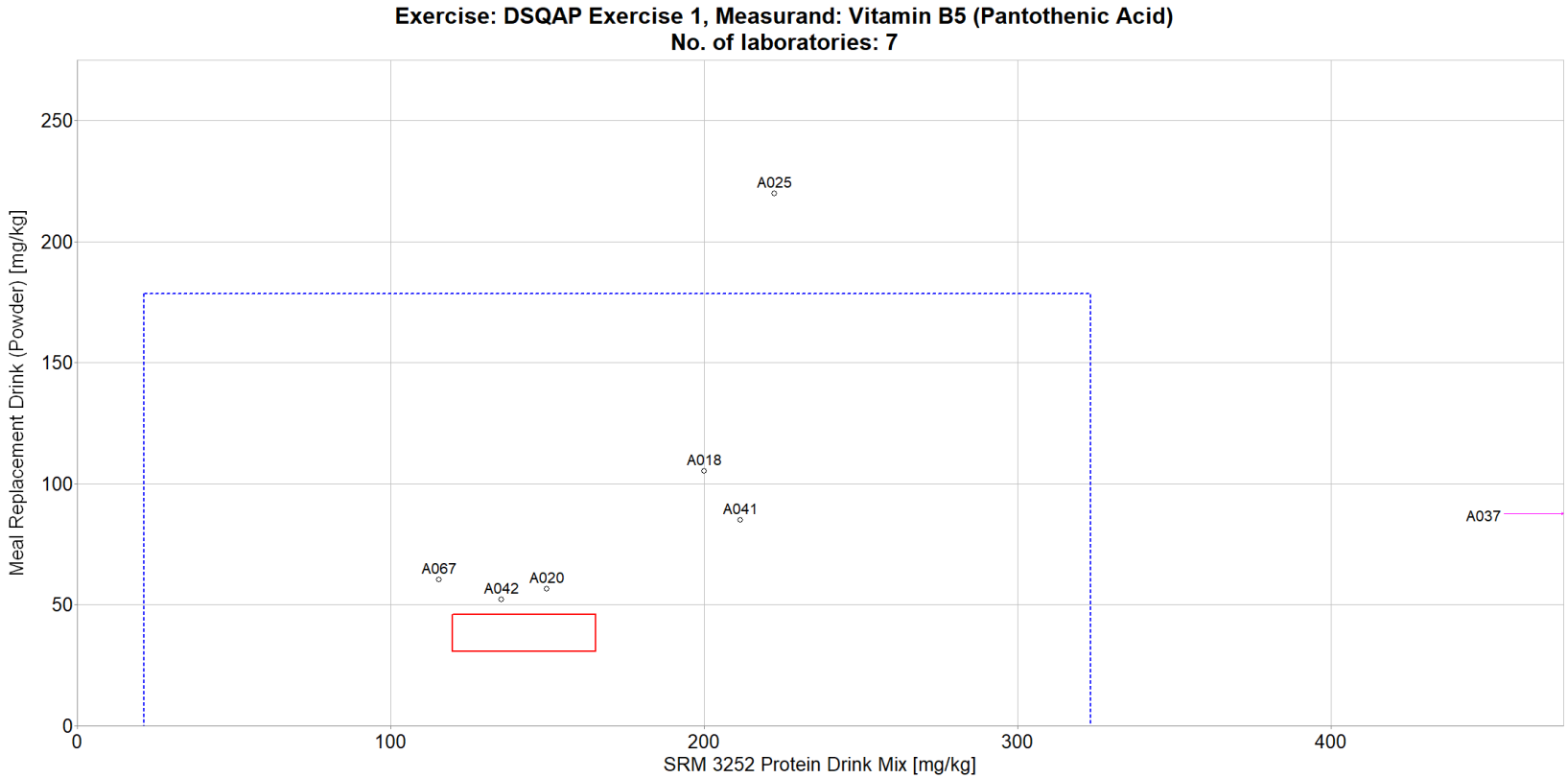

Fig. F-8. Laboratory means for pantothenic acid in SRM 3252 Protein Drink Mix and Meal Replacement Drink (Powder) (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3252, is compared to the individual laboratory mean for a second sample, Meal Replacement Drink (Powder). The solid red box represents the NIST range of tolerance for the two samples, SRM 3252 (x-axis) and Meal Replacement Drink (Powder) (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3252 (x-axis) and Meal Replacement Drink (Powder) (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

Table F-6. Data summary table for pyridoxine in Meal Replacement Drink (Liquid), Meal Replacement Drink (Powder), and SRM 3252 Protein Drink Mix.

		Vitamin B6 (Pyridoxine)														
		Meal Replacement Drink (Liquid) (mg/kg)					Meal Replacement Drink (Powder) (mg/kg)					SRM 3252 Protein Drink Mix (mg/kg)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				1.75	0.18				19.2	1.92				27.7	1.52
	A001															
	A002															
	A016															
	A018	2.56	2.99		2.78	0.30		19.9	20	20.0	0.07			91.9	91.9	
	A020	3.5	3.3	3.1	3.30	0.20	25.5	44.3	41.6	37.1	10.16	40.8	42.8	46.8	43.5	3.06
	A021	< 100.0	< 100.0	< 100.0			< 100.0	< 100.0	< 100.0			< 100.0	< 100.0	< 100.0		
	A022															
	A025															
	A027															
	A032															
	A034															
	A035	2.77	2.81	2.91	2.83	0.07										
	A036						8.67			8.67						
	A037						< 30.0	< 30.0	< 30.0			< 30.0	< 30.0	< 30.0		
	A039															
	A041	< 0.5					33	19	33	28.3	8.08	40	49	51	46.7	5.86
	A042	2.83	2.35	2.66	2.61	0.24	19.1	20.5		19.8	0.99	19.2	21.3		20.3	1.48
	A043	< 1.0	< 1.0	< 1.0			< 1.0	< 1.0	< 1.0							
	A046															
	A057															
	A062															
	A063															
	A067	3.12	3.12	3.16	3.13	0.02	23.8	25.3	32.4	27.2	4.59	23.8	26.4	25.4	25.2	1.31
	A068															
	A075	< 470.5	< 470.5	< 470.5			< 497.5	< 497.5	< 497.5			< 535.3	< 535.3	< 535.3		
	A077															
Community Results		Consensus Mean			2.93		Consensus Mean			23.5		Consensus Mean			45.5	
		Consensus Standard Deviation			0.41		Consensus Standard Deviation			12.4		Consensus Standard Deviation			36.0	
		Maximum			3.30		Maximum			37.1		Maximum			91.9	
		Minimum			2.61		Minimum			8.67		Minimum			20.3	
		N			5		N			5		N			4	

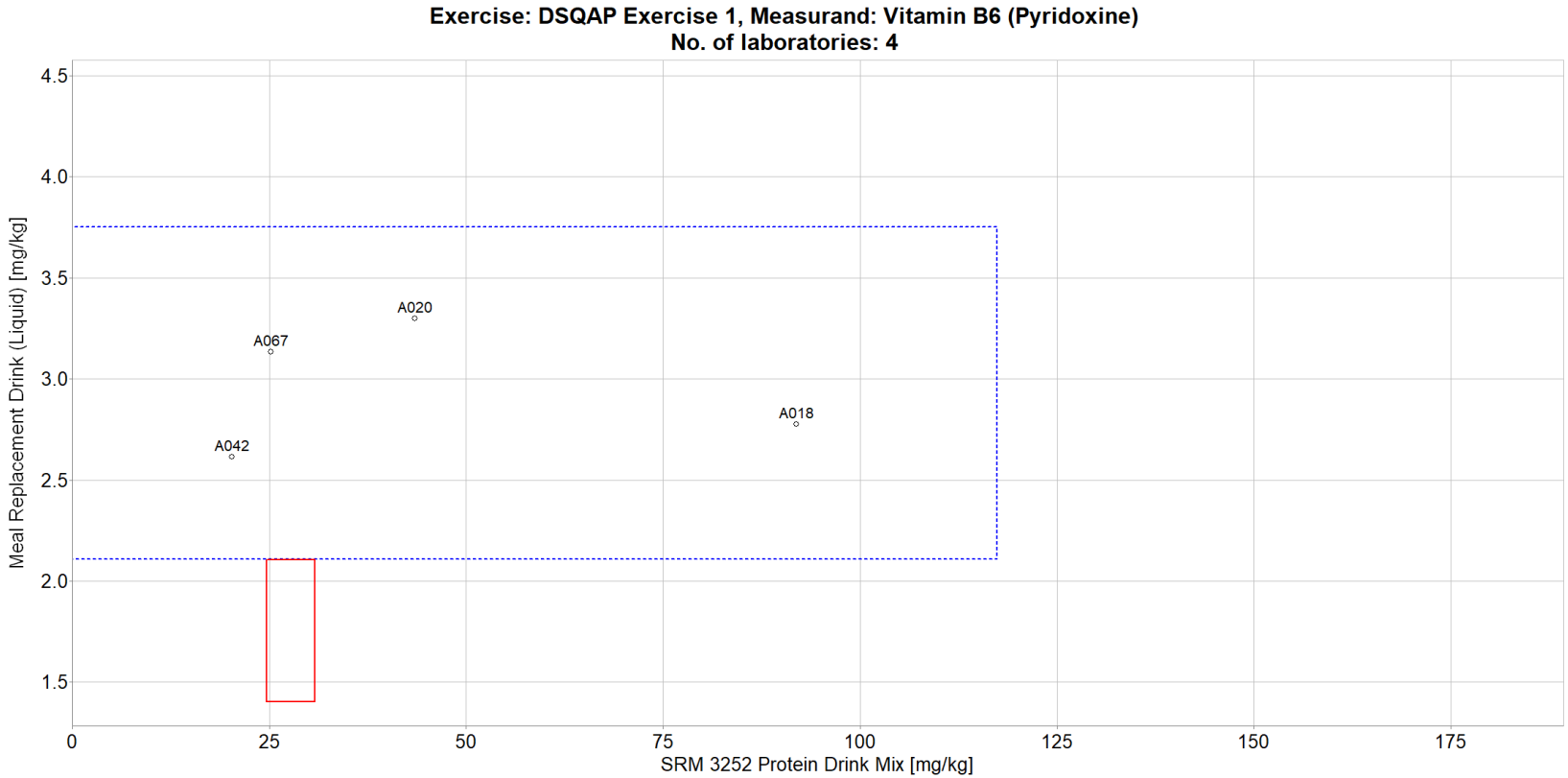

Fig. F-9. Laboratory means for pyridoxine in SRM 3252 Protein Drink Mix and Meal Replacement Drink (Liquid) (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3252, is compared to the individual laboratory mean for a second sample, Meal Replacement Drink (Liquid). The solid red box represents the NIST range of tolerance for the two samples, SRM 3252 (x-axis) and Meal Replacement Drink (Liquid) (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3252 (x-axis) and Meal Replacement Drink (Liquid) (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

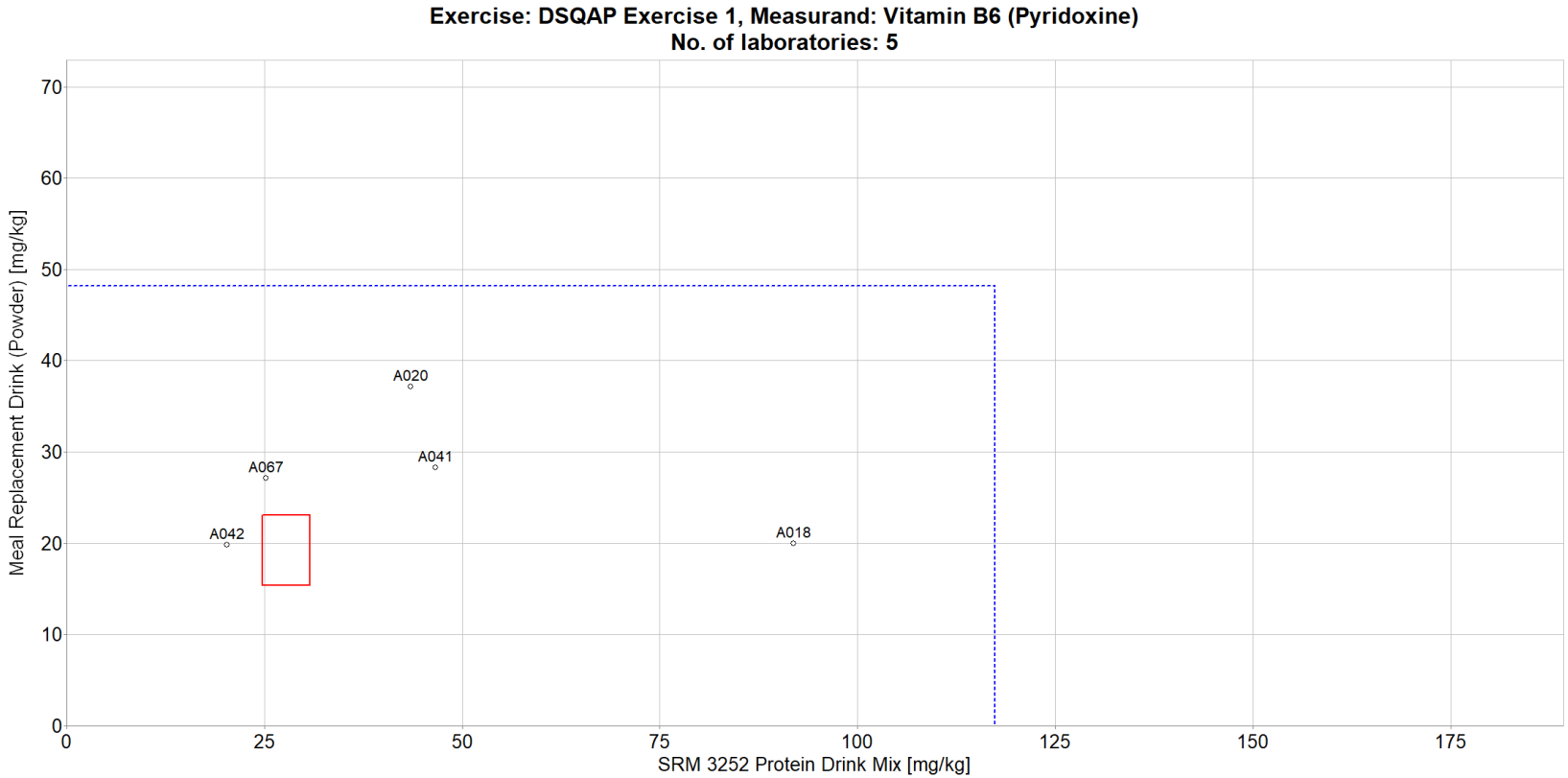

Fig. F-10. Laboratory means for pyridoxine in SRM 3252 Protein Drink Mix and Meal Replacement Drink (Powder) (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3252, is compared to the individual laboratory mean for a second sample, Meal Replacement Drink (Powder). The solid red box represents the NIST range of tolerance for the two samples, SRM 3252 (x-axis) and Meal Replacement Drink (Powder) (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3252 (x-axis) and Meal Replacement Drink (Powder) (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

Table F-7. Data summary table for biotin in Meal Replacement Drink (Liquid), Meal Replacement Drink (Powder), and SRM 3252 Protein Drink Mix.

Data points highlighted in blue have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable Z'_{comm} score, $|Z'_{\text{comm}}| \geq 2$.

		Vitamin B7 (Biotin)														
		Meal Replacement Drink (Liquid) (mg/kg)					Meal Replacement Drink (Powder) (mg/kg)					SRM 3252 Protein Drink Mix (mg/kg)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
Individual Results	Target				0.031	0.003				0.385	0.038				4.21	0.181
	A001															
	A002															
	A018	0.038	0.041		0.040	0.002		0.54	0.47	0.505	0.049		3.35	2.78	3.07	0.403
	A020	0.0903	0.0905	0.09	0.090	0.0003	0.3875	0.396	0.4604	0.415	0.040		3.6547	3.8232	3.74	0.119
	A021	< 1.0	< 1.0	< 1.0			< 1.0	< 1.0	< 1.0			3.68	4.18	3.4	3.75	0.395
	A022															
	A025															
	A027															
	A032															
	A034															
	A035	0.095	0.093	0.099	0.096	0.003	0.344	0.416	0.353	0.371	0.039					
	A036															
	A037															
	A039															
	A041	< 0.59					< 2.95	< 2.95	< 2.95			3.2	3.9	3.8	3.63	0.379
	A042		0.14	0.14	0.137	0.006	0.51	0.47	0.5	0.493	0.021	3.37	3.46	3.27	3.37	0.095
	A043	0.2	0.19	0.15	0.180	0.026	1.3	0.9	1	1.07	0.208	3	4.1	4.4	3.83	0.737
	A046															
	A057															
	A062															
	A063															
	A067	0.101	0.0983	0.0998	0.100	0.001	0.371	0.439	0.371	0.394	0.039	3.57	3.98	4.51	4.02	0.471
	A068															
	A075															
	A077															
Community Results		Consensus Mean			0.107		Consensus Mean			0.436		Consensus Mean			3.63	
		Consensus Standard Deviation			0.072		Consensus Standard Deviation			0.111		Consensus Standard Deviation			0.556	
		Maximum			0.180		Maximum			1.07		Maximum			4.02	
		Minimum			0.040		Minimum			0.371		Minimum			3.07	
		N			6		N			6		N			7	

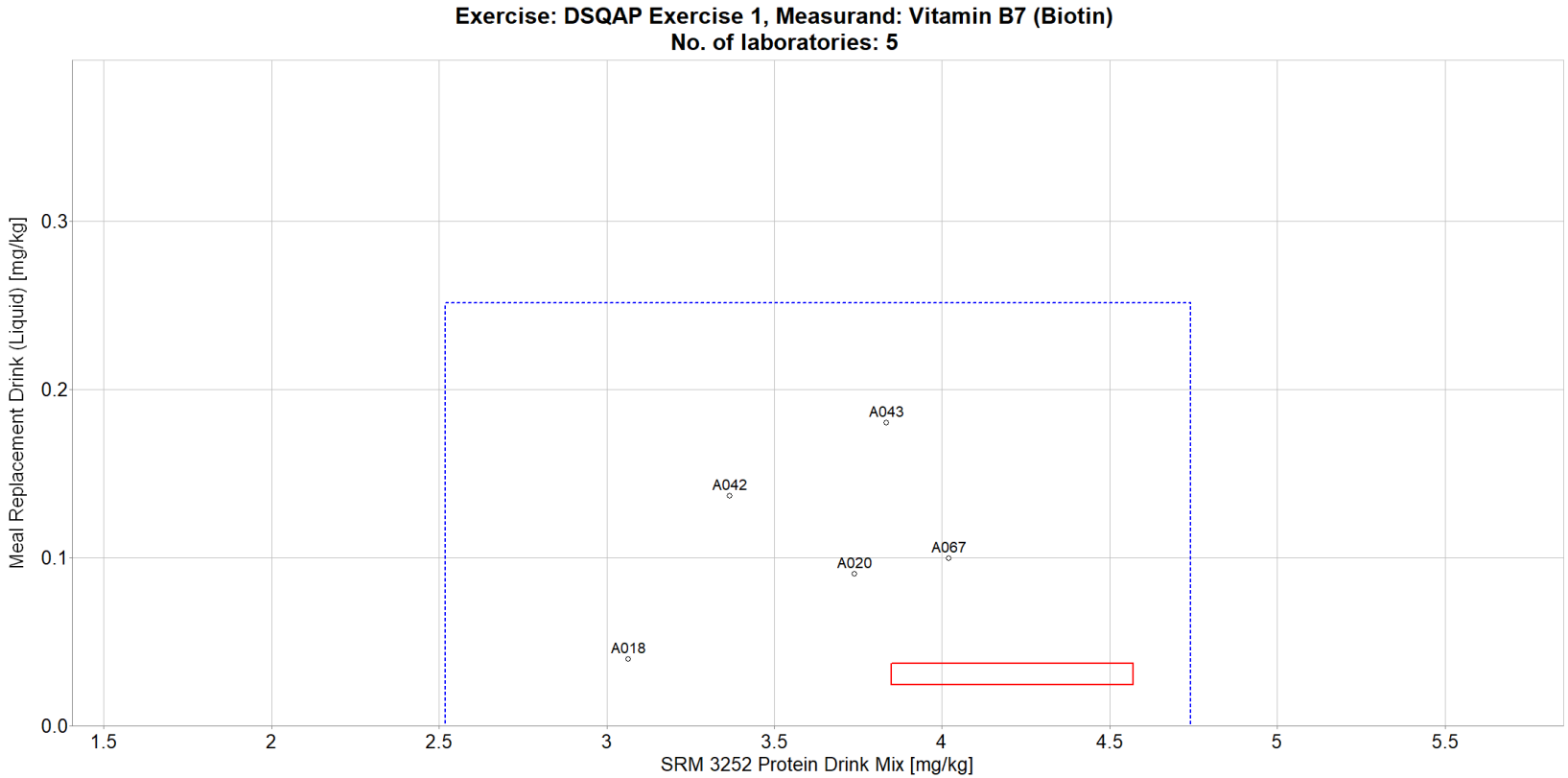

Fig. F-11. Laboratory means for biotin in SRM 3252 Protein Drink Mix and Meal Replacement Drink (Liquid) (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3252, is compared to the individual laboratory mean for a second sample, Meal Replacement Drink (Liquid). The solid red box represents the NIST range of tolerance for the two samples, SRM 3252 (x-axis) and Meal Replacement Drink (Liquid) (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3252 (x-axis) and Meal Replacement Drink (Liquid) (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

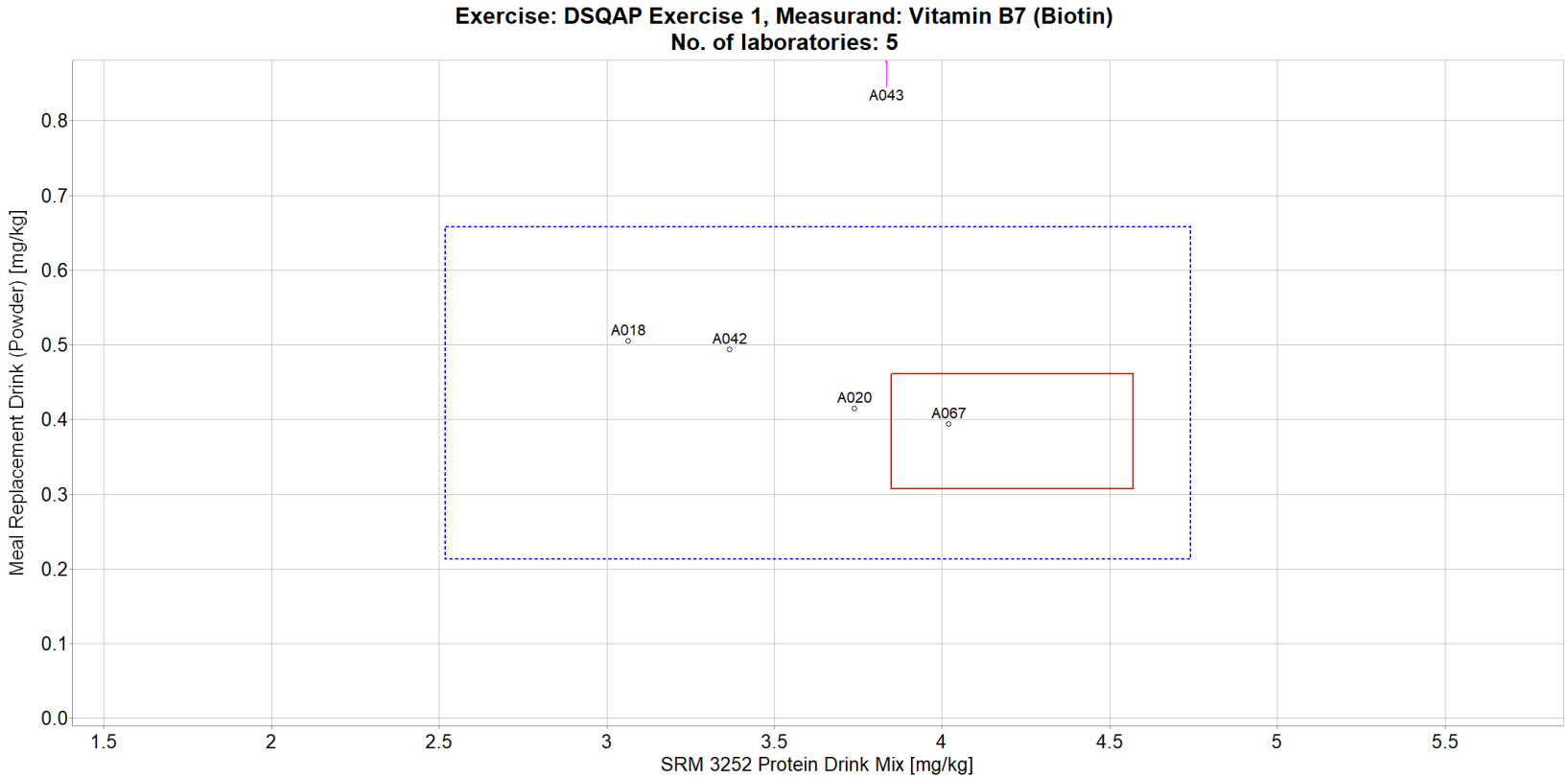

Fig. F-12. Laboratory means for biotin in SRM 3252 Protein Drink Mix and Meal Replacement Drink (Powder) (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3252, is compared to the individual laboratory mean for a second sample, Meal Replacement Drink (Powder). The solid red box represents the NIST range of tolerance for the two samples, SRM 3252 (x-axis) and Meal Replacement Drink (Powder) (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3252 (x-axis) and Meal Replacement Drink (Powder) (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

Table F-8. Data summary table for folic acid in Meal Replacement Drink (Liquid), Meal Replacement Drink (Powder), and SRM 3252 Protein Drink Mix.

Data points highlighted in blue have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable Z'_{comm} score, $|Z'_{\text{comm}}| \geq 2$.

		Vitamin B9 (Folic Acid)														
		Meal Replacement Drink (Liquid) (mg/kg)					Meal Replacement Drink (Powder) (mg/kg)					SRM 3252 Protein Drink Mix (mg/kg)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target				0.246	0.025				2.31	0.231				7.22	1.81
	A001															
	A002															
	A018	0.84	0.98	0.95	0.923	0.074		6.8	7.4	7.10	0.424		26.1	31	28.55	3.46
	A020	0.6175	0.6664	0.6183	0.634	0.028	2.6432	2.6404	3.1026	2.80	0.266	7.3629	7.0963	7.3129	7.26	0.142
	A021	< 10.0	< 10.0	< 10.0			< 10.0	< 10.0	< 10.0			< 10.0	< 10.0	< 10.0		
	A022															
	A025															
	A027															
	A032															
	A034															
	A035	0.477	0.465	0.497	0.480	0.016										
	A036															
	A037	0.648	0.685	0.708	0.680	0.030	3.25	2.88	2.98	3.04	0.191					
	A039															
	A041	< 1.8					< 1.8	< 1.8	< 1.8			< 1.8	< 1.8	< 1.8		
	A042	0.44	0.41	0.47	0.440	0.030	2.74	2.69	2.85	2.76	0.082	5	5.14	5.09	5.08	0.071
	A043	0.43	0.5	0.4	0.443	0.051	3	2.7	2.9	2.87	0.153	5.3	4.9	4.6	4.93	0.351
	A046															
	A057	0.372	0.394	0.426	0.397	0.027	0.618	0.604	0.742	0.655	0.076	1.51	1.68	1.2	1.46	0.243
	A062															
	A063															
	A067	0.662	0.626	0.719	0.669	0.047	3.13	3.47	3.24	3.28	0.173	8.47	7.53	8.07	8.02	0.472
	A068															
	A075															
	A077	0.459	0.459	0.45	0.456	0.005										
Community Results		Consensus Mean			0.550		Consensus Mean			2.87		Consensus Mean			5.59	
		Consensus Standard Deviation			0.131		Consensus Standard Deviation			0.583		Consensus Standard Deviation			5.37	
		Maximum			0.923		Maximum			7.10		Maximum			28.6	
		Minimum			0.397		Minimum			0.655		Minimum			1.46	
		N			9		N			7		N			6	

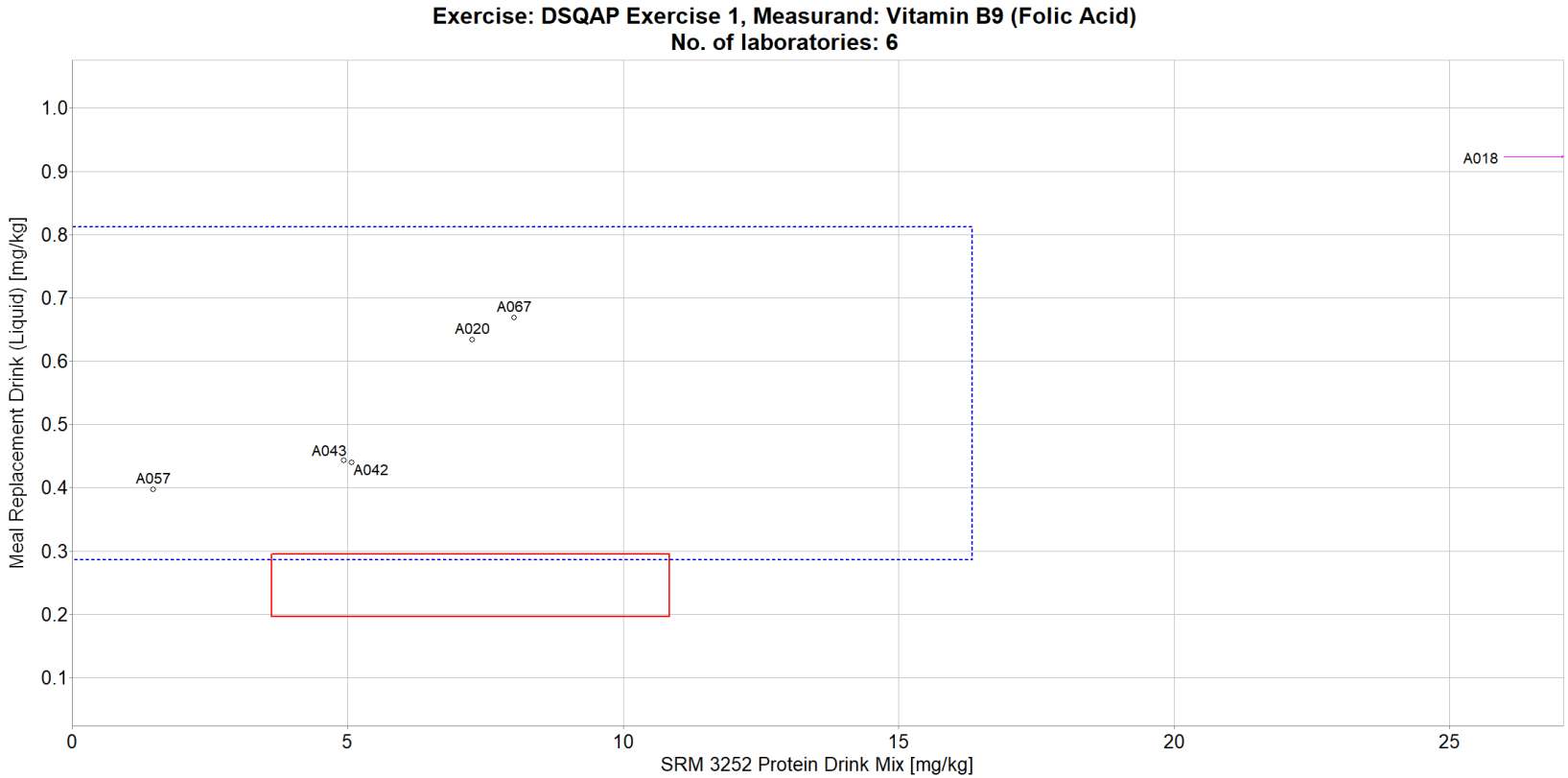

Fig. F-13. Laboratory means for folic acid in SRM 3252 Protein Drink Mix and Meal Replacement Drink (Liquid) (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3252, is compared to the individual laboratory mean for a second sample, Meal Replacement Drink (Liquid). The solid red box represents the NIST range of tolerance for the two samples, SRM 3252 (x-axis) and Meal Replacement Drink (Liquid) (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3252 (x-axis) and Meal Replacement Drink (Liquid) (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

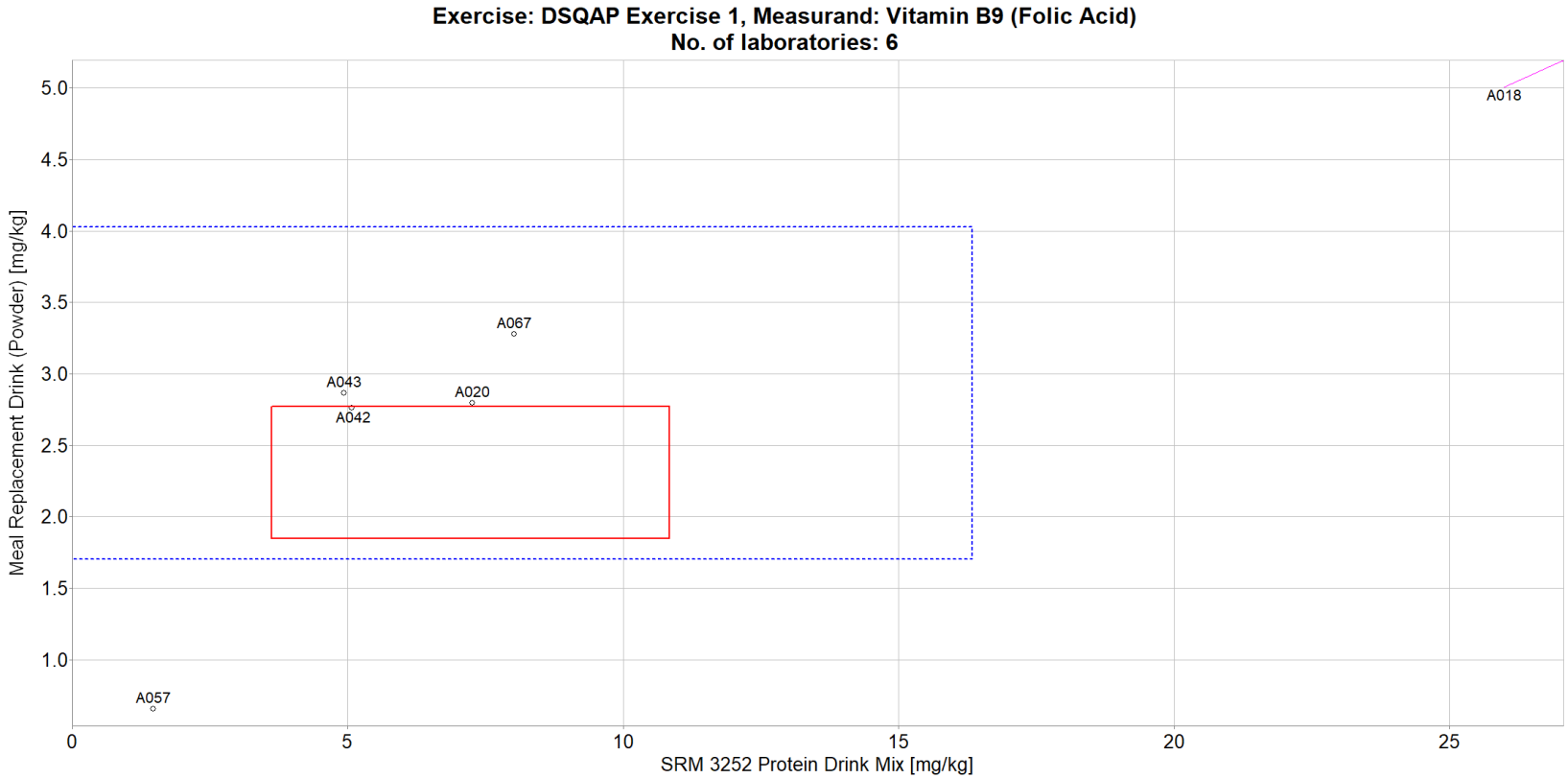

Fig. F-14. Laboratory means for folic acid in SRM 3252 Protein Drink Mix and Meal Replacement Drink (Powder) (sample/sample comparison view).

In this view, the individual laboratory mean for one sample, SRM 3252, is compared to the individual laboratory mean for a second sample, Meal Replacement Drink (Powder). The solid red box represents the NIST range of tolerance for the two samples, SRM 3252 (x-axis) and Meal Replacement Drink (Powder) (y-axis), which encompasses the target values bounded by their uncertainties (U_{NIST}) and represents the range that results in an acceptable Z_{NIST} score, $|Z_{\mathrm{NIST}}| \leq 2$. The dotted blue box represents the consensus range of tolerance for SRM 3252 (x-axis) and Meal Replacement Drink (Powder) (y-axis), calculated as the values above and below the consensus means that result in an acceptable Z'_{comm} score, $|Z'_{\mathrm{comm}}| \leq 2$.

Table F-9. Data summary table for cobalamin in Meal Replacement Drink (Liquid), Meal Replacement Drink (Powder), and SRM 3252 Protein Drink Mix.

Data points highlighted in blue have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable Z'_{comm} score, $|Z'_{\text{comm}}| \geq 2$.

		Vitamin B12 (cobalamin)														
		Meal Replacement Drink (Liquid) (mg/kg)					Meal Replacement Drink (Powder) (mg/kg)					SRM 3252 Protein Drink Mix (mg/kg)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
Individual Results	Target				0.007	0.001				0.038	0.004				0.103	0.025
	A001															
	A002															
	A004						0.0373	0.0392	0.0379	0.038	0.001					
	A018															
	A020	0.0045	0.0044	0.044	0.018	0.023	0.0453	0.0484	0.0451	0.046	0.002	0.0652	0.0643	0.0576	0.062	0.004
	A021	< 5.0	< 5.0	< 5.0			< 5.0	< 5.0	< 5.0			< 5.0	< 5.0	< 5.0		
	A022															
	A025															
	A027															
	A032															
	A034															
	A035	0.0036	0.0035	0.0035	0.004	0.0001										
	A036															
	A037	0.005	0.005	0.005	0.005	0										
	A039															
	A041	< 0.068					< 0.340	< 0.340	< 0.340			< 0.340	< 0.340	< 0.340		
	A042	0.0014	0.0022	0.0019	0.002	0.0004	0.0543	0.047	0.049	0.050	0.004	0.0493	0.0527	0.0537	0.052	0.002
	A043	< 0.5	< 0.5	< 0.5			< 0.5	< 0.5	< 0.5			< 0.5	< 0.5	< 0.5		
	A046															
	A057															
	A062															
	A063															
	A067	0.0058	0.00585	0.00575	0.006	0.0001	0.0576	0.0512	0.0568	0.055	0.003	0.0656	0.069	0.0651	0.067	0.002
	A068															
	A075															
	A077															
Community Results		Consensus Mean			0.004		Consensus Mean			0.047		Consensus Mean			0.060	
		Consensus Standard Deviation			0.003		Consensus Standard Deviation			0.013		Consensus Standard Deviation			0.011	
		Maximum			0.018		Maximum			0.055		Maximum			0.067	
		Minimum			0.002		Minimum			0.038		Minimum			0.052	
		N			5		N			4		N			3	

Appendix G. Botanicals II Supplemental Tables and Figures

Table G-1. Individualized data summary table (example) for botanicals in ashwagandha root powder and root powder extract.

(Laboratory Name)

Exercise 1 - Botanicals II - Ashwagandha

	Lab Code:	**(code)**	**1. Your Results**				**2. Community Results**			**3. Target**	
Analyte	Ashwagandha Sample	Units	x_i	s_i	Z'_{comm}	Z_{NIST}	N	x*	s*	x_{NIST}	*U*
12-deoxywithastromonolide	Root Powder	% w/w	*Individual laboratory results will appear in this section; Laboratory-specific results were provided to each participant separately from this report*				7	0.033	0.023		
withaferin A	Root Powder	% w/w					10	0.066	0.027		
withanolide A	Root Powder	% w/w					11	0.036	0.021		
withanolide B	Root Powder	% w/w					10	0.012	0.007		
withanoside IV	Root Powder	% w/w					8	0.077	0.029		
withanoside V	Root Powder	% w/w					8	0.050	0.020		
12-deoxywithastromonolide	Root Powder Extract	% w/w					7	0.141	0.043		
withaferin A	Root Powder Extract	% w/w					10	0.340	0.120		
withanolide A	Root Powder Extract	% w/w					11	0.179	0.065		
withanolide B	Root Powder Extract	% w/w					10	0.049	0.023		
withanoside IV	Root Powder Extract	% w/w					8	0.676	0.049		
withanoside V	Root Powder Extract	% w/w					8	0.389	0.064		

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

Table G-2. Data summary table for 12-deoxywithastromonolide in ashwagandha root powder and ashwagandha root powder extract.

Data points highlighted in blue have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable Z'_{comm} score, $|Z'_{\text{comm}}| \geq 2$.

		12-deoxywithastromonolide									
		Ashwagandha Root Powder (% w/w)					Ashwagandha Root Powder Extract (% w/w)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
Individual Results	Target										
	A001										
	A002										
	A019	0.03	0.03	0.02	0.027	0.01	0.13	0.13	0.13	0.13	0
	A021	0.05	0.05	0.05	0.05	0	0.12	0.12	0.12	0.12	0
	A023	0.0851	0.0844	0.0841	0.085	0.001	0.3424	0.3452	0.3454	0.344	0.002
	A030										
	A037	0.011	0.011	0.011	0.011	0	0.122	0.126	0.123	0.124	0.002
	A043										
	A046	0.02	0.02	0.01	0.017	0.01	0.15	0.15	0.15	0.15	0
	A049										
	A056	0.033	0.032	0.032	0.032	0.001	0.143	0.146	0.149	0.146	0.003
	A060										
	A076										
	A077										
	A078	0.0233	0.0267	0.0244	0.025	0.002	0.173	0.176	0.178	0.176	0.003
Community Results		Consensus Mean			0.033		Consensus Mean			0.141	
		Consensus Standard Deviation			0.023		Consensus Standard Deviation			0.043	
		Maximum			0.085		Maximum			0.344	
		Minimum			0.011		Minimum			0.120	
		N			7		N			7	

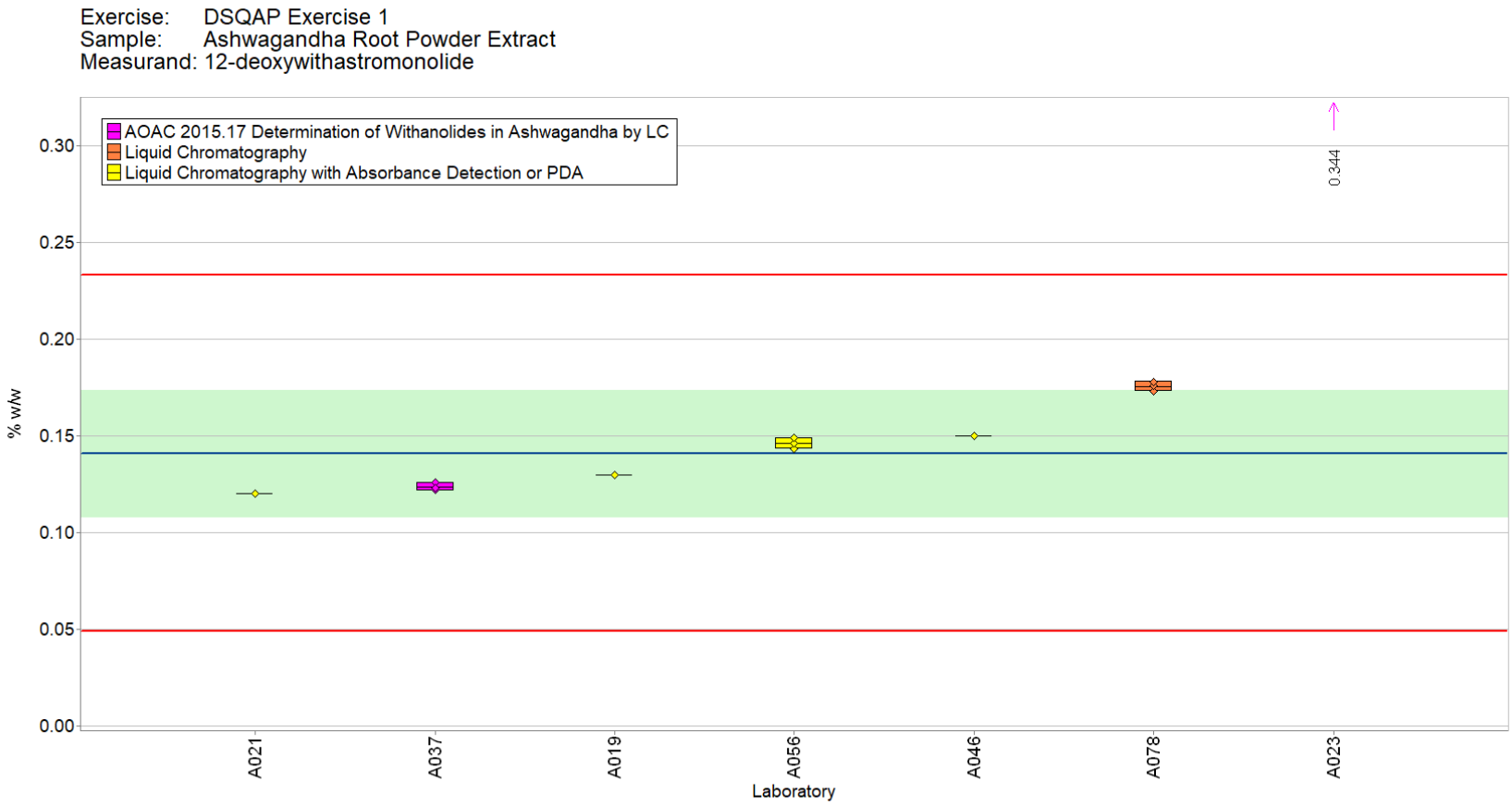

Fig. G-1. 12-deoxywithastromonolide in ashwagandha root powder extract (data summary view – analytical method).

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 red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

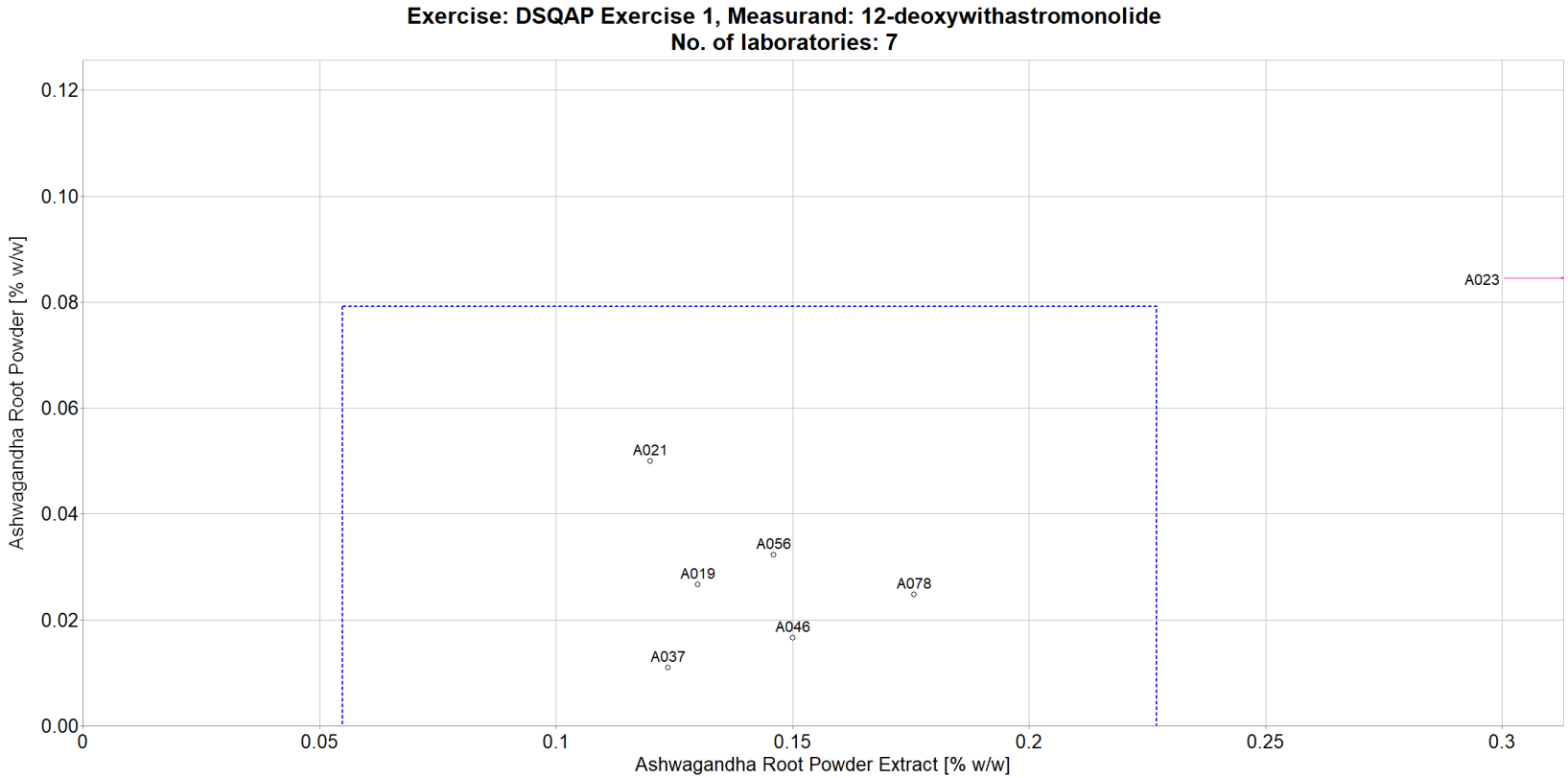

Fig. G-2. Laboratory means for 12-deoxywithastromonolide in ashwagandha root powder and ashwagandha root powder extract (sample/sample comparison view).

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

Table G-3. Data summary table for withaferin A in ashwagandha root powder and ashwagandha root powder extract.

Data points highlighted in blue have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable Z'_{comm} score, $|Z'_{\text{comm}}| \geq 2$.

		withaferin A									
		Ashwagandha Root Powder (% w/w)					Ashwagandha Root Powder Extract (% w/w)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
Individual Results	Target										
	A001										
	A002										
	A019	0.05	0.05	0.05	0.050	0	0.29	0.28	0.27	0.280	0.010
	A021	0.1	0.1	0.1	0.1	0	0.29	0.29	0.3	0.293	0.006
	A023	0.2429	0.2446	0.2443	0.244	0.001	0.9883	1.0082	1.0331	1.01	0.022
	A030										
	A037	0.029	0.029	0.029	0.029	0	0.241	0.253	0.214	0.236	0.020
	A043	0.0654	0.0675	0.0588	0.064	0.005	0.5191	0.5123	0.5562	0.529	0.024
	A046	0.06	0.06	0.06	0.060	0	0.35	0.37	0.38	0.367	0.015
	A049										
	A056	0.094	0.088	0.086	0.089	0.004	0.357	0.367	0.376	0.367	0.010
	A058	0.07	0.072	0.068	0.070	0.002	0.29	0.28	0.28	0.283	0.006
	A060										
	A076										
	A077	0.071	0.071	0.071	0.071	0	0.297	0.306	0.301	0.301	0.005
	A078	0.0583	0.0637	0.058	0.060	0.003	0.421	0.424	0.415	0.420	0.005
Community Results		Consensus Mean			0.066		Consensus Mean			0.342	
		Consensus Standard Deviation			0.027		Consensus Standard Deviation			0.125	
		Maximum			0.244		Maximum			1.010	
		Minimum			0.029		Minimum			0.236	
		N			10		N			10	

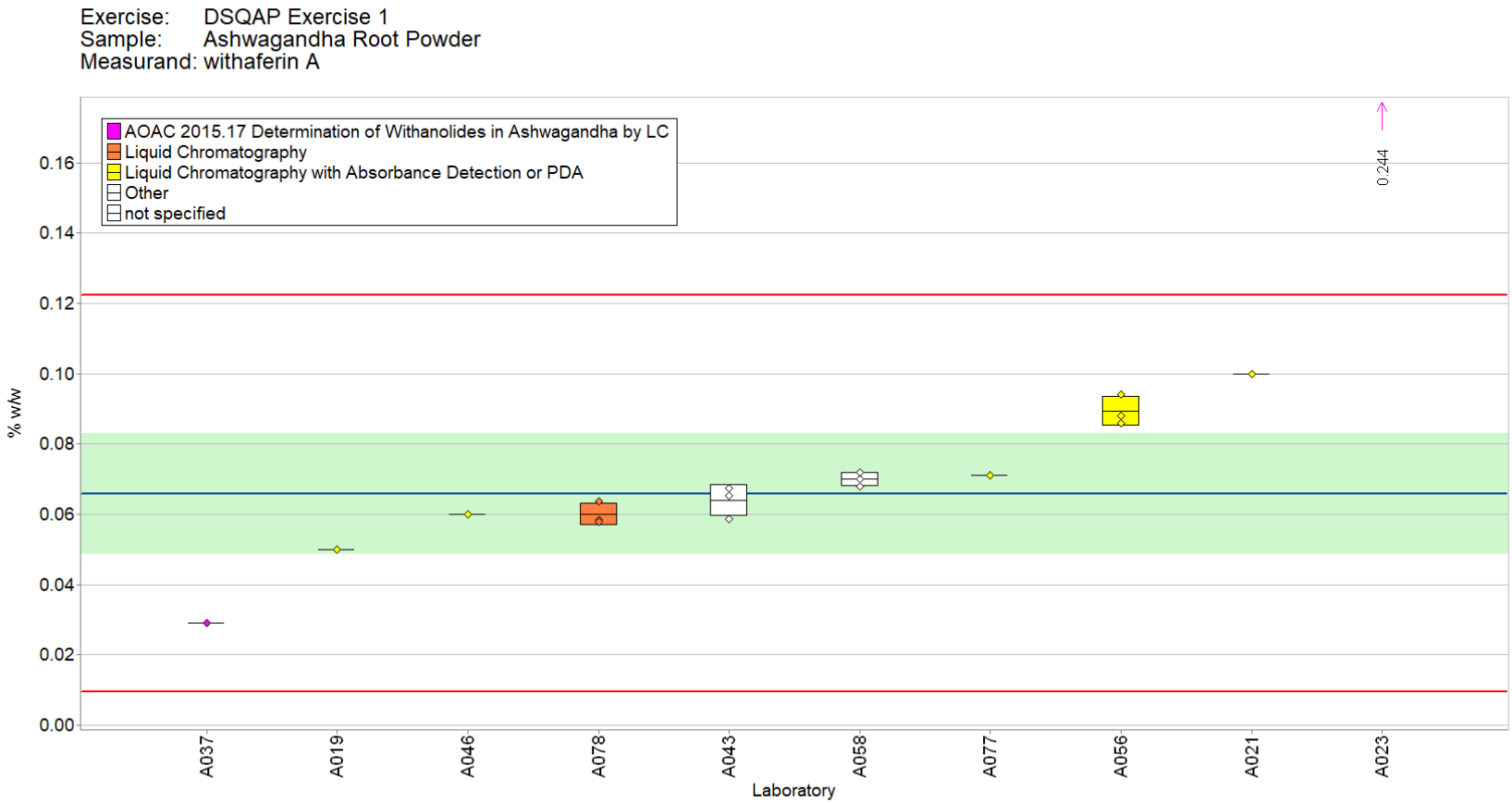

Fig. G-3. Withaferin A in ashwagandha root powder (data summary view – analytical method).

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 red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

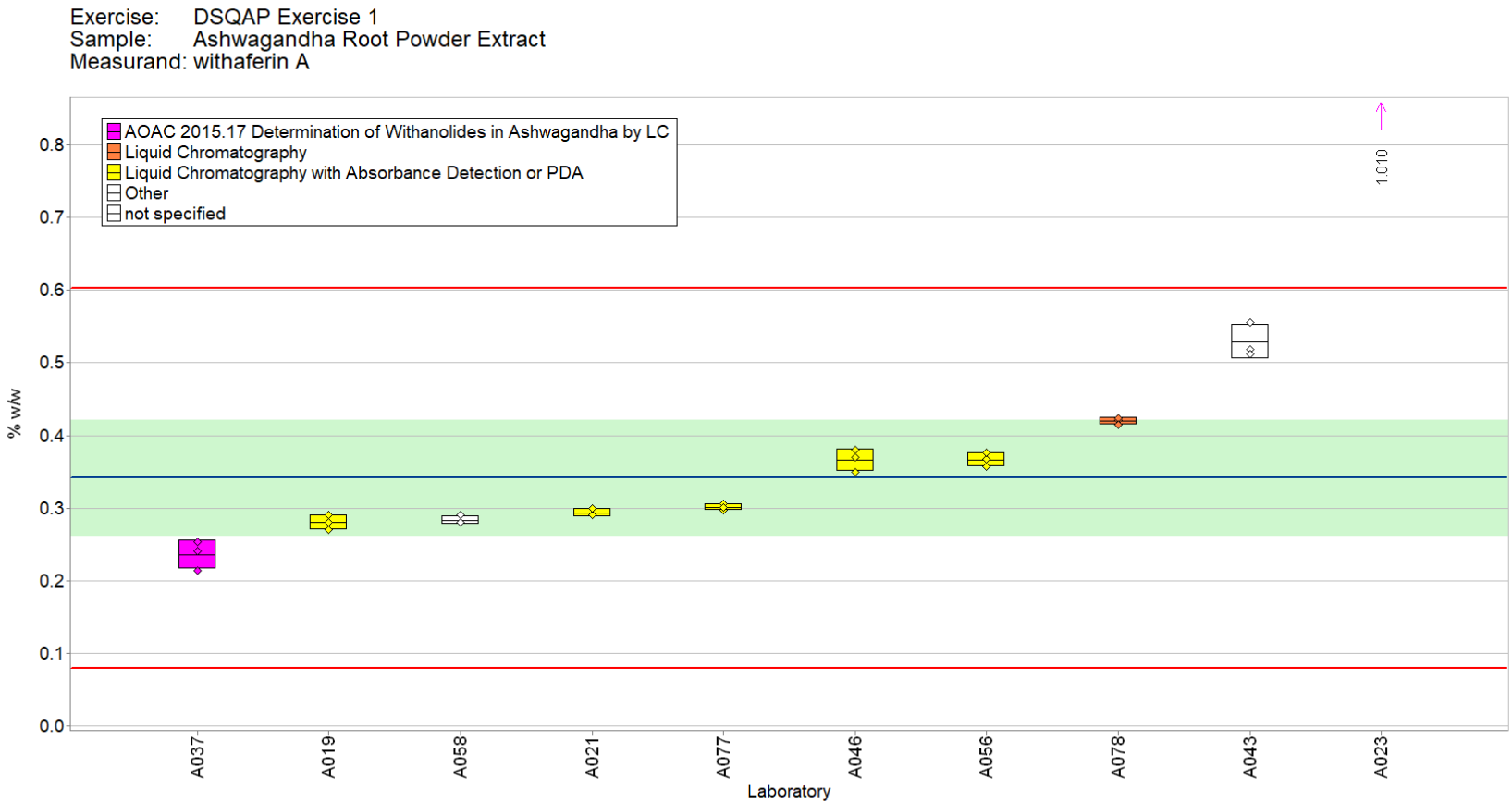

Fig. G-4. Withaferin A in ashwagandha root powder extract (data summary view – analytical method).

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 red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

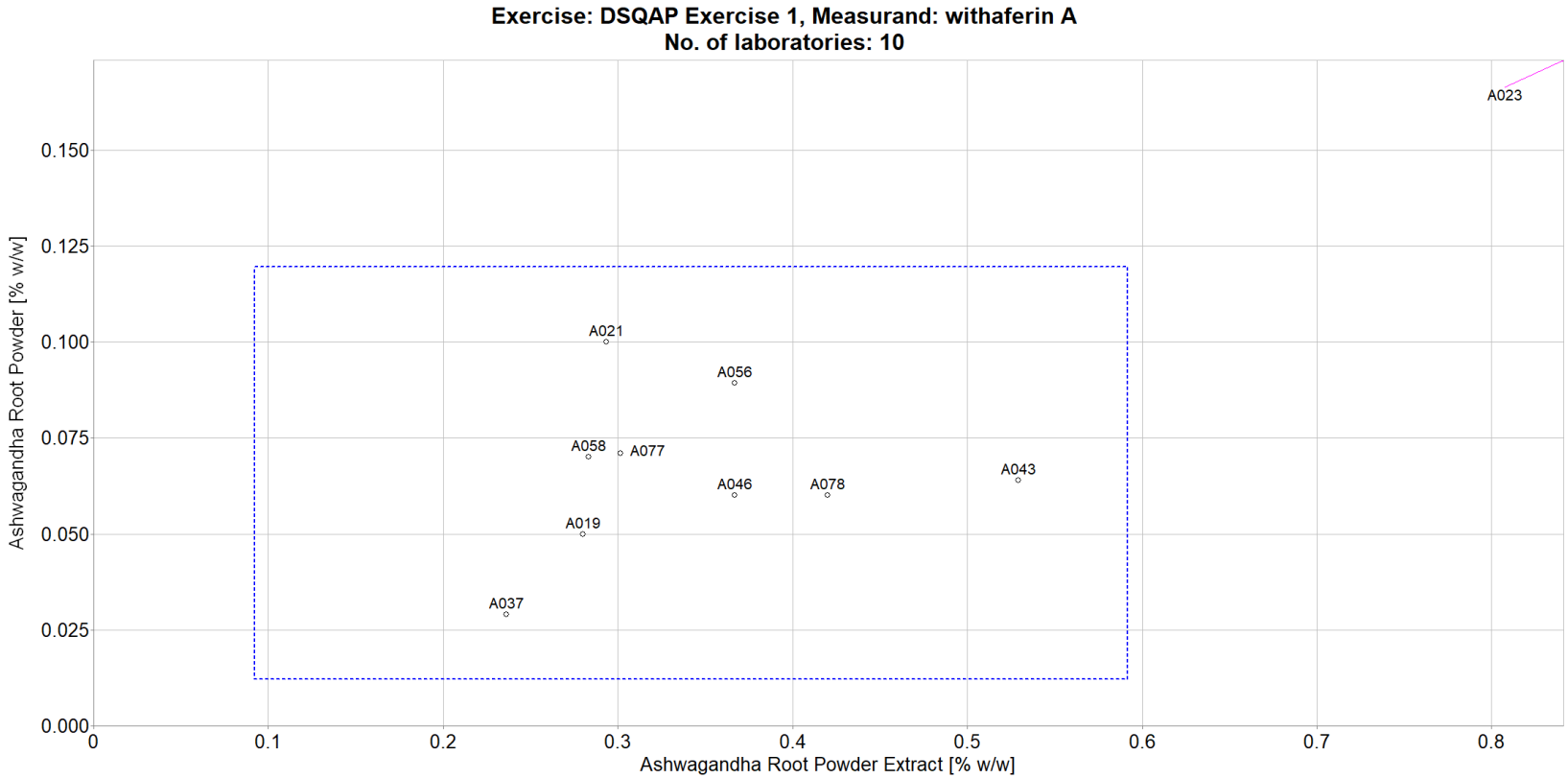

Fig. G-5. Laboratory means for withaferin A in ashwagandha root powder and ashwagandha root powder extract (sample/sample comparison view).

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

Table G-4. Data summary table for withanolide A in ashwagandha root powder and ashwagandha root powder extract.

Data points highlighted in blue have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable Z'_{comm} score, $|Z'_{\text{comm}}| \geq 2$.

		withanolide A									
		Ashwagandha Root Powder (% w/w)					Ashwagandha Root Powder Extract (% w/w)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
Individual Results	Target										
	A001										
	A002										
	A019	0.04	0.04	0.03	0.037	0.006	0.19	0.19	0.18	0.187	0.006
	A021	0.05	0.05	0.05	0.05	0	0.13	0.13	0.14	0.133	0.006
	A023	0.132	0.1303	0.1304	0.131	0.001	0.5274	0.5376	0.5333	0.533	0.005
	A024	0.1	0.1	0.1	0.1	0	0.23	0.23	0.22	0.227	0.006
	A030										
	A037	0.016	0.016	0.016	0.016	0	0.119	0.122	0.119	0.120	0.002
	A043	0.0364	0.0373	0.0324	0.035	0.003	0.2731	0.2759	0.285	0.278	0.006
	A046	0.03	0.02	0.02	0.023	0.006	0.16	0.17	0.16	0.163	0.006
	A049										
	A056	0.037	0.037	0.037	0.037	0	0.146	0.151	0.15	0.149	0.003
	A058	0.039	0.037	0.035	0.037	0.002	0.15	0.145	0.15	0.148	0.003
	A060										
	A076										
	A077	0.027	0.026	0.026	0.026	0.001	0.159	0.166	0.167	0.164	0.004
	A078	0.0259	0.0286	0.027	0.027	0.001	0.218	0.221	0.224	0.221	0.003
Community Results		Consensus Mean			0.036		Consensus Mean			0.179	
		Consensus Standard Deviation			0.021		Consensus Standard Deviation			0.065	
		Maximum			0.131		Maximum			0.533	
		Minimum			0.016		Minimum			0.120	
		N			11		N			11	

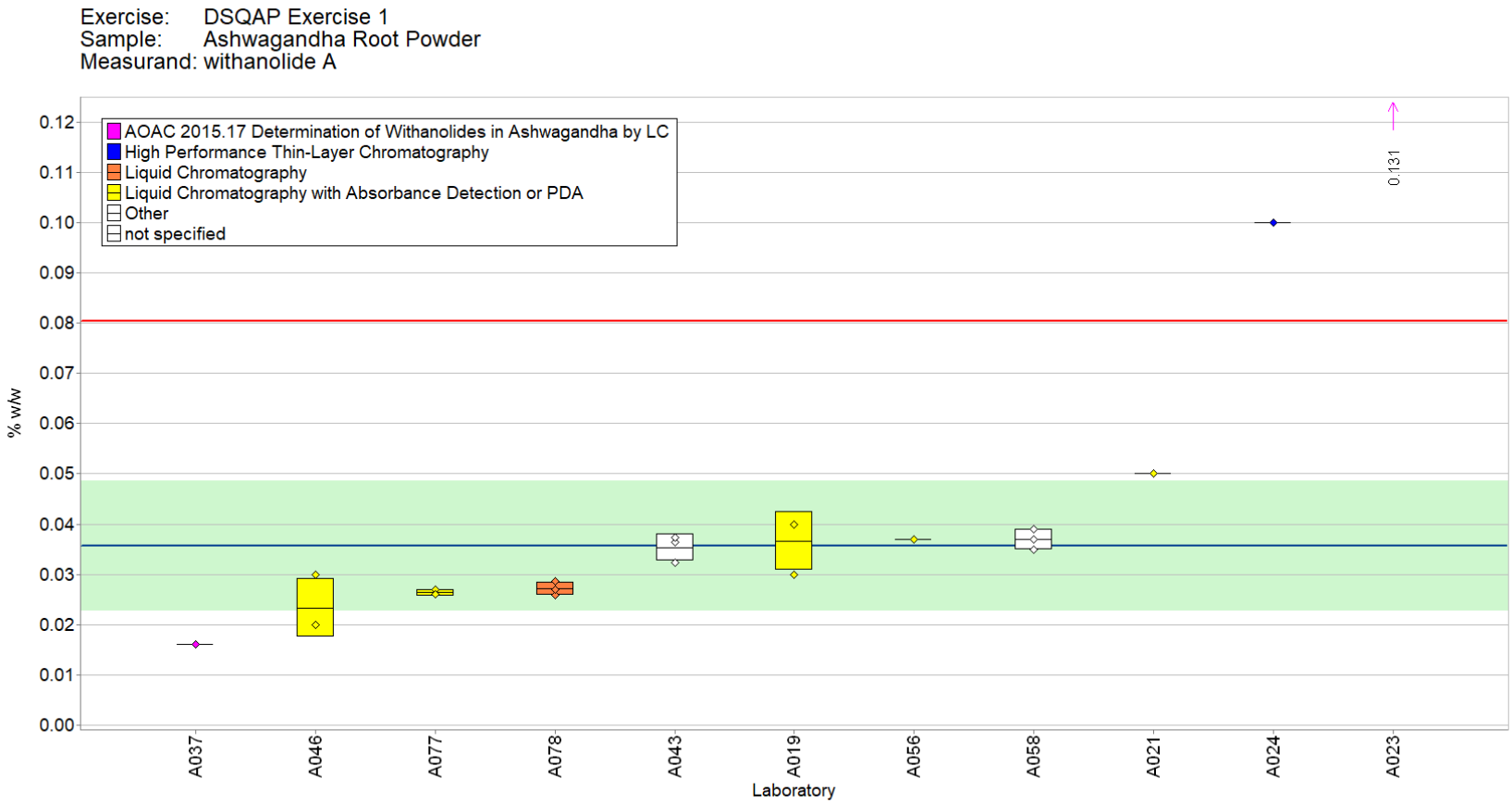

Fig. G-6. Withanolide A in ashwagandha root powder (data summary view – analytical method).

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 red solid line represents the upper bound of the consensus range of tolerance, calculated as the value above the consensus mean that results in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$, with the lower bound set to zero.

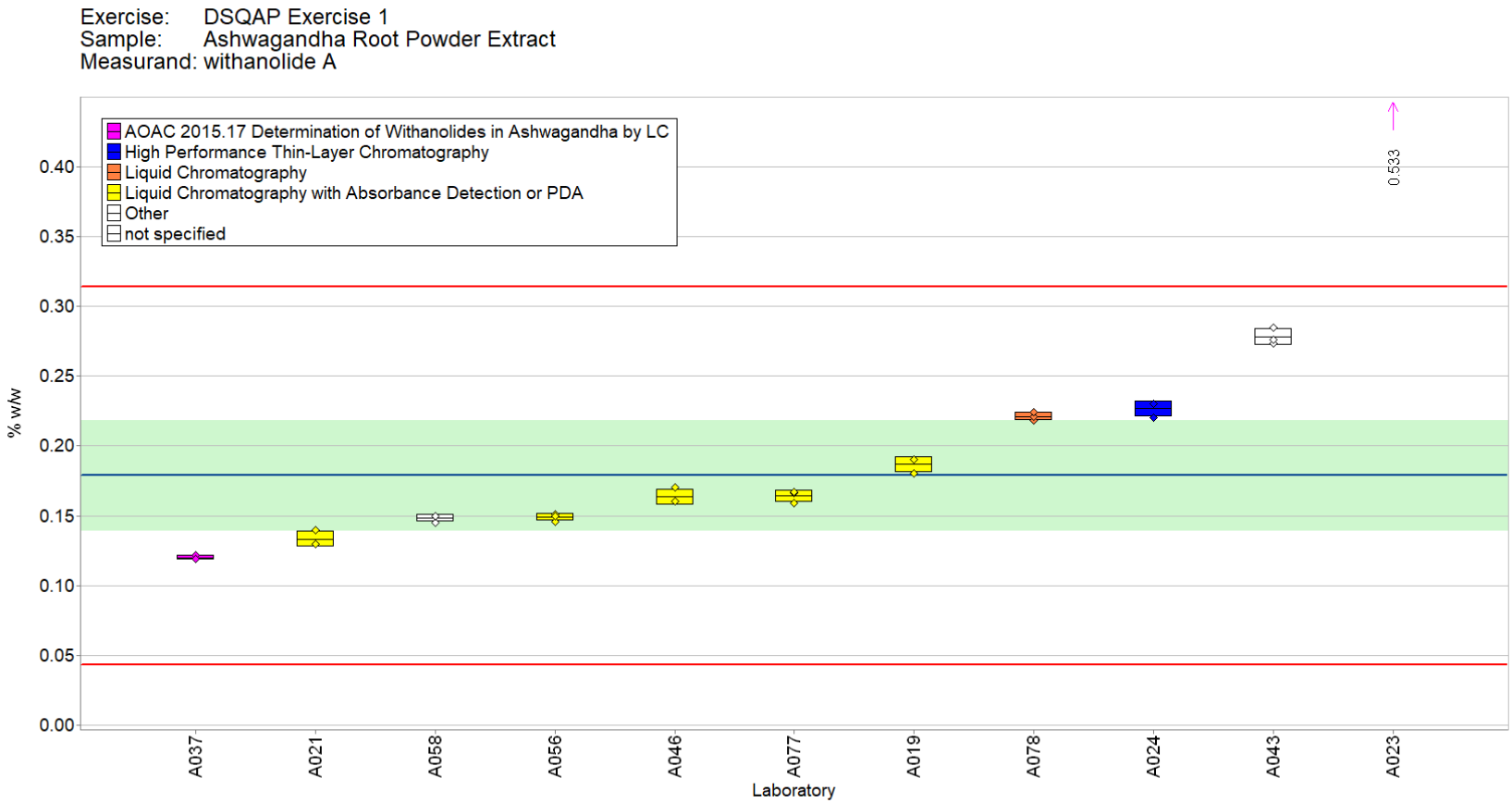

Fig. G-7. Withanolide A in ashwagandha root powder extract (data summary view – analytical method).

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 red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

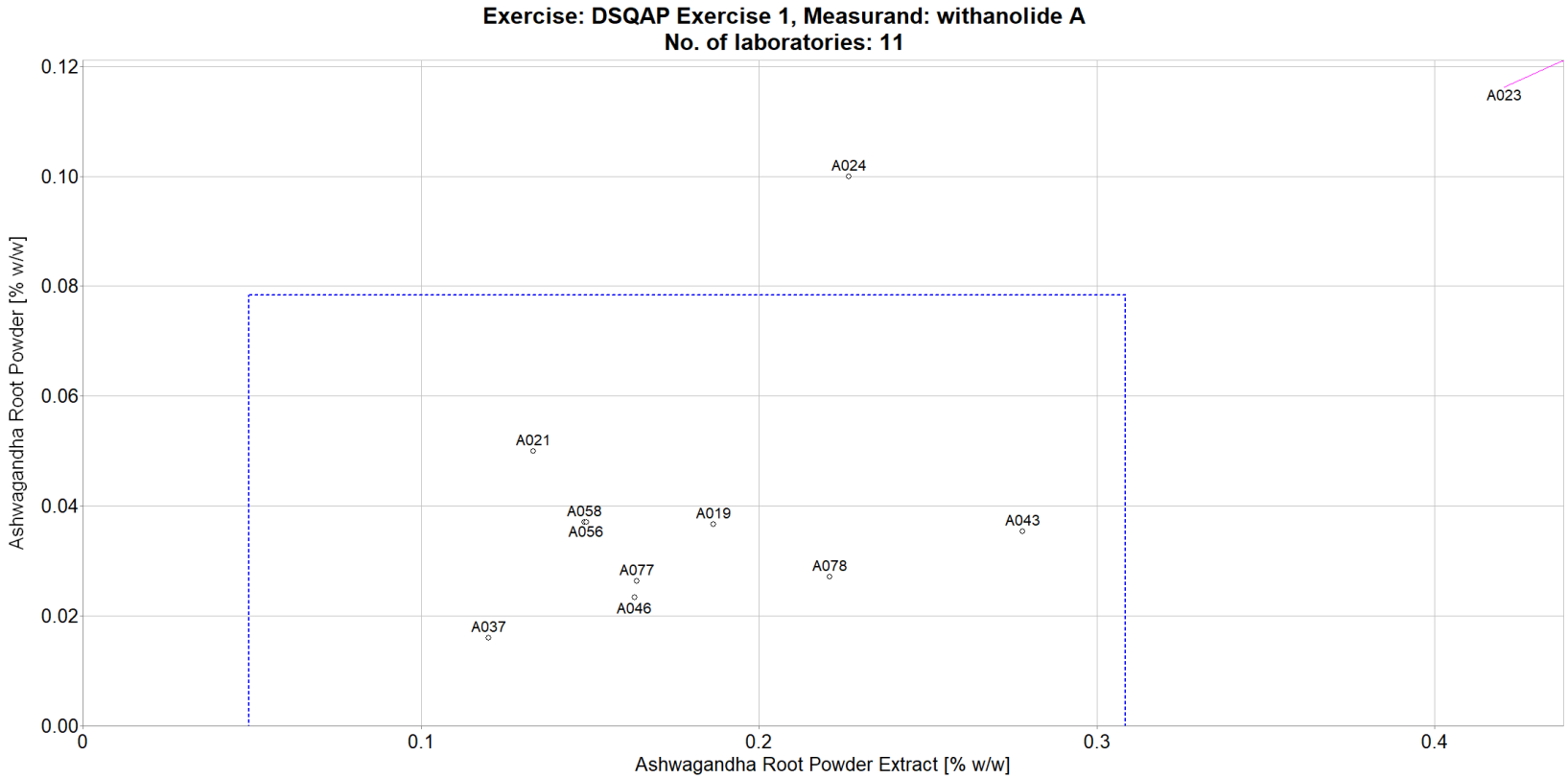

Fig. G-8. Laboratory means for withanolide A in ashwagandha root powder and ashwagandha root powder extract (sample/sample comparison view).

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

Table G-5. Data summary table for withanolide B in ashwagandha root powder and ashwagandha root powder extract.

Data points highlighted in blue have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable Z'_{comm} score, $|Z'_{\text{comm}}| \geq 2$.

		withanolide B									
		Ashwagandha Root Powder (% w/w)					**Ashwagandha Root Powder Extract (% w/w)**				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
Individual Results	Target										
	A001										
	A002										
	A019	0.02	0.02	0.02	0.02	0	0.08	0.08	0.08	0.08	0
	A021	0.03	0.03	0.03	0.03	0	0.07	0.07	0.07	0.07	0
	A023	0.0288	0.0282	0.0284	0.028	0.0003	0.1242	0.1274	0.1261	0.126	0.002
	A024	0.006	0.006	0.007	0.006	0.001	0.037	0.031	0.03	0.033	0.004
	A030										
	A037	0.005	0.005	0.005	0.005	0	0.036	0.037	0.036	0.036	0.001
	A043										
	A046	0.004	0.004	0.004	0.004	0	0.03	0.03	0.02	0.027	0.006
	A049										
	A056	0.014	0.016	0.015	0.015	0.001	0.043	0.047	0.046	0.045	0.002
	A058	0.0077	0.0071	0.0068	0.007	0.0005	0.032	0.033	0.031	0.032	0.001
	A060										
	A076										
	A077	0.005	0.005	0.005	0.005	0	0.031	0.035	0.034	0.033	0.002
	A078	0.00939	0.0104	0.00954	0.010	0.001	0.0576	0.0587	0.0589	0.058	0.001
Community Results		Consensus Mean			0.012		Consensus Mean			0.049	
		Consensus Standard Deviation			0.007		Consensus Standard Deviation			0.023	
		Maximum			0.030		Maximum			0.126	
		Minimum			0.004		Minimum			0.027	
		N			10		N			10	

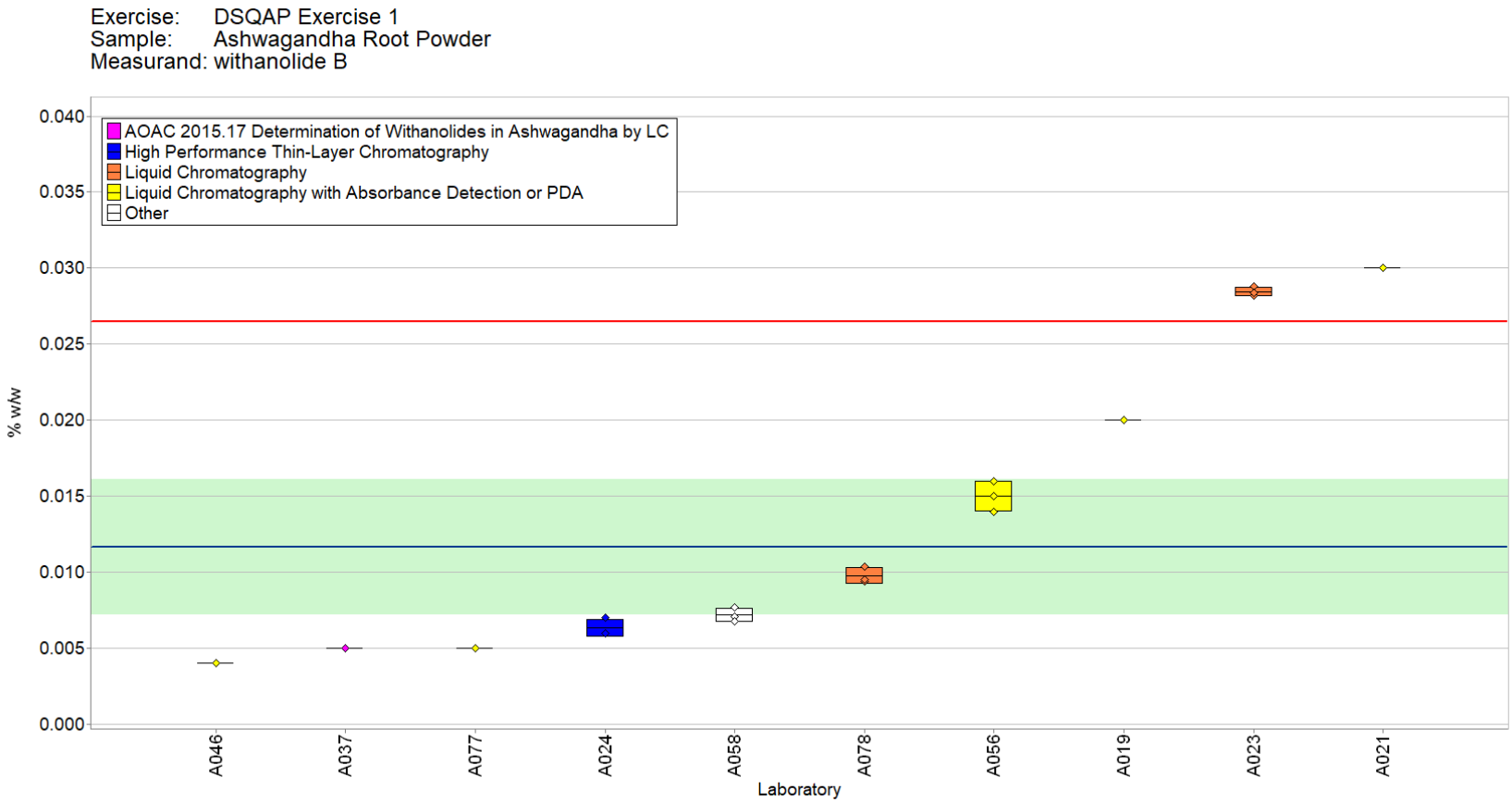

Fig. G-9. Withanolide B in ashwagandha root powder (data summary view – analytical method).

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 red solid line represents the upper bound of the consensus range of tolerance, calculated as the value above the consensus mean that results in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$, with the lower bound set to zero.

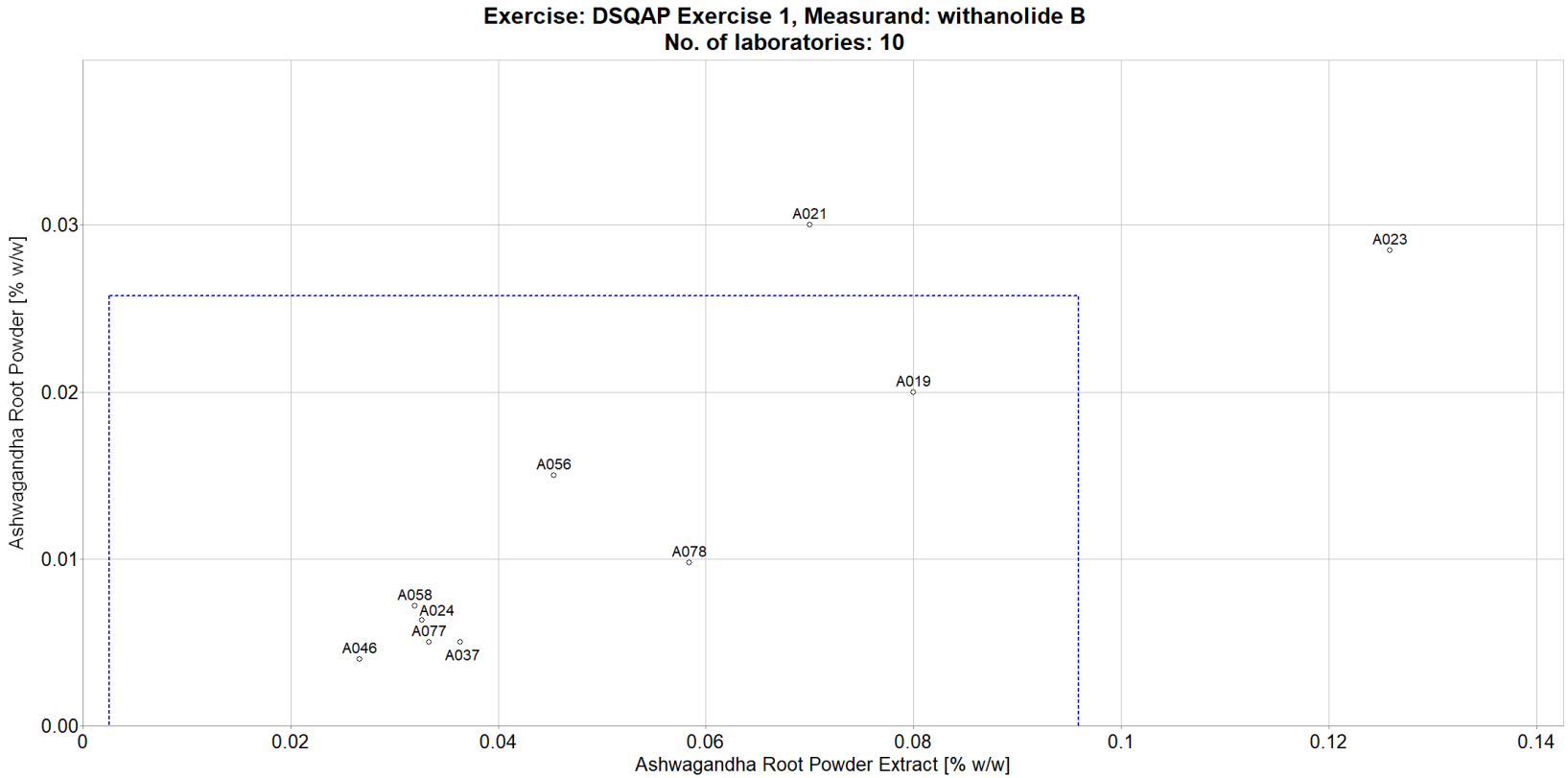

Fig. G-10. Laboratory means for withanolide B in ashwagandha root powder and ashwagandha root powder extract (sample/sample comparison view).

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

Table G-6. Data summary table for withanoside IV in ashwagandha root powder and ashwagandha root powder extract.

Data points highlighted in blue have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable Z'_{comm} score, $|Z'_{\text{comm}}| \geq 2$.

		withanoside IV									
		Ashwagandha Root Powder (% w/w)					Ashwagandha Root Powder Extract (% w/w)				
	Lab	A	B	C	Avg	SD	A	B	C	Avg	SD
Individual Results	Target										
	A001										
	A002										
	A019	0.06	0.06	0.05	0.057	0.006	0.66	0.68	0.63	0.657	0.025
	A021	0.06	0.06	0.06	0.06	0	0.66	0.65	0.66	0.657	0.006
	A023	0.108	0.107	0.1076	0.108	0.001	0.7624	0.7745	0.7697	0.769	0.006
	A030										
	A037	0.06	0.059	0.06	0.060	0.001	0.684	0.672	0.71	0.689	0.019
	A043										
	A046	0.07	0.07	0.07	0.07	0	0.63	0.65	0.69	0.657	0.031
	A049										
	A056										
	A058	0.096	0.097	0.094	0.096	0.002	0.64	0.64	0.65	0.643	0.006
	A060										
	A076										
	A077	0.077	0.077	0.077	0.077	0	0.669	0.683	0.686	0.679	0.009
	A078	0.184	0.205	0.192	0.194	0.011	1.34	1.35	1.37	1.353	0.015
Community Results		Consensus Mean			0.077		Consensus Mean			0.676	
		Consensus Standard Deviation			0.029		Consensus Standard Deviation			0.049	
		Maximum			0.194		Maximum			1.353	
		Minimum			0.057		Minimum			0.643	
		N			8		N			8	

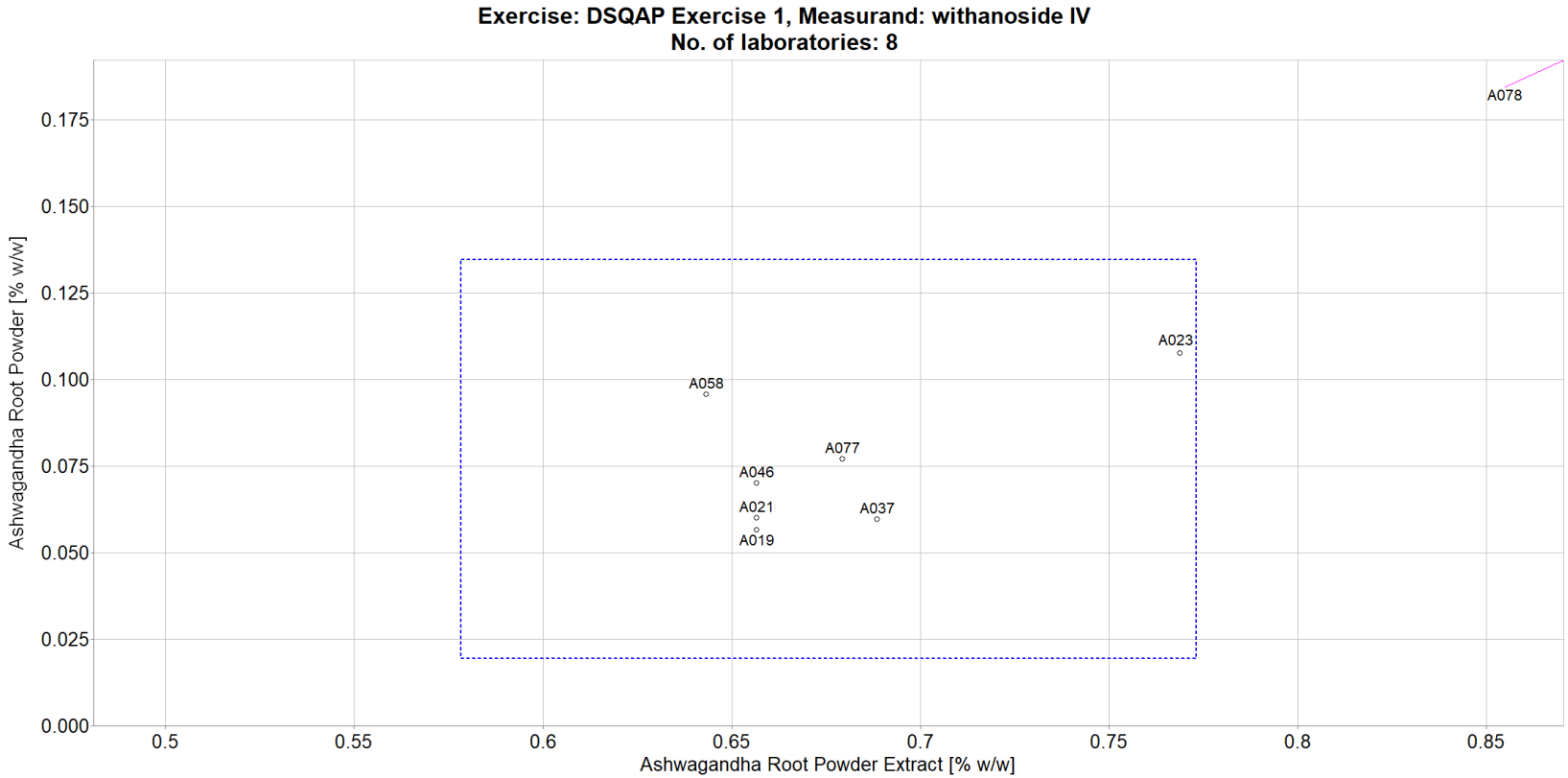

Fig. G-11. Laboratory means for withanoside IV in ashwagandha root powder and ashwagandha root powder extract (sample/sample comparison view).

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

Table G-7. Data summary table for withanoside V in ashwagandha root powder and ashwagandha root powder extract.

Data points highlighted in blue have been identified as outside the consensus tolerance limits and would be estimated to result in an unacceptable Z'_{comm} score, $|Z'_{\text{comm}}| \geq 2$.

		withanoside V									
		Ashwagandha Root Powder (% w/w)					Ashwagandha Root Powder Extract (% w/w)				
	Lab	**A**	**B**	**C**	**Avg**	**SD**	**A**	**B**	**C**	**Avg**	**SD**
Individual Results	Target										
	A001										
	A002										
	A019	0.04	0.04	0.03	0.037	0.006	0.36	0.37	0.34	0.357	0.015
	A021	0.04	0.04	0.04	0.04	0	0.34	0.34	0.35	0.343	0.006
	A023	0.0712	0.0704	0.0705	0.071	0.0004	0.4309	0.4366	0.4484	0.439	0.009
	A030										
	A037	0.037	0.037	0.039	0.038	0.001	0.355	0.326	0.311	0.331	0.022
	A043										
	A046	0.06	0.06	0.06	0.06	0	0.39	0.4	0.43	0.407	0.021
	A049										
	A056										
	A058	0.069	0.07	0.068	0.069	0.001	0.4	0.39	0.39	0.393	0.006
	A060										
	A076										
	A077	0.065	0.065	0.066	0.065	0.001	0.516	0.591	0.586	0.564	0.042
	A078	0.00961	0.0111	0.0103	0.010	0.001	0.361	0.366	0.346	0.358	0.010
Community Results		Consensus Mean			0.050		Consensus Mean			0.389	
		Consensus Standard Deviation			0.020		Consensus Standard Deviation			0.064	
		Maximum			0.071		Maximum			0.564	
		Minimum			0.010		Minimum			0.331	
		N			8		N			8	

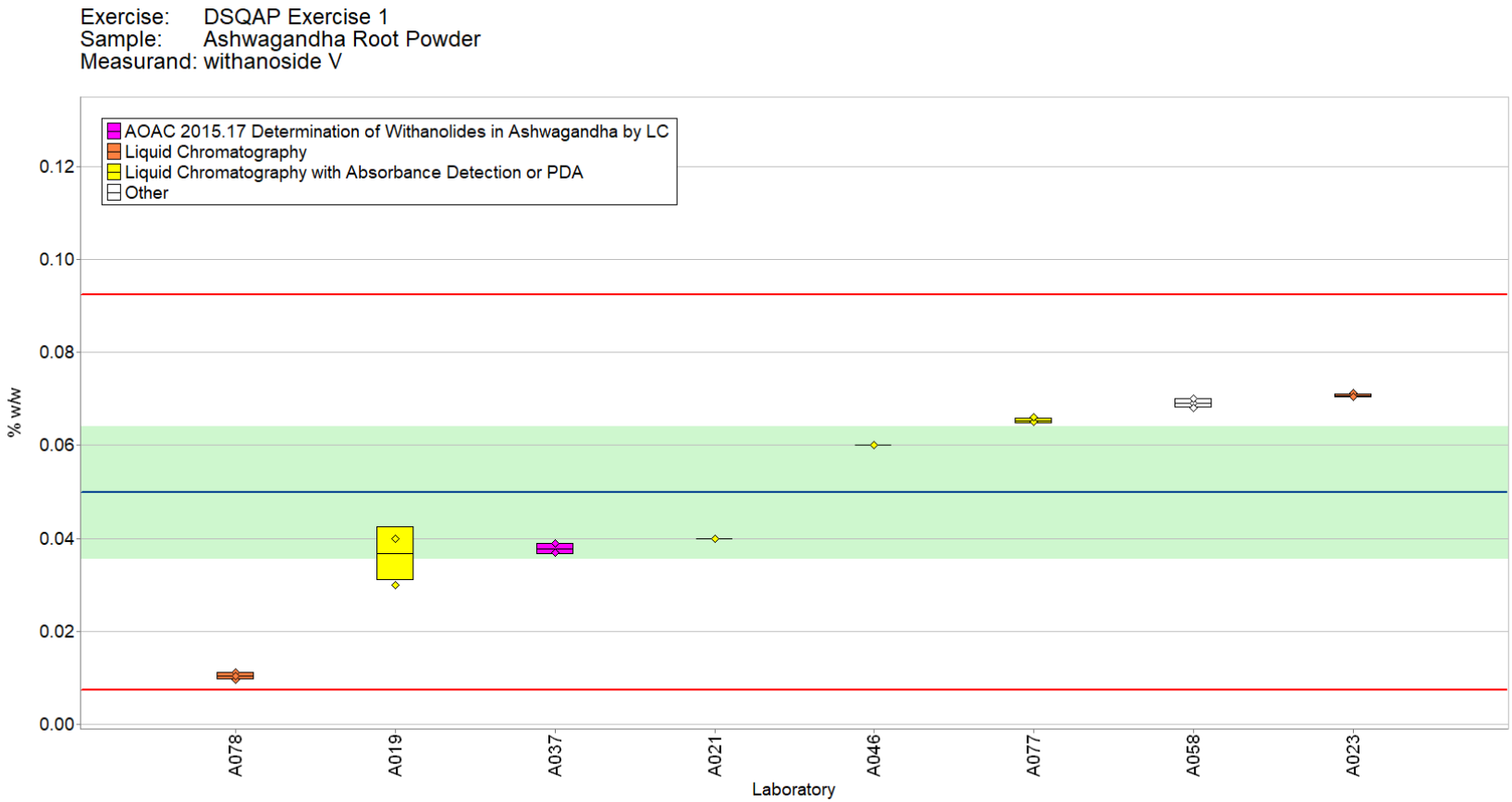

Fig. G-12. Withanoside V in ashwagandha root powder (data summary view – analytical method).

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 red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

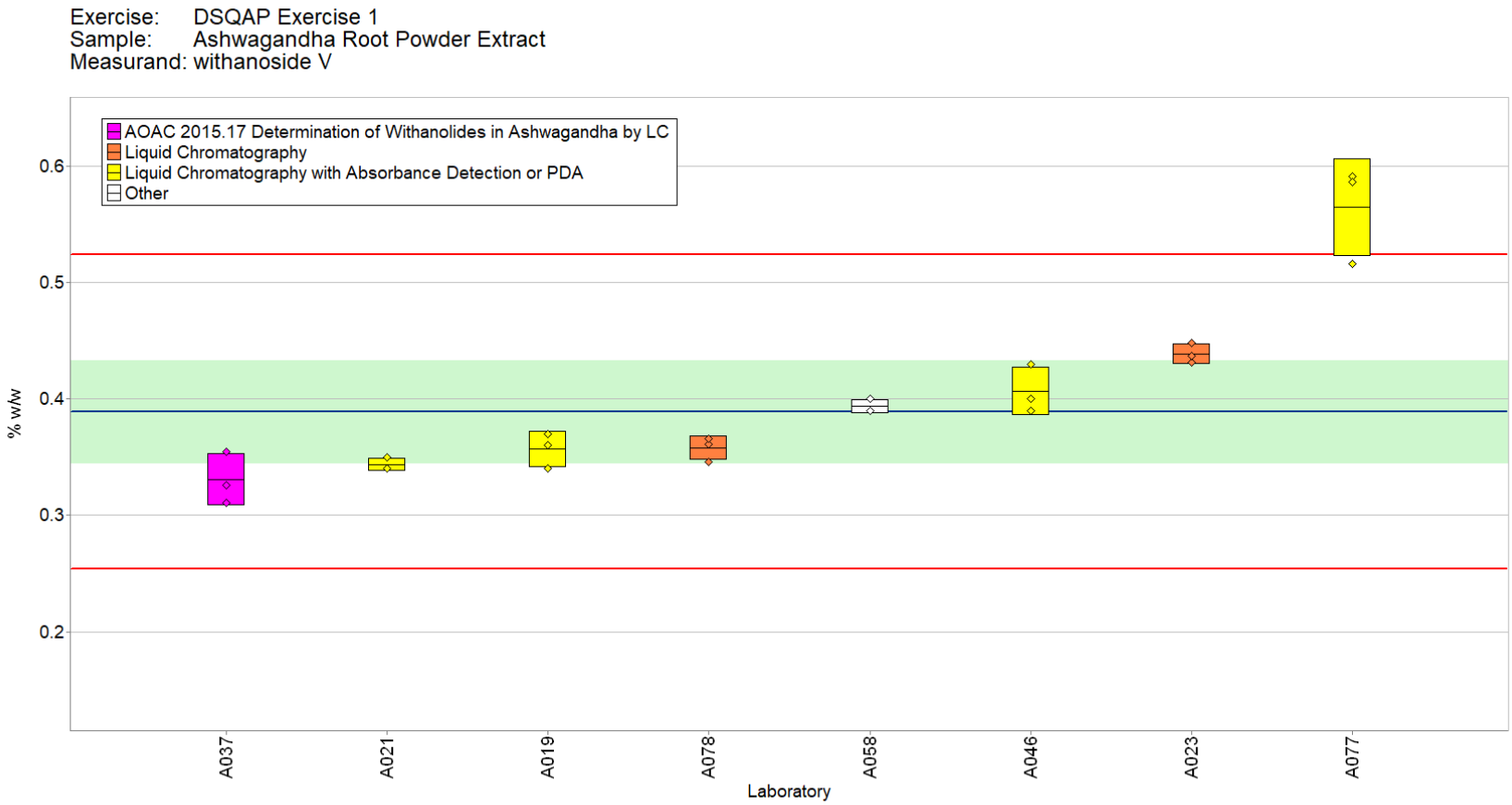

Fig. G-13. Withanoside V in ashwagandha root powder extract (data summary view – analytical method).

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 red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

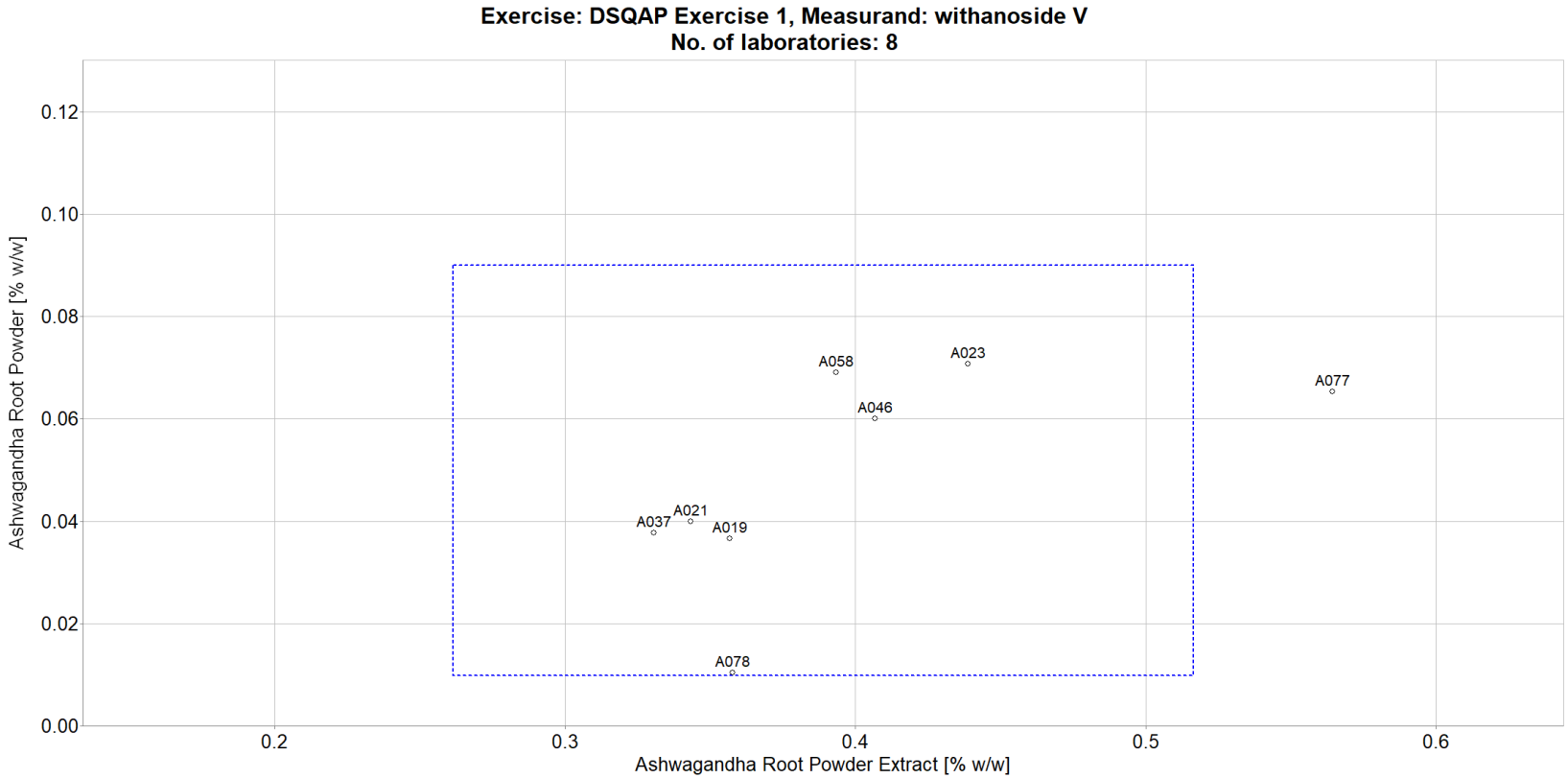

Fig. G-14. Laboratory means for withanoside V in ashwagandha root powder and ashwagandha root powder extract (sample/sample comparison view).

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