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NIST Micronutrients Measurement Quality Assurance Program Winter and Summer 2000 Comparability Studies

Results for Round Robins XLVII and XLVIII Fat-Soluble Vitamins and Carotenoids in Human Serum and Round Robin 13 Ascorbic Acid in Human Serum

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National Institute of Standards and Technology
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July, 2013

U.S. Department of Commerce
Penny Pritzker, Secretary

National Institute of Standards and Technology
Patrick D. Gallagher, Under Secretary of Commerce for Standards and Technology and Director

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Abstract

The National Institute of Standards and Technology coordinates the Micronutrients Measurement Quality Assurance Program (MMQAP) for laboratories that measure fat- and water-soluble vitamins and carotenoids in human serum and plasma. This report describes the design of and results for the Winter and Summer 2000 MMQAP measurement comparability improvement studies: 1) Round Robin XLVII Fat-Soluble Vitamins and Carotenoids in Human Serum, 2) Round Robin XLVIII Fat-Soluble Vitamins and Carotenoids in Human Serum and 3) Round Robin 13 Total Ascorbic Acid in Human Serum. The materials for Round Robin XLVII were shipped to participants in February 2000; participants were requested to provide their measurement results by May 12, 2000. The materials for Round Robin XLIII and Round Robin 13 were shipped to participants in June 2000; participants were requested to provide their measurement results by September 15, 2000.

Keywords

Human Serum
Retinol, α-Tocopherol, γ-Tocopherol, Total and *Trans*-β-Carotene
Total Ascorbic Acid

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Introduction

Beginning in 1988, the National Institute of Standards and Technology (NIST) has coordinated the Micronutrients Measurement Quality Assurance Program (MMQAP) for laboratories that measure fat- and water-soluble vitamins and carotenoids in human serum and plasma. The MMQAP provides participants with measurement comparability assessment through use of interlaboratory studies, Standard Reference Materials (SRMs) and control materials, and methods development and validation. Serum-based samples with assigned values for the target analytes (retinol, alpha-tocopherol, gamma/beta-tocopherol, *trans*- and total beta-carotene, and total ascorbic acid) and performance-evaluation standards are distributed by NIST to laboratories for analysis.

Participants use the methodology of their choice to determine analyte content in the control and study materials. Participants provide their data to NIST, where it is compiled and evaluated for trueness relative to the NIST value, within-laboratory precision, and concordance within the participant community. NIST provides the participants with a technical summary report concerning their performance for each exercise and suggestions for methods development and refinement. Participants who have concerns regarding their laboratory's performance are encouraged to consult with the MMQAP coordinators.

All MMQAP interlaboratory studies consist of individual units of batch-prepared samples that are distributed to each participant. For historical reasons these studies are referred to as "Round Robins". The MMQAP program and the nature of its studies are described elsewhere. [1,2]

Round Robin XLVII: Fat-Soluble Vitamins and Carotenoids in Human Serum

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin XLVII comparability study (hereafter referred to as RR47) received three lyophilized and two liquid-frozen human serum test samples for analysis. Unless multiple vials were previously requested, participants received one vial of each serum. These sera were shipped on dry ice to participants in February 2000. The communication materials included in the sample shipment are provided in Appendix A.

Participants are requested to report values for all fat-soluble vitamin-related analytes that are of interest to their organizations. Not all participants report values for the target analytes, and many participants report values for non-target analytes.

The final report delivered to every participant in RR47 consists of three documents:

- A cover letter for the current study, a brief description of the other two documents, and a discussion of our analysis of the overall results that may be of broad interest. This cover letter is reproduced as Appendix B.

- The "All-Lab Report" that lists all of the reported measurement results, a number of consensus statistics for analytes reported by more than one participant, and the mean median and pooled SD from any prior distributions of the serum. This report also provides a numerical "score card" for each participant's measurement comparability for the more commonly reported analytes. This report is reproduced as Appendix C.

- An “Individualized Report” that graphically analyzes each participant’s results for selected analytes. This report also provides a graphical summary of their measurement comparability. The graphical tools used in this report are described in detail elsewhere [3]. An example “Individualized Report” is reproduced as Appendix D.

Round Robin XLVIII: Fat-Soluble Vitamins and Carotenoids in Human Serum

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin XLVIII comparability study (hereafter referred to as RR48) received three lyophilized and two liquid-frozen human serum test samples for analysis. Unless multiple vials were previously requested, participants received one vial of each serum. These sera were shipped on dry ice to participants in June 2000. The communication materials included in the sample shipment are provided in Appendix E.

Participants are requested to report values for all fat-soluble vitamin-related analytes that are of interest to their organizations. Not all participants report values for the target analytes, and many participants report values for non-target analytes.

The final report delivered to every participant in RR48 consists of three documents:

- A cover letter for the current study, a brief description of the other two documents, and a discussion of our analysis of the overall results that may be of broad interest. This cover letter is reproduced as Appendix F.

- The “All-Lab Report” that lists all of the reported measurement results, a number of consensus statistics for analytes reported by more than one participant, and the mean median and pooled SD from any prior distributions of the serum. This report also provides a numerical “score card” for each participant’s measurement comparability for the more commonly reported analytes. This report is reproduced as Appendix G.

- An “Individualized Report” that graphically analyzes each participant’s results for selected analytes. This report also provides a graphical summary of their measurement comparability. The graphical tools used in this report are described in detail elsewhere [3]. An example “Individualized Report” is reproduced as Appendix H.

Round Robin 13: Vitamin C in Human Serum

Participants in the MMQAP Vitamin C in Human Serum Round Robin 13 comparability study (hereafter referred to as RR13) received four frozen serum test samples and a solid ascorbic acid control material for analysis. Unless multiple vials were previously requested, participants received one vial of each material. These sample materials were shipped on dry ice to participants in June 2000. The communication materials included in the sample shipment are provided in Appendix I.

The test and control serum materials were prepared by adding equal volumes of 10 % metaphosphoric acid (MPA) to human serum that had been spiked with ascorbic acid. While these samples contain some dehydroascorbic acid, its content is variable. Therefore, the participants report only total ascorbic acid (TAA, ascorbic acid plus dehydroascorbic acid). Participants are also

encouraged to prepare calibration solutions from the supplied solid control to enable calibrating their serum measurements to the same reference standard.

The final report delivered to every participant in RR13 consists of three documents:

- A cover letter for the current study, a brief description of the other two documents, and a discussion of our analysis of overall results that may be of broad interest. This cover letter is reproduced as Appendix J.

- The "All-Lab Report" that summarizes all of the reported measurement results and provides several consensus statistics. This report is reproduced as Appendix K.

- An "Individualized Report" that graphically analyzes each participant's results for TAA, including a graphical summary of their measurement comparability. The graphical tools used in this report are described in detail elsewhere [3]. An example "Individualized Report" is reproduced as Appendix L.

References

1 Duewer DL, Brown Thomas J, Kline MC, MacCrehan WA, Schaffer R, Sharpless KE, May WE, Crowell JA. NIST/NCI Micronutrients Measurement Quality Assurance Program: Measurement Repeatabilities and Reproducibilities for Fat-Soluble Vitamin-Related Compounds in Human Sera. Anal Chem 1997;69(7):1406-1413.

2 Margolis SA, Duewer DL. Measurement Of Ascorbic Acid in Human Plasma and Serum: Stability, Intralaboratory Repeatability, and Interlaboratory Reproducibility. Clin Chem 1996;42(8):1257-1262.

3 Duewer DL, Kline MC, Sharpless KE, Brown Thomas J, Gary KT, Sowell AL. Micronutrients Measurement Quality Assurance Program: Helping Participants Use Interlaboratory Comparison Exercise Results to Improve Their Long-Term Measurement Performance. Anal Chem 1999;71(9):1870-1878.

Appendix A. Shipping Package Inserts for RR47

The following two items were included in each package shipped to an RR47 participant:

- Cover letter
- Datasheet

The cover letter and datasheet were enclosed in a sealed waterproof bag along with the samples themselves.

UNITED STATES DEPARTMENT OF COMMERCE
National Institute of Standards and Technology
Gaithersburg, Maryland 20899-

February 17, 2000

Dear Colleague:

Enclosed is the set of samples for the first quality assurance round robin exercise (Round Robin XVII) for 2000. You will find one vial of each of two lyophilized and two liquid-frozen serum samples for analysis along with a form for reporting your results. When reporting your results, please submit one value for each analyte for a given serum sample. If an obtained value is below your limit of quantitation, please indicate this result on the form by using NQ *(Not Quantitated)*. For analytes not measured, please leave a blank. Results are due to NIST by **May 12, 2000**. Results received two weeks after the due date will not be included in the summary report for this round robin study. The feedback report concerning the study will be provided around mid-June.

Samples should be reconstituted with 1.0 mL of HPLC-grade water or equivalent. We recommend that dissolution be facilitated with 3 to 5 min agitation in an ultrasonic bath or at least 30 min at room temperature with intermittent swirling. (CAUTION: Vigorous shaking will cause foaming and possibly interfere with accurate measurement. The rubber stopper contains phthalate esters that will leach into the sample upon intermittent contact of the liquid sample with the stopper. These esters absorb strongly in the UV region and elute very near retinol in most LC systems creating analytical problems.) Pipette a known volume of serum from the vial for analysis. (The final volume of the reconstituted sample is greater than 1.0 mL.). For consistency, we request that laboratories use the following absorptivities (E 1% cm) in ethanol: retinol, 1843 at 325 nm; retinyl palmitate, 975 at 325 nm; α-tocopherol, 75.8 at 292 nm; γ-tocopherol, 91.4 at 298 nm; α-carotene, 2800 at 444 nm (in hexane); β-carotene, 2560 at 450 nm (in ethanol), 2592 at 452 nm (in hexane); lycopene, 3450 at 472 nm (in hexane).

Please mail or fax your results for Round Robin XLVII to:

Micronutrients Measurement Quality Assurance Program
NIST
100 Bureau Drive Stop 8392
Gaithersburg, MD 20899-8392
Fax: (301) 977-0685

If you have questions regarding this round robin exercise, please call me at (301) 975-3120;e-mail me at jbthomas@nist.gov; or mail/fax queries to the above address. Please note: the second set of samples for the fat-soluble vitamins and carotenoids will be shipped during the week of June 19; results will be due by September 15. The feedback report will be provided mid-October.

Sincerely,

Jeanice Brown Thomas
Research Chemist
Analytical Chemistry Division
Chemical Science and Technology Laboratory

Enclosures
cc: L. C. Sander

NIST

Participant #: _____ Date: _____

Round Robin XLVII

NIST Micronutrients Measurement Quality Assurance Program

Analyte	259	260	261	262	263	Units*
total retinol						
trans-retinol						
retinyl palmitate						
α-tocopherol						
γ-tocopherol						
δ-tocopherol						
total β-carotene						
trans-β-carotene						
total cis-β-carotene						
total α-carotene						
trans-α-carotene						
total lycopene						
trans-lycopene						
total β-cryptoxanthin						
total α-cryptoxanthin						
total lutein						
total zeaxanthin						
total lutein&zeaxanthin						
ubiquinone-10 (Q_{10})						
phylloquinone (K_1)						
25-hydroxyvitamin D						

Other analytes?						

* We prefer μg/mL

Were sera 259 & 260 frozen when received? Yes | No

Mail: M^2QAP
NIST, Stop 8392
Gaithersburg, MD 20899-8392

Please return results on-or-before
12-May-2000

Fax: 301-977-0685
Email: David.Duewer@NIST.gov

Appendix B. Final Report for RR47

The following four pages are the final report as provided to all participants:

- Cover letter
- An information sheet that:
  - describes the contents of the “All-Lab” report
  - describes the content of the “Individualized” report
  - describes the nature of the test samples and details any previous distributions
  - summarizes aspects of the study that we believe may be of interest to the participants

UNITED STATES DEPARTMENT OF COMMERCE
National Institute of Standards and Technology
Gaithersburg, Maryland 20899-

June 16, 2000

Dear Colleague:

Enclosed is the summary report of the results for Round Robin XLVII (RR 47). Included in this report are: a summary of data for all laboratories; the measurement comparability summary for evaluating laboratory performance; a summary of individual laboratory performance and interlaboratory accuracy and precision; and a summary of the NIST assigned value (NAV) vs. your laboratory value for the analytes you measured. As in previous reports, the NIST assigned values are derived from the equally weighted means of the medians from this interlaboratory comparison exercise and the means from the analyses performed by NIST.

Data for evaluating laboratory performance in RR 47 are provided in the comparability summary (Score Card) on page 6 of the "All Lab Report." Laboratory comparability is summarized as follows: results rated 1 to 3 are within 1 to 3 standard deviation(s) of the assigned value, respectively; those rated 4 are >3 standard deviations from the assigned value.

If you have concerns regarding your laboratory's performance, we suggest that you obtain and analyze a unit of SRM 968c, Fat-Soluble Vitamins, Carotenoids, and Cholesterol in Human Serum. If your measured values do not agree with the certified values, we suggest that you contact us for consultation.

The following are newly released or forthcoming publications. Reprints will be provided upon request.

"NIST Micronutrients Measurement Quality Assurance Program: Characterizing Individual Participant Measurement Performance Over Time," Duewer et al., Anal. Chem. 1999, 71, 1870-1878. http://dx.doi.org/10.1021/ac991481b

"Liquid Chromatographic Methods for the Determination of Vitamins in Food-Matrix Standard Reference Materials," Sharpless et al., J. Chromatogr. A, 2000, 881(1-2), 171-181. www.elsevier.nl/oasis

"Analysis of Anthocyanins in Foods by Liquid Chromatography, Liquid Chromatography Mass Spectrometry and Capillary Electrophoresis," da Costa et al., J. Chromatogr. A, 2000, 881(1-2), 403-410.www.elsevier.nl/oasis

"NIST Micronutrients Measurement Quality Assurance Program: Characterizing the Measurement Community's Performance Over Time," Duewer et al., Anal. Chem., in press.

"Preparation and Value Assignment of Standard Reference Material 968c: Fat-Soluble Vitamins, Carotenoids, and Cholesterol in Human Serum, Brown Thomas et al., submitted to Clin. Chim. Acta.

As you are aware, the participation fees for the QA program will increase in 2001. The participation fees for the fat-soluble vitamins and carotenoids in serum studies (two exercises per year) will be $1600 for U.S. laboratories and $2000 for laboratories outside of the U.S. The fees for the vitamin C in serum study (one exercise per year) will be $800 for U.S. laboratories and $1000 for non-U.S. laboratories. Please complete and return the Intent-to-Participate form no later than **August 4, 2000**.

Invoicing for the 2001 program will take place after shipment of the first set of samples which will be in January 2001.

If you have any questions regarding this report, please contact David Duewer at 301/975-3935; e-mail: david.duewer@nist.gov, or me at 301/975-3120; e-mail: jbthomas@nist.gov; fax: 301/977-0685.

Sincerely,

Jeanice B Thomas

Jeanice Brown Thomas
Research Chemist
Analytical Chemistry Division
Chemical Science and Technology Laboratory

cc: L. C. Sander

Enclosures

The NIST M^2QAP Round Robin XLVII (RR47) report includes the usual

Page	"Individualized" Report
1	Your values, the number of labs reporting values, and our assigned values.
2+	"Four Plot" summaries of your current and past measurement performance, one page for each analyte you report that is also reported by at least 10 other participants.

Page	"All Lab" Report
1-5	A listing of all results and statistics for analytes reported by at least two laboratories
6	A list of results for the four analytes reported by only one laboratory. A legend for the above two lists
7	The "Comparability Summary" (or "Score Card")

We have modified the definition of the "Comparability Summary" scores. Previously, the scores reflected your "worst" value for the given measurand. The scores are now calculated from the concordance and apparent precision characteristic of all your values for the measurand, as displayed in the "target" plots in your Individualized Report and defined in Anal Chem 1999; 71:1074-9. We have also included, on an experimental basis, a graphical version of the "Comparability Summary" that displays the full target plots for 14 measurands all on one page. If you like it – or hate it – please let us know.

Samples. Five sera were distributed in RR47: two pairs of newly prepared {liquid frozen, lyophilized} sera and a redistribution of SRM 968c Level I. The {liquid frozen, lyophilized} pairs were designed to enable investigation of potential measurement differences between "fresh" and lyophilized sera. SRM 968c Level I was distributed as a known reference for the matrix studies and to further assess measurand stability in the SRM.

Sera {#259, #261} are {liquid frozen, lyophilized} material prepared from a freshly drawn male-donor native serum pool. These samples were prepared and aliquoted into vials as a single batch.

Sera {#260, #262} are {liquid frozen, lyophilized} material prepared from a freshly drawn female-donor native serum pool. These samples were prepared and aliquoted into vials as a single batch.

Serum #263 is Level I of SRM 968c, previously distributed as #249 (RR44, 10/98) and #258 (RR46,10/99). This material is a blended serum that was augmented with retinol and γ-tocopherol, and contains native carotenoid levels.

Qualitative Results.

1) A number of participants "reconstituted" the liquid frozen samples with 1 mL water. All such results have been mathematically corrected (2x measured). We will be distributing some liquid frozen samples in the next several Round Robins.

2) A number of participants did not receive their samples in frozen form. We have instituted a more aggressive sample delivery and storage tracking program to help resolve these issues. We ask that you examine carefully all future RR samples upon delivery and quickly provide us with the information requested in the shipment's manifest.

Quantitative Results

1) With the possible exception of total lutien, total zeaxanthin, and total lutein&zeaxanthin, there have been no changes in median level or measurement variability in SRM 968c Level 1. We believe that the small changes in the lutein and zeaxanthin measurands may be more a reflection of measurement systems changes rather than a result of SRM degradation. We will continue to monitor this material and will revise the SRM 968c Certificate if/when necessary.

2) Since the lyophilized materials are reconstituted with 1.0 mL water rather than TO a total volume of 1.0 mL, we expect that the measurand concentrations in the lyophilized member of each {liquid frozen, lyophilized} pair will be about 0.95x that of the liquid frozen. In general, this is true for the retinol and tocopherol measurands. It appears less true for the carotenoids. We will be exploring this issue in greater detail in future round robins.

Appendix C. "All-Lab Report" for RR47

The following seven pages are the "All-Lab Report" as provided to all participants, with two exceptions:

- the participant identifiers (Lab) have been altered.
- the order in which the participant results are listed has been altered.

The data summary in the "All-Lab Report" has been altered to ensure confidentiality of identification codes assigned to laboratories. The only attributed results are those reported by NIST. The NIST results are not used in the assessment of the consensus summary results of the study.

Round Robin XLVII Laboratory Results

All Results are in µg/mL

	Total Retinol					trans-Retinol					Retinyl Palmitate					α-Tocopherol				
Lab	259	260	261	262	263	259	260	261	262	263	259	260	261	262	263	259	260	261	262	263
FSV-BA	0.598	0.705	0.560	0.646	0.878						0.111	0.101	0.097	0.092	0.049	12.1	19.3	10.9	17.4	7.42
FSV-BB	0.520	0.615	0.505	0.608	0.865						0.105	0.100	0.099	0.091	0.030	12.0	19.2	11.5	18.3	7.89
FSV-BD	0.585	0.690	0.564	0.643	0.880											12.5	18.5	10.8	16.9	8.10
FSV-BE	0.710	0.760	0.620	0.710	0.920											13.3	20.0	11.8	18.5	7.50
FSV-BF	0.545	0.640	0.555	0.625	0.865											11.9	19.0	11.6	18.2	7.75
FSV-BG	0.615	0.718	0.547	0.655	0.872						0.127	0.147	0.125	0.132	0.072	13.0	20.8	11.4	18.8	7.44
FSV-BH	0.546	0.649	0.509	0.604	0.774						0.117	0.115	0.126	0.105	0.043	11.8	18.6	10.9	17.0	7.36
FSV-BI	0.603	0.726	0.565	0.684	0.881						0.157	0.151	0.143	0.138	0.042	12.0	18.4	11.2	17.8	7.64
FSV-BJ	0.579	0.668	0.556	0.629	0.866						0.140	0.148	0.121	0.135	0.034	12.7	20.0	12.5	18.5	7.31
FSV-BK	0.548	0.649	0.529	0.627	0.845											11.9	18.5	11.1	17.6	7.60
FSV-BL	0.659	0.716	0.602	0.659	0.802											9.5	13.4	9.5	12.1	8.18
FSV-BM	0.594	0.736	0.603	0.717	0.901											12.2	19.8	11.5	17.6	7.80
FSV-BN	0.514	0.598	0.534	0.636	0.856						0.109	0.092	0.089	0.102	0.039	12.2	18.2	11.6	18.8	8.47
FSV-BO	*>0.521*	*>0.593*	*>0.518*	*>0.536*	*>0.722*	0.521	0.593	0.518	0.536	0.722						9.8	15.2	9.0	13.9	6.37
FSV-BP	0.564	0.664	0.549	0.643	0.817											11.4	15.9	10.1	15.3	7.91
FSV-BQ	0.609	0.711	0.558	0.657	0.873											13.0	20.4	11.7	18.4	8.15
FSV-BR	*>0.600*	*>0.700*	*>0.510*	*>0.650*	*>0.840*	0.600	0.700	0.510	0.650	0.840										
FSV-BU	0.581	0.691	0.520	0.629	0.833											11.5	19.7	11.0	18.7	7.03
FSV-BV	0.480	0.560	0.440	0.517	0.720											12.3	20.2	11.0	17.7	6.83
FSV-BW	0.530	0.660	0.510	0.580	0.790						0.154	0.150	0.160	0.150	0.041	10.4	17.2	9.8	15.4	6.51
FSV-BX	*>0.599*	*>0.699*	*>0.568*	*>0.641*	*>0.857*	0.599	0.699	0.568	0.641	0.857						12.8	20.4	12.2	18.8	8.00
FSV-BZ																9.6	16.2	8.9	14.3	7.00
FSV-CB	0.521	0.603	0.526	0.593	0.866											11.7	18.0	11.4	17.7	7.74
FSV-CC	0.550	0.670	0.590	0.670	0.910	0.550	0.660	0.540	0.630	0.820						11.7	19.3	11.4	18.2	7.70
FSV-CD	*>0.567*	*>0.624*	*>0.500*	*>0.598*	*>0.786*	0.567	0.624	0.500	0.598	0.786	0.091	0.090	0.083	0.090	0.048	10.9	16.0	9.1	14.3	6.21
FSV-CE	0.540	0.630	0.520	0.610	0.840											12.7	19.6	11.8	18.7	6.52
FSV-CF	0.602	0.710	0.568	0.673	0.877											12.8	19.8	11.9	18.9	8.10
FSV-CG	0.623	0.761	0.744	0.853	1.232											10.2	16.3	9.4	15.2	6.22
FSV-CH	0.483	0.567	0.468	0.538	0.706											10.7	16.3	10.0	14.9	6.36
FSV-CI	0.670	0.760	0.760	0.730	1.000						0.130	0.070	0.160	0.130	0.060	11.4	17.9	12.9	17.2	7.70
FSV-CL	0.579	0.769	0.594	0.646	0.857											14.4	26.7	14.9	22.4	9.24
FSV-CN	0.586	0.705	0.566	0.658	0.886											12.5	20.1	12.1	18.7	8.02
FSV-CR	0.650	0.370	0.700	0.730	0.900											12.3	9.3	12.5	19.8	7.30
FSV-CS	*>0.577*	*>0.695*	*>0.529*	*>0.646*	*>0.886*	0.577	0.695	0.529	0.646	0.886						10.4	18.1	10.3	17.4	6.74
FSV-CU	*>0.613*	*>0.695*	*>0.541*	*>0.608*	*>0.782*	0.613	0.695	0.541	0.608	0.782	0.127	0.132	0.124	0.115	0.074	11.1	18.1	9.9	16.0	6.91
FSV-CV	0.529	0.487	0.585	0.564	0.961						0.122	0.139	0.127	0.123	0.022	12.3	18.0	13.2	15.6	7.14
FSV-CW	*>0.473*	*>0.543*	*>0.459*	*>0.597*	*>1.019*	0.473	0.543	0.459	0.597	1.019	0.102	0.114	0.100	0.115	0.026	11.9	18.2	12.3	19.7	6.75
FSV-CX	*>0.643*	*>0.769*	*>0.600*	*>0.715*	*>0.921*	0.643	0.769	0.600	0.715	0.921										
FSV-DA	*>0.619*	*>0.745*	*>0.563*	*>0.667*	*>0.855*	0.619	0.745	0.563	0.667	0.855	0.181	0.145	0.163	0.108	0.039	11.8	20.0	11.6	17.2	7.32
FSV-DB	0.490	0.580	0.486	0.616	0.842											11.0	17.5	10.9	17.6	8.23
FSV-DF	0.505	0.625	0.485	0.564	0.768															
FSV-DI	*>0.582*	*>0.690*	*>0.547*	*>0.643*	*>0.841*	0.582	0.690	0.547	0.643	0.841	0.118	0.110	0.095	0.092	0.027	12.7	20.1	11.8	18.8	7.91
FSV-DK	0.533	0.627	0.506	0.593	0.803						0.065	0.062	0.067	0.045	0.023	11.2	18.4	10.7	16.2	6.13
FSV-DP	*>0.609*	*>0.723*	*>0.586*	*>0.683*	*>0.853*	0.609	0.723	0.586	0.683	0.853										
FSV-DQ																10.1	16.0	8.1	13.6	6.89
FSV-DR	0.615	0.687	0.548	0.636	0.822											12.0	19.2	11.0	18.3	6.89
FSV-DU	0.770	0.750	0.560	0.690	0.930											16.8	24.1	13.4	21.7	10.86
FSV-EM	0.520	0.570	0.490	0.640	0.790											10.0	16.6	10.7	18.0	7.37
FSV-EQ	0.698	0.810	0.647	0.757	0.989											13.9	21.2	12.4	19.5	8.48
FSV-ES	*>0.626*	*>0.742*	*>0.522*	*>0.677*	*>0.870*	0.626	0.742	0.522	0.677	0.870						12.6	19.3	10.6	17.8	7.90
FSV-FH	0.668	0.799	0.655	0.769	1.051											11.1	17.1	10.4	16.0	7.10
FSV-FT	0.740	0.890	0.730	0.870	1.350											10.9	17.6	11.0	15.3	8.29
FSV-FW	0.558	0.705	0.582	0.604	0.797															
N	39	39	39	39	39	13	13	13	13	13	16	16	16	16	16	48	48	48	48	48
Min	0.480	0.370	0.440	0.517	0.706	0.473	0.543	0.459	0.536	0.722	0.065	0.062	0.067	0.045	0.022	9.5	9.3	8.1	12.1	6.13
Median	0.579	0.687	0.558	0.643	0.866	0.599	0.695	0.540	0.643	0.853	0.120	0.115	0.123	0.112	0.040	11.9	18.5	11.2	17.7	7.47
Max	0.770	0.890	0.760	0.870	1.350	0.643	0.769	0.600	0.715	1.019	0.181	0.151	0.163	0.150	0.074	16.8	26.7	14.9	22.4	10.86
eSD	0.065	0.085	0.056	0.052	0.065	0.032	0.052	0.034	0.050	0.049	0.019	0.036	0.036	0.029	0.014	1.1	1.9	1.0	1.5	0.80
eCV	11	12	10	8	8	5	7	6	8	6	16	32	30	26	35	10	11	9	9	11
N_{past}	0	0	0	0	47	0	0	0	0	7	0	0	0	0	14	0	0	0	0	48
$Median_{past}$					0.849					0.830					0.036					7.45
SD_{past}					0.057					0.050					0.012					0.73
NISTa	*>0.592*	*>0.703*	*>0.569*	*>0.677*	0.877	0.592	0.703	0.569	0.677	0.848						11.4	19.3	11.5	18.3	7.44
NISTb	*>0.596*	*>0.712*	*>0.526*	*>0.643*	*>0.799*	0.596	0.712	0.526	0.643	0.799						12.6	19.4	11.6	19.3	7.74
N_{NIST}	5	5	5	5	5	5	5	5	5	5						5	5	5	5	5
$Mean_{NIST}$	0.594	0.708	0.547	0.660	0.838	0.594	0.708	0.547	0.660	0.824						12.0	19.4	11.5	18.8	7.59
S_{rep}	0.027	0.019	0.021	0.023	0.021	0.027	0.019	0.021	0.023	0.016						0.4	0.3	0.3	0.4	0.12
S_{het}	0.021	0.024	0.030	0.006	0.004	0.021	0.024	0.030	0.006	0.005						0.5	0.4	0.3	0.5	0.10
S_{anl}	0.003	0.007	0.030	0.024	0.055	0.003	0.007	0.030	0.024	0.034						0.9	0.1	0.0	0.7	0.22
S_{NIST}	0.035	0.032	0.048	0.034	0.059	0.035	0.032	0.048	0.034	0.038						1.0	0.5	0.4	0.9	0.27
NAV	0.587	0.699	0.553	0.651	0.852	0.597	0.701	0.544	0.651	0.838	0.118	0.114	0.121	0.108	0.041	11.9	19.0	11.3	18.3	7.57
NAU	0.056	0.072	0.055	0.056	0.070	0.048	0.056	0.048	0.052	0.070	0.032	0.037	0.033	0.030	0.019	1.1	1.9	1.0	2.1	0.80

Round Robin XLVII Laboratory Results

All Results are in µg/mL

	γ-Tocopherol					δ-Tocopherol					Total β-Carotene					trans-β-Carotene				
Lab	259	260	261	262	263	259	260	261	262	263	259	260	261	262	263	259	260	261	262	263
FSV-BA	2.35	1.14	2.12	1.01	3.80						0.231	0.306	0.213	0.289	0.182	0.212	0.284	0.195	0.267	0.164
FSV-BB	1.99	0.94	1.93	0.90	3.57						0.211	0.297	0.196	0.286	0.158	0.202	0.284	0.187	0.272	0.150
FSV-BD																				
FSV-BE	2.70	1.20	2.40	1.10	4.00						0.266	0.356	0.247	0.347	0.193					
FSV-BF	2.15	0.99	2.10	0.94	3.95						0.246	0.312	0.237	0.311	0.176					
FSV-BG	2.34	1.04	2.02	1.00	3.63						0.267	0.334	0.238	0.305	0.177					
FSV-BH	2.34	1.12	2.16	1.02	4.00						0.232	0.333	0.240	0.317	0.175	0.222	0.319	0.228	0.303	0.167
FSV-BI	2.21	1.06	2.02	1.04	3.79						0.252	0.316	0.223	0.308	0.168					
FSV-BJ	2.63	1.14	2.45	1.04	3.83						0.281	0.354	0.245	0.334	0.184					
FSV-BK																				
FSV-BL																				
FSV-BM																				
FSV-BN	1.84	0.74	1.75	0.86	3.11	0.380	0.362	0.389	0.377	0.348	0.265	0.315	0.228	0.334	0.208	0.236	0.286	0.208	0.304	0.184
FSV-BO											*0.186*	*0.258*	*0.192*	*0.248*	*0.148*	0.171	0.237	0.179	0.238	0.137
FSV-BP											0.233	0.328	0.241	0.329	0.193					
FSV-BQ																				
FSV-BR																				
FSV-BU	2.64	1.19	2.80	1.11	4.15						0.237	0.294	0.231	0.293	0.162					
FSV-BV	2.40	1.12	2.22	1.02	4.17						0.228	0.297	0.196	0.276	0.160					
FSV-BW	2.45	1.18	2.39	1.14	4.21						0.210	0.280	0.220	0.280	0.170					
FSV-BX	2.41	1.06	2.26	0.99	4.14						*>0.237*	*>0.324*	*>0.220*	*>0.274*	*>0.150*	0.237	0.324	0.220	0.274	0.150
FSV-BZ	1.90	0.80	2.20	0.90	3.90						*>0.190*	*>0.327*	*>0.235*	*>0.290*	*>0.162*	0.190	0.327	0.235	0.290	0.162
FSV-CB											0.241	0.284	0.231	0.296	0.189					
FSV-CC																				
FSV-CD	2.24	1.17	1.90	1.08	3.23						0.192	0.291	0.190	0.285	0.142					
FSV-CE											0.270	0.350	0.250	0.340	0.120					
FSV-CF																				
FSV-CG	2.43	1.29	2.29	1.21	3.88	0.198	0.198	0.147	0.179	0.156	0.282	0.387	0.268	0.360	0.196	0.261	0.359	0.248	0.334	0.180
FSV-CH	1.90	0.90	1.82	0.82	3.05						0.261	0.334	0.256	0.311	0.174					
FSV-CI	2.70	1.90	3.10	1.80	4.30															
FSV-CL	2.54	1.68	2.62	1.41	4.52						0.292	0.343	0.296	0.387	0.229					
FSV-CN	2.28	1.09	2.21	1.01	4.16						*>0.239*	*>0.321*	*>0.234*	*>0.302*	*>0.175*	0.239	0.321	0.234	0.302	0.175
FSV-CR																				
FSV-CS	2.02	1.02	1.93	0.97	3.62						0.215	0.317	0.217	0.310	0.177	0.200	0.292	0.198	0.280	0.153
FSV-CU																				
FSV-CV	2.52	1.27	2.62	0.91	3.20						0.208	0.329	0.275	0.270	0.215					
FSV-CW	2.46	1.14	2.48	1.22	4.02	0.080	0.040	0.110	0.070	0.120	0.216	0.286	0.216	0.298	0.144	0.202	0.267	0.200	0.279	0.132
FSV-CX											*>0.202*	*>0.279*	*>0.206*	*>0.260*	*>0.150*	0.202	0.279	0.206	0.260	0.150
FSV-DA	2.41	1.23	2.34	1.11	4.04	0.115	0.077	0.150	0.052	0.105	0.304	0.379	0.268	0.299	0.146	0.265	0.350	0.241	0.256	0.122
FSV-DB											0.180	0.261	0.194	0.281	0.164					
FSV-DF																				
FSV-DI	2.20	1.01	2.04	0.93	3.80	0.112	0.070	0.102	0.063	0.141	*>0.262*	*>0.346*	*>0.216*	*>0.286*	*>0.167*	0.262	0.346	0.216	0.286	0.167
FSV-DK	2.11	0.92	1.91	0.79	3.17						0.331	0.426	0.271	0.406	0.216					
FSV-DP																				
FSV-DQ	2.10	1.40	1.74	0.99	2.99						0.132	0.190	0.095	0.182	0.110					
FSV-DR											0.275	0.358	0.249	0.339	0.127					
FSV-DU											*>0.090*	*>0.160*	*>0.110*	*>0.170*	*>0.090*	0.090	0.160	0.110	0.170	0.090
FSV-EM											0.320	0.410	0.280	0.410	0.210					
FSV-EQ											*>0.267*	*>0.300*	*>0.256*	*>0.338*	*>0.186*	0.267	0.300	0.256	0.338	0.186
FSV-ES											*>0.254*	*>0.344*	*>0.200*	*>0.298*	*>0.174*	0.254	0.344	0.200	0.298	0.174
FSV-FH																				
FSV-FT																				
FSV-FW																				
N	27	27	27	27	27	5	5	5	5	5	29	29	29	29	29	17	17	17	17	17
Min	1.84	0.74	1.74	0.79	2.99	0.080	0.040	0.102	0.052	0.105	0.132	0.190	0.095	0.182	0.110	0.090	0.160	0.110	0.170	0.090
Median	2.34	1.12	2.20	1.01	3.88	0.115	0.077	0.147	0.070	0.141	0.241	0.317	0.237	0.308	0.175	0.222	0.300	0.208	0.280	0.162
Max	2.70	1.90	3.10	1.80	4.52	0.380	0.362	0.389	0.377	0.348	0.331	0.426	0.296	0.410	0.229	0.267	0.359	0.256	0.338	0.186
eSD	0.27	0.15	0.30	0.13	0.39	0.052	0.055	0.055	0.027	0.031	0.043	0.039	0.030	0.039	0.027	0.033	0.035	0.030	0.029	0.018
eCV	11	13	13	13	10	45	71	37	38	22	18	12	13	13	15	15	12	14	10	11
N_{past}	0	0	0	0	26	0	0	0	0	8	0	0	0	0	33	0	0	0	0	15
$Median_{past}$					3.83					0.130					0.173					0.165
SD_{past}					0.34					0.067					0.022					0.017
NISTa	2.31	1.19	2.12	1.00	4.00	*nq*	*nq*	*nq*	*nq*	0.138	0.211	0.336	0.265	0.353	0.172					
NISTb	2.36	1.21	2.13	1.10	4.05	0.142	*nq*	0.101	*nq*	0.143	0.217	0.278	0.230	0.322	0.159	0.204	0.257	0.192	0.271	0.134
N_{NIST}	5	5	5	5	5	2		2		5	5	5	5	5	5	2	2	2	2	2
$Mean_{NIST}$	2.34	1.20	2.13	1.05	4.03	0.146		0.101		0.140	0.217	0.307	0.248	0.338	0.165	0.204	0.257	0.192	0.271	0.134
S_{rep}	0.10	0.09	0.06	0.04	0.16	0.001		0.000		0.027	0.007	0.020	0.024	0.018	0.018	0.019	0.013	0.003	0.010	0.016
S_{het}	0.09	0.06	0.13	0.07	0.11	0.020		0.016		0.035	0.015	0.006	0.013	0.010	0.009	0.022	0.012	0.016	0.013	0.004
S_{anl}	0.04	0.02	0.00	0.07	0.03					0.003	0.007	0.041	0.025	0.022	0.010					
S_{NIST}	0.14	0.11	0.14	0.10	0.19	0.020		0.016		0.044	0.018	0.046	0.037	0.031	0.022	0.029	0.018	0.017	0.016	0.016
NAV	2.34	1.16	2.15	1.03	3.96	0.131	0.077	0.124	0.070	0.141	0.229	0.311	0.239	0.323	0.170	0.213	0.279	0.200	0.276	0.148
NAU	0.24	0.14	0.32	0.12	0.38	0.068	0.055	0.044	0.027	0.044	0.043	0.046	0.039	0.049	0.028	0.041	0.044	0.029	0.031	0.027

Round Robin XLVII Laboratory Results

All Results are in µg/mL

	Total cis-β-Carotene					Total α-Carotene					trans-α-Carotene					Total Lycopene				
Lab	259	260	261	262	263	259	260	261	262	263	259	260	261	262	263	259	260	261	262	263
FSV-BA	0.018	0.022	0.017	0.022	0.017	0.039	0.068	0.036	0.065	0.018										
FSV-BB	0.009	0.013	0.009	0.014	0.008	0.032	0.062	0.033	0.063	0.011						0.419	0.47	0.488	0.58	0.406
FSV-BD																				
FSV-BE																				
FSV-BF						0.042	0.075	0.043	0.072	0.019						0.422	0.43	0.425	0.43	0.384
FSV-BG						0.046	0.069	0.038	0.060	0.031						0.366	0.36	0.332	0.34	0.290
FSV-BH	0.010	0.014	0.012	0.015	0.008	0.040	0.075	0.039	0.071	0.012						0.375	0.41	0.385	0.40	0.325
FSV-BI						0.039	0.072	0.039	0.067	0.016						0.340	0.33	0.297	0.32	0.240
FSV-BJ						0.052	0.092	0.042	0.086	*nq*						0.450	0.44	0.408	0.43	0.354
FSV-BK																				
FSV-BL																				
FSV-BM																				
FSV-BN	*0.029*	*0.029*	*0.020*	*0.030*	*0.024*	0.040	0.068	0.037	0.067	0.020						0.388	0.36	0.351	0.41	0.337
FSV-BO	0.015	0.020	0.013	0.010	0.011	0.053	0.082	0.049	0.088	0.028						0.357	0.38	0.370	0.38	0.298
FSV-BP						0.046	0.090	0.049	0.087	0.021						0.366	0.50	0.398	0.47	0.338
FSV-BQ																				
FSV-BR																				
FSV-BU						0.042	0.070	0.042	0.065	0.017						0.406	0.40	0.380	0.37	0.280
FSV-BV						0.037	0.069	0.031	0.063	0.009						0.405	0.41	0.368	0.38	0.305
FSV-BW						0.038	0.068	0.038	0.068	0.014						0.330	0.35	0.340	0.34	0.270
FSV-BX						0.051	0.087	0.049	0.071	0.019										
FSV-BZ						0.040	0.086	0.057	0.089	0.020										
FSV-CB						0.032	0.052	0.033	0.058	0.011						0.342	0.30	0.332	0.32	0.290
FSV-CC																				
FSV-CD						0.045	0.091	0.048	0.087	0.016						0.344	0.38	0.309	0.39	0.269
FSV-CE																				
FSV-CF																				
FSV-CG	0.021	0.028	0.020	0.026	0.016	0.050	0.092	0.048	0.083	0.018						0.435	0.46	0.412	0.43	0.326
FSV-CH						0.039	0.064	0.038	0.053	0.013						0.363	0.37	0.364	0.34	0.273
FSV-CI																				
FSV-CL						0.057	0.093	0.058	0.094	0.021						0.637	0.65	0.656	0.64	0.553
FSV-CN						0.050	0.080	0.049	0.076	0.027						0.444	0.46	0.441	0.42	0.359
FSV-CR																				
FSV-CS	*0.015*	*0.025*	*0.019*	*0.030*	*0.024*	0.048	0.095	0.044	0.090	0.017										
FSV-CU																				
FSV-CV						0.039	0.069	0.048	0.058	*nq*						0.337	0.34	0.474	0.28	0.312
FSV-CW	*0.014*	*0.019*	*0.016*	*0.019*	*0.012*	*>0.032*	*>0.056*	*>0.033*	*>0.058*	*>0.012*	0.032	0.056	0.033	0.058	0.012	0.402	0.42	0.405	0.42	0.309
FSV-CX						0.036	0.071	0.038	0.066	0.014										
FSV-DA	0.039	0.029	0.027	0.043	0.024	0.044	0.079	0.042	0.057	0.013						0.448	0.48	0.438	0.36	0.237
FSV-DB																0.381	0.42	0.416	0.41	0.353
FSV-DF																				
FSV-DI																				
FSV-DK						0.045	0.074	0.042	0.074	0.019										
FSV-DP																				
FSV-DQ						*nq*	0.048	0.022	0.042	0.018						0.420	0.43	0.300	0.31	0.327
FSV-DR																				
FSV-DU																				
FSV-EM						*0.030*	0.050	0.030	0.050	0.010						0.340	0.35	0.310	0.37	0.260
FSV-EQ						0.046	0.070	0.043	0.080	0.015						0.463	0.40	0.437	0.46	0.309
FSV-ES						*>0.047*	*>0.084*	*>0.037*	*>0.074*	*>0.017*	0.047	0.084	0.037	0.074	0.017	0.390	0.40	0.310	0.39	0.302
FSV-FH																				
FSV-FT																				
FSV-FW																				
N	9	9	9	9	9	28	29	29	29	27	2	2	2	2	2	26	26	26	26	26
Min	0.009	0.013	0.009	0.010	0.008	0.030	0.048	0.022	0.042	0.009	0.032	0.056	0.033	0.058	0.012	0.330	0.30	0.297	0.28	0.237
Median	0.015	0.022	0.017	0.022	0.016	0.042	0.072	0.042	0.068	0.017	0.039	0.070	0.035	0.066	0.015	0.389	0.41	0.383	0.39	0.309
Max	0.039	0.029	0.027	0.043	0.024	0.057	0.095	0.058	0.094	0.031	0.047	0.084	0.037	0.074	0.017	0.637	0.65	0.656	0.64	0.553
eSD	0.008	0.009	0.004	0.012	0.011	0.006	0.011	0.009	0.015	0.004	0.011	0.021	0.003	0.012	0.004	0.048	0.06	0.069	0.06	0.043
eCV	50	40	22	54	70	14	16	21	22	26	28	29	7	18	26	12	14	18	15	14
N_{past}	0	0	0	0	9	0	0	0	0	28	0	0	0	0	0	0	0	0	0	27
$Median_{past}$					0.014					0.016										0.313
SD_{past}					0.004					0.004										0.061
NISTa						*nq*	*>0.081*	*>0.042*	*>0.090*	*nq*	*nq*	0.081	0.042	0.090	*nq*					
NISTb	*0.013*	*0.021*	*0.038*	*0.051*	*0.024*	0.038	0.064	0.046	0.070	0.013	0.029	0.048	0.033	0.054	0.012	0.452	0.54	0.458	0.55	0.418
N_{NIST}						2	5	5	5	2	2	5	5	5	2	2	2	2	2	2
$Mean_{NIST}$						0.039	0.072	0.044	0.080	0.013	0.031	0.065	0.037	0.073	0.011	0.452	0.54	0.458	0.55	0.404
S_{rep}						0.003	0.004	0.008	0.010	0.000	0.004	0.005	0.004	0.007	0.005	0.032	0.08	0.054	0.05	0.030
S_{het}						0.004	0.004	0.004	0.009	0.003	0.008	0.009	0.002	0.008	0.002	0.045	0.05	0.025	0.01	0.057
S_{anl}							0.012	0.003	0.015			0.023	0.006	0.025						
S_{NIST}						0.005	0.013	0.009	0.020	0.003	0.009	0.025	0.007	0.027	0.005	0.055	0.09	0.059	0.05	0.065
NAV	0.014	0.022	0.028	0.036	0.018	0.041	0.073	0.043	0.076	0.015						0.424	0.47	0.420	0.47	0.357
NAU	0.008	0.009	0.004	0.012	0.009	0.014	0.022	0.013	0.022	0.007						0.094	0.13	0.099	0.14	0.097

Round Robin XLVII Laboratory Results

All Results are in µg/mL

	trans-Lycopene					Total β-Cryptoxanthin					Total α-Cryptoxanthin					Total Lutein				
Lab	259	260	261	262	263	259	260	261	262	263	259	260	261	262	263	259	260	261	262	263
FSV-BA	0.239	0.292	0.219	0.271	0.228	0.099	0.087	0.092	0.083	0.107										
FSV-BB	0.177	0.236	0.169	0.216	0.161	0.099	0.089	0.092	0.085	0.089	0.036	0.046	0.042	0.035	0.024	0.107	0.205	0.094	0.197	0.053
FSV-BD																				
FSV-BE																				
FSV-BF						0.080	0.069	0.083	0.065	0.077										
FSV-BG	0.183	0.205	0.168	0.185	0.159	0.082	0.082	0.078	0.079	0.077						0.135	0.233	0.124	0.212	0.053
FSV-BH						0.139	0.115	0.138	0.111	0.105						0.082	0.163	0.078	0.151	0.038
FSV-BI						0.101	0.085	0.088	0.082	0.089						0.103	0.229	0.100	0.210	0.055
FSV-BJ						0.089	0.078	0.083	0.073	0.082						0.129	0.200	0.121	0.179	0.059
FSV-BK																				
FSV-BL																				
FSV-BM																				
FSV-BN	0.170	0.179	0.156	0.192	0.162	0.095	0.082	0.091	0.089	0.089	0.038	0.038	0.038	0.041	0.028	0.086	0.169	0.098	0.179	0.058
FSV-BO						0.067	0.059	0.063	0.050	0.061						0.136	0.245	0.114	0.219	0.049
FSV-BP						0.059	0.058	0.059	0.058	0.053										
FSV-BQ																				
FSV-BR																				
FSV-BU						0.071	0.056	0.066	0.055	0.063										
FSV-BV						0.060	0.050	0.055	0.046	0.055										
FSV-BW																				
FSV-BX	0.150	0.195	0.140	0.172	0.119	0.066	0.060	0.059	0.048	0.051						0.120	0.216	0.109	0.183	0.050
FSV-BZ																0.073	0.089	0.073	0.090	0.061
FSV-CB						0.049	0.045	0.049	0.044	0.049						-	-	-	-	0.045
FSV-CC																				
FSV-CD						0.144	0.137	0.137	0.135	0.125										
FSV-CE																				
FSV-CF																				
FSV-CG	0.195	0.250	0.185	0.229	0.166	0.086	0.075	0.079	0.070	0.064						0.134	0.228	0.128	0.216	0.085
FSV-CH																				
FSV-CI																				
FSV-CL						0.083	0.079	0.085	0.070	0.082	0.035	0.038	0.034	0.032	0.022					
FSV-CN						0.074	0.064	0.070	0.061	0.067										
FSV-CR																				
FSV-CS						0.081	0.073	0.073	0.065	0.046										
FSV-CU																				
FSV-CV																				
FSV-CW	0.162	0.195	0.161	0.203	0.139	0.090	0.077	0.087	0.078	0.077	0.036	0.035	0.036	0.035	0.022	*>0.085*	*>0.156*	*>0.081*	*>0.169*	*>0.059*
FSV-CX	0.160	0.214	0.158	0.193	0.144	0.085	0.079	0.082	0.073	0.081						0.075	0.173	0.069	0.159	0.038
FSV-DA	0.210	0.270	0.196	0.191	0.123	0.091	0.079	0.085	0.056	0.060	0.032	0.032	0.034	0.027	0.018	0.127	0.210	0.112	0.184	0.058
FSV-DB						0.078	0.075	0.088	0.073	0.075										
FSV-DF																				
FSV-DI	0.214	0.228	0.179	0.192	0.152											0.096	0.194	0.091	0.181	0.050
FSV-DK																				
FSV-DP																				
FSV-DQ						0.080	0.082	0.053	0.046	0.076						0.106	0.221	0.086	0.186	0.052
FSV-DR																				
FSV-DU																				
FSV-EM						0.070	0.070	0.070	0.060	0.070										
FSV-EQ						0.151	0.121	0.140	0.131	0.104										
FSV-ES	0.236	0.305	0.186	0.263	0.205	0.085	0.079	0.074	0.071	0.074						0.139	0.266	0.106	0.242	0.057
FSV-FH																				
FSV-FT																				
FSV-FW																				
N	11	11	11	11	11	27	27	27	27	27	5	5	5	5	5	15	15	15	15	16
Min	0.150	0.179	0.140	0.172	0.119	0.049	0.045	0.049	0.044	0.046	0.032	0.032	0.034	0.027	0.018	0.073	0.089	0.069	0.090	0.038
Median	0.183	0.228	0.169	0.193	0.159	0.083	0.078	0.082	0.070	0.076	0.036	0.038	0.036	0.035	0.022	0.107	0.210	0.100	0.184	0.053
Max	0.239	0.305	0.219	0.271	0.228	0.151	0.137	0.140	0.135	0.125	0.038	0.046	0.042	0.041	0.028	0.139	0.266	0.128	0.242	0.085
eSD	0.034	0.048	0.019	0.015	0.022	0.018	0.012	0.015	0.018	0.019	0.001	0.004	0.003	0.004	0.002	0.032	0.028	0.020	0.037	0.007
eCV	19	21	11	8	14	21	15	18	25	25	4	12	8	12	10	30	13	20	20	13
N_{past}	0	0	0	0	13	0	0	0	0	28	0	0	0	0	6	0	0	0	0	17
$Median_{past}$					0.157					0.074					0.022					0.056
SD_{past}					0.031					0.011					0.007					0.017
NISTa						0.054	0.094	0.074	0.078	0.070						0.101	0.251	0.114	0.248	0.055
NISTb						0.085	0.087	0.091	0.087	0.089	0.056	0.055	0.052	0.056	0.039	*>0.089*	*>0.179*	*>0.083*	*>0.169*	*>0.047*
N_{NIST}						5	5	5	5	5	2	2	2	2	2	3	3	3	3	3
$Mean_{NIST}$						0.070	0.090	0.082	0.082	0.079	0.057	0.055	0.052	0.056	0.039	0.101	0.251	0.114	0.252	0.055
S_{rep}						0.009	0.012	0.009	0.004	0.003	0.003	0.012	0.005	0.009	0.001	0.006	0.011	0.000	0.016	0.002
S_{het}						0.008	0.002	0.014	0.007	0.005	0.005	0.014	0.010	0.002	0.001	0.004	0.012	0.006	0.014	0.001
S_{anl}						0.021	0.005	0.012	0.007	0.013										
S_{NIST}						0.024	0.013	0.020	0.010	0.014	0.006	0.019	0.011	0.009	0.001	0.007	0.016	0.006	0.022	0.002
NAV	0.183	0.228	0.169	0.193	0.159	0.077	0.084	0.082	0.076	0.078	0.047	0.046	0.044	0.045	0.030	0.104	0.231	0.107	0.218	0.054
NAU	0.034	0.044	0.030	0.034	0.028	0.026	0.020	0.020	0.020	0.019	0.016	0.022	0.016	0.017	0.012	0.030	0.047	0.022	0.058	0.011

Round Robin XLVII Laboratory Results

All Results are in µg/mL

	trans-Lutein					Total Zeaxanthin					Total Lutein&Zeaxanthin					Coenzyme Q10				
Lab	259	260	261	262	263	259	260	261	262	263	259	260	261	262	263	259	260	261	262	263
FSV-BA											0.169	0.290	0.158	0.27	0.149					
FSV-BB						0.052	0.053	0.048	0.051	0.042	*0.159*	*0.258*	*0.142*	*0.25*	*0.095*					
FSV-BD																				
FSV-BE																				
FSV-BF											0.165	0.266	0.165	0.26	0.093					
FSV-BG						0.037	0.052	0.028	0.048	0.011	0.184	0.303	0.161	0.28	0.067					
FSV-BH						0.039	0.037	0.021	0.035	0.028	0.121	0.200	0.099	0.19	0.066					
FSV-BI						0.031	0.049	0.031	0.045	0.036	*0.134*	*0.278*	*0.131*	*0.26*	*0.091*					
FSV-BJ																				
FSV-BK																				
FSV-BL																				
FSV-BM																				
FSV-BN						0.028	0.043	0.030	0.045	0.046	0.106	0.199	0.118	0.21	0.090					
FSV-BO						0.026	0.037	0.021	0.037	0.021	*0.162*	*0.282*	*0.134*	*0.26*	*0.070*					
FSV-BP											0.178	0.364	0.167	0.34	0.105					
FSV-BQ																				
FSV-BR																				
FSV-BU											0.150	0.254	0.144	0.24	0.086					
FSV-BV											0.144	0.255	0.145	0.24	0.105					
FSV-BW																				
FSV-BX						0.022	0.033	0.020	0.026	0.019	0.142	0.248	0.129	0.21	0.069					
FSV-BZ																				
FSV-CB						-	-	-	-	0.028	0.148	0.266	0.127	0.24	*0.073*					
FSV-CC																				
FSV-CD											0.197	0.262	0.155	0.25	0.126					
FSV-CE																				
FSV-CF																				
FSV-CG						0.027	0.045	0.025	0.042	0.022	0.162	0.273	0.153	0.26	0.107					
FSV-CH																0.614	0.826	0.571	0.610	0.464
FSV-CI																				
FSV-CL											0.149	0.286	0.150	0.24	0.093					
FSV-CN											0.136	0.223	0.128	0.21	0.085					
FSV-CR																				
FSV-CS											0.175	0.308	0.145	0.27	0.097					
FSV-CU																				
FSV-CV											0.134	0.239	0.159	0.24	0.079	0.341	0.510	0.371	0.576	0.507
FSV-CW	0.085	0.156	0.081	0.169	0.059	0.024	0.035	0.023	0.039	0.041	*>0.109*	*>0.191*	*>0.104*	*>0.208*	*>0.100*	0.560	0.800	0.550	0.740	0.520
FSV-CX						0.022	0.043	0.023	0.038	0.023	*0.097*	*0.216*	*0.092*	*0.20*	*0.061*					
FSV-DA						0.039	0.058	0.032	0.049	0.027	0.166	0.268	0.144	0.23	0.085					
FSV-DB											0.118	0.193	0.119	0.21	0.104					
FSV-DF																				
FSV-DI																0.606	0.816	0.484	0.677	0.535
FSV-DK																				
FSV-DP																				
FSV-DQ						0.018	0.028	0.011	0.022	0.026	*0.124*	*0.249*	*0.097*	*0.21*	*0.078*					
FSV-DR																				
FSV-DU																				
FSV-EM											0.090	0.200	0.090	0.19	0.040					
FSV-EQ											0.176	0.312	0.159	0.33	0.101					
FSV-ES						0.041	0.053	0.028	0.047	0.035	*0.180*	*0.319*	*0.134*	*0.29*	*0.092*					
FSV-FH																				
FSV-FT																				
FSV-FW																				
N	1	1	1	1	1	13	13	13	13	14	26	26	26	26	26	4	4	4	4	4
Min						0.018	0.028	0.011	0.022	0.011	0.090	0.193	0.090	0.19	0.040	0.341	0.510	0.371	0.576	0.464
Median	0.085	0.156	0.081	0.169	0.059	0.028	0.043	0.025	0.042	0.027	0.150	0.264	0.143	0.24	0.091	0.583	0.808	0.517	0.644	0.514
Max						0.052	0.058	0.048	0.051	0.046	0.197	0.364	0.167	0.34	0.149	0.614	0.826	0.571	0.740	0.535
eSD						0.010	0.012	0.007	0.007	0.011	0.027	0.035	0.022	0.04	0.019	0.040	0.019	0.064	0.075	0.021
eCV						34	28	27	18	39	18	13	16	18	21	7	2	12	12	4
N_{past}	0	0	0	0	0	0	0	0	0	15	0	0	0	0	26	0	0	0	0	0
$Median_{past}$										0.034					0.095					
SD_{past}										0.008					0.017					
NISTa						0.025	0.030	0.021	0.029	0.026	*0.126*	*0.281*	*0.136*	*0.28*	*0.081*					
NISTb	0.089	0.179	0.083	0.169	0.047	0.029	0.036	0.027	0.035	0.033	*>0.119*	*>0.215*	*>0.110*	*>0.204*	*>0.080*					
N_{NIST}	2	2	2	2	2	5	5	5	5	5	5	5	5	5	5					
$Mean_{NIST}$	0.089	0.179	0.083	0.169	0.047	0.027	0.033	0.024	0.032	0.030	0.123	0.248	0.123	0.20	0.080					
S_{rep}	0.008	0.006	0.003	0.009	0.004	0.003	0.004	0.004	0.004	0.007	0.009	0.009	0.005	0.12	0.009					
S_{het}	0.008	0.005	0.000	0.003	0.001	0.002	0.002	0.003	0.003	0.002	0.007	0.009	0.005	0.05	0.001					
S_{anl}						0.004	0.004	0.004	0.005	0.005	0.006	0.047	0.018	0.01	0.002					
S_{NIST}	0.011	0.007	0.003	0.010	0.004	0.006	0.006	0.006	0.006	0.009	0.013	0.048	0.019	0.13	0.009					
NAV						0.027	0.038	0.025	0.037	0.029	0.139	0.257	0.133	0.22	0.086					
NAU						0.011	0.014	0.007	0.014	0.010	0.039	0.058	0.033	0.14	0.021					

Round Robin XLVII Laboratory Results

All Results are in µg/mL

Analytes Reported By One Laboratory

Analyte	Code	259	260	261	262	263
trans-β-Cryptoxanthin	NISTb	0.07	0.06	0.07	0.06	0.06
25-hydroxyvitamin D	FSV-BF	0.027	0.030	0.030	0.031	0.021
25-hydroxyvitamin D2	FSV-BN	0.0134	0.0083	0.0059	0.0163	*nd*
Phylloquinone (K1) x1000	FSV-DI	0.78	0.38	0.73	0.30	0.57
Phytofluene	FSV-CL	0.032	0.026	0.032	0.023	0.037
Phytoene	FSV-CL	0.120	0.140	0.133	0.130	0.075

Legend

Term	Definition
N	Number of (non-NIST) quantitative values reported for this analyte
Min	Minimum (non-NIST) quantitative value reported
$Median_{part}$	Median (non-NIST) quantitative value reported
Max	Maximum (non-NIST) quantitative value reported
eSD	Robust estimate of standard deviation for (non-NIST) results
eCV	Coefficient of Variation for (non-NIST) results: 100*eSD/Median
N_{past}	Mean of N(s) from past RR(s)
$Median_{past}$	Mean of Median(s) from past RR(s)
SD_{past}	Pooled SD from past RR(s)
N_{NIST}	Number of total vials analyzed in duplicate by NIST analysts
$Mean_{NIST}$	Mean of the NIST-analyzed vial means
S_{rep}	Within-vial pooled standard deviation
S_{het}	Among-vial pooled standard deviation
S_{anl}	Between NIST analyst standard deviation
S_{NIST}	Total standard deviation for NIST analyses: $(S_{rep}^2 + S_{het}^2 + S_{anl}^2)^{0.5}$
NAV	NIST Assigned Value = $(Median_{part} + Mean_{NIST})/2$ for analytes reported by NIST analyst(s) = $Median_{part}$ for analytes reported by ≥ 10 labs but not NIST
NAU	NIST Assigned Uncertainty: $(S^2 + S_{btw}^2)^{0.5}$ S is the maximum of (0.05*NAV, SD, S_{NIST}, eSD) and S_{btw} is the standard deviation between $Median_{part}$ and $Mean_{NIST}$. The expected long-term SD, eSD, is defined in: Duewer, et al. Anal Chem 1997;69(7):1406-1413.
nq	Detected but not quantitatively determined
>x	Concentration larger than x
italics	Not explicitly reported but calculated by NIST from reported values

Round Robin XLVII Laboratory Results

Comparability Summary

Lab	TR	aT	gT	bC	tbC	aC	TLy	TbX	TLu	TZ	L&Z
FSV-BA	1	1	1	1	1	1		1	1	1	1
FSV-BB	1	1	2	1	1	1	1	1	1	1	1
FSV-BD	1	1									
FSV-BE	2	1	1	1							1
FSV-BF	1	1	1	1		1	1	1			
FSV-BG	1	1	1	1		1	1	1			2
FSV-BH	1	1	1	1	1	1	1	3	1	2	1
FSV-BI	1	1	1	1		1	1	1	1	1	2
FSV-BJ	1	1	1	1		1	1	1	1	1	1
FSV-BK	1	1							2		
FSV-BL	1	3									
FSV-BM	1	1							1		
FSV-BN	1	1	2	1	1	1	1	1			
FSV-BO	2	2		2	2	1	1	1			1
FSV-BP	1	2		1		1	1	1			
FSV-BQ	1	1									
FSV-BR	1										1
FSV-BU	1	1	2	1		1	1	1	2	1	2
FSV-BV	3	1	1	1		1	1	2			1
FSV-BW	1	2	1	1		1	1				2
FSV-BX	1	1	1	1	1	1		2	1	1	1
FSV-BZ		2	2	1	1	1			1	1	1
FSV-CB	1	1		1		1	1	2			
FSV-CC	1	1									
FSV-CD	1	2	2	1		1	1	3	2	1	1
FSV-CE	1	1		2							
FSV-CF	1	1									
FSV-CG	4	2	1	2	2	1	1	1			2
FSV-CH	2	2	2	1		1	1				
FSV-CI	3	1	4								1
FSV-CL	1	4	3	2		2	3	1			1
FSV-CN	1	1	1	1	1	1	1	1	1	1	1
FSV-CR	3	3									
FSV-CS	1	1	1	1	1	1		1			1
FSV-CU	1	1									1
FSV-CV	2	2	2	2		1	1				
FSV-CW	2	1	1	1	1	1	1	1			
FSV-CX	2			1	1	1		1			
FSV-DA	1	1	1	2	2	1	1	1			1
FSV-DB	2	1		2			1	1			
FSV-DF	2										
FSV-DI	1	1	1	1	1				1	2	1
FSV-DK	1	1	2	2		1					2
FSV-DP	1										
FSV-DQ		2	2	3		2	1	1			
FSV-DR	1	1		2					1	1	1
FSV-DU	2	4		4	4						
FSV-EM	2	1		2		1	1	1			
FSV-EQ	2	2		1	2	1	1	3			1
FSV-ES	1	1		1	1	1	1	1	1		
FSV-FH	3	1									
FSV-FT	4	1									
FSV-FW	1								1	1	1
NISTa	1	1	1	1		1		1	1	1	1
NISTb	1	1	1	1	1	1	1	1	1	1	1
n	53	50	29	39	18	33	27	29	18	15	29

	TR	aT	g/bT	bC	tbC	aC	TLy	TbX	TLu	TZ	L&Z
% 1	68	72	62	69	72	94	96	79	83	87	79
% 2	21	20	31	26	22	6	0	10	17	13	21
% 3	8	4	3	3	0	0	4	10	0	0	0
% 4	4	4	3	3	6	0	0	0	0	0	0

Label	Definition
Lab	Participant code
TR	Total Retinol
aT	α-Tocopherol
gT	γ/β-Tocopherol
bC	Total β-Carotene
tbC	trans-β-Carotene
aC	Total α-Carotene
TLy	Total Lycopene
TbX	Total β-Cryptoxanthin
TLu	Total Lutein
TZ	Total Zeaxanthin
L&Z	Total Lutein & Zeaxanthin
n	number of participants providing quantitative data
% 1	Percent of CS = 1 (within 1 SD of medians)
% 2	Percent of CS = 2 (within 2 SD of medians)
% 3	Percent of CS = 3 (within 3 SD of medians)
% 4	Percent of CS = 4 (3 or more SD from medians)

"Comparability Score"

The Comparability Score (CS) summarizes your measurement performance for a given analyte relative to the consensus medians in this study. CS is the average distance (in units of standard deviation) of your measurement performance characteristics from the consensus performance. CS is calculated when the number of quantitative values you reported N_{you}, is at least two and at least six participants reported quantitative values for the analyte.

We define CS as follows:

$$CS = \text{MINIMUM}\left(4, \text{INTEGER}\left(1+\sqrt{C^2+AP^2}\right)\right)$$

$$C = \text{Concordance} = \frac{\sum_{i=1}^{N_{you}} \frac{You_i - Median_i}{NAU_i}}{N_{you}}$$

$$AP = \text{Apparent Precision} = \sqrt{\frac{\sum_{i=1}^{N_{you}} \left(\frac{You_i - Median_i}{NAU_i}\right)^2}{N_{you}-1}}$$

NAU = NIST Assigned Uncertainty

For further details, please see

Duewer DL, Kline MC, Sharpless KE, Brown Thomas J, Gar KT. Micronutrients Measurement Quality Assurance Program: Helping participants use interlaboratory comparisc exercise results to improve their long-term measurement performance. Anal Chem 1999;71(9):1870-8.

Appendix D. Representative "Individualized Report" for RR47

Each participant in RR47 received an "Individualized Report" reflecting their reported results. Each report included a detailed analysis of the results they reported for the following analytes:

- Total Retinol
- *trans*-Retinol
- Retinyl Palmitate
- α-Tocopherol
- γ-Tocopherol
- δ-Tocopherol
- Total β-Carotene
- *trans*-β-Carotene
- Total *cis*-β-Carotene
- Total α-Carotene
- Total Lycopene
- *trans*-Lycopene
- Total β-Cryptoxanthin
- Total α-Cryptoxanthin
- Total Lutein
- Total Zeaxanthin
- Total Lutein & Zeaxanthin

The following twelve pages are the "Individualized Report" for the analytes evaluated by participant FSV-BA.

Individualized Round Robin XLVII Report: FSV-BA

Summary

	Serum 259			Serum 260			Serum 261			Serum 262			Serum 263		
Analyte	You	NAV	n	You	NAV	n	You	NAV	n	You	NAV	n	You	NAV	n
Total Retinol	0.598	0.587	39	0.705	0.699	39	0.560	0.553	39	0.646	0.651	39	0.878	0.852	39
Retinyl Palmitate	0.11	0.12	16	0.1	0.1	16	0.1	0.1	16	0.09	0.11	16	0.05	0.04	16
α-Tocopherol	12.06	11.93	48	19.35	18.96	48	10.92	11.35	48	17.35	18.25	48	7.42	7.57	48
γ-Tocopherol	2.354	2.338	27	1.143	1.160	27	2.123	2.153	27	1.006	1.032	27	3.799	3.959	27
Total β-Carotene	0.231	0.229	29	0.306	0.311	29	0.213	0.239	29	0.289	0.323	29	0.182	0.170	29
trans-β-Carotene	0.212	0.213	17	0.284	0.279	17	0.195	0.200	17	0.267	0.276	17	0.164	0.148	17
Total cis-β-Carotene	0.018		9	0.022		9	0.017		9	0.022		9	0.017		9
Total α-Carotene	0.039	0.041	28	0.068	0.073	29	0.036	0.043	29	0.065	0.076	29	0.018	0.015	27
trans-Lycopene	0.239	0.183	11	0.292	0.228	11	0.219	0.169	11	0.271	0.193	11	0.228	0.159	11
Total β-Cryptoxanthin	0.099	0.077	27	0.087	0.084	27	0.092	0.082	27	0.083	0.076	27	0.107	0.078	27
Total Lutein&Zeaxanthin	0.169	0.139	26	0.290	0.257	26	0.158	0.133	26	0.273	0.221	26	0.149	0.086	26

D2

You : Your reported values for the listed analytes (micrograms/milliliter)
NAV : NIST Assigned Values, equal to (NIST's average-of-averages + this RR's median) / 2
n : Number of non-NIST laboratories reporting quantitative values for this analyte in this serum

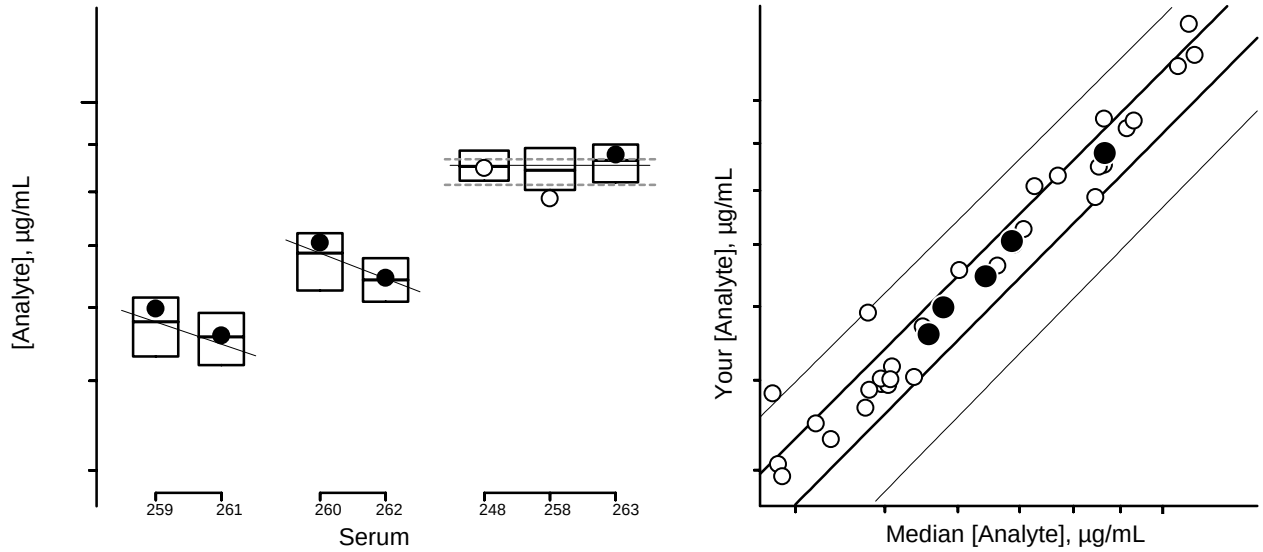
Please check our records against your records. Send corrections and/or updates to...

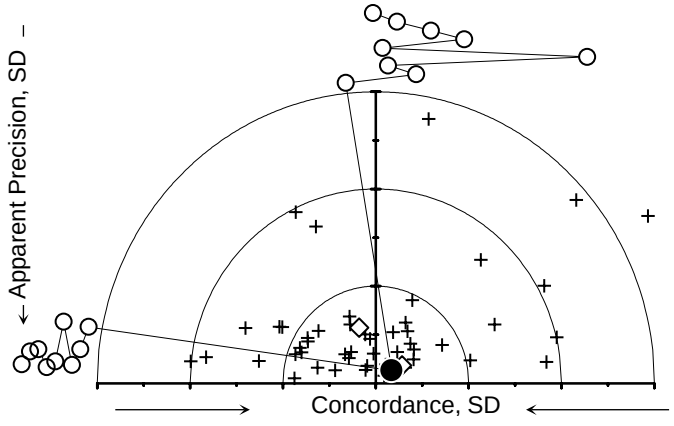
Micronutrients Measurement Quality Assurance Program
National Institute of Standards and Technology
100 Bureau Drive Stop 8392
Gaithersburg, MD 20899-8392 USA

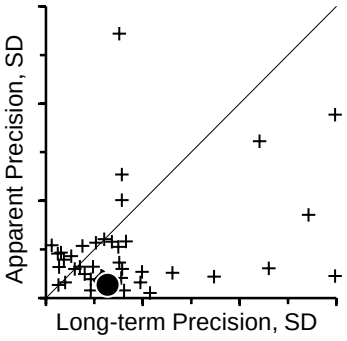
Tel: (301) 975-3935
Fax: (301) 977-0685
Email: david.duewer@nist.gov

Individualized RR XLVII Report: FSV-BA

Total Retinol







3rd Quartile (75%)
Median (50%)
1st Quartile (25%)

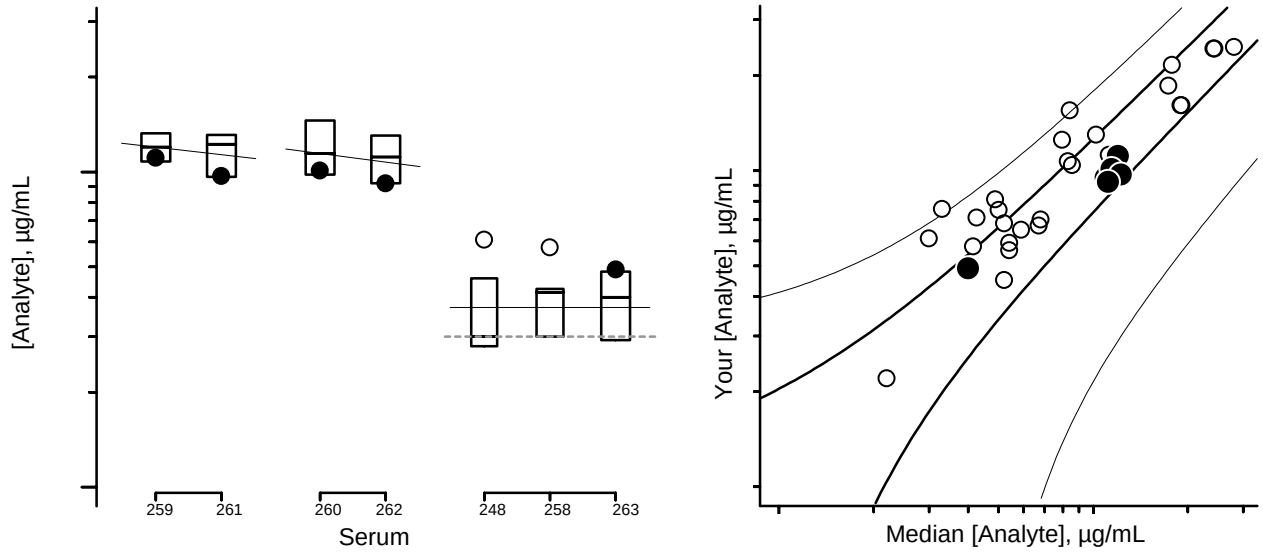
● You, this RR
○ You, past RRs
▲ You, >x, this RR
△ You, >x, past RRs
◇ NIST, this RR
+ Others, this RR
SRM 968c 95% confidence limits

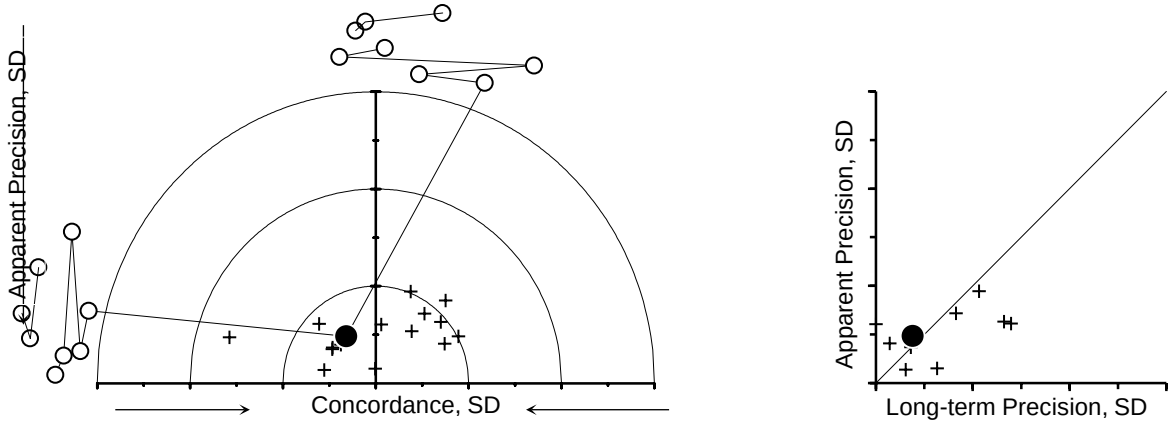
For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem May 1, 1999.

Serum	History	Comments
#259		Fresh frozen
#260		Fresh frozen
#261		Lyophilized, same pool as #259
#262		Lyophilized, same pool as #260
#263	#248 in RR44 (9/98), #258 in RR46 (9/99)	SRM 968c, Level I

Individualized RR XLVII Report: FSV-BA

Retinyl Palmitate





3rd Quartile (75%)
Median (50%)
1st Quartile (25%)

● You, this RR
○ You, past RRs
▲ You, >x, this RR
△ You, >x, past RRs
◇ NIST, this RR
+ Others, this RR

SRM 968c
95% confidence limits

For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem May 1, 1999.

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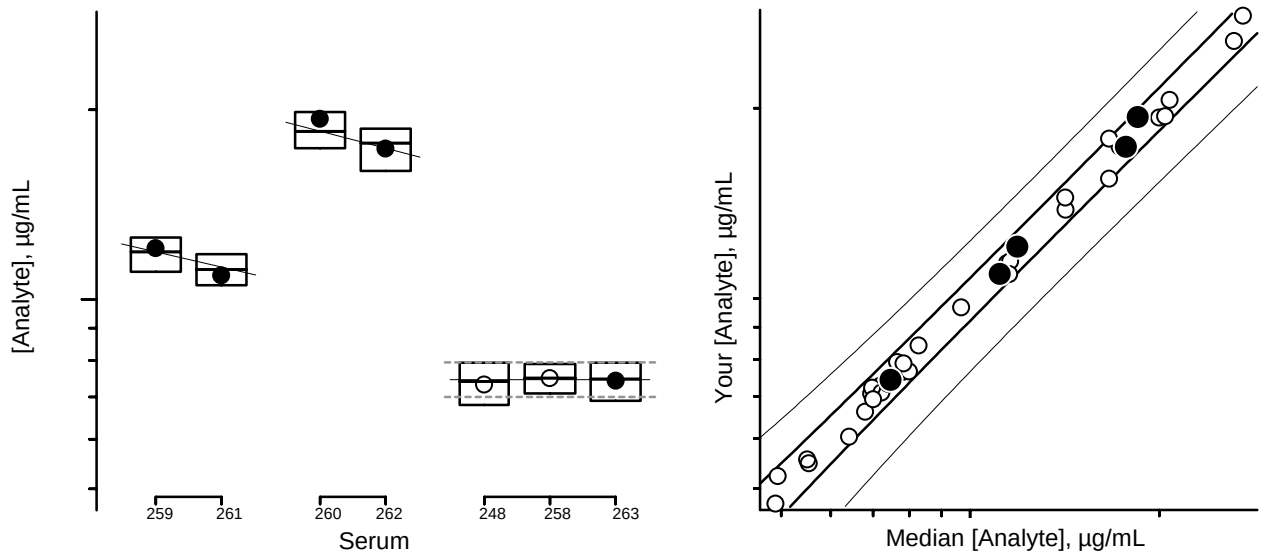
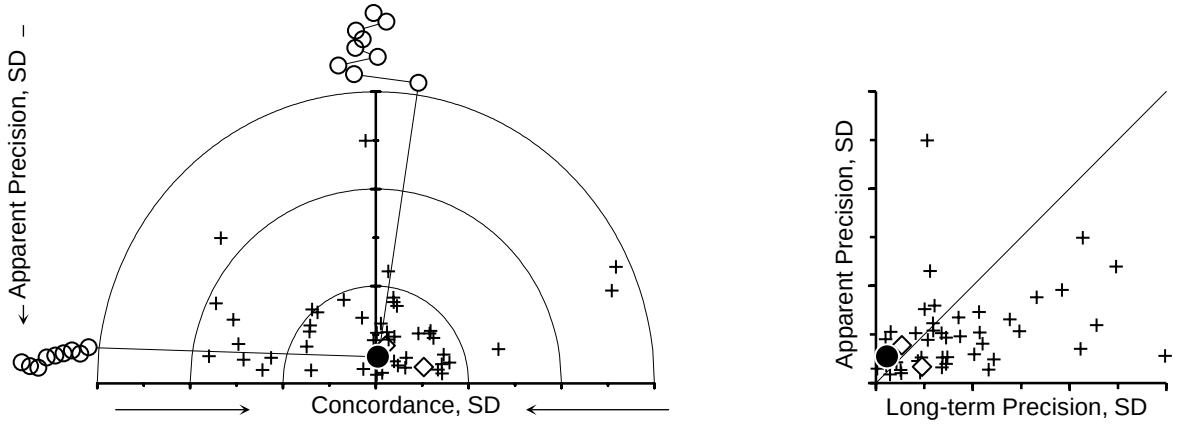
#263	#248 in RR44 (9/98), #258 in RR46 (9/99)	SRM 968c, Level I

Individualized RR XLVII Report: FSV-BA

α-Tocopherol





3rd Quartile (75%)
Median (50%)
1st Quartile (25%)

● You, this RR
○ You, past RRs
▲ You, >x, this RR
△ You, >x, past RRs
◇ NIST, this RR
+ Others, this RR
SRM 968c 95% confidence limits

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Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem May 1, 1999.

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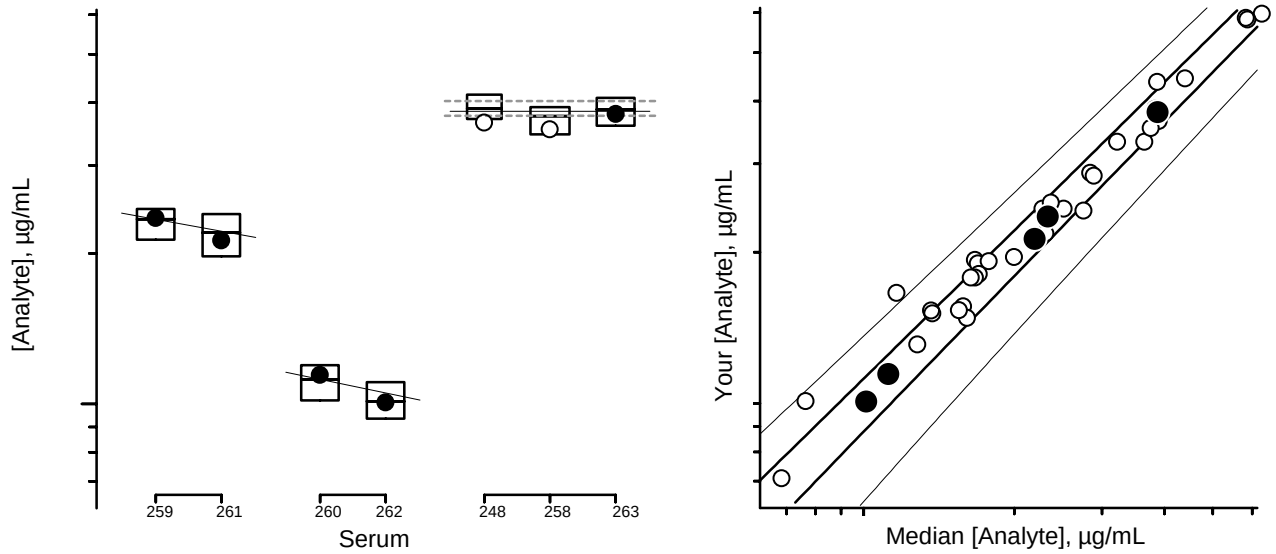
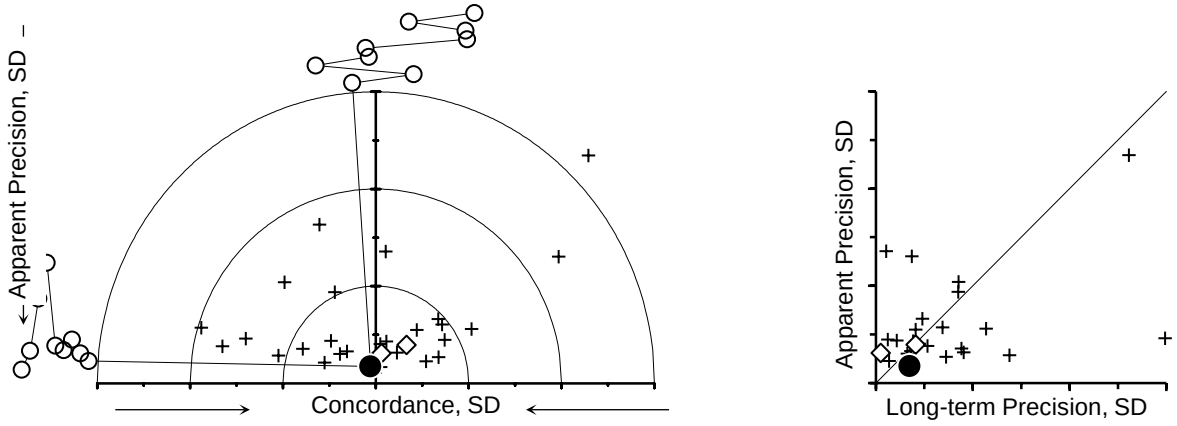
#263	#248 in RR44 (9/98), #258 in RR46 (9/99)	SRM 968c, Level I

Individualized RR XLVII Report: FSV-BA

γ-Tocopherol





3rd Quartile (75%)
Median (50%)
1st Quartile (25%)

● You, this RR
○ You, past RRs
▲ You, >x, this RR
△ You, >x, past RRs
◇ NIST, this RR
+ Others, this RR

SRM 968c
95% confidence limits

For details of the construction and interpretation of these plots, see:
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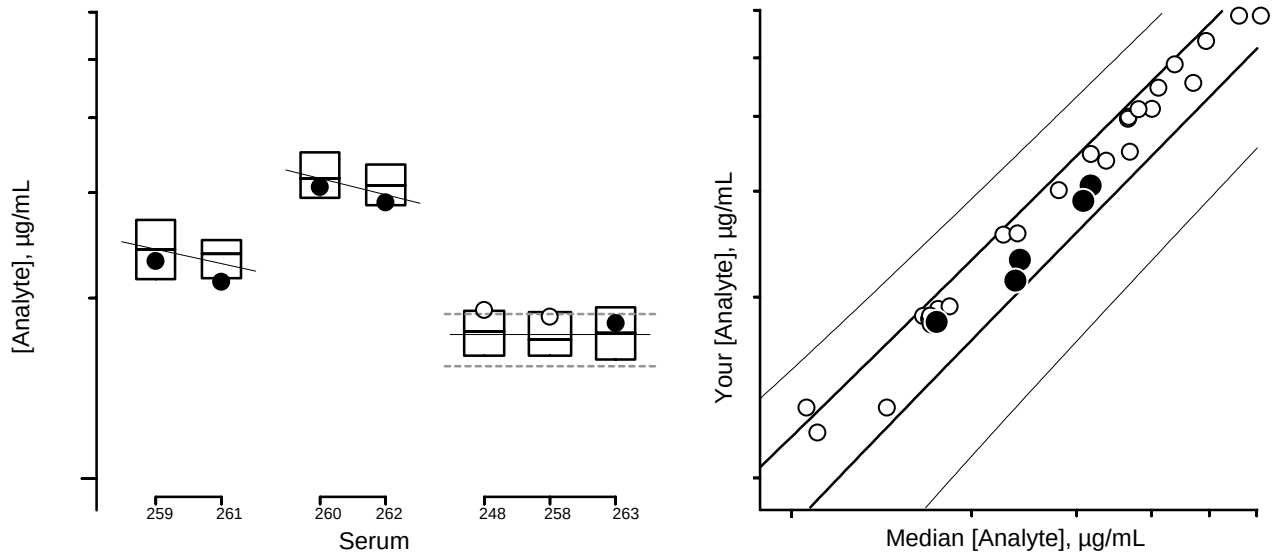
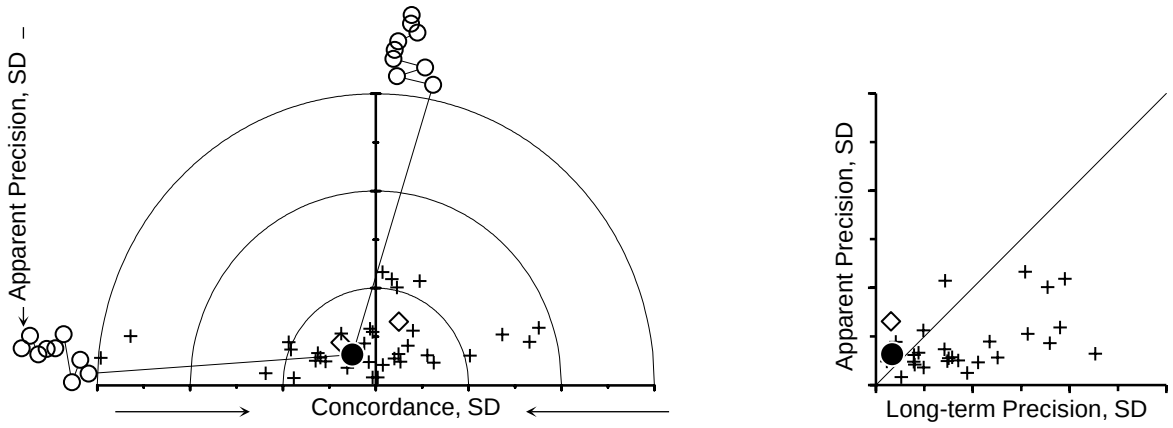
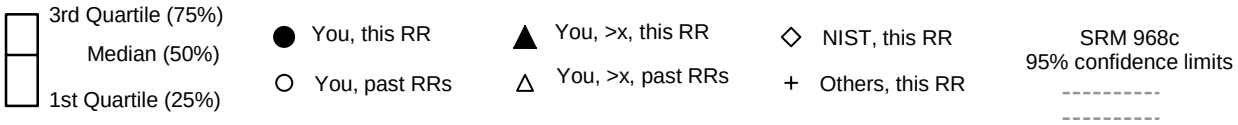
#263	#248 in RR44 (9/98), #258 in RR46 (9/99)	SRM 968c, Level I

Individualized RR XLVII Report: FSV-BA

Total β-Carotene







For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem May 1, 1999.

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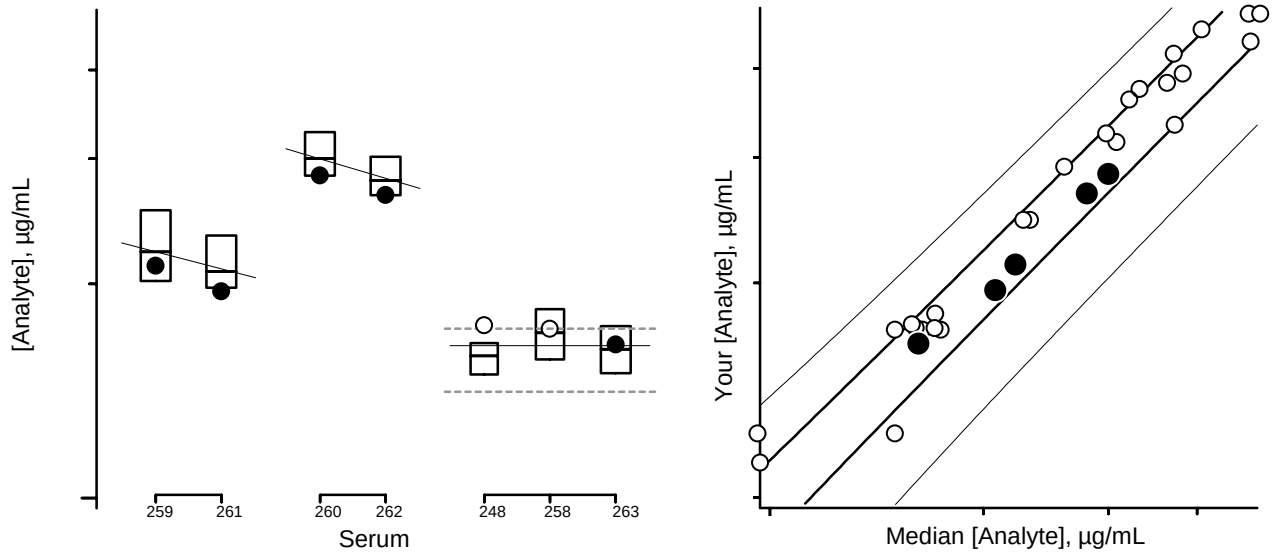
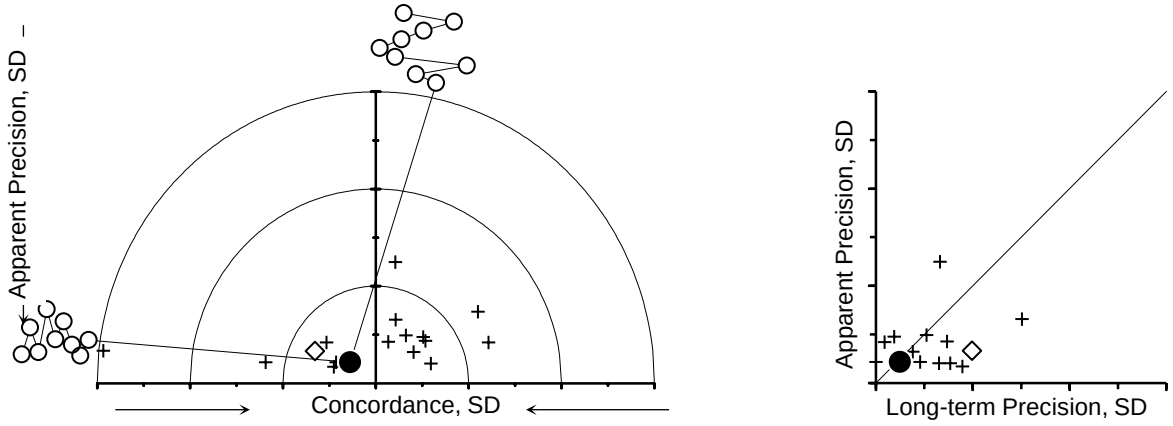
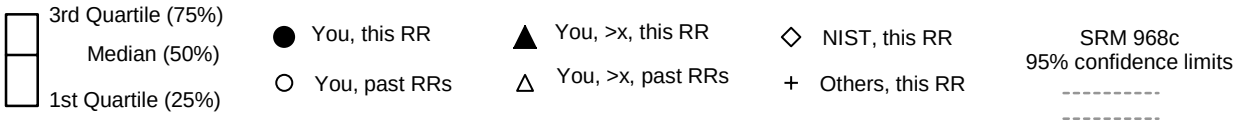
#262		Lyophilized, same pool as #260
#263	#248 in RR44 (9/98), #258 in RR46 (9/99)	SRM 968c, Level I

Individualized RR XLVII Report: FSV-BA

trans-β-Carotene







For details of the construction and interpretation of these plots, see:
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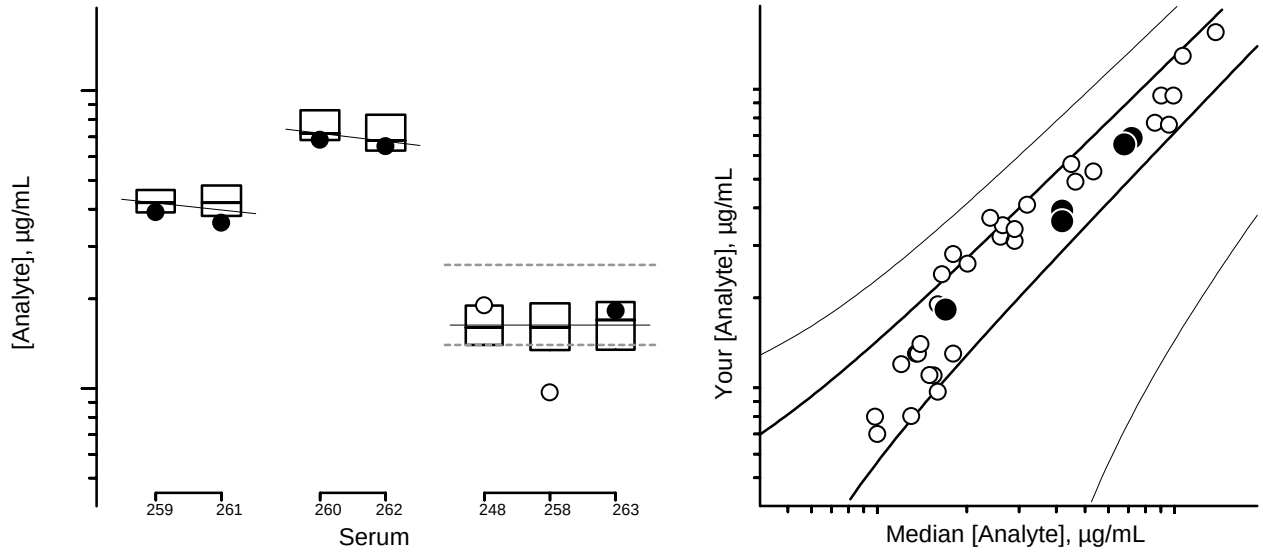
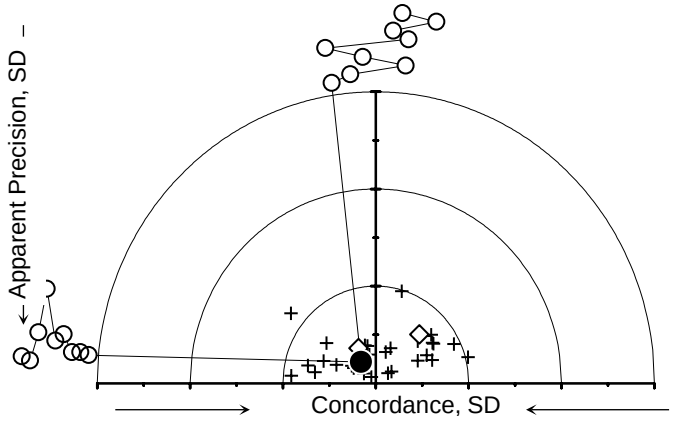
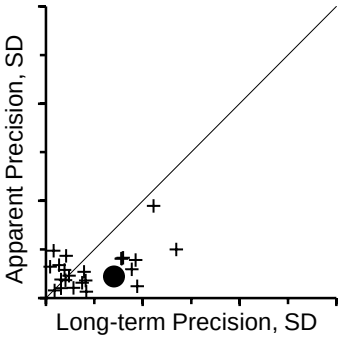
#262		Lyophilized, same pool as #260
#263	#248 in RR44 (9/98), #258 in RR46 (9/99)	SRM 968c, Level I

Individualized RR XLVII Report: FSV-BA

Total α-Carotene







3rd Quartile (75%)
Median (50%)
1st Quartile (25%)

● You, this RR
○ You, past RRs
▲ You, >x, this RR
△ You, >x, past RRs
◇ NIST, this RR
+ Others, this RR

SRM 968c
95% confidence limits

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Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem May 1, 1999.

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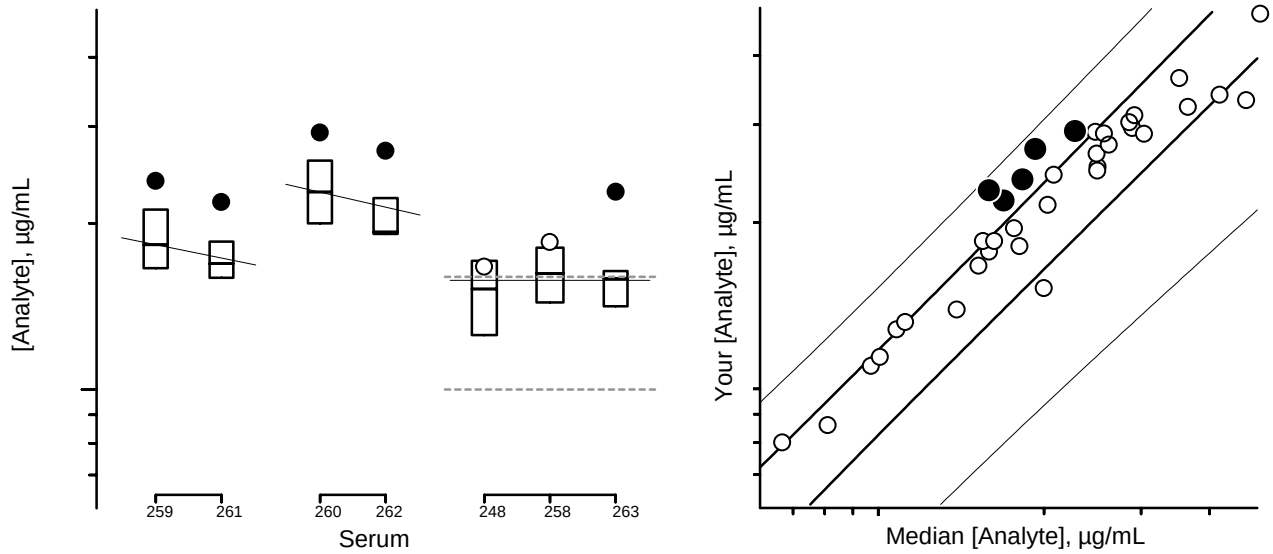
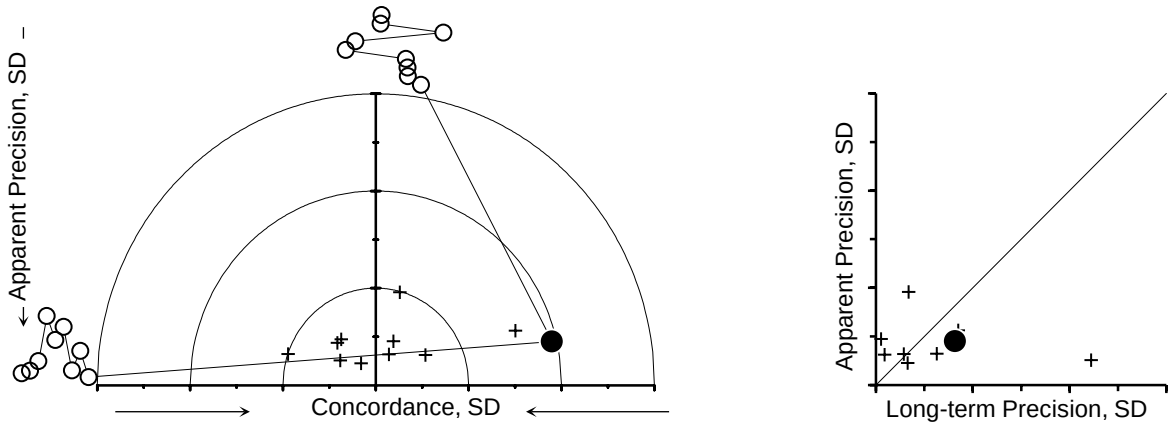
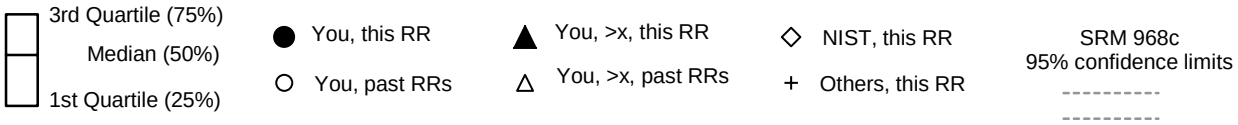
Individualized RR XLVII Report: FSV-BA

trans-Lycopene







For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem May 1, 1999.

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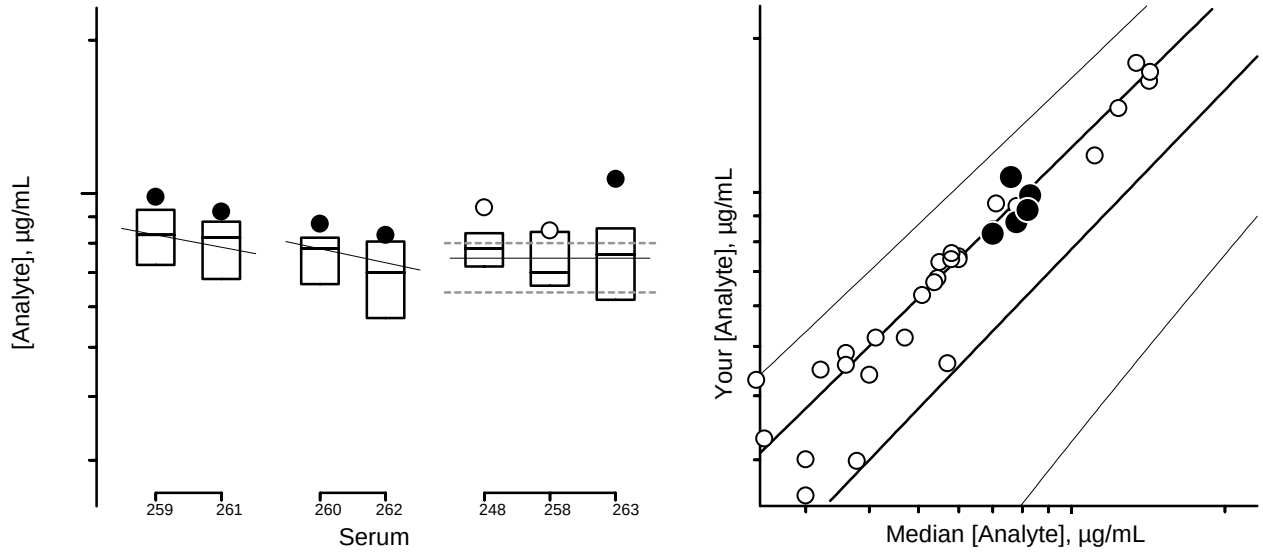
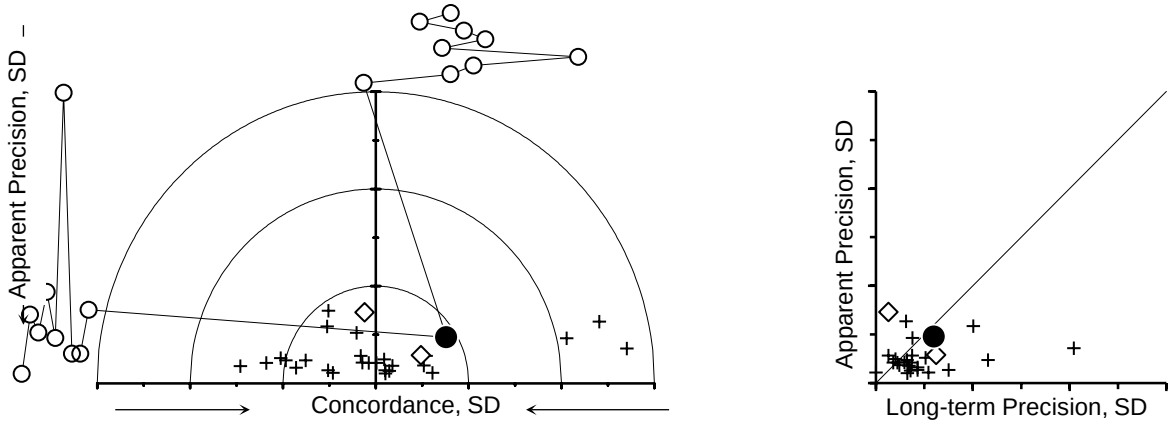
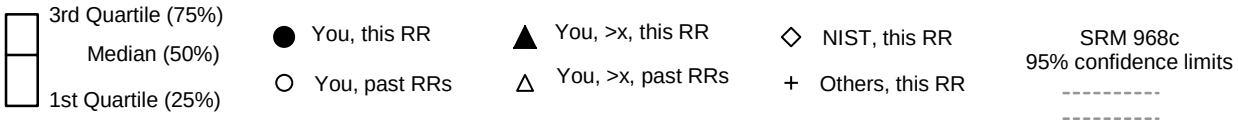
Individualized RR XLVII Report: FSV-BA

Total β-Cryptoxanthin





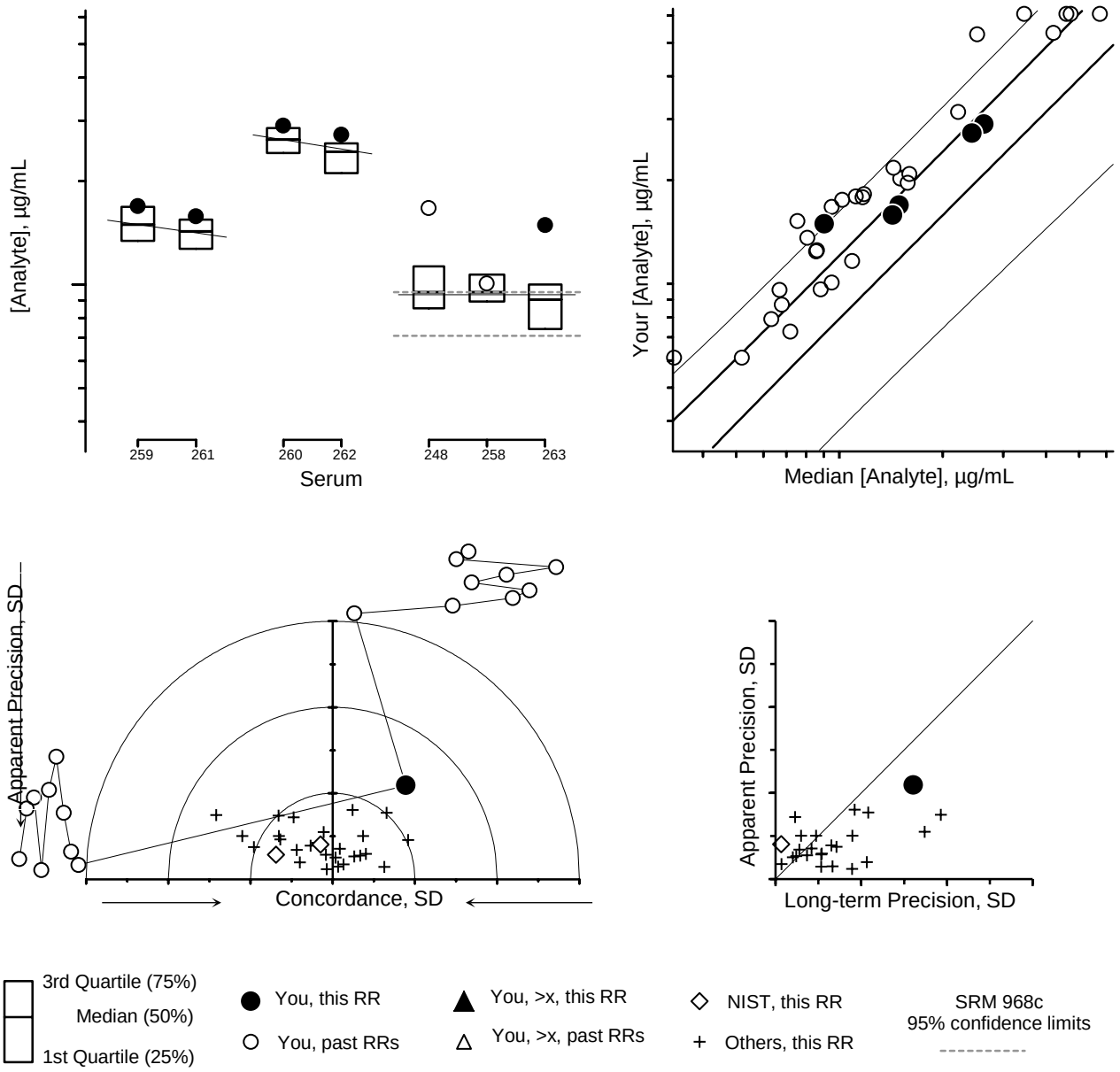


For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem May 1, 1999.

Serum	History	Comments
#259		Fresh frozen
#260		Fresh frozen
#261		Lyophilized, same pool as #259
#262		Lyophilized, same pool as #260
#263	#248 in RR44 (9/98), #258 in RR46 (9/99)	SRM 968c, Level I

Individualized RR XLVII Report: FSV-BA

Total Lutein&Zeaxanthin



For details of the construction and interpretation of these plots, see:
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Serum	History	Comments
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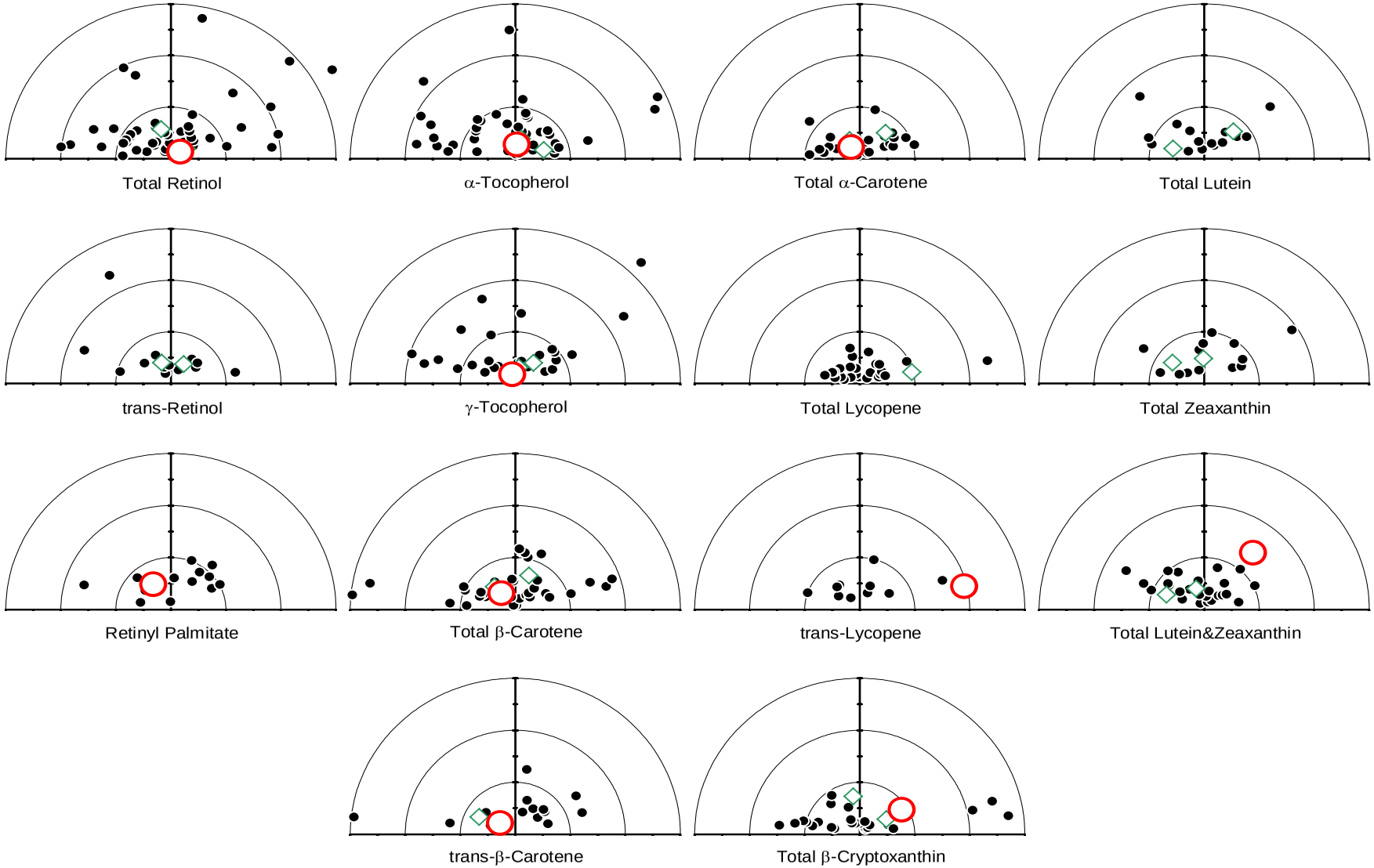
#262		Lyophilized, same pool as #260
#263	#248 in RR44 (9/98), #258 in RR46 (9/99)	SRM 968c, Level I

Individualized Round Robin XLVII Report: FSV-BA

Graphical Comparability Summary



D13

Appendix E. Shipping Package Inserts for RR48

The following three items were included in each package shipped to an RR48 participant:

- Cover letter
- Datasheet
- Packing List and Shipment Receipt Confirmation Form

The cover letter and datasheet were enclosed in a sealed waterproof bag along with the samples themselves. The packing list was placed at the top of the shipping box, between the cardboard covering and the foam insulation.

UNITED STATES DEPARTMENT OF COMMERCE
National Institute of Standards and Technology
Gaithersburg, Maryland 20899-

June 13, 2000

Dear Colleague:

Enclosed is the set of samples for the second quality assurance round robin exercise (Round Robin XVIII) for 2000. You will find one vial of each of three lyophilized and two liquid-frozen serum samples for analysis along with a form for reporting your results. When reporting your results, please submit one value for each analyte for a given serum sample. If an obtained value is below your limit of quantitation, please indicate this result on the form by using NQ *(Not Quantitated)*. For analytes not measured, please leave a blank. Results are due to NIST by **September 15, 2000**. Results received two weeks after the due date will not be included in the summary report for this round robin study. The feedback report concerning the study will be provided around mid-October.

Lyophilized samples should be reconstituted with 1.0 mL of HPLC-grade water or equivalent. We recommend that dissolution be facilitated with 3 to 5 min agitation in an ultrasonic bath or at least 30 min at room temperature with intermittent swirling. (CAUTION: Vigorous shaking will cause foaming and possibly interfere with accurate measurement. The rubber stopper contains phthalate esters that will leach into the sample upon intermittent contact of the liquid sample with the stopper. These esters absorb strongly in the UV region and elute near retinol in most LC systems creating analytical problems.) Pipette a known volume of serum from the vial for analysis. (The final volume of the reconstituted sample is greater than 1.0 mL). Liquid-frozen samples 267 and 268 should **not be** reconstituted.

For consistency, we request that laboratories use the following absorptivities (E 1% cm) in ethanol: retinol, 1843 at 325 nm; retinyl palmitate, 975 at 325 nm; α-tocopherol, 75.8 at 292 nm; γ-tocopherol, 91.4 at 298 nm; α-carotene, 2800 at 444 nm (in hexane); β-carotene, 2560 at 450 nm (in ethanol), 2592 at 452 nm (in hexane); lycopene, 3450 at 472 nm (in hexane).

Please mail or fax your results for Round Robin XLVIII to:

Micronutrients Measurement Quality Assurance Program
NIST
100 Bureau Drive, Stop 8392
Gaithersburg, MD 20899-8392
Fax: (301) 977-0685

If you have questions regarding this round robin exercise, please call me at (301) 975-3120; e-mail me at jbthomas@nist.gov; or mail/fax queries to the above address.

Sincerely,

Jeanice Brown Thomas
Research Chemist
Analytical Chemistry Division
Chemical Science and Technology Laboratory

Enclosures

cc: L. C. Sander

Participant #: _____ Date: _____

Round Robin XLVIII

NIST Micronutrients Measurement Quality Assurance Program

Analyte	264	265	266	267	268	Units*
total retinol						
trans-retinol						
retinyl palmitate						
α-tocopherol						
γ-tocopherol						
δ-tocopherol						
total β-carotene						
trans-β-carotene						
total cis-β-carotene						
total α-carotene						
trans-α-carotene						
total lycopene						
trans-lycopene						
total β-cryptoxanthin						
total α-cryptoxanthin						
total lutein						
total zeaxanthin						
total lutein&zeaxanthin						
ubiquinone-10 (Q_{10})						
phylloquinone (K_1)						
25-hydroxyvitamin D						
cholesterol						

Other analytes?						

* we prefer μg/mL

Were sera 267 & 268 frozen when received? Yes | No

Mail: M^2QAP
NIST, Stop 8392
Gaithersburg, MD 20899-8392

Please return results on-or-before
15-Sep-2000

Fax: 301-977-0685
Email: David.Duewer@NIST.gov

Participant #: _____ Date: _____

Round Robin XLVIII

NIST Micronutrients Measurement Quality Assurance Program

Packing List and Shipment Receipt Confirmation Form

The enclosed bubble-pack (should) contain one vial each of the following **five** sera:

Serum	Form	Reconstitute?
#264	Lyophilized	Yes (1 ml H_2O)
#265	Lyophilized	Yes (1 ml H_2O)
#266	Lyophilized	Yes (1 ml H_2O)
#267	Liquid frozen	No
#268	Liquid frozen	No

Please
1) Open the pack immediately
2) Check that it contains one vial each of the above samples
3) Check if sera #267 and #268 arrived frozen
4) Store the RR XLVIII samples upright at -20 ˚C or below until analysis
5) Complete the following information
6) Fax the completed form to us at 301-977-0685
(or email requested information to david.duewer@nist.gov)

1) Date this shipment arrived: _____

2) Are all five vials intact? Yes | No
If "No", which one(s) were damaged?

3) Was there any dry-ice left in cooler? Yes | No

4) Did sera #267 and #268 arrive frozen? Yes | No

5) At what temperature are you storing the samples? _____ ˚C

6) When do you expect to analyse these samples? _____

Thank you in advance for your prompt response.

The M^2QAP Gang

Mail: M^2QAP
NIST, Stop 8392
Gaithersburg, MD 20899-8392

Fax: 301-977-0685
Email: David.Duewer@NIST.gov

Appendix F. Final Report for RR48

The following three pages are the final report as provided to all participants:

- Cover letter
- An information sheet that:
  - describes the contents of the “All-Lab” report
  - describes the content of the “Individualized” report
  - describes the nature of the test samples and details any previous distributions
  - summarizes aspects of the study that we believe may be of interest to the participants

UNITED STATES DEPARTMENT OF COMMERCE
National Institute of Standards and Technology
Gaithersburg, Maryland 20899-0001

October 23, 2000

Dear Colleague:

Enclosed is the summary report of the results for Round Robin XLVIII (RR 48). Included in this report are: a summary of data for all laboratories; the measurement comparability summary for evaluating laboratory performance; a summary of individual laboratory performance and interlaboratory accuracy and precision; and a summary of the NIST assigned value (NAV) vs. your laboratory value for the analytes you measured. As in previous reports, the NIST assigned values are derived from the equally weighted means of the medians from this interlaboratory comparison exercise and the means from the analyses performed by NIST.

Data for evaluating laboratory performance in RR 48 are provided in the comparability summary (Score Card) on page 8 of the "All Lab Report." Laboratory comparability is summarized as follows: results rated 1 to 3 are within 1 to 3 standard deviation(s) of the assigned value, respectively; those rated 4 are >3 standard deviations from the assigned value.

If you have concerns regarding your laboratory's performance, we suggest that you obtain and analyze a unit of SRM 968c, Fat-Soluble Vitamins, Carotenoids, and Cholesterol in Human Serum. If your measured values do not agree with the certified values, we suggest that you contact us for consultation.

The following are newly released publications. Reprints will be provided upon request.

"NIST Micronutrients Measurement Quality Assurance Program: Characterizing the Measurement Community's Performance Over Time," Duewer et al., Anal. Chem., 72, 17, 4163- 4170 (2000).

"C_{30} Stationary Phases for the Analysis of Food by Liquid Chromatography," Sander, L.C., Sharpless, K.E., and Pursch M., J. Chromatogr. A. 880, 189-202 (2000).

The next NIST Micronutrients Measurement Quality Assurance Workshop will be held on **April 4, 2001** as a symposium at the Experimental Biology meeting in Orlando, Florida. This workshop will be held to allow participants to discuss analytical methods, results of intercomparison exercises from the past year, and other related topics. Details about the meeting will be sent under a separate cover as soon as meeting plans are finalized. A tentative workshop agenda is enclosed. There is no registration fee for the workshop. However, if you plan on attending the Experimental Biology conference on March 31-April 4, a registration fee is required. Please contact the secretariat for the American Institute of Nutrition at 301-530-7050; fax: 301-571-1892; e-mail:sec@asns.faseb.org for details regarding the conference.

If you have any questions regarding this report, please contact David Duewer at 301/975-3935; e-mail: david.duewer@nist.gov, or me at 301/975-3120; e-mail: jbthomas@nist.gov; fax: 301/977-0685.

Sincerely,

Jeanice Brown Thomas
Research Chemist
Analytical Chemistry Division
Chemical Science and Technology Laboratory

cc: L. C. Sander
Enclosures

NIST

The NIST M^2QAP Round Robin XLVIII (RR48) report consists of

Page	"Individualized" Report
1	Your values, the number of labs reporting values, and our assigned values.
2 to n	"Four Plot" summaries of your current and past measurement performance, one page for each analyte you report that is also reported by at least 10 other participants.
n+1	The "target" plot version of your "Comparability Summary" scores.

Page	"All Lab" Report
1-5	A listing of all results and statistics for analytes reported by at least two laboratories
6	A list of results for the four analytes reported by only one laboratory. A legend for the above two lists
7	The "Comparability Summary" (or "Score Card")

Samples. Five sera were distributed in RR48: the third distribution of SRM 968c Level II, a newly prepared {liquid-frozen, lyophilized} pair, a single new lyophilized serum, and a single new liquid-frozen serum. The new {liquid-frozen, lyophilized} pair furthers our investigation of measurement differences between "fresh" and lyophilized sera. SRM 968c Level II was distributed as a known reference for the new sera and to further assess measurand stability in the SRM.

Serum #264 is Level II of SRM 968c, previously distributed as #248 (RR44, 10/98) and #256 (RR46,10/99). This material is a blended serum with native carotenoid levels, but it was augmented with α- and δ-tocopherol.

Sera {#268, #265} are {liquid-frozen, lyophilized} material prepared from a native serum pool. These samples were prepared and aliquoted into vials as a single batch.

Serum #266 is a lyophilized material prepared from a native serum pool.

Serum #267 is a liquid-frozen material prepared from a native serum pool.

Qualitative Results.

1) Three participants noted (and, we think, all of you had) a small amount of precipitated material in Serum #266. We failed to re-filter the final blend of the serum pool used for this sample after a relatively small volume of a (filtered) stock serum was added to bring the pool to adequate volume. The resulting solids do not appear to affect anything other than aesthetics, but add it to the list of things we won't do again…

2) Two participants again diluted the liquid-frozen samples with 1 mL water. In addition to providing reconstitution instructions in the sample manifest and study cover letter, in the future we will label the vials themselves.

3) Nearly all participants did receive their samples still frozen. We thank all of you who promptly confirmed receipt of the samples. We will request similar confirmation in all future studies.

Quantitative Results

1) There have been no changes in median level or measurement variability in SRM 968c Level II. We will continue to monitor this material.

2) Since the lyophilized materials are reconstituted WITH 1.0 mL water rather than TO a total volume of 1.0 mL, we expect that the measurand concentrations in serum #265 (lyophilized) should be about 0.95x those of serum #268 (liquid-frozen). The observed medians of the participant ratios ranged from 0.97 for total a-carotene to 0.92 for retinyl palmitate, all with a fairly constant SD of ±0.05. The ratios for individual participants ranged from quite constant across all reported measurands to quite different for different measurands. All very confusing… we will try to sort some of it out for the Workshop coming up this Spring (see enclosed workshop agenda). If your average #265/#268 ratio was **not** in the 0.90 to 1.00 range, you should re-evaluate how you reconstitute lyophilized materials.

Appendix G. "All-Lab Report" for RR48

The following seven pages are the "All-Lab Report" as provided to all participants, with two exceptions:

- the participant identifiers (Lab) have been altered.
- the order in which the participant results are listed has been altered.

The data summary in the "All-Lab Report" has been altered to ensure confidentiality of identification codes assigned to laboratories. The only attributed results are those reported by NIST. The NIST results are not used in the assessment of the consensus summary results of the study.

Round Robin XLVIII Laboratory Results

All Results in µg/mL

	Total Retinol					trans-Retinol					Retinyl Palmitate					α-Tocopherol					γ-Tocopherol				
Lab	264	265	266	267	268	264	265	266	267	268	264	265	266	267	268	264	265	266	267	268	264	265	266	267	268
FSV-BA	0.504	0.628	0.457	0.687	0.673						0.102	0.214	0.061	*nd*	0.232	16.4	15.4	6.92	6.37	16.4	1.48	1.92	1.88	2.40	2.05
FSV-BB	0.497	0.578	0.456	0.675	0.632						0.071	0.186	0.046	*nd*	0.216	17.9	15.7	7.13	6.61	16.8	1.51	1.66	1.77	2.23	1.79
FSV-BD	0.479	0.571	0.440	0.658	0.615											15.1	13.8	6.40	6.20	14.8					
FSV-BE	0.450	0.570	0.420	0.640	0.650											15.4	13.8	6.30	5.80	15.6	1.60	1.90	1.90	2.40	2.20
FSV-BF	0.465	0.575	0.390	0.645	0.615											15.6	15.0	5.90	6.05	16.2	1.45	1.66	1.85	2.34	1.82
FSV-BG	0.467	0.597	0.439	0.695	0.680						0.082	0.203	0.077	0.069	0.236	16.8	15.6	6.95	6.57	17.4	1.57	1.72	1.93	2.40	1.91
FSV-BH	0.443	0.540	0.395	0.607	0.571						0.112	0.248	0.053	0.048	0.284	16.5	14.3	6.60	6.25	15.2	1.77	1.95	1.97	2.58	2.01
FSV-BI	0.489	0.595	0.457	0.687	0.644						0.084	0.234	0.058	0.022	0.270	16.8	14.2	6.99	6.46	15.3	1.47	1.67	1.91	2.31	1.83
FSV-BJ	0.481	0.580	0.431	0.647	0.603						0.063	0.189	0.032	*nq*	0.208	17.4	16.8	6.92	5.94	17.2	1.58	1.80	1.80	2.22	1.91
FSV-BK	0.494	0.556	0.450	0.668	0.624											16.4	13.4	6.35	5.53	14.6					
FSV-BL	0.490	0.600	0.460	0.720	0.690											16.8	15.1	9.04	6.89	13.8					
FSV-BM	0.467	0.574	0.421	0.651	0.593											15.3	13.9	6.40	5.70	14.6					
FSV-BN	0.446	0.503	0.440	0.595	0.570						0.084	0.217	0.064	0.027	0.233	16.1	12.5	6.82	5.80	14.2	1.21	1.20	1.46	1.67	1.42
FSV-BO	0.426	0.536	0.402	0.622	0.595											16.4	14.0	7.00	6.90	15.7					
FSV-BP	0.525	0.605	0.490	0.708	0.658											17.0	12.7	7.89	9.80	12.7					
FSV-BQ	0.492	0.581	0.411	0.631	0.595											15.2	12.9	6.14	5.98	13.0					
FSV-BR	>0.510	>0.620	>0.480	>0.685	>0.630	0.510	0.620	0.480	0.685	0.630						20.0	17.1	7.82	7.62	17.5					
FSV-BT	0.471	0.660	0.474	0.791	0.753											14.6	13.1	6.55	5.65	13.4	1.74	1.84	1.78	2.02	1.54
FSV-BU	0.386	0.524	0.370	0.584	0.573											14.2	9.0	5.68	5.21	14.9	1.28	1.55	1.73	2.12	1.67
FSV-BV	0.451	0.536	0.406	0.579	0.542											19.1	16.7	7.84	6.72	15.9	1.65	1.89	1.99	2.45	1.89
FSV-BW	0.510	0.630	0.460	0.710	0.660						0.082	0.340	0.056	0.022	0.340	18.0	15.5	7.32	6.45	15.4	1.90	2.08	2.28	2.67	2.20
FSV-BX	>0.489	>0.588	>0.435	>0.682	>0.649	0.489	0.588	0.435	0.682	0.649						16.3	14.6	6.71	6.30	16.1	1.64	1.79	1.91	2.52	1.97
FSV-CB	0.409	0.520	0.385	0.604	0.554											16.4	14.3	6.82	6.48	15.4					
FSV-CC	0.540	0.580	0.460	0.650	0.600	0.490	0.560	0.440	0.640	0.600						17.2	13.5	6.73	6.16	14.4					
FSV-CD	>0.501	>0.605	>0.447	>0.668	>0.620	0.501	0.605	0.447	0.668	0.620	0.093	0.181	0.056	0.035	0.220	16.4	14.8	6.40	6.00	14.4	1.62	1.95	1.94	2.38	1.86
FSV-CE	0.490	0.550	0.400	0.620	0.340											26.4	25.2	11.10	9.98	11.9					
FSV-CF	0.527	0.679	0.477	0.718	0.661											18.5	17.4	7.60	7.20	17.1					
FSV-CG	0.672	0.707	0.529	0.705	0.690											14.1	12.8	5.59	5.56	14.1	1.55	1.80	1.77	2.52	1.97
FSV-CH	0.452	0.553	0.384	0.588	0.601											18.6	15.5	6.85	7.08	16.9	1.13	1.34	1.26	1.78	1.44
FSV-CI	0.500	0.610	0.460	0.720	0.690						0.080	0.210	0.050	0.030	0.210	16.2	12.6	6.20	5.80	12.6	1.40	1.70	1.70	2.10	1.70
FSV-CK	0.370	0.440	0.340	0.510	0.500											13.3	12.2	5.06	4.80	12.6	1.24	2.10	1.44	1.91	2.24
FSV-CL	0.485	0.579	0.462	0.688	0.632											18.8	18.1	8.88	7.79	20.3	2.02	2.62	2.98	4.08	6.59
FSV-CN	0.501	0.636	0.452	0.755	0.682											16.3	14.8	6.44	6.37	15.6	1.56	1.74	1.79	2.55	1.90
FSV-CR	0.560	0.690	0.490	0.750	0.720											18.7	16.1	7.80	7.30	16.9					
FSV-CS	>0.484	>0.595	>0.442	>0.719	>0.623	0.484	0.595	0.442	0.719	0.623						17.2	14.7	6.80	6.02	14.4	1.64	1.84	1.97	2.40	1.86
FSV-CU	>0.431	>0.559	>0.402	>0.623	>0.621	0.431	0.559	0.402	0.623	0.621	0.126	0.202	0.098	0.081	0.231	15.4	12.7	6.69	6.55	14.9					
FSV-CV	0.523	0.432	0.205	0.325	0.400											18.0	14.9	6.17	6.07	15.4	1.65	1.71	1.74	2.33	1.88
FSV-CW	>0.441	>0.511	>0.388	>0.687	>0.558	0.441	0.511	0.388	0.687	0.558	0.066	0.207	0.040	0.013	0.216	18.9	15.8	7.26	6.65	17.7	1.78	1.99	2.08	2.64	2.11
FSV-CX	>0.420	>0.533	>0.386	>0.562	>0.535	0.420	0.533	0.386	0.562	0.535						15.2	13.3	6.04	5.34	13.4					
FSV-DA	0.466	0.579	0.442	0.656	0.615						0.068	0.263	0.041	0.028	0.294	15.9	14.6	6.85	6.28	15.8	1.47	1.61	1.67	2.09	1.68
FSV-DB	0.533	0.613	0.468	0.673	0.668											16.9	15.5	7.65	6.35	17.0					
FSV-DF	0.414	0.516	0.393	0.568	0.540																				
FSV-DI	>0.496	>0.610	>0.442	>0.687	>0.646	0.496	0.610	0.442	0.687	0.646	0.060	0.185	0.035	0.010	0.203	17.1	14.6	6.81	6.23	15.4	1.61	1.79	1.94	2.45	1.90
FSV-DK	0.497	0.605	0.434	0.751	0.662						0.023	0.114	0.022	*nq*	0.123	16.3	12.4	5.36	5.35	11.0	1.48	1.53	1.49	2.08	1.46
FSV-DP	>0.507	>0.606	>0.442	>0.667	>0.622	0.507	0.606	0.442	0.667	0.622															
FSV-DQ																14.9	12.5	4.05	4.29	10.5	1.49	1.39	1.30	1.45	1.72
FSV-DR	0.490	0.575	0.459	0.700	0.580											18.2	14.6	7.39	7.19	14.6					
FSV-DU	0.650	0.580	0.370	0.610	0.660											27.9	18.9	7.10	6.40	20.8					
FSV-EM	0.410	0.490	0.370	0.550	0.490											14.1	11.8	5.60	4.80	12.2					
FSV-EQ	0.484	0.592	0.461	0.804	0.640											16.7	15.1	6.90	8.40	15.6					
FSV-ES	>0.471	>0.587	>0.402	>0.667	>0.625	0.471	0.587	0.402	0.667	0.625						16.0	14.6	6.37	6.50	15.0					
FSV-FH	0.606	0.715	0.516	0.806	0.747											16.9	13.2	6.70	6.50	13.7					
FSV-FJ	0.690	0.950	0.700	1.060	0.980											18.6	17.4	8.10	7.30	18.4	1.90	2.30	2.40	3.00	2.40
FSV-FT	0.530	0.700	0.520	0.750	0.660											20.0	14.2	8.70	7.20	13.9					
FSV-FW	0.504	0.645	0.492	0.742	0.726																				
N	44	44	44	44	44	11	11	11	11	11	15	15	15	11	15	52	52	52	52	52	29	29	29	29	29
Min	0.370	0.432	0.205	0.325	0.340	0.420	0.511	0.386	0.562	0.535	0.023	0.114	0.022	0.010	0.123	13.3	9.0	4.05	4.29	10.5	1.13	1.20	1.26	1.45	1.42
Median	0.490	0.580	0.446	0.671	0.632	0.489	0.588	0.440	0.668	0.622	0.082	0.207	0.053	0.028	0.231	16.6	14.6	6.81	6.36	15.3	1.57	1.79	1.85	2.38	1.89
Max	0.690	0.950	0.700	1.060	0.980	0.510	0.620	0.480	0.719	0.649	0.126	0.340	0.098	0.081	0.340	27.9	25.2	11.10	9.98	20.8	2.02	2.62	2.98	4.08	6.59
eSD	0.043	0.047	0.044	0.073	0.059	0.027	0.033	0.010	0.028	0.012	0.021	0.031	0.016	0.010	0.031	1.8	1.6	0.67	0.70	1.7	0.13	0.19	0.17	0.25	0.23
eCV	9	8	10	11	9	5	6	2	4	2	25	15	31	37	13	11	11	10	11	11	8	11	9	10	12
N_{past}	47	0	0	0	0	7	0	0	0	0	16	0	0	0	0	48	0	0	0	0	26	0	0	0	0
$Median_{past}$	0.479					0.485					0.081					16.6					1.58				
SD_{past}	0.038					0.050					0.022					1.3					0.14				
NISTa	>0.491	>0.633	>0.460	>0.717	>0.671	0.491	0.633	0.460	0.717	0.671						16.8	15.5	7.18	6.29	15.4	1.55	1.98	1.98	2.53	1.98
NISTb	>0.475	>0.602	>0.439	>0.673	>0.644	0.475	0.602	0.439	0.673	0.644						17.2	15.2	6.92	6.26	15.5	1.60	1.82	1.91	2.40	1.88
N_{NIST}	6	5	6	6	6	6	5	6	6	6						6	5	6	6	6	6	5	6	6	6
$Mean_{NIST}$	0.483	0.618	0.449	0.695	0.657	0.483	0.618	0.449	0.695	0.657						17.0	15.4	7.05	6.28	15.4	1.58	1.90	1.94	2.46	1.93
S_{rep}	0.015	0.005	0.012	0.011	0.012	0.015	0.005	0.012	0.011	0.012						0.3	0.2	0.18	0.11	0.1	0.06	0.06	0.09	0.08	0.08
S_{het}	0.019	0.016	0.009	0.011	0.022	0.019	0.016	0.009	0.011	0.022						0.5	0.4	0.17	0.22	0.4	0.03	0.12	0.04	0.06	0.10
S_{anl}	0.011	0.022	0.017	0.031	0.019	0.011	0.022	0.017	0.031	0.019						0.3	0.2	0.19	0.02	0.1	0.04	0.11	0.05	0.09	0.07
S_{NIST}	0.026	0.028	0.022	0.035	0.031	0.026	0.028	0.022	0.035	0.031						0.7	0.5	0.31	0.25	0.4	0.08	0.17	0.11	0.14	0.15
NAV	0.486	0.599	0.449	0.682	0.645	0.486	0.603	0.444	0.682	0.640	0.082	0.207	0.053	0.028	0.231	16.8	15.0	6.93	6.30	15.3	1.57	1.85	1.90	2.40	1.91
NAU	0.040	0.056	0.043	0.080	0.065	0.040	0.051	0.036	0.056	0.055	0.026	0.050	0.021	0.018	0.055	1.6	1.7	0.70	0.64	1.7	0.17	0.21	0.21	0.28	0.22

Round Robin XLVIII Laboratory Results

All Results in µg/mL

	δ-Tocopherol					Total β-Carotene					trans-β-Carotene					Total cis-β-Carotene				
Lab	264	265	266	267	268	264	265	266	267	268	264	265	266	267	268	264	265	266	267	268
FSV-BA						0.419	0.276	0.326	0.515	0.302	0.385	0.255	0.300	0.491	0.281	0.034	0.021	0.025	0.024	0.021
FSV-BB						0.386	0.254	0.296	0.475	0.271	0.367	0.244	0.284	0.461	0.261	0.019	0.011	0.013	0.014	0.010
FSV-BD																				
FSV-BE						0.189	0.109	0.143	0.224	0.117										
FSV-BF						0.405	0.183	0.307	0.530	0.252										
FSV-BG						0.452	0.301	0.338	0.614	0.319										
FSV-BH	0.23	*nd*	*nd*	*nd*	*nd*	0.461	0.290	0.337	0.533	0.311	0.430	0.273	0.314	0.511	0.292	0.031	0.018	0.023	0.022	0.019
FSV-BI						0.460	0.290	0.353	0.582	0.313										
FSV-BJ						0.391	0.205	0.271	0.394	0.197										
FSV-BK																				
FSV-BL																				
FSV-BM																				
FSV-BN	0.96	0.45	0.46	0.47	0.46	0.455	0.265	0.368	0.508	0.298	0.409	0.243	0.341	0.478	0.277	*0.046*	*0.022*	*0.027*	*0.030*	*0.021*
FSV-BO						*0.396*	*0.248*	*0.190*	*0.247*	*0.272*	0.388	0.237	0.179	0.238	0.267	0.008	0.011	0.011	0.009	0.005
FSV-BP						0.415	0.245	0.290	0.465	0.243										
FSV-BQ																				
FSV-BR																				
FSV-BT	1.65	0.80	0.82	0.54	0.50	0.346	0.236	0.272	0.442	0.242	0.316	0.219	0.254	0.418	0.222	0.034	0.017	0.018	0.024	0.021
FSV-BU						0.483	0.335	0.373	0.590	0.356										
FSV-BV						0.404	0.269	0.333	0.527	0.288										
FSV-BW						0.420	0.270	0.330	0.520	0.270										
FSV-BX						>0.413	>0.271	>0.315	>0.525	>0.294	0.413	0.271	0.315	0.525	0.294					
FSV-CB						0.530	0.316	0.399	0.535	0.320										
FSV-CC																				
FSV-CD						0.426	0.258	0.299	0.494	0.263										
FSV-CE						0.420	0.340	0.380	0.550	0.140										
FSV-CF																				
FSV-CG	0.83	0.17	0.16	0.12	0.20	0.400	0.271	0.304	0.498	0.287	0.369	0.249	0.280	0.470	0.265	0.031	0.022	0.023	0.028	0.022
FSV-CH						0.441	0.284	0.307	0.528	0.304										
FSV-CI																				
FSV-CK						0.258	0.170	0.213	0.329	0.172										
FSV-CL						0.506	0.323	0.397	0.558	0.350										
FSV-CN						>0.405	>0.260	>0.307	>0.523	>0.263	0.405	0.260	0.307	0.523	0.263					
FSV-CR																				
FSV-CS						0.452	0.302	0.356	0.567	0.310	0.407	0.276	0.327	0.528	0.285	*0.044*	*0.026*	*0.030*	*0.039*	*0.025*
FSV-CU																				
FSV-CV						0.490	0.165	0.400	0.587	0.174										
FSV-CW	0.47	0.08	0.08	0.07	0.09	0.391	0.248	0.310	0.472	0.271	0.363	0.233	0.290	0.448	0.252	*0.028*	*0.015*	*0.020*	*0.024*	*0.019*
FSV-CX						>0.332	>0.223	>0.259	>0.418	>0.208	0.332	0.223	0.259	0.418	0.208					
FSV-DA	0.56	0.15	0.14	0.11	0.12	0.280	0.275	0.227	0.362	0.288	0.250	0.244	0.208	0.331	0.255	0.030	0.031	0.019	0.031	0.033
FSV-DB						0.350	0.252	0.295	0.457	0.245										
FSV-DF																				
FSV-DI	0.67	0.14	0.14	0.12	0.14	>0.356	>0.216	>0.287	>0.452	>0.247	0.356	0.216	0.287	0.452	0.247					
FSV-DK						0.340	0.232	0.283	0.461	0.246										
FSV-DP																				
FSV-DQ						0.305	0.149	0.197	0.433	0.268										
FSV-DR						0.450	0.326	0.440	0.705	0.324										
FSV-DU						>0.280	>0.130	>0.200	>0.470	>0.090	0.280	0.130	0.200	0.470	0.090					
FSV-EM						0.429	0.290	0.135	0.515	0.268										
FSV-EQ						0.477	0.310	0.455	0.634	0.790	0.463	0.301	0.444	0.622	0.630	0.014	0.009	0.011	0.012	0.160
FSV-ES						>0.349	>0.227	>0.263	>0.468	>0.246	0.349	0.227	0.263	0.468	0.246					
FSV-FH																				
FSV-FJ						0.306	0.193	0.247	0.424	0.199										
FSV-FT																				
FSV-FW																				
N	7	6	6	6	6	33	33	33	33	33	17	17	17	17	17	11	11	11	11	11
Min	0.23	0.08	0.08	0.07	0.09	0.189	0.109	0.135	0.224	0.117	0.250	0.130	0.179	0.238	0.090	0.008	0.009	0.011	0.009	0.005
Median	0.67	0.16	0.15	0.12	0.17	0.419	0.269	0.307	0.515	0.271	0.369	0.244	0.287	0.470	0.263	0.031	0.018	0.020	0.024	0.021
Max	1.65	0.80	0.82	0.54	0.50	0.530	0.340	0.455	0.705	0.790	0.463	0.301	0.444	0.622	0.630	0.046	0.031	0.030	0.039	0.160
eSD	0.30	0.07	0.06	0.05	0.10	0.049	0.047	0.068	0.077	0.046	0.055	0.026	0.042	0.061	0.025	0.004	0.006	0.007	0.009	0.003
eCV	45	47	41	40	57	12	18	22	15	17	15	11	14	13	9	14	33	37	37	14
N_{past}	10	0	0	0	0	33	0	0	0	0	15	0	0	0	0	10	0	0	0	0
$Median_{past}$	0.80					0.424					0.389					0.031				
SD_{past}	0.41					0.048					0.029					0.010				
NISTa	0.53	*nd*	*nd*	*nd*	*nd*	0.424	0.249	0.326	0.506	0.241	0.378	0.215	<0.326	0.438	0.200	*0.047*	*0.033*	*nd*	*0.068*	*0.041*
NISTb	0.54	0.11	0.10	0.09	0.11	0.425	0.275	0.329	0.526	0.289	0.379	0.243	0.300	0.486	0.259	*0.046*	*0.032*	*0.029*	*0.040*	*0.030*
N_{NIST}	6	2	3	3	3	6	5	6	6	6	6	5	3	6	6					
$Mean_{NIST}$	0.54	0.11	0.10	0.09	0.11	0.425	0.264	0.328	0.516	0.264	0.385	0.229	0.300	0.462	0.230					
S_{rep}	0.02	0.01	0.02	0.01	0.01	0.009	0.007	0.020	0.017	0.012	0.009	0.005	0.005	0.026	0.010					
S_{het}	0.03	0.02	0.01	0.01	0.01	0.023	0.012	0.024	0.031	0.011	0.032	0.010	0.003	0.024	0.006					
S_{anl}	0.00					0.007	0.022	0.002	0.014	0.033	0.004	0.020		0.033	0.042					
S_{NIST}	0.03	0.02	0.02	0.02	0.02	0.025	0.026	0.031	0.038	0.036	0.034	0.022	0.006	0.049	0.043					
NAV	0.61	0.14	0.12	0.11	0.14	0.422	0.266	0.317	0.512	0.268	0.377	0.237	0.293	0.466	0.246	0.031	0.018	0.020	0.024	0.021
NAU	0.30	0.18	0.18	0.20	0.21	0.059	0.040	0.064	0.071	0.046	0.044	0.029	0.042	0.050	0.049	0.011	0.007	0.007	0.008	0.007

Round Robin XLVIII Laboratory Results

All Results in µg/mL

	Total α-Carotene					trans-α-Carotene					Total Lycopene					trans-Lycopene				
Lab	264	265	266	267	268	264	265	266	267	268	264	265	266	267	268	264	265	266	267	268
FSV-BA	0.096	0.102	0.030	0.020	0.112						!0.544	!0.39	!0.465	!0.313	!0.426	0.229	0.159	0.187	0.155	0.176
FSV-BB	0.077	0.093	0.020	0.015	0.099						0.410	0.310	0.334	0.233	0.330	0.169	0.122	0.145	0.119	0.130
FSV-BD																				
FSV-BE																				
FSV-BF	0.093	0.101	0.039	0.030	0.099						0.428	0.318	0.306	0.214	0.308					
FSV-BG	0.098	0.106	0.036	0.036	0.102						0.447	0.305	0.313	0.232	0.313	0.237	0.155	0.178	0.150	0.159
FSV-BH	0.112	0.116	0.026	0.019	0.124						0.434	0.311	0.330	0.229	0.325					
FSV-BI	0.100	0.101	0.029	0.025	0.115						0.349	0.241	0.269	0.192	0.281					
FSV-BJ	0.104	0.095	0.026	0.017	0.098						0.340	0.234	0.244	0.148	0.217					
FSV-BK																				
FSV-BL																				
FSV-BM																				
FSV-BN	0.090	0.088	0.031	0.016	0.108						0.485	0.330	0.417	0.270	0.370	0.259	0.160	0.220	0.159	0.182
FSV-BO	0.192	0.175	0.064	0.076	0.207						0.351	0.241	0.310	0.228	0.295					
FSV-BP	0.121	0.117	0.036	0.027	0.109						0.546	0.355	0.469	0.346	0.337					
FSV-BQ																				
FSV-BR																				
FSV-BT	0.074	0.083	0.026	0.020	0.086						0.425	0.325	0.334	0.248	0.336	0.174	0.132	0.139	0.102	0.132
FSV-BU	0.111	0.141	0.040	0.031	0.148						0.338	0.265	0.265	0.132	0.278					
FSV-BV	0.098	0.111	0.030	0.019	0.105						0.427	0.324	0.354	0.246	0.337					
FSV-BW	0.104	0.112	0.038	0.026	0.110						0.410	0.310	0.330	0.250	0.310					
FSV-BX	0.110	0.121	0.036	0.026	0.126											0.196	0.137	0.163	0.136	0.144
FSV-CB	0.085	0.084	0.022	0.013	0.085						0.355	0.227	0.282	0.150	0.220					
FSV-CC																				
FSV-CD	0.106	0.092	0.028	0.016	0.105						0.407	0.289	0.308	0.226	0.318					
FSV-CE																				
FSV-CF																				
FSV-CG	0.098	0.110	0.032	0.024	0.118						0.424	0.324	0.332	0.246	0.344	0.212	0.156	0.173	0.146	0.166
FSV-CH	0.094	0.104	0.028	0.021	0.111						0.367	0.267	0.260	0.215	0.319					
FSV-CI																				
FSV-CK	0.053	0.098	0.033	0.019	0.098						0.283	0.230	0.252	0.158	0.197					
FSV-CL	0.112	0.115	0.029	0.022	0.116						0.519	0.392	0.435	0.274	0.408					
FSV-CN	0.105	0.104	0.038	0.030	0.103						0.428	0.323	0.343	0.262	0.347					
FSV-CR																				
FSV-CS	0.137	0.151	0.035	0.027	0.158						0.473	0.369	0.388	0.271	0.378					
FSV-CU																				
FSV-CV											0.480	0.326	0.332	0.278	0.343					
FSV-CW	*>0.076*	*>0.081*	*>0.025*	*>0.018*	*>0.087*	0.076	0.081	0.025	0.018	0.087	0.423	0.314	0.346	0.228	0.329	0.189	0.131	0.163	0.124	0.142
FSV-CX	*>0.097*	*>0.100*	*>0.026*	*>0.017*	*>0.091*	0.097	0.100	0.026	0.017	0.091						0.204	0.143	0.167	0.134	0.132
FSV-DA	0.069	0.102	0.026	0.019	0.112						0.332	0.318	0.269	0.183	0.365	0.161	0.154	0.135	0.111	0.172
FSV-DB											0.493	0.357	0.402	0.302	0.344					
FSV-DF																				
FSV-DI																0.212	0.140	0.175	0.126	0.163
FSV-DK	0.096	0.113	0.032	0.021	0.117															
FSV-DP																				
FSV-DQ	0.073	0.087	0.027	0.026	0.052						0.305	0.209	0.258	0.200	0.243					
FSV-DR																				
FSV-DU																				
FSV-EM	0.102	0.102	0.027	0.016	0.097						0.381	0.268	0.268	0.193	0.247					
FSV-EQ	0.117	0.118	0.046	0.114	0.120						0.397	0.295	0.399	0.724	0.756					
FSV-ES	*>0.088*	*>0.099*	*>0.023*	*>0.018*	*>0.100*	0.088	0.099	0.023	0.018	0.100	0.315	0.231	0.256	0.205	0.267	0.188	0.135	0.158	0.139	0.151
FSV-FH																				
FSV-FJ	0.070	0.075	0.021	*nq*	0.075															
FSV-FT																				
FSV-FW																				
N	29	29	29	28	29	3	3	3	3	3	29	29	29	29	29	12	12	12	12	12
Min	0.053	0.075	0.020	0.013	0.052	0.076	0.081	0.023	0.017	0.087	0.283	0.209	0.244	0.132	0.197	0.161	0.122	0.135	0.102	0.130
Median	0.098	0.104	0.030	0.022	0.109	0.088	0.099	0.025	0.018	0.091	0.410	0.310	0.330	0.229	0.325	0.200	0.142	0.165	0.135	0.155
Max	0.192	0.175	0.064	0.114	0.207	0.097	0.100	0.026	0.018	0.100	0.546	0.392	0.469	0.724	0.756	0.259	0.160	0.220	0.159	0.182
eSD	0.017	0.016	0.006	0.007	0.015						0.081	0.031	0.086	0.049	0.033	0.028	0.017	0.017	0.019	0.022
eCV	17	15	20	33	14						20	10	26	21	10	14	12	10	14	14
N_{past}	28	0	0	0	0	0	0	0	0	0	27	0	0	0	0	13	0	0	0	0
$Median_{past}$	0.095										0.404					0.206				
SD_{past}	0.023										0.076					0.041				
NISTa	0.106	0.109	0.040	*nd*	0.109															
NISTb	0.092	0.091	0.029	0.020	0.097	0.080	0.082	0.024	0.015	0.089	0.439	0.291	0.334	0.241	0.319					
N_{NIST}	6	5	5	3	6	3	2	3	3	3	3	2	3	3	3					
$Mean_{NIST}$	0.100	0.100	0.034	0.020	0.103	0.082	0.082	0.024	0.015	0.089	0.449	0.291	0.334	0.241	0.319					
S_{rep}	0.008	0.004	0.004	0.005	0.004	0.009	0.002	0.003	0.004	0.005	0.047	0.015	0.028	0.013	0.030					
S_{het}	0.009	0.004	0.005	0.006	0.004	0.009	0.003	0.001	0.001	0.004	0.047	0.013	0.026	0.023	0.025					
S_{anl}	0.009	0.012	0.008		0.008															
S_{NIST}	0.015	0.013	0.010	0.008	0.010	0.013	0.004	0.003	0.004	0.006	0.066	0.019	0.038	0.026	0.039					
NAV	0.099	0.102	0.032	0.020	0.106						0.429	0.300	0.332	0.235	0.319	0.200	0.142	0.165	0.135	0.155
NAU	0.028	0.030	0.011	0.008	0.031						0.092	0.071	0.074	0.055	0.072	0.036	0.025	0.029	0.024	0.027

Round Robin XLVIII Laboratory Results

All Results in µg/mL

	Total β-Cryptoxanthin					Total α-Cryptoxanthin					Total Lutein					trans-Lutein				
Lab	264	265	266	267	268	264	265	266	267	268	264	265	266	267	268	264	265	266	267	268
FSV-BA	0.059	0.070	0.083	0.078	0.078															
FSV-BB	0.034	0.062	0.054	0.054	0.066	0.014	0.042	0.024	0.026	0.043	0.098	0.172	0.089	0.102	0.189					
FSV-BD																				
FSV-BE																				
FSV-BF	0.027	0.053	0.039	0.043	0.058															
FSV-BG	0.032	0.052	0.048	0.047	0.066						0.089	0.180	0.089	0.065	0.209					
FSV-BH	0.043	0.079	0.061	0.062	0.081						0.071	0.142	0.073	0.078	0.153					
FSV-BI	0.036	0.071	0.064	0.064	0.077						0.078	0.172	0.091	0.089	0.189					
FSV-BJ	0.025	0.047	0.042	0.041	0.049						0.080	0.189	0.075	0.080	0.189					
FSV-BK																				
FSV-BL																				
FSV-BM																				
FSV-BN	0.037	0.061	0.063	0.065	0.074	0.016	0.040	0.026	0.033	0.049	0.064	0.123	0.074	0.073	0.141					
FSV-BO	0.047	0.053	0.042	0.046	0.059						0.142	0.276	0.179	0.184	0.307					
FSV-BP	0.055	0.061	0.065	0.056	0.064															
FSV-BQ																				
FSV-BR																				
FSV-BT	0.038	0.066	0.061	0.060	0.070	0.031	0.052	0.035	0.039	0.053	0.090	0.181	0.100	0.138	0.211					
FSV-BU	0.017	0.113	0.079	0.076	0.105															
FSV-BV	0.020	0.039	0.037	0.036	0.042															
FSV-BW																				
FSV-BX	0.035	0.056	0.051	0.054	0.062						0.082	0.167	0.082	0.103	0.181					
FSV-CB	0.019	0.032	0.031	0.032	0.035						-	0.159	0.086	0.083	0.166					
FSV-CC																				
FSV-CD	0.026	0.054	0.054	0.045	0.072															
FSV-CE																				
FSV-CF																				
FSV-CG	0.041	0.064	0.049	0.048	0.070															
FSV-CH																				
FSV-CI																				
FSV-CK	0.037	0.086	0.054	0.059	0.086	0.034	0.034	0.021	0.032	0.038						0.069	0.159	0.075	0.076	0.167
FSV-CL	0.036	0.072	0.063	0.066	0.088	0.018	0.054	0.035	0.030	0.060										
FSV-CN	0.035	0.066	0.055	0.046	0.067															
FSV-CR																				
FSV-CS	0.031	0.054	0.047	0.047	0.060															
FSV-CU																				
FSV-CV																				
FSV-CW	0.044	0.066	0.056	0.054	0.072	0.022	0.043	0.028	0.029	0.046	0.078	0.140	0.074	0.099	0.155	0.074	0.133	0.070	0.094	0.146
FSV-CX	0.024	0.051	0.041	0.042	0.050						0.063	0.128	0.064	0.061	0.129					
FSV-DA	0.032	0.066	0.048	0.048	0.076	0.015	0.040	0.020	0.024	0.044	0.090	0.158	0.084	0.094	0.170					
FSV-DB	0.044	0.063	0.063	0.053	0.072															
FSV-DF																				
FSV-DI											0.074	0.156	0.076	0.084	0.165					
FSV-DK																				
FSV-DP																				
FSV-DQ	0.020	0.023	0.025	0.026	0.025						*nd*	0.041	0.060	0.190	0.262					
FSV-DR																				
FSV-DU																				
FSV-EM	0.028	0.055	0.044	0.050	0.055															
FSV-EQ	0.023	0.065	0.075	0.052	0.060															
FSV-ES	0.032	0.062	0.053	0.059	0.071						0.108	0.210	0.098	0.095	0.233					
FSV-FH																				
FSV-FJ																				
FSV-FT																				
FSV-FW																				
N	29	29	29	29	29	7	7	7	7	7	14	16	16	16	16	2	2	2	2	2
Min	0.017	0.023	0.025	0.026	0.025	0.014	0.034	0.020	0.024	0.038	0.063	0.041	0.060	0.061	0.129	0.069	0.133	0.070	0.076	0.146
Median	0.034	0.062	0.054	0.052	0.067	0.018	0.042	0.026	0.030	0.046	0.081	0.163	0.083	0.092	0.185	0.072	0.146	0.073	0.085	0.157
Max	0.059	0.113	0.083	0.078	0.105	0.034	0.054	0.035	0.039	0.060	0.142	0.276	0.179	0.190	0.307	0.074	0.159	0.075	0.094	0.167
eSD	0.010	0.012	0.013	0.010	0.013	0.006	0.003	0.007	0.004	0.004	0.013	0.029	0.013	0.017	0.037					
eCV	31	19	25	20	19	33	7	29	15	10	16	18	15	19	20					
N_{past}	28	0	0	0	0	6	0	0	0	0	16	0	0	0	0	0	0	0	0	0
$Median_{past}$	0.031					0.018					0.084									
SD_{past}	0.008					0.007					0.016									
NISTa	0.041	0.065	0.067	0.073	0.067						0.088	0.187	0.100	0.096	0.195					
NISTb	0.026	0.049	0.048	0.050	0.054	0.021	0.039	0.028	0.029	0.042	0.075	0.153	0.076	0.078	0.158					
N_{NIST}	5	5	6	6	6	3	2	3	3	3	6	5	6	6	6					
$Mean_{NIST}$	0.034	0.057	0.057	0.062	0.061	0.021	0.039	0.028	0.029	0.042	0.081	0.170	0.088	0.087	0.177					
S_{rep}	0.001	0.004	0.002	0.004	0.005	0.003	0.004	0.003	0.001	0.005	0.006	0.002	0.006	0.005	0.010					
S_{het}	0.002	0.005	0.001	0.006	0.002	0.003	0.004	0.003	0.002	0.002	0.002	0.003	0.003	0.003	0.004					
S_{anl}	0.011	0.011	0.014	0.016	0.009						0.009	0.024	0.017	0.013	0.027					
S_{NIST}	0.011	0.013	0.014	0.017	0.010	0.004	0.006	0.004	0.002	0.005	0.011	0.024	0.018	0.014	0.029					
NAV	0.034	0.059	0.056	0.056	0.063	0.020	0.041	0.027	0.029	0.043	0.081	0.166	0.085	0.089	0.181					
NAU	0.011	0.015	0.014	0.019	0.016	0.008	0.006	0.007	0.006	0.005	0.016	0.030	0.018	0.018	0.035					

Round Robin XLVIII Laboratory Results

All Results in µg/mL

	Total Zeaxanthin					Total Lutein&Zeaxanthin					Coenzyme Q10					Phylloquinone (K1) x1000				
Lab	264	265	266	267	268	264	265	266	267	268	264	265	266	267	268	264	265	266	267	268
FSV-BA						0.122	0.228	0.121	0.150	0.256										
FSV-BB	0.042	0.045	0.035	0.043	0.048	*0.140*	*0.217*	*0.124*	*0.145*	*0.237*										
FSV-BD																				
FSV-BE																0.94	1.16	0.42	1.64	1.38
FSV-BF						0.106	0.217	0.100	0.129	0.255										
FSV-BG	0.021	0.040	0.020	0.011	0.041	0.117	0.235	0.116	0.079	0.265										
FSV-BH	0.017	0.041	0.029	0.034	0.043	0.088	0.183	0.101	0.112	0.196										
FSV-BI	0.019	0.040	0.027	0.035	0.044	*0.097*	*0.212*	*0.118*	*0.124*	*0.233*										
FSV-BJ																				
FSV-BK																				
FSV-BL																				
FSV-BM																				
FSV-BN	0.028	0.052	0.035	0.061	0.054	0.078	0.149	0.092	0.104	0.169										
FSV-BO	0.088	0.095	0.065	0.077	0.095	*0.230*	*0.371*	*0.244*	*0.261*	*0.402*										
FSV-BP						0.093	0.200	0.096	0.104	0.222										
FSV-BQ																				
FSV-BR																				
FSV-BT	0.021	0.047	0.026	0.029	0.057	0.111	0.228	0.125	0.167	0.268										
FSV-BU						0.093	0.273	0.123	0.156	0.273										
FSV-BV						0.102	0.212	0.114	0.134	0.224										
FSV-BW																				
FSV-BX	0.023	0.048	0.028	0.039	0.045	0.105	0.214	0.110	0.142	0.226										
FSV-CB	-	0.037	0.027	0.041	0.037	0.105	*0.196*	*0.114*	*0.124*	*0.204*										
FSV-CC																				
FSV-CD						0.110	0.208	0.097	0.118	0.224										
FSV-CE																				
FSV-CF																				
FSV-CG						0.124	0.233	0.122	0.152	0.261										
FSV-CH											1.202	0.882	0.884	1.245	0.872					
FSV-CI																				
FSV-CK	0.001	0.002	0.001	0.002	0.002	0.090	0.186	0.099	0.102	0.179										
FSV-CL						0.121	0.219	0.123	0.152	0.256										
FSV-CN						0.100	0.207	0.106	0.133	0.217										
FSV-CR																				
FSV-CS						0.109	0.241	0.118	0.144	0.296										
FSV-CU																				
FSV-CV						0.102	0.200	0.105	0.116	0.214										
FSV-CW	0.021	0.038	0.026	0.047	0.042	*0.099*	*0.178*	*0.100*	*0.146*	*0.197*	0.820	0.640	0.610	0.680	0.630					
FSV-CX	0.021	0.044	0.024	0.031	0.043	*0.084*	*0.172*	*0.088*	*0.092*	*0.172*										
FSV-DA	0.030	0.025	0.036	0.032	0.038	0.120	0.178	0.120	0.126	0.208										
FSV-DB						0.108	0.207	0.131	0.136	0.240										
FSV-DF																				
FSV-DI											0.388	0.267	0.317	0.318	0.292	1.43	1.28	0.42	1.42	1.61
FSV-DK																				
FSV-DP																				
FSV-DQ	0.018	0.014	0.011	0.018	0.028	*0.018*	*0.056*	*0.071*	*0.207*	*0.290*										
FSV-DR																				
FSV-DU																				
FSV-EM						0.080	0.165	0.074	0.074	0.165										
FSV-EQ						0.127	0.200	0.132	0.242	0.254										
FSV-ES	0.024	0.048	0.028	0.040	0.055	*0.132*	*0.258*	*0.126*	*0.135*	*0.287*										
FSV-FH																				
FSV-FJ																				
FSV-FT																				
FSV-FW																				
N	14	15	15	15	15	29	29	29	29	29	3	3	3	3	3	2	2	2	2	2
Min	0.001	0.002	0.001	0.002	0.002	0.018	0.056	0.071	0.074	0.165	0.388	0.267	0.317	0.318	0.292	0.94	1.16	0.42	1.42	1.38
Median	0.021	0.041	0.027	0.035	0.043	0.105	0.208	0.114	0.134	0.233	0.820	0.640	0.610	0.680	0.630	1.19	1.22	0.42	1.53	1.50
Max	0.088	0.095	0.065	0.077	0.095	0.230	0.371	0.244	0.261	0.402	1.202	0.882	0.884	1.245	0.872	1.43	1.28	0.42	1.64	1.61
eSD	0.005	0.008	0.005	0.009	0.007	0.018	0.030	0.017	0.027	0.042										
eCV	23	21	18	27	17	17	14	15	20	18										
N_{past}	15	0	0	0	0	27	0	0	0	0	0	0	0	0	0	0	0	0	0	0
$Median_{past}$	0.024					0.110														
SD_{past}	0.007					0.028														
NISTa	0.019	0.030	*nd*	0.040	0.036	*0.107*	*0.217*	*0.100*	*0.136*	*0.231*										
NISTb	0.031	0.050	0.031	0.041	0.050	*0.107*	*0.202*	*0.107*	*0.119*	*0.208*										
N_{NIST}	6	5	3	6	6	6	5	6	6	6										
$Mean_{NIST}$	0.024	0.040	0.031	0.040	0.043	0.107	0.210	0.103	0.128	0.219										
S_{rep}	0.003	0.002	0.003	0.003	0.005	0.005	0.004	0.008	0.006	0.010										
S_{het}	0.004	0.000	0.002	0.003	0.003	0.006	0.002	0.004	0.004	0.003										
S_{anl}	0.008	0.013		0.001	0.010	0.002	0.011	0.005	0.012	0.016										
S_{NIST}	0.010	0.014	0.004	0.004	0.012	0.008	0.011	0.010	0.014	0.019										
NAV	0.023	0.041	0.029	0.039	0.043	0.106	0.210	0.109	0.129	0.222										
NAU	0.010	0.014	0.008	0.011	0.012	0.022	0.044	0.025	0.028	0.047										

Round Robin XLVIII Laboratory Results

All Results in µg/mL

Analytes Reported By One Laboratory

Analyte	Code	264	265	266	267	268
trans-β-Cryptoxanthin	NISTb	0.025	0.044	0.042	0.043	0.051
3'-dehydro-Lutein	FSV-BH	0.03	0.05	0.03	0.03	0.06
Total Carotenoids	FSV-BT	1.16	1.15	0.95	1.08	1.30
Phytofluene	FSV-CL	0.051	0.030	0.046	0.024	0.152
Phytoene	FSV-CL	0.109	0.122	0.109	0.038	0.282
25-hydroxyvitamin D	FSV-BN	*nq*	0.018	0.041	0.009	0.017
25-hydroxyvitamin D3	FSV-EM	0.03	0.02	0.01	0.00	0.02

Table Legend

Term	Definition
N	Number of (non-NIST) quantitative values reported for this analyte
Min	Minimum (non-NIST) quantitative value reported
$Median_{part}$	Median (non-NIST) quantitative value reported
Max	Maximum (non-NIST) quantitative value reported
SD	Standard deviation for (non-NIST) results: 0.741*(3rd Quartile - 1st Quartile)
CV	Coefficient of Variation for (non-NIST) results: 100*SD/Median
N_{past}	Mean of N(s) from past RR(s)
$Median_{past}$	Mean of Median(s) from past RR(s)
SD_{past}	Pooled SD from past RR(s)
NISTa, NISTb	Mean of all analyses (vials x duplicates) reported by given NIST analyst
N_{NIST}	Number of total vials analyzed in duplicate by NIST analysts
$Mean_{NIST}$	Mean of the NIST-analyzed vial means
S_{rep}	Within-vial pooled standard deviation
S_{het}	Among-vial pooled standard deviation
S_{anl}	Between NIST analyst standard deviation
S_{NIST}	Total standard deviation for NIST analyses: $(S_{rep}^2 + S_{het}^2 + S_{anl}^2)^{0.5}$
NAV	NIST Assigned Value = $(Median_{part} + Mean_{NIST})/2$ for analytes reported by NIST analyst(s) = $Median_{part}$ for analytes reported by ≥ 10 labs but not NIST
NAU	NIST Assigned Uncertainty: $(S^2 + S_{btw}^2)^{0.5}$ S is the maximum of (0.05*NAV, SD, S_{NIST}, eSD) and S_{btw} is the standard deviation between $Median_{part}$ and $Mean_{NIST}$. The expected long-term SD, eSD, is defined in: Duewer, et al. Anal Chem 1997;69(7):1406-1413.
-	Not analyzed
nd	Not detected (i.e., no detectable peak for analyte)
nq	Detected but not quantitatively determined
<x	Concentration greater than x
!	Non-quantitative value: heterogeneous serum, damaged sample, malfunction, etc.
italics	Not explicitly reported but calculated by NIST from reported values

Round Robin XLVIII Laboratory Results

Comparability Summary

Lab	TR	aT	gT	bC	tbC	aC	TLy	TbX	TLu	TZ	L&Z
FSV-BA	1	1	1	1	1	1		2	1	1	1
FSV-BB	1	1	1	1	1	1	1	1	1	2	2
FSV-BD	1	1									
FSV-BE	1	1	1	4							1
FSV-BF	1	1	1	2		1	1	1			
FSV-BG	1	1	1	1		1	1	1			1
FSV-BH	1	1	1	1	1	1	1	1	1	2	1
FSV-BI	1	1	1	1		1	1	1	1	1	1
FSV-BJ	1	1	1	2		1	2	1		3	1
FSV-BK	1	1							1	1	1
FSV-BL	1	2									
FSV-BM	1	1							1		
FSV-BN	2	1	3	1	1	1	1	1			
FSV-BO	2	1		3	3	4	1	1			1
FSV-BP	1	3		1		1	2	2			
FSV-BQ	1	1									
FSV-BR	1	2									1
FSV-BT	2	1	2	1	1	1	1	1	2	1	2
FSV-BU	2	2	2	2		2	2	3	1	1	1
FSV-BV	2	2	1	1		1	1	2			1
FSV-BW	1	1	2	1		1	1		2	2	2
FSV-BX	1	1	1	1	1	1		1	4	4	4
FSV-CB	2	1		2		1	2	2	2	1	1
FSV-CC	1	1									
FSV-CD	1	1	1	1		1	1	1			
FSV-CE	3	4		2							1
FSV-CF	2	2									
FSV-CG	3	2	1	1	1	1	1	1			
FSV-CH	1	1	3	1		1	1				1
FSV-CI	1	2	1								
FSV-CK	3	3	2	3		1	2	1			1
FSV-CL	1	3	4	2		1	2	1			1
FSV-CN	1	1	1	1	1	1	1	1	1	1	1
FSV-CR	2	2									
FSV-CS	1	1	1	1	1	2	1	1			1
FSV-CU	1	1									1
FSV-CV	4	1	1	2			1				
FSV-CW	2	2	2	1	1	1	1	1			
FSV-CX	2	2		2	1	1		1			
FSV-DA	1	1	1	2	3	1	1	1			1
FSV-DB	1	1		1			1	1			
FSV-DF	2										
FSV-DI	1	1	1	1	1				4	2	4
FSV-DK	1	2	2	1		1					2
FSV-DP	1										
FSV-DQ		3	3	2		2	2	3			
FSV-DR	1	1		2					2	1	1
FSV-DU	3	4		3	3						
FSV-EM	2	2		2		1	1	1			
FSV-EQ	1	2		4	4	4	4	1			2
FSV-ES	1	1		1	1	1	1	1	1		
FSV-FH	3	1									
FSV-FJ	4	2	3	2		1					
FSV-FT	2	2							1	1	1
FSV-FW	2										
NISTa	1	1	1	1	1	1		1	1	1	1
NISTb	1	1	1	1	1	1	1	1	1	1	1
n	56	54	31	41	19	34	30	31	18	17	31

	TR	aT	g/bT	bC	tbC	aC	TLy	TbX	TLu	TZ	L&Z
% 1	63	61	65	56	79	85	73	81	67	65	77
% 2	25	28	19	32	0	9	23	13	22	24	16
% 3	9	7	13	7	16	0	0	6	0	6	0
% 4	4	4	3	5	5	6	3	0	11	6	6

Label	Definition
Lab	Participant code
TR	Total Retinol
aT	α-Tocopherol
gT	γ/β-Tocopherol
bC	Total β-Carotene
tbC	trans-β-Carotene
aC	Total α-Carotene
TLy	Total Lycopene
TbX	Total β-Cryptoxanthin
TLu	Total Lutein
TZ	Total Zeaxanthin
L&Z	Total Lutein & Zeaxanthin

n	number of participants providing quantitative data
% 1	Percent of CS = 1 (within 1 SD of medians)
% 2	Percent of CS = 2 (within 2 SD of medians)
% 3	Percent of CS = 3 (within 3 SD of medians)
% 4	Percent of CS = 4 (3 or more SD from medians)

"Comparability Score"

The Comparability Score (CS) summarizes your measurement performance for a given analyte relative to the consensus medians in this study. CS is the average distance (in units of standard deviation) of your measurement performance characteristics from the consensus performance. CS is calculated when the number of quantitative values you reported N_{you}, is at least two and at least six participants reported quantitative values for the analyte.

We define CS as follows:

$$CS = \text{MINIMUM}\left(4, \text{INTEGER}\left(1+\sqrt{C^2+AP^2}\right)\right)$$

$$C = \text{Concordance} = \frac{\sum_{i=1}^{N_{you}} \frac{You_i - Median_i}{NAU_i}}{N_{you}}$$

$$AP = \text{Apparent Precision} = \sqrt{\frac{\sum_{i=1}^{N_{you}} \left(\frac{You_i - Median_i}{NAU_i}\right)^2}{N_{you}-1}}$$

NAU = NIST Assigned Uncertainty

For further details, please see

Duewer DL, Kline MC, Sharpless KE, Brown Thomas J, Gar KT. Micronutrients Measurement Quality Assurance Program: Helping participants use interlaboratory comparisc exercise results to improve their long-term measurement performance. Anal Chem 1999;71(9):1870-8.

Appendix H. Representative "Individualized Report" for RR48

Each participant in RR48 received an "Individualized Report" reflecting their reported results. Each report included a detailed analysis of the results they reported for the following analytes:

- Total Retinol
- *trans*-Retinol
- Retinyl Palmitate
- α-Tocopherol
- γ-Tocopherol
- δ-Tocopherol
- Total β-Carotene
- *trans*-β-Carotene
- Total *cis*-β-Carotene
- Total α-Carotene
- Total Lycopene
- *trans*-Lycopene
- Total β-Cryptoxanthin
- Total α-Cryptoxanthin
- Total Lutein
- Total Zeaxanthin
- Total Lutein & Zeaxanthin

The following twelve pages are the "Individualized Report" for the analytes evaluated by participant FSV-BA.

Individualized Round Robin XLVIII Report: FSV-BA

Summary

	Serum 264			Serum 265			Serum 266			Serum 267			Serum 268		
Analyte	You	NAV	n	You	NAV	n	You	NAV	n	You	NAV	n	You	NAV	n
Total Retinol	0.504	0.486	44	0.628	0.599	44	0.457	0.449	44	0.687	0.682	44	0.673	0.645	44
Retinyl Palmitate	0.10	0.08	15	0.2	0.2	15	0.1	0.1	15	nd	0.03	11	0.23	0.23	15
α-Tocopherol	16.37	16.82	52	15.45	14.99	52	6.92	6.93	52	6.37	6.30	52	16.42	15.26	52
γ/β-Tocopherol	1.482	1.574	29	1.921	1.846	29	1.876	1.897	29	2.401	2.401	29	2.048	1.909	29
Total β-Carotene	0.419	0.422	33	0.276	0.266	33	0.326	0.317	33	0.515	0.512	33	0.302	0.268	33
trans-β-Carotene	0.385	0.377	17	0.255	0.237	17	0.300	0.293	17	0.491	0.466	17	0.281	0.246	17
Total cis-β-Carotene	0.034	0.031	11	0.021	0.018	11	0.025	0.020	11	0.024	0.024	11	0.021	0.021	11
Total α-Carotene	0.096	0.099	29	0.102	0.102	29	0.030	0.032	29	0.020	0.020	28	0.112	0.106	29
Total Lycopene	!0.544	0.429	29	!0.39	0.300	29	!0.465	0.332	29	!0.313	0.235	29	!0.426	0.319	29
trans-Lycopene	0.229	0.200	12	0.159	0.142	12	0.187	0.165	12	0.155	0.135	12	0.176	0.155	12
Total β-Cryptoxanthin	0.059	0.034	29	0.070	0.059	29	0.083	0.056	29	0.078	0.056	29	0.078	0.063	29
Total Lutein&Zeaxanthin	0.122	0.106	29	0.23	0.21	29	0.12	0.11	29	0.150	0.129	29	0.26	0.22	29

H2

You : Your reported values for the listed analytes (micrograms/milliliter)
NAV : NIST Assigned Values, equal to (NIST's average-of-averages + this RR's median) / 2
n : Number of non-NIST laboratories reporting quantitative values for this analyte in this serum
* : Non-quantitative value: heterogeneous serum, damaged sample, procedural error, etc.

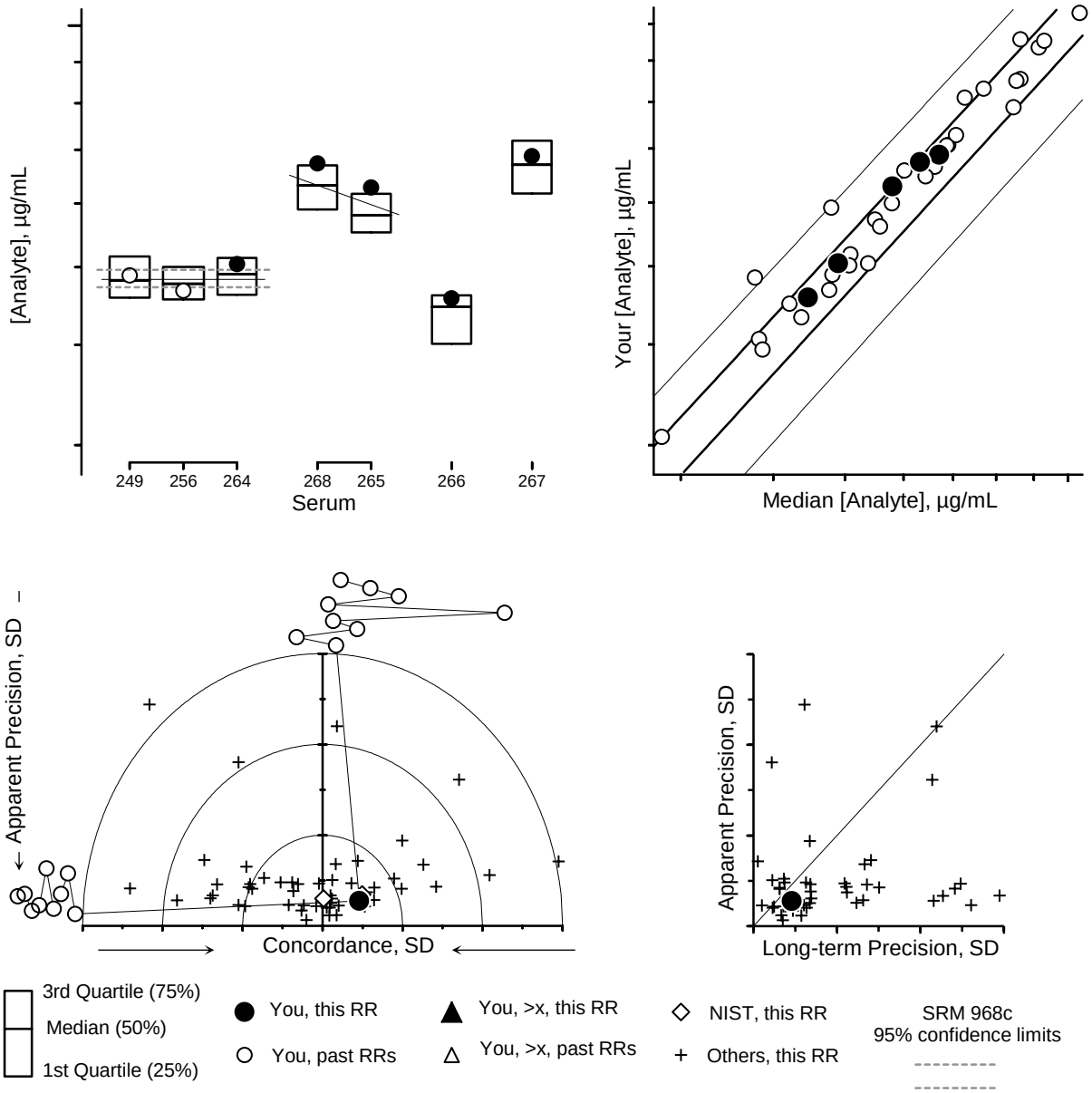
Please check our records against your records. Send corrections and/or updates to...

Micronutrients Measurement Quality Assurance Program
National Institute of Standards and Technology
100 Bureau Drive Stop 8392
Gaithersburg, MD 20899-8392 USA

Tel: (301) 975-3935
Fax: (301) 977-0685
Email: david.duewer@nist.gov

Individualized RR XLVIII Report: FSV-BA

Total Retinol

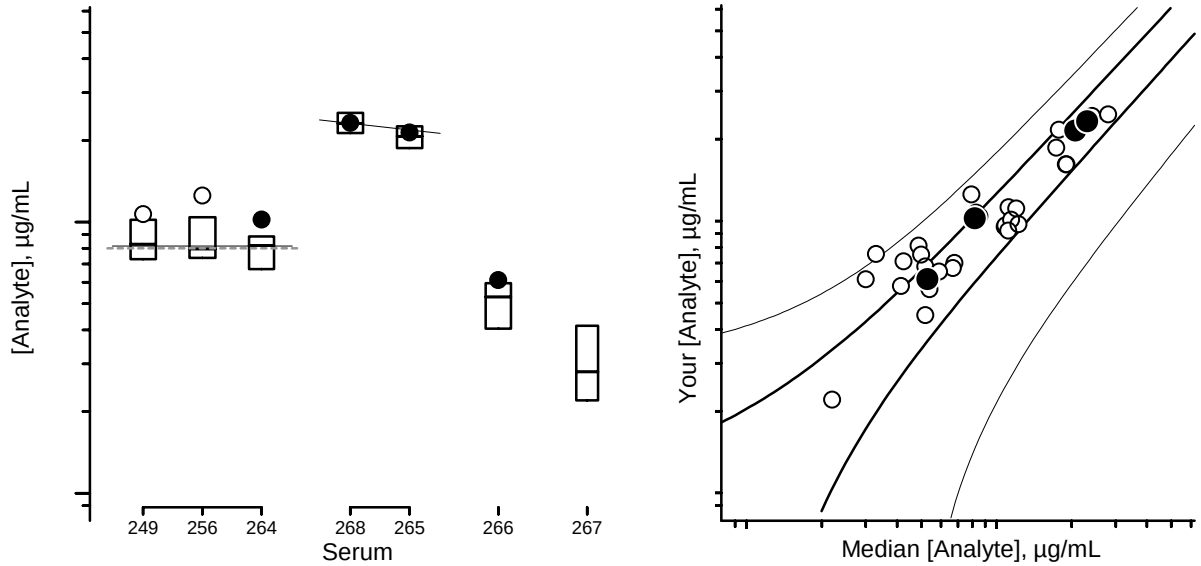


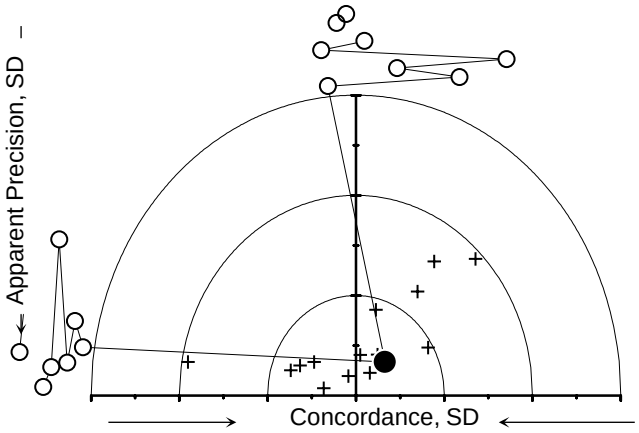
For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

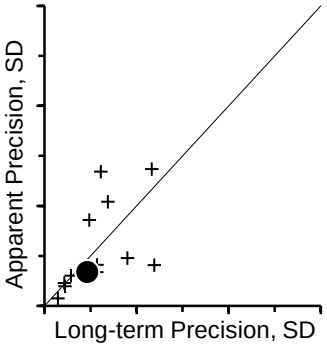
Serum	History	Comments
#264	#249 in RR44 (9/98), #256 in RR46 (9/99)	SRM 968c, Level II
#265		Lyophilized, same pool as #268
#266		Lyophilized
#267		Fresh frozen
#268		Fresh frozen, same pool as #265

Individualized RR XLVIII Report: FSV-BA

Retinyl Palmitate







3rd Quartile (75%)
Median (50%)
1st Quartile (25%)

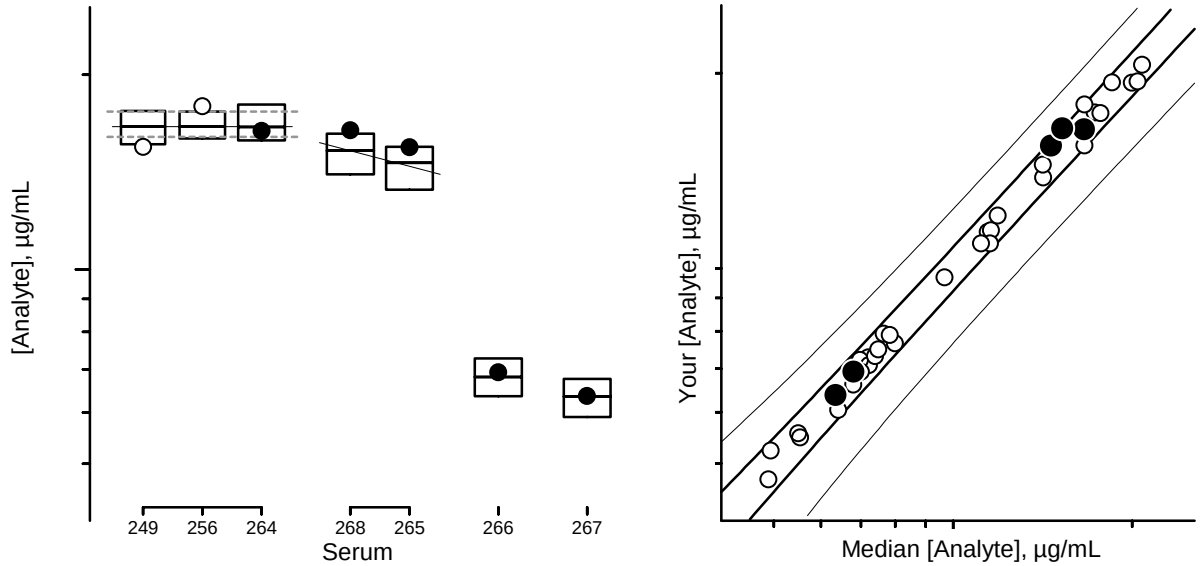
You, this RR
You, past RRs
You, >x, this RR
You, >x, past RRs
NIST, this RR
Others, this RR
SRM 968c
95% confidence limits

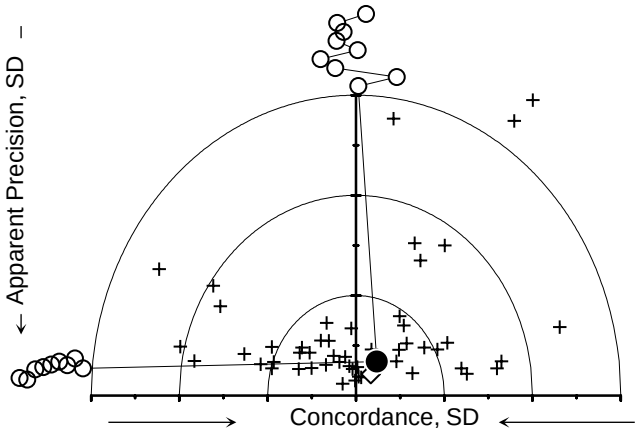
For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

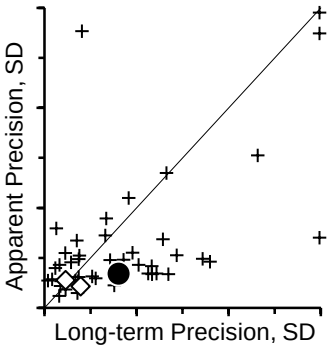
Serum	History	Comments
#264	#249 in RR44 (9/98), #256 in RR46 (9/99)	SRM 968c, Level II
#265		Lyophilized, same pool as #268
#266		Lyophilized
#267		Fresh frozen
#268		Fresh frozen, same pool as #265

Individualized RR XLVIII Report: FSV-BA

α-Tocopherol







3rd Quartile (75%)
Median (50%)
1st Quartile (25%)

● You, this RR
○ You, past RRs
▲ You, >x, this RR
△ You, >x, past RRs
◇ NIST, this RR
+ Others, this RR

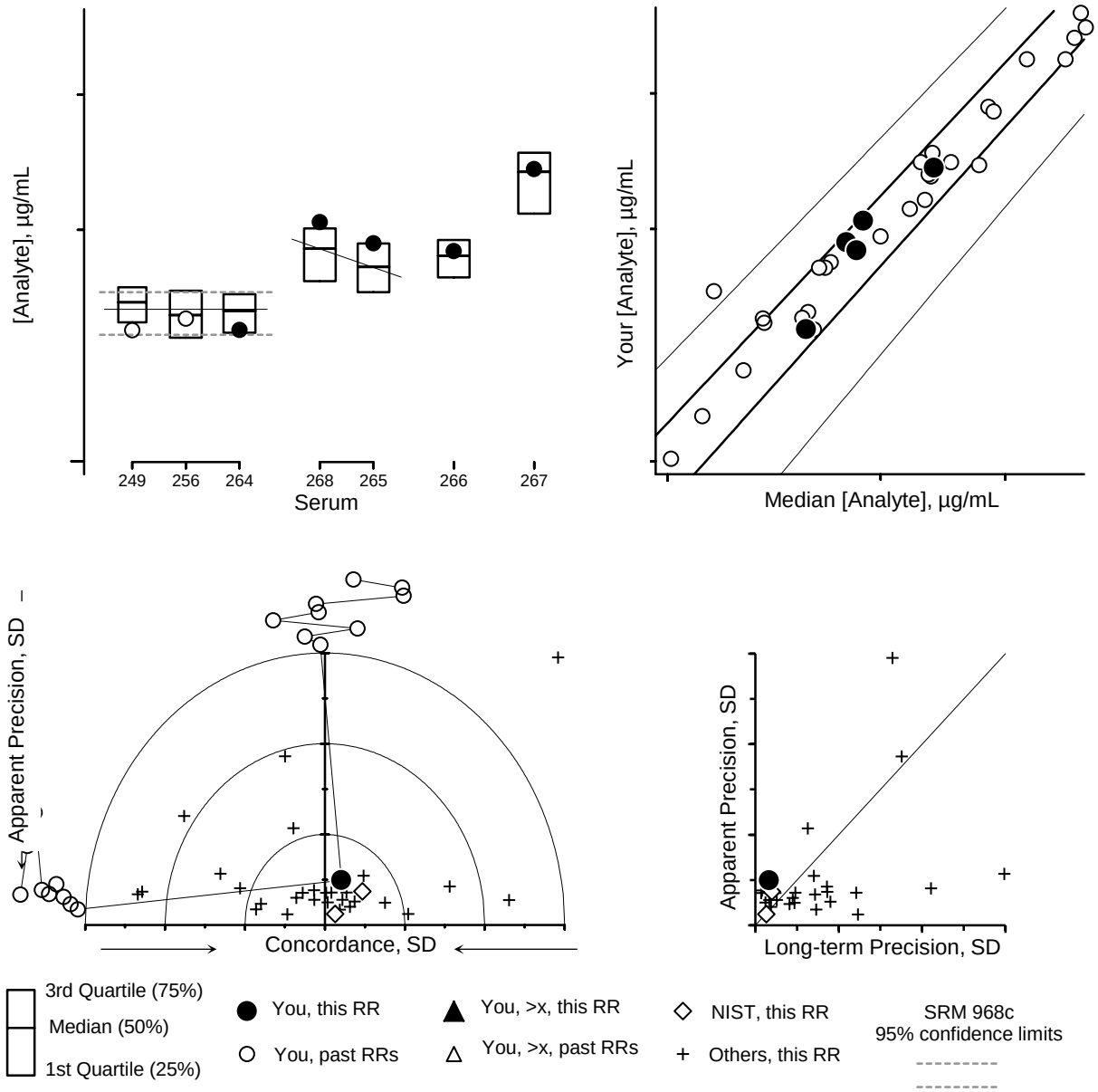
SRM 968c
95% confidence limits

For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

Serum	History	Comments
#264	#249 in RR44 (9/98), #256 in RR46 (9/99)	SRM 968c, Level II
#265		Lyophilized, same pool as #268
#266		Lyophilized
#267		Fresh frozen
#268		Fresh frozen, same pool as #265

Individualized RR XLVIII Report: FSV-BA

γ/β-Tocopherol

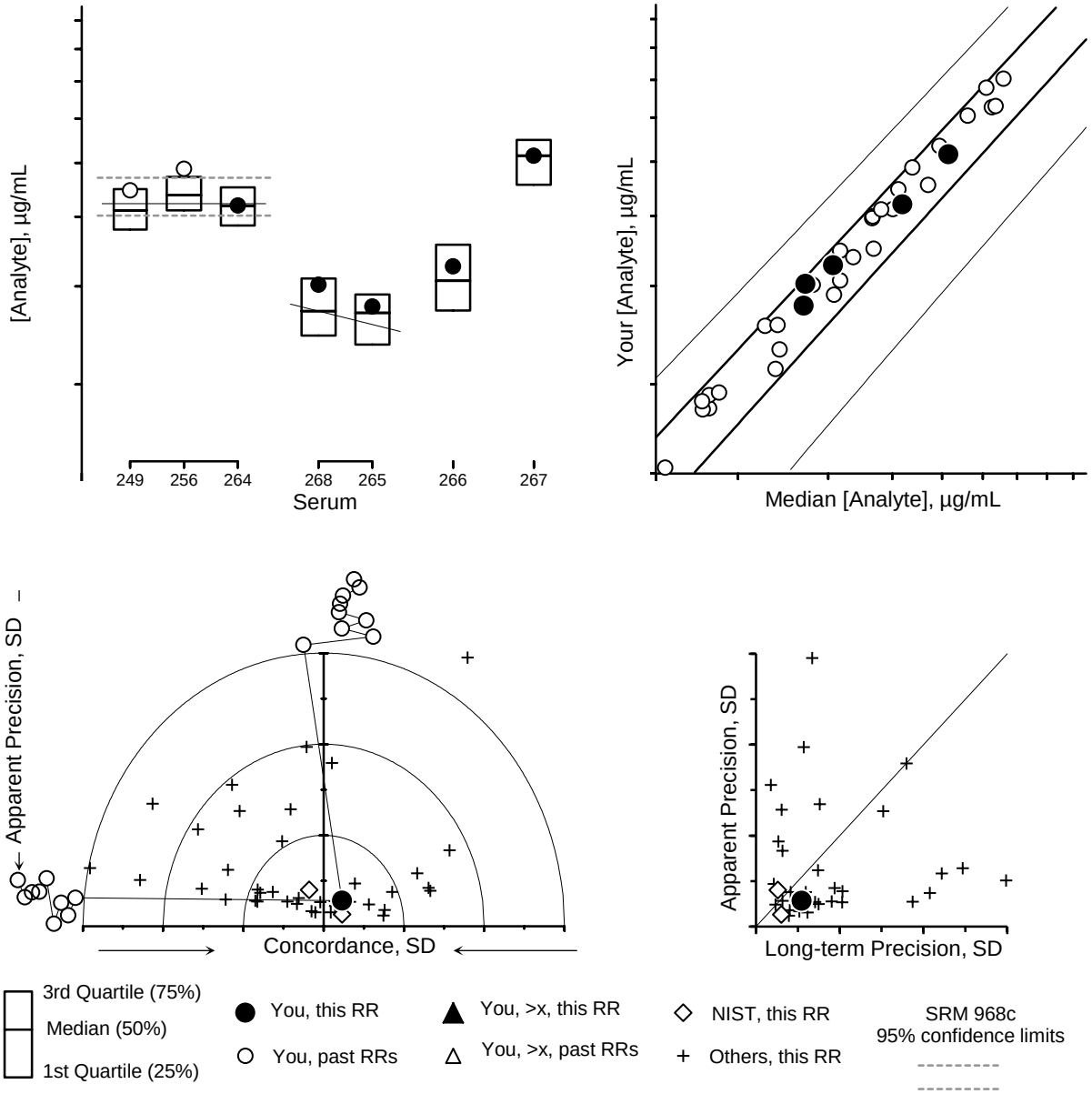


For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

Serum	History	Comments
#264	#249 in RR44 (9/98), #256 in RR46 (9/99)	SRM 968c, Level II
#265		Lyophilized, same pool as #268
#266		Lyophilized
#267		Fresh frozen
#268		Fresh frozen, same pool as #265

Individualized RR XLVIII Report: FSV-BA

Total β-Carotene

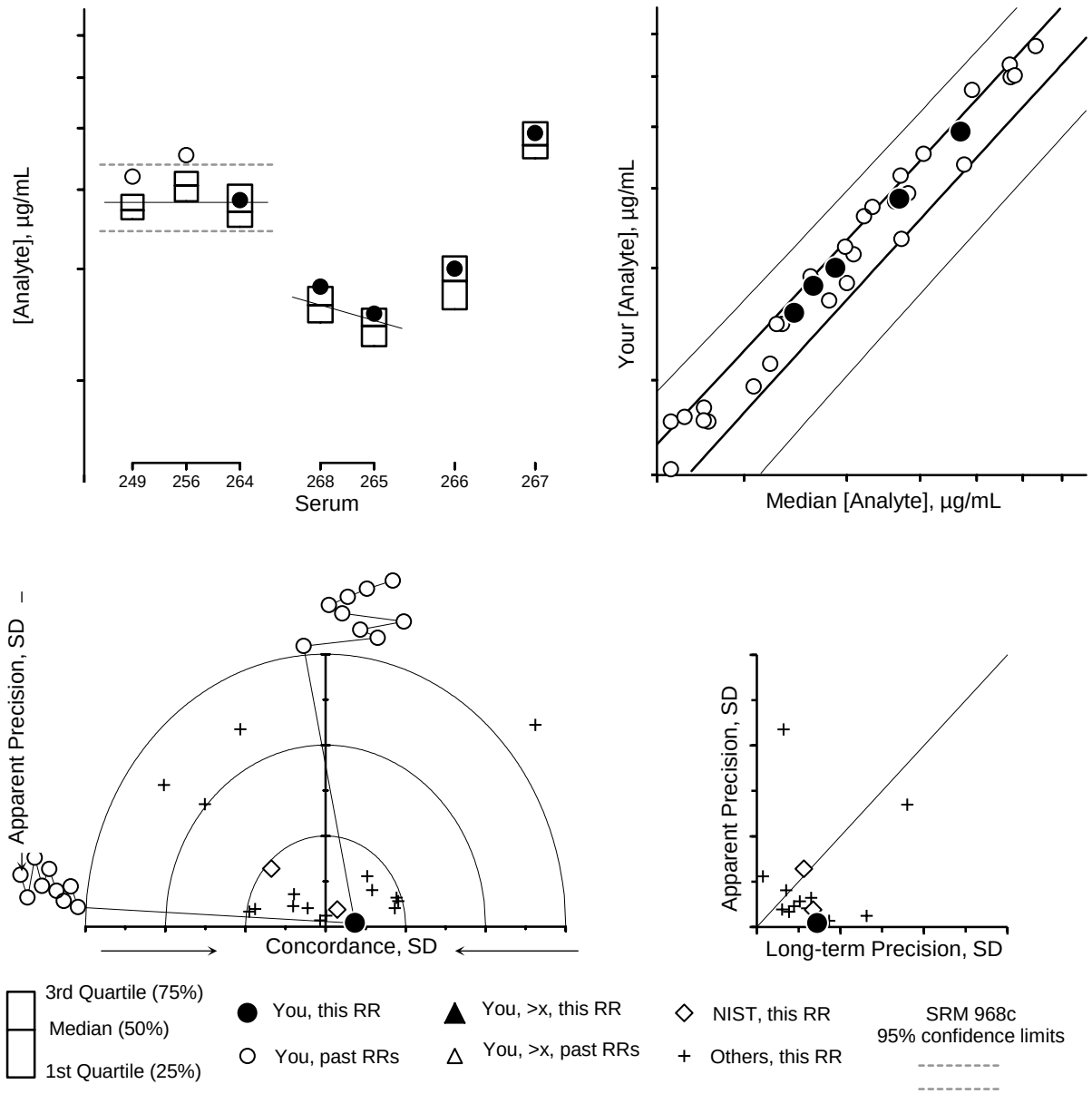


For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

Serum	History	Comments
#264	#249 in RR44 (9/98), #256 in RR46 (9/99)	SRM 968c, Level II
#265		Lyophilized, same pool as #268
#266		Lyophilized
#267		Fresh frozen
#268		Fresh frozen, same pool as #265

Individualized RR XLVIII Report: FSV-BA

trans-β-Carotene

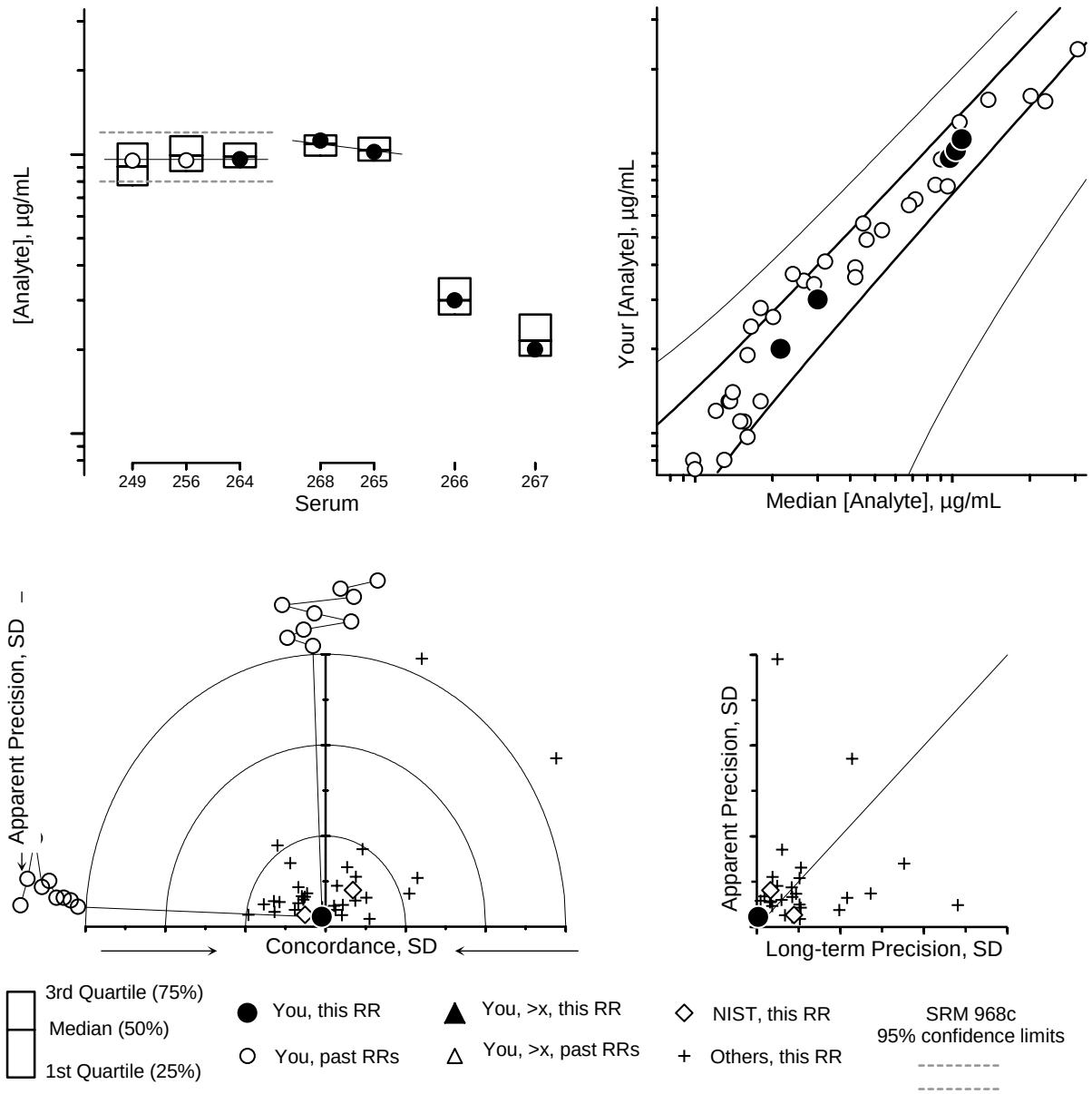


For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

Serum	History	Comments
#264	#249 in RR44 (9/98), #256 in RR46 (9/99)	SRM 968c, Level II
#265		Lyophilized, same pool as #268
#266		Lyophilized
#267		Fresh frozen
#268		Fresh frozen, same pool as #265

Individualized RR XLVIII Report: FSV-BA

Total α-Carotene

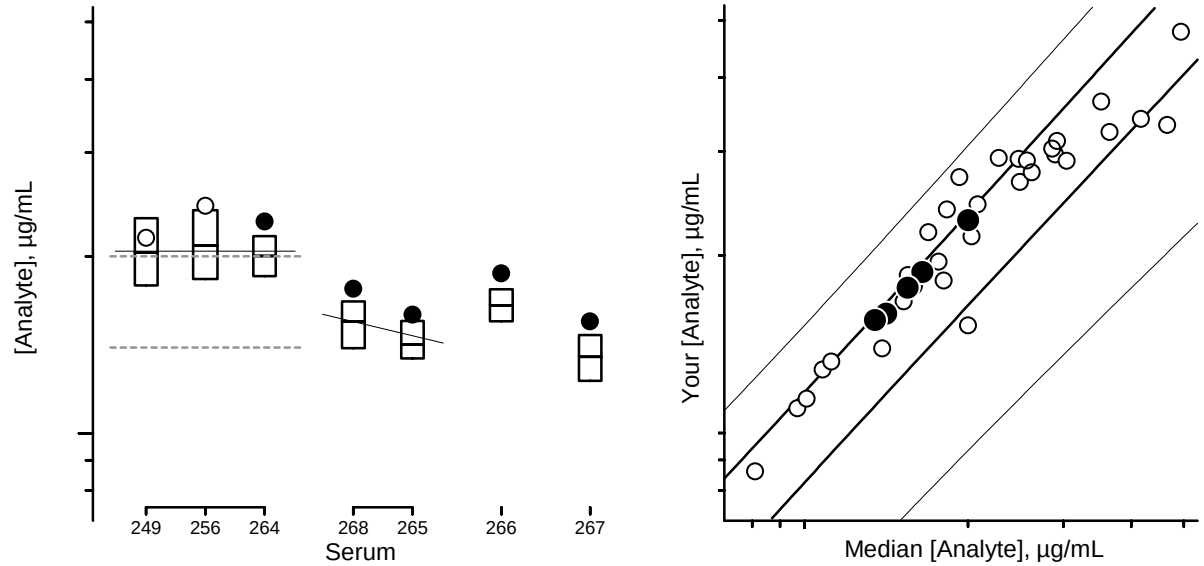


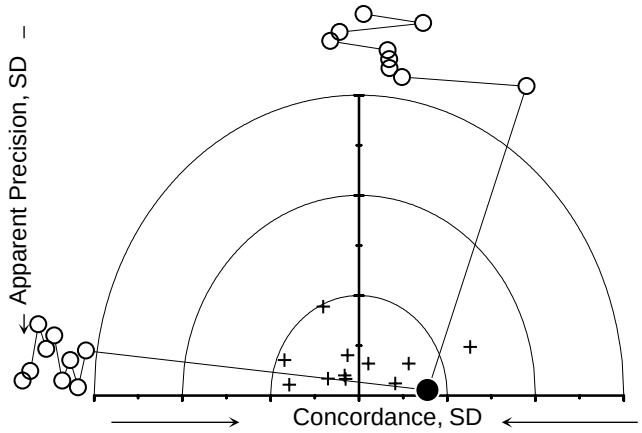
For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

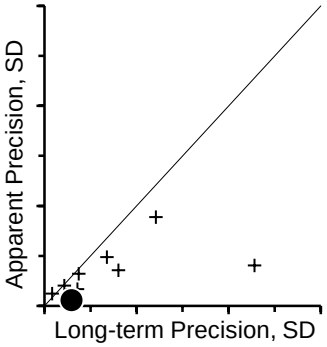
Serum	History	Comments
#264	#249 in RR44 (9/98), #256 in RR46 (9/99)	SRM 968c, Level II
#265		Lyophilized, same pool as #268
#266		Lyophilized
#267		Fresh frozen
#268		Fresh frozen, same pool as #265

Individualized RR XLVIII Report: FSV-BA

trans-Lycopene







3rd Quartile (75%)
Median (50%)
1st Quartile (25%)

● You, this RR
○ You, past RRs
▲ You, >x, this RR
△ You, >x, past RRs
◇ NIST, this RR
+ Others, this RR

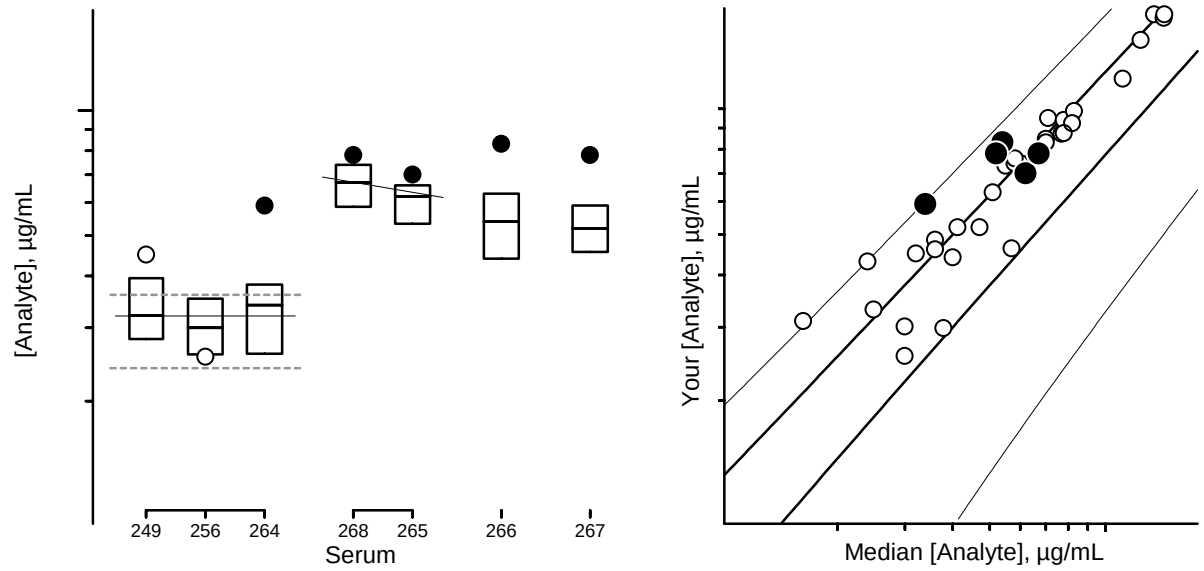
SRM 968c
95% confidence limits

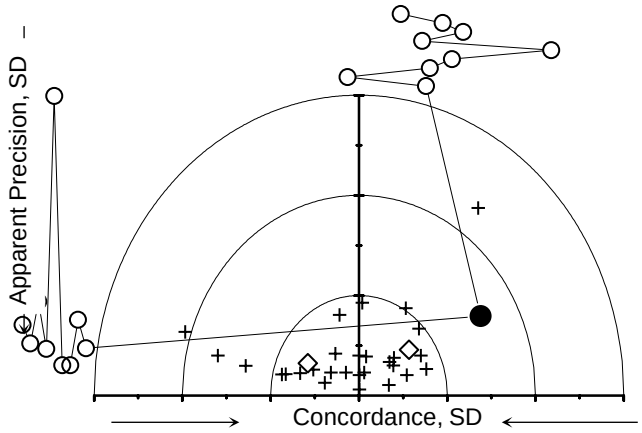
For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

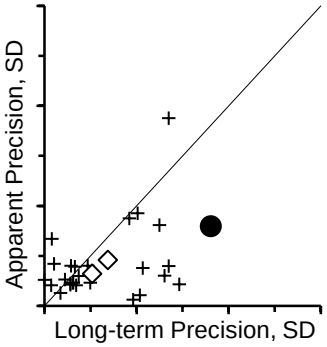
Serum	History	Comments
#264	#249 in RR44 (9/98), #256 in RR46 (9/99)	SRM 968c, Level II
#265		Lyophilized, same pool as #268
#266		Lyophilized
#267		Fresh frozen
#268		Fresh frozen, same pool as #265

Individualized RR XLVIII Report: FSV-BA

Total β-Cryptoxanthin







3rd Quartile (75%)
Median (50%)
1st Quartile (25%)

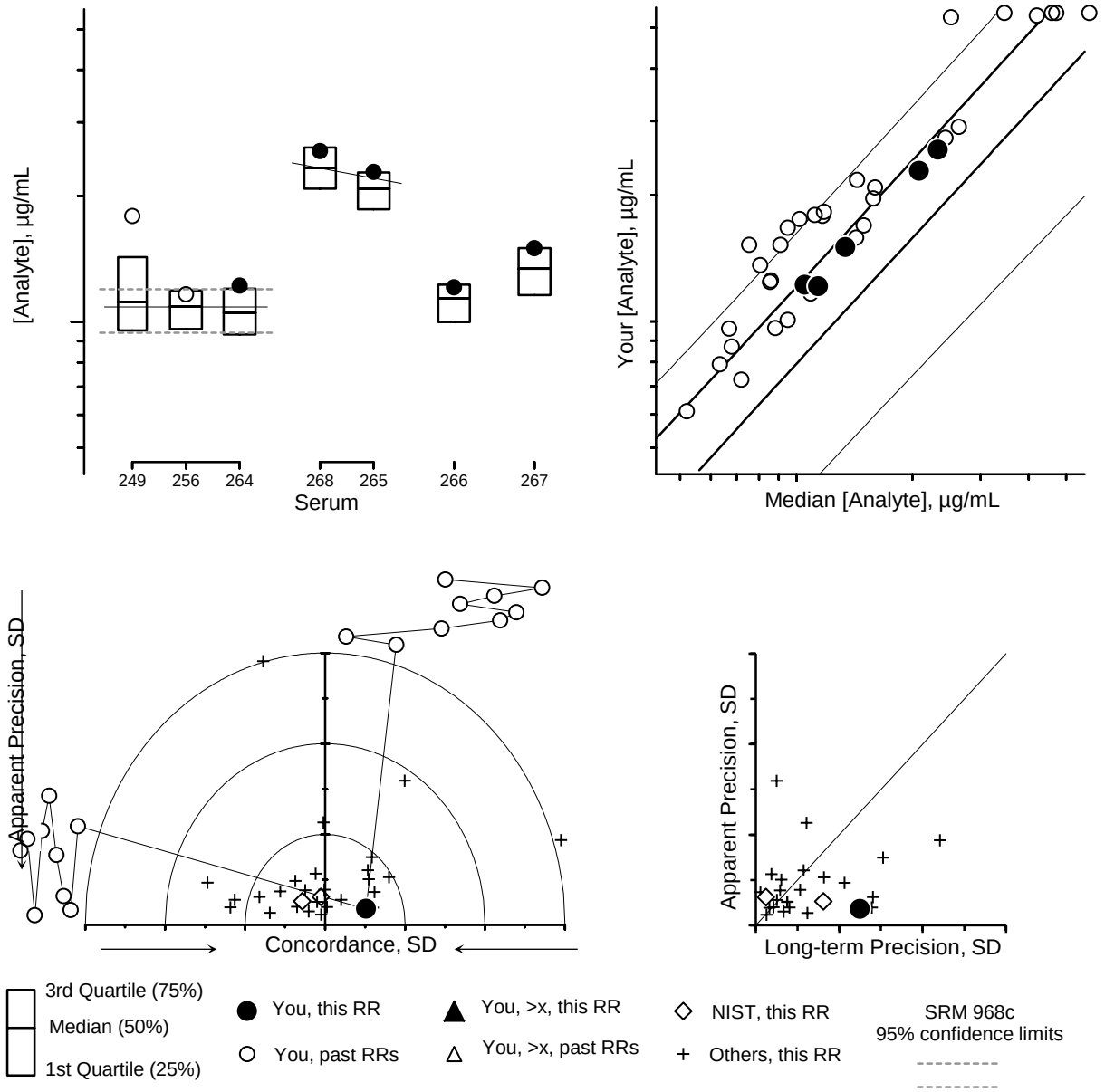
You, this RR
You, past RRs
You, >x, this RR
You, >x, past RRs
NIST, this RR
Others, this RR
SRM 968c
95% confidence limits

For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

Serum	History	Comments
#264	#249 in RR44 (9/98), #256 in RR46 (9/99)	SRM 968c, Level II
#265		Lyophilized, same pool as #268
#266		Lyophilized
#267		Fresh frozen
#268		Fresh frozen, same pool as #265

Individualized RR XLVIII Report: FSV-BA

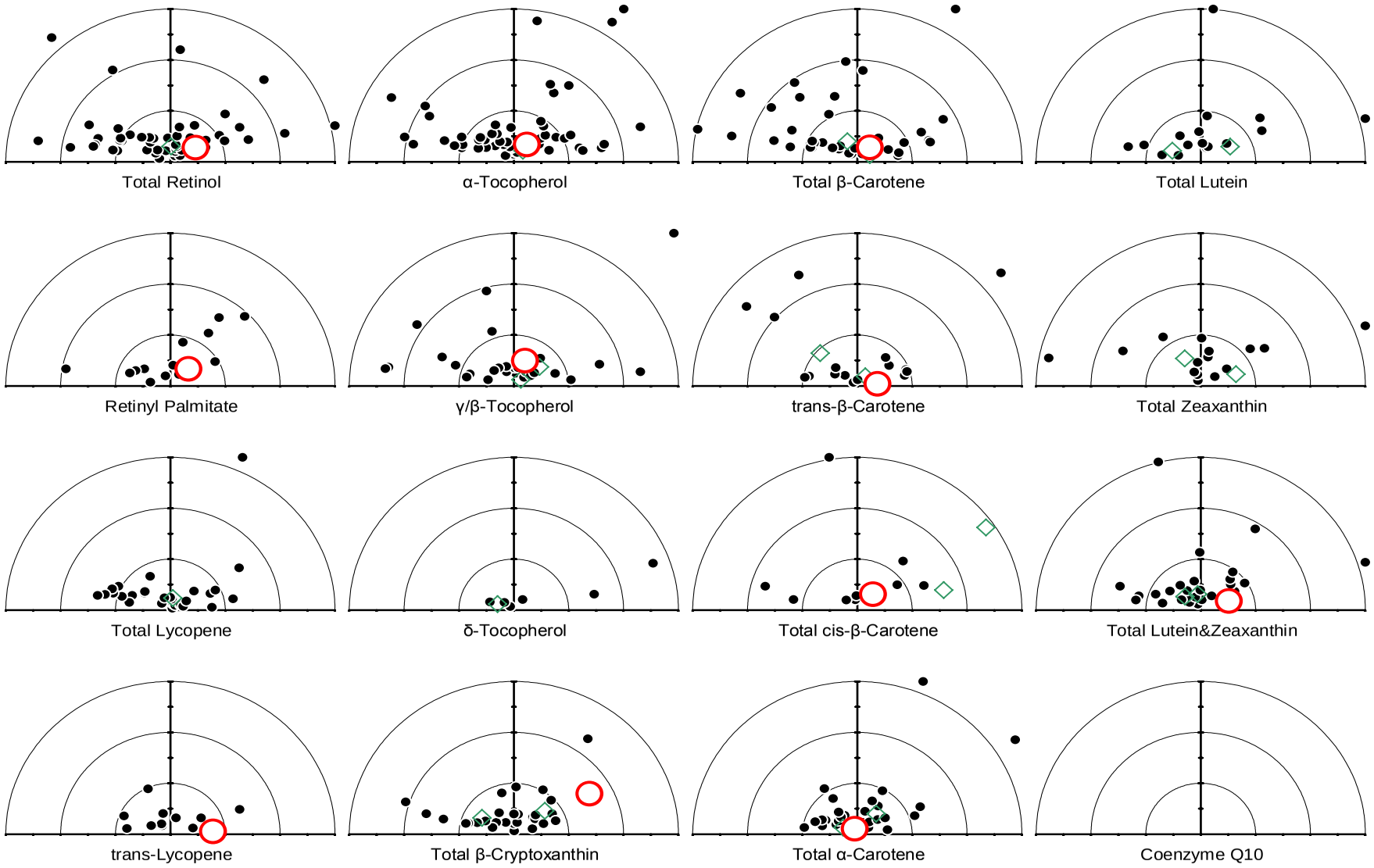
Total Lutein&Zeaxanthin



For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

Serum	History	Comments
#264	#249 in RR44 (9/98), #256 in RR46 (9/99)	SRM 968c, Level II
#265		Lyophilized, same pool as #268
#266		Lyophilized
#267		Fresh frozen
#268		Fresh frozen, same pool as #265

Individualized Round Robin XLVIII Report: FSV-BA
Graphical Comparability Summary



H13

Appendix I. Shipping Package Inserts for RR13

The following four items were included in each package shipped to an RR13 participant:

- Cover letter
- Protocol for Analyzing Samples
- Preparation of Stock Solution and Diluted Solution Datasheet
- Control and Test Samples Datasheet

The cover letter, preparation protocol, and the two datasheets were enclosed in a sealed waterproof bag along with the samples themselves.

UNITED STATES DEPARTMENT OF COMMERCE
National Institute of Standards and Technology
Gaithersburg, Maryland 20899-

June 13, 2000

Dear Colleague:

For the past 15 years the National Institute of Standards and Technology (NIST) has coordinated a Micronutrients Measurement Quality Assurance (QA) Program for laboratories making vitamin measurements in human serum. Frozen and/or freeze dried sera are sent to laboratories for analysis as an interlaboratory comparison exercise. Results are returned to NIST for data tabulation and evaluation. Value-assignment of the sample pools is based on the median of all the laboratory results, with confirmation based on measurements at NIST. We provide consultation and trouble-shooting regarding methods of analysis, and a certificate of participation in the QA program is issued at the end of each calendar year. We also host a micronutrient analysis QA workshop for fat-soluble vitamin, carotenoid, and ascorbic acid measurements in serum.

The enclosed set of samples constitute the round robin exercise for vitamin C (Round Robin XIII) for 2000. Four vials of frozen serum (*test samples),* and a vial of solid ascorbic acid (*a control sample),* are enclosed. Please follow the attached protocol when you analyze these samples.

Report your results using the attached form by **July 14, 2000**. We also request that you send us a representative chromatogram from the analysis of each sample and indicate whether peak height or peak area was used in the calculation of the ascorbic acid concentration. Your results will be kept confidential. Results received two weeks after the due date will not be included in the summary report of this round robin study. The summary report concerning this study will be provided near the end of June.

Please mail your results to:

Micronutrients Measurement Quality Assurance Program
NIST
100 Bureau Drive, Stop 8392
Gaithersburg, MD 20899-8392

or Fax: 301.977.0685 E-mail: sam.margolis@nist.gov

If you have any questions or concerns please call me at 301.975.3137 or contact me by Fax or E-mail.

Thank you for your participation and we look forward to receiving your results.

Sincerely,

Sam A. Margolis

Sam A. Margolis, Ph.D.
Research Chemist
Analytical Chemistry Division
Chemical Science and Technology Laboratory

Enclosures

NIST

Protocol for analyzing samples

The *control sample* consists of a sample of solid ascorbic acid in an amber vial and should be used in the following manner (please record your weights on the attached report form):

1. Prepare 250 mL of 5% metaphosphoric acid (MPA) in distilled water.

2. Weigh out **180-220 mg** of the solid ascorbic acid sample to 0.1 mg (if possible) and dissolve it in 100 mL of 5% MPA using a 100 mL volumetric flask. **Weigh the amount of MPA solution that was added.** This will be referred to as the Stock Solution.

3. Dilute the Stock Solution by **weighing** 0.5 mL of the stock solution into a 100 mL volumetric flask. Then add 5% MPA solution to 100 mL and **weigh the amount of MPA solution that was added**.

4. Record the ultraviolet spectrum of the diluted solution against 5% MPA solution as the blank using paired cuvettes. Record the wavelength in the region of 240-245 nm at which you observe the maximum absorbance and record the absorbance at that wavelength.

5. Record the absorbance of the sample at 243 and 244 nm.

6. Measure the concentration of the ascorbic acid in the **dilute solution** in duplicate along with the ampuled Test Samples.

The purpose of measuring the absorbance at the wave-length maximum is to check the concentration of your sample. If your spectrophotometer is properly calibrated the maximum absorbance should be between 243 and 244 nm and if the concentration is correct the molar extinction coefficient ($E^{1\%}$) of ascorbic acid at this wavelength (using a cell with a 1 cm path length) should be close to 550 ± 30. The extinction coefficient of your solution can be calculated using the following equation:

$$E^{1\%} = \text{observed OD} \div \text{g AA/100 mL of the diluted solution}$$

In the last three collaborative studies a significant number of laboratories reported total ascorbic acid (AA) values that were above the AA level of the samples but were significantly lower than the values for the total AA. This would suggest that the dehydroascorbic acid (DHAA) was not completely reduced to AA. In order to evaluate this possibility, we are asking those who utilize either dithiothreitol (DTT) or homocysteine (HS) to reduce DHAA to AA to use one of the following protocols.

DTT Add 0.200 mL serum sample to 0.200 mL of a solution containing 0.5 mol/L K_2HPO_4 and 1 mg/mL DTT, mix, and incubate for 30 min at 21 °C. Add 0.160 mL of metaphosphoric acid (MPA) 400 g/L and assay by your method.

HS Add 0.200 mL serum sample to 0.200 mL of a solution containing 0.5 mol/L K_2HPO_4 and 10 mmol/L HS, mix, and incubate for 30 min at 21 °C. Add 0.160 mL of metaphosphoric acid (MPA) 400 g/L and assay by your method.

If you require larger samples increase the volumes but maintain the ratio of the sample to the reducing mixture.

The *test samples* are in sealed ampules and were prepared by adding equal volumes of 10% metaphosphoric acid to spiked human serum. We have checked the samples for stability and homogeneity and the total ascorbic acid appears to be sufficiently stable however, these samples contain some dehydroascorbic acid. The *test samples* should be defrosted by warming at 20 °C for not more than 10 min otherwise some irreversible oxidation of ascorbic acid may occur.

Each ampule should contain between **0** and **120** µmol of ascorbic acid/L of diluted serum and each ampule should be analyzed in duplicate by the method(s) used in your laboratory (preferably one measuring total ascorbic acid).

REPORT OF ANALYSIS

NAME:

ADDRESS:

Telephone no.:_____
Fax no.:_____

Method of Analysis:

Please attach representative chromatograms.

Method used for calculating ascorbic acid concentration.

Peak height _____ Peak area _____

Manufacturer of ascorbic acid used to make in-house standards _____

Were samples frozen upon receipt? _____ Yes _____ No

Date of Analysis:_____

PREPARATION OF STOCK SOLUTION AND DILUTED SOLUTIONS

STOCK SOLUTION

Weight of ascorbic acid in the Stock Solution _____ mg
Weight of 5% MPA added to the 100 mL volumetric flask _____ g

DILUTE SOLUTION

Weight of added stock solution (0.5 mL) _____ mg
Weight of 5% MPA added to the 100 mL volumetric flask _____ g

Absorbance of Dilute Solution 1 at **243 nm** _____
Absorbance of Dilute Solution 1 at **244 nm** _____

Wavelength of maximum absorbance _____ nm
Calculated molar absorptivity _____

COMMENTS: (use other side if necessary)

Mail by July 14, 2000 to:
Micronutrients Measurement Quality Program
NIST, 100 Bureau Drive, Stop 8392
Gaithersburg, MD 20899-8392

Fax: 301-977-0685 Micronutrients
E-mail: sam.margolis@nist.gov

REPORT OF ANALYSIS

RESULTS (µmol/L of Sample)

CONTROL SAMPLE 1

REPLICATE 1 _____µmol/L
REPLICATE 2 _____ µmol/L

TEST SAMPLE #1

REPLICATE 1 _____µmol/L
REPLICATE 2 _____ µmol/L

TEST SAMPLE #2

REPLICATE 1 _____µmol/L
REPLICATE 2 _____ µmol/L

TEST SAMPLE #3

REPLICATE 1 _____µmol/L
REPLICATE 2 _____ µmol/L

TEST SAMPLE #4

REPLICATE 1 _____µmol/L
REPLICATE 2 _____ µmol/L

Mail by July 14, 2000 to:
Micronutrients Measurement Quality Program
NIST, 100 Bureau Drive, Stop 8392
Gaithersburg, MD 20899-8392

Fax: 301-977-0685 Micronutrients
E-mail: sam.margolis@nist.gov

Appendix J. Final Report for RR13

The following ten pages are the final report as provided to all participants:

- Cover letter
- An information sheet that:
  - describes the contents of the “All-Lab” report
  - describes the content of the “Individualized” report
  - describes the nature of the test samples and details any previous distributions
  - summarizes aspects of the study that we believe may be of interest to the participants

December 4, 2000

«Name»
«Company»
«Address1»
«Address2»
«City», «State» «PostalCode»
«Country»

Dr. Margolis printed a separate cover letter for each RR13 participant. The words within the "«»" are parameters for a mail-merge macro routine.

Dear «Name»:

This report describes the results of Round Robin 13 (RR13) for the measurement of total ascorbic acid (TAA, ascorbic acid (AA) + dehydroascorbic acid (DHAA))) in human serum. Your results are designated as Lab. No. ~ in the accompanying table and figures of the "All laboratory" report. We also include an "Individualized" report of your own results that is very similar in format to that used in the fat-soluble Vitamin Quality Assurance Program (QAP).

RR13 involved the duplicate analyses of a solid ascorbic acid sample and four human serum samples:

S13:1, SRM 970 Level I, distributed as blind replicates in RRs 11 and 12
S13:2, SRM 970 Level II, distributed as blind replicates in RRs 11 and 12
S13:3, a 1:1 serum:metaphosphoric (MPA) material prepared 3/95 to have a 28.9 µmol/L TAA level
S13:4, a 1:1 serum:MPA material prepared 8/93 to have a 77.2 µmol/L TAA level.

Table 1 summarizes the data for these samples and for the control solution each participant prepared from the solid ascorbic acid sample. As in the fat-soluble QAP, the estimated standard deviations (eSD) for the measurements are defined as 0.74*(interquartile range) and the estimated coefficients of variation (eCV) are defined as 100*eSD/median. NIST data are not included in the statistical analyses.

Figures 1 through 4 detail the individual measurements reported by all participants. These plots suggest that many of the laboratories are not measuring all of the DHAA or that the AA is being degraded at some point during the assay procedure.

Figure 5 presents an analysis of the components of variation, using data from RR11 and RR12 as well as RR13. The average duplicability ($SD_{duplicability}$, within-sample within-RR) and repeatability ($SD_{repeatability}$, among-blind replicate within RR) represent the short-term precision typical of the vitamin QAP participants. The agreement between these two short-term precision estimates suggests both that participants are reporting "true" duplicates (independently processed aliquots from one sample) and that both levels of the

SRM are homogenous. The average reproducibility ($SD_{reproducibility}$, within-laboratory among RR) represents the long-term precision typical of the vitamin QAP participants. The interlaboratory variability ($SD_{interlaboratory}$) is somewhat larger than $SD_{reproducibility}$, reflecting among-participant discordances as well as within-participant imprecision. The dashed denotes Horwitz's predicted interlaboratory variance for generic analytes at these concentrations (Horwitz W, Britton P, Chirtel SJ. A simple method for evaluating data from an interlaboratory study. JAOAC International 1998;81(6):1257-1265.) The $SD_{interlaboratory}$ are about equal to Horwitz's values, suggesting that TAA is not an unusually difficult measurand. However, the difference between $SD_{interlaboratory}$ and $SD_{reproducibility}$ suggests that interlaboratory comparability can be improved.

Using the data that you submitted to us, we calculated the density (D_{MPA}) of your MPA solution. This value was low for one laboratory indicating a possible error in the preparation of the MPA solution. We also calculated the concentration of the TAA in the standard solution (AA_{cal}, Table 1) that each of you made and assumed that the error in concentration was no greater than the error in weighing solutions. The amount of TAA that you actually measured in your assay of the standard solution is listed in Table 1, AA_{obs}. To compare the measurements on the standard solutions, we calculated the ratio of the observed value to the calculated value. The median ratio was 0.97±0.06. If the estimated error in weighing and in filling the volumetric flasks is small (1 - 2%), then the major source of error is in the assay itself. Potential sources of such error include: 1) inaccurate volume delivery of sample standard and/or serum delivered to the assay mixture, 2) inaccurate standards (impurities and/or degradation), 3) inaccurate constants and/or calculations, and 4) instability of the AA during the preparation and analysis steps (e.g., in the glassware, vials etc).

We also asked each of you to measure the absorbance of your standard solution at 243 nm and 244 nm (AA in MPA exhibits its maximum absorbance within these wavelengths). Every laboratory obtained similar values at each wavelength indicating that the wavelength was correct. The median $E^{1\%}$ for a 1 cm cell was 565 AU/mole/cm, very close to the literature value of 560 AU/mole/cm at 243 nm. However, the range of results (526 AU/mole/cm to 613 AU/mole/cm) indicates a need for some laboratories to calibrate the absorbance scale of their spectrophotometers (e.g., NIST SRM 2031, Metal-on-Quartz Filters, Transmittance).

After the distribution of this set of samples, we made several significant observations that may affect the accuracy of the measurement of TAA. The first of these was the accidental observation that autosampler vials may catalyze the degradation of AA. In many vials we observed significant degradation (7%) within 4 hours. This occurs to some extent in all vials as well as other tubes used for sample handling. We believe that the variation in the composition of the glassware may account for a significant amount of interlaboratory variation and low values. It may also account for the formation of DHAA acid during the processing of blood samples. A manuscript describing several cleaning procedures to remedy this problem is in preparation and will be provided at a later date.

We also observed a significant **increase** in the AA levels in sample S13:3 between the time we distributed the samples to most participants and the time we assayed the samples in our laboratory. Two laboratories (VC-MR and VC-NQ) received samples late and their results are similar to the results that we obtained. This event was not unique to a single sample but was characteristic of four sets of samples prepared in 1995; we have not observed any change in any samples prepared in 1989, 1993, or 1998. All samples are sealed in glass ampoules and have been continuously stored together at –70 °C. Samples from different years were assayed together and only the 1995 samples were high thus this was not a function of the assay method. We have no explanation as to the cause of this increase. However, all of the material in the AA peak was electrochemically active and treatment with ascorbic acid oxidase chemically modified all of the material in the AA peak. We do not use S13:3 results in any of the comparability calculations.

In a very recent study we observed that AA dissolved in 5% MPA is not as stable as AA dissolved in a solution of 75% acetonitrile, 25% water and 1 mg dithiothreitol (DTT)/mL. The AA in the MPA started degrading within 24 h (15% at the end of 24 h) whereas the DTT solution remained stable for 5 days.

In conclusion we can identify the following sources of systematic bias in the results reported by participating laboratories:

1) Pipetting of aqueous solutions (median 3-4% above expected value).
2) Weighing of samples 0.5-100 g (CV of 1 and 2% respectively).
3) Measurement of the concentration of a 50 μmol/L standard solution (CV of 9-10%). The median value is close to the expected value; therefore the error probably lies in the accuracy of the measurement of the sample or the values used for making your calculations.
4) Measurement of the serum TAA. This could either reflect inaccurate dispensing of the total sample to the assay because of the viscosity of the sample, inaccurate values used in calculating the TAA concentration, or incomplete reduction of the DHAA.
5) The variable degradation of TAA caused by the variation in the composition of the glassware or autosampler vials.
6) Degradation of the TAA in a 5% MPA solution

If your values differed from the medians by more than 2 eSDs (see target plot on your individual report) we suggest that you evaluate whether you accurately deliver the correct sample volume. This can vary either as a function of the sample viscosity or the accuracy of the pipette. We also suggest that you evaluate 1) the accuracy of the constants that you use in converting the assay results to a final AA concentration in the serum, 2) the completeness of the reduction of the DHAA, or 3) the possible oxidation of the AA to DHAA. If your results still differ significantly from the interlaboratory medians, you should reexamine your methods for possible systematic errors particularly degradation due to contaminated glassware (see enclosed preprint). If you need to validate your method, SRM 970 Ascorbic Acid in Frozen Human Serum is now available for purchase (http:/ts.nist.gov/ts/htdocs/230/232/232.htm).

If you have any questions concerning this report please contact Dave Duewer at david.duewer@nist,gov, or me at e-mail: sam.margolis@nist.gov; tel: 301/975-3137; fax: 301/977-0685.

Sincerely,

Sam A. Margolis, Ph.D.
Research Chemist
Analytical Chemistry Division
Chemical Science and Technology Laboratory

Enclosures

Figure Captions

Figure 1. Summary of the Results of Round Robin 13 for the Measurement of TAA in Sample 1. Each point represents an individual measurement. The solid line represent the NIST certified value for AA + DHAA.

Figure 2. Summary of the Results of Round Robin 13 for the Measurement of TAA in Sample 2. Each point represents an individual measurement. The solid line represent the NIST certified value for AA + DHAA.

Figure 3. Summary of the Results of Round Robin 13 for the Measurement of TAA in Sample 3. Each point represents an individual measurement. The solid line represents the NIST value for AA + DHAA (combined gravimetric and LC measurements) assigned at the time the samples were prepared.

Figure 4. Summary of the Results of Round Robin 13 for the Measurement of TAA in Sample 4. Each point represents an individual measurement. The solid lines represent the NIST value for AA + DHAA (combined gravimetric and LC measurements) assigned at the time the samples were prepared.

Figure 5. Summary of the Statistical Analysis of the Combined Results of Round Robins 11- 13.

Figure 1.

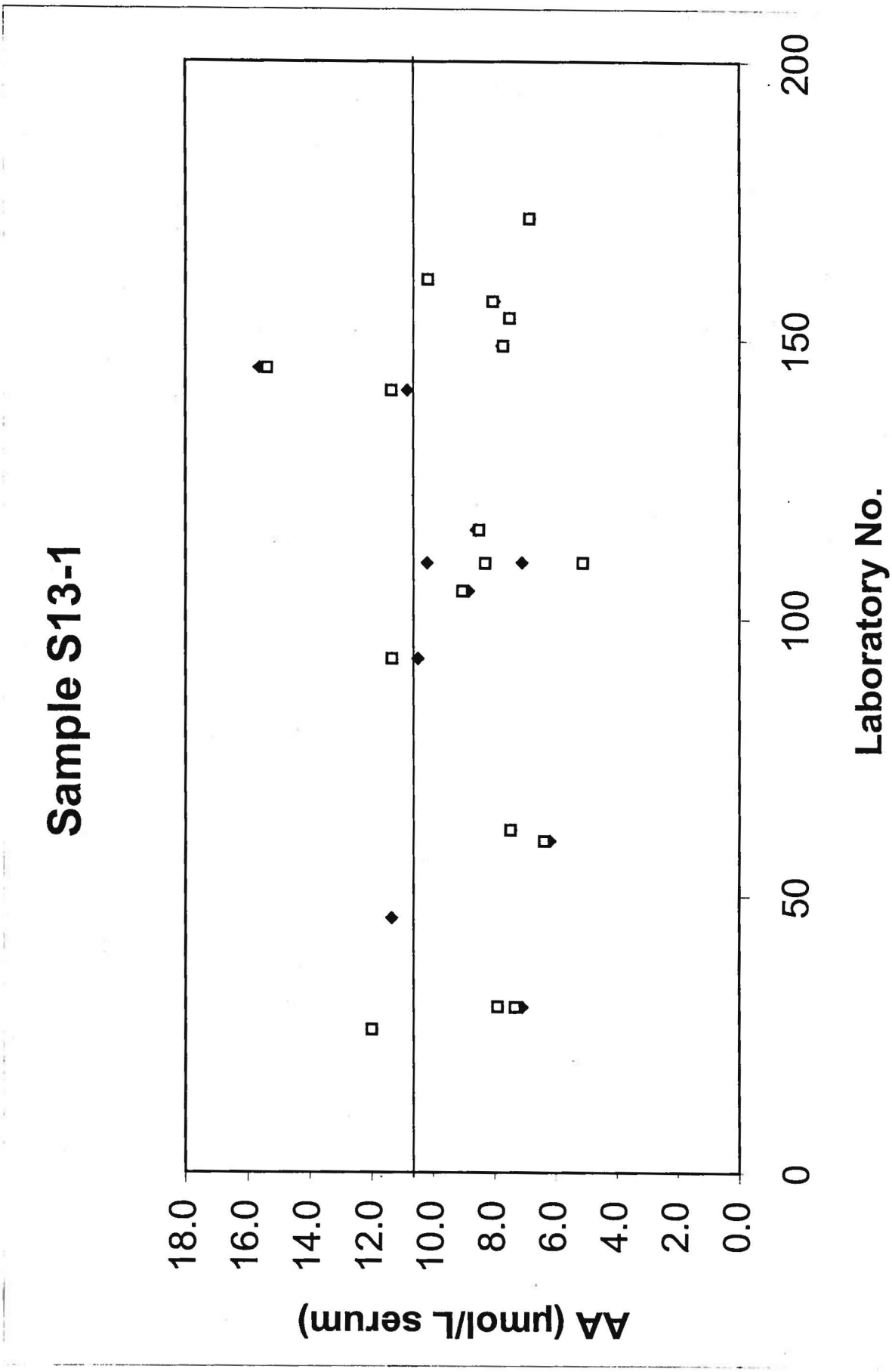

Sample S13-1
AA (µmol/L serum)
18.0
16.0
14.0
12.0
10.0
8.0
6.0
4.0
2.0
0.0
0
50
100
150
200
Laboratory No.

Figure 2.

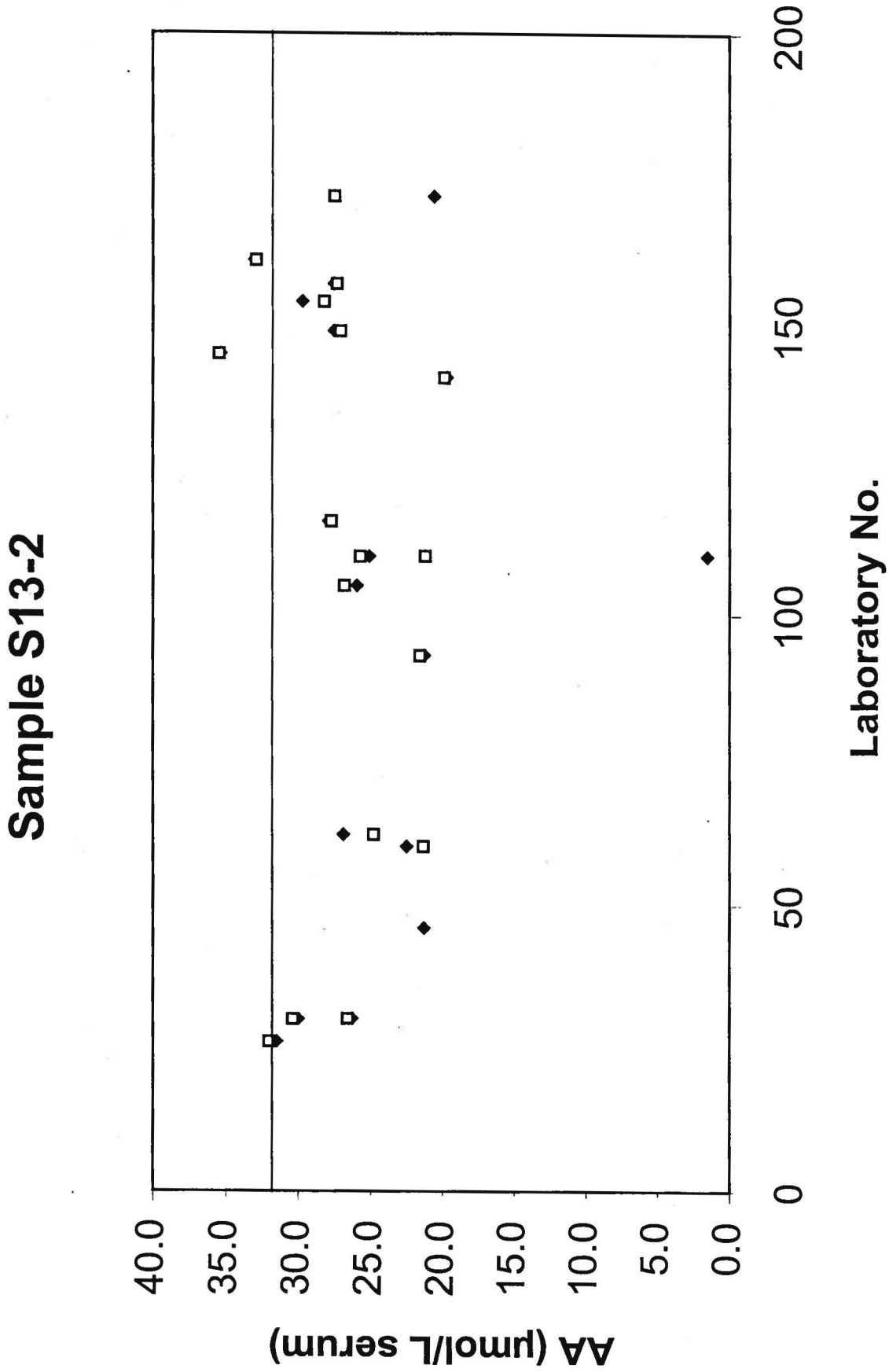

Sample S13-2
AA (µmol/L serum)
40.0
35.0
30.0
25.0
20.0
15.0
10.0
5.0
0.0
0
50
100
150
200
Laboratory No.

Figure 3.

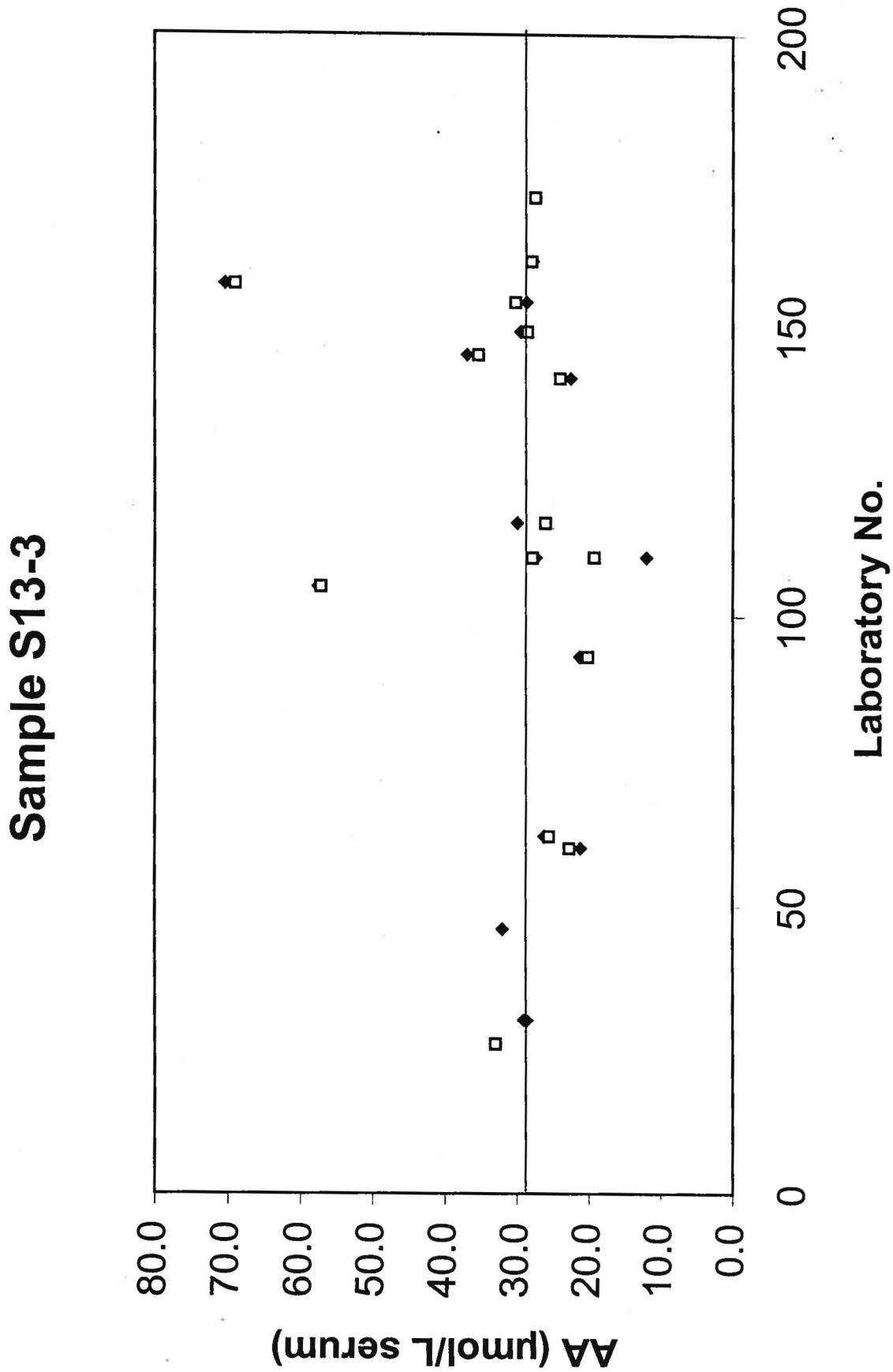

Sample S13-3
AA (µmol/L serum)
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0.0
0
50
100
150
200
Laboratory No.

Figure 4.

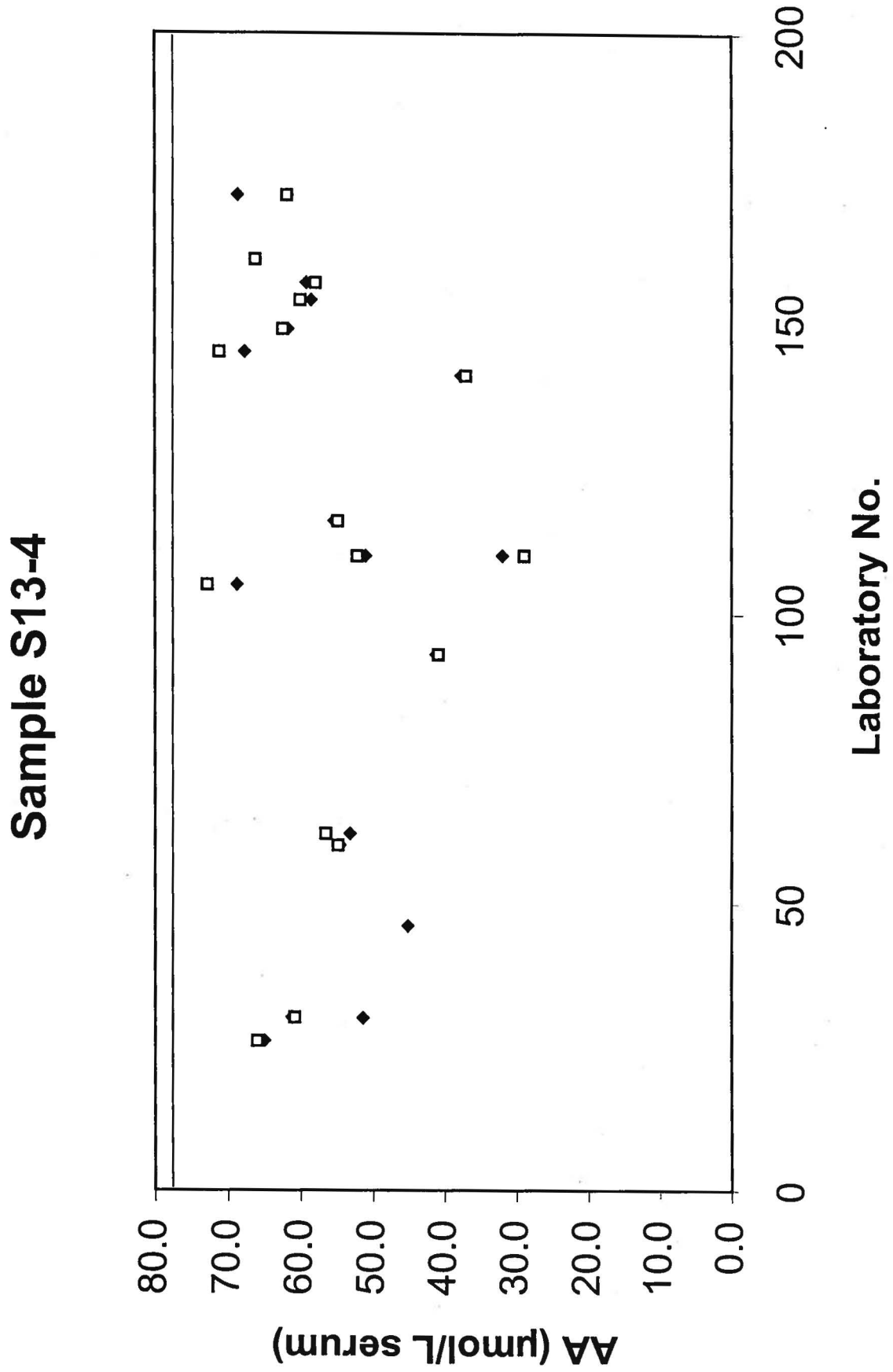
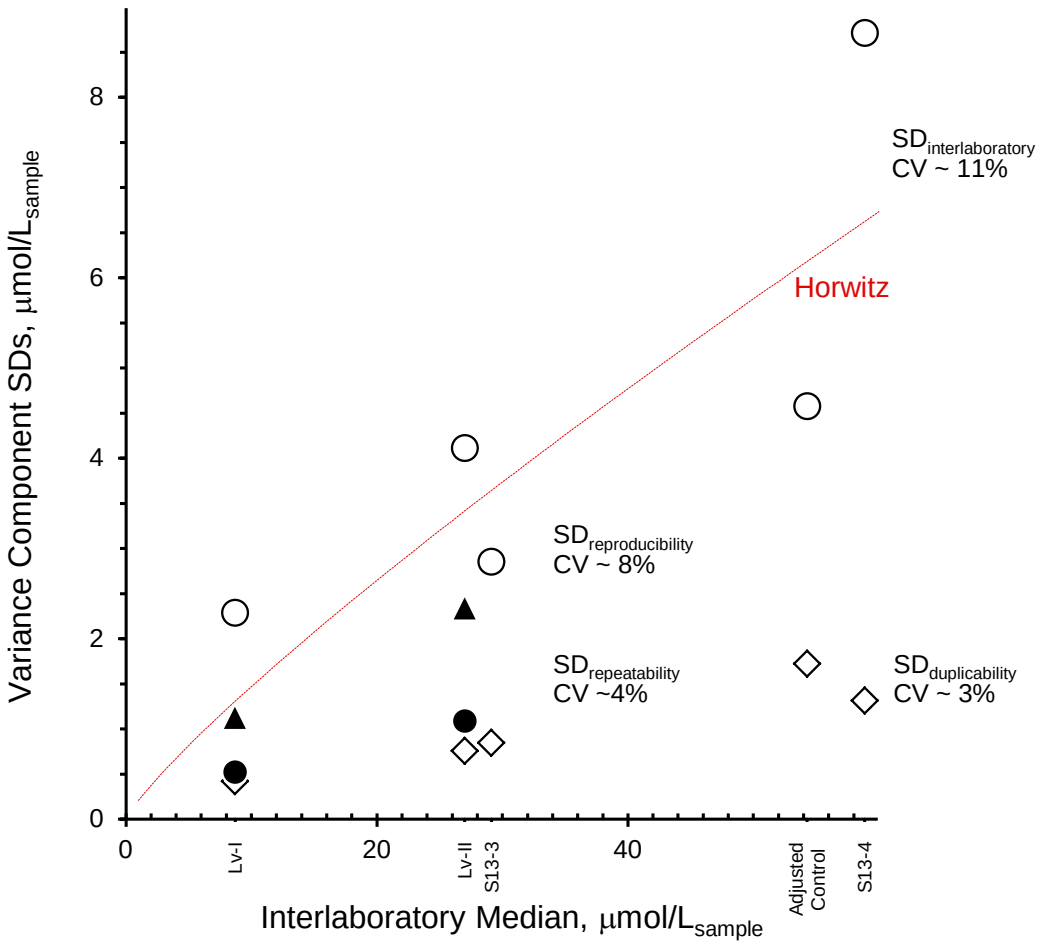

Sample S13-4
AA (µmol/L serum)
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0.0
0
50
100
150
200
Laboratory No.

Figure 5.


Variance Component SDs, µmol/L$_{sample}$
8
6
4
2
0
0
20
40
Lv-I
Lv-II
S13-3
Adjusted Control
S13-4
Interlaboratory Median, µmol/L$_{sample}$
SD$_{interlaboratory}$
CV ~ 11%
Horwitz
SD$_{reproducibility}$
CV ~ 8%
SD$_{repeatability}$
CV ~4%
SD$_{duplicability}$
CV ~ 3%

Appendix K. "All-Lab Report" for RR13

The following single page is the "All-Lab Report" as provided to all participants, with two exceptions:

- the participant identifiers (Lab) have been altered.
- the order in which the participant results are listed has been altered.

The data summary in the "All-Lab Report" has been altered to ensure confidentiality of identification codes assigned to laboratories. The only attributed results are those reported by NIST. The NIST results are not used in the assessment of the consensus summary results of the study.

Vitamin C "Round Robin" 13 Laboratory Results

		Control Solution						Total Ascorbic Acid, µmol/L			
		OD_{max}	D_{MPA}	$E_{1\%}$	[TAA] µmol/L						
Lab	Method	au	$^{g}/_{mL}$	$^{dL}/_{gcm}$	Cal	Obs	$^{Obs}/_{Cal}$	S13:1	S13:2	S13:3	S13:4
VC-MA	HPLC-EC	0.523	1.031	572	56.0	55.4	0.99	8.5	27.7	57.1	69.2
VC-MB	AO-OPD, Cobas Fara	0.578	1.032	559	58.7	63.5	1.08	12.0	31.8	33.0	65.5
VC-MC	HPLC-EC	0.565	1.031	573	56.2	56.9	1.01	7.9	26.5	29.3	61.0
VC-MD	24DNPH	0.565	"	"	56.2	46.6	0.83	7.2	30.2	28.7	51.4
VC-ME	HPLC-UV	0.520	1.030	574	51.4	48.9	0.95	6.3	21.9	22.0	54.7
VC-MG	HPLC-UV	0.614	1.036	613	56.9	56.5	0.99	15.5	35.5	36.1	69.4
VC-MH	HPLC-OPD/f	0.581	1.033	564	58.6	57.0	0.97	8.6	27.8	29.6	55.1
VC-MI	HPLC-UV		1.032		62.3	61.6	0.99	10.2	33.0	27.9	66.2
VC-ML	HPLC-UV	0.573	1.032	543	60.7	48.7	0.80	9.3	25.4	27.5	51.6
VC-MO	HPLC-OPD	0.551	1.030	550	56.9	61.4	1.08	7.8	27.3	29.0	62.0
VC-MP	Ferrozine/Beckman LX20	0.539	1.006	566	54.1	13.6	0.25	1.7	6.0	6.8	16.2
VC-MR	HPLC-EC	0.588	1.034	569	58.7	53.5	0.91	9.0	26.4	57.4	70.8
VC-MS	HPLC-EC	0.471	1.031	571	46.8	39.5	0.84	10.9	21.4	20.7	41.0
VC-NE	HPLC-UV	0.545	1.018	533	58.4	56.3	0.96	22.2	39.6	46.6	74.6
VC-NI	AO-OPD, Cobas Mira S	0.531	1.031	550	55.4	54.5	0.98	7.5	29.0	29.5	59.3
VC-NK	24DNPH	0.527	1.031	526	56.9	71.3	1.25	11.4	21.3	32.1	45.1
VC-NL	HPLC-EC	0.576	1.035	569	57.4	56.0	0.98	7.5	25.8	26.0	54.9
VC-NQ	HPLC-OPD/f	0.549	1.030	547	57.0	54.1	0.95	7.8	27.4	58.5	69.7
	N	17	17	16	18	18	18	18	18	18	18
	Average	0.553	1.029	561	56.6	53.1	0.94	9.5	26.9	33.2	57.6
	Min	0.471	1.006	526	46.8	13.6	0.25	1.7	6.0	6.8	16.2
	Median	0.551	1.031	565	56.9	55.7	0.97	8.5	27.3	29.4	60.1
	Max	0.614	1.036	613	62.3	71.3	1.25	22.2	39.6	58.5	74.6
	eSD	0.035	0.001	12.7	2.3	5.9	0.05	1.7	3.5	5.2	12.8
	eCV	6	0.1	2	4	11	5	20	13	18	21
							N_{NIST}	6	6	6	6
							Mean	9.7	29.6	63.9	75.5
							Expected	10.7	31.8	28.9	77.2
							S_{rep}	0.42	0.52	1.52	1.27
							S_{het}	0.20	0.61	0.09	0.66
							S_{NIST}	0.47	0.80	1.52	1.43

Note

The S13:3 samples were intended to be the material originally designated "682b". Examination of Dr. Margolis's sample storage containers in 2007 strongly suggested that some of the S13:3 samples were actually a material originally designated "188a". Likewise, the S13:4 samples were intended to have all been the "179b" material but it is likely some were actually the "682b" material.

Appendix L. Representative "Individualized Report" for RR13

Each participant in RR13 received an "Individualized Report" reflecting their reported results. The following two pages are the "Individualized Report" for participant "VC-MA".

Vitamin C "Round Robin" 13 Report: Participant VC-MA

RR	Date	Sample	AA mg	MPA g	Stock mg	MPA g	$[TAA]_{cal}$ µmol/L	A_{243} OD	A_{244} OD	$E^{1\%}_{max}$ dL/gcm
11	09/23/98	Control	200.0	103.09	526.0	102.45	57.8	0.0525	0.0527	
12	04/02/99	Control	215.0	103.10	517.1	102.52	61.1	0.0721	0.0721	
13	09/17/01	Control	200.8	103.16	508.0	102.58	0.0	0.5650	0.5232	0
										FALSE

Method	RR	Date	Sample	Replicates, $[TAA]_{obs}$ µmol/L_{sample} Rep_1	Rep_2	*Factor*	Mean	SD_{dup}	Duplicates, $[TAA]_{obs}/[TAA]_{cal}$ Mean	SD_{dup}	SD_{reprod}
HPLC-EC (Height)	11	09/23/98	11-Ctrl	6.3	5.9	1.0	6.1	0.3	0.487	0.004	0.541
HPLC-EC (Height)	12	04/02/99	12-Ctrl	53.3	53.0	1.0	53.1	0.2			
HPLC-EC (Height)	13	09/17/01	13-Ctrl	55.9	55.0	1.0	55.4	0.7			

				Replicates, [TAA] µmol/L_{sample}					Duplicates, [TAA] µmol/L_{sample}			
HPLC-EC (Height)	11	09/23/98	SRM Lv I, S11:1	15.5	13.9	0.5	7.4	0.6	Mean	SD_{dup}	SD_{repeat}	SD_{reprod}
HPLC-EC (Height)	11	09/23/98	SRM Lv I, S11:1	14.0	14.5	0.5	7.1	0.2	7.8	0.4	0.1	0.6
HPLC-EC (Height)	12	04/02/99	SRM Lv I, S12:1	14.5	15.8	0.5	7.6	0.5				
HPLC-EC (Height)	12	04/02/99	SRM Lv I, S12:1	16.1	15.1	0.5	7.8	0.3				
HPLC-EC (Height)	13	09/17/01	SRM Lv I, S13:1	8.4	8.5	1.0	8.5	0.1				
HPLC-EC (Height)	11	09/23/98	SRM Lv II, S11;2	50.7	47.7	0.5	24.6	1.1	Mean	SD_{dup}	SD_{repeat}	SD_{reprod}
HPLC-EC (Height)	11	09/23/98	SRM Lv II, S11;2	48.8	54.0	0.5	25.7	1.8	25.5	1.1	0.6	2.0
HPLC-EC (Height)	12	04/02/99	SRM Lv II, S12:2	49.5	45.9	0.5	23.9	1.3				
HPLC-EC (Height)	12	04/02/99	SRM Lv II, S12:2	46.2	47.9	0.5	23.5	0.6				
HPLC-EC (Height)	13	09/17/01	SRM Lv II, S13:2	27.6	27.7	1.0	27.7	0.1				
HPLC-EC (Height)	13	09/17/01	S13:3 (682B)	58.3	55.9	1.0	57.1	1.7				
HPLC-EC (Height)	13	09/17/01	S13:4 (179B)	69.6	68.7	1.0	69.2	0.7				

L2

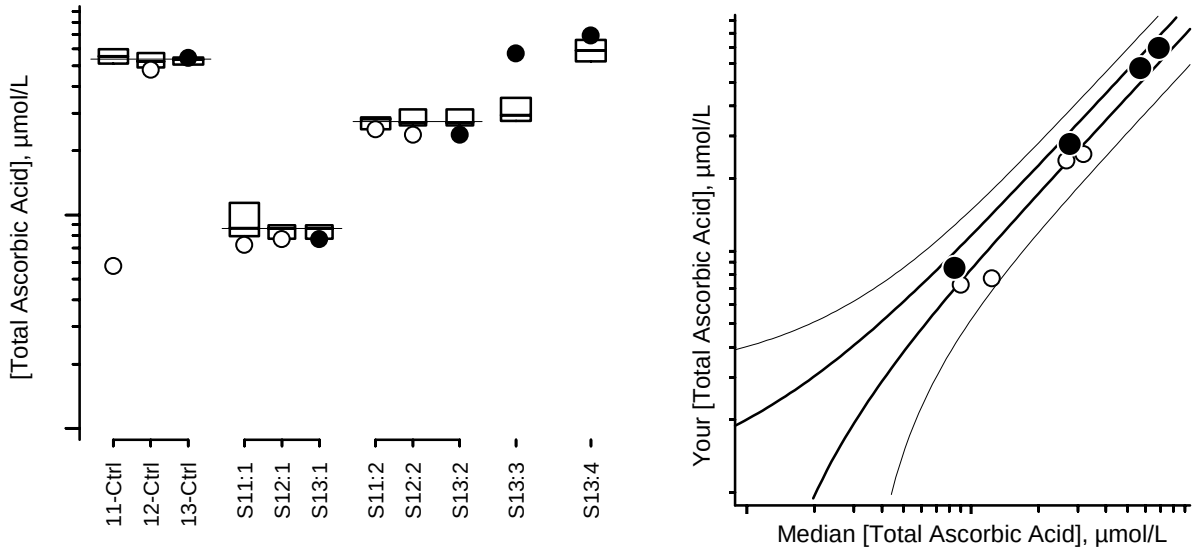
Please check our records against your records. Send corrections and/or updates to...

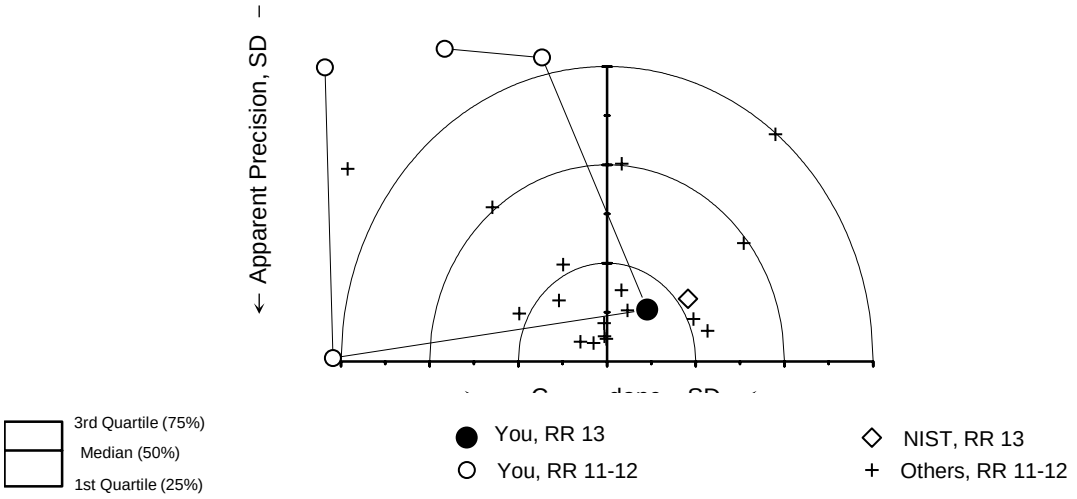
Micronutrients Measurement Quality Assurance Program
National Institute of Standards and Technology
100 Bureau Drive Stop 8392
Gaithersburg, MD 20899-8392 USA

Fax: (301) 977-0685
Email: sam.margolis@nist.gov

Vitamin C "Round Robin" 13 Report: Participant VC-MA

Total Ascorbic Acid





For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem May 1, 1999.

Sample	Comments
Ctrl	Nominal 100 mg/L (56 µmol/mL) standard "control" solution prepared by participant from solid ascorbic acid provided by NIST. These data are not used in the target-plot calculations.
S13:1	SRM 970 Level I
S13:2	SRM 970 Level II
S13:3	Serum 682B, prepared 3/1995. NIST's and the last-distributed RR samples of this serum assayed much higher than the historical value. These data are not used in the target-plot calculations.
S13:4	Serum 179B, prepared 4/1993.