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NIST Micronutrients Measurement Quality Assurance Program Winter 2005 Comparability Studies

Results for Round Robin LVII
Fat-Soluble Vitamins and Carotenoids in Human Serum
and Round Robin 22 Ascorbic Acid in Human Serum

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

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U.S. Department of Commerce
Cameron F. Kerry, Acting 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 2005 MMQAP measurement comparability improvement studies: 1) Round Robin LVII Fat-Soluble Vitamins and Carotenoids in Human Serum and 2) Round Robin 22 Total Ascorbic Acid in Human Serum. The materials for both studies were shipped to participants in November 2004; participants were requested to provide their measurement results by March 2, 2005.

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 LVII: Fat-Soluble Vitamins and Carotenoids in Human Serum

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin LVII comparability study (hereafter referred to as RR57) received four lyophilized and one 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 November 2004. 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 RR57 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 all analytes reported by at least five participants. 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 22: Vitamin C in Human Serum

Participants in the MMQAP Vitamin C in Human Serum Round Robin 22 comparability study (hereafter referred to as RR22) received four frozen serum test samples, two frozen control sera, 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 November 2004. The communication materials included in the sample shipment are provided in Appendix E.

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 RR22 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 F.

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

- 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 H.

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 RR57

The following three items were included in each package shipped to an RR57 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 20899-0001

November 8, 2004

Dear Colleague:

Enclosed are the samples (Sera 309 – 313) for the first fat-soluble vitamins and carotenoids in serum round robin study (Round Robin LVII) for the fiscal year (FY) 05 NIST Micronutrients Measurement Quality Assurance Program. You will find one vial of each of one liquid-frozen and four lyophilized 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 a value is obtained below your limit of quantification, please indicate this result on the form by using NQ *(Not Quantified)*. Results are due to NIST by **March 2, 2005**. Results received more than 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 in April.

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 may 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. **Water should not be added to the liquid-frozen sample 313.**

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

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

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

If you have questions or comments regarding this study, 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

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

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Participant #: ________ Date: ____________

Round Robin LVII

NIST Micronutrients Measurement Quality Assurance Program

Analyte	309	310	311	312	313	Units*
total retinol						
trans-retinol						
didehydroretinol						
retinyl palmitate						
α-tocopherol						
γ/β-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						
total Coenzyme Q10						
ubiquinol (QH_2)						
ubiquinone (Qox)						
phylloquinone (K_1)						
25-hydroxyvitamin D						

Other analytes?						

* we prefer µg/mL

Was serum 309 frozen when received? Yes | No

Comments:

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

Please return results **before** 2-Mar-2005

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

Participant #: ________ Date: ____________

Fat-Soluble Vitamins Round Robin LVII

NIST Micronutrients Measurement Quality Assurance Program

Packing List and Shipment Receipt Confirmation Form

This box contains (we hope) one vial each of the following **five** FSV M^{2}QAP sera:

Serum	Form	Reconstitute?
#309	Lyophilized	Yes (1 ml H_2O)
#310	Lyophilized	Yes (1 ml H_2O)
#311	Lyophilized	Yes (1 ml H_2O)
#312	Lyophilized	Yes (1 ml H_2O)
#313	Liquid frozen	No

Please 1) Open the pack immediately
2) Check that it contains one vial each of the above samples
3) Check if serum 313 arrived frozen
4) Store the samples 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 serum 313 arrive frozen? Yes | No

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

6) When do you anticipate analyzing these samples? ________________________

Your prompt return of this information is appreciated.

The M^{2}QAP Gang

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

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

Appendix B. Final Report for RR57

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 their previous distributions, if any, and
  - 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

April 14, 2005

Dear Colleague:

Enclosed is the summary report of the results for round robin LVII (RR57) of the 2005 NIST Micronutrients Measurement Quality Assurance Program (M^2QAP) for the fat-soluble vitamins and carotenoids in human serum. Included in this report are: (1) a summary of data and measurement comparability scores for all laboratories, (2) a detailed graphical analysis of your results; and (3) a graphical summary of your measurement comparabilities relative to the NIST assigned values. The NIST-assigned values are 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 57 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 deviations 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.

Samples for the second 2005 QA interlaboratory exercise will be shipped **starting the week of May 16**. We will send you a reminder via e-mail or fax a week prior to shipment. It is critical that you carefully inspect all samples upon arrival and that you promptly confirm to us that they have arrived. We will replace samples (lost or damaged in shipment or miss-packaged by us) only for participants who report the problem within <u>one calendar week</u> after the package arrives.

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

Sincerely,

Jeanice B Thomas

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

Enclosures

NIST

The NIST M^2QAP Round Robin LVII (RR57) report consists of:

Page	"All Lab" Report
1-4	A listing of all results and statistics for analytes reported by at least two laboratories.
5a	A list of results for the analytes reported by only one laboratory.
5b	A legend for the above two lists.
6	The text version of the "Comparability Summary" (or "Score Card").

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 8 other participants.
n+1	The "target" plot version of your "Comparability Summary" scores.

Samples. The five sera below were distributed in RR57.

Serum	Description	Prior Distributions
309	Lyophilized blended serum with native carotenoid levels, augmented with α- and δ-tocopherol; SRM 968c Level II.	#249:RR44-9/98, #256:RR46-6/99, #264:RR48-3/01, #284:RR52-9/02, #299:RR55-3/04
310	Lyophilized blend of the #311 augmented serum and stripped serum, in 1+3 ratio (*i.e.*, measurand levels should be ¼ those of #311).	#197:RR31-6/94, #211:RR35-9/95, #245:RR43-6/98
311	Lyophilized blended serum with native carotenoid levels, augmented with retinol, retinyl palmitate, and α- and γ-tocopherol.	#198:RR31-6/94, #212:RR35-9/95, #246:RR43-6/98
312	Lyophilized, native, single-donor, commercially obtained serum prepared in 2002. The same material was used to prepare #313.	#290:RR53-2/03, #300:RR55-3/04
313	Fresh-frozen, native, single-donor, commercially obtained serum prepared in 2002. The same material was used to prepare #312.	#292:RR53-2/03, #301:RR55-3/04

Results

1) Sera Stability. There was no significant change in the median level or increase in the variability of any measurand in any of the sera. Note that Sera 310 and 311 were prepared more than 10 years ago.

2) Relative Accuracy at Low Measurand Levels: Sera 310 and 311 were prepared to evaluate relative accuracy of measurements made at low measurand levels. The Serum 311 material was created by augmenting a blended serum pool having with fairly high native carotenoid levels with retinol, retinyl palmitate, α-tocopherol, and γ-tocopherol. The Serum #310 material was created by blending one part of the Serum 311 material with 3 parts stripped serum. The measurand levels in the

resulting Serum 310 pool should be ≈ 25% of those in the Serum 311 pool. The Table below reports the observed ratios for the measurands reported by at least 8 participants. Only the ratio for retinyl palmitate is significantly different from the expected 0.25 value in Serum 310; this could result from systematic underestimation of low retinyl palmitate levels **or** about half of the retinyl palmitate in Serum 310 has been lost since the materials were prepared **or** about half of the retinyl palmitate that should be in Serum 310 was lost during preparation. The ratio data from earlier comparisons of these materials agree well with the current values, suggesting that retinyl palmitate has not degraded during storage. However, we cannot yet distinguish between the other two possibilities.

	Ratio, Sera 310/311		
Measurand	Mean	-SD	+SD
Retinyl Palmitate	0.15	0.13	0.17
trans-β-Carotene	0.21	0.19	0.24
trans-Lycopene	0.21	0.20	0.23
Total β-Carotene	0.22	0.18	0.27
Total Lycopene	0.23	0.19	0.29
Total α-Carotene	0.24	0.16	0.36
α-Tocopherol	0.24	0.23	0.26
Total Lutein&Zeaxanthin	0.25	0.21	0.29
Total Retinol	0.25	0.23	0.27
Total Lutein	0.26	0.21	0.32
γ/β -Tocopherol	0.26	0.24	0.28
Total β-Cryptoxanthin	0.28	0.22	0.38

If your serum 310/311 ratio for any measurand is much different from 0.25, you should review the manner by which you integrate low-level signals.

3) Matrix (Lyophilized vs. Fresh-Frozen) Differences. Sera 312 and 313 were prepared from the same serum pool. Since we suggest that you reconstitute our lyophilized samples with 1.0 mL water rather than to a total volume of 1.0 mL, the measurand levels in Serum 312 should be ≈ 95% of those in Serum 313. The observed average ratio ± SD over all measurands with 8 or more quantitative measurements is 0.952 ± 0.008. If any of your Sera 312/313 ratios are much different than 0.95, you should look at your measurement system for those measurands. If your lyophilized/fresh frozen ratios are consistently much different from 0.95, you should review how you reconstitute lyophilized materials.

Appendix C. "All-Lab Report" for RR57

The following six 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 LVII Laboratory Results
All Results in µg/mL

Lab	Total Retinol 309	310	311	312	313	trans-Retinol 309	310	311	312	313
FSV-BA	0.489	0.204	0.787	0.649	0.670					
FSV-BB	0.487	0.205	0.778	0.641	0.657					
FSV-BC	0.477	0.111	0.730	0.613	0.647					
FSV-BD	0.490	0.190	0.740	0.720	0.700					
FSV-BE	0.520	0.250	0.790	0.600	0.690					
FSV-BF	0.480	0.170	0.700	0.610	0.640					
FSV-BG	0.477	0.191	0.760	0.595	0.627					
FSV-BH	0.412	0.145	0.648	0.579	0.586					
FSV-BI	0.459	0.179	0.707	0.567	0.542					
FSV-BJ	0.480	0.188	0.724	0.551	0.596					
FSV-BK	0.509	0.190	0.784	0.643	0.679					
FSV-BL	0.490	0.110	0.460	0.690	0.720					
FSV-BM	0.486	0.180	0.752	0.623	0.646					
FSV-BN	0.456	0.190	0.693	0.545	0.601					
FSV-BO	0.470	-	0.705	0.690	0.667					
FSV-BP	0.482	-	-	0.596	0.632					
FSV-BQ	0.580	0.183	0.908	0.708	0.731					
FSV-BR	≥0.510	≥0.180	≥0.730	≥0.600	≥0.660	0.510	0.180	0.730	0.600	0.660
FSV-BS	0.493	0.176	0.873	0.668	0.714					
FSV-BU	0.489	0.177	0.748	0.574	0.637					
FSV-BV	0.600	0.192	0.733	0.681	0.681					
FSV-BW	0.470	0.170	0.750	0.600	0.620					
FSV-BX	≥0.470	≥0.183	≥0.750	≥0.596	≥0.618	0.470	0.183	0.750	0.596	0.618
FSV-CC	0.510	0.200	0.770	0.600	0.620	0.480	0.190	0.740	0.600	0.620
FSV-CD	≥0.496	≥0.196	≥0.813	≥0.627	≥0.657	0.496	0.196	0.813	0.627	0.657
FSV-CE	0.536	0.207	0.458	0.523	0.518					
FSV-CF	0.480	0.178	0.802	0.626	0.652					
FSV-CG	0.539	0.189	0.729	0.561	0.618					
FSV-CI	0.439	0.151	0.618	0.561	0.584					
FSV-CP										
FSV-CS	0.485	0.198	0.802	0.668	0.662					
FSV-CT										
FSV-CW	0.403	0.263	0.748	0.569	0.562					
FSV-CZ	0.490	0.210	0.790	0.630	0.650					
FSV-DA	≥0.471	≥0.213	≥0.758	≥0.601	≥0.633	0.471	0.213	0.758	0.601	0.633
FSV-DB	0.481	0.171	0.712	0.582	0.617					
FSV-DD	0.456	0.187	0.700	0.575	0.580					
FSV-DF	0.477	0.180	0.776	0.607	0.645					
FSV-DI	0.518	0.141	0.770	0.660	0.670					
FSV-DV	1.250	0.450	1.720	1.360	1.450					
FSV-DW	0.520	0.220	0.750	0.630	0.630					
FSV-ET	0.430	0.180	0.790	0.560	0.580					
N	36	34	35	36	36	5	5	5	5	5
Min	0.403	0.110	0.458	0.523	0.518	0.470	0.180	0.730	0.596	0.618
Median	0.486	0.187	0.750	0.609	0.643	0.480	0.190	0.750	0.600	0.633
Max	1.250	0.450	1.720	1.360	1.450	0.510	0.213	0.813	0.627	0.660
SD	0.025	0.017	0.057	0.057	0.042					
CV	5	9	8	9	7	0	0	0	0	0
N_{past}	42	44	44	33	33	7	0	0	7	7
$Median_{past}$	0.482	0.196	0.730	0.601	0.638	0.480			0.605	0.648
SD_{past}	0.036	0.020	0.065	0.034	0.045	0.037			0.025	0.024
NIST	0.497	≥0.188	≥0.789	≥0.689	≥0.673	0.489	0.188	0.789	0.689	0.673
N_{NIST}	3	3	3	3	3	3				
S_{rep}	0.009	0.008	0.029	0.019	0.017	0.006				
S_{het}	0.005	0.004	0.008	0.016	0.005	0.007				
S_{NIST}	0.010	0.009	0.031	0.025	0.018	0.009				
NAV	0.491	0.188	0.769	0.650	0.658	0.484	0.190	0.750	0.600	0.633
NAU	0.040	0.016	0.065	0.080	0.055	0.039	0.016	0.059	0.048	0.050

Lab	Retinyl Palmitate 309	310	311	312	313	α-Tocopherol 309	310	311	312	313
FSV-BA	0.088	0.031	0.219	0.110	0.115	16.63	3.46	14.75	10.05	10.40
FSV-BB	0.069	0.017	0.225	0.066	0.073	17.11	3.52	14.99	10.09	10.33
FSV-BC										
FSV-BD						16.80	3.90	14.40	10.30	11.40
FSV-BE						18.10	4.00	14.90	10.20	10.80
FSV-BF						17.00	1.40	13.70	10.10	10.40
FSV-BG	0.081	0.036	0.250	0.123	0.123	16.41	3.42	14.28	9.64	10.09
FSV-BH						15.92	3.30	13.73	10.57	9.88
FSV-BI	0.097	0.048	0.296	0.107	0.103	18.32	3.73	15.34	10.73	10.23
FSV-BJ	0.086	*nq*	0.278	0.094	0.095	17.05	3.52	13.70	9.53	10.17
FSV-BK						16.96	3.03	13.91	9.42	9.96
FSV-BL						15.07	3.01	12.49	9.48	9.91
FSV-BM						16.40	3.60	15.20	10.30	10.90
FSV-BN	0.073	0.048	0.272	0.086	0.092	14.69	3.02	12.15	8.37	9.25
FSV-BO						15.69	4.05	13.70	9.48	9.86
FSV-BP						16.65	-	-	10.70	9.57
FSV-BQ						16.60	3.50	14.30	10.10	10.70
FSV-BR						14.84	3.10	14.35	9.92	10.93
FSV-BS						12.16	3.13	10.73	7.28	8.63
FSV-BU						16.02	3.84	13.94	9.97	10.44
FSV-BV						15.69	3.29	13.36	9.83	9.66
FSV-BW	0.110	0.050	0.340	0.120	0.130	15.55	3.74	13.48	9.12	10.00
FSV-BX						12.11	3.39	14.15	10.08	10.45
FSV-CC						18.82	4.01	16.50	10.55	11.28
FSV-CD	0.075	0.068	0.493	0.153	0.212	18.92	2.98	16.13	10.10	10.40
FSV-CE						18.46	3.26	10.94	6.55	9.87
FSV-CF						15.70	4.70	13.80	9.40	10.00
FSV-CG						14.65	2.37	12.20	7.93	8.96
FSV-CI	0.077	0.033	0.254	0.091	0.099	16.21	3.50	14.82	9.59	10.23
FSV-CP						16.71	5.89	14.51	10.49	10.41
FSV-CS						17.05	3.13	15.64	10.36	11.15
FSV-CT										
FSV-CW	0.046	0.041	0.220	0.059	0.051	18.60	3.80	15.20	10.20	11.60
FSV-CZ						16.20	3.40	14.40	9.80	10.10
FSV-DA	0.070	0.048	0.291	0.084	0.086	17.63	3.80	14.72	10.46	11.02
FSV-DB						16.12	3.89	13.57	9.44	9.99
FSV-DD										
FSV-DF										
FSV-DI	0.065	0.048	0.249	0.060	0.084	17.30	3.68	14.60	10.50	11.20
FSV-DV						17.10	2.90	14.30	8.50	9.80
FSV-DW						16.65	3.31	13.71	10.17	10.45
FSV-ET						14.60	3.68	14.41	9.23	9.64
N	12	11	12	12	12	38	37	37	38	38
Min	0.046	0.017	0.219	0.059	0.051	12.11	1.40	10.73	6.55	8.63
Median	0.076	0.048	0.263	0.093	0.097	16.61	3.50	14.30	10.01	10.23
Max	0.110	0.068	0.493	0.153	0.212	18.92	5.89	16.50	10.73	11.60
SD	0.012	0.010	0.037	0.024	0.023	1.04	0.50	0.78	0.61	0.56
CV	16	21	14	26	24	6	14	5	6	5
N_{past}	14	9	11	13	13	44	44	44	36	37
$Median_{past}$	0.081	0.052	0.277	0.095	0.103	16.61	3.59	14.20	9.85	10.47
SD_{past}	0.017	0.014	0.055	0.020	0.023	1.28	0.54	1.25	0.75	0.71
NIST						16.76	3.49	14.54	10.95	10.47
N_{NIST}						3	3	3	3	3
S_{rep}						0.08	0.11	0.14	0.01	0.38
S_{het}						0.26	0.04	0.18	0.21	0.10
S_{NIST}						0.27	0.11	0.23	0.21	0.39
NAV	0.076	0.048	0.263	0.093	0.097	16.69	3.49	14.42	10.51	10.37
NAU	0.021	0.016	0.060	0.025	0.026	1.29	0.42	1.11	1.00	0.81

Lab	γ/β-Tocopherol 309	310	311	312	313	δ-Tocopherol 309	310	311	312	313
FSV-BA	1.589	0.706	2.693	1.843	1.915	0.581	0.046	0.154	0.055	0.057
FSV-BB	1.537	0.687	2.512	1.727	1.764	0.553	0.049	0.128	0.062	0.059
FSV-BC										
FSV-BD										
FSV-BE	1.700	0.900	2.800	1.800	1.900					
FSV-BF	1.700	0.300	2.600	1.800	1.800					
FSV-BG	1.561	0.678	2.575	1.754	1.833					
FSV-BH	1.449	0.639	2.461	1.694	1.678					
FSV-BI	1.750	0.660	2.830	1.890	1.740					
FSV-BJ	1.527	0.718	2.429	1.611	1.729					
FSV-BK										
FSV-BL										
FSV-BM										
FSV-BN	1.289	0.587	2.013	1.299	1.489	0.466	*nq*	0.143	*nq*	0.048
FSV-BO										
FSV-BP										
FSV-BQ										
FSV-BR										
FSV-BS	1.029	0.504	1.668	1.126	1.281					
FSV-BU	1.548	0.719	2.432	1.715	1.798					
FSV-BV	1.570	0.670	2.540	1.770	1.810					
FSV-BW										
FSV-BX	1.617	0.633	2.637	1.712	1.800					
FSV-CC										
FSV-CD	1.905	0.859	3.288	1.966	2.031					
FSV-CE										
FSV-CF										
FSV-CG	1.544	0.634	2.523	1.660	1.828	0.663	0.038	0.246	0.202	0.210
FSV-CI	1.430	0.660	2.470	1.580	1.690					
FSV-CP	1.910	0.740	2.980	2.410	2.800					
FSV-CS	1.553	0.707	3.318	2.623	2.512					
FSV-CT										
FSV-CW	1.700	0.700	2.600	1.800	2.000	0.690	0.020	0.140	0.030	0.030
FSV-CZ										
FSV-DA	1.550	0.704	2.690	1.860	1.984	0.466	0.024	0.084	0.024	0.033
FSV-DB	1.536	0.574	2.392	1.736	1.856	0.577	*nq*	0.074	*nq*	*nq*
FSV-DD										
FSV-DF										
FSV-DI	1.480	0.620	2.510	1.690	1.780	0.600	0.033	0.140	0.044	0.044
FSV-DV										
FSV-DW										
FSV-ET	1.240	0.570	2.570	1.580	1.680					
N	23	23	23	23	23	8	6	8	6	7
Min	1.029	0.300	1.668	1.126	1.281	0.466	0.020	0.074	0.024	0.030
Median	1.550	0.670	2.570	1.736	1.800	0.579	0.036	0.140	0.050	0.048
Max	1.910	0.900	3.318	2.623	2.800	0.690	0.049	0.246	0.202	0.210
SD	0.115	0.059	0.168	0.109	0.128	0.063	0.013	0.021	0.020	0.014
CV	7	9	7	6	7	11	37	15	40	30
N_{past}	25	20	20	22	22	6	0	0	5	5
$Median_{past}$	1.579	0.701	2.610	1.750	1.848	0.714			0.083	0.075
SD_{past}	0.123	0.130	0.178	0.108	0.146	0.265			0.026	0.030
NIST	1.549	0.695	2.570	1.798	1.778	0.526	*nq*	*nq*	*nq*	*nq*
N_{NIST}	3	3	3	3	3	3				
S_{rep}	0.019	0.004	0.092	0.020	0.024	0.015				
S_{het}	0.012	0.012	0.047	0.043	0.018	0.007				
S_{NIST}	0.022	0.013	0.103	0.047	0.030	0.017				
NAV	1.550	0.684	2.571	1.769	1.789	0.553	0.036	0.140	0.050	0.048
NAU	0.171	0.090	0.255	0.191	0.193	0.099	0.016	0.029	0.020	0.017

C2

Round Robin LVII Laboratory Results

All Results in µg/mL

	Total β-Carotene					trans-β-Carotene					Total cis-β-Carotene					Total α-Carotene					Total Lycopene				
Lab	309	310	311	312	313	309	310	311	312	313	309	310	311	312	313	309	310	311	312	313	309	310	311	312	313
FSV-BA	0.443	0.167	0.689	0.130	0.133	0.414	0.155	0.643	0.123	0.127	0.029	0.012	0.046	0.007	0.006	0.090	0.006	0.029	0.073	0.075	0.459	0.034	0.156	0.528	0.554
FSV-BB	0.424	0.148	0.692	0.111	0.117	0.394	0.139	0.643	0.106	0.111	0.030	0.009	0.048	0.005	0.006	0.090	0.008	0.035	0.064	0.068	0.418	0.037	0.156	0.468	0.483
FSV-BC																									
FSV-BD																									
FSV-BE	0.531	0.124	0.800	0.136	0.142																				
FSV-BF	0.390	0.113	0.695	0.090	0.094											0.105	0.016	0.033	0.094	0.096	0.426	0.017	0.128	0.474	0.476
FSV-BG	0.461	0.137	0.729	0.144	0.157											0.096	0.014	0.038	0.080	0.084	0.442	0.045	0.158	0.551	0.544
FSV-BH	0.395	0.151	0.775	0.139	0.123	0.369	0.138	0.718	0.139	0.123	0.026	0.013	0.057	*nq*	*nq*	0.076	*nq*	0.040	0.071	0.069	0.331	*nq*	0.141	0.434	0.463
FSV-BI	0.473	0.160	0.688	0.122	0.113											0.108	0.008	0.031	0.082	0.079	0.405	0.035	0.124	0.428	0.423
FSV-BJ	0.491	0.133	0.788	0.124	0.119											0.118	*nq*	0.040	0.085	0.086	0.515	0.026	0.162	0.522	0.563
FSV-BK																									
FSV-BL																									
FSV-BM																									
FSV-BN	0.401	0.139	0.606	0.103	0.114	0.361	0.122	0.547	0.095	0.105	0.035	0.012	0.054	0.004	0.004	0.095	0.002	0.031	0.073	0.080	0.362	0.027	0.120	0.393	0.428
FSV-BO	0.456	0.130	0.723	0.110	0.117											0.095	*nq*	0.038	0.070	0.074	0.436	0.042	0.156	0.550	0.561
FSV-BP	0.444	-	-	0.109	0.122											0.085	-	-	0.065	0.079	0.405	-	-	0.482	0.390
FSV-BQ																									
FSV-BR																									
FSV-BS	≥0.414	≥0.150	≥0.633	≥0.130	≥0.144	0.414	0.150	0.633	0.130	0.144						0.108	0.017	0.046	0.093	0.099	0.480	0.061	0.191	0.548	0.582
FSV-BU	0.441		0.688	0.112	0.120											0.099		0.035	0.075	0.080	0.438		0.130	0.465	0.509
FSV-BV	0.403	0.160	0.637	0.106	0.113											0.068	0.005	0.022	0.054	0.057	0.454	0.035	0.150	0.509	0.539
FSV-BW	0.490	0.130	0.740	0.120	0.120											0.150	*nq*	0.031	0.102	0.108	0.530	0.030	0.170	0.570	0.590
FSV-BX	≥0.380	≥0.134	≥0.628	≥0.115	≥0.122	0.380	0.134	0.628	0.115	0.122						0.093	*0.007*	0.032	0.085	0.087					
FSV-CC																									
FSV-CD	0.436	0.103	0.583	0.081	0.107											0.102	0.009	0.038	0.065	0.081	0.404	0.048	0.247	0.278	0.360
FSV-CE	0.496	0.132	0.547	0.049	0.123																				
FSV-CF																									
FSV-CG	0.369	0.111	0.569	0.097	0.111	0.339	0.099	0.519	0.091	0.104	0.030	0.012	0.050	0.006	0.008	0.106	0.008	0.040	0.075	0.086	0.398	0.028	0.143	0.408	0.469
FSV-CI	≥0.367	≥0.114	≥0.582	≥0.113	≥0.123	0.37	0.11	0.58	0.11	0.12						0.078	<0.016	0.031	0.061	0.067					
FSV-CP	0.417	0.165	0.665	0.122	0.121											0.105	0.011	0.043	0.082	0.081	0.403	0.042	0.143	0.466	0.459
FSV-CS	0.449	0.180	0.684	0.117	0.124											0.106	0.010	0.037	0.079	0.083	0.458	0.043	0.174	0.492	0.521
FSV-CT	0.544	0.178	0.748	0.127	0.135																0.538	0.042	0.152	0.575	0.613
FSV-CW						0.375	0.183	0.718	0.116	0.122						0.092	0.020	0.045	0.080	0.082					
FSV-CZ	0.440	0.170	0.560	0.130	0.160																				
FSV-DA	0.440	0.157	0.724	0.130	0.140	0.409	0.134	0.663	0.123	0.130	0.031	0.023	0.061	0.007	0.008	0.100	0.008	0.040	0.079	0.085	0.442	0.033	0.152	0.511	0.552
FSV-DB	0.377	0.131	0.620	0.106	0.114																0.399	0.028	0.141	0.467	0.503
FSV-DD																									
FSV-DF																									
FSV-DI	0.417	0.150	0.680	0.065	0.075																0.385	0.022	0.097	0.445	0.490
FSV-DV																									
FSV-DW	0.260	0.140	0.380	0.060	0.050											0.048	0.006	0.014	0.041	0.041	0.229	0.021	0.059	0.286	0.293
FSV-ET	0.380	0.140	0.690	0.110	0.120																				
N	26	24	25	26	26	10	10	10	10	10	6	6	6	5	5	23	16	22	23	23	23	20	22	23	23
Min	0.260	0.103	0.380	0.049	0.050	0.339	0.099	0.519	0.091	0.104	0.026	0.009	0.046	0.004	0.004	0.048	0.002	0.014	0.041	0.041	0.229	0.017	0.059	0.278	0.293
Median	0.440	0.140	0.688	0.111	0.120	0.378	0.136	0.638	0.116	0.123	0.030	0.012	0.052	0.006	0.006	0.096	0.008	0.036	0.075	0.081	0.426	0.035	0.151	0.474	0.503
Max	0.544	0.180	0.800	0.144	0.160	0.414	0.183	0.718	0.139	0.144	0.035	0.023	0.061	0.007	0.008	0.150	0.020	0.046	0.102	0.108	0.538	0.061	0.247	0.575	0.613
SD	0.043	0.022	0.077	0.017	0.008	0.028	0.017	0.048	0.011	0.009	0.002	0.001	0.006	0.001	0.001	0.012	0.004	0.007	0.011	0.008	0.041	0.011	0.019	0.063	0.068
CV	10	16	11	15	7	7	12	7	10	7	5	5	11	19	25	12	45	19	14	10	10	32	12	13	14
N_{past}	30	32	32	26	26	14	10	10	13	13	10	8	8	7	7	26	22	26	23	23	25	22	24	22	22
$Median_{past}$	0.424	0.154	0.678	0.112	0.120	0.386	0.152	0.634	0.108	0.115	0.030	0.016	0.052	0.007	0.007	0.096	0.009	0.034	0.071	0.076	0.411	0.027	0.127	0.479	0.504
SD_{past}	0.047	0.032	0.089	0.014	0.018	0.034	0.020	0.048	0.010	0.012	0.009	0.009	0.020	0.002	0.002	0.018	0.004	0.010	0.012	0.013	0.065	0.009	0.046	0.059	0.071
NIST	0.431	≥0.140	≥0.669	≥0.124	≥0.125	0.394	0.140	0.669	0.124	0.125	0.037	*nq*	*nq*	*nq*	*nq*	0.100	*nq*	*nq*	0.076	0.076					
N_{NIST}	3	3	3	3	3	3					3					3			3	3					
S_{rep}	0.007	0.002	0.017	0.004	0.001	0.016					0.011					0.003			0.002	0.003					
S_{het}	0.006	0.001	0.021	0.002	0.001	0.010					0.010					0.006			0.002	0.001					
S_{NIST}	0.009	0.003	0.027	0.004	0.001	0.019					0.015					0.007			0.003	0.003					
NAV	0.436	0.140	0.679	0.118	0.123	0.386	0.136	0.638	0.116	0.123	0.034	0.012	0.052	0.006	0.006	0.098	0.008	0.036	0.076	0.078	0.431	0.035	0.152	0.478	0.500
NAU	0.062	0.022	0.094	0.021	0.020	0.042	0.017	0.066	0.014	0.015	0.016	0.004	0.021	0.003	0.003	0.028	0.004	0.012	0.022	0.024	0.092	0.012	0.039	0.100	0.103

Round Robin LVII Laboratory Results

All Results in µg/mL

Lab	trans-Lycopene					Total β-Cryptoxanthin					Total α-Cryptoxanthin					Total Lutein					Total Zeaxanthin				
	309	310	311	312	313	309	310	311	312	313	309	310	311	312	313	309	310	311	312	313	309	310	311	312	313
FSV-BA	0.215	0.016	0.068	0.277	0.290	0.040	0.006	0.025	0.068	0.070	0.022	*nq*	0.013	0.033	0.033										
FSV-BB	0.184	0.011	0.057	0.229	0.240	0.030	0.005	0.019	0.051	0.053	0.014	0.002	0.007	0.022	0.023	0.107	0.016	0.067	0.099	0.101	0.041	0.005	0.025	0.053	0.055
FSV-BC																									
FSV-BD																									
FSV-BE																									
FSV-BF						0.026	0.012	0.017	0.047	0.045															
FSV-BG	0.229	0.017	0.078	0.300	0.307	0.030	0.006	0.020	0.054	0.057						0.097	0.013	0.057	0.078	0.072	0.023	0.007	0.027	0.026	0.031
FSV-BH						0.030	*nq*	0.021	0.058	0.058						0.062	0.008	0.032	0.056	0.059	0.021	*nq*	0.012	0.027	0.030
FSV-BI						0.035	0.005	0.023	0.065	0.062						0.086	0.013	0.049	0.075	0.072	0.036	0.007	0.023	0.041	0.039
FSV-BJ						0.032	*nq*	0.016	0.052	0.054						0.087	0.016	0.050	0.085	0.090					
FSV-BK																									
FSV-BL																									
FSV-BM																									
FSV-BN	0.187	0.011	0.052	0.224	0.245	0.021	nq	0.010	0.043	0.048	0.005	*nq*	*nq*	0.017	0.016	0.062	0.015	0.039	0.055	0.062	0.021	nq	0.015	0.024	0.030
FSV-BO						0.023	*nq*	0.015	0.044	0.046						0.073	*nq*	0.050	0.071	0.073	0.011	*nq*	0.017	0.024	0.025
FSV-BP						0.014	-	-	0.050	0.040															
FSV-BQ																									
FSV-BR																									
FSV-BS						0.048	0.008	0.019	0.073	0.060															
FSV-BU						0.029		0.017	0.052	0.058															
FSV-BV						0.023	0.003	0.013	0.043	0.045															
FSV-BW						0.027	0.007	0.012	0.049	0.055															
FSV-BX	0.179	0.014	0.065	0.244	0.253	0.028	0.006	0.018	0.049	0.051						0.086	0.012	0.053	0.082	0.085	0.018	0.003	0.015	0.028	0.028
FSV-CC																									
FSV-CD						0.036	*nq*	0.014	0.056	0.062															
FSV-CE																									
FSV-CF																									
FSV-CG	0.194	0.011	0.061	0.221	0.255	0.043	0.004	0.024	0.065	0.073															
FSV-CI																0.061	0.013	0.040	0.064	0.067	0.018	<0.009	0.015	0.026	0.032
FSV-CP						0.039	0.007	0.023	0.065	0.066															
FSV-CS						0.036	0.005	0.021	0.050	0.053						0.095	0.017	0.065	0.080	0.084	0.021	0.005	0.022	0.030	0.030
FSV-CT						0.039	0.007	0.018	0.050	0.056						0.115	nq	0.057	0.092	0.094	0.024	nq	0.006	0.033	0.034
FSV-CW	0.211	0.022	0.080	0.289	0.306	0.040	0.008	0.030	0.059	0.063						0.085	0.015	0.061	0.087	0.084	0.021	0.009	0.019	0.027	0.027
FSV-CZ																									
FSV-DA	0.224	0.016	0.073	0.287	0.309	0.044	0.007	0.025	0.064	0.067	0.019	0.002	0.008	0.029	0.030	0.098	0.016	0.062	0.087	0.092	0.028	0.006	0.022	0.041	0.043
FSV-DB						0.027	0.003	0.016	0.049	0.050															
FSV-DD																									
FSV-DF																									
FSV-DI																0.072	0.012	0.046	0.064	0.068					
FSV-DV																									
FSV-DW						0.026	0.006	0.016	0.044	0.046						0.070	0.015	0.043	0.056	0.059	0.021	0.005	0.016	0.024	0.025
FSV-ET																									
N	8	8	8	8	8	24	17	23	24	24	4	2	3	4	4	15	13	15	15	15	13	8	13	13	13
Min	0.179	0.011	0.052	0.221	0.240	0.014	0.003	0.010	0.043	0.040	0.005	0.002	0.007	0.017	0.016	0.061	0.008	0.032	0.055	0.059	0.011	0.003	0.006	0.024	0.025
Median	0.203	0.015	0.067	0.260	0.273	0.030	0.006	0.018	0.052	0.055	0.016	0.002	0.008	0.026	0.027	0.086	0.015	0.050	0.078	0.073	0.021	0.006	0.017	0.027	0.030
Max	0.229	0.022	0.080	0.300	0.309	0.048	0.012	0.030	0.073	0.073	0.022	0.002	0.013	0.033	0.033	0.115	0.017	0.067	0.099	0.101	0.041	0.009	0.027	0.053	0.055
SD	0.023	0.004	0.010	0.044	0.041	0.009	0.001	0.004	0.008	0.009	0.006			0.007	0.007	0.018	0.002	0.011	0.016	0.015	0.003	0.002	0.005	0.005	0.004
CV	11	26	16	17	15	30	24	24	16	16	35			27	27	21	15	22	21	21	12	28	31	19	13
N_{past}	11	8	8	10	10	26	14	19	22	22	7	5	7	7	0	16	10	12	15	15	14	8	8	13	13
$Median_{past}$	0.208	0.021	0.077	0.248	0.268	0.032	0.007	0.022	0.051	0.053	0.017	0.005	0.012	0.021		0.085	0.014	0.053	0.078	0.083	0.024	0.007	0.018	0.032	0.033
SD_{past}	0.036	0.008	0.020	0.042	0.053	0.008	0.003	0.007	0.007	0.010	0.006		0.002			0.014	0.004	0.011	0.017	0.016	0.007	0.003	0.008	0.008	0.008
NIST						0.029	*nq*	*nq*	0.050	0.049						0.085	*nq*	0.052	0.076	0.075	0.018	*nq*	*nq*	0.028	0.024
N_{NIST}						3			3	3						3		3	3	3	3			3	3
S_{rep}						0.003			0.002	0.002						0.003		0.002	0.002	0.003	0.003			0.004	0.001
S_{het}						0.001			0.003	0.002						0.002		0.002	0.003	0.001	0.001			0.002	0.002
S_{NIST}						0.003			0.003	0.003						0.004		0.003	0.004	0.003	0.003			0.005	0.002
NAV	0.203	0.015	0.067	0.260	0.273	0.030	0.006	0.019	0.051	0.053	0.016		0.008	0.026	0.027	0.085	0.015	0.051	0.077	0.074	0.019	0.006	0.017	0.027	0.027
NAU	0.036	0.006	0.012	0.047	0.049	0.009	0.002	0.005	0.013	0.014						0.018	0.004	0.011	0.016	0.015	0.007	0.003	0.005	0.008	0.010

Round Robin LVII Laboratory Results
All Results in µg/mL

	Total Lutein&Zeaxanthin					Coenzyme Q10					Ubiquinol					Ubiquinone					Phylloquinone (K1)				
Lab	309	310	311	312	313	309	310	311	312	313	309	310	311	312	313	309	310	311	312	313	309	310	311	312	313
FSV-BA	0.107	0.022	0.072	0.111	0.115																				
FSV-BB	0.148	0.021	0.092	0.152	0.156																				
FSV-BC																									
FSV-BD																									
FSV-BE																					1.32	0.21	1.20	0.36	0.41
FSV-BF	0.111	0.013	0.068	0.109	0.112																				
FSV-BG	0.109	0.016	0.069	0.091	0.086																				
FSV-BH	0.083	0.008	0.044	0.083	0.089																				
FSV-BI	*0.122*	*0.020*	*0.072*	*0.116*	*0.111*																				
FSV-BJ						0.867	0.110	0.539	0.678	0.745															
FSV-BK																									
FSV-BL																									
FSV-BM																									
FSV-BN	0.083	0.015	0.055	0.079	0.092																				
FSV-BO	0.085	*nq*	0.0669	0.0940	0.0982																				
FSV-BP	0.116	-	-	0.099	0.115																				
FSV-BQ																									
FSV-BR																									
FSV-BS	0.105	0.029	0.071	0.116	0.107																				
FSV-BU	0.102		0.065	0.107	0.122																				
FSV-BV	0.096	0.014	0.060	0.106	0.111																				
FSV-BW	0.089	0.013	0.057	0.089	0.096	0.980	0.100	0.520	0.670	0.690	0.780	0.053	0.400	0.500	0.350	0.150	0.047	0.120	0.170	0.340					
FSV-BX	0.105	0.015	0.068	0.110	0.114																				
FSV-CC																									
FSV-CD	0.137	*nq*	0.074	0.118	0.110																				
FSV-CE																									
FSV-CF																									
FSV-CG	0.125	0.019	0.078	0.126	0.141																				
FSV-CI	*0.079*	*<0.022*	*0.055*	*0.090*	*0.099*																0.83	0.24	0.86	0.33	0.31
FSV-CP	0.131	0.023	0.082	0.136	0.137																				
FSV-CS	*0.116*	*0.022*	*0.087*	*0.110*	*0.114*																				
FSV-CT	*0.139*	*nq*	*0.063*	*0.125*	*0.128*																				
FSV-CW	*0.106*	*0.024*	*0.080*	*0.114*	*0.111*	1.100	0.030	0.070	0.090	0.090															
FSV-CZ						0.690	<0.2	0.360	0.390	0.430															
FSV-DA	0.131	0.023	0.086	0.132	0.140	0.916	0.045	0.660	0.808	0.836	*nd*	*nd*	0.208	0.138	0.234	0.916	0.045	0.452	0.670	0.602					
FSV-DB	0.093	0.013	0.058	0.103	0.115																				
FSV-DD																									
FSV-DF																									
FSV-DI						0.992	0.090	0.560	0.620	0.690											0.71	0.19	0.65	0.18	0.16
FSV-DV																									
FSV-DW	*0.091*	*0.020*	*0.059*	*0.080*	*0.084*																				
FSV-ET																									
N	24	18	23	24	24	6	5	6	6	6	1	1	2	2	2	2	2	2	2	2	3	3	3	3	3
Min	0.079	0.008	0.044	0.079	0.084	0.690	0.030	0.070	0.090	0.090			0.208	0.138	0.234	0.150	0.045	0.120	0.170	0.340	0.71	0.19	0.65	0.18	0.16
Median	0.107	0.020	0.068	0.109	0.112	0.948	0.090	0.530	0.645	0.690	0.780	0.053	0.304	0.319	0.292	0.533	0.046	0.286	0.420	0.471	0.83	0.21	0.86	0.33	0.31
Max	0.148	0.029	0.092	0.152	0.156	1.100	0.110	0.660	0.808	0.836			0.400	0.500	0.350	0.916	0.047	0.452	0.670	0.602	1.32	0.24	1.20	0.36	0.41
SD	0.022	0.006	0.012	0.017	0.013	0.081	0.041	0.115	0.170	0.175															
CV	21	29	18	16	12	9	46	22	26	25															
N_{past}	25	17	18	23	23	14	0	0	5	5	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
$Median_{past}$	0.109	0.021	0.074	0.107	0.112	0.950			0.739	0.810															
SD_{past}	0.023	0.010	0.013	0.018	0.019	0.081			0.035	0.047															
NIST	*0.102*	*nq*	*≥0.052*	*0.104*	*0.099*																				
N_{NIST}	3			3	3																				
S_{rep}	0.003			0.005	0.002																				
S_{het}	0.002			0.005	0.002																				
S_{NIST}	0.004			0.007	0.003																				
NAV	0.105	0.020	0.069	0.107	0.105	0.948	0.090	0.530	0.645	0.690											0.830	0.212	0.860	0.330	0.310
NAU	0.023	0.005	0.014	0.023	0.025	0.081	0.041	0.115	0.170	0.175															

Round Robin LVII Laboratory Results

All Results in µg/mL

Analytes Reported By One Laboratory

Analyte	Code	309	310	311	312	313
25-hydroxyvitamin D	FSV-BN	*nq*	0.0270	0.0170	0.0350	0.0230
trans-Lutein	FSV-DA	0.090	0.015	0.058	0.082	0.087
Phytofluene	FSV-DA	0.068	0.011	0.048	0.107	0.114
Phytoene	FSV-DA	0.051	0.009	0.052	0.125	0.132
Retinyl stearate	FSV-DA	0.030	0.005	0.037	0.033	0.034

Legend

Term	Definition
N	Number of (non-NIST) quantitative values reported for this analyte
Min	Minimum (non-NIST) quantitative value reported
Median	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)
NIST	Mean of all analyses (vials x duplicates) reported by a NIST analyst
N_{NIST}	Number of total vials analyzed in duplicate by NIST analysts
S_{rep}	Within-vial pooled standard deviation
S_{het}	Among-vial pooled standard deviation
S_{NIST}	Total standard deviation for NIST analyses: $(S_{rep}^2 + S_{het}^2)^{0.5}$
NAV	NIST Assigned Value = (Median + $Mean_{NIST}$)/2 for analytes reported by NIST analyst(s) = Median 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 at or below the limit of quantification, x
≥x	Concentration greater than or equal to x

Comparability Summary

Lab	TR	aT	g/bT	bC	tbC	aC	TLy	TbX	TLu	TZ	L&Z
FSV-BA	1	1	1	1	1	1	1	2	1	2	1
FSV-BB	1	1	1	1	1	1	1	1	1	1	1
FSV-BC	3										
FSV-BD	1	1									1
FSV-BE	2	1	2	2							
FSV-BF	1	3	3	2		2	1	2			1
FSV-BG	1	1	1	2		1	1	1	2	3	2
FSV-BH	2	1	1	1	1	1	1	1			
FSV-BI	2	1	1	1		1	1	1	2	1	2
FSV-BJ	1	1	1	1		1	1	1	1	1	1
FSV-BK	1	1									
FSV-BL	4	2							1		
FSV-BM	1	1									
FSV-BN	1	2	2	1	2	1	1	2			1
FSV-BO	1	1		1		1	1	1			1
FSV-BP	1	1		1		1	1	2	2	1	2
FSV-BQ	2	1									1
FSV-BR	1	1							2	2	1
FSV-BS	2	3	3	1	1	2	2	2	1	1	1
FSV-BU	1	1	1	1		1	1	1			
FSV-BV	2	1	1	1		2	1	1			
FSV-BW	1	1		1		2	1	1			1
FSV-BX	1	2	1	1	1	1		1			
FSV-CC	1	2									1
FSV-CD	1	2	3	2		1	2	1			1
FSV-CE	3	3		2							1
FSV-CF	1	2							1	2	2
FSV-CG	1	2	1	2	2	1	1	2			1
FSV-CI	2	1	1	1	1	2					
FSV-CP		3	4	1		1	1	1			1
FSV-CS	1	1	4	1		1	1	1			
FSV-CT				2			1	1	1	1	1
FSV-CW	3	2	1		2	2		2	1	1	1
FSV-CZ	1	1		2							
FSV-DA	1	1	1	1	1	1	1	2			
FSV-DB	1	1	1	1			1	1	1	1	1
FSV-DD	1										
FSV-DF	1								1		
FSV-DI	2	1	1	2			1		1	1	1
FSV-DV	4	2									
FSV-DW	2	1		3		2	2	1			
FSV-ET	1	1	2	1							
NISTa	1	1	1	1	1	1		1	1	1	1
n	41	39	24	30	11	24	23	25	16	14	25

	TR	aT	g/bT	bC	tbC	aC	TLy	TbX	TLu	TZ	L&Z
% 1	66	67	67	67	73	71	87	68	75	71	84
% 2	22	23	13	30	27	29	13	32	25	21	16
% 3	7	10	13	3	0	0	0	0	0	7	0
% 4	5	0	8	0	0	0	0	0	0	0	0

Label	Definition
Lab	Participant code
TR	Total Retinol
aT	α-Tocopherol
g/bT	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) of summarizes your measurement performance for a given measurand, relative to the consensus medians. CS is the average distance, in standard deviation units, that your measurement performance characteristics are from the consensus performance. CS is calculated when the number of quantitative values you reported for a measurand, N_{you}, is at least two and the measurand has been reported by 10 or more participants.

$$CS = \mathrm{MIN}(4, \mathrm{INT}(1 + \sqrt{C^2 + AP^2}))$$

$$C = \mathrm{Concordance} = \sum_{i}^{N_{you}} \frac{You_i - Median_i}{NAU_i} \Big/ N_{you}$$

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

NAU = NIST Assigned Uncertainty, our estimate of the overall measurement standard deviation for each sample. The estimate includes serum heterogeneity, analytical repeatability, and among-participant reproducibility variance components.

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

Appendix D. Representative "Individualized Report" for RR57

Each participant in RR57 received an "Individualized Report" reflecting their reported results. Each report included a detailed analysis for analytes that were assayed by at least five participants. The following analytes met this criterion in RR57:

- Total Retinol
- *trans*-Retinol
- Retinyl Palmitate
- α-Tocopherol
- γ/β-Tocopherol
- δ-Tocopherol
- Total β-Carotene
- *trans*-β-Carotene
- Total *cis*-β-Carotene
- Total α-Carotene
- Total Lycopene
- *trans*-Lycopene
- Total β-Cryptoxanthin
- Total Lutein
- Total Zeaxanthin
- Total Lutein & Zeaxanthin
- Coenzyme Q10

The following 15 pages are the "Individualized Report" for the analytes evaluated by participant FSV-BA.
`

Individualized Round Robin LVII Report: FSV-BA

Summary

	Serum 309			Serum 310			Serum 311			Serum 312			Serum 313		
Analyte	You	NAV	n	You	NAV	n	You	NAV	n	You	NAV	n	You	NAV	n
Total Retinol	0.489	0.491	36	0.204	0.188	34	0.787	0.769	35	0.649	0.650	36	0.670	0.658	36
Retinyl Palmitate	0.09	0.08	12	0.0	0.0	11	0.2	0.3	12	0.11	0.09	12	0.12	0.10	12
α-Tocopherol	16.63	16.69	38	3.46	3.49	37	14.75	14.42	37	10.05	10.51	38	10.40	10.37	38
γ/β-Tocopherol	1.589	1.550	23	0.706	0.684	23	2.693	2.571	23	1.843	1.769	23	1.915	1.789	23
δ-Tocopherol	0.581	0.553	8	0.046	0.036	6	0.154	0.140	8	0.055	0.050	6	0.057	0.048	7
Total β-Carotene	0.443	0.436	26	0.167	0.140	24	0.689	0.679	25	0.130	0.118	26	0.133	0.123	26
trans-β-Carotene	0.414	0.386	10	0.155	0.136	10	0.643	0.638	10	0.123	0.116	10	0.127	0.123	10
Total cis-β-Carotene	0.029	0.034	6	0.012	0.012	6	0.046	0.052	6	0.007	0.006	5	0.006	0.006	5
Total α-Carotene	0.090	0.098	23	0.006	0.008	16	0.029	0.036	22	0.073	0.076	23	0.075	0.078	23
Total Lycopene	0.459	0.431	23	0.034	0.035	20	0.156	0.152	22	0.528	0.478	23	0.554	0.500	23
trans-Lycopene	0.215	0.203	8	0.016	0.015	8	0.068	0.067	8	0.277	0.260	8	0.290	0.273	8
Total β-Cryptoxanthin	0.040	0.030	24	0.006	0.006	17	0.025	0.019	23	0.068	0.051	24	0.070	0.053	24
Total α-Cryptoxanthin	0.022	0.016	4	nq		2	0.013	0.008	3	0.033	0.026	4	0.033	0.027	4
Total Lutein&Zeaxanthin	0.107	0.105	24	0.02	0.02	18	0.07	0.07	23	0.111	0.107	24	0.12	0.11	24

You : Your reported values for the listed analytes (micrograms/milliliter)
NAV : NIST Assigned Values, here equal to this RR's median
n : Number of non-NIST laboratories reporting quantitative values for this analyte in this serum
nq : Detected, but not quantitatively determined

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

Micronutrients Measurement Quality Assurance Program
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100 Bureau Drive Stop 8392
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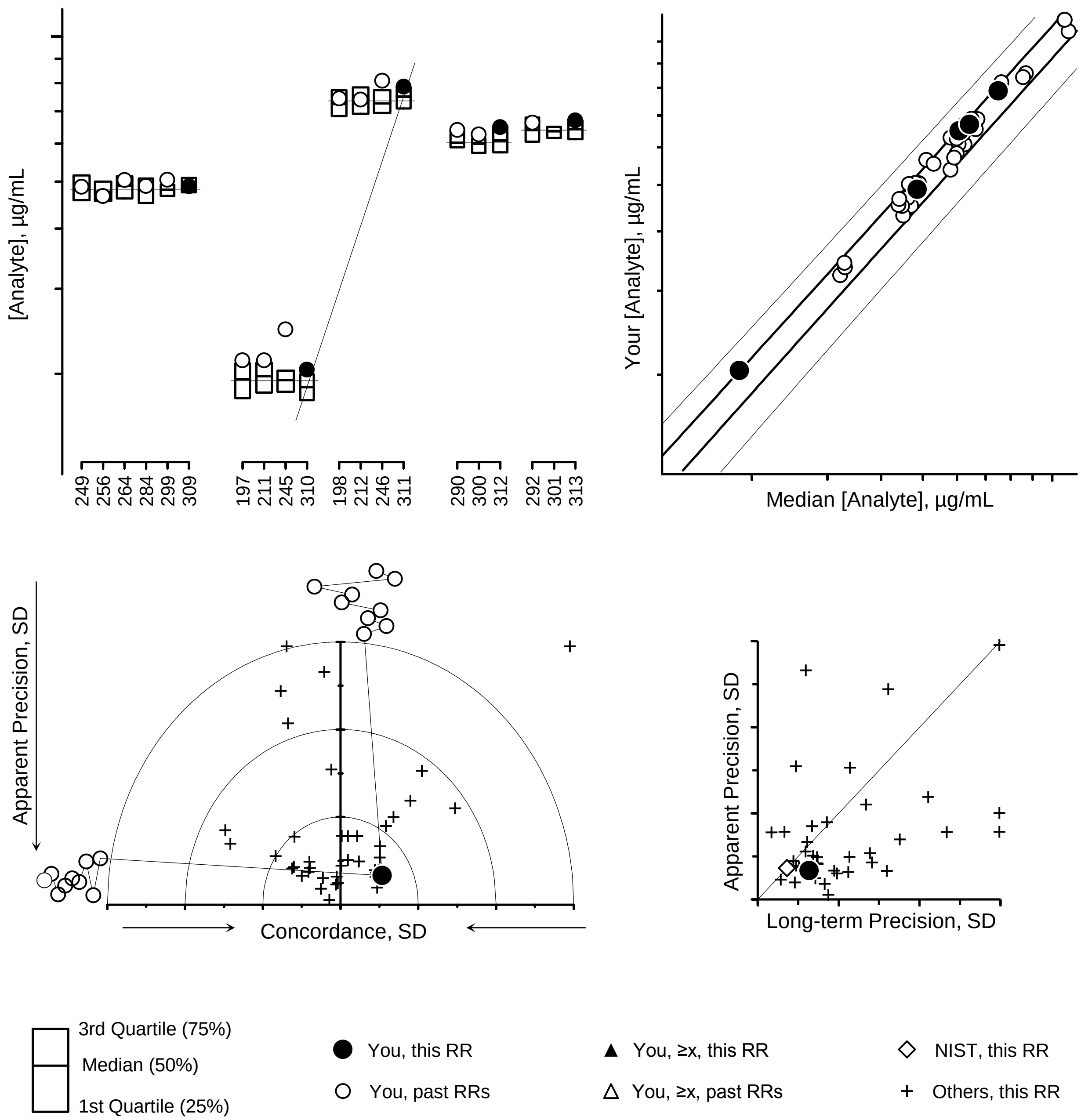
Individualized RR LVII Report: FSV-BA

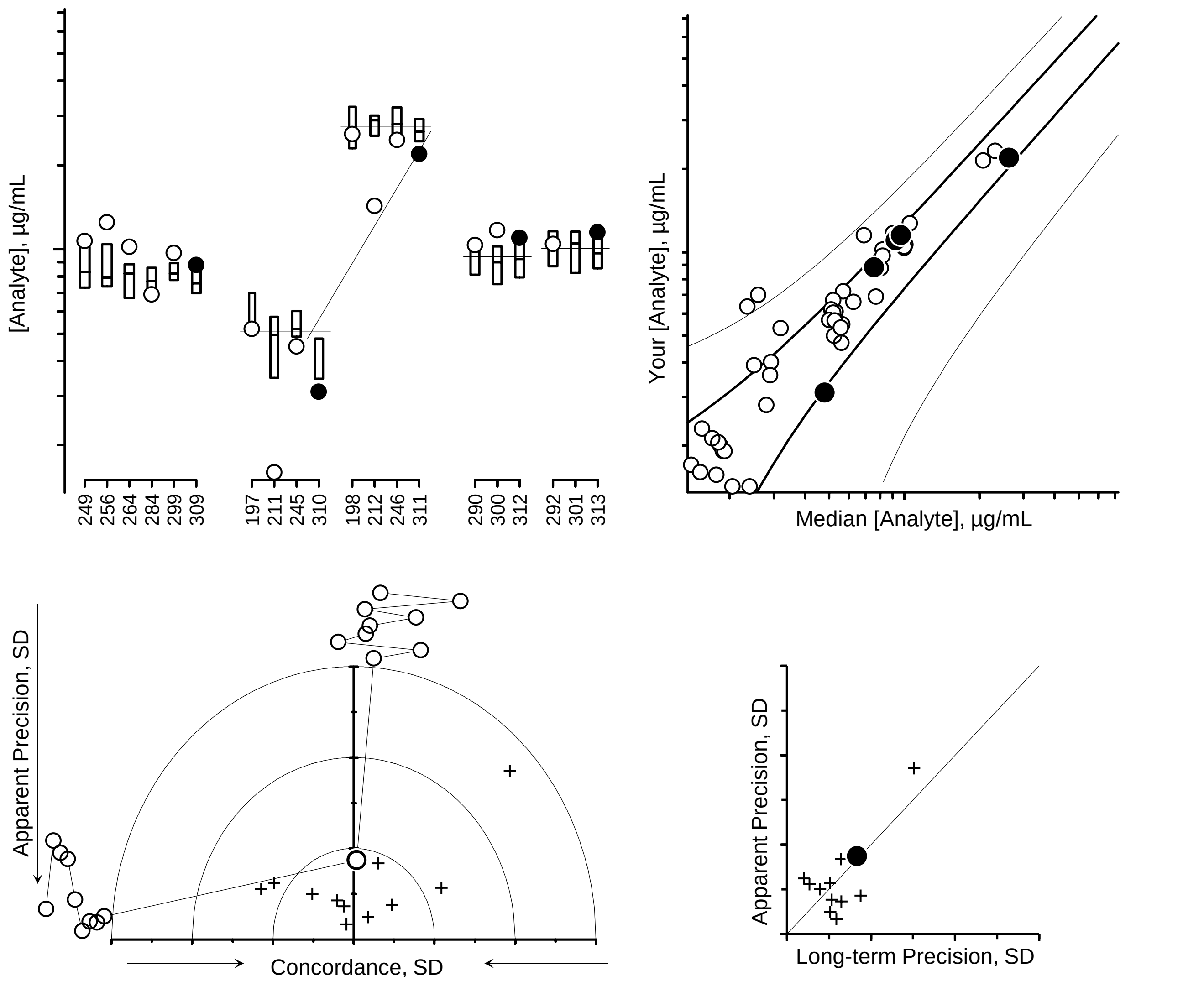
Total Retinol



Serum	History	Comments
#309	Lyophilized - 44:249, 46:256, 48:264, 52:284, 55:299	Augmented, multi-source (SRM 968c Level II)
#310	Lyophilized - 31:197, 35:211, 43:245	Same as #311, 1:3 diluted with stripped serum
#311	Lyophilized - 31:198, 35:212, 43:246	Augmented, multi-source
#312	Lyophilized - 53:290, 55:300	Native, single-source
#313	Fresh-frozen: 53:292, 55:301	Native, single-source, hemolyzed

Individualized RR LVII Report: FSV-BA

Retinyl Palmitate



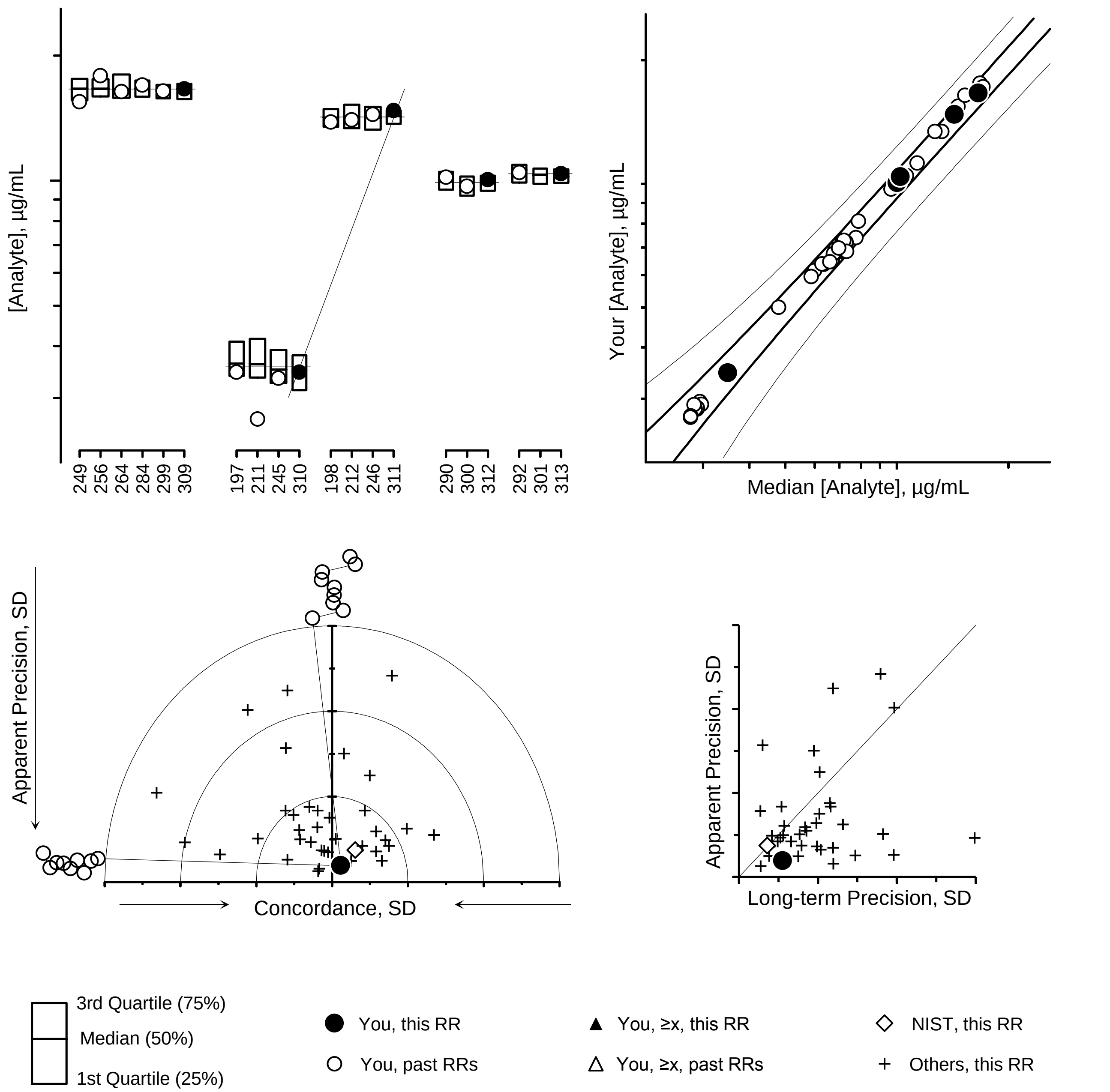
3rd Quartile (75%)
Median (50%)
1st Quartile (25%)

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

Serum	History	Comments
#309	Lyophilized - 44:249, 46:256, 48:264, 52:284, 55:299	Augmented, multi-source (SRM 968c Level II)
#310	Lyophilized - 31:197, 35:211, 43:245	Same as #311, 1:3 diluted with stripped serum
#311	Lyophilized - 31:198, 35:212, 43:246	Augmented, multi-source
#312	Lyophilized - 53:290, 55:300	Native, single-source
#313	Fresh-frozen: 53:292, 55:301	Native, single-source, hemolyzed

Individualized RR LVII Report: FSV-BA

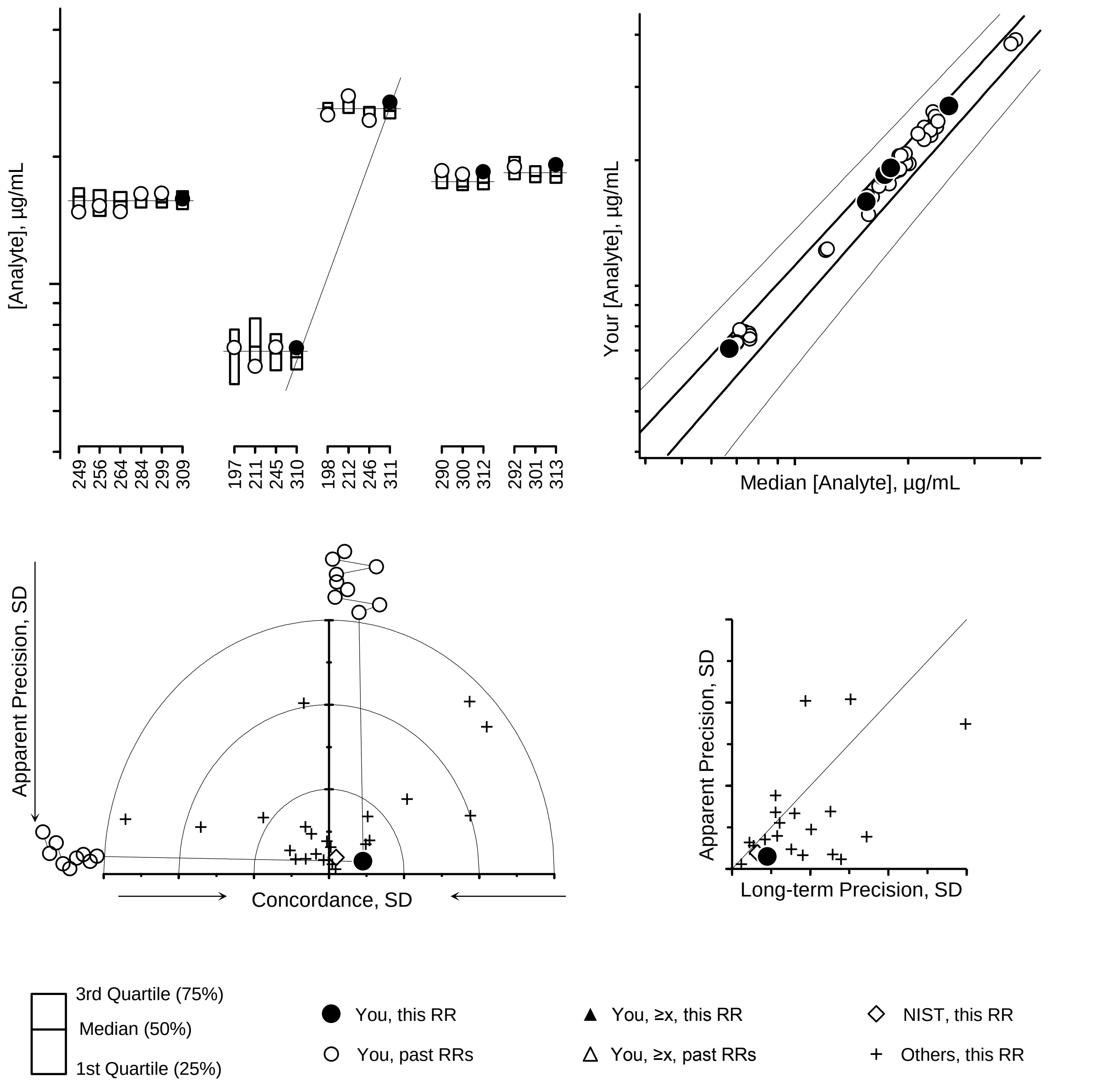
α-Tocopherol



Serum	History	Comments
#309	Lyophilized - 44:249, 46:256, 48:264, 52:284, 55:299	Augmented, multi-source (SRM 968c Level II)
#310	Lyophilized - 31:197, 35:211, 43:245	Same as #311, 1:3 diluted with stripped serum
#311	Lyophilized - 31:198, 35:212, 43:246	Augmented, multi-source
#312	Lyophilized - 53:290, 55:300	Native, single-source
#313	Fresh-frozen: 53:292, 55:301	Native, single-source, hemolyzed

Individualized RR LVII Report: FSV-BA

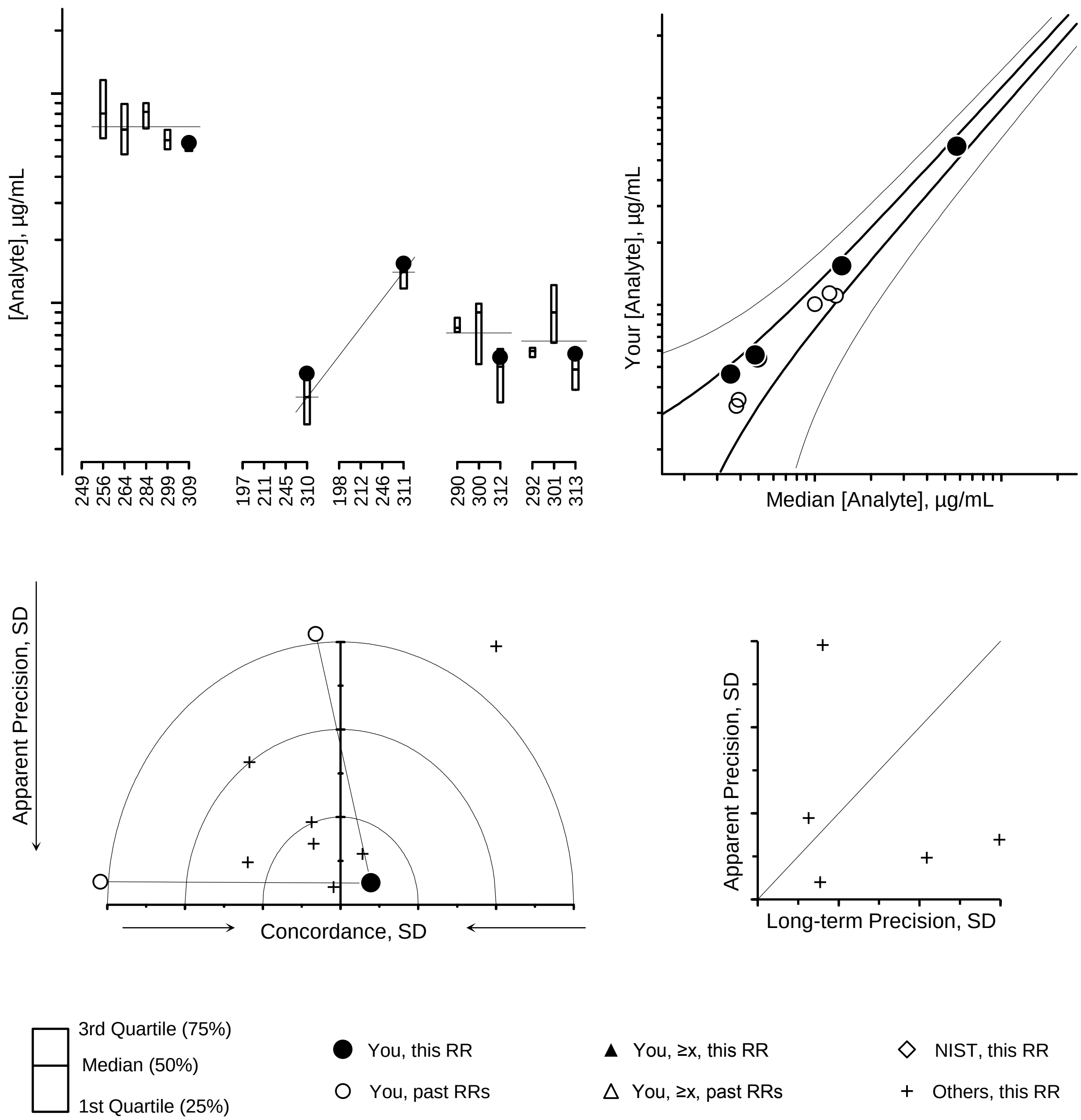
γ/β-Tocopherol



Serum	History	Comments
#309	Lyophilized - 44:249, 46:256, 48:264, 52:284, 55:299	Augmented, multi-source (SRM 968c Level II)
#310	Lyophilized - 31:197, 35:211, 43:245	Same as #311, 1:3 diluted with stripped serum
#311	Lyophilized - 31:198, 35:212, 43:246	Augmented, multi-source
#312	Lyophilized - 53:290, 55:300	Native, single-source
#313	Fresh-frozen: 53:292, 55:301	Native, single-source, hemolyzed

Individualized RR LVII Report: FSV-BA

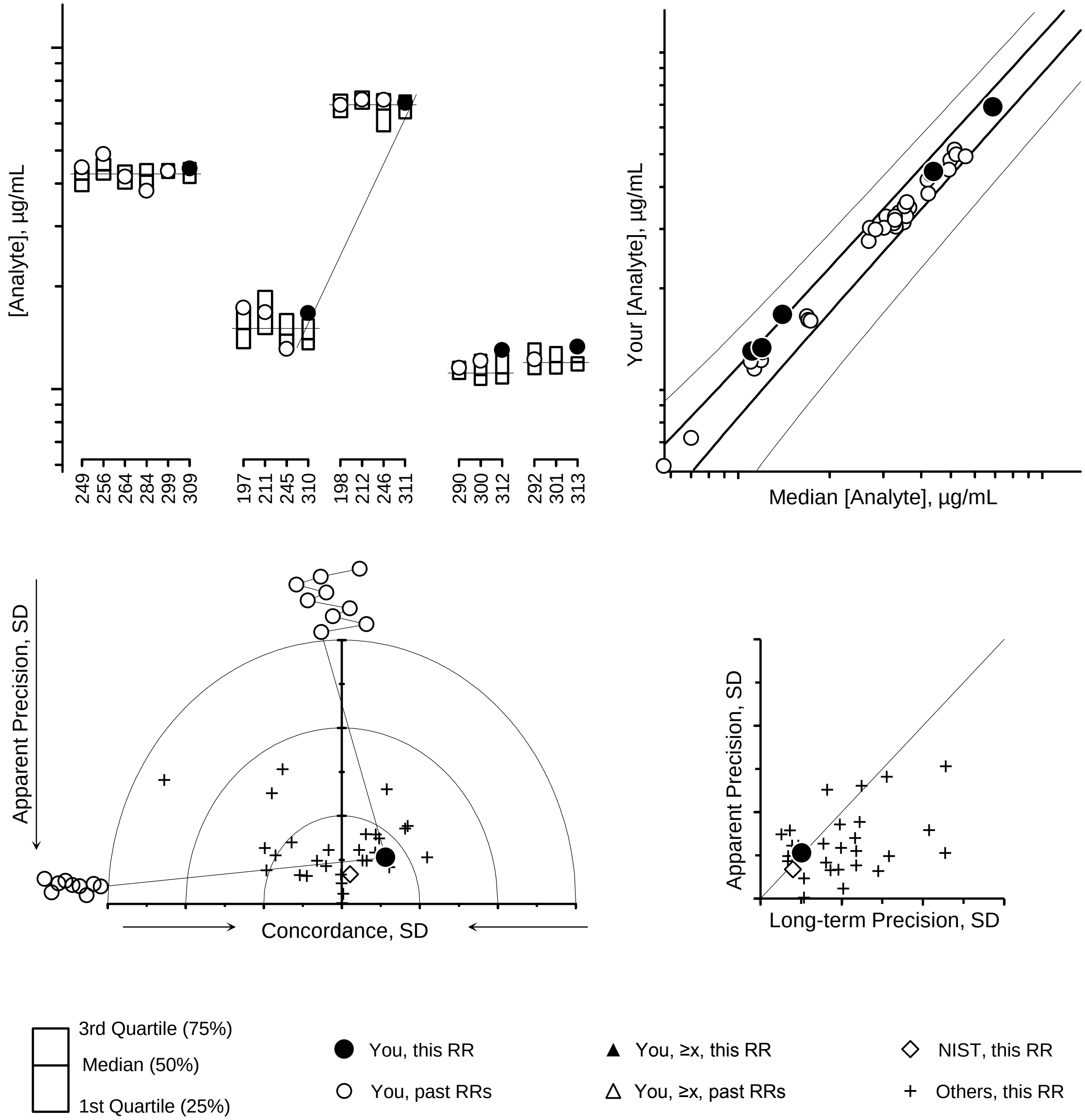
δ-Tocopherol



Serum	History	Comments
#309	Lyophilized - 44:249, 46:256, 48:264, 52:284, 55:299	Augmented, multi-source (SRM 968c Level II)
#310	Lyophilized - 31:197, 35:211, 43:245	Same as #311, 1:3 diluted with stripped serum
#311	Lyophilized - 31:198, 35:212, 43:246	Augmented, multi-source
#312	Lyophilized - 53:290, 55:300	Native, single-source
#313	Fresh-frozen: 53:292, 55:301	Native, single-source, hemolyzed

Individualized RR LVII Report: FSV-BA

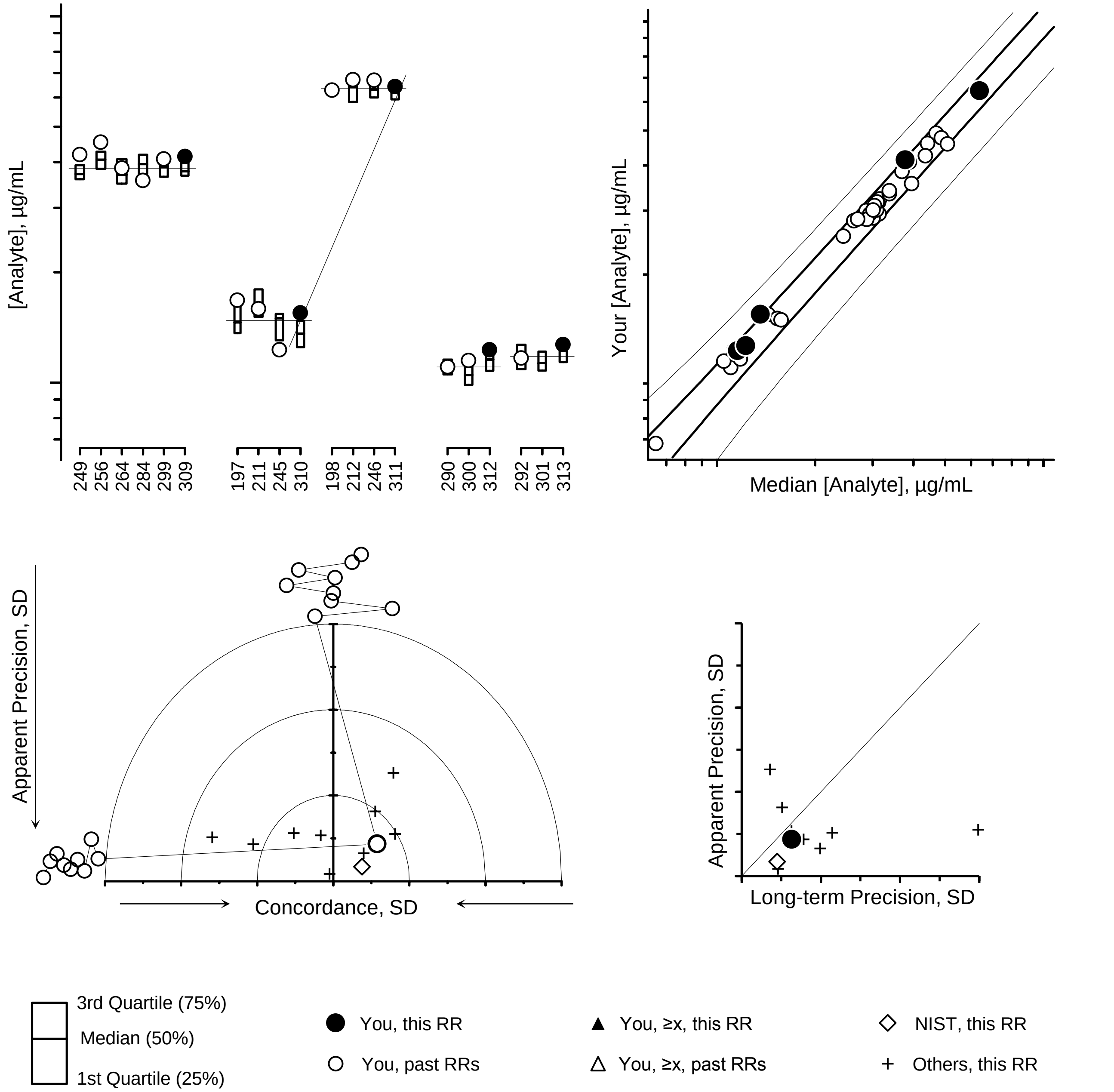
Total β-Carotene



Serum	History	Comments
#309	Lyophilized - 44:249, 46:256, 48:264, 52:284, 55:299	Augmented, multi-source (SRM 968c Level II)
#310	Lyophilized - 31:197, 35:211, 43:245	Same as #311, 1:3 diluted with stripped serum
#311	Lyophilized - 31:198, 35:212, 43:246	Augmented, multi-source
#312	Lyophilized - 53:290, 55:300	Native, single-source
#313	Fresh-frozen: 53:292, 55:301	Native, single-source, hemolyzed

Individualized RR LVII Report: FSV-BA

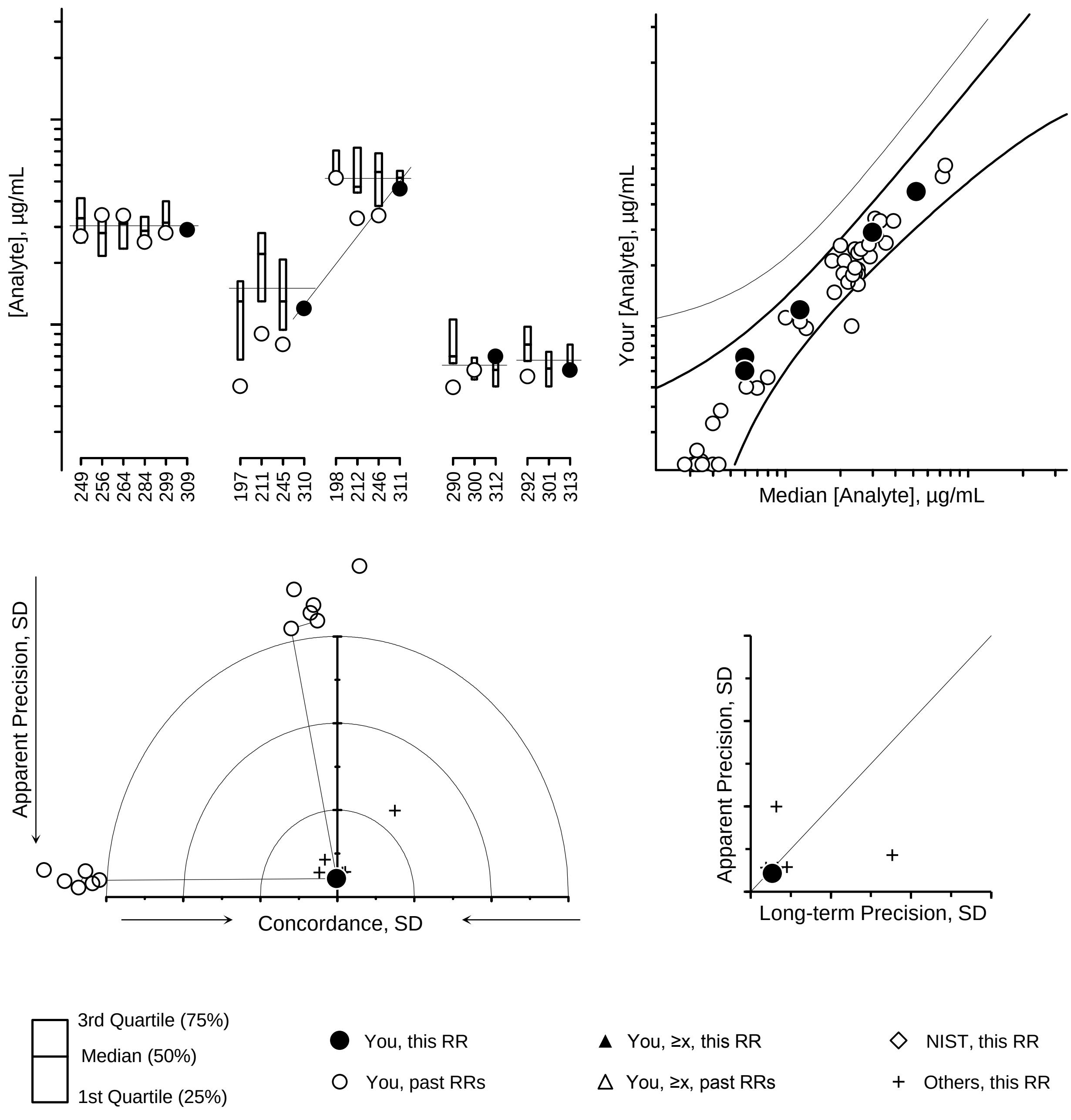
trans-β-Carotene



Serum	History	Comments
#309	Lyophilized - 44:249, 46:256, 48:264, 52:284, 55:299	Augmented, multi-source (SRM 968c Level II)
#310	Lyophilized - 31:197, 35:211, 43:245	Same as #311, 1:3 diluted with stripped serum
#311	Lyophilized - 31:198, 35:212, 43:246	Augmented, multi-source
#312	Lyophilized - 53:290, 55:300	Native, single-source
#313	Fresh-frozen: 53:292, 55:301	Native, single-source, hemolyzed

Individualized RR LVII Report: FSV-BA

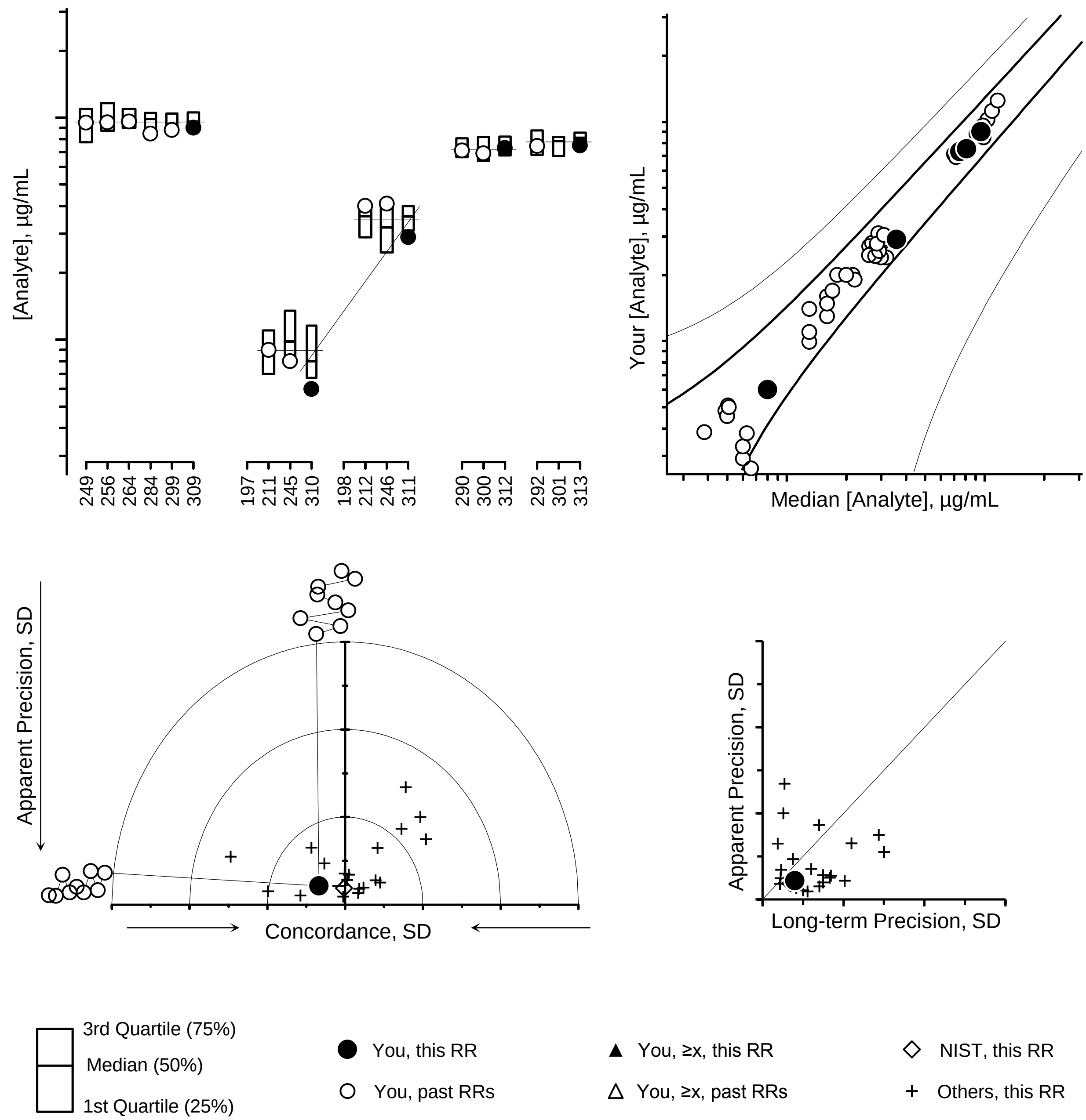
Total cis-β-Carotene



Serum	History	Comments
#309	Lyophilized - 44:249, 46:256, 48:264, 52:284, 55:299	Augmented, multi-source (SRM 968c Level II)
#310	Lyophilized - 31:197, 35:211, 43:245	Same as #311, 1:3 diluted with stripped serum
#311	Lyophilized - 31:198, 35:212, 43:246	Augmented, multi-source
#312	Lyophilized - 53:290, 55:300	Native, single-source
#313	Fresh-frozen: 53:292, 55:301	Native, single-source, hemolyzed

Individualized RR LVII Report: FSV-BA

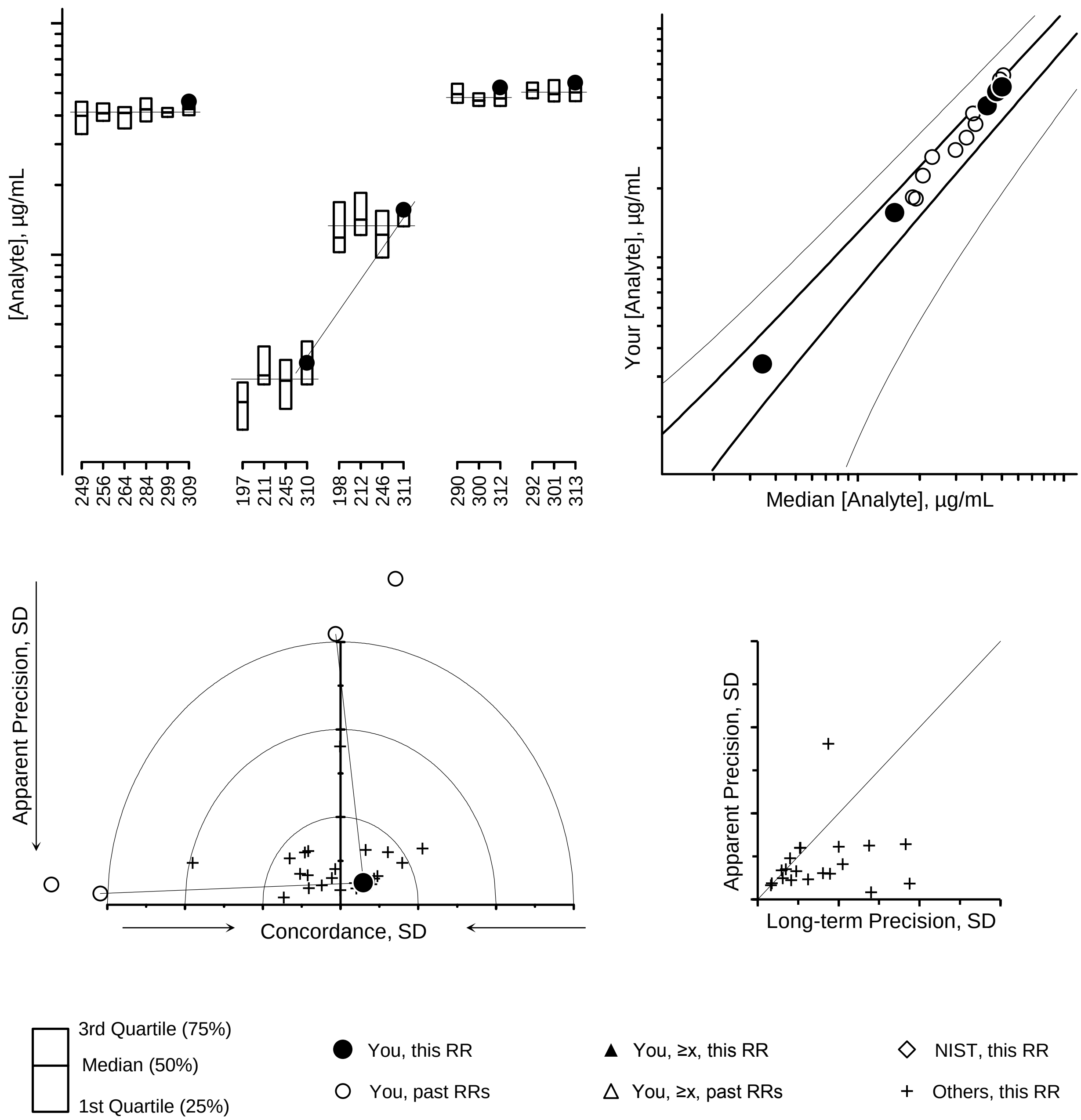
Total α-Carotene



Serum	History	Comments
#309	Lyophilized - 44:249, 46:256, 48:264, 52:284, 55:299	Augmented, multi-source (SRM 968c Level II)
#310	Lyophilized - 31:197, 35:211, 43:245	Same as #311, 1:3 diluted with stripped serum
#311	Lyophilized - 31:198, 35:212, 43:246	Augmented, multi-source
#312	Lyophilized - 53:290, 55:300	Native, single-source
#313	Fresh-frozen: 53:292, 55:301	Native, single-source, hemolyzed

Individualized RR LVII Report: FSV-BA

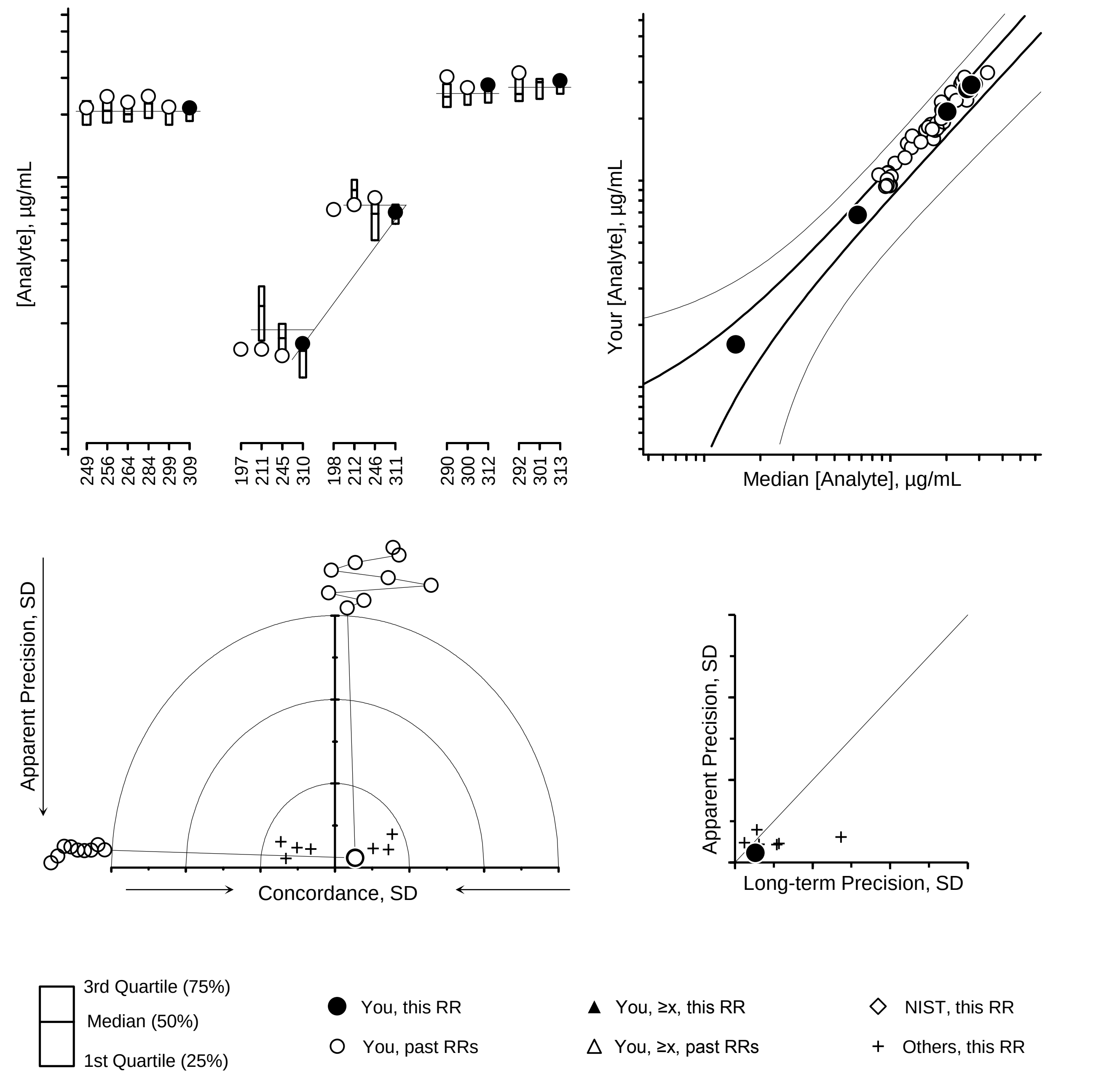
Total Lycopene



Serum	History	Comments
#309	Lyophilized - 44:249, 46:256, 48:264, 52:284, 55:299	Augmented, multi-source (SRM 968c Level II)
#310	Lyophilized - 31:197, 35:211, 43:245	Same as #311, 1:3 diluted with stripped serum
#311	Lyophilized - 31:198, 35:212, 43:246	Augmented, multi-source
#312	Lyophilized - 53:290, 55:300	Native, single-source
#313	Fresh-frozen: 53:292, 55:301	Native, single-source, hemolyzed

Individualized RR LVII Report: FSV-BA

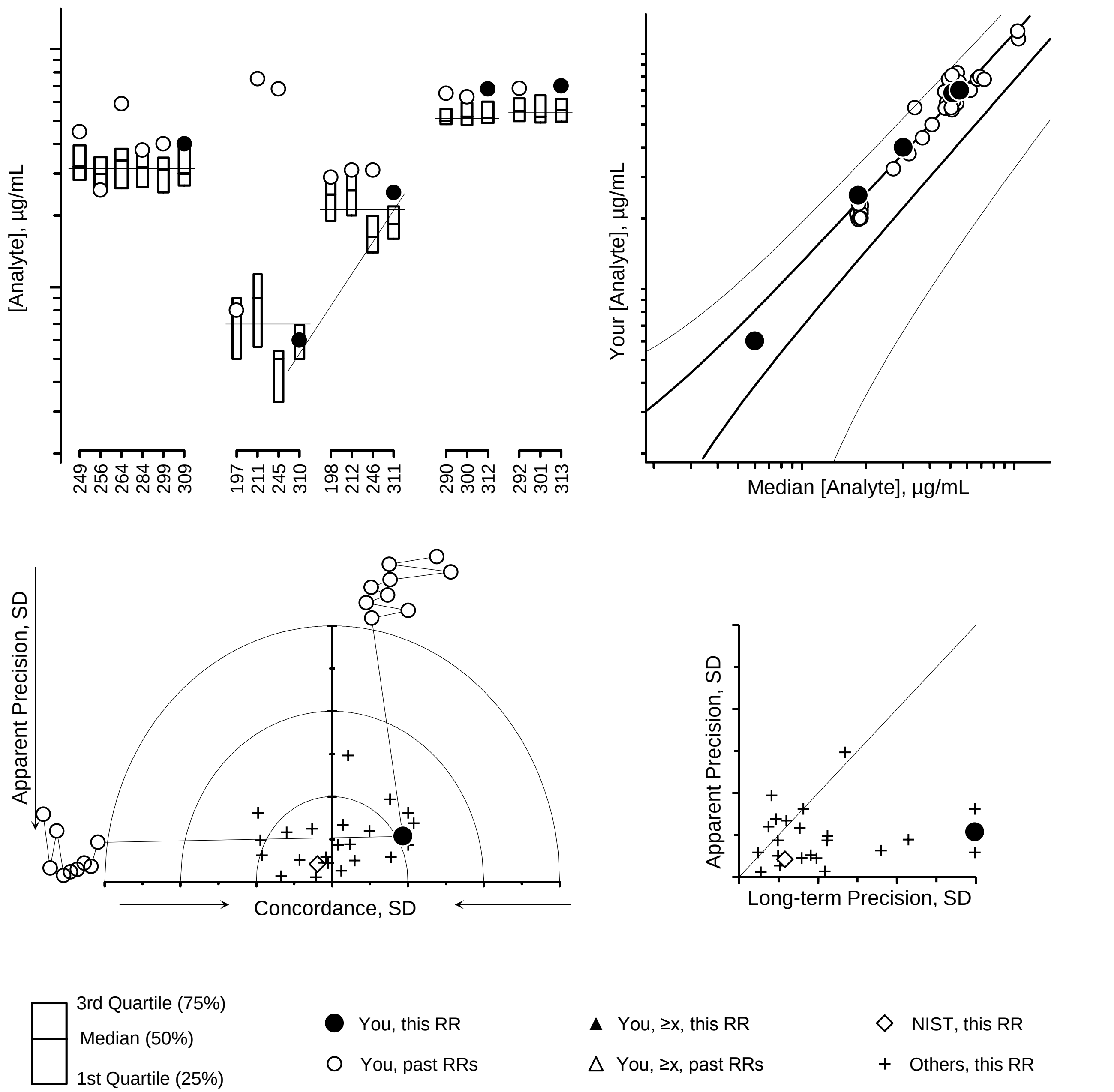
trans-Lycopene



Serum	History	Comments
#309	Lyophilized - 44:249, 46:256, 48:264, 52:284, 55:299	Augmented, multi-source (SRM 968c Level II)
#310	Lyophilized - 31:197, 35:211, 43:245	Same as #311, 1:3 diluted with stripped serum
#311	Lyophilized - 31:198, 35:212, 43:246	Augmented, multi-source
#312	Lyophilized - 53:290, 55:300	Native, single-source
#313	Fresh-frozen: 53:292, 55:301	Native, single-source, hemolyzed

Individualized RR LVII Report: FSV-BA

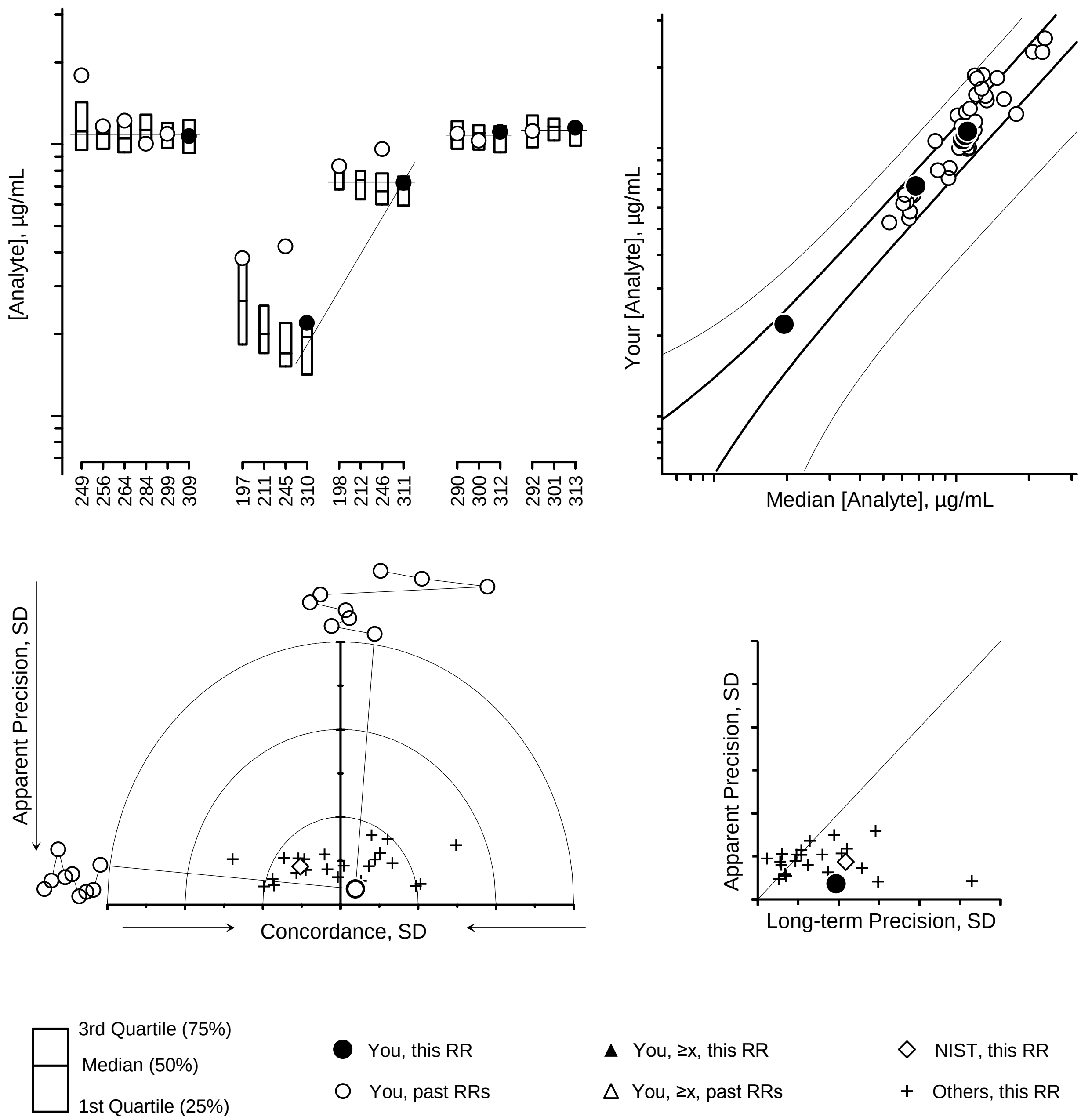
Total β-Cryptoxanthin



Serum	History	Comments
#309	Lyophilized - 44:249, 46:256, 48:264, 52:284, 55:299	Augmented, multi-source (SRM 968c Level II)
#310	Lyophilized - 31:197, 35:211, 43:245	Same as #311, 1:3 diluted with stripped serum
#311	Lyophilized - 31:198, 35:212, 43:246	Augmented, multi-source
#312	Lyophilized - 53:290, 55:300	Native, single-source
#313	Fresh-frozen: 53:292, 55:301	Native, single-source, hemolyzed

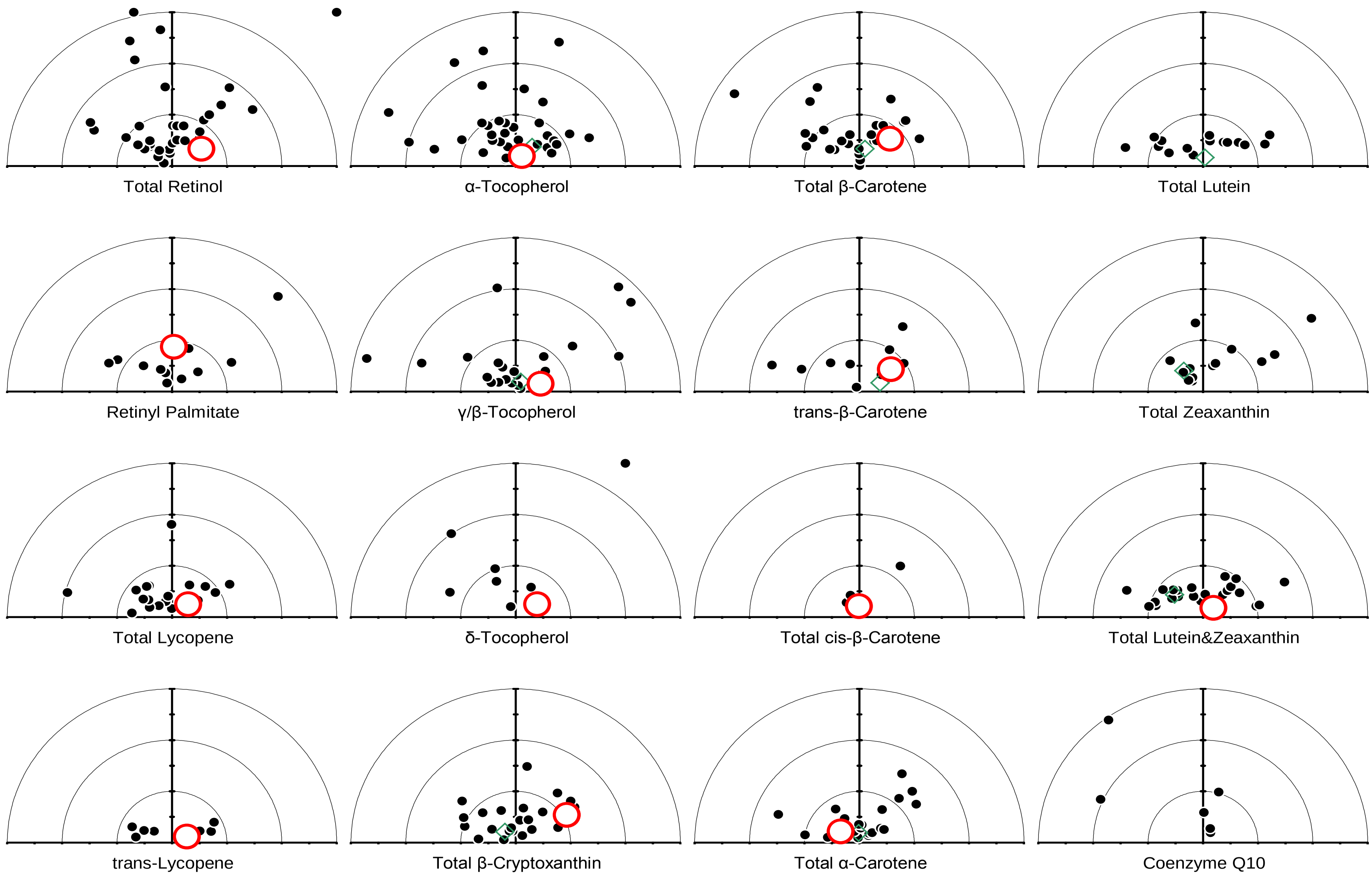
Individualized RR LVII Report: FSV-BA

Total Lutein&Zeaxanthin



Serum	History	Comments
#309	Lyophilized - 44:249, 46:256, 48:264, 52:284, 55:299	Augmented, multi-source (SRM 968c Level II)
#310	Lyophilized - 31:197, 35:211, 43:245	Same as #311, 1:3 diluted with stripped serum
#311	Lyophilized - 31:198, 35:212, 43:246	Augmented, multi-source
#312	Lyophilized - 53:290, 55:300	Native, single-source
#313	Fresh-frozen: 53:292, 55:301	Native, single-source, hemolyzed

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



Appendix E. Shipping Package Inserts for RR22

The following five items were included in each package shipped to an RR22 participant:

- Cover letter
- Protocol for Preparation and Analysis of the Ascorbic Acid Solid Control Material
- Preparation and Validation of Ascorbic Acid Solid Control Material Datasheet
- Analysis of Control Materials and Test Samples Datasheet
- Packing List and Shipment Receipt Confirmation Form

The cover letter, preparation protocol, and the two datasheets 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 20899-0001

November 8, 2004

Dear Colleague:

The samples within this package constitute Vitamin C Round Robin 22 (RR22) of the 2005 Micronutrients Measurement Quality Assurance Program.

RR22 consists of four vials of frozen serum *test samples* (#23, #33, #42, and #70), one vial of ascorbic acid *solid control material* (Control), and two vials of frozen *serum control materials* (Control #1 and Control #2). Please follow the attached protocols when you prepare and analyze these samples. If you cannot prepare the *solid control* solutions gravimetrically, please prepare equivalent solutions volumetrically and report the exact volumes used. (Routine 0.5 g gravimetric measurements are generally 10-fold more accurate than routine 0.5 mL volumetric measurements.)

The two *serum control materials* are a new component of the M^2QAP for Vitamin C. Please use these materials to validate the performance of your measurement system before you analyze the *test samples*. The target value and ≈95% confidence interval for *Control #1* is 8.41 ±0.61 µmol/L sample; the target value and ≈95% confidence interval for *Control #2* is 28.05 ±0.49 µmol/L sample.

Please be aware that sample contact with any oxidant-contaminated surface (vials, glassware, etc.) may degrade your measurement system's performance (SA Margolis and E Park, "Stability of Ascorbic Acid in Solutions in Autosampler Vials", *Clinical Chemistry* **2001**, *47*(8), 1463-1464). You should suspect such degradation if you observe unusually large variation in replicate analyses.

The report for RR21 was mailed the week of Nov 1, 2004. If you find your results for RR21 unsatisfactory, we recommend that you obtain **Standard Reference Material (SRM) 970 Ascorbic Acid in Serum** to validate your methodology and value assign in-house control materials. This SRM may be purchased from the Standard Materials Reference Program at NIST (Tel: 301-975-6776, Fax: 301-948-3730, or e-mail: srminfo@nist.gov).

If you have any questions or concerns about the Vitamin C Micronutrients Measurement Quality Assurance Program please contact Jeanice Brown Thomas at tel: 301-975-3120, fax: 301-977-0685, or e-mail: jbthomas@nist.gov.

We ask that you return your results for these RR22 samples *before* **March 2, 2005**. We would appreciate receiving your results as soon as they become available. Please use the attached form. Your results will be kept confidential.

Sincerely,

Jeanice Brown Thomas

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

Enclosures: Protocols, Preparation and Analysis of Control Materials and Analysis of Test Samples
RR22 Report Form for Ascorbic Acid Solid Control Material Preparation
RR22 Report Form for Control Material and Test Sample Analyses

NIST

Micronutrient Measurement Quality Assurance Program for
Vitamin C

Please Read Through Completely BEFORE Analyzing Samples

Protocol for Preparation and Analysis of the Ascorbic Acid Solid Control Material

The *ascorbic acid solid control material* (in the amber vial) should be prepared and used in the following manner:

1) Prepare at least 500 mL of 5% mass fraction metaphosphoric acid (MPA) in distilled water. This solution will be referred to as the “Diluent” below.

2) Weigh 0.20 to 0.22 g of the ascorbic acid solid control material to 0.0001 g (if possible), dissolve it in the Diluent in a 100 mL volumetric flask, and dilute with the Diluent to the 100 mL mark. Weigh the amount of Diluent added to 0.1 g. Record the weights. The resulting material will be referred to as the “Stock Solution” below.

3) Prepare three dilute solutions of the Stock Solution as follows:

   Dilute Solution 1: Weigh 0.500 mL of the Stock Solution to 0.0001 g into a 100 mL volumetric flask; dilute with Diluent to the 100 mL mark. Record the weight.

   Dilute Solution 2: Weigh 0.250 mL of the Stock Solution to 0.0001 g into a 100 mL volumetric flask; dilute with Diluent to the 100 mL mark. Record the weight.

   Dilute Solution 3: Weigh 0.125 mL of the Stock Solution to 0.0001 g into a 100 mL volumetric flask; dilute with Diluent to the 100 mL mark. Record the weight.

4) Calculate and record the total ascorbic acid concentrations, [TAA], in these Dilute Solutions. If you follow the above gravimetric preparation directions, the [TAA] in µmol/L is calculated:

$$[\mathrm{TAA}]_{\mathrm{DS}} = \frac{(\text{g Stock Solution in Dilute Solution})\cdot(\text{g AA in Stock Solution})\cdot(56785\ \mu\text{mol/g}\cdot\text{L})}{(\text{g AA in Stock Solution})+(\text{g Diluent in Stock Solution})}$$

   For example, if you prepared the Stock Solution with 0.2000 g of solid ascorbic acid and 103.0 g of Diluent, then 0.5 mL of the Stock Solution should weigh (0.2+103)/200 = 0.52 g and $[\mathrm{TAA}]_{\mathrm{DS1}}$ = (0.52 g)(0.2 g)·(56785 µmol/g·L)/(0.2 + 103 g) = 57.2 µmol/L. Likewise, 0.25 mL of the Stock Solution should weigh 0.26 g and $[\mathrm{TAA}]_{\mathrm{DS2}}$ = 28.4 µmol/L and 0.125 mL should weigh 0.13 g and $[\mathrm{TAA}]_{\mathrm{DS3}}$ = 14.2 µmol/L.

5) Measure the ultraviolet absorbance spectrum of Dilute Solution 1 against the Diluent as the blank using paired 1 cm path length cuvettes. Record the absorbance at 242, 243, 244, and 245 nm. Record the maximum absorbance (A_{max}) within this region. Record the wavelength (λ_{max}) at which this maximum occurs.

   The extinction coefficient ($E^{1\%}$) of ascorbic acid at λ_{max} (using a cell with a 1 cm path length) of Dilute Solution #1 can be calculated:

$$E^{1\%}\left(\frac{dL}{g\cdot cm}\right)=\frac{(A_{max})\cdot((g\ AA\ in\ Stock\ Solution)+(g\ Diluent\ in\ Stock\ Solution))}{(g\ Stock\ Solution\ in\ Dilute\ Solution\ 1)\cdot(g\ AA\ in\ Stock\ Solution)}$$

If your spectrophotometer is properly calibrated, λ_{max} should be between 243 and 244 nm and $E^{1\%}$ should be 550 ± 30 dL/g·cm. If they are not, you should calibrate the wavelength and/or absorbance axes of your spectrophotometer and repeat the measurements.

6) Measure and record the concentration of total ascorbic acid in all three dilute solutions and in the 5% MPA Diluent in duplicate using *exactly* the same method that you will use for the serum control materials and test samples, including any enzymatic treatment. We recommend that you analyze these solutions in the following order: Diluent, Dilute Solution 1, Dilute Solution 2, Dilute Solution 3, Dilute Solution 3, Dilute Solution 2, Dilute Solution 1, Diluent.

   a) Compare the values of the duplicate measurements. *Are you satisfied that your measurement precision is adequate?*

   b) Compare the measured with the calculated [TAA] values. This is most conveniently done by plotting the measured values on the y-axis of a scatterplot against the calculated values on the x-axis. The line through the four {calculated, measured} data pairs should go through the origin with a slope of 1.0. *Are you satisfied with the agreement between the measured and calculated values?*

   Do **not** analyze the serum control materials or test samples until you are satisfied that your system is performing properly!

7) Once you have confirmed that your system is properly calibrated, analyze the serum control materials (see protocol below). The target values for these materials are:

   Control #1: 8.5 ±0.5 µmol/L of sample

   Control #2: 28.1 ±1.0 µmol/L of sample.

   If your measured values are not close to these target values, please review your sample preparation procedure and whether you followed *exactly* the same measurement protocol the solutions prepared from the solid control material as you used for these serum controls. If the protocols differ, please repeat from Step 6 using the proper protocol. If the proper protocol was used, your measurement system may not be suitable for MPA-preserved samples. Please contact us: 301-975-3120 or Jeanice.BrownThomas@NIST.gov.

   Do **not** analyze the test samples until you are satisfied that your system is performing properly and is suitable for the analysis of MPA-preserved serum!

Protocol for Analysis of the Serum Control Materials and Test Samples

The *serum control materials* and *test samples* are in sealed ampoules. They were prepared by adding equal volumes of 10% MPA to spiked human serum. We have checked the samples for stability and homogeneity. Only the total ascorbic acid is stable. While these samples contain some dehydroascorbic acid, its content is variable. Therefore, only total ascorbic acid should be reported. The *serum control materials* and *test samples* should be defrosted by warming at 20 °C for not more than 10 min otherwise some irreversible degradation may occur.

Each *serum test sample* contains between 0.0 and 80.0 µmol of total ascorbic acid/L of solution. The total ascorbic acid in each ampoule should be measured in duplicate. Please report your results in µmol/(L of the sample solution) rather than µmol/(L of serum NIST used to prepare the sample).

Participant #: _______ Date: __________

Vitamin C Round Robin 22

NIST Micronutrient Measurement Quality Assurance Program

Preparation and Validation of Ascorbic Acid Solid Control Material

STOCK SOLUTION

Mass of ascorbic acid in the Stock Solution .... __________ g

Mass of 5% MPA Diluent added to the 100 mL volumetric flask................ __________ g

DILUTE SOLUTION 1

Mass of added stock solution (0.5 mL).. __________ g

Mass of 5% MPA Diluent added to the 100 mL volumetric flask................ __________ g

Absorbance of Dilute Solution 1 at 242 nm.. __________ AU

Absorbance of Dilute Solution 1 at 243 nm.. __________ AU

Absorbance of Dilute Solution 1 at 244 nm.. __________ AU

Absorbance of Dilute Solution 1 at 245 nm.. __________ AU

Absorbance of Dilute Solution absorbance maximum .... __________ AU

Wavelength of maximum absorbance.. __________ nm

Calculated $E^{1\%}$ .... __________ dL/g·cm

Calculated $[TAA]_{DS1}$ .... __________ µmol/L

DILUTE SOLUTION 2

Mass of added stock solution (0.25 mL)... __________ g

Mass of 5% MPA Diluent added to the 100 mL volumetric flask................ __________ g

Calculated $[TAA]_{DS2}$ .... __________ µmol/L

DILUTE SOLUTION 3

Mass of added stock solution (0.125 mL)... __________ g

Mass of 5% MPA Diluent added to the 100 mL volumetric flask................ __________ g

Calculated $[TAA]_{DS3}$ .... __________ µmol/L

Please return *before* **March 2, 2005** to:

MMQAP
100 Bureau Drive, Stop 8392
Gaithersburg, MD 20899-8392

Fax: 301-977-0685
Email: david.duewer@nist.gov

Participant #: _______ Date: __________

Vitamin C Round Robin 22

NIST Micronutrient Measurement Quality Assurance Program

Analysis of Control Materials and Test Samples

Sample	Replicate 1	Replicate 2	Units
Dilute Solution 1			µmol/L of Dilute Solution
Dilute Solution 2			µmol/L of Dilute Solution
Dilute Solution 3			µmol/L of Dilute Solution
5% MPA Diluent			µmol/L of Diluent
Serum Control #1			µmol/L of Sample *Target:* 8.5 ±0.5 µmol/L
Serum Control #2			µmol/L of Sample *Target:* 28.1 ±1.0 µmol/L
Serum Test Sample #23			µmol/L of Sample
Serum Test Sample #33			µmol/L of Sample
Serum Test Sample #42			µmol/L of Sample
Serum Test Sample #70			µmol/L of Sample

Were samples frozen upon receipt? Yes | No

Analysis method: HPLC-EC | HPLC-Fluor DAB | HPLC-OPD | HPLC-UV | AO-OPD | Other
If "Other", please describe:

COMMENTS:

Please return *before* **March 2, 2005** to:

MMQAP
100 Bureau Drive, Stop 8392
Gaithersburg, MD 20899-8392

Fax: 301-977-0685
Email: david.duewer@nist.gov

Participant #: ________ Date: ____________

Vitamin C Round Robin 22

NIST Micronutrients Measurement Quality Assurance Program

Packing List and Shipment Receipt Confirmation Form

This box contains one vial each of the following **seven** VitC M^{2}QAP samples:

Sample	Form
VitC #23	Liquid frozen (1:1 serum:10% MPA)
VitC #33	Liquid frozen (1:1 serum:10% MPA)
VitC #42	Liquid frozen (1:1 serum:10% MPA)
VitC #70	Liquid frozen (1:1 serum:10% MPA)
Control #1	Liquid frozen (1:1 serum:10% MPA)
Control #2	Liquid frozen (1:1 serum:10% MPA)
Control	Solid AA

Please 1) Open the pack immediately
2) Check that it contains one vial each of the above samples
3) Check if the samples arrived frozen
4) Store the samples 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 of the vials intact? Yes | No
If "No", which one(s) were damaged?

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

4) Did the samples arrive frozen? Yes | No

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

6) When do you anticipate analyzing these samples? ________________________

Your prompt return of this information is appreciated.

The M^{2}QAP Gang

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

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

Appendix F. Final Report for RR22

The following two 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 their previous distributions, if any, and
    - 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

April 14, 2005

Dear Colleague:

Enclosed is the summary report of the results for Round Robin 22 (RR22) for the measurement of total ascorbic acid (TAA, ascorbic acid plus dehydroascorbic acid) in human serum. Included in this report are: a summary of data for all laboratories and a summary of individual laboratory performance and interlaboratory accuracy and repeatability. As in previous reports, the estimated standard deviations (eSD) for the measurements are defined as 0.74x interquartile range and the estimate coefficients of variation (eCV) are defined as 100x eSD/median.

RR 22 consists of four *test samples* (#23, #33, #42, and #70), two *serum control materials*, and one *solid control material* for preparation of TAA control solutions. Details regarding the samples can be found in the enclosed report.

If you have concerns regarding your laboratory's performance, we suggest that you obtain and analyze a unit of Standard Reference Material (SRM) 970, Vitamin C in Frozen Human Serum. SRM 970 can be purchased from the NIST SRM Program at phone: 301-975-6776; fax: 301-948-3730. If your measured values do not agree with the certified values, we suggest that you contact us for consultation.

Samples for the second vitamin C round robin study (RR 23) for the 2005 Vitamin C in Serum QA Program will be shipped (**during the week of May 16**). If you have questions or concerns 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; or fax: 301-977-0685.

Sincerely,

Jeanice B Thomas

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

Enclosures

NIST

The NIST M^{2}QAP Vitamin C Round Robin 22 (RR22) report consists of

Page	"Individualized" Report
1	Summarizes your reported values for the nominal 55 mmol/L solution you prepared from the ascorbic acid solid control sample, the two serum control samples, and the four serum test samples.
2	Graphical summary of your RR 22 sample measurements.

Page	"All Lab" Report
1	A tabulation of results and summary statistics for Total Ascorbic Acid [TAA] in the RR22 samples and control/calibration solutions.

Serum-based Samples. Two serum controls and four unknowns were distributed in RR22.

CS1 SRM 970 level 1, ampouled in mid-1998.

CS2 SRM 970 level 2, ampouled in mid-1998.

S22:1 Serum 23, ampouled in late 2001, previously distributed as sample S17:1 (RR17, Sep-02), S19:2 (RR19, Sep-03), and S21:2 (Sep-04). An augmented serum.

S22:2 Serum 33, ampouled in late 2001, previously distributed as sample S17:2 (RR17, Sep-02), S18:1 (RR18, Mar-03) and S20:1 (RR20, Mar-04). An augmented serum.

S22:3 Serum 42, ampouled in late 2001, previously distributed as sample S18:2 (RR18, Mar-03), S19:3 (RR19, Sep-03) and S21:3 (RR21, Sep-04). An augmented serum.

S22:4 Serum 70, SRM 970 level 2, ampouled in mid-1998. This material was distributed with identification in RR11 (Oct-98) and RR12 (Mar-99) and as samples S13-2 (RR13, Mar-00), S14-4 (RR14, Mar-01), S15:2 (RR15, Sep-01), S18:3 (RR18, Mar-03), and S20:4 (RR20, Mar-04). An augmented serum.

Results.

1) All participants who prepared the four control/calibration solutions (the three "Dilute Solutions" and the 5% MPA "Diluent") did so correctly. The criteria used to evaluate this success are: the density of the 5% MPA (≈1.03 g/mL), the observed wavelength maximum of "Dilute Solution 1"(≈244 nm), the observed absorbance at that maximum (≈0.55 OD), the calculated $E^{1\%}$ of Solution 1"(≈550 dL/g·cm).

2) Judging from the calibration parameters calculated for the control/calibration solutions (intercepts close to 0.0 and slopes close to 1.0), the measurement systems for most participants are well calibrated, although the slope for one participant was 11% lower than expected and the intercept for another participant was significantly different from zero.

3) Several participants reported values for high-level control that were well outside the target range of 26 µmol/L to 30 µmol/L. If the measured values for the control samples are not close to the targets, even if your measured and calculated values for the calibration solutions agree, there is a problem with your measurement system.

4) There is no evidence of sample degradation with any of the samples distributed.

Appendix G. “All-Lab Report” for RR22

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.

Micronutrients Measurement Quality Assurance Program for Total Ascorbic Acid
"Round Robin" 22 - March 2005

		Control / Calibration Samples											MPA	Dilute Solution 1			Samples											
		Gravimetric, µmol/L			Measured, µmol/L				Calibration Parameters				Density	Spectrophotometry			Measured, µmol/L						Corrected, µmol/L					
Lab	Date	Dil:1	Dil:2	Dil:3	Dil:1	Dil:2	Dil:3	MPA	Inter	Slope	R^2	SEE	g/mL	λ_{max}	A_{max}	$E^{1\%}$	CS#1	CS#2	S22:1	S22:2	S22:3	S22:4	CS#1	CS#2	S22:1	S22:2	S22:3	S22:4
VC-MA	08/03/05	56.4	28.5	14.0	59.9	30.6	15.3	0.0	0.21	1.06	1.000	0.2	1.032	243.	0.5591	562.9	8.6	29.0	9.6	22.7	35.6	29.4	7.9	27.1	8.9	21.2	33.4	27.6
VC-MB	02/03/05	56.6	28.0	14.3	53.7	26.1	13.7	0.0	-0.07	0.95	1.000	0.3	1.032	244.	0.5612	563.3	8.3	28.9	7.7	22.0	35.1	28.4	8.8	30.6	8.2	23.2	37.1	30.0
VC-MC	29/11/04	57.2	27.5	13.5	50.8	25.5	12.5	0.0	0.40	0.89	0.999	0.6	1.030	243.	0.5553	551.1	8.3	27.0	10.3	23.9	37.6	27.0	8.9	30.0	11.1	26.4	42.0	30.0
VC-ME	14/03/03	56.7	28.3	14.0	57.9	29.5	14.8	0.0	0.33	1.02	1.000	0.4	1.033	243.	0.5315	532.5	8.4	27.7	10.2	22.6	31.3	28.2	7.9	26.9	9.7	21.8	30.4	27.4
VC-MG	08/02/05	57.6	29.1	14.5	58.4	28.1	13.3	0.0	-0.83	1.02	0.999	1.0	1.033	243.9	0.5700	561.6	8.7	30.4	11.4	24.7	40.6	34.1	9.3	30.6	12.0	25.1	40.7	34.3
VC-MH	13/01/05	62.5	31.2	15.2	62.9	31.8	15.7	0.2	0.34	1.00	1.000	0.2	1.030	244.	0.6120	556.2	8.3	27.9	8.5	22.5	34.3	28.2	7.9	27.5	8.1	22.1	33.9	27.8
VC-MI	12/01/05	56.0	28.0	14.3	55.2	26.8	13.4	1.0	0.15	0.97	0.999	1.0	1.033				7.1	26.5	4.1	16.1	26.4	26.0	7.1	27.0	4.0	16.4	26.9	26.5
VC-MJ	15/12/04	58.7	28.8	15.0	57.4	30.4	15.0	0.4	0.80	0.97	0.998	1.3	1.023				10.3	27.2	11.6	22.6	31.1	26.5	9.7	27.0	11.1	22.3	31.1	26.4
VC-MK	25/02/05	58.9	28.4	13.9	65.2	35.2	19.8	1.5	3.40	1.07	0.996	2.2	1.032	244.	0.5854	564.0	12.4	35.3	14.6	29.7	41.3	35.0	8.4	29.9	10.5	24.6	35.5	29.6
VC-MT	01/03/05	56.7	27.8	14.4	56.9	28.4	13.9	0.0	-0.12	1.01	1.000	0.5	1.032	244.	0.5603	561.3	8.7	28.9	9.5	21.2	35.7	29.8	8.8	28.8	9.6	21.1	35.5	29.6
VC-MY	22/02/05																11.5	49.8	11.1	34.5	54.9	46.0	11.5	49.8	11.1	34.5	54.9	46.0
	N	10	10	10	10	10	10	10				N	10	8	8	8	11	11	11	11	11	11	11	11	11	11	11	11
	Average	57.7	28.5	14.3	57.8	29.2	14.7	0.3				Average	1.031	243.6	0.5669	556.6	9.1	30.8	9.9	23.8	36.7	30.8	8.8	30.5	9.5	23.5	36.5	30.5
	SD	1.9	1.0	0.5	4.2	2.9	2.0	0.5				SD	0.003	0.5	0.0236	10.7	1.6	6.7	2.7	4.7	7.4	5.8	1.2	6.6	2.2	4.5	7.5	5.6
	Min	56.0	27.50	13.5	50.8	25.47	12.5	0.0				Min	1.023	243.0	0.5315	532.5	7.1	26.5	4.1	16.1	26.4	26.0	7.1	26.9	4.0	16.4	26.9	26.4
	%25	56.6	27.98	14.0	55.6	27.09	13.4	0.0				%25	1.031	243.0	0.5582	554.9	8.3	27.4	9.0	22.2	32.8	27.6	7.9	27.1	8.5	21.5	32.3	27.5
	Median	56.9	28.32	14.3	57.6	28.97	14.4	0.0				Median	1.032	244.0	0.5608	561.5	8.6	28.9	10.2	22.6	35.6	28.4	8.8	28.8	9.7	22.3	35.5	29.6
	%75	58.5	28.73	14.5	59.5	30.53	15.2	0.3				%75	1.033	244.0	0.5739	563.0	9.5	29.7	11.3	24.3	39.1	31.9	9.1	30.3	11.1	24.8	38.9	30.0
	Max	62.5	31.19	15.2	65.2	35.21	19.8	1.5				Max	1.033	244.0	0.6120	564.0	12.4	49.8	14.6	34.5	54.9	46.0	11.5	49.8	12.0	34.5	54.9	46.0
	MADe	0.9	0.6	0.4	3.5	2.8	1.4	0.0				eSD	0.002	0.1	0.0109	3.3	0.4	2.1	1.8	1.9	6.4	2.1	1.3	2.6	2.1	1.8	6.5	3.0
	CV	2	2	3	6	10	10					CV	0.19	0.03	1.9	0.6	5	7	18	8	18	7	14	9	22	8	18	10

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Appendix H. Representative "Individualized Report" for RR22

Each participant in RR22 received an "Individualized Report" reflecting their reported results. The following two pages are the "Individualized Report" for participant "VC-MA".

Vitamin C "Round Robin" 22 Report: Participant VC-MA

Date	RR	Method	MPA Density g/mL	Dilute Solution 1 Spectrophotometry λ_{max}	A_{max}	$E^{1\%}$	Control/Calibration Solutions Y_{meas} = Inter + Slope* X_{grav} Inter	Slope	R^2	SEE
12/12/02	17	HPLC-EC	1.026	242.0	0.552	551.0	-0.3	1.06	1.000	0.49
03/20/03	18	HPLC-EC	1.026	244.0	0.509	563.1	-0.1	1.02	1.000	0.18
11/13/03	19	HPLC-EC	1.026	243.0	0.584	561.9	1.1	1.03	0.998	1.24
02/23/04	20	HPLC-EC	1.031	243.0	0.552	560.7	-0.4	1.05	1.000	0.65
09/13/04	21	HPLC-EC	1.030	244.0	0.555	562.2	-0.1	0.99	1.000	0.10
03/08/05	22	HPLC-EC	1.034	243.0	0.559	562.9	0.2	1.06	1.000	0.24
		Mean	1.029	243.2	0.55	560.3				0.48
		SD	0.003	0.8	0.02	4.6				0.42
		CV	0.32	0.31	4.4	0.8				

[TAA] mmol/Lsample

Date	RR	Sample	Rep_1	Rep_2	F_{adj}	Mean	SD_{dup}	N	Mean	SD_{repeat}	SD_{reprod}
02/23/04	20	CS#1	7.8	8.0	1.0	7.9	0.1	3	8.2	0.1	0.4
09/13/04	21	CS#1	8.1	7.9	1.0	8.0	0.1				
03/08/05	22	CS#1	8.5	8.7	1.0	8.6	0.1				
02/23/04	20	CS#2	25.8	26.2	1.0	26.0	0.3	3	27.2	0.4	1.6
09/13/04	21	CS#2	26.2	27.2	1.0	26.7	0.7				
03/08/05	22	CS#2	29.0	29.0	1.0	29.0	0.0				
12/12/02	17	S17:1	9.9	9.1	1.0	9.5	0.6	4	9.3	0.3	0.4
11/13/03	19	S19:2	9.2	9.1	1.0	9.2	0.1				
09/13/04	21	S21:2	8.8	8.7	1.0	8.7	0.1				
03/08/05	22	S22:1	9.6	9.6	1.0	9.6	0.0				
12/12/02	17	S17:2	23.3	23.4	1.0	23.4	0.1	4	23.5	0.5	0.8
03/20/03	18	S18:1	22.7	23.7	1.0	23.2	0.7				
02/23/04	20	S20:1	25.1	24.1	1.0	24.6	0.7				
03/08/05	22	S22:2	22.7	22.7	1.0	22.7	0.0				
03/20/03	18	S18:2	35.1	36.0	1.0	35.6	0.6	4	35.0	0.3	1.3
11/13/03	19	S19:3	35.9	35.8	1.0	35.9	0.1				
09/13/04	21	S21:3	33.2	32.9	1.0	33.0	0.2				
03/08/05	22	S22:3	35.7	35.6	1.0	35.6	0.1				
09/23/98	11	S11:2	49.2	51.4	0.5	25.2	0.8	8	26.5	0.4	2.0
04/02/99	12	S12:2	47.7	47.0	0.5	23.7	0.2				
09/17/01	13	S13:2	27.6	27.7	1.0	27.7	0.1				
09/27/01	14	S14:4	25.7	26.4	1.0	26.0	0.5				
09/18/01	15	S15:2	25.4	25.6	1.0	25.5	0.2				
03/20/03	18	S18:3	28.8	29.2	1.0	29.0	0.3				
02/23/04	20	S20:4	25.9	25.2	1.0	25.5	0.5				
03/08/05	22	S22:4	29.4	29.4	1.0	29.4	0.0				

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

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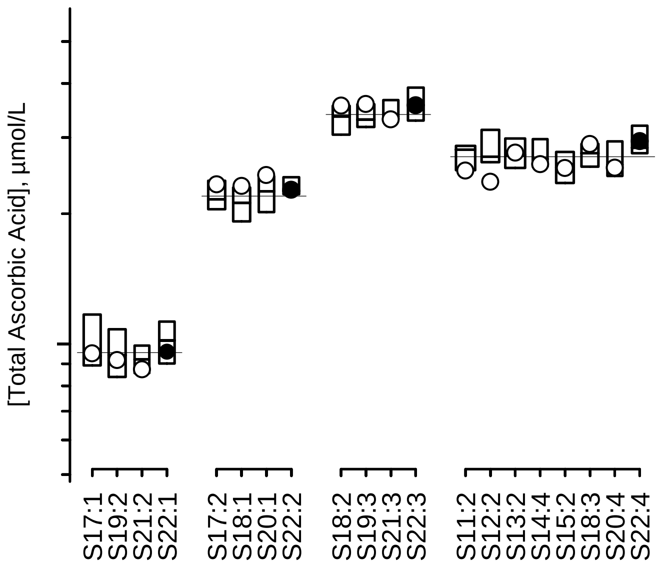
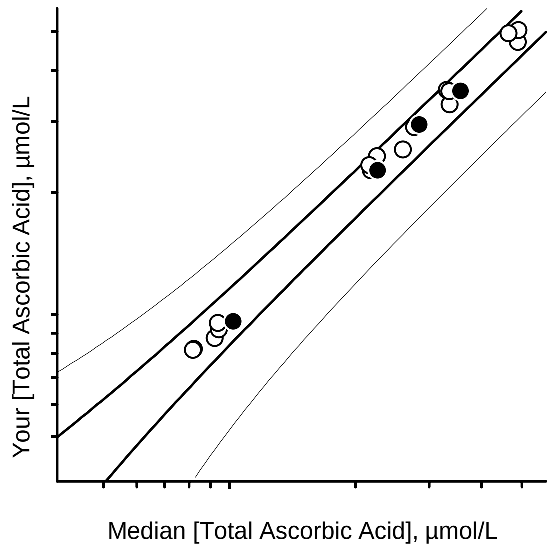
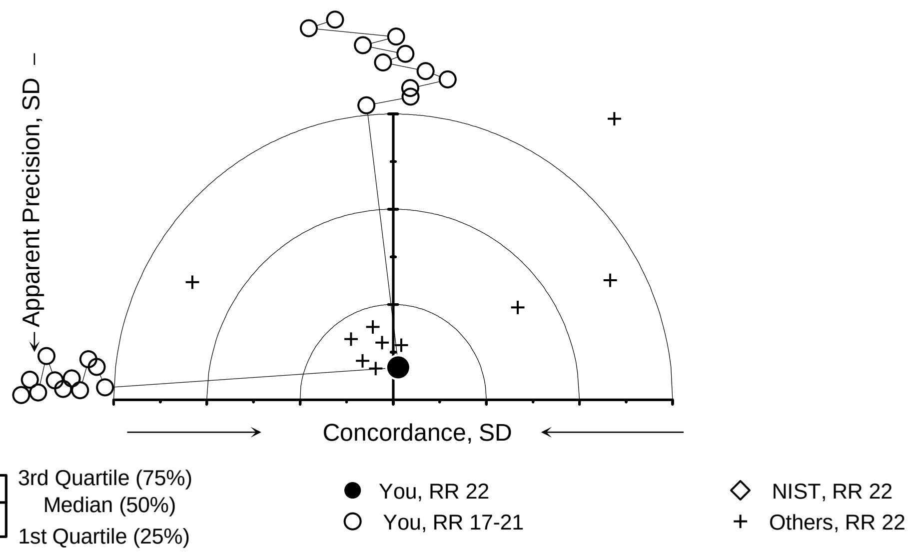
Vitamin C "Round Robin" 22 Report: Participant VC-MA

Total Ascorbic Acid







For details of the construction and interpretation of these plots, see:

Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

Sample	Comments
S21:1	Serum 23, previously distributed in RRs 17, 19, and 21
S21:2	Serum 33, previously distributed in RRs 17, 18, and 20
S21:3	Serum 42, previously distributed in RRs 18, 19, and 21
S21:4	SRM 970 Level 2, previously distributed in RRs 11, 12, 13, 14, 15, 18, and 20