NISTIR 7880-7

NIST Micronutrients Measurement Quality Assurance Program Winter 2009 Comparability Studies

Results for Round Robin LXV
Fat-Soluble Vitamins and Carotenoids in Human Serum
and Round Robin 30 Ascorbic Acid in Human Serum

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http://dx.doi.org/10.6028/NIST.IR.7880-7

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

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April, 2013

U.S. Department of Commerce
Rebecca Blank, 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 2009 MMQAP measurement comparability improvement studies: 1) Round Robin LXV Fat-Soluble Vitamins and Carotenoids in Human Serum and 2) Round Robin 30 Total Ascorbic Acid in Human Serum. The materials for both studies were shipped to participants in December 2008; participants were requested to provide their measurement results by March 31, 2009.

Keywords

Human Serum
Retinol, α-Tocopherol, γ-Tocopherol, Total and *Trans*-β-Carotene
SRM 1950, 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 LXV: Fat-Soluble Vitamins and Carotenoids in Human Serum

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin LXV comparability study (hereafter referred to as RR65) received one lyophilized and four 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 December 2008. 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 RR65 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 30: Vitamin C in Human Serum

Participants in the MMQAP Vitamin C in Human Serum Round Robin 30 comparability study (hereafter referred to as RR30) received four frozen serum test samples, one frozen control serum, a solid ascorbic acid control material, and one optional unknown (SRM 1950 Metabolites in Human Serum) 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 December 2008. 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.

SRM 1950 is a human plasma pool designed for the evaluation of the measurement of metabolites in humans to provide insight into various disease states. The SRM 1950 plasma contains lithium heparin as an anticoagulant but does not contain MPA. Participants were asked to analyze SRM 1950 as an unknown to help evaluate whether TAA could be usefully certified in this material.

The final report delivered to every participant in RR30 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 RR65

The following three items were included in each package shipped to an RR65 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-

December 3, 2008

Dear Colleague:

Enclosed are samples for the first fat-soluble vitamins and carotenoids in serum study (Round Robin LXV) for the 2009 NIST Micronutrients Measurement Quality Assurance Program. The set of samples (Sera 352 - 356) consists of one vial of each of four liquid-frozen and one lyophilized serum samples for analysis along with a form for reporting your results. These samples should be stored in the dark at or below –20 °C upon receipt. When reporting your results, please submit one value for each analyte for a given serum sample. If a value obtained is below your limit of quantification, please indicate this result on the form by using NQ *(Not Quantified)*. Results are due to NIST by **March 31, 2009**. Results received more than two weeks after the due date may not be included in the summary report for this round robin study. The feedback report concerning the study will be distributed in April 2009.

Lyophilized samples should be reconstituted with 1.0 mL of HPLC-grade water or equivalent. Before reconstitution, samples should be allowed to stand at room temperature under subdued light until thawed. 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 samples.**

For consistency, we request that laboratories use the following absorptivities (dL/g • 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); and lycopene, 3450 at 472 nm (hexane).

Please report your results for Round Robin LXV by e-mail to david.duewer@nist.gov or fax to 301-977-0685. If you have questions or comments regarding this study, please call me at (301) 975-3120 or e-mail me at jbthomas@nist.gov.

Sincerely,

Jeanice B Thomas

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

Enclosures

NIST

Participant #: ________ Date: _____________

Round Robin LXV: Human Sera

NIST Micronutrients Measurement Quality Assurance Program

Analyte	352	353	354	355	356	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 measurands?						

* we prefer **µg/mL**

Were the liquid-frozen samples (353 to 356) frozen when received? Yes | No

Comments:

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

Please return by March 31, 2009

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

Participant #: ________ Date: ____________

Fat-Soluble Vitamins Round Robin LXV

NIST Micronutrients Measurement Quality Assurance Program

Packing List and Shipment Receipt Confirmation Form

This box contains: one vial each of the following five FSV M^2QAP sera

Serum	Form	Reconstitute?	Vial/Cap
#352	Lyophilized	Yes (1 ml H_2O)	2 mL amber, silver
#353	Liquid frozen	No	2 mL amber, green cap
#354	Liquid frozen	No	2 mL amber, red cap
#355	Liquid frozen	No	2 mL amber, blue cap
#356	Liquid frozen	No	2 mL amber, green cap

Please 1) Open the pack immediately
2) Check that it contains all of the above samples
3) Check if the vials are intact
4) Store the sera 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 sera 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 liquid frozen samples arrive frozen? Yes | No

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

6) When do you anticipate analyzing these samples? ________________________

Your prompt return of this information is appreciated.

The M^2QAP Gang

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

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

Appendix B. Final Report for RR65

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 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-

May 8, 2009

Dear Colleague:

Enclosed is the summary report of the results for intercomparison RR65 of the 2009 NIST Micronutrients Measurement Quality Assurance Program (MMQAP) 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 comparability.

Your overall measurement comparability is summarized in the "Score Card" summary, page 6 of the All Lab Report. Combined 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. Similar information is presented graphically in the "target plots" that are the last page of your Individualized Report. If you have concerns regarding your laboratory's performance, please contact us for consultation.

SRM 968d, Fat-Soluble Vitamins, Carotenoids, and Cholesterol in Human Serum is now available. Orders can be placed directly through the NIST on-line SRM order request system at: https://srmors.nist.gov/index.cfm. You may also call the SRM office directly at (301) 975-2200 if you have purchasing questions.

Samples for the second 2009 QA intercomparison study will be shipped during the week of June 1, 2009. 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 B. Thomas, M.B.A.
Research Chemist
Analytical Chemistry Division
Chemical Science and Technology Laboratory

Dave Duewer

Dave L. Duewer, Ph.D.
Research Chemist
Analytical Chemistry Division
Chemical Science and Technology Laboratory

Cc: L.C. Sander

NIST

The NIST M^2QAP Round Robin LXV (RR65) report consists of:

Page	"All Lab" Report
1-4	A listing of all results and statistics for analytes reported by two or more participants.
5	A listing of results reported by only one laboratory and the legend for pages 1-4.
6	The text Comparability Summary ("Score Card") of measurement performance.

Page	"Individualized" Report
1	Your values, the number of labs reporting values, and our assigned values.
2-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 eight other participants.
n+1	The graphical Comparability Summary (target plot) of measurement performance.

Samples. Five samples were distributed in RR65.

Serum	Description	Prior Distributions
352	Lyophilized 1+1 blend of the pools used to produce SRM 968c Levels I and II.	#247:RR44-9/98, #317:RR58-9/05
353	Fresh-frozen 1+0.03 blend of a normal serum and a low-normal serum spiked with *trans*-β-carotene in a lipoprotein carrier. The same materials were used to prepare #354 and #355.	New, produced Fall 2008
354	Fresh-frozen 1+0.08 blend of the materials used to prepare #353.	New, produced Fall 2008
355	Fresh-frozen 1+0.19 blend of the materials used to prepare #353.	New, produced Fall 2008
356	Fresh-frozen 1+0.56 blend of the residual pools used to produce SRM 968c Levels I and II.	New, produced Fall 2008

Results

1) Serum #352: This material is a 1+1 mixture of the left-over pools for SRM 968c Level I and Level II (where 1+1 means equal volumes of the two pools and left-over means the material left in the carboy after stirring became problematic). The material was prepared in 1998, soon after the SRM 968c units were packed away, to test how well we could predict measurand levels in a mixture. This is the third time this material has been distributed. There has been no statistically significant change in the level or variability of any measurand. However, the measured level of total lutein and total lutein plus zeaxanthin has somewhat decreased with time. The level of total zeaxanthin does not appear to have changed. Given the relatively few measurements available, the apparent decline in lutein is mostly a curiosity. We will, however, re-examine the archived data for other materials that have been stored for ten or more years.

2) <u>Serum #356:</u> This material is also a mixture of the left-over pools for SRM 968c Level I and Level II. However, this material was mixed and ampouled in 2009. While there was no surety that results for SRM 968c were really applicable to these residual materials, we were again interested in how well we can predict the levels in a blend. Table 1 contrasts our predictions for the #352 material (lyophilized, stored for 11 years in 2-mL glass vials) with those for #356 (liquid frozen in Teflon for 11 years). The "Calc" values are our predictions based upon MMQAP results for the six distributions of the SRM 968c Level I and Level II materials over the past ten years, "Obs" are the median results in RR LXV, and "%Dif" is 100*(Calc – Obs)/Obs. Note: The "Calc" results of Table 1 define the dark blue "expectation" lines for these two SRM 968c-related materials in your "Individualized" Report.

Table 1: Calculated and Observed Results for Mixtures of the SRM 968c Level I and II Pools

	Serum #352			Serum #356		
Analyte	Calc	Obs	%Dif	Calc	Obs	%Dif
Total Retinol	0.67	0.69	-2	0.72	0.76	-5
α-Tocopherol	12.1	11.4	6	10.8	10.6	2
γ/β-Tocopherol	2.7	2.6	3	3.0	2.5	24
Total β-Carotene	0.30	0.31	-4	0.26	0.27	-1
Total α-Carotene	0.056	0.054	4	0.045	0.044	3
Total Lycopene	0.36	0.38	-4	0.35	0.37	-7
trans-Lycopene	0.18	0.17	7	0.18	0.18	0
Total β-Cryptoxanthin	0.052	0.056	-7	0.058	0.059	-2
Total Lutein	0.071	0.058	23	0.066	0.055	20
Total Zeaxanthin	0.027	0.024	16	0.029	0.030	-5
Total Lutein&Zeaxanthin	0.100	0.091	10	0.097	0.097	0

Most of the predicted levels are within about 10 % of those observed. Interestingly, total lutein is about 20 % less than predicted in both materials. While total zeaxanthin and total lutein plus zeaxanthin are somewhat lower than expected in the #352 material but not in the #356, the major difference between the two materials is for γ/β-tocopherol, where the level in the #356 material is about 25 % lower than expected. We as yet have no defensible explanation for this anomaly.

3) <u>Sera #353, #354, and #355.</u> These three materials are the culmination of an experiment begun in late 1999 testing whether carotenoids could be successfully augmented. The materials are all mixtures of the same β-carotene-deficient but otherwise fairly normal serum and a *trans*-β-carotene spiking solution. Following a number of false-starts, this solution was prepared in the late 1990's by mixing *trans*-β-carotene and high- and low-density lipoproteins into a low-normal serum followed by extensive mixing and filtering. Due to resource constraints, the solution sat unused until this spring.

Following further mixing and filtering, the three materials were prepared at the same time as #356. Serum #353 was spiked with just enough of the solution to raise the level of *trans*-β-carotene to about that of the #352 and #356 materials. Serum #354 was spiked to a reasonably high *trans*-β-carotene level and #355 was spike to an unreasonably high level, just to determine the limits of augmentation.

Figure 1 displays the results for all of the β-carotene results reported in RR65. Each line represents the results for a single participant. The line segments o the left connect results for #352 and #356; the lines to the right connect results for #353, #354, and #355. The x-axis spacing for the three spiked materials reflects the relative proportion of the spike, so that ideal results will produce a straight line.

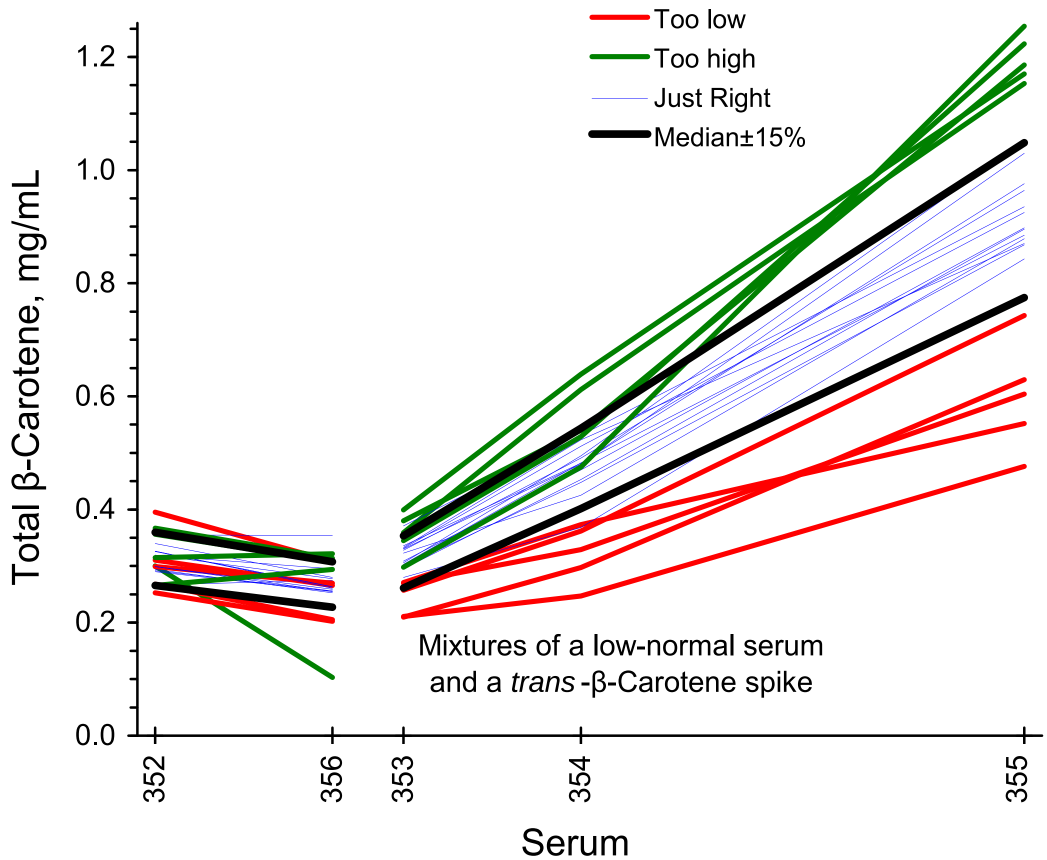

Figure 1: RR65 Results for Total β-Carotene

The thick black lines bound the region ± 15 % about the median result. The thin blue lines denote the participant results that (mostly) fall within this region. The thick green lines denote the results for #355 that are more than 15 % higher than the median; the thick red lines denote the results that are (mostly) more than 15 % lower than expected.

We hypothesize that the "too low" results arise from extraction processes that are not adequate enough to completely extract β-carotene. However, there is no correlation between the relative location of the "too low" results of the spiked materials and in the two SRM 968c-related mixtures. Therefore, the relative under-extraction in the artificial matrix does not imply under-extraction in a native matrix. Also, the "too low" results are quite consistently low for all three of the spiked materials, thereby indicating that the problem is with the nature of the spike and not its proportion in the mixture.

The "too high" results are less easily explained, since we do not believe that extraction from the artificial matrix should be easier than from the native. However, we actually expected the spiked levels to be somewhat higher than the median results so it is quite plausible that the "too high" values are in fact the "true" values for a really complete extraction.

Regardless of the causes, the RR65 results strongly suggest that we cannot (yet) successfully augment carotenoid levels. We will continue to try to provide you with sera that have interesting levels and patterns of analytes, but we are constrained in what we can deliver by the native levels in commercially available sera.

Appendix C. “All-Lab Report” for RR65

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 LXV Laboratory Results

Lab	Total Retinol, µg/mL 352	353	354	355	356	trans-Retinol, µg/mL 352	353	354	355	356	Retinyl Palmitate, µg/mL 352	353	354	355	356
FSV-BA	0.666	0.318	0.320	0.292	0.781						0.058	0.066	0.072	0.096	0.053
FSV-BB	0.750	0.326	0.333	0.300	0.799						0.049	0.017	0.021	0.031	0.040
FSV-BC	0.683	0.335	0.331	0.301	0.804										
FSV-BD	0.593	0.310	0.312	0.289	0.778										
FSV-BE	0.525	0.239	0.250	0.233	0.589										
FSV-BF	0.790	0.340	0.350	0.310	0.800										
FSV-BG	0.700	0.325	0.324	0.297	0.774						0.054	0.037	0.036	0.036	0.051
FSV-BH	0.545	0.247	0.246	0.215	0.647										
FSV-BJ	0.698	0.315	0.319	0.293	0.749						0.061	*nq*	*nq*	*nq*	*nq*
FSV-BK															
FSV-BL	0.690	0.320	0.320	0.290	0.740										
FSV-BM	0.690	0.360	0.360	0.350	0.770										
FSV-BN	0.574	0.246	0.245	0.235	0.624						0.040	0.001	0.002	0.001	0.034
FSV-BO	0.673	0.327	0.347	0.295	0.768										
FSV-BP	0.689	0.339	0.335	0.304	0.680										
FSV-BQ	0.740	0.330	0.320	0.290	0.800										
FSV-BR	*≥0.650*	*≥0.290*	*≥0.290*	*≥0.260*	*≥0.620*	0.650	0.290	0.290	0.260	0.620					
FSV-BS	*≥0.698*	*≥0.356*	*≥0.353*	*≥0.326*	*≥0.745*	0.698	0.356	0.353	0.326	0.745					
FSV-BU	0.650	0.264	0.313	0.294	0.702										
FSV-BV	0.727	0.317	0.312	0.261	0.776										
FSV-BW	0.728	0.338	0.342	0.316	0.805						0.058	0.010	0.010	0.012	0.077
FSV-CC	0.565	0.310	0.314	0.340	0.806	0.529	0.310	0.314	0.340	0.806					
FSV-CD	0.620	0.280	0.290	0.260	0.670						0.040	0.010	0.020	0.010	0.040
FSV-CE	0.668	0.264	0.286	0.267	0.599										
FSV-CF	0.843	0.349	0.399	0.291	0.862										
FSV-CG	0.803	0.337	0.334	0.314	0.737										
FSV-CI	0.818	0.321	0.323	0.302	0.812						0.071	0.039	0.051	0.034	0.056
FSV-CP															
FSV-CW	0.697	0.268	0.285	0.241	0.752						0.034	0.006	0.008	0.006	0.036
FSV-CZ	0.664	0.309	0.397	0.293	0.584										
FSV-DD	0.590	0.270	0.280	0.260	0.690										
FSV-DV	*≥0.638*	*≥0.259*	*≥0.258*	*≥0.230*	*≥0.594*	0.638	0.259	0.258	0.230	0.594					
FSV-EE	0.612	0.304	0.294	0.279	0.643										
FSV-EZ	*≥0.572*	*≥0.259*	*≥0.251*	*≥0.233*	*≥0.708*	0.572	0.259	0.251	0.233	0.708	0.049	<0.02	<0.02	<0.02	0.045
N	28	28	28	28	28	5	5	5	5	5	10	8	8	8	9
Min	0.525	0.239	0.245	0.215	0.584	0.529	0.259	0.251	0.230	0.594	0.034	0.001	0.002	0.001	0.034
Median	0.686	0.318	0.320	0.293	0.760	0.638	0.290	0.290	0.260	0.708	0.052	0.014	0.021	0.022	0.045
Max	0.843	0.360	0.399	0.350	0.862	0.698	0.356	0.353	0.340	0.806	0.071	0.066	0.072	0.096	0.077
SD	0.071	0.027	0.027	0.019	0.066	0.089	0.046	0.047	0.044	0.130	0.012	0.015	0.021	0.020	0.012
CV	10	9	9	6	9	14	16	16	17	18	23	112	103	94	26
N_{past}	41	0	0	0	0	6	0	0	0	0	13	0	0	0	0
$Median_{past}$	0.673					0.681					0.052				
SD_{past}	0.051					0.059					0.015				
NIST	0.683	0.309	0.308	0.276	0.722										
N_{NIST}	2	2	2	2	2										
Mean	0.683	0.309	0.308	0.276	0.722										
S_{rep}	0.006	0.004	0.010	0.011	0.027										
S_{het}	0.017	0.011	0.007	0.002	0.013										
S_{NIST}	0.018	0.012	0.012	0.011	0.030										
NAV	0.686	0.313	0.314	0.285	0.745	0.638	0.290	0.290	0.260	0.708	0.052	0.014	0.021	0.022	0.045
NAU	0.059	0.027	0.028	0.027	0.068	0.089	0.046	0.047	0.044	0.130	0.016	0.015	0.021	0.020	0.015

Lab	α-Tocopherol, µg/mL 352	353	354	355	356	γ/β-Tocopherol, µg/mL 352	353	354	355	356	δ-Tocopherol, µg/mL 352	353	354	355	356
FSV-BA	10.86	7.73	7.69	6.73	10.24	2.80	3.50	3.49	3.06	2.73	0.338	0.104	0.106	0.098	0.290
FSV-BB	11.96	8.03	8.13	7.13	10.55	2.78	3.23	3.27	2.75	2.52	0.320	0.072	0.067	0.060	0.253
FSV-BC															
FSV-BD	12.20	8.80	8.40	7.30	10.70										
FSV-BE	10.57	6.83	6.79	6.25	9.58	2.00	2.41	2.42	2.19	1.90					
FSV-BF	11.20	7.70	7.70	6.80	11.10										
FSV-BG	12.28	8.54	8.37	7.38	11.44	2.75	3.54	3.51	3.09	2.72					
FSV-BH	10.85	7.59	7.91	6.74	10.44	3.00	3.67	3.67	3.17	2.90					
FSV-BJ	12.55	8.48	8.51	7.47	11.07	2.73	3.43	3.49	3.06	2.49					
FSV-BK	13.44	8.27	7.97	7.32	10.35										
FSV-BL	12.06	8.18	8.18	7.32	10.77										
FSV-BM	11.90	8.80	9.10	8.10	11.70										
FSV-BN	9.25	6.07	5.98	5.49	8.20	2.06	2.39	2.34	2.17	1.88					
FSV-BO	9.50	7.80	7.70	6.90	10.20	2.40	3.30	3.30	2.90	2.40					
FSV-BP	10.67	9.03	8.45	7.25	9.66										
FSV-BQ	11.40	6.90	7.80	6.90	10.70										
FSV-BR	12.54	8.40	8.20	6.20	9.70										
FSV-BS															
FSV-BU	11.32	7.58	7.38	6.79	10.26	2.28	2.96	2.82	2.68	2.34					
FSV-BV	13.17	8.98	9.11	7.30	11.74	2.58	3.26	3.28	2.61	2.37					
FSV-BW	12.69	8.55	8.53	7.67	11.05	2.95	3.65	3.65	3.22	2.70					
FSV-CC	9.37	7.85	8.26	7.56	10.70										
FSV-CD	13.91	9.27	6.67	8.51	12.40	2.66	3.06	3.19	2.73	2.46					
FSV-CE	10.07	7.70	8.04	6.91	7.80										
FSV-CF	12.90	8.10	8.70	6.90	10.80										
FSV-CG	10.67	7.37	7.03	6.22	9.52	2.63	3.17	3.08	2.75	2.40	0.251	0.110	0.060	0.062	0.201
FSV-CI	11.94	7.70	7.70	6.93	10.57	3.24	3.80	3.77	3.37	2.98					
FSV-CP	9.58	6.41	6.26	5.32	8.24	2.55	2.68	2.87	2.45	1.90					
FSV-CW	12.07	8.27	7.97	7.13	10.57	2.62	3.15	3.08	2.75	2.45	0.370	0.080	0.100	0.050	0.280
FSV-CZ	12.64	6.98	7.25	6.25	9.95	2.73	3.15	3.27	2.79	2.32					
FSV-DD															
FSV-DV	10.90	7.40	7.20	6.40	9.70										
FSV-EE	9.70	7.80	7.40	6.60	9.60										
FSV-EZ	11.43	6.89	6.98	6.58	11.48	2.41	3.00	3.09	2.92	2.71					
N	31	31	31	31	31	18	18	18	18	18	4	4	4	4	4
Min	9.25	6.07	5.98	5.32	7.80	2.00	2.39	2.34	2.17	1.88	0.251	0.072	0.060	0.050	0.201
Median	11.43	7.80	7.91	6.90	10.55	2.65	3.20	3.27	2.77	2.46	0.329	0.092	0.084	0.061	0.267
Max	13.91	9.27	9.11	8.51	12.40	3.24	3.80	3.77	3.37	2.98	0.370	0.110	0.106	0.098	0.290
SD	1.260	0.697	0.755	0.593	0.812	0.212	0.351	0.328	0.337	0.285	0.037	0.022	0.029	0.009	0.027
CV	11	9	10	9	8	8	11	10	12	12	11	24	35	15	10
N_{past}	41	0	0	0	0	23	0	0	0	0	6	0	0	0	0
$Median_{past}$	11.619					2.730					0.369				
SD_{past}	0.993					0.183					0.057				
NIST	11.65	7.87	8.09	6.93	10.71	2.61	2.95	3.11	2.82	2.34	0.331	*nq*	*nq*	*nq*	0.287
N_{NIST}	2	2	2	2	2	2	2	2	2	2	2				2
Mean	11.653	7.872	8.094	6.927	10.715	2.608	2.954	3.106	2.816	2.341	0.331				0.287
S_{rep}	0.601	0.429	0.110	0.026	0.284	0.059	0.013	0.086	0.076	0.063	0.016				0.023
S_{het}	0.270	0.398	0.109	0.201	0.227	0.123	0.058	0.110	0.070	0.048	0.011				0.013
S_{NIST}	0.659	0.585	0.155	0.203	0.363	0.136	0.060	0.139	0.103	0.080	0.019				0.026
NAV	11.542	7.836	8.002	6.914	10.633	2.627	3.076	3.187	2.794	2.398	0.330	0.092	0.084	0.061	0.277
NAU	1.270	0.699	0.766	0.593	0.827	0.262	0.391	0.348	0.338	0.296	0.051				0.047

Round Robin LXV Laboratory Results

Lab	Total β-Carotene, µg/mL					trans-β-Carotene, µg/mL					Total cis-β-Carotene, µg/mL				
	352	353	354	355	356	352	353	354	355	356	352	353	354	355	356
FSV-BA	0.292	0.299	0.448	0.868	0.256	0.274	0.285	0.428	0.833	0.243	0.019	0.014	0.020	0.036	0.013
FSV-BB	0.294	0.295	0.453	0.896	0.253	0.274	0.281	0.433	0.855	0.239	0.020	0.014	0.021	0.041	0.013
FSV-BC															
FSV-BD															
FSV-BE	0.311	0.211	0.247	0.476	0.266										
FSV-BF	0.267	0.306	0.495	1.050	0.275										
FSV-BG	0.356	0.380	0.540	1.186	0.307										
FSV-BH	0.299	0.336	0.482	0.976	0.259	0.283	0.322	0.462	0.933	0.248	0.016	0.014	0.020	0.043	0.011
FSV-BJ	0.319	0.280	0.368	0.843	0.295										
FSV-BK															
FSV-BL															
FSV-BM															
FSV-BN	0.340	0.229	0.329	0.629	0.269	0.304	0.210	0.301	0.569	0.252	*0.036*	*0.019*	*0.028*	*0.060*	*0.017*
FSV-BO	0.326	0.309	0.470	0.898	0.263										
FSV-BP	0.315	0.298	0.475	1.254	0.322										
FSV-BQ															
FSV-BR															
FSV-BS	*≥0.290*	*≥0.333*	*≥0.523*	*≥0.870*	*≥0.255*	0.290	0.333	0.523	0.870	0.255					
FSV-BU	0.326	0.371	0.531	0.936	0.261										
FSV-BV	0.367	0.399	0.639	1.170	0.314										
FSV-BW	0.355	0.323	0.425	0.879	0.354										
FSV-CC															
FSV-CD	0.340	0.330	0.480	1.030	0.280										
FSV-CE	0.300	0.359	0.612	1.153	0.103										
FSV-CF															
FSV-CG	0.299	0.328	0.491	0.964	0.255	0.274	0.303	0.455	0.901	0.233	0.025	0.024	0.036	0.063	0.021
FSV-CI	0.268	0.270	0.329	0.604	0.205										
FSV-CP	0.253	0.257	0.362	0.743	0.202										
FSV-CW	0.319	0.332	0.512	0.925	0.277										
FSV-CZ	0.395	0.271	0.373	0.552	0.308										
FSV-DD															
FSV-DV															
FSV-EE															
FSV-EZ	*≥0.266*	*≥0.345*	*≥0.529*	*≥1.223*	*≥0.294*	0.266	0.345	0.529	1.223	0.294					
N	20	20	20	20	20	7	7	7	7	7	5	5	5	5	5
Min	0.253	0.211	0.247	0.476	0.103	0.266	0.210	0.301	0.569	0.233	0.016	0.014	0.020	0.036	0.011
Median	0.317	0.307	0.472	0.912	0.268	0.274	0.303	0.455	0.870	0.248	0.020	0.014	0.021	0.043	0.013
Max	0.395	0.399	0.639	1.254	0.354	0.304	0.345	0.529	1.223	0.294	0.036	0.024	0.036	0.063	0.021
SD	0.034	0.042	0.079	0.190	0.020	0.012	0.033	0.040	0.055	0.010	0.006	0.000	0.001	0.010	0.003
CV	11	14	17	21	7	4	11	9	6	4	30	0	7	24	23
N_{past}	28	0	0	0	0	13	0	0	0	0	9	0	0	0	0
$Median_{past}$	0.320					0.301					0.024				
SD_{past}	0.043					0.034					0.008				
NIST	0.326	0.303	0.482	0.885	0.264										
N_{NIST}	2	2	2	2	2										
Mean	0.326	0.303	0.482	0.885	0.264										
S_{rep}	0.021	0.007	0.020	0.030	0.020										
S_{het}	0.006	0.011	0.009	0.035	0.001										
S_{NIST}	0.022	0.013	0.022	0.046	0.020										
NAV	0.321	0.305	0.477	0.898	0.266	0.274	0.303	0.455	0.870	0.248	0.020	0.014	0.021	0.043	0.013
NAU	0.046	0.045	0.079	0.191	0.039	0.030	0.033	0.048	0.090	0.027	0.007	0.005	0.007	0.017	0.005

Lab	Total α-Carotene, µg/mL					Total Lycopene, µg/mL					trans-Lycopene, µg/mL				
	352	353	354	355	356	352	353	354	355	356	352	353	354	355	356
FSV-BA	0.049	0.032	0.032	0.030	0.043	0.416	0.581	0.597	0.571	0.401	0.218	0.322	0.330	0.310	0.228
FSV-BB	0.048	0.028	0.029	0.024	0.042	0.376	0.538	0.533	0.505	0.382	0.156	0.228	0.230	0.201	0.170
FSV-BC															
FSV-BD															
FSV-BE															
FSV-BF															
FSV-BG	0.056	0.038	0.034	0.033	0.051	0.375	0.519	0.497	0.449	0.376	0.189	0.271	0.255	0.235	0.194
FSV-BH	0.043	*nq*	*nq*	*nq*	0.040	0.367	0.518	0.515	0.428	0.372					
FSV-BJ	0.058	0.032	0.034	0.031	0.057	0.416	0.533	0.572	0.490	0.390					
FSV-BK															
FSV-BL															
FSV-BM															
FSV-BN	0.056	0.025	0.025	0.024	0.044	0.331	0.437	0.464	0.467	0.341	0.161	0.223	0.233	0.239	0.163
FSV-BO	0.024	0.026	0.026	0.027	0.044	0.262	0.443	0.441	0.391	0.350					
FSV-BP	0.057	0.021	0.047	0.038	0.065	0.396	0.321	0.408	0.313	0.383					
FSV-BQ															
FSV-BR															
FSV-BS	0.050	0.037	0.039	0.040	0.040	0.404	0.531	0.544	0.412	0.368	0.150	0.211	0.215	0.166	0.149
FSV-BU	0.056	0.040	0.034	0.027	0.056	0.381	0.566	0.543	0.441	0.339					
FSV-BV	0.071	0.040	0.043	0.033	0.063	0.442	0.584	0.627	0.511	0.439					
FSV-BW	0.047	0.019	0.020	0.021	0.039	0.448	0.644	0.628	0.577	0.487					
FSV-CC															
FSV-CD	0.050	0.030	0.030	0.040	0.040	0.320	0.380	0.430	0.520	0.280					
FSV-CE															
FSV-CF															
FSV-CG	0.066	0.049	0.048	0.045	0.060	0.311	0.506	0.507	0.472	0.351	0.229	0.320	0.316	0.296	0.221
FSV-CI	0.082	0.062	0.058	0.059	0.062										
FSV-CP	0.052	0.030	0.033	0.028	0.042	0.280	0.365	0.364	0.293	0.251					
FSV-CW	0.054	0.028	0.027	0.026	0.049						0.171	0.223	0.217	0.187	0.176
FSV-CZ															
FSV-DD															
FSV-DV															
FSV-EE															
FSV-EZ															
N	17	16	16	16	17	15	15	15	15	15	7	7	7	7	7
Min	0.024	0.019	0.020	0.021	0.039	0.262	0.321	0.364	0.293	0.251	0.150	0.211	0.215	0.166	0.149
Median	0.054	0.031	0.033	0.031	0.044	0.376	0.519	0.515	0.467	0.372	0.171	0.228	0.233	0.235	0.176
Max	0.082	0.062	0.058	0.059	0.065	0.448	0.644	0.628	0.577	0.487	0.229	0.322	0.330	0.310	0.228
SD	0.006	0.009	0.009	0.008	0.007	0.059	0.092	0.085	0.066	0.033	0.026	0.025	0.027	0.072	0.027
CV	11	29	26	26	16	16	18	16	14	9	15	11	11	30	16
N_{past}	25	0	0	0	0	24	0	0	0	0	11	0	0	0	0
$Median_{past}$	0.055					0.370					0.175				
SD_{past}	0.012					0.078					0.033				
NIST	0.048	0.029	0.031	0.031	0.050						0.201	0.225	0.223	0.225	0.191
N_{NIST}	2	2	2	2	2						2	2	2	2	2
Mean	0.048	0.029	0.031	0.031	0.050						0.201	0.225	0.223	0.225	0.191
S_{rep}	0.002	0.002	0.004	0.004	0.002						0.020	0.011	0.012	0.010	0.008
S_{het}	0.001	0.001	0.000	0.001	0.003						0.012	0.000	0.006	0.011	0.001
S_{NIST}	0.002	0.002	0.004	0.005	0.003						0.023	0.011	0.013	0.015	0.008
NAV	0.051	0.030	0.032	0.031	0.047	0.376	0.519	0.515	0.467	0.372	0.186	0.227	0.228	0.230	0.183
NAU	0.017	0.010	0.011	0.010	0.015	0.082	0.107	0.106	0.098	0.081	0.037	0.041	0.042	0.072	0.033

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Round Robin LXV Laboratory Results

Lab	Total β-Cryptoxanthin, µg/mL 352	353	354	355	356	Total α-Cryptoxanthin, µg/mL 352	353	354	355	356
FSV-BA	0.056	0.097	0.098	0.090	0.061	0.020	0.034	0.035	0.031	0.020
FSV-BB	0.056	0.093	0.096	0.084	0.061	0.017	0.032	0.033	0.028	0.020
FSV-BC										
FSV-BD										
FSV-BE										
FSV-BF										
FSV-BG	0.058	0.100	0.097	0.088	0.061					
FSV-BH	0.060	0.100	0.102	0.095	0.066					
FSV-BJ	0.056	0.089	0.092	0.082	0.058					
FSV-BK										
FSV-BL										
FSV-BM										
FSV-BN	0.051	0.088	0.088	0.081	0.054	0.007	0.019	0.022	0.017	0.007
FSV-BO	0.070	0.077	0.078	0.074	0.050					
FSV-BP	0.056	0.043	0.044	0.036	0.052					
FSV-BQ										
FSV-BR										
FSV-BS	0.046	0.074	0.081	0.076	0.049	0.028	0.041	0.039	0.028	0.026
FSV-BU	0.075	0.110	0.131	0.111	0.080					
FSV-BV	0.050	0.089	0.093	0.076	0.057					
FSV-BW	0.042	0.090	0.089	0.075	0.046					
FSV-CC										
FSV-CD	0.070	0.110	0.120	0.130	0.070	0.060	0.100	0.110	0.100	0.060
FSV-CE										
FSV-CF										
FSV-CG	0.061	0.132	0.135	0.134	0.082					
FSV-CI										
FSV-CP	0.054	0.091	0.090	0.079	0.060					
FSV-CW	0.046	0.080	0.075	0.070	0.050					
FSV-CZ										
FSV-DD										
FSV-DV										
FSV-EE										
FSV-EZ										
N	16	16	16	16	16	5	5	5	5	5
Min	0.042	0.043	0.044	0.036	0.046	0.007	0.019	0.022	0.017	0.007
Median	0.056	0.091	0.093	0.082	0.059	0.020	0.034	0.035	0.028	0.020
Max	0.075	0.132	0.135	0.134	0.082	0.060	0.100	0.110	0.100	0.060
SD	0.007	0.014	0.011	0.011	0.010	0.012	0.010	0.006	0.004	0.009
CV	13	15	12	13	18	59	31	17	16	44
N_{past}	26	0	0	0	0	6	0	0	0	0
$Median_{past}$	0.057					0.019				
SD_{past}	0.011					0.002				
NIST	0.058	0.100	0.101	0.083	0.060					
N_{NIST}	2	2	2	2	2					
Mean	0.058	0.100	0.101	0.083	0.060					
S_{rep}	0.002	0.002	0.003	0.001	0.001					
S_{het}	0.001	0.003	0.000	0.001	0.004					
S_{NIST}	0.002	0.004	0.003	0.002	0.004					
NAV	0.057	0.095	0.097	0.082	0.059	0.020	0.034	0.035	0.028	0.020
NAU	0.014	0.022	0.022	0.019	0.014	0.012	0.010	0.006	0.004	0.009

Lab	Total Lutein, µg/mL 352	353	354	355	356	Total Zeaxanthin, µg/mL 352	353	354	355	356
FSV-BA										
FSV-BB	0.067	0.130	0.132	0.113	0.063	0.040	0.068	0.066	0.050	0.040
FSV-BC										
FSV-BD										
FSV-BE										
FSV-BF										
FSV-BG										
FSV-BH	0.052	0.101	0.096	0.087	0.050	0.027	0.060	0.057	0.050	0.030
FSV-BJ	0.069	0.137	0.139	0.125	0.068					
FSV-BK										
FSV-BL										
FSV-BM										
FSV-BN	0.054	0.107	0.109	0.100	0.054	0.020	0.047	0.047	0.043	0.023
FSV-BO	0.058	0.120	0.117	0.108	0.056	*nd*	0.086	0.071	0.071	0.045
FSV-BP										
FSV-BQ										
FSV-BR										
FSV-BS	0.047	0.092	0.090	0.075	0.042	0.019	0.045	0.044	0.031	0.021
FSV-BU										
FSV-BV										
FSV-BW										
FSV-CC										
FSV-CD										
FSV-CE										
FSV-CF										
FSV-CG										
FSV-CI	0.057	0.119	0.101	0.093	0.052	0.013	0.026	0.024	0.023	0.014
FSV-CP										
FSV-CW	0.073	0.133	0.139	0.122	0.074	0.039	0.077	0.083	0.076	0.044
FSV-CZ										
FSV-DD										
FSV-DV										
FSV-EE										
FSV-EZ										
N	8	8	8	8	8	6	7	7	7	7
Min	0.047	0.092	0.090	0.075	0.042	0.013	0.026	0.024	0.023	0.014
Median	0.058	0.120	0.113	0.104	0.055	0.024	0.060	0.057	0.050	0.030
Max	0.073	0.137	0.139	0.125	0.074	0.040	0.086	0.083	0.076	0.045
SD	0.011	0.019	0.027	0.021	0.010	0.011	0.022	0.019	0.028	0.015
CV	19	16	24	20	17	48	37	34	56	49
N_{past}	16	0	0	0	0	15	0	0	0	0
$Median_{past}$	0.068					0.027				
SD_{past}	0.015					0.009				
NIST	0.059	0.121	0.118	0.101	0.061	0.034	0.062	0.062	0.048	0.034
N_{NIST}	2	2	2	2	2	2	2	2	2	2
Mean	0.059	0.121	0.118	0.101	0.061	0.034	0.062	0.062	0.048	0.034
S_{rep}	0.001	0.002	0.002	0.001	0.002	0.005	0.003	0.002	0.004	0.002
S_{het}	0.004	0.005	0.005	0.011	0.003	0.001	0.001	0.002	0.003	0.004
S_{NIST}	0.004	0.006	0.005	0.011	0.004	0.005	0.003	0.003	0.005	0.004
NAV	0.058	0.120	0.115	0.103	0.058	0.029	0.061	0.059	0.049	0.032
NAU	0.012	0.022	0.027	0.021	0.012	0.013	0.022	0.020	0.028	0.015

Lab	Total Lutein&Zeaxanthin, µg/mL 352	353	354	355	356	Coenzyme Q10, µg/mL 352	353	354	355	356
FSV-BA	0.082	0.171	0.172	0.154	0.082					
FSV-BB	0.107	0.199	0.197	0.163	0.103					
FSV-BC										
FSV-BD										
FSV-BE						0.941	1.160	1.150	1.060	0.925
FSV-BF										
FSV-BG	0.098	0.174	0.162	0.142	0.097					
FSV-BH	0.079	0.161	0.153	0.137	0.080					
FSV-BJ						0.770	0.853	0.919	0.789	0.776
FSV-BK										
FSV-BL										
FSV-BM										
FSV-BN	0.074	0.151	0.152	0.140	0.077					
FSV-BO	0.058	0.206	0.188	0.179	0.101					
FSV-BP	0.094	0.088	0.090	0.102	0.097					
FSV-BQ										
FSV-BR										
FSV-BS	0.066	0.137	0.134	0.106	0.063					
FSV-BU	0.103	0.196	0.212	0.181	0.122					
FSV-BV	0.107	0.234	0.232	0.187	0.112					
FSV-BW	0.082	0.204	0.203	0.169	0.085	1.349	1.585	1.817	1.334	1.083
FSV-CC										
FSV-CD	0.230	0.360	0.400	0.360	0.230					
FSV-CE										
FSV-CF										
FSV-CG	0.122	0.251	0.252	0.224	0.129					
FSV-CI	*0.070*	*0.145*	*0.125*	*0.116*	*0.066*	0.780	1.000	0.970	0.770	0.770
FSV-CP	0.083	0.168	0.165	0.150	0.093					
FSV-CW	*0.113*	*0.209*	*0.221*	*0.199*	*0.118*	1.130	1.080	0.980	0.910	1.280
FSV-CZ						1.043	1.077	1.080	0.981	0.900
FSV-DD										
FSV-DV										
FSV-EE						0.813	0.959	0.971	0.933	0.793
FSV-EZ										
N	16	16	16	16	16	7	7	7	7	7
Min	0.058	0.088	0.090	0.102	0.063	0.770	0.853	0.919	0.770	0.770
Median	0.088	0.185	0.180	0.159	0.097	0.941	1.077	0.980	0.933	0.900
Max	0.230	0.360	0.400	0.360	0.230	1.349	1.585	1.817	1.334	1.280
SD	0.024	0.036	0.044	0.033	0.024	0.239	0.123	0.090	0.188	0.184
CV	28	19	25	21	24	25	11	9	20	20
N_{past}	24	0	0	0	0	7	0	0	0	0
$Median_{past}$	0.100					0.776				
SD_{past}	0.025					0.135				
NIST	*0.093*	*0.183*	*0.179*	*0.149*	*0.095*					
N_{NIST}	2	2	2	2	2					
Mean	0.093	0.183	0.179	0.149	0.095					
S_{rep}	0.005	0.005	0.003	0.005	0.000					
S_{het}	0.005	0.004	0.007	0.014	0.007					
S_{NIST}	0.007	0.006	0.007	0.015	0.007					
NAV	0.091	0.184	0.180	0.154	0.096	0.941	1.077	0.980	0.933	0.900
NAU	0.025	0.039	0.044	0.034	0.024	0.239	0.123	0.098	0.188	0.184

Round Robin LXV Laboratory Results

	Phylloquinone (K1), ng/mL					25-hydroxyvitamin D, µg/mL				
Lab	352	353	354	355	356	352	353	354	355	356
FSV-BA										
FSV-BB						0.013	0.008	0.009	0.010	0.014
FSV-BC										
FSV-BD										
FSV-BE	1.188	0.587	0.578	0.605	1.132					
FSV-BF										
FSV-BG										
FSV-BH						0.012	0.009	0.010	0.010	0.012
FSV-BJ										
FSV-BK										
FSV-BL										
FSV-BM										
FSV-BN										
FSV-BO										
FSV-BP										
FSV-BQ										
FSV-BR										
FSV-BS										
FSV-BU										
FSV-BV										
FSV-BW										
FSV-CC										
FSV-CD										
FSV-CE										
FSV-CF										
FSV-CG										
FSV-CI	1.837	0.820	0.714	<0.03	1.253					
FSV-CP										
FSV-CW										
FSV-CZ										
FSV-DD										
FSV-DV										
FSV-EE										
FSV-EZ										
N	2	2	2	1	2	2	2	2	2	2
Min	1.188	0.587	0.578		1.132	0.012	0.008	0.009	0.010	0.012
Median	1.513	0.704	0.646	0.605	1.193	0.013	0.008	0.009	0.010	0.013
Max	1.837	0.820	0.714		1.253	0.013	0.009	0.010	0.010	0.014
SD										
CV										
N_{past}	0	0	0	0	0	0	0	0	0	0
$Median_{past}$										
SD_{past}										
NIST										
N_{NIST}										
Mean										
S_{rep}										
S_{het}										
S_{NIST}										
NAV										
NAU										

Round Robin LXV Laboratory Results

Analytes Reported By One Laboratory

Analyte	Code	352	353	354	355	356
Ubiquinol	FSV-BW	1.283	1.265	1.543	1.207	0.555
Ubiquinone	FSV-BW	0.066	0.320	0.274	0.126	0.528

Term	Legend
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)
$Mean_{NIST}$	Mean of NIST results
S_{rep}	NIST's within-vial pooled standard deviation
S_{het}	NIST's among-vial pooled standard deviation
S_{NIST}	Combined standard deviation for NIST analyses: $\sqrt{(S_{rep}^2 + S_{het}^2)}$
NAV	NIST Assigned Value = (Median + $Mean_{NIST}$)/2 for analytes reported by NIST analyst(s) = Median for analytes reported by ≥ 5 labs but not NIST
NAU	NIST Assigned Uncertainty: $\sqrt{(S^2 + S_{btw}^2)}$ S is the maximum of (0.05*NAV, SD, S_{NIST}, eSD) and S_{btw} is the standard deviation between Median and $Mean_{NIST}$. The expected long-term SD, eSD, is defined in: Duewer et al., Anal Chem 1997;69(7):1406-1413.
nd	Not detected (i.e., no detectable peak for analyte)
nq	Detected but not quantitatively determined
≥x	Concentration greater than or equal to x
italics	Not explicitly reported but calculated by NIST from reported values

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	1			1
FSV-BB	1	1	1	1	1	1	1	1	1	1	1
FSV-BC	1										
FSV-BD	1	1									
FSV-BE	3	2	3	2							
FSV-BF	2	1		1							
FSV-BG	1	1	1	2		1	1	1			1
FSV-BH	3	1	2	1	1	1	1	1	1	1	1
FSV-BJ	1	1	1	1		1	1	1	1		
FSV-BK		1									
FSV-BL	1	1									
FSV-BM	2	2									
FSV-BN	3	3	3	2	3	1	1	1	1	1	1
FSV-BO	1	1	1	1		1	1	1	1	1	1
FSV-BP	1	2		2		2	2	2			2
FSV-BQ	1	1									
FSV-BR	2	2									
FSV-BS	2			1	1	1	1	1	2	1	2
FSV-BU	2	1	1	1		1	1	2			1
FSV-BV	1	2	1	2		1	1	1			1
FSV-BW	1	1	2	2		1	2	1			1
FSV-CC	2	2									
FSV-CD	2	3	1	1		1	1	2			4
FSV-CE	2	2		3							
FSV-CF	3	1									
FSV-CG	2	2	1	1	1	2	1	2			2
FSV-CI	2	1	2	2		3			1	2	2
FSV-CP		3	2	2		1	2	1			1
FSV-CW	2	1	1	1		1		1	2	2	1
FSV-CZ	2	2	1	2							
FSV-DD	2										
FSV-DV	3	1									
FSV-EE	2	1									
FSV-EZ	3	2	1	2	3						
NIST	1	1	1	1		1		1	1	1	1
n	33	32	19	23	7	18	15	17	9	8	17

	TR	aT	g/bT	bC	tbC	aC	TLy	TbX	TLu	TZ	L&Z
% 1	39	59	68	52	71	83	80	76	78	75	71
% 2	42	31	21	43	0	11	20	24	22	25	24
% 3	18	9	11	4	29	6	0	0	0	0	0
% 4	0	0	0	0	0	0	0	0	0	0	6

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.

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

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

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

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 RR65

Each participant in RR65 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 RR65:

- Total Retinol
- *trans*-Retinol
- Retinyl Palmitate
- α-Tocopherol
- γ/β-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
- Coenzyme Q10

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

Individualized Round Robin LXV Report: FSV-BA

Summary

Analyte	Serum 352 You	Serum 352 NAV	Serum 352 n	Serum 353 You	Serum 353 NAV	Serum 353 n	Serum 354 You	Serum 354 NAV	Serum 354 n	Serum 355 You	Serum 355 NAV	Serum 355 n	Serum 356 You	Serum 356 NAV	Serum 356 n
Total Retinol	0.666	0.685	28	0.318	0.313	28	0.320	0.314	28	0.292	0.284	28	0.781	0.741	28
Retinyl Palmitate	0.06	0.05	10	0.1	0.0	8	0.1	0.0	8	0.10	0.02	8	0.05	0.05	9
α-Tocopherol	10.86	11.54	31	7.73	7.84	31	7.69	8.00	31	6.73	6.91	31	10.24	10.63	31
γ/β-Tocopherol	2.795	2.627	18	3.495	3.076	18	3.485	3.187	18	3.064	2.794	18	2.734	2.398	18
δ-Tocopherol	0.338	0.330	4	0.104	0.092	4	0.106	0.084	4	0.098	0.061	4	0.290	0.277	4
Total β-Carotene	0.292	0.319	20	0.299	0.305	20	0.448	0.477	20	0.868	0.898	20	0.256	0.266	20
trans-β-Carotene	0.274	0.274	7	0.285	0.303	7	0.428	0.455	7	0.833	0.870	7	0.243	0.248	7
Total cis-β-Carotene	0.019	0.020	4	0.014	0.014	4	0.020	0.021	4	0.036	0.043	5	0.013	0.013	5
Total α-Carotene	0.049	0.051	17	0.032	0.030	16	0.032	0.032	16	0.030	0.031	16	0.043	0.047	17
Total Lycopene	0.416	0.376	15	0.581	0.519	15	0.597	0.515	15	0.571	0.467	15	0.401	0.372	15
trans-Lycopene	0.218	0.186	7	0.322	0.227	7	0.330	0.228	7	0.310	0.230	7	0.228	0.183	7
Total β-Cryptoxanthin	0.056	0.057	16	0.097	0.095	16	0.098	0.097	16	0.090	0.082	16	0.061	0.059	16
Total α-Cryptoxanthin	0.020	0.020	5	0.034	0.034	5	0.035	0.035	5	0.031	0.028	5	0.020	0.020	5
Total Lutein&Zeaxanthin	0.082	0.092	16	0.171	0.186	16	0.172	0.181	16	0.154	0.154	16	0.082	0.096	16

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

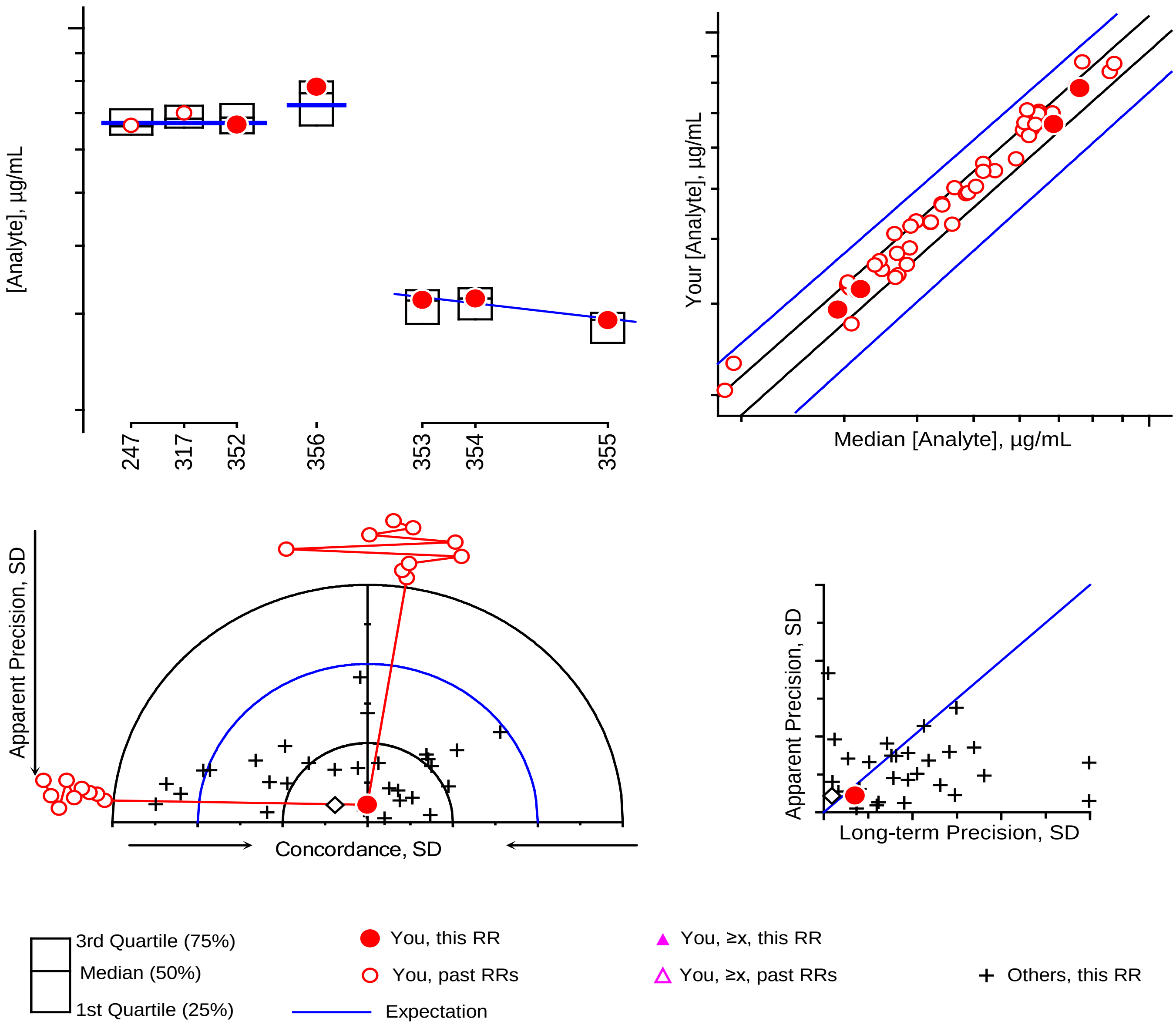
Please check our records against your records. Send corrections and/or updates to...

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 LXV Report: FSV-BA

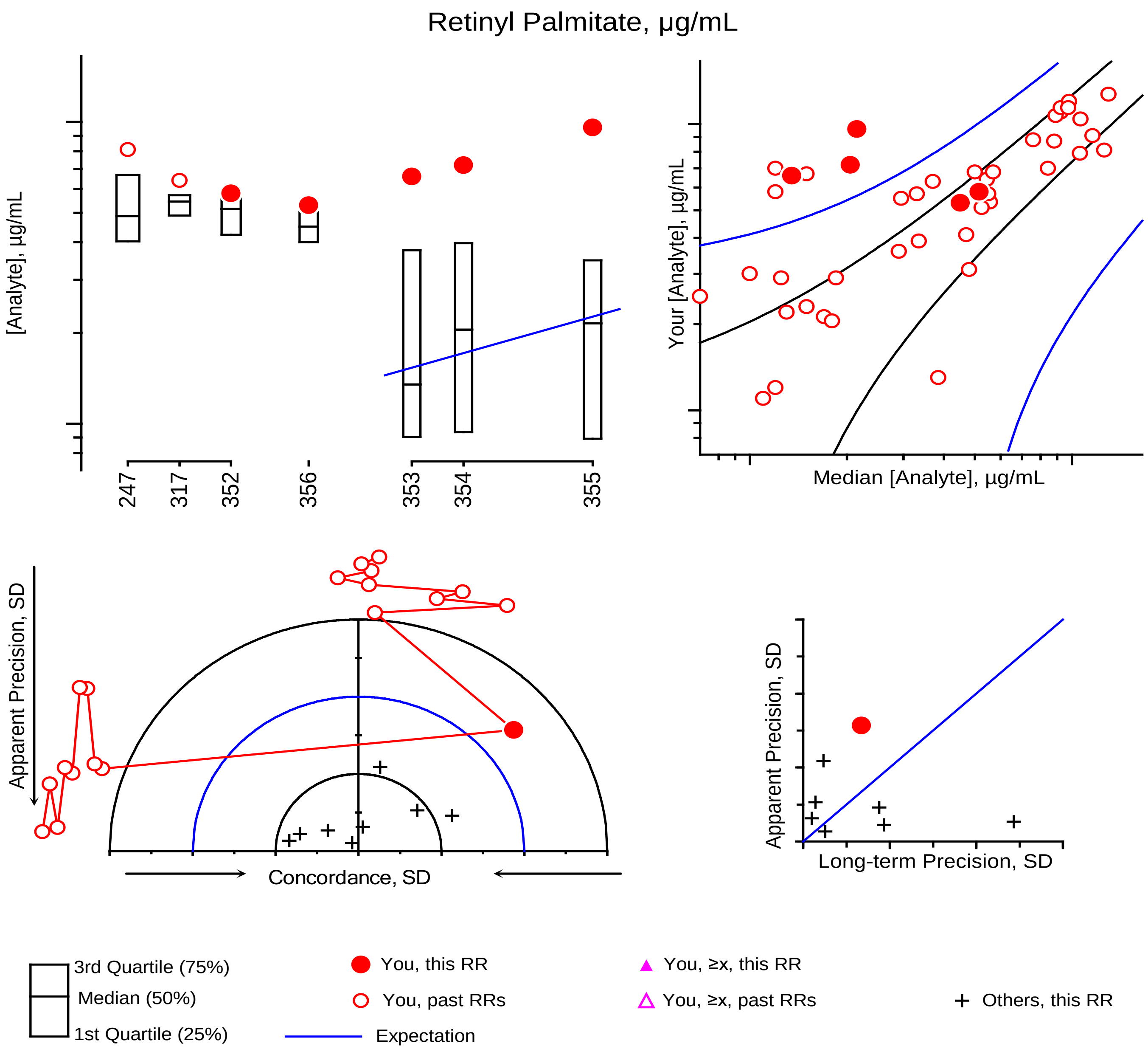
Total Retinol, µg/mL



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	Comments	History
#352	1+1 mixture of multi-donor pools #248 and #249	44:#247, 58:#317
#356	1+0.56 mixture of multi-donor pools #248 and #249	New
#353	126+4 mixture of single-donor #353 with spike pool	New
#354	120+10 mixture of single-donor #353 with spike pool	New
#355	105+25 mixture of single-donor #353 with spike pool	New

Individualized RR LXV Report: FSV-BA



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.

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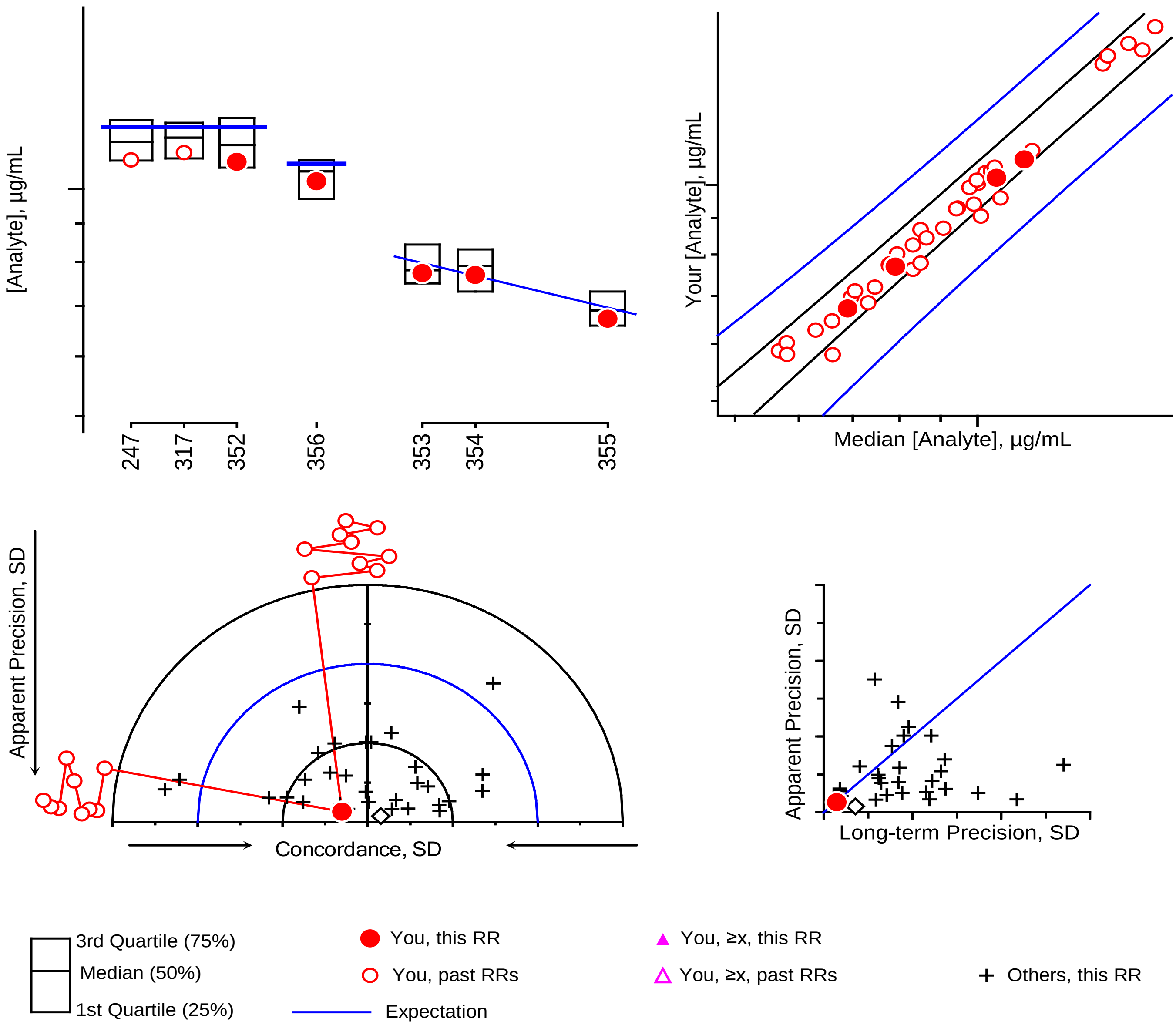
#354	120+10 mixture of single-donor #353 with spike pool	New
#355	105+25 mixture of single-donor #353 with spike pool	New

Individualized RR LXV Report: FSV-BA

α-Tocopherol, µg/mL

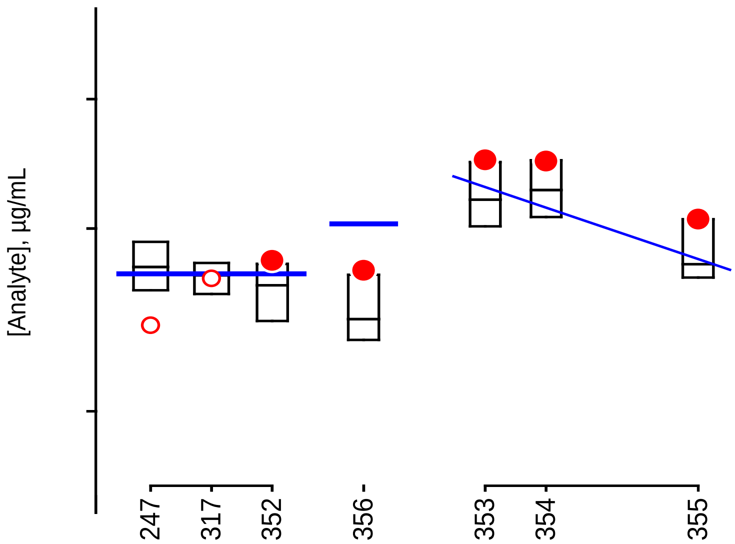


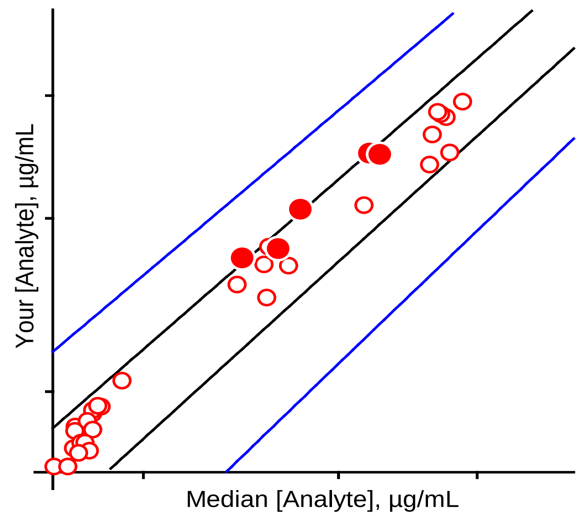
For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

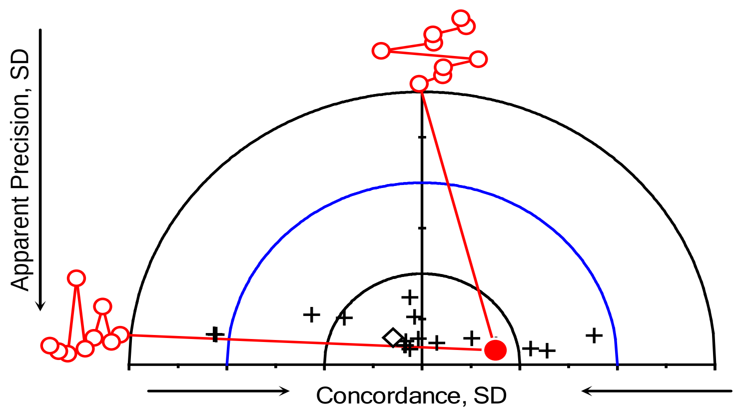
Serum	Comments	History
#352	1+1 mixture of multi-donor pools #248 and #249	44:#247, 58:#317
#356	1+0.56 mixture of multi-donor pools #248 and #249	New
#353	126+4 mixture of single-donor #353 with spike pool	New
#354	120+10 mixture of single-donor #353 with spike pool	New
#355	105+25 mixture of single-donor #353 with spike pool	New

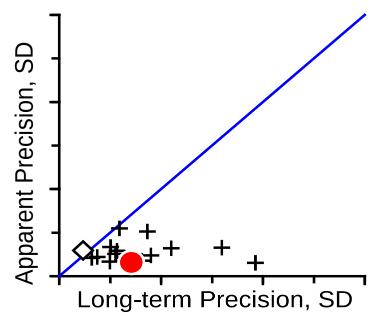
Individualized RR LXV Report: FSV-BA

γ/β-Tocopherol, µg/mL









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

You, this RR
You, past RRs
Expectation

You, ≥x, this RR
You, ≥x, past RRs

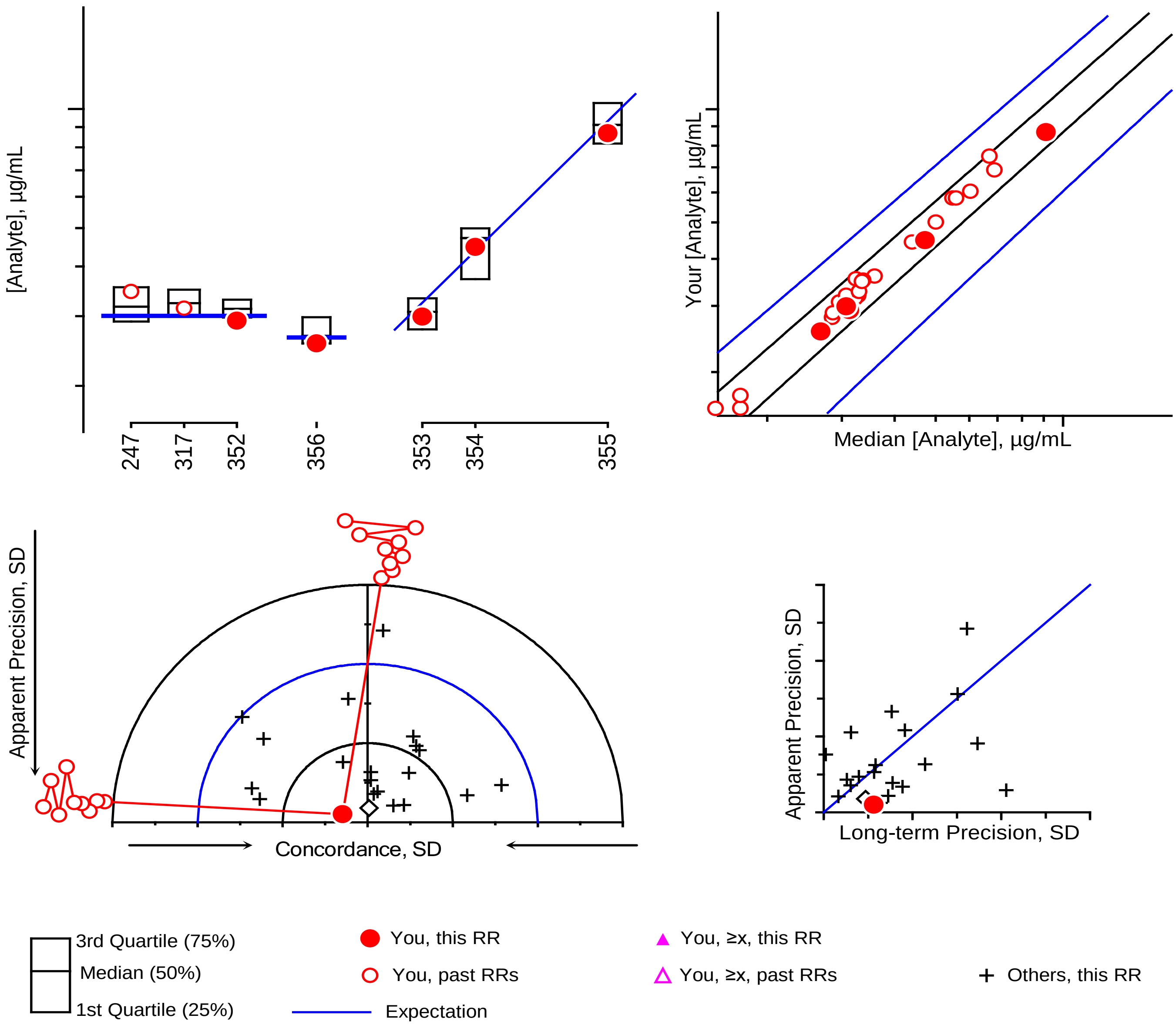
Others, this RR

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	Comments	History
#352	1+1 mixture of multi-donor pools #248 and #249	44:#247, 58:#317
#356	1+0.56 mixture of multi-donor pools #248 and #249	New
#353	126+4 mixture of single-donor #353 with spike pool	New
#354	120+10 mixture of single-donor #353 with spike pool	New
#355	105+25 mixture of single-donor #353 with spike pool	New

Individualized RR LXV Report: FSV-BA

Total β-Carotene, µg/mL

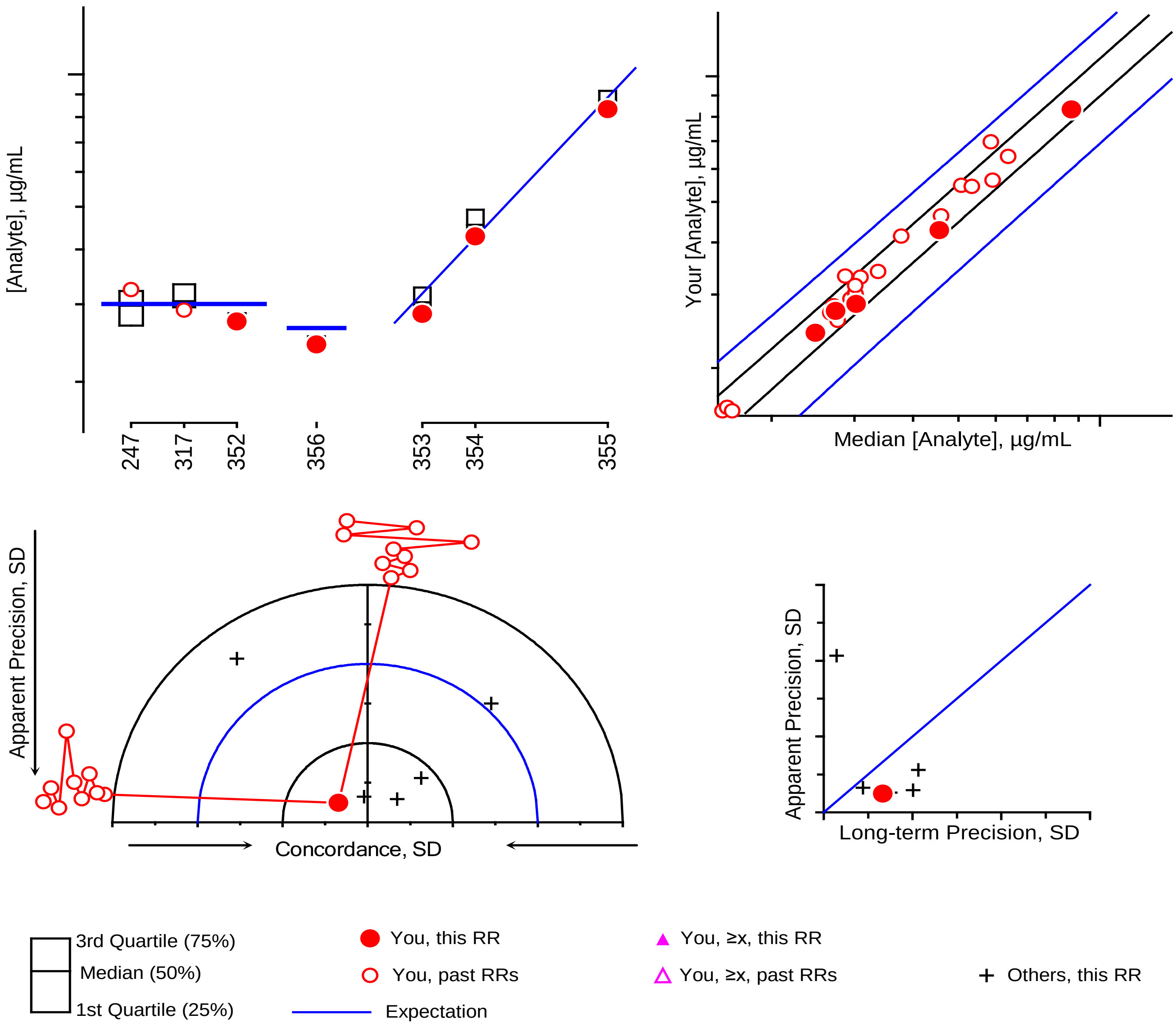


For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

Serum	Comments	History
#352	1+1 mixture of multi-donor pools #248 and #249	44:#247, 58:#317
#356	1+0.56 mixture of multi-donor pools #248 and #249	New
#353	126+4 mixture of single-donor #353 with spike pool	New
#354	120+10 mixture of single-donor #353 with spike pool	New
#355	105+25 mixture of single-donor #353 with spike pool	New

Individualized RR LXV Report: FSV-BA

trans-β-Carotene, µg/mL

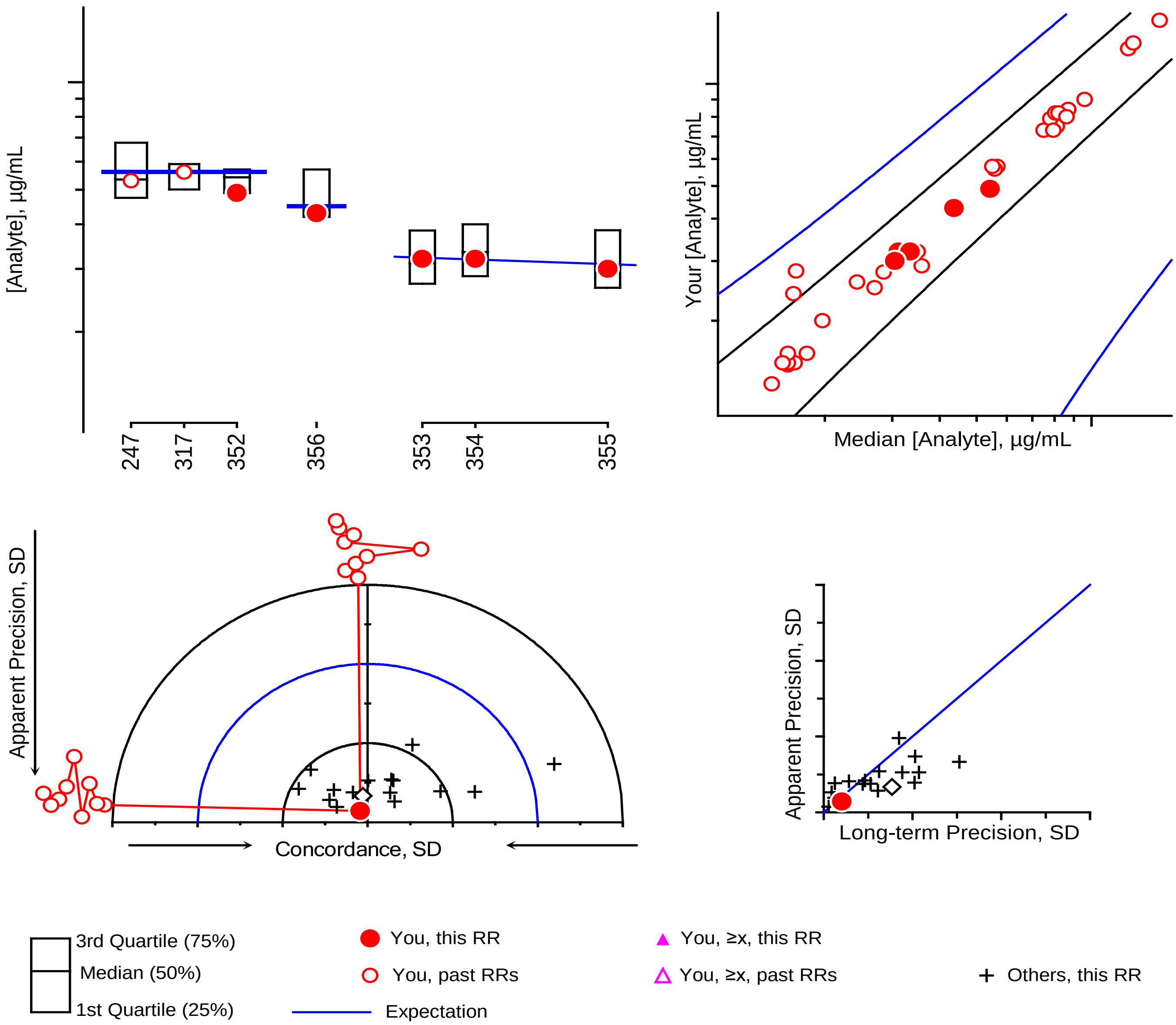


For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

Serum	Comments	History
#352	1+1 mixture of multi-donor pools #248 and #249	44:#247, 58:#317
#356	1+0.56 mixture of multi-donor pools #248 and #249	New
#353	126+4 mixture of single-donor #353 with spike pool	New
#354	120+10 mixture of single-donor #353 with spike pool	New
#355	105+25 mixture of single-donor #353 with spike pool	New

Individualized RR LXV Report: FSV-BA

Total α-Carotene, µg/mL

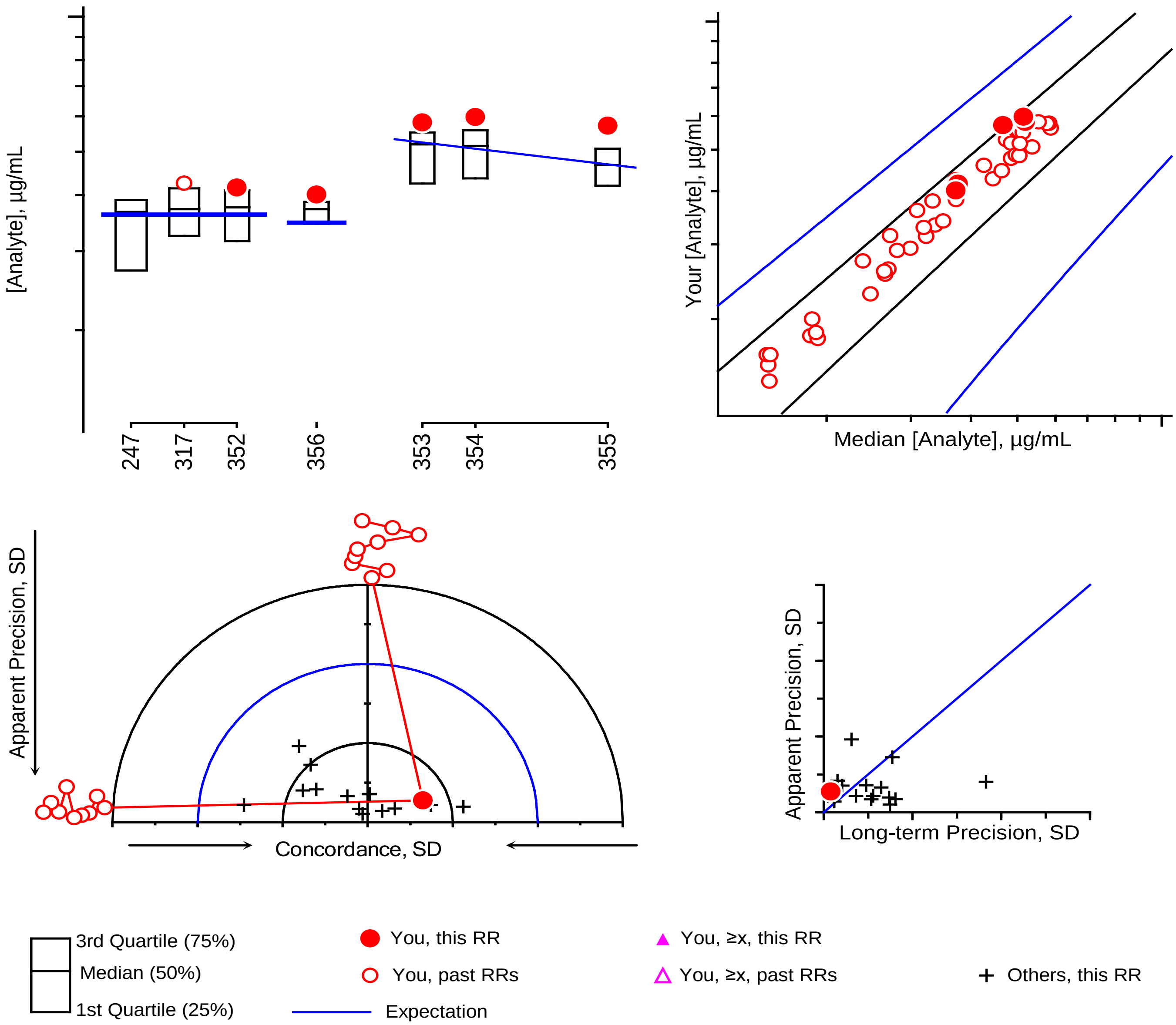


For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

Serum	Comments	History
#352	1+1 mixture of multi-donor pools #248 and #249	44:#247, 58:#317
#356	1+0.56 mixture of multi-donor pools #248 and #249	New
#353	126+4 mixture of single-donor #353 with spike pool	New
#354	120+10 mixture of single-donor #353 with spike pool	New
#355	105+25 mixture of single-donor #353 with spike pool	New

Individualized RR LXV Report: FSV-BA

Total Lycopene, µg/mL

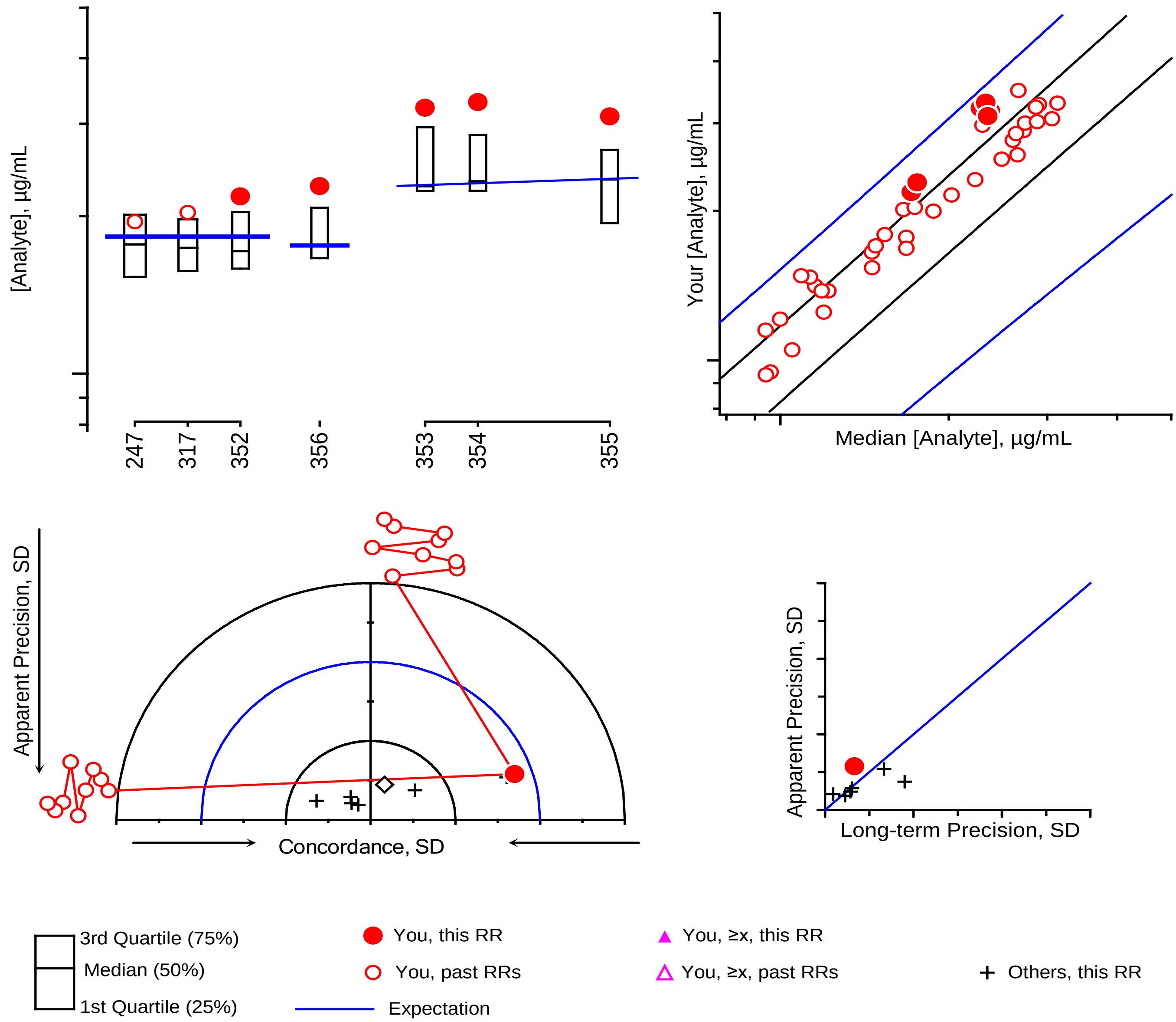


For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

Serum	Comments	History
#352	1+1 mixture of multi-donor pools #248 and #249	44:#247, 58:#317
#356	1+0.56 mixture of multi-donor pools #248 and #249	New
#353	126+4 mixture of single-donor #353 with spike pool	New
#354	120+10 mixture of single-donor #353 with spike pool	New
#355	105+25 mixture of single-donor #353 with spike pool	New

Individualized RR LXV Report: FSV-BA

trans-Lycopene, µg/mL

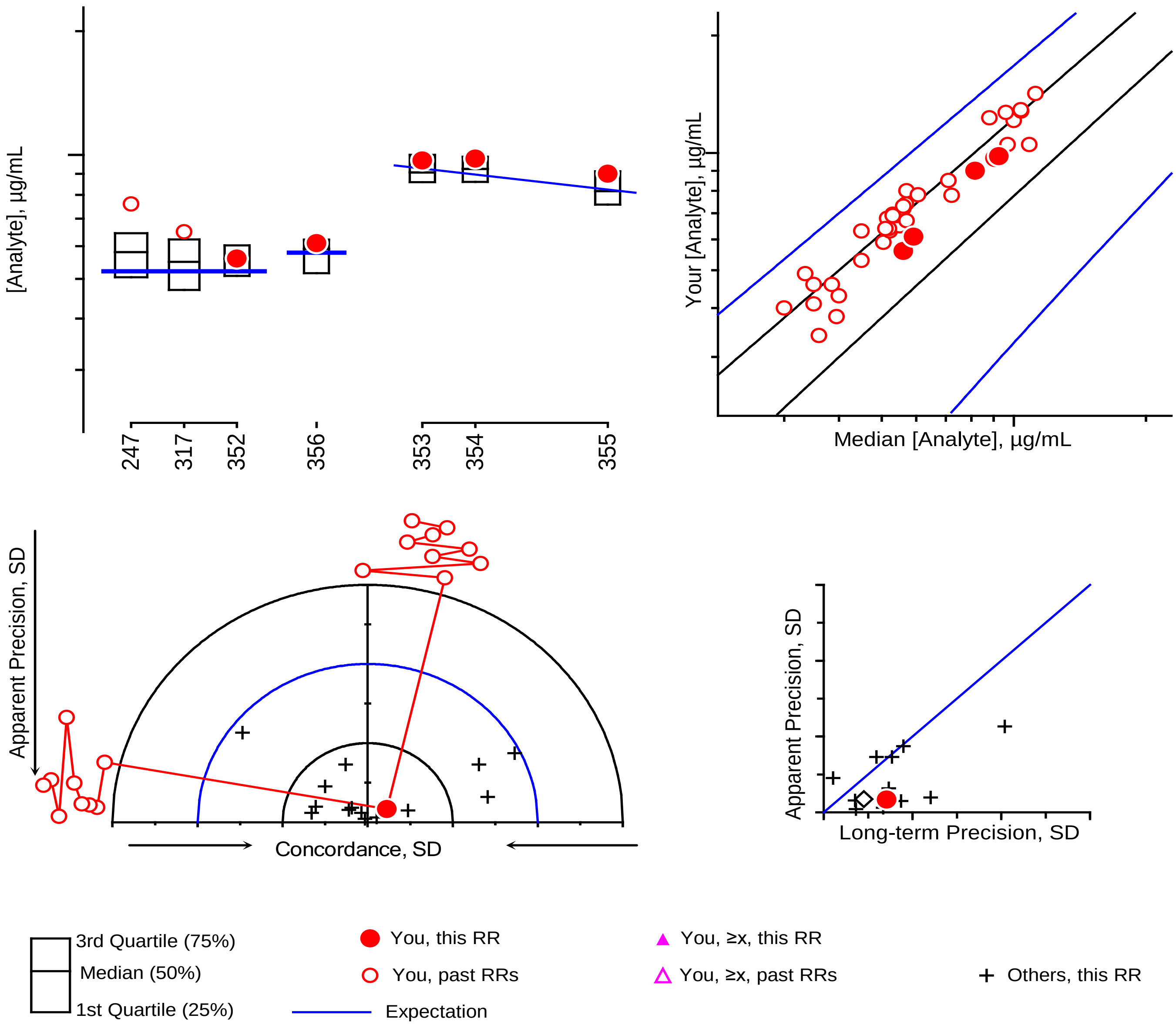


For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

Serum	Comments	History
#352	1+1 mixture of multi-donor pools #248 and #249	44:#247, 58:#317
#356	1+0.56 mixture of multi-donor pools #248 and #249	New
#353	126+4 mixture of single-donor #353 with spike pool	New
#354	120+10 mixture of single-donor #353 with spike pool	New
#355	105+25 mixture of single-donor #353 with spike pool	New

Individualized RR LXV Report: FSV-BA

Total β-Cryptoxanthin, µg/mL

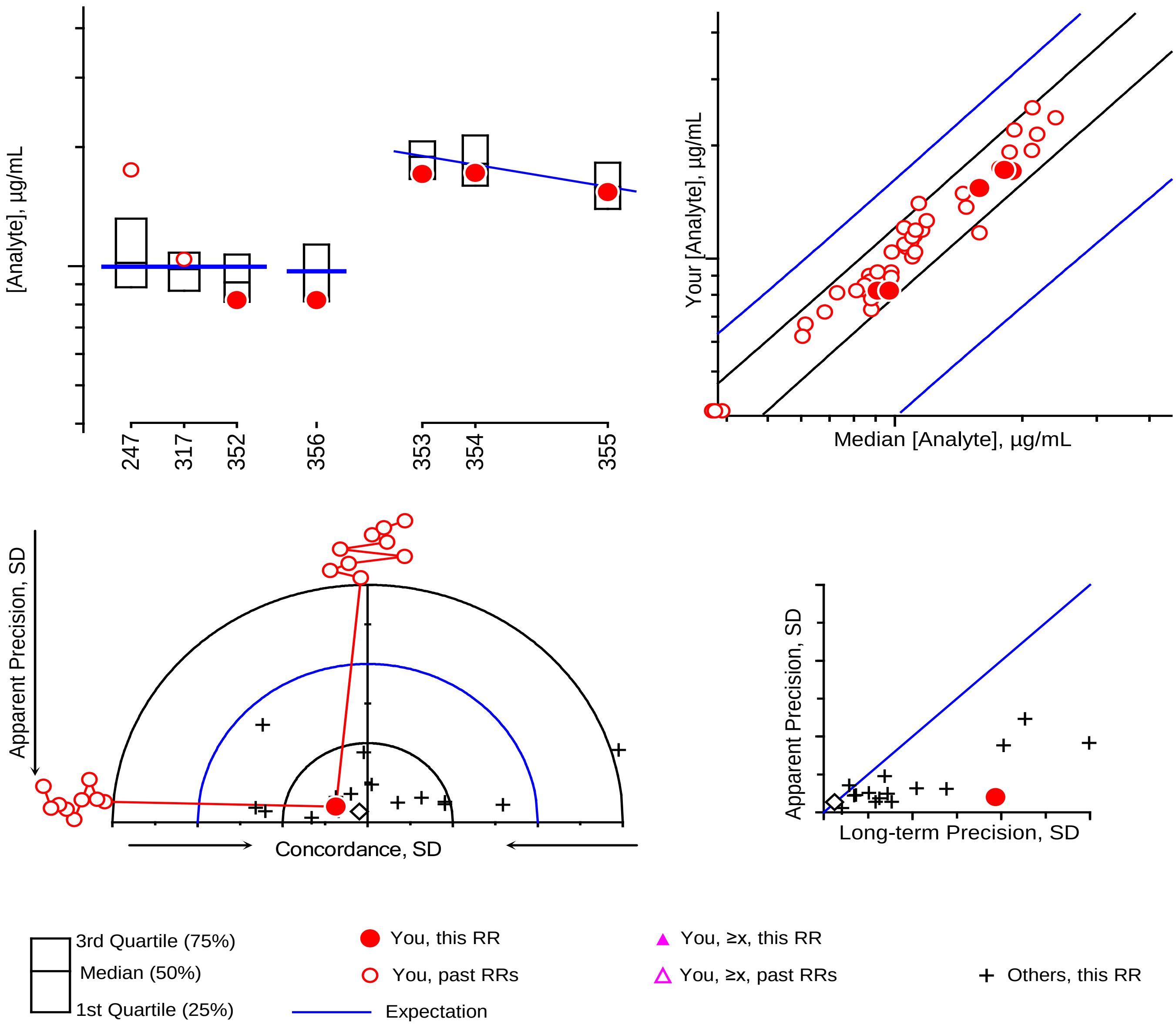


For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

Serum	Comments	History
#352	1+1 mixture of multi-donor pools #248 and #249	44:#247, 58:#317
#356	1+0.56 mixture of multi-donor pools #248 and #249	New
#353	126+4 mixture of single-donor #353 with spike pool	New
#354	120+10 mixture of single-donor #353 with spike pool	New
#355	105+25 mixture of single-donor #353 with spike pool	New

Individualized RR LXV Report: FSV-BA

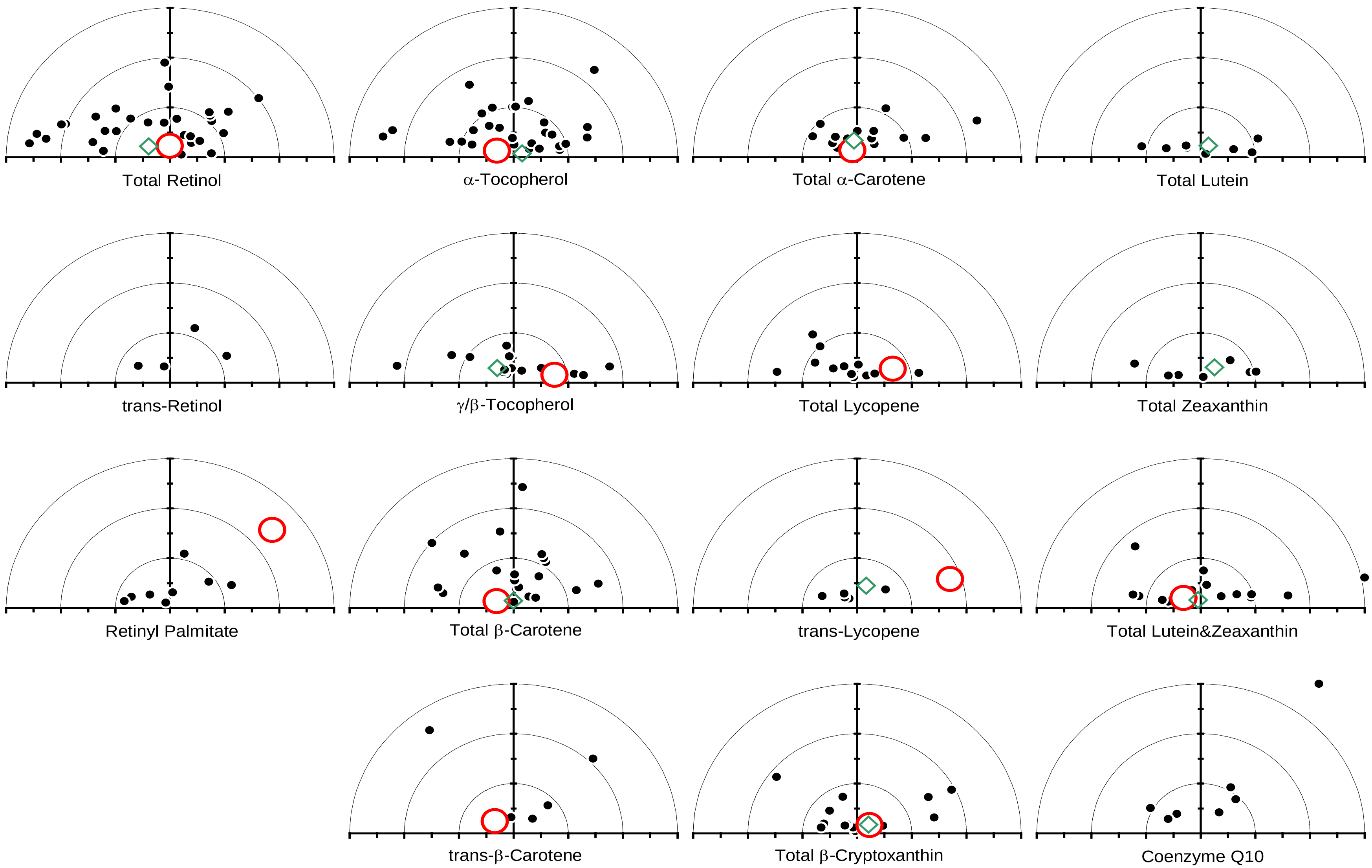
Total Lutein&Zeaxanthin, µg/mL



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	Comments	History
#352	1+1 mixture of multi-donor pools #248 and #249	44:#247, 58:#317
#356	1+0.56 mixture of multi-donor pools #248 and #249	New
#353	126+4 mixture of single-donor #353 with spike pool	New
#354	120+10 mixture of single-donor #353 with spike pool	New
#355	105+25 mixture of single-donor #353 with spike pool	New

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



D14

Appendix E. Shipping Package Inserts for RR30

The following five items were included in each package shipped to an RR30 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-

December 3, 2008

Dear Colleague:

The samples within this package constitute Vitamin C Round Robin 30 (RR30) of the 2009 Micronutrients Measurement Quality Assurance Program.

RR30 consists of four vials of frozen serum *test samples* (#27, #57, #117, and #119), one vial of frozen *control serum* (CS #1), and one vial of ascorbic acid *solid control material* (Control). 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.)

Also included for analysis is one vial of SRM 1950 Metabolites in Human Serum. We would appreciate your assistance in the value-assignment of ascorbic acid in this material. SRM 1950 is a human plasma pool that was collected from healthy male and female donors. It contains lithium heparin as an anticoagulant. **Please note: SRM 1950 does not contain metaphosphoric acid.** Therefore, please prepare this sample as you would a routine plasma sample and provide us with any preparation and dilution details you consider relevant.

Please use the control serum to validate the performance of your measurement system <u>before</u> you analyze the *test samples*. The target value and ≈95% confidence interval for *CS* #1 is 8.4 ± 0.7 µmol/L of sample.

The report for RR29 was e-mailed November 25, 2008. If you find your results for RR29 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).

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.

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 RR30 samples by **March 31, 2009**. 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 B 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
RR30 Report Form for Ascorbic Acid Solid Control Material Preparation
RR30 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 $[\mathrm{TAA}]_{\mathrm{DS2}}$ = 29.4 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 recalibrate 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 CS #1 (see protocol below). The target values for this materials is 8.4 ±0.7 µmol/L of sample.

   If your measured values are not close to this value, 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 at 301-975-3120 or jbthomas@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 material* 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 material* 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 30

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 by **March 31, 2009**

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 30

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
CS #1			µmol/L of Sample *Target:* 8.4 ±0.7 µmol/L
Serum Test Sample #27			µmol/L of Sample
Serum Test Sample #57			µmol/L of Sample
Serum Test Sample #117			µmol/L of Sample
Serum Test Sample #119			µmol/L of Sample
SRM 1950			µmol/L of Plasma
Preparation details you consider relevant to our interpretation of these results:			

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 by **March 31, 2009**

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 30

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^2QAP samples:

Label	Form
VitC #27	Liquid frozen (1:1 serum:10% MPA)
VitC #57	Liquid frozen (1:1 serum:10% MPA)
VitC #117	Liquid frozen (1:1 serum:10% MPA)
VitC #119	Liquid frozen (1:1 serum:10% MPA)
CS #1	Liquid frozen (1:1 serum:10% MPA)
Control	Solid AA
SRM 1950	Liquid frozen (plasma)

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^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 RR30

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-

May 15, 2009

Dear Colleague:

Enclosed is the summary report of the results for intercomparison study RR30 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 an individualized summary of your laboratory's measurement performance. The robust median is used to estimate the consensus value for all samples, the "median absolute deviation from the median" (MADe) is used to estimate the expected standard deviation, and the coefficient of variation (CV) is defined as 100×MADe/median.

RR30 consisted of one *serum control material* (CS#1), one *solid control material* for preparation of TAA control solutions, four *test samples* (#27 #57, #117, #119), and one optional unknown (SRM 1950). 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 study (RR31) of the 2009 M^2QAP will be shipped during the week of June 1, 2009. 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　　David Lee Duewer

Jeanice Brown Thomas, M.B.A.
Research Chemist
Analytical Chemistry Division
Chemical Science and Technology Laboratory
Laboratory

David Lee Duewer, Ph.D.
Research Chemometrician
Analytical Chemistry Division
Chemical Science and Technology

Cc: L. C. Sander

NIST

The NIST M^2QAP summary report for vitamin C intercomparison study RR30 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 serum control sample, and the four serum test samples.
2	Graphical summary of your RR30 sample measurements.

Page	"All Lab" Report
1	A tabulation of results and summary statistics for Total Ascorbic Acid [TAA] in the RR30 samples and control/calibration solutions.

Serum-based Samples. One serum control, four test unknowns, and an optional unknown (SRM 1950) were distributed in RR30.

CS#1 SRM 970 level 1, ampouled in mid-1998.

S30:1 VitC #27, ampouled in late 2001, previously distributed as sample S17:1 (RR17, Fall 02), S19:2 (RR19, Fall 03), S21:2 (RR21, Fall 04), S22:1 (RR24, Spring 05), S24:1 (RR24, Spring 06), and S28:1 (Spring 08).

S30:2 VitC #57. ampouled in late 2001, previously distributed as sample S16:3 (RR16, Spring 02), S17:3 (RR17, Fall 02), S20:2 (RR20, Spring 04), S21:4 (RR21, Fall 04), S23:3 (RR23, Fall 05), S27:2 (RR27, Fall 07).

S30:3 VitC #117, ampouled in 1995, previously distributed sample 682a=S08:3 (RR8, 1996), 682a=S10:2 (RR10, 1997), S25:4 (RR25, Fall 06), and S28:3 (Spring 08). (Unfortunately, two participants received miss-labeled units of a different serum. We apologize for the error.)

S30:4 VitC #119, ampouled in 1995, previously distributed sample 688b=S07:2 (RR7, Spring 1995), 688b=S08:2 (RR8, 1997), S24:4 (RR24, Spring 06), and S28:4 (Spring 08)

SRM 1950 Metabolites in Human Serum: A human plasma pool that was prepared in the Spring 07 from healthy male and female donors. This sample was designed for the evaluation of the measurement of metabolites in humans to provide insight into various disease states.

Results.

1) All participants who prepared the four 5% MPA control/calibration solutions (the three "Dilute Solutions" and the "Diluent") did so correctly. The criteria used to evaluate this success are: the density of the 5% MPA (≈1.03 gm/mL), the observed wavelength maximum of "Dilute Solution #1"(≈244 nm), the observed absorbance at that maximum (≈0.58 OD), the calculated $E^{1\%}$ #1"(≈560 dL/g·cm).

2) The Measured = a+b*Gravimetric calibration parameters for the control/calibration solutions (columns 10 to 13 of the All Lab Report) indicate that the measurement systems for most participants are linear (R^2 close to 1 and RMS close to 0.0) and reasonably well calibrated (intercepts close to zero and slopes close to 1). However, one participant's RMS is larger than expected and "re-calibration" to the gravimetric values degrades their results for the unknowns. This suggests that their measurement process may be non-linear and that it is sensitive to the sample matrix (5 % MPA vs. a 1+1 blend of serum and 10 % MPA).

3) One participant reported that they were unable to obtain a reasonable value for the serum control while results for the gravimetrically prepared solutions were as expected. On evaluation, they found

that an enzymatic reagent was insufficiently active, giving rise to plausible but incorrect results for the test unknown. This emphasizes the importance of validating your methods with a well-characterized control material having composition similar to that of your samples.

4) There is no evidence of sample degradation in any of the materials.

5) We appreciate your efforts to help us value-assign ascorbic acid in SRM 1950. With one exception, all of the reported results were at or below the detection limits for your methods. Since SRM 1950 plasma is not stabilized to oxidation/reduction, this was the expected result.

Appendix G. “All-Lab Report” for RR30

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" 30 - March 2009

		Control / Calibration Samples											MPA	Dilute Solution 1		
		Grav, µmol/L			Measured, µmol/L				Measured = a + b*Grav				Density	Spectrophotometry		
Lab	Date	Dil:1	Dil:2	Dil:3	Dil:1	Dil:2	Dil:3	MPA	Inter	Slope	R^2	RMS	g/mL	λ_{max}	A_{max}	$E^{1\%}$
VC-MA	03/03/09	58.1	29.6	14.5	60.0	31.3	15.1	0.0	0.19	1.03	1.000	0.4	1.037	242.	0.5690	555.6
VC-MB	17/12/08	59.5	29.8	14.7	58.3	27.9	13.4	0.0	-0.68	0.98	0.999	0.8	1.031	243.	0.5880	561.2
VC-MC	09/03/09	58.2	29.2	14.7	61.2	30.3	14.2	0.0	-0.57	1.06	1.000	0.7	1.024	243.	0.5496	536.3
VC-ME	10/02/09	56.6	28.4	14.3	57.0	28.2	13.8	0.0	-0.33	1.01	1.000	0.4	1.031	243.	0.5804	582.4
VC-MG	17/03/09	60.9	31.4	16.8	65.1	32.8	16.2	0.0	-0.89	1.08	0.999	1.0	1.029	243.6	0.5850	545.8
VC-MH	20/03/09	62.8	31.5	15.4	63.9	32.2	15.7	0.0	0.05	1.02	1.000	0.1	1.030	244.	0.6117	552.7
VC-MI	10/02/09	57.0	28.3	14.2	60.1	28.8	14.4	0.0	-0.43	1.06	1.000	0.6	1.030			
VC-MJ	27/03/09	55.7	27.8	13.4	60.5	29.7	14.9	0.4	0.31	1.08	1.000	0.5	1.018	253[a]	0.473[a]	482.5[a]
VC-MK	26/03/09	62.6	30.7	15.2	63.1	34.6	16.8	8.5	6.41	0.90	0.991	2.8	1.030	244.	0.5810	526.8
VC-MN	24/09/09	63.0	31.6	15.8	62.5	30.8	14.3	0.0	-0.64	1.00	0.999	0.7	1.029	243.7	0.6178	556.7
VC-MP	30/03/09															
VC-MU	06/03/09															
VC-NE	11/02/09															
VC-NF	17/03/09	60.5	29.5	14.9	61.2	30.7	14.9	0.2	0.16	1.01	1.000	0.6	1.031	243.	0.5930	556.9
	N	11	11	11	11	11	11	11				N	11	9	9	9
	Average	59.5	29.8	14.9	61.2	30.6	14.9	0.8				Average	1.029	243.3	0.5862	552.7
	SD	2.6	1.3	0.9	2.4	2.0	1.0	2.6				SD	0.005	0.6	0.0206	15.7
	Min	55.7	27.83	13.4	57.0	27.87	13.4	0.0				Min	1.018	242.0	0.5496	526.8
	%25	57.6	28.78	14.4	60.0	29.28	14.3	0.0				%25	1.029	243.0	0.5804	545.8
	Median	59.5	29.63	14.7	61.2	30.72	14.9	0.0				Median	1.030	243.0	0.5850	555.6
	%75	61.7	31.05	15.3	62.8	31.70	15.4	0.1				%75	1.031	243.7	0.5930	556.9
	Max	63.0	31.58	16.8	65.1	34.56	16.8	8.5				Max	1.037	244.0	0.6178	582.4
	MADe	3.7	1.8	0.7	1.9	2.1	1.0	0.0				eSD	0.0	0.9	0.0119	8.3
	CV	6	6	5	3	7	6					CV	0.15	0.37	2.0	1.5

	Samples														SRM
	Measured, µmol/L					Measured = p+q*Median				Calibrated, µmol/L					
Lab	CS#1	S30:1	S30:2	S30:3	S30:4	Inter	Slope	R^2	RMS	CS#1	S30:1	S30:2	S30:3	S30:4	1950
VC-MA	8.1	9.4	50.8	44.6	28.6	-0.16	1.07	1.000	0.4	7.7	9.0	49.0	43.1	27.5	*nd*
VC-MB	9.0	8.8	48.0	43.9	28.7	0.16	1.02	0.998	1.0	9.9	9.6	49.5	45.3	29.8	1.04
VC-MC	8.4	8.5	45.8	42.4	26.9	0.07	0.98	0.998	0.9	8.5	8.6	43.8	40.6	26.0	1.09
VC-ME	11.0	10.8	59.5	54.1	34.9	-0.06	1.27	0.999	0.9	11.3	11.0	59.2	53.9	34.9	
VC-MG	7.9	7.6	50.4	47.4	28.4	-2.33	1.14	0.997	1.4	8.2	7.9	47.7	44.8	27.2	*nd*
VC-MH	8.4	9.2	42.0	41.6	26.7	1.92	0.89	0.986	2.2	8.2	9.0	41.2	40.9	26.2	0.80
VC-MI	8.3	6.3	47.8	*b*	26.9	-2.94	1.08	0.999	1.0	8.3	6.3	45.7		25.9	*nd*
VC-MJ	*na*	13.4	49.9	42.5	27.0	3.83	0.93	0.989	2.1		12.1	46.0	39.2	24.8	8.05
VC-MK	8.4	10.8	49.8	41.1	23.4	-0.07	1.00	0.975	3.4	2.2	4.9	48.3	38.6	18.9	
VC-MN	*na*	10.7	44.9	*b*	25.3	2.25	0.89	0.998	1.1		11.4	45.6		26.0	6.56
VC-MP	8.5	8.7	46.8	42.4	26.9	-0.05	0.99	1.000	0.4						
VC-MU	6.3	7.4	42.0	*b*	23.9	-0.56	0.90	1.000	0.3						0.57
VC-NE	9.0	9.2	47.2	41.4	27.0										
VC-NF	8.9	8.7	44.5	41.0	26.8	0.80	0.94	0.998	0.9	8.6	8.5	43.8	40.3	26.3	*nd*
N	12	14	14	11	14					9	11	11	9	11	
Average	8.5	9.3	47.8	43.8	27.2					8.1	8.9	47.3	43.0	26.7	
SD	1.1	1.8	4.4	3.9	2.7					2.5	2.1	4.7	4.7	3.8	
Min	6.3	6.3	42.0	41.0	23.4					2.2	4.9	41.2	38.6	18.9	
%25	8.3	8.6	45.1	41.5	26.7					8.2	8.2	44.7	40.3	25.9	
Median	8.4	9.0	47.5	42.4	26.9					8.3	9.0	46.0	40.9	26.2	
%75	8.9	10.4	49.8	44.3	28.0					8.6	10.3	48.7	44.8	27.3	
Max	11.0	13.4	59.5	54.1	34.9					11.3	12.1	59.2	53.9	34.9	
eSD	0.5	1.3	3.7	2.0	1.2					0.5	1.5	3.3	3.2	1.4	
CV	6	15	8	5	5					5	17	7	8	5	

a) 5% Trichloroacetic acid solution
b) Mislabeled sample

Appendix H. Representative "Individualized Report" for RR30

Each participant in RR30 received an "Individualized Report" reflecting their reported results. The following two pages are the "Individualized Report" for participant "VC-MA".

Vitamin C "Round Robin" 30 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
08/28/06	25	HPLC-EC	1.039	242.0	0.555	557.4	0.8	0.95	0.999	0.92
03/20/07	26	HPLC-EC	1.033	244.0	0.573	554.3	0.3	1.00	1.000	0.31
10/05/07	27	HPLC-EC	1.032	242.0	0.561	557.2	-0.1	0.99	1.000	0.14
03/04/08	28	HPLC-EC	1.035	243.0	0.572	562.2	0.7	1.03	0.999	0.99
08/11/08	29	HPLC-EC	1.037	243.0	0.567	553.2	0.3	1.03	1.000	0.64
03/03/09	30	HPLC-EC	1.037	242.0	0.569	555.6	0.2	1.03	1.000	0.40
		Mean	1.036	242.7	0.57	556.6			Pooled SEE	0.65
		SD	0.002	0.8	0.01	3.2				
		CV	0.24	0.34	1.2	0.6				

[TAA] mmol/Lsample

Date	RR	Sample	Rep_1	Rep_2	F_{adj}	Mean	SD_{dup}	N	Mean	SD_{repeat}	SD_{reprod}
08/28/06	25	CS#1	8.3	8.6	1.0	8.4	0.2	4	8.5	0.2	0.4
03/20/07	26	CS#1	8.6	8.3	1.0	8.5	0.2				
03/04/08	28	CS#1	9.1	9.0	1.0	9.0	0.1				
03/03/09	30	CS#1	**8.3**	**8.0**	1.0	8.1	0.2				
12/12/02	17	S17:1	9.9	9.1	1.0	9.5	0.6	7	9.5	0.2	0.5
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				
03/09/06	24	S24:1	9.8	9.6	1.0	9.7	0.2				
03/04/08	28	S28:1	10.4	10.3	1.0	10.4	0.1				
03/03/09	30	S30:1	**9.5**	**9.4**	1.0	9.4	0.1				
11/18/02	16	S16:3	49.9	44.9	1.0	47.4	3.5	7	48.9	1.4	1.5
12/12/02	17	S17:3	49.7	49.1	1.0	49.4	0.4				
02/23/04	20	S20:2	50.6	50.0	1.0	50.3	0.4				
09/13/04	21	S21:4	47.1	47.0	1.0	47.0	0.0				
10/17/05	23	S23:3	49.8	48.8	1.0	49.3	0.7				
10/05/07	27	S27:2	48.6	47.6	1.0	48.1	0.8				
03/03/09	30	S30:2	**51.2**	**50.4**	1.0	50.8	0.6				
ND	08							3	42.5	0.5	2.3
08/20/97	10	S10:2	81.5	80.6	0.5	40.5	0.3				
08/28/06	25	S25:4	42.0	42.2	1.0	42.1	0.2				
03/04/08	28	S28:3	45.6	44.4	1.0	45.0	0.9				
03/03/09	30	S30:3	**44.8**	**44.5**	1.0	44.6	0.2				
05/23/95	07	S07:2	51.1	49.8	0.5	25.2	0.5	3	27.8	0.5	2.3
ND	08										
03/09/06	24	S24:4	29.0	29.5	1.0	29.2	0.4				
03/04/08	28	S28:4	29.5	28.7	1.0	29.1	0.5				
03/03/09	30	S30:4	**28.5**	**28.6**	1.0	28.6	0.0				

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

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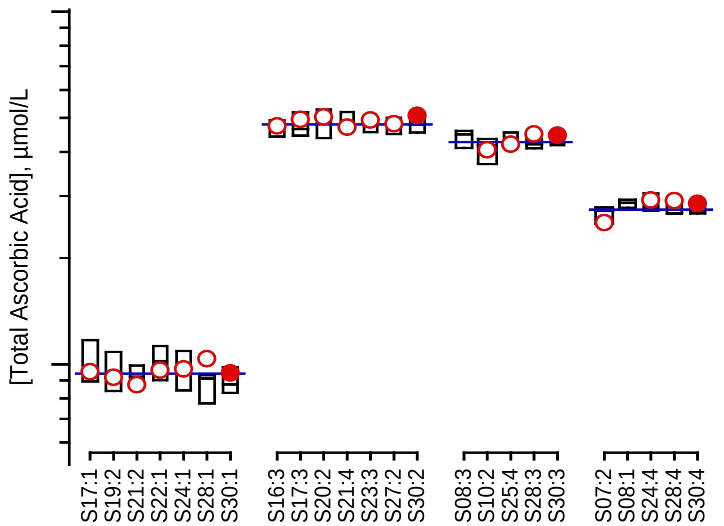
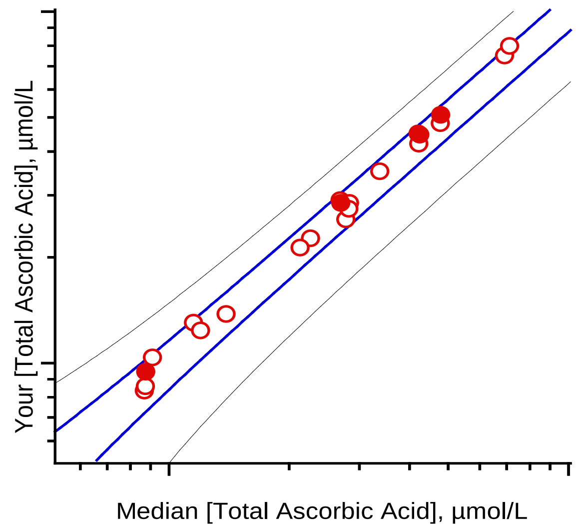
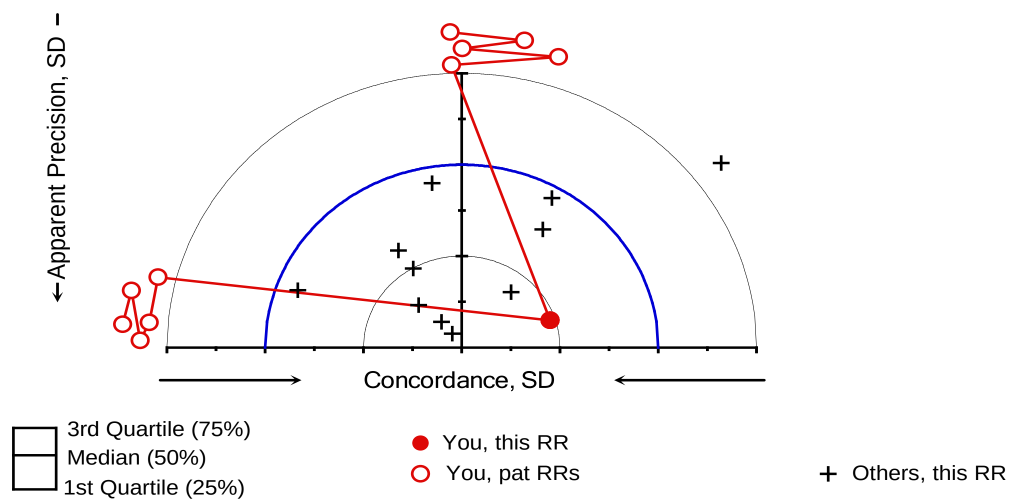
Vitamin C "Round Robin" 30 Report: Participant VC-MA

Total Ascorbic Acid, µmol/mL







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
S30:1	VitC #27 previously distributed in RRs 17, 19, 21, 22, 24, and 28
S30:2	VitC #57, previously distributed in RRs 16, 17, 20, 21, 23, and 27
S30:3	VitC #117, previously distributed in RRs 8, 10, 25, and 28
S30:4	VitC #119, previously distributed in RR 7, 8, 24, and 28