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NIST Micronutrients Measurement Quality Assurance Program Summer 2002 Comparability Studies

Results for Round Robin LII
Fat-Soluble Vitamins and Carotenoids in Human Serum
and Round Robin 17 Ascorbic Acid in Human Serum

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-XQH2013

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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 Summer 2002 MMQAP measurement comparability improvement studies: 1) Round Robin LII Fat-Soluble Vitamins and Carotenoids in Human Serum and 2) Round Robin 17 Total Ascorbic Acid in Human Serum. The materials for both studies were shipped to participants in June 2002; participants were requested to provide their measurement results by September 13, 2002.

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 LII: Fat-Soluble Vitamins and Carotenoids in Human Serum

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin LII comparability study (hereafter referred to as RR52) 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 June 2002. 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 RR52 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 17: Vitamin C in Human Serum

Participants in the MMQAP Vitamin C in Human Serum Round Robin 17 comparability study (hereafter referred to as RR17) received three frozen serum test samples and a solid ascorbic acid control material for analysis. Unless multiple vials were previously requested, participants received one vial of each material. These sample materials were shipped on dry ice to participants in June 2002. The communication materials included in the sample shipment are provided in Appendix E.

The test 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 RR17 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 RR52

The following three items were included in each package shipped to an RR52 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

June 10, 2002

Dear Colleague:

Enclosed is the set of samples for the second quality assurance round robin exercise (Round Robin LII) for the fat-soluble vitamins and carotenoids in serum. You will find one vial of each of four liquid-frozen and one 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 an obtained value is below your limit of quantification, please indicate this result on the form by using NQ *(Not Quantified)*. Results are due to NIST by **September 13, 2002.** 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 around mid-September.

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 289.**

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 LII to:

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

As you are aware, the intent-to-participate forms for the 2003 QA Program were mailed in May. Please return all forms by September 1, 2002. Laboratories will be invoiced for the 2003 program at the end of September 2002. Samples for the first fat-soluble vitamins/carotenoids and vitamin C in serum round robins will be shipped during the first week of November 2002. Please call me at (301) 975-3120; e-mail me at jbthomas@nist.gov; or mail/fax queries to the above address if you have any questions or comments.

Sincerely,

Jeanice B. Thomas

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

Enclosures

NIST

Participant #: ________ Date: ____________

Round Robin LII

NIST Micronutrients Measurement Quality Assurance Program

Analyte	284	285	286	287	288	Units*
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						
trans-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

Were sera {284,285,286,287} frozen when received? Yes | No

Comments:

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

Please return results **before**
16-Sep-2002

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

Participant #: ________ Date: ____________

Fat-Soluble Vitamins Round Robin LII

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^2QAP sera:

Serum	Form	Reconstitute?
#284	Lyophilized	Yes (1 ml H_2O)
#285	Liquid frozen	No
#286	Liquid frozen	No
#287	Liquid frozen	No
#288	Liquid frozen	No

Please 1) Open the pack immediately
2) Check that it contains one vial each of the above samples
3) Check if sera {285,286,287,288} arrived frozen
4) Store the samples upright at -20 ˚C or below until analysis
5) Complete the following information
6) Fax the completed form to us at 301-977-0685
(or email requested information to david.duewer@nist.gov)

1) Date this shipment arrived: ___________________________

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

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

4) Did sera {285,286,287,288} 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 B. Final Report for RR52

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 20899-0001

October 15, 2002

Dear Colleague:

Enclosed is the summary report of the results for Round Robin LII (RR 52) for fat-soluble vitamins and carotenoids. Included in this report are: a summary of data for all laboratories; the measurement comparability summary for evaluating laboratory performance; a summary of individual laboratory performance and interlaboratory accuracy and precision; and a summary of the NIST assigned value (NAV) vs. your laboratory value for the analytes that you measured. As in previous reports, 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 52 are provided in the comparability summary (Score Card) on page 5 of the "All Lab Report." Laboratory comparability is summarized as follows: results rated 1 to 3 are within 1 to 3 standard deviation(s) of the assigned value, respectively; those rated 4 are >3 standard deviations from the assigned value.

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

Confirmation notices acknowledging the receipt of your intent-to-participate form for the 2003 QA Program have been mailed. All participating laboratories have been invoiced and should have received billing information as well. **If you plan to participate in the 2003 program and have not received a confirmation from us, please contact me immediately.**

Samples for the first round robin exercise (RR53) will be distributed during **the week of November 4, 2002.** We will send you a reminder via e-mail or fax a week prior to shipment. Please notify us, preferably before the first week of November, if you have special shipping instructions.

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

Sincerely,

Jeanice B Thomas

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

cc: L.C. Sander
S.A. Wise

Enclosures

NIST

The NIST M^{2}QAP Round Robin LI (RR52) report consists of:

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

Page	"All Lab" Report
1-3	A listing of all results and statistics for analytes reported by at least two laboratories.
4a	A list of results for the four analytes reported by only one laboratory.
4b	A legend for the above two lists.
5	The text version of the "Comparability Summary" (or "Score Card").

Samples. The five sera below were distributed in RR52.

Serum	Description	Prior Distributions
284	Lyophilized blended serum with native carotenoid levels, augmented with α- and δ-tocopherol; SRM 968c Level II.	#249 in RR44 (9/98), #256 in RR46 (6/99), #264 in RR48 (3/01).
285	The same fresh-frozen blended serum as #288, augmented with ≈0.3 µg/mL *trans*-retinol	Initial distribution.
286	The same fresh-frozen blended serum as #288, augmented with ≈0.3 µg/mL 13-*cis*-retinol	Initial distribution.
287	Fresh-frozen single-donor hemolyzed serum with endogenous augmented carotenoid levels. Provided to the M^{2}QAP by the CDC.	Initial distribution.
288	Fresh-frozen blended serum, stored for five years at –80 °C.	Initial distribution.

Observations

1) Several participants noted that serum #287 was somewhat hemolyzed. With the exception of one participant who noted that the hemolysis interfered with their carotenoid measurements, the hemolysis had little or no apparent influence on measurements.

2) Sera Stability. There has been no significant change in the median level or in the variability of any measurand in serum #284, SRM 968c Level II.

3) Total versus *trans*-retinol. Sera #285, #286, and #288 were intentionally designed to help sort out the "Total/trans"-retinol issues we commented on in the RR51 report. The three sera were prepared from the same base material, a serum pool low in retinol and most other measurands. The three sera were produced at the same time using as close to the same preparation, handling, and storage

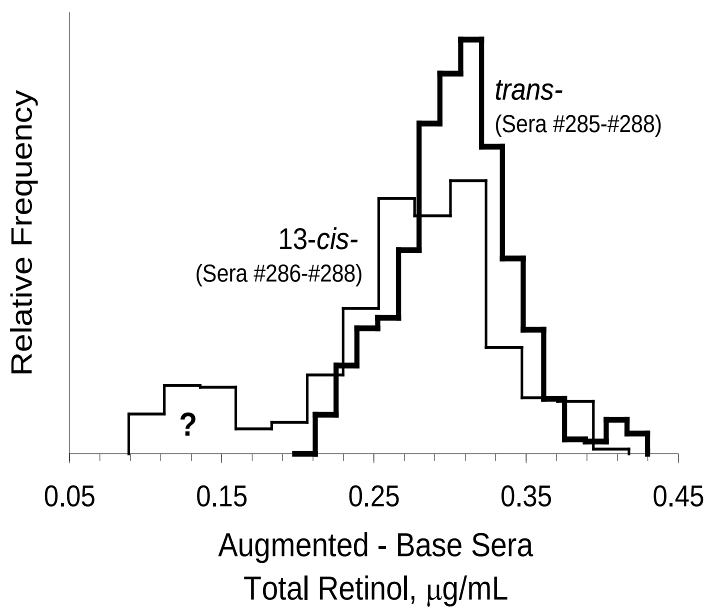
protocols as we could manage. The only intentional difference in the three materials was retinol: #285 was augmented from with 0.3 µg/mL *trans*-retinol and #286 was augmented with the same concentration of 13-*cis*-retinol. The chemical identity and purity of both materials was checked chromatographically. The *trans*-retinol spiking solution was prepared gravimetrically; the 13-*cis*-retinol was prepared spectrophotometrically to match the *trans*-retinol concentration using the extinction coefficients given in Table II of Hubbard R, Brown PK, Bownds D, Methods of Enzymology 1971;18c:615-653 (after adjusting to the correct molecular weight, the coefficients in ethanol are, respectively, 1843 dL/g·cm and 1688 dL/g·cm) . The base serum and spiking level were chosen to provide a not-unreasonably-high "total" level in the two spiked samples while providing similar 13-*cis*- and *trans*-retinol levels in serum #286.

Results

a) After discussion of their original reported values, several participants re-evaluated their "retinol" identification and/or measurement protocol and decided that they were reporting the wrong component.

b) The "*trans*-retinol" for the three sera is about what we expect, although there are only seven complete sets (including two NIST sets) so the "robust" medians are not very robust: 0.33 µg/mL in the base serum pool #288, 0.38 µg/mL in the 13-*cis*-augmented #286, and 0.61 µg/mL in the *trans*-augmented #285.

c) The "total retinol" for the three sera is (mostly) also about what we expect: 0.33 µg/mL in the base serum pool #288, 0.58 µg/mL in the 13-*cis*-augmented #286, and 0.64 µg/mL in the *trans*-augmented #285. However, five of the 38 values for the 13-*cis*-augmented #286 are halfway between (about 0.45 µg/mL) what we expect for total and *trans*-retinol. Given the normal among-participant variation in the reported values, the most quantitative way of evaluating these data is by examining the differences between the two augmented sera (#285 and #286) and the base serum (#288). The graph to the left displays the distributions of these differences as smoothed histograms. Note the small "bump" on the left tail of the #286 - #288 distribution: these five values might arise from some systematic integration decision regarding a partially resolved doublet, but we do not yet understand what is going on. We would appreciate suggestions!



d) Excluding the five sets of values where the total retinol for #286 is suspect, the median difference in total retinol for #285 - #288 is 0.300 µg/mL and for #286 - #288 is 0.275 µg/mL. Given the 1688/1843 = 0.92 ratio between the extinction coefficients, this is exactly the expected result. This implies that few if any of the participants took into account the reduced extinction coefficient for 13-*cis*-retinol when calculating the total.

4) <u>Repeatability.</u> With the exception of total and *trans*-retinol measurands, Sera #285, #286, and #288 were intended to be identical. From the summary statistics, all other measurands reported by at least ten participants do appear to be the same in all three sera. If you reported results that differ significantly (chemically, *not* statistically) among these three sera, you should re-examine your overall measurement process.

Request

We need to better understand the chromatography that produced the "halfway between" total retinols for serum #286. If your total retinol for #286 is less than 0.20 μg/mL larger then for #288, please carefully re-reevaluate the relevant sections of your chromatograms. We would appreciate hearing about your analysis and would particularly like to see the chromatogram with annotated integration parameters.

Please note!

1) We prepared a fair large batch of the 13-*cis*-augmented sera. It will be distributed again, though (probably) not in conjunction with both the *trans*-augmented and base sera. If you need to analyze a 13-*cis*-augmented serum again to confirm that you're indeed measuring what you think you're measuring, let us know before the Nov 4th shipment of RR LIII and we will include it in your shipment.

2) It is critical that you carefully inspect all samples on arrival and that you promptly confirm to us that they have arrived. We will now replace samples that have been lost or damaged in shipment or mis-packaged by us provided that participants report the problem within *one calendar* week after the package's arrival.

Appendix C. “All-Lab Report” for RR52

The following five 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 analysts. The NIST results are not used in the assessment of the consensus summary results of the study.

Round Robin LII Laboratory Results

All Results in µg/mL

Lab	Total Retinol 284	285	286	287	288	trans-Retinol 284	285	286	287	288
FSV-BA	0.490	0.640	0.538	0.609	0.323					
FSV-BB	0.483	0.607	0.598	0.598	0.315					
FSV-BE	0.560	0.720	0.630	0.680	0.380					
FSV-BF	0.500	0.600	0.470	0.600	0.320					
FSV-BG	0.464	0.597	0.578	0.603	0.303					
FSV-BH	0.442	0.575	0.418	0.558	0.306					
FSV-BI	0.510	0.682	0.647	0.677	0.351					
FSV-BJ	0.472	0.604	0.587	0.591	0.313					
FSV-BK	0.488	0.546	0.554	0.552	0.315					
FSV-BL	0.400	0.570	0.600	0.570	0.290					
FSV-BM	0.481	0.659	0.626	0.660	0.331					
FSV-BN	0.457	0.540	0.544	0.525	0.299					
FSV-BO	0.450	0.636	0.579	0.573	0.329					
FSV-BP	0.496	0.656	0.582	0.637	0.348					
FSV-BQ	0.580	0.690	0.580	0.660	0.380					
FSV-BR	>0.530	>0.620		>0.640	>0.340	0.53	0.62	-	0.64	0.34
FSV-BS	>0.356	>0.418		>0.435	>0.208	0.356	0.418	0.382	0.435	0.208
FSV-BT	0.437	0.718	0.802	0.676	0.463					
FSV-BU										
FSV-BV	0.546	0.715	0.498	0.696	0.370					
FSV-BW	0.432	0.634	0.616	0.618	0.323					
FSV-BX	0.484	0.637	0.635	0.616	0.333	0.484	0.637	0.307	0.616	0.333
FSV-CB	0.493	0.542	0.497	0.523	0.302					
FSV-CC	0.570	0.580	0.560	0.590	0.300	0.530	0.580	0.410	0.590	0.300
FSV-CD	0.458	0.583	0.580	0.579	0.317					
FSV-CE	0.472	0.578	0.566	0.577	0.308					
FSV-CF	0.523	0.690	0.502	0.668	0.377					
FSV-CG	0.595	0.678	0.649	0.680	0.361					
FSV-CI	0.410	0.550	0.420	0.530	0.330					
FSV-CL	0.431	0.593	0.564	0.679	0.308					
FSV-CP										
FSV-CR	0.490	0.710	0.650	0.730	0.350					
FSV-CW	>0.461	>0.597	0.579	>0.529	>0.326	0.461	0.597	0.415	0.529	0.326
FSV-CZ	0.450	0.590	0.600	0.580	0.310					
FSV-DD	0.519	-	0.565	0.594	0.353					
FSV-DF	0.471	0.608	0.582	0.618	0.351	0.471	0.608	0.367	0.618	0.351
FSV-DI	0.519	0.789	0.699	0.712	0.384					
FSV-DQ										
FSV-DR	0.480	0.640	0.680	0.640	0.320					
FSV-DW	0.520	0.640	0.590	0.620	0.340					
FSV-EQ	0.445	-	0.546	0.676	0.302					
FSV-ET	0.460	0.550	0.540	0.560	0.270					
FSV-FB	0.460	0.660	0.690	0.570	0.320					
N	37	35	38	37	37	6	6	5	6	6
Min	0.400	0.540	0.418	0.523	0.270	0.356	0.418	0.307	0.435	0.208
Median	0.481	0.634	0.580	0.609	0.323	0.478	0.603	0.382	0.603	0.330
Max	0.595	0.789	0.802	0.730	0.463	0.530	0.637	0.415	0.640	0.351
SD	0.039	0.065	0.056	0.068	0.032	0.041	0.030	0.032	0.054	0.024
CV	8	10	10	11	10	9	5	8	9	7
N_{past}	46	0	0	0	0	9	0	0	0	0
$Median_{past}$	0.483					0.487				
SD_{past}	0.038					0.042				
NISTa	0.486	0.668	0.691	0.610	0.364	0.486	0.668	0.417	0.610	0.364
NISTb	0.497	0.649	0.647	0.637	0.348	0.484	0.644	0.367	0.620	0.348
N_{NIST}	4	4	4	4	4	4	4	4	4	4
Mean	0.492	0.658	0.669	0.624	0.356	0.485	0.656	0.392	0.615	0.356
S_{rep}	0.006	0.026	0.010	0.020	0.009	0.006	0.027	0.008	0.020	0.009
S_{het}	0.003	0.007	0.003	0.004	0.001	0.003	0.009	0.004	0.007	0.001
S_{anl}	0.007	0.013	0.031	0.019	0.011	0.002	0.017	0.035	0.007	0.011
S_{NIST}	0.010	0.030	0.033	0.028	0.014	0.007	0.033	0.036	0.022	0.014
NAV	0.487	0.647	0.624	0.618	0.340	0.482	0.629	0.387	0.609	0.343
NAU	0.043	0.069	0.086	0.070	0.040	0.041	0.061	0.037	0.055	0.033

Lab	Retinyl Palmitate 284	285	286	287	288	α-Tocopherol 284	285	286	287	288
FSV-BA	0.069	0.017	0.017	0.028	0.016	16.97	2.90	2.95	13.41	2.90
FSV-BB	0.078	0.014	0.012	0.053	0.011	18.01	2.97	2.97	14.16	3.03
FSV-BE						16.00	3.00	2.80	12.30	2.90
FSV-BF						16.90	3.00	2.90	13.30	3.20
FSV-BG	0.081	0.028	0.028	0.054	0.024	16.70	2.90	2.84	13.50	2.80
FSV-BH						16.66	2.94	2.95	13.44	2.91
FSV-BI	0.094	*nq*	*nq*	*nq*	*nq*	16.66	2.96	2.94	13.62	2.92
FSV-BJ	0.076	*nq*	*nq*	*nq*	*nq*	16.86	2.84	2.84	13.83	2.81
FSV-BK						16.30	2.50	2.50	10.80	2.70
FSV-BL						13.78	3.01	3.01	12.49	3.01
FSV-BM						16.00	3.00	3.10	13.10	3.10
FSV-BN	0.072	0.007	0.015	*nq*	0.007	17.38	2.71	2.76	12.33	2.71
FSV-BO						19.10	2.60	3.40	15.10	3.20
FSV-BP						11.47	3.53	3.50	10.61	3.78
FSV-BQ						19.50	3.30	3.10	14.30	3.40
FSV-BR						17.47	3.84	3.78	16.50	4.06
FSV-BS										
FSV-BT	0.093	0.008	0.009	0.029	0.007	16.41	3.20	3.12	14.78	2.98
FSV-BU										
FSV-BV						15.80	2.70	2.63	11.92	2.61
FSV-BW	0.094	0.020	0.018	0.031	0.019	15.83	3.02	2.99	13.35	2.99
FSV-BX						16.62	3.00	3.01	8.68	2.95
FSV-CB						15.48	2.69	2.62	11.65	2.69
FSV-CC						18.73	3.03	3.03	13.91	2.99
FSV-CD	0.083	0.013	0.013	0.023	0.015	15.64	2.76	2.72	12.29	3.06
FSV-CE						16.26	2.66	3.22	12.29	2.59
FSV-CF						16.90	3.00	2.90	12.90	3.00
FSV-CG						14.48	2.59	2.61	10.13	2.58
FSV-CI	0.060	*nq*	*nq*	0.020	*nq*	14.90	2.70	2.60	11.40	2.70
FSV-CL						20.45	4.35	4.25	21.23	4.12
FSV-CP						15.07	2.78	2.45	13.22	3.42
FSV-CR						17.10	2.60	2.50	13.40	2.60
FSV-CW	0.057	0.011	0.009	0.018	0.012	16.28	2.80	2.77	12.81	2.77
FSV-CZ						16.20	2.54	2.62	9.30	2.46
FSV-DD										
FSV-DF										
FSV-DI	0.066	0.010	0.014	0.017	0.021	18.19	3.45	3.38	13.98	3.41
FSV-DQ						18.14	2.70	2.84	15.80	3.10
FSV-DR						17.76	3.19	3.09	13.24	2.97
FSV-DW						18.30	3.13	2.97	13.50	3.26
FSV-EQ						17.20	-	2.80	13.70	2.70
FSV-ET						16.30	2.70	2.70	12.50	2.60
FSV-FB						15.46	4.77	3.49	13.07	3.67
N	12	9	9	9	9	39	38	39	39	39
Min	0.057	0.007	0.009	0.017	0.007	11.47	2.50	2.45	8.68	2.46
Median	0.077	0.013	0.014	0.028	0.015	16.66	2.95	2.94	13.24	2.97
Max	0.094	0.028	0.028	0.054	0.024	20.45	4.77	4.25	21.23	4.12
SD	0.013	0.005	0.004	0.008	0.006	1.12	0.24	0.26	1.09	0.33
CV	17	41	27	29	39	7	8	9	8	11
N_{past}	15	0	0	0	0	49	0	0	0	0
$Median_{past}$	0.082					16.628				
SD_{past}	0.020					1.429				
NISTa						17.08	2.98	3.46	12.63	3.11
NISTb						16.90	2.77	2.80	12.67	2.80
N_{NIST}						4	4	4	4	4
Mean						16.99	2.88	3.13	12.65	2.95
S_{rep}						0.52	0.22	0.44	0.19	0.12
S_{het}						0.48	0.14	0.15	0.31	0.09
S_{anl}						0.12	0.15	0.47	0.03	0.22
S_{NIST}						0.72	0.30	0.66	0.36	0.26
NAV	0.076	0.014	0.014	0.029	0.014	16.82	2.97	2.97	12.96	2.97
NAU	0.021	0.006	0.006	0.013	0.006	1.31	0.51	0.51	1.19	0.51

Lab	γ/β-Tocopherol 284	285	286	287	288	δ-Tocopherol 284	285	286	287	288
FSV-BA	1.63	0.78	0.76	2.24	0.77					
FSV-BB	1.52	0.67	0.67	1.98	0.66					
FSV-BE	1.60	0.70	0.70	2.10	0.70					
FSV-BF	1.70	0.60	0.70	2.30	0.70					
FSV-BG	1.56	0.77	0.79	2.21	0.75					
FSV-BH	1.34	0.68	0.74	1.79	0.67					
FSV-BI	1.65	0.84	0.84	2.30	0.85					
FSV-BJ	1.56	0.71	0.71	2.25	0.71					
FSV-BK										
FSV-BL										
FSV-BM										
FSV-BN	1.50	0.60	0.63	1.80	0.71	0.527	0.025	0.047	0.119	0.050
FSV-BO										
FSV-BP										
FSV-BQ										
FSV-BR										
FSV-BS										
FSV-BT	1.51	0.59	0.58	1.86	0.54	0.899	0.116	0.077	0.165	0.023
FSV-BU										
FSV-BV	1.65	0.77	0.75	2.19	0.76					
FSV-BW	1.65	0.82	0.84	2.45	0.83					
FSV-BX	1.52	0.67	0.73	1.73	0.72					
FSV-CB										
FSV-CC										
FSV-CD	1.60	0.80	0.80	2.10	0.81					
FSV-CE										
FSV-CF										
FSV-CG	1.72	0.85	0.83	2.47	0.82	0.817	0.110	0.094	0.262	0.081
FSV-CI	1.50	0.70	0.70	2.20	0.70					
FSV-CL	1.91	0.97	0.96	2.83	0.91					
FSV-CP	1.65	0.86	0.77	2.57	1.28					
FSV-CR										
FSV-CW	1.61	0.76	0.76	2.20	0.76	0.680	0.040	0.030	0.120	0.040
FSV-CZ										
FSV-DD										
FSV-DF										
FSV-DI	1.67	0.86	0.84	2.37	0.85	0.957	0.067	0.065	0.199	0.065
FSV-DQ	1.67	0.69	0.74	2.66	0.76					
FSV-DR										
FSV-DW										
FSV-EQ										
FSV-ET	1.40	0.60	0.60	2.10	0.60					
FSV-FB										
N	22	22	22	22	22	5	5	5	5	5
Min	1.34	0.59	0.58	1.73	0.54	0.527	0.025	0.030	0.119	0.023
Median	1.61	0.73	0.75	2.21	0.76	0.817	0.067	0.065	0.165	0.050
Max	1.91	0.97	0.96	2.83	1.28	0.957	0.116	0.094	0.262	0.081
SD	0.10	0.11	0.07	0.19	0.09	0.163	0.052	0.022	0.059	0.019
CV	6	15	9	8	12	20	77	35	35	37
N_{past}	27	0	0	0	0	9	0	0	0	0
$Median_{past}$	1.577					0.738				
SD_{past}	0.141					0.350				
NISTa	1.58	1.07	0.97	2.50	0.89					
NISTb	1.52	0.73	0.70	1.96	0.71	0.562	0.056	0.057	0.114	0.058
N_{NIST}	4	4	4	4	4	2	2	2	2	2
Mean	1.55	0.89	0.83	2.23	0.80	0.562	0.056	0.057	0.114	0.058
S_{rep}	0.07	0.05	0.12	0.11	0.05	0.021	0.007	0.008	0.013	0.004
S_{het}	0.10	0.09	0.06	0.25	0.10	0.043	0.029	0.030	0.013	0.028
S_{anl}	0.05	0.23	0.19	0.38	0.13					
S_{NIST}	0.13	0.25	0.24	0.47	0.17	0.048	0.030	0.031	0.018	0.028
NAV	1.58	0.79	0.79	2.22	0.79	0.690	0.059	0.059	0.140	0.059
NAU	0.18	0.24	0.24	0.47	0.24	0.243	0.039	0.039	0.069	0.039

Lab	Total β-Carotene 284	285	286	287	288
FSV-BA	0.381	0.046	0.044	1.276	0.045
FSV-BB	0.422	0.049	0.048	1.373	0.047
FSV-BE	0.463	0.049	0.044	1.503	0.045
FSV-BF	0.432	0.057	0.054	1.662	0.053
FSV-BG	0.461	0.046	0.046	1.499	0.049
FSV-BH	0.449	0.056	0.054	1.522	0.055
FSV-BI	0.464	0.059	0.056	1.733	0.058
FSV-BJ	0.450	0.060	0.059	1.627	0.058
FSV-BK					
FSV-BL					
FSV-BM					
FSV-BN	0.440	0.039	0.041	1.405	0.039
FSV-BO	0.576	0.092	0.053	2.110	0.092
FSV-BP	0.466	0.076	0.109	1.221	0.087
FSV-BQ					
FSV-BR					
FSV-BS	>1.060	>0.024	>0.007	>3.180	>0.026
FSV-BT	0.418	0.067	0.070	1.512	0.065
FSV-BU	0.391	0.044	0.041	1.480	0.040
FSV-BV	0.413	0.052	0.051	1.390	0.063
FSV-BW	0.377	0.049	0.049	1.467	0.049
FSV-BX	>0.389	>0.050	>0.050	>1.330	>0.050
FSV-CB	0.346	0.048	0.044	1.161	0.052
FSV-CC					
FSV-CD	0.428	0.046	0.048	1.255	0.043
FSV-CE	0.426	0.052	0.042	1.368	0.047
FSV-CF					
FSV-CG	0.456	0.049	0.048	1.634	0.049
FSV-CI	0.337	0.041	0.041	-	0.043
FSV-CL	0.329	0.052	0.049	1.375	0.049
FSV-CP					
FSV-CR					
FSV-CW	0.400	0.053	0.052	1.295	0.052
FSV-CZ	0.380	0.380	0.370	1.170	0.040
FSV-DD					
FSV-DF					
FSV-DI	>0.419	>0.055	>0.053	>1.440	>0.054
FSV-DQ	0.502	0.052	0.021	1.860	0.059
FSV-DR	0.390	0.080	0.070	1.300	0.030
FSV-DW	0.481	0.060	0.053	1.038	0.053
FSV-EQ	0.352	>0.048	>0.048	>1.341	>0.052
FSV-ET	0.380	0.050	0.050	1.350	0.050
FSV-FB	0.400	0.038	0.038	1.450	0.042
N	29	28	28	27	28
Min	0.329	0.038	0.021	1.038	0.030
Median	0.422	0.052	0.049	1.405	0.049
Max	0.576	0.380	0.370	2.110	0.092
SD	0.056	0.009	0.008	0.163	0.008
CV	13	17	15	12	17
N_{past}	33	0	0	0	0
$Median_{past}$	0.423				
SD_{past}	0.049				
NISTa	0.439	*nq*	0.084	1.385	0.056
NISTb	0.469	0.050	0.054	1.592	0.054
N_{NIST}	4	2	4	4	4
Mean	0.454	0.050	0.069	1.488	0.055
S_{rep}	0.014	0.003	0.004	0.048	0.003
S_{het}	0.016	0.001	0.005	0.054	0.001
S_{anl}	0.021		0.021	0.147	0.002
S_{NIST}	0.030	0.003	0.022	0.164	0.003
NAV	0.437	0.054	0.054	1.458	0.054
NAU	0.064	0.018	0.018	0.188	0.018

C3

Round Robin LII Laboratory Results

All Results in µg/mL

Lab	trans-β-Carotene 284	285	286	287	288	Total cis-β-Carotene 284	285	286	287	288	Total α-Carotene 284	285	286	287	288
FSV-BA	0.356	0.044	0.042	1.221	0.043	0.025	0.002	0.002	0.055	0.002	0.085	0.005	0.004	0.029	0.005
FSV-BB	0.395	0.047	0.046	1.310	0.044	0.027	0.002	0.002	0.064	0.003	0.093	0.003	0.004	0.031	0.003
FSV-BE															
FSV-BF											0.105	0.008	0.007	0.034	0.009
FSV-BG											0.098	0.012	0.012	0.042	0.014
FSV-BH	0.417	0.056	0.054	1.445	0.055	0.032	*nq*	*nq*	0.077	*nq*	0.100	*nq*	*nq*	0.033	*nq*
FSV-BI											0.099	0.004	0.004	0.034	0.004
FSV-BJ											0.124	*nq*	*nq*	0.032	*nq*
FSV-BK															
FSV-BL															
FSV-BM															
FSV-BN	0.402	0.036	0.038	1.325	0.037	0.028	*nq*	*nq*	0.070	*nq*	0.097	*nq*	*nq*	0.023	*nq*
FSV-BO											0.130	*nq*	*nq*	0.044	*nq*
FSV-BP											0.101	*nq*	*nq*	0.038	0.013
FSV-BQ															
FSV-BR															
FSV-BS	1.060	0.024	0.007	3.180	0.026										
FSV-BT	0.384	0.062	0.064	1.424	0.060	0.034	0.005	0.006	0.089	0.005	0.084	0.009	0.011	0.036	0.009
FSV-BU											0.098	0.003	0.003	0.027	0.004
FSV-BV											0.075	0.005	0.003	0.021	0.004
FSV-BW											0.110	*nq*	*nq*	0.021	*nq*
FSV-BX	0.389	0.050	0.050	1.330	0.050						0.105	0.010	0.009	0.037	0.010
FSV-CB											0.074	0.003	0.002	0.018	0.002
FSV-CC															
FSV-CD											0.099	0.006	0.005	0.027	0.005
FSV-CE															
FSV-CF															
FSV-CG	0.430	0.047	0.046	1.562	0.046	0.026	0.002	0.002	0.073	0.002	0.103	0.004	0.003	0.034	0.004
FSV-CI											0.109	0.009	<0.009	-	0.010
FSV-CL											0.075	0.006	0.005	0.025	0.005
FSV-CP															
FSV-CR															
FSV-CW	0.371	0.048	0.048	1.231	0.048	*0.029*	*0.005*	*0.004*	*0.064*	*0.004*	>0.091	>0.005	>0.006	>0.029	>0.007
FSV-CZ															
FSV-DD															
FSV-DF															
FSV-DI	0.419	0.055	0.053	1.440	0.054										
FSV-DQ											0.124	0.003	0.003	0.023	0.003
FSV-DR															
FSV-DW	0.419	0.054	0.048	0.964	0.049	0.062	0.006	0.005	0.074	0.004					
FSV-EQ	0.344	0.048	0.048	1.341	0.052	0.008	*nq*	*nq*	*nq*	*nq*	0.077	0.002	0.003	0.020	0.002
FSV-ET															
FSV-FB	0.320	0.038	0.038	1.260	0.042	0.080	*nq*	*nq*	0.190	*nq*	0.080	*nq*	*nq*	0.030	0.005
N	13	13	13	13	13	10	6	6	9	6	23	16	15	22	18
Min	0.320	0.024	0.007	0.964	0.026	0.008	0.002	0.002	0.055	0.002	0.074	0.002	0.002	0.018	0.002
Median	0.395	0.048	0.048	1.330	0.048	0.029	0.003	0.003	0.073	0.003	0.099	0.005	0.004	0.030	0.005
Max	1.060	0.062	0.064	3.180	0.060	0.080	0.006	0.006	0.190	0.005	0.130	0.012	0.012	0.044	0.014
SD	0.036	0.008	0.006	0.134	0.007	0.005	0.002	0.002	0.010	0.001	0.015	0.004	0.002	0.008	0.004
CV	9	16	13	10	14	18	54	62	13	35	16	73	58	26	78
N_{past}	15	0	0	0	0	10	0	0	0	0	28	0	0	0	0
$Median_{past}$	0.382					0.031					0.096				
SD_{past}	0.034					0.010					0.020				
NISTa															
NISTb	0.426	0.050	0.054	1.509	0.054	*0.043*	*0.000*	*0.000*	*0.083*	*0.000*	0.085	0.004	0.005	0.035	0.008
N_{NIST}	2	2	2	2	2						2	1	2	2	2
Mean	0.426	0.050	0.054	1.509	0.054						0.085	0.004	0.005	0.035	0.008
S_{rep}	0.007	0.003	0.003	0.020	0.003						0.001	0.000	0.002	0.004	
S_{het}	0.003	0.001	0.001	0.065	0.002						0.001		0.000	0.007	0.002
S_{anl}															
S_{NIST}	0.007	0.003	0.003	0.068	0.004						0.002		0.002	0.008	
NAV	0.410	0.050	0.050	1.419	0.050	0.029					0.092	0.005	0.005	0.033	0.005
NAU	0.047	0.010	0.010	0.186	0.010	0.010					0.030	0.004	0.004	0.011	0.004

Lab	trans-α-Carotene 284	285	286	287	288	Total Lycopene 284	285	286	287	288	trans-Lycopene 284	285	286	287	288	Total β-Cryptoxanthin 284	285	286	287	288
FSV-BA											0.244	0.109	0.107	0.235	0.108	0.038	0.023	0.021	0.116	0.022
FSV-BB						0.425	0.196	0.196	0.422	0.187	0.204	0.092	0.091	0.189	0.088	0.033	0.018	0.018	0.104	0.018
FSV-BE																				
FSV-BF						0.429	0.189	0.211	0.449	0.179						0.027	0.020	0.018	0.105	0.019
FSV-BG						0.463	0.183	0.181	0.365	0.177	0.254	0.102	0.097	0.192	0.100	0.031	0.015	0.020	0.099	0.017
FSV-BH						0.474	0.213	0.221	0.484	0.214						0.032	0.019	0.018	0.127	0.020
FSV-BI						0.350	0.159	0.161	0.378	0.161						0.030	0.019	0.019	0.129	0.017
FSV-BJ						0.498	0.214	0.217	0.489	0.214						0.027	0.016	0.017	0.114	0.016
FSV-BK																				
FSV-BL																				
FSV-BM																				
FSV-BN						0.420	0.153	0.163	0.394	0.181	0.226	0.078	0.086	0.203	0.093	0.022	0.007	0.005	0.117	0.005
FSV-BO						0.555	0.293	0.221	0.580	0.288						0.056	0.030	0.024	0.180	0.033
FSV-BP						0.504	0.229	0.300	0.390	0.250						0.050	0.036	0.034	0.086	0.023
FSV-BQ																				
FSV-BR																				
FSV-BS						1.040	0.391	0.361	0.841	0.395	0.405	0.222	0.211	0.338	0.224					
FSV-BT						0.397	0.220	0.226	0.434	0.211	0.167	0.094	0.095	0.180	0.089	0.045	0.027	0.031	0.146	0.029
FSV-BU						0.386	0.162	0.162	0.395	0.159						0.019	0.012	0.011	0.082	0.011
FSV-BV						0.461	0.194	0.197	0.437	0.190						0.018	0.011	0.010	0.074	0.011
FSV-BW						0.373	0.178	0.186	0.408	0.175										
FSV-BX											0.180	0.084	0.085	0.167	0.083	0.025	0.018	0.018	0.092	0.018
FSV-CB						0.299	0.150	0.142	0.338	0.148						0.026	0.014	0.014	0.099	0.013
FSV-CC																				
FSV-CD						0.487	0.188	0.187	0.474	0.178						0.040	0.022	0.020	0.096	0.022
FSV-CE																				
FSV-CF																				
FSV-CG						0.427	0.182	0.182	0.461	0.179	0.226	0.097	0.098	0.229	0.097	0.033	0.018	0.019	0.104	0.018
FSV-CI																				
FSV-CL						0.377	0.191	0.181	0.460	0.179						0.029	0.019	0.018	0.141	0.019
FSV-CP																				
FSV-CR																				
FSV-CW	0.091	0.005	0.006	0.029	0.007	0.423	0.201	0.197	0.412	0.203	0.178	0.089	0.087	0.170	0.088	0.037	0.020	0.020	0.100	0.019
FSV-CZ																				
FSV-DD																				
FSV-DF																				
FSV-DI											0.300	0.133	0.133	0.307	0.133					
FSV-DQ						0.346	0.205	0.343	0.368	0.245						0.032	0.019	0.016	0.133	0.020
FSV-DR																				
FSV-DW											0.223	0.109	0.096	0.169	0.096	0.050	0.027	0.025	0.130	0.025
FSV-EQ						0.338	0.155	0.157	0.351	0.155						0.043	0.022	0.023	0.118	0.020
FSV-ET																				
FSV-FB																0.023	0.016	0.015	0.098	0.016
N	1	1	1	1	1	21	21	21	21	21	11	11	11	11	11	23	23	23	23	23
Min						0.299	0.150	0.142	0.338	0.148	0.167	0.078	0.085	0.167	0.083	0.018	0.007	0.005	0.074	0.005
Median	0.091	0.005	0.006	0.029	0.007	0.425	0.191	0.196	0.422	0.181	0.226	0.097	0.096	0.192	0.096	0.032	0.019	0.018	0.105	0.019
Max						1.040	0.391	0.361	0.841	0.395	0.405	0.222	0.211	0.338	0.224	0.056	0.036	0.034	0.180	0.033
SD						0.072	0.026	0.029	0.053	0.027	0.042	0.014	0.010	0.042	0.012	0.009	0.004	0.003	0.022	0.003
CV						17	14	15	12	15	19	14	11	22	12	29	24	17	21	17
N_{past}	0	0	0	0	0	27	0	0	0	0	13	0	0	0	0	28	0	0	0	0
$Median_{past}$						0.406					0.204					0.032				
SD_{past}						0.072					0.036					0.008				
NISTa																*nq*	*nq*	*nq*	0.127	*nq*
NISTb	0.086	0.004	0.005	0.029	0.007	0.472	0.217	0.207	0.465	0.222						0.035	0.015	0.014	0.105	0.014
N_{NIST}	2	1	2	2	2	2	2	2	2	2						2	2	2	4	2
Mean	0.086	0.004	0.005	0.029	0.007	0.472	0.217	0.207	0.465	0.222						0.035	0.015	0.014	0.116	0.013
S_{rep}	0.002	0.000	0.001	0.002		0.002	0.017	0.013	0.016	0.010						0.007	0.003	0.003	0.008	0.003
S_{het}	0.000		0.000	0.005	0.003	0.000	0.023	0.007	0.021	0.033						0.004	0.002	0.001	0.013	0.003
S_{anl}																			0.016	
S_{NIST}	0.002		0.002	0.005		0.002	0.028	0.015	0.026	0.034						0.008	0.004	0.003	0.022	0.004
NAV						0.448	0.203	0.203	0.441	0.203	0.226	0.096	0.096	0.192	0.096	0.033	0.016	0.016	0.113	0.016
NAU						0.097	0.051	0.051	0.096	0.051	0.042	0.017	0.017	0.042	0.017	0.009	0.006	0.006	0.025	0.006

Round Robin LII Laboratory Results

All Results in µg/mL

Lab	Total α-Cryptoxanthin 284	285	286	287	288	Total Lutein 284	285	286	287	288
FSV-BA										
FSV-BB	0.014	0.010	0.010	0.030	0.009	0.088	0.044	0.044	0.102	0.042
FSV-BE										
FSV-BF										
FSV-BG						0.086	0.048	0.051	0.150	0.047
FSV-BH						0.068	0.036	0.032	0.087	0.036
FSV-BI						0.093	0.048	0.046	0.131	0.046
FSV-BJ						0.087	0.044	0.045	0.136	0.047
FSV-BK										
FSV-BL										
FSV-BM										
FSV-BN	0.005	0.001	*nq*	0.032	*nq*	0.121	0.059	0.073	0.190	0.069
FSV-BO						0.108	0.076	0.067	0.181	0.074
FSV-BP										
FSV-BQ										
FSV-BR										
FSV-BS						0.228	0.091	0.111	0.752	0.095
FSV-BT	0.032	0.021	0.022	0.055	0.019	0.085	0.061	0.073	0.173	0.066
FSV-BU										
FSV-BV										
FSV-BW										
FSV-BX						0.079	0.044	0.043	0.111	0.046
FSV-CB						-	0.037	0.038	0.077	0.024
FSV-CC										
FSV-CD										
FSV-CE										
FSV-CF										
FSV-CG						0.102	0.060	0.061	0.166	0.060
FSV-CI						0.070	0.038	0.037	-	0.036
FSV-CL	0.014	0.013	0.009	0.053	0.010	0.098	0.060	0.061	0.204	0.058
FSV-CP										
FSV-CR										
FSV-CW	0.017	0.011	0.011	0.030	0.010	>0.077	>0.038	>0.038	>0.085	>0.038
FSV-CZ										
FSV-DD										
FSV-DF										
FSV-DI						0.093	0.062	0.060	0.169	0.061
FSV-DQ						0.100	0.046	0.041	0.130	0.048
FSV-DR										
FSV-DW						0.085	0.046	0.042	0.101	0.045
FSV-EQ										
FSV-ET										
FSV-FB						0.049	0.029	0.027	0.069	0.024
N	5	5	4	5	4	17	18	18	17	18
Min	0.005	0.001	0.009	0.030	0.009	0.049	0.029	0.027	0.069	0.024
Median	0.014	0.011	0.010	0.032	0.010	0.088	0.047	0.046	0.136	0.047
Max	0.032	0.021	0.022	0.055	0.019	0.228	0.091	0.111	0.752	0.095
SD	0.002	0.002		0.017		0.012	0.012	0.015	0.053	0.014
CV	17	22		53		13	26	32	39	29
N_{past}	6	0	0	0	0	15	0	0	0	0
$Median_{past}$	0.018					0.083				
SD_{past}	0.007					0.015				
NISTa						0.088	0.077	*nq*	0.145	0.069
NISTb	0.024	0.013	0.014	0.056	0.013	0.078	0.043	0.040	0.095	0.039
N_{NIST}	1	1	1	1	1	4	4	2	4	4
Mean	0.024	0.013	0.014	0.056	0.013	0.083	0.060	0.040	0.120	0.043
S_{rep}	0.002	0.000	0.000	0.000	0.002	0.004	0.004	0.003	0.013	0.004
S_{het}						0.002	0.004	0.006	0.011	0.018
S_{anl}						0.007	0.024		0.035	0.037
S_{NIST}						0.008	0.025	0.007	0.039	0.041
NAV	0.019	0.012		0.044		0.085	0.047	0.047	0.128	0.047
NAU						0.017	0.030	0.030	0.054	0.030

Lab	trans-Lutein 284	285	286	287	288	Total Zeaxanthin 284	285	286	287	288
FSV-BB						0.046	0.025	0.026	0.072	0.023
FSV-BG						0.021	0.012	0.022	0.051	0.013
FSV-BH						0.022	0.016	0.016	0.075	0.015
FSV-BI						0.025	0.021	0.020	0.068	0.019
FSV-BN						0.071	0.020	0.034	0.109	0.028
FSV-BO						0.014	0.017	0.015	0.068	*nq*
FSV-BT						0.032	0.024	0.027	0.058	0.026
FSV-BX						0.018	0.018	0.016	0.060	0.019
FSV-CB						-	0.015	0.016	0.049	0.021
FSV-CG						0.027	0.019	0.019	0.048	0.021
FSV-CI						0.031	0.019	0.019	-	0.019
FSV-CW	0.077	0.038	0.038	0.085	0.038	0.022	0.021	0.020	0.062	0.019
FSV-DQ						0.024	0.019	0.023	0.084	0.021
FSV-DW						0.061	0.037	0.033	0.100	0.037
FSV-FB						0.013	0.015	0.016	0.055	0.014
N	1	1	1	1	1	14	15	15	14	14
Min						0.013	0.012	0.015	0.048	0.013
Median	0.077	0.038	0.038	0.085	0.038	0.025	0.019	0.020	0.065	0.020
Max						0.071	0.037	0.034	0.109	0.037
SD						0.008	0.004	0.006	0.014	0.003
CV						32	19	31	21	14
N_{past}	0	0	0	0	0	14	0	0	0	0
$Median_{past}$						0.023				
SD_{past}						0.007				
NISTa						*nq*	*nq*	*nq*	0.055	*nq*
NISTb	0.078	0.043	0.040	0.095	0.039	0.037	0.023	0.021	0.063	0.020
N_{NIST}	2	2	2	2	2	2	2	2	4	2
Mean	0.078	0.043	0.040	0.095	0.039	0.037	0.023	0.021	0.059	0.009
S_{rep}	0.002	0.005	0.003	0.008	0.005	0.002	0.005	0.000	0.006	0.004
S_{het}	0.002	0.005	0.006	0.013	0.006	0.000	0.004	0.007	0.009	0.013
S_{anl}	0.003	0.007	0.007	0.015	0.007				0.006	
S_{NIST}						0.002	0.006	0.007	0.012	0.014
NAV						0.031	0.019	0.019	0.062	0.019
NAU						0.012	0.011	0.011	0.018	0.011

Lab	Total Lutein & Zeaxanthin 284	285	286	287	288
FSV-BA	0.100	0.067	0.055	0.134	0.058
FSV-BB	*0.134*	*0.069*	*0.069*	*0.173*	*0.065*
FSV-BF	0.104	0.052	0.058	0.180	0.059
FSV-BG	0.097	0.054	0.063	0.175	0.052
FSV-BH	0.090	0.052	0.048	0.162	0.051
FSV-BI	*0.118*	*0.069*	*0.066*	*0.199*	*0.065*
FSV-BN	*0.192*	*0.080*	*0.107*	*0.299*	*0.097*
FSV-BO	0.122	0.092	0.083	0.249	>0.074
FSV-BP	0.113	0.088	0.078	0.143	0.070
FSV-BT	0.117	0.085	0.100	0.230	0.092
FSV-BU	0.128	0.074	0.074	0.231	0.075
FSV-BV	0.097	0.058	0.057	0.152	0.058
FSV-BX	*0.103*	*0.061*	*0.059*	*0.171*	*0.064*
FSV-CB	0.103	*0.051*	*0.054*	*0.126*	*0.045*
FSV-CD	0.101	0.051	0.050	0.135	0.053
FSV-CG	0.129	0.079	0.079	0.214	0.081
FSV-CI	*0.101*	*0.057*	*0.056*	-	*0.055*
FSV-CW	*>0.099*	*>0.059*	*>0.058*	*>0.147*	*>0.057*
FSV-DQ	0.124	0.065	0.064	0.214	0.069
FSV-DW	0.146	0.083	0.075	0.201	0.082
FSV-EQ	0.247	0.138	0.137	0.419	0.112
FSV-FB	0.062	0.044	0.043	0.124	0.038
N	21	21	21	20	20
Min	0.062	0.044	0.043	0.124	0.038
Median	0.113	0.067	0.064	0.178	0.064
Max	0.247	0.138	0.137	0.419	0.112
SD	0.021	0.019	0.016	0.051	0.016
CV	18	29	26	29	25
N_{past}	27	0	0	0	0
$Median_{past}$	0.109				
SD_{past}	0.025				
NISTa	*>0.088*	*>0.077*	*nq*	*0.200*	*>0.069*
NISTb	0.115	0.066	0.061	0.158	0.054
N_{NIST}	2	2	2	4	2
Mean	0.115	0.066	0.061	0.179	0.027
S_{rep}	0.005	0.009	0.003	0.018	0.009
S_{het}	0.002	0.009	0.013	0.018	0.038
S_{anl}				0.030	
S_{NIST}	0.005	0.013	0.013	0.039	0.039
NAV	0.115	0.059	0.059	0.179	0.059
NAU	0.024	0.031	0.031	0.048	0.031

Lab	Coenzyme Q10 284	285	286	287	288	Phylloquinone (K1) x1000 284	285	286	287	288
FSV-BE						0.68	0.14	0.30	0.63	0.19
FSV-CI						0.81	0.11	0.13	0.61	0.13
FSV-CW	1.200	0.700	0.600	1.300	0.600					
FSV-CZ	0.780	0.510	0.500	1.000	0.420					
FSV-DI	1.010	0.498	0.506	1.090	0.503	0.79	0.18	0.16	0.64	0.17
N	3	3	3	3	3	3	3	3	3	3
Min	0.780	0.498	0.500	1.000	0.420	0.68	0.11	0.13	0.61	0.13
Median	1.010	0.510	0.506	1.090	0.503	0.79	0.14	0.16	0.63	0.17
Max	1.200	0.700	0.600	1.300	0.600	0.81	0.18	0.30	0.64	0.19
N_{past}	0	0	0	0	0	0	0	0	0	0

C4

Round Robin LII Laboratory Results

All Results in µg/mL

Analytes Reported By One Laboratory

Analyte	Code	284	285	286	287	288
trans-β-Cryptoxanthin	NISTb	0.028	0.014	0.014	0.098	0.05
trans-Zeaxanthin	NISTb	0.037	0.023	0.021	0.063	0.02
Total Carotenoids	FSV-BT	1.116	0.433	0.474	2.477	0.439
Phytofluene	FSV-CL	0.03	0.02	0.02	*nq*	0.020
Phytoene	FSV-CL	0.07	0.05	0.05	0.06	0.013

Legend

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

Comparability Summary

Lab	TR	aT	g/bT	bC	tbC	aC	TLy	tLy	TbX	TLu	TZ	L&Z
FSV-BA	1	1	1	1	1	1		1	1	1	1	1
FSV-BB	1	1	1	1	1	1	1	1	1	1	1	1
FSV-BE	2	1	1	1								1
FSV-BF	1	1	1	1		1	1		1			
FSV-BG	1	1	1	1		2	1	1	1			1
FSV-BH	2	1	1	1	1	1	1		1	1	1	1
FSV-BI	1	1	1	1		1	1		1	1	1	1
FSV-BJ	1	1	1	1		1	1		1	1	1	1
FSV-BK	1	2										
FSV-BL	2	2								1		
FSV-BM	1	1										
FSV-BN	1	1	1	1	1	1	1	1	2			
FSV-BO	1	2		3		2	2		3	1		
FSV-BP	1	3		3		2	2		3	1	1	1
FSV-BQ	2	2										1
FSV-BR		2								2	3	3
FSV-BT	3	1	1	1	2	2	1	1	2	1	1	1
FSV-BU				1		1	1		2	1	1	1
FSV-BV	2	1	1	1		1	1		2			
FSV-BW	1	1	1	1		1	1					
FSV-BX	1	2	1		1	2		1	1	1	1	1
FSV-CB	2	1		1		1	1		1			
FSV-CC	2	1										
FSV-CD	1	1	1	1		1	1		1			1
FSV-CE	1	1		1								1
FSV-CF	2	1										1
FSV-CG	2	2	1	1	1	1	1	1	1			
FSV-CI	2	2	1	1		2						
FSV-CL	1	4	2	1		1	1		1			
FSV-CP		1	2									
FSV-CR	2	1								1	1	1
FSV-CW		1	1	1	1		1	1	1	1	1	1
FSV-CZ	1	2		4								
FSV-DD	1											
FSV-DF	1									1	3	1
FSV-DI	2	1	1		1			3				4
FSV-DQ		2	1	2		1	2		1			
FSV-DR	1	1		2						1		
FSV-DW	1	1		2	2			1	2		1	
FSV-EQ	1	1			1	1	1		1	2	1	2
FSV-ET	1	1	1	1								
FSV-FB	1	2		1	2	1			1			
NISTa	1	1	1	2						1		
NISTb	1	1	1	1	1	1	1		1	1	1	1
n	39	41	24	30	13	24	21	10	24	19	16	22

	TR	aT	g/bT	bC	tbC	aC	TLy	tLy	TbX	TLu	TZ	L&Z
% 1	67	68	92	77	77	75	86	90	71	89	88	86
% 2	31	27	8	13	23	25	14	0	21	11	0	5
% 3	3	2	0	7	0	0	0	10	8	0	13	5
% 4	0	2	0	3	0	0	0	0	0	0	0	5

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
tLy	trans-Lycopene
TbX	Total β-Cryptoxanthin
TLu	Total Lutein
TZ	Total Zeaxanthin
L&Z	Total Lutein & Zeaxanthin

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

"Comparability Score"

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

We define CS as follows:

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

$$\text{C} = \text{Concordance} = \frac{\sum_{i=1}^{N_{you}} \frac{\text{You}_i - \text{Median}_i}{\text{NAU}_i}}{N_{you}}$$

$$\text{AP} = \text{Apparent Precision} = \sqrt{\frac{\sum_{i=1}^{N_{you}} \left(\frac{\text{You}_i - \text{Median}_i}{\text{NAU}_i}\right)^2}{N_{you}-1}}$$

NAU = NIST Assigned Uncertainty

For further details, please see

Duewer DL, Kline MC, Sharpless KE, Brown Thomas J, 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 RR52

Each participant in RR52 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 RR52:

- 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 α-Cryptoxanthin
- Total Lutein
- Total Zeaxanthin
- Total Lutein & Zeaxanthin

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

Individualized Round Robin LII Report: FSV-BA

Summary

	Serum 284			Serum 285			Serum 286			Serum 287			Serum 288		
Analyte	You	NAV	n	You	NAV	n	You	NAV	n	You	NAV	n	You	NAV	n
Total Retinol	0.490	0.487	38	0.640	0.647	36	0.538	0.624	39	0.609	0.618	38	0.323	0.340	38
Retinyl Palmitate	0.07	0.08	12	0.0	0.0	9	0.0	0.0	9	0.03	0.03	9	0.02	0.01	9
α-Tocopherol	16.97	16.82	39	2.90	2.97	38	2.95	2.97	39	13.41	12.96	39	2.90	2.97	39
γ/β-Tocopherol	1.634	1.581	22	0.776	0.788	22	0.755	0.788	22	2.240	2.222	22	0.769	0.788	22
Total β-Carotene	0.381	0.437	29	0.046	0.054	28	0.044	0.054	28	1.276	1.458	27	0.045	0.054	28
trans-β-Carotene	0.356	0.410	13	0.044	0.050	12	0.042	0.050	12	1.221	1.419	13	0.043	0.050	12
Total cis-β-Carotene	0.025	0.029	10	0.002		6	0.002		6	0.055		9	0.002		6
Total α-Carotene	0.085	0.092	23	0.005	0.005	16	0.004	0.005	15	0.029	0.033	22	0.005	0.005	18
trans-Lycopene	0.244	0.226	11	0.109	0.096	11	0.107	0.096	11	0.235	0.192	11	0.108	0.096	11
Total β-Cryptoxanthin	0.038	0.033	23	0.023	0.016	23	0.021	0.016	23	0.116	0.113	23	0.022	0.016	23
Total Lutein&Zeaxanthin	0.100	0.115	21	0.067	0.059	21	0.055	0.059	21	0.134	0.179	20	0.058	0.059	20

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

D2

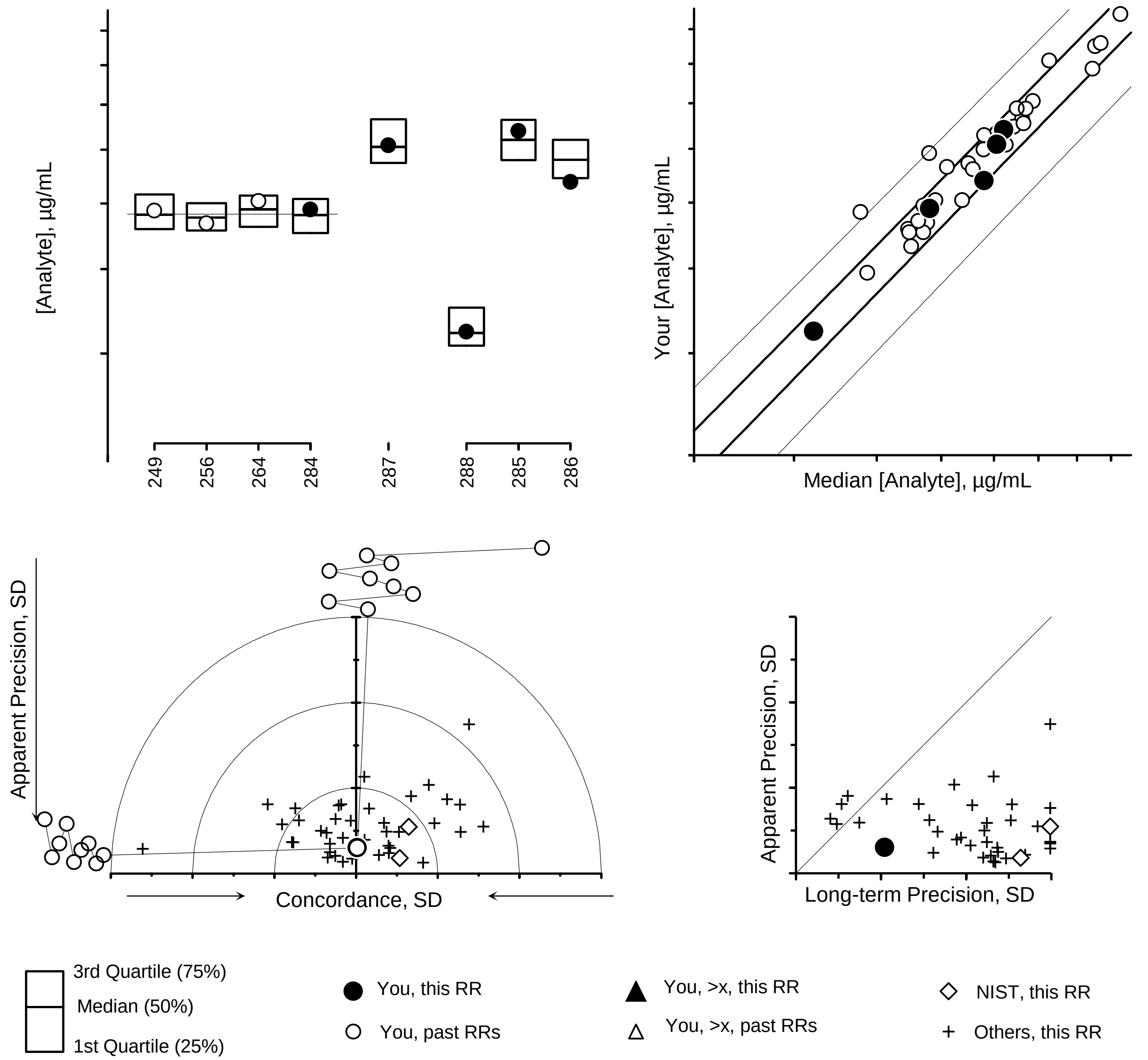
Please check our records against your records. Send corrections and/or updates to...

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 LII Report: FSV-BA

Total Retinol

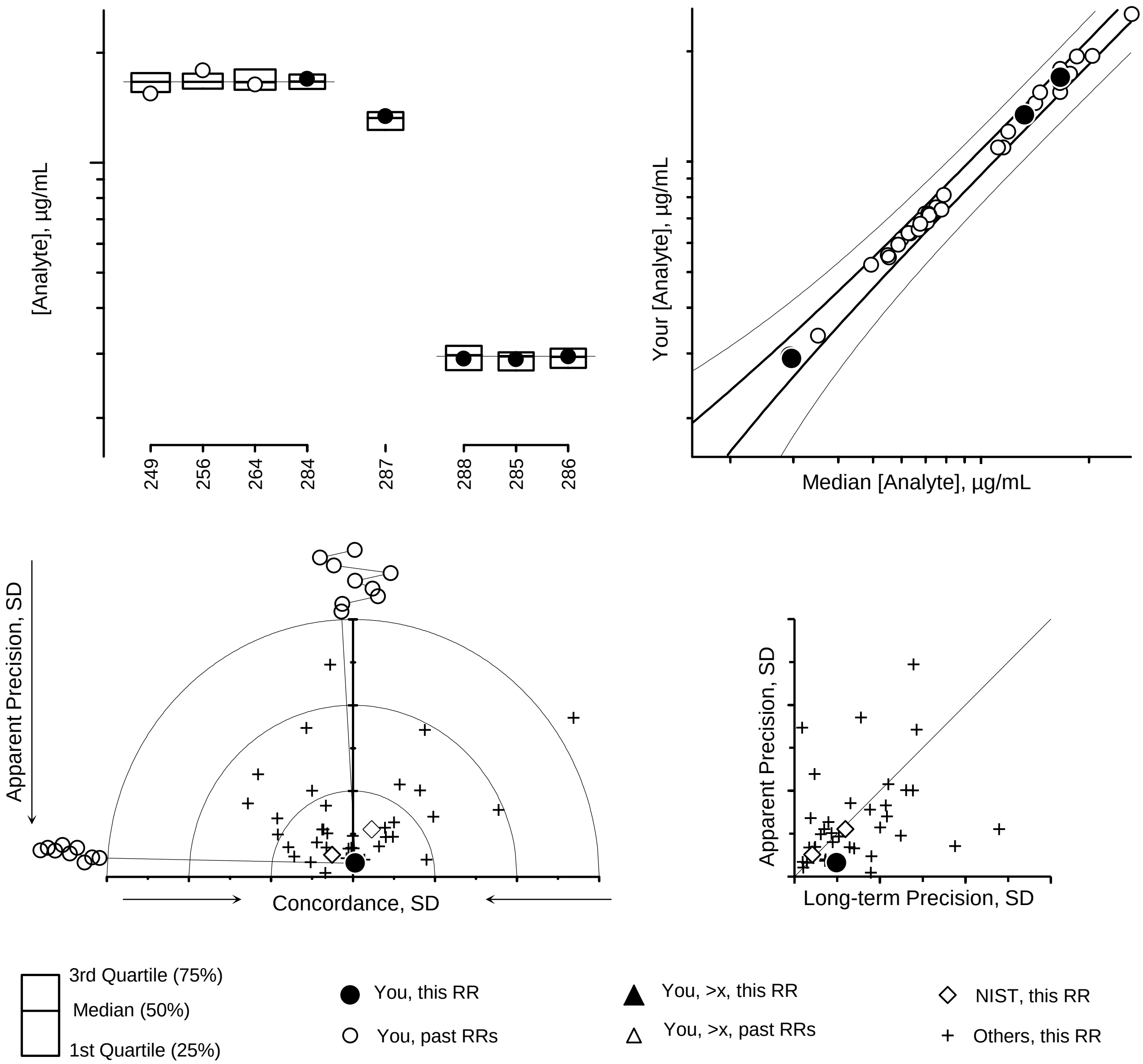


For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

Serum	History	Comments
#284	#249 RR44, #256 RR46, #264 RR48	SRM 968c Level II
#285	New, fresh-frozen	Same pool as #288 & ~0.3 µg/mL trans-retinol
#286	New, fresh-frozen	Same pool as #288 & ~0.3 µg/mL 13-cis-retinol
#287	New, fresh-frozen	Native serum, high in many measurands
#288	New, fresh-frozen	Native serum, low in most measurands

Individualized RR LII Report: FSV-BA

α-Tocopherol



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

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#284	#249 RR44, #256 RR46, #264 RR48	SRM 968c Level II
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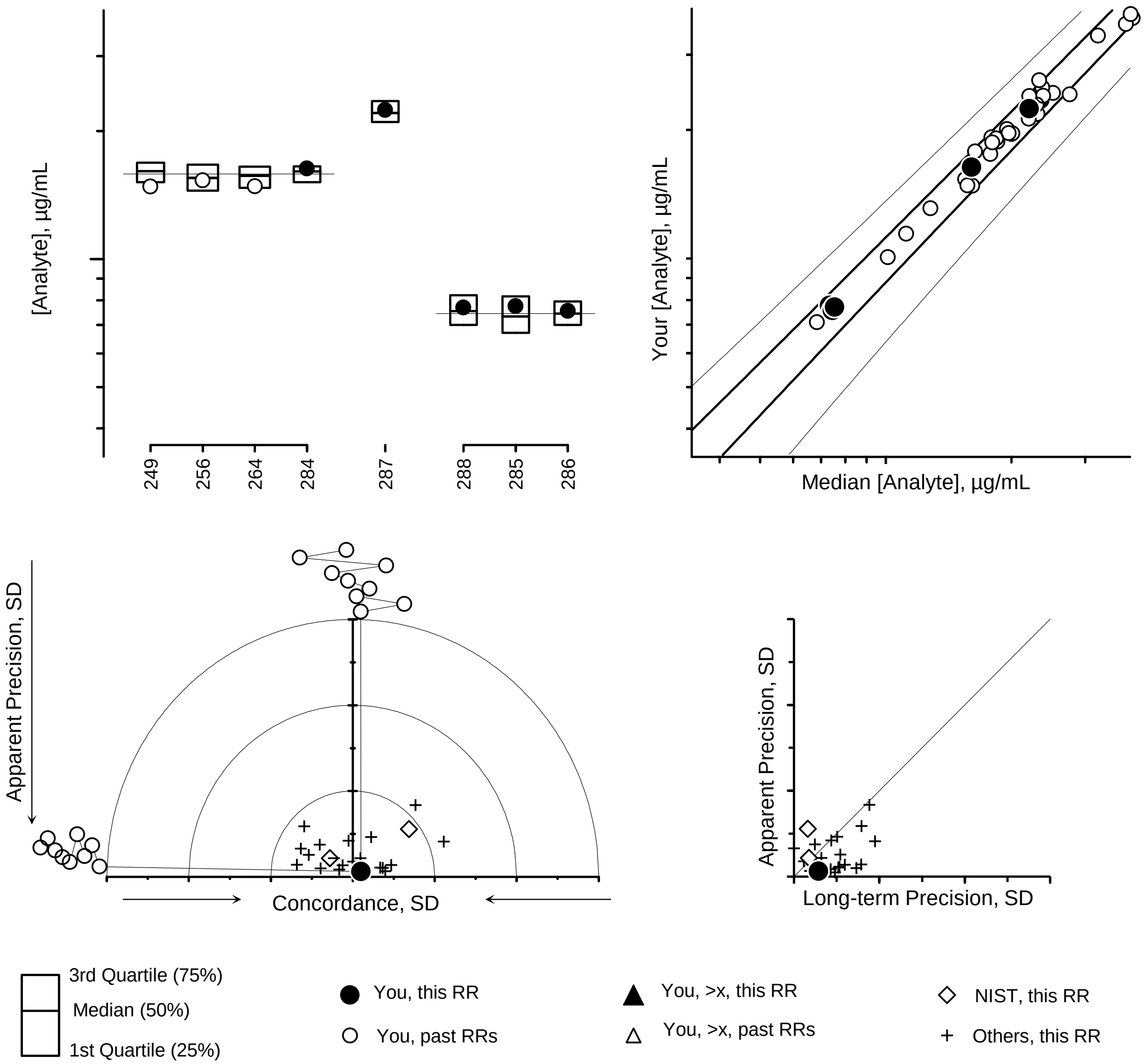
#286	New, fresh-frozen	Same pool as #288 & ~0.3 µg/mL 13-cis-retinol
#287	New, fresh-frozen	Native serum, high in many measurands
#288	New, fresh-frozen	Native serum, low in most measurands

Individualized RR LII Report: FSV-BA

γ/β-Tocopherol



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

Serum	History	Comments
#284	#249 RR44, #256 RR46, #264 RR48	SRM 968c Level II
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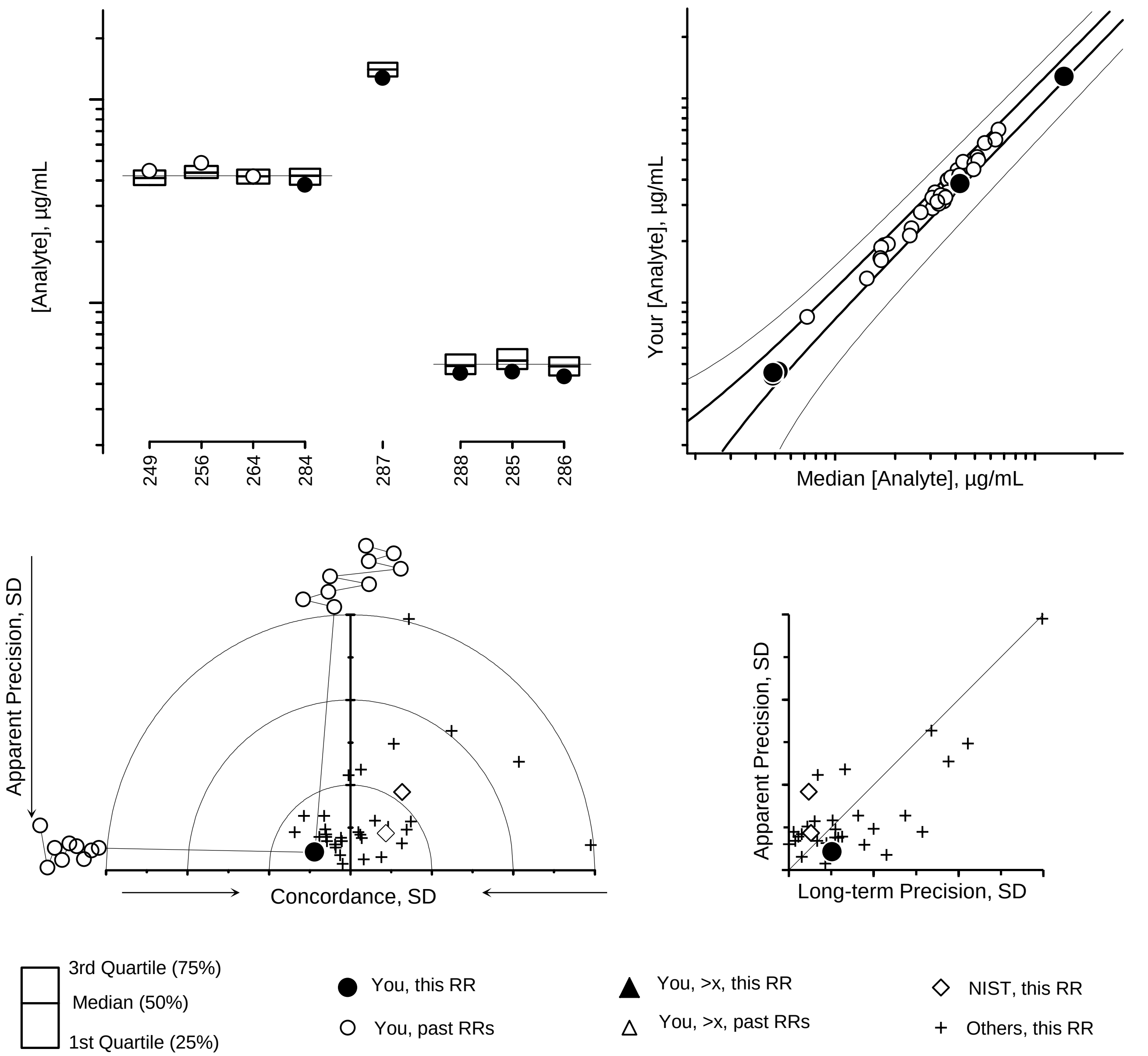
#286	New, fresh-frozen	Same pool as #288 & ~0.3 µg/mL 13-cis-retinol
#287	New, fresh-frozen	Native serum, high in many measurands
#288	New, fresh-frozen	Native serum, low in most measurands

Individualized RR LII Report: FSV-BA

Total β-Carotene



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

Serum	History	Comments
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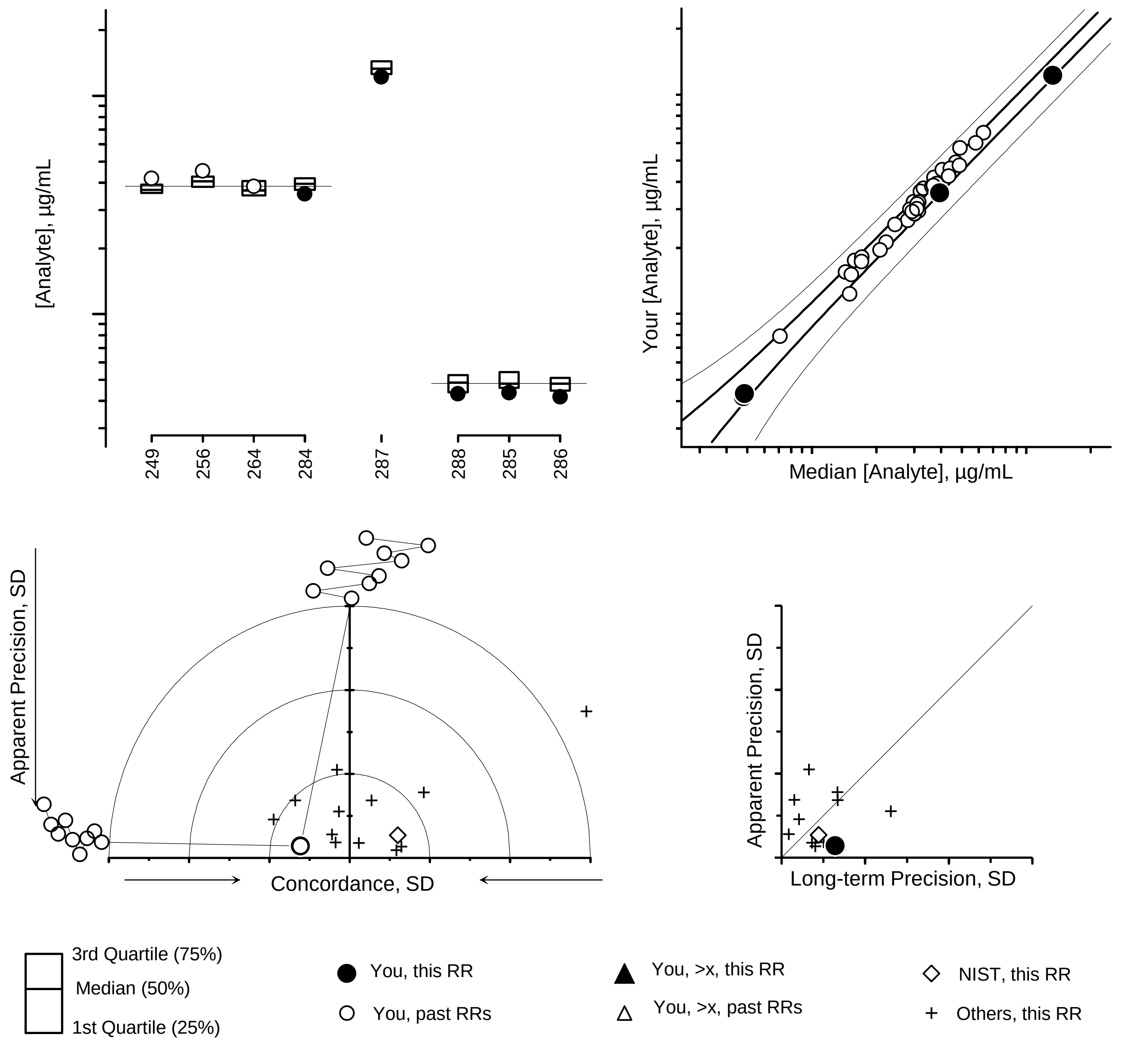
#287	New, fresh-frozen	Native serum, high in many measurands
#288	New, fresh-frozen	Native serum, low in most measurands

Individualized RR LII Report: FSV-BA

trans-β-Carotene

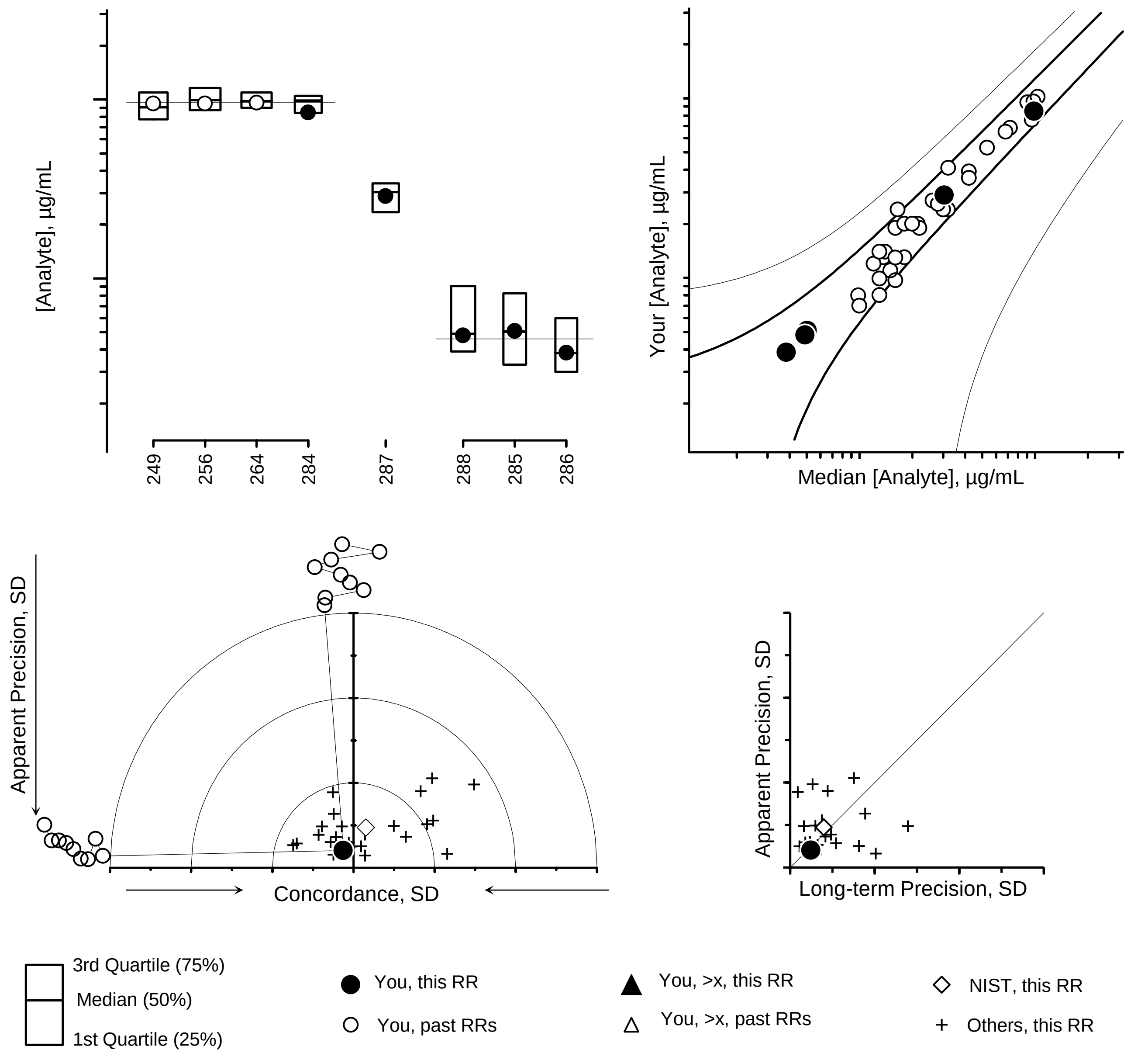


For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

Serum	History	Comments
#284	#249 RR44, #256 RR46, #264 RR48	SRM 968c Level II
#285	New, fresh-frozen	Same pool as #288 & ~0.3 µg/mL trans-retinol
#286	New, fresh-frozen	Same pool as #288 & ~0.3 µg/mL 13-cis-retinol
#287	New, fresh-frozen	Native serum, high in many measurands
#288	New, fresh-frozen	Native serum, low in most measurands

Individualized RR LII Report: FSV-BA

Total α-Carotene



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

Serum	History	Comments
#284	#249 RR44, #256 RR46, #264 RR48	SRM 968c Level II
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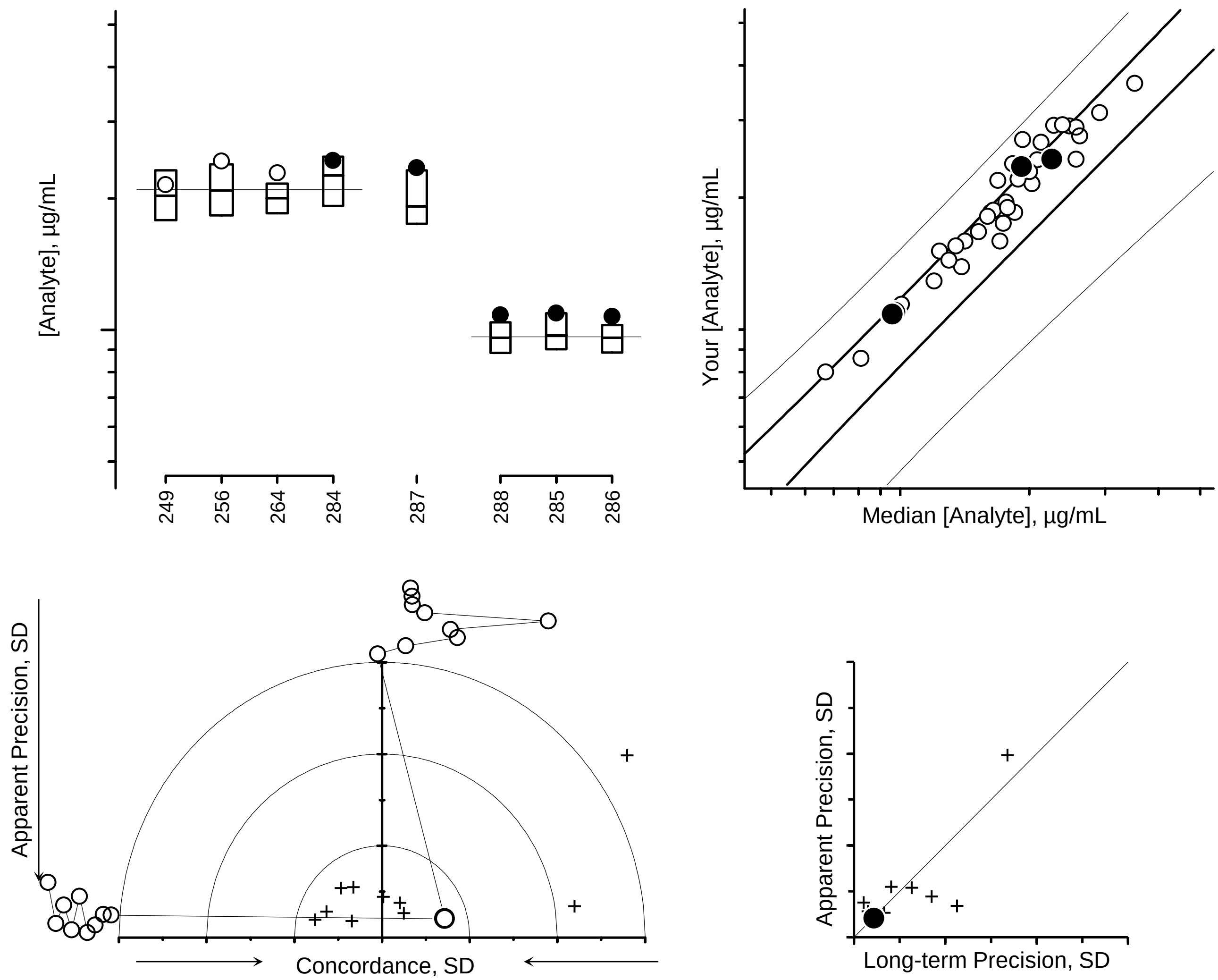
#286	New, fresh-frozen	Same pool as #288 & ~0.3 µg/mL 13-cis-retinol
#287	New, fresh-frozen	Native serum, high in many measurands
#288	New, fresh-frozen	Native serum, low in most measurands

Individualized RR LII Report: FSV-BA

trans-Lycopene



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

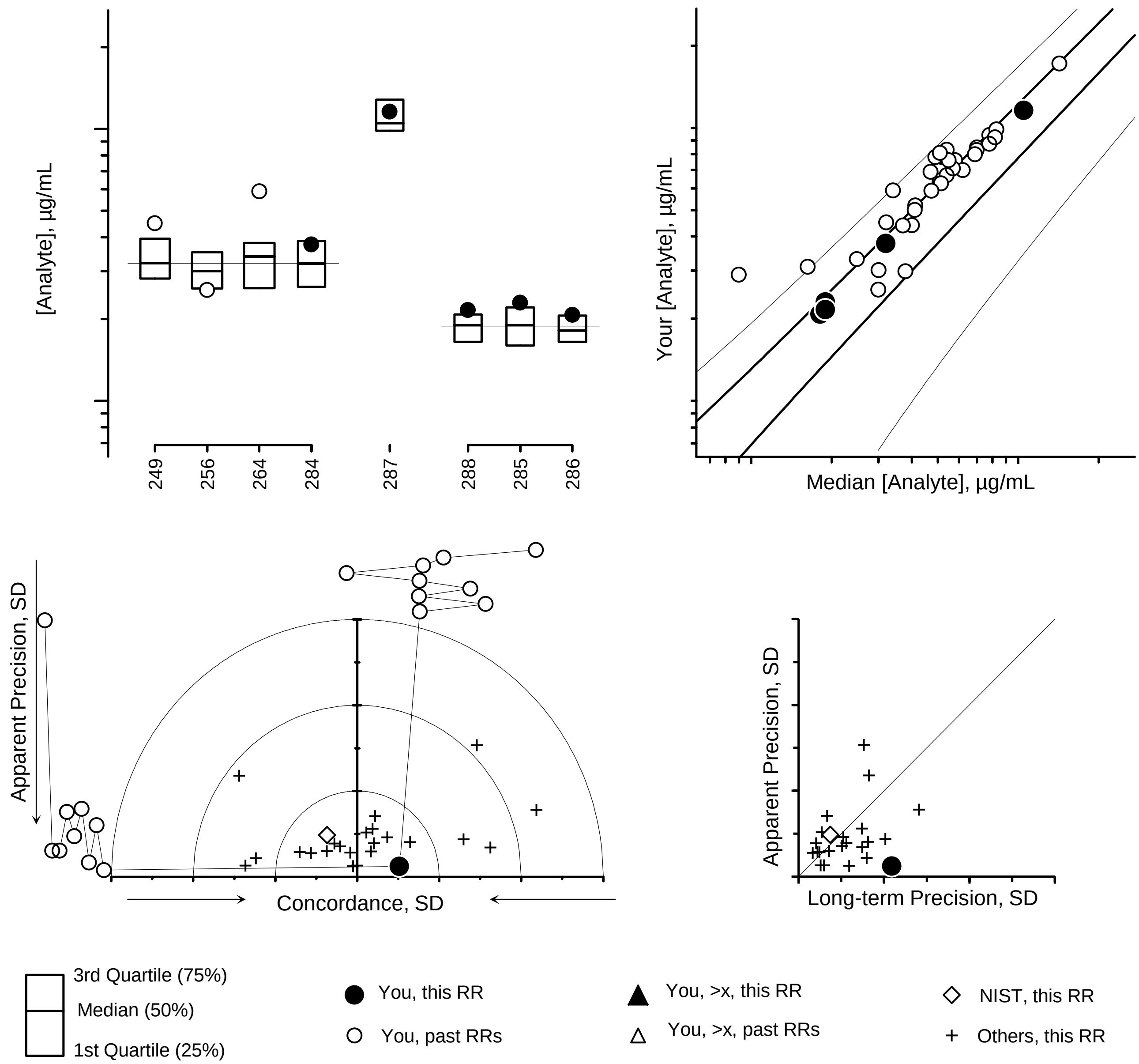
● You, this RR
○ You, past RRs
▲ You, >x, this RR
△ You, >x, past RRs
◇ NIST, this RR
\+ Others, this RR

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

Serum	History	Comments
#284	#249 RR44, #256 RR46, #264 RR48	SRM 968c Level II
#285	New, fresh-frozen	Same pool as #288 & ~0.3 µg/mL trans-retinol
#286	New, fresh-frozen	Same pool as #288 & ~0.3 µg/mL 13-cis-retinol
#287	New, fresh-frozen	Native serum, high in many measurands
#288	New, fresh-frozen	Native serum, low in most measurands

Individualized RR LII Report: FSV-BA

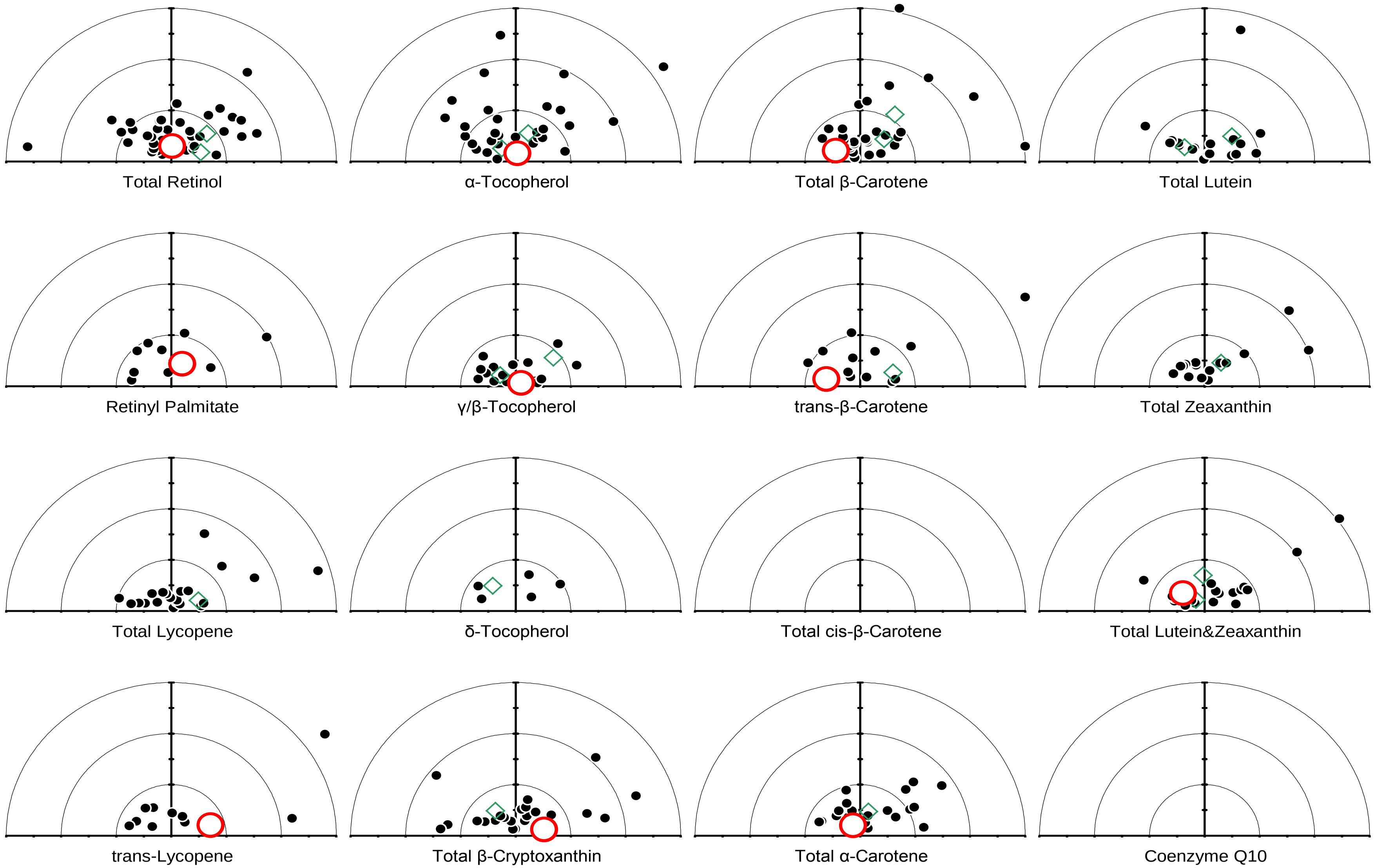
Total β-Cryptoxanthin



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

Serum	History	Comments
#284	#249 RR44, #256 RR46, #264 RR48	SRM 968c Level II
#285	New, fresh-frozen	Same pool as #288 & ~0.3 µg/mL trans-retinol
#286	New, fresh-frozen	Same pool as #288 & ~0.3 µg/mL 13-cis-retinol
#287	New, fresh-frozen	Native serum, high in many measurands
#288	New, fresh-frozen	Native serum, low in most measurands

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



Appendix E. Shipping Package Inserts for RR17

The following five items were included in each package shipped to an RR17 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

June 14, 2002

Dear Colleague:

Enclosed are samples for the second Vitamin C Round Robin 17 (RR17) of the 2002 Micronutrients Measurement Quality Assurance Program. This study is provided to you at no additional cost through financial support from the Centers for Disease Control and Prevention.

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).

RR17 consists of three vials of frozen serum (*test samples)* and one vial of solid ascorbic acid (*control sample*). Please follow the attached protocol when you prepare and analyze these samples. If you cannot prepare the control sample solutions gravimetrically, please prepare equivalent solutions volumetrically and report the exact volumes used.

Please be reminded that sample contact with any oxidant-contaminated surface (sample 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, particularly of your calibration solutions and/or control samples.

We ask that you return your results for these RR17 samples using the attached form before **September 30, 2002**. Your results will be kept confidential.

If you have any questions or concerns please contact Jeanice Brown Thomas at tel: 301-975-3120, fax: 301-977-0685, or e-mail: jbthomas@nist.gov.

Sincerely,

Jeanice Brown Thomas
Research Chemist

Sam Margolis, Ph.D.
Research Chemist

Enclosures

NIST

NIST/CDC Micronutrient Measurement Quality Assurance Program

Please Read Through Completely BEFORE Analyzing Samples

Protocol for Preparation and Analysis of the Control Sample

The *control sample* consists of a sample of solid ascorbic acid in an amber vial. It 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 0.18 to 0.22 g of the solid ascorbic acid sample 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:

   <u>*Dilute Solution* 1</u>: 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.

   <u>*Dilute Solution 2*</u>: 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.

   <u>*Dilute Solution 3*</u>: 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:

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

   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 $[\mathrm{TAA}]_{DS1}$ = (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 $[\mathrm{TAA}]_{DS2}$ = 28.4 µmol/L and 0.125 mL should weigh 0.13 g and $[\mathrm{TAA}]_{DS3}$ = 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 of the sample 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, λmax 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 you will use for the test samples, including any enzymatic treatment. Compare the replicate values. *Are you satisfied that your measurement precision is adequate?* Do **not** evaluate the test samples until you are satisfied that your system is performing properly!

7. Compare the measured with the calculated $[TAA]_{DS}$ 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** evaluate the test samples until you are satisfied that your system is performing properly!

Protocol for Analysis of the Test Samples

The *test samples* are in sealed ampoules. They were prepared by adding equal volumes of 10% metaphosphoric acid 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 *test samples* should be defrosted by warming at 20 °C for not more than 10 min otherwise some irreversible degradation may occur.

Each *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 17

NIST/CDC Micronutrient Measurement Quality Assurance Program

Preparation and Validation of Control Samples

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 **September 30, 2002** 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 17

NIST/CDC Micronutrient Measurement Quality Assurance Program

Analysis of Control 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
Test Sample #21			µmol/L of Sample
Test Sample #34			µmol/L of Sample
Test Sample #53			µmol/L of Sample

Were samples frozen upon receipt? Yes | No

Was SRM 970 used to validate your method or value-assign your in-house controls? Yes | No

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

COMMENTS:

Please return by **September 30, 2002** 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 17

NIST/CDC Micronutrients Measurement Quality Assurance Program

Packing List and Shipment Receipt Confirmation Form

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

Sample	Form
VitC #21	Liquid frozen (1:1 serum:10% MPA)
VitC #34	Liquid frozen (1:1 serum:10% MPA)
VitC #53	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 samples VitC:1, VitC:11, and VitC:51 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 four vials intact? Yes | No
If "No", which one(s) were damaged?

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

4) Did samples VitC #21, VitC #34, and VitC #53 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 RR17

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

November 20, 2002

Dear Colleague:

Enclosed is the summary report of the results for Round Robin 17 (RR17) 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.

RR17 consists of three unknowns (*test samples*) and one solid reference ascorbic acid for preparation of 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 2003 QA vitamin C round robin study (RR 18) were shipped on November 18. If you have not received these samples, please contact us immediately. The results for RR 18 are due by **March 3, 2003**.

If you have questions or concerns regarding this report, please contact me at 301-975-3120; e-mail: jbthomas@nist.gov; or fax: 301-977-0685.

Sincerely,

Jeanice Brown Thomas

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

Enclosures

NIST

The NIST/CDC M^2QAP Vitamin C Round Robin 17 (RR17) report consists of

Page	"Individualized" Report
1	Tabular summary of your reported values for the nominal 55 mmol/L solution you prepared from the solid ascorbic acid control sample and for three recently prepared test samples.
2	Graphical summary of your RR 17 sample measurements.
3	Graphical summary of your RR 17 control solution measurements.

Page	"All Lab" Report
1	Tabular summary of results and statistics for Total Ascorbic Acid [TAA] in the RR17 samples and control/calibration solutions.

Test Samples. Three unknowns were distributed in RR17.

Three unknowns (Sera 21. 34, and 53) were distributed in RR17. Each serum sample was prepared from a serum pool augmented with solid, high-purity ascorbic acid.

Qualitative Observations.

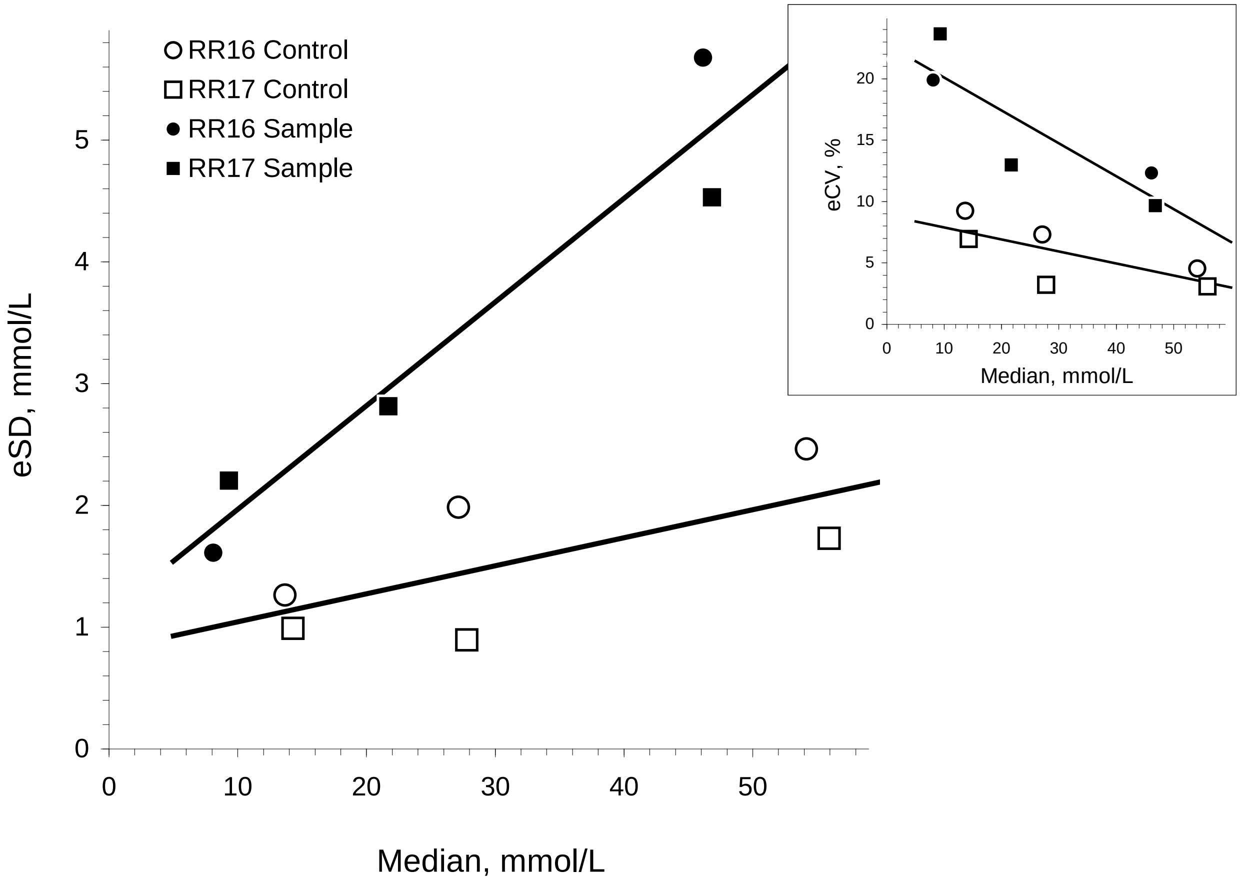
1) All participants successfully prepared the four control/calibration solutions (the three dilute solutions and the 5% metaphosphoric acid (MPA) diluent). The criteria used to evaluate this success are: the density of the 5% MPA (≈1.03 gm/mL), the observed wavelength maximum of dilute solution 1 (≈244 nm), the observed absorbance at that maximum (≈0.55 A.U.), the calculated $E^{1\%}$ #1"(≈590 dL/g·cm), and the extent of correlation between the expected and observed [TAA] for the four solutions (R^2 of 0.99 or better).

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 all participants appear to be well calibrated. The overall among-participant agreement (concordance) was only slightly improved by correcting the reported test sample results with the observed calibration parameters.

Quantitative Results

As shown in Figure 1, the interlaboratory estimated standard deviations (eSD) of both the test and control/calibration measurements appear to be linear functions of the median [TAA]. (These robust estimates of the location and dispersion of the measurement distributions are less sensitive to atypical values then are the mean and standard deviations. (See Robust Statistics - How Not to Reject Outliers 1. Basic Concepts, ANALYST 1989;114(12):1693-1697.) While the eSD is linearly related to the median, the estimated coefficient of variance (eCV, 100×eSD/median) is **not** constant (see inset) for either the test or control samples. This suggests that the observed interlaboratory measurement dispersions have both measurand-dependent and measurand-independent sources. The observed eSD for the test sample measurements are much larger than those for the control/calibration solutions. These observations will be addressed in future "Round Robin" studies.

Figure 1.



RR16 Control
RR17 Control
RR16 Sample
RR17 Sample
eSD, mmol/L
Median, mmol/L
0
1
2
3
4
5
0
10
20
30
40
50
eCV, %
Median, mmol/L
0
5
10
15
20
0
10
20
30
40
50

Appendix G. “All-Lab Report” for RR17

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

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

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

Micronutrients Measurement Quality Assurance Program for Total Ascorbic Acid
"Round Robin" 17 - September 2002

		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}	$E^{1\%}$	S17:1	S17:2	S17:3	S17:1	S17:2	S17:3
VC-MA	12/12/02	56.9	28.5	14.2	60.1	30.2	14.2	0.0	-0.32	1.06	1.000	0.5	1.030	242.	0.5521	551.0	9.5	23.4	49.4	9.3	22.3	46.8
VC-MB	01/07/02	56.9	29.7	14.8	54.5	30.0	19.5	0.0	2.41	0.93	0.989	2.9	1.031	244.	0.5457	544.0	15.0	27.3	49.5	13.5	26.7	50.7
VC-MC	01/07/02	51.5	24.7	12.9	50.7	24.9	12.4	0.0	0.02	0.99	1.000	0.4	1.031	243.	0.4847	534.7	8.4	21.4	46.5	8.5	21.6	47.0
VC-ME	30/09/02	55.1	27.8	13.5	56.0	28.8	14.3	0.7	0.75	1.00	1.000	0.1	1.030	244.	0.5651	582.6	8.9	17.2	41.7	8.1	16.3	40.7
VC-MG	11/10/02	57.2	28.4	13.9	58.6	29.4	15.2	0.0	0.43	1.02	1.000	0.5	1.030	243.6	0.544	539.5	11.7	26.6	57.9	11.1	25.7	56.4
VC-MH	11/10/02	58.8	29.4	14.6	57.8	28.8	14.4	0.0	-0.01	0.98	1.000	0.1	1.030	243.1	0.5987	578.2	9.3	21.4	45.8	9.5	21.8	46.7
VC-MI	05/09/02	60.3	30.9	15.0	62.1	32.8	16.4	0.0	0.58	1.03	1.000	0.7	1.028				13.5	23.8	51.8	12.6	22.7	49.9
VC-MO	27/09/02	57.8	27.5	13.5	56.8	27.9	13.8	1.3	1.12	0.96	1.000	0.3	1.029	243.	0.5549	545.5	10.0	22.4	44.9	9.2	22.1	45.4
VC-MQ	31/10/02	54.1	27.1	13.7	58.2	29.3	14.6	0.0	-0.06	1.08	1.000	0.1	1.029	244.	0.547	574.4	9.4	21.6	52.4	8.8	20.1	48.6
VC-MR	03/07/02	56.0	29.3	14.5	56.0	28.0	14.0	0.0	-0.44	1.00	0.999	0.8	1.030	244.	0.533	540.8	9.0	20.5	44.5	9.4	20.9	44.9
VC-MT	05/09/02	56.0	27.6	14.3	58.2	24.8	12.3	0.0	-1.79	1.05	0.994	2.3	1.028	243.5	0.5655	572.9	15.6	26.8	59.3	16.6	27.3	58.4
VC-MY	08/08/02	57.4	29.8	14.4	64.8	31.5	15.6	0.0	-0.64	1.13	0.999	1.2	1.031	244.	0.555	548.6	7.1	13.3	40.8	6.8	12.4	36.8
VC-NH	23/09/02	55.7	27.8	13.8	56.7	28.0	13.8	0.0	-0.18	1.02	1.000	0.2	1.031	242.5	0.5624	573.0	8.4	18.4	41.7	8.4	18.2	41.1
NIST	15/10/02	56.1	27.6	13.8	56.6	28.8	14.5	0.0	0.41	1.01	1.000	0.5	1.029	243.	0.5428	549.1	9.2	21.4	47.1	8.8	20.8	46.4

	Dil:1	Dil:2	Dil:3	MPA
N	13	13	13	13
Average	57.7	28.8	14.6	0.2
SD	3.5	2.3	1.8	0.4
Min	50.7	24.80	12.3	0.0
%25	56.0	27.95	13.8	0.0
Median	57.8	28.80	14.3	0.0
%75	58.6	30.00	15.2	0.0
Max	64.8	32.85	19.5	1.3
eSD	2.6	1.35	0.7	0.0
CV	4	5	5	

	g/mL	λ_{max}	A_{max}	$E^{1\%}$	S17:1	S17:2	S17:3	S17:1	S17:2	S17:3
N	13	12	12	12	13	13	13	13	13	13
Average	1.030	243.4	0.5507	557.1	10.4	21.8	48.2	10.1	21.4	47.2
SD	0.001	0.7	0.0264	17.5	2.7	4.0	5.9	2.7	4.1	6.0
Min	1.028	242.0	0.4847	534.7	7.1	13.3	40.8	6.8	12.4	36.8
%25	1.029	243.0	0.5453	543.2	8.9	20.5	44.5	8.5	20.1	44.9
Median	1.030	243.6	0.5535	549.8	9.4	21.6	46.5	9.3	21.8	46.8
%75	1.031	244.0	0.5631	573.3	11.7	23.8	51.8	11.1	22.7	49.9
Max	1.031	244.0	0.5987	582.6	15.6	27.3	59.3	16.6	27.3	58.4
eSD	0.001	0.7	0.0136	18.8	1.4	3.3	7.1	1.3	2.5	4.5
CV	0.09	0.27	2	3	15	15	15	14	11	10

Appendix H. Representative "Individualized Report" for RR17

Each participant in RR17 received an "Individualized Report" reflecting their reported results. The following two pages are the "Individualized Report" for participant "VC-MA".

Vitamin C 'Round Robin' 17 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
04/02/99	12	HPLC-EC	1.030	243.0	0.072					
09/17/01	13	HPLC-EC	1.031	244.0	0.523	572.5				
09/27/01	14	HPLC-EC	1.028	243.0	0.541	547.7	-0.9	1.03	0.999	1.09
09/18/01	15	HPLC-EC	1.027	243.0	0.547	556.5	0.0	1.04	1.000	0.05
11/18/02	16	HPLC-EC	1.032	242.0	0.575	576.5	-0.4	1.07	0.999	0.90
12/12/02	17	HPLC-EC	1.026	242.0	0.552	551.0	-0.3	1.06	1.000	0.49
		Mean	1.029	242.8	0.47	560.8				
		SD	0.002	0.8	0.19	12.9				
		CV	0.23	0.31	41.6	2.3				

RR	Sample	[TAA] mmol/Lsample Rep_1	Rep_2	F_{adj}	Mean	SD_{dup}
17	S17:1	9.9	9.1	1.0	9.5	0.6
17	S17:2	23.3	23.4	1.0	23.4	0.1
16	S16:3	49.9	44.9	1.0	47.4	3.5
17	S17:3	49.7	49.1	1.0	49.4	0.4

N	Mean	SD_{repeat}	SD_{reprod}
2	48.4	2.5	2.3

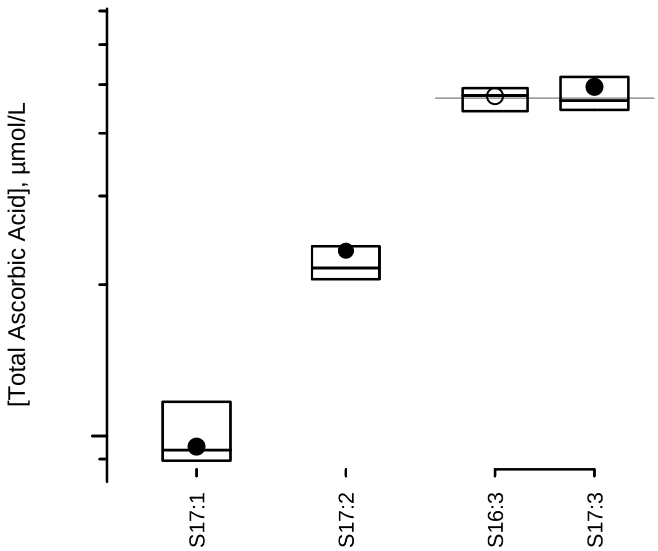
Please check our records against your records. Send corrections and/or updates to...

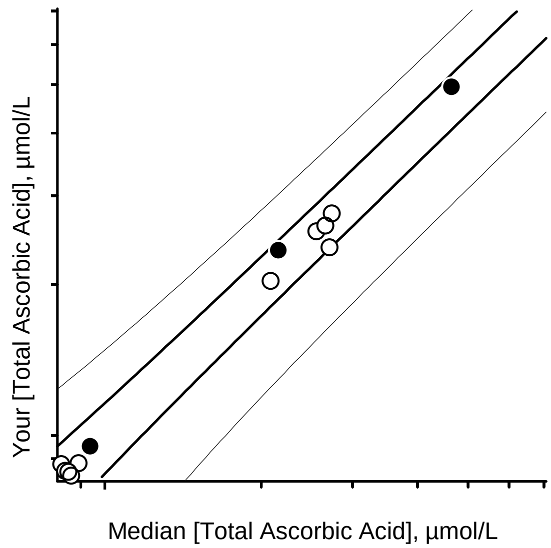
Micronutrients Measurement Quality Assurance Program
National Institute of Standards and Technology
100 Bureau Drive Stop 8392
Gaithersburg, MD 20899-8392 USA

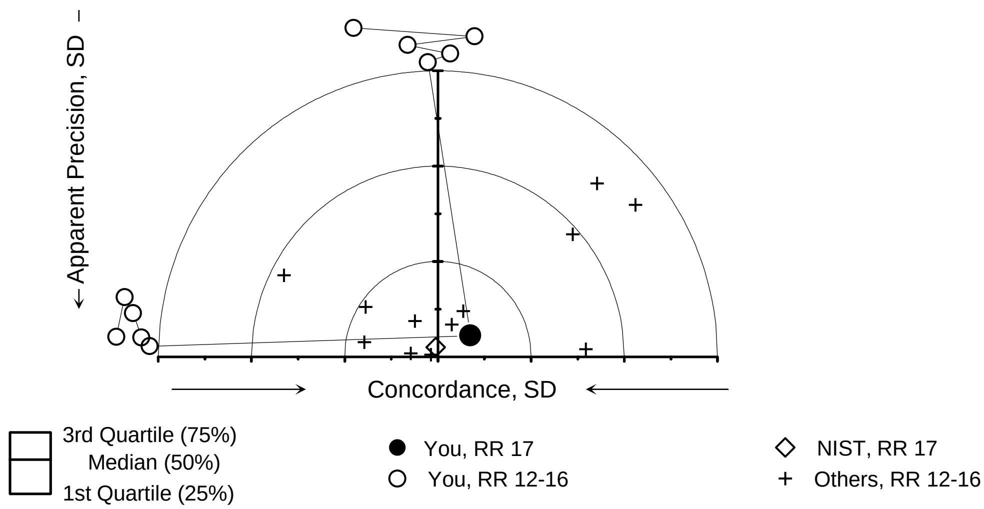
Fax: (301) 977-0685
Email: david.duewer@nist.gov

Vitamin C 'Round Robin' 17 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
S17:1	Serum 21, augmented to 9.1 µmol/L sample
S17:2	Serum 34, augmented to 23.8 µmol/L sample
S17:3	Serum 53, augmented to 47.9 µmol/L sample. Distributed as S16:3 in RR 16.