

**NISTIR 7880-13**

**NIST Micronutrients Measurement  
Quality Assurance Program  
Winter 2006  
Comparability Studies**

Results for Round Robin LIX  
Fat-Soluble Vitamins and Carotenoids in Human Serum  
and Round Robin 24 Ascorbic Acid in Human Serum

David L. Duewer  
Jeanice B. Thomas

<http://dx.doi.org/10.6028/NIST.IR.7880-13>



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U.S. Department of Commerce  
&DPHRQ) . HU, 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 2006 MMQAP measurement comparability improvement studies: 1) Round Robin LIX Fat-Soluble Vitamins and Carotenoids in Human Serum and 2) Round Robin 24 Total Ascorbic Acid in Human Serum. The materials for both studies were shipped to participants in November 2005; participants were requested to provide their measurement results by March 3, 2006.

## **Keywords**

Human Serum  
Retinol,  $\alpha$ -Tocopherol,  $\gamma$ -Tocopherol, Total and *Trans*- $\beta$ -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 LIX: Fat-Soluble Vitamins and Carotenoids in Human Serum**

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin LIX comparability study (hereafter referred to as RR59) received four lyophilized and one liquid-frozen human serum test samples for analysis. Unless multiple vials were previously requested, participants received one vial of each serum. These sera were shipped on dry ice to participants in November 2005. 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 RR59 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 24: Vitamin C in Human Serum**

Participants in the MMQAP Vitamin C in Human Serum Round Robin 24 comparability study (hereafter referred to as RR24) received four frozen serum test samples, two frozen control sera, and a solid ascorbic acid control material for analysis. Unless multiple vials were previously requested, participants received one vial of each material. These sample materials were shipped on dry ice to participants in November 2005. The communication materials included in the sample shipment are provided in Appendix E.

The test and control serum materials were prepared by adding equal volumes of 10 % metaphosphoric acid (MPA) to human serum that had been spiked with ascorbic acid. While these samples contain some dehydroascorbic acid, its content is variable. Therefore, the participants report only total ascorbic acid (TAA, ascorbic acid plus dehydroascorbic acid). Participants are also encouraged to prepare calibration solutions from the supplied solid control to enable calibrating their serum measurements to the same reference standard.

The final report delivered to every participant in RR24 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 RR59**

The following three items were included in each package shipped to an RR59 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-

November 28, 2005

Dear Colleague:

Enclosed are the samples (Sera 319 – 323) for the first fat-soluble vitamins and carotenoids in serum round robin study (Round Robin LIX) for the fiscal year (FY) 06 NIST Micronutrients Measurement Quality Assurance Program. You will find one vial of each of one liquid-frozen and four lyophilized serum samples for analysis along with a form for reporting your results. When reporting your results, please submit one value for each analyte for a given serum sample. If a value is obtained below your limit of quantification, please indicate this result on the form by using NQ (*Not Quantified*). Results are due to NIST by **March 3, 2006**. Results received more than two weeks after the due date will not be included in the summary report for this round robin study. The feedback report concerning the study will be provided in April.

Lyophilized samples should be reconstituted with 1.0 mL of HPLC-grade water or equivalent. We recommend that dissolution be facilitated with 3 to 5 min agitation in an ultrasonic bath or at least 30 min at room temperature with intermittent swirling. (CAUTION: Vigorous shaking will cause foaming and possibly interfere with accurate measurement. The rubber stopper contains phthalate esters that may leach into the sample upon intermittent contact of the liquid sample with the stopper. These esters absorb strongly in the UV region and elute near retinol in most LC systems creating analytical problems.) Pipette a known volume of serum from the vial for analysis. The final volume of the reconstituted sample is greater than 1.0 mL. **Water should not be added to the liquid-frozen sample (Serum 323).**

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);  $\alpha$ -tocopherol, 75.8 at 292 nm (ethanol);  $\gamma$ -tocopherol, 91.4 at 298 nm (ethanol);  $\alpha$ -carotene, 2800 at 444 nm (hexane);  $\beta$ -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 LIX to:

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

If you have questions or comments regarding this study, please call me at (301) 975-3120; e-mail me at [jbthomas@nist.gov](mailto:jbthomas@nist.gov); or mail/fax queries to the above address.

Sincerely,

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

Enclosures

Participant #: \_\_\_\_\_

Date: \_\_\_\_\_

**Round Robin LIX: Human Sera**  
**NIST Micronutrients Measurement Quality Assurance Program**

Analyte	319	320	321	322	323	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 <sub>2</sub> )						
ubiquinone (Qox)						
phylloquinone (K <sub>1</sub> )						
25-hydroxyvitamin D						
Other measurands?						

\* we prefer µg/mL

Was the liquid frozen sample #323 frozen when received? Yes | No

Comments:

Participant #: \_\_\_\_\_

Date: \_\_\_\_\_

**Fat-Soluble Vitamins Round Robin LIX**  
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<sup>2</sup>QAP sera

<u>Serum</u>	<u>Form</u>	<u>Reconstitute?</u>
#319	Lyophilized	Yes (1 ml H <sub>2</sub> O)
#320	Lyophilized	Yes (1 ml H <sub>2</sub> O)
#321	Lyophilized	Yes (1 ml H <sub>2</sub> O)
#322	Lyophilized	Yes (1 ml H <sub>2</sub> O)
#323	Liquid frozen	No

- Please**
- 1) Open the pack immediately
  - 2) Check that it contains all of the above samples
  - 3) Check if the ampoules and 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 sample #323 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<sup>2</sup>QAP Gang

## **Appendix B. Final Report for RR59**

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

April 10, 2006

Dear Colleague:

Enclosed is the summary report of the results for round robin LIX (RR59) of the 2006 NIST Micronutrients Measurement Quality Assurance Program (M<sup>2</sup>QAP) 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 relative to the NIST assigned values. The NIST-assigned values are equally weighted means of the medians from this interlaboratory comparison exercise and the means from the analyses performed by NIST.

Data for evaluating laboratory performance in RR59 are provided in text "Score Card" summary, page 6 of the All Lab Report. Laboratory comparability is summarized as follows: results rated 1 to 3 are within 1 to 3 standard deviations of the assigned value, respectively; those rated 4 are >3 standard deviations from the assigned value. Similar information is presented in the graphical "target plot" summary, last page of your Individualized Report.

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

Samples for the second 2006 QA interlaboratory exercise will be shipped **starting the week of May 8, 2006**. We will send you a reminder via e-mail or fax a week prior to shipment. It is critical that you carefully inspect all samples upon arrival and that you promptly confirm to us that they have arrived. We will replace samples (lost or damaged in shipment or miss-packaged by us) only for participants who report the problem within one calendar week after the package arrives.

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

Sincerely,

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

Cc: L.C. Sander  
D.L. Duewer

Enclosures

The NIST M<sup>2</sup>QAP Round Robin LVII (RR59) report consists of:

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

Page	“Individualized” Report
1	Your values, the number of labs reporting values, and our assigned values.
2 to n	“Four Plot” summaries of your current and past measurement performance, one page for each analyte you report that is also reported by at least 8 other participants.
n+1	The graphical Comparability Summary (target plot) of measurement performance.

**Samples.** The five sera below were distributed in RR59.

Serum	Description	Prior Distributions
319	Lyophilized, native, single-donor, commercially obtained serum.	#184:RR28-6/93
320	Lyophilized blend of the #311 augmented serum and stripped serum, in 1+3 ratio ( <i>i.e.</i> , measurand levels should be ¼ those of #321).	#197:RR31-6/94, #211:RR35-9/95; #245:RR43-6/98; #310:RR57-3/05
321	Lyophilized blended serum with native carotenoid levels, augmented with retinol, retinyl palmitate, and $\alpha$ - and $\gamma$ -tocopherol.	#198:RR31-6/94, #212:RR35-9/95; #246:RR43-6/98; #311:RR57-3/05
322	Lyophilized, native, single-donor, commercially obtained serum prepared in 2002. The same material was used to prepare #323.	#290:RR53-2/03, #300:RR55-3/04; #312:RR57-3/05
323	Fresh-frozen, native, single-donor, commercially obtained serum prepared in 2002. The same material was used to prepare #322.	#292:RR53-2/03, #301:RR55-3/04; #313:RR57-3/05

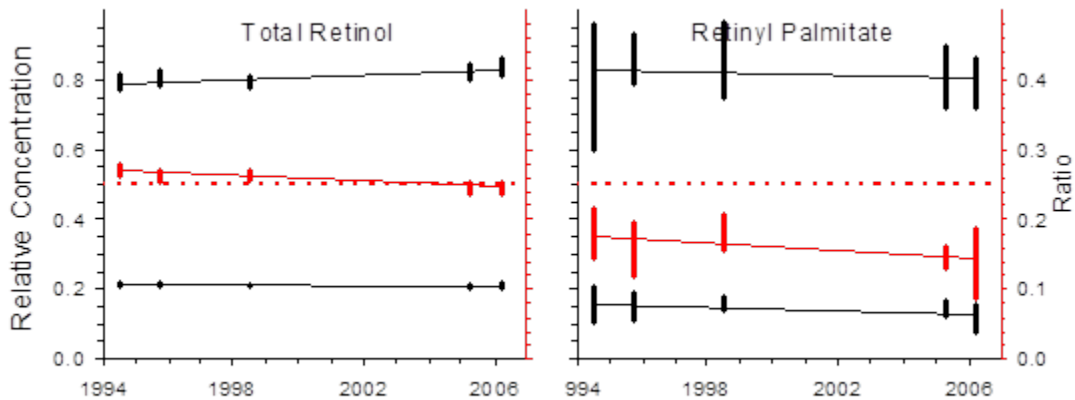
## Results

- 1) Sera Stability. There was no significant change in the median level for any measurand in any of the sera. The measurement variability for some of the very low-level measurands in #320 was marginally higher than expected. Note that #319, 320, and 321 were prepared more than 10 years ago.
- 2) Follow-up on Retinyl Palmitate Issue from RR 57: Sera #320 and #321 provide the fifth set of paired data for an experiment begun in April, 1994. The material analyzed as serum #320 was prepared from a single-source pool spiked with retinol,  $\alpha$ -tocopherol, and retinyl palmitate. The material analyzed as serum #321 was prepared as a 1:3 blend of the spiked pool and a stripped serum. These



materials were also analyzed in last year's RR57 as sera #310 and #311. At that time, we'd noted that there was considerably less retinyl palmitate in the low-level material than expected and speculated on the cause. We are in the process of re-examining all of the available data for these materials, and have at least a tentative answer: retinyl palmitate was lost when the blended material was filtered.

The following Figure shows the trends with time for total retinol and retinyl palmitate. The trend for the spiked material is represented by the upper solid line, that for the "25%-of-spike" low level material is the lower solid line, and the trend of the ratio between the two is the middle solid line. The expected value for the ratio is 0.25 (dotted line). All of the bars represent an approximate 95% confidence interval on the estimated mean values. The ratios are estimated directly from the "raw" low-level / spiked pairs, not just from the summary statistics.



The pattern for total retinol is about as expected, although the small apparent increase in the values for the spiked material is curious. However, the retinyl palmitate ratio for the low-level/spike has been considerably less than the expected 0.25 from the beginning. Simplest explanation for the difference between the patterns is that some of the hydrophobic retinyl palmitate spike was lost to filtration after blending the spiked-pool and the stripped serum. The variability in the estimates is too great to definitively judge whether the ratio actually changed over time.

We intend to provide a complete analysis of the results for other measurands in these materials within the next year. However, your values in serum #320 should be very close to 25% of those in #231 for total retinol,  $\alpha$  and  $\gamma/\beta$ -tocopherol, and  $\beta$ -carotene; they should be at least "fairly" close for all of the other measurands except retinyl palmitate.

- 3) Matrix (Lyophilized vs. Fresh-Frozen) Differences. Sera 322 and 323 were prepared from the same serum pool. Since we suggest that you reconstitute our lyophilized samples with 1.0 mL water rather than to a total volume of 1.0 mL, the measurand levels in #322 should be  $\approx 95\%$  of those in #323. The observed average ratio  $\pm 95\%$  confidence interval over all measurands with 8 or more quantitative measurements is  $0.953 \pm 0.007$ . If any of your #322/#323 ratios are much different than 0.95, you should look at your measurement system for those measurands. If your lyophilized/fresh frozen ratios are consistently much different from 0.95, you should review how you reconstitute lyophilized materials.

## **Appendix C. “All-Lab Report” for RR59**

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

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

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



# Round Robin LIX Laboratory Results

## All Results in µg/mL

Lab	δ-Tocopherol					Total β-Carotene					trans-β-Carotene					Total cis-β-Carotene					Total α-Carotene				
	319	320	321	322	323	319	320	321	322	323	319	320	321	322	323	319	320	321	322	323	319	320	321	322	323
FSV-BA	0.040	0.029	0.150	0.051	0.058	0.354	0.103	0.750	0.137	0.140	0.332	0.097	0.697	0.130	0.134	0.022	0.006	0.053	0.007	0.005	0.015	0.003	0.032	0.079	0.082
FSV-BB	0.052	0.020	0.123	0.039	0.042	0.321	0.075	0.669	0.113	0.122	0.299	0.069	0.617	0.108	0.116	0.022	0.006	0.051	0.005	0.006	0.010	0.004	0.035	0.067	0.072
FSV-BC																									
FSV-BD						0.326	0.133	0.752	0.121	0.133											0.013	0.012	0.036	0.090	0.079
FSV-BE						0.324	0.250	0.778	0.073	0.082											0.022	0.007	0.044	0.076	0.075
FSV-BF						0.369	0.135	0.803	0.154	0.156											<i>nq</i>	<i>nq</i>	<i>nq</i>	0.069	0.070
FSV-BG						0.280	0.123	0.599	0.101	0.106	0.280	0.123	0.556	0.101	0.106	<i>nq</i>	<i>nq</i>	0.043	<i>nq</i>	<i>nq</i>	0.014	0.007	0.027	0.065	0.068
FSV-BH	0.046	0.040	0.161	0.059	0.054	0.272	0.140	0.590	0.095	0.103											0.013	<i>nq</i>	0.035	0.079	0.083
FSV-BI						0.326	0.115	0.719	0.111	0.122															
FSV-BJ																									
FSV-BK																									
FSV-BL																									
FSV-BM																									
FSV-BN	0.072	0.048	0.198	0.084	0.077	0.296	0.147	0.611	0.108	0.113	0.269	0.127	0.553	0.102	0.106	0.032	0.025	0.063	0.011	0.013	0.019	0.012	0.031	0.076	0.081
FSV-BO						0.529	0.161	0.597	0.115	0.121											0.030	<i>nq</i>	0.045	0.085	0.090
FSV-BP						0.316	0.148	1.508	0.122	0.095											<i>nq</i>	<i>nq</i>	0.034	0.078	0.060
FSV-BQ																									
FSV-BR						0.444	0.250	1.200	0.174	0.183	0.294	0.113	0.556	0.107	0.111	0.025	0.014	0.061	0.011	0.010	0.017	0.007	0.027	0.078	0.080
FSV-BS						0.325	0.128	0.625	0.119	0.123											0.013	<i>nd</i>	0.033	0.062	0.077
FSV-BT						0.299	0.171	0.582	0.121	0.145											0.011	0.006	0.027	0.061	0.062
FSV-BU						0.367	0.175	0.775	0.129	0.130											<i>nq</i>	<i>nq</i>	0.030	0.112	0.121
FSV-BV						0.371	0.152	0.790	0.120	0.130															
FSV-BW																									
FSV-BX																									
FSV-CC						0.304	0.067	0.768	0.057	0.112															
FSV-CE																									
FSV-CF																									
FSV-CG	<i>nd</i>		0.212	<i>nd</i>	<i>nd</i>	0.288	0.132	0.596	0.105	0.111	0.268	0.119	0.552	0.100	0.107	0.019	0.013	0.043	0.005	0.005	0.016	0.009	0.043	0.088	0.095
FSV-CH						≥0.272	≥0.079	≥0.624	≥0.093	≥0.102	0.272	0.079	0.624	0.093	0.102						0.013	0.003	0.026	0.058	0.063
FSV-CI						0.358	0.169	0.670	0.145	0.144											0.018	0.011	0.044	0.101	0.100
FSV-CP						0.424	0.234	0.844	0.148	0.149											0.017	0.011	0.041	0.091	0.094
FSV-CS						0.353	0.155	0.830	0.135	0.134															
FSV-CT						≥0.380	≥0.182	≥0.804	≥0.107	≥0.115	0.380	0.182	0.804	0.107	0.115						0.031	0.019	0.051	0.089	0.089
FSV-CW	0.029	0.004	0.150	0.014	0.020	0.280	0.163	0.534	0.186	0.199															
FSV-CZ	<i>nq</i>					0.270	0.114	0.534	0.098	0.119															
FSV-DB						0.300	0.091	0.663	0.125	0.122											0.023	0.006	0.035	0.073	0.087
FSV-DF						0.290	0.104	0.651	0.133	0.145															
FSV-DI																									
FSV-DJ																									
FSV-DK																									
FSV-DV																									
N	6	6	8	7	7	25	25	25	24	25	8	8	8	8	8	5	5	6	5	5	17	14	19	20	20
Min	0.029	0.004	0.123	0.014	0.020	0.270	0.067	0.534	0.073	0.082	0.268	0.069	0.552	0.093	0.102	0.019	0.006	0.043	0.005	0.005	0.010	0.003	0.026	0.058	0.060
Median	0.049	0.032	0.156	0.051	0.054	0.324	0.140	0.670	0.121	0.123	0.287	0.116	0.587	0.105	0.109	0.022	0.013	0.052	0.007	0.006	0.016	0.007	0.035	0.078	0.081
Max	0.072	0.048	0.212	0.272	0.103	0.529	0.250	1.508	0.186	0.199	0.380	0.182	0.804	0.130	0.134	0.032	0.025	0.063	0.011	0.013	0.031	0.019	0.051	0.112	0.121
SD	0.009	0.012	0.025	0.021	0.016	0.046	0.036	0.133	0.019	0.023	0.027	0.023	0.065	0.005	0.007	0.002	0.006	0.010	0.004	0.004	0.004	0.004	0.008	0.015	0.013
CV	19	38	16	41	30	14	25	20	15	19	9	20	11	5	6	10	46	20	64	62	28	56	24	19	16
N <sub>pass</sub>	0	10	12	5	6	32	25	30	26	26	5	10	10	12	12	5	7	7	6	6	16	20	24	23	23
Median <sub>pass</sub>						0.036	0.140	0.072	0.066	0.353	0.150	0.681	0.111	0.120	0.019	0.015	0.052	0.006	0.007	0.015	0.009	0.035	0.072	0.078	
SD <sub>pass</sub>						0.013	0.021	0.024	0.026	0.047	0.029	0.086	0.015	0.015	0.010	0.008	0.017	0.002	0.002	0.006	0.004	0.009	0.012	0.011	
NIST						0.329	0.173	0.755	0.125	0.126															
Srep						0.008	0.002	0.014	0.002	0.001															
Shet						0.002	0.000	0.012	0.001	0.000															
SNIST						0.008	0.002	0.018	0.002	0.001															
NAV	0.049	0.032	0.156	0.051	0.054	0.326	0.156	0.713	0.123	0.125	0.287	0.116	0.587	0.105	0.109	0.022	0.013	0.052	0.007	0.006	0.016	0.007	0.035	0.078	0.081
NAU	0.017	0.016	0.031	0.021	0.018	0.047	0.042	0.146	0.020	0.024	0.031	0.023	0.065	0.013	0.014	0.008	0.006	0.021	0.004	0.004	0.006	0.004	0.011	0.023	0.024

# Round Robin LIX Laboratory Results

## All Results in µg/mL

Lab	Total Lycopene					trans-Lycopene					Total β-Cryptoxanthin					Total α-Cryptoxanthin					
	319	320	321	322	323	319	320	321	322	323	319	320	321	322	323	319	320	321	322	323	
FSV-BA	0.200	0.023	0.165	0.518	0.549	0.115	0.010	0.073	0.297	0.318	0.069	0.002	0.026	0.072	0.074	0.032	nd	0.012	0.035	0.035	
FSV-BB	0.178	0.027	0.137	0.429	0.453	0.082	0.010	0.051	0.210	0.226	0.050	nd	0.019	0.051	0.054	0.021	nd	0.006	0.022	0.023	
FSV-BC																					
FSV-BD																					
FSV-BE	0.139	0.035	0.144	0.499	0.526	0.106	0.014	0.077	0.281	0.287	0.045	0.009	0.015	0.046	0.047						
FSV-BF	0.214	0.043	0.202	0.542	0.550						0.056	0.006	0.024	0.064	0.075						
FSV-BG	0.202	0.035	0.151	0.532	0.555						0.058	nd	nd	0.061	0.064						
FSV-BH	0.157	0.027	0.125	0.375	0.397						0.054	0.004	0.018	0.048	0.051						
FSV-BI	0.194	0.024	0.156	0.552	0.580						0.049	nd	0.015	0.049	0.053						
FSV-BJ																					
FSV-BK																					
FSV-BL																					
FSV-BM	0.173	0.037	0.143	0.421	0.442	0.093	0.021	0.065	0.230	0.239	0.054	0.009	0.020	0.056	0.059	0.028	nd	0.012	0.030	0.031	
FSV-BN	0.183	nd	0.163	0.467	0.509						0.021	nd	0.018	0.018	0.018						
FSV-BO	0.161	nd	0.398	0.500	0.427						0.135	nd	0.098	0.102	0.092						
FSV-BP																					
FSV-BQ																					
FSV-BR																					
FSV-BS	0.142	0.031	0.170	0.511	0.527	0.087	0.012	0.060	0.201	0.206	0.053	0.007	0.021	0.048	0.051	0.019	0.003	0.011	0.021	0.023	
FSV-BT	0.201	0.030	0.164	0.474	0.487						0.058	nd	0.027	0.054	0.055						
FSV-BU	0.198	0.067	0.169	0.434	0.541						0.052	0.003	0.015	0.051	0.051						
FSV-BV	0.211	0.035	0.162	0.550	0.552						0.063	nd	0.015	0.060	0.063						
FSV-BW	0.190	0.015	0.153	0.520	0.580																
FSV-BX																					
FSV-C																					
FSV-CC																					
FSV-CE																					
FSV-CF																					
FSV-CG	0.190	0.029	0.152	0.469	0.507	0.110	0.012	0.073	0.286	0.313	0.071	0.005	0.027	0.075	0.081						
FSV-CH																					
FSV-CI																					
FSV-CJ																					
FSV-CK																					
FSV-CL																					
FSV-CM																					
FSV-CN																					
FSV-CO																					
FSV-CP	0.175	0.033	0.128	0.477	0.483	0.082	0.010	0.051	0.201	0.206	0.021	0.002	0.014	0.018	0.018	0.019	0.006	0.021	0.023	0.023	
FSV-CQ	0.282	0.060	0.249	0.705	0.718	0.094	0.012	0.067	0.230	0.239	0.054	0.006	0.020	0.056	0.057	0.025	0.003	0.012	0.026	0.027	
FSV-CS	0.201	nd	0.146	0.494	0.501	0.115	0.022	0.077	0.297	0.318	0.135	0.009	0.098	0.102	0.092	0.032		0.012	0.035	0.035	
FSV-CT						0.013	0.005	0.008	0.055	0.057	0.007	0.002	0.006	0.010	0.008	0.006		0.002	0.007	0.007	
FSV-CU																					
FSV-CV	14	19	13	11	11	14	40	12	24	24	12	30	32	19	14	26		15	27	25	
FSV-CW																					
FSV-CX																					
FSV-CY																					
FSV-CZ	0.183	nd	0.134	0.441	0.517						0.049	nd	0.014	0.047	0.055						
FSV-DB																					
FSV-DC																					
FSV-DD																					
FSV-DE																					
FSV-DF																					
FSV-DG																					
FSV-DH																					
FSV-DI	0.163	0.030	0.120	0.466	0.468						0.043	0.006	0.017	0.063	0.062						
FSV-DJ	0.159	0.022	0.117	0.405	0.465																
FSV-DK																					
FSV-DL																					
FSV-DM																					
FSV-DN																					
FSV-DO																					
FSV-DP																					
FSV-DQ																					
FSV-DR																					
FSV-DS																					
FSV-DT																					
FSV-DU																					
FSV-DV																					
N	22	18	22	22	22	7	7	7	7	7	21	11	19	21	21	4	1	4	4	4	
Min	0.139	0.015	0.117	0.375	0.397	0.082	0.010	0.051	0.201	0.206	0.021	0.002	0.014	0.018	0.018	0.019	0.006	0.021	0.023	0.023	
Median	0.187	0.031	0.153	0.485	0.513	0.094	0.012	0.067	0.230	0.239	0.054	0.006	0.020	0.056	0.057	0.025	0.003	0.012	0.026	0.027	
Max	0.282	0.067	0.398	0.705	0.718	0.115	0.022	0.077	0.297	0.318	0.135	0.009	0.098	0.102	0.092	0.032		0.012	0.035	0.035	
SD	0.026	0.006	0.019	0.053	0.058	0.013	0.005	0.008	0.055	0.057	0.007	0.002	0.006	0.010	0.008	0.006		0.002	0.007	0.007	
CV	14	19	13	11	11	14	40	12	24	24	12	30	32	19	14	26		15	27	25	
Npast	16	21	24	22	22	0	8	8	9	9	11	15	20	23	23	0	7	10	6	10	
Mediampast	0.183	0.029	0.133	0.477	0.504	0.019	0.074	0.252	0.270	0.270	0.065	0.007	0.021	0.051	0.054	0.005	0.012	0.023	0.027	0.027	
SDpast	0.042	0.010	0.041	0.060	0.070	0.007	0.017	0.043	0.049	0.049	0.012	0.003	0.006	0.008	0.010	0.002	0.002	0.002	0.007	0.007	
NIST																					
Step											0.042	nd	nd	0.049	0.051						
Sheet											0.003			0.002	0.004						
SNIST											0.000			0.002	0.002						
											0.003			0.003	0.005						
NAV	0.187	0.031	0.153	0.485	0.513	0.094	0.012	0.067	0.230	0.239	0.048	0.006	0.020	0.053	0.054	0.025		0.012	0.026	0.027	
NAU	0.046	0.011	0.039	0.101	0.106	0.016	0.006	0.012	0.055	0.057	0.016	0.002	0.006	0.014	0.014						

# Round Robin LIX Laboratory Results

## All Results in µg/mL

Lab	Total Lutein					Total Zeaxanthin					Total Lutein&Zeaxanthin				
	319	320	321	322	323	319	320	321	322	323	319	320	321	322	323
FSV-BA	0.089	0.012	0.065	0.085	0.089	0.024	0.004	0.022	0.041	0.043	0.109	0.022	0.081	0.114	0.119
FSV-BB											0.113	0.016	0.087	0.126	0.132
FSV-BC															
FSV-BD															
FSV-BE															
FSV-BF	0.086	0.015	0.060	0.114	0.134	0.018	0.005	0.020	0.029	0.032	0.086	0.017	0.074	0.110	0.119
FSV-BG	0.073	0.009	0.040	0.065	0.063	0.026	<i>nq</i>	0.017	0.034	0.033	0.093	0.016	0.066	0.126	0.147
FSV-BH	0.088	0.016	0.058	0.079	0.085	0.023	0.004	0.016	0.028	0.030	0.099	0.009	0.054	0.099	0.096
FSV-BI	0.063	0.014	0.046	0.075	0.082						0.111	0.020	0.074	0.107	0.115
FSV-BJ															
FSV-BK															
FSV-BL															
FSV-BM	0.084	0.030	0.063	0.079	0.082	0.024	<i>nd</i>	<i>nd</i>	0.022	0.022	0.108	0.030	0.063	0.101	0.104
FSV-BN	0.096	<i>nq</i>	0.051	0.080	0.090	0.012	<i>nq</i>	0.019	0.054	0.055	0.109	<i>nq</i>	0.071	0.134	0.144
FSV-BO											0.090	<i>nq</i>	0.162	0.110	0.103
FSV-BP															
FSV-BQ															
FSV-BR															
FSV-BS	0.066	0.024	0.050	0.074	0.082	0.014	0.006	0.011	0.021	0.033	0.096	<i>nd</i>	0.089	0.094	0.117
FSV-BT											0.071	0.026	0.054	0.087	0.110
FSV-BU											0.089	0.017	0.068	0.106	0.113
FSV-BV											0.105	0.014	0.072	0.122	0.124
FSV-BW											0.095	<i>nq</i>	0.054	0.082	0.098
FSV-CC															
FSV-CE															
FSV-CF															
FSV-CG															
FSV-CI	0.057	0.007	0.033	0.047	0.051	0.017	0.003	0.012	0.022	0.024	0.118	0.021	0.083	0.136	0.147
FSV-CP											0.074	0.010	0.045	0.069	0.075
FSV-CS	0.086	0.017	0.051	0.069	0.071	0.023	0.008	0.032	0.049	0.052	0.144	0.025	0.099	0.171	0.173
FSV-CT	0.116	<i>nq</i>	0.081	0.137	0.139	0.026	<i>nq</i>	<i>nq</i>	0.030	0.026	0.110	0.025	0.083	0.119	0.123
FSV-CW	0.087	0.021	0.062	0.082	0.082	0.019	0.008	0.021	0.029	0.030	0.142	<i>nq</i>	0.081	0.167	0.165
FSV-CZ											0.106	0.029	0.083	0.112	0.111
FSV-DB											0.084	<i>nq</i>	0.060	0.093	0.112
FSV-DF															
FSV-DI	0.065	0.011	0.039	0.060	0.064	0.052	0.004	0.016	0.031	0.024	0.122	≥0.004	0.050	0.102	0.107
FSV-DQ	0.070	<i>nd</i>	0.034	0.071	0.083										
FSV-DV															
N	14	11	14	14	14	12	8	10	12	12	22	15	22	22	22
Min	0.057	0.007	0.033	0.047	0.051	0.012	0.003	0.011	0.021	0.022	0.071	0.009	0.045	0.069	0.075
Median	0.085	0.015	0.051	0.077	0.082	0.023	0.005	0.018	0.030	0.031	0.105	0.020	0.073	0.110	0.116
Max	0.116	0.030	0.081	0.137	0.139	0.052	0.008	0.032	0.054	0.055	0.144	0.030	0.162	0.171	0.173
SD	0.015	0.006	0.015	0.009	0.011	0.005	0.002	0.004	0.007	0.007	0.015	0.007	0.016	0.019	0.017
CV	18	38	29	12	13	22	40	21	23	24	14	34	23	17	14
N <sub>past</sub>	7	11	13	15	15	0	8	10	13	13	8	18	20	23	23
Median <sub>past</sub>	0.066	0.014	0.052	0.078	0.079						0.115	0.021	0.072	0.108	0.112
SD <sub>past</sub>	0.019	0.004	0.011	0.017	0.016	0.003	0.007	0.007	0.007	0.007	0.017	0.009	0.013	0.017	0.017
NIIST	0.058	<i>nq</i>	0.043	0.068	0.067	0.020	<i>nq</i>	<i>nq</i>	0.022	0.023	0.077	<i>nq</i>	≥0.043	0.090	0.090
Strep	0.004		0.003	0.000	0.002	0.000			0.002	0.001	0.004			0.002	0.001
Sheet	0.006		0.004	0.001	0.002	0.001			0.001	0.002	0.006			0.000	0.004
SNIST	0.007		0.005	0.001	0.003	0.001			0.002	0.002	0.008			0.002	0.004
NAV	0.071	0.015	0.047	0.073	0.074	0.021	0.005	0.018	0.026	0.027	0.091	0.020	0.073	0.100	0.103
NAU	0.026	0.006	0.016	0.017	0.019	0.007	0.003	0.006	0.010	0.010	0.030	0.007	0.016	0.027	0.030

# Round Robin LIX Laboratory Results

## All Results in µg/mL

Lab	Coenzyme Q10				Ubiquinol				Ubiquinone				25-hydroxyvitamin D			
	319	320	321	322	319	320	321	322	319	320	321	322	319	320	321	322
FSV-BA																
FSV-BB																
FSV-BC																
FSV-BD																
FSV-BE																
FSV-BF																
FSV-BG																
FSV-BH																
FSV-BI																
FSV-BJ	0.607	0.140	0.528	0.675									0.0048	0.0070	0.0166	0.0158
FSV-BK				0.730												
FSV-BL																
FSV-BM																
FSV-BN																
FSV-BO																
FSV-BP																
FSV-BQ																
FSV-BR																
FSV-BS																
FSV-BT																
FSV-BU																
FSV-BV																
FSV-BW					0.136	0.014	0.099	0.100	0.018	0.012	0.026	0.037	0.0039	0.0274	0.0167	0.0176
FSV-BX																
FSV-BY																
FSV-CZ																
FSV-CC																
FSV-CE																
FSV-CF																
FSV-CG																
FSV-CH																
FSV-CI																
FSV-CJ																
FSV-CK																
FSV-CL																
FSV-CM																
FSV-CN																
FSV-CO																
FSV-CP																
FSV-CQ																
FSV-CR																
FSV-CS																
FSV-CT																
FSV-CU																
FSV-CV																
FSV-CW	0.780	0.310	0.780	0.740	0.700	0.076	0.533	0.622	0.140	0.050	0.188	0.263	0.0044	0.0033	0.0027	0.0017
FSV-CX																
FSV-CY																
FSV-DZ	0.720	0.230	0.620	0.730												
FSV-DB																
FSV-DC																
FSV-DD																
FSV-DE																
FSV-DF																
FSV-DI	0.694	0.110	0.595	0.660												
FSV-DJ																
FSV-DK																
FSV-DL																
FSV-DM																
FSV-DN																
FSV-DO																
FSV-DP																
FSV-DQ																
FSV-DR																
FSV-DS																
FSV-DT																
FSV-DU																
FSV-DV																
N	5	5	5	5	2	2	2	2	2	2	2	2	4	4	4	4
Min	0.155	0.026	0.125	0.137	0.136	0.014	0.099	0.100	0.018	0.012	0.026	0.037	0.0039	0.0033	0.0027	0.0017
Median	0.694	0.140	0.595	0.675	0.418	0.045	0.316	0.361	0.079	0.031	0.107	0.150	0.0042	0.0070	0.0167	0.0154
Max	0.780	0.310	0.780	0.740	0.700	0.076	0.533	0.622	0.140	0.050	0.188	0.263	0.0048	0.0274	0.0200	0.0210
SD	0.084	0.089	0.068	0.052	0.084	0.089	0.068	0.052	0.0004	0.0045	0.0033	0.0044	0.0004	0.0045	0.0033	0.0044
CV	12	64	11	8	12	64	11	8	9	64	20	26	9	64	20	26
Npast	0	6	7	5	0	0	0	0	0	0	0	0	0	0	0	0
Medianpast		0.090	0.530	0.707												
SDpast		0.041	0.115	0.122												
NIST																
Step																
Sheet																
SNIST																
NAV	0.694	0.140	0.595	0.675	0.694	0.140	0.595	0.675	0.694	0.140	0.595	0.675	0.694	0.140	0.595	0.675
NAU	0.084	0.089	0.068	0.068	0.084	0.089	0.068	0.068	0.084	0.089	0.068	0.068	0.084	0.089	0.068	0.068

# Round Robin LIX Laboratory Results

## All Results in µg/mL

### Analytes Reported By One Laboratory

Analyte	Code	319	320	321	322	323
Phylloquinone (K1) x1000	FSV-CI	0.140	0.210	0.740	0.260	0.260
26-Cyclolycopene-15-diol	FSV-DQ	0.066	0.015	0.041	0.058	0.072

### Legend

Term	Definition
N	Number of (non-NIST) quantitative values reported for this analyte
Min	Minimum (non-NIST) quantitative value reported
Median	Median (non-NIST) quantitative value reported
Max	Maximum (non-NIST) quantitative value reported
SD	Standard deviation for (non-NIST) results: $0.741 \times (\text{3rd Quartile} - \text{1st Quartile})$
CV	Coefficient of Variation for (non-NIST) results: $100 \times \text{SD} / \text{Median}$
$N_{\text{past}}$	Mean of N(s) from past RR(s)
$\text{Median}_{\text{past}}$	Mean of Median(s) from past RR(s)
$\text{SD}_{\text{past}}$	Pooled SD from past RR(s)
NIST	Mean of all analyses (vials x duplicates) reported by a NIST analyst
$S_{\text{rep}}$	Within-vial pooled standard deviation
$S_{\text{het}}$	Among-vial pooled standard deviation
$S_{\text{NIST}}$	Total standard deviation for NIST analyses: $(S_{\text{rep}}^2 + S_{\text{het}}^2)^{0.5}$
NAV	NIST Assigned Value = $(\text{Median} + \text{Mean}_{\text{NIST}}) / 2$ for analytes reported by NIST analyst(s) = Median for analytes reported by $\geq 10$ labs but not NIST
NAU	NIST Assigned Uncertainty: $(S^2 + S_{\text{btw}}^2)^{0.5}$ S is the maximum of $(0.05 \times \text{NAV}, \text{SD}, S_{\text{NIST}}, \text{eSD})$ and $S_{\text{btw}}$ is the standard deviation between $\text{Median}_{\text{part}}$ and $\text{Mean}_{\text{NIST}}$ . 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 at or below the limit of quantification, x
≥x	Concentration greater than or equal to x
!	Non-quantitative value: heterogeneous serum, damaged sample, malfunction, etc.



# Round Robin LIX Laboratory Results

## Comparability Summary

Lab	TR	aT	g/bT	bC	tbC	aC	TLy	TbX	TLu	TZ	L&Z	Label	Definition
FSV-BA	2	1	1	1	2	1	1	2	1	1	1	Lab	Participant code
FSV-BB	1	1	2	1	2	1	1	1	2	1	1	TR	Total Retinol
FSV-BC	1											aT	α-Tocopherol
FSV-BD	2	3									1	g/bT	γ/β-Tocopherol
FSV-BE	1	1	1	1								bC	Total β-Carotene
FSV-BF	1	2	1	2		1	1	1			1	tbC	trans-β-Carotene
FSV-BG	1	1	1	2		1	1	1	1	1	1	aC	Total α-Carotene
FSV-BH	2	1	1	1	1	1	1	1				TLy	Total Lycopene
FSV-BI	2	1	2	1		1	1	1	1	1	1	TbX	Total β-Cryptoxanthin
FSV-BJ	1	1	1	1		1	1	1				TLu	Total Lutein
FSV-BK	2	2							1			TZ	Total Zeaxanthin
FSV-BL	1	1										L&Z	Total Lutein & Zeaxanthin
FSV-BM	2	1									2		
FSV-BN	1	1	1	1	1	1	1	1			4	n	number of participants providing quantitative data
FSV-BO	2	2		3		2	1	3	2	1	1	% 1	Percent of CS = 1 (within 1 SD of medians)
FSV-BP	3	2		3		1	4	4			1	% 2	Percent of CS = 2 (within 2 SD of medians)
FSV-BQ	1	1							3	1	2	% 3	Percent of CS = 3 (within 3 SD of medians)
FSV-BR	1	2							1	3	1	% 4	Percent of CS = 4 (3 or more SD from medians)
FSV-BS	4	4	4	3			1		1	1	1		
FSV-BT	3	2	1	1	1	1	1	1					"Comparability Score"
FSV-BU	1	1	2	1		1	2	1					
FSV-BV	4	2	2	1		1	1	1			1		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 <sub>you</sub> , is at least two and the measurand has been reported by 10 or more participants.
FSV-BW	1	1	1	1		2	1	1					
FSV-CC	1	2									1		
FSV-CE	2	1		2							1		
FSV-CF	1	1									1		
FSV-CG	1	2	1	1	1	1	1	2					CS = MIN(4, INT(1 + √(C <sup>2</sup> + AP <sup>2</sup> )))
FSV-CI	1	1	1	2	1	1					1		C = Concordance = $\sum_i^{N_{you}} \frac{You_i - Median_i}{NAU_i} / N_{you}$
FSV-CP	1	2	3	1		1	1	2					
FSV-CS	1	2	3	2		1	3	1	1	2	1		
FSV-CT				1			1	1	1	3	2		
FSV-CW	2	3	1	1	3	2		1	2	1	2		C = Concordance = $\sum_i^{N_{you}} \frac{You_i - Median_i}{NAU_i} / N_{you}$
FSV-CZ	1	1		3									
FSV-DB	1	1	2	1			1	1					
FSV-DF	1								1				AP = Apparent Precision = $\sqrt{\sum_i^{N_{you}} \left( \frac{You_i - Median_i}{NAU_i} \right)^2 / (N_{you} - 1)}$
FSV-DI	1	1	1	1			1		1	1	1		
FSV-DQ		1	1	1		1	1	1					
FSV-DV	3	1											
NISTa	1	1	1	1				1	1	1	2		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.
n	36	36	23	28	8	20	22	22	15	13	23		
	TR	aT	g/bT	bC	tbC	aC	TLy	TbX	TLu	TZ	L&Z		
% 1	61	61	65	68	63	85	86	77	73	77	74		
% 2	25	31	22	18	25	15	5	14	20	8	22		
% 3	8	6	9	14	13	0	5	5	7	15	0		
% 4	6	3	4	0	0	0	5	5	0	0	4		

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 RR59

Each participant in RR59 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 RR59:

- Total Retinol
- Retinyl Palmitate
- $\alpha$ -Tocopherol
- $\gamma/\beta$ -Tocopherol
- $\delta$ -Tocopherol
- Total  $\beta$ -Carotene
- *trans*- $\beta$ -Carotene
- Total *cis*- $\beta$ -Carotene
- Total  $\alpha$ -Carotene
- Total Lycopene
- *trans*-Lycopene
- Total  $\beta$ -Cryptoxanthin
- Total Lutein
- Total Zeaxanthin
- Total Lutein & Zeaxanthin
- Coenzyme Q10

The following 14 pages are the “Individualized Report” for the analytes evaluated by participant FSV-BA.

# Individualized Round Robin LIX Report: FSV-BA

## Summary

Analyte	Serum 319			Serum 320			Serum 321			Serum 322			Serum 323		
	You	NAV	n	You	NAV	n	You	NAV	n	You	NAV	n	You	NAV	n
Total Retinol	0.375	0.376	33	0.230	0.185	33	0.878	0.744	33	0.670	0.622	32	0.705	0.653	33
Retinyl Palmitate	0.08	0.11	10	0.0	0.0	10	0.2	0.3	10	0.11	0.09	10	0.11	0.09	10
α-Tocopherol	4.35	4.28	35	1.80	3.30	35	15.14	14.34	35	9.92	9.93	34	10.46	10.19	35
γ/β-Tocopherol	1.686	1.653	22	0.486	0.683	22	2.808	2.662	22	1.824	1.766	22	1.930	1.804	22
δ-Tocopherol	0.040	0.049	6	0.029	0.032	6	0.150	0.156	8	0.051	0.051	7	0.058	0.054	7
Total β-Carotene	0.354	0.326	25	0.103	0.156	25	0.750	0.713	25	0.137	0.123	24	0.140	0.125	25
trans-β-Carotene	0.332	0.287	8	0.097	0.116	8	0.697	0.587	8	0.130	0.105	8	0.134	0.109	8
Total cis-β-Carotene	0.022	0.022	5	0.006	0.013	5	0.053	0.052	6	0.007	0.007	5	0.005	0.006	5
Total α-Carotene	0.015	0.016	17	0.003	0.007	14	0.032	0.035	19	0.079	0.078	20	0.082	0.081	20
Total Lycopene	0.200	0.187	22	0.023	0.031	18	0.165	0.153	22	0.518	0.485	22	0.549	0.513	22
trans-Lycopene	0.115	0.094	7	0.010	0.012	7	0.073	0.067	7	0.297	0.230	7	0.318	0.239	7
Total β-Cryptoxanthin	0.069	0.048	21	0.002	0.006	11	0.026	0.020	19	0.072	0.053	21	0.074	0.054	21
Total α-Cryptoxanthin	0.032	0.025	4	nd		1	0.012	0.012	4	0.035	0.026	4	0.035	0.027	4
Total Lutein&Zeaxanthin	0.109	0.091	22	0.02	0.02	15	0.08	0.07	22	0.114	0.100	22	0.12	0.10	22
25-hydroxyvitamin D	0.005	0.004	4	0.007	0.007	4	0.017	0.017	4	0.016	0.017	4	0.015	0.015	3

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

nd : Not detected

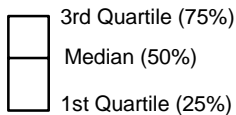
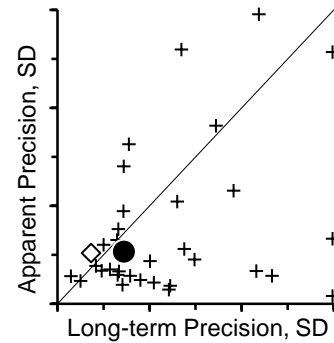
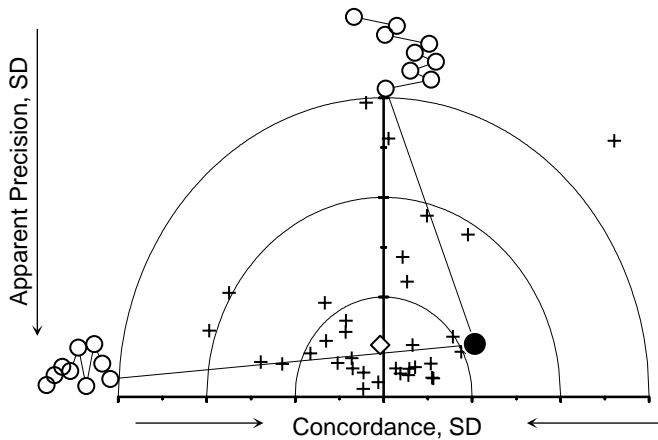
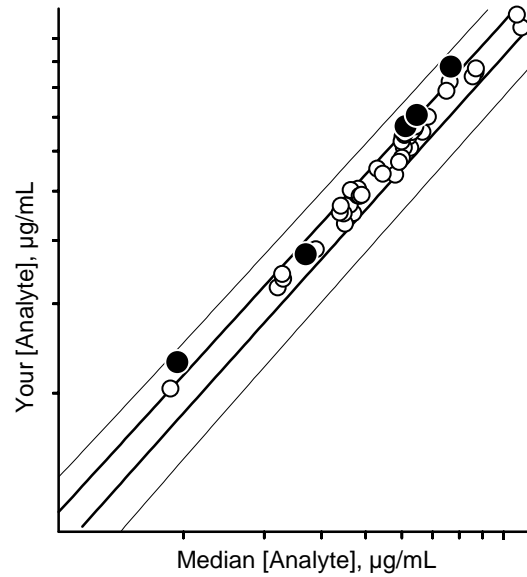
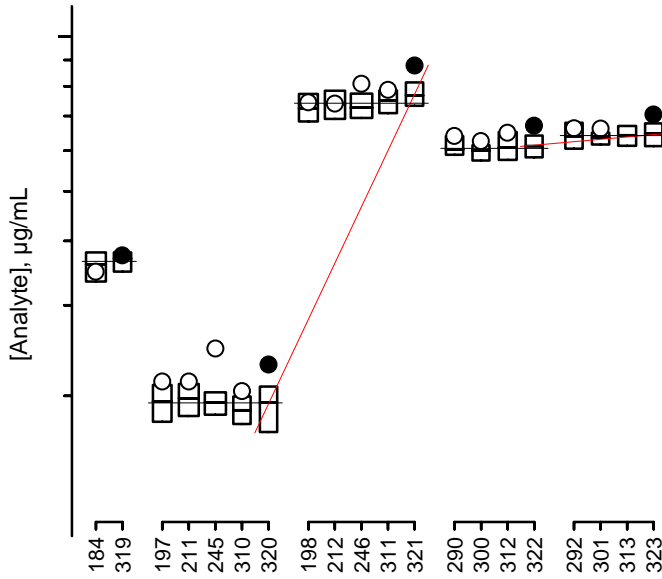
Please check our records against your records. Send corrections and/or updates to...

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 LIX Report: FSV-BA

## Total Retinol



● You, this RR  
○ You, past RRs

▲ You, ≥x, this RR  
△ You, ≥x, past RRs

◇ NIST, this RR  
+ Others, this RR

### Serum

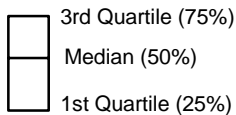
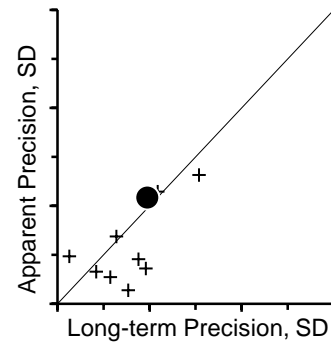
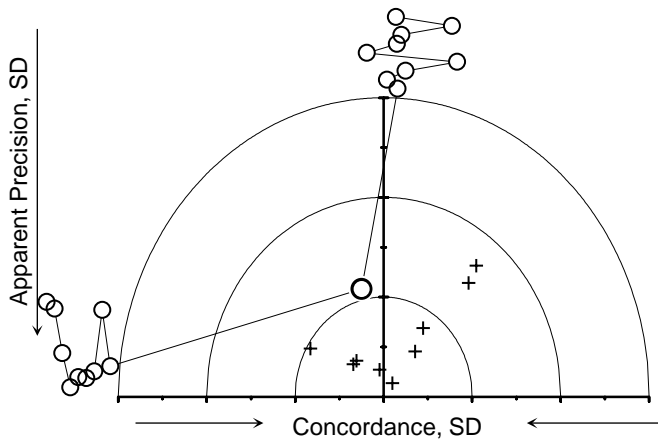
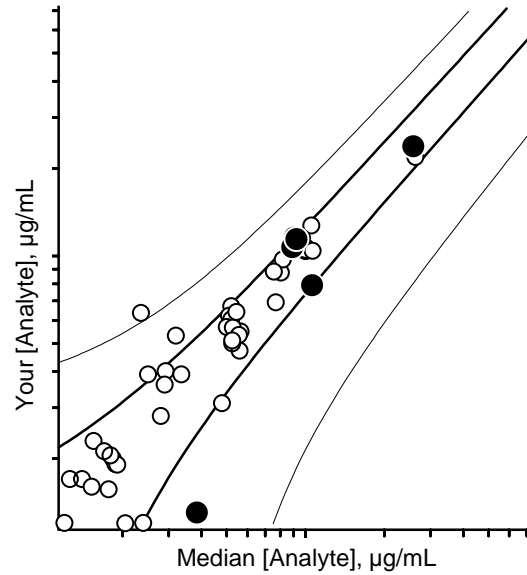
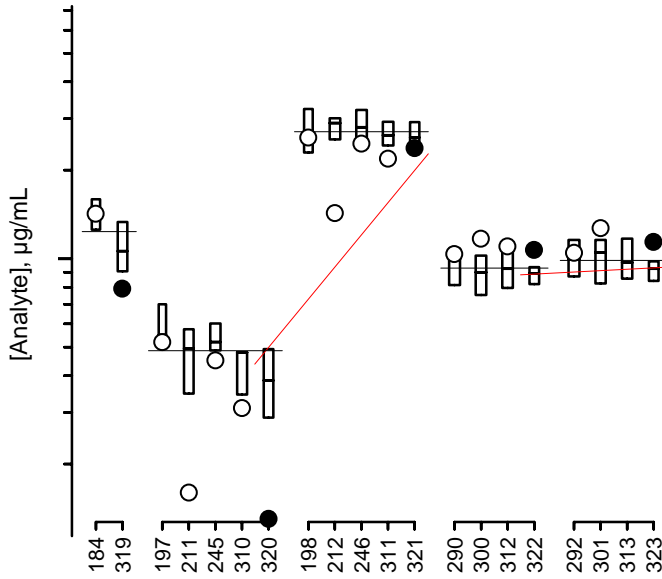
Serum	History
#319	Lyophilized - 28:184
#320	Lyophilized - 31:197, 35:211, 43:245, 57:310
#321	Lyophilized - 31:198, 35:212, 43:246, 57:311
#322	Lyophilized - 53:290, 55:300, 57:312
#323	Fresh-frozen: 53:292, 55:301, 57:313

### Comments

Native, single-source
Same as #321, 1:3 diluted with stripped serum
Augmented, multi-source
Native, single-source
Native, single-source, hemolyzed

# Individualized RR LIX Report: FSV-BA

## Retinyl Palmitate



● You, this RR  
○ You, past RRs

▲ You, ≥x, this RR  
△ You, ≥x, past RRs

◇ NIST, this RR  
+ Others, this RR

### Serum

Serum ID	History
#319	Lyophilized - 28:184
#320	Lyophilized - 31:197, 35:211, 43:245, 57:310
#321	Lyophilized - 31:198, 35:212, 43:246, 57:311
#322	Lyophilized - 53:290, 55:300, 57:312
#323	Fresh-frozen: 53:292, 55:301, 57:313

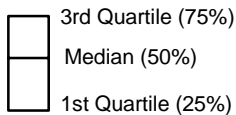
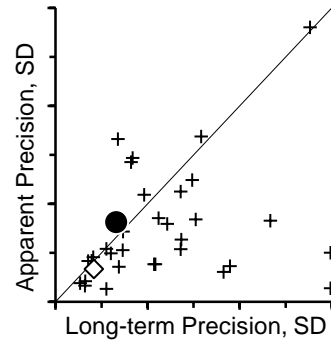
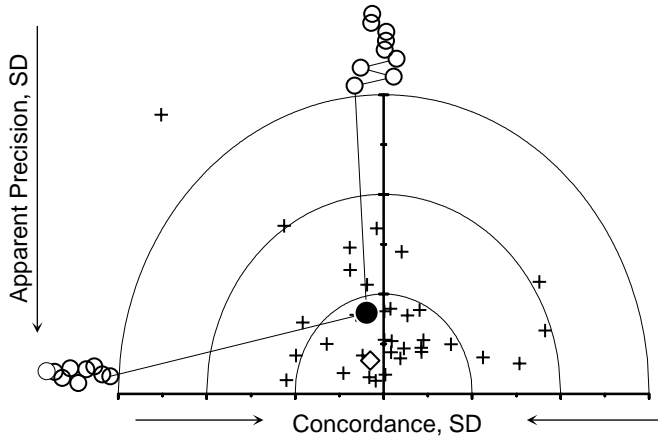
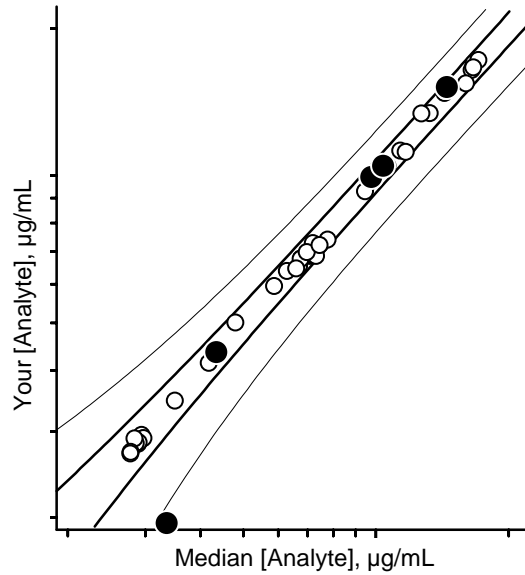
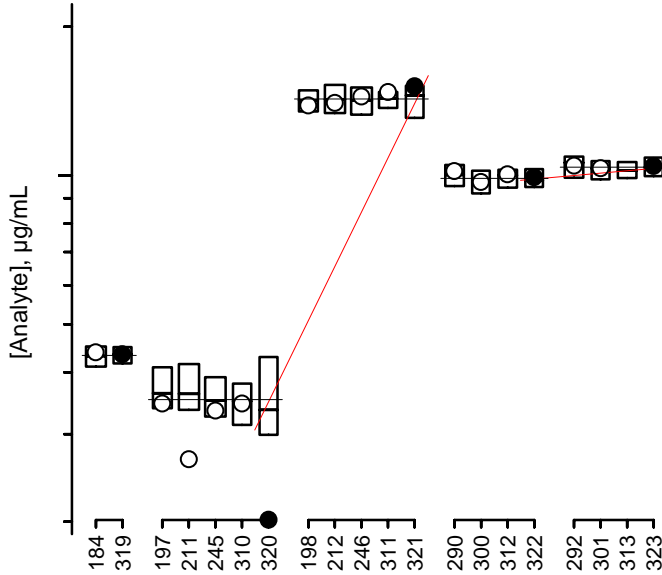
### History

### Comments

Native, single-source
Same as #321, 1:3 diluted with stripped serum
Augmented, multi-source
Native, single-source
Native, single-source, hemolyzed

# Individualized RR LIX Report: FSV-BA

## α-Tocopherol



● You, this RR  
○ You, past RRs

▲ You, ≥x, this RR  
△ You, ≥x, past RRs

◇ NIST, this RR  
+ Others, this RR

### Serum

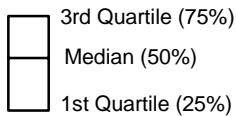
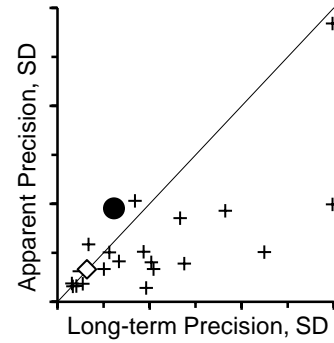
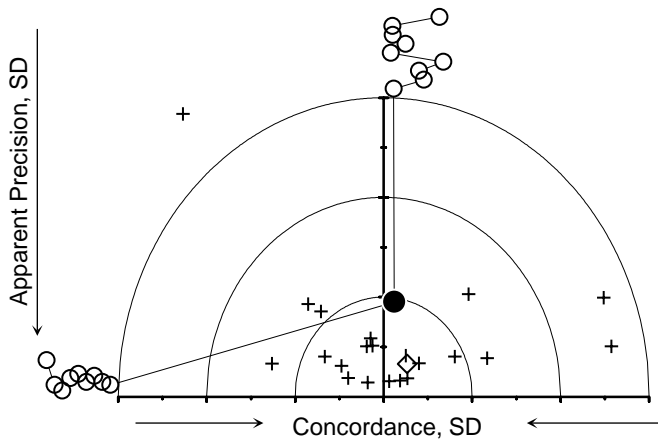
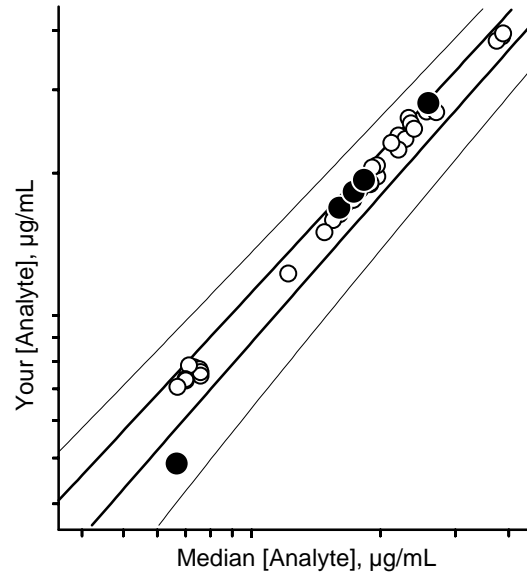
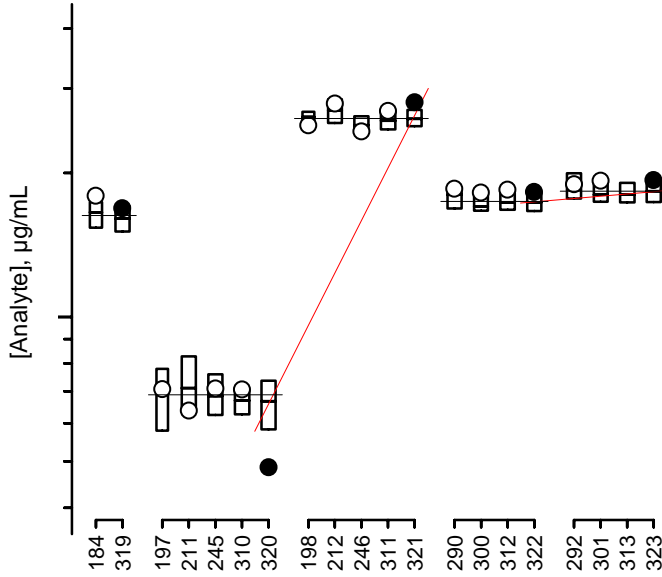
Serum	History
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#320	Lyophilized - 31:197, 35:211, 43:245, 57:310
#321	Lyophilized - 31:198, 35:212, 43:246, 57:311
#322	Lyophilized - 53:290, 55:300, 57:312
#323	Fresh-frozen: 53:292, 55:301, 57:313

### Comments

Native, single-source
Same as #321, 1:3 diluted with stripped serum
Augmented, multi-source
Native, single-source
Native, single-source, hemolyzed

# Individualized RR LIX Report: FSV-BA

$\gamma/\beta$ -Tocopherol



● You, this RR  
○ You, past RRs

▲ You,  $\geq x$ , this RR  
△ You,  $\geq x$ , past RRs

◇ NIST, this RR  
+ Others, this RR

Serum

#319	Lyophilized - 28:184
#320	Lyophilized - 31:197, 35:211, 43:245, 57:310
#321	Lyophilized - 31:198, 35:212, 43:246, 57:311
#322	Lyophilized - 53:290, 55:300, 57:312
#323	Fresh-frozen: 53:292, 55:301, 57:313

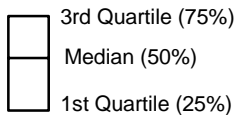
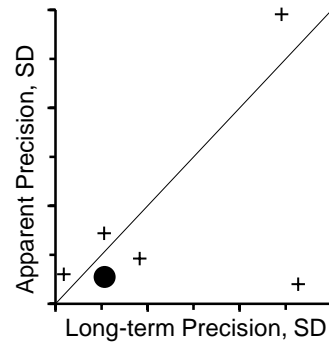
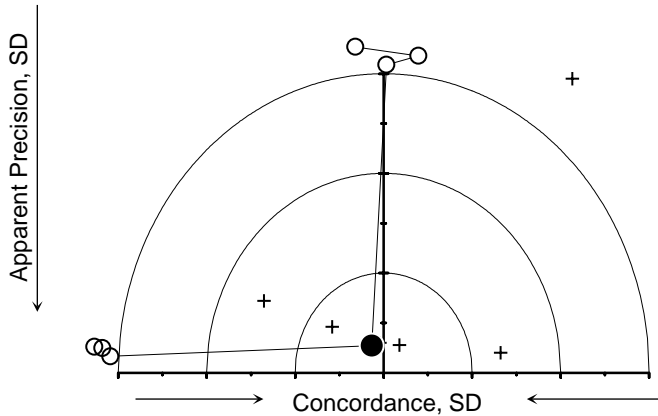
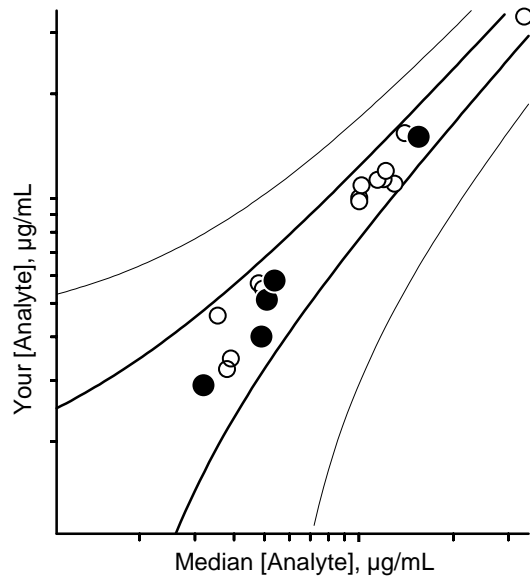
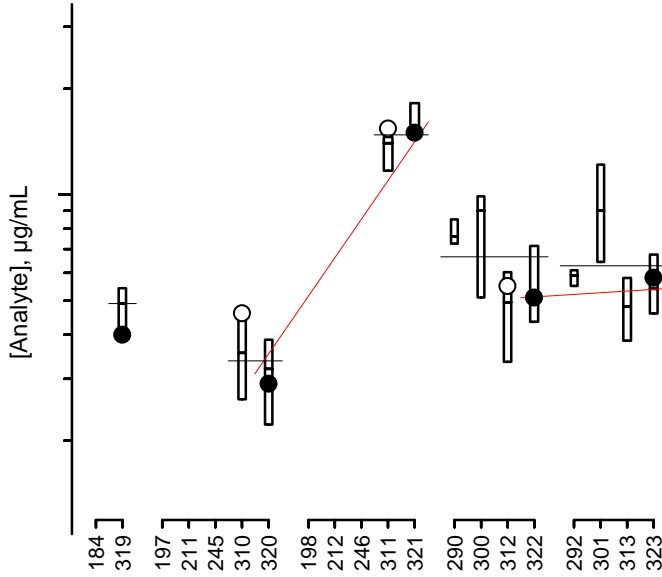
History

Comments

Native, single-source  
Same as #321, 1:3 diluted with stripped serum  
Augmented, multi-source  
Native, single-source  
Native, single-source, hemolyzed

# Individualized RR LIX Report: FSV-BA

## δ-Tocopherol



● You, this RR  
○ You, past RRs

▲ You, ≥x, this RR  
△ You, ≥x, past RRs

◇ NIST, this RR  
+ Others, this RR

### Serum

Serum ID	History
#319	Lyophilized - 28:184
#320	Lyophilized - 31:197, 35:211, 43:245, 57:310
#321	Lyophilized - 31:198, 35:212, 43:246, 57:311
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### History

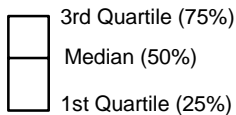
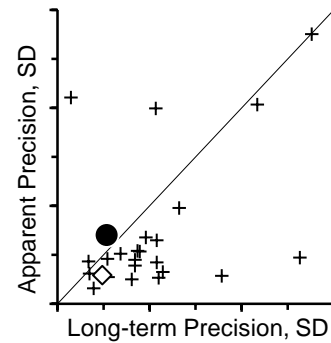
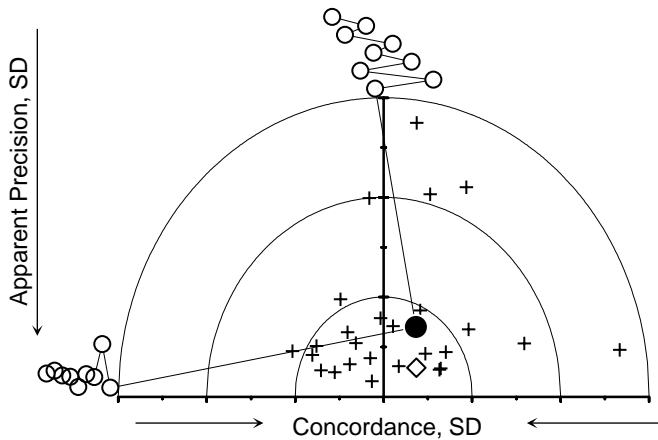
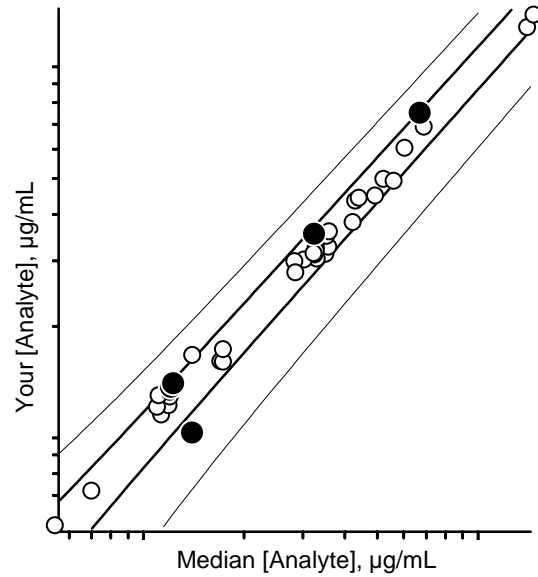
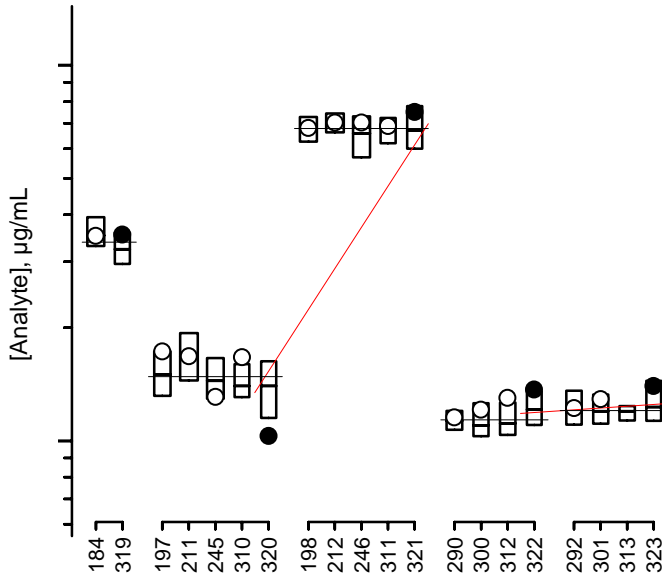
### Comments

Native, single-source
Same as #321, 1:3 diluted with stripped serum
Augmented, multi-source
Native, single-source
Native, single-source, hemolyzed



# Individualized RR LIX Report: FSV-BA

## Total β-Carotene



● You, this RR  
○ You, past RRs

▲ You, ≥x, this RR  
△ You, ≥x, past RRs

◇ NIST, this RR  
+ Others, this RR

### Serum

#319	Lyophilized - 28:184
#320	Lyophilized - 31:197, 35:211, 43:245, 57:310
#321	Lyophilized - 31:198, 35:212, 43:246, 57:311
#322	Lyophilized - 53:290, 55:300, 57:312
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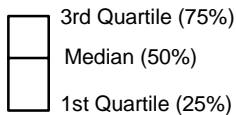
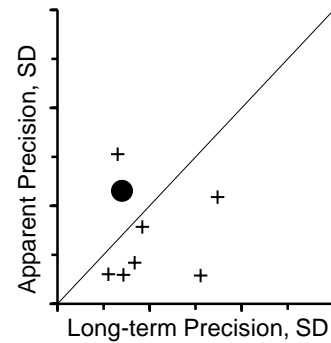
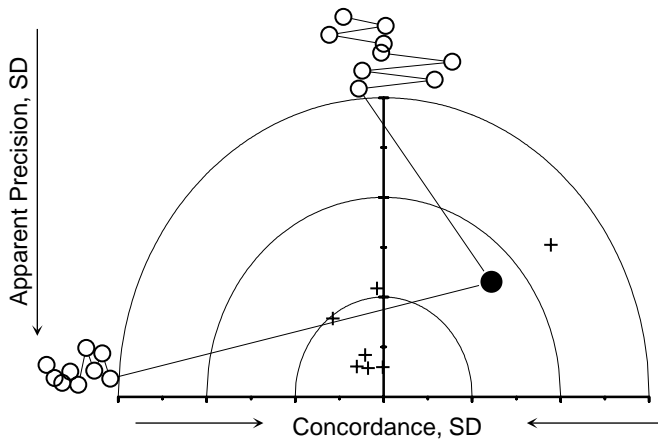
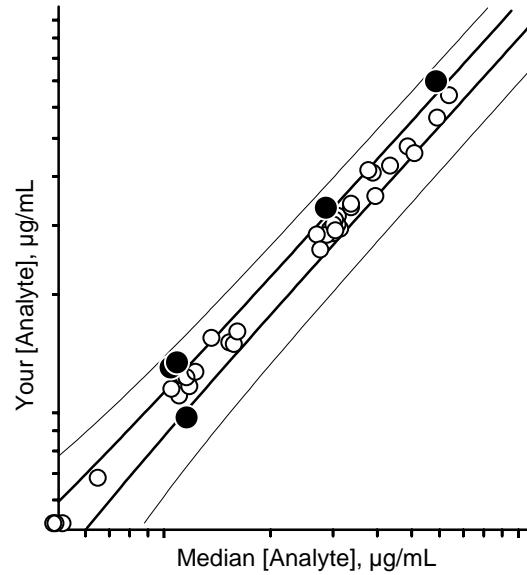
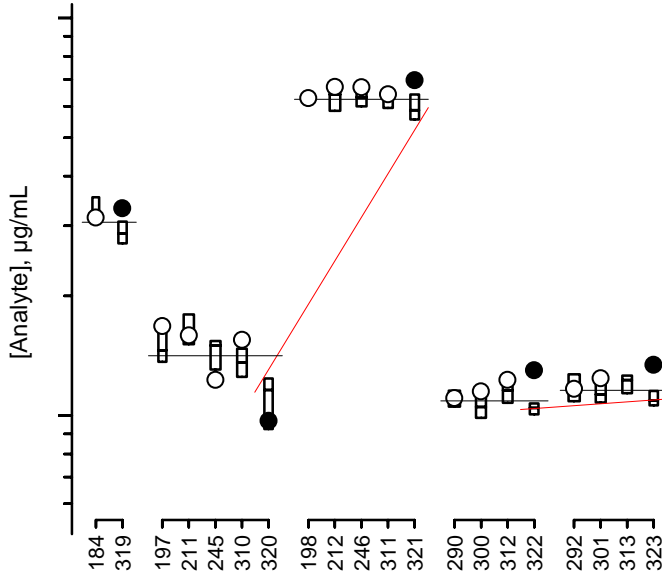
### History

### Comments

Native, single-source  
Same as #321, 1:3 diluted with stripped serum  
Augmented, multi-source  
Native, single-source  
Native, single-source, hemolyzed

# Individualized RR LIX Report: FSV-BA

trans-β-Carotene



● You, this RR  
○ You, past RRs

▲ You, ≥x, this RR  
△ You, ≥x, past RRs

◇ NIST, this RR  
+ Others, this RR

Serum

#319	Lyophilized - 28:184
#320	Lyophilized - 31:197, 35:211, 43:245, 57:310
#321	Lyophilized - 31:198, 35:212, 43:246, 57:311
#322	Lyophilized - 53:290, 55:300, 57:312
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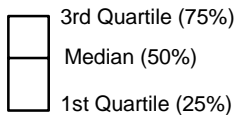
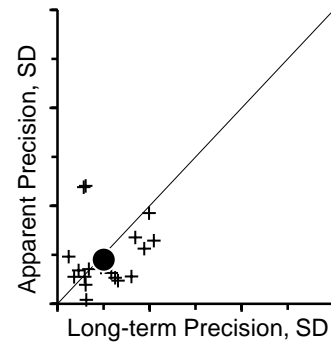
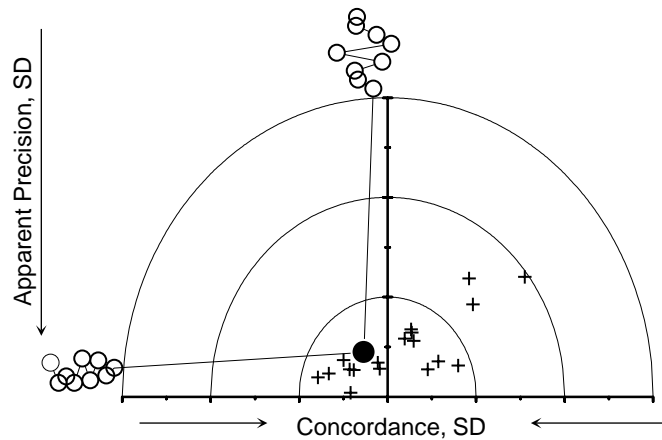
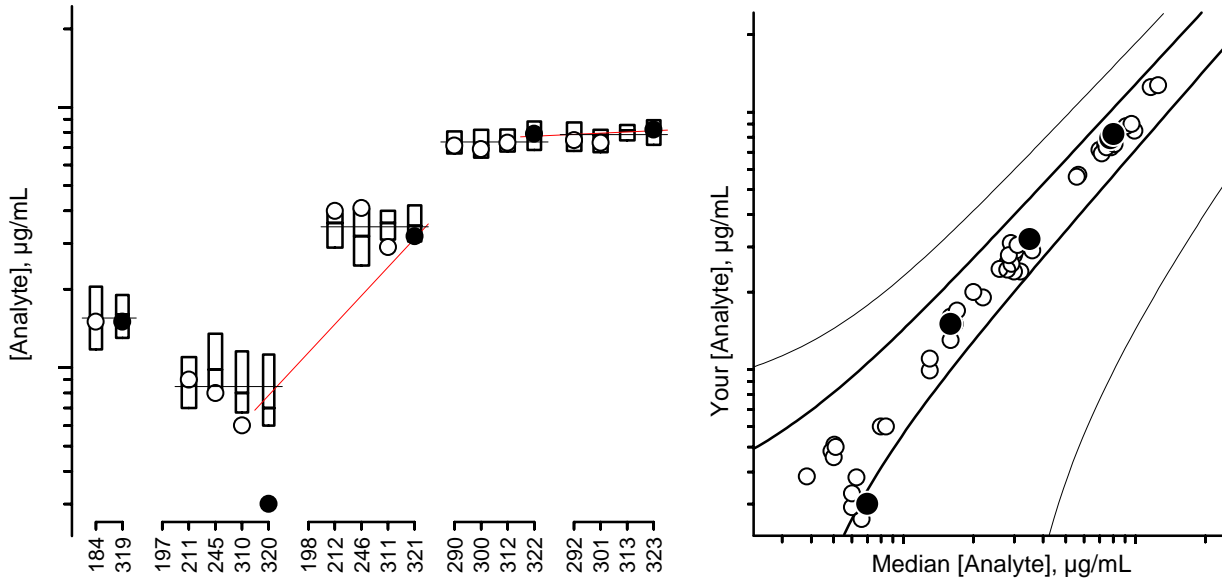
History

Comments

Native, single-source
Same as #321, 1:3 diluted with stripped serum
Augmented, multi-source
Native, single-source
Native, single-source, hemolyzed

# Individualized RR LIX Report: FSV-BA

## Total α-Carotene



● You, this RR  
○ You, past RRs

▲ You, ≥x, this RR  
△ You, ≥x, past RRs

◇ NIST, this RR  
+ Others, this RR

### Serum

Sample ID	History
#319	Lyophilized - 28:184
#320	Lyophilized - 31:197, 35:211, 43:245, 57:310
#321	Lyophilized - 31:198, 35:212, 43:246, 57:311
#322	Lyophilized - 53:290, 55:300, 57:312
#323	Fresh-frozen: 53:292, 55:301, 57:313

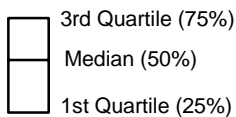
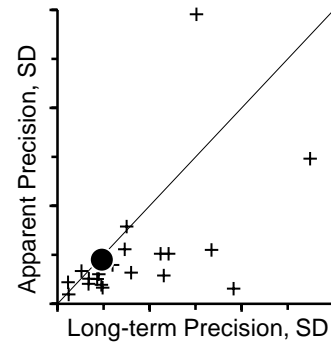
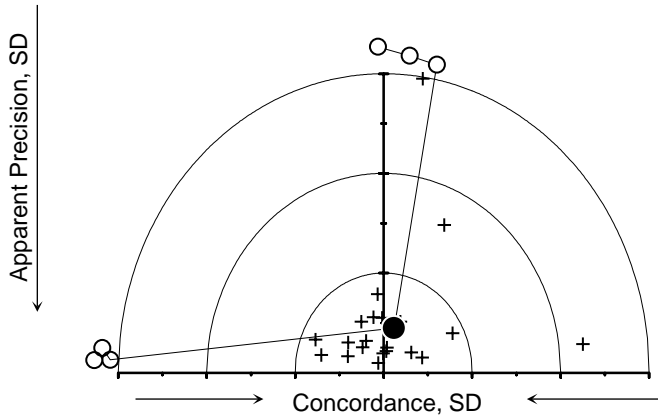
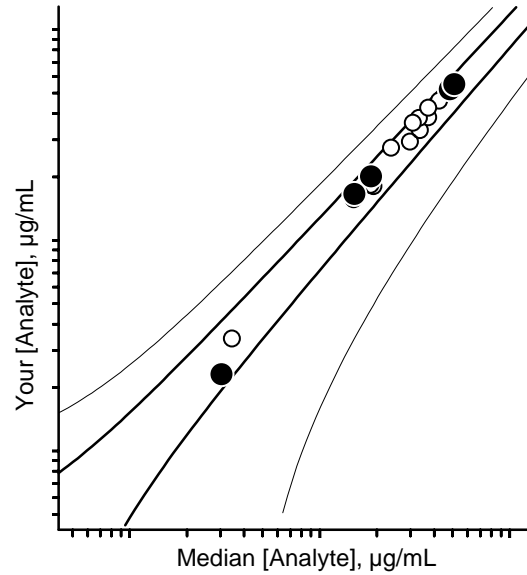
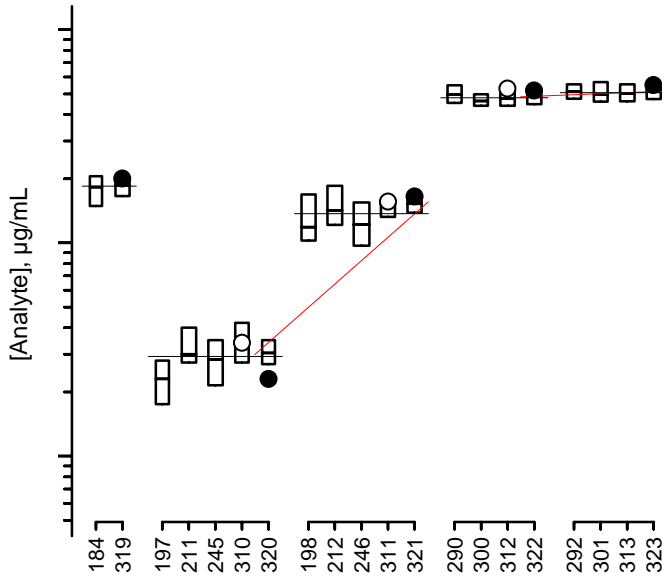
### History

### Comments

Native, single-source
Same as #321, 1:3 diluted with stripped serum
Augmented, multi-source
Native, single-source
Native, single-source, hemolyzed

# Individualized RR LIX Report: FSV-BA

## Total Lycopene



● You, this RR  
○ You, past RRs

▲ You, ≥x, this RR  
△ You, ≥x, past RRs

◇ NIST, this RR  
+ Others, this RR

### Serum

#319	Lyophilized - 28:184
#320	Lyophilized - 31:197, 35:211, 43:245, 57:310
#321	Lyophilized - 31:198, 35:212, 43:246, 57:311
#322	Lyophilized - 53:290, 55:300, 57:312
#323	Fresh-frozen: 53:292, 55:301, 57:313

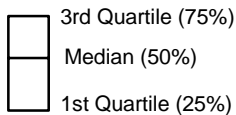
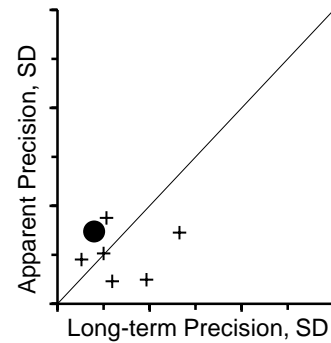
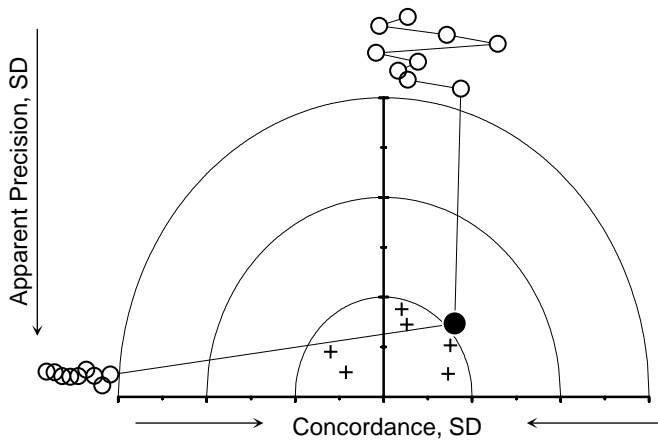
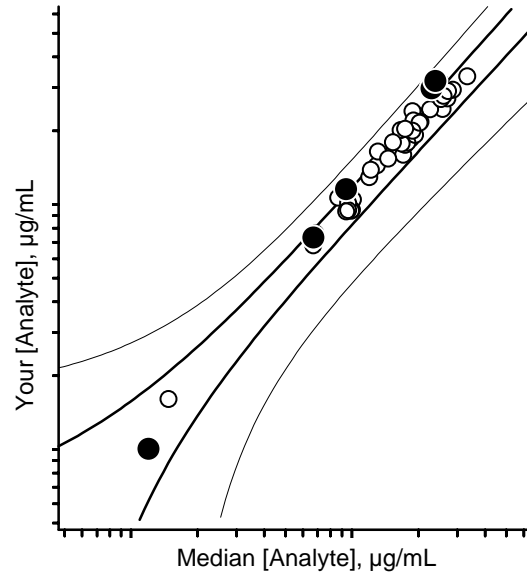
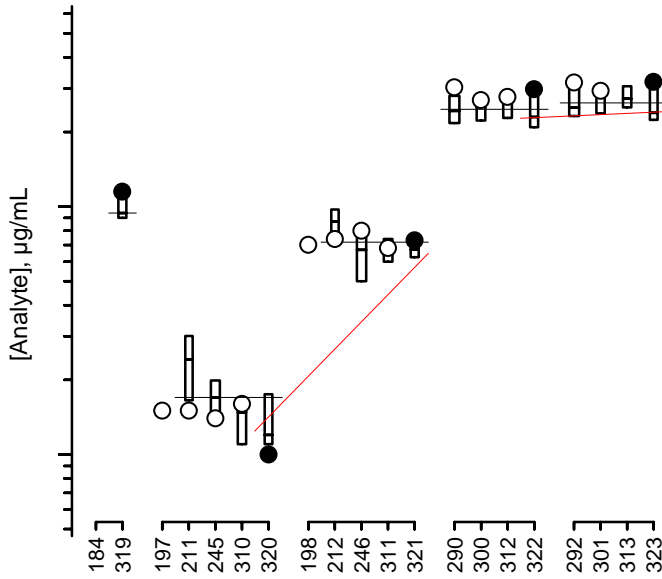
### History

### Comments

Native, single-source
Same as #321, 1:3 diluted with stripped serum
Augmented, multi-source
Native, single-source
Native, single-source, hemolyzed

# Individualized RR LIX Report: FSV-BA

## trans-Lycopene



● You, this RR  
○ You, past RRs

▲ You, ≥x, this RR  
△ You, ≥x, past RRs

◇ NIST, this RR  
+ Others, this RR

### Serum

#319	Lyophilized - 28:184
#320	Lyophilized - 31:197, 35:211, 43:245, 57:310
#321	Lyophilized - 31:198, 35:212, 43:246, 57:311
#322	Lyophilized - 53:290, 55:300, 57:312
#323	Fresh-frozen: 53:292, 55:301, 57:313

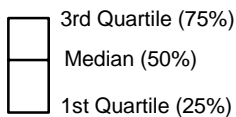
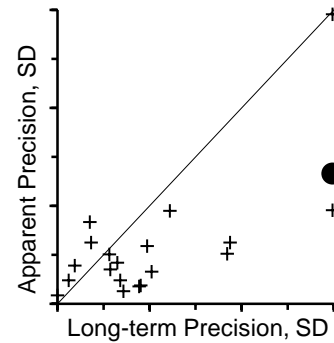
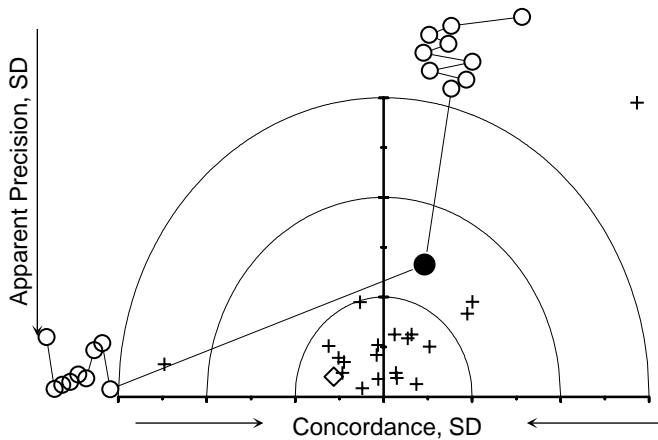
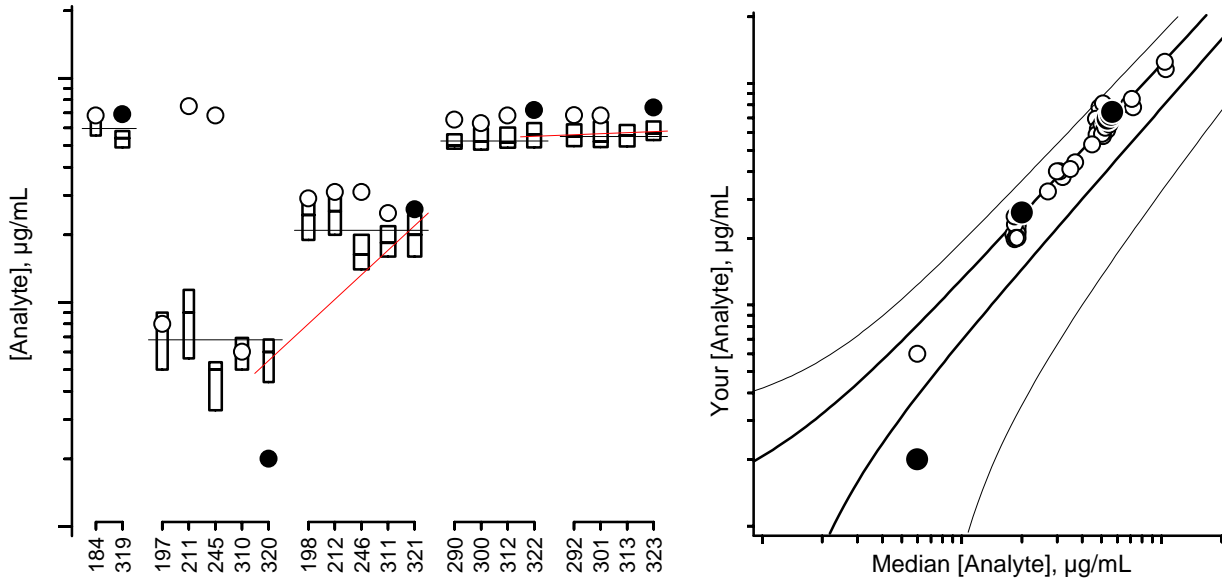
### History

### Comments

Native, single-source  
Same as #321, 1:3 diluted with stripped serum  
Augmented, multi-source  
Native, single-source  
Native, single-source, hemolyzed

# Individualized RR LIX Report: FSV-BA

## Total $\beta$ -Cryptoxanthin



● You, this RR  
○ You, past RRs

▲ You,  $\geq x$ , this RR  
△ You,  $\geq x$ , past RRs

◇ NIST, this RR  
+ Others, this RR

### Serum

#319	Lyophilized - 28:184
#320	Lyophilized - 31:197, 35:211, 43:245, 57:310
#321	Lyophilized - 31:198, 35:212, 43:246, 57:311
#322	Lyophilized - 53:290, 55:300, 57:312
#323	Fresh-frozen: 53:292, 55:301, 57:313

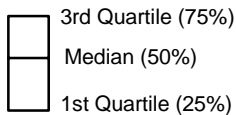
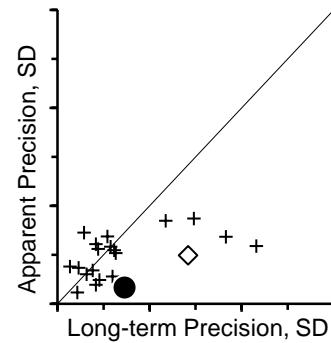
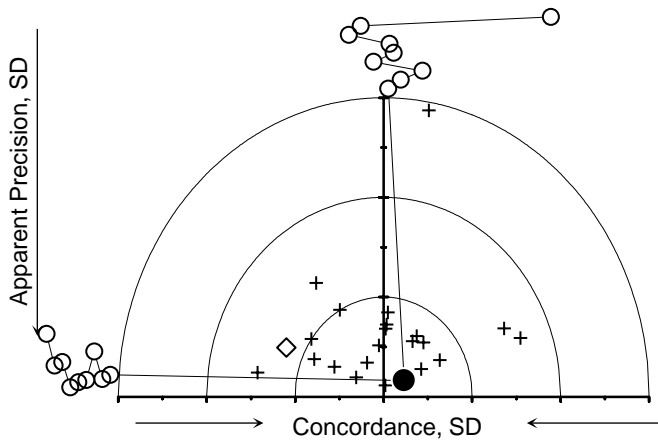
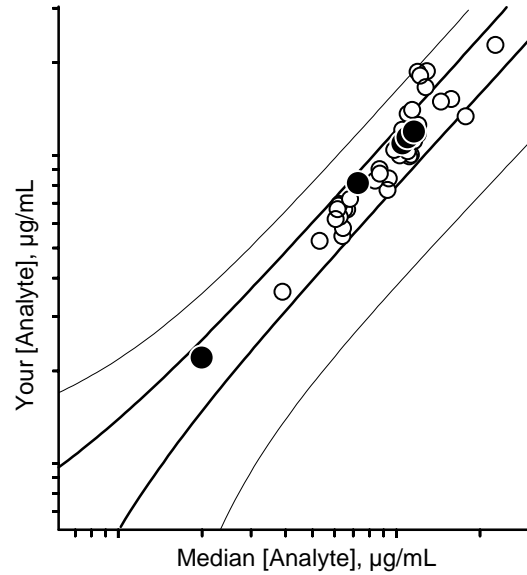
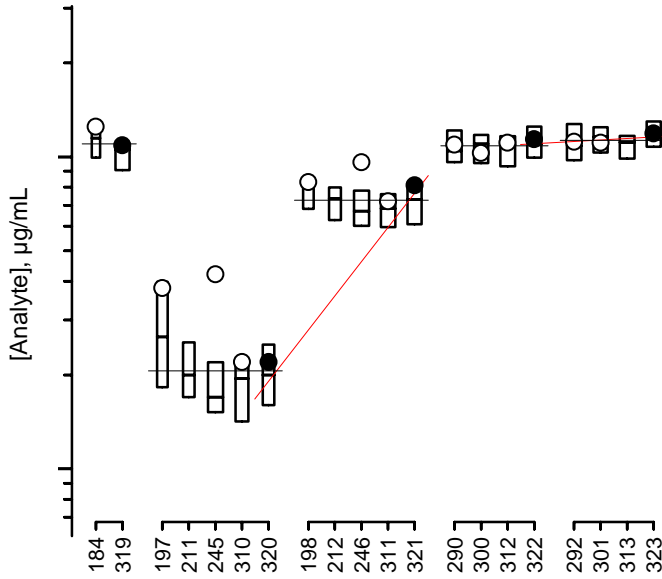
### History

### Comments

Native, single-source
Same as #321, 1:3 diluted with stripped serum
Augmented, multi-source
Native, single-source
Native, single-source, hemolyzed

# Individualized RR LIX Report: FSV-BA

## Total Lutein&Zeaxanthin



● You, this RR  
○ You, past RRs

▲ You, ≥x, this RR  
△ You, ≥x, past RRs

◇ NIST, this RR  
+ Others, this RR

### Serum

#319	Lyophilized - 28:184
#320	Lyophilized - 31:197, 35:211, 43:245, 57:310
#321	Lyophilized - 31:198, 35:212, 43:246, 57:311
#322	Lyophilized - 53:290, 55:300, 57:312
#323	Fresh-frozen: 53:292, 55:301, 57:313

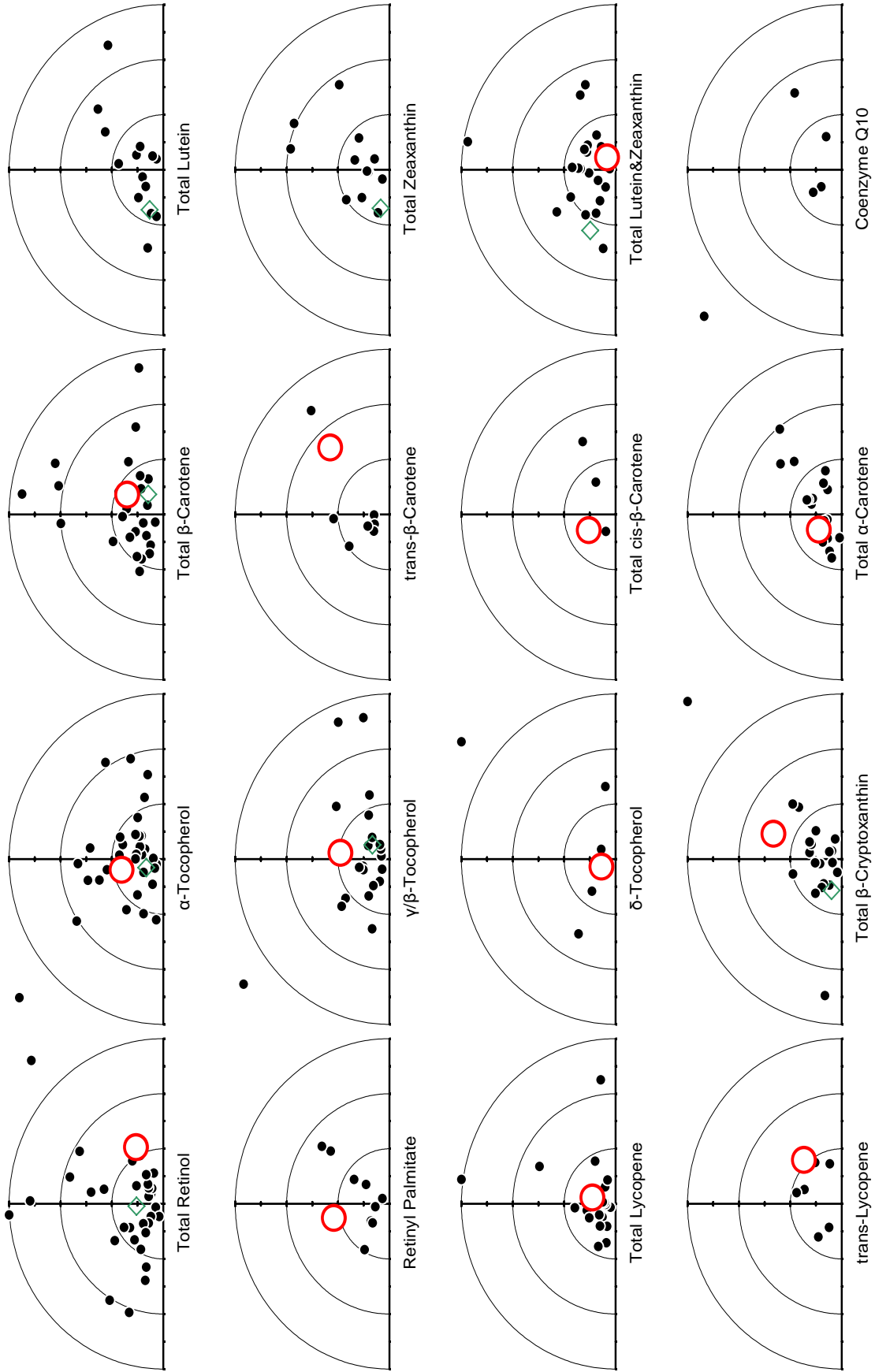
### History

### Comments

Native, single-source
Same as #321, 1:3 diluted with stripped serum
Augmented, multi-source
Native, single-source
Native, single-source, hemolyzed

# Individualized Round Robin LIX Report: FSV-BA

## Graphical Comparability Summary





## **Appendix E. Shipping Package Inserts for RR24**

The following five items were included in each package shipped to an RR24 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.

November 28, 2005



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

Dear Colleague:

The samples within this package constitute Vitamin C Round Robin 24 (RR24) of the fiscal year (FY) 06 Micronutrients Measurement Quality Assurance Program.

RR24 consists of four vials of frozen serum *test samples* (#22, #43, #117, and #119), one vial of ascorbic acid *solid control material* (Control), and two vials of frozen *serum control materials* (Control #1 and Control #2). Please follow the attached protocols when you prepare and analyze these samples. If you cannot prepare the *solid control* solutions gravimetrically, please prepare equivalent solutions volumetrically and report the exact volumes used. (Routine 0.5 g gravimetric measurements are generally 10-fold more accurate than routine 0.5 mL volumetric measurements.)

The two *serum control materials* are a new component of the M<sup>2</sup>QAP for Vitamin C. Please use these materials to validate the performance of your measurement system before you analyze the *test samples*. The target value and  $\approx 95\%$  confidence interval for *Control #1* is  $8.41 \pm 0.61 \mu\text{mol/L}$  sample; the target value and  $\approx 95\%$  confidence interval for *Control #2* is  $28.05 \pm 0.49 \mu\text{mol/L}$  sample.

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

The report for RR23 was mailed during the first week of November. If you find your results for RR23 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](mailto:srminfo@nist.gov)).

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

We ask that you return your results for the RR24 samples by **March 3, 2006**. We would appreciate receiving your results as soon as they become available. Please use the attached form. Your results will be kept confidential.

Sincerely,

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

Enclosures: Protocols, Preparation and Analysis of Control Materials and Analysis of Test Samples  
RR24 Report Form for Ascorbic Acid Solid Control Material Preparation  
RR24 Report Form for Control Material and Test Sample Analyses

# 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  $\mu\text{mol/L}$  is calculated:

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

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  $[\text{TAA}]_{\text{DS1}} = (0.52 \text{ g})(0.2 \text{ g}) \cdot (56785 \mu\text{mol/g} \cdot \text{L}) / (0.2 + 103 \text{ g}) = 57.2 \mu\text{mol/L}$ . Likewise, 0.25 mL of the Stock Solution should weigh 0.26 g and  $[\text{TAA}]_{\text{DS2}} = 28.4 \mu\text{mol/L}$  and 0.125 mL should weigh 0.13 g and  $[\text{TAA}]_{\text{DS3}} = 14.2 \mu\text{mol/L}$ .

- 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 ( $A_{\text{max}}$ ) within this region. Record the wavelength ( $\lambda_{\text{max}}$ ) at which this maximum occurs.

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

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

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

- 6) Measure and record the concentration of total ascorbic acid in all three dilute solutions and in the 5% MPA Diluent in duplicate using *exactly* the same method that you will use for the serum control materials and test samples, including any enzymatic treatment. We recommend that you analyze these solutions in the following order: Diluent, Dilute Solution 1, Dilute Solution 2, Dilute Solution 3, Dilute Solution 3, Dilute Solution 2, Dilute Solution 1, Diluent.
  - a) Compare the values of the duplicate measurements. *Are you satisfied that your measurement precision is adequate?*
  - b) Compare the measured with the calculated [TAA] values. This is most conveniently done by plotting the measured values on the y-axis of a scatterplot against the calculated values on the x-axis. The line through the four {calculated, measured} data pairs should go through the origin with a slope of 1.0. *Are you satisfied with the agreement between the measured and calculated values?*

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

- 7) Once you have confirmed that your system is properly calibrated, analyze the serum control CS #2 (see protocol below). The target values for this materials is  $28.1 \pm 1.0 \mu\text{mol/L}$  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: 301-975-3120 or Jeanice.BrownThomas@NIST.gov.

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

### **Protocol for Analysis of the Serum Control Materials and Test Samples**

The *serum control 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  $\mu\text{mol}$  of total ascorbic acid/L of solution. The total ascorbic acid in each ampoule should be measured in duplicate. Please report your results in  $\mu\text{mol}/(\text{L of the sample solution})$  rather than  $\mu\text{mol}/(\text{L of serum NIST used to prepare the sample})$ .

Participant #: \_\_\_\_\_

Date: \_\_\_\_\_

**Vitamin C Round Robin 24**  
**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]<sub>DS1</sub> ..... \_\_\_\_\_ 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]<sub>DS2</sub> ..... \_\_\_\_\_ 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]<sub>DS3</sub> ..... \_\_\_\_\_ μmol/L

Please return by **March 3, 2006**

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 24**  
**NIST Micronutrient Measurement Quality Assurance Program**

**Analysis of Control Materials and Test Samples**

<b>Sample</b>	<b>Replicate 1</b>	<b>Replicate 2</b>	<b>Units</b>
Dilute Solution 1	_____	_____	µmol/L of Dilute Solution
Dilute Solution 2	_____	_____	µmol/L of Dilute Solution
Dilute Solution 3	_____	_____	µmol/L of Dilute Solution
5% MPA Diluent	_____	_____	µmol/L of Diluent
Serum Control #1	_____	_____	µmol/L of Sample <i>Target: 8.5 ±0.5 µmol/L</i>
Serum Control #2	_____	_____	µmol/L of Sample <i>Target: 28.1 ±1.0 µmol/L</i>
Serum Test Sample #22	_____	_____	µmol/L of Sample
Serum Test Sample #43	_____	_____	µmol/L of Sample
Serum Test Sample #117	_____	_____	µmol/L of Sample
Serum Test Sample #119	_____	_____	µmol/L of Sample

Were samples frozen upon receipt? Yes | No

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

**COMMENTS:**

Please return by **March 3, 2006**

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 24**  
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<sup>2</sup>QAP samples:

Sample	Form
VitC #22	Liquid frozen (1:1 serum:10% MPA)
VitC #43	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)
CS #2	Liquid frozen (1:1 serum:10% MPA)
Control	Solid AA

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

1) Date this shipment arrived: \_\_\_\_\_

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

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

4) Did the samples arrive frozen? Yes | No

5) At what temperature are you storing the samples? \_\_\_\_\_ °C

6) When do you anticipate analyzing these samples? \_\_\_\_\_

**Your prompt return of this information is appreciated.**

The M<sup>2</sup>QAP Gang

Mail: M<sup>2</sup>QAP  
NIST, Stop 8392  
Gaithersburg, MD 20899-8392

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

## **Appendix F. Final Report for RR24**

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

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





April 18, 2006

Dear Colleague:

Enclosed is the summary report of the results for Round Robin 24 (RR 24) 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  $100 \times \text{MADe} / \text{median}$ .

RR 24 consisted of four *test samples* (#22, #43, #117, and #119), two *serum control materials*, and one *solid control material* for preparation of TAA control solutions. Details regarding the samples can be found in the enclosed report.

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

Samples for the second vitamin C round robin (RR 25) of the 2006 M<sup>2</sup>QAP will be shipped **during the week of May 8, 2006.**

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

Enclosures

The NIST M<sup>2</sup>QAP Vitamin C Round Robin 24 (RR24) report consists of

Page	“Individualized” Report
1	Summarizes your reported values for the nominal 55 mmol/L solution you prepared from the ascorbic acid solid control sample, the two serum control samples, and the four serum test samples.
2	Graphical summary of your RR24 sample measurements.
Page	“All Lab” Report
1	A tabulation of results and summary statistics for Total Ascorbic Acid [TAA] in the RR24 samples and control/calibration solutions.

**Serum-based Samples.** Two serum controls and four unknowns were distributed in RR24.

- CS1 SRM 970 level 1, ampouled in mid-1998.
- CS2 SRM 970 level 2, ampouled in mid-1998.
- S24:1 Serum 22, ampouled in late 2001, previously distributed as sample S17:1 (RR17, Fall 02), S19:2 (RR19, Fall 03) and S21:2 (RR21, Fall 04).
- S24:2 Serum 43, ampouled in late 2001, previously distributed as sample S18:2 (RR18, Spring 03), S19:3 (RR19, Fall 03), S21:3 (RR21, Fall 04), and S22:3 (RR22, Spring 05).
- S24:3 Serum 117, ampouled in 1995, previously distributed sample 682b=S07:3 (RR7, Spring 95), 682b=S10:1 (RR10, 1997), and S13:4 (RR13, 2000).
- S24:4 Serum 119, ampouled in 1995, previously distributed sample 688b=S07:2 (RR7, Spring 1995), 688b=S08:2 (RR8, 1997), and S13:4 (RR13, 2000).

## Results.

- 1) Nearly all participants who prepared the four 5% metaphosphoric acid (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 ( $\approx 1.03$  gm/mL), the observed wavelength maximum of “Dilute Solution #1” ( $\approx 244$  nm), the observed absorbance at that maximum ( $\approx 0.55$  OD), the calculated  $E^{1\%}_{1\text{cm}}$  ( $\approx 550$  dL/g·cm).

One participant chose to make the solutions in trichloroacetic acid (TCA) rather than MPA. One participant appears to have made simple aqueous solutions. Since the test samples are in 5% MPA (i.e., 1:1 dilutions of augmented serum in 10% MPA), their measurements of the test samples are not necessarily related to their control solution measurements.

- 2) Judging from the calibration parameters (intercepts close to 0.0, slopes close to 1.0, correlation ( $R^2$ ) close to 1, and the root mean square error or “standard estimate of the error” (SEE) close to 0.0) calculated for the control/calibration solutions in the report summary, the measurement systems for all participants are linear and reasonably well calibrated. However, several participants have measurement systems that perform somewhat differently for the control solutions and the test samples. This suggests either that the serum matrix can induce a measurement bias and/or that the measurement system used for the control solutions isn’t *exactly* the same as for the test materials. Figuring this out is one of our future projects.
- 3) Perhaps somewhat surprisingly, there is no evidence for sample degradation in any of the samples – including the two that were produced in 1995.

## **Appendix G. “All-Lab Report” for RR24**

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" 24 - March 2006**

Lab	Date	Control / Calibration Samples						MPA				Dilute Solution 1				Samples																																										
		Gravimetric, $\mu\text{mol/L}$		Measured, $\mu\text{mol/L}$		Calibration Parameters		Density g/mL	Spectrophotometry		Measured, $\mu\text{mol/L}$		Calibrated to Gravimetric Solution, $\mu\text{mol/L}$		CS#1		CS#2		CS#1		CS#2		S24:1		S24:2		S24:3		S24:4																													
		Dil:1	Dil:2	Dil:3	Dil:1	Dil:2	Dil:3		MPA	Inter	Slope	R <sup>2</sup>	RMSE	$\lambda_{\text{max}}$	A <sub>max</sub>	E%	S24:1	S24:2	S24:3	S24:4	CS#1	CS#2	S24:1	S24:2	S24:3	S24:4	CS#1	CS#2	S24:1	S24:2	S24:3	S24:4																										
VC-MA	09/03/06	55.0	27.8	13.9	62.3	31.3	16.3	0.0	0.21	1.13	1.000	0.4	1.031	244.	0.5680	586.7	9.3	28.2	9.7	35.6	59.1	29.2	8.0	25.7	8.4	31.4	52.2	25.7	VC-MB	29/12/05	55.9	27.7	14.0	55.0	27.7	13.9	0.0	0.13	0.98	1.000	0.2	1.033	244.	0.5481	565.5	7.5	26.6	8.5	33.3	60.9	27.6	7.5	26.9	8.6	33.7	61.9	28.0	
VC-MC	26/01/06	55.4	27.6	13.7	59.0	30.4	14.2	0.0	-0.01	1.07	0.999	0.7	1.031	243.	0.5757	589.7	7.5	30.0	11.1	38.3	60.4	27.1	7.0	28.0	10.4	35.8	56.4	25.3	VC-ME	03/03/06	56.8	29.2	14.1	58.0	28.9	14.3	0.9	0.33	1.01	0.999	0.8	1.029	243.	0.5712	571.2	8.7	27.5	9.6	32.7	59.7	27.5	8.3	27.0	9.2	32.1	58.9	27.0	
VC-MG	09/02/06	58.8	30.4	16.0	59.8	27.7	13.7	0.0	-1.65	1.02	0.996	2.1	1.031	243.7	0.5730	553.1	8.1	30.2	10.7	38.2	69.7	31.3	9.5	31.1	12.1	38.9	69.6	32.2	VC-MH	09/03/06	62.2	31.1	15.4	63.9	32.2	15.9	0.1	0.15	1.02	1.000	0.1	1.031	244.1	0.6198	565.6	8.9	29.0	9.8	34.6	b	27.6	8.5	28.1	9.4	33.6	26.8		
VC-MI	02/03/06	56.6	28.1	13.8	53.9	26.5	12.6	0.0	-0.31	0.95	1.000	0.3	1.031	244.1	0.6198	565.6	8.5	28.0	8.3	29.4	62.5	27.4	9.2	29.6	9.0	31.1	65.8	29.0	VC-MJ	31/01/06	59.4	29.1	15.1	58.3	30.5	15.3	0.7	0.98	0.97	0.999	1.0	1.022 <sup>a</sup>	250 <sup>a</sup>	0.3650 <sup>a</sup>	349.1 <sup>a</sup>	11.8	31.4	13.6	37.5	59.7	30.2	11.1	31.2	12.9	37.5	60.4	30.0	
VC-MK	03/02/06	58.9	30.8	14.9	59.8	31.6	14.9	1.2	0.67	1.00	1.000	0.6	1.031	244.	0.5980	576.8	9.6	31.8	12.3	35.6	c	31.7	8.9	31.0	11.6	34.8	31.0	VC-ML	08/03/06	56.5	28.3	14.3	56.2	27.6	13.1	0.0	-0.56	1.00	1.000	0.6	1.026	242.	0.5580	560.9	7.1	27.0	6.8	29.9	55.4	24.8	7.6	27.5	7.3	30.5	55.9	25.4		
VC-MP	03/03/06	55.4	28.9	11.8	51.2	25.9	10.8	0.2	-0.10	0.92	1.000	0.5	0.992	243.	0.5135	526.6	6.7	32.2	7.9	40.8	78.8	32.8	7.4	35.1	8.6	44.4	85.7	35.8	VC-MS	06/03/06	55.4	28.9	11.8	51.2	25.9	10.8	0.2	-0.10	0.92	1.000	0.5	0.992	243.	0.5135	526.6	6.7	32.2	7.9	40.8	78.8	32.8	7.4	35.1	8.6	44.4	85.7	35.8	
N	11	11	11	11	11	11	11	11	N	10	9	9	9	10	9	9	9	12	12	11	10	12	11	11	11	11	11	9	11	VC-MB	29/12/05	57.3	29.0	14.3	57.9	29.1	14.1	0.3	0.7	0.9298	19.2	28.7	8.5	29.2	9.8	34.9	62.4	28.7	8.5	29.2	9.8	34.9	63.0	28.7	9.2	33.7	60.4	28.0
Average	57.3	29.0	14.3	57.9	29.1	14.1	0.3	Average	1.026	243.4	0.5695	565.2	1.031	243.4	0.5695	565.2	8.5	29.3	9.8	34.9	62.4	28.7	8.5	29.2	9.8	34.9	63.0	28.7	VC-MC	26/01/06	55.0	27.6	13.8	55.6	27.64	13.4	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0				
SD	2.2	1.3	1.1	3.7	2.2	1.6	0.4	SD	0.012	0.7	0.0298	19.2	1.031	243.7	0.5712	565.6	7.5	27.9	8.4	32.8	59.3	27.3	7.5	27.2	7.3	30.5	52.2	25.3	VC-ME	03/03/06	56.6	28.92	14.1	58.3	28.90	14.2	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0				
Min	55.0	27.60	11.8	51.2	25.90	10.8	0.0	Min	0.992	242.0	0.5135	526.6	0.992	242.0	0.5135	526.6	6.7	26.6	6.8	29.4	55.4	24.8	7.0	25.7	7.3	30.5	52.2	25.3	VC-MG	09/02/06	58.8	29.82	15.0	59.8	30.92	15.1	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4		
%25	55.7	27.97	13.8	55.6	27.64	13.4	0.0	%25	1.029	243.0	0.5580	556.5	1.029	243.0	0.5580	556.5	7.5	27.9	8.4	32.8	59.3	27.3	7.5	27.2	7.3	30.5	52.2	25.3	VC-MH	09/03/06	62.2	31.07	16.0	63.9	32.15	16.3	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2		
Median	56.6	28.92	14.1	58.3	28.90	14.2	0.0	Median	1.031	244.0	0.5712	565.6	1.031	244.0	0.5712	565.6	8.5	28.1	9.7	35.1	60.0	27.6	8.3	28.1	9.2	33.7	60.4	28.0	VC-MI	02/03/06	56.6	28.1	13.8	53.9	26.5	12.6	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0		
%75	58.8	29.82	15.0	59.8	30.92	15.1	0.4	%75	1.031	244.0	0.5712	565.6	1.031	244.0	0.5712	565.6	8.5	28.1	9.7	35.1	60.0	27.6	8.3	28.1	9.2	33.7	60.4	28.0	VC-MJ	31/01/06	59.4	29.1	15.1	58.3	30.5	15.3	0.7	0.98	0.97	0.999	1.0	1.022 <sup>a</sup>	250 <sup>a</sup>	0.3650 <sup>a</sup>	349.1 <sup>a</sup>	11.8	31.4	13.6	37.5	59.7	30.2	11.1	31.2	12.9	37.5	60.4	30.0	
Max	62.2	31.07	16.0	63.9	32.15	16.3	1.2	Max	1.033	244.1	0.6198	589.7	1.033	244.1	0.6198	589.7	11.8	32.2	13.6	40.8	78.8	32.8	11.1	35.1	12.9	44.4	85.7	35.8	VC-MK	03/02/06	58.9	30.8	14.9	59.8	31.6	14.9	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2	1.2		
MADe	1.9	1.6	0.6	3.1	2.4	1.6	0.0	MADe	0.000	0.6	0.0196	16.6	0.000	0.6	0.0196	16.6	1.3	2.0	2.1	3.6	2.3	1.6	1.3	2.3	1.2	3.5	6.6	3.4	VC-ML	08/03/06	56.5	28.3	14.3	56.2	27.6	13.1	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0			
CV	3	6	4	5	8	11	0.0	CV	0.04	0.24	3.4	2.9	0.04	0.24	3.4	2.9	16	7	21	10	4	15	8	13	10	11	12	VC-MP	03/03/06	55.4	28.9	11.8	51.2	25.9	10.8	0.2	-0.10	0.92	1.000	0.5	0.992	243.	0.5135	526.6	6.7	32.2	7.9	40.8	78.8	32.8	7.4	35.1	8.6	44.4	85.7	35.8		

- a) 5% Trichloroacetic acid solution
- b) Miss-labeled sample
- c) Broken ampoule

## **Appendix H. Representative “Individualized Report” for RR24**

Each participant in RR24 received an “Individualized Report” reflecting their reported results. The following two pages are the “Individualized Report” for participant “VC-MA”.

## Vitamin C "Round Robin" 24 Report: Participant VC-MA

Date	RR	Method	MPA	Dilute Solution 1			Control/Calibration Solutions			
			Density	Spectrophotometry			$Y_{\text{meas}} = \text{Inter} + \text{Slope} * X_{\text{grav}}$			
			g/mL	$\lambda_{\text{max}}$	$A_{\text{max}}$	$E^{1\%}$	Inter	Slope	$R^2$	SEE
11/13/03	19	HPLC-EC	1.026	243.0	0.584	561.9	1.1	1.03	0.998	1.24
02/23/04	20	HPLC-EC	1.031	243.0	0.552	560.7	-0.4	1.05	1.000	0.65
09/13/04	21	HPLC-EC	1.030	244.0	0.555	562.2	-0.1	0.99	1.000	0.10
03/08/05	22	HPLC-EC	1.034	243.0	0.559	562.9	0.2	1.06	1.000	0.24
10/17/05	23	HPLC-EC	1.030	244.0	0.562	567.9	-0.6	1.09	0.998	1.47
03/09/06	24	HPLC-EC	1.031	244.0	0.568	586.7	0.2	1.13	1.000	0.41
		Mean	1.03	243.5	0.56	567.0				0.68
		SD	0.003	0.5	0.01	9.9				0.56
		CV	0.25	0.22	2.0	1.8				

Date	RR	Sample	[TAA] mmol/Lsample								
			Rep <sub>1</sub>	Rep <sub>2</sub>	F <sub>adj</sub>	Mean	SD <sub>dup</sub>	N	Mean	SD <sub>repeat</sub>	SD <sub>reprod</sub>
02/23/04	20	CS#1	7.8	8.0	1.0	7.9	0.1	5	8.6	0.1	0.7
09/13/04	21	CS#1	8.1	7.9	1.0	8.0	0.1				
03/08/05	22	CS#1	8.5	8.7	1.0	8.6	0.1				
10/17/05	23	CS#1	9.3	9.5	1.0	9.4	0.1				
03/09/06	24	CS#1	<b>9.3</b>	<b>9.2</b>	1.0	9.3	0.0				
02/23/04	20	CS#2	25.8	26.2	1.0	26.0	0.3	5	28.2	0.5	1.7
09/13/04	21	CS#2	26.2	27.2	1.0	26.7	0.7				
03/08/05	22	CS#2	29.0	29.0	1.0	29.0	0.0				
10/17/05	23	CS#2	29.4	30.5	1.0	30.0	0.8				
03/09/06	24	CS#2	<b>29.2</b>	<b>29.1</b>	1.0	29.2	0.1				
11/13/03	19	S19:2	9.2	9.1	1.0	9.2	0.1	4	9.3	0.1	0.4
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	<b>9.8</b>	<b>9.6</b>	1.0	9.7	0.2				
11/13/03	19	S19:3	35.9	35.8	1.0	35.9	0.1	4	35.0	0.2	1.3
09/13/04	21	S21:3	33.2	32.9	1.0	33.0	0.2				
03/08/05	22	S22:3	35.7	35.6	1.0	35.6	0.1				
03/09/06	24	S24:2	<b>35.8</b>	<b>35.5</b>	1.0	35.6	0.2				
03/09/06	24	S24:3	<b>59.1</b>	<b>59.1</b>	1.0	59.1	0.1				
03/09/06	24	S24:4	<b>29.0</b>	<b>29.5</b>	1.0	29.2	0.4				

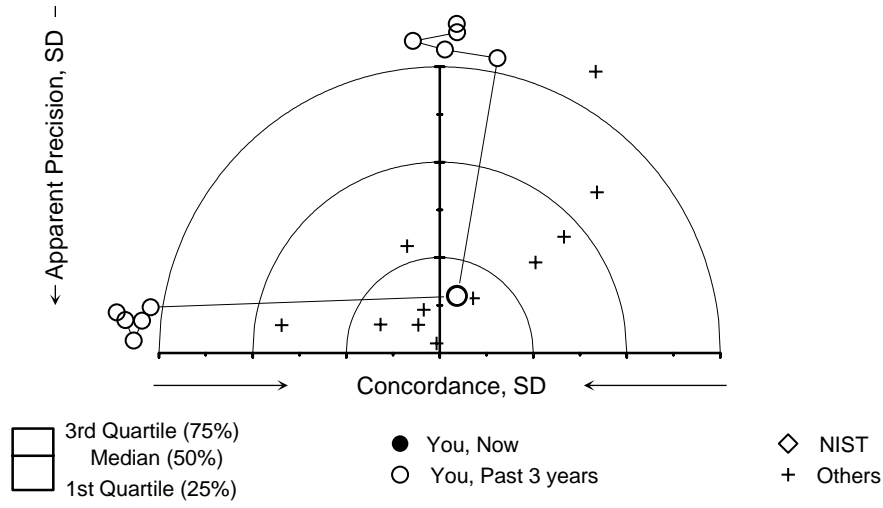
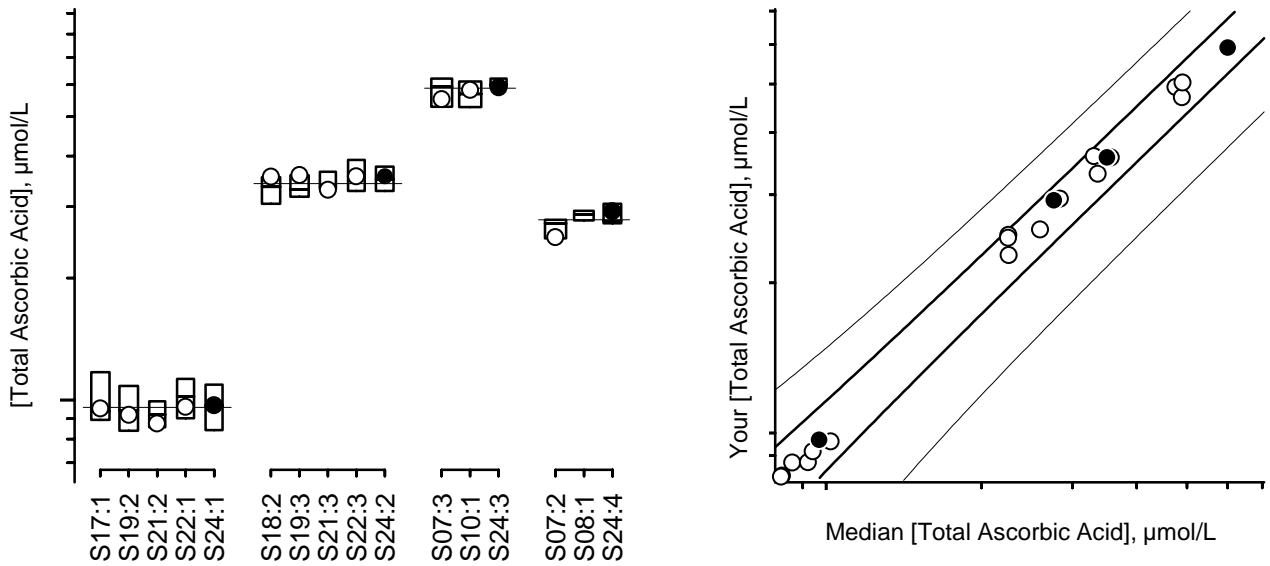
Please check our records against your records. Send corrections and/or updates to...

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# Vitamin C "Round Robin" 24 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

- S24:1 Serum 22, previously distributed in RRs 17, 19, 21, and 22
- S24:2 Serum 43, previously distributed in RRs 18, 19, 21, and 22
- S24:3 Serum 117, previously distributed in RRs 7 and 10
- S24:4 Serum 119, previously distributed in RRs 7 and 8