NISTIR 7880-14

NIST Micronutrients Measurement Quality Assurance Program Summer 2005 Comparability Studies

Results for Round Robin LVIII
Fat-Soluble Vitamins and Carotenoids in Human Serum
and Round Robin 23 Ascorbic Acid in Human Serum

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

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Materials Measurement Laboratory

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U.S. Department of Commerce
Cameron F. Kerry, Acting Secretary

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

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Abstract

The National Institute of Standards and Technology coordinates the Micronutrients Measurement Quality Assurance Program (MMQAP) for laboratories that measure fat- and water-soluble vitamins and carotenoids in human serum and plasma. This report describes the design of and results for the Summer 2005 MMQAP measurement comparability improvement studies: 1) Round Robin LVIII Fat-Soluble Vitamins and Carotenoids in Human Serum and 2) Round Robin 23 Total Ascorbic Acid in Human Serum. The materials for both studies were shipped to participants in May 2005; participants were requested to provide their measurement results by September 12, 2005.

Keywords

Human Serum
Retinol, α-Tocopherol, γ-Tocopherol, Total and *Trans*-β-Carotene
SRM 3276
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 LVIII: Fat-Soluble Vitamins and Carotenoids in Human Serum

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin LVIII comparability study (hereafter referred to as RR58) received two ampoules of SRM 3276 Carrot Extract in Sunflower Oil, two lyophilized human serum test samples, and three 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 May 2005. The "carrot oil" ampoules were shipped in the same package, but isolated from the dry ice. 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 RR58 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. Anonymized results for the “carrot oil” are also included. 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 23: Vitamin C in Human Serum

Participants in the MMQAP Vitamin C in Human Serum Round Robin 23 comparability study (hereafter referred to as RR23) 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 May 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 RR23 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 RR58

The following four items were included in each package shipped to an RR58 participant:

- Cover letter
- Datasheet for SRM 3276 carrot Extract in Sunflower Oil
- Datasheet for serum samples
- Packing List and Shipment Receipt Confirmation Form

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

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

May 9, 2005

Dear Colleague:

Enclosed are two sets of samples for the second fat-soluble vitamins and carotenoids in serum round robin study (Round Robin LVIII) for the fiscal year (FY) 05 NIST Micronutrients Measurement Quality Assurance Program. The first set consists of two ampoules of **Standard Reference Material (SRM) 3276 Carrot Extract in Sunflower Oil**, which is being prepared as part of a collaborative effort among NIST, the Food and Drug Administration (FDA) and the National Institutes of Health's Office of Dietary Supplements (NIH/ODS). This material should be refrigerated upon receipt. The material was prepared by extracting carrots into sunflower oil and readily dissolves in ethanol. The "carrot oil" was spiked with 2,6-di-*tert*-butyl-4-methylphenol (BHT) to a concentration of approximately 670 mg/kg. SRM 2376 was ampouled under argon in amber ampoules. Each ampoule contains approximately 1 mL of material. This material is very viscous, therefore we recommend that you weigh out your sample for analysis rather than relying on an accurate volumetric reading from a pipet. Your results should therefore be best expressed as mass fractions (ug/g).

Please analyze a test portion from each of the two ampoules. We would like you to measure α-carotene, β-carotene (total, trans, and any other isomers that you are able to quantify), and tocopherols in this material. Also, feel free to report values for any other carotenoids that you see. When reporting your results, please submit one value for each analyte from each ampoule. Also, please submit a chromatogram of your carrot oil analyses and specify the chromatographic conditions used.

The second set (**Sera 314 – 318**) consists of one vial of each of three liquid-frozen and two lyophilized serum samples for analysis along with a form for reporting your results. These samples should be stored in the dark at or below –20 °C upon receipt. When reporting your results, please submit one value for each analyte for a given serum sample. If a value obtained is below your limit of quantification, please indicate this result on the form by using NQ *(Not Quantified)*. Results are due to NIST by **September 12, 2005**. Results received more than two weeks after the due date will not be included in the summary report for this round robin study. The feedback report concerning the study will be distributed in October.

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

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

Please mail or fax your results for Round Robin LVIII and the carrot oil study to:

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

NIST

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

Sincerely,

Jeanice B Thomas

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

Enclosures

Participant #: ________ Date: _____________

Round Robin LVIII: Carrot Oil

NIST Micronutrients Measurement Quality Assurance Program

Analyte	Ampoule 1	Ampoule 2	Units*
α-tocopherol			
γ/β-tocopherol			
δ-tocopherol			
total β-carotene			
trans-β-carotene			
total cis-β-carotene			
9-cis-β-carotene			
13-cis-β-carotene			
15-cis-β-carotene			
total α-carotene			
trans-α-carotene			

Other measurands?			

* we prefer **µg/g**

Method:

Comments:

Please attach representative chromatogram(s)!

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

Please return results **before** 12-Sep-2005

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

Participant #: ________ Date: _____________

Round Robin LVIII: Human Sera

NIST Micronutrients Measurement Quality Assurance Program

Analyte	314	315	316	317	318	Units*
total retinol						
trans-retinol						
didehydroretinol						
retinyl palmitate						
α-tocopherol						
γ/β-tocopherol						
δ-tocopherol						
total β-carotene						
trans-β-carotene						
total cis-β-carotene						
total α-carotene						
total lycopene						
trans-lycopene						
total β-cryptoxanthin						
total α-cryptoxanthin						
total lutein						
total zeaxanthin						
total lutein&zeaxanthin						
total coenzyme Q10						
ubiquinol (QH_2)						
ubiquinone (Qox)						
phylloquinone (K_1)						
25-hydroxyvitamin D						

Other measurands?						

* we prefer **μg/mL**

Were the liquid frozen samples frozen when received? Yes | No

Comments:

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

Please return results **before** 12-Sep-2005

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

Participant #: ________ Date: ____________

Fat-Soluble Vitamins Round Robin LVIII

NIST Micronutrients Measurement Quality Assurance Program

Packing List and Shipment Receipt Confirmation Form

This box contains: two ampoules of **carrot oil**
one vial each of the following five FSV M2QAP **sera**

Serum	Form	Reconstitute?
#314	Liquid frozen	No
#315	Liquid frozen	No
#316	Liquid frozen	No
#317	Lyophilized	Yes (1 ml H_2O)
#318	Lyophilized	Yes (1 ml H_2O)

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
4a) Store the **carrot oil** at 4 ˚C until analysis
4b) 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 both carrot oil ampoules and 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 sera arrive frozen? Yes **|** No

5a) At what temperature are you storing the **carrot oil** ampoules? _______ ˚C

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

6) When do you anticipate analyzing these samples? ________________________

Your prompt return of this information is appreciated.

The M^2QAP Gang

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

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

Appendix B. Final Report for RR58

The following eight 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-

November 8, 2005

Dear Colleague:

Enclosed is the summary report of the results for round robin LVIII (RR58) of the 2005 NIST Micronutrients Measurement Quality Assurance Program (M^2QAP) for the fat-soluble vitamins and carotenoids in human serum. Included in this report are: (1) a summary of data and measurement comparability scores for all laboratories, (2) a detailed graphical analysis of your results; and (3) a graphical summary of your measurement comparabilities relative to the NIST assigned values. The NIST-assigned values are equally weighted means of the medians from this interlaboratory comparison exercise and the means from the analyses performed by NIST.

Data for evaluating laboratory performance in RR58 are provided in the comparability summary (Score Card) on page 6 of the All Lab Report. Laboratory comparability is summarized as follows: results rated 1 to 3 are within 1 to 3 standard deviations of the assigned value, respectively; those rated 4 are >3 standard deviations from the assigned value.

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

Samples for the first 2006 QA interlaboratory exercise will be shipped **starting the last week of November 2005**. 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

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

Enclosures

NIST

The NIST M²QAP Round Robin LVIII (RR58) report consists of:

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

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

Samples. The five sera below were distributed in RR58.

Serum	Description	Prior Distributions
314	Fresh-frozen, native, multi-donor serum prepared in 2005. This serum was expected to have moderate to high levels of many analytes.	None
315	Fresh-frozen, native, multi-donor serum prepared in 2005. This serum was prepared as a 77:110 volumetric blend of sera #314:#316.	None
316	Fresh-frozen, native, multi-donor serum prepared in 2005. This serum was expected to have low levels of most analytes.	None
317	Lyophilized blended serum, a 1:1 blend of the pools used to produce SRM 968c Levels I and II.	#248 RR44 (9/98)
318	Lyophilized blended serum with native carotenoid levels, augmented with *trans*-retinol and γ/β-tocopherol; SRM 968c Level I.	#248 RR44 (9/98), #258 RR46 (6/99), #263 RR47 (5/00), #280 RR51 (3/02), #304 RR46 (9/04)

Results

1) Sera Stability: There was no significant change in the median level nor increase in the variability of any measurand in either sera #317 nor #318.

2) Relative Accuracy: Sera #314, #315, and #316 were prepared to evaluate the relative accuracy of your assays. Serum #314 was prepared from a serum pool known to have relatively high levels of many analytes. Serum #316 was prepared from a serum pool known to have relatively low levels of most analytes. Serum #315 was prepared as a 77:110 volumetric blend of the two pools. If your assay imprecision is small and the response is linear over the range from the low level of #316 to the

high level of #314, assuming that the blend is equal to the sum of its parts, then the result for #315 should be $77/187\times\#314+110/187\times\#316$. Likewise, serum #317 was prepared as a 1:1 volumetric blend of the pools used to produce SRM 968c Levels I (serum #318) and II (serum #309 in RR57). With the same caveats as above (and with greater uncertainty since serum #309 was analyzed in the previous RR), the result for #317 should be $1/2\times\#309+1/2\times\#318$.

The median values for *nearly* all analytes follow the expected behavior with both blended sera. For total β-carotene and total α-carotene, the observed medians for the blends are a bit above the line connecting the respective endpoints; however, they are definitively in-between the endpoints. However, the behavior of γ/β-tocopherol is unexpected: the median value for #315 is larger than either #314 or #316.

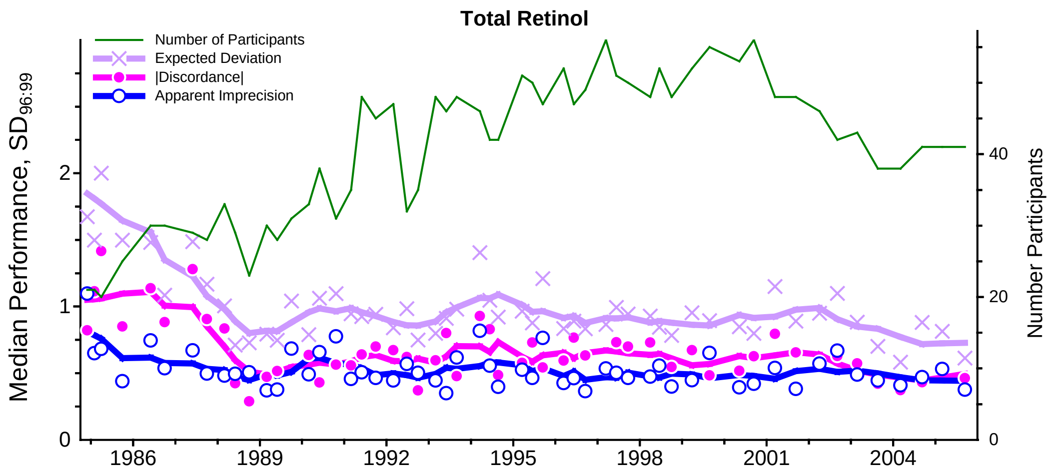
While the level of γ/β-tocopherol is relatively low in all three of these sera, it is well above the level where detection problems are expected. We obtain identical results with fluorescence detection as with absorbance and with peak height as with peak area, so it is unlikely to be an artifact of incomplete chromatographic separation or integration. We have also resolved the two isomers on a high-performance column and found that the ratio between the γ- and β-isomers is about the same in the two endpoints… and that *both* isomers are higher in #315 than in either endpoint. It really does look like the blending process either made the γ/β more readily extractable or that the relatively high level of α-tocopherol in #314 somehow suppresses the extraction of the γ/β. At any rate, we're puzzled. Other ideas and suggestions are most welcome.

3) Community Measurement Performance Characteristics: The graphs on the following four pages summarize the among-participant concordance, expected apparent precision, expected overall comparability, and number of participants reporting values for all of the "generally-reported" analytes. These summary graphs are described in detail in: Duewer DL, Kline MC, Sharpless KE, Brown Thomas J. *NIST Micronutrients Measurement Quality Assurance Program: Characterizing the measurement community's performance over time*. Anal Chem 2000;72(17):4163-4170.

The following table summarizes our interpretation of the information in these graphics:

Analyte	Comparability Relative to 2003
Total Retinol	Holding to slight improvement
Retinyl palmitate	Improving
α-Tocopherol	Declining after improvement
γ/β-Tocopherol	Holding to slight improvement
Total β-Carotene	Holding to slight decline
Total α-Carotene	Holding
Total Lycopene	Holding to slight decline
trans-Lycopene	Holding
Total β-Cryptoxanthin	Holding to slight decline
Total Lutein	Holding to slight improvement
Total Zeaxanthin	Holding
Total Lutein&Zeaxanthin	Holding

We intend to investigate possible causes for the loss of α-tocopherol comparability in the coming year.


Total Retinol
Number of Participants
Expected Deviation
|Discordance|
Apparent Imprecision
Median Performance, SD96:99
Number Participants
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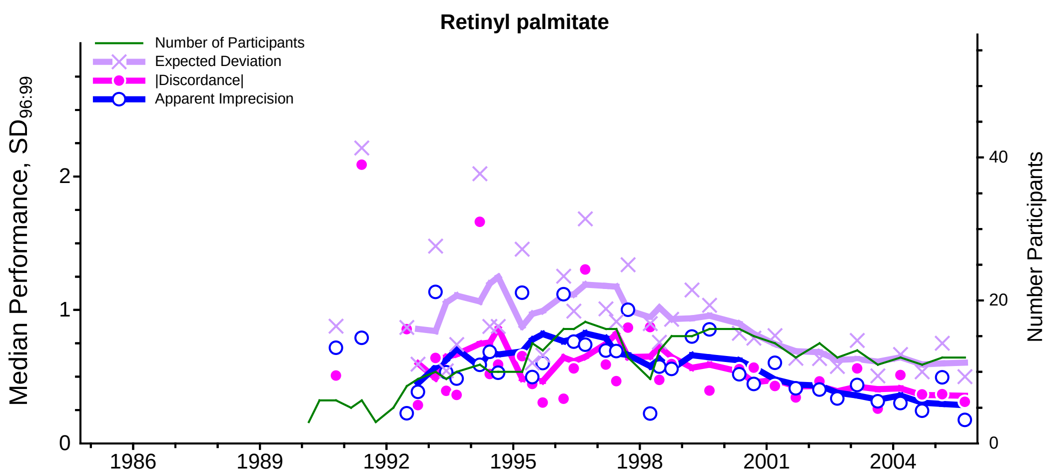
Retinyl palmitate
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Expected Deviation
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Apparent Imprecision
Median Performance, SD96:99
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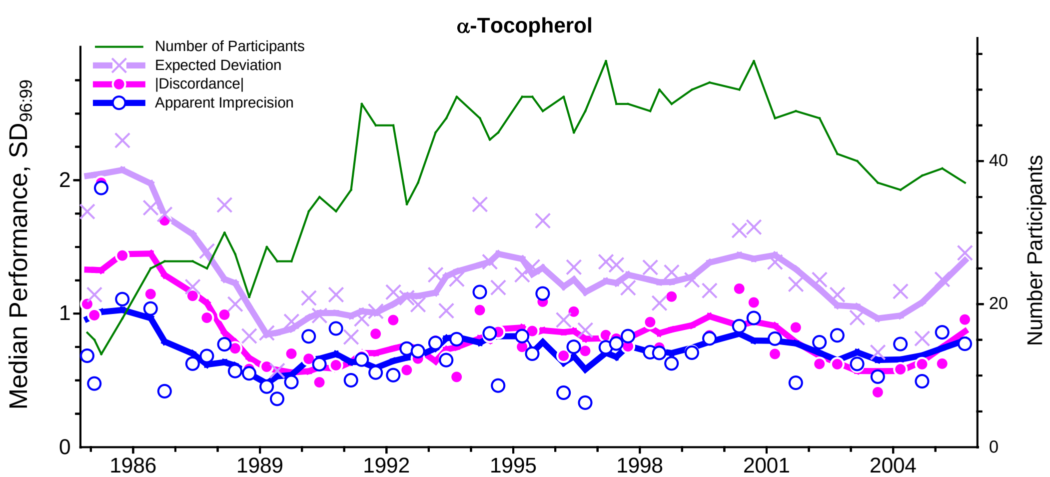
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Number of Participants
Expected Deviation
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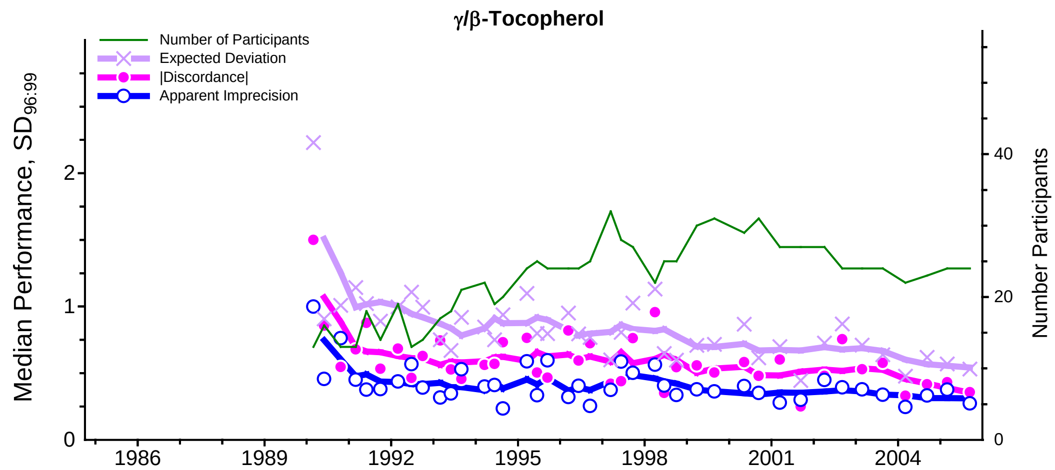
γ/β-Tocopherol
Number of Participants
Expected Deviation
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Apparent Imprecision
Median Performance, SD96:99
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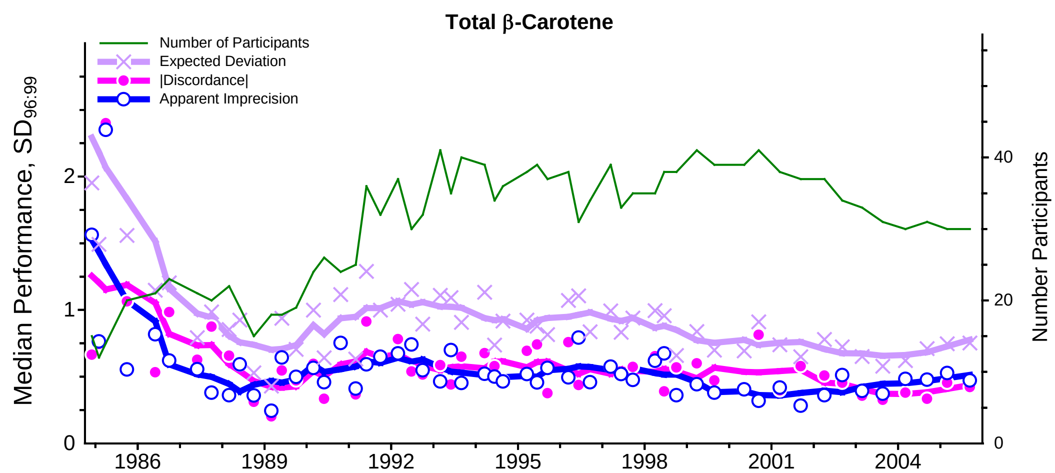
Total β-Carotene
Number of Participants
Expected Deviation
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Apparent Imprecision
Median Performance, SD96:99
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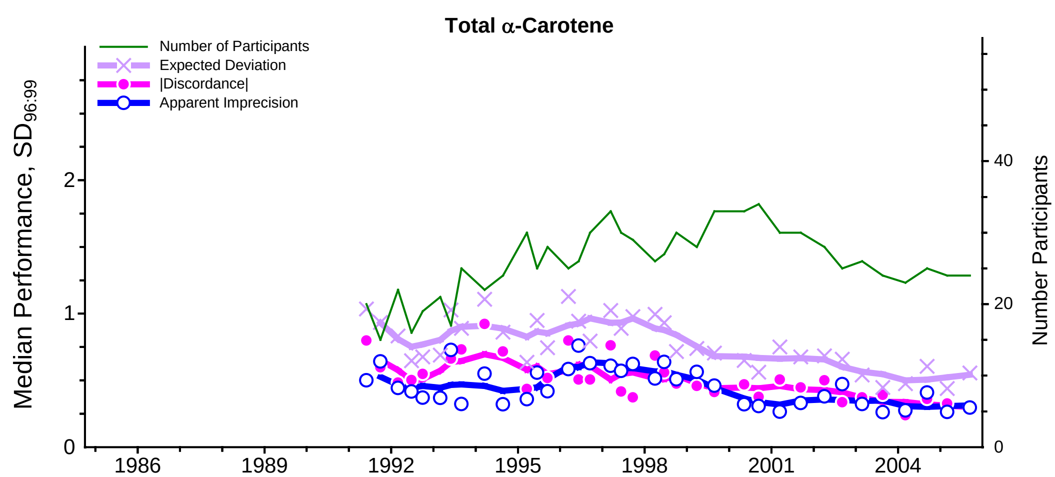
Total α-Carotene
Number of Participants
Expected Deviation
|Discordance|
Apparent Imprecision
Median Performance, SD96:99
Number Participants
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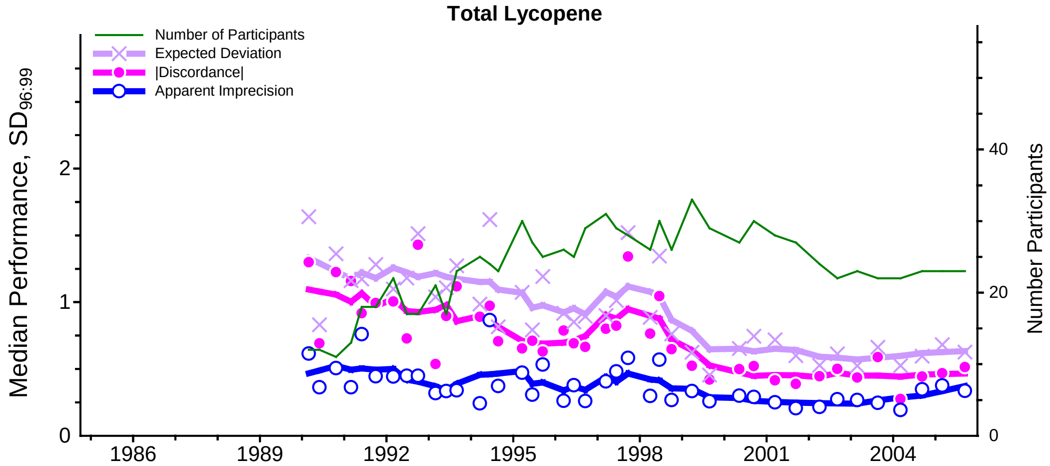
Total Lycopene
Number of Participants
Expected Deviation
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Apparent Imprecision
Median Performance, SD96:99
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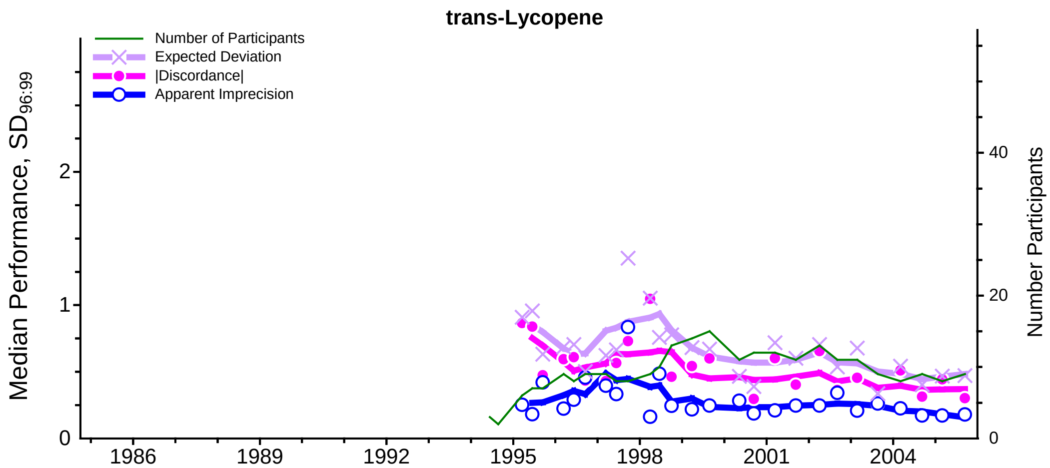
trans-Lycopene
Number of Participants
Expected Deviation
|Discordance|
Apparent Imprecision
Median Performance, SD96:99
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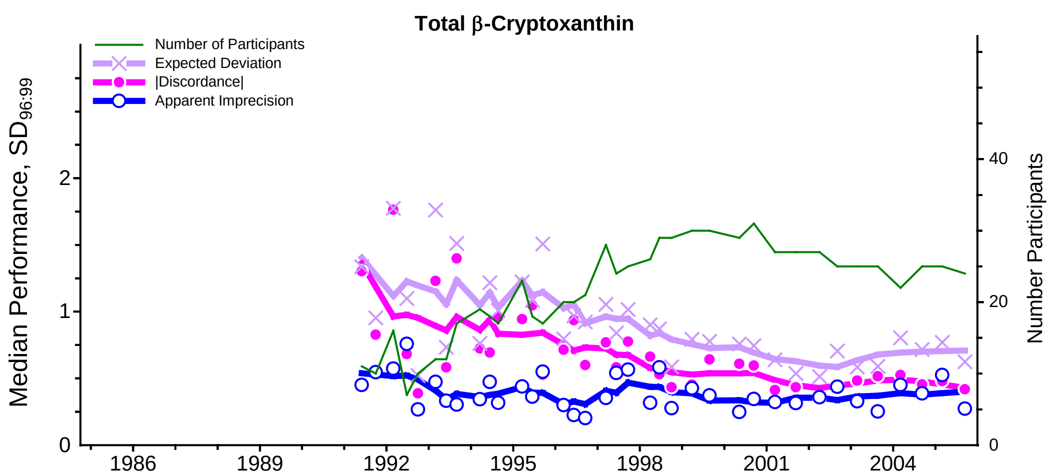
Total β-Cryptoxanthin
Number of Participants
Expected Deviation
|Discordance|
Apparent Imprecision
Median Performance, SD96:99
Number Participants
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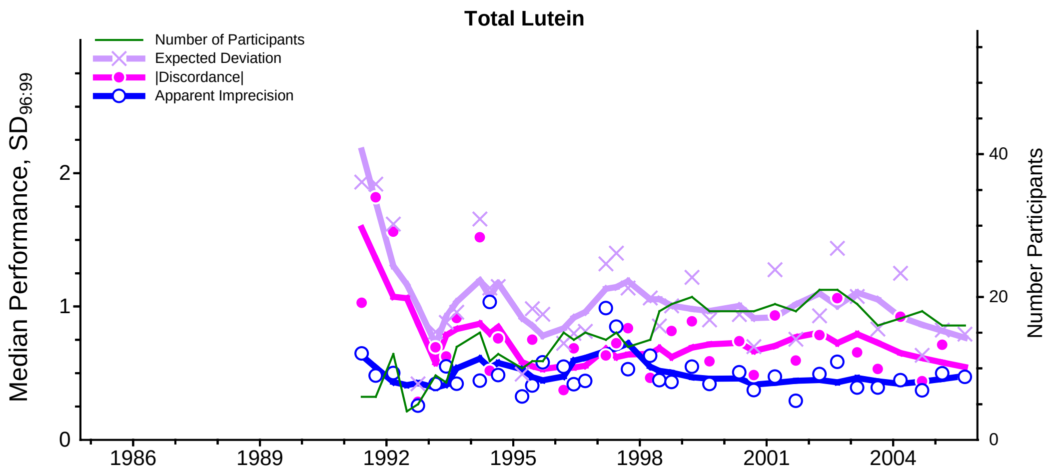
Total Lutein
Number of Participants
Expected Deviation
|Discordance|
Apparent Imprecision
Median Performance, $SD_{96:99}$
Number Participants
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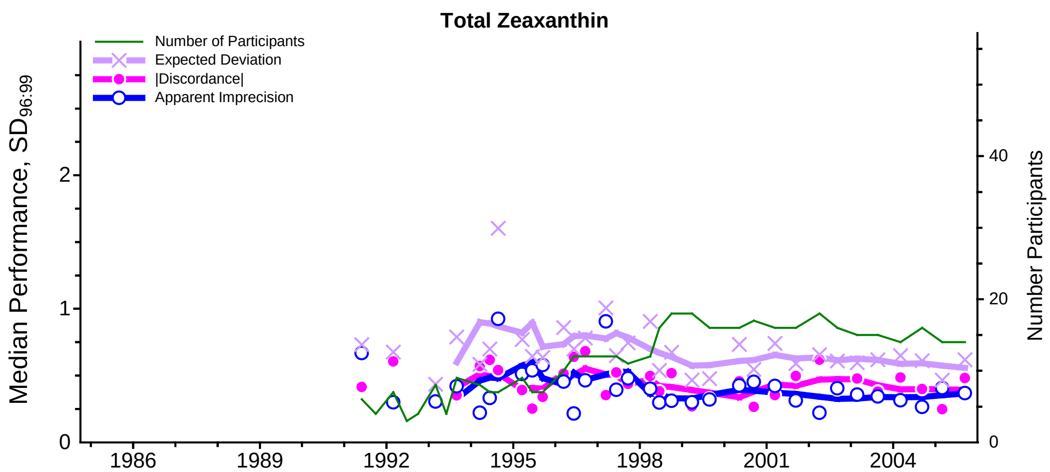
Total Zeaxanthin
Number of Participants
Expected Deviation
|Discordance|
Apparent Imprecision
Median Performance, $SD_{96:99}$
Number Participants
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40
1986
1989
1992
1995
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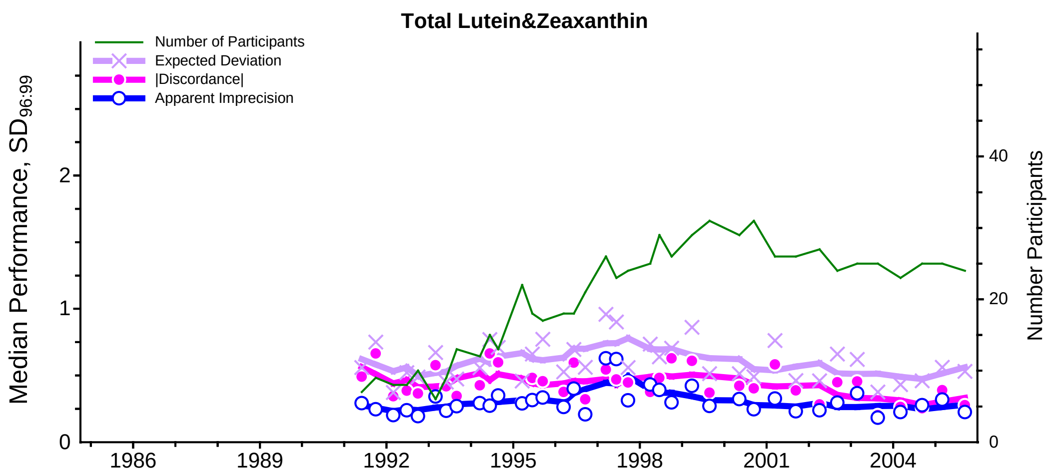
2004


Total Lutein&Zeaxanthin
Number of Participants
Expected Deviation
|Discordance|
Apparent Imprecision
Median Performance, $SD_{96:99}$
Number Participants
0
1
2
0
20
40
1986
1989
1992
1995
1998
2001
2004

4) Carrot oil observations/conclusions: We appreciate the effort that all of you who evaluated the "carrot oil" ampoules put into your analyses! About half of you provided some results or reported that no results could be obtained using available methods.

   We chose not to provide specific instructions for the analysis of this material to prevent biasing your results. We wanted to get an idea of the handling and measurement capabilities for this matrix in the hands of experienced analysts. Unsurprisingly, those of you who also participate in our "food" SRM value assignment studies reported more complete sets of results than those who do not. We were somewhat surprised that more of you did not investigate dissolving the material in ethanol (or whatever alcohol is most compatible with your chromatographic liquid phase). This simplistic approach generally has worked well for us.

   Also surprisingly, the among-participant variation for the carotenoids was considerably smaller than for the tocopherols. We believe much of the scatter in the tocopherol results arises from incomplete chromatographic (baseline) separation… and the unusual and unexpected isomer ratios: only a trace of α-tocopherol level is present while both the δ- and γ-tocopherols are about 400 µg/g. We strongly suspect that the carrier for the carrot-extract is ***not*** predominantly "sunflower oil" as stated by the supplier.

   We intend to design a second study of this material, both to better characterize the material and to evaluate its potential for use as a calibrant. If we do ask you to evaluate this material again, we will provide detailed instructions.

Appendix C. "All-Lab Report" for RR58

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

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

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

Round Robin LVIII Serum Results

All Values in µg/mL

Lab	Total Retinol 314	315	316	317	318	trans-Retinol 314	315	316	317	318
FSV-BA	0.541	0.491	0.384	0.700	0.872					
FSV-BB	0.533	0.486	0.396	0.709	0.868					
FSV-BC	0.565	0.497	0.409	0.697	0.875					
FSV-BD	0.460	0.420	0.360	0.610	0.780					
FSV-BE	0.538	0.481	0.384	0.680	0.880					
FSV-BF	0.534	,474	0.383	0.700	0.870					
FSV-BG	0.574	0.501	0.383	0.682	0.904					
FSV-BH	0.578	0.491	0.411	0.752	0.885					
FSV-BI	0.493	0.434	0.339	0.571	0.739					
FSV-BJ	0.527	0.477	0.391	0.672	0.836					
FSV-BK	0.643	0.547	0.405	0.675	0.888					
FSV-BL	0.610	0.550	0.440	0.750	1.000					
FSV-BM	0.573	0.486	0.407	0.628	0.894					
FSV-BN	0.558	0.507	0.408	0.724	0.926					
FSV-BO	0.565	0.469	0.387	0.631	0.833					
FSV-BP	0.560	0.480	0.360	0.620	0.800					
FSV-BQ	0.574	0.539	0.403	0.737	0.894					
FSV-BR	≥0.540	≥0.465	≥0.415	≥0.770	≥0.880	0.540	0.465	0.415	0.770	0.880
FSV-BS	≥0.384	≥0.320	≥0.225	≥0.529	≥0.671	0.384	0.320	0.225	0.529	0.671
FSV-BT	0.536	0.458	0.355	0.661	0.866					
FSV-BU	0.535	0.518	0.402	0.721	0.923					
FSV-BV	0.670	0.605	0.479	0.862	1.089					
FSV-BW	0.530	0.490	0.400	0.660	0.830					
FSV-BX	0.555	0.483	0.370	0.684	0.899					
FSV-CC	0.570	0.510	0.410	0.730	0.910	0.570	0.510	0.410	0.690	0.880
FSV-CD	≥0.572	≥0.505	≥0.394	≥0.672	≥0.842	0.572	0.505	0.394	0.672	0.842
FSV-CE	0.485	0.434	0.343	0.696	0.850					
FSV-CF	0.577	0.526	0.403	0.763	0.947					
FSV-CG	0.551	0.490	0.374	0.919	0.932					
FSV-CI	0.510	0.452	0.370	0.627	0.797					
FSV-CS	0.541	0.513	0.408	0.674	0.854					
FSV-CT										
FSV-CW	0.546	0.478	0.404	0.687	0.800					
FSV-CZ	0.520	0.480	0.380	0.660	0.850					
FSV-DA	0.537	0.471	0.374	0.644	0.827	0.537	0.471	0.374	0.644	0.827
FSV-DB	0.542	0.476	0.356	0.652	0.835					
FSV-DD	≥0.589	≥0.576	≥0.438	≥0.797	≥1.099	0.589	0.576	0.438	0.797	1.099
FSV-DF	0.543	0.492	0.393	0.706	0.889					
FSV-DI	0.568	0.517	0.386	0.674	0.858					
FSV-DV	1.210	1.100	0.870	1.530	1.940					
FSV-DW	0.520	0.490	0.380	0.650	0.810					
N	36	35	36	36	36	6	6	6	6	6
Min	0.460	0.420	0.339	0.571	0.739	0.384	0.320	0.225	0.529	0.671
Median	0.545	0.490	0.389	0.683	0.871	0.555	0.488	0.402	0.681	0.861
Max	1.210	1.100	0.870	1.530	1.940	0.589	0.576	0.438	0.797	1.099
SD	0.027	0.025	0.023	0.047	0.049					
CV	5	5	6	7	6	0	0	0	0	0
N_{past}	0	0	0	46	42	0	0	0	0	8
$Median_{past}$				0.662	0.858					0.839
SD_{past}				0.054	0.056					0.041
NIST	0.557	0.496	0.394	0.681	0.844					
N_{NIST}	2	2	2	2	2	2	2	2	2	2
S_{rep}	0.010	0.009	0.005	0.018	0.009					
S_{het}	0.003	0.008	0.001	0.033	0.010					
S_{NIST}	0.010	0.012	0.005	0.037	0.013					
NAV	0.551	0.493	0.391	0.682	0.858	0.540	0.471	0.410	0.690	0.880
NAU	0.044	0.040	0.032	0.054	0.070	0.043	0.038	0.033	0.054	0.069

Lab	Retinyl Palmitate 314	315	316	317	318	α-Tocopherol 314	315	316	317	318
FSV-BA	0.104	0.051	0.012	0.064	0.039	15.43	9.28	4.14	11.17	7.20
FSV-BB	0.100	0.045	0.009	0.049	0.024	16.47	9.29	4.18	12.10	7.71
FSV-BC										
FSV-BD						15.80	9.70	4.70	12.00	8.00
FSV-BE						17.70	10.00	4.40	13.10	8.40
FSV-BF						15.60	9.50	4.20	11.90	7.80
FSV-BG	0.103	0.056	0.024	0.049	0.041	17.07	9.80	5.16	12.65	8.89
FSV-BH						17.91	10.53	4.82	13.45	8.31
FSV-BI	0.132	0.058	*nd*	0.055	0.035	14.59	8.53	3.73	10.22	6.53
FSV-BJ	0.120	0.054	*nq*	0.057	0.037	17.04	10.03	4.41	12.42	7.91
FSV-BK						16.19	9.26	3.57	10.39	6.99
FSV-BL						14.21	7.75	3.88	10.77	7.32
FSV-BM						15.00	9.80	4.80	11.30	7.60
FSV-BN	0.093	0.044	0.007	0.048	0.025	14.36	8.71	3.92	11.16	7.09
FSV-BO						14.69	8.31	4.13	10.06	6.85
FSV-BP						10.58	6.93	2.92	12.82	7.51
FSV-BQ						13.30	8.00	3.30	9.50	6.10
FSV-BR						16.43	9.73	4.69	11.09	7.02
FSV-BS										
FSV-BT						15.86	9.78	4.49	10.56	7.41
FSV-BU						16.32	9.91	4.22	12.19	7.79
FSV-BV						16.59	9.90	4.37	11.71	8.15
FSV-BW	0.121	0.061	*nq*	0.066	0.041	14.08	8.77	4.10	10.98	6.95
FSV-BX						16.57	9.35	4.02	12.10	7.82
FSV-CC						14.50	8.84	4.07	11.08	6.91
FSV-CD	0.139	0.072	0.012	0.058	0.027	17.13	9.76	4.18	12.62	7.28
FSV-CE						16.62	9.76	4.27	10.83	7.66
FSV-CF						14.70	9.40	5.20	11.70	7.80
FSV-CG						13.92	8.14	2.19	10.97	6.36
FSV-CI	0.109	0.052	<0.01	0.054	0.033	15.38	9.37	4.18	11.91	7.24
FSV-CS						17.34	10.38	5.03	12.56	8.70
FSV-CT										
FSV-CW	0.112	0.053	0.024	0.056	0.034	19.80	9.25	2.73	12.36	6.85
FSV-CZ						14.10	8.70	4.10	11.40	7.70
FSV-DA	0.091	0.040	0.004	0.040	0.021	16.24	9.62	4.35	11.68	7.31
FSV-DB						16.61	9.70	4.03	11.48	7.57
FSV-DD										
FSV-DF										
FSV-DI	0.100	0.050	0.012	0.053	0.031	16.15	9.63	4.18	12.00	7.37
FSV-DV						15.10	8.70	4.00	8.10	5.00
FSV-DW						22.39	11.36	5.40	12.84	7.66
N	12	12	8	12	12	36	36	36	36	36
Min	0.091	0.040	0.004	0.040	0.021	10.58	6.93	2.19	8.10	5.00
Median	0.107	0.053	0.012	0.055	0.034	16.00	9.45	4.18	11.69	7.46
Max	0.139	0.072	0.024	0.066	0.041	22.39	11.36	5.40	13.45	8.89
SD	0.015	0.006	0.005	0.006	0.008	1.44	0.76	0.30	0.93	0.58
CV	14	11	42	11	25	9	8	7	8	8
N_{past}	0	0	0	14	13	0	0	0	46	45
$Median_{past}$				0.049	0.034				11.55	7.49
SD_{past}				0.020	0.011				1.05	0.66
NIST						15.99	9.42	3.98	12.21	7.52
N_{NIST}						2	2	2	2	2
S_{rep}						0.21	0.21	0.07	0.12	0.03
S_{het}						0.11	0.05	0.02	0.12	0.15
S_{NIST}						0.24	0.22	0.07	0.16	0.15
NAV	0.104	0.052	0.012	0.054	0.034	16.07	9.41	4.08	11.95	7.54
NAU	0.027	0.017	0.011	0.017	0.013	1.47	0.79	0.46	0.99	0.62

Lab	γ/β-Tocopherol 314	315	316	317	318	δ-Tocopherol 314	315	316	317	318
FSV-BA	1.50	1.84	1.75	2.69	3.94	0.109	0.113	0.098	0.342	0.120
FSV-BB	1.32	1.65	1.63	2.61	3.68	0.095	0.103	0.093	0.331	0.122
FSV-BC										
FSV-BD										
FSV-BE	1.40	1.70	1.70	2.80	4.20					
FSV-BF	1.39	1.81	1.77	2.75	4.16					
FSV-BG	1.57	1.74	1.75	2.75	4.00					
FSV-BH	2.06	2.43	2.37	3.70	5.34					
FSV-BI	1.48	1.71	1.69	2.47	3.53					
FSV-BJ	1.40	1.72	1.68	2.65	3.78					
FSV-BK										
FSV-BL										
FSV-BM										
FSV-BN	1.18	1.46	1.44	2.26	3.30	0.133	0.116	0.132	0.312	0.143
FSV-BO										
FSV-BP										
FSV-BQ										
FSV-BR										
FSV-BS	1.15	1.45	1.23	2.39	5.71					
FSV-BT	1.50	1.80	1.73	2.59	3.71					
FSV-BU	1.40	1.67	1.67	2.61	3.83					
FSV-BV	1.55	1.89	1.83	2.76	4.27					
FSV-BW	1.69	2.13	2.20	3.25	4.78					
FSV-BX	1.58	1.89	1.74	2.75	4.07					
FSV-CC										
FSV-CD	1.41	1.70	1.59	2.50	3.68					
FSV-CE										
FSV-CF										
FSV-CG	1.44	1.74	1.67	2.87	3.85	0.153	0.128	0.124	0.411	0.124
FSV-CI	1.55	1.98	1.73	2.83	3.86					
FSV-CS	1.98	2.28	2.42	2.94	4.23					
FSV-CT										
FSV-CW	1.65	1.79	1.75	2.74	3.90	0.101	0.115	0.086	0.419	0.108
FSV-CZ										
FSV-DA	1.42	1.81	1.76	2.61	3.88	0.074	0.115	0.100	0.276	0.095
FSV-DB	1.75	2.01	1.72	2.70	3.83	*nq*	*nq*	*nq*	0.156	*nq*
FSV-DD										
FSV-DF										
FSV-DI	1.31	1.66	1.61	2.51	3.67	0.102	0.114	0.104	0.370	0.130
FSV-DV										
FSV-DW										
N	23	23	23	23	23	7	7	7	8	7
Min	1.15	1.45	1.23	2.26	3.30	0.074	0.103	0.086	0.156	0.095
Median	1.48	1.79	1.73	2.70	3.88	0.102	0.115	0.100	0.337	0.122
Max	2.06	2.43	2.42	3.70	5.71	0.153	0.128	0.132	0.419	0.143
SD	0.13	0.14	0.09	0.14	0.32	0.017	0.006	0.014	0.057	0.010
CV	9	8	5	5	8	17	5	14	17	8
N_{past}	0	0	0	23	25	0	0	0	4	6
$Median_{past}$				2.76	3.83				0.402	0.145
SD_{past}				0.22	0.34					0.047
NIST	1.43	1.82	1.72	2.73	4.00	0.105	0.107	0.102	0.331	0.130
N_{NIST}	2	2	2	2	2	2	2	2	2	2
S_{rep}	0.02	0.03	0.03	0.05	0.05	0.010	0.001	0.008	0.003	0.000
S_{het}	0.01	0.01	0.03	0.02	0.08	0.006	0.003	0.008	0.010	0.003
S_{NIST}	0.03	0.03	0.04	0.06	0.09	0.012	0.003	0.011	0.010	0.003
NAV	1.46	1.81	1.73	2.73	3.94	0.103	0.111	0.101	0.334	0.126
NAU	0.17	0.19	0.19	0.27	0.36	0.024	0.026	0.024	0.057	0.027

Round Robin LVIII Serum Results

All Values in µg/mL

Lab	Total β-Carotene 314	315	316	317	318	trans-β-Carotene 314	315	316	317	318
FSV-BA	0.604	0.279	0.040	0.314	0.173	0.564	0.260	0.037	0.291	0.161
FSV-BB	0.601	0.282	0.042	0.318	0.167	0.569	0.267	0.039	0.300	0.157
FSV-BC										
FSV-BD										
FSV-BE	0.631	0.281	0.040	0.350	0.192					
FSV-BF	0.654	0.317	0.034	0.373	0.186					
FSV-BG	0.736	0.355	0.079	0.425	0.195					
FSV-BH	0.685	0.313	0.039	0.370	0.190	0.653	0.295	0.039	0.348	0.177
FSV-BI	0.558	0.244	0.036	0.281	0.153					
FSV-BJ	0.593	0.267	0.037	0.319	0.161					
FSV-BK										
FSV-BL										
FSV-BM										
FSV-BN	0.558	0.262	0.042	0.323	0.163	0.517	0.242	0.037	0.293	0.150
FSV-BO	0.558	0.212	0.043	0.276	0.139					
FSV-BP	0.279	0.350	*nq*	0.330	0.170					
FSV-BQ										
FSV-BR										
FSV-BS	≥0.621	≥0.284	≥0.064	≥0.343	≥0.202	0.621	0.284	0.064	0.343	0.202
FSV-BT	0.606	0.304	0.044	0.351	0.196	0.564	0.284	0.042	0.326	0.183
FSV-BU	0.529	0.269	0.067	0.299	0.174					
FSV-BV	0.661	0.303	0.041	0.341	0.191					
FSV-BW	0.580	0.275	0.036	0.301	0.151					
FSV-BX	≥0.569	≥0.258	≥0.034	≥0.302	≥0.161	0.569	0.258	0.034	0.302	0.161
FSV-CC										
FSV-CD	0.729	0.298	0.045	0.337	0.169					
FSV-CE	0.684	0.310	0.050	0.292	0.171					
FSV-CF										
FSV-CG	0.571	0.260	0.035	0.319	0.159	0.530	0.241	0.032	0.296	0.147
FSV-CI	≥0.613	≥0.269	≥0.039	≥0.285	≥0.162	0.613	0.269	0.039	0.285	0.162
FSV-CS	0.701	0.365	0.093	0.389	0.219	0.645	0.335	0.082	0.357	0.200
FSV-CT	1.034	0.444	0.060	0.491	0.273					
FSV-CW	≥0.632	≥0.291	≥0.023	≥0.330	≥0.152	0.632	0.291	0.023	0.330	0.152
FSV-CZ	0.504	0.260	0.080	0.296	0.180					
FSV-DA	0.794	0.352	0.050	0.340	0.177	0.738	0.326	0.045	0.308	0.158
FSV-DB	0.531	0.279	0.033	0.267	0.137					
FSV-DD										
FSV-DF										
FSV-DI	0.600	0.285	0.040	0.335	0.185					
FSV-DV										
FSV-DW	0.806	0.287	0.006	0.294	0.130					
N	25	25	24	25	25	12	12	12	12	12
Min	0.279	0.212	0.006	0.267	0.130	0.517	0.241	0.023	0.285	0.147
Median	0.604	0.285	0.042	0.323	0.173	0.591	0.277	0.039	0.305	0.161
Max	1.034	0.444	0.093	0.491	0.273	0.738	0.335	0.082	0.357	0.202
SD	0.094	0.033	0.010	0.038	0.022	0.053	0.024	0.005	0.028	0.017
CV	16	11	24	12	12	9	9	12	9	10
N_{past}	0	0	0	31	30	0	0	0	14	14
$Median_{past}$				0.317	0.173				0.298	0.160
SD_{past}				0.047	0.022				0.039	0.017
NIST	0.599	0.307	*nq*	0.315	0.172	0.509	0.256	*nq*	0.272	0.162
N_{NIST}	2	2		2	2	2	2		2	2
S_{rep}	0.016	0.017		0.013	0.003	0.024	0.009		0.014	0.005
S_{het}	0.003	0.014		0.007	0.005	0.005	0.000		0.001	0.002
S_{NIST}	0.016	0.022		0.015	0.005	0.024	0.009		0.014	0.005
NAV	0.601	0.295	0.041	0.318	0.173	0.550	0.266	0.039	0.289	0.162
NAU	0.094	0.045	0.010	0.047	0.027	0.085	0.033	0.008	0.040	0.019

Lab	Total cis-β-Carotene 314	315	316	317	318	Total α-Carotene 314	315	316	317	318
FSV-BA	0.040	0.018	0.003	0.022	0.013	0.127	0.057	0.006	0.056	0.015
FSV-BB	0.032	0.015	0.003	0.018	0.011	0.109	0.051	0.005	0.051	0.011
FSV-BC										
FSV-BD										
FSV-BE										
FSV-BF						0.127	0.059	0.008	0.052	0.016
FSV-BG						0.120	0.062	0.013	0.057	0.018
FSV-BH	0.032	0.018	*nq*	0.023	0.013	0.122	0.053	*nq*	0.053	*nq*
FSV-BI						0.120	0.052	0.004	0.049	0.018
FSV-BJ						0.119	0.052	0.010	0.052	0.012
FSV-BK										
FSV-BL										
FSV-BM										
FSV-BN	0.044	0.023	0.008	0.033	0.016	0.119	0.055	0.010	0.057	0.017
FSV-BO						0.132	0.057	*nq*	0.056	0.010
FSV-BP						0.112	0.029	*nq*	0.030	*nq*
FSV-BQ										
FSV-BR										
FSV-BS						0.138	0.076	0.033	0.079	0.044
FSV-BT	0.044	0.020	0.003	0.026	0.013	0.121	0.063	0.008	0.061	0.022
FSV-BU						0.110	0.052	0.009	0.049	0.013
FSV-BV						0.096	0.042	0.004	0.040	0.010
FSV-BW						0.165	0.070	*nq*	0.056	*nq*
FSV-BX						0.125	0.055	0.009	0.054	0.013
FSV-CC										
FSV-CD						0.163	0.073	0.014	0.070	0.022
FSV-CE										
FSV-CF										
FSV-CG	0.041	0.019	0.003	0.023	0.012	0.145	0.065	0.006	0.069	0.018
FSV-CI						0.132	0.060	<0.009	0.057	0.024
FSV-CS	0.056	0.029	0.011	0.032	0.019	0.135	0.065	0.008	0.063	0.017
FSV-CT										
FSV-CW						0.143	0.069	0.011	0.067	0.022
FSV-CZ										
FSV-DA	0.057	0.026	0.006	0.032	0.019	0.124	0.054	0.006	0.049	0.014
FSV-DB										
FSV-DD										
FSV-DF										
FSV-DI										
FSV-DV										
FSV-DW						0.148	0.056	0.011	0.048	0.016
N	8	8	7	8	8	23	23	18	23	20
Min	0.032	0.015	0.003	0.018	0.011	0.096	0.029	0.004	0.030	0.010
Median	0.043	0.020	0.003	0.025	0.013	0.125	0.057	0.008	0.056	0.017
Max	0.057	0.029	0.011	0.033	0.019	0.165	0.076	0.033	0.079	0.044
SD	0.007	0.004	0.003	0.007	0.003	0.012	0.008	0.004	0.007	0.004
CV	16	21	100	27	23	10	15	42	12	26
N_{past}	0	0	0	10	9	0	0	0	26	26
$Median_{past}$				0.024	0.014				0.053	0.016
SD_{past}				0.008	0.006				0.015	0.004
NIST	0.090	0.052	*nq*	0.042	0.010	0.118	0.058	*nq*	0.059	*nq*
N_{NIST}	2	2		2	2	2	2		2	
S_{rep}	0.020	0.022		0.003	0.003	0.005	0.005		0.005	
S_{het}	0.008	0.014		0.005	0.007	0.001	0.005		0.000	
S_{NIST}	0.021	0.026		0.006	0.007	0.005	0.007		0.005	
NAV	0.066	0.036	0.003	0.033	0.011	0.121	0.057	0.008	0.057	0.016
NAU	0.040	0.035	0.003	0.015	0.008	0.035	0.017	0.004	0.017	0.006

Lab	Total Lycopene 314	315	316	317	318	trans-Lycopene 314	315	316	317	318
FSV-BA	0.379	0.274	0.165	0.425	0.360	0.201	0.138	0.076	0.203	0.179
FSV-BB	0.331	0.243	0.150	0.372	0.309	0.166	0.113	0.062	0.162	0.140
FSV-BC										
FSV-BD										
FSV-BE										
FSV-BF	0.331	0.256	0.150	0.405	0.371					
FSV-BG	0.411	0.340	0.197	0.499	0.405	0.234	0.172	0.103	0.252	0.207
FSV-BH	0.276	0.186	0.114	0.291	0.246					
FSV-BI	0.285	0.189	0.121	0.280	0.244					
FSV-BJ	0.324	0.228	0.146	0.387	0.319					
FSV-BK										
FSV-BL										
FSV-BM										
FSV-BN	0.289	0.227	0.134	0.335	0.294	0.154	0.112	0.069	0.174	0.154
FSV-BO	0.333	0.204	0.173	0.351	0.296					
FSV-BP	0.403	0.375	0.217	0.420	0.292					
FSV-BQ										
FSV-BR										
FSV-BS	0.497	0.359	0.257	0.538	0.493					
FSV-BT	0.328	0.250	0.153	0.375	0.370	0.161	0.124	0.076	0.184	0.186
FSV-BU	0.352	0.272	0.189	0.408	0.354					
FSV-BV	0.414	0.291	0.167	0.445	0.405					
FSV-BW	0.288	0.206	0.125	0.324	0.273					
FSV-BX						0.148	0.098	0.055	0.150	0.139
FSV-CC										
FSV-CD	0.526	0.319	0.150	0.451	0.336					
FSV-CE										
FSV-CF										
FSV-CG	0.342	0.238	0.128	0.386	0.313	0.189	0.128	0.066	0.197	0.166
FSV-CI										
FSV-CS	0.428	0.322	0.202	0.500	0.456					
FSV-CT	0.369	0.244	0.120	0.335	0.318					
FSV-CW						0.149	0.102	0.067	0.157	0.132
FSV-CZ										
FSV-DA	0.336	0.232	0.144	0.315	0.273	0.189	0.122	0.072	0.155	0.138
FSV-DB	0.297	0.232	0.138	0.313	0.275					
FSV-DD										
FSV-DF										
FSV-DI	0.300	0.230	0.160	0.325	0.300					
FSV-DV										
FSV-DW	0.349	0.217	0.157	0.292	0.247					
N	23	23	23	23	23	9	9	9	9	9
Min	0.276	0.186	0.114	0.280	0.244	0.148	0.098	0.055	0.150	0.132
Median	0.336	0.243	0.150	0.375	0.313	0.166	0.122	0.069	0.174	0.154
Max	0.526	0.375	0.257	0.538	0.493	0.234	0.172	0.103	0.252	0.207
SD	0.059	0.041	0.025	0.073	0.061	0.026	0.012	0.007	0.030	0.030
CV	17	17	17	19	19	15	10	11	17	19
N_{past}	0	0	0	25	25	0	0	0	12	12
$Median_{past}$				0.367	0.307				0.177	0.158
SD_{past}				0.088	0.051				0.036	0.026
NAV	0.335	0.241	0.152	0.374	0.311	0.166	0.122	0.069	0.174	0.154
NAU	0.075	0.057	0.039	0.082	0.070	0.029	0.021	0.012	0.031	0.030

Round Robin LVIII Serum Results

All Values in µg/mL

Lab	Total β-Cryptoxanthin 314	315	316	317	318	Total α-Cryptoxanthin 314	315	316	317	318
FSV-BA	0.063	0.053	0.041	0.065	0.085	0.027	0.018	0.012	0.023	0.027
FSV-BB	0.049	0.043	0.032	0.052	0.065	0.016	0.012	0.008	0.016	0.017
FSV-BC										
FSV-BD										
FSV-BE										
FSV-BF	0.048	0.045	0.036	0.055	0.078					
FSV-BG	0.050	0.035	0.023	0.042	0.068					
FSV-BH	0.093	0.071	0.047	0.083	0.099					
FSV-BI	0.048	0.042	0.033	0.046	0.065					
FSV-BJ	0.045	0.040	0.035	0.053	0.070					
FSV-BK										
FSV-BL										
FSV-BM										
FSV-BN	0.050	0.045	0.036	0.057	0.076	0.019	0.015	0.011	0.021	0.024
FSV-BO	0.053	0.041	0.036	0.053	0.065					
FSV-BP	0.057	0.056	0.020	0.110	0.072					
FSV-BQ										
FSV-BR										
FSV-BS	0.068	0.055	0.042	0.076	0.074					
FSV-BT	0.052	0.044	0.032	0.055	0.074	0.020	0.013	0.007	0.018	0.019
FSV-BU	0.051	0.048	0.040	0.055	0.074					
FSV-BV	0.045	0.041	0.032	0.047	0.067					
FSV-BW	0.048	0.035	0.025	0.040	0.053					
FSV-BX	0.045	0.039	0.029	0.044	0.062					
FSV-CC										
FSV-CD	0.064	0.044	0.030	0.058	0.066					
FSV-CE										
FSV-CF										
FSV-CG	0.074	0.061	0.043	0.078	0.093					
FSV-CI										
FSV-CS	0.060	0.051	0.035	0.061	0.076	0.017	0.015	0.009	0.021	0.022
FSV-CT	0.050	0.040	0.022	0.047	0.058					
FSV-CW	0.056	0.045	0.035	0.059	0.069					
FSV-CZ										
FSV-DA	0.067	0.054	0.037	0.059	0.073	0.022	0.015	0.009	0.018	0.020
FSV-DB	0.061	0.047	0.031	0.039	0.070					
FSV-DD										
FSV-DF										
FSV-DI										
FSV-DV										
FSV-DW	0.082	0.064	0.059	0.067	0.083					
N	24	24	24	24	24	6	6	6	6	6
Min	0.045	0.035	0.020	0.039	0.053	0.016	0.012	0.007	0.016	0.017
Median	0.052	0.045	0.035	0.055	0.071	0.020	0.015	0.009	0.020	0.021
Max	0.093	0.071	0.059	0.110	0.099	0.027	0.018	0.012	0.023	0.027
SD	0.011	0.009	0.005	0.011	0.008	0.003	0.001	0.002	0.002	0.003
CV	20	20	14	21	11	13	9	20	11	14
N_{past}	0	0	0	27	26	0	0	0	6	5
$Median_{past}$				0.058	0.073				0.018	0.021
SD_{past}				0.010	0.012				0.002	0.005
NIST										
N_{NIST}	2	2	2	2	2					
S_{rep}										
S_{het}										
S_{NIST}										
NAV	0.052	0.045	0.035	0.055	0.072	0.020	0.015	0.009	0.020	0.021
NAU	0.013	0.011	0.009	0.013	0.017	0.003	0.001	0.002	0.002	0.003

Lab	Total Lutein 314	315	316	317	318	Total Zeaxanthin 314	315	316	317	318
FSV-BA										
FSV-BB	0.113	0.066	0.025	0.078	0.056	0.028	0.025	0.024	0.046	0.047
FSV-BC										
FSV-BD										
FSV-BE										
FSV-BF										
FSV-BG	0.126	0.085	0.039	0.097	0.066	0.037	0.029	0.018	0.036	0.030
FSV-BH	0.101	0.055	0.015	0.054	0.041	0.024	0.017	0.013	0.028	0.032
FSV-BI	0.100	0.056	0.017	0.055	0.042	0.020	0.018	0.014	0.023	0.027
FSV-BJ	0.118	0.068	0.026	0.073	0.062					
FSV-BK										
FSV-BL										
FSV-BM										
FSV-BN	0.113	0.065	0.028	0.070	0.052	0.040	0.025	0.015	0.038	0.036
FSV-BO	0.123	0.056	*nq*	0.057	0.043	0.049	*nd*	0.015	0.025	0.040
FSV-BP										
FSV-BQ										
FSV-BR										
FSV-BS										
FSV-BT	0.078	0.051	0.023	0.063	0.063	0.031	0.016	0.008	0.020	0.022
FSV-BU										
FSV-BV										
FSV-BW										
FSV-BX	0.136	0.066	0.030	0.076	0.073	0.035	0.027	0.022	0.033	0.191
FSV-CC										
FSV-CD										
FSV-CE										
FSV-CF										
FSV-CG										
FSV-CI	0.102	0.063	0.035	0.061	0.055	0.030	0.022	0.019	0.026	0.032
FSV-CS	0.111	0.065	0.024	0.067	0.050	0.022	0.020	0.017	0.023	0.026
FSV-CT	0.111	0.054	*nq*	0.053	0.044	*nq*	*nq*	*nq*	0.016	0.019
FSV-CW	0.114	0.065	0.025	0.071	0.052	0.024	0.021	0.017	0.027	0.027
FSV-CZ										
FSV-DA	0.114	0.067	0.028	0.075	0.058	0.041	0.030	0.021	0.033	0.037
FSV-DB										
FSV-DD										
FSV-DF										
FSV-DI	0.105	0.057	0.017	0.058	0.050					
FSV-DV										
FSV-DW	0.113	0.037	0.015	0.032	0.014	0.101	0.084	0.080	0.092	0.089
N	16	16	14	16	16	13	12	13	14	14
Min	0.078	0.037	0.015	0.032	0.014	0.020	0.016	0.008	0.016	0.019
Median	0.113	0.064	0.025	0.065	0.052	0.031	0.023	0.017	0.028	0.032
Max	0.136	0.085	0.039	0.097	0.073	0.101	0.084	0.080	0.092	0.191
SD	0.008	0.008	0.007	0.013	0.011	0.012	0.006	0.004	0.009	0.009
CV	7	12	28	20	22	38	24	25	32	28
N_{past}	0	0	0	16	17	0	0	0	15	15
$Median_{past}$				0.071	0.055				0.026	0.032
SD_{past}				0.016	0.013				0.009	0.009
NIST										
N_{NIST}	2	2	2	2	2	2	2	2	2	2
S_{rep}										
S_{het}										
S_{NIST}										
NAV	0.113	0.064	0.025	0.065	0.052	0.031	0.023	0.017	0.028	0.032
NAU	0.021	0.013	0.007	0.013	0.011	0.012	0.007	0.005	0.009	0.009

Lab	Total Lutein&Zeaxanthin 314	315	316	317	318	Coenzyme Q10 314	315	316	317	318
FSV-BA	0.149	0.090	0.036	0.104	0.087					
FSV-BB	0.141	0.091	0.049	0.124	0.103					
FSV-BC										
FSV-BD										
FSV-BE										
FSV-BF	0.140	0.085	0.034	0.100	0.088					
FSV-BG	0.142	0.096	0.047	0.111	0.077					
FSV-BH	0.125	0.072	0.028	0.082	0.073					
FSV-BI	*0.120*	*0.074*	*0.031*	*0.078*	*0.069*					
FSV-BJ						0.612	0.656	0.535	0.817	0.599
FSV-BK										
FSV-BL										
FSV-BM										
FSV-BN	0.153	0.090	0.043	0.108	0.088					
FSV-BO	0.172	0.056	*nq*	0.081	0.083					
FSV-BP	0.230	0.120	0.030	0.160	0.083					
FSV-BQ										
FSV-BR										
FSV-BS	0.140	0.087	0.047	0.097	0.085					
FSV-BT	0.115	0.069	0.033	0.086	0.089					
FSV-BU	0.155	0.085	0.037	0.098	0.091					
FSV-BV	0.145	0.086	0.037	0.093	0.089					
FSV-BW	0.124	0.067	0.027	0.062	0.059	0.049	0.521	0.608	0.735	0.580
FSV-BX	0.171	0.093	0.052	0.109	0.264					
FSV-CC										
FSV-CD	0.167	0.092	0.036	0.104	0.092					
FSV-CE										
FSV-CF										
FSV-CG	0.184	0.110	0.048	0.132	0.117					
FSV-CI	*0.132*	*0.085*	*0.054*	*0.087*	*0.087*					
FSV-CS	*0.132*	*0.085*	*0.041*	*0.090*	*0.076*					
FSV-CT	*≥0.111*	*≥0.054*	*nq*	*0.069*	*0.063*					
FSV-CW	*0.138*	*0.086*	*0.042*	*0.098*	*0.079*	0.656	0.819	0.696	0.935	0.747
FSV-CZ						0.610	0.530	0.460	0.650	0.640
FSV-DA	0.155	0.097	0.048	0.108	0.095	0.850	0.690	0.440	1.080	1.110
FSV-DB	0.159	0.092	0.038	0.099	0.091					
FSV-DD										
FSV-DF										
FSV-DI						0.560	0.580	0.570	0.720	0.600
FSV-DV										
FSV-DW	*0.214*	*0.121*	*0.095*	*0.124*	*0.103*					
N	23	23	22	24	24	6	6	6	6	6
Min	0.115	0.056	0.027	0.062	0.059	0.049	0.521	0.440	0.650	0.580
Median	0.145	0.087	0.039	0.098	0.087	0.611	0.618	0.553	0.776	0.620
Max	0.230	0.121	0.095	0.160	0.264	0.850	0.819	0.696	1.080	1.110
SD	0.021	0.006	0.010	0.016	0.010	0.054	0.103	0.089	0.135	0.090
CV	14	7	25	16	11	9	17	16	17	14
N_{past}	0	0	0	24	25	0	0	0	0	6
$Median_{past}$				0.102	0.092					0.630
SD_{past}				0.032	0.017					0.246
NIST										
N_{NIST}										
S_{rep}										
S_{het}										
S_{NIST}										
NAV	0.144	0.087	0.041	0.098	0.087	0.611	0.618	0.553	0.776	0.620
NAU	0.030	0.018	0.010	0.020	0.018	0.054	0.103	0.089	0.135	0.090

Round Robin LVIII Serum Results
All Values in µg/mL

Lab	Ubiquinol 314	Ubiquinol 315	Ubiquinol 316	Ubiquinol 317	Ubiquinol 318	Ubiquinone 314	Ubiquinone 315	Ubiquinone 316	Ubiquinone 317	Ubiquinone 318	Phylloquinone (K1) x1000 314	Phylloquinone (K1) x1000 315	Phylloquinone (K1) x1000 316	Phylloquinone (K1) x1000 317	Phylloquinone (K1) x1000 318
FSV-BA															
FSV-BB															
FSV-BC															
FSV-BD															
FSV-BE											1.571	0.774	0.154	1.116	1.063
FSV-BF															
FSV-BG															
FSV-BH															
FSV-BI															
FSV-BJ															
FSV-BK															
FSV-BL															
FSV-BM															
FSV-BN															
FSV-BO															
FSV-BP															
FSV-BQ															
FSV-BR															
FSV-BS															
FSV-BT															
FSV-BU															
FSV-BV															
FSV-BW	*nd*	0.195	0.488	0.472	0.452	0.049	0.326	0.120	0.263	0.127					
FSV-BX															
FSV-CC															
FSV-CD															
FSV-CE															
FSV-CF															
FSV-CG															
FSV-CI											1.180	0.520	0.100	0.770	0.670
FSV-CS															
FSV-CT															
FSV-CW															
FSV-CZ															
FSV-DA	0.850	0.690	0.440	1.080	1.110										
FSV-DB															
FSV-DD															
FSV-DF															
FSV-DI	0.150	0.370	0.540	0.585	0.600	0.310	0.220	0.050	0.160	0.105	0.890	0.490	0.220	0.730	0.740
FSV-DV															
FSV-DW															
N	2	3	3	3	3	2	2	2	2	2	3	3	3	3	3
Min	0.150	0.195	0.440	0.472	0.452	0.049	0.220	0.050	0.160	0.105	0.890	0.490	0.100	0.730	0.670
Median	0.500	0.370	0.488	0.585	0.600	0.180	0.273	0.085	0.212	0.116	1.180	0.520	0.154	0.770	0.740
Max	0.850	0.690	0.540	1.080	1.110	0.310	0.326	0.120	0.263	0.127	1.571	0.774	0.220	1.116	1.063
SD															
CV															
N_{past}	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
$Median_{past}$															
SD_{past}															
NIST															
N_{NIST}															
S_{rep}															
S_{het}															
S_{NIST}															
NAV		0.370	0.488	0.585	0.600						1.180	0.520	0.154	0.770	0.740
NAU															

Round Robin LVIII Serum Results
All Values in µg/mL

Analytes Reported By One Laboratory

Analyte	Code	314	315	316	317	318
trans-Lutein	FSV-DA	0.107	0.061	0.023	0.068	0.051
25-hydroxyvitamin D	FSV-BN	0.0381	0.0200	0.0090		
Phytofluene	FSV-DA	0.051	0.030	0.013	0.049	0.039
Phytoene	FSV-DA	0.047	0.028	0.024	0.048	0.024
Retinyl stearate	FSV-DA	0.040	0.012	*nd*	0.016	0.008
9-cis-Lycopene	FSV-BN	0.083	0.065	0.041	0.103	0.086
13-cis-Lycopene	FSV-BN	0.045	0.038	0.026	0.055	0.051
15-cis-Lycopene	FSV-BN	0.016	0.022	0.008	0.014	0.013

Legend

Term	Definition
N	Number of (non-NIST) quantitative values reported for this analyte
Min	Minimum (non-NIST) quantitative value reported
Median	Median (non-NIST) quantitative value reported
Max	Maximum (non-NIST) quantitative value reported
SD	Standard deviation for (non-NIST) results: 0.741*(3rd Quartile - 1st Quartile)
CV	Coefficient of Variation for (non-NIST) results: 100*SD/Median
N_{past}	Mean of N(s) from past RR(s)
$Median_{past}$	Mean of Median(s) from past RR(s)
SD_{past}	Pooled SD from past RR(s)
NIST	Mean of all analyses (vials x duplicates) reported by a NIST analyst
N_{NIST}	Number of total vials analyzed in duplicate by NIST analysts
S_{rep}	Within-vial pooled standard deviation
S_{het}	Among-vial pooled standard deviation
S_{NIST}	Total standard deviation for NIST analyses: $(S_{rep}^2 + S_{het}^2)^{0.5}$
NAV	NIST Assigned Value = (Median + $Mean_{NIST}$)/2 for analytes reported by NIST analyst(s) = Median for analytes reported by ≥ 10 labs but not NIST
NAU	NIST Assigned Uncertainty: $(S^2 + S_{btw}^2)^{0.5}$ S is the maximum of (0.05*NAV, SD, S_{NIST}, eSD) and S_{btw} is the standard deviation between $Median_{part}$ and $Mean_{NIST}$. The expected long-term SD, eSD, is defined in: Duewer, et al. Anal Chem 1997;69(7):1406-1413.
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

Round Robin LVIII "Score Card" for Serum Samples

Comparability Summary

Lab	TR	aT	g/bT	bC	tbC	aC	TLy	TbX	TLu	TZ	L&Z
FSV-BA	1	1	1	1	1	1	1	1	1	1	1
FSV-BB	1	1	1	1	1	1	1	1	2	1	1
FSV-BC	1										
FSV-BD	2	1									1
FSV-BE	1	2	1	1							
FSV-BF	1	1	1	1		1	1	1			1
FSV-BG	1	2	1	3		1	2	1	1	2	1
FSV-BH	1	2	4	1	1	1	1	3			
FSV-BI	2	2	1	1		1	1	1	1	1	1
FSV-BJ	1	1	1	1		1	1	1	2	4	4
FSV-BK	2	1									
FSV-BL	2	2							1		
FSV-BM	1	1									
FSV-BN	1	1	2	1	1	1	1	1			3
FSV-BO	1	2		2		1	1	1	1	1	1
FSV-BP	1	3		3		2	2	3			1
FSV-BQ	1	3							1	2	2
FSV-BR	1	1							1	2	2
FSV-BS	4		4	2	2	4	3	2	2	2	1
FSV-BT	1	1	1	1	1	1	1	1			
FSV-BU	1	1	1	2		1	1	1			
FSV-BV	3	1	1	1		1	1	1			2
FSV-BW	1	1	3	1		1	1	1			
FSV-BX	1	1	1	1	1	1		1			1
FSV-CC	1	1									1
FSV-CD	1	1	1	1		2	2	1			1
FSV-CE	2	1		1					1	1	1
FSV-CF	2	2									1
FSV-CG	3	3	1	1	1	1	1	2			
FSV-CI	1	1	1	1	1	1					2
FSV-CS	1	2	3	3	3	1	2	1			
FSV-CT				4			1	1	1	1	1
FSV-CW	1	3	1	1	2	1		1	1	1	1
FSV-CZ	1	1		2							
FSV-DA	1	1	1	2	2	1	1	1			
FSV-DB	1	1	1	1			1	1	3	4	3
FSV-DD	3										
FSV-DF	1								1		
FSV-DI	1	1	1	1			1		1	1	1
FSV-DV	4	3									
FSV-DW	1	3		3		1	1	2			
NIST	1	1	1	1	1	1					
n	41	37	24	30	13	24	23	24	16	14	24

	TR	aT	g/bT	bC	tbC	aC	TLy	TbX	TLu	TZ	L&Z
% 1	73	62	79	67	69	88	78	79	75	57	71
% 2	15	22	4	17	23	8	17	13	19	29	17
% 3	7	16	8	13	8	0	4	8	6	0	8
% 4	5	0	8	3	0	4	0	0	0	14	4

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

"Comparability Score"

The Comparability Score (CS) of summarizes your measurement performance for a given measurand, relative to the consensus medians. CS is the average distance, in standard deviation units, that your measurement performance characteristics are from the consensus performance. CS is calculated when the number of quantitative values you reported for a measurand, N_{you}, is at least two and the measurand has been reported by 10 or more participants.

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

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

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

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

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

Appendix D. Representative "Individualized Report" for RR58

Each participant in RR58 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 RR58:

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

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

Individualized Round Robin LVIII Report: FSV-BA

Summary

Analyte	Serum 314 You	Serum 314 NAV	Serum 314 n	Serum 315 You	Serum 315 NAV	Serum 315 n	Serum 316 You	Serum 316 NAV	Serum 316 n	Serum 317 You	Serum 317 NAV	Serum 317 n	Serum 318 You	Serum 318 NAV	Serum 318 n
Total Retinol	0.541	0.551	36	0.491	0.493	35	0.384	0.391	36	0.700	0.682	36	0.872	0.858	36
Retinyl Palmitate	0.10	0.10	12	0.1	0.1	12	0.0	0.0	8	0.06	0.05	12	0.04	0.03	12
α-Tocopherol	15.43	16.07	36	9.28	9.41	36	4.14	4.08	36	11.17	11.95	36	7.20	7.54	36
γ/β-Tocopherol	1.497	1.458	23	1.839	1.809	23	1.752	1.728	23	2.686	2.728	23	3.942	3.945	23
δ-Tocopherol	0.109	0.103	7	0.113	0.111	7	0.098	0.101	7	0.342	0.334	8	0.120	0.126	7
Total β-Carotene	0.604	0.601	25	0.279	0.295	25	0.040	0.041	24	0.314	0.318	25	0.173	0.173	25
trans-β-Carotene	0.564	0.550	12	0.260	0.266	12	0.037	0.039	12	0.291	0.289	12	0.161	0.162	12
Total cis-β-Carotene	0.040	0.066	8	0.018	0.036	8	0.003	0.003	7	0.022	0.033	8	0.013	0.011	8
Total α-Carotene	0.127	0.121	23	0.057	0.057	23	0.006	0.008	18	0.056	0.057	23	0.015	0.016	20
Total Lycopene	0.379	0.335	23	0.274	0.241	23	0.165	0.152	23	0.425	0.374	23	0.360	0.311	23
trans-Lycopene	0.201	0.166	9	0.138	0.122	9	0.076	0.069	9	0.203	0.174	9	0.179	0.154	9
Total β-Cryptoxanthin	0.063	0.052	24	0.053	0.045	24	0.041	0.035	24	0.065	0.055	24	0.085	0.072	24
Total α-Cryptoxanthin	0.027	0.020	6	0.018	0.015	6	0.012	0.009	6	0.023	0.020	6	0.027	0.021	6
Total Lutein&Zeaxanthin	0.149	0.144	23	0.09	0.09	23	0.04	0.04	22	0.104	0.098	24	0.09	0.09	24

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

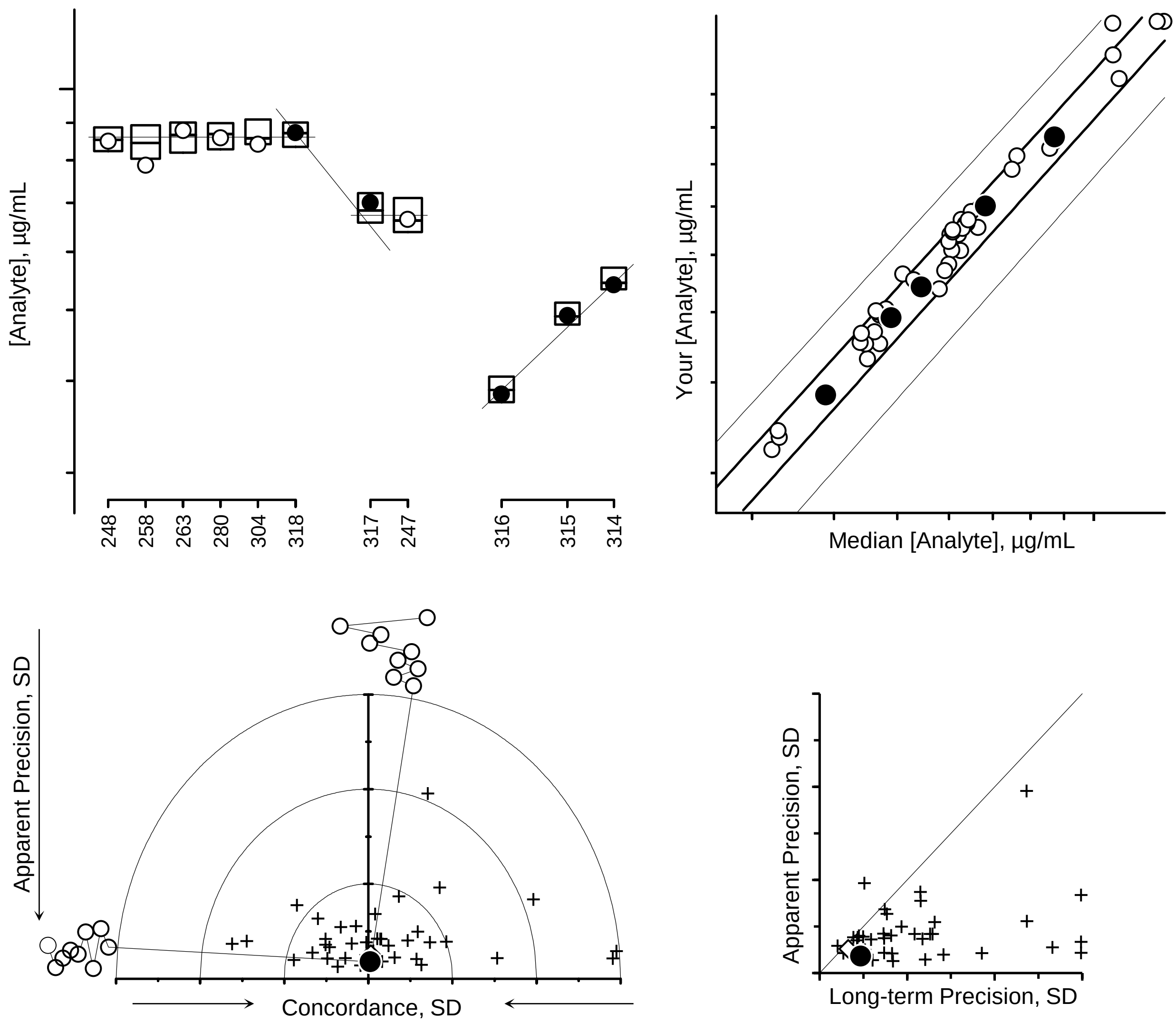
Please check our records against your records. Send corrections and/or updates to...

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 LVIII Report: FSV-BA

Total Retinol



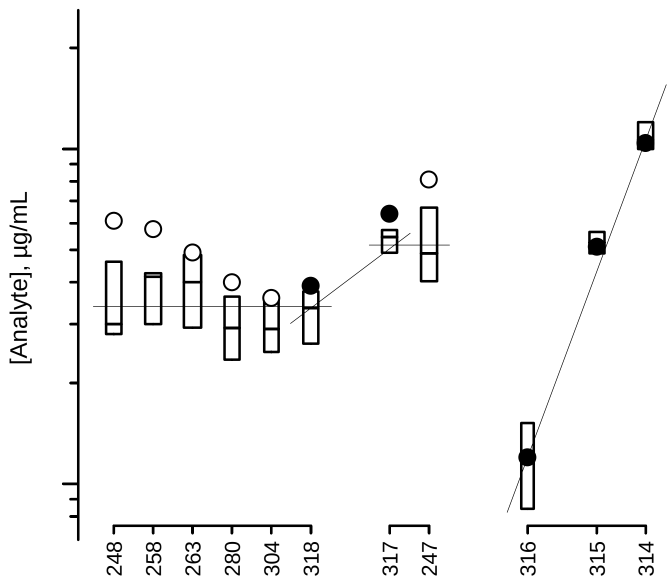
3rd Quartile (75%)
Median (50%)
1st Quartile (25%)

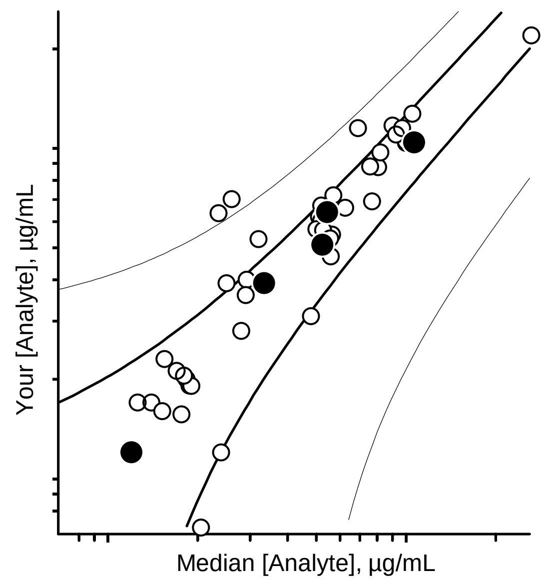
● You, this RR
○ You, past RRs
▲ You, ≥x, this RR
△ You, ≥x, past RRs
◇ NIST, this RR
\+ Others, this RR

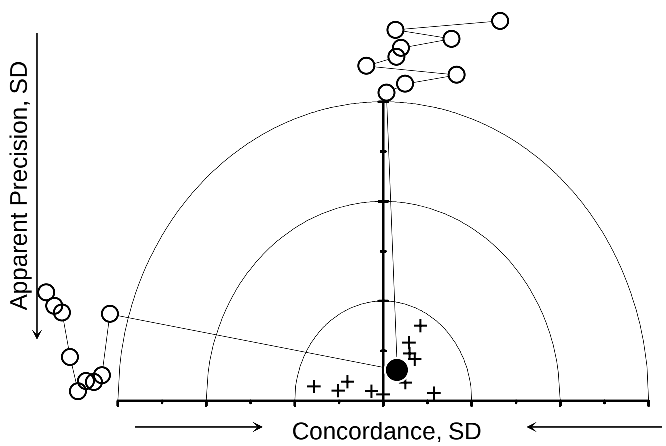
Serum	History	Comments
#314	Fresh frozen	Native, multi-source
#315	Fresh frozen	77:110 blend of #314 and #316
#316	Fresh frozen	Native, multi-source
#317	Lyophilized - 44:247	1:1 blend of SRMc Level I and II
#318	Lyophilized - 44:248, 46:258, 47:263, 51:280, 56:304	Augmented, multi-source (SRM 968c Level I)

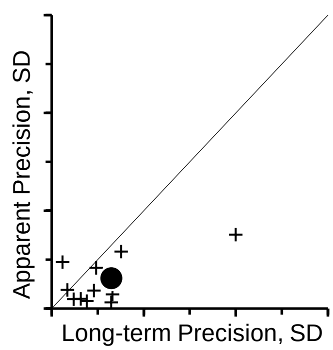
Individualized RR LVIII Report: FSV-BA

Retinyl Palmitate









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

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

Serum	History	Comments
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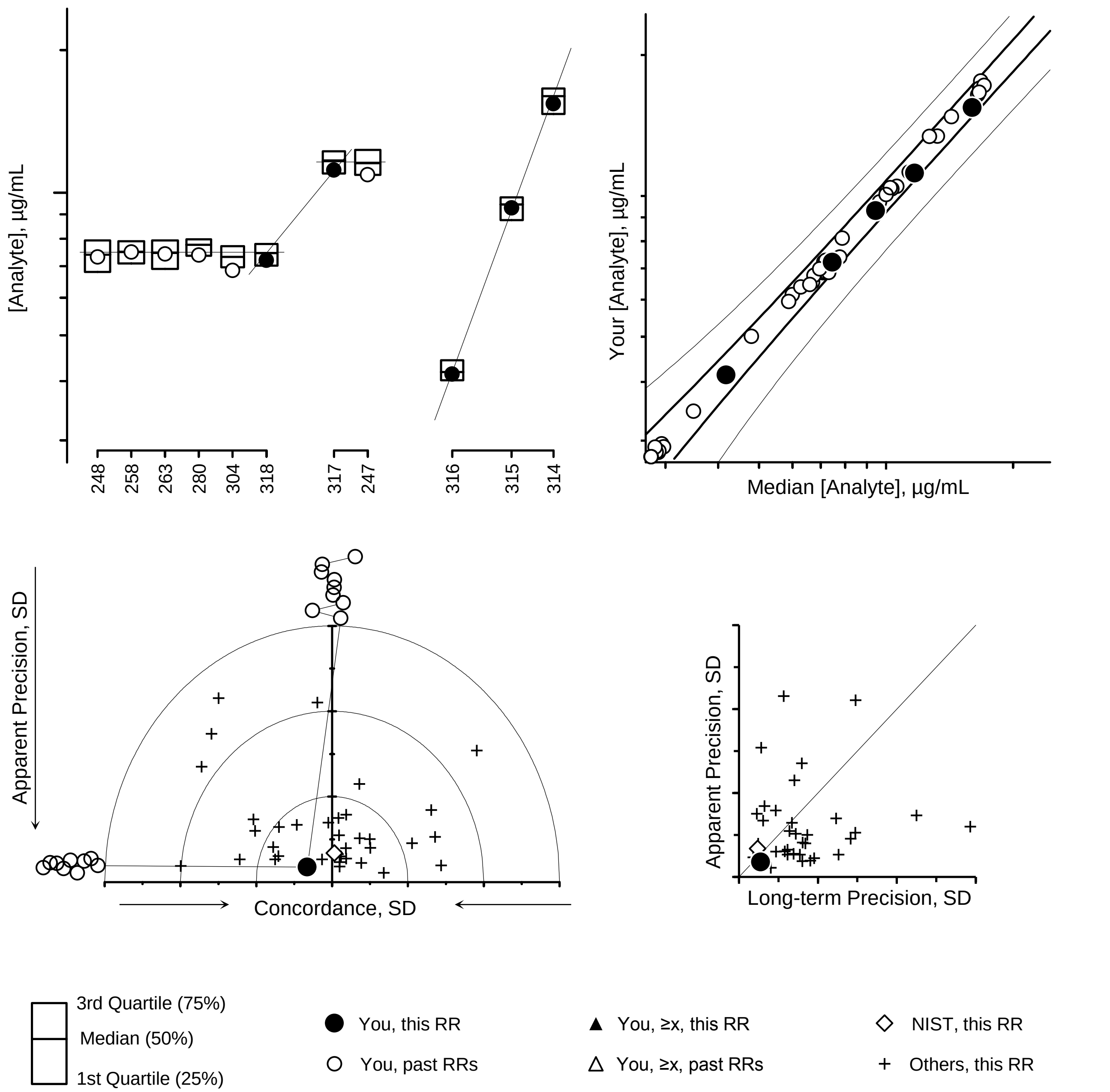
#318	Lyophilized - 44:248, 46:258, 47:263, 51:280, 56:304	Augmented, multi-source (SRM 968c Level I)

Individualized RR LVIII Report: FSV-BA

α-Tocopherol



Serum	History	Comments
#314	Fresh frozen	Native, multi-source
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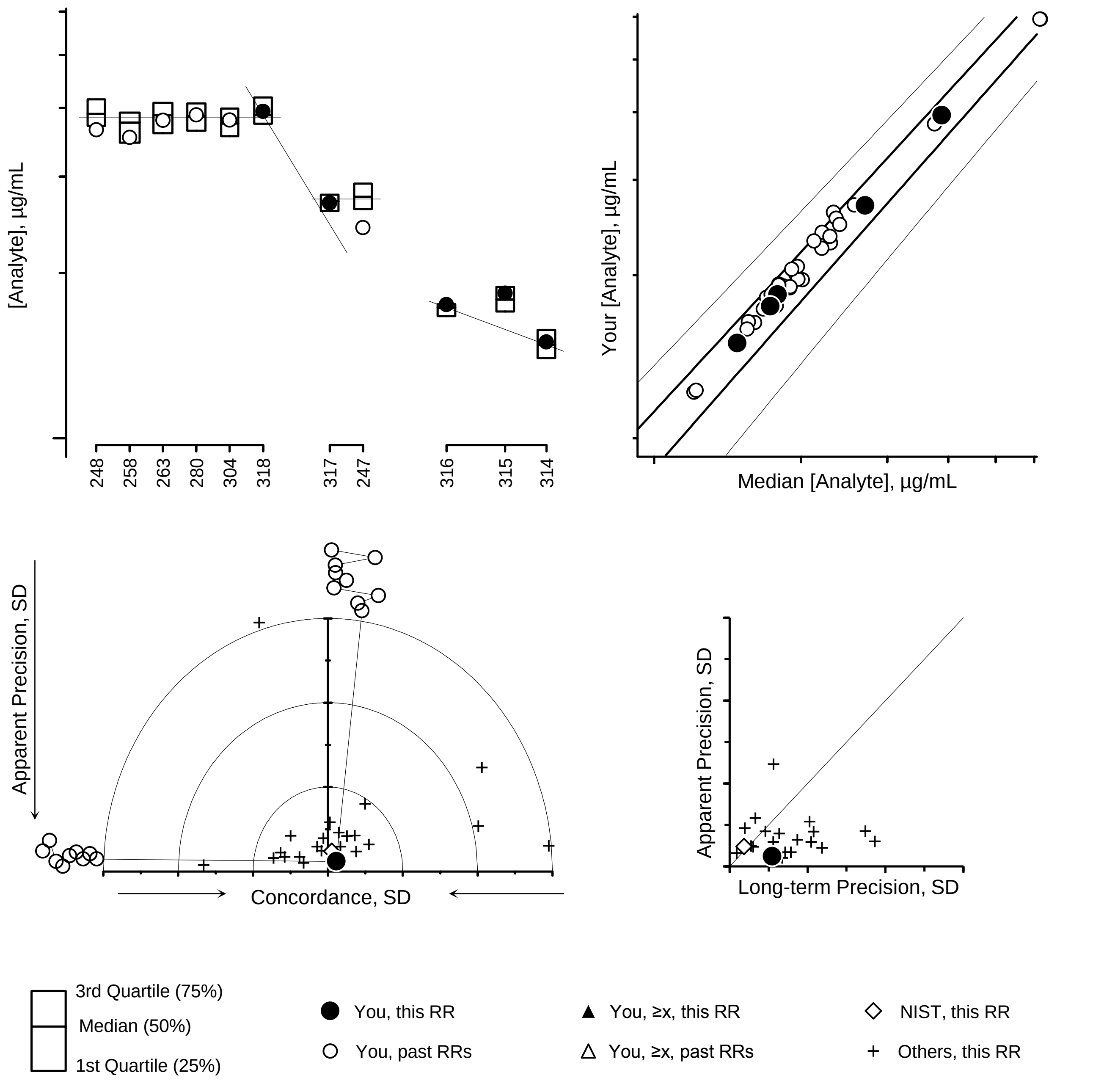
#318	Lyophilized - 44:248, 46:258, 47:263, 51:280, 56:304	Augmented, multi-source (SRM 968c Level I)

Individualized RR LVIII Report: FSV-BA

γ/β-Tocopherol



Serum	History	Comments
#314	Fresh frozen	Native, multi-source
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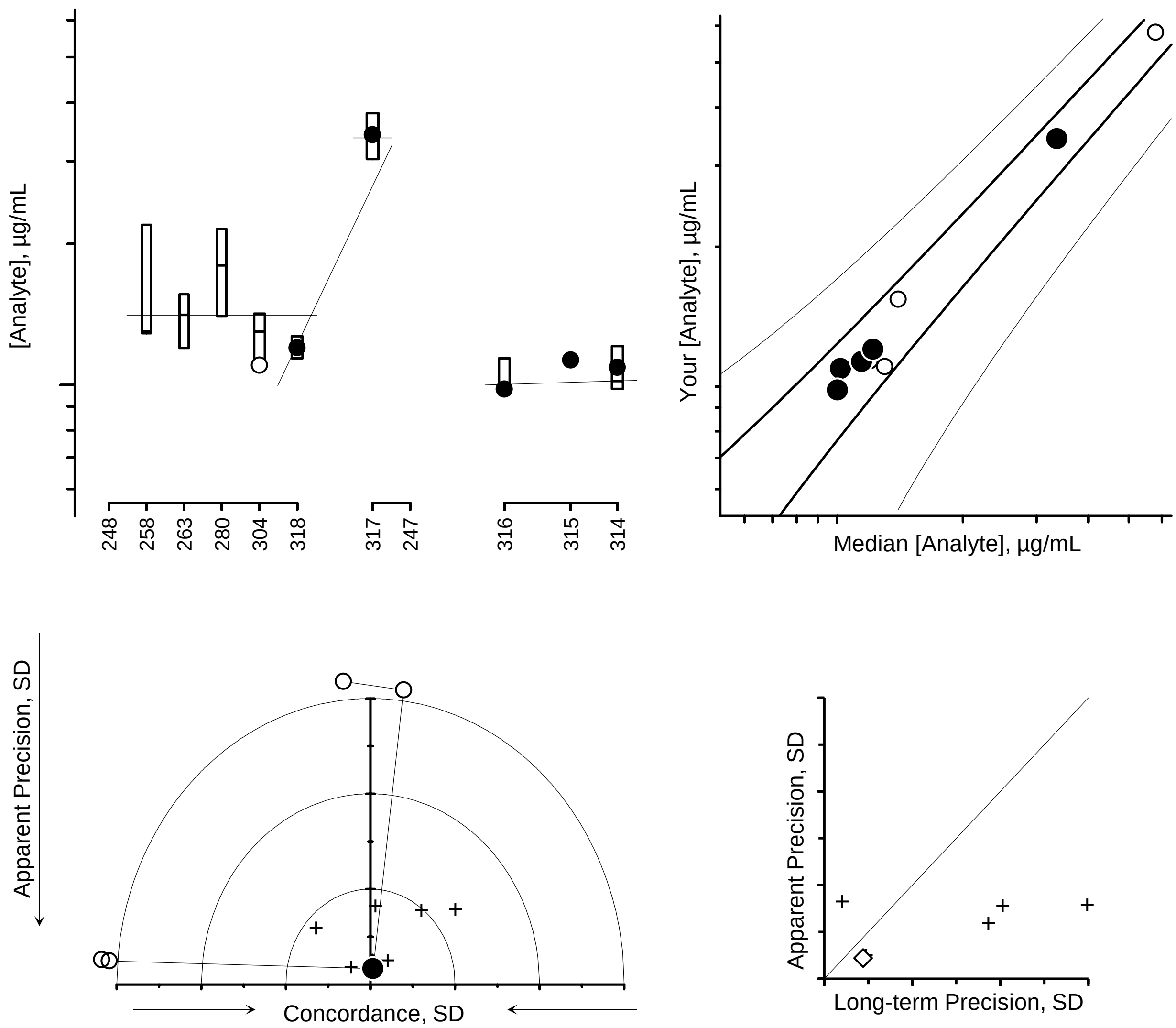
Individualized RR LVIII Report: FSV-BA

δ-Tocopherol



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

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

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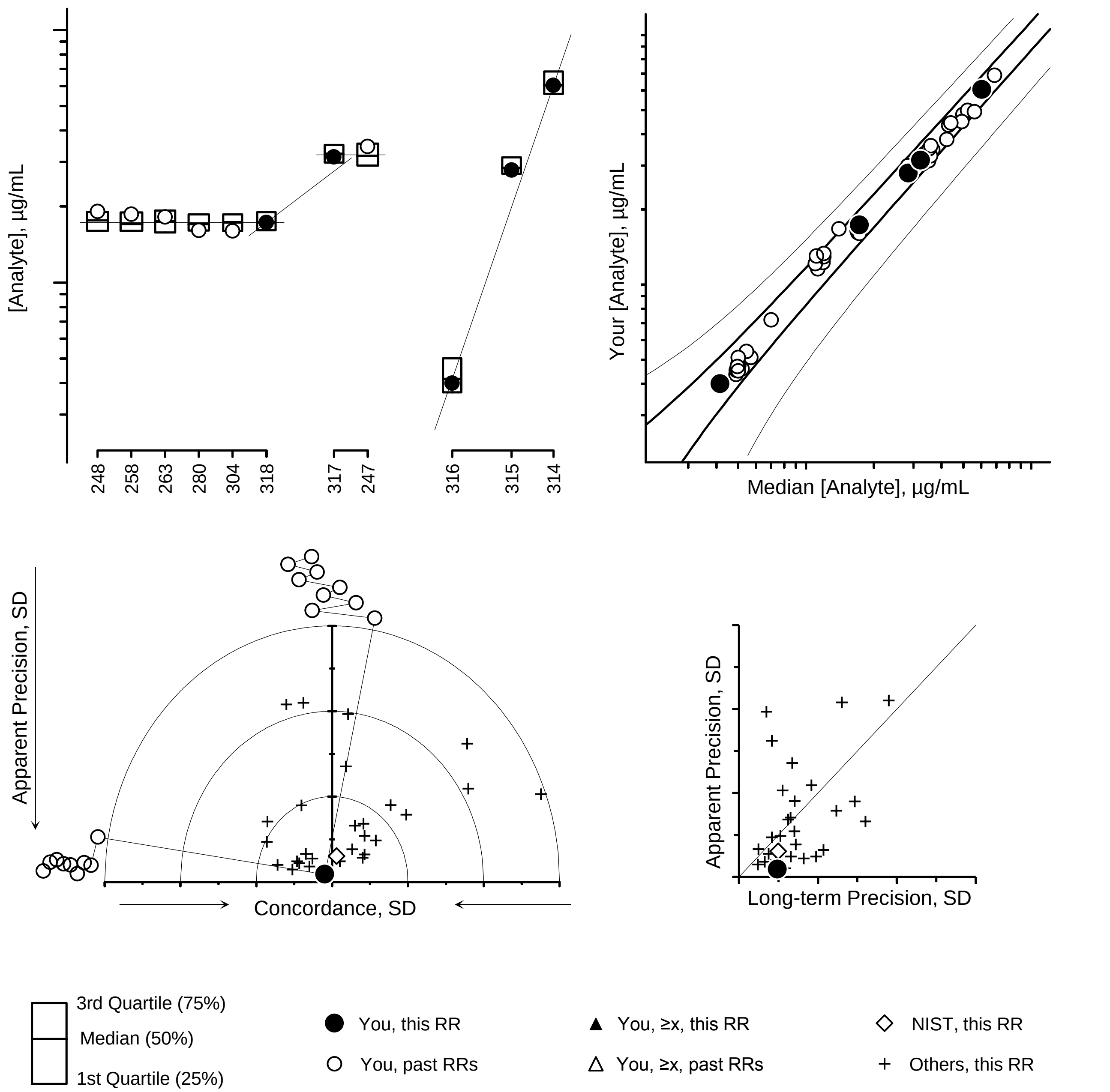
#318	Lyophilized - 44:248, 46:258, 47:263, 51:280, 56:304	Augmented, multi-source (SRM 968c Level I)

Individualized RR LVIII Report: FSV-BA

Total β-Carotene



<u>Serum</u>	<u>History</u>	<u>Comments</u>
#314	Fresh frozen	Native, multi-source
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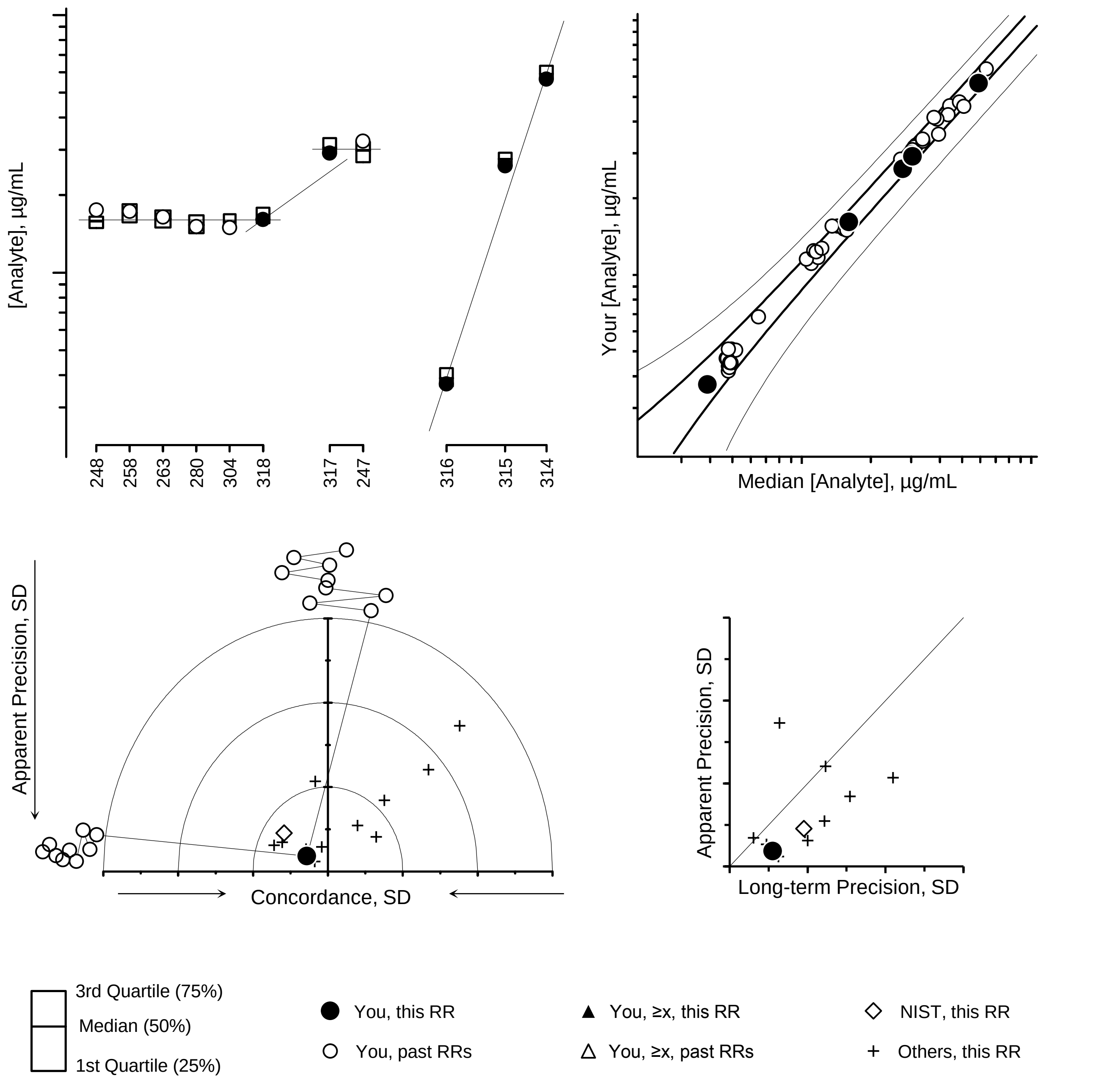
Individualized RR LVIII Report: FSV-BA

trans-β-Carotene



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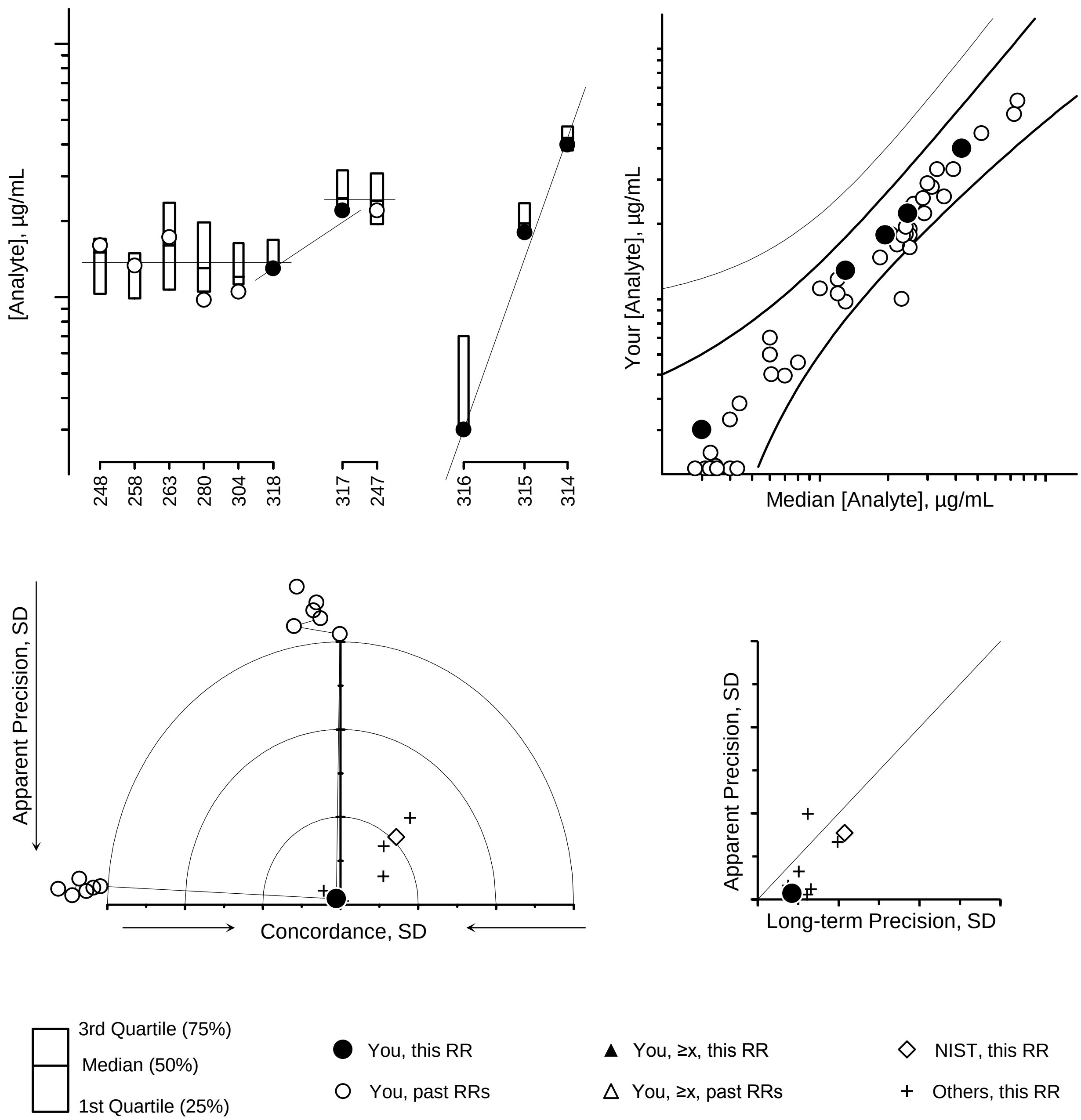
#318	Lyophilized - 44:248, 46:258, 47:263, 51:280, 56:304	Augmented, multi-source (SRM 968c Level I)

Individualized RR LVIII Report: FSV-BA

Total cis-β-Carotene



Serum	History	Comments
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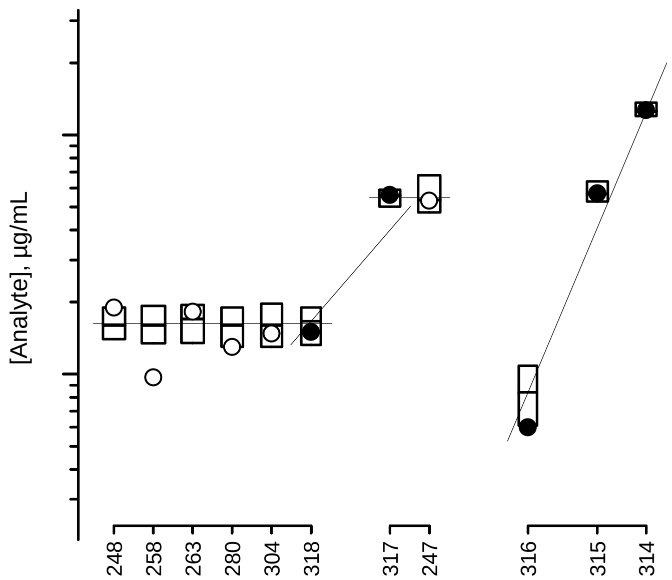
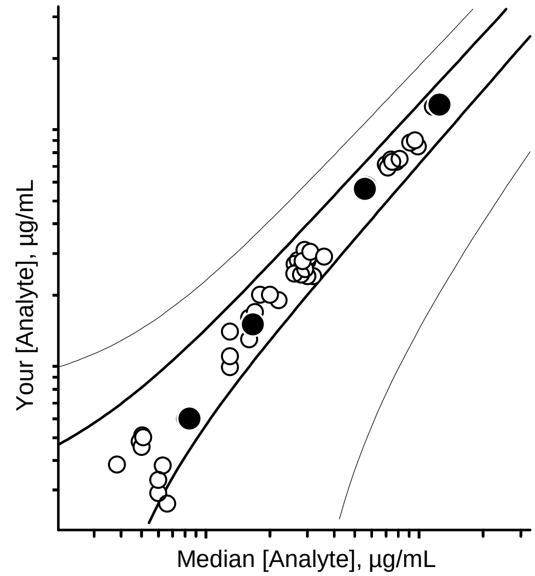
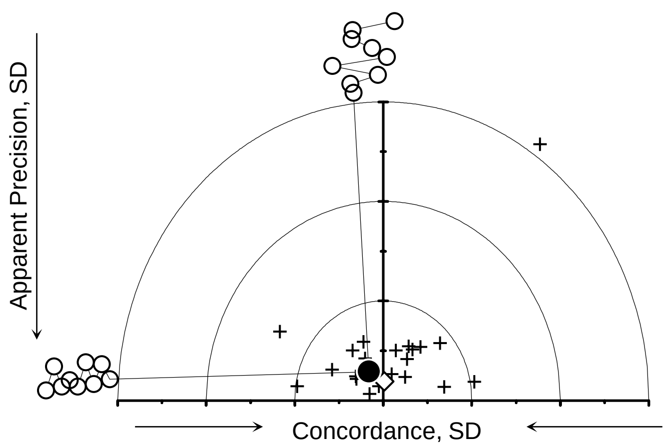
#318	Lyophilized - 44:248, 46:258, 47:263, 51:280, 56:304	Augmented, multi-source (SRM 968c Level I)

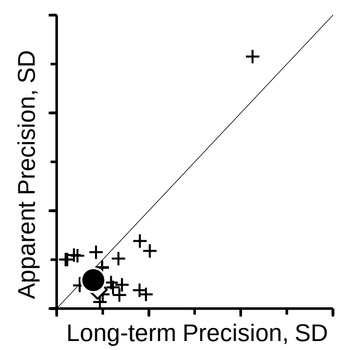
Individualized RR LVIII Report: FSV-BA

Total α-Carotene









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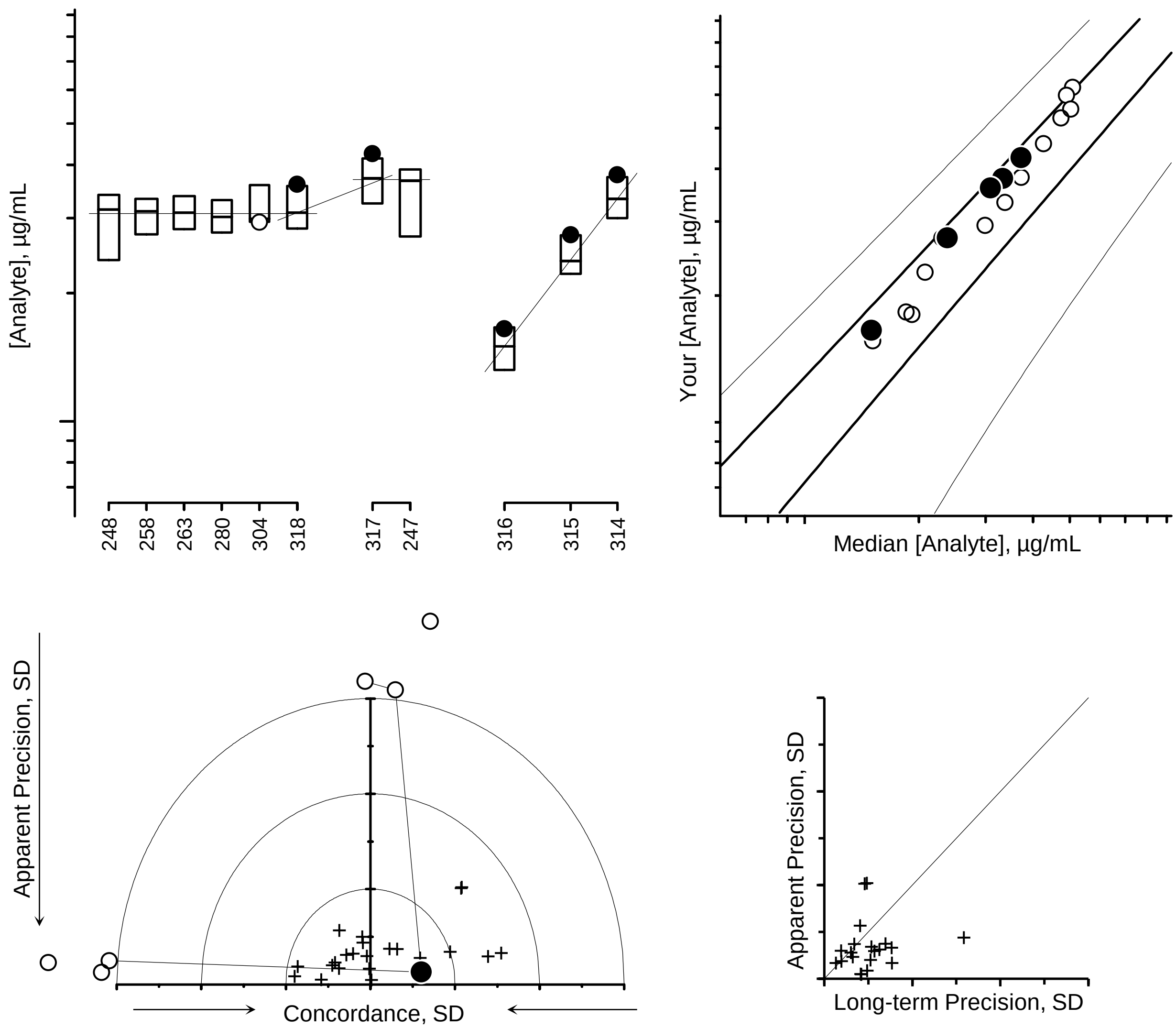
Individualized RR LVIII Report: FSV-BA

Total Lycopene



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

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

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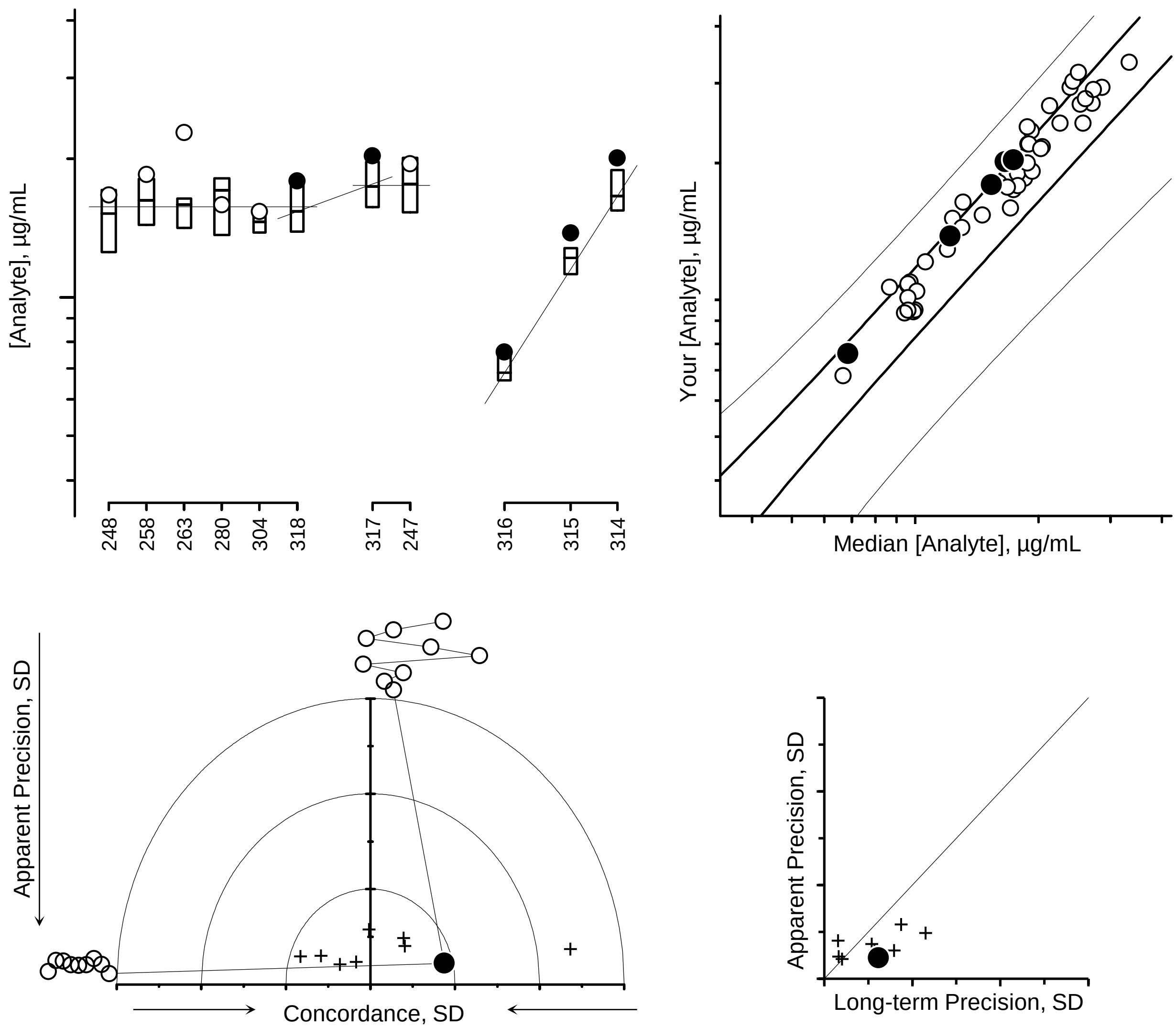
Individualized RR LVIII Report: FSV-BA

trans-Lycopene



3rd Quartile (75%)
Median (50%)
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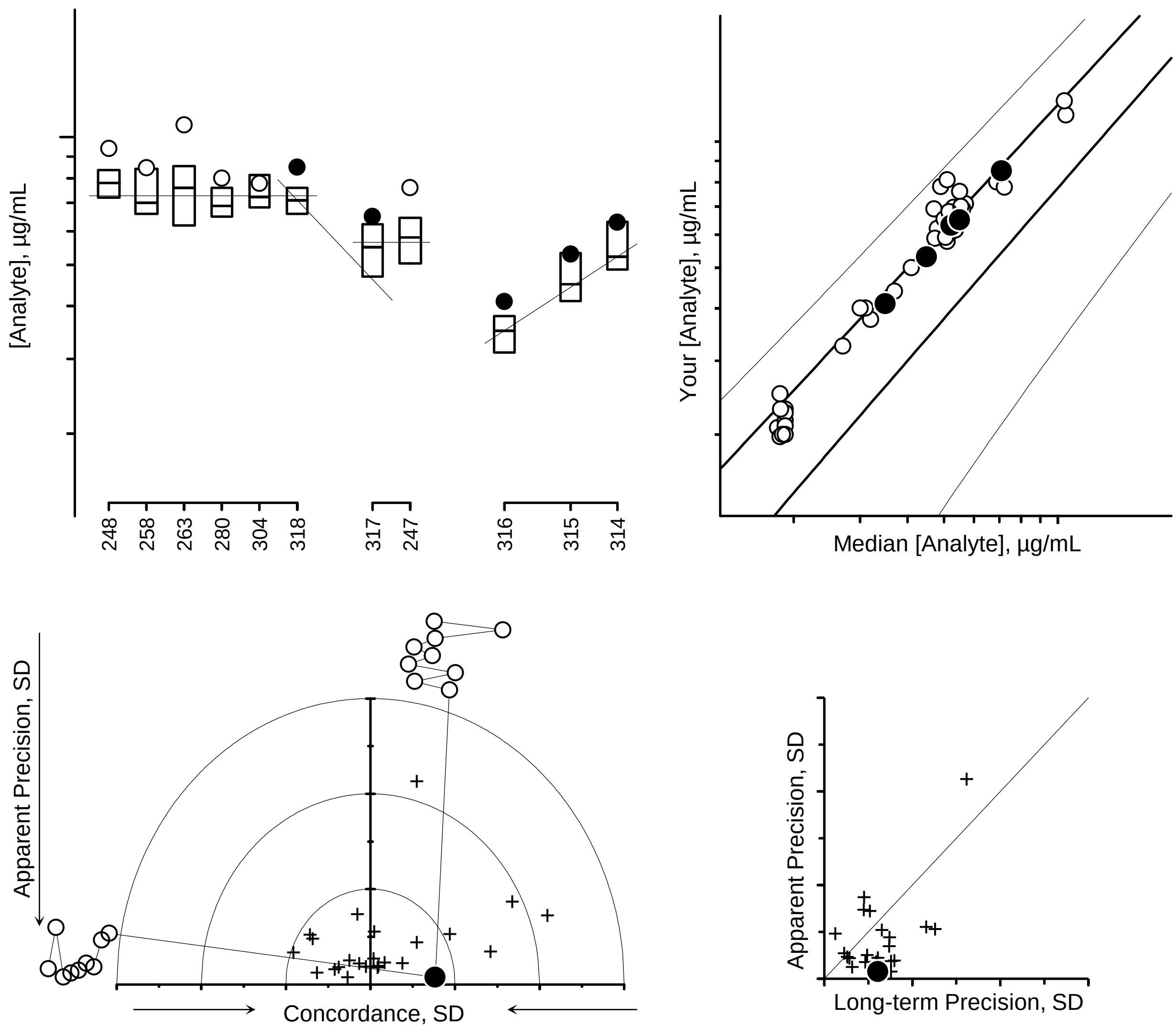
Individualized RR LVIII Report: FSV-BA

Total β-Cryptoxanthin



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

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△ You, ≥x, past RRs
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+ Others, this RR

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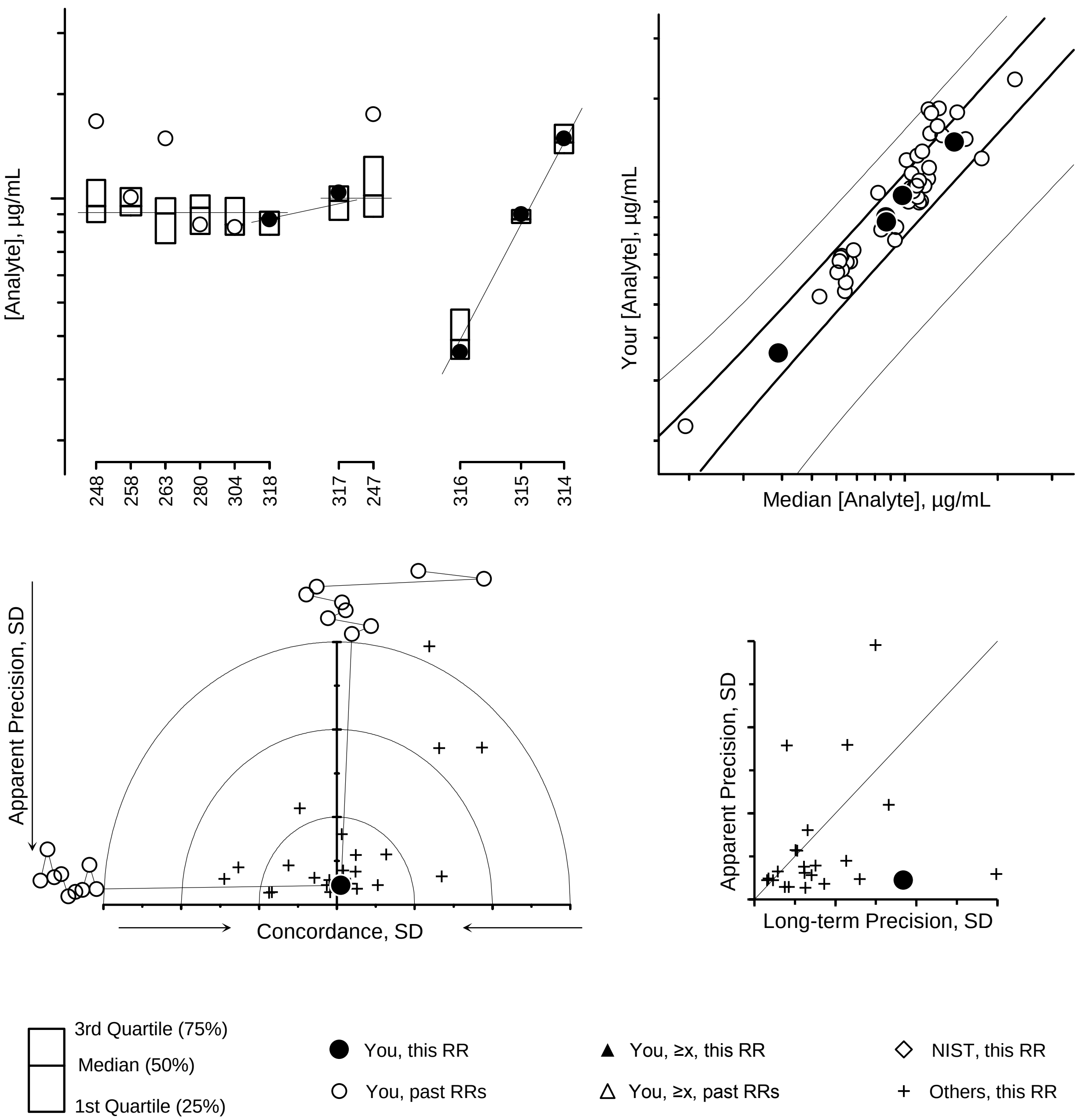
Individualized RR LVIII Report: FSV-BA

Total Lutein&Zeaxanthin



Serum	History	Comments
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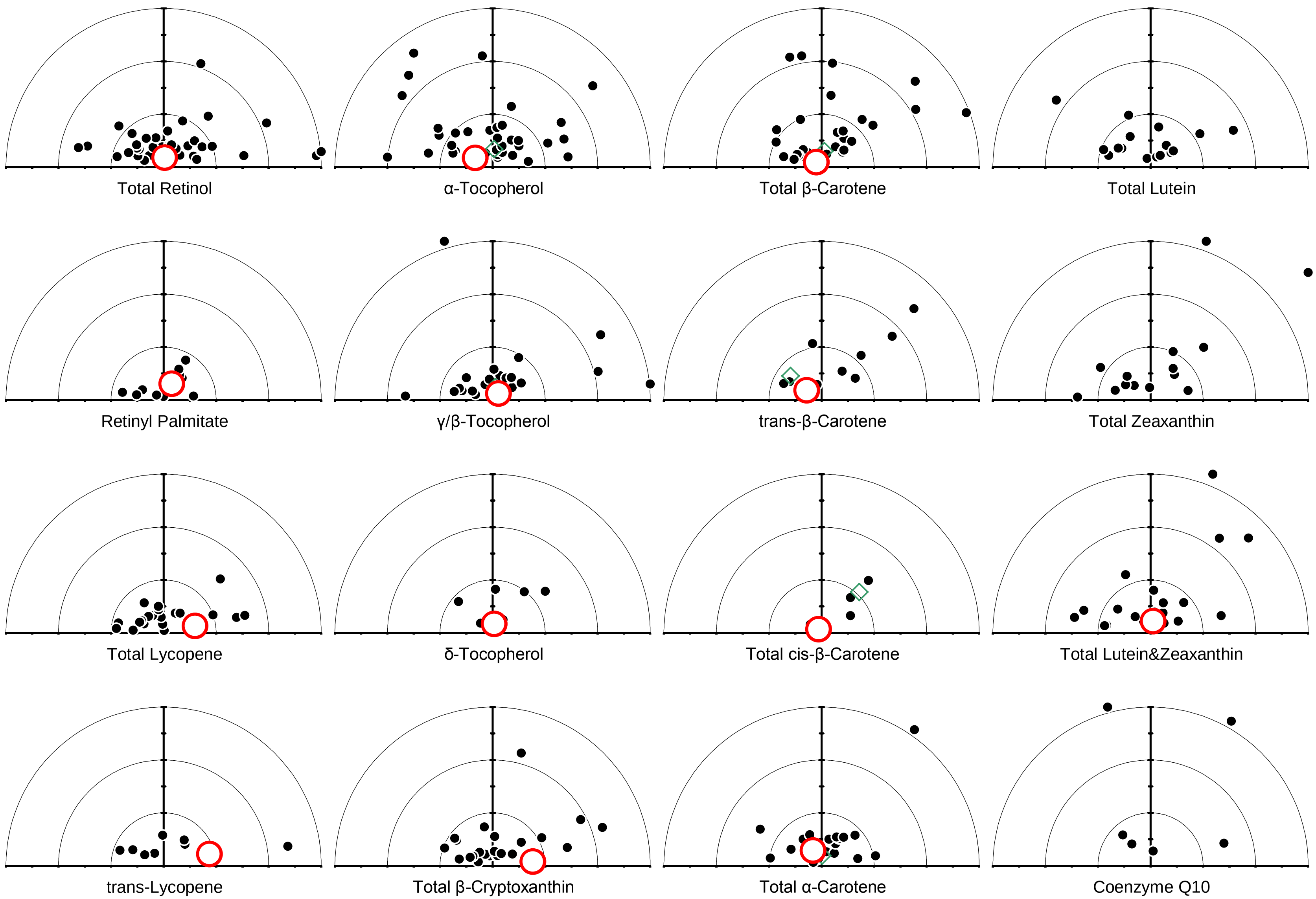
Individualized Round Robin LVIII Report: FSV-BA
Graphical Comparability Summary



Appendix E. Shipping Package Inserts for RR23

The following five items were included in each package shipped to an RR23 participant:

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

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

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

May 11, 2005

Dear Colleague:

The samples within this package constitute Vitamin C Round Robin 23 (RR23) of the fiscal year (FY) 05 Micronutrients Measurement Quality Assurance Program.

RR23consists of four vials of frozen serum *test samples* (#14, #31, #54, and #71), one vial of ascorbic acid *solid control material* (Control), and two vials of frozen *serum control materials* (Control #1 and Control #2). Please follow the attached protocols when you prepare and analyze these samples. If you cannot prepare the *solid control* solutions gravimetrically, please prepare equivalent solutions volumetrically and report the exact volumes used. (Routine 0.5 g gravimetric measurements are generally 10-fold more accurate than routine 0.5 mL volumetric measurements.)

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

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

The report for RR22 was mailed the week of April 14, 2005. If you find your results for RR22 unsatisfactory, we recommend that you obtain **Standard Reference Material (SRM) 970 Ascorbic Acid in Serum** to validate your methodology and value assign in-house control materials. This SRM may be purchased from the Standard Materials Reference Program at NIST (Tel: 301-975-6776, Fax: 301-948-3730, or e-mail: srminfo@nist.gov).

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

We ask that you return your results for these RR23 samples *before* **September 30, 2005**. We would appreciate receiving your results as soon as they become available. Please use the attached form. Your results will be kept confidential.

Sincerely,

Jeanice Thomas

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

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

NIST

Micronutrient Measurement Quality Assurance Program for
Vitamin C

Please Read Through Completely BEFORE Analyzing Samples

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

7) Once you have confirmed that your system is properly calibrated, analyze the serum control CS #2 (see protocol below). The target values for this materials is 28.1 ±1.0 µmol/L of sample.

   If your measured values are not close to this value, please review your sample preparation procedure and whether you followed *exactly* the same measurement protocol the solutions prepared from the solid control material as you used for these serum controls. If the protocols differ, please repeat from Step 6 using the proper protocol. If the proper protocol was used, your measurement system may not be suitable for MPA-preserved samples. Please contact us: 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 µmol of total ascorbic acid/L of solution. The total ascorbic acid in each ampoule should be measured in duplicate. Please report your results in µmol/(L of the sample solution) rather than µmol/(L of serum NIST used to prepare the sample).

Participant #: _______ Date: __________

Vitamin C Round Robin 23

NIST Micronutrient Measurement Quality Assurance Program

Preparation and Validation of Ascorbic Acid Solid Control Material

STOCK SOLUTION

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

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

DILUTE SOLUTION 1

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

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

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

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

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

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

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

Wavelength of maximum absorbance.. __________ nm

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

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

DILUTE SOLUTION 2

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

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

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

DILUTE SOLUTION 3

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

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

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

Please return by **Sept 12, 2005**

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 23

NIST Micronutrient Measurement Quality Assurance Program

Analysis of Control Materials and Test Samples

Sample	Replicate 1	Replicate 2	Units
Dilute Solution 1			µmol/L of Dilute Solution
Dilute Solution 2			µmol/L of Dilute Solution
Dilute Solution 3			µmol/L of Dilute Solution
5% MPA Diluent			µmol/L of Diluent
Serum Control #1			µmol/L of Sample *Target:* 8.5 ±0.5 µmol/L
Serum Control #2			µmol/L of Sample *Target:* 28.1 ±1.0 µmol/L
Serum Test Sample #14			µmol/L of Sample
Serum Test Sample #31			µmol/L of Sample
Serum Test Sample #54			µmol/L of Sample
Serum Test Sample #71			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 **Sept 12, 2005**

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 23

NIST Micronutrients Measurement Quality Assurance Program

Packing List and Shipment Receipt Confirmation Form

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

Sample	Form
VitC #14	Liquid frozen (1:1 serum:10% MPA)
VitC #31	Liquid frozen (1:1 serum:10% MPA)
VitC #54	Liquid frozen (1:1 serum:10% MPA)
VitC #71	Liquid frozen (1:1 serum:10% MPA)
Control #1	Liquid frozen (1:1 serum:10% MPA)
Control #2	Liquid frozen (1:1 serum:10% MPA)
Control	Solid AA

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

1) Date this shipment arrived: ___________________________

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

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

4) Did the samples arrive frozen? Yes | No

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

6) When do you anticipate analyzing these samples? ________________________

Your prompt return of this information is appreciated.

The M^{2}QAP Gang

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

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

Appendix F. Final Report for RR23

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-

November 8, 2005

Dear Colleague:

Enclosed is the summary report of the results for Round Robin 23 (RR23) for the measurement of total ascorbic acid (TAA, ascorbic acid plus dehydroascorbic acid) in human serum. Included in this report are: a summary of data for all laboratories and a summary of individual laboratory performance and interlaboratory accuracy and repeatability. The robust median is used to estimate the consensus value for all samples, the median absolute deviation from the median (MADe) is used to estimate the expected standard deviation, and the coefficient of variation (CV) is defined as 100×MADe/median.

RR 23 consisted of four *test samples* (#14, #31, #54, and #71), 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 first vitamin C round robin study of the 2006 M^2QAP (RR24) will be shipped **starting the last week of November 2005**.

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

Sincerely,

Jeanice B Thomas

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

Enclosures

NIST

The NIST M^2QAP Vitamin C Round Robin 23 (RR23) 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 RR23 sample measurements.

Page	"All Lab" Report
1	A tabulation of results and summary statistics for Total Ascorbic Acid [TAA] in the RR23 samples and control/calibration solutions.

Serum-based Samples. Two serum controls and four unknowns were distributed in RR23.

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

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

S23:1 Serum 14, ampouled in late 2001, previously distributed as sample S19:1 (RR19, Sep-03) and S21:1 (RR21, Sep-04). A "blank" stripped serum.

S23:2 Serum 31, ampouled in late 2001, previously distributed as sample S17:2 (RR17, Sep-02), S18:1 (RR18, Mar-03) S20:1 (RR20, Mar-04), and S22:1 (RR22, Mar-05). An augmented serum.

S23:3 Serum 54, ampouled in late 2001, previously distributed as sample 16:3 (RR13, Mar-02), S17:3 (RR17, Sep-02), S20:2 (RR20, Mar-04), and S21:4 (RR21, Sep-04). An augmented serum.

S23:4 Serum 71, SRM 970 level 1, ampouled in mid-1998. This material was distributed with identification in RR11 (Oct-98) and RR12 (Mar-99) and as samples S13-1 (RR13, Mar-00), S14-3 (RR14, Mar-01), S15:1 (RR15, Sep-01), S16:1 (RR16, Mar-02), S19:4 (Sep-03), and S20:3 (Mar-04). An augmented serum.

Results.

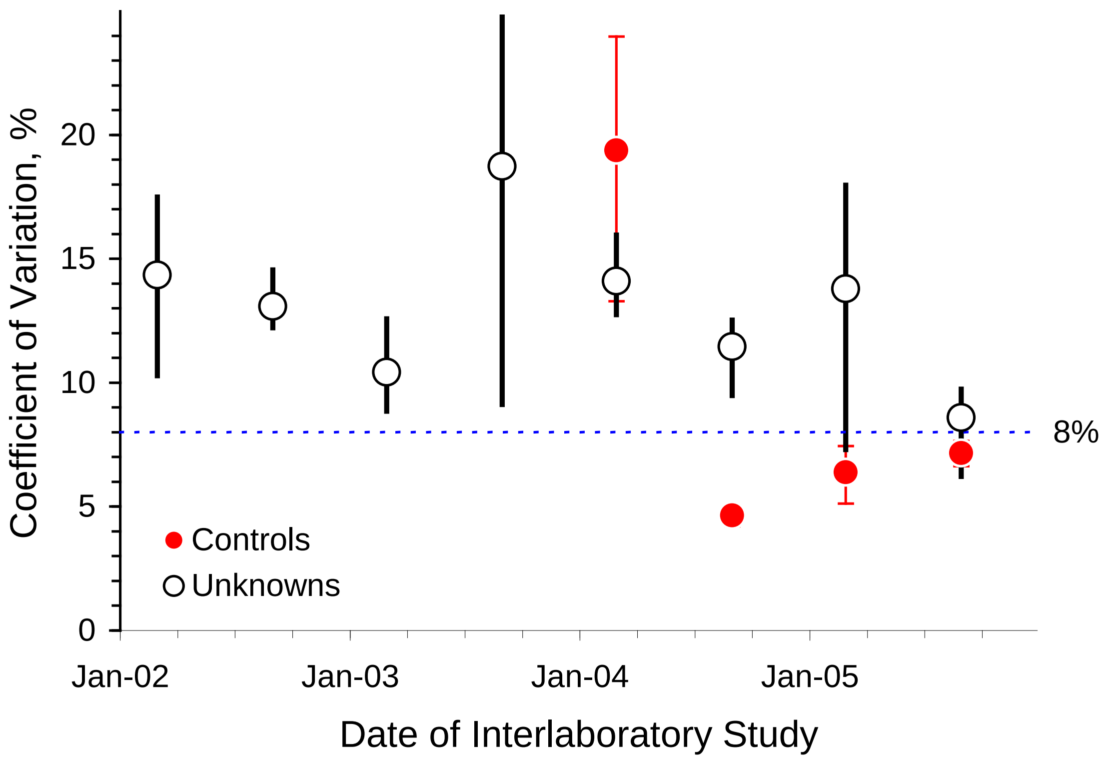
1) All participants who prepared the four control/calibration solutions (the three dilute solutions and the 5% MPA diluent) did so correctly. The criteria used to evaluate this success are: the density of the 5% MPA (≈1.03 gm/mL), the observed wavelength maximum of "Dilute Solution #1"(≈244 nm), the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

2) Judging from the calibration parameters calculated for the control/calibration solutions (intercepts close to 0.0 and slopes close to 1.0), the measurement systems for all participants are well calibrated.

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

4) The following Figure displays the expected %relative standard deviation or "coefficient of variation" (CV) for the control and unknown samples as a function of time for the past four years. The expected CV is estimated by pooling the individual CVs of all non-blank samples in a given study. The individual sample CVs are estimated as 100×MADe/median. The "error bars" span from the lowest to the highest observed CVs.

   While the data are as yet insufficient for any great confidence, the general reduction in the expected CV with time since the introduction of the control samples in 2004 suggests that analysis of the controls helps control the observed variability of the unknown samples. Based on the convergence of the expected CV of the control materials with that for the unknowns, the currently achievable among-laboratory CV for the analysis of total ascorbic acid is about 8%.



Appendix G. "All-Lab Report" for RR23

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

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

The data summary in the "All-Lab Report" has been altered to ensure confidentiality of identification codes assigned to laboratories.

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

Control / Calibration Samples

Lab	Date	Gravimetric, μmol/L			Measured, μmol/L				Calibration Parameters			
		Dil:1	Dil:2	Dil:3	Dil:1	Dil:2	Dil:3	MPA	Inter	Slope	R^2	SEE
VC-MA	17/10/05	56.2	27.8	13.7	61.3	28.4	13.6	0.6	-0.59	1.09	0.998	1.5
VC-MB	25/05/05	59.3	29.1	15.1	56.3	26.1	13.4	0.0	-0.66	0.95	0.999	0.9
VC-MC	02/06/05	57.4	28.5	14.2	56.4	29.6	14.7	0.0	0.63	0.98	0.999	1.0
VC-ME	20/09/05	58.2	29.2	14.1	57.9	29.6	15.2	0.0	0.58	0.99	1.000	0.7
VC-MG	22/09/05	57.0	29.8	15.8	56.1	28.0	13.9	0.0	-0.87	0.99	0.999	1.0
VC-MH	03/10/05	47.2	23.5	11.6	45.9	24.0	13.0	0.0	0.95	0.96	0.998	1.1
VC-MI	12/09/05	57.2	28.4	14.1	57.4	27.6	13.0	0.0	-0.67	1.01	0.999	0.7
VC-MJ	24/06/05	60.8	30.4	15.8	61.8	31.4	16.0	0.2	0.20	1.02	1.000	0.3
VC-MK	09/09/05	59.5	29.6	14.5	60.1	31.3	16.8	1.9	2.25	0.97	1.000	0.4
VC-MP	12/08/05											
	N	9	9	9	9	9	9	9				
	Average	57.0	28.5	14.3	57.0	28.4	14.4	0.3				
	SD	3.9	2.0	1.3	4.7	2.4	1.4	0.6				
	Min	47.2	23.53	11.6	45.9	24.00	13.0	0.0				
	%25	57.0	28.39	14.1	56.3	27.60	13.4	0.0				
	Median	57.4	29.14	14.2	57.4	28.35	13.9	0.0				
	%75	59.3	29.58	15.1	60.1	29.64	15.2	0.2				
	Max	60.8	30.38	15.8	61.8	31.40	16.8	1.9				
	MADe	1.8	1.0	0.7	1.9	1.9	1.3	0.0				
	CV	3	3	5	3	7	9					

Lab	Date	MPA Density	Dilute Solution 1 Spectrophotometry			Samples: Measured, μmol/L						Samples: Corrected, μmol/L					
		g/mL	λ_{max}	A_{max}	$E^{1\%}$	CS#1	CS#2	S23:1	S23:2	S23:3	S23:4	CS#1	CS#2	S23:1	S23:2	S23:3	S23:4
VC-MA	17/10/05	1.031	244.	0.5621	567.9	9.4	30.0	0.0	24.9	49.3	8.7	9.2	28.1	0.5	23.5	45.9	8.6
VC-MB	25/05/05	1.030	244.	0.6247	597.8	7.5	27.9	0.0	21.7	42.6	7.7	8.6	30.0	0.7	23.5	45.5	8.8
VC-MC	02/06/05	1.035	243.	0.5831	576.7	8.6	29.2	0.0	20.7	45.3	8.1	8.1	29.1	0.0	20.5	45.5	7.6
VC-ME	20/09/05	1.030	243.6	0.5798	565.5	8.2	26.8	0.0	21.8	46.9	8.9	7.7	26.5	0.0	21.5	46.8	8.4
VC-MG	22/09/05	1.031	243.2	0.5420	539.5	8.0	30.1	0.0	24.8	54.2	7.9	9.0	31.3	0.9	26.0	55.7	8.9
VC-MH	03/10/05	1.029	243.9	0.4538	545.4	8.9	27.4	1.3	23.4	48.3	9.3	8.3	27.5	0.3	23.4	49.3	8.7
VC-MI	12/09/05	1.025				8.5	28.1	0.0	19.3	43.3	8.2	9.0	28.5	0.7	19.8	43.5	8.8
VC-MJ	24/06/05	1.016	254.4	0.3680	343.9	10.6	30.3	3.8	24.6	48.7	11.4	10.2	29.6	3.5	24.0	47.7	11.0
VC-MK	09/09/05	1.031	244.	0.5947	567.5	12.0	33.6	3.8	23.3	52.5	12.7	10.0	32.1	1.5	21.6	51.6	10.7
VC-MP	12/08/05					8.4	28.0	0.0	21.6	46.2	8.5						
	N	9	8	8	8	10	10	10	10	10	10	9	9	9	9	9	9
	Average	1.029	245.0	0.5385	538.0	9.0	29.1	0.9	22.6	47.7	9.1	8.9	29.2	0.9	22.6	47.9	9.1
	SD	0.005	3.8	0.0854	80.5	1.4	2.0	1.6	1.9	3.7	1.6	0.8	1.8	1.1	1.9	3.7	1.1
	Min	1.016	243.0	0.3680	343.9	7.5	26.8	0.0	19.3	42.6	7.7	7.7	26.5	0.0	19.8	43.5	7.6
	%25	1.029	243.5	0.5200	543.9	8.2	27.9	0.0	21.6	45.5	8.1	8.3	28.1	0.3	21.5	45.5	8.6
	Median	1.030	244.0	0.5710	566.5	8.5	28.6	0.0	22.6	47.6	8.6	9.0	29.1	0.7	23.4	46.8	8.8
	%75	1.031	244.0	0.5860	570.1	9.3	30.0	0.9	24.3	49.1	9.2	9.2	30.0	0.9	23.5	49.3	8.9
	Max	1.035	254.4	0.6247	597.8	12.0	33.6	3.8	24.9	54.2	12.7	10.2	32.1	3.5	26.0	55.7	11.0
	eSD	0.001	0.3	0.0391	23.2	0.7	1.9	0.0	2.1	3.0	0.8	1.1	1.5	0.5	2.6	1.9	0.3
	CV	0.12	0.12	6.8	4.1	8	7		9	6	10	12	5	77	11	4	4

Appendix H. Representative "Individualized Report" for RR23

Each participant in RR23 received an "Individualized Report" reflecting their reported results. The following two pages are the "Individualized Report" for participant "VC-MA".

Vitamin C "Round Robin" 23 Report: Participant VC-MA

Date	RR	Method	MPA Density g/mL	Dilute Solution 1 Spectrophotometry λ_{max}	A_{max}	$E^{1\%}$	Control/Calibration Solutions Y_{meas} = Inter + Slope* X_{grav} Inter	Slope	R^2	SEE
03/20/03	18	HPLC-EC	1.026	244.0	0.509	563.1	-0.1	1.02	1.000	0.18
11/13/03	19	HPLC-EC	1.026	243.0	0.584	561.9	1.1	1.03	0.998	1.24
02/23/04	20	HPLC-EC	1.031	243.0	0.552	560.7	-0.4	1.05	1.000	0.65
09/13/04	21	HPLC-EC	1.030	244.0	0.555	562.2	-0.1	0.99	1.000	0.10
03/08/05	22	HPLC-EC	1.034	243.0	0.559	562.9	0.2	1.06	1.000	0.24
10/17/05	23	HPLC-EC	1.030	244.0	0.562	567.9	-0.6	1.09	0.998	1.47
		Mean	1.029	243.5	0.55	563.1				0.65
		SD	0.003	0.5	0.02	2.5				0.58
		CV	0.29	0.22	4.4	0.4				

[TAA] mmol/Lsample

Date	RR	Sample	Rep_1	Rep_2	F_{adj}	Mean	SD_{dup}	N	Mean	SD_{repeat}	SD_{reprod}
02/23/04	20	CS#1	7.8	8.0	1.0	7.9	0.1	4	8.5	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				
02/23/04	20	CS#2	25.8	26.2	1.0	26.0	0.3	4	27.9	0.6	1.9
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				
11/13/03	19	S19:1	nd	nd	1.0			1	0.0	0.0	
09/13/04	21	S21:1	nd	nd	1.0						
10/17/05	23	S23:1	0.0	0.0	1.0	0.0	0.0				
03/20/03	18	S18:1	22.7	23.7	1.0	23.2	0.7	4	23.9	0.6	1.1
02/23/04	20	S20:1	25.1	24.1	1.0	24.6	0.7				
03/08/05	22	S22:2	22.7	22.7	1.0	22.7	0.0				
10/17/05	23	S23:2	25.5	24.4	1.0	24.9	0.8				
02/23/04	20	S20:2	50.6	50.0	1.0	50.3	0.4	3	48.9	0.5	1.7
09/13/04	21	S21:4	47.1	47.0	1.0	47.0	0.0				
10/17/05	23	S23:3	49.8	48.8	1.0	49.3	0.7				
11/13/03	19	S19:4	7.8	8.6	1.0	8.2	0.5	3	8.4	0.3	0.4
02/23/04	20	S20:3	8.3	8.1	1.0	8.2	0.1				
10/17/05	23	S23:4	8.6	8.8	1.0	8.7	0.1				

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

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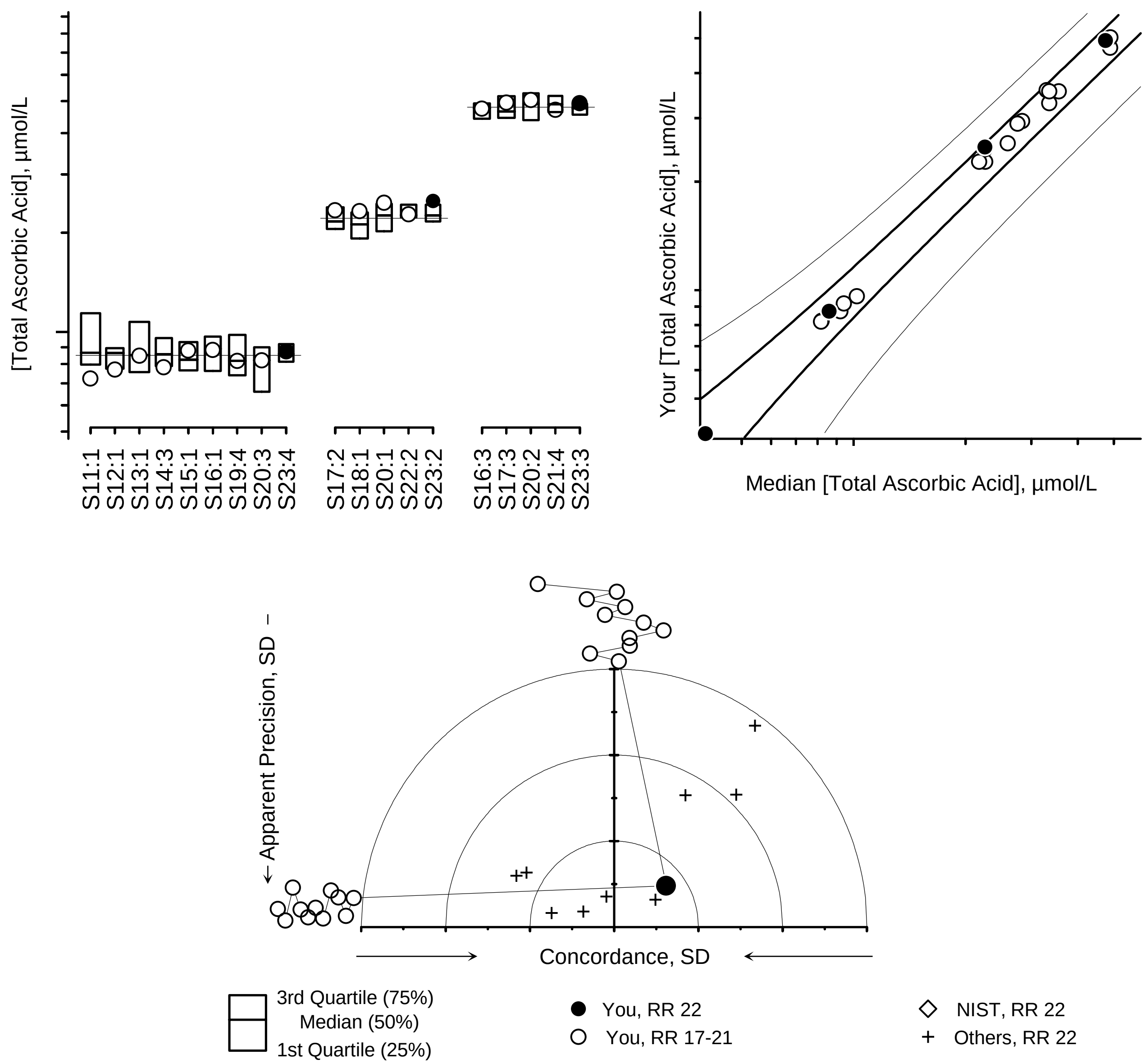
Vitamin C "Round Robin" 23 Report: Participant VC-MA

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
S23:1	Serum 14, a "blank" previously distributed in RRs 16, 19, 21, and 22
S23:2	Serum 31, previously distributed in RRs 17, 18, 20, and 22
S23:3	Serum 54, previously distributed in RRs 16, 17, 20, and 21
S23:4	SRM 970 Level 1, previously distributed in RRs 11, 12, 13, 14, 15, 16, 19, and 20