NISTIR 7880-23

# NIST Micronutrients Measurement Quality Assurance Program Winter 2001 Comparability Studies

Results for Round Robin XLIX
Fat-Soluble Vitamins and Carotenoids in Human Serum
and Round Robin 14 Ascorbic Acid in Human Serum

David L. Duewer
Sam A. Margolis (Retired)
Katherine E. Sharpless
Jeanice B. Thomas

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

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*Materials Measurement Laboratory*

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July, 2013

U.S. Department of Commerce
*Penny Pritzker, Secretary*

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

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## Abstract

The National Institute of Standards and Technology coordinates the Micronutrients Measurement Quality Assurance Program (MMQAP) for laboratories that measure fat- and water-soluble vitamins and carotenoids in human serum and plasma. This report describes the design of and results for the Winter 2001 MMQAP measurement comparability improvement studies: 1) Round Robin XLIX Fat-Soluble Vitamins and Carotenoids in Human Serum and 2) Round Robin 14 Total Ascorbic Acid in Human Serum. The materials for both studies were shipped to participants in January 2001. Participants in Round Robin XLIX were requested to provide their measurement results by March 16, 2001. Participants in Round Robin 14 were requested to provide their measurement results by May 12, 2001.



## Keywords

Human Serum
Retinol, α-Tocopherol, γ-Tocopherol, Total and *Trans*-β-Carotene
Total Ascorbic Acid

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## Introduction

Beginning in 1988, the National Institute of Standards and Technology (NIST) has coordinated the Micronutrients Measurement Quality Assurance Program (MMQAP) for laboratories that measure fat- and water-soluble vitamins and carotenoids in human serum and plasma. The MMQAP provides participants with measurement comparability assessment through use of interlaboratory studies, Standard Reference Materials (SRMs) and control materials, and methods development and validation. Serum-based samples with assigned values for the target analytes (retinol, alpha-tocopherol, gamma/beta-tocopherol, *trans*- and total beta-carotene, and total ascorbic acid) and performance-evaluation standards are distributed by NIST to laboratories for analysis.

Participants use the methodology of their choice to determine analyte content in the control and study materials. Participants provide their data to NIST, where it is compiled and evaluated for trueness relative to the NIST value, within-laboratory precision, and concordance within the participant community. NIST provides the participants with a technical summary report concerning their performance for each exercise and suggestions for methods development and refinement. Participants who have concerns regarding their laboratory's performance are encouraged to consult with the MMQAP coordinators.

All MMQAP interlaboratory studies consist of individual units of batch-prepared samples that are distributed to each participant. For historical reasons these studies are referred to as "Round Robins". The MMQAP program and the nature of its studies are described elsewhere. [1,2]

## Round Robin XLIX: Fat-Soluble Vitamins and Carotenoids in Human Serum

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin XLIX comparability study (hereafter referred to as RR49) received four lyophilized and one liquid-frozen human serum test samples for analysis. Unless multiple vials were previously requested, participants received one vial of each serum. These sera were shipped on dry ice to participants in January 2001. 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 RR49 consists of three documents:

- A cover letter for the current study, a brief description of the other two documents, and a discussion of our analysis of the overall results that may be of broad interest. This cover letter is reproduced as Appendix B.

- The "All-Lab Report" that lists all of the reported measurement results, a number of consensus statistics for analytes reported by more than one participant, and the mean median and pooled SD from any prior distributions of the serum. This report also provides a numerical "score card" for each participant's measurement comparability for the more commonly reported analytes. This report is reproduced as Appendix C.

- An “Individualized Report” that graphically analyzes each participant’s results for selected analytes. 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 14: Vitamin C in Human Serum

Participants in the MMQAP Vitamin C in Human Serum Round Robin 14 comparability study (hereafter referred to as RR14) received four frozen serum test samples and a solid ascorbic acid control material for analysis. Unless multiple vials were previously requested, participants received one vial of each material. These sample materials were shipped on dry ice to participants in January 2001. The communication materials included in the sample shipment are provided in Appendix E.

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

The final report delivered to every participant in RR14 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 RR49

The following three items were included in each package shipped to an RR49 participant:

- Cover letter
- Datasheet
- Packing List and Shipment Receipt Confirmation Form

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

NIST CENTENNIAL 1901-2001

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

January 16, 2001

Dear Colleague:

Happy New Year! Enclosed is the first set of samples for the quality assurance round robin exercise (Round Robin XLIX) for 2001. You will find one vial of each of one liquid-frozen and four lyophilized serum samples for analysis along with a form for reporting your results. When reporting your results, please submit one value for each analyte for a given serum sample. If an obtained value is below your limit of quantitation, please indicate this result on the form by using NQ *(Not Quantitated)*. For analytes not measured, please leave a blank. Results are due to NIST by **March 16, 2001**. Results received two weeks after the due date will not be included in the summary report for this round robin study. The feedback report concerning the study will be provided during the week of April 9, 2001.

Lyophilized samples should be reconstituted with 1.0 mL of HPLC-grade water or equivalent. We recommend that dissolution be facilitated with 3 to 5 min agitation in an ultrasonic bath or at least 30 min at room temperature with intermittent swirling. (CAUTION: Vigorous shaking will cause foaming and possibly interfere with accurate measurement. The rubber stopper contains phthalate esters that will 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). Liquid-frozen sample 271 should **not be** reconstituted.

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

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

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

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

Sincerely,

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

Enclosures

NIST

Participant #: _____ Date: _____

# Round Robin XLIX

## NIST Micronutrients Measurement Quality Assurance Program

| Analyte | 269 | 270 | 271 | 272 | 273 | Units* |
|---|---|---|---|---|---|---|
| total retinol | | | | | | |
| trans-retinol | | | | | | |
| retinyl palmitate | | | | | | |
| α-tocopherol | | | | | | |
| γ-tocopherol | | | | | | |
| δ-tocopherol | | | | | | |
| total β-carotene | | | | | | |
| trans-β-carotene | | | | | | |
| total cis-β-carotene | | | | | | |
| total α-carotene | | | | | | |
| trans-α-carotene | | | | | | |
| total lycopene | | | | | | |
| trans-lycopene | | | | | | |
| total β-cryptoxanthin | | | | | | |
| total α-cryptoxanthin | | | | | | |
| total lutein | | | | | | |
| total zeaxanthin | | | | | | |
| total lutein&zeaxanthin | | | | | | |
| ubiquinone-10 ($Q_{10}$) | | | | | | |
| phylloquinone ($K_1$) | | | | | | |
| 25-hydroxyvitamin D | | | | | | |
| cholesterol | | | | | | |

| Other analytes? | | | | | | |
|---|---|---|---|---|---|---|
| | | | | | | |
| | | | | | | |
| | | | | | | |

* we prefer μg/mL

Was serum 271 frozen when received? Yes | No

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

Please return results on-or-before
16-Mar-2001

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

Participant #: _____ Date: _____

# Round Robin XLIX

NIST Micronutrients Measurement Quality Assurance Program

## Packing List and Shipment Receipt Confirmation Form

The enclosed bubble-pack (should) contain one vial each of the following **five** sera:

| Serum | Form | Reconstitute? |
|---|---|---|
| #269 | Lyophilized | Yes (1 ml $H_2O$) |
| #270 | Lyophilized | Yes (1 ml $H_2O$) |
| #271 | Liquid frozen | No |
| #272 | Lyophilized | Yes (1 ml $H_2O$) |
| #273 | Lyophilized | Yes (1 ml $H_2O$) |

**Please** 1) Open the pack immediately
2) Check that it contains one vial each of the above samples
3) Check if serum #271 arrived frozen
4) Store the RR XLIX samples upright at -20 ˚C or below until analysis
5) Complete the following information
6) Fax the completed form to us at 301-977-0685
(or email requested information to david.duewer@nist.gov)

1) Date this shipment arrived: _____

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

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

4) Did serum #271 arrive frozen? Yes | No

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

6) When do you anticipate analyzing these samples? _____

Thank you in advance for your prompt response.

The $M^2$QAP Gang

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

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

## Appendix B. Final Report for RR49 and Folate Questionnaire

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

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

The fifth page of this section is a questionnaire that was distributed in the same mailing as the final report. This questionnaire was intended to assess the level of interest in the measurement of folates in serum and plasma.

NIST CENTENNIAL 1901-2001

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

April 25, 2001

Dear Colleague:

Enclosed is the summary report of the results for Round Robin XLIX (RR 49) for fat-soluble vitamins and carotenoids. Included in this report are: a summary of data for all laboratories; the measurement comparability summary for evaluating laboratory performance; lyophilized vs. fresh-frozen commutability data, a summary of individual laboratory performance and interlaboratory accuracy and precision; and a summary of the NIST assigned value (NAV) vs. your laboratory value for the analytes that you measured. As in previous reports, the NIST-assigned values are equally weighted means of the medians from this interlaboratory comparison exercise and the means from the analyses performed by NIST.

Data for evaluating laboratory performance in RR 49 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 deviation(s) of the assigned value, respectively; those rated 4 are >3 standard deviations from the assigned value.

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

The following are newly released or forthcoming publications. Reprints will be provided upon request.

***"Preparation and Value Assignment of Standard Reference Material 968c, Fat-Soluble Vitamins, Carotenoids, and Cholesterol in Human Serum,"*** **Brown Thomas et al., Clinica Chimica Acta 305 (2001) 141-155.**

***"The Stability of Ascorbic Acid in Autosampler Vials,"*** **Margolis, S.A. and Park, E., accepted by Clinical Chemistry, March 2001.**

The NIST Micronutrients Measurement Quality Assurance Workshop was held on April 4, 2001 as a symposium at the Experimental Biology (EB) meeting in Orlando, Florida. At the workshop, results from the past three interlaboratory comparison exercises (Round Robins 47-49), methodology updates, and program needs were discussed. There were 41 attendees at the workshop. As discussed at the session, the "better-than-expected" attendance was thought to be due to the workshop being held at the end of the conference instead of at the beginning. Since the EB meetings now begin a day earlier (on Saturday) than in the past, having the QA workshop at the end of the conference allows conferees to attend the EB meetings first and focus on the QA workshop afterwards. It was agreed that the next workshop will be held in conjunction with EB, which provides the appropriate audience, and will be held at the end of the conference. It was also suggested that our next QA workshop be held at EB 2003 in San Diego, CA. You will be notified about the next QA workshop as plans are made.

Most of the discussion for the afternoon session at the workshop concerned the measurement of folates in serum. Attendees expressed that quality assurance of folate measurements in serum and a folate-in-serum/plasma reference material are needed, and that they would like to start participating in round robin studies for folates in serum/plasma. To help us assess the level of interest in the measurement of folates in serum/plasma among the current QA program participants, **please return the enclosed questionnaire by May 31.**

NIST

Another topic discussed at the workshop was the need to have more than one vitamin C round robin study so that laboratories can meet the requirements of the Clinical Laboratory Improvement Amendments (CLIA). Since there were not enough representatives from laboratories that routinely measure vitamin C present at the workshop, a decision about increasing the number of studies could not be made at that time. Again, we would appreciate your feedback regarding this issue by completing the enclosed questionnaire. Additionally, please be reminded that **SRM 970, Vitamin C in Frozen Human Serum,** is now available. This material can be purchased through the Standard Reference Materials Program at 301-975-6776; fax: 301-948-3730; or at srminfo@nist.gov.

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

Sincerely,

Jeanice B Thomas

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

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

Enclosures

The NIST $M^2$QAP Round Robin XLIX (RR49) report consists of

| Page | "Individualized" Report |
|---|---|
| 1 | Your values, the number of labs reporting values, and our assigned values. |
| 2 to n | "Four Plot" summaries of your current and past measurement performance, one page for each analyte you report that is also reported by at least 10 other participants. |
| n+1 | The "target" plot version of your "Comparability Summary" scores. |
| n+2 | An experimental "Commutability" plot summarizing your measurements in the five liquid-frozen / lyophilized sample pairs distributed in RR47, RR48, and RR49. |

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

**Samples**. Five sera were distributed in RR49.

Serum #269 is a lyophilized material that has been distributed in three previous studies, as #193 in RR30 (3/94), #254 in RR45 (3/99), and #255 in RR46 (6/99). It was prepared from a native serum pool augmented with "retinol" that we now believe was highly enriched in *cis*-isomers. Up to 30% of the total retinol in this sample may be in the form of *cis*-retinols.

Serum #270 is a lyophilized material prepared from a native serum pool. The liquid-frozen partner of this sample was distributed as #267 in RR48. Sera #267 and #270 were prepared and aliquoted into vials as a single batch.

Serum #271 is a liquid-frozen material prepared from a native serum pool. The lyophilized partner of this sample was distributed as #266 in RR48. Sera #266 and #271 were prepared and aliquoted into vials as a single batch.

Serum #272 is a lyophilized material that has been distributed in two previous studies, as #223 and #225 in RR38 (9/96) and #228 in RR39 (3/97). It was prepared from a native serum pool augmented with retinyl palmitate.

Serum #273 is a lyophilized material that was distributed as #203 in RR33 (3/95). It was prepared from a native carotenoid-deficient serum augmented with retinol, retinyl palmitate, ($\alpha$, $\gamma$, $\delta$)-tocopherols, ($\alpha$, $\beta$)-carotenes, and lycopene.

**Qualitative Observations**.

1) Several participants noted the presence an "insoluble stringy clot" in Serum #271. As noted in the RR48 report for #266, we failed to re-filter the final blend of the serum pool used for this sample. As with its lyophilized partner, the resulting solids do **not** appear to have affected measurements.

2) One participant noted that what we've been reporting as $\gamma$-tocopherol is "really" $\gamma$- and/or $\beta$-tocopherol. We now denote this measurand as "$\gamma/\beta$-tocopherol.

3) All participants received their samples still frozen. We thank all of you who promptly confirmed receipt of the samples. Such prompt confirmation greatly simplifies our package delivery tracking and thus helps contain $M^2$QAP costs. *We will be requesting similar confirmation in all future studies.*

**Quantitative Results**

1) With the possible exception of zeaxanthin, there have been no changes in the median level of any measurand in sera #269 (7 years storage), #272 (5 years), or #273 (6 years). There is, however, evidence of increased variability in retinol and α-tocopherol measurements, particularly in sera #272 and #273. We believe that this increased variability may be related to contamination of many of the lyophilized sera prepared in the past 6 years. We will discuss this more fully in the RR50 report.

   As discussed in the RR45 report for serum #254, perhaps 30% of the retinol in Serum #269 is in the form of *cis*-isomers. While we did not intentionally spike this serum with *cis*-retinol, our methods indicate an unusually large retinol shoulder peak that is compatible with the presence of one or more of the *cis*-forms.

2) The distribution of reported "retinol-related measurand" interlaboratory values is bimodal, with approximately equal numbers centered at about 0.9 μg/mL and 1.1 μg/mL. Somewhat distressingly, many of your are **not** correctly specifying the form you are actually measuring. Many of the reported "Total Retinol" values are more compatible with being "*trans*-Retinol"; many of reported "*trans*-Retinol" values are more compatible with being "Total".

   If you report "Total Retinol" and your #269 value isn't closer to 0.9 μg/mL than to 1.1 μg/mL, we suggest that you check your integration protocol to ensure that you are including *cis*-retinol isomers. If you report "*trans*-Retinol" and your #269 value isn't closer to 1.1 μg/mL than to 0.9 μg/mL, we suggest that you confirm that your separation system does adequately resolve *cis*- and *trans*-retinol isomers. We intend to address this issue in the near future with explicitly *cis*-augmented samples.

3) A total of five matched {liquid-frozen, lyophilized} matrix pairs have now been distributed: {261,259} and {262,260} in RR47, {265,268} in RR48, and {266,271} and {270,267} in RR48 and RR49. Since these pairs were prepared at the same time from the same serum pools, the ratio of the values in the two matrices (liquid-frozen / lyophilized) is a direct assessment of measurement commutability between the two matrices. The last page of your "Individualized Report" summarizes the community's and your individual commutability ratios.

   We have not yet completed the detailed analysis of these data; however, a number of interesting results are already apparent:

   The average (liquid-frozen / lyophilized) ratio is 0.94, essentially identical to the 0.95 expected from serum density consideration. There is no systematic variation in this ratio across measurands. While there is considerable variability about this average with most of the "minor" carotenoids, much of this variability in the ratios is a consequence of limited reporting precision at low measurand levels (e.g., 0.0034/0.0036 = 0.94 but 0.003/0.004 = 0.75).

   There are several participants with retinol and/or tocopherol commutability ratios that are consistently above or below 0.94, indicating a systematic difference between the matrices in their measurement systems. We believe that this arises from contamination of the lyophilized samples used rather than a lyophilization-specific matrix effect.

   Note: since the {266,271} and {270,267} ratios involve measurements made half a year apart, the average of these two ratios (in logarithmic form) estimates commutability while the difference between the ratios estimates within-laboratory calibration differences between RR48 and RR49.

Lab number:_____
Phone: _____
Fax:_____
E-mail:_____

## Questionnaire

## NIST Micronutrients Measurement Quality Assurance Program

*Please provide us with your input by completing this form and returning it by* ***May 31, 2001*** *to:*

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

_____ Folates in serum/plasma are currently measured in my organization by:

_____ _____ _____
*Name of the contact* *Phone* *Fax*

_____
*E-mail address*

_____ I would participate and be willing to pay for a second annual QA interlaboratory comparison study for vitamin C if it were available.

_____ I am required to comply with the Clinical Laboratory Improvement Amendments and/or another laboratory standards program for the measurement of:

☐ vitamin A
☐ carotenes
☐ vitamin C
☐ vitamin E
☐ vitamin D
☐ vitamin $K_1$
☐ coenzyme $Q_{10}$
☐ other *(please specify)*_____

Comments:

## Appendix C. "All-Lab Report" for RR49

The following eight 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 XLIX Laboratory Results

All Values in µg/mL

| | Total Retinol | | | | | trans-Retinol | | | | | Retinyl Palmitate | | | | | α-Tocopherol | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Lab | 269 | 270 | 271 | 272 | 273 | 269 | 270 | 271 | 272 | 273 | 269 | 270 | 271 | 272 | 273 | 269 | 270 | 271 | 272 | 273 |
| FSV-BA | 1.25 | 0.688 | 0.495 | 0.564 | 1.35 | | | | | | 0.070 | *nd* | 0.072 | 0.115 | 0.066 | 28.9 | 6.15 | 7.21 | 8.11 | 17.6 |
| FSV-BB | 1.27 | 0.714 | 0.519 | 0.542 | 1.32 | | | | | | 0.017 | 0.008 | 0.054 | 0.068 | 0.050 | 29.8 | 6.52 | 8.01 | 8.82 | 18.6 |
| FSV-BD | 1.02 | 0.619 | 0.451 | 0.487 | 1.15 | | | | | | | | | | | 26.6 | 6.50 | 6.50 | 7.40 | 16.0 |
| FSV-BE | 1.04 | 0.660 | 0.450 | 0.570 | 1.37 | | | | | | | | | | | 25.1 | 5.50 | 6.40 | 7.40 | 16.0 |
| FSV-BF | 1.01 | 0.640 | 0.430 | 0.500 | 1.26 | | | | | | | | | | | 29.1 | 6.20 | 7.10 | 7.90 | 17.5 |
| FSV-BG | 1.24 | 0.653 | 0.483 | 0.533 | 1.35 | | | | | | 0.046 | 0.047 | 0.065 | 0.049 | 0.055 | 30.1 | 6.07 | 7.67 | 8.28 | 17.5 |
| FSV-BH | 0.97 | 0.612 | 0.431 | 0.425 | 1.12 | | | | | | | | | | | 27.2 | 6.00 | 6.85 | 7.50 | 16.0 |
| FSV-BI | 1.22 | 0.680 | 0.503 | 0.582 | 1.37 | | | | | | 0.024 | 0.023 | 0.066 | 0.068 | 0.063 | 27.7 | 6.18 | 7.53 | 8.56 | 17.1 |
| FSV-BJ | 1.11 | 0.634 | 0.460 | 0.509 | 1.21 | | | | | | *nq* | *nq* | 0.049 | 0.091 | 0.074 | 26.4 | 5.94 | 7.14 | 7.85 | 17.6 |
| FSV-BK | 1.03 | 0.649 | 0.421 | 0.516 | 1.28 | | | | | | | | | | | 25.5 | 5.25 | 5.70 | 6.87 | 16.0 |
| FSV-BL | 0.40 | 0.600 | 0.460 | 0.400 | 0.69 | | | | | | | | | | | 21.5 | 10.34 | 9.04 | 8.61 | 10.8 |
| FSV-BM | 0.98 | 0.668 | 0.511 | 0.516 | 1.11 | | | | | | | | | | | 32.8 | 6.00 | 7.40 | 8.40 | 19.7 |
| FSV-BN | 1.08 | 0.630 | 0.470 | 0.494 | 1.31 | | | | | | 0.018 | 0.016 | 0.048 | 0.050 | 0.043 | 25.6 | 5.89 | 7.30 | 7.68 | 17.0 |
| FSV-BO | 1.01 | 0.610 | 0.513 | 0.504 | 1.35 | | | | | | | | | | | 24.9 | 5.30 | 7.30 | 7.20 | 16.8 |
| FSV-BP | 1.00 | 0.590 | 0.436 | 0.460 | 1.12 | | | | | | | | | | | 27.2 | 6.52 | 7.07 | 7.28 | 16.2 |
| FSV-BQ | 1.17 | 0.674 | 0.496 | 0.550 | 1.33 | | | | | | | | | | | 24.4 | 5.07 | 6.22 | 7.44 | 15.8 |
| FSV-BR | >1.04 | >0.600 | >0.440 | >0.500 | >1.15 | 1.04 | 0.600 | 0.440 | 0.500 | 1.15 | | | | | | 24.5 | 4.90 | 5.86 | 6.96 | 14.9 |
| FSV-BS | >1.05 | >0.590 | >0.440 | >0.500 | >1.18 | 1.05 | 0.590 | 0.440 | 0.500 | 1.18 | | | | | | | | | | |
| FSV-BT | 1.12 | 0.599 | 0.643 | 0.506 | 1.24 | | | | | | 0.026 | 0.014 | 0.046 | 0.081 | 0.070 | 26.8 | 5.83 | 5.56 | 7.59 | 16.6 |
| FSV-BU | 0.88 | 0.603 | 0.458 | 0.413 | 1.18 | | | | | | | | | | | 27.6 | 5.43 | 6.83 | 7.35 | 16.8 |
| FSV-BV | 0.94 | 0.570 | 0.440 | 0.450 | 1.10 | | | | | | | | | | | 26.7 | 5.94 | 7.71 | 7.71 | 16.8 |
| FSV-BW | 1.19 | 0.660 | 0.490 | 0.540 | 1.36 | | | | | | 0.022 | 0.018 | 0.062 | 0.071 | 0.076 | 28.4 | 6.04 | 7.32 | 8.10 | 17.5 |
| FSV-BX | >1.02 | >0.62 | >0.46 | >0.50 | >1.14 | 1.02 | 0.620 | 0.455 | 0.501 | 1.14 | | | | | | 25.0 | 5.65 | 6.90 | 7.54 | 15.9 |
| FSV-CB | 0.95 | 0.591 | 0.419 | 0.473 | 1.18 | | | | | | | | | | | 25.7 | 5.96 | 7.03 | 7.67 | 16.2 |
| FSV-CC | 1.11 | 0.670 | 0.460 | 0.510 | 1.26 | 1.03 | 0.630 | 0.450 | 0.490 | 1.21 | | | | | | 27.9 | 6.10 | 7.00 | 7.50 | 16.2 |
| FSV-CD | >1.18 | >0.628 | >0.450 | >0.505 | >1.19 | 1.18 | 0.628 | 0.450 | 0.505 | 1.19 | 0.013 | 0.033 | 0.022 | 0.179 | 0.041 | 27.6 | 5.94 | 6.99 | 8.36 | 17.0 |
| FSV-CE | 1.16 | 0.670 | 0.510 | 0.570 | 1.30 | | | | | | | | | | | 29.5 | 6.70 | 8.07 | 9.08 | 17.2 |
| FSV-CF | 1.08 | 0.663 | 0.474 | 0.512 | 1.31 | | | | | | | | | | | 25.5 | 6.50 | 7.90 | 8.20 | 16.5 |
| FSV-CG | 1.13 | 0.715 | 0.510 | 0.560 | 1.28 | | | | | | | | | | | 24.7 | 5.59 | 6.75 | 7.19 | 13.8 |
| FSV-CH | 0.94 | 0.542 | 0.376 | 0.431 | 1.01 | | | | | | | | | | | 26.3 | 5.92 | 7.01 | 7.92 | 15.3 |
| FSV-CI | 0.88 | 0.710 | 0.520 | 0.550 | 1.42 | | | | | | 0.030 | 0.030 | 0.060 | 0.060 | 0.050 | 31.2 | 5.60 | 6.70 | 7.60 | 16.9 |
| FSV-CL | 0.99 | 0.534 | 0.389 | 0.396 | 1.08 | | | | | | | | | | | 31.6 | 7.54 | 9.01 | 9.89 | 19.3 |
| FSV-CR | 1.14 | 0.720 | 0.490 | 0.550 | 1.35 | | | | | | | | | | | 28.3 | 6.00 | 6.90 | 7.90 | 18.2 |
| FSV-CV | 0.49 | 0.488 | 0.283 | 0.205 | 0.58 | | | | | | 0.110 | 0.083 | 0.103 | 0.144 | 0.120 | 30.3 | 7.69 | 7.82 | 9.04 | 16.9 |
| FSV-CW | >0.99 | >0.575 | >0.401 | >0.441 | >1.15 | 0.99 | 0.575 | 0.401 | 0.441 | 1.15 | 0.012 | 0.016 | 0.046 | 0.065 | 0.065 | 27.4 | 5.82 | 6.86 | 7.60 | 16.7 |
| FSV-CZ | 1.12 | 0.690 | 0.510 | 0.550 | 1.47 | | | | | | | | | | | 15.9 | 1.60 | 2.38 | 2.67 | 10.7 |
| FSV-DB | 1.14 | 0.650 | 0.470 | 0.540 | 1.34 | | | | | | | | | | | 28.1 | 6.43 | 7.57 | 8.60 | 18.0 |
| FSV-DD | >1.04 | >0.600 | >0.417 | >0.525 | >1.09 | 1.04 | 0.600 | 0.417 | 0.525 | 1.09 | | | | | | | | | | |
| FSV-DF | 1.06 | 0.605 | 0.433 | 0.492 | 1.16 | | | | | | | | | | | | | | | |
| FSV-DI | >0.98 | >0.606 | >0.428 | >0.466 | >1.14 | 0.98 | 0.606 | 0.428 | 0.466 | 1.14 | 0.031 | 0.036 | 0.028 | 0.046 | 0.040 | 28.4 | 6.21 | 7.28 | 8.11 | 17.4 |
| FSV-DQ | | | | | | | | | | | | | | | | 36.8 | 7.04 | 10.24 | 13.42 | 26.2 |
| FSV-DR | 1.19 | 0.650 | 0.490 | 0.510 | 1.33 | | | | | | | | | | | 31.9 | 6.58 | 7.99 | 8.23 | 19.2 |
| FSV-DU | >1.16 | >0.576 | >0.406 | >0.491 | >1.20 | 1.16 | 0.576 | 0.406 | 0.491 | 1.21 | | | | | | 24.4 | 4.32 | 5.51 | 6.19 | 14.3 |
| FSV-EH | >1.07 | >0.549 | >0.468 | >0.508 | >1.26 | 1.07 | 0.549 | 0.468 | 0.508 | 1.26 | 0.031 | 0.032 | 0.070 | 0.070 | 0.062 | 28.1 | 5.75 | 6.59 | 7.00 | 16.6 |
| FSV-EQ | 1.24 | 0.678 | 0.523 | 0.572 | 1.34 | | | | | | | | | | | 31.3 | 6.90 | 8.00 | 8.80 | 11.6 |
| FSV-FB | >1.33 | >0.760 | >0.660 | >0.773 | >1.76 | 1.33 | 0.760 | 0.660 | 0.773 | 1.76 | | | | | | 28.0 | 5.96 | 7.15 | 8.37 | 18.0 |
| FSV-FJ | 1.01 | 0.640 | 0.470 | 0.470 | 1.05 | | | | | | | | | | | 27.7 | 6.08 | 7.43 | 8.06 | 15.4 |
| N | 36 | 36 | 36 | 36 | 36 | 11 | 11 | 11 | 11 | 11 | 13 | 12 | 14 | 14 | 14 | 44 | 44 | 44 | 44 | 44 |
| Min | 0.40 | 0.488 | 0.283 | 0.205 | 0.58 | 0.98 | 0.549 | 0.401 | 0.441 | 1.09 | 0.012 | 0.008 | 0.022 | 0.046 | 0.040 | 15.9 | 1.60 | 2.38 | 2.67 | 10.7 |
| Median | 1.07 | 0.650 | 0.470 | 0.510 | 1.28 | 1.04 | 0.600 | 0.440 | 0.500 | 1.18 | 0.026 | 0.027 | 0.057 | 0.069 | 0.063 | 27.6 | 6.00 | 7.12 | 7.87 | 16.8 |
| Max | 1.27 | 0.720 | 0.643 | 0.582 | 1.47 | 1.33 | 0.760 | 0.660 | 0.773 | 1.76 | 0.110 | 0.083 | 0.103 | 0.179 | 0.120 | 36.8 | 10.34 | 10.24 | 13.42 | 26.2 |
| eSD | 0.13 | 0.050 | 0.050 | 0.059 | 0.11 | 0.04 | 0.036 | 0.022 | 0.013 | 0.05 | 0.012 | 0.015 | 0.015 | 0.023 | 0.018 | 2.8 | 0.56 | 0.62 | 0.70 | 1.2 |
| eCV | 12 | 8 | 11 | 12 | 9 | 4 | 6 | 5 | 3 | 4 | 46 | 56 | 26 | 34 | 28 | 10 | 9 | 9 | 9 | 7 |
| $N_{past}$ | 47 | 44 | 44 | 49 | 49 | 7 | 11 | 11 | 0 | 0 | 11 | 11 | 15 | 15 | 9 | 47 | 52 | 52 | 47 | 47 |
| $Median_{past}$ | 1.04 | 0.671 | 0.446 | 0.498 | 1.27 | 1.00 | 0.668 | 0.440 | | | 0.041 | 0.028 | 0.053 | 0.091 | 0.129 | 26.9 | 6.36 | 6.81 | 7.72 | 17.1 |
| $SD_{past}$ | 0.10 | 0.073 | 0.044 | 0.039 | 0.11 | 0.15 | 0.028 | 0.010 | | | 0.040 | 0.010 | 0.016 | 0.044 | 0.061 | 2.2 | 0.70 | 0.67 | 0.59 | 1.5 |
| NISTa | 0.91 | 0.599 | 0.454 | 0.478 | 1.13 | 0.71 | 0.599 | 0.454 | 0.478 | 1.13 | | | | | | 23.9 | 5.28 | 6.76 | 7.14 | 16.3 |
| NISTb | 0.92 | 0.621 | 0.403 | 0.468 | 1.11 | | | | | | | | | | | 28.5 | 5.55 | 5.94 | 7.21 | 15.5 |
| $N_{NIST}$ | 3 | 4 | 4 | 4 | 4 | 2 | 2 | 2 | 2 | 2 | | | | | | 3 | 4 | 4 | 4 | 4 |
| Mean | 0.92 | 0.610 | 0.428 | 0.473 | 1.13 | 0.71 | 0.599 | 0.454 | 0.478 | 1.13 | | | | | | 26.2 | 5.41 | 6.35 | 7.18 | 15.9 |
| $S_{rep}$ | 0.02 | 0.017 | 0.016 | 0.008 | 0.02 | 0.02 | 0.024 | 0.019 | 0.010 | 0.00 | | | | | | 0.3 | 0.14 | 0.29 | 0.12 | 0.2 |
| $S_{het}$ | 0.00 | 0.038 | 0.004 | 0.037 | 0.05 | 0.02 | 0.009 | 0.003 | 0.009 | 0.01 | | | | | | 0.4 | 0.32 | 0.10 | 0.40 | 0.1 |
| $S_{anl}$ | 0.01 | 0.016 | 0.036 | 0.007 | 0.00 | | | | | | | | | | | 3.3 | 0.19 | 0.58 | 0.05 | 0.5 |
| $S_{NIST}$ | 0.02 | 0.045 | 0.040 | 0.038 | 0.06 | 0.03 | 0.025 | 0.019 | 0.014 | 0.01 | | | | | | 3.3 | 0.39 | 0.65 | 0.42 | 0.6 |
| NAV | 0.99 | 0.630 | 0.449 | 0.491 | 1.20 | 0.88 | 0.599 | 0.447 | 0.489 | 1.16 | 0.026 | 0.027 | 0.057 | 0.069 | 0.063 | 26.9 | 5.71 | 6.74 | 7.52 | 16.3 |
| NAU | 0.17 | 0.059 | 0.058 | 0.065 | 0.16 | 0.25 | 0.048 | 0.037 | 0.043 | 0.10 | 0.012 | 0.015 | 0.017 | 0.023 | 0.019 | 3.5 | 0.69 | 0.85 | 0.86 | 1.5 |

# Round Robin XLIX Laboratory Results

All Values in µg/mL

| | γ/β-Tocopherol | | | | | δ-Tocopherol | | | | | Total β-Carotene | | | | | trans-β-Carotene | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Lab | 269 | 270 | 271 | 272 | 273 | 269 | 270 | 271 | 272 | 273 | 269 | 270 | 271 | 272 | 273 | 269 | 270 | 271 | 272 | 273 |
| FSV-BA | 6.42 | 2.29 | 1.96 | 1.76 | 1.22 | | | | | | 0.347 | 0.480 | 0.331 | 0.165 | 0.051 | 0.316 | 0.460 | 0.323 | 0.155 | 0.051 |
| FSV-BB | 6.18 | 2.30 | 2.03 | 1.78 | 1.21 | | | | | | 0.366 | 0.490 | 0.355 | 0.158 | 0.063 | 0.338 | 0.470 | 0.338 | 0.155 | 0.045 |
| FSV-BD | | | | | | | | | | | 0.376 | 0.548 | 0.367 | 0.192 | 0.074 | | | | | |
| FSV-BE | 5.90 | 2.10 | 1.80 | 1.70 | 1.10 | | | | | | 0.357 | 0.486 | 0.295 | 0.156 | 0.050 | | | | | |
| FSV-BF | 5.60 | 2.50 | 1.90 | 1.70 | 1.10 | | | | | | 0.440 | 0.586 | 0.410 | 0.169 | 0.031 | | | | | |
| FSV-BG | 6.72 | 2.23 | 2.06 | 1.88 | 1.20 | | | | | | 0.366 | 0.543 | 0.350 | 0.174 | 0.058 | | | | | |
| FSV-BH | 6.56 | 2.43 | 2.06 | 1.84 | 1.24 | | | | | | 0.375 | 0.505 | 0.357 | 0.168 | 0.048 | 0.342 | 0.481 | 0.336 | 0.159 | 0.048 |
| FSV-BI | 6.47 | 2.43 | 2.17 | 2.01 | 1.23 | | | | | | 0.360 | 0.518 | 0.373 | 0.172 | 0.056 | | | | | |
| FSV-BJ | 6.23 | 2.33 | 2.05 | 1.81 | 1.31 | | | | | | 0.397 | 0.565 | 0.451 | 0.172 | 0.060 | | | | | |
| FSV-BK | | | | | | | | | | | | | | | | | | | | |
| FSV-BL | | | | | | | | | | | | | | | | | | | | |
| FSV-BM | | | | | | | | | | | | | | | | | | | | |
| FSV-BN | 4.72 | 1.79 | 1.62 | 1.37 | 0.96 | 0.700 | 0.250 | 0.310 | 0.340 | 3.39 | 0.336 | 0.471 | 0.352 | 0.148 | 0.053 | 0.300 | 0.442 | 0.324 | 0.140 | 0.052 |
| FSV-BO | | | | | | | | | | | 0.367 | 0.506 | 0.410 | 0.180 | 0.064 | | | | | |
| FSV-BP | | | | | | | | | | | 0.380 | 0.453 | 0.310 | 0.183 | *nd* | | | | | |
| FSV-BQ | | | | | | | | | | | | | | | | | | | | |
| FSV-BR | | | | | | | | | | | | | | | | | | | | |
| FSV-BS | | | | | | | | | | | >0.222 | >0.292 | >0.180 | >0.082 | >0.034 | 0.222 | 0.292 | 0.180 | 0.082 | 0.034 |
| FSV-BT | 5.86 | 2.32 | 1.56 | 1.68 | 1.20 | 0.757 | 0.748 | 0.233 | 0.336 | 3.38 | 0.314 | 0.436 | 0.317 | 0.143 | 0.057 | 0.288 | 0.416 | 0.298 | 0.135 | 0.053 |
| FSV-BU | 6.16 | 2.30 | 2.05 | 1.73 | 1.12 | | | | | | 0.375 | 0.535 | 0.362 | 0.175 | 0.058 | | | | | |
| FSV-BV | 6.44 | 2.48 | 2.21 | 1.89 | 1.28 | | | | | | 0.351 | 0.506 | 0.365 | 0.169 | 0.057 | | | | | |
| FSV-BW | 6.75 | 2.46 | 2.17 | 1.96 | 1.24 | | | | | | 0.350 | 0.500 | 0.350 | 0.160 | 0.054 | | | | | |
| FSV-BX | 6.02 | 2.22 | 1.96 | 1.75 | 1.13 | | | | | | >0.317 | >0.467 | >0.336 | >0.165 | >0.076 | 0.317 | 0.467 | 0.336 | 0.165 | 0.076 |
| FSV-CB | | | | | | | | | | | 0.367 | 0.497 | 0.345 | 0.170 | 0.056 | | | | | |
| FSV-CC | | | | | | | | | | | | | | | | | | | | |
| FSV-CD | 5.77 | 2.24 | 1.94 | 1.90 | 1.24 | | | | | | 0.390 | 0.515 | 0.332 | 0.140 | 0.060 | | | | | |
| FSV-CE | | | | | | | | | | | 0.390 | 0.570 | 0.430 | 0.200 | 0.050 | | | | | |
| FSV-CF | | | | | | | | | | | | | | | | | | | | |
| FSV-CG | 6.32 | 2.29 | 2.11 | 1.82 | 1.21 | 0.492 | 0.123 | 0.171 | 0.228 | 2.48 | 0.368 | 0.472 | 0.352 | 0.164 | 0.059 | 0.334 | 0.446 | 0.328 | 0.154 | 0.052 |
| FSV-CH | 5.65 | 2.17 | 1.87 | 1.61 | 1.06 | | | | | | 0.233 | 0.440 | 0.322 | 0.153 | 0.051 | | | | | |
| FSV-CI | 6.00 | 1.80 | 1.70 | 1.60 | 1.10 | | | | | | | | | | | | | | | |
| FSV-CL | 6.84 | 2.73 | 2.39 | 1.95 | 0.98 | | | | | | 0.321 | 0.432 | 0.292 | 0.153 | 0.049 | | | | | |
| FSV-CR | | | | | | | | | | | | | | | | | | | | |
| FSV-CV | 16.17 | 5.81 | 4.36 | 4.15 | 2.53 | 0.432 | 0.111 | 0.118 | 0.166 | 2.64 | 0.467 | 0.691 | 0.394 | 0.173 | 0.038 | | | | | |
| FSV-CW | 6.31 | 2.32 | 2.01 | 1.80 | 1.23 | 0.390 | 0.090 | 0.110 | 0.160 | 3.14 | 0.344 | 0.469 | 0.332 | 0.157 | 0.055 | 0.310 | 0.442 | 0.308 | 0.147 | 0.051 |
| FSV-CZ | | | | | | | | | | | 0.380 | 0.530 | 0.410 | 0.210 | 0.110 | | | | | |
| FSV-DB | | | | | | | | | | | 0.400 | 0.530 | 0.360 | 0.170 | 0.070 | | | | | |
| FSV-DD | | | | | | | | | | | | | | | | | | | | |
| FSV-DF | | | | | | | | | | | | | | | | | | | | |
| FSV-DI | 5.60 | 2.03 | 1.73 | 1.53 | 1.01 | 0.378 | 0.084 | 0.106 | 0.153 | 3.22 | >0.320 | >0.439 | >0.293 | >0.135 | >0.039 | 0.320 | 0.439 | 0.293 | 0.135 | 0.039 |
| FSV-DQ | 9.39 | 2.76 | 2.73 | 2.83 | 1.92 | | | | | | 0.485 | 0.431 | 0.483 | 0.337 | 0.106 | | | | | |
| FSV-DR | | | | | | | | | | | 0.350 | 0.528 | 0.390 | 0.119 | 0.070 | | | | | |
| FSV-DU | | | | | | | | | | | >0.274 | >0.376 | >0.252 | >0.118 | >0.021 | 0.274 | 0.376 | 0.252 | 0.118 | 0.021 |
| FSV-EH | 6.06 | 2.01 | 1.92 | 1.65 | 1.24 | 0.313 | 0.106 | 0.118 | 0.196 | 2.15 | 0.339 | 0.463 | 0.337 | 0.158 | 0.070 | 0.281 | 0.419 | 0.296 | 0.135 | 0.048 |
| FSV-EQ | | | | | | | | | | | 0.335 | 0.471 | 0.328 | 0.165 | 0.052 | 0.323 | 0.463 | 0.328 | 0.162 | 0.052 |
| FSV-FB | | | | | | | | | | | 0.326 | 0.390 | 0.313 | 0.178 | 0.073 | 0.215 | 0.317 | 0.256 | 0.134 | 0.042 |
| FSV-FJ | 5.53 | 2.18 | 1.89 | 1.63 | 0.95 | | | | | | 0.310 | 0.320 | 0.150 | 0.160 | 0.030 | | | | | |
| N | 25 | 25 | 25 | 25 | 25 | 7 | 7 | 7 | 7 | 7 | 32 | 32 | 32 | 32 | 31 | 14 | 14 | 14 | 14 | 14 |
| Min | 4.72 | 1.79 | 1.56 | 1.37 | 0.95 | 0.313 | 0.084 | 0.106 | 0.153 | 2.15 | 0.233 | 0.320 | 0.150 | 0.119 | 0.030 | 0.215 | 0.292 | 0.180 | 0.082 | 0.021 |
| Median | 6.18 | 2.30 | 2.01 | 1.78 | 1.21 | 0.432 | 0.111 | 0.118 | 0.196 | 3.14 | 0.366 | 0.499 | 0.352 | 0.169 | 0.057 | 0.313 | 0.442 | 0.316 | 0.144 | 0.050 |
| Max | 16.17 | 5.81 | 4.36 | 4.15 | 2.53 | 0.757 | 0.748 | 0.310 | 0.340 | 3.39 | 0.485 | 0.691 | 0.483 | 0.337 | 0.110 | 0.342 | 0.481 | 0.338 | 0.165 | 0.076 |
| eSD | 0.48 | 0.20 | 0.18 | 0.16 | 0.11 | 0.089 | 0.031 | 0.018 | 0.053 | 0.37 | 0.030 | 0.047 | 0.034 | 0.015 | 0.009 | 0.034 | 0.036 | 0.030 | 0.016 | 0.005 |
| eCV | 8 | 9 | 9 | 9 | 9 | 21 | 28 | 15 | 27 | 12 | 8 | 9 | 10 | 9 | 16 | 11 | 8 | 9 | 11 | 10 |
| $N_{past}$ | 26 | 29 | 29 | 25 | 22 | 10 | 6 | 6 | 7 | 0 | 35 | 33 | 33 | 34 | 35 | 13 | 17 | 17 | 12 | 11 |
| $Median_{past}$ | 6.03 | 2.38 | 1.85 | 1.71 | 1.22 | 0.550 | 0.121 | 0.152 | 0.165 | | 0.369 | 0.515 | 0.307 | 0.171 | 0.056 | 0.325 | 0.470 | 0.287 | 0.158 | 0.056 |
| $SD_{past}$ | 0.65 | 0.25 | 0.17 | 0.15 | 0.17 | 0.249 | 0.048 | 0.062 | 0.038 | | 0.051 | 0.077 | 0.068 | 0.022 | 0.017 | 0.047 | 0.061 | 0.042 | 0.020 | 0.012 |
| NISTa | 5.90 | 2.08 | 1.96 | 1.66 | 1.08 | 0.268 | *nq* | *nq* | *nq* | 2.06 | 0.327 | 0.402 | 0.273 | 0.139 | 0.046 | 0.246 | 0.345 | 0.251 | <0.139 | <0.046 |
| NISTb | 6.18 | 2.04 | 1.57 | 1.58 | 1.11 | 0.403 | 0.072 | 0.128 | 0.136 | 2.50 | 0.371 | >0.453 | >0.285 | >0.153 | >0.048 | 0.343 | 0.453 | 0.285 | 0.153 | 0.048 |
| $N_{NIST}$ | 3 | 4 | 4 | 4 | 4 | 3 | 2 | 2 | 2 | 4 | 3 | 2 | 2 | 2 | 2 | 3 | 4 | 4 | 2 | 1 |
| Mean | 6.04 | 2.06 | 1.76 | 1.62 | 1.12 | 0.337 | 0.072 | 0.128 | 0.136 | 2.30 | 0.349 | 0.402 | 0.273 | 0.139 | 0.046 | 0.295 | 0.399 | 0.268 | 0.153 | 0.048 |
| $S_{rep}$ | 0.22 | 0.06 | 0.09 | 0.05 | 0.03 | 0.023 | 0.004 | 0.008 | 0.009 | 0.08 | 0.005 | 0.015 | 0.009 | 0.005 | 0.007 | 0.005 | 0.010 | 0.012 | 0.003 | 0.003 |
| $S_{het}$ | 0.02 | 0.15 | 0.10 | 0.08 | 0.15 | 0.010 | 0.001 | 0.011 | 0.011 | 0.10 | 0.001 | 0.008 | 0.006 | 0.007 | 0.001 | 0.008 | 0.026 | 0.003 | 0.009 | 0.000 |
| $S_{anl}$ | 0.19 | 0.02 | 0.27 | 0.05 | 0.06 | 0.093 | | | | 0.34 | 0.031 | | | | | 0.069 | 0.076 | 0.024 | | |
| $S_{NIST}$ | 0.29 | 0.17 | 0.30 | 0.11 | 0.17 | 0.096 | 0.004 | 0.014 | 0.014 | 0.36 | 0.032 | 0.017 | 0.011 | 0.008 | 0.007 | 0.069 | 0.081 | 0.027 | 0.010 | 0.003 |
| NAV | 6.11 | 2.18 | 1.89 | 1.70 | 1.16 | 0.385 | 0.091 | 0.123 | 0.166 | 2.72 | 0.358 | 0.450 | 0.313 | 0.154 | 0.052 | 0.304 | 0.420 | 0.292 | 0.148 | 0.049 |
| NAU | 0.52 | 0.29 | 0.35 | 0.22 | 0.18 | 0.117 | 0.042 | 0.027 | 0.068 | 0.70 | 0.054 | 0.097 | 0.075 | 0.034 | 0.014 | 0.070 | 0.087 | 0.048 | 0.018 | 0.009 |

# Round Robin XLIX Laboratory Results

All Values in µg/mL

| | Total cis-β-Carotene | | | | | Total α-Carotene | | | | | trans-α-Carotene | | | | | Total Lycopene | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Lab | 269 | 270 | 271 | 272 | 273 | 269 | 270 | 271 | 272 | 273 | 269 | 270 | 271 | 272 | 273 | 269 | 270 | 271 | 272 | 273 |
| FSV-BA | 0.033 | 0.023 | 0.010 | 0.011 | *nd* | 0.014 | 0.020 | 0.029 | 0.027 | 0.028 | | | | | | 0.62 | 0.274 | 0.425 | 0.60 | 0.227 |
| FSV-BB | 0.028 | 0.020 | 0.018 | 0.003 | 0.019 | 0.009 | 0.018 | 0.029 | 0.020 | 0.017 | | | | | | 0.59 | 0.260 | 0.421 | 0.57 | 0.237 |
| FSV-BD | | | | | | | | | | | | | | | | | | | | |
| FSV-BE | | | | | | | | | | | | | | | | | | | | |
| FSV-BF | | | | | | 0.015 | 0.020 | 0.031 | 0.030 | 0.035 | | | | | | 0.60 | 0.209 | 0.377 | 0.53 | 0.196 |
| FSV-BG | | | | | | 0.023 | 0.034 | 0.043 | 0.031 | 0.030 | | | | | | 0.60 | 0.316 | 0.415 | 0.52 | 0.234 |
| FSV-BH | 0.033 | 0.024 | 0.022 | 0.010 | *nd* | 0.011 | 0.015 | 0.027 | 0.024 | 0.024 | | | | | | 0.54 | 0.236 | 0.376 | 0.52 | 0.205 |
| FSV-BI | | | | | | 0.014 | 0.022 | 0.028 | 0.031 | 0.031 | | | | | | 0.44 | 0.204 | 0.338 | 0.43 | 0.175 |
| FSV-BJ | | | | | | 0.015 | 0.028 | 0.042 | 0.034 | 0.028 | | | | | | 0.49 | 0.186 | 0.330 | 0.45 | 0.189 |
| FSV-BK | | | | | | | | | | | | | | | | | | | | |
| FSV-BL | | | | | | | | | | | | | | | | | | | | |
| FSV-BM | | | | | | | | | | | | | | | | | | | | |
| FSV-BN | 0.032 | 0.026 | 0.024 | 0.004 | *nd* | 0.001 | 0.011 | 0.023 | 0.018 | 0.025 | | | | | | 0.48 | 0.217 | 0.363 | 0.47 | 0.218 |
| FSV-BO | | | | | | 0.014 | 0.018 | 0.033 | 0.032 | 0.029 | | | | | | 0.49 | 0.211 | 0.395 | 0.27 | 0.248 |
| FSV-BP | | | | | | 0.009 | 0.015 | 0.026 | 0.022 | *nd* | | | | | | 0.54 | 0.416 | 0.362 | 0.27 | *nd* |
| FSV-BQ | | | | | | | | | | | | | | | | | | | | |
| FSV-BR | | | | | | | | | | | | | | | | | | | | |
| FSV-BS | | | | | | *nd* | >0.009 | >0.015 | >0.017 | >0.012 | *nd* | 0.009 | 0.015 | 0.017 | 0.012 | 0.48 | 0.278 | 0.330 | 0.42 | 0.276 |
| FSV-BT | 0.024 | 0.019 | 0.018 | 0.008 | 0.003 | 0.010 | 0.017 | 0.027 | 0.026 | 0.026 | | | | | | 0.43 | 0.198 | 0.311 | 0.42 | 0.190 |
| FSV-BU | | | | | | 0.020 | 0.021 | 0.035 | 0.032 | 0.034 | | | | | | 0.57 | 0.134 | 0.346 | 0.57 | 0.142 |
| FSV-BV | | | | | | 0.006 | 0.012 | 0.021 | 0.017 | 0.019 | | | | | | 0.40 | 0.181 | 0.299 | 0.40 | 0.173 |
| FSV-BW | | | | | | >0.020 | >0.016 | | | | 0.020 | 0.016 | <0.015 | <0.015 | <0.015 | 0.53 | 0.230 | 0.350 | 0.50 | 0.240 |
| FSV-BX | | | | | | 0.019 | 0.023 | 0.038 | 0.037 | 0.035 | | | | | | | | | | |
| FSV-CB | | | | | | 0.010 | 0.016 | 0.026 | 0.022 | 0.025 | | | | | | 0.45 | 0.200 | 0.305 | 0.44 | 0.177 |
| FSV-CC | | | | | | | | | | | | | | | | | | | | |
| FSV-CD | | | | | | 0.012 | 0.018 | 0.032 | 0.023 | 0.027 | | | | | | 0.60 | 0.231 | 0.338 | 0.41 | 0.158 |
| FSV-CE | | | | | | | | | | | | | | | | | | | | |
| FSV-CF | | | | | | | | | | | | | | | | | | | | |
| FSV-CG | 0.034 | 0.026 | 0.025 | 0.010 | 0.007 | 0.016 | 0.021 | 0.036 | 0.030 | 0.029 | | | | | | 0.54 | 0.233 | 0.381 | 0.50 | 0.207 |
| FSV-CH | | | | | | 0.013 | 0.018 | 0.038 | 0.023 | 0.029 | | | | | | 0.40 | 0.196 | 0.297 | 0.40 | 0.179 |
| FSV-CI | | | | | | | | | | | | | | | | | | | | |
| FSV-CL | | | | | | 0.013 | 0.020 | 0.027 | 0.026 | 0.022 | | | | | | 0.49 | 0.230 | 0.324 | 0.46 | 0.180 |
| FSV-CR | | | | | | | | | | | | | | | | | | | | |
| FSV-CV | | | | | | 0.115 | 0.017 | 0.032 | 0.203 | 0.015 | | | | | | 1.15 | 0.487 | 0.632 | 1.09 | 0.314 |
| FSV-CW | 0.034 | 0.027 | 0.024 | 0.010 | 0.004 | >0.013 | >0.021 | >0.031 | >0.025 | >0.028 | 0.013 | 0.021 | 0.031 | 0.025 | 0.028 | 0.46 | 0.203 | 0.339 | 0.45 | 0.200 |
| FSV-CZ | | | | | | | | | | | | | | | | | | | | |
| FSV-DB | | | | | | | | | | | | | | | | 0.65 | 0.280 | 0.440 | 0.61 | 0.270 |
| FSV-DD | | | | | | | | | | | | | | | | | | | | |
| FSV-DF | | | | | | | | | | | | | | | | | | | | |
| FSV-DI | | | | | | | | | | | | | | | | | | | | |
| FSV-DQ | | | | | | 0.025 | 0.017 | 0.045 | 0.042 | 0.049 | | | | | | 0.89 | 0.149 | 0.594 | 1.07 | 0.361 |
| FSV-DR | | | | | | | | | | | | | | | | | | | | |
| FSV-DU | | | | | | | | | | | | | | | | | | | | |
| FSV-EH | 0.058 | 0.044 | 0.041 | 0.023 | 0.022 | 0.008 | 0.013 | 0.026 | 0.018 | 0.020 | | | | | | 0.51 | 0.224 | 0.363 | 0.49 | 0.208 |
| FSV-EQ | 0.012 | 0.008 | 0.008 | 0.003 | *nd* | 0.008 | 0.012 | 0.024 | 0.019 | 0.017 | | | | | | 0.51 | 0.236 | 0.364 | 0.54 | 0.213 |
| FSV-FB | 0.111 | 0.073 | 0.057 | 0.044 | 0.031 | >0.003 | >0.012 | >0.020 | >0.014 | >0.018 | 0.003 | 0.012 | 0.020 | 0.014 | 0.018 | 0.46 | 0.242 | 0.396 | 0.58 | 0.229 |
| FSV-FJ | | | | | | 0.010 | *nq* | 0.020 | 0.020 | 0.020 | | | | | | | | | | |
| N | 10 | 10 | 10 | 10 | 6 | 24 | 23 | 24 | 24 | 23 | 3 | 4 | 3 | 3 | 3 | 27 | 27 | 27 | 27 | 26 |
| Min | 0.012 | 0.008 | 0.008 | 0.003 | 0.003 | 0.001 | 0.011 | 0.020 | 0.017 | 0.015 | 0.003 | 0.009 | 0.015 | 0.014 | 0.012 | 0.40 | 0.134 | 0.297 | 0.27 | 0.142 |
| Median | 0.033 | 0.025 | 0.023 | 0.010 | 0.013 | 0.013 | 0.018 | 0.029 | 0.026 | 0.027 | 0.013 | 0.014 | 0.020 | 0.017 | 0.018 | 0.51 | 0.230 | 0.363 | 0.49 | 0.208 |
| Max | 0.111 | 0.073 | 0.057 | 0.044 | 0.031 | 0.115 | 0.034 | 0.045 | 0.203 | 0.049 | 0.020 | 0.021 | 0.031 | 0.025 | 0.028 | 1.15 | 0.487 | 0.632 | 1.09 | 0.361 |
| eSD | 0.004 | 0.005 | 0.008 | 0.006 | 0.013 | 0.005 | 0.004 | 0.005 | 0.008 | 0.006 | | 0.005 | | | | 0.09 | 0.044 | 0.049 | 0.10 | 0.042 |
| eCV | 13 | 21 | 33 | 63 | 103 | 38 | 25 | 17 | 29 | 22 | | 35 | | | | 18 | 19 | 13 | 21 | 20 |
| $N_{past}$ | 10 | 11 | 11 | 7 | 6 | 25 | 28 | 29 | 27 | 28 | 0 | 0 | 0 | 0 | 0 | 28 | 29 | 29 | 29 | 29 |
| $Median_{past}$ | 0.037 | 0.024 | 0.020 | 0.012 | 0.006 | 0.013 | 0.022 | 0.030 | 0.027 | 0.028 | | | | | | 0.50 | 0.229 | 0.330 | 0.46 | 0.197 |
| $SD_{past}$ | 0.019 | 0.009 | 0.007 | 0.006 | 0.004 | 0.005 | 0.007 | 0.006 | 0.012 | 0.006 | | | | | | 0.11 | 0.049 | 0.086 | 0.10 | 0.050 |
| NISTa | 0.080 | 0.057 | 0.023 | *nq* | *nq* | *nq* | *nq* | *nq* | >0.034 | >0.028 | *nq* | *nq* | *nq* | 0.034 | 0.028 | | | | | |
| NISTb | 0.028 | *nd* | *nd* | *nd* | *nd* | >0.017 | >0.014 | >0.021 | >0.018 | >0.023 | 0.017 | 0.014 | 0.021 | 0.018 | 0.023 | 0.40 | 0.185 | 0.296 | 0.47 | 0.184 |
| $N_{NIST}$ | | | | | | | 1 | | 2 | 2 | 1 | 2 | 2 | 2 | 1 | 1 | 2 | 2 | 2 | 1 |
| Mean | | | | | | | 0.012 | | 0.034 | 0.028 | 0.017 | 0.014 | 0.021 | 0.018 | 0.023 | 0.40 | 0.185 | 0.296 | 0.47 | 0.184 |
| $S_{rep}$ | | | | | | | 0.000 | | 0.001 | 0.001 | 0.000 | 0.004 | 0.003 | 0.001 | 0.000 | 0.05 | 0.008 | 0.019 | 0.02 | 0.038 |
| $S_{het}$ | | | | | | | 0.000 | | 0.001 | 0.001 | 0.000 | 0.001 | 0.001 | 0.002 | 0.000 | 0.00 | 0.013 | 0.003 | 0.04 | 0.000 |
| $S_{anl}$ | | | | | | | | | | | | | | | | | | | | |
| $S_{NIST}$ | | | | | | | 0.000 | | 0.001 | 0.001 | 0.000 | 0.004 | 0.004 | 0.002 | 0.000 | 0.05 | 0.016 | 0.019 | 0.05 | 0.038 |
| NAV | 0.033 | 0.025 | 0.023 | 0.010 | | 0.013 | 0.015 | 0.029 | 0.030 | 0.028 | | | | | | 0.45 | 0.208 | 0.329 | 0.48 | 0.196 |
| NAU | 0.012 | 0.009 | 0.008 | 0.006 | | 0.005 | 0.008 | 0.010 | 0.011 | 0.009 | | | | | | 0.13 | 0.063 | 0.093 | 0.10 | 0.053 |

# Round Robin XLIX Laboratory Results

All Values in µg/mL

| | trans-Lycopene | | | | | Total β-Cryptoxanthin | | | | | Total α-Cryptoxanthin | | | | | Total Lutein | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Lab | 269 | 270 | 271 | 272 | 273 | 269 | 270 | 271 | 272 | 273 | 269 | 270 | 271 | 272 | 273 | 269 | 270 | 271 | 272 | 273 |
| FSV-BA | 0.293 | 0.151 | 0.220 | 0.267 | 0.121 | 0.050 | 0.064 | 0.071 | 0.067 | 0.069 | | | | | | | | | | |
| FSV-BB | 0.235 | 0.128 | 0.186 | 0.219 | 0.101 | 0.039 | 0.049 | 0.056 | 0.052 | 0.055 | 0.016 | 0.024 | 0.025 | 0.019 | 0.031 | 0.062 | 0.101 | 0.097 | 0.118 | 0.064 |
| FSV-BD | | | | | | | | | | | | | | | | | | | | |
| FSV-BE | | | | | | | | | | | | | | | | | | | | |
| FSV-BF | | | | | | 0.036 | 0.037 | 0.043 | 0.043 | 0.041 | | | | | | | | | | |
| FSV-BG | 0.322 | 0.172 | 0.221 | 0.256 | 0.126 | 0.032 | 0.042 | 0.042 | 0.044 | 0.033 | | | | | | 0.065 | 0.058 | 0.074 | 0.077 | 0.060 |
| FSV-BH | | | | | | 0.061 | 0.064 | 0.078 | 0.083 | 0.080 | | | | | | 0.040 | 0.068 | 0.063 | 0.085 | 0.054 |
| FSV-BI | | | | | | 0.042 | 0.061 | 0.066 | 0.059 | 0.066 | | | | | | 0.070 | 0.106 | 0.113 | 0.143 | 0.082 |
| FSV-BJ | | | | | | 0.034 | 0.043 | 0.054 | 0.044 | 0.055 | | | | | | 0.062 | 0.082 | 0.090 | 0.119 | 0.086 |
| FSV-BK | | | | | | | | | | | | | | | | | | | | |
| FSV-BL | | | | | | | | | | | | | | | | | | | | |
| FSV-BM | | | | | | | | | | | | | | | | | | | | |
| FSV-BN | 0.217 | 0.119 | 0.191 | 0.207 | 0.117 | 0.035 | 0.056 | 0.065 | 0.052 | 0.066 | 0.009 | 0.023 | 0.024 | 0.014 | 0.033 | 0.049 | 0.088 | 0.084 | 0.104 | 0.068 |
| FSV-BO | | | | | | 0.038 | 0.049 | 0.057 | 0.051 | 0.059 | | | | | | 0.059 | 0.111 | 0.108 | 0.205 | 0.080 |
| FSV-BP | | | | | | 0.035 | 0.064 | 0.059 | 0.064 | 0.032 | | | | | | | | | | |
| FSV-BQ | | | | | | | | | | | | | | | | | | | | |
| FSV-BR | | | | | | | | | | | | | | | | | | | | |
| FSV-BS | 0.243 | 0.103 | 0.122 | 0.156 | 0.103 | 0.042 | 0.051 | 0.052 | 0.064 | 0.058 | | | | | | *nd* | 0.033 | 0.033 | 0.052 | 0.023 |
| FSV-BT | 0.188 | 0.088 | 0.138 | 0.178 | 0.084 | 0.055 | 0.063 | 0.064 | 0.066 | 0.077 | 0.033 | 0.035 | 0.035 | 0.039 | 0.043 | 0.072 | 0.105 | 0.115 | 0.119 | 0.095 |
| FSV-BU | | | | | | 0.044 | 0.056 | 0.060 | 0.056 | 0.062 | | | | | | | | | | |
| FSV-BV | | | | | | 0.021 | 0.029 | 0.033 | 0.030 | 0.032 | | | | | | | | | | |
| FSV-BW | | | | | | | | | | | | | | | | | | | | |
| FSV-BX | 0.220 | 0.111 | 0.170 | 0.199 | 0.100 | 0.039 | 0.051 | 0.054 | 0.051 | 0.051 | | | | | | 0.057 | 0.097 | 0.092 | 0.118 | 0.069 |
| FSV-CB | | | | | | 0.041 | 0.046 | 0.054 | 0.049 | 0.053 | | | | | | 0.048 | 0.074 | 0.084 | 0.133 | 0.063 |
| FSV-CC | | | | | | | | | | | | | | | | | | | | |
| FSV-CD | | | | | | 0.039 | 0.045 | 0.054 | 0.054 | 0.048 | | | | | | | | | | |
| FSV-CE | | | | | | | | | | | | | | | | | | | | |
| FSV-CF | | | | | | | | | | | | | | | | | | | | |
| FSV-CG | 0.283 | 0.139 | 0.208 | 0.245 | 0.116 | 0.047 | 0.048 | 0.058 | 0.058 | 0.051 | | | | | | 0.082 | 0.119 | 0.115 | 0.138 | 0.099 |
| FSV-CH | | | | | | | | | | | | | | | | | | | | |
| FSV-CI | | | | | | | | | | | | | | | | | | | | |
| FSV-CL | | | | | | 0.047 | 0.059 | 0.050 | 0.043 | 0.056 | 0.037 | 0.041 | 0.034 | 0.029 | 0.042 | | | | | |
| FSV-CR | | | | | | | | | | | | | | | | | | | | |
| FSV-CV | | | | | | | | | | | | | | | | | | | | |
| FSV-CW | 0.204 | 0.106 | 0.155 | 0.185 | 0.101 | 0.042 | 0.045 | 0.053 | 0.051 | 0.052 | 0.020 | 0.023 | 0.027 | 0.022 | 0.029 | >0.048 | >0.073 | >0.073 | >0.093 | >0.056 |
| FSV-CZ | | | | | | | | | | | | | | | | | | | | |
| FSV-DB | | | | | | 0.045 | 0.063 | 0.060 | 0.056 | 0.072 | | | | | | | | | | |
| FSV-DD | | | | | | | | | | | | | | | | | | | | |
| FSV-DF | | | | | | | | | | | | | | | | | | | | |
| FSV-DI | 0.342 | 0.150 | 0.217 | 0.304 | 0.109 | | | | | | | | | | | 0.038 | 0.064 | 0.063 | 0.086 | 0.052 |
| FSV-DQ | | | | | | 0.049 | 0.067 | 0.087 | 0.093 | 0.107 | | | | | | 0.047 | 0.207 | 0.089 | 0.072 | 0.033 |
| FSV-DR | | | | | | | | | | | | | | | | | | | | |
| FSV-DU | | | | | | | | | | | | | | | | | | | | |
| FSV-EH | 0.215 | 0.107 | 0.164 | 0.197 | 0.089 | 0.041 | 0.052 | 0.059 | 0.056 | 0.062 | | | | | | >0.051 | >0.080 | >0.077 | >0.092 | >0.061 |
| FSV-EQ | | | | | | 0.037 | 0.045 | 0.049 | 0.051 | 0.052 | | | | | | | | | | |
| FSV-FB | 0.413 | 0.208 | 0.345 | 0.503 | 0.196 | 0.038 | 0.062 | 0.063 | 0.057 | 0.067 | | | | | | 0.052 | 0.063 | 0.065 | 0.100 | 0.044 |
| FSV-FJ | | | | | | | | | | | | | | | | | | | | |
| N | 12 | 12 | 12 | 12 | 12 | 25 | 25 | 25 | 25 | 25 | 5 | 5 | 5 | 5 | 5 | 14 | 15 | 15 | 15 | 15 |
| Min | 0.188 | 0.088 | 0.122 | 0.156 | 0.084 | 0.021 | 0.029 | 0.033 | 0.030 | 0.032 | 0.009 | 0.023 | 0.024 | 0.014 | 0.029 | 0.038 | 0.033 | 0.033 | 0.052 | 0.023 |
| Median | 0.239 | 0.124 | 0.189 | 0.213 | 0.106 | 0.041 | 0.051 | 0.057 | 0.054 | 0.056 | 0.020 | 0.024 | 0.027 | 0.022 | 0.033 | 0.058 | 0.088 | 0.089 | 0.118 | 0.064 |
| Max | 0.413 | 0.208 | 0.345 | 0.503 | 0.196 | 0.061 | 0.067 | 0.087 | 0.093 | 0.107 | 0.037 | 0.041 | 0.035 | 0.039 | 0.043 | 0.082 | 0.207 | 0.115 | 0.205 | 0.099 |
| eSD | 0.058 | 0.028 | 0.044 | 0.050 | 0.015 | 0.006 | 0.012 | 0.008 | 0.007 | 0.012 | 0.016 | 0.001 | 0.004 | 0.010 | 0.006 | 0.014 | 0.030 | 0.029 | 0.031 | 0.024 |
| eCV | 24 | 23 | 24 | 23 | 14 | 14 | 23 | 14 | 13 | 21 | 82 | 6 | 16 | 47 | 18 | 25 | 34 | 32 | 26 | 37 |
| $N_{past}$ | 14 | 12 | 12 | 8 | 5 | 24 | 29 | 29 | 22 | 22 | 6 | 7 | 7 | 0 | 0 | 16 | 16 | 16 | 14 | 8 |
| $Median_{past}$ | 0.260 | 0.135 | 0.165 | 0.250 | 0.114 | 0.044 | 0.052 | 0.054 | 0.054 | 0.060 | 0.019 | 0.030 | 0.026 | | | 0.065 | 0.092 | 0.083 | 0.125 | 0.079 |
| $SD_{past}$ | 0.061 | 0.019 | 0.017 | 0.039 | 0.031 | 0.010 | 0.010 | 0.013 | 0.016 | 0.016 | 0.010 | 0.004 | 0.007 | | | 0.026 | 0.017 | 0.013 | 0.016 | 0.014 |
| NISTa | | | | | | *nq* | 0.029 | 0.065 | *nq* | 0.036 | | | | | | 0.055 | 0.101 | 0.110 | 0.137 | 0.075 |
| NISTb | | | | | | 0.042 | 0.064 | 0.069 | 0.065 | >0.045 | 0.030 | 0.047 | 0.047 | 0.038 | 0.036 | >0.054 | >0.067 | >0.068 | >0.091 | >0.050 |
| $N_{NIST}$ | | | | | | 1 | 4 | 4 | 2 | 2 | 1 | 2 | 2 | 2 | 1 | 2 | 2 | 2 | 2 | 2 |
| Mean | | | | | | 0.042 | 0.047 | 0.067 | 0.065 | 0.036 | 0.030 | 0.047 | 0.047 | 0.038 | 0.036 | 0.055 | 0.101 | 0.110 | 0.137 | 0.075 |
| $S_{rep}$ | | | | | | 0.000 | 0.001 | 0.010 | 0.014 | 0.003 | 0.000 | 0.001 | 0.009 | 0.006 | 0.007 | 0.001 | 0.010 | 0.005 | 0.004 | 0.004 |
| $S_{het}$ | | | | | | 0.000 | 0.007 | 0.004 | 0.003 | 0.002 | 0.000 | 0.002 | 0.002 | 0.001 | 0.000 | 0.006 | 0.002 | 0.002 | 0.003 | 0.000 |
| $S_{anl}$ | | | | | | | 0.025 | 0.003 | | | | | | | | | | | | |
| $S_{NIST}$ | | | | | | 0.000 | 0.026 | 0.011 | 0.015 | 0.003 | 0.000 | 0.002 | 0.009 | 0.007 | 0.007 | 0.006 | 0.010 | 0.006 | 0.005 | 0.004 |
| NAV | 0.239 | 0.124 | 0.189 | 0.213 | 0.106 | 0.041 | 0.049 | 0.062 | 0.060 | 0.046 | 0.025 | 0.035 | 0.037 | 0.030 | 0.035 | 0.057 | 0.094 | 0.099 | 0.127 | 0.069 |
| NAU | 0.058 | 0.028 | 0.044 | 0.050 | 0.018 | 0.010 | 0.026 | 0.015 | 0.017 | 0.020 | 0.018 | 0.016 | 0.017 | 0.015 | 0.007 | 0.015 | 0.031 | 0.032 | 0.033 | 0.025 |

# Round Robin XLIX Laboratory Results

All Values in µg/mL

| | trans-Lutein | | | | | Total Zeaxanthin | | | | | trans-Zeaxanthin | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Lab | 269 | 270 | 271 | 272 | 273 | 269 | 270 | 271 | 272 | 273 | 269 | 270 | 271 | 272 | 273 |
| FSV-BA | | | | | | | | | | | | | | | |
| FSV-BB | | | | | | 0.051 | 0.048 | 0.041 | 0.054 | 0.035 | | | | | |
| FSV-BD | | | | | | | | | | | | | | | |
| FSV-BE | | | | | | | | | | | | | | | |
| FSV-BF | | | | | | | | | | | | | | | |
| FSV-BG | | | | | | 0.019 | 0.012 | 0.020 | 0.018 | 0.020 | | | | | |
| FSV-BH | | | | | | 0.020 | 0.033 | 0.023 | 0.025 | 0.035 | | | | | |
| FSV-BI | | | | | | 0.026 | 0.042 | 0.033 | 0.038 | 0.039 | | | | | |
| FSV-BJ | | | | | | | | | | | | | | | |
| FSV-BK | | | | | | | | | | | | | | | |
| FSV-BL | | | | | | | | | | | | | | | |
| FSV-BM | | | | | | | | | | | | | | | |
| FSV-BN | | | | | | 0.017 | 0.027 | 0.022 | 0.023 | 0.032 | | | | | |
| FSV-BO | | | | | | 0.020 | 0.024 | 0.018 | 0.019 | 0.027 | | | | | |
| FSV-BP | | | | | | | | | | | | | | | |
| FSV-BQ | | | | | | | | | | | | | | | |
| FSV-BR | | | | | | | | | | | | | | | |
| FSV-BS | | | | | | | | | | | | | | | |
| FSV-BT | | | | | | >0.028 | >0.033 | >0.038 | >0.044 | >0.041 | 0.028 | 0.033 | 0.038 | 0.044 | 0.041 |
| FSV-BU | | | | | | | | | | | | | | | |
| FSV-BV | | | | | | | | | | | | | | | |
| FSV-BW | | | | | | | | | | | | | | | |
| FSV-BX | | | | | | 0.022 | 0.034 | 0.024 | 0.026 | 0.034 | | | | | |
| FSV-CB | | | | | | 0.020 | 0.035 | 0.033 | 0.007 | 0.036 | | | | | |
| FSV-CC | | | | | | | | | | | | | | | |
| FSV-CD | | | | | | | | | | | | | | | |
| FSV-CE | | | | | | | | | | | | | | | |
| FSV-CF | | | | | | | | | | | | | | | |
| FSV-CG | | | | | | 0.018 | 0.025 | 0.024 | 0.034 | 0.022 | | | | | |
| FSV-CH | | | | | | | | | | | | | | | |
| FSV-CI | | | | | | | | | | | | | | | |
| FSV-CL | | | | | | | | | | | | | | | |
| FSV-CR | | | | | | | | | | | | | | | |
| FSV-CV | | | | | | | | | | | | | | | |
| FSV-CW | 0.048 | 0.073 | 0.073 | 0.093 | 0.056 | 0.023 | 0.034 | 0.025 | 0.026 | 0.033 | | | | | |
| FSV-CZ | | | | | | | | | | | | | | | |
| FSV-DB | | | | | | | | | | | | | | | |
| FSV-DD | | | | | | | | | | | | | | | |
| FSV-DF | | | | | | | | | | | | | | | |
| FSV-DI | | | | | | | | | | | | | | | |
| FSV-DQ | | | | | | 0.019 | 0.064 | 0.017 | 0.008 | 0.025 | | | | | |
| FSV-DR | | | | | | | | | | | | | | | |
| FSV-DU | | | | | | | | | | | | | | | |
| FSV-EH | 0.051 | 0.080 | 0.077 | 0.092 | 0.061 | 0.023 | 0.036 | 0.028 | 0.029 | 0.033 | | | | | |
| FSV-EQ | | | | | | | | | | | | | | | |
| FSV-FB | | | | | | 0.020 | 0.037 | 0.016 | 0.012 | 0.030 | | | | | |
| FSV-FJ | | | | | | | | | | | | | | | |
| N | 2 | 2 | 2 | 2 | 2 | 13 | 13 | 13 | 13 | 13 | 1 | 1 | 1 | 1 | 1 |
| Min | 0.048 | 0.073 | 0.073 | 0.092 | 0.056 | 0.017 | 0.012 | 0.016 | 0.007 | 0.020 | | | | | |
| Median | 0.050 | 0.077 | 0.075 | 0.093 | 0.059 | 0.020 | 0.034 | 0.024 | 0.025 | 0.033 | 0.028 | 0.033 | 0.038 | 0.044 | 0.041 |
| Max | 0.051 | 0.080 | 0.077 | 0.093 | 0.061 | 0.051 | 0.064 | 0.041 | 0.054 | 0.039 | | | | | |
| eSD | | | | | | 0.002 | 0.010 | 0.007 | 0.010 | 0.004 | | | | | |
| eCV | | | | | | 12 | 31 | 28 | 42 | 13 | | | | | |
| $N_{past}$ | 0 | 0 | 0 | 0 | 0 | 12 | 15 | 15 | 11 | 8 | 0 | 0 | 0 | 0 | 0 |
| $Median_{past}$ | | | | | | 0.027 | 0.035 | 0.027 | 0.032 | 0.035 | | | | | |
| $SD_{past}$ | | | | | | 0.010 | 0.009 | 0.005 | 0.012 | 0.008 | | | | | |
| NISTa | | | | | | *nq* | *nq* | 0.020 | 0.015 | 0.024 | | | | | |
| NISTb | 0.054 | 0.067 | 0.068 | 0.091 | 0.050 | >0.029 | >0.036 | >0.026 | >0.034 | >0.033 | 0.029 | 0.036 | 0.026 | 0.034 | 0.033 |
| $N_{NIST}$ | 1 | 2 | 2 | 2 | 1 | | | 2 | 2 | 2 | 1 | 2 | 2 | 2 | 1 |
| Mean | 0.054 | 0.067 | 0.068 | 0.091 | 0.050 | | | 0.020 | 0.015 | 0.024 | 0.029 | 0.036 | 0.026 | 0.034 | 0.033 |
| $S_{rep}$ | 0.009 | 0.004 | 0.005 | 0.004 | 0.001 | | | 0.001 | 0.001 | 0.001 | 0.000 | 0.002 | 0.002 | 0.004 | 0.003 |
| $S_{het}$ | 0.000 | 0.005 | 0.003 | 0.005 | 0.000 | | | 0.000 | 0.003 | 0.000 | 0.000 | 0.004 | 0.003 | 0.005 | 0.000 |
| $S_{anl}$ | | | | | | | | | | | | | | | |
| $S_{NIST}$ | 0.009 | 0.006 | 0.006 | 0.007 | 0.001 | | | 0.001 | 0.003 | 0.001 | 0.000 | 0.004 | 0.004 | 0.007 | 0.003 |
| NAV | | | | | | 0.020 | 0.034 | 0.022 | 0.020 | 0.028 | | | | | |
| NAU | | | | | | 0.006 | 0.010 | 0.007 | 0.012 | 0.011 | | | | | |

# Round Robin XLIX Laboratory Results

All Values in µg/mL

| | Total Lutein&Zeaxanthin | | | | | Coenzyme Q10 | | | | | Phylloquinone (K1) x1000 | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Lab | 269 | 270 | 271 | 272 | 273 | 269 | 270 | 271 | 272 | 273 | 269 | 270 | 271 | 272 | 273 |
| FSV-BA | 0.106 | 0.156 | 0.158 | 0.182 | 0.132 | | | | | | | | | | |
| FSV-BB | 0.113 | 0.149 | 0.138 | 0.172 | 0.099 | | | | | | | | | | |
| FSV-BD | | | | | | | | | | | | | | | |
| FSV-BE | | | | | | | | | | | 0.38 | 0.91 | 0.38 | 0.42 | 0.16 |
| FSV-BF | 0.107 | 0.157 | 0.153 | 0.203 | 0.138 | | | | | | | | | | |
| FSV-BG | 0.089 | 0.073 | 0.102 | 0.101 | 0.087 | | | | | | | | | | |
| FSV-BH | 0.060 | 0.101 | 0.086 | 0.110 | 0.089 | | | | | | | | | | |
| FSV-BI | 0.096 | 0.141 | 0.145 | 0.187 | 0.130 | | | | | | | | | | |
| FSV-BJ | | | | | | | | | | | | | | | |
| FSV-BK | | | | | | | | | | | | | | | |
| FSV-BL | | | | | | | | | | | | | | | |
| FSV-BM | | | | | | | | | | | | | | | |
| FSV-BN | 0.060 | 0.104 | 0.098 | 0.118 | 0.087 | | | | | | | | | | |
| FSV-BO | 0.079 | 0.134 | 0.126 | 0.224 | 0.107 | | | | | | | | | | |
| FSV-BP | 0.080 | 0.120 | 0.110 | 0.130 | 0.006 | | | | | | | | | | |
| FSV-BQ | | | | | | | | | | | | | | | |
| FSV-BR | | | | | | | | | | | | | | | |
| FSV-BS | | | | | | | | | | | | | | | |
| FSV-BT | 0.192 | 0.191 | 0.217 | 0.263 | 0.192 | | | | | | | | | | |
| FSV-BU | 0.091 | 0.153 | 0.144 | 0.203 | 0.130 | | | | | | | | | | |
| FSV-BV | 0.071 | 0.113 | 0.109 | 0.136 | 0.099 | | | | | | | | | | |
| FSV-BW | | | | | | | | | | | | | | | |
| FSV-BX | 0.079 | 0.130 | 0.115 | 0.144 | 0.103 | | | | | | | | | | |
| FSV-CB | 0.069 | 0.109 | 0.117 | 0.140 | 0.099 | | | | | | | | | | |
| FSV-CC | | | | | | | | | | | | | | | |
| FSV-CD | 0.080 | 0.116 | 0.143 | 0.130 | 0.089 | | | | | | | | | | |
| FSV-CE | | | | | | | | | | | | | | | |
| FSV-CF | | | | | | | | | | | | | | | |
| FSV-CG | 0.100 | 0.143 | 0.139 | 0.171 | 0.120 | | | | | | | | | | |
| FSV-CH | | | | | | 0.487 | 0.45 | 0.52 | 0.335 | 0.48 | | | | | |
| FSV-CI | | | | | | | | | | | | | | | |
| FSV-CL | 0.084 | 0.114 | 0.100 | 0.125 | 0.100 | | | | | | | | | | |
| FSV-CR | | | | | | | | | | | | | | | |
| FSV-CV | 0.092 | 0.146 | 0.125 | 0.172 | 0.110 | 1.948 | 0.96 | 0.99 | 0.942 | 1.78 | | | | | |
| FSV-CW | >0.071 | >0.107 | >0.098 | >0.119 | >0.089 | 0.910 | 0.83 | 0.95 | 0.570 | 1.07 | | | | | |
| FSV-CZ | | | | | | 0.790 | 0.94 | 1.10 | 0.540 | 1.00 | | | | | |
| FSV-DB | 0.098 | 0.129 | 0.115 | 0.152 | 0.120 | | | | | | | | | | |
| FSV-DD | | | | | | | | | | | | | | | |
| FSV-DF | | | | | | | | | | | | | | | |
| FSV-DI | | | | | | 0.855 | 0.72 | 0.79 | 0.493 | 0.85 | 0.68 | 1.37 | 0.53 | 0.47 | *nd* |
| FSV-DQ | 0.066 | 0.271 | 0.106 | 0.080 | 0.058 | | | | | | | | | | |
| FSV-DR | | | | | | | | | | | | | | | |
| FSV-DU | | | | | | | | | | | | | | | |
| FSV-EH | >0.074 | >0.116 | >0.105 | >0.121 | >0.094 | | | | | | | | | | |
| FSV-EQ | 0.017 | 0.196 | 0.183 | 0.245 | 0.178 | | | | | | | | | | |
| FSV-FB | 0.072 | 0.100 | 0.081 | 0.112 | 0.074 | | | | | | | | | | |
| FSV-FJ | | | | | | | | | | | | | | | |
| N | 22 | 22 | 22 | 22 | 22 | 5 | 5 | 5 | 5 | 5 | 2 | 2 | 2 | 2 | 1 |
| Min | 0.017 | 0.073 | 0.081 | 0.080 | 0.006 | 0.487 | 0.45 | 0.52 | 0.335 | 0.48 | 0.38 | 0.91 | 0.38 | 0.42 | |
| Median | 0.082 | 0.132 | 0.121 | 0.148 | 0.101 | 0.855 | 0.83 | 0.95 | 0.540 | 1.00 | 0.53 | 1.14 | 0.46 | 0.45 | 0.16 |
| Max | 0.192 | 0.271 | 0.217 | 0.263 | 0.192 | 1.948 | 0.96 | 1.10 | 0.942 | 1.78 | 0.68 | 1.37 | 0.53 | 0.47 | |
| eSD | 0.020 | 0.030 | 0.030 | 0.047 | 0.024 | 0.096 | 0.16 | 0.22 | 0.070 | 0.22 | | | | | |
| eCV | 25 | 22 | 25 | 32 | 24 | 11 | 20 | 23 | 13 | 22 | | | | | |
| $N_{past}$ | 22 | 29 | 29 | 22 | 21 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| $Median_{past}$ | 0.089 | 0.134 | 0.114 | 0.154 | 0.117 | | | | | | | | | | |
| $SD_{past}$ | 0.016 | 0.027 | 0.017 | 0.034 | 0.018 | | | | | | | | | | |
| NISTa | >0.055 | >0.101 | 0.130 | 0.151 | 0.098 | | | | | | | | | | |
| NISTb | >0.083 | >0.103 | >0.093 | >0.125 | >0.082 | | | | | | | | | | |
| $N_{NIST}$ | 1 | 2 | 4 | 4 | 3 | | | | | | | | | | |
| Mean | 0.077 | 0.103 | 0.112 | 0.138 | 0.090 | | | | | | | | | | |
| $S_{rep}$ | 0.000 | 0.005 | 0.007 | 0.008 | 0.004 | | | | | | | | | | |
| $S_{het}$ | 0.000 | 0.009 | 0.005 | 0.000 | 0.000 | | | | | | | | | | |
| $S_{anl}$ | | | 0.026 | 0.019 | 0.011 | | | | | | | | | | |
| $S_{NIST}$ | 0.000 | 0.010 | 0.028 | 0.020 | 0.012 | | | | | | | | | | |
| NAV | 0.080 | 0.118 | 0.117 | 0.143 | 0.096 | | | | | | | | | | |
| NAU | 0.021 | 0.036 | 0.030 | 0.048 | 0.026 | | | | | | | | | | |

# Round Robin XLIX Laboratory Results

## Analytes Reported By One Laboratory

Values in µg/mL

| Analyte | Code | 269 | 270 | 271 | 272 | 273 |
|---|---|---|---|---|---|---|
| 25-hydroxyvitamin D | FSV-BN | 0.01 | 0.06 | 0.02 | 0.02 | 0.01 |
| 3'-dehydro-Lutein | FSV-BH | 0.04 | 0.03 | 0.04 | 0.06 | 0.03 |
| Phytofluene | FSV-CL | 0.059 | 0.014 | 0.042 | 0.078 | 0.024 |
| Total cis-Lutein&Zeaxanthin | FSV-BT | 0.092 | 0.052 | 0.064 | 0.100 | 0.056 |
| trans-Lutein&Zeaxanthin | FSV-BT | 0.100 | 0.138 | 0.153 | 0.163 | 0.136 |
| trans-β-Cryptoxanthin | NISTb | 0.040 | 0.056 | 0.059 | 0.053 | 0.045 |

## 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 | Adjusted median absolute deviation from the median of the non-NIST results |
| 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 NIST results |
| NAV | NIST Assigned Value<br>= (Median + NIST)/2 for analytes reported by NIST<br>= Median for analytes reported by ≥ 5 labs but not NIST |
| NAU | NIST Assigned Uncertainty: $\sqrt{(S^2 + S_{btw}^2)}$<br>S is the maximum of (0.05*NAV, SD, $SD_{past}$, eSD) and $S_{btw}$ is the standard deviation between Median and NIST. The expected long-term SD, eSD, is defined in: Duewer et al., Anal Chem 1997;69(7):1406-1413. |
| $N_{NIST}$ | Number of total vials analyzed in duplicate by NIST analysts |
| $Mean_{NIST}$ | Mean of the NIST-analyzed vial means |
| $S_{rep}$ | Within-vial pooled standard deviation |
| $S_{het}$ | Among-vial pooled standard deviation |
| $S_{anl}$ | Between NIST analyst standard deviation |
| $S_{NIST}$ | Total standard deviation for NIST analyses: $(S_{rep}^2 + S_{het}^2 + S_{anl}^2)^{0.5}$ |
| *nd* | Not detected (i.e., no detectable peak for analyte) |
| *nq* | Detected but not quantitatively determined |
| <x | Concentration less than the limit of quantification, x |
| >x | Concentration greater than x |
| *italics* | Not explicitly reported but calculated by NIST from reported values |

# Round Robin XLIX Laboratory Results

## 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 |
| FSV-BB | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 2 | 2 |
| FSV-BD | 1 | 1 | | 1 | | | | | | | |
| FSV-BE | 1 | 1 | 1 | 1 | | | | | | | 2 |
| FSV-BF | 1 | 1 | 1 | 2 | | 1 | 1 | 1 | | | |
| FSV-BG | 1 | 1 | 1 | 1 | | 2 | 1 | 1 | 1 | | 2 |
| FSV-BH | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 2 | 1 | 3 | 1 |
| FSV-BI | 1 | 1 | 1 | 1 | | 1 | 1 | 1 | 1 | 1 | 1 |
| FSV-BJ | 1 | 1 | 1 | 1 | | 1 | 1 | 1 | 1 | 1 | 1 |
| FSV-BK | 1 | 2 | | | | | | | | | |
| FSV-BL | 3 | 4 | | | | | | | | | |
| FSV-BM | 1 | 2 | | | | | | | | | |
| FSV-BN | 1 | 1 | 2 | 1 | 1 | 2 | 1 | 1 | 1 | | 1 |
| FSV-BO | 1 | 1 | | 1 | | 1 | 2 | 1 | 2 | | |
| FSV-BP | 1 | 1 | | 1 | | 1 | 3 | 1 | | | 1 |
| FSV-BQ | 1 | 1 | | | | | | | | 2 | 1 |
| FSV-BR | 1 | 2 | | | | | | | | | 2 |
| FSV-BS | 1 | | | 3 | 3 | 2 | 1 | 1 | 2 | 1 | 1 |
| FSV-BT | 2 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| FSV-BU | 1 | 1 | 1 | 1 | | 1 | 2 | 1 | | 1 | 1 |
| FSV-BV | 2 | 1 | 1 | 1 | | 1 | 1 | 2 | | 2 | 4 |
| FSV-BW | 1 | 1 | 1 | 1 | | 2 | 1 | | | | |
| FSV-BX | 1 | 1 | 1 | 1 | 2 | 1 | | 1 | 1 | | |
| FSV-CB | 1 | 1 | | 1 | | 1 | 1 | 1 | 1 | 1 | 1 |
| FSV-CC | 1 | 1 | | | | | | | | | |
| FSV-CD | 1 | 1 | 1 | 1 | | 1 | 1 | 1 | | | |
| FSV-CE | 1 | 1 | | 1 | | | | | | | 1 |
| FSV-CF | 1 | 1 | | | | | | | | | 1 |
| FSV-CG | 1 | 2 | 1 | 1 | 1 | 1 | 1 | 1 | 2 | | 1 |
| FSV-CH | 2 | 1 | 1 | 2 | | 1 | 1 | | | | 1 |
| FSV-CI | 2 | 1 | 2 | | | | | | | 1 | 1 |
| FSV-CL | 2 | 2 | 2 | 1 | | 1 | 1 | 1 | | | |
| FSV-CR | 1 | 1 | | | | | | | | | |
| FSV-CV | 4 | 2 | 4 | 2 | | 4 | 4 | | | | |
| FSV-CW | 2 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | |
| FSV-CZ | 1 | 4 | | 2 | | | | | | | |
| FSV-DB | 1 | 1 | | 1 | | | 2 | 1 | | 2 | 3 |
| FSV-DD | 1 | | | | | | | | | | |
| FSV-DF | 1 | | | | | | | | | | |
| FSV-DI | 1 | 1 | 2 | 1 | 1 | | | | 1 | | |
| FSV-DQ | | 4 | 4 | 4 | | 2 | 4 | 2 | 3 | | 3 |
| FSV-DR | 1 | 2 | | 1 | | | | | | | |
| FSV-DU | 1 | 2 | | 2 | 2 | | | | | | |
| FSV-EH | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 |
| FSV-EQ | 1 | 3 | | 1 | 1 | 1 | 1 | 1 | | | |
| FSV-FB | 3 | 1 | | 1 | 2 | 2 | 1 | 1 | 1 | 1 | 1 |
| FSV-FJ | 1 | 1 | 1 | 2 | | 1 | | | | | |
| NISTa | 1 | 1 | 1 | 1 | 1 | 1 | | 1 | 1 | 1 | 1 |
| NISTb | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 |
| n | 48 | 46 | 27 | 38 | 16 | 30 | 28 | 27 | 19 | 16 | 26 |

| | TR | aT | g/bT | bC | tbC | aC | TLy | TbX | TLu | TZ | L&Z |
|---|---|---|---|---|---|---|---|---|---|---|---|
| % 1 | 81 | 74 | 78 | 79 | 75 | 77 | 79 | 89 | 79 | 69 | 73 |
| % 2 | 13 | 17 | 15 | 16 | 19 | 20 | 11 | 11 | 16 | 25 | 15 |
| % 3 | 4 | 2 | 0 | 3 | 6 | 0 | 4 | 0 | 5 | 6 | 8 |
| % 4 | 2 | 7 | 7 | 3 | 0 | 3 | 7 | 0 | 0 | 0 | 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) summarizes your measurement performance for a given analyte relative to the consensus medians in this study. CS is the average distance (in units of standard deviation) of your measurement performance characteristics from the consensus performance. CS is calculated when the number of quantitative values you reported, $N_{you}$, is at least two and at least six participants reported quantitative values for the analyte.

We define CS as follows:

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

$$C = \text{Concordance} = \frac{\sum_{i=1}^{N_{you}} \frac{You_i - Median_i}{NAU_i}}{N_{you}}$$

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

NAU = NIST Assigned Uncertainty

For further details, please see

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

## Appendix D. Representative "Individualized Report" for RR49

Each participant in RR49 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 RR49:

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

The following 13 pages are the "Individualized Report" for the analytes evaluated by participant FSV-BA.

# Individualized Round Robin XLIX Report: FSV-BA

## Summary

| | Serum 269 | | | Serum 270 | | | Serum 271 | | | Serum 272 | | | Serum 273 | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Analyte | You | NAV | n | You | NAV | n | You | NAV | n | You | NAV | n | You | NAV | n |
| Total Retinol | 1.252 | 0.979 | 36 | 0.688 | 0.630 | 36 | 0.495 | 0.449 | 36 | 0.564 | 0.491 | 36 | 1.350 | 1.204 | 36 |
| Retinyl Palmitate | 0.07 | 0.03 | 13 | nd | 0.0 | 12 | 0.1 | 0.1 | 14 | 0.12 | 0.07 | 14 | 0.07 | 0.06 | 14 |
| α-Tocopherol | 28.85 | 26.88 | 44 | 6.15 | 5.71 | 44 | 7.21 | 6.72 | 44 | 8.11 | 7.52 | 44 | 17.58 | 16.33 | 44 |
| γ/β-Tocopherol | 6.423 | 6.124 | 25 | 2.292 | 2.179 | 25 | 1.961 | 1.890 | 25 | 1.755 | 1.705 | 25 | 1.215 | 1.165 | 25 |
| Total β-Carotene | 0.347 | 0.358 | 32 | 0.480 | 0.452 | 32 | 0.331 | 0.314 | 32 | 0.165 | 0.154 | 32 | 0.051 | 0.052 | 31 |
| trans-β-Carotene | 0.316 | 0.305 | 14 | 0.460 | 0.420 | 14 | 0.323 | 0.288 | 14 | 0.155 | 0.150 | 14 | 0.051 | 0.048 | 14 |
| Total cis-β-Carotene | 0.033 | 0.035 | 10 | 0.023 | 0.025 | 10 | 0.010 | 0.022 | 10 | 0.011 | 0.011 | 10 | nd | 0.010 | 6 |
| Total α-Carotene | 0.014 | 0.013 | 24 | 0.020 | 0.015 | 23 | 0.029 | 0.030 | 24 | 0.027 | 0.030 | 24 | 0.028 | 0.028 | 23 |
| Total Lycopene | 0.624 | 0.465 | 27 | 0.274 | 0.206 | 27 | 0.425 | 0.329 | 27 | 0.598 | 0.484 | 27 | 0.227 | 0.194 | 26 |
| trans-Lycopene | 0.293 | 0.259 | 12 | 0.151 | 0.134 | 12 | 0.220 | 0.197 | 12 | 0.267 | 0.232 | 12 | 0.121 | 0.105 | 12 |
| Total β-Cryptoxanthin | 0.050 | 0.041 | 25 | 0.064 | 0.049 | 25 | 0.071 | 0.062 | 25 | 0.067 | 0.060 | 25 | 0.069 | 0.046 | 25 |
| Total Lutein&Zeaxanthin | 0.106 | 0.082 | 22 | 0.156 | 0.118 | 22 | 0.158 | 0.117 | 22 | 0.182 | 0.143 | 22 | 0.132 | 0.096 | 22 |

D2

You : Your reported values for the listed analytes (micrograms/milliliter)
NAV : NIST Assigned Values, equal to (NIST's average-of-averages + this RR's median) / 2
n : Number of non-NIST laboratories reporting quantitative values for this analyte in this serum
nd : Not detected

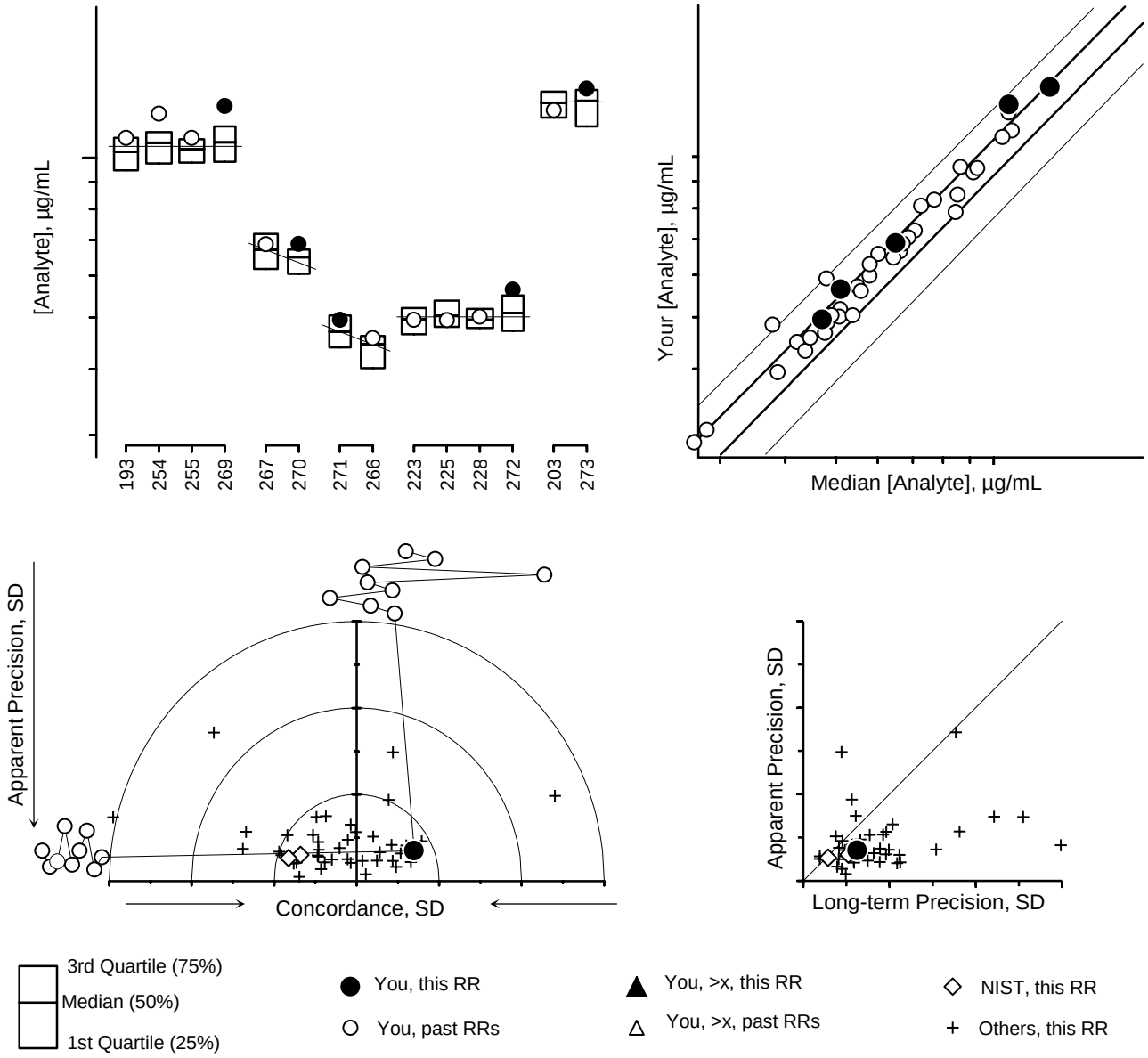
Please check our records against your records. Send corrections and/or updates to...

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

Tel: (301) 975-3935
Fax: (301) 977-0685
Email: david.duewer@nist.gov

# Individualized RR XLIX Report: FSV-BA

## Total Retinol

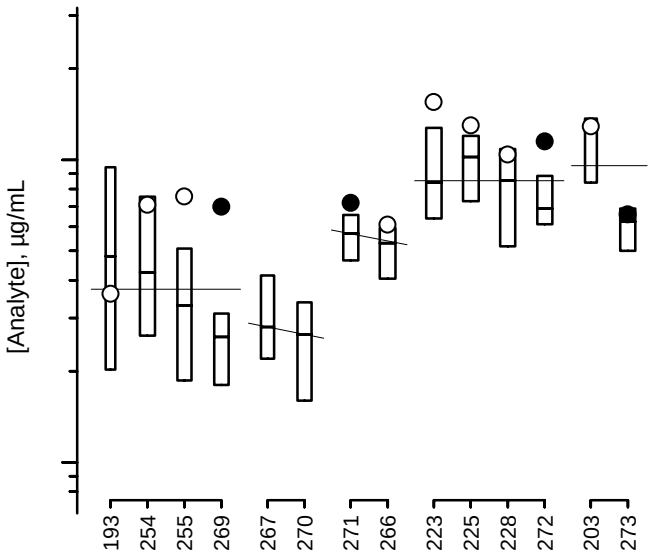


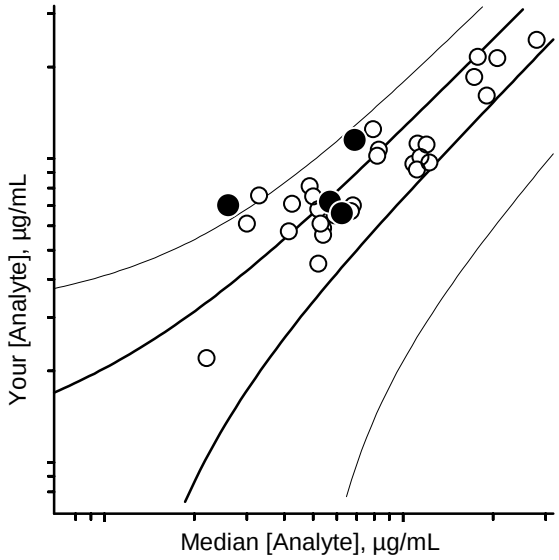

For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

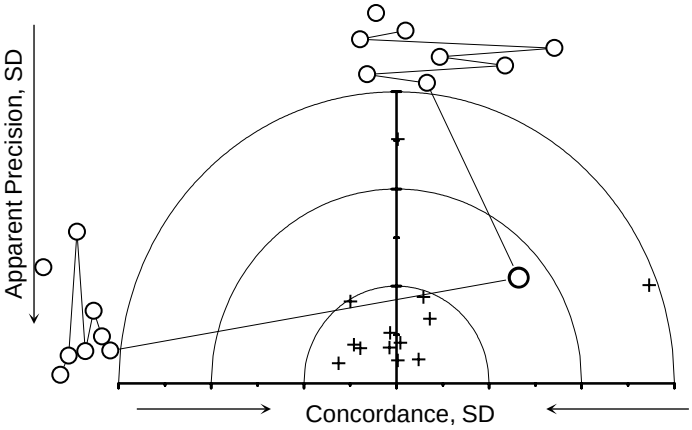
| Serum | History | Comments |
|---|---|---|
| #269 | #193 RR30 3/94, #254 RR45 3/99, #255 RR46 6/99 | Lyophilized |
| #270 | Same pool as #267 (lq fz) RR48 9/00 | Lyophilized |
| #271 | Same pool as #266 (ly) RR48 9/00 | Fresh frozen |
| #272 | #223 & #225 RR38 9/96, #228 RR39 3/97 | Lyophilized |
| #273 | #203 RR33 3/95 | Lyophilized |

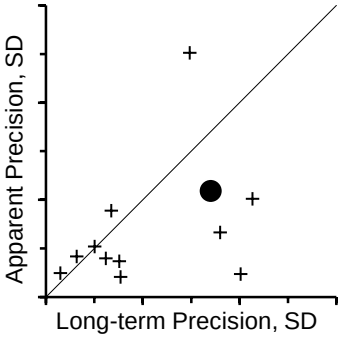
# Individualized RR XLIX Report: FSV-BA

## Retinyl Palmitate




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

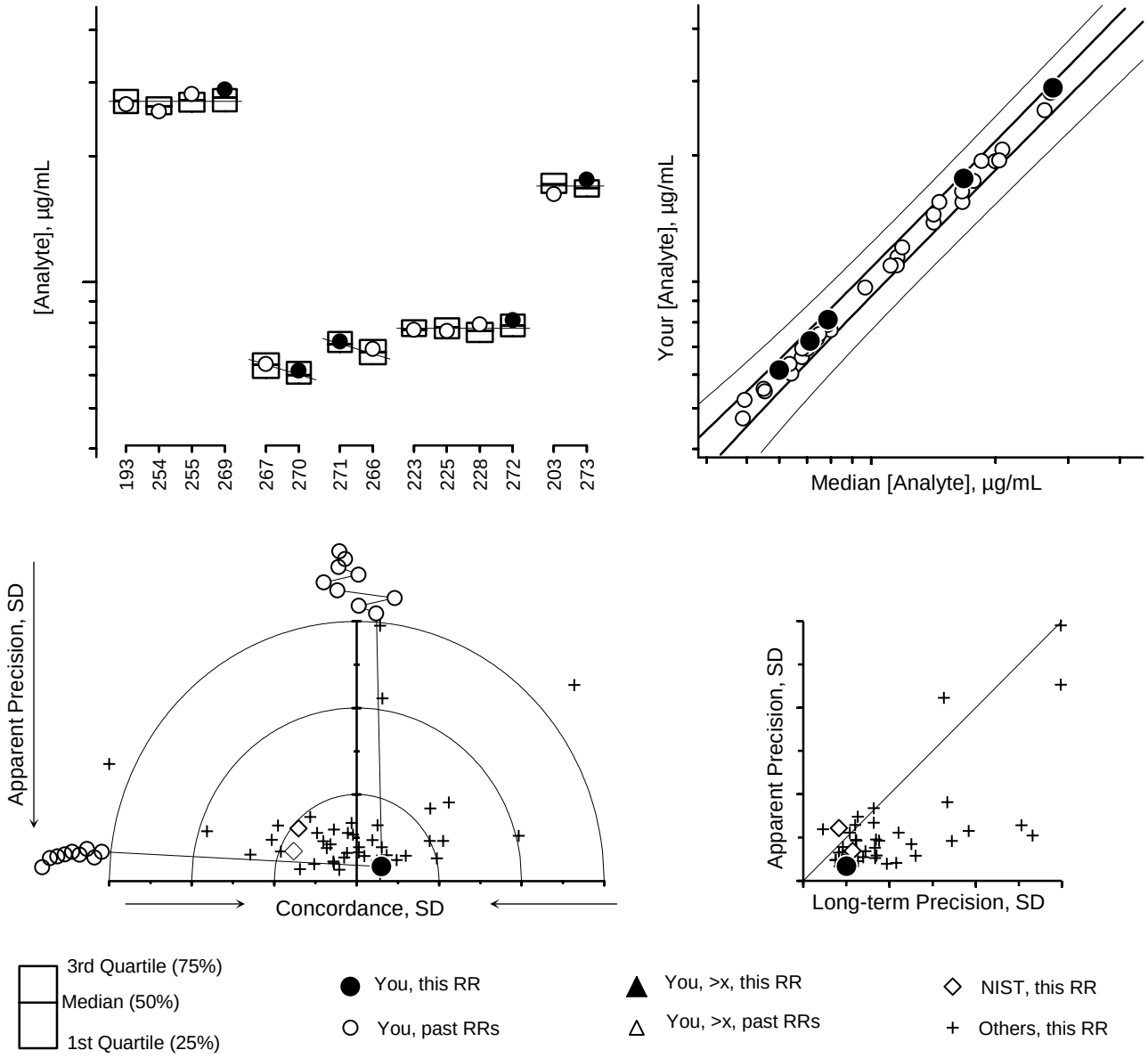
● You, this RR
○ You, past RRs
▲ You, >x, this RR
△ You, >x, past RRs
◇ NIST, this RR
+ Others, this RR

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

| Serum | History | Comments |
|---|---|---|
| #269 | #193 RR30 3/94, #254 RR45 3/99, #255 RR46 6/99 | Lyophilized |
| #270 | Same pool as #267 (lq fz) RR48 9/00 | Lyophilized |
| #271 | Same pool as #266 (ly) RR48 9/00 | Fresh frozen |
| #272 | #223 & #225 RR38 9/96, #228 RR39 3/97 | Lyophilized |
| #273 | #203 RR33 3/95 | Lyophilized |

# Individualized RR XLIX Report: FSV-BA

## α-Tocopherol

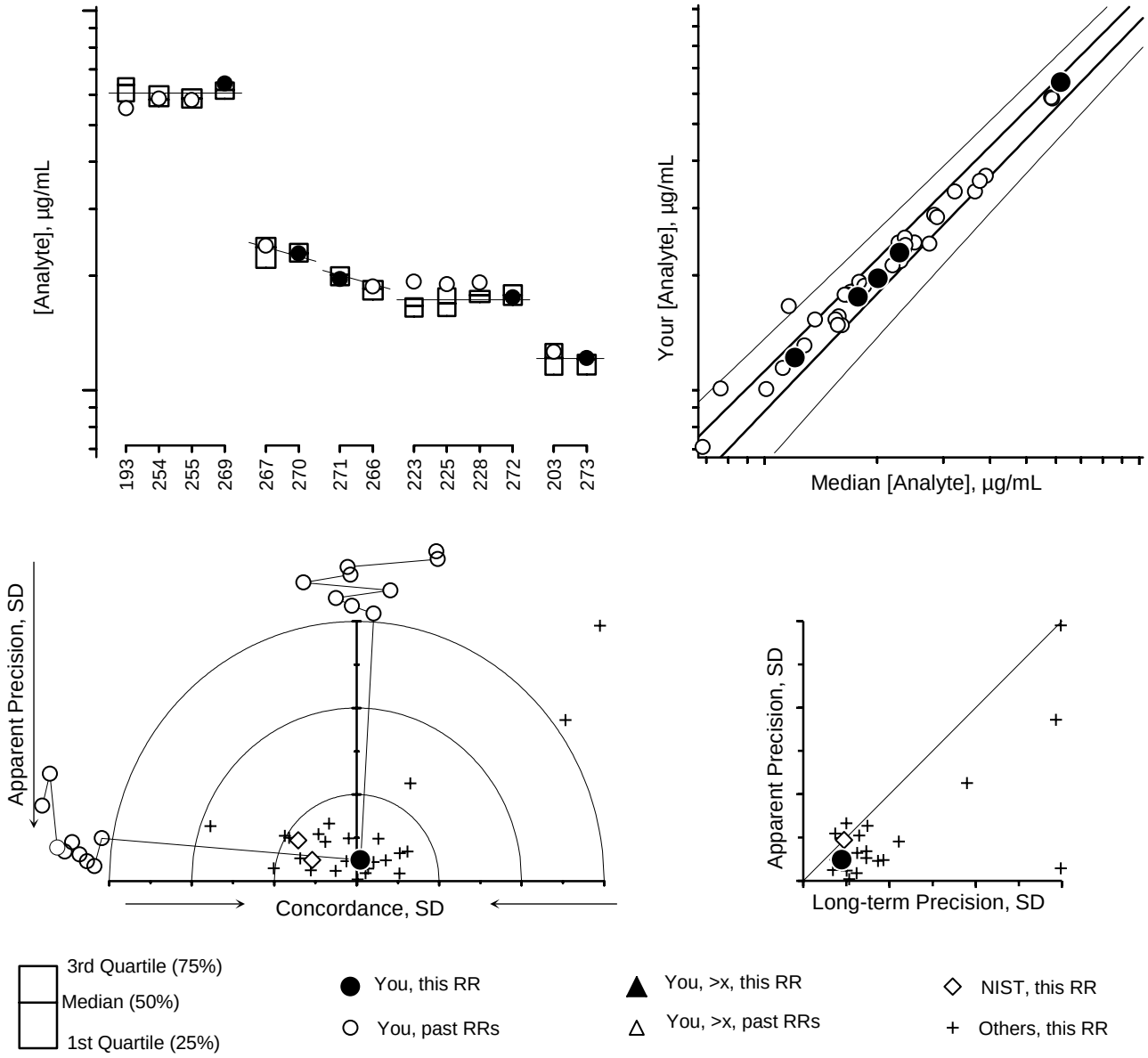



For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

| Serum | History | Comments |
|---|---|---|
| #269 | #193 RR30 3/94, #254 RR45 3/99, #255 RR46 6/99 | Lyophilized |
| #270 | Same pool as #267 (lq fz) RR48 9/00 | Lyophilized |
| #271 | Same pool as #266 (ly) RR48 9/00 | Fresh frozen |
| #272 | #223 & #225 RR38 9/96, #228 RR39 3/97 | Lyophilized |
| #273 | #203 RR33 3/95 | Lyophilized |

# Individualized RR XLIX Report: FSV-BA

γ/β-Tocopherol

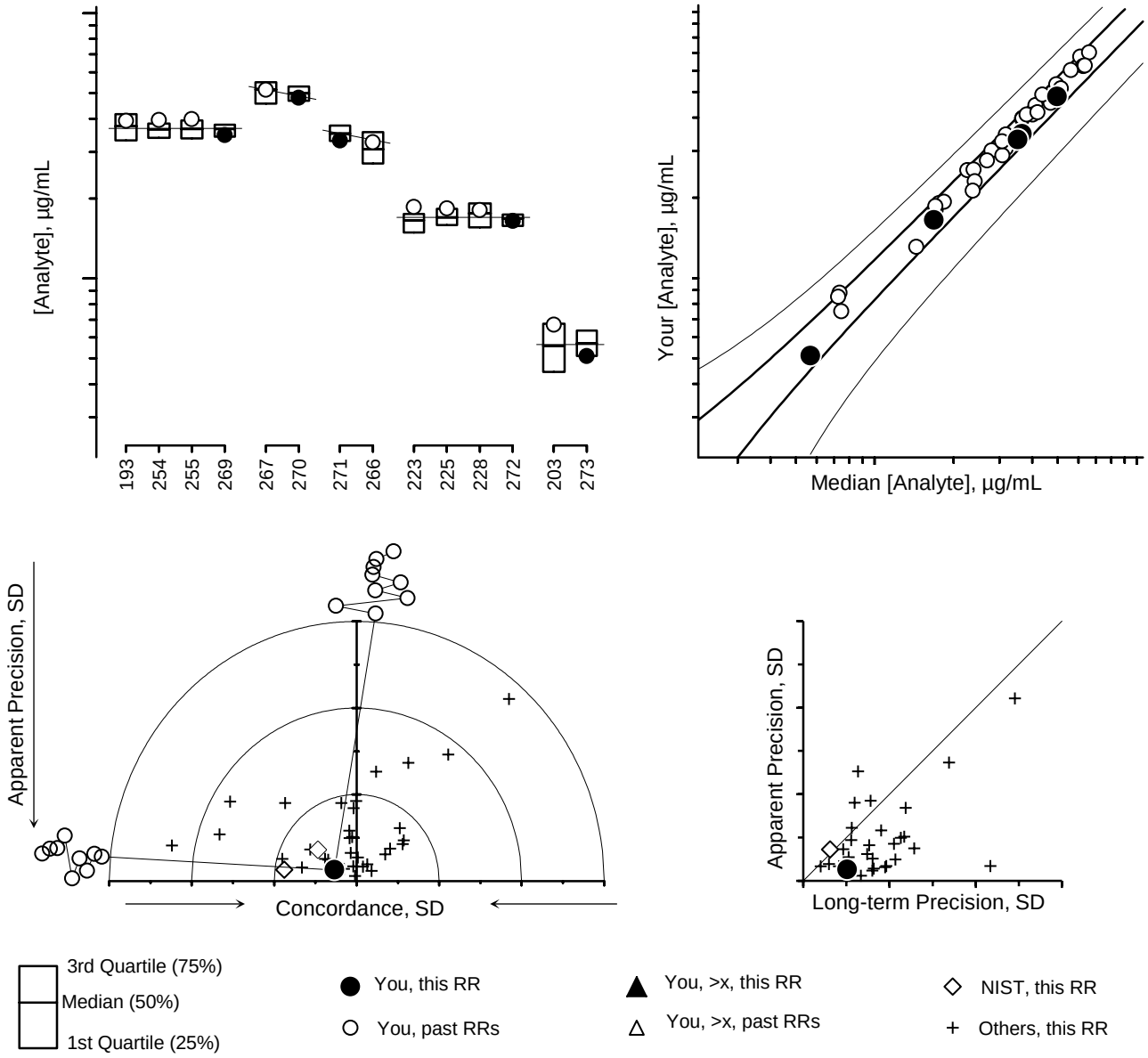



For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

| Serum | History | Comments |
|---|---|---|
| #269 | #193 RR30 3/94, #254 RR45 3/99, #255 RR46 6/99 | Lyophilized |
| #270 | Same pool as #267 (lq fz) RR48 9/00 | Lyophilized |
| #271 | Same pool as #266 (ly) RR48 9/00 | Fresh frozen |
| #272 | #223 & #225 RR38 9/96, #228 RR39 3/97 | Lyophilized |
| #273 | #203 RR33 3/95 | Lyophilized |

# Individualized RR XLIX Report: FSV-BA

## Total β-Carotene

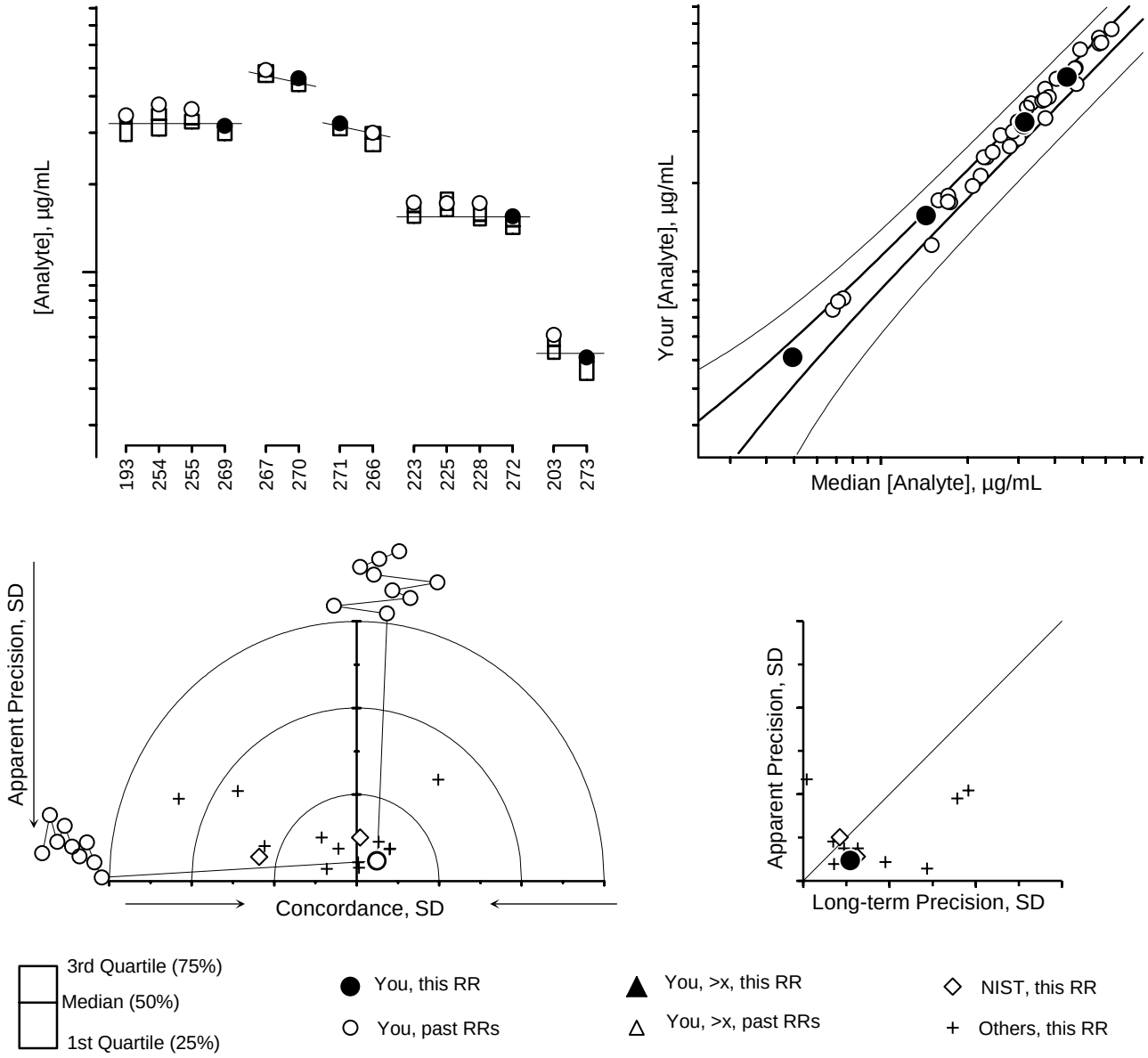



For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

| Serum | History | Comments |
|---|---|---|
| #269 | #193 RR30 3/94, #254 RR45 3/99, #255 RR46 6/99 | Lyophilized |
| #270 | Same pool as #267 (lq fz) RR48 9/00 | Lyophilized |
| #271 | Same pool as #266 (ly) RR48 9/00 | Fresh frozen |
| #272 | #223 & #225 RR38 9/96, #228 RR39 3/97 | Lyophilized |
| #273 | #203 RR33 3/95 | Lyophilized |

# Individualized RR XLIX Report: FSV-BA

## trans-β-Carotene

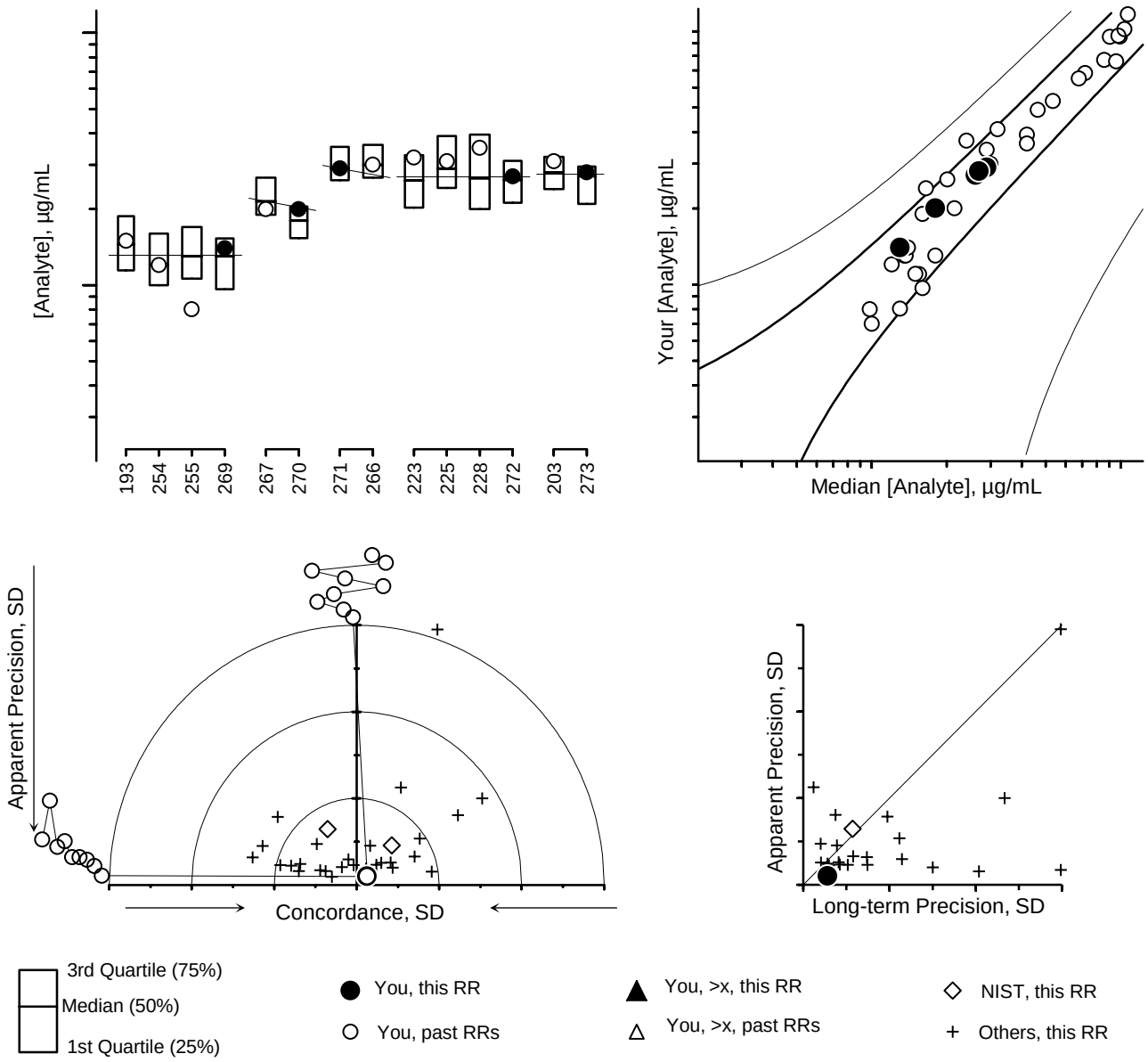



For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

| Serum | History | Comments |
|---|---|---|
| #269 | #193 RR30 3/94, #254 RR45 3/99, #255 RR46 6/99 | Lyophilized |
| #270 | Same pool as #267 (lq fz) RR48 9/00 | Lyophilized |
| #271 | Same pool as #266 (ly) RR48 9/00 | Fresh frozen |
| #272 | #223 & #225 RR38 9/96, #228 RR39 3/97 | Lyophilized |
| #273 | #203 RR33 3/95 | Lyophilized |

# Individualized RR XLIX Report: FSV-BA

## Total α-Carotene

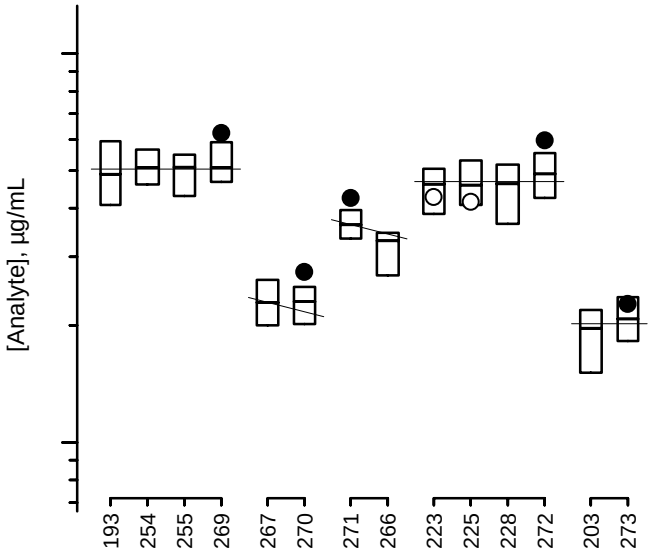


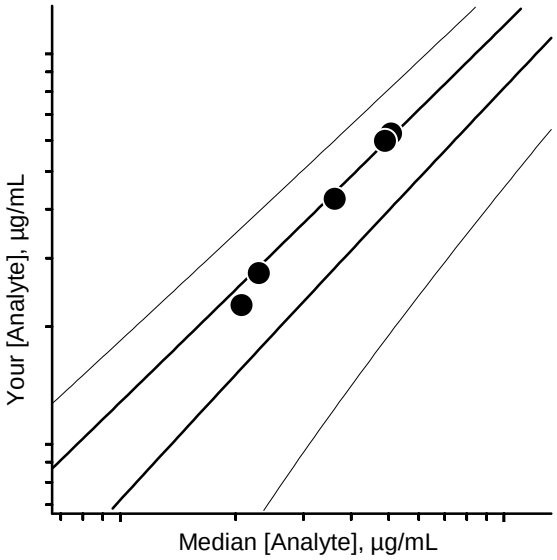

For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

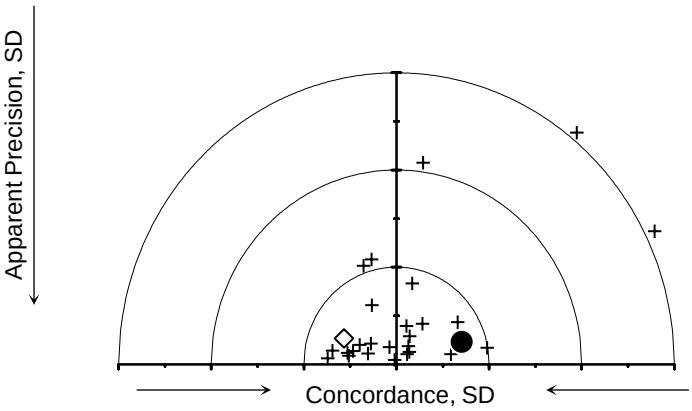
| Serum | History | Comments |
|---|---|---|
| #269 | #193 RR30 3/94, #254 RR45 3/99, #255 RR46 6/99 | Lyophilized |
| #270 | Same pool as #267 (lq fz) RR48 9/00 | Lyophilized |
| #271 | Same pool as #266 (ly) RR48 9/00 | Fresh frozen |
| #272 | #223 & #225 RR38 9/96, #228 RR39 3/97 | Lyophilized |
| #273 | #203 RR33 3/95 | Lyophilized |

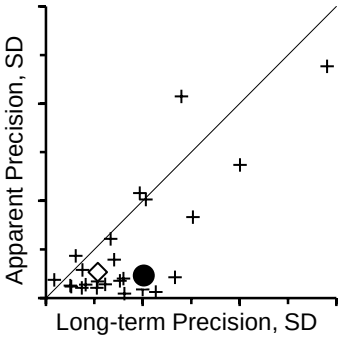
# Individualized RR XLIX Report: FSV-BA

## Total Lycopene




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

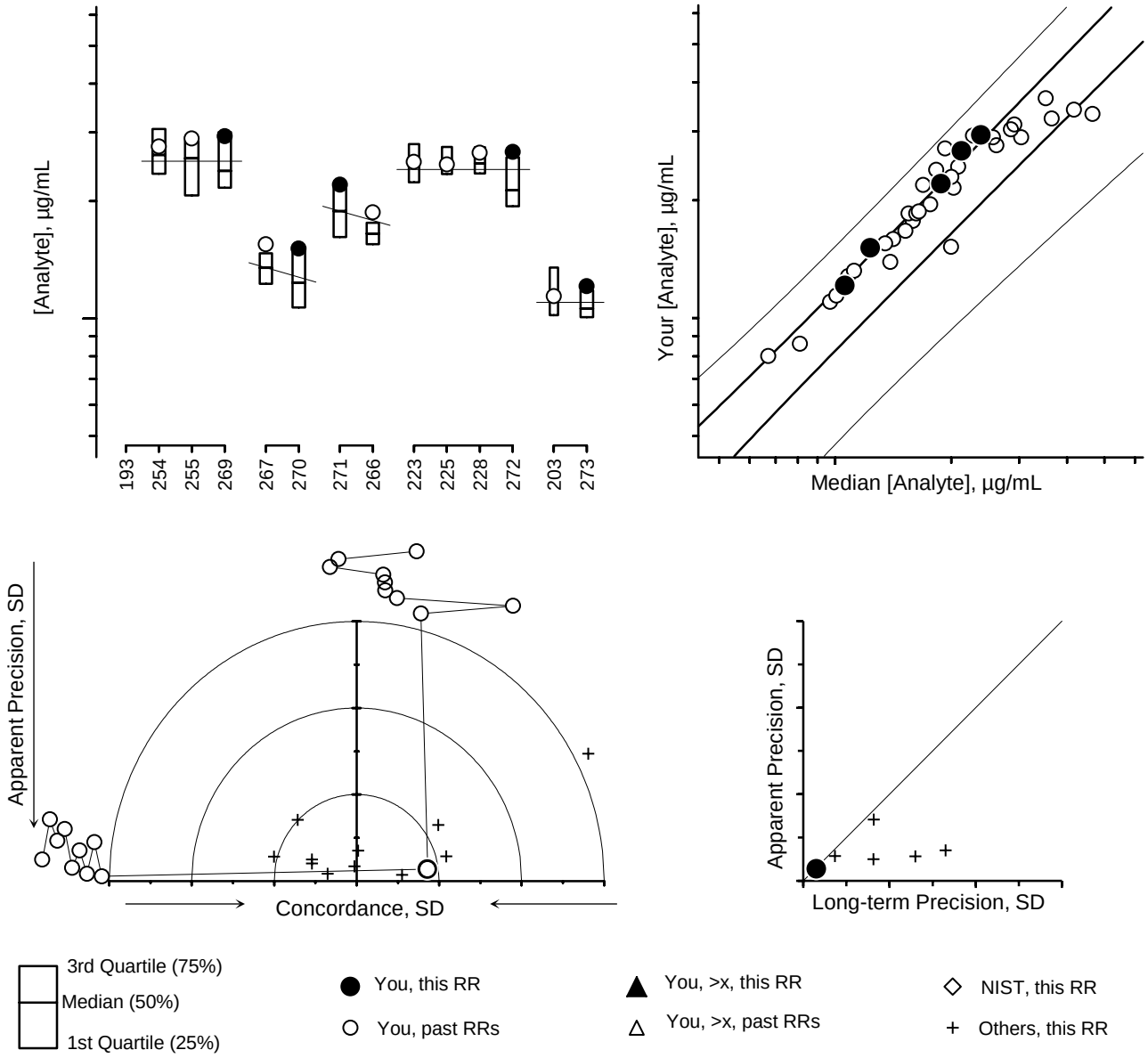
● You, this RR
○ You, past RRs
▲ You, >x, this RR
△ You, >x, past RRs
◇ NIST, this RR
+ Others, this RR

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

| Serum | History | Comments |
|---|---|---|
| #269 | #193 RR30 3/94, #254 RR45 3/99, #255 RR46 6/99 | Lyophilized |
| #270 | Same pool as #267 (lq fz) RR48 9/00 | Lyophilized |
| #271 | Same pool as #266 (ly) RR48 9/00 | Fresh frozen |
| #272 | #223 & #225 RR38 9/96, #228 RR39 3/97 | Lyophilized |
| #273 | #203 RR33 3/95 | Lyophilized |

# Individualized RR XLIX Report: FSV-BA

trans-Lycopene

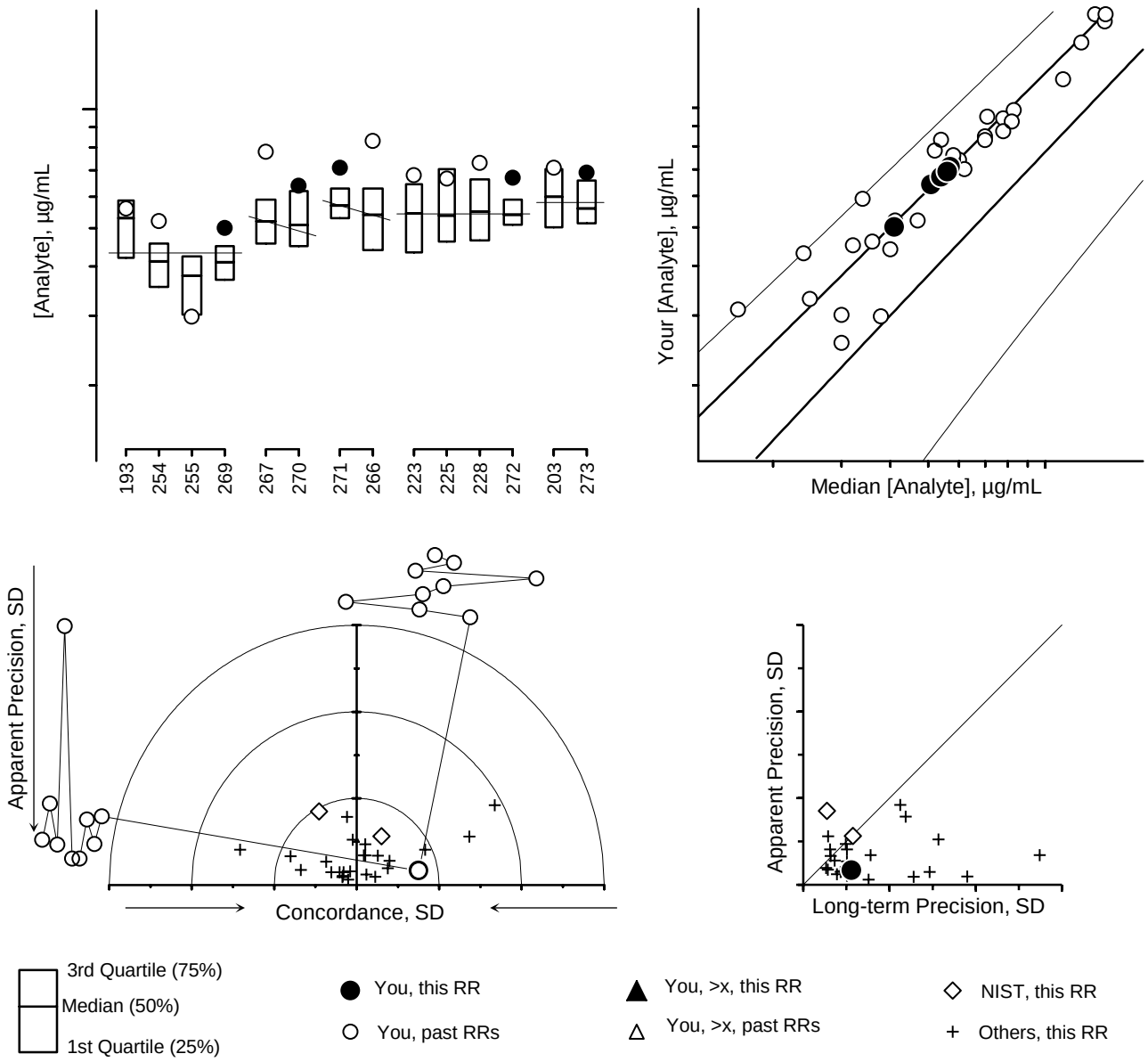



For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

| Serum | History | Comments |
|---|---|---|
| #269 | #193 RR30 3/94, #254 RR45 3/99, #255 RR46 6/99 | Lyophilized |
| #270 | Same pool as #267 (lq fz) RR48 9/00 | Lyophilized |
| #271 | Same pool as #266 (ly) RR48 9/00 | Fresh frozen |
| #272 | #223 & #225 RR38 9/96, #228 RR39 3/97 | Lyophilized |
| #273 | #203 RR33 3/95 | Lyophilized |

# Individualized RR XLIX Report: FSV-BA

## Total β-Cryptoxanthin

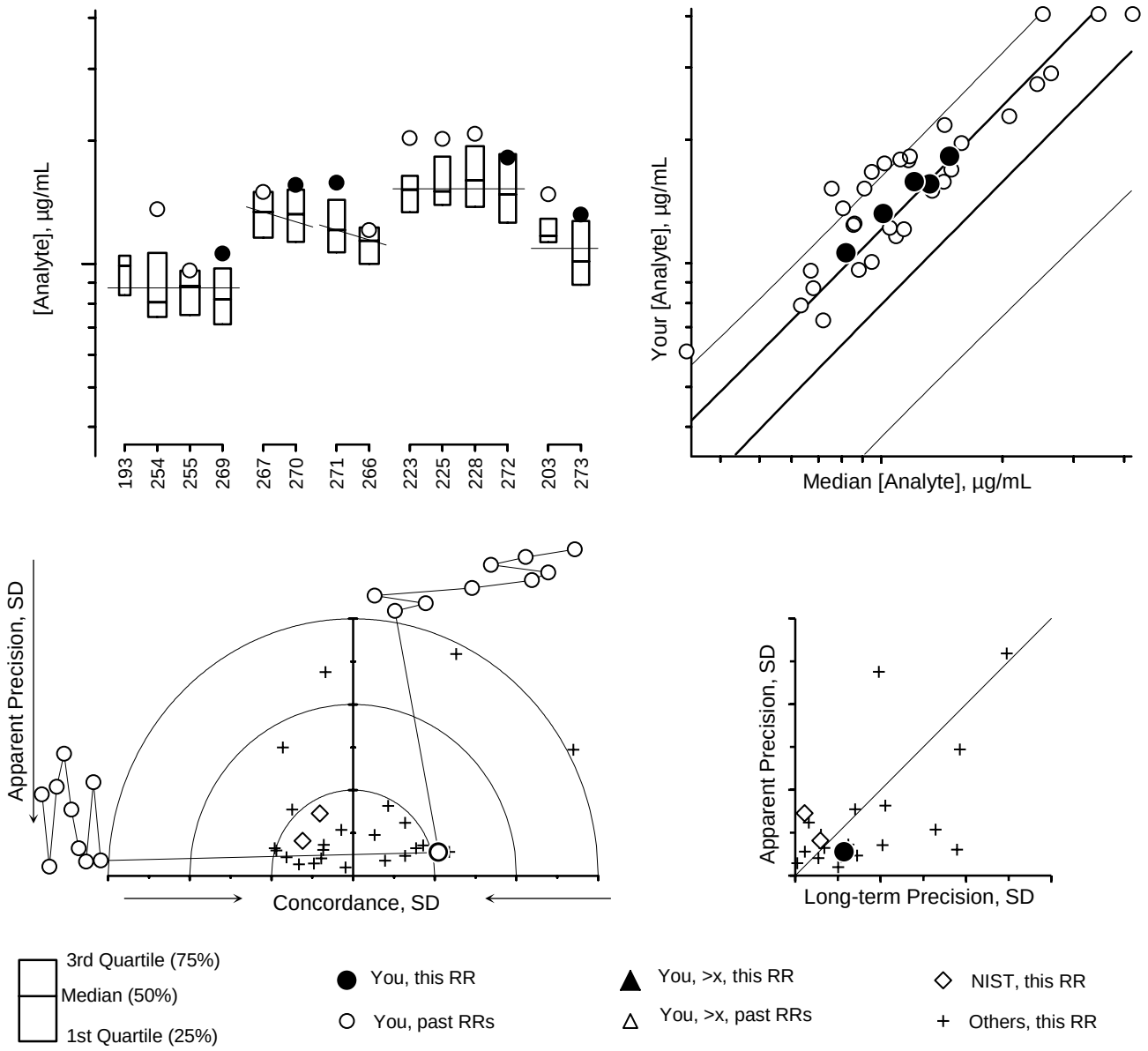



For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

| Serum | History | Comments |
|---|---|---|
| #269 | #193 RR30 3/94, #254 RR45 3/99, #255 RR46 6/99 | Lyophilized |
| #270 | Same pool as #267 (lq fz) RR48 9/00 | Lyophilized |
| #271 | Same pool as #266 (ly) RR48 9/00 | Fresh frozen |
| #272 | #223 & #225 RR38 9/96, #228 RR39 3/97 | Lyophilized |
| #273 | #203 RR33 3/95 | Lyophilized |

# Individualized RR XLIX Report: FSV-BA

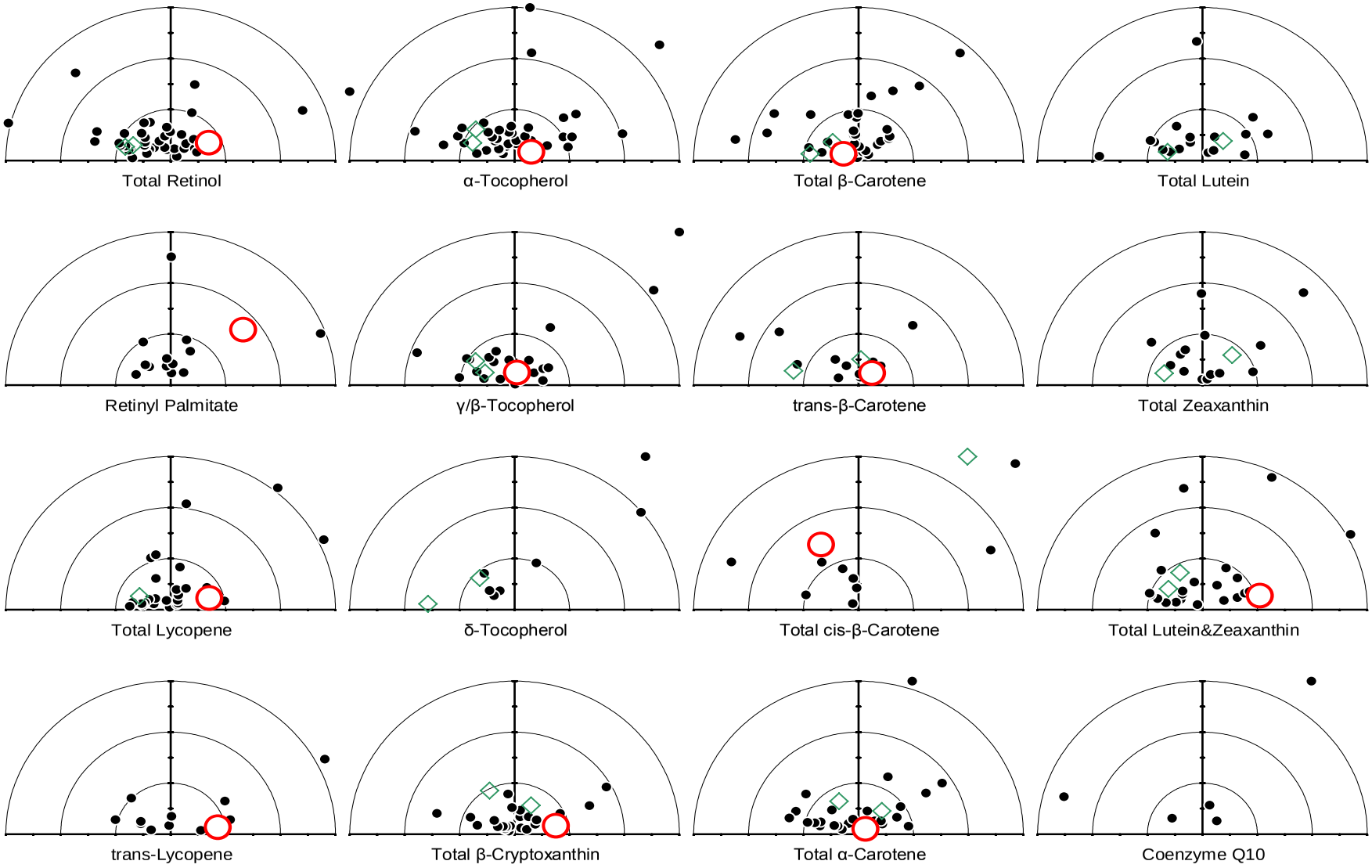
## Total Lutein&Zeaxanthin




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

| Serum | History | Comments |
|---|---|---|
| #269 | #193 RR30 3/94, #254 RR45 3/99, #255 RR46 6/99 | Lyophilized |
| #270 | Same pool as #267 (lq fz) RR48 9/00 | Lyophilized |
| #271 | Same pool as #266 (ly) RR48 9/00 | Fresh frozen |
| #272 | #223 & #225 RR38 9/96, #228 RR39 3/97 | Lyophilized |
| #273 | #203 RR33 3/95 | Lyophilized |

# Individualized Round Robin XLIX Report: FSV-BA
## Graphical Comparability Summary




D14

## Appendix E. Shipping Package Inserts for RR14

The following three items were included in each package shipped to an RR14 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

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.

We have no record, either electronic or hardcopy, of a packing list for the RR14 study.

January, 2001

Dear Colleague:

For the past 16 years the National Institute of Standards and Technology (NIST) has coordinated a Micronutrients Measurement Quality Assurance (QA) Program for laboratories making vitamin measurements in human serum. Frozen and/or freeze dried sera are sent to laboratories for analysis as an interlaboratory comparison exercise. Results are returned to NIST for data tabulation and evaluation. Value-assignment of the sample pools is based on the median of all the laboratory results, with confirmation based on measurements at NIST. We provide consultation and trouble-shooting regarding methods of analysis, and a certificate of participation in the QA program is issued at the end of each calendar year. We also host a micronutrient analysis QA workshop for fat-soluble vitamin, carotenoid, and ascorbic acid measurements in serum.

The enclosed set of samples constitute the round robin exercise for vitamin C (Round Robin XIV) for 2001. Four vials of frozen serum (*test samples*), and a vial of solid ascorbic acid (*a control sample*), are enclosed. Please follow the attached protocol when you analyze these samples.

Report your results using the attached form by **May 12, 2001**. We also request that you send us a representative chromatogram from the analysis of each sample and indicate whether peak height or peak area was used in the calculation of the ascorbic acid concentration. Your results will be kept confidential. Results received two weeks after the due date will not be included in the summary report of this round robin study. The summary report concerning this study will be provided near the end of June.

Please mail your results to:

Micronutrients Measurement Quality Assurance Program NIST
100 Bureau Drive, Stop 8392
Gaithersburg, MD 20899-8392

or Fax: 301.977.0685 E-mail: sam.margolis@nist.gov

If you have any questions or concerns please call me at 301.975.3137 or contact me by Fax or E-mail.

Thank you for your participation and we look forward to receiving your results.

Sincerely,

Sam A. Margolis, Ph.D.
Research Chemist
Analytical Chemistry Division
Chemical Science and Technology Laboratory

Enclosures

**Protocol for analyzing samples**

The *control sample* consists of a sample of solid ascorbic acid in an amber vial and should be used in the following manner (please record your weights on the attached report form):

1. Prepare 250 mL of 5% metaphosphoric acid (MPA) in distilled water.

2. Weigh **180-220 mg** of the solid ascorbic acid sample to 0.1 mg (if possible), dissolve it in 5% MPA in a 100 mL volumetric flask, and dilute to the 100 mL mark. **Weigh the amount of MPA solution that was added.** This will be referred to as the Stock Solution.

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

   *Dilute Solution* 1: **Weigh** 0.500 mL of the stock solution into a 100 mL volumetric flask. Then dilute with 5% MPA solution to 100 mL mark and **weigh the amount of MPA solution that was added**.

   *Dilute Solution* 2: **Weigh** 0.250 mL of the stock solution into a 100 mL volumetric flask. Then dilute with 5% MPA solution to 100 mL mark and **weigh the amount of MPA solution that was added**.

   *Dilute Solution* 3: **Weigh** 0.125 mL of the stock solution into a 100 mL volumetric flask. Then dilute with 5% MPA solution to 100 mL mark and **weigh the amount of MPA solution that was added**.

4. Record the ultraviolet absorbance spectrum of *Dilute Solution 1* against 5% MPA solution as the blank using paired cuvettes. Record the wavelength in the region of 240-245 nm at which you observe the maximum absorbance and record the absorbance at that wavelength.

5. Record the absorbance of the sample at 242, 243 and 244 nm.

6. Measure the concentration of the ascorbic acid in all three **dilute solutions** and the **5% MPA diluent** in duplicate along with the ampouled *test samples* using your usual methods.

The purpose of measuring the absorbance at the wavelength maximum is to check the concentration of your sample. If your spectrophotometer is properly calibrated, the maximum absorbance should be between 243 and 244 nm. If the concentration is correct, the molar extinction coefficient ($E^{1\%}$) of ascorbic acid at this wavelength (using a cell with a 1 cm path length) should be close to 550 ± 30 nm. The extinction coefficient of your solution can be calculated using the following equation:

$$E^{1\%}\ \text{dl/g·cm} = \frac{\text{Observed Absorbance}_{\lambda max}}{\dfrac{(\text{g AA/100 mL stock})(\text{g stock in 100 mL dilute solution})}{(\text{g AA stock solution}) + (\text{g MPA solution in 100 mL dilute solution 1})}}$$

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

Each *test sample* should contain between **0** and **100** µmol of ascorbic acid/L of solution. The total ascorbic acid in each ampoule should be measured in duplicate by the method(s) used in your laboratory. Please report your results in µg/L of sample.

# REPORT OF ANALYSIS

**NAME:**

**ADDRESS:**

**Telephone no.:**_____
**Fax no.:**_____

**Method of Analysis:**

*Please note the type of method that you use.* _____

*Please attach representative chromatograms.*

Method used for calculating ascorbic acid concentration.

Peak height _____ Peak area _____

Manufacturer of ascorbic acid used to make in-house standards _____

Were samples frozen upon receipt? _____ Yes _____ No

Date of Analysis:_____

## PREPARATION OF STOCK SOLUTION AND DILUTED SOLUTIONS

***STOCK SOLUTION***

Weight of ascorbic acid in the Stock Solution _____ mg
Weight of 5% MPA added to the 100 mL volumetric flask _____ g

**DILUTE SOLUTION 1**

Weight of added stock solution (0.5 mL) _____ mg
Weight of 5% MPA added to the 100 mL volumetric flask _____ g

Absorbance of Dilute Solution 1 at **243 nm** _____ AU
Absorbance of Dilute Solution 1 at **244 nm** _____ AU

Wavelength of maximum absorbance _____ nm
Calculated molar absorptivity _____ dL/g·cm

**DILUTE SOLUTION 2**

Weight of added stock solution (0.25 mL) _____ mg
Weight of 5% MPA added to the 100 mL volumetric flask _____ g

**DILUTE SOLUTION 3**

Weight of added stock solution (0.125 mL) _____ mg
Weight of 5% MPA added to the 100 mL volumetric flask _____ g

COMMENTS: (use other side if necessary)

Mail by May 12, 2001 to:
Micronutrients Measurement Quality Program
NIST, 100 Bureau Drive, Stop 8392
Gaithersburg, MD 20899-8392

Fax: 301-977-0685 Micronutrients
E-mail: sam.margolis@nist.gov

# REPORT OF ANALYSIS

## RESULTS (µmol/L of Sample)

**DILUTE SOLUTION 1**

REPLICATE 1 _____µmol/L of dilute solution 1
REPLICATE 2 _____ µmol/L of dilute solution 1

**DILUTE SOLUTION 2**

REPLICATE 1 _____µmol/L of dilute solution 2
REPLICATE 2 _____ µmol/L of dilute solution 2

**DILUTE SOLUTION 3**

REPLICATE 1 _____µmol/L of dilute solution 3
REPLICATE 2 _____ µmol/L of dilute solution 3

**TEST SAMPLE #1**

REPLICATE 1 _____µmol/L of Sample 1
REPLICATE 2 _____ µmol/L of Sample 1

**TEST SAMPLE #2**

REPLICATE 1 _____µmol/L of Sample 2
REPLICATE 2 _____ µmol/L of Sample 2

**TEST SAMPLE #3**

REPLICATE 1 _____µmol/L of Sample 3
REPLICATE 2 _____ µmol/L of Sample 3

**TEST SAMPLE #4**

REPLICATE 1 _____µmol/L of Sample 4
REPLICATE 2 _____ µmol/L of Sample 4

Mail by May 12, 2001 to:
Micronutrients Measurement Quality Program
NIST, 100 Bureau Drive, Stop 8392
Gaithersburg, MD 20899-8392

Fax: 301-977-0685 Micronutrients
E-mail: sam.margolis@nist.gov

## Appendix F. Final Report for RR14

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

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

NIST CENTENNIAL 1901-2001

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

August 8, 2001

Dear Colleague:

Enclosed is the summary report of the results for Round Robin 14 (RR14) 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; the measurement comparability summary for evaluating laboratory performance; and a summary of individual laboratory performance and interlaboratory accuracy and precision. As in previous reports, the estimated standard deviations (eSD) for the measurements are defined as 0.74 x interquartile range and the estimate coefficients of variation (eCV) are defined as 100 x eSD/median. NIST data are not included in the statistical analysis.

RR14 consists of four unknowns and one solid reference ascorbic acid for control solutions. Details regarding the samples can be found in the enclosed report.

Data for evaluating laboratory performance in RR14 are provided in the comparability summary (Score Card) on page 1 of the "All Lab Report." Laboratory comparability is summarized as follows: results rated 1 to 3 are within 1 to 3 standard deviation(s) of the assigned values; 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 970, Vitamin C in Frozen Human Serum. SRM 970 can be purchased from the NIST SRM Office 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.

If you have any questions regarding this report, please contact David Duewer at david.duewer@nist.gov, phone: 301-975-3935 or Sam Margolis at sam.margolis@nist.gov; phone: 301-975-3137; fax: 301-977-0685.

Sincerely,

Jeanice Brown Thomas

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

Sam A. Margolis, Ph.D.
Research Chemist
Analytical Chemistry Division
Chemical Science and Technology Laboratory

enclosure

NIST

The NIST $M^2QAP$ Vitamin C Round Robin 14 (RR14) report consists of

| Page | "Individualized" Report |
|---|---|
| 1 | Summarizes your reported values for the nominal 55 mmol/L control solution and the SRM 970 Level 1 and 2 samples distributed in RR11 through RR14. |
| 2 | Graphical summary of your RR 14 sample measurements. |
| 3 | Graphical summary of your RR 14 control solution measurements. |

| Page | "All Lab" Report |
|---|---|
| 1 | A listing of all results and statistics for Total Ascorbic Acid [TAA] in the RR14 samples and control solutions, the density of the 5% metaphosphoric acid (MPA) used to prepare the control solutions, the maximum absorbance reported between 243 nm and 245 nm for control solution #1, and the molar extinction coefficient. |

**Samples.** Four unknowns and a control material (ascorbic acid) were distributed in RR14. . Serum sample 1 was prepared (04/93) as sample 180a. Serum sample 2 was prepared (03/95) as sample 188b. Sera samples 3 and 4 were prepared (05/98) as SRM 970, Level 1 and Level 2, respectively. Each serum sample was prepared by adding ascorbic acid to a serum pool that was depleted of ascorbic acid.

**Qualitative Observations**.

1) Too few sets of data have been returned to place a great deal of faith in the statistical summaries. If the program is to continue, the number of active participants must be increased. Suggestions are more than welcome!

2) Many participants that reported "Not detected" for the "blank" (S14-2) sample had significantly non-zero intercepts for their external calibration curve. While these intercepts averaged about 1 μmol/L (the expected value for [TAA] in S14-2), they ranged as high as 13 μmol/L. Future exercises will request a "true" 5% MPA blank to help sort things out.

3) Almost all participants correctly prepared the control solutions, although one participant appears to be using about 3% MPA and one about 10%. While the actual concentration of MPA in the nominal 5% is not believed to be a critical parameter, these two participants reported the most extreme (low and high) molar extinction coefficients ($E^{1\%}$). These differences may influence calibrant purity corrections.

4) Based upon 1% to 10% differences between reported and calculated E1% values, several participants based their calculations on volume rather than weight of stock solution delivered to control solution #1. In general, 0.500 g gravimetric measurements are more accurate than are 0.500 mL volumetric measurements. These differences may influence calibrant purity corrections.

5) All participants are now reporting "reasonable" maximum absorbance values ($OD_{max}$) for the nominal 55 μmol/L control solution #1. However, several participants report the location of maximal absorbance 1 nm to 2 nm offset from its nominal location of 244 nm. For most accurate results, spectrophotometers require periodic calibration of both their absorbance and wavelength axes.

**Quantitative Results.**

1) There is no sign of degradation (change in median [TAA] or increase in estimated standard deviation) in either SRM 970 Level 1 or Level 2 since their Certification in 1998. We will continue to periodically monitor these materials.

## Appendix G. "All-Lab Report" for RR14

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

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

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

# Vitamin C 'Round Robin' 14
## Laboratory Results

| | | | Total Ascorbic Acid | | | | $[AA]_{nom}$ ($[AA]_{obs}$ / $[AA]_{calc}$) | | | Ctrl1 Properties | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | S14:1 | S14:2 | S14:3 | S14:4 | 14-Ctrl1 | 14-Ctrl2 | 14-Ctrl3 | $D_{5\%MPA}$ | $A_{max}$ | $OD_{max}$ | $E^{1\%}$ |
| Lab | Date | Method | µmol/L solution | | | | Nominal µmol/L solution | | | g/mL | nm | AU | dL/g·cm |
| VC-MA | 27/09/01 | HPLC-EC | 20.0 | 0.00 | 7.8 | 26.0 | 56.03 | 26.54 | 13.83 | 1.034 | 243.0 | 0.5409 | 548 |
| VC-MB | 26/03/01 | AO-OPD, Cobas Fara | 23.0 | 3.60 | 9.8 | 30.2 | 57.54 | 29.32 | 15.38 | 1.033 | 245.0 | 0.5908 | 566 |
| VC-MC | 02/05/01 | HPLC-EC | 26.0 | 6.32 | 11.4 | 33.0 | 60.08 | 30.72 | 14.60 | 1.032 | 243.0 | 0.5773 | 572 |
| VC-ME | 17/05/01 | Cobas Bio Fluoro | 18.7 | 0.04 | 6.5 | 25.7 | 54.03 | 26.83 | 13.42 | 1.032 | 243.6 | 0.6280 | 568 |
| VC-MG | 02/03/01 | HPLC-UV-ionpair | 18.0 | 0.00 | 9.1 | 27.5 | 55.35 | 24.49 | 10.91 | 1.032 | 243.6 | 0.5870 | 584 |
| VC-MH | 11/04/01 | HPLC-Fluor OPD | 19.8 | 0.00 | 7.9 | | 54.95 | 27.59 | 13.97 | 1.033 | 243.0 | 0.5461 | 569 |
| VC-MI | 11/05/01 | HPLC-UV | 29.3 | 0.00 | | 31.4 | 78.76 | 33.67 | 15.30 | 1.035 | | | |
| VC-MO | 15/02/01 | HPLC-OPD | 16.7 | 0.26 | 8.1 | 23.7 | 54.47 | 29.50 | 15.45 | 1.032 | 243.0 | 0.5617 | 556 |
| VC-MQ | 05/06/01 | HPLC-EC | 18.7 | 1.49 | 7.9 | 24.8 | 53.78 | | | 1.043 | 242.0 | 0.5090 | 527 |
| VC-MR | 13/03/01 | HPLC-OPDA | 20.5 | 0.00 | 9.0 | 28.5 | 57.17 | 28.67 | 15.25 | 1.032 | 244.0 | 0.5520 | 538 |
| VC-MS | 13/04/01 | HPLC-EC | 19.0 | 0.00 | 7.9 | 25.7 | | | | 1.031 | 244.0 | 0.5369 | 542 |
| VC-NH | 23/04/01 | HPLC-Fluor DAB | 22.8 | | 9.5 | | 53.49 | 27.11 | 13.73 | 1.034 | 243.0 | 0.5215 | 565 |
| VC-NK | 27/04/01 | 24DNPH | 36.7 | 0.00 | 12.2 | 21.0 | 58.25 | 36.12 | 23.63 | 1.029 | 243.0 | 0.5470 | 543 |
| NIST | 18/04/01 | HPLC-EC | 20.6 | 1.7 | 9.1 | 27.1 | 52.58 | 25.79 | 13.31 | 1.031 | 243.6 | 0.5643 | 572 |
| | | N | 13 | 12 | 12 | 11 | 12 | 11 | 11 | 13 | 12 | 12 | 12 |
| | | Average | 22.2 | 1.0 | 8.9 | 27.0 | 57.8 | 29.1 | 15.0 | 1.033 | 243.4 | 0.558 | 557 |
| | | Min | 16.7 | 0.00 | 6.5 | 21.0 | 53.49 | 24.49 | 10.91 | 1.029 | 242.0 | 0.509 | 527 |
| | | %25 | 18.7 | 0.00 | 7.9 | 25.2 | 54.36 | 26.97 | 13.78 | 1.032 | 243.0 | 0.540 | 543 |
| | | Median | 20.0 | 0.00 | 8.6 | 26.0 | 55.69 | 28.67 | 14.60 | 1.032 | 243.0 | 0.550 | 561 |
| | | %75 | 23.0 | 0.57 | 9.6 | 29.4 | 57.72 | 30.11 | 15.34 | 1.034 | 243.7 | 0.580 | 568 |
| | | Max | 36.7 | 6.32 | 12.2 | 33.0 | 78.76 | 36.12 | 23.63 | 1.043 | 245.0 | 0.628 | 584 |
| | | eSD | 3.0 | 0.00 | 1.1 | 3.4 | 2.60 | 2.71 | 1.15 | 0.001 | 0.44 | 0.030 | 18 |
| | | eCV | 15 | | 13 | 13 | 5 | 9 | 8 | 0.1 | 0 | 5.4 | 3.2 |

G2

## Appendix H. Representative "Individualized Report" for RR14

Each participant in RR14 received an "Individualized Report" reflecting their reported results. The following three pages are the "Individualized Report" for participant "VC-MA".

# Vitamin C 'Round Robin' 14 Report: Participant VC-MA

| Date | Method | RR | Control | AA mg | MPA g | Stock mg | MPA g | [AA] µmol/L Calc | [AA] µmol/L Obs | $A_{243}$ OD | $A_{244}$ OD | $A_{245}$ OD | $E^{1\%}_{max}$ dL/gcm |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 09/23/98 | HPLC-EC (Height) | 11 | 55 µmol/L | 200.0 | 103.09 | 526.0 | 102.45 | 57.8 | 6.1 | 0.0525 | 0.0527 | | 0 |
| 04/02/99 | HPLC-EC (Height) | 12 | 55 µmol/L | 215.0 | 103.10 | 517.1 | 102.52 | 61.1 | 53.1 | 0.0721 | 0.0721 | | 67 |
| 09/17/01 | HPLC-EC | 13 | 55 µmol/L | 200.8 | 103.16 | 508.0 | 102.58 | 56.0 | 55.4 | 0.5650 | 0.5232 | | 572 |
| 10/02/01 | HPLC-EC (Height) | 14 | 55 µmol/L, Crtl-1 | 200.3 | 103.23 | 510.0 | 102.31 | 56.1 | 57.1 | 0.5409 | 0.5193 | | 548 |
| | | | | | | | | | | | | | 297 ±305 |

| RR | Sample | [TAA] µmol/$L_{sample}$ $Rep_1$ | $Rep_2$ | *Factor* | Mean | $SD_{dup}$ | Grand Average Mean | $SD_{repeat}$ | $SD_{reprod}$ |
|---|---|---|---|---|---|---|---|---|---|
| 11 | SRM Lv 1, A | 15.5 | 13.9 | 0.5 | 7.4 | 0.6 | 7.8 | 0.3 | 0.5 |
| 11 | SRM Lv 1, B | 14.0 | 14.5 | 0.5 | 7.1 | 0.2 | | | |
| 12 | SRM Lv 1, A | 14.5 | 15.8 | 0.5 | 7.6 | 0.5 | | | |
| 12 | SRM Lv 1, B | 16.1 | 15.1 | 0.5 | 7.8 | 0.3 | | | |
| 13 | SRM Lv 1, S13-1 | 8.4 | 8.5 | 1.0 | 8.5 | 0.1 | | | |
| 14 | SRM Lv 1, S14-3 | 8.0 | 7.8 | 1.0 | 7.9 | 0.2 | | | |
| 11 | SRM Lv 2, A | 50.7 | 47.7 | 0.5 | 24.6 | 1.1 | 25.6 | 1.1 | 1.7 |
| 11 | SRM Lv 2, B | 48.8 | 54.0 | 0.5 | 25.7 | 1.8 | | | |
| 12 | SRM Lv 2, A | 49.5 | 45.9 | 0.5 | 23.9 | 1.3 | | | |
| 12 | SRM Lv 2, B | 46.2 | 47.9 | 0.5 | 23.5 | 0.6 | | | |
| 13 | SRM Lv 2, S13-2 | 27.6 | 27.7 | 1.0 | 27.7 | 0.1 | | | |
| 14 | SRM Lv 2, S14-4 | 25.7 | 26.4 | 1.0 | 26.0 | 0.5 | | | |
| 14 | S14-1 (180B) | 20.4 | 19.6 | 1.0 | 20.0 | 0.6 | | | |
| 14 | S14-2 (188B) | | | 1.0 | 0.0 | 0.0 | | | |

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

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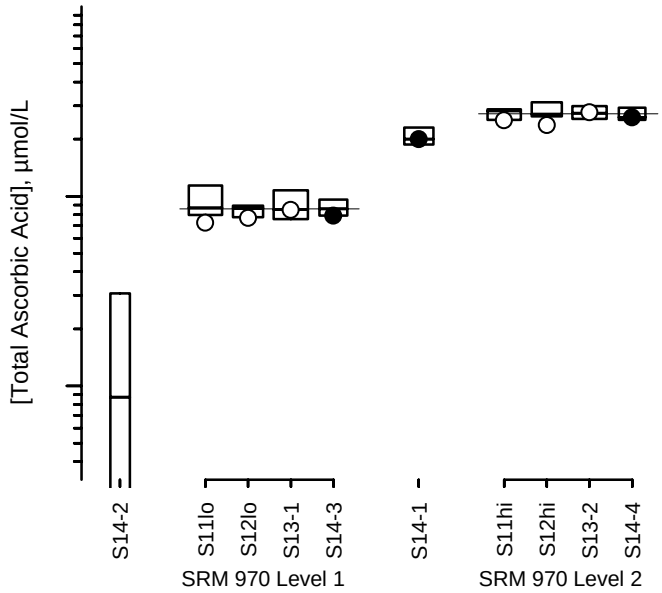
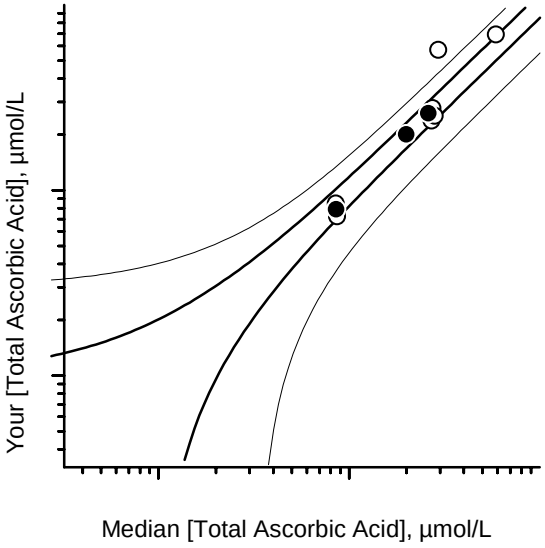
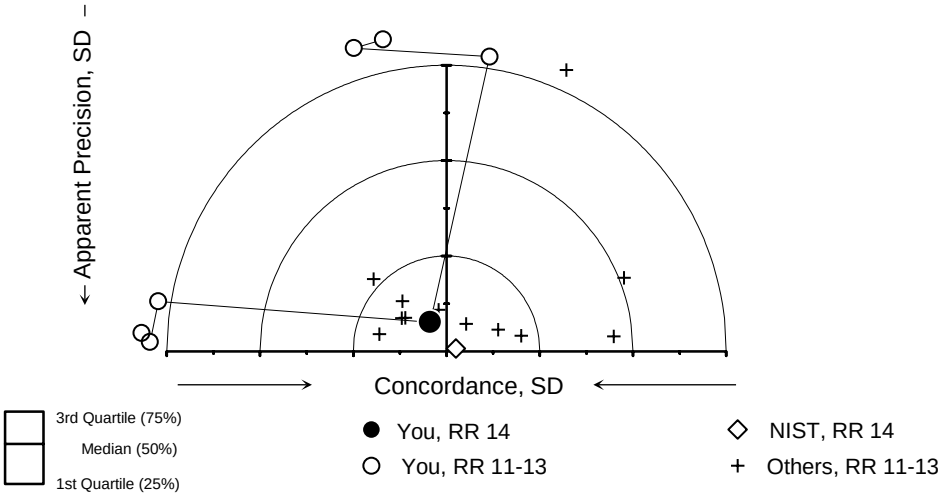
H2

# Vitamin C 'Round Robin' 14 Report: Participant VC-MA

## Total Ascorbic Acid: Samples



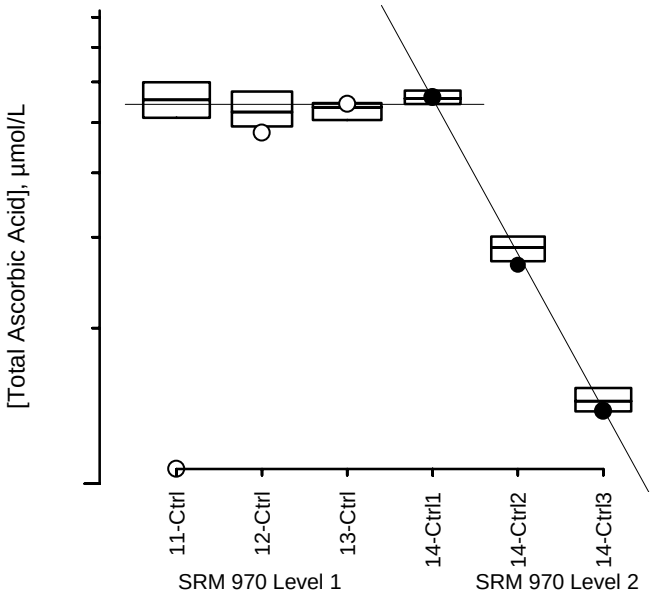



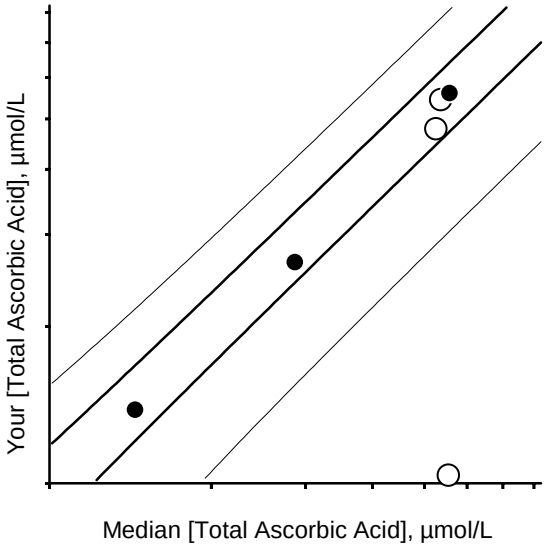

For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

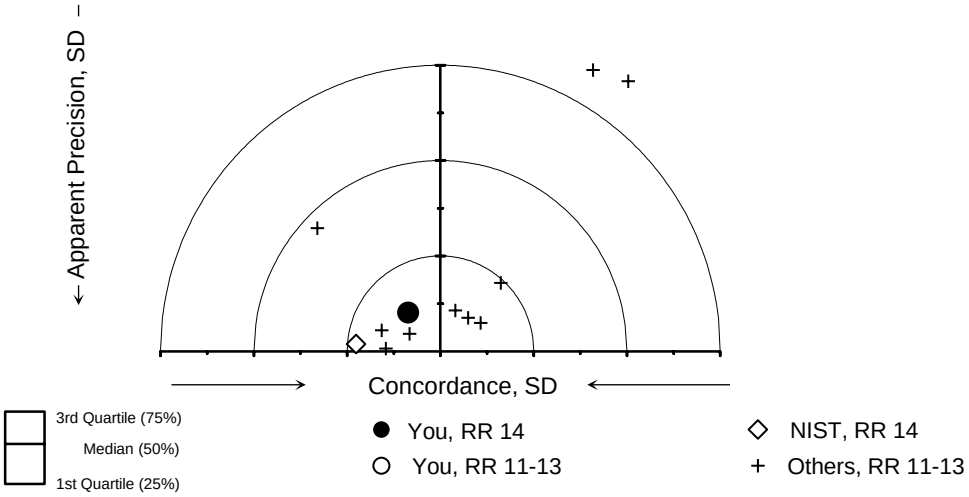
| Sample | | Comments |
|---|---|---|
| S14-1 | Serum 180B, augmented | |
| S14-2 | Serum 188B, no augmentation | |
| S14-3 | SRM 970 Level 1 | |
| S14-4 | SRM 970 Level 2 | |

# Vitamin C 'Round Robin' 14 Report: Participant VC-MA

## Total Ascorbic Acid: Control Solutions




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 |
|---|---|
| 14Ctrl-1 | Control Solution 1, nominally 55.0 µm/mL. Displayed Value = (Nominal)(Measured)/(Calculated) |
| 14Ctrl-2 | Control Solution 2, nominally 27.5 µm/mL. Displayed Value = (Nominal)(Measured)/(Calculated) |
| 14Ctrl-3 | Control Solution 3, nominally 13.75 µm/mL. Displayed Value = (Nominal)(Measured)/(Calculated) |