**NISTIR 7880-18**

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Results for Round Robin LIV
Fat-Soluble Vitamins and Carotenoids in Human Serum
and Round Robin 19 Ascorbic Acid in Human Serum

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

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*Materials Measurement Laboratory*

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June 2013

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

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

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## Abstract

The National Institute of Standards and Technology coordinates the Micronutrients Measurement Quality Assurance Program (MMQAP) for laboratories that measure fat- and water-soluble vitamins and carotenoids in human serum and plasma. This report describes the design of and results for the Summer 2003 MMQAP measurement comparability improvement studies: 1) Round Robin LIV Fat-Soluble Vitamins and Carotenoids in Human Serum and 2) Round Robin 19 Total Ascorbic Acid in Human Serum. The materials for both studies were shipped to participants in March 2003; participants were requested to provide their measurement results by September 19, 2003.



## 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 LIV: Fat-Soluble Vitamins and Carotenoids in Human Serum

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin LIV comparability study (hereafter referred to as RR54) received two lyophilized and three liquid-frozen human serum test samples for analysis. Unless multiple vials were previously requested, participants received one vial of each serum. These sera were shipped on dry ice to participants in March 2003. 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 RR54 consists of three documents:

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

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

- An “Individualized Report” that graphically analyzes each participant’s results for all analytes reported by at least five participants. This report also provides a graphical summary of their measurement comparability. The graphical tools used in this report are described in detail elsewhere [3]. An example “Individualized Report” is reproduced as Appendix D.

## Round Robin 19: Vitamin C in Human Serum

Participants in the MMQAP Vitamin C in Human Serum Round Robin 19 comparability study (hereafter referred to as RR19) 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 March 2003. 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 RR19 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 RR54

The following three items were included in each package shipped to an RR54 participant:

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

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

**UNITED STATES DEPARTMENT OF COMMERCE**
**National Institute of Standards and Technology**
Gaithersburg, Maryland 20899-0001

March 26, 2003

Dear Colleague:

Enclosed are the samples (Sera 294 – 298) for the second fat-soluble vitamins and carotenoids in serum round robin study (Round Robin LIV) for the 2003 NIST Micronutrients Measurement Quality Assurance Program. You will find one vial of each of two liquid-frozen and three lyophilized serum samples for analysis along with a form for reporting your results. When reporting your results, please submit one value for each analyte for a given serum sample. If a value is obtained below your limit of quantification, please indicate this result on the form by using NQ *(Not Quantified)*. Results are due to NIST by **September 19, 2003**. Results received more than two weeks after the due date will not be included in the summary report for this round robin study. The feedback report concerning the study will be provided around mid-October.

Lyophilized samples should be reconstituted with 1.0 mL of HPLC-grade water or equivalent. We recommend that dissolution be facilitated with 3 to 5 min agitation in an ultrasonic bath or at least 30 min at room temperature with intermittent swirling. (CAUTION: Vigorous shaking will cause foaming and possibly interfere with accurate measurement. The rubber stopper contains phthalate esters that may leach into the sample upon intermittent contact of the liquid sample with the stopper. These esters absorb strongly in the UV region and elute near retinol in most LC systems creating analytical problems.) Pipette a known volume of serum from the vial for analysis. The final volume of the reconstituted sample is greater than 1.0 mL. **Water should not be added to the liquid-frozen samples 296 and 298.**

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

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

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

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

Sincerely,

Jeanice B Thomas

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

Enclosures

NIST

Participant #: ________ Date: ____________

# Round Robin LIV

## NIST Micronutrients Measurement Quality Assurance Program

| Analyte | 294 | 295 | 296 | 297 | 298 | Units* |
|---|---|---|---|---|---|---|
| total retinol | | | | | | |
| trans-retinol | | | | | | |
| didehydroretinol | | | | | | |
| retinyl palmitate | | | | | | |
| $\alpha$-tocopherol | | | | | | |
| $\gamma/\beta$-tocopherol | | | | | | |
| $\delta$-tocopherol | | | | | | |
| total $\beta$-carotene | | | | | | |
| trans-$\beta$-carotene | | | | | | |
| total cis-$\beta$-carotene | | | | | | |
| total $\alpha$-carotene | | | | | | |
| total lycopene | | | | | | |
| trans-lycopene | | | | | | |
| total $\beta$-cryptoxanthin | | | | | | |
| total $\alpha$-cryptoxanthin | | | | | | |
| total lutein | | | | | | |
| total zeaxanthin | | | | | | |
| total lutein&zeaxanthin | | | | | | |
| total Coenzyme Q10 | | | | | | |
| ubiquinol ($QH_2$) | | | | | | |
| ubiquinone (Qox) | | | | | | |
| phylloquinone ($K_1$) | | | | | | |
| 25-hydroxyvitamin D | | | | | | |

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

* we prefer µg/mL

Were sera {296,297,298} frozen when received? Yes | No

Comments:

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

Please return results **before**
19-Sep-2003

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

Participant #: ________ Date: ____________

Fat-Soluble Vitamins Round Robin LIV

NIST Micronutrients Measurement Quality Assurance Program

## Packing List and Shipment Receipt Confirmation Form

This box contains (we hope) one vial each of the following **five** FSV $M^2$QAP sera:

| Serum | Form | Reconstitute? |
|---|---|---|
| #294 | Lyophilized | Yes (1 ml $H_2O$) |
| #295 | Lyophilized | Yes (1 ml $H_2O$) |
| #296 | Liquid frozen | No |
| #297 | Liquid frozen | No |
| #298 | Liquid frozen | No |

**Please** 1) Open the pack immediately
2) Check that it contains one vial each of the above samples
3) Check if sera {296,297,298} arrived frozen
4) Store the samples at -20 ˚C or below until analysis
5) Complete the following information
6) Fax the completed form to us at 301-977-0685
(or email requested information to david.duewer@nist.gov)

1) Date this shipment arrived: ___________________________

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

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

4) Did sera {296,297,298} arrive frozen? Yes **|** No

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

6) When do you anticipate analyzing these samples? ________________________

Your prompt return of this information is appreciated.

The $M^2$QAP Gang

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

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

## Appendix B. Final Report for RR54

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

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

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

October 15, 2003

Dear Colleague:

Enclosed is the summary report of the results for Round Robin LIV (RR 54) for fat-soluble vitamins and carotenoids. Included in this report are: (1) a summary of data and measurement comparability scores for all laboratories, (2) a detailed graphical analysis of your results; and (3) a graphical summary of your measurement comparabilities relative to the NIST assigned values. 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 54 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.

Samples for the first round robin exercise (RR55) for the 2004 QA program will be distributed during **the week of November 17, 2003.** We will send you a reminder via e-mail or fax a week prior to shipment. It is critical that you carefully inspect all samples upon arrival and that you promptly confirm to us that they have arrived. We will replace samples (lost or damaged in shipment or miss-packaged by us) only for participants who report the problem within one calendar week after the package arrives.

Please mark your calendars. Our next QA workshop will be held in conjunction with Experimental Biology '04 on Wednesday, April 21, 2004 in Washington, DC. A preliminary workshop agenda can be found at: www.asns.org. We will send you more information about the workshop as our plans are finalized.

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

Sincerely,

Jeanice B Thomas

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

Cc: D.L. Duewer
L.C. Sander
S.A. Wise

Enclosures

NIST

The NIST $M^2QAP$ Round Robin LIV (RR54) report consists of:

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

| 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. |

**Samples**. The five sera below were distributed in RR54.

| Serum | Description | Prior Distributions |
|---|---|---|
| 294 | Lyophilized, low-carotenoid pool augmented with trans-retinol; retinyl palmitate; α-, γ-, and δ-tocopherol, *trans*-α- and β-carotene, and *trans*-lycopene; serum pool was prepared in 1995. | Serum 203 RR33 (Mar-95)<br>Serum 273 RR16 (Mar-01) |
| 295 | Lyophilized, native, single donor serum prepared in 1999. The same material was used to prepare Serum 296. | Serum 266 RR48 (Sep-00)<br>Serum 277 RR50 (Sep-01)<br>Serum 282 RR51 (Mar-02) |
| 296 | Fresh-frozen, native, single donor serum prepared in 1999. The same material was used to prepare Serum 295. | Serum 271 RR49 (Mar-01)<br>Serum 275 RR50 (Sep-01)<br>Serum 279 RR51 (Mar-02) |
| 297 | The same fresh-frozen blended serum as Serum 298, augmented with ≈0.3 µg/mL *trans*-retinol; serum pool was prepared in 2002. | Serum 285 RR52 (Sep-02) |
| 298 | The same fresh-frozen blended serum as Serum 297, augmented wit`h ≈0.3 µg/mL 13-*cis*-retinol; serum pool was prepared in 2002. | Serum 286 RR52 (Sep-02) |

**Results**

1) Sera Stability. There was no significant change in the median level or in the variability of most measurands in the lyophilized Serum 294 after 8 years; however, there was an apparent decline in retinyl palmitate between the first distribution in 1995 and the second in 2001. Since this serum was prepared using an experimental augmentation protocol, this may reflect a real change in level. Given that the 2003 results are nearly the same as those of 2001, the apparent decline could also be a

measurement artifact.

There has been no significant change in the median level or variability for any measurand in the fresh-frozen Serum 296 after two years nor in Sera 297 and 298 after 1 year.

2) Matrix (Lyophilized Vs Fresh-Frozen) Differences. Sera 295 and 296 were prepared from the same serum pool. Since we suggest that you reconstitute our lyophilized samples with 1.0 mL water rather than to a total volume of 1.0 mL, the measurand levels in Serum 295 should be ≈ 95% of those in Serum 296. The observed average ±SD over all measurands with 10 or more quantitative measurements is 94.0 ±1.2%. If any of your Sera 295/296 ratios are much different than 0.95, you may want to take a hard look at your measurement system for those measurands.

3) Total Retinol and *trans*-retinol. Sera 297 and 298 are identical except that Serum 297 was augmented with about 0.3 µg/mL *trans*-retinol and Serum 297 was augmented with the same concentration of 13-*cis*-retinol. This is a repeat of last year's evaluation of "total retinol" and "*trans*-retinol" reporting. At that time, there were three different types of anomalous reports: (1) total values reported as *trans*, (2) *trans* values reported as total, and (3) something about half-way in-between total and *trans* reported as total retinol.

   The good news: there were no type (1) or (2) anomalies reported in this exercise. All participants who reported *trans*-retinol reported the expected values for both the *trans*- and *cis*-spiked sera.

   The less good news: there are still type (3) anomalies. Several participants reported a "Total retinol" value for the *cis*-retinol spiked Serum 298 that is roughly half-way between the value expected for *trans*- and Total retinol. We believe that this results from quantitation by peak *height* in combination with modest *chromatographic separation* between the 13-*cis*- and *trans*-retinol isomers. If you quantitate by peak height and your value for total retinol for Serum 298 was more than 0.05 µg/mL lower than that for Serum 297, you should consider using peak area to quantify total retinol.

## Appendix C. “All-Lab Report” for RR54

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

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

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

# Round Robin LIV Laboratory Results

All Values in µg/mL

| Lab | Total Retinol | | | | | trans-Retinol | | | | | Retinyl Palmitate | | | | | α-Tocopherol | | | | | γ/β-Tocopherol | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 294 | 295 | 296 | 297 | 298 | 294 | 295 | 296 | 297 | 298 | 294 | 295 | 296 | 297 | 298 | 294 | 295 | 296 | 297 | 298 | 294 | 295 | 296 | 297 | 298 |
| FSV-BA | 1.263 | 0.454 | 0.500 | 0.671 | 0.582 | | | | | | 0.047 | 0.050 | 0.055 | 0.019 | 0.016 | 17.21 | 6.76 | 7.27 | 2.84 | 2.83 | 1.22 | 1.90 | 2.07 | 0.75 | 0.76 |
| FSV-BB | 1.169 | 0.439 | 0.430 | 0.583 | 0.561 | | | | | | 0.045 | 0.046 | 0.049 | 0.012 | 0.013 | 17.05 | 6.68 | 6.70 | 2.76 | 2.69 | 1.16 | 1.81 | 1.82 | 0.69 | 0.68 |
| FSV-BD | 1.252 | 0.452 | 0.467 | 0.641 | 0.621 | | | | | | | | | | | 18.20 | 8.00 | 7.50 | 3.00 | 3.20 | | | | | |
| FSV-BE | 1.370 | 0.470 | 0.540 | 0.680 | 0.600 | | | | | | | | | | | 18.00 | 7.20 | 7.80 | 3.30 | 3.30 | 1.20 | 1.90 | 2.00 | 0.70 | 0.70 |
| FSV-BF | 0.970 | 0.410 | 0.410 | 0.580 | 0.460 | | | | | | | | | | | 12.70 | 7.10 | 7.30 | 2.60 | 2.60 | 1.30 | 1.84 | 1.77 | 0.77 | 0.82 |
| FSV-BG | 1.274 | 0.453 | 0.492 | 0.651 | 0.622 | | | | | | 0.031 | 0.049 | 0.061 | 0.031 | 0.033 | 17.12 | 7.03 | 7.63 | 3.03 | 2.98 | 1.26 | 1.83 | 2.00 | 0.81 | 0.77 |
| FSV-BH | 1.255 | 0.478 | 0.493 | 0.700 | 0.474 | | | | | | | | | | | 18.05 | 7.44 | 7.50 | 3.10 | 3.06 | 1.45 | 2.33 | 2.40 | 0.90 | 0.89 |
| FSV-BI | 1.287 | 0.449 | 0.472 | 0.638 | 0.611 | | | | | | 0.064 | 0.045 | 0.058 | 0.023 | 0.018 | 17.54 | 7.09 | 7.22 | 2.94 | 2.92 | 1.23 | 1.93 | 1.97 | 0.77 | 0.77 |
| FSV-BJ | 1.233 | 0.432 | 0.450 | 0.612 | 0.600 | | | | | | 0.050 | *nq* | *nq* | *nq* | *nq* | 17.66 | 6.70 | 7.00 | 2.77 | 2.76 | 1.21 | 1.79 | 1.87 | 0.67 | 0.69 |
| FSV-BK | 1.250 | 0.410 | 0.420 | 0.560 | 0.530 | | | | | | | | | | | 15.55 | 5.90 | 6.05 | 3.20 | 3.10 | | | | | |
| FSV-BL | - | 0.400 | 0.460 | 0.600 | 0.600 | | | | | | | | | | | - | 7.32 | 8.18 | 3.45 | 3.45 | | | | | |
| FSV-BM | 1.218 | 0.489 | 0.509 | 0.651 | 0.673 | | | | | | | | | | | 18.00 | 7.30 | 7.80 | 4.00 | 4.20 | | | | | |
| FSV-BN | 1.260 | 0.436 | 0.422 | 0.639 | 0.624 | | | | | | 0.042 | 0.042 | 0.038 | 0.012 | 0.015 | 14.83 | 5.91 | 5.55 | 2.61 | 2.57 | 0.94 | 1.46 | 1.34 | 0.57 | 0.60 |
| FSV-BO | 1.120 | 0.415 | 0.444 | 0.599 | 0.570 | | | | | | | | | | | 16.45 | 6.55 | 7.06 | 2.80 | 2.84 | | | | | |
| FSV-BP | 1.191 | 0.455 | 0.475 | 0.613 | 0.607 | | | | | | | | | | | 16.75 | 6.75 | 6.66 | 3.06 | 3.070 | | | | | |
| FSV-BQ | 1.320 | 0.458 | 0.494 | 0.642 | 0.554 | | | | | | | | | | | 16.80 | 6.50 | 7.20 | 2.90 | 2.70 | | | | | |
| FSV-BR | ≥1.180 | ≥0.440 | ≥0.460 | ≥0.610 | ≥0.350 | 1.180 | 0.440 | 0.460 | 0.610 | 0.350 | | | | | | 17.50 | 7.10 | 7.30 | 2.89 | 3.11 | | | | | |
| FSV-BS | 1.261 | 0.362 | 0.417 | 0.616 | 0.579 | | | | | | | | | | | | | | | | | | | | |
| FSV-BT | 0.978 | 0.434 | 0.515 | 0.728 | 0.784 | | | | | | | | | | | 18.91 | 6.01 | 7.22 | 2.90 | 2.81 | 0.69 | 2.06 | 2.54 | 0.96 | 0.91 |
| FSV-BU | 1.154 | 0.429 | 0.461 | 0.625 | 0.574 | | | | | | | | | | | 17.64 | 6.54 | 7.11 | 2.58 | 2.50 | 1.33 | 2.16 | 2.24 | 0.78 | 0.80 |
| FSV-BV | 1.351 | 0.493 | 0.520 | 0.681 | 0.517 | | | | | | | | | | | 16.54 | 6.69 | 6.98 | 2.79 | 3.11 | 1.28 | 2.01 | 2.13 | 0.77 | 0.81 |
| FSV-BW | 1.250 | 0.440 | 0.470 | 0.625 | 0.618 | | | | | | 0.064 | 0.065 | 0.073 | 0.021 | 0.020 | 16.66 | 6.58 | 6.98 | 2.78 | 2.75 | 1.19 | 1.94 | 2.04 | 0.78 | 0.75 |
| FSV-BX | 1.190 | 0.424 | 0.457 | 0.627 | 0.608 | | | | | | | | | | | 16.52 | 6.63 | 7.15 | 2.93 | 2.90 | 1.13 | 1.80 | 1.96 | 0.75 | 0.74 |
| FSV-CB | 1.057 | 0.421 | 0.399 | 0.552 | 0.521 | | | | | | | | | | | 14.99 | 5.97 | 6.43 | 2.55 | 2.59 | | | | | |
| FSV-CC | 1.360 | 0.460 | 0.470 | 0.630 | 0.590 | 1.340 | 0.450 | 0.470 | 0.630 | 0.420 | | | | | | 17.42 | 6.65 | 7.23 | 2.83 | 2.68 | | | | | |
| FSV-CD | 1.278 | 0.453 | 0.465 | 0.631 | 0.623 | | | | | | 0.072 | 0.056 | 0.067 | 0.017 | 0.018 | 16.89 | 7.11 | 7.26 | 2.90 | 2.95 | 1.34 | 1.90 | 1.94 | 0.91 | 0.91 |
| FSV-CE | 1.269 | 0.427 | 0.472 | 0.586 | 0.617 | | | | | | | | | | | 17.51 | 7.34 | 8.01 | 2.66 | 2.62 | | | | | |
| FSV-CF | 1.125 | 0.433 | 0.474 | 0.648 | 0.627 | | | | | | | | | | | 17.50 | 7.70 | 8.50 | 3.90 | 3.90 | | | | | |
| FSV-CG | 1.204 | 0.493 | 0.467 | 0.605 | 0.572 | | | | | | | | | | | 14.70 | 6.56 | 6.82 | 2.80 | 2.75 | 1.23 | 1.80 | 1.93 | 0.74 | 0.72 |
| FSV-CI | 1.120 | 0.420 | 0.440 | 0.590 | 0.470 | | | | | | 0.110 | 0.070 | 0.040 | *nq* | *nq* | 18.40 | 7.10 | 7.10 | 3.10 | 2.80 | 1.20 | 1.80 | 1.90 | 0.70 | 0.60 |
| FSV-CP | | | | | | | | | | | | | | | | 14.14 | 6.40 | 6.87 | 3.14 | 3.20 | 1.07 | 1.64 | 1.82 | 0.57 | 0.76 |
| FSV-CS | 1.310 | 0.460 | 0.490 | 0.690 | 0.670 | | | | | | | | | | | 16.08 | 6.79 | 7.08 | 2.98 | 2.89 | 1.41 | 1.95 | 2.04 | 0.94 | 0.92 |
| FSV-CT | | | | | | | | | | | | | | | | | | | | | | | | | |
| FSV-CZ | 1.240 | 0.410 | 0.440 | 0.580 | 0.580 | | | | | | | | | | | 15.60 | 5.50 | 5.90 | 2.30 | 2.40 | | | | | |
| FSV-DA | 1.278 | 0.437 | 0.462 | 0.617 | 0.607 | | | | | | 0.061 | 0.055 | 0.058 | 0.014 | 0.014 | 16.72 | 6.80 | 7.22 | 2.85 | 2.86 | 1.21 | 1.86 | 1.95 | 0.72 | 0.72 |
| FSV-DF | 1.289 | 0.445 | 0.489 | 0.633 | 0.612 | 1.289 | 0.445 | 0.489 | 0.633 | 0.407 | | | | | | | | | | | | | | | |
| FSV-DI | 1.410 | 0.483 | 0.510 | 0.713 | 0.673 | | | | | | 0.056 | 0.057 | 0.055 | 0.023 | 0.023 | 21.59 | 8.19 | 8.34 | 3.26 | 3.21 | 1.37 | 2.22 | 2.28 | 0.79 | 0.78 |
| FSV-ET | 1.240 | 0.430 | 0.430 | 0.560 | 0.560 | | | | | | | | | | | 17.10 | 6.70 | 6.70 | 2.70 | 2.80 | 1.10 | 1.90 | 1.80 | 0.60 | 0.70 |
| N | 34 | 35 | 35 | 35 | 35 | 3 | 3 | 3 | 3 | 3 | 11 | 10 | 10 | 9 | 9 | 34 | 35 | 35 | 35 | 35 | 22 | 22 | 22 | 22 | 22 |
| Min | 0.970 | 0.362 | 0.399 | 0.552 | 0.460 | 1.180 | 0.440 | 0.460 | 0.610 | 0.350 | 0.031 | 0.042 | 0.038 | 0.012 | 0.013 | 12.70 | 5.50 | 5.55 | 2.30 | 2.40 | 0.69 | 1.46 | 1.34 | 0.57 | 0.60 |
| Median | 1.251 | 0.439 | 0.467 | 0.627 | 0.600 | 1.289 | 0.445 | 0.470 | 0.630 | 0.407 | 0.056 | 0.052 | 0.057 | 0.019 | 0.018 | 17.07 | 6.75 | 7.20 | 2.90 | 2.86 | 1.22 | 1.90 | 1.96 | 0.76 | 0.76 |
| Max | 1.410 | 0.493 | 0.540 | 0.728 | 0.784 | 1.340 | 0.450 | 0.489 | 0.633 | 0.420 | 0.110 | 0.070 | 0.073 | 0.031 | 0.033 | 21.59 | 8.19 | 8.50 | 4.00 | 4.20 | 1.45 | 2.33 | 2.54 | 0.96 | 0.92 |
| SD | 0.065 | 0.023 | 0.036 | 0.037 | 0.040 | | | | | | 0.013 | 0.008 | 0.007 | 0.007 | 0.004 | 0.85 | 0.41 | 0.36 | 0.23 | 0.28 | 0.10 | 0.11 | 0.14 | 0.06 | 0.07 |
| CV | 5 | 5 | 8 | 6 | 7 | | | | | | 24 | 15 | 13 | 35 | 21 | 5 | 6 | 5 | 8 | 10 | 8 | 6 | 7 | 8 | 10 |
| $N_{past}$ | 43 | 41 | 38 | 36 | 39 | 11 | 8 | 8 | 5 | 4 | 12 | 13 | 13 | 9 | 9 | 46 | 47 | 44 | 38 | 39 | 24 | 26 | 25 | 22 | 22 |
| $Median_{past}$ | 1.275 | 0.448 | 0.467 | 0.621 | 0.580 | 1.180 | 0.437 | 0.451 | 0.608 | 0.389 | 0.096 | 0.051 | 0.054 | 0.013 | 0.014 | 16.96 | 6.76 | 7.13 | 2.95 | 2.94 | 1.21 | 1.83 | 1.98 | 0.73 | 0.75 |
| $SD_{past}$ | 0.127 | 0.045 | 0.045 | 0.063 | 0.057 | 0.059 | 0.037 | 0.033 | 0.030 | 0.044 | 0.030 | 0.012 | 0.014 | 0.005 | 0.004 | 1.25 | 0.62 | 0.63 | 0.24 | 0.26 | 0.14 | 0.13 | 0.17 | 0.11 | 0.07 |
| NISTa | 1.298 | 0.479 | 0.480 | 0.649 | 0.624 | 1.273 | 0.479 | 0.480 | 0.649 | 0.373 | | | | | | 16.61 | 7.11 | 6.92 | 2.79 | 2.74 | 1.22 | 1.86 | 1.87 | 0.73 | 0.73 |
| NISTb | 1.278 | 0.477 | 0.483 | 0.667 | 0.654 | 1.217 | 0.470 | 0.483 | 0.662 | 0.369 | | | | | | 17.31 | 6.93 | 7.10 | 2.78 | 2.80 | 1.06 | 1.71 | 1.75 | 0.65 | 0.65 |
| $N_{NIST}$ | 4 | 4 | 4 | 4 | 4 | 4 | 4 | 4 | 4 | 4 | | | | | | 4 | 4 | 4 | 4 | 4 | 4 | 4 | 4 | 4 | 4 |
| Mean | 1.288 | 0.478 | 0.482 | 0.659 | 0.639 | 1.245 | 0.474 | 0.481 | 0.657 | 0.371 | | | | | | 16.96 | 7.02 | 7.01 | 2.77 | 2.77 | 1.14 | 1.79 | 1.81 | 0.69 | 0.69 |
| $S_{rep}$ | 0.036 | 0.021 | 0.008 | 0.008 | 0.017 | 0.037 | 0.021 | 0.008 | 0.007 | 0.006 | | | | | | 0.36 | 0.26 | 0.03 | 0.05 | 0.09 | 0.04 | 0.01 | 0.03 | 0.02 | 0.03 |
| $S_{het}$ | 0.032 | 0.026 | 0.006 | 0.007 | 0.019 | 0.039 | 0.026 | 0.006 | 0.008 | 0.009 | | | | | | 0.39 | 0.39 | 0.21 | 0.06 | 0.08 | 0.02 | 0.07 | 0.02 | 0.01 | 0.04 |
| $S_{anl}$ | 0.014 | 0.001 | 0.002 | 0.014 | 0.021 | 0.039 | 0.006 | 0.002 | 0.011 | 0.003 | | | | | | 0.49 | 0.13 | 0.13 | 0.02 | 0.04 | 0.11 | 0.11 | 0.09 | 0.06 | 0.06 |
| $S_{NIST}$ | 0.050 | 0.033 | 0.010 | 0.018 | 0.033 | 0.066 | 0.033 | 0.010 | 0.015 | 0.011 | | | | | | 0.73 | 0.49 | 0.25 | 0.08 | 0.13 | 0.12 | 0.13 | 0.10 | 0.06 | 0.07 |
| NAV | 1.269 | 0.459 | 0.474 | 0.643 | 0.619 | 1.267 | 0.460 | 0.476 | 0.643 | 0.389 | 0.056 | 0.052 | 0.057 | 0.019 | 0.018 | 17.02 | 6.89 | 7.10 | 2.83 | 2.81 | 1.18 | 1.84 | 1.89 | 0.72 | 0.72 |
| NAU | 0.099 | 0.045 | 0.039 | 0.055 | 0.055 | 0.103 | 0.042 | 0.039 | 0.053 | 0.045 | 0.017 | 0.017 | 0.017 | 0.011 | 0.011 | 1.33 | 0.60 | 0.61 | 0.41 | 0.40 | 0.15 | 0.22 | 0.23 | 0.11 | 0.11 |

# Round Robin LIV Laboratory Results

All Values in µg/mL

| | δ-Tocopherol | | | | | Total β-Carotene | | | | | trans-β-Carotene | | | | | Total cis-β-Carotene | | | | | Total α-Carotene | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Lab | 294 | 295 | 296 | 297 | 298 | 294 | 295 | 296 | 297 | 298 | 294 | 295 | 296 | 297 | 298 | 294 | 295 | 296 | 297 | 298 | 294 | 295 | 296 | 297 | 298 |
| FSV-BA | | | | | | 0.054 | 0.326 | 0.350 | 0.048 | 0.047 | 0.051 | 0.310 | 0.334 | 0.047 | 0.047 | 0.003 | 0.017 | 0.016 | 0.001 | 0.001 | 0.025 | 0.024 | 0.026 | 0.003 | 0.003 |
| FSV-BB | | | | | | 0.054 | 0.308 | 0.309 | 0.044 | 0.044 | 0.052 | 0.293 | 0.292 | 0.043 | 0.043 | 0.002 | 0.016 | 0.017 | 0.001 | 0.001 | 0.030 | 0.021 | 0.024 | 0.002 | 0.002 |
| FSV-BD | | | | | | | | | | | | | | | | | | | | | | | | | |
| FSV-BE | | | | | | 0.045 | 0.315 | 0.327 | 0.048 | 0.047 | | | | | | | | | | | | | | | |
| FSV-BF | | | | | | 0.061 | 0.377 | 0.370 | 0.061 | 0.046 | | | | | | | | | | | 0.030 | 0.045 | 0.031 | 0.015 | 0.009 |
| FSV-BG | | | | | | 0.054 | 0.360 | 0.401 | 0.055 | 0.055 | | | | | | | | | | | 0.029 | 0.043 | 0.047 | 0.017 | 0.017 |
| FSV-BH | | | | | | 0.054 | 0.360 | 0.364 | 0.050 | 0.048 | 0.054 | 0.340 | 0.345 | 0.050 | 0.048 | *nq* | 0.020 | 0.018 | *nq* | *nq* | 0.022 | 0.025 | 0.024 | *nq* | *nq* |
| FSV-BI | | | | | | 0.059 | 0.343 | 0.354 | 0.049 | 0.049 | | | | | | | | | | | 0.031 | 0.028 | 0.029 | 0.008 | 0.006 |
| FSV-BJ | | | | | | 0.049 | 0.320 | 0.344 | 0.047 | 0.043 | | | | | | | | | | | 0.030 | 0.030 | *nq* | *nq* | *nq* |
| FSV-BK | | | | | | | | | | | | | | | | | | | | | | | | | |
| FSV-BL | | | | | | | | | | | | | | | | | | | | | | | | | |
| FSV-BM | | | | | | | | | | | | | | | | | | | | | | | | | |
| FSV-BN | 2.522 | 0.112 | 0.124 | 0.053 | 0.064 | 0.059 | 0.308 | 0.288 | 0.053 | 0.049 | 0.055 | 0.282 | 0.266 | 0.048 | 0.047 | *nd* | *nd* | 0.025 | 0.008 | 0.005 | 0.029 | 0.033 | 0.029 | 0.008 | 0.008 |
| FSV-BO | | | | | | 0.052 | 0.374 | 0.398 | 0.056 | 0.065 | | | | | | | | | | | 0.026 | 0.026 | 0.030 | *nq* | *nq* |
| FSV-BP | | | | | | 0.055 | 0.318 | 0.318 | 0.057 | 0.060 | | | | | | | | | | | 0.026 | 0.039 | 0.036 | *nq* | *nq* |
| FSV-BQ | | | | | | | | | | | | | | | | | | | | | | | | | |
| FSV-BR | | | | | | | | | | | | | | | | | | | | | | | | | |
| FSV-BS | | | | | | ≥0.033 | ≥0.288 | ≥0.325 | ≥0.035 | ≥0.033 | 0.033 | 0.288 | 0.325 | 0.035 | 0.033 | | | | | | *nq* | 0.013 | 0.015 | *nq* | *nq* |
| FSV-BT | 1.962 | 0.140 | 0.210 | 0.150 | 0.098 | 0.053 | 0.323 | 0.429 | 0.053 | 0.054 | 0.050 | 0.323 | 0.396 | 0.050 | 0.050 | 0.004 | 0.024 | 0.027 | 0.003 | 0.004 | 0.026 | 0.033 | 0.044 | 0.007 | 0.007 |
| FSV-BU | | | | | | 0.068 | 0.282 | 0.298 | 0.060 | 0.057 | | | | | | | | | | | 0.026 | 0.025 | 0.029 | 0.007 | 0.007 |
| FSV-BV | | | | | | 0.054 | 0.342 | 0.372 | 0.053 | 0.052 | | | | | | | | | | | 0.025 | 0.024 | 0.028 | 0.004 | 0.005 |
| FSV-BW | | | | | | 0.051 | 0.307 | 0.340 | 0.045 | 0.043 | | | | | | | | | | | 0.026 | 0.024 | 0.021 | *nd* | *nd* |
| FSV-BX | | | | | | ≥0.063 | ≥0.286 | ≥0.346 | ≥0.053 | ≥0.051 | 0.063 | 0.286 | 0.346 | 0.053 | 0.051 | | | | | | 0.030 | 0.025 | 0.031 | 0.007 | 0.007 |
| FSV-CB | | | | | | 0.076 | 0.359 | 0.356 | 0.059 | 0.050 | | | | | | | | | | | 0.018 | 0.021 | 0.022 | 0.003 | 0.002 |
| FSV-CC | | | | | | | | | | | | | | | | | | | | | | | | | |
| FSV-CD | | | | | | 0.065 | 0.361 | 0.353 | 0.042 | 0.042 | | | | | | | | | | | 0.036 | 0.039 | 0.039 | *nd* | *nd* |
| FSV-CE | | | | | | 0.052 | 0.337 | 0.383 | 0.049 | 0.057 | | | | | | | | | | | | | | | |
| FSV-CF | | | | | | | | | | | | | | | | | | | | | | | | | |
| FSV-CG | 1.174 | 0.080 | 0.076 | 0.038 | 0.038 | 0.055 | 0.328 | 0.361 | 0.049 | 0.051 | 0.051 | 0.306 | 0.335 | 0.047 | 0.048 | *nq* | 0.022 | 0.025 | *nq* | *nq* | 0.024 | 0.028 | 0.034 | 0.005 | 0.005 |
| FSV-CI | | | | | | ≥0.058 | ≥0.294 | ≥0.340 | ≥0.040 | ≥0.049 | 0.06 | 0.29 | 0.34 | 0.04 | 0.05 | | | | | | 0.033 | 0.028 | 0.032 | 0.008 | 0.008 |
| FSV-CP | | | | | | 0.047 | 0.280 | 0.304 | 0.044 | 0.046 | | | | | | | | | | | 0.022 | 0.025 | 0.027 | *nq* | *nq* |
| FSV-CS | | | | | | 0.050 | 0.330 | 0.350 | 0.050 | 0.050 | 0.046 | 0.302 | 0.322 | 0.047 | 0.045 | 0.01 | 0.03 | 0.03 | 0.00 | 0.00 | 0.026 | 0.030 | 0.032 | 0.005 | 0.004 |
| FSV-CT | | | | | | 0.057 | 0.305 | 0.318 | 0.054 | 0.051 | | | | | | | | | | | | | | | |
| FSV-CZ | | | | | | 0.080 | 0.300 | 0.320 | 0.050 | 0.060 | | | | | | | | | | | | | | | |
| FSV-DA | 3.890 | 0.139 | 0.126 | 0.063 | 0.064 | 0.071 | 0.358 | 0.384 | 0.059 | 0.055 | 0.065 | 0.328 | 0.357 | 0.054 | 0.051 | 0.006 | 0.030 | 0.027 | 0.005 | 0.004 | 0.028 | 0.029 | 0.031 | 0.006 | 0.005 |
| FSV-DF | | | | | | | | | | | | | | | | | | | | | | | | | |
| FSV-DI | 5.480 | 0.211 | 0.200 | 0.077 | 0.078 | ≥0.047 | ≥0.413 | ≥0.408 | ≥0.043 | ≥0.043 | 0.047 | 0.413 | 0.408 | 0.043 | 0.043 | | | | | | | | | | |
| FSV-ET | | | | | | 0.060 | 0.360 | 0.370 | 0.050 | 0.060 | | | | | | | | | | | | | | | |
| N | 5 | 5 | 5 | 5 | 5 | 25 | 25 | 25 | 25 | 25 | 12 | 12 | 12 | 12 | 12 | 5 | 7 | 8 | 6 | 6 | 22 | 23 | 22 | 15 | 15 |
| Min | 1.174 | 0.080 | 0.076 | 0.038 | 0.038 | 0.045 | 0.280 | 0.288 | 0.042 | 0.042 | 0.033 | 0.282 | 0.266 | 0.035 | 0.033 | 0.002 | 0.016 | 0.016 | 0.001 | 0.001 | 0.018 | 0.013 | 0.015 | 0.002 | 0.002 |
| Median | 2.522 | 0.139 | 0.126 | 0.063 | 0.064 | 0.054 | 0.328 | 0.353 | 0.050 | 0.050 | 0.052 | 0.304 | 0.338 | 0.047 | 0.048 | 0.004 | 0.022 | 0.025 | 0.003 | 0.004 | 0.026 | 0.028 | 0.029 | 0.007 | 0.006 |
| Max | 5.480 | 0.211 | 0.210 | 0.150 | 0.098 | 0.080 | 0.377 | 0.429 | 0.061 | 0.065 | 0.065 | 0.413 | 0.408 | 0.054 | 0.051 | 0.006 | 0.031 | 0.032 | 0.008 | 0.005 | 0.036 | 0.045 | 0.047 | 0.017 | 0.017 |
| SD | 1.431 | 0.021 | 0.056 | 0.018 | 0.010 | 0.006 | 0.038 | 0.037 | 0.005 | 0.006 | 0.005 | 0.024 | 0.018 | 0.005 | 0.004 | 0.001 | 0.006 | 0.007 | 0.002 | 0.002 | 0.004 | 0.005 | 0.004 | 0.003 | 0.002 |
| CV | 57 | 15 | 45 | 28 | 16 | 11 | 12 | 11 | 10 | 12 | 9 | 8 | 5 | 11 | 7 | 32 | 30 | 28 | 69 | 44 | 14 | 18 | 15 | 39 | 38 |
| $N_{past}$ | 8 | 6 | 6 | 5 | 5 | 33 | 31 | 31 | 28 | 28 | 13 | 15 | 14 | 12 | 12 | 6 | 10 | 10 | 6 | 6 | 26 | 27 | 25 | 16 | 15 |
| $Median_{past}$ | 3.140 | 0.145 | 0.138 | 0.067 | 0.065 | 0.056 | 0.321 | 0.347 | 0.052 | 0.049 | 0.053 | 0.294 | 0.313 | 0.048 | 0.048 | 0.010 | 0.023 | 0.023 | 0.003 | 0.003 | 0.028 | 0.029 | 0.030 | 0.005 | 0.004 |
| $SD_{past}$ | 0.549 | 0.115 | 0.087 | 0.052 | 0.022 | 0.014 | 0.050 | 0.038 | 0.009 | 0.008 | 0.008 | 0.030 | 0.025 | 0.006 | 0.005 | 0.009 | 0.008 | 0.006 | 0.002 | 0.002 | 0.006 | 0.007 | 0.008 | 0.004 | 0.002 |
| NISTa | | | | | | 0.054 | 0.340 | 0.311 | ≥ 0.044 | ≥ 0.042 | 0.049 | 0.283 | 0.275 | 0.044 | 0.042 | 0.005 | 0.057 | 0.036 | ≤ 0.005 | ≤ 0.005 | | | | | |
| NISTb | 3.114 | 0.103 | 0.109 | 0.033 | 0.037 | 0.058 | 0.339 | 0.355 | 0.045 | 0.048 | 0.054 | 0.315 | 0.330 | 0.045 | 0.046 | 0.008 | 0.024 | 0.025 | *nq* | *nq* | 0.027 | 0.028 | 0.029 | 0.005 | 0.005 |
| $N_{NIST}$ | 2 | 2 | 2 | 2 | 2 | 4 | 4 | 4 | 4 | 4 | 4 | 4 | 4 | 4 | 4 | 4 | 4 | 4 | 1 | 1 | 2 | 2 | 2 | 2 | 2 |
| Mean | 3.114 | 0.103 | 0.109 | 0.034 | 0.037 | 0.056 | 0.339 | 0.333 | 0.045 | 0.045 | 0.051 | 0.299 | 0.303 | 0.045 | 0.044 | 0.007 | 0.040 | 0.030 | | | 0.027 | 0.028 | 0.029 | 0.004 | 0.005 |
| $S_{rep}$ | 0.036 | 0.009 | 0.004 | 0.012 | 0.007 | 0.006 | 0.007 | 0.006 | 0.002 | 0.003 | 0.003 | 0.011 | 0.011 | 0.002 | 0.002 | 0.003 | 0.006 | 0.008 | | | 0.003 | 0.002 | 0.003 | 0.000 | 0.000 |
| $S_{het}$ | 0.043 | 0.005 | 0.007 | 0.003 | 0.002 | 0.002 | 0.019 | 0.008 | 0.003 | 0.002 | 0.001 | 0.004 | 0.012 | 0.003 | 0.001 | 0.002 | 0.016 | 0.006 | | | 0.001 | 0.002 | 0.002 | 0.001 | 0.001 |
| $S_{anl}$ | | | | | | 0.002 | 0.000 | 0.031 | 0.001 | 0.004 | 0.004 | 0.023 | 0.039 | 0.001 | 0.003 | 0.002 | 0.023 | 0.008 | | | | | | | |
| $S_{NIST}$ | 0.056 | 0.010 | 0.008 | 0.012 | 0.007 | 0.007 | 0.020 | 0.032 | 0.003 | 0.006 | 0.005 | 0.026 | 0.042 | 0.003 | 0.004 | 0.003 | 0.029 | 0.012 | | | 0.003 | 0.003 | 0.003 | 0.001 | 0.001 |
| NAV | 2.818 | 0.121 | 0.118 | 0.048 | 0.051 | 0.055 | 0.334 | 0.343 | 0.047 | 0.048 | 0.051 | 0.301 | 0.320 | 0.046 | 0.046 | 0.005 | 0.031 | 0.028 | | | 0.026 | 0.028 | 0.029 | 0.006 | 0.006 |
| NAU | 1.491 | 0.038 | 0.058 | 0.028 | 0.027 | 0.011 | 0.048 | 0.052 | 0.011 | 0.012 | 0.009 | 0.033 | 0.049 | 0.009 | 0.009 | 0.004 | 0.032 | 0.013 | | | 0.009 | 0.009 | 0.010 | 0.004 | 0.003 |

# Round Robin LIV Laboratory Results

All Values in µg/mL

| | Total Lycopene | | | | | trans-Lycopene | | | | | Total β-Cryptoxanthin | | | | | Total α-Cryptoxanthin | | | | | Total Lutein | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Lab | 294 | 295 | 296 | 297 | 298 | 294 | 295 | 296 | 297 | 298 | 294 | 295 | 296 | 297 | 298 | 294 | 295 | 296 | 297 | 298 | 294 | 295 | 296 | 297 | 298 |
| FSV-BA | | | | | | 0.104 | 0.178 | 0.192 | 0.094 | 0.095 | 0.061 | 0.058 | 0.061 | 0.021 | 0.020 | | | | | | | | | | |
| FSV-BB | 0.183 | 0.299 | 0.287 | 0.162 | 0.163 | 0.095 | 0.134 | 0.141 | 0.074 | 0.076 | 0.054 | 0.048 | 0.048 | 0.018 | 0.018 | 0.030 | 0.021 | 0.021 | 0.010 | 0.010 | 0.068 | 0.091 | 0.087 | 0.044 | 0.041 |
| FSV-BD | | | | | | | | | | | | | | | | | | | | | | | | | |
| FSV-BE | | | | | | | | | | | | | | | | | | | | | | | | | |
| FSV-BF | 0.231 | 0.352 | 0.354 | 0.209 | 0.185 | | | | | | 0.043 | 0.039 | 0.042 | 0.016 | 0.013 | | | | | | | | | | |
| FSV-BG | 0.190 | 0.358 | 0.399 | 0.196 | 0.194 | 0.117 | 0.230 | 0.252 | 0.117 | 0.113 | 0.056 | 0.051 | 0.059 | 0.020 | 0.025 | | | | | | 0.054 | 0.094 | 0.104 | 0.051 | 0.048 |
| FSV-BH | 0.194 | 0.344 | 0.349 | 0.187 | 0.181 | | | | | | 0.080 | 0.078 | 0.080 | 0.028 | 0.028 | | | | | | 0.064 | 0.074 | 0.081 | 0.037 | 0.038 |
| FSV-BI | 0.178 | 0.278 | 0.291 | 0.144 | 0.154 | | | | | | 0.063 | 0.056 | 0.058 | 0.020 | 0.020 | | | | | | 0.084 | 0.101 | 0.105 | 0.051 | 0.051 |
| FSV-BJ | 0.165 | 0.294 | 0.313 | 0.163 | 0.158 | | | | | | 0.056 | 0.053 | 0.052 | 0.020 | 0.018 | | | | | | 0.086 | 0.080 | 0.085 | 0.044 | 0.044 |
| FSV-BK | | | | | | | | | | | | | | | | | | | | | | | | | |
| FSV-BL | | | | | | | | | | | | | | | | | | | | | | | | | |
| FSV-BM | | | | | | | | | | | | | | | | | | | | | | | | | |
| FSV-BN | 0.183 | 0.313 | 0.302 | 0.184 | 0.179 | 0.101 | 0.169 | 0.165 | 0.098 | 0.100 | 0.055 | 0.049 | 0.048 | 0.017 | 0.018 | 0.028 | 0.021 | *nd* | 0.009 | 0.009 | 0.070 | 0.085 | 0.092 | 0.055 | 0.053 |
| FSV-BO | 0.258 | 0.425 | 0.426 | 0.251 | 0.223 | | | | | | 0.052 | 0.049 | 0.051 | 0.017 | 0.017 | | | | | | 0.049 | 0.066 | 0.069 | 0.027 | 0.036 |
| FSV-BP | 0.232 | 0.386 | 0.347 | 0.254 | 0.250 | | | | | | 0.046 | 0.058 | 0.061 | 0.019 | 0.019 | | | | | | | | | | |
| FSV-BQ | | | | | | | | | | | | | | | | | | | | | | | | | |
| FSV-BR | | | | | | | | | | | | | | | | | | | | | | | | | |
| FSV-BS | 0.202 | 0.330 | 0.366 | 0.198 | 0.195 | | | | | | 0.041 | 0.045 | 0.051 | 0.017 | 0.015 | | | | | | | | | | |
| FSV-BT | 0.242 | 0.383 | 0.490 | 0.259 | 0.253 | 0.094 | 0.151 | 0.193 | 0.099 | 0.096 | 0.054 | 0.046 | 0.063 | 0.025 | 0.023 | 0.038 | 0.038 | 0.052 | 0.023 | 0.023 | 0.077 | 0.076 | 0.115 | 0.065 | 0.072 |
| FSV-BU | 0.180 | 0.284 | 0.305 | 0.164 | 0.163 | | | | | | 0.060 | 0.052 | 0.054 | 0.026 | 0.026 | | | | | | | | | | |
| FSV-BV | 0.234 | 0.419 | 0.455 | 0.223 | 0.221 | | | | | | 0.037 | 0.034 | 0.037 | 0.011 | 0.011 | | | | | | | | | | |
| FSV-BW | 0.178 | 0.321 | 0.347 | 0.172 | 0.172 | | | | | | 0.050 | 0.042 | 0.043 | 0.010 | 0.013 | | | | | | | | | | |
| FSV-BX | | | | | | 0.092 | 0.129 | 0.169 | 0.086 | 0.082 | 0.048 | 0.046 | 0.051 | 0.017 | 0.018 | | | | | | 0.072 | 0.086 | 0.094 | 0.045 | 0.046 |
| FSV-CB | 0.161 | 0.262 | 0.279 | 0.152 | 0.149 | | | | | | 0.016 | 0.055 | 0.062 | 0.023 | 0.024 | | | | | | 0.060 | 0.063 | 0.070 | - | - |
| FSV-CC | | | | | | | | | | | | | | | | | | | | | | | | | |
| FSV-CD | 0.192 | 0.423 | 0.421 | 0.156 | 0.169 | | | | | | 0.051 | 0.062 | 0.064 | 0.031 | 0.033 | | | | | | | | | | |
| FSV-CE | | | | | | | | | | | | | | | | | | | | | | | | | |
| FSV-CF | | | | | | | | | | | | | | | | | | | | | | | | | |
| FSV-CG | 0.196 | 0.344 | 0.380 | 0.193 | 0.197 | 0.100 | 0.182 | 0.202 | 0.101 | 0.102 | 0.054 | 0.053 | 0.056 | 0.021 | 0.020 | | | | | | | | | | |
| FSV-CI | | | | | | | | | | | | | | | | | | | | | 0.057 | 0.065 | 0.074 | 0.033 | 0.038 |
| FSV-CP | 0.119 | 0.193 | 0.210 | 0.108 | 0.112 | | | | | | 0.040 | 0.037 | 0.040 | 0.012 | 0.012 | | | | | | | | | | |
| FSV-CS | 0.190 | 0.360 | 0.380 | 0.210 | 0.200 | | | | | | 0.047 | 0.051 | 0.054 | 0.019 | 0.018 | | | | | | 0.059 | 0.082 | 0.087 | 0.047 | 0.045 |
| FSV-CT | 0.277 | 0.392 | 0.420 | 0.241 | 0.228 | | | | | | 0.045 | 0.037 | 0.039 | 0.014 | 0.013 | | | | | | 0.068 | 0.101 | 0.107 | 0.039 | 0.039 |
| FSV-CZ | | | | | | | | | | | | | | | | | | | | | | | | | |
| FSV-DA | 0.215 | 0.366 | 0.395 | 0.197 | 0.203 | 0.113 | 0.189 | 0.207 | 0.102 | 0.104 | 0.066 | 0.062 | 0.066 | 0.021 | 0.023 | 0.030 | 0.023 | 0.025 | 0.011 | 0.011 | 0.060 | 0.075 | 0.079 | 0.040 | 0.041 |
| FSV-DF | | | | | | | | | | | | | | | | | | | | | | | | | |
| FSV-DI | | | | | | 0.156 | 0.282 | 0.284 | 0.148 | 0.143 | | | | | | | | | | | 0.090 | 0.096 | 0.091 | 0.047 | 0.053 |
| FSV-ET | | | | | | | | | | | | | | | | | | | | | | | | | |
| N | 21 | 21 | 21 | 21 | 21 | 9 | 9 | 9 | 9 | 9 | 23 | 23 | 23 | 23 | 23 | 4 | 4 | 3 | 4 | 4 | 15 | 15 | 15 | 14 | 14 |
| Min | 0.119 | 0.193 | 0.210 | 0.108 | 0.112 | 0.092 | 0.129 | 0.141 | 0.074 | 0.076 | 0.016 | 0.034 | 0.037 | 0.010 | 0.011 | 0.028 | 0.021 | 0.021 | 0.009 | 0.009 | 0.049 | 0.063 | 0.069 | 0.027 | 0.036 |
| Median | 0.192 | 0.344 | 0.354 | 0.193 | 0.185 | 0.101 | 0.178 | 0.193 | 0.099 | 0.100 | 0.052 | 0.051 | 0.054 | 0.019 | 0.018 | 0.030 | 0.022 | 0.025 | 0.010 | 0.011 | 0.068 | 0.082 | 0.087 | 0.045 | 0.045 |
| Max | 0.277 | 0.425 | 0.490 | 0.259 | 0.253 | 0.156 | 0.282 | 0.284 | 0.148 | 0.143 | 0.080 | 0.078 | 0.080 | 0.031 | 0.033 | 0.038 | 0.038 | 0.052 | 0.023 | 0.023 | 0.090 | 0.101 | 0.115 | 0.065 | 0.072 |
| SD | 0.038 | 0.062 | 0.070 | 0.035 | 0.029 | 0.013 | 0.028 | 0.029 | 0.006 | 0.007 | 0.008 | 0.007 | 0.010 | 0.003 | 0.005 | 0.002 | 0.004 | | 0.003 | 0.003 | 0.011 | 0.013 | 0.014 | 0.008 | 0.008 |
| CV | 20 | 18 | 20 | 18 | 16 | 13 | 16 | 15 | 6 | 7 | 15 | 15 | 18 | 16 | 28 | 7 | 20 | | 31 | 30 | 17 | 16 | 16 | 18 | 18 |
| $N_{past}$ | 28 | 26 | 25 | 21 | 21 | 9 | 12 | 12 | 11 | 11 | 24 | 26 | 25 | 23 | 23 | 5 | 5 | 5 | 5 | 4 | 12 | 16 | 16 | 18 | 18 |
| $Median_{past}$ | 0.202 | 0.320 | 0.346 | 0.191 | 0.196 | 0.110 | 0.166 | 0.184 | 0.097 | 0.096 | 0.058 | 0.051 | 0.054 | 0.019 | 0.018 | 0.033 | 0.024 | 0.025 | 0.011 | 0.010 | 0.071 | 0.085 | 0.090 | 0.046 | 0.044 |
| $SD_{past}$ | 0.046 | 0.050 | 0.046 | 0.026 | 0.029 | 0.020 | 0.035 | 0.043 | 0.014 | 0.010 | 0.013 | 0.010 | 0.008 | 0.004 | 0.003 | 0.008 | 0.004 | 0.005 | 0.002 | 0.003 | 0.017 | 0.013 | 0.019 | 0.015 | 0.016 |
| NISTa | | | | | | | | | | | 0.056 | 0.059 | 0.061 | *nq* | *nq* | | | | | | 0.086 | 0.086 | 0.084 | 0.042 | 0.043 |
| NISTb | 0.230 | 0.386 | 0.396 | 0.200 | 0.206 | | | | | | 0.053 | 0.057 | 0.056 | 0.019 | 0.015 | 0.040 | 0.035 | 0.036 | 0.014 | 0.014 | | | | | |
| $N_{NIST}$ | 2 | 2 | 2 | 2 | 2 | | | | | | 4 | 4 | 4 | 2 | 2 | 2 | 2 | 2 | 2 | 2 | 2 | 2 | 2 | 2 | 2 |
| Mean | 0.230 | 0.386 | 0.396 | 0.206 | 0.206 | | | | | | 0.054 | 0.058 | 0.059 | 0.020 | 0.015 | 0.040 | 0.035 | 0.036 | 0.014 | 0.014 | 0.086 | 0.086 | 0.084 | 0.042 | 0.043 |
| $S_{rep}$ | 0.006 | 0.019 | 0.007 | 0.010 | 0.008 | | | | | | 0.004 | 0.003 | 0.004 | 0.000 | 0.010 | 0.001 | 0.001 | 0.001 | 0.001 | 0.003 | 0.002 | 0.004 | 0.004 | | 0.004 |
| $S_{het}$ | 0.020 | 0.009 | 0.013 | 0.024 | 0.024 | | | | | | 0.004 | 0.003 | 0.004 | 0.002 | 0.006 | 0.004 | 0.001 | 0.000 | 0.001 | 0.001 | 0.002 | 0.002 | 0.001 | 0.000 | 0.001 |
| $S_{anl}$ | | | | | | | | | | | 0.002 | 0.001 | 0.003 | | | | | | | | | | | | |
| $S_{NIST}$ | 0.021 | 0.021 | 0.015 | 0.026 | 0.026 | | | | | | 0.006 | 0.005 | 0.006 | 0.002 | 0.012 | 0.004 | 0.001 | 0.001 | 0.001 | 0.003 | 0.003 | 0.004 | 0.004 | | 0.005 |
| NAV | 0.211 | 0.365 | 0.375 | 0.199 | 0.196 | 0.101 | 0.178 | 0.193 | 0.099 | 0.100 | 0.053 | 0.054 | 0.056 | 0.019 | 0.016 | 0.035 | 0.029 | | 0.012 | 0.012 | 0.077 | 0.084 | 0.086 | 0.043 | 0.044 |
| NAU | 0.055 | 0.082 | 0.083 | 0.049 | 0.049 | 0.018 | 0.031 | 0.034 | 0.017 | 0.017 | 0.013 | 0.013 | 0.013 | 0.005 | 0.012 | 0.008 | 0.010 | | 0.004 | 0.004 | 0.019 | 0.016 | 0.017 | 0.010 | 0.010 |

# Round Robin LIV Laboratory Results

All Values in µg/mL

| | Total Zeaxanthin | | | | | Total Lutein&Zeaxanthin | | | | | Coenzyme Q10 | | | | | Phylloquinone (K1) x1000 | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Lab | 294 | 295 | 296 | 297 | 298 | 294 | 295 | 296 | 297 | 596 | 294 | 295 | 296 | 297 | 298 | 294 | 295 | 296 | 297 | 298 |
| FSV-BA | | | | | | 0.100 | 0.115 | 0.125 | 0.069 | 0.068 | | | | | | | | | | |
| FSV-BB | 0.042 | 0.041 | 0.043 | 0.027 | 0.026 | *0.111* | *0.131* | *0.131* | *0.070* | *0.067* | | | | | | | | | | |
| FSV-BD | | | | | | | | | | | | | | | | | | | | |
| FSV-BE | | | | | | | | | | | | | | | | 0.15 | 0.41 | 0.49 | <0.05 | 0.19 |
| FSV-BF | | | | | | 0.106 | 0.110 | 0.114 | 0.060 | 0.061 | | | | | | | | | | |
| FSV-BG | 0.018 | 0.024 | 0.023 | 0.013 | 0.011 | 0.064 | 0.108 | 0.116 | 0.060 | 0.054 | | | | | | | | | | |
| FSV-BH | 0.051 | 0.032 | 0.035 | 0.017 | 0.017 | 0.115 | 0.106 | 0.116 | 0.054 | 0.055 | | | | | | | | | | |
| FSV-BI | 0.037 | 0.032 | 0.031 | 0.017 | 0.017 | *0.121* | *0.133* | *0.136* | *0.068* | *0.068* | | | | | | | | | | |
| FSV-BJ | | | | | | | | | | | 1.051 | 0.672 | 0.645 | 0.304 | 0.337 | | | | | |
| FSV-BK | | | | | | | | | | | | | | | | | | | | |
| FSV-BL | | | | | | | | | | | | | | | | | | | | |
| FSV-BM | | | | | | | | | | | | | | | | | | | | |
| FSV-BN | 0.036 | 0.016 | 0.009 | 0.011 | 0.012 | 0.099 | 0.102 | 0.105 | 0.068 | 0.067 | | | | | | | | | | |
| FSV-BO | 0.046 | 0.063 | 0.046 | 0.023 | 0.017 | 0.0950 | 0.1286 | 0.1149 | 0.0506 | 0.0535 | | | | | | | | | | |
| FSV-BP | | | | | | 0.110 | 0.114 | 0.120 | 0.057 | 0.059 | | | | | | | | | | |
| FSV-BQ | | | | | | | | | | | | | | | | | | | | |
| FSV-BR | | | | | | | | | | | | | | | | | | | | |
| FSV-BS | | | | | | 0.089 | 0.100 | 0.120 | 0.076 | 0.074 | | | | | | | | | | |
| FSV-BT | 0.024 | 0.018 | 0.033 | 0.021 | 0.021 | 0.103 | 0.095 | 0.150 | 0.088 | 0.094 | | | | | | | | | | |
| FSV-BU | | | | | | 0.103 | 0.103 | 0.019 | 0.058 | 0.056 | | | | | | | | | | |
| FSV-BV | | | | | | 0.112 | 0.114 | 0.124 | 0.065 | 0.066 | | | | | | | | | | |
| FSV-BW | | | | | | 0.087 | 0.093 | 0.094 | 0.042 | 0.054 | 1.020 | 0.750 | 0.830 | 0.450 | 0.440 | | | | | |
| FSV-BX | 0.035 | 0.032 | 0.027 | 0.017 | 0.017 | 0.107 | 0.119 | 0.122 | 0.063 | 0.063 | | | | | | | | | | |
| FSV-CB | 0.029 | 0.019 | 0.023 | - | - | *0.088* | *0.083* | *0.092* | 0.049 | 0.050 | | | | | | | | | | |
| FSV-CC | | | | | | | | | | | | | | | | | | | | |
| FSV-CD | | | | | | 0.099 | 0.107 | 0.104 | 0.056 | 0.057 | | | | | | | | | | |
| FSV-CE | | | | | | | | | | | | | | | | | | | | |
| FSV-CF | | | | | | | | | | | | | | | | | | | | |
| FSV-CG | | | | | | 0.114 | 0.118 | 0.126 | 0.068 | 0.067 | | | | | | | | | | |
| FSV-CI | 0.035 | 0.025 | 0.028 | 0.015 | 0.017 | *0.092* | *0.090* | *0.102* | *0.048* | *0.055* | | | | | | 0.16 | 0.51 | 0.49 | 0.18 | 0.18 |
| FSV-CP | | | | | | 0.136 | 0.142 | 0.155 | 0.081 | 0.084 | | | | | | | | | | |
| FSV-CS | 0.037 | 0.029 | 0.032 | 0.018 | 0.017 | *0.096* | *0.111* | *0.119* | *0.065* | *0.062* | | | | | | | | | | |
| FSV-CT | 0.038 | 0.024 | 0.025 | 0.011 | *nq* | 0.106 | 0.125 | 0.132 | 0.050 | 0.039 | | | | | | | | | | |
| FSV-CZ | | | | | | | | | | | 1.200 | 0.860 | 1.000 | 0.490 | 0.510 | | | | | |
| FSV-DA | 0.034 | 0.026 | 0.029 | 0.017 | 0.017 | 0.108 | 0.114 | 0.121 | 0.063 | 0.064 | | | | | | | | | | |
| FSV-DF | | | | | | | | | | | | | | | | | | | | |
| FSV-DI | | | | | | | | | | | 1.120 | 0.953 | 0.940 | 0.517 | 0.514 | 0.10 | 0.39 | 0.41 | 0.14 | 0.14 |
| FSV-ET | | | | | | | | | | | | | | | | | | | | |
| N | 13 | 13 | 13 | 12 | 11 | 23 | 23 | 23 | 23 | 23 | 4 | 4 | 4 | 4 | 4 | 3 | 3 | 3 | 2 | 3 |
| Min | 0.018 | 0.016 | 0.009 | 0.011 | 0.011 | 0.064 | 0.083 | 0.019 | 0.042 | 0.039 | 1.020 | 0.672 | 0.645 | 0.304 | 0.337 | 0.10 | 0.39 | 0.41 | 0.14 | 0.14 |
| Median | 0.036 | 0.026 | 0.029 | 0.017 | 0.017 | 0.103 | 0.111 | 0.120 | 0.063 | 0.062 | 1.086 | 0.805 | 0.885 | 0.470 | 0.475 | 0.15 | 0.41 | 0.49 | 0.16 | 0.18 |
| Max | 0.051 | 0.063 | 0.046 | 0.027 | 0.026 | 0.136 | 0.142 | 0.155 | 0.088 | 0.094 | 1.200 | 0.953 | 1.000 | 0.517 | 0.514 | 0.16 | 0.51 | 0.49 | 0.18 | 0.19 |
| SD | 0.003 | 0.006 | 0.006 | 0.003 | 0.001 | 0.011 | 0.012 | 0.012 | 0.010 | 0.009 | 0.072 | 0.113 | 0.127 | 0.062 | 0.072 | | | | | |
| CV | 8 | 23 | 20 | 19 | 5 | 11 | 11 | 10 | 15 | 15 | 7 | 14 | 14 | 13 | 15 | | | | | |
| $N_{past}$ | 11 | 15 | 15 | 15 | 15 | 22 | 26 | 23 | 21 | 21 | 5 | 8 | 6 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| $Median_{past}$ | 0.034 | 0.025 | 0.026 | 0.019 | 0.020 | 0.109 | 0.115 | 0.120 | 0.067 | 0.064 | 1.000 | 0.844 | 0.898 | | | | | | | |
| $SD_{past}$ | 0.006 | 0.007 | 0.006 | 0.004 | 0.006 | 0.022 | 0.016 | 0.022 | 0.019 | 0.016 | 0.161 | 0.256 | 0.213 | | | | | | | |
| NISTa | 0.034 | *nq* | *nq* | *nq* | *nq* | 0.120 | ≥0.086 | ≥0.086 | ≥0.084 | ≥0.042 | | | | | | | | | | |
| NISTb | 0.037 | 0.034 | 0.038 | 0.021 | 0.022 | 0.107 | 0.118 | 0.122 | 0.063 | 0.064 | | | | | | | | | | |
| $N_{NIST}$ | 4 | 2 | 2 | 2 | 2 | 4 | 2 | 2 | 2 | 2 | | | | | | | | | | |
| Mean | 0.036 | 0.034 | 0.038 | 0.021 | 0.022 | 0.114 | 0.118 | 0.122 | 0.047 | 0.048 | | | | | | | | | | |
| $S_{rep}$ | 0.002 | 0.003 | 0.002 | 0.002 | 0.001 | 0.007 | 0.004 | 0.006 | 0.031 | 0.035 | | | | | | | | | | |
| $S_{het}$ | 0.002 | 0.000 | 0.001 | 0.001 | 0.004 | 0.000 | 0.003 | 0.001 | 0.023 | 0.018 | | | | | | | | | | |
| $S_{anl}$ | 0.002 | | | | | 0.009 | | | | | | | | | | | | | | |
| $S_{NIST}$ | 0.004 | 0.003 | 0.002 | 0.003 | 0.004 | 0.011 | 0.004 | 0.006 | 0.039 | 0.040 | | | | | | | | | | |
| NAV | 0.036 | 0.030 | 0.033 | 0.019 | 0.020 | 0.108 | 0.115 | 0.121 | 0.055 | 0.055 | | | | | | | | | | |
| NAU | 0.010 | 0.010 | 0.010 | 0.006 | 0.007 | 0.023 | 0.024 | 0.025 | 0.040 | 0.041 | | | | | | | | | | |

# Round Robin LIV Laboratory Results

All Values in µg/mL

## Analytes Reported By One Laboratory

| Analyte | Code | 294 | 295 | 296 | 297 | 298 |
|---|---|---|---|---|---|---|
| Total cis-b-Cryptoxanthin | FSV-BT | 0.03 | 0.02 | 0.03 | 0.02 | 0.160 |
| Ubiquinol | FSV-BW | 0.630 | 0.640 | 0.520 | 0.290 | 0.280 |
| Ubiquinone | FSV-BW | 0.390 | 0.110 | 0.310 | 0.160 | 0.01 |
| 25-hydroxyvitamin D | FSV-CF | 0.0220 | <0.007 | <0.007 | 0.0100 | 0.042 |
| Phytoene | FSV-DA | 0.013 | 0.024 | 0.032 | 0.011 | 0.018 |
| Retinyl stearate | FSV-DA | 0.005 | 0.017 | 0.018 | 0.007 | 0.005 |

## Legend

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

## Comparability Summary

| Lab | TR | aT | g/bT | bC | tbC | aC | TLy | tLy | TbX | TLu | TZ | L&Z |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| FSV-BA | 1 | 1 | 1 | 1 | 1 | 1 | | 1 | 1 | 1 | 1 | 1 |
| FSV-BB | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 2 | 1 | 1 | 2 | 1 |
| FSV-BD | 1 | 2 | | | | | | | | | | |
| FSV-BE | 2 | 1 | 1 | 1 | | | | | | | | 1 |
| FSV-BF | 2 | 2 | 1 | 1 | | 2 | 1 | | 1 | | | |
| FSV-BG | 1 | 1 | 1 | 1 | | 3 | 1 | 2 | 1 | | | 1 |
| FSV-BH | 2 | 1 | 2 | 1 | 1 | 1 | 1 | | 2 | 1 | 2 | 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 | 2 | 2 | | | | | | | | | | |
| FSV-BL | 1 | 2 | | | | | | | | 1 | | |
| FSV-BM | 1 | 3 | | | | | | | | | | |
| FSV-BN | 1 | 2 | 3 | 1 | 1 | 1 | 1 | 1 | 1 | | | 2 |
| FSV-BO | 1 | 1 | | 1 | | 1 | 2 | | 1 | 1 | 1 | 1 |
| FSV-BP | 1 | 1 | | 1 | | 1 | 1 | | 1 | | | 1 |
| FSV-BQ | 1 | 1 | | | | | | | | 1 | 2 | 1 |
| FSV-BR | | 1 | | | | | | | | | | 1 |
| FSV-BS | 2 | | | | 2 | 2 | 1 | | 1 | 1 | 1 | 1 |
| FSV-BT | 3 | 1 | 3 | 1 | 1 | 1 | 2 | 1 | 1 | 2 | 3 | 1 |
| FSV-BU | 1 | 1 | 1 | 2 | | 1 | 1 | | 1 | 2 | 1 | 1 |
| FSV-BV | 2 | 1 | 1 | 1 | | 1 | 1 | | 2 | | | |
| FSV-BW | 1 | 1 | 1 | 1 | | 1 | 1 | | 1 | | | |
| FSV-BX | 1 | 1 | 1 | | 1 | 1 | | 1 | 1 | | | 1 |
| FSV-CB | 2 | 2 | | 2 | | 1 | 1 | | 2 | | | |
| FSV-CC | 1 | 1 | | | | | | | | | | 1 |
| FSV-CD | 1 | 1 | 2 | 1 | | 2 | 1 | | 2 | | | 2 |
| FSV-CE | 1 | 1 | | 1 | | | | | | 1 | 1 | 1 |
| FSV-CF | 1 | 3 | | | | | | | | | | 1 |
| FSV-CG | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | | |
| FSV-CI | 2 | 1 | 1 | | 1 | 1 | | | | | | 1 |
| FSV-CP | | 2 | 2 | 1 | | 1 | 2 | | 2 | | | |
| FSV-CS | 1 | 1 | 2 | 1 | 1 | 1 | 1 | | 1 | 1 | 1 | 1 |
| FSV-CT | | | | 1 | | | 2 | | 1 | 1 | 1 | 1 |
| FSV-CZ | 1 | 2 | | 2 | | | | | | | | |
| FSV-DA | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | | |
| FSV-DF | 1 | | | | | | | | | 1 | | |
| FSV-DI | 2 | 3 | 2 | | 2 | | | 3 | | | | |
| FSV-ET | 1 | 1 | 1 | 1 | | | | | | | | |
| NISTa | 1 | 1 | 1 | 1 | 1 | | | | 1 | 1 | | |
| NISTb | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | 1 | | 1 | 1 |
| n | 37 | 37 | 24 | 27 | 14 | 24 | 22 | 9 | 25 | 16 | 14 | 24 |

| | TR | aT | g/bT | bC | tbC | aC | TLy | tLy | TbX | TLu | TZ | L&Z |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| % 1 | 73 | 70 | 71 | 89 | 86 | 83 | 82 | 67 | 80 | 88 | 71 | 92 |
| % 2 | 24 | 22 | 21 | 11 | 14 | 13 | 18 | 22 | 20 | 13 | 21 | 8 |
| % 3 | 3 | 8 | 8 | 0 | 0 | 4 | 0 | 11 | 0 | 0 | 7 | 0 |
| % 4 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |

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

### "Comparability Score"

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

We define CS as follows:

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

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

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

NAU = NIST Assigned Uncertainty

For further details, please see

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

## Appendix D. Representative "Individualized Report" for RR54

Each participant in RR54 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 RR54:

- Total 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 Lutein
- Total Zeaxanthin
- Total Lutein & Zeaxanthin

The following 12 pages are the "Individualized Report" for the analytes evaluated by participant FSV-BA.

# Individualized Round Robin LIV Report: FSV-BA

## Summary

| Analyte | Serum 294 | | | Serum 295 | | | Serum 296 | | | Serum 297 | | | Serum 298 | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | You | NAV | n | You | NAV | n | You | NAV | n | You | NAV | n | You | NAV | n |
| Total Retinol | 1.263 | 1.269 | 34 | 0.454 | 0.459 | 35 | 0.500 | 0.474 | 35 | 0.671 | 0.643 | 35 | 0.582 | 0.619 | 35 |
| Retinyl Palmitate | 0.05 | 0.06 | 11 | 0.0 | 0.1 | 10 | 0.1 | 0.1 | 10 | 0.02 | 0.02 | 9 | 0.02 | 0.02 | 9 |
| α-Tocopherol | 17.21 | 17.02 | 34 | 6.76 | 6.89 | 35 | 7.27 | 7.10 | 35 | 2.84 | 2.83 | 35 | 2.83 | 2.81 | 35 |
| γ/β-Tocopherol | 1.225 | 1.181 | 22 | 1.903 | 1.844 | 22 | 2.074 | 1.887 | 22 | 0.745 | 0.724 | 22 | 0.758 | 0.724 | 22 |
| Total β-Carotene | 0.054 | 0.055 | 25 | 0.326 | 0.334 | 25 | 0.350 | 0.343 | 25 | 0.048 | 0.047 | 25 | 0.047 | 0.048 | 25 |
| trans-β-Carotene | 0.051 | 0.051 | 12 | 0.310 | 0.301 | 12 | 0.334 | 0.320 | 12 | 0.047 | 0.046 | 12 | 0.047 | 0.046 | 12 |
| Total cis-β-Carotene | 0.003 | 0.005 | 5 | 0.017 | 0.031 | 7 | 0.016 | 0.028 | 8 | 0.001 | | 6 | 0.001 | | 6 |
| Total α-Carotene | 0.025 | 0.026 | 22 | 0.024 | 0.028 | 23 | 0.026 | 0.029 | 22 | 0.003 | 0.006 | 15 | 0.003 | 0.006 | 15 |
| trans-Lycopene | 0.104 | 0.101 | 9 | 0.178 | 0.178 | 9 | 0.192 | 0.193 | 9 | 0.094 | 0.099 | 9 | 0.095 | 0.100 | 9 |
| Total β-Cryptoxanthin | 0.061 | 0.053 | 23 | 0.058 | 0.054 | 23 | 0.061 | 0.056 | 23 | 0.021 | 0.019 | 23 | 0.020 | 0.016 | 23 |
| Total Lutein&Zeaxanthin | 0.100 | 0.108 | 23 | 0.115 | 0.115 | 23 | 0.125 | 0.121 | 23 | 0.069 | 0.055 | 23 | 0.068 | 0.055 | 23 |

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

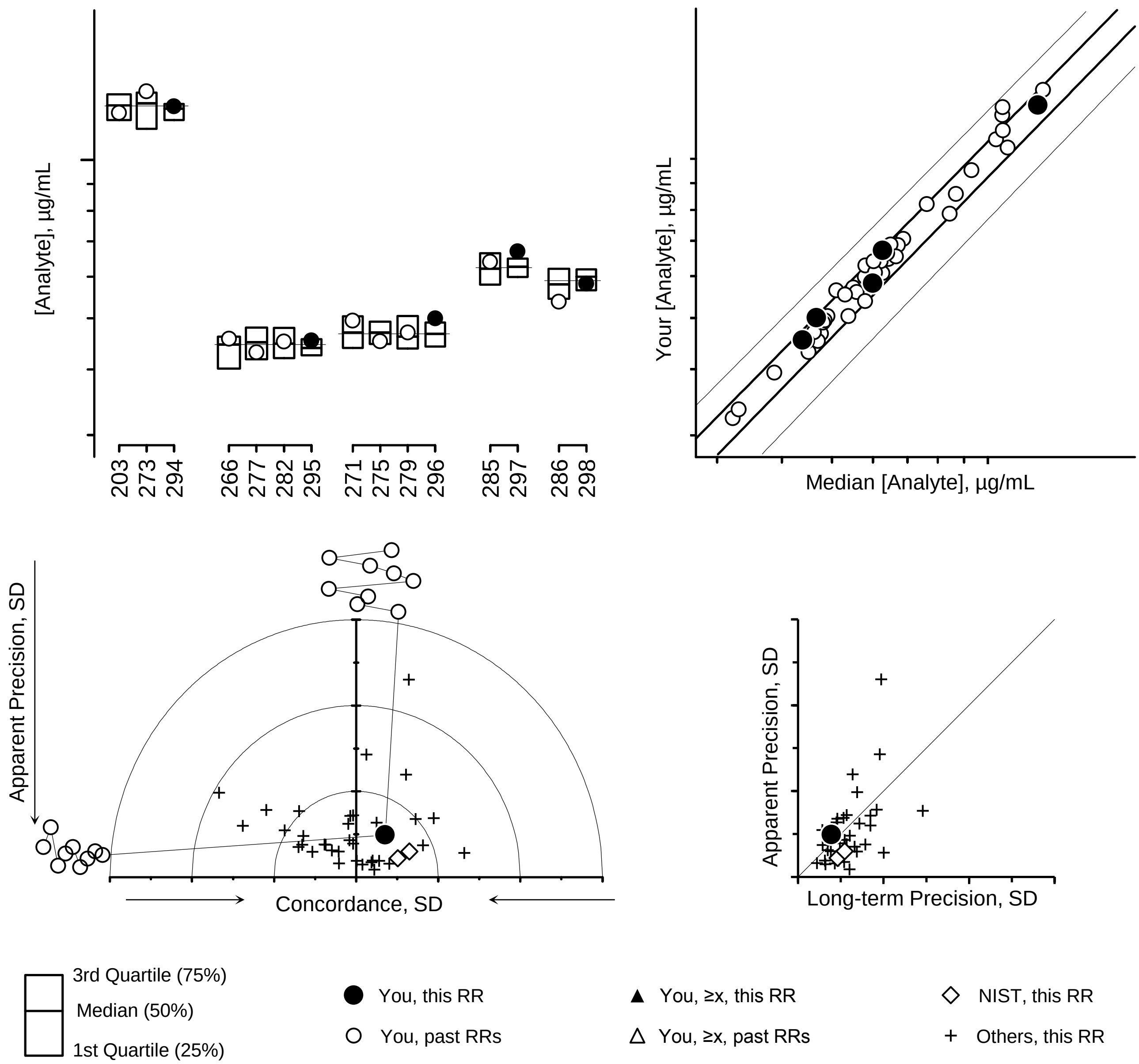
Please check our records against your records. Send corrections and/or updates to...

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 LIV Report: FSV-BA

## Total Retinol

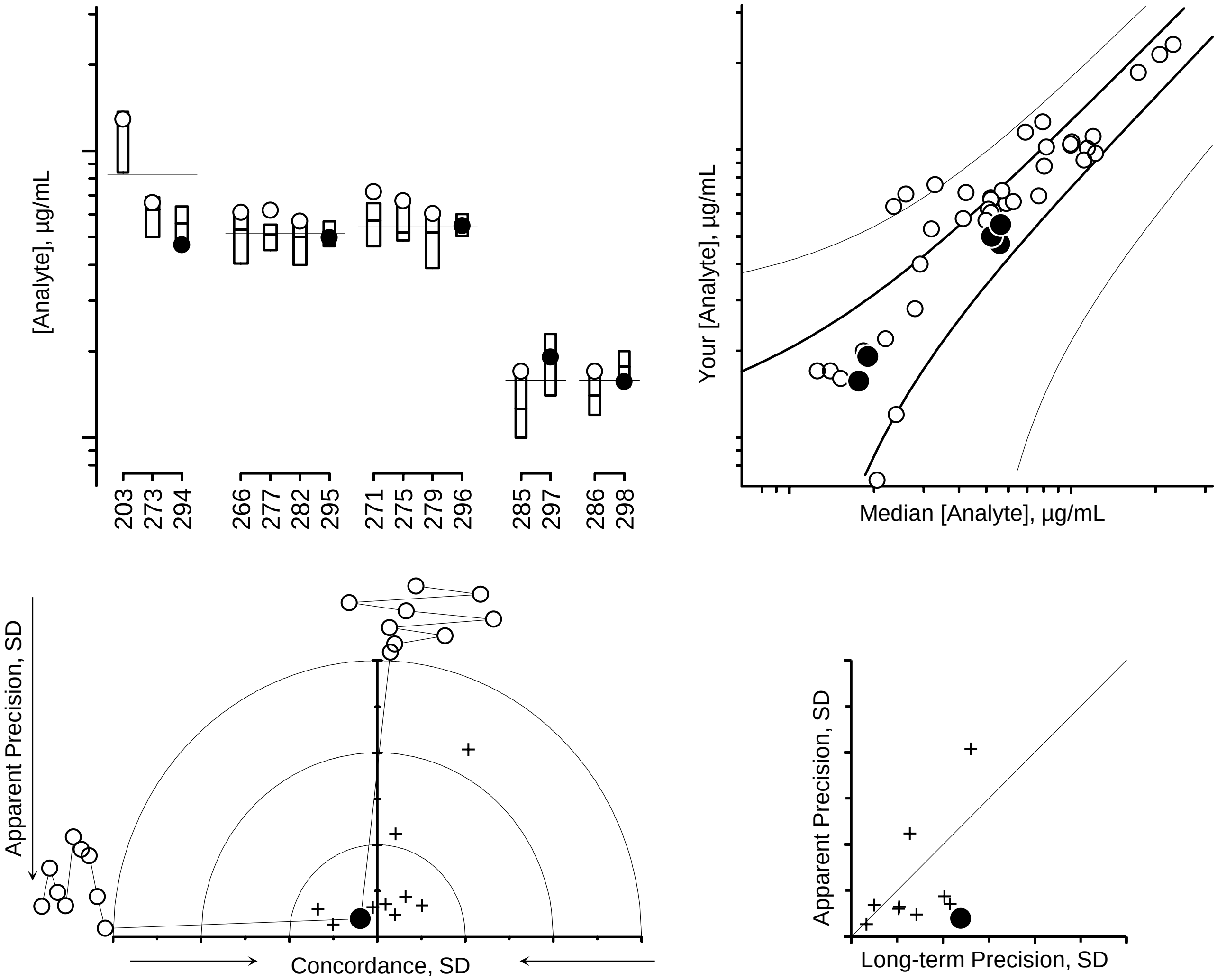



For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

| Serum | History | Comments |
|---|---|---|
| #294 | Lyophilized: #203 RR33, #273 RR49 | Augmented with R, RP, αT, γT, and δT |
| #295 | Lyophilized: #266 RR48, #277 RR50, #282 RR51 | Same pool as #296, native |
| #296 | Fresh-frozen: #271 RR49, #275 RR50, #279 RR51 | Same pool as #295, native |
| #297 | Fresh-frozen: #285 RR52 | Same pool as #298, trans-Retinol augmented |
| #298 | Fresh-frozen: #286 RR52 | Same pool as #297, cis-Retinol augmented |

# Individualized RR LIV Report: FSV-BA

## Retinyl Palmitate




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

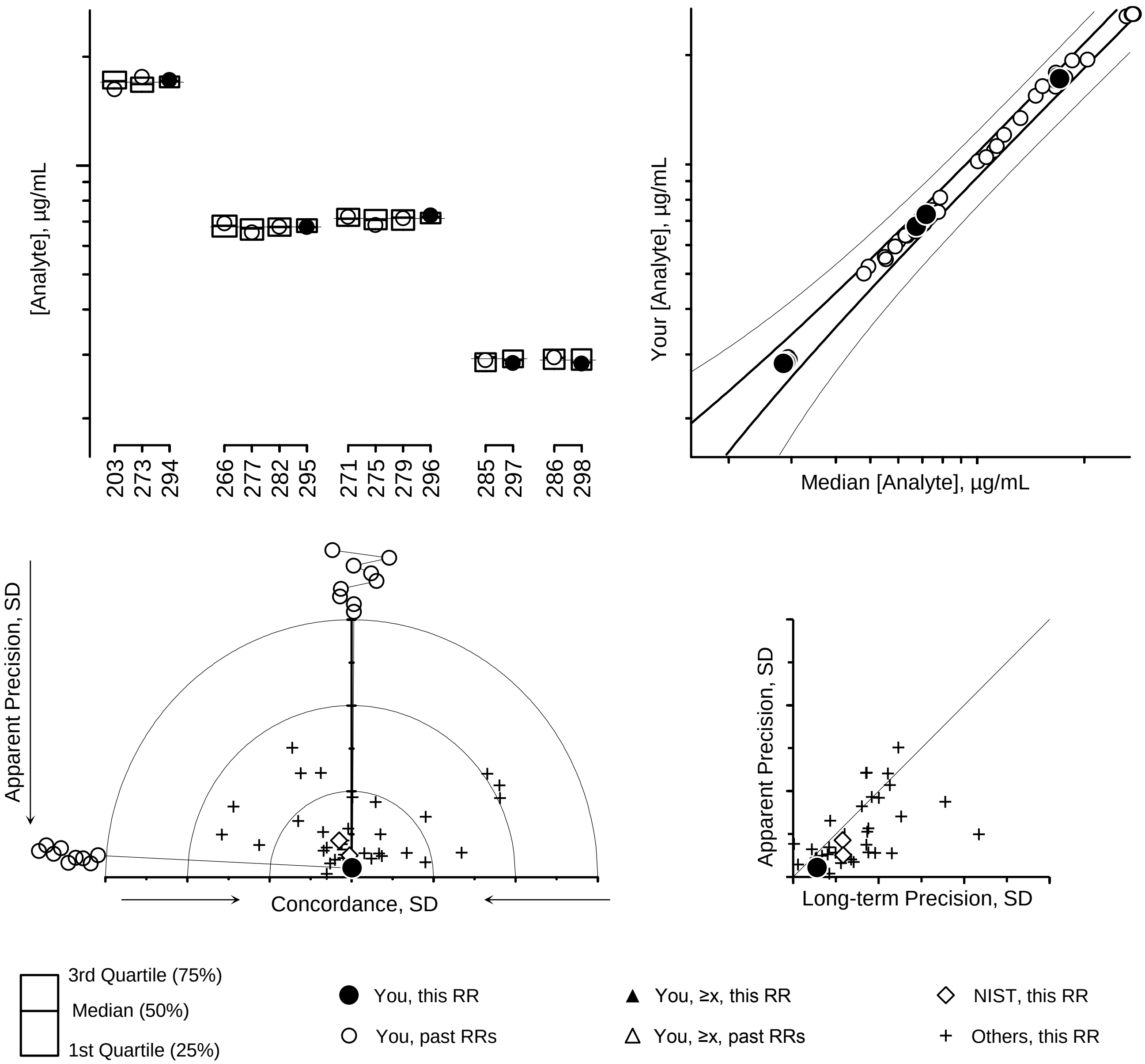
● You, this RR
○ You, past RRs
▲ You, ≥x, this RR
△ You, ≥x, past RRs
◇ NIST, this RR
+ Others, this RR

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 |
|---|---|---|
| #294 | Lyophilized: #203 RR33, #273 RR49 | Augmented with R, RP, $\alpha$T, $\gamma$T, and $\delta$T |
| #295 | Lyophilized: #266 RR48, #277 RR50, #282 RR51 | Same pool as #296, native |
| #296 | Fresh-frozen: #271 RR49, #275 RR50, #279 RR51 | Same pool as #295, native |
| #297 | Fresh-frozen: #285 RR52 | Same pool as #298, trans-Retinol augmented |
| #298 | Fresh-frozen: #286 RR52 | Same pool as #297, cis-Retinol augmented |

# Individualized RR LIV Report: FSV-BA

## α-Tocopherol

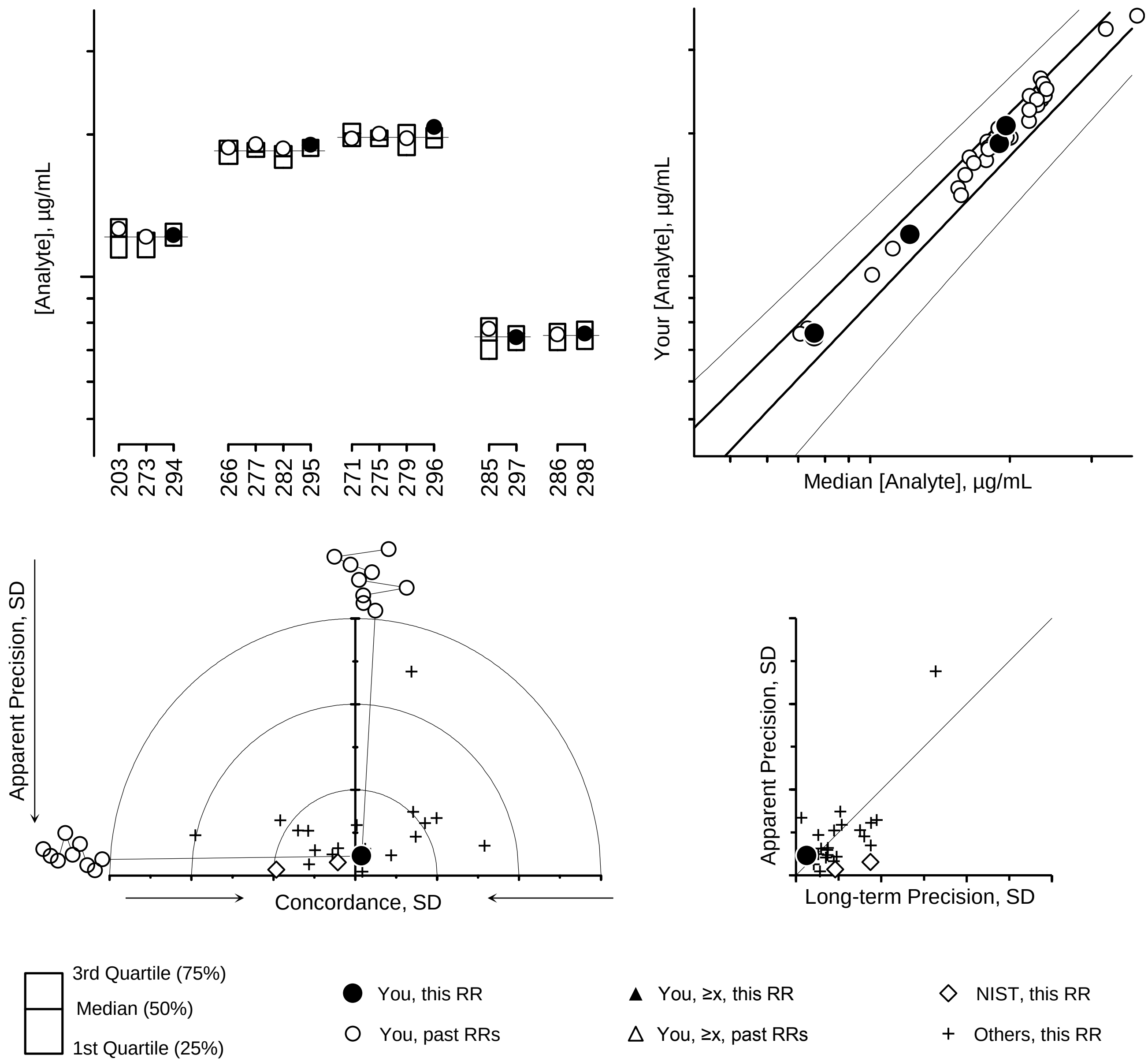



For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

| Serum | History | Comments |
|---|---|---|
| #294 | Lyophilized: #203 RR33, #273 RR49 | Augmented with R, RP, αT, γT, and δT |
| #295 | Lyophilized: #266 RR48, #277 RR50, #282 RR51 | Same pool as #296, native |
| #296 | Fresh-frozen: #271 RR49, #275 RR50, #279 RR51 | Same pool as #295, native |
| #297 | Fresh-frozen: #285 RR52 | Same pool as #298, trans-Retinol augmented |
| #298 | Fresh-frozen: #286 RR52 | Same pool as #297, cis-Retinol augmented |

# Individualized RR LIV Report: FSV-BA

## γ/β-Tocopherol

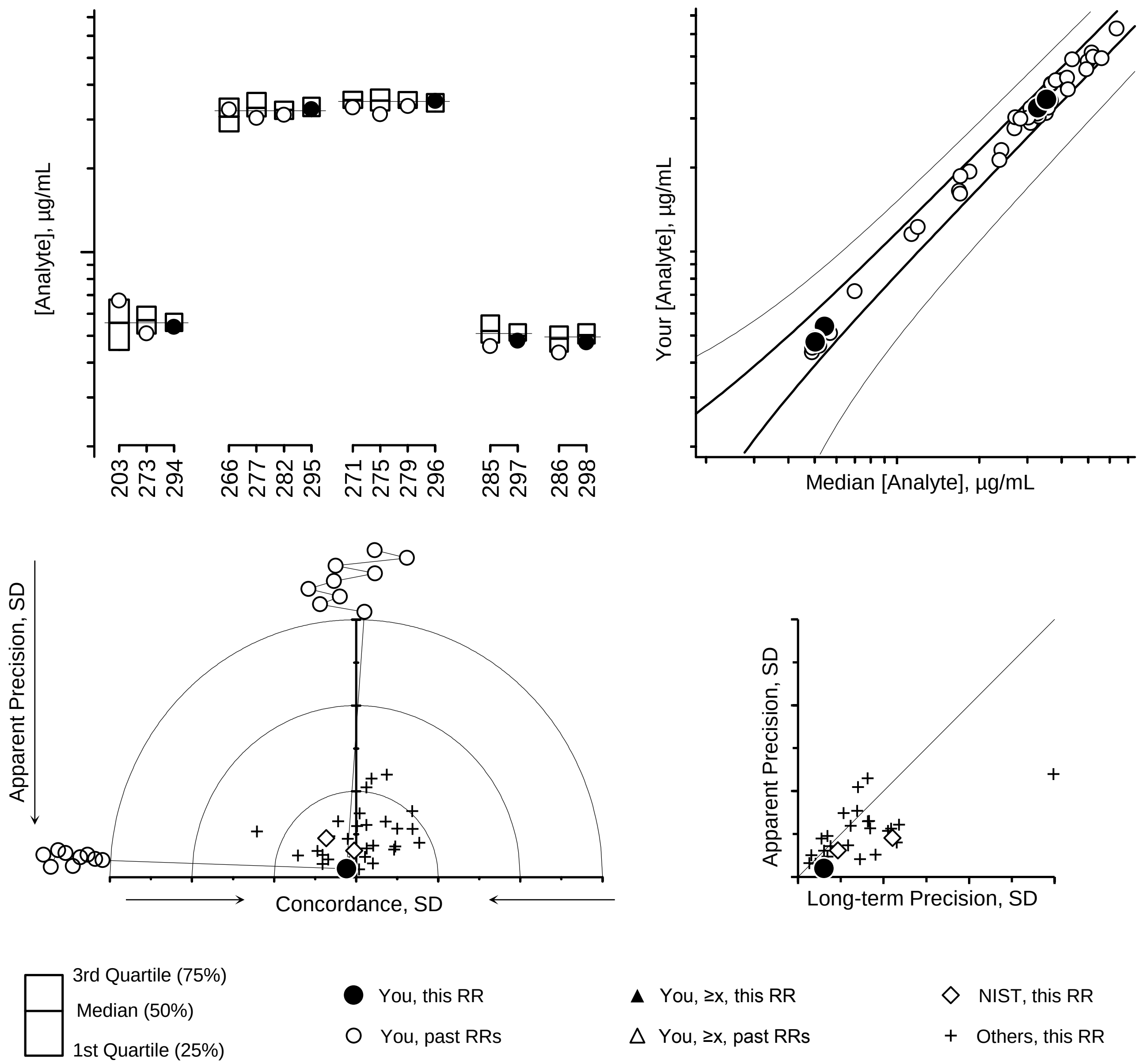



For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

| Serum | History | Comments |
|---|---|---|
| #294 | Lyophilized: #203 RR33, #273 RR49 | Augmented with R, RP, αT, γT, and δT |
| #295 | Lyophilized: #266 RR48, #277 RR50, #282 RR51 | Same pool as #296, native |
| #296 | Fresh-frozen: #271 RR49, #275 RR50, #279 RR51 | Same pool as #295, native |
| #297 | Fresh-frozen: #285 RR52 | Same pool as #298, trans-Retinol augmented |
| #298 | Fresh-frozen: #286 RR52 | Same pool as #297, cis-Retinol augmented |

# Individualized RR LIV Report: FSV-BA

## Total β-Carotene

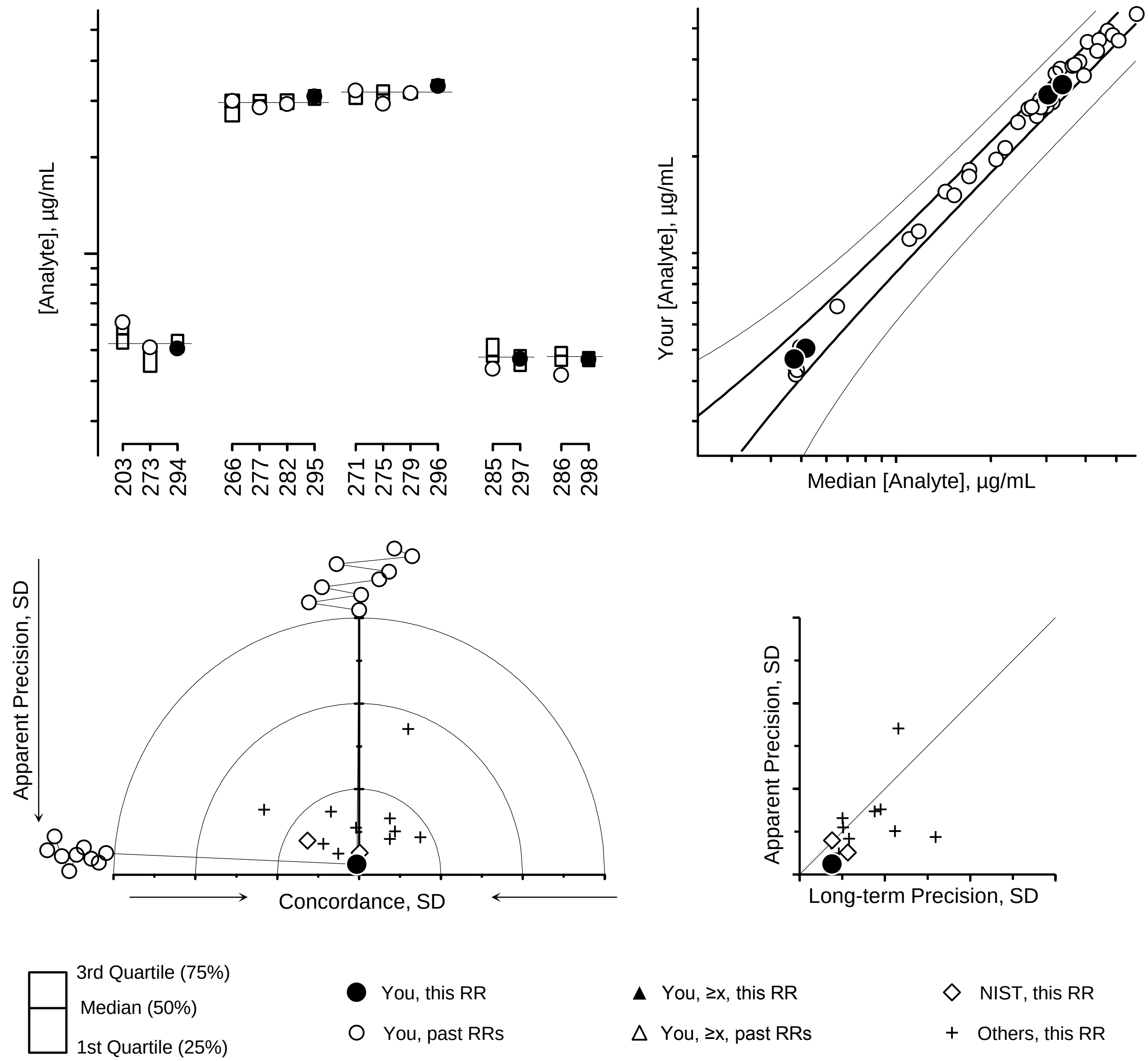



For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

| Serum | History | Comments |
|---|---|---|
| #294 | Lyophilized: #203 RR33, #273 RR49 | Augmented with R, RP, $\alpha$T, $\gamma$T, and $\delta$T |
| #295 | Lyophilized: #266 RR48, #277 RR50, #282 RR51 | Same pool as #296, native |
| #296 | Fresh-frozen: #271 RR49, #275 RR50, #279 RR51 | Same pool as #295, native |
| #297 | Fresh-frozen: #285 RR52 | Same pool as #298, trans-Retinol augmented |
| #298 | Fresh-frozen: #286 RR52 | Same pool as #297, cis-Retinol augmented |

# Individualized RR LIV Report: FSV-BA

## trans-β-Carotene

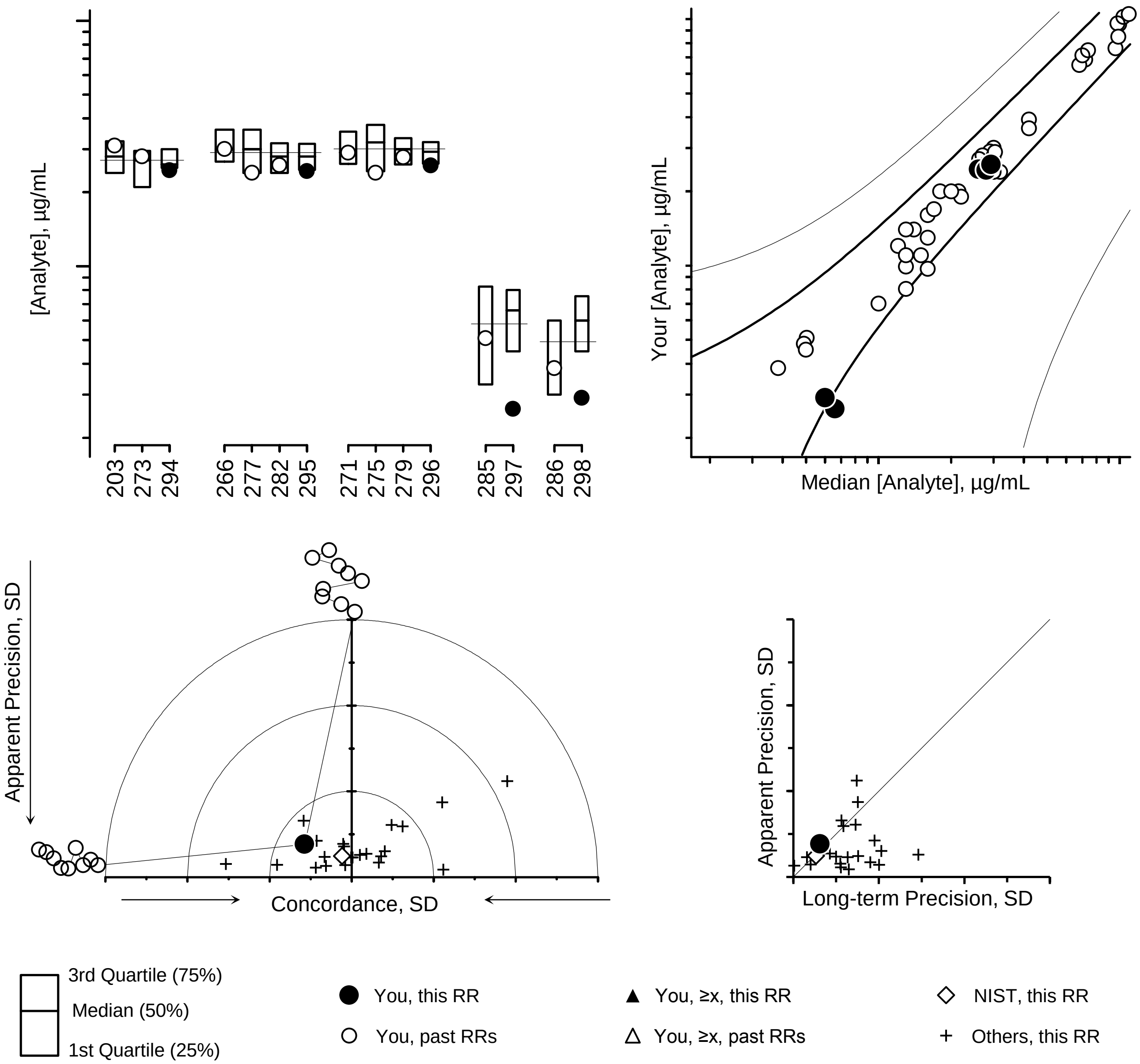



For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

| Serum | History | Comments |
|---|---|---|
| #294 | Lyophilized: #203 RR33, #273 RR49 | Augmented with R, RP, αT, γT, and δT |
| #295 | Lyophilized: #266 RR48, #277 RR50, #282 RR51 | Same pool as #296, native |
| #296 | Fresh-frozen: #271 RR49, #275 RR50, #279 RR51 | Same pool as #295, native |
| #297 | Fresh-frozen: #285 RR52 | Same pool as #298, trans-Retinol augmented |
| #298 | Fresh-frozen: #286 RR52 | Same pool as #297, cis-Retinol augmented |

# Individualized RR LIV Report: FSV-BA

## Total α-Carotene

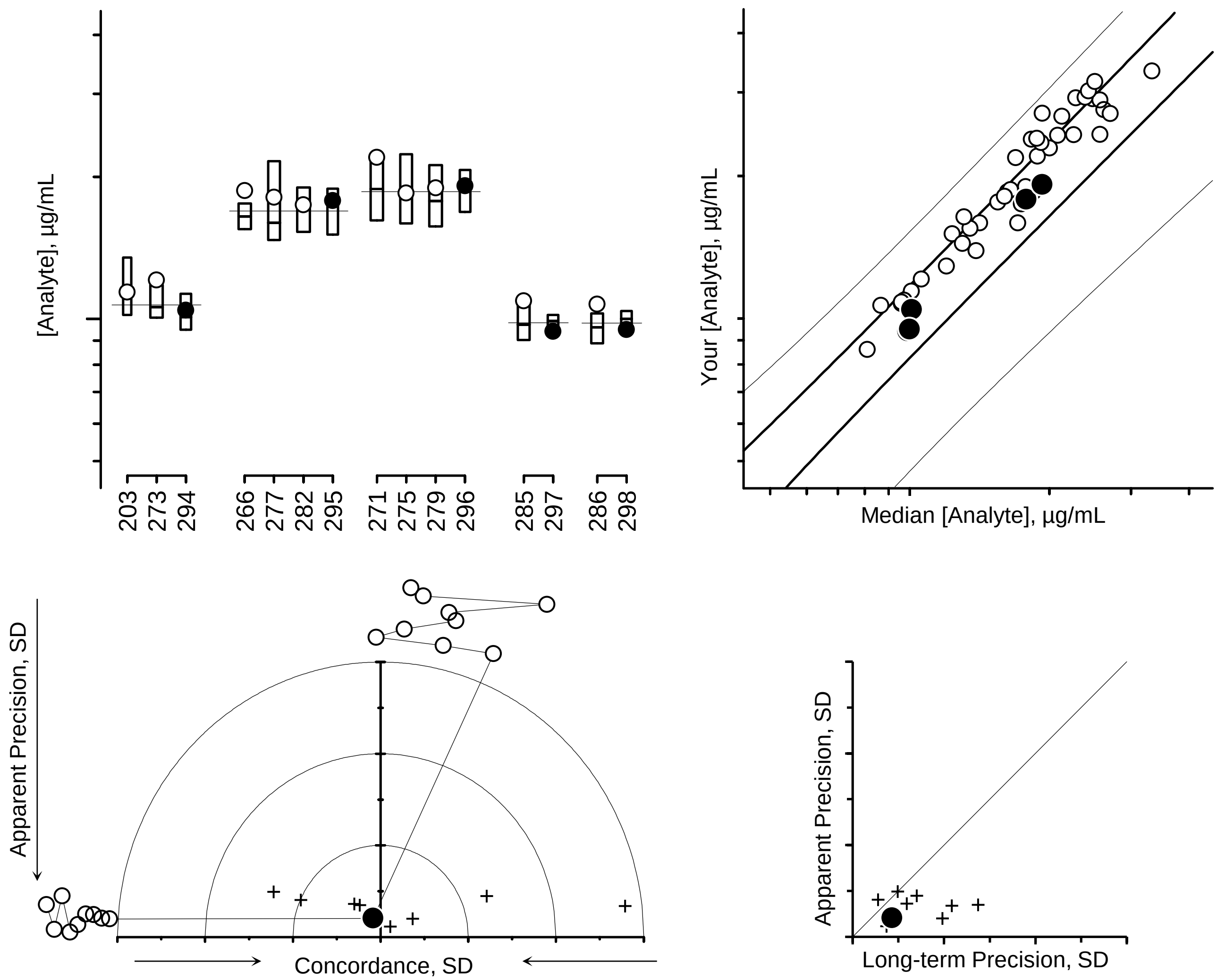



For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

| Serum | History | Comments |
|---|---|---|
| #294 | Lyophilized: #203 RR33, #273 RR49 | Augmented with R, RP, αT, γT, and δT |
| #295 | Lyophilized: #266 RR48, #277 RR50, #282 RR51 | Same pool as #296, native |
| #296 | Fresh-frozen: #271 RR49, #275 RR50, #279 RR51 | Same pool as #295, native |
| #297 | Fresh-frozen: #285 RR52 | Same pool as #298, trans-Retinol augmented |
| #298 | Fresh-frozen: #286 RR52 | Same pool as #297, cis-Retinol augmented |

# Individualized RR LIV Report: FSV-BA

## trans-Lycopene




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

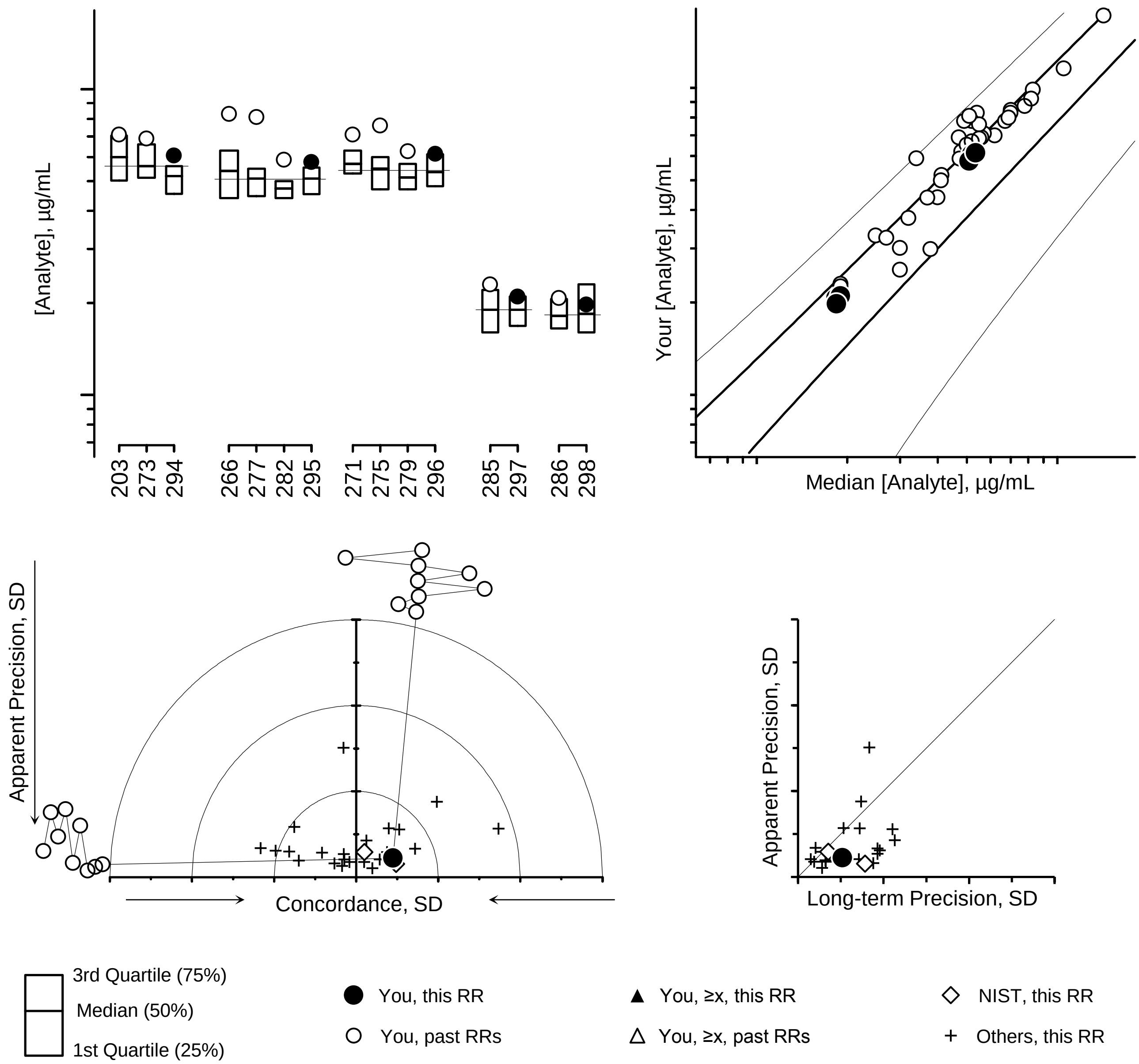
● You, this RR
○ You, past RRs
▲ You, ≥x, this RR
△ You, ≥x, past RRs
◇ NIST, this RR
\+ Others, this RR

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

| Serum | History | Comments |
|---|---|---|
| #294 | Lyophilized: #203 RR33, #273 RR49 | Augmented with R, RP, αT, γT, and δT |
| #295 | Lyophilized: #266 RR48, #277 RR50, #282 RR51 | Same pool as #296, native |
| #296 | Fresh-frozen: #271 RR49, #275 RR50, #279 RR51 | Same pool as #295, native |
| #297 | Fresh-frozen: #285 RR52 | Same pool as #298, trans-Retinol augmented |
| #298 | Fresh-frozen: #286 RR52 | Same pool as #297, cis-Retinol augmented |

# Individualized RR LIV Report: FSV-BA

## Total β-Cryptoxanthin

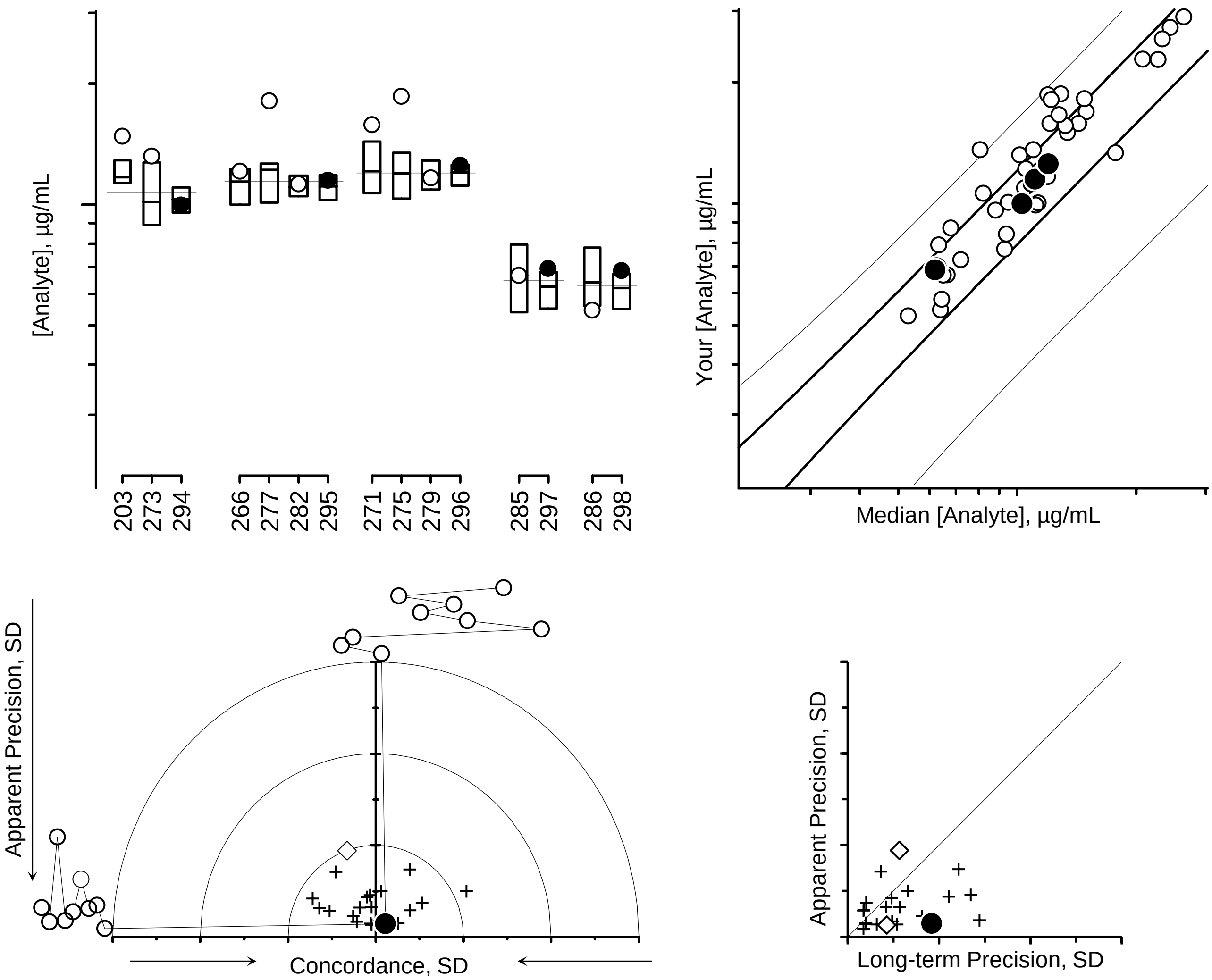



For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

| Serum | History | Comments |
|---|---|---|
| #294 | Lyophilized: #203 RR33, #273 RR49 | Augmented with R, RP, αT, γT, and δT |
| #295 | Lyophilized: #266 RR48, #277 RR50, #282 RR51 | Same pool as #296, native |
| #296 | Fresh-frozen: #271 RR49, #275 RR50, #279 RR51 | Same pool as #295, native |
| #297 | Fresh-frozen: #285 RR52 | Same pool as #298, trans-Retinol augmented |
| #298 | Fresh-frozen: #286 RR52 | Same pool as #297, cis-Retinol augmented |

# Individualized RR LIV Report: FSV-BA

## Total Lutein&Zeaxanthin



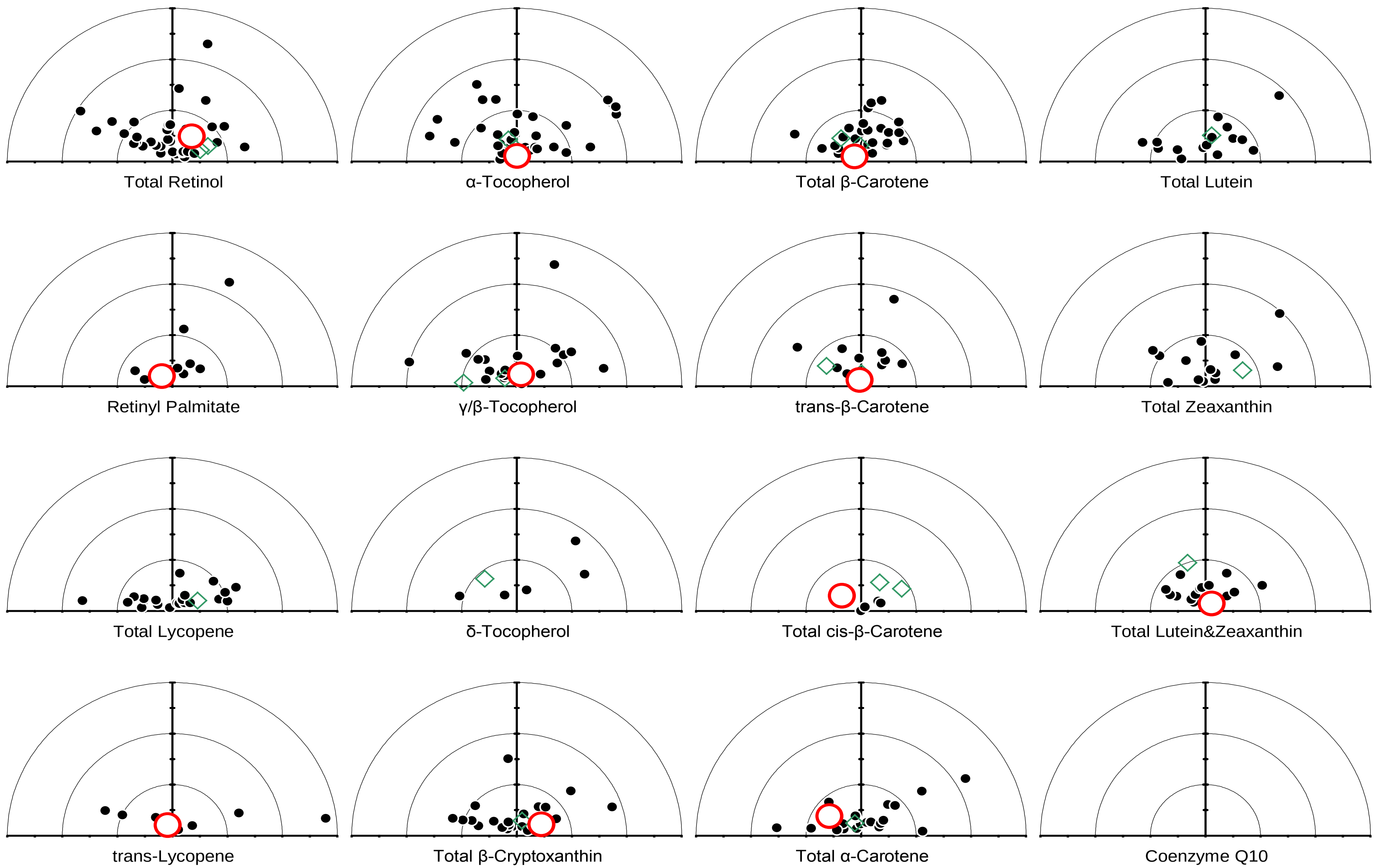

3rd Quartile (75%)
Median (50%)
1st Quartile (25%)

● 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 |
|---|---|---|
| #294 | Lyophilized: #203 RR33, #273 RR49 | Augmented with R, RP, αT, γT, and δT |
| #295 | Lyophilized: #266 RR48, #277 RR50, #282 RR51 | Same pool as #296, native |
| #296 | Fresh-frozen: #271 RR49, #275 RR50, #279 RR51 | Same pool as #295, native |
| #297 | Fresh-frozen: #285 RR52 | Same pool as #298, trans-Retinol augmented |
| #298 | Fresh-frozen: #286 RR52 | Same pool as #297, cis-Retinol augmented |

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



## Appendix E. Shipping Package Inserts for RR19

The following five items were included in each package shipped to an RR19 participant:

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

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

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

March 26, 2003

Dear Colleague:

Enclosed are samples for the vitamin C round robin study (RR 19) for the 2003 NIST Micronutrients Measurement Quality Assurance Program. RR 19 consists of three vials of frozen serum (*test samples)* and one vial of solid ascorbic acid (*control sample*). Please follow the attached protocol when you prepare and analyze these samples. If you cannot prepare the control sample solutions gravimetrically, please prepare equivalent solutions volumetrically and report the exact volumes used.

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

We recommend that you obtain **Standard Reference Material (SRM) 970 Ascorbic Acid in Serum** to validate your methodology and value assign in-house control materials. This SRM may be purchased from the Standard Reference Materials Program at NIST. (Tel: 301-975-6776, Fax: 301-948-3730, or e-mail: srminfo@nist.gov)

Please return your results for RR 19 using the attached form by **September 19, 2003** to:

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

If you have questions or concerns about this study, please contact me at 301-975-3120; e-mail: jbthomas@nist.gov; or mail/fax queries to the above address.

Sincerely,

Jean B Thomas

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

Enclosures

NIST

## Micronutrient Measurement Quality Assurance Program for
# Vitamin C

***Please Read Through Completely BEFORE Analyzing Samples***

**Protocol for Preparation and Analysis of the Control Sample**

The *control sample* consists of a sample of solid ascorbic acid in an amber vial. It should be prepared and used in the following manner:

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

2. Weigh 0.19 to 0.22 g of the solid ascorbic acid sample to 0.0001 g (if possible), dissolve it in the Diluent in a 100 mL volumetric flask, and dilute with the Diluent to the 100 mL mark. Weigh the amount of Diluent added to 0.1 g. Record the weights. The resulting material will be referred to as the "Stock Solution" below.

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

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

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

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

4. Calculate and record the total ascorbic acid concentrations, [TAA], in these Dilute Solutions.

   If you follow the above gravimetric preparation directions, the [TAA] in µmol/L is calculated:

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

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

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

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

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

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

6. Measure and record the concentration of total ascorbic acid in all three dilute solutions and in the 5% MPA Diluent in duplicate using *exactly* the same method used for the test samples, including any enzymatic treatment. Compare the replicate values. *Are you satisfied that your measurement precision is adequate?* Do **not** evaluate the test samples until you are satisfied that your system is performing properly!

7. Compare the measured with the calculated $[TAA]_{DS}$ values. This is most conveniently done by plotting the measured values on the y-axis of a scatterplot against the calculated values on the x-axis. The line through the four {calculated, measured} data pairs should go through the origin with a slope of 1.0. *Are you satisfied with the agreement between the measured and calculated values?* Do **not** evaluate the test samples until you are satisfied that your system is performing properly!

**Protocol for Analysis of the Test Samples**

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

Each *test sample* contains between 0.0 and 80.0 µmol of total ascorbic acid/L of solution. The total ascorbic acid in each ampoule should be measured in duplicate. Please report your results in µmol/(L of the sample solution) rather than µmol/(L of serum NIST used to prepare the sample).

Participant #: _______ Date: __________

# Vitamin C Round Robin 19

## NIST Micronutrient Measurement Quality Assurance Program

## Preparation and Validation of Control Samples

**STOCK SOLUTION**

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

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

**DILUTE SOLUTION 1**

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

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

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

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

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

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

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

Wavelength of maximum absorbance..........................................................__________ nm

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

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

**DILUTE SOLUTION 2**

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

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

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

**DILUTE SOLUTION 3**

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

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

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

Please return *before* **September 19, 2003** to:

MMQAP
100 Bureau Drive, Stop 8392
Gaithersburg, MD 20899-8392

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

Participant #: _______ Date: __________

# Vitamin C Round Robin 19

## NIST Micronutrient Measurement Quality Assurance Program

## Analysis of Control and Test Samples

| Sample | Replicate 1 | Replicate 2 | Units |
|---|---|---|---|
| Dilute Solution 1 | | | µmol/L of Dilute Solution |
| Dilute Solution 2 | | | µmol/L of Dilute Solution |
| Dilute Solution 3 | | | µmol/L of Dilute Solution |
| 5% MPA Diluent | | | µmol/L of Diluent |
| Test Sample #12 | | | µmol/L of Sample |
| Test Sample #25 | | | µmol/L of Sample |
| Test Sample #44 | | | µmol/L of Sample |
| Test Sample #66 | | | µmol/L of Sample |

Were samples frozen upon receipt? Yes | No

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

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

**COMMENTS:**

Please return *before* **September 19, 2003** to:

MMQAP
100 Bureau Drive, Stop 8392
Gaithersburg, MD 20899-8392

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

Participant #: ________ Date: _____________

# Vitamin C Round Robin 19

NIST Micronutrients Measurement Quality Assurance Program

## Packing List and Shipment Receipt Confirmation Form

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

| Sample | Form |
|---|---|
| VitC #12 | Liquid frozen (1:1 serum:10% MPA) |
| VitC #25 | Liquid frozen (1:1 serum:10% MPA) |
| VitC #44 | Liquid frozen (1:1 serum:10% MPA) |
| VitC #66 | Liquid frozen (1:1 serum:10% MPA) |
| Control | Solid AA |

**Please** 1) Open the pack immediately
2) Check that it contains one vial each of the above samples
3) Check if samples VitC #12, #25, #44, and #66 arrived frozen
4) Store the samples at -20 ˚C or below until analysis
5) Complete the following information
6) Fax the completed form to us at 301-977-0685
(or email requested information to david.duewer@nist.gov)

1) Date this shipment arrived: ____________________________

2) Are all 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 samples VitC #12, #25, #44, and #66 arrive frozen? Yes | No

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

6) When do you anticipate analyzing these samples? _________________________

Your prompt return of this information is appreciated.

The M$^2$QAP Gang

Mail: M$^2$QAP
NIST, Stop 8392
Gaithersburg, MD 20899-8392
Fax: 301-977-0685
Email: David.Duewer@NIST.gov

## Appendix F.  Final Report for RR19

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

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

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

November 18, 2003

Dear Colleague:

Enclosed is the summary report of the results for Round Robin 19 (RR19) for the measurement of total ascorbic acid (TAA, ascorbic acid plus dehydroascorbic acid) in human serum. Included in this report are: a summary of data for all laboratories and a summary of individual laboratory performance and interlaboratory accuracy and repeatability. As in previous reports, the estimated standard deviations (eSD) for the measurements are defined as 0.74x interquartile range and the estimate coefficients of variation (eCV) are defined as 100x eSD/median.

RR19 consists of four unknowns (*test samples*) and one solid reference ascorbic acid for preparation of control solutions. Details regarding the samples can be found in the enclosed report.

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

As you are aware, samples for the vitamin C round robin study (RR 20) for the 2004 NIST Vitamin C in Serum QA Program were recently shipped (during the **week of November 17**). It is critical that you carefully inspect all samples upon arrival and that you promptly confirm to us that they have arrived, if you have not done so already. We will replace samples (lost or damaged in shipment or miss-packaged by us) **only** for participants who report the problem within one calendar week after the package arrives.

We are pleased to announce that due to the generous funding support from the Centers for Disease Control in Atlanta, a second Vitamin C Round Robin (RR21) will be distributed, at no cost to you, during the **week of May 3, 2004**. As before, we will send you a reminder via e-mail or fax about a week prior to shipment.

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

Sincerely,

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

Enclosures



NIST

The NIST $M^2$QAP Vitamin C Round Robin 19 (RR19) report consists of

| Page | "Individualized" Report |
|---|---|
| 1 | Summarizes your reported values for the nominal 55 mmol/L solution you prepared from the ascorbic acid solid control sample and for four serum test samples. |
| 2 | Graphical summary of your RR 19 sample measurements. |

| Page | "All Lab" Report |
|---|---|
| 1 | A tabulation of results and summary statistics for Total Ascorbic Acid [TAA] in the RR19 samples and control/calibration solutions. |

**Test Samples**. Three unknowns were distributed in RR19.

S19:1 Serum 12, ampouled in late 2001, previously distributed as sample S16:2 (RR16, Mar-02). This is the base serum used to prepare the augmented test samples. The TAA level in this material is low (1 to 2 mmol/L), but it is not zero.

S19:2 Serum 25, ampouled in late 2001, previously distributed as sample 17:1 (RR17, Sep-02). An augmented serum.

S19:3 Serum 44, ampouled in late 2001, previously distributed as sample 18:2 (RR18, Mar-03). An augmented serum.

S19:4 Serum 66, SRM 970 level 1, ampouled in mid-1998. This material was distributed with identification in RR11 (Oct-98) and RR12 (Mar-99) and as samples S13-1 (RR13, Mar-00), S14-3 (RR14, Mar-01), S15:1 (RR15, Sep-01), and 16:1 (RR16, Mar-02).

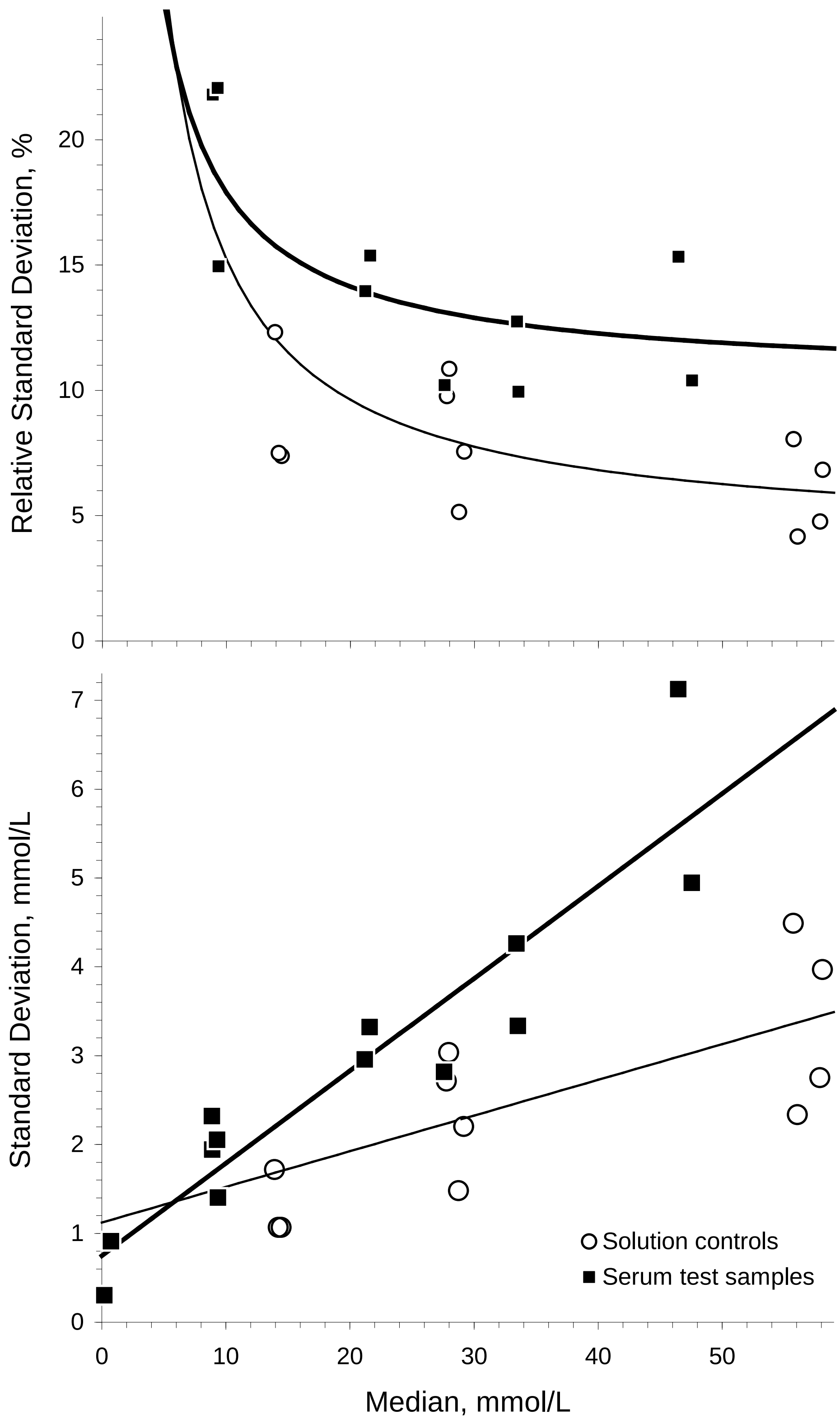
**Results**.

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

2) Judging from the Calibration Parameters calculated for the control / calibration solutions (intercepts close to 0.0 and slopes close to 1.0), the measurement systems for most participants are well calibrated. The overall among-participant agreement (concordance) is little changed by "correcting" the reported test sample results with the observed Calibration Parameters.

3) There has been no significant change in TAA average level or among-participant variability in any of the test samples.

4) The enclosed figure displays the interlaboratory estimated standard deviations (SDs) as a function of the median [TAA] for both the dilute solutions prepared from the solid control and for the serum-based test samples of RR16, RR17, RR18, and RR19. The SDs of both matrices appear to be linearly related to the TAA level. Since both a level-independent SD viability component of ≈1 mmol/L, the estimated percent relative SDs (%RSDs) of the two matrices are not constant. The limiting %RSD of the solutions prepared from the control material is ≈6%; the limiting %RSD of the serum-based test samples is about 12%.




**Expected Variability at Given Total Ascorbic Acid Levels for Solution Controls and Serum Test Samples, RR 16 – 19.**

## Appendix G. "All-Lab Report" for RR19

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

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

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

# Micronutrients Measurement Quality Assurance Program for Total Ascorbic Acid
## "Round Robin" 19 - September 2003

| Lab | Date | Control / Calibration Samples: Gravimetric, µmol/L Dil:1 | Dil:2 | Dil:3 | Measured, µmol/L Dil:1 | Dil:2 | Dil:3 | MPA | Calibration Parameters Inter | Slope | $R^2$ | SEE | MPA Density g/mL | Dilute Solution 1 Spectrophotometry $\lambda_{max}$ | $A_{max}$ | $E^{1\%}$ | Samples: Measured, µmol/L S19:1 | S19:2 | S19:3 | S19:4 | Corrected, µmol/L S19:1 | S19:2 | S19:3 | S19:4 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| VC-MA | 13/11/03 | 59.0 | 29.5 | 14.6 | 61.7 | 31.8 | 17.5 | 0.0 | 1.08 | 1.03 | 0.998 | 1.2 | 1.029 | 243. | 0.5839 | 561.9 | nq | 9.2 | 35.9 | 8.2 | | 7.8 | 33.6 | 6.9 |
| VC-MB | 01/05/03 | 59.9 | 29.9 | 15.1 | 50.9 | 25.0 | 13.2 | 2.6 | 1.60 | 0.81 | 0.998 | 1.2 | 1.031 | 244. | 0.6116 | 579.7 | 3.1 | 10.8 | 33.0 | 9.8 | 1.8 | 11.4 | 38.7 | 10.1 |
| VC-MC | 12/05/03 | 57.2 | 28.6 | 14.3 | 58.0 | 32.9 | 16.1 | 0.1 | 1.44 | 1.01 | 0.995 | 2.2 | 1.031 | 243. | 0.5294 | 525.8 | 0.7 | 12.0 | 41.1 | 9.8 | 0.0 | 10.4 | 39.1 | 8.3 |
| VC-ME | 12/09/03 | 55.0 | 27.5 | 13.8 | 52.5 | 26.1 | 12.9 | 0.2 | -0.02 | 0.95 | 1.000 | 0.2 | 1.026 | 244. | 0.5473 | 564.6 | 0.2 | 8.1 | 29.5 | 7.0 | 0.2 | 8.5 | 31.0 | 7.4 |
| VC-MG | 17/09/03 | 57.4 | 28.9 | 14.8 | 62.3 | 30.5 | 18.1 | 0.0 | 0.65 | 1.07 | 0.998 | 1.4 | 1.032 | 243.8 | 0.5685 | 562.1 | 4.5 | 10.0 | 36.8 | 10.5 | 3.6 | 8.7 | 33.7 | 9.2 |
| VC-MH | 21/05/03 | 58.5 | 29.4 | 14.2 | 58.8 | 29.7 | 14.1 | 0.0 | -0.05 | 1.01 | 1.000 | 0.2 | 1.030 | 243.7 | 0.5719 | 555.4 | 0.1 | 9.4 | 33.9 | 8.2 | 0.1 | 9.4 | 33.6 | 8.2 |
| VC-MI | 22/05/03 | 58.6 | 28.7 | 14.0 | 62.9 | 30.1 | 14.6 | 0.0 | -0.32 | 1.07 | 1.000 | 0.4 | 1.030 | | | | nq | 11.2 | 41.3 | 11.6 | | 10.7 | 38.8 | 11.1 |
| VC-MK | 15/09/03 | 60.5 | 29.8 | 14.8 | 52.7 | 28.5 | 14.3 | 0.8 | 1.60 | 0.86 | 0.998 | 1.3 | 1.033 | 244. | 0.5984 | 561.6 | 4.6 | 13.7 | 34.5 | 10.5 | 3.4 | 14.1 | 38.4 | 10.4 |
| VC-MO | 19/09/03 | 57.5 | 28.4 | 14.2 | 59.2 | 29.5 | 16.1 | 1.5 | 1.59 | 1.14 | 1.000 | 0.5 | 1.028 | 244. | 0.5395 | 532.7 | 0.7 | 9.1 | 31.9 | 9.6 | 0.0 | 6.6 | 26.6 | 7.0 |
| VC-MR | 24/06/03 | 57.3 | 29.1 | 14.4 | 55.9 | 28.3 | 14.1 | 0.0 | 0.02 | 0.97 | 1.000 | 0.1 | 1.028 | 243. | 0.5610 | 555.8 | 0.1 | 8.4 | 32.6 | 7.4 | 0.1 | 8.6 | 33.4 | 7.6 |
| VC-MS | 21/04/03 | 56.8 | 28.2 | 13.8 | | 24.2 | 10.1 | 0.0 | -0.59 | 0.86 | 0.993 | 1.4 | 1.028 | 244. | 0.5637 | 563.5 | nq | 5.1 | 19.1 | 4.2 | | 6.6 | 23.0 | 5.6 |
| VC-MT | 15/12/03 | | | | | | | | 0.00 | 1.00 | | | | | | | 0.9 | 9.4 | 31.7 | 8.2 | 0.9 | 9.4 | 31.7 | 8.2 |
| VC-MY | 10/09/03 | | | | | | | | 0.00 | 1.00 | | | | | | | < 5.7 | 7.5 | 28.8 | 6.8 | < 5.7 | 7.5 | 28.8 | 6.8 |
| NIST | 24/10/03 | 59.1 | 29.2 | 14.0 | 58.7 | 32.1 | 13.6 | 0.0 | 0.47 | 1.00 | 0.996 | 2.0 | 1.024 | 243. | 0.5723 | 549.7 | 1.4 | 9.8 | 34.0 | 8.6 | 0.9 | 9.4 | 33.5 | 8.1 |

| | Grav. Dil:1 | Dil:2 | Dil:3 | Meas. Dil:1 | Dil:2 | Dil:3 | MPA | | Density g/mL | $\lambda_{max}$ | $A_{max}$ | $E^{1\%}$ | Meas. S19:1 | S19:2 | S19:3 | S19:4 | Corr. S19:1 | S19:2 | S19:3 | S19:4 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| N | 11 | 11 | 11 | 10 | 11 | 11 | 11 | N | 11 | 10 | 10 | 10 | 9 | 13 | 13 | 13 | 9 | 13 | 13 | 13 |
| Average | 58.0 | 28.9 | 14.4 | 57.5 | 28.8 | 14.6 | 0.5 | Average | 1.030 | 243.7 | 0.5675 | 556.3 | 1.6 | 9.5 | 33.1 | 8.6 | 1.1 | 9.2 | 33.1 | 8.2 |
| SD | 1.5 | 0.7 | 0.4 | 4.3 | 2.7 | 2.2 | 0.9 | SD | 0.002 | 0.5 | 0.0256 | 15.8 | 1.9 | 2.1 | 5.7 | 2.0 | 1.5 | 2.1 | 5.0 | 1.6 |
| Min | 55.0 | 27.5 | 13.8 | 50.9 | 24.19 | 10.1 | 0.0 | Min | 1.026 | 243.0 | 0.5294 | 525.8 | 0.1 | 5.1 | 19.1 | 4.2 | 0.0 | 6.6 | 23.0 | 5.6 |
| %25 | 57.2 | 28.5 | 14.1 | 53.5 | 27.18 | 13.6 | 0.0 | %25 | 1.028 | 243.2 | 0.5507 | 555.5 | 0.2 | 8.4 | 31.7 | 7.4 | 0.1 | 7.8 | 31.0 | 7.0 |
| Median | 57.5 | 28.9 | 14.3 | 58.4 | 29.46 | 14.3 | 0.0 | Median | 1.030 | 243.9 | 0.5661 | 561.8 | 0.7 | 9.4 | 33.0 | 8.2 | 0.2 | 8.7 | 33.6 | 8.2 |
| %75 | 58.8 | 29.5 | 14.7 | 61.0 | 30.32 | 16.1 | 0.5 | %75 | 1.031 | 244.0 | 0.5809 | 563.1 | 3.1 | 10.8 | 35.9 | 9.8 | 1.8 | 10.4 | 38.4 | 9.2 |
| Max | 60.5 | 29.9 | 15.1 | 62.9 | 32.91 | 18.1 | 2.6 | Max | 1.033 | 244.0 | 0.6116 | 579.7 | 4.6 | 13.7 | 41.3 | 11.6 | 3.6 | 14.1 | 39.1 | 11.1 |
| MADe | 1.4 | 0.8 | 0.5 | 5.3 | 1.79 | 2.1 | 0.0 | MADe | 0.002 | 0.1 | 0.0271 | 6.5 | 0.8 | 2.0 | 4.2 | 2.1 | 0.3 | 1.7 | 7.1 | 1.7 |
| CV | 2 | 3 | 4 | 9 | 6 | 15 | | CV | 0.19 | 0.06 | 5 | 1 | | 22 | 13 | 25 | | 20 | 21 | 21 |

## Appendix H. Representative "Individualized Report" for RR19

Each participant in RR19 received an "Individualized Report" reflecting their reported results. The following two pages are the "Individualized Report" for participant "VC-MA".

# Vitamin C "Round Robin" 19 Report: Participant VC-MA

| Date | RR | Method | MPA Density g/mL | Dilute Solution 1 Spectrophotometry $\lambda_{max}$ | $A_{max}$ | $E^{1\%}$ | Control/Calibration Solutions $Y_{meas}$ = Inter + Slope* $X_{grav}$ Inter | Slope | $R^2$ | SEE |
|---|---|---|---|---|---|---|---|---|---|---|
| 09/27/01 | 14 | HPLC-EC | 1.028 | 243.0 | 0.541 | 547.7 | -0.9 | 1.03 | 0.999 | 1.09 |
| 09/18/01 | 15 | HPLC-EC | 1.027 | 243.0 | 0.547 | 556.5 | 0.0 | 1.04 | 1.000 | 0.05 |
| 11/18/02 | 16 | HPLC-EC | 1.032 | 242.0 | 0.575 | 576.5 | -0.4 | 1.07 | 0.999 | 0.90 |
| 12/12/02 | 17 | HPLC-EC | 1.026 | 242.0 | 0.552 | 551.0 | -0.3 | 1.06 | 1.000 | 0.49 |
| 03/20/03 | 18 | HPLC-EC | 1.026 | 244.0 | 0.509 | 563.1 | -0.1 | 1.02 | 1.000 | 0.18 |
| 11/13/03 | 19 | HPLC-EC | 1.026 | 243.0 | 0.584 | 561.9 | 1.1 | 1.03 | 0.998 | 1.24 |
| | | Mean | 1.027 | 242.8 | 0.55 | 559.5 | | | | |
| | | SD | 0.002 | 0.8 | 0.03 | 10.3 | | | | |
| | | CV | 0.22 | 0.31 | 4.8 | 1.8 | | | | |

[TAA] mmol/Lsample

| Date | RR | Sample | $Rep_1$ | $Rep_2$ | $F_{adj}$ | Mean | $SD_{dup}$ | N | Mean | $SD_{repeat}$ | $SD_{reprod}$ |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 11/18/02 | 16 | S16:2 | 0.0 | 0.1 | 1.0 | 0.0 | 0.0 | 1 | 0.0 | 0.0 | |
| 11/13/03 | 19 | S19:1 | na | na | 1.0 | | | | | | |
| 12/12/02 | 17 | S17:1 | 9.9 | 9.1 | 1.0 | 9.5 | 0.6 | 2 | 9.4 | 0.4 | 0.4 |
| 11/13/03 | 19 | S19:2 | 9.2 | 9.1 | 1.0 | 9.2 | 0.1 | | | | |
| 03/20/03 | 18 | S18:2 | 35.1 | 36.0 | 1.0 | 35.6 | 0.6 | 2 | 35.7 | 0.4 | 0.4 |
| 11/13/03 | 19 | S19:3 | 35.9 | 35.8 | 1.0 | 35.9 | 0.1 | | | | |
| 09/23/98 | 11 | S11:1:A | 15.5 | 13.9 | 0.5 | 7.4 | 0.6 | 9 | 8.0 | 0.4 | 0.6 |
| 09/23/98 | 11 | S11:1:B | 15.5 | 13.9 | 0.5 | 7.4 | 0.6 | | | | |
| 04/02/99 | 12 | S12:1:A | 14.5 | 15.8 | 0.5 | 7.6 | 0.5 | | | | |
| 04/02/99 | 12 | S12:1:B | 14.5 | 15.8 | 0.5 | 7.6 | 0.5 | | | | |
| 09/17/01 | 13 | S13:1 | 8.4 | 8.5 | 1.0 | 8.5 | 0.1 | | | | |
| 09/27/01 | 14 | S14:3 | 8.0 | 7.7 | 1.0 | 7.8 | 0.2 | | | | |
| 09/18/01 | 15 | S15:1 | 8.9 | 8.7 | 1.0 | 8.8 | 0.1 | | | | |
| 11/18/02 | 16 | S16:1 | 8.8 | 8.8 | 1.0 | 8.8 | 0.0 | | | | |
| 11/13/03 | 19 | S19:4 | 7.8 | 8.6 | 1.0 | 8.2 | 0.5 | | | | |

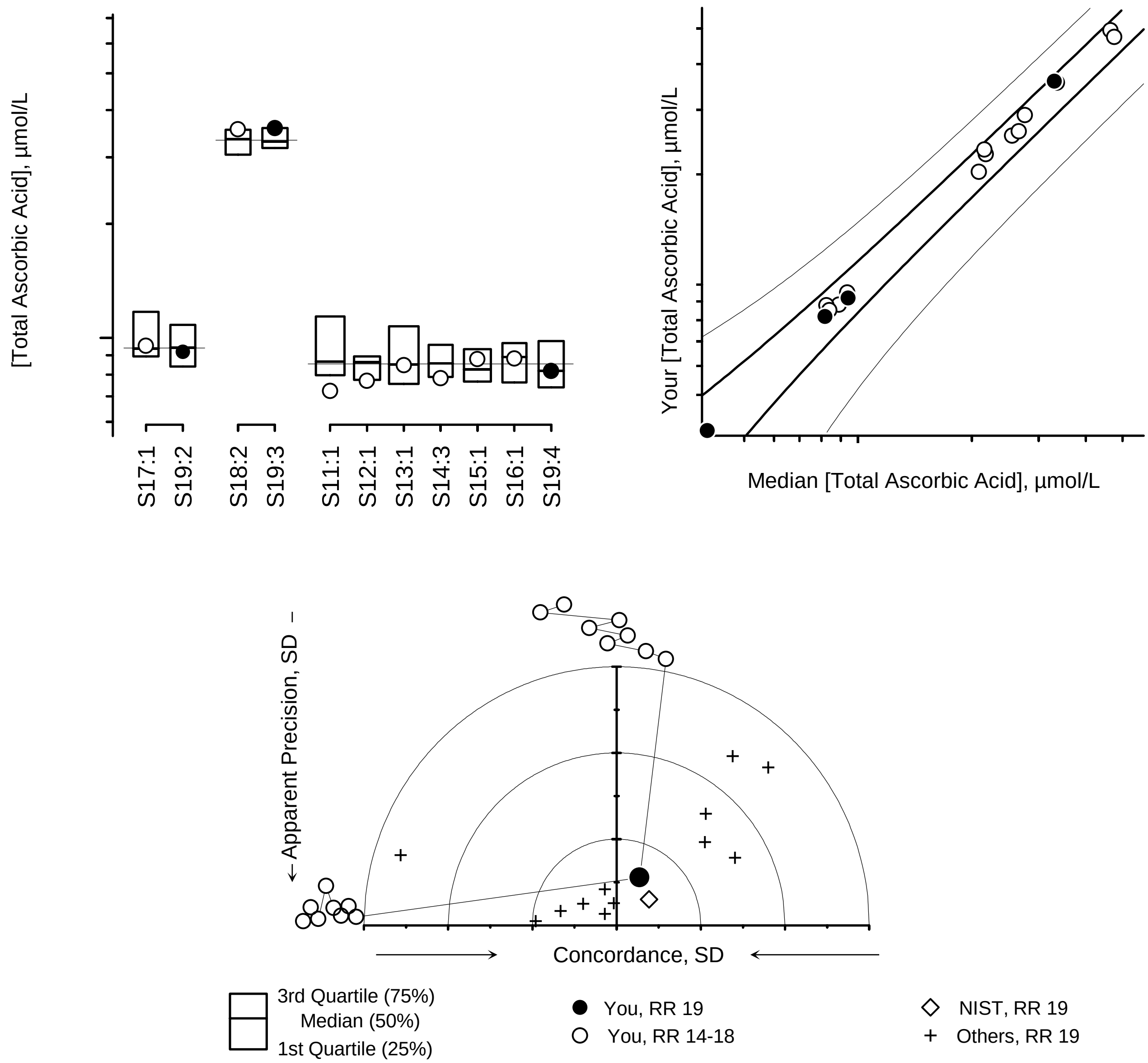
Please check our records against your records. Send corrections and/or updates to...

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

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

# Vitamin C "Round Robin" 19 Report: Participant VC-MA

## Total Ascorbic Acid




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

| Sample | Comments |
|---|---|
| S19:1 | Serum 12, previously distributed in RR 16, is a "blank" control: not displayed in the box-plot. |
| S19:2 | Serum 25, previously distributed in RR 17. |
| S19:3 | Serum 44, previously distributed in RR 18. |
| S19:4 | SRM 970 Level 1, previously distributed as in RRs 11, 12, 13, 14, 15, and 16 |