NISTIR 7880-26

# NIST Micronutrients Measurement Quality Assurance Program Winter, Spring, and Fall 1998 Comparability Studies

## Results for Round Robin XLII, XLIII, and XLIV Fat-Soluble Vitamins and Carotenoids in Human Serum and Round Robin 11 Ascorbic Acid in Human Serum

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http://dx.doi.org/10.6028/NIST.IR.7880-26

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

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David L. Duewer
Sam A. Margolis (Retired)
Katherine E. Sharpless
Jeanice B. Thomas
*Chemical Sciences Division*
*Materials Measurement Laboratory*

Margaret C. Kline
*Biomolecular Measurement Division*
*Material Measurement Laboratory*

http://dx.doi.org/10.6028/NIST.IR.7880-26

July, 2013


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

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

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## Abstract

The National Institute of Standards and Technology coordinates the Micronutrients Measurement Quality Assurance Program (MMQAP) for laboratories that measure fat- and water-soluble vitamins and carotenoids in human serum and plasma. This report describes the design of and results for the Winter, Spring and Fall 1998 MMQAP measurement comparability improvement studies: 1) Round Robin XLII Fat-Soluble Vitamins and Carotenoids in Human Serum, 2) Round Robin XLIII Fat-Soluble Vitamins and Carotenoids in Human Serum, 3) Round Robin XLIV Fat-Soluble Vitamins and Carotenoids in Human Serum, and 4) Round Robin 11 Ascorbic Acid in Human Serum. The materials for Round Robin XLII were shipped to participants in February 1998; participants were requested to provide their measurement results by April 3, 1998. The materials for Round Robin XLIII were shipped to participants in May 1998; participants were requested to provide their measurement results by June 22, 1998. The materials for Round Robin XLIV were shipped to participants in August 1998; participants were requested to provide their measurement results by October 9, 1998. The sample materials for Round Robin 11 were distributed in September 1998 with results due by October 20, 1998.



## Keywords

Human Serum
Retinol, α-Tocopherol, γ-Tocopherol, Total and *Trans*-β-Carotene
Ascorbic Acid, SRM 970

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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 XLII: Fat-Soluble Vitamins and Carotenoids in Human Serum

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin XLII comparability study (hereafter referred to as RR42) received four lyophilized 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 February 1998. 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 RR42 consists of three documents:

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

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

- An "Individualized Report" that graphically analyzes each participant's results for selected analytes. This report also provides a graphical summary of their measurement comparability. The graphical tools used in this report are described in detail elsewhere [3]. An example "Individualized Report" is reproduced as Appendix D.

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

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin XLIII comparability study (hereafter referred to as RR43) received four lyophilized human serum test samples for analysis. Unless multiple vials were previously requested, participants received one vial of each material. These sample materials were shipped on dry ice to participants in June 1998. The communication materials included in the sample shipment are provided in Appendix E.

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 RR43 consists of three documents:

- A cover letter for the current study, a brief description of the other document, 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 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 G.

- An "Individualized Report" that graphically analyzes each participant's results for selected analytes. This report also provides a graphical summary of their measurement comparability. The graphical tools used in this report are described in detail elsewhere [3]. An example "Individualized Report" is reproduced as Appendix H.

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

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin XLIV comparability study (hereafter referred to as RR44) received four lyophilized human serum test samples 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 August 1998. The communication materials included in the sample shipment are provided in Appendix G.

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 RR43 consists of three documents:

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

- 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 I.

- An "Individualized Report" that graphically analyzes each participant's results for selected analytes. This report also provides a graphical summary of their measurement comparability. The graphical tools used in this report are described in detail elsewhere [3]. An example "Individualized Report" is reproduced as Appendix H.

## Round Robin 11: Vitamin C in Human Serum

Participants in the MMQAP Vitamin C in Human Serum Round Robin 11 comparability study (hereafter referred to as RR11) received four frozen serum test samples and a solid ascorbic acid control material for analysis. The four test samples were stated to be duplicate ampoules of Levels 1 and 2 of candidate SRM 970 Ascorbic Acid in Human Serum. These sample materials were shipped on dry ice to participants in September 1998. The communication materials included in the sample shipment are provided in Appendix M.

The test materials were prepared by adding equal volumes of 10 % metaphosphoric acid (MPA) to human serum that had been spiked with ascorbic acid. Participants were asked to provide two results for each vial. Participants were also asked to prepare and evaluate a standard solution of 50 µmol ascorbic acid (AA) per L solution of 5 % by mass metaphosphoric acid.

No report on participant results was prepared for RR11 itself. A combined report on the results obtained in RR11 and/or in Round Robin 12 was prepared and reported to participants in August of 1999. This report is presented in full in Appendix J in NISTIR 7880-25; Table 2 of this report is equivalent to the usual "All Lab Report" for RR11.

## 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 RR42

The following two items were included in each package shipped to an RR42 participant:

- Cover letter
- Datasheet

The cover letter and datasheet were enclosed in a sealed waterproof bag along with the samples themselves.

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

February 4, 1998

Dear Colleague:

Enclosed is the set of samples for the first quality assurance round robin exercise (Round Robin XLII) for FY98. You will find one vial of each of four lyophilized serum samples for analysis along with a form for reporting your results. When reporting your results, please submit one value for each analyte for a given serum sample. If an obtained value is below your limit of quantitation, please indicate this result on the form by using NQ *(Not Quantitated)*. For analytes not measured, please leave a blank. Results are due to NIST by April 3, 1998. Results received two weeks after the due date will not be included in the summary report for this round robin study. Written feedback concerning the study will be provided around April 30.

Samples should be reconstituted with 1.0 mL of HPLC-grade water or equivalent. We recommend that dissolution be facilitated with 3 to 5 min agitation in an ultrasonic bath or at least 30 min. at room temperature with intermittent swirling. (CAUTION: Vigorous shaking will cause foaming and possibly interfere with accurate measurement. The rubber stopper contains phthalate esters that will leach into the sample upon intermittent contact of the liquid sample with the stopper. These esters absorb strongly in the UV region and elute very 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.). For consistency, we request that laboratories use the following absorptivities (E 1% cm) in ethanol: retinol, 1843 at 325 nm; retinyl palmitate, 975 at 325 nm; α-tocopherol, 75.8 at 292 nm; γ-tocopherol, 91.4 at 298 nm; α-carotene, 2800 at 444 nm (in hexane); β-carotene, 2560 at 450 nm (in ethanol), 2592 at 452 nm (in hexane); lycopene, 3450 at 472 nm (in hexane).

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

Micronutrients Measurement Quality Assurance Program
NIST
Bldg. 222, Rm. B208
Gaithersburg, MD 20899
Fax: (301) 977-0685

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

Sincerely,

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

Enclosures

cc: S. Wise

*NIST*

# *Micronutrients Measurement Quality Assurance Program*

## Round Robin XLII Results from Laboratory #_____

| | Serum | | | | |
|---|---|---|---|---|---|
| Analyte | 239 | 240 | 241 | 242 | Units* |
| retinol | | | | | |
| retinyl palmitate | | | | | |
| α-tocopherol | | | | | |
| γ-tocopherol | | | | | |
| δ-tocopherol | | | | | |
| total β-carotene | | | | | |
| trans-β-carotene | | | | | |
| total cis-β-carotene | | | | | |
| total α-carotene | | | | | |
| trans-α-carotene | | | | | |
| total lycopene | | | | | |
| trans-lycopene | | | | | |
| β-cryptoxanthin | | | | | |
| α-cryptoxanthin | | | | | |
| lutein | | | | | |
| zeaxanthin | | | | | |
| lutein&zeaxanthin | | | | | |
| Other Analytes? | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |

* We prefer mg/mL

Today's Date:

Comments?

Mail: MMQAP, 222/B208
NIST
Gaithersburg, MD 20899

Please return results on-or-before
April 3, 1998

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

## Appendix B. Final Report for RR42

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

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

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

May 28, 1998

Dear Colleague:

Enclosed is the summary report of the results for Round Robin XLII (RR42). Included in this report are: a summary of data for all laboratories; the measurement comparability summary for evaluating laboratory performance; a summary of individual laboratory performance for the past three years; a summary of the interlaboratory accuracy and precision over the same period of time for the measurement of retinol, α- and γ-tocopherol, and *trans*- and total β-carotene; and a graphical summary of the NIST assigned value (NAV) vs. your laboratory value for these analytes. As in previous reports, the NIST assigned values are derived from the equally weighted results from the analyses performed by NIST and the laboratories that participated in this interlaboratory comparison exercise.

We regret the delay in issuing this report. This year has been marked by both the sorrow of endings--Dave's mother passed away in late April-and the joy of beginnings--Kathy and her new baby are doing well! An interim report of interlaboratory median values was faxed (on April 10, about two weeks after the closing date) to all participants who had by then submitted quantitative values.

The experimental design for this interlaboratory comparison exercise (RR42) is summarized in the attached report. The four serum samples (Sera 239-242) distributed are all new samples that were used to further evaluate our analyte augmentation techniques. These samples, however, were from an aged (about 10 years) serum pool which apparently caused some of the observed interferences. About 10% of the reporting laboratories indicated that they saw interferences in the retinol and/or tocopherol chromatographic region. As indicated in the attached report, we believe the interference is plasticizers extracted from one or more of the bags used to store the plasma units. While plasticizer contamination of samples is not an unexpected problem, recognition of such a contamination is an important consideration for method development and validation. We apologize for any incovenience that this may have caused.

Data for evaluating laboratory performance in RR42 are provided in the comparability summary (Score Card) on page 6 of the "All Lab Report." Due to the observed interferences noted in this round robin exercise, the criteria used to evaluate laboratory performance are as follows: results rated A (within 1 SD of the assigned value) indicate EXCEPTIONAL performance, results rated B (within 2 SD) indicate ACCEPTABLE performance, results rated C (greater than 2 SD of the assigned value) are MARGINAL relative to the current state-of-the-practice for these measurements.

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



NIST

As you are aware, the intercomparison exercises for fat-soluble vitamins in human serum in which you are currently participating have been supported in part by the National Cancer Institute (NCI). Last September, our interagency agreement with NCI for the Micronutrient Measurement Quality Assurance (QA) Program expired. To date, NCI has not expressed interest in continuing support for this program. Because of the uncertainty of the funding since last October, NIST has committed to continue the intercomparison exercises through FY 98. We will keep you informed concerning the status of QA activities beyond 1998.

Samples for RR43 were shipped during the first week of May. Results are due July 10; feedback to labs will be provided during the first week of August.

If you have any questions regarding this report, please contact me at 301/975-3120; FAX: 301/977-0685; e-mail: jeanice.brownthomas@nist.gov.

Sincerely,

Jeanice Brown Thomas

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

cc: S. Wise

Enclosures

# Analysis of NIST M²QAP Round Robin XLII Results: Sera 239 to 242

**Background.** Four samples were distributed in RR42.

Serum 239:a new serum, prepared from a composite of three plasma units purchased several years ago and stored since then at –80°C. The combined pool was mixed thoroughly and twice-filtered, but was otherwise unadulterated – at least by us!

Serum 241:a new serum, prepared from the same base material as Serum 239 but spiked with: retinyl palmitate, γ-tocopherol, δ-tocopherol, *trans*-β-carotene, *trans*-α-carotene, zeaxanthin, and *trans*-lycopene.

Serum 242:a new serum, prepared from the same base material as Serum 239 but spiked with: retinol, retinyl palmitate, α-tocopherol, δ-tocopherol, *trans*-β-carotene, *trans*-α-carotene, lutein, and *trans*-β-cryptoxanthin.

Serum 240:a new serum, prepared as a 1:1 by volume blend of Sera 241 and 242.

The spiking procedure used with Sera 241 and 242 adds no organic solvent and does not create non-extractable analyte-protein associations; however, it does dilute the non-spiked analytes by approximately 20% and may modify the lipoprotein composition.

## Qualitative Results.

Interferences: About 20% of participants reported, either spontaneously or after we issued a "heads up" in the interim report, some degree of interference in the retinol and tocopherol retention zone of their chromatograms. The magnitude of the reported problems ranged from "we can't analyze these samples" to "an unusual baseline." Further, two participants volunteered "We see nothing unusual." (On retrospective examination, our chromatograms have a "humpogram" baseline in the retinol/tocopherol retention zone.)

From the preparation history, the quite diverse impact of the interferences, and examination of the chromatograms kindly provided by the afflicted participants, we believe the interference is plasticizers extracted from one or more of the bags the plasma units were supplied / stored / thawed in. Unfortunately, the bags themselves are long since disposed of so this must remain a working hypothesis. The erratic impact of the plasticizer is attributed to differences among your extraction, chromatography, and detection systems.

Action: Plasticizer contamination of samples is a "not unexpected" problem. Whether or not quantitative results can be obtained in the presence of interferences, recognition of such problems is an important consideration for method development and validation. Everyone who recognized that their results for these samples were questionable deserves a gold star!

While we apologize for having the *same* interferences in all four samples, we consider all four to be "valid." Serum 239 is no more, but you may see Sera 240, 241, and 242 again (but not all at the same time!). Now that we know what to look for, we intend to examine the historical data/archived chromatograms for evidence interference in previous samples.

In recognition of the unusual nature of the four sera distributed in RR42, we have modified our "grading scheme" from four levels (1-4) to three (A-C).

"Lutein&Zeaxanthin": One of our participants recently tried to make sense of the various "Lutein", "Zeaxanthin", and "Lutein&Zeaxanthin" data we've been reporting and found it all very confusing. About half of the participants who report the composite analyte "Lutein&Zeaxanthin" do not report either of the component analytes. Only about half of the participants who do report both "Lutein" and "Zeaxanthin" also explicitly report "Lutein&Zeaxanthin". While some of these participants clearly estimate the composite independently, others appear to calculate it as the simple sum of the components.

Action: As we stated some time ago, we calculate "Lutein&Zeaxanthin" results as the simple sum of the constituents whenever both "Lutein" and "Zeaxanthin" are reported. As of RR 42, we distinguish between explicitly reported values for "Lutein&Zeaxanthin" and values that we calculate from the components: the explicitly reported values are in standard font, our calculated values are *italicized.*

**Quantitative Results.** The following NIST Data and Value/Uncertainty Assignments table presents all NIST data, summary statistics for the NIST data, summary results for RR42, and the NIST assigned values and uncertainties. Since one of the two NIST analysts had higher priority responsibilities during the past several months, these statistics reflect results from only one NIST chromatographic system. The entries are defined as follows:

NIST Data and Summary Statistics

A:1 to C:2 two aliquots ("1" and "2") of three vials (A, B, and C) of each serum were extracted and analyzed.

n number of quantitative values for this analyte for this serum

$Mean_{NIST}$ arithmetic average

SD simple standard deviation

$SD_{rep}$ within-vial pooled standard deviation, reflecting variation in extraction, chromatography, peak integration, etc.

$SD_{het}$ among-sample standard deviation, reflecting heterogeneity in preparing and reconstituting the serum samples

$SD_{NIST}$ $\sqrt{SD_{rep}^2 + SD_{het}^2}$, total standard deviation.

$CV_{NIST}$ $100 \times SD_{NIST}/Mean_{NIST}$

Round Robin XLII Summary Statistics

$n_n$ number of non-NIST laboratories reporting quantitative values for this analyte for this serum in this Round Robin

$Median_n$ median of the reported values

$eSD_n$ 0.741 × InterQuartile Range (IQR)

$SD_{labs}$ $\sqrt{eSD_n^2 - SD_{NIST}^2}$, the residual non-NIST interlaboratory biases after correction for measurement-, sample-, and NIST-analyst-related sources of variance. When $SD_{NIST}$ is greater than $eSD_n$, $SD_{labs} = 0$.

$CV_{labs}$ $100 \times SD_{labs}/Median_n$

NIST Assigned Values and Uncertainties

NAV $(\text{Mean}_{\text{NIST}} + \text{Median}_{n}) / 2$, our best guess of the "true" analyte level

NAU $\text{Maximum}\left(0.05 \times \text{NAV}, \sqrt{\text{SD}^2_{\text{NIST}} + \text{SD}^2_{\text{labs}}}\right)$, our best guess for the "true" interlaboratory standard deviation characterizing measurement, sample heterogeneity, inter-analyst, and interlaboratory sources of variation.

CV $100 \times \text{NAU} / \text{NAV}$

Expected Interlaboratory Uncertainties

eCV $100 \times \sqrt{L^2_{qc} + (\beta_0 \times \text{NAV}^{\beta_1})^2} \Big/ \text{NAV}$ where $L_{qc}$, $\beta_0$, and $\beta_1$ are empirical constants describing historical results (Duewer *et al*, Anal Chem 1997:69;1406-1413.)

**Measurement Performance Summary.**

Measurement Variability: The observed CVs are larger than expected for γ-tocopherol, *trans*-β-carotene, and total α-carotene. They are about as expected for retinol, α-tocopherol, total β-carotene, and β-cryptoxanthin. The approximately 100% increase in the γ-tocopherol CV is likely related to plasticizer interference. The 50% increase in the α-carotene CV is less plausibly related to plasticizers, and may represent interferences from one or more of the spiking solutions and/or underestimation of the "true" expected analytical variability.

Expected Results: The design of the analyte spiking protocol allows the prediction of relative analyte levels among the sera. The expected levels are:

retinol, α-tocopherol, β-cryptoxanthin, and lutein...
- Serum 242... highest level (from analyte-specific spikes)
- Serum 241... lowest level (from ~15% dilution by spikes of other analytes)
- Serum 240... average of Serum 242 and Serum 241 levels
- Serum 239... somewhere between levels of Serum 241 and Serum 240

γ-tocopherol, *trans*-lycopene, and zeaxanthin...
- Serum 242... lowest level (from ~21% dilution by spikes of other analytes)
- Serum 241... highest level (from analyte-specific spikes)
- Serum 240... average of Serum 242 and Serum 241 levels
- Serum 239... somewhere between levels of Serum 241 and Serum 240

retinyl palmitate, δ-tocopherol, *trans*-β–carotene, and "lutein&zeaxanthin"...
- Serum 242... highest level (larger analyte-specific spikes)
- Serum 241... next-to-lowest level (smaller analyte-specific spikes)
- Serum 240... average of Serum 242 and Serum 241 levels
- Serum 239... lowest level

*trans*-α-carotene...
- Serum 242... next-to-lowest level (smaller analyte-specific spike)
- Serum 241... highest level (larger analyte-specific spike)
- Serum 240... average of Serum 242 and Serum 241 levels
- Serum 239... lowest level

any other analyte you may have analyzed...
- Serum 242... lowest level (~21% dilution)
- Serum 241... next to highest level (~15% dilution)
- Serum 240... average of Serum 242 and Serum 241 levels
- Serum 239... highest level

Observed Results: Too few results are available to evaluate retinyl palmitate, δ-tocopherol, and the "any other" analytes. For those analytes displayed in the boxplot comparisons (pages 2 and 3 of your Individualized Report), the observed results are as follows:

Fully as expected..... retinol, α-tocopherol, γ-tocopherol, β-cryptoxanthin

Nearly as expected... β-carotene, α-carotene, lutein, and "lutein&zeaxanthin". The median analyte levels in Serum 240 are slightly larger than the average of medians for Sera 241 and 242. Further, the observed "high spike" analyte levels were less than expected.

Unexpected..... lycopene, where the levels are virtually are the same for all sera, and zeaxanthin, where the Serum 240 level is highest.

Actions: Some fraction of the carotenoid analytes continue to be lost and/or non-extractably bound in "high spike" sera, keeping us from achieving our 95% population targets. If analytical resources become available, we will continue to (attempt to) refine our augmentation techniques.

The lycopene spike either was irrecoverably bound or degraded before/during/after lyophilization. If analytical resources become available, we will address this problem before attempting any further multi-analyte augmentation.

The elevated Serum 240 zeaxanthin result is inexplicable. **All** analysts reporting zeaxanthin uniformly found a higher level in Serum 240 than in either of it's constituent components! In our chromatograms the putative "lutein" and "zeaxanthin" peaks are well resolved, more-or-less typically shaped, and have characteristic retention times, thus we do not believe that chromatographic resolution is the cause of the problem (although it may contribute to the increased variability of the Serum 240 result relative to Sera 241 and 242). If analytical resources become available, we will certainly pursue this puzzle! Right now, though, we're clueless as to what might be going on. Any ideas?

Dave Duewer
Research Chemist
David.Duewer@NIST.gov

Margaret Kline
Research Biologist
Margaret.Kline@NIST.gov

# NIST Data and Value/Uncertainty Assignments

| | Retinol | | | | α-Tocopherol | | | | γ-Tocopherol | | | | Total β-Carotene | | | | trans-β-Carotene | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | NIST1 | | | | NIST1 | | | | NIST1 | | | | NIST1 | | | | NIST1 | | | |
| | 239 | 240 | 241 | 242 | 239 | 240 | 241 | 242 | 239 | 240 | 241 | 242 | 239 | 240 | 241 | 242 | 239 | 240 | 241 | 242 |
| A:1 | 0.536 | 0.697 | 0.412 | 0.976 | 7.35 | 14.9 | 6.63 | 19.8 | 1.78 | 3.19 | 3.55 | 1.76 | 0.089 | 0.522 | 0.196 | 0.798 | 0.081 | 0.487 | 0.185 | 0.763 |
| A:2 | 0.529 | 0.711 | 0.402 | 0.973 | 7.26 | 14.7 | 5.76 | 20.0 | 1.58 | 2.50 | 3.53 | 1.86 | 0.094 | 0.526 | 0.194 | 0.849 | 0.082 | 0.485 | 0.186 | 0.834 |
| B:1 | 0.545 | 0.680 | 0.414 | 0.914 | 7.34 | 14.3 | 6.49 | 19.2 | 1.64 | 2.82 | 3.60 | 2.19 | 0.086 | 0.638 | *nq* | 0.684 | 0.077 | 0.591 | *nq* | 0.628 |
| B:2 | 0.531 | 0.705 | 0.431 | 0.881 | 6.87 | 14.2 | 6.62 | 20.5 | 1.75 | 2.76 | 3.37 | 1.83 | 0.086 | 0.657 | 0.194 | 0.711 | 0.078 | 0.607 | 0.178 | 0.664 |
| C:1 | 0.544 | 0.638 | 0.419 | 1.095 | 6.92 | 13.4 | 6.85 | 23.8 | 1.72 | 2.89 | 3.18 | 1.94 | 0.087 | 0.600 | 0.192 | 0.769 | 0.087 | 0.557 | 0.186 | 0.753 |
| C:2 | 0.507 | 0.696 | 0.410 | 0.884 | 7.27 | 14.0 | 7.42 | 17.3 | 1.74 | 2.78 | 3.32 | 1.96 | 0.088 | 0.631 | 0.188 | 0.709 | 0.081 | 0.576 | 0.174 | 0.669 |
| $n_x$ | 6 | 6 | 6 | 6 | 6 | 6 | 6 | 6 | 6 | 6 | 6 | 6 | 6 | 6 | 5 | 6 | 6 | 6 | 5 | 6 |
| $Mean_{NIST}$ | 0.532 | 0.688 | 0.415 | 0.954 | 7.17 | 14.2 | 6.63 | 20.1 | 1.70 | 2.82 | 3.43 | 1.92 | 0.088 | 0.595 | 0.193 | 0.753 | 0.081 | 0.551 | 0.182 | 0.718 |
| SD | 0.014 | 0.027 | 0.010 | 0.081 | 0.22 | 0.5 | 0.54 | 2.1 | 0.07 | 0.22 | 0.16 | 0.15 | 0.003 | 0.058 | 0.003 | 0.063 | 0.003 | 0.052 | 0.006 | 0.078 |
| $SD_{rep}$ | 0.017 | 0.026 | 0.009 | 0.087 | 0.24 | 0.2 | 0.43 | 2.7 | 0.09 | 0.29 | 0.11 | 0.15 | 0.002 | 0.015 | 0.002 | 0.034 | 0.002 | 0.010 | 0.005 | 0.047 |
| $SD_{bet}$ | 0.006 | 0.019 | 0.008 | 0.049 | 0.12 | 0.5 | 0.47 | 0.4 | 0.02 | 0.03 | 0.15 | 0.10 | 0.003 | 0.064 | 0.003 | 0.064 | 0.003 | 0.058 | 0.004 | 0.077 |
| $SD_{NIST}$ | 0.018 | 0.032 | 0.012 | 0.100 | 0.27 | 0.6 | 0.64 | 2.7 | 0.09 | 0.29 | 0.19 | 0.19 | 0.004 | 0.066 | 0.003 | 0.073 | 0.004 | 0.059 | 0.006 | 0.090 |
| $CV_{NIST}$ | 3.3 | 4.7 | 2.9 | 11 | 3.8 | 4.2 | 9.7 | 13 | 5.5 | 10 | 5.5 | 9.7 | 4.0 | 11 | 1.7 | 9.6 | 4.8 | 11 | 3.5 | 13 |
| | RRXLII | | | | RRXLII | | | | RRXLII | | | | RRXLII | | | | RRXLII | | | |
| | 239 | 240 | 241 | 242 | 239 | 240 | 241 | 242 | 239 | 240 | 241 | 242 | 239 | 240 | 241 | 242 | 239 | 240 | 241 | 242 |
| $n_n$ | 44 | 44 | 44 | 44 | 44 | 44 | 44 | 44 | 21 | 21 | 21 | 21 | 29 | 29 | 29 | 29 | 8 | 8 | 8 | 8 |
| $Median_n$ | 0.507 | 0.704 | 0.436 | 0.909 | 7.83 | 14.2 | 6.42 | 20.9 | 1.58 | 2.89 | 3.65 | 1.67 | 0.073 | 0.472 | 0.173 | 0.626 | 0.074 | 0.478 | 0.176 | 0.585 |
| $eSD_n$ | 0.047 | 0.053 | 0.045 | 0.088 | 0.64 | 1.3 | 0.50 | 1.7 | 0.24 | 0.43 | 0.53 | 0.25 | 0.016 | 0.059 | 0.034 | 0.082 | 0.004 | 0.064 | 0.013 | 0.103 |
| $SD_{labs}$ | 0.043 | 0.042 | 0.044 | 0 | 0.58 | 1.1 | 0 | 0 | 0.22 | 0.33 | 0.50 | 0.17 | 0.016 | 0 | 0.034 | 0.037 | 0.000 | 0.026 | 0.011 | 0.050 |
| $CV_{labs}$ | 8.5 | 5.9 | 10 | 0 | 7.4 | 7.9 | 0 | 0 | 14 | 11 | 14 | 10 | 22 | 0 | 20 | 5.9 | 0.7 | 5.3 | 6.2 | 8.5 |
| NAV | 0.520 | 0.696 | 0.425 | 0.931 | 7.50 | 14.2 | 6.52 | 20.5 | 1.64 | 2.86 | 3.54 | 1.80 | 0.081 | 0.534 | 0.183 | 0.690 | 0.077 | 0.514 | 0.179 | 0.652 |
| NAU | 0.047 | 0.053 | 0.045 | 0.100 | 0.64 | 1.3 | 0.64 | 2.7 | 0.24 | 0.43 | 0.53 | 0.25 | 0.016 | 0.066 | 0.034 | 0.082 | 0.004 | 0.064 | 0.013 | 0.103 |
| CV | 9.0 | 7.6 | 11 | 11 | 8.5 | 9.0 | 9.8 | 13 | 15 | 15 | 15 | 14 | 20 | 12 | 19 | 12 | 5.1 | 12 | 7.1 | 16 |
| eCV | 8.8 | 8.8 | 8.9 | 9.0 | 7.7 | 7.3 | 8.2 | 7.7 | 9.3 | 7.8 | 7.5 | 8.9 | 23 | 15 | 19 | 14 | 12 | 9.1 | 9.6 | 9.0 |

# NIST Data and Value/Uncertainty Assignments

| | Total α-Carotene | | | | β-Cryptoxanthin | | | | "Lutein" | | | | "Zeaxanthin" | | | | "Lutein&Zeaxanthin" | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | NIST1 | | | | NIST3 | | | | NIST3 | | | | NIST3 | | | | NIST3 | | | |
| | 239 | 240 | 241 | 242 | 239 | 240 | 241 | 242 | 239 | 240 | 241 | 242 | 239 | 240 | 241 | 242 | 239 | 240 | 241 | 242 |
| A:1 | *nd* | 0.225 | 0.288 | 0.073 | 0.067 | 0.141 | 0.070 | 0.178 | 0.060 | 0.312 | 0.051 | 0.553 | 0.037 | 0.236 | 0.126 | 0.064 | 0.097 | 0.548 | 0.177 | 0.617 |
| A:2 | *nd* | 0.231 | 0.291 | 0.081 | 0.064 | 0.147 | 0.068 | 0.186 | 0.061 | 0.313 | 0.047 | 0.521 | 0.037 | 0.223 | 0.126 | 0.048 | 0.097 | 0.536 | 0.173 | 0.568 |
| B:1 | *nd* | 0.250 | 0.206 | 0.067 | 0.065 | 0.146 | 0.067 | 0.170 | 0.063 | 0.300 | 0.059 | 0.517 | 0.029 | 0.233 | 0.153 | 0.057 | 0.091 | 0.533 | 0.212 | 0.574 |
| B:2 | *nd* | 0.239 | 0.255 | 0.072 | 0.066 | 0.149 | 0.075 | 0.168 | 0.059 | 0.296 | 0.049 | 0.484 | 0.029 | 0.218 | 0.123 | 0.051 | 0.087 | 0.514 | 0.172 | 0.535 |
| C:1 | *nd* | 0.192 | 0.301 | 0.062 | 0.065 | 0.138 | 0.078 | 0.184 | 0.063 | 0.279 | 0.050 | 0.597 | 0.029 | 0.225 | 0.122 | 0.053 | 0.092 | 0.504 | 0.172 | 0.651 |
| C:2 | *nd* | 0.224 | 0.280 | 0.077 | 0.063 | 0.146 | 0.072 | 0.178 | 0.059 | 0.296 | 0.054 | 0.488 | 0.025 | 0.220 | 0.124 | 0.048 | 0.084 | 0.517 | 0.177 | 0.536 |
| $n_x$ | 0 | 6 | 6 | 6 | 6 | 6 | 6 | 6 | 6 | 6 | 6 | 6 | 6 | 6 | 6 | 6 | 6 | 6 | 6 | 6 |
| $Mean_{NIST}$ | | 0.227 | 0.270 | 0.072 | 0.065 | 0.144 | 0.072 | 0.177 | 0.061 | 0.299 | 0.052 | 0.527 | 0.031 | 0.226 | 0.129 | 0.053 | 0.092 | 0.525 | 0.181 | 0.580 |
| SD | | 0.020 | 0.035 | 0.007 | 0.001 | 0.004 | 0.004 | 0.007 | 0.002 | 0.013 | 0.004 | 0.043 | 0.005 | 0.007 | 0.012 | 0.006 | 0.005 | 0.017 | 0.015 | 0.046 |
| $SD_{rep}$ | | 0.014 | 0.021 | 0.007 | 0.002 | 0.005 | 0.004 | 0.004 | 0.002 | 0.007 | 0.005 | 0.048 | 0.002 | 0.008 | 0.012 | 0.007 | 0.004 | 0.011 | 0.016 | 0.053 |
| $SD_{bet}$ | | 0.018 | 0.034 | 0.004 | 0.001 | 0.003 | 0.003 | 0.007 | 0.001 | 0.012 | 0.002 | 0.023 | 0.005 | 0.004 | 0.008 | 0.003 | 0.005 | 0.016 | 0.010 | 0.022 |
| $SD_{NIST}$ | | 0.023 | 0.041 | 0.008 | 0.002 | 0.005 | 0.005 | 0.008 | 0.002 | 0.015 | 0.005 | 0.053 | 0.005 | 0.009 | 0.014 | 0.008 | 0.006 | 0.019 | 0.019 | 0.058 |
| $CV_{NIST}$ | | 10 | 15 | 11 | 2.7 | 3.6 | 7.3 | 4.5 | 4.1 | 4.9 | 10 | 10 | 18 | 4.0 | 11 | 15 | 6.5 | 3.7 | 11 | 9.9 |
| | RRXLII | | | | RRXLII | | | | RRXLII | | | | RRXLII | | | | RRXLII | | | |
| | 239 | 240 | 241 | 242 | 239 | 240 | 241 | 242 | 239 | 240 | 241 | 242 | 239 | 240 | 241 | 242 | 239 | 240 | 241 | 242 |
| $n_n$ | 21 | 24 | 24 | 24 | 24 | 24 | 24 | 24 | 12 | 12 | 12 | 11 | 10 | 10 | 10 | 8 | 23 | 23 | 23 | 23 |
| $Median_n$ | 0.013 | 0.230 | 0.304 | 0.086 | 0.072 | 0.111 | 0.060 | 0.142 | 0.062 | 0.241 | 0.046 | 0.397 | 0.028 | 0.171 | 0.108 | 0.050 | 0.091 | 0.420 | 0.158 | 0.472 |
| $eSD_n$ | 0.006 | 0.076 | 0.100 | 0.028 | 0.016 | 0.022 | 0.013 | 0.034 | 0.010 | 0.026 | 0.014 | 0.041 | 0.005 | 0.080 | 0.025 | 0.013 | 0.011 | 0.068 | 0.036 | 0.077 |
| $SD_{labs}$ | 0.006 | 0.072 | 0.092 | 0.027 | 0.016 | 0.021 | 0.012 | 0.033 | 0.010 | 0.022 | 0.013 | 0 | 0 | 0.080 | 0.021 | 0.011 | 0.009 | 0.065 | 0.031 | 0.051 |
| $CV_{labs}$ | 46 | 31 | 30 | 31 | 22 | 19 | 20 | 23 | 15 | 8.9 | 29 | 0 | 0 | 47 | 19 | 21 | 9.8 | 16 | 19 | 11 |
| NAV | 0.013 | 0.229 | 0.287 | 0.079 | 0.069 | 0.128 | 0.066 | 0.160 | 0.061 | 0.270 | 0.049 | 0.462 | 0.029 | 0.199 | 0.118 | 0.052 | 0.091 | 0.473 | 0.169 | 0.526 |
| NAU | 0.006 | 0.076 | 0.100 | 0.028 | 0.016 | 0.022 | 0.013 | 0.034 | 0.010 | 0.026 | 0.014 | 0.053 | 0.005 | 0.080 | 0.025 | 0.013 | 0.011 | 0.068 | 0.036 | 0.077 |
| CV | 46 | 33 | 35 | 35 | 23 | 17 | 20 | 21 | 16 | 9.6 | 29 | 12 | 18 | 40 | 21 | 26 | 12 | 14 | 21 | 15 |
| eCV | 37 | 25 | 25 | 27 | 26 | 23 | 26 | 22 | | | | | | | | | | | | |

## Appendix C. "All-Lab Report" for RR42

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

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

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

# Round Robin XLII Laboratory Results

All values in µg/mL

| | Retinol | | | | Retinyl Palmitate | | | | α-Tocopherol | | | | γ-Tocopherol | | | | δ-Tocopherol | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Lab | 239 | 240 | 241 | 242 | 239 | 240 | 241 | 242 | 239 | 240 | 241 | 242 | 239 | 240 | 241 | 242 | 239 | 240 | 241 | 242 |
| FSV-BA | 0.501 | 0.727 | 0.432 | 0.93 | 0.059 | 0.070 | 0.056 | 0.067 | 7.9 | 13.8 | 6.0 | 20.6 | 1.56 | 2.84 | 3.32 | 1.78 | | | | |
| FSV-BD | 0.512 | 0.697 | 0.442 | 0.91 | | | | | 6.7 | 13.1 | 5.3 | 19.4 | | | | | | | | |
| FSV-BE | 0.510 | 0.700 | 0.400 | 0.88 | | | | | 7.6 | 13.5 | 6.2 | 19.7 | 1.40 | 2.70 | 3.50 | 1.50 | | | | |
| FSV-BF | 0.549 | 0.725 | 0.513 | 0.94 | | | | | 8.0 | 14.1 | 6.2 | 21.7 | 1.55 | 2.88 | 3.65 | 1.66 | | | | |
| FSV-BG | 0.538 | 0.756 | 0.452 | 0.98 | 0.037 | 0.054 | 0.035 | 0.068 | 8.2 | 14.9 | 6.8 | 22.2 | 1.61 | 3.02 | 3.85 | 1.67 | | | | |
| FSV-BH | 0.537 | 0.715 | 0.455 | 0.96 | *nq* | *nq* | *nq* | *nq* | 8.2 | 14.9 | 6.7 | 21.7 | 1.83 | 3.55 | 4.57 | 1.99 | | | | |
| FSV-BI | 0.476 | 0.658 | 0.390 | 0.87 | *nd* | *nd* | *nd* | *nd* | 7.3 | 13.8 | 6.0 | 20.1 | 1.41 | 2.69 | 3.40 | 1.61 | | | | |
| FSV-BJ | 0.472 | 0.690 | 0.412 | 0.90 | *nq* | *nq* | *nq* | *nq* | 8.3 | 14.7 | 6.5 | 21.7 | 1.67 | 3.14 | 4.16 | 1.84 | | | | |
| FSV-BK | 0.491 | 0.707 | 0.437 | 0.87 | | | | | 7.5 | 14.7 | 6.2 | 20.0 | | | | | | | | |
| FSV-BL | 0.520 | 0.720 | 0.460 | 0.92 | | | | | 8.2 | 15.1 | 5.6 | 21.5 | | | | | | | | |
| FSV-BM | 0.537 | 0.694 | 0.430 | 0.92 | | | | | 7.7 | 14.5 | 6.3 | 21.3 | | | | | | | | |
| FSV-BN | 0.458 | 0.667 | 0.392 | 0.84 | 0.013 | 0.035 | 0.023 | 0.045 | 8.0 | 15.4 | 6.4 | 22.2 | 1.34 | 2.71 | 3.35 | 1.40 | *nq* | 0.301 | 0.105 | 0.587 |
| FSV-BO | 0.444 | 0.616 | 0.358 | 0.80 | | | | | 6.7 | 11.8 | 5.2 | 17.3 | | | | | | | | |
| FSV-BP | 0.357 | 0.624 | 0.326 | 0.57 | | | | | 7.4 | 9.8 | 6.5 | 12.8 | | | | | | | | |
| FSV-BQ | 0.568 | 0.724 | 0.475 | 0.98 | | | | | 7.9 | 14.2 | 6.9 | 20.9 | | | | | | | | |
| FSV-BR | 0.530 | 0.720 | 0.430 | 0.92 | | | | | | | | | | | | | | | | |
| FSV-BS | 0.507 | 0.647 | 0.446 | 0.83 | | | | | | | | | | | | | | | | |
| FSV-BU | 0.645 | 0.693 | 0.475 | 0.92 | | | | | 7.2 | 13.1 | 6.5 | 20.4 | 1.67 | 2.71 | 3.51 | 1.59 | | | | |
| FSV-BV | 0.610 | 0.810 | 0.470 | 0.99 | | | | | 8.6 | 15.8 | 6.7 | 22.1 | 1.73 | 3.59 | 4.20 | 1.78 | | | | |
| FSV-BW | 0.475 | 0.688 | 0.380 | 0.90 | *nq* | 0.016 | *nq* | 0.017 | 7.4 | 14.0 | 5.3 | 20.6 | 1.73 | 3.40 | 4.16 | 1.75 | | | | |
| FSV-BZ | | | | | | | | | 8.4 | 13.8 | 8.1 | 20.9 | 2.30 | 3.10 | 4.60 | 3.20 | | | | |
| FSV-CB | 0.417 | 0.632 | 0.360 | 0.80 | | | | | 5.6 | 11.5 | 4.7 | 16.2 | | | | | | | | |
| FSV-CBa | 0.560 | 0.830 | 0.560 | 0.98 | | | | | 7.4 | 14.5 | 6.4 | 20.4 | | | | | | | | |
| FSV-CC | 0.507 | 0.699 | 0.499 | 0.87 | | | | | 7.5 | 14.2 | 6.3 | 20.7 | | | | | | | | |
| FSV-CD | 0.453 | 0.669 | 0.433 | 0.85 | 0.070 | 0.111 | 0.109 | 0.131 | 8.2 | 14.8 | 6.7 | 22.1 | 1.41 | 2.77 | 3.61 | 1.49 | | | | |
| FSV-CE | 0.507 | 0.707 | 0.434 | 0.91 | | | | | 8.4 | 15.3 | 6.4 | 21.5 | | | | | | | | |
| FSV-CF | 0.526 | 0.750 | 0.454 | 1.00 | | | | | 8.5 | 14.1 | 6.9 | 21.9 | | | | | | | | |
| FSV-CH | 0.421 | 0.552 | 0.395 | 0.80 | | | | | 6.8 | 11.6 | 6.3 | 18.3 | 1.05 | 2.09 | 2.91 | 1.19 | | | | |
| FSV-CI | 0.570 | 0.770 | 0.450 | 0.99 | 0.070 | 0.080 | 0.070 | 0.090 | 6.8 | 12.4 | 5.4 | 18.7 | | | | | | | | |
| FSV-CK | 0.696 | 0.736 | 0.516 | 1.17 | | | | | 10.5 | 15.1 | 7.8 | 26.2 | 2.08 | 3.30 | 5.08 | 2.17 | | | | |
| FSV-CL | 0.363 | 0.469 | 0.307 | 0.62 | | | | | 7.8 | 12.5 | 6.8 | 18.8 | 1.14 | 1.91 | 2.96 | 0.80 | | | | |
| FSV-CM | | | | | | | | | 4.1 | 13.7 | 5.0 | 19.6 | | | | | | | | |
| FSV-CN | 0.506 | 0.718 | 0.418 | 0.97 | | | | | 7.3 | 15.6 | 6.4 | 23.1 | 1.23 | 2.81 | 3.45 | 1.23 | | | | |
| FSV-CP | | | | | | | | | 7.9 | 13.8 | 6.4 | 20.0 | 1.75 | 3.20 | 3.72 | 2.14 | | | | |
| FSV-CQ | 0.500 | 0.713 | 0.443 | 0.97 | | | | | 7.9 | 15.2 | 6.6 | 21.6 | | | | | | | | |
| FSV-CR | 0.520 | 0.700 | 0.460 | 0.80 | | | | | 6.6 | 11.7 | 5.4 | 18.5 | | | | | <0.3 | 0.400 | 0.400 | 0.800 |
| FSV-CS | 0.464 | 0.636 | 0.365 | 0.87 | | | | | 7.5 | 14.8 | 6.5 | 22.3 | 1.34 | 2.89 | 3.51 | 1.64 | | | | |
| FSV-CT | 0.541 | 0.777 | 0.459 | 1.06 | | | | | 9.5 | 16.8 | 7.2 | 27.2 | | | | | | | | |
| FSV-CU | 0.458 | 0.643 | 0.394 | 0.80 | 0.058 | 0.068 | 0.054 | 0.071 | 7.1 | 13.3 | 5.4 | 17.9 | | | | | | | | |
| FSV-CX | 0.500 | 0.750 | 0.420 | 0.93 | 0.050 | 0.070 | 0.050 | 0.050 | 8.0 | 15.9 | 6.6 | 22.2 | 1.58 | 3.48 | 4.16 | 1.98 | 0.030 | 0.530 | 0.010 | 0.840 |
| FSV-DB | 0.518 | 0.757 | 0.502 | 1.02 | | | | | 8.5 | 15.3 | 7.1 | 23.0 | | | | | | | | |
| FSV-DF | 0.510 | 0.800 | 0.460 | 1.03 | | | | | | | | | | | | | | | | |
| FSV-DJ | 0.510 | 0.720 | 0.430 | 0.92 | | | | | 9.2 | 16.0 | 7.2 | 21.0 | | | | | | | | |
| FSV-DK | 0.450 | 0.650 | 0.360 | 0.85 | 0.004 | 0.006 | *nq* | 0.005 | 7.5 | 13.8 | 5.9 | 20.3 | 1.61 | 3.41 | 4.18 | 1.73 | | | | |
| FSV-DP | 0.485 | 0.677 | 0.425 | 0.89 | | | | | | | | | | | | | | | | |
| FSV-DR | 0.532 | 0.751 | 0.440 | 0.97 | | | | | 8.0 | 14.7 | 6.5 | 22.0 | | | | | | | | |
| FSV-DW | 0.493 | 0.663 | 0.440 | 0.86 | | | | | 8.6 | 14.8 | 7.0 | 20.0 | | | | | | | | |
| FSV-EM | 0.570 | 0.800 | 0.550 | 0.89 | | | | | 7.2 | 12.8 | 5.5 | 18.3 | | | | | | | | |
| FSV-ES | 0.475 | 0.696 | 0.406 | 0.88 | | | | | 8.1 | 14.7 | 6.6 | 21.4 | | | | | | | | |
| FSV-FG | 0.610 | 0.830 | 0.640 | 1.02 | | | | | 6.6 | 12.6 | 5.2 | 17.8 | | | | | | | | |
| FSV-FH | | | | | | | | | *nq*: Interfering peaks | | | | | | | | | | | |
| n | 47 | 47 | 47 | 47 | 8 | 9 | 7 | 9 | 46 | 46 | 46 | 46 | 21 | 21 | 21 | 21 | 1 | 3 | 3 | 3 |
| Min | 0.357 | 0.469 | 0.307 | 0.57 | 0.004 | 0.006 | 0.023 | 0.005 | 4.1 | 9.8 | 4.7 | 12.8 | 1.05 | 1.91 | 2.91 | 0.80 | | 0.301 | 0.010 | 0.587 |
| Median | 0.507 | 0.707 | 0.437 | 0.91 | 0.054 | 0.068 | 0.054 | 0.067 | 7.8 | 14.2 | 6.4 | 20.8 | 1.58 | 2.89 | 3.65 | 1.67 | 0.030 | 0.400 | 0.105 | 0.800 |
| Max | 0.696 | 0.830 | 0.640 | 1.17 | 0.070 | 0.111 | 0.109 | 0.131 | 10.5 | 16.8 | 8.1 | 27.2 | 2.30 | 3.59 | 5.08 | 3.20 | | 0.530 | 0.400 | 0.840 |
| eSD | 0.046 | 0.059 | 0.037 | 0.08 | 0.024 | 0.021 | 0.024 | 0.033 | 0.7 | 1.2 | 0.5 | 1.7 | 0.25 | 0.31 | 0.49 | 0.26 | | | | |
| eCV | 9 | 8 | 8 | 9 | 44 | 31 | 44 | 49 | 8 | 9 | 8 | 8 | 16 | 11 | 13 | 15 | | | | |
| NISTa | 0.532 | 0.688 | 0.415 | 0.95 | | | | | 7.2 | 14.2 | 6.6 | 20.1 | 1.70 | 2.82 | 3.43 | 1.92 | | | | |
| NAV | 0.520 | 0.698 | 0.426 | 0.93 | 0.054 | 0.068 | 0.054 | 0.067 | 7.5 | 14.2 | 6.5 | 20.4 | 1.64 | 2.86 | 3.54 | 1.80 | | | | |
| NAU | 0.049 | 0.060 | 0.045 | 0.10 | 0.024 | 0.021 | 0.024 | 0.033 | 0.8 | 1.2 | 0.7 | 2.8 | 0.26 | 0.44 | 0.56 | 0.31 | | | | |

# Round Robin XLII Laboratory Results

All values in µg/mL

| | Total β-Carotene | | | | trans-β-Carotene | | | | Total cis-β-Carotene | | | | Total α-Carotene | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Lab | 239 | 240 | 241 | 242 | 239 | 240 | 241 | 242 | 239 | 240 | 241 | 242 | 239 | 240 | 241 | 242 |
| FSV-BA | 0.088 | 0.45 | 0.180 | 0.63 | 0.081 | 0.436 | 0.172 | 0.60 | 0.007 | 0.018 | 0.008 | 0.027 | 0.013 | 0.153 | 0.235 | 0.077 |
| FSV-BD | !0.099 | !0.66 | !0.256 | !0.86 | | | | | | | | | | | | |
| FSV-BE | 0.099 | 0.56 | 0.145 | 0.63 | | | | | | | | | | | | |
| FSV-BF | 0.074 | 0.42 | 0.120 | 0.63 | | | | | | | | | 0.018 | 0.340 | 0.494 | 0.121 |
| FSV-BG | 0.090 | 0.61 | 0.205 | 0.70 | | | | | | | | | 0.022 | 0.021 | 0.308 | 0.089 |
| FSV-BH | 0.073 | 0.47 | 0.190 | 0.67 | 0.073 | 0.449 | 0.190 | 0.65 | *nq* | 0.019 | *nq* | 0.024 | 0.014 | 0.268 | 0.439 | 0.123 |
| FSV-BI | 0.072 | 0.53 | 0.169 | 0.68 | | | | | | | | | 0.012 | 0.197 | 0.280 | 0.086 |
| FSV-BJ | 0.098 | 0.50 | 0.195 | 0.73 | | | | | | | | | *nq* | 0.163 | 0.242 | 0.082 |
| FSV-BK | | | | | | | | | | | | | | | | |
| FSV-BL | | | | | | | | | | | | | | | | |
| FSV-BM | | | | | | | | | | | | | | | | |
| FSV-BN | 0.103 | 0.60 | 0.204 | 0.56 | 0.077 | 0.560 | 0.189 | 0.52 | 0.002 | 0.013 | 0.003 | 0.014 | 0.006 | 0.194 | 0.245 | 0.065 |
| FSV-BO | 0.048 | 0.44 | 0.101 | 0.41 | | | | | | | | | *nq* | 0.164 | 0.251 | 0.068 |
| FSV-BP | 0.100 | 0.48 | 0.288 | 0.58 | | | | | | | | | | | | |
| FSV-BQ | | | | | | | | | | | | | | | | |
| FSV-BR | | | | | | | | | | | | | | | | |
| FSV-BS | 0.095 | 0.51 | 0.186 | 0.71 | | | | | | | | | 0.020 | 0.248 | 0.377 | 0.060 |
| FSV-BU | 0.071 | 0.49 | 0.181 | 0.63 | | | | | | | | | 0.017 | 0.253 | 0.343 | 0.097 |
| FSV-BV | 0.071 | 0.41 | 0.150 | 0.67 | | | | | | | | | 0.021 | 0.290 | 0.500 | 0.126 |
| FSV-BW | 0.048 | 0.36 | 0.110 | 0.59 | | | | | | | | | 0.013 | 0.230 | 0.420 | 0.102 |
| FSV-BZ | >0.087 | >0.480 | >0.190 | >0.430 | 0.087 | 0.480 | 0.190 | 0.43 | *nd* | *nd* | *nd* | 0.010 | 0.027 | 0.230 | 0.250 | 0.086 |
| FSV-CB | 0.052 | 0.39 | 0.136 | 0.41 | | | | | | | | | 0.013 | 0.159 | 0.230 | 0.067 |
| FSV-CBa | | | | | | | | | | | | | | | | |
| FSV-CC | | | | | | | | | | | | | | | | |
| FSV-CD | 0.060 | 0.42 | 0.160 | 0.61 | | | | | | | | | 0.011 | 0.157 | 0.210 | 0.065 |
| FSV-CE | 0.092 | 0.62 | 0.208 | 0.80 | | | | | | | | | | | | |
| FSV-CF | | | | | | | | | | | | | | | | |
| FSV-CH | 0.059 | 0.50 | 0.167 | 0.68 | | | | | | | | | 0.002 | 0.255 | 0.357 | 0.074 |
| FSV-CI | | | | | | | | | | | | | | | | |
| FSV-CK | 0.081 | 0.38 | 0.129 | 0.61 | | | | | | | | | 0.021 | 0.279 | 0.301 | 0.118 |
| FSV-CL | 0.071 | 0.41 | 0.142 | 0.54 | | | | | | | | | 0.018 | 0.249 | 0.404 | 0.109 |
| FSV-CM | | | | | | | | | | | | | | | | |
| FSV-CN | >0.073 | >0.570 | >0.177 | >0.784 | 0.073 | 0.570 | 0.177 | 0.78 | | | | | *nq* | 0.258 | 0.348 | 0.083 |
| FSV-CP | 0.079 | 0.50 | 0.191 | 0.74 | | | | | | | | | 0.012 | 0.268 | 0.346 | 0.100 |
| FSV-CQ | 0.067 | 0.46 | 0.164 | 0.75 | | | | | | | | | | | | |
| FSV-CR | | | | | | | | | | | | | | | | |
| FSV-CS | 0.071 | 0.57 | 0.188 | 0.70 | 0.064 | 0.523 | 0.169 | 0.64 | *0.007* | *0.045* | *0.019* | *0.054* | 0.013 | 0.305 | 0.425 | 0.113 |
| FSV-CT | 0.077 | 0.50 | 0.186 | 0.75 | | | | | | | | | | | | |
| FSV-CU | 0.066 | 0.44 | 0.138 | 0.59 | | | | | | | | | | | | |
| FSV-CX | 0.060 | 0.39 | 0.180 | 0.48 | | | | | | | | | 0.010 | 0.150 | 0.280 | 0.080 |
| FSV-DB | 0.049 | 0.34 | 0.137 | 0.49 | | | | | | | | | | | | |
| FSV-DF | | | | | | | | | | | | | | | | |
| FSV-DJ | | | | | | | | | | | | | | | | |
| FSV-DK | 0.077 | 0.47 | 0.173 | 0.62 | | | | | | | | | 0.006 | 0.119 | 0.215 | 0.047 |
| FSV-DP | | | | | | | | | | | | | | | | |
| FSV-DR | 0.078 | 0.47 | 0.176 | 0.61 | | | | | | | | | | | | |
| FSV-DW | 0.090 | 0.45 | 0.157 | 0.43 | 0.085 | 0.427 | 0.149 | 0.40 | 0.005 | 0.019 | 0.008 | 0.026 | 0.025 | 0.160 | 0.330 | 0.120 |
| FSV-EM | >0.074 | >0.386 | >0.174 | >0.572 | 0.074 | 0.386 | 0.174 | 0.57 | | | | | 0.028 | 0.156 | 0.277 | 0.294 |
| FSV-ES | >0.072 | >0.476 | >0.172 | >0.472 | 0.072 | 0.476 | 0.172 | 0.47 | | | | | | | | |
| FSV-FG | | | | | | | | | | | | | | | | |
| FSV-FH | | | | | | | | | | | | | | | | |
| n | 30 | 30 | 30 | 30 | 9 | 9 | 9 | 9 | 4 | 5 | 4 | 6 | 22 | 25 | 25 | 25 |
| Min | 0.048 | 0.34 | 0.101 | 0.41 | 0.064 | 0.386 | 0.149 | 0.40 | 0.002 | 0.013 | 0.003 | 0.010 | 0.002 | 0.021 | 0.210 | 0.047 |
| Median | 0.074 | 0.47 | 0.171 | 0.63 | 0.074 | 0.476 | 0.174 | 0.57 | 0.006 | 0.019 | 0.008 | 0.025 | 0.014 | 0.230 | 0.308 | 0.086 |
| Max | 0.103 | 0.62 | 0.288 | 0.80 | 0.087 | 0.570 | 0.190 | 0.78 | 0.007 | 0.045 | 0.019 | 0.054 | 0.028 | 0.340 | 0.500 | 0.294 |
| eSD | 0.020 | 0.07 | 0.031 | 0.09 | 0.004 | 0.069 | 0.007 | 0.11 | | 0.001 | | 0.010 | 0.007 | 0.089 | 0.093 | 0.028 |
| eCV | 27 | 14 | 18 | 14 | 6 | 15 | 4 | 20 | | 8 | | 39 | 49 | 39 | 30 | 33 |
| NISTa | 0.088 | 0.60 | 0.193 | 0.75 | 0.081 | 0.551 | 0.182 | 0.72 | *0.007* | *0.045* | *0.011* | *0.035* | nd | 0.227 | 0.270 | 0.072 |
| NAV | 0.081 | 0.53 | 0.182 | 0.69 | 0.077 | 0.513 | 0.178 | 0.65 | 0.007 | 0.032 | 0.012 | 0.030 | 0.014 | 0.229 | 0.289 | 0.079 |
| NAU | 0.020 | 0.11 | 0.037 | 0.12 | 0.012 | 0.083 | 0.021 | 0.16 | | 0.020 | | 0.021 | 0.006 | 0.073 | 0.098 | 0.031 |

# Round Robin XLII Laboratory Results

All values in µg/mL

| | Total Lycopene | | | | trans-Lycopene | | | | Total β-Cryptoxanthin | | | | Total α-Cryptoxanthin | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Lab | 239 | 240 | 241 | 242 | 239 | 240 | 241 | 242 | 239 | 240 | 241 | 242 | 239 | 240 | 241 | 242 |
| FSV-BA | | | | | 0.289 | 0.32 | 0.340 | 0.303 | 0.095 | 0.118 | 0.074 | 0.165 | | | | |
| FSV-BD | 0.426 | 0.44 | 0.46 | 0.38 | | | | | 0.063 | 0.082 | 0.043 | 0.111 | | | | |
| FSV-BE | | | | | | | | | | | | | | | | |
| FSV-BF | 0.527 | 0.50 | 0.51 | 0.43 | | | | | 0.069 | 0.094 | 0.054 | 0.126 | | | | |
| FSV-BG | 0.556 | 0.60 | 0.66 | 0.50 | 0.314 | 0.37 | 0.419 | 0.286 | 0.089 | 0.124 | 0.082 | 0.173 | | | | |
| FSV-BH | 0.563 | 0.57 | 0.62 | 0.54 | | | | | 0.101 | 0.141 | 0.086 | 0.184 | | | | |
| FSV-BI | 0.436 | 0.47 | 0.45 | 0.41 | | | | | 0.087 | 0.128 | 0.073 | 0.175 | | | | |
| FSV-BJ | 0.630 | 0.65 | 0.70 | 0.63 | | | | | | | | | | | | |
| FSV-BK | | | | | | | | | | | | | | | | |
| FSV-BL | | | | | | | | | | | | | | | | |
| FSV-BM | | | | | | | | | | | | | | | | |
| FSV-BN | 0.552 | 0.65 | 0.63 | 0.54 | 0.304 | 0.42 | 0.417 | 0.324 | 0.078 | 0.114 | 0.063 | 0.142 | 0.034 | 0.029 | 0.026 | 0.032 |
| FSV-BO | 0.496 | 0.48 | 0.39 | 0.42 | | | | | 0.066 | 0.086 | 0.057 | 0.114 | | | | |
| FSV-BP | 0.355 | 0.37 | 0.41 | 0.37 | | | | | 0.141 | 0.135 | 0.113 | 0.204 | | | | |
| FSV-BQ | | | | | | | | | | | | | | | | |
| FSV-BR | | | | | | | | | | | | | | | | |
| FSV-BS | 0.509 | 0.52 | 0.56 | 0.43 | 0.215 | 0.26 | 0.302 | 0.182 | 0.071 | 0.098 | 0.058 | 0.124 | | | | |
| FSV-BU | 0.431 | 0.46 | 0.45 | 0.37 | | | | | 0.078 | 0.109 | 0.057 | 0.142 | | | | |
| FSV-BV | 0.490 | 0.45 | 0.49 | 0.43 | | | | | 0.064 | 0.089 | 0.049 | 0.122 | | | | |
| FSV-BW | 0.410 | 0.54 | 0.50 | 0.64 | | | | | | | | | | | | |
| FSV-BZ | | | | | 0.480 | 0.56 | 0.600 | 0.410 | | | | | | | | |
| FSV-CB | 0.092 | 0.11 | 0.12 | 0.10 | | | | | 0.037 | 0.073 | 0.036 | 0.083 | | | | |
| FSV-CBa | | | | | | | | | | | | | | | | |
| FSV-CC | | | | | | | | | | | | | | | | |
| FSV-CD | 0.451 | 0.56 | 0.53 | 0.55 | | | | | 0.084 | 0.112 | 0.071 | 0.142 | 0.032 | 0.036 | 0.029 | 0.039 |
| FSV-CE | | | | | | | | | | | | | | | | |
| FSV-CF | | | | | | | | | | | | | | | | |
| FSV-CH | 0.376 | 0.39 | 0.43 | 0.34 | | | | | | | | | | | | |
| FSV-CI | | | | | | | | | | | | | | | | |
| FSV-CK | 0.354 | 0.47 | 0.30 | 0.40 | | | | | 0.100 | 0.122 | 0.077 | 0.160 | 0.051 | 0.051 | 0.040 | 0.057 |
| FSV-CL | 0.419 | 0.51 | 0.49 | 0.48 | | | | | 0.053 | 0.073 | 0.047 | 0.099 | 0.040 | 0.037 | 0.036 | 0.037 |
| FSV-CM | | | | | | | | | | | | | | | | |
| FSV-CN | 0.400 | 0.50 | 0.44 | 0.40 | | | | | 0.067 | 0.116 | 0.074 | 0.176 | | | | |
| FSV-CP | 0.259 | 0.29 | 0.30 | 0.22 | | | | | 0.085 | 0.107 | 0.066 | 0.138 | | | | |
| FSV-CQ | | | | | | | | | | | | | | | | |
| FSV-CR | | | | | | | | | | | | | | | | |
| FSV-CS | 0.483 | 0.59 | 0.53 | 0.50 | | | | | 0.070 | 0.100 | 0.058 | 0.120 | | | | |
| FSV-CT | | | | | 0.310 | 0.37 | 0.421 | 0.285 | 0.062 | 0.084 | 0.045 | 0.099 | | | | |
| FSV-CU | | | | | | | | | | | | | | | | |
| FSV-CX | | | | | 0.610 | 0.58 | 0.660 | 0.560 | 0.070 | 0.140 | 0.060 | 0.180 | | | | |
| FSV-DB | 0.430 | 0.51 | 0.52 | 0.43 | | | | | 0.090 | 0.124 | 0.075 | 0.165 | | | | |
| FSV-DF | | | | | | | | | | | | | | | | |
| FSV-DJ | | | | | | | | | | | | | | | | |
| FSV-DK | 0.471 | 0.37 | 0.40 | 0.31 | | | | | | | | | | | | |
| FSV-DP | | | | | | | | | | | | | | | | |
| FSV-DR | | | | | | | | | | | | | | | | |
| FSV-DW | 0.155 | 0.15 | 0.16 | 0.13 | 0.099 | 0.10 | 0.111 | 0.082 | 0.071 | 0.101 | 0.055 | 0.132 | | | | |
| FSV-EM | 0.671 | 0.52 | 0.80 | 0.61 | | | | | 0.065 | 0.111 | 0.060 | 0.148 | | | | |
| FSV-ES | 0.365 | 0.40 | 0.44 | 0.36 | 0.230 | 0.28 | 0.314 | 0.227 | 0.073 | 0.111 | 0.057 | 0.144 | | | | |
| FSV-FG | | | | | | | | | | | | | | | | |
| FSV-FH | | | | | | | | | | | | | | | | |
| n | 26 | 26 | 26 | 26 | 9 | 9 | 9 | 9 | 25 | 25 | 25 | 25 | 4 | 4 | 4 | 4 |
| Min | 0.092 | 0.11 | 0.12 | 0.10 | 0.099 | 0.10 | 0.111 | 0.082 | 0.037 | 0.073 | 0.036 | 0.083 | 0.032 | 0.029 | 0.026 | 0.032 |
| Median | 0.434 | 0.49 | 0.47 | 0.43 | 0.304 | 0.37 | 0.417 | 0.286 | 0.071 | 0.111 | 0.060 | 0.142 | 0.037 | 0.037 | 0.033 | 0.038 |
| Max | 0.671 | 0.65 | 0.80 | 0.64 | 0.610 | 0.58 | 0.660 | 0.560 | 0.141 | 0.141 | 0.113 | 0.204 | 0.051 | 0.051 | 0.040 | 0.057 |
| eSD | 0.097 | 0.10 | 0.09 | 0.11 | 0.110 | 0.13 | 0.153 | 0.087 | 0.012 | 0.019 | 0.016 | 0.034 | | | | |
| eCV | 22 | 20 | 19 | 25 | 36 | 36 | 37 | 31 | 17 | 17 | 27 | 24 | | | | |
| NISTa | | | | | | | | | 0.065 | 0.144 | 0.072 | 0.177 | | | | |
| NAV | 0.434 | 0.49 | 0.47 | 0.43 | 0.304 | 0.37 | 0.417 | 0.286 | 0.068 | 0.128 | 0.066 | 0.160 | | | | |
| NAU | 0.097 | 0.11 | 0.10 | 0.10 | 0.063 | 0.11 | 0.086 | 0.072 | 0.018 | 0.035 | 0.017 | 0.041 | | | | |

# Round Robin XLII Laboratory Results

All values in µg/mL

| | Total Lutein | | | | Total Zeaxanthin | | | | Total Lutein&Zeaxanthin | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Lab | 239 | 240 | 241 | 242 | 239 | 240 | 241 | 242 | 239 | 240 | 241 | 242 |
| FSV-BA | | | | | | | | | 0.152 | 0.53 | 0.196 | 0.72 |
| FSV-BD | 0.053 | 0.225 | 0.048 | 0.40 | 0.029 | 0.172 | 0.102 | 0.062 | *0.082* | *0.40* | *0.150* | *0.46* |
| FSV-BE | | | | | | | | | | | | |
| FSV-BF | | | | | | | | | 0.092 | 0.43 | 0.141 | 0.49 |
| FSV-BG | 0.069 | 0.225 | 0.065 | 0.36 | 0.022 | 0.068 | 0.017 | 0.110 | *0.091* | *0.29* | *0.082* | *0.47* |
| FSV-BH | 0.059 | 0.239 | 0.045 | 0.44 | 0.028 | 0.129 | 0.104 | 0.050 | *0.087* | *0.37* | *0.149* | *0.49* |
| FSV-BI | 0.061 | 0.245 | 0.045 | 0.43 | 0.028 | 0.197 | 0.112 | 0.050 | *0.089* | *0.44* | *0.157* | *0.48* |
| FSV-BJ | | | | | | | | | | | | |
| FSV-BK | | | | | | | | | | | | |
| FSV-BL | | | | | | | | | | | | |
| FSV-BM | | | | | | | | | | | | |
| FSV-BN | 0.064 | 0.211 | 0.034 | 0.37 | 0.040 | 0.349 | 0.140 | 0.083 | 0.083 | 0.47 | 0.158 | 0.42 |
| FSV-BO | | | | | | | | | 0.087 | 0.45 | 0.172 | 0.39 |
| FSV-BP | | | | | | | | | | | | |
| FSV-BQ | | | | | | | | | | | | |
| FSV-BR | | | | | | | | | | | | |
| FSV-BS | | | | | | | | | 0.091 | 0.45 | 0.118 | 0.37 |
| FSV-BU | | | | | | | | | 0.103 | 0.54 | 0.192 | 0.57 |
| FSV-BV | | | | | | | | | 0.091 | 0.33 | 0.137 | 0.37 |
| FSV-BW | | | | | | | | | | | | |
| FSV-BZ | 0.078 | 0.270 | 0.144 | 0.43 | | | | | | | | |
| FSV-CB | 0.047 | 0.243 | 0.045 | 0.40 | 0.028 | 0.264 | 0.143 | *nd* | *0.075* | *0.51* | *0.188* | *0.40* |
| FSV-CBa | | | | | | | | | | | | |
| FSV-CC | | | | | | | | | | | | |
| FSV-CD | | | | | | | | | 0.072 | 0.35 | 0.133 | 0.34 |
| FSV-CE | | | | | | | | | | | | |
| FSV-CF | | | | | | | | | | | | |
| FSV-CH | | | | | | | | | | | | |
| FSV-CI | | | | | | | | | | | | |
| FSV-CK | | | | | | | | | 0.165 | 0.30 | 0.199 | 0.56 |
| FSV-CL | | | | | | | | | 0.066 | 0.26 | 0.103 | 0.29 |
| FSV-CM | | | | | | | | | | | | |
| FSV-CN | 0.060 | 0.198 | 0.046 | | 0.039 | 0.302 | 0.158 | | 0.099 | 0.50 | 0.204 | 0.47 |
| FSV-CP | | | | | | | | | 0.110 | 0.41 | 0.179 | 0.51 |
| FSV-CQ | | | | | | | | | | | | |
| FSV-CR | | | | | | | | | | | | |
| FSV-CS | | | | | | | | | 0.091 | 0.39 | 0.133 | 0.42 |
| FSV-CT | 0.076 | 0.265 | 0.064 | 0.45 | 0.024 | 0.170 | 0.098 | 0.047 | *0.100* | *0.44* | *0.162* | *0.50* |
| FSV-CU | | | | | | | | | | | | |
| FSV-CX | 0.030 | 0.180 | 0.020 | 0.34 | 0.010 | 0.130 | 0.060 | 0.020 | *0.040* | *0.31* | *0.080* | *0.36* |
| FSV-DB | | | | | | | | | 0.087 | 0.41 | 0.167 | 0.40 |
| FSV-DF | | | | | | | | | | | | |
| FSV-DJ | | | | | | | | | | | | |
| FSV-DK | 0.095 | 0.317 | 0.121 | 0.40 | | | | | | | | |
| FSV-DP | | | | | | | | | | | | |
| FSV-DR | | | | | | | | | | | | |
| FSV-DW | 0.068 | >0.200 | 0.061 | >0.200 | 0.049 | 0.191 | 0.145 | 0.073 | *0.117* | *>0.391* | *0.206* | *>0.273* |
| FSV-EM | | | | | | | | | 0.127 | 0.43 | 0.211 | 0.56 |
| FSV-ES | 0.062 | 0.254 | 0.047 | 0.44 | 0.032 | 0.167 | 0.112 | 0.051 | *0.094* | *0.42* | *0.159* | *0.49* |
| FSV-FG | | | | | | | | | | | | |
| FSV-FH | | | | | | | | | | | | |
| n | 13 | 12 | 13 | 11 | 11 | 11 | 11 | 9 | 24 | 23 | 24 | 23 |
| Min | 0.030 | 0.180 | 0.020 | 0.34 | 0.010 | 0.068 | 0.017 | 0.020 | 0.040 | 0.26 | 0.080 | 0.29 |
| Median | 0.062 | 0.241 | 0.047 | 0.40 | 0.028 | 0.172 | 0.112 | 0.051 | 0.091 | 0.42 | 0.158 | 0.47 |
| Max | 0.095 | 0.317 | 0.144 | 0.45 | 0.049 | 0.349 | 0.158 | 0.110 | 0.165 | 0.54 | 0.211 | 0.72 |
| eSD | 0.010 | 0.030 | 0.019 | 0.05 | 0.007 | 0.062 | 0.042 | 0.017 | 0.013 | 0.07 | 0.038 | 0.11 |
| eCV | 17 | 12 | 40 | 12 | 23 | 36 | 37 | 33 | 14 | 17 | 24 | 24 |
| NISTa | 0.061 | 0.299 | 0.052 | 0.53 | 0.031 | 0.226 | 0.129 | 0.053 | *0.092* | *0.53* | *0.181* | *0.58* |
| NAV | 0.061 | 0.270 | 0.049 | 0.46 | 0.029 | 0.199 | 0.120 | 0.052 | 0.091 | 0.47 | 0.169 | 0.53 |
| NAU | 0.014 | 0.068 | 0.015 | 0.13 | 0.008 | 0.072 | 0.035 | 0.017 | 0.020 | 0.12 | 0.042 | 0.13 |

# Round Robin XLII Laboratory Results

## Analytes Reported By One Laboratory

Values in µg/mL

| Analyte | Code | 239 | 240 | 241 | 242 |
|---|---|---|---|---|---|
| Coenzyme Q10 | FSV-CH | 0.441 | 0.376 | 0.339 | 0.421 |
| Phytoene | FSV-CL | 0.071 | 0.064 | 0.058 | 0.078 |
| Phytofluene | FSV-CL | 0.042 | 0.028 | 0.032 | 0.021 |
| Total Carotenoids | FSV-EM | 1.01 | 2.26 | 1.41 | 2.74 |
| trans-α-Carotene | FSV-ES | 0.021 | 0.235 | 0.336 | 0.092 |

## Table Legend

| Term | Definition |
|---|---|
| N | Number of (non-NIST) quantitative values reported for this analyte |
| Min | Minimum (non-NIST) quantitative value reported |
| Median | Median (non-NIST) quantitative value reported |
| Max | Maximum (non-NIST) quantitative value reported |
| eSD | Estimated standard deviation, calculated from the median absolute deviation from the median of the non-NIST results |
| eCV | Coefficient of Variation for (non-NIST) results: 100*eSD/Median |
| NAV | NIST Assigned Value, our estimate of the "true" analyte concentration |
| NAU | NIST Assigned Uncertainty, our estimate of the total (serum hetergeniety and inter- and intra-laboratory) standard deviation<br>For details on how we assign these quantities, see the "Analysis of Results." |
| *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 great that x |
| ! | Discrepant value: heterogeneous serum, damaged sample, malfunction, etc. |
| *italics* | Not explicitly reported but calculated by NIST from reported values |

# Round Robin XLII Laboratory Results

## Comparability Summary

| Lab | R | aT | gT | bC | tbC |
|---|---|---|---|---|---|
| FSV-BA | A | A | A | A | |
| FSV-BD | A | B | | | |
| FSV-BE | A | A | A | A | |
| FSV-BF | B | A | A | B | |
| FSV-BG | A | A | A | A | |
| FSV-BH | A | A | B | A | |
| FSV-BI | A | A | A | A | A |
| FSV-BJ | A | A | B | A | A |
| FSV-BK | A | A | | | |
| FSV-BL | A | B | | | B |
| FSV-BM | A | A | | | |
| FSV-BN | B | B | B | B | |
| FSV-BO | B | C | | C | |
| FSV-BP | C | C | | C | |
| FSV-BQ | B | A | | | |
| FSV-BR | A | | | | |
| FSV-BS | A | | | A | |
| FSV-BU | C | A | A | A | |
| FSV-BV | B | B | B | B | |
| FSV-BW | B | B | B | B | |
| FSV-BZ | | C | C | | |
| FSV-CB | C | C | | C | |
| FSV-CBa | C | A | | | A |
| FSV-CC | B | A | | | |
| FSV-CD | B | A | A | B | |
| FSV-CE | A | B | | A | |
| FSV-CF | A | B | | | |
| FSV-CH | C | C | C | B | |
| FSV-CI | B | B | | | |
| FSV-CK | C | C | C | B | |
| FSV-CL | C | B | C | B | |
| FSV-CM | | C | | | |
| FSV-CN | A | B | B | | |
| FSV-CP | | A | B | A | |
| FSV-CQ | A | A | | A | |
| FSV-CR | B | C | | | |
| FSV-CS | B | A | B | A | |
| FSV-CT | B | C | | A | A |
| FSV-CU | B | B | | B | |
| FSV-CX | A | B | B | B | |
| FSV-DB | B | B | | B | |
| FSV-DF | B | | | | B |
| FSV-DJ | A | C | | | |
| FSV-DK | B | A | B | A | B |
| FSV-DP | A | | | | |
| FSV-DR | A | A | | A | |
| FSV-DW | A | B | | C | |
| FSV-EM | C | B | | | A |
| FSV-ES | A | A | | | |
| FSV-FG | C | B | | | B |
| NISTa | A | A | A | A | A |
| n | 47 | 46 | 21 | 30 | 9 |

| Label | Definition |
|---|---|
| Lab | Participant code |
| R | "Standard Score" for Retinol |
| aT | "Standard Score" for α-Tocopherol |
| gT | "Standard Score" for γ-Tocopherol |
| bC | "Standard Score" for Total β-Carotene |
| tbC | "Standard Score" for trans-β-Carotene |
| n | number of laboratories providing data for this analyte for at least one serum |

"Standard Score"

Given that our knowledge of the shapre, location, and width of the measurement distributions is approximate and that a limited number of laboratories are involved, we summarize comparability with the following 3-level "Standard Score" (StS)…

| StS | Definition | |
|---|---|---|
| A | All StV within ±*t*(1-0.683,n-1) | (i.e., ±1 SD) |
| B | All StV within ±*t*(1-0.954,n-1) | (i.e., ±2 SD) |
| C | At least one StV > ±*t*(1-0.954,n-1) | (i.e., ≥2 SD) |

where:

| | |
|---|---|
| StV | Standardized Value, the distance in standard deviation units your value is from the "true" concentration: StV = (your value - NAV) / NAU |
| NAV | NIST Assigned Value, our estimate of the "true" analyte concentration |
| NAU | NIST Assigned Uncertainty, our estimate of the total (serum heterogeneity and inter- and intra-laboratory measurement) standard deviation |
| *t*(1-α,n-1) | Two-tailed Student's t for coverage of ±1 and ±2 NAU about NAV assuming a normal population of size n |

| StS | % Observed | | | | | Expected |
|---|---|---|---|---|---|---|
| A | 47 | 43 | 33 | 50 | 56 | 68.3 % |
| B | 34 | 35 | 48 | 37 | 44 | 27.2 % |
| C | 19 | 22 | 19 | 13 | 0 | 4.6 % |

These are the observed and normal-population-expected proportions of each Standard Score (StS), based upon each laboratory's largest StV for the four sera.

## Appendix D. Representative "Individualized Report" for RR42

Each participant in RR42 received an "Individualized Report" reflecting their reported results. Each report included a detailed analysis of the results they reported for the following analytes:

- Total Retinol
- α-Tocopherol
- γ-Tocopherol
- Total β-Carotene
- *trans*-β-Carotene
- Total α-Carotene
- *trans*-Lycopene

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

**Note**

The design file and program used to generate the original RR42 "Individualized Reports" are no longer available. The following pages were produced using the 2013 version of the program (a linear descendant of that used in 1998) and a re-created design file.

# Individualized Round Robin XLII Report: FSV-BA

## Summary

| | Serum 239 | | | Serum 240 | | | Serum 241 | | | Serum 242 | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Analyte | You | NAV | n | You | NAV | n | You | NAV | n | You | NAV | n |
| Retinol | 0.501 | 0.520 | 47 | 0.727 | 0.698 | 47 | 0.432 | 0.426 | 47 | 0.934 | 0.934 | 47 |
| Retinyl Palmitate | 0.059 | 0.054 | 8 | 0.070 | 0.068 | 9 | 0.056 | 0.054 | 7 | 0.067 | 0.067 | 9 |
| α-Tocopherol | 7.89 | 7.50 | 46 | 13.8 | 14.2 | 46 | 6.0 | 6.5 | 46 | 20.60 | 20.45 | 46 |
| γ-Tocopherol | 1.56 | 1.64 | 21 | 2.84 | 2.86 | 21 | 3.32 | 3.54 | 21 | 1.78 | 1.80 | 21 |
| Total β-Carotene | 0.088 | 0.081 | 30 | 0.454 | 0.533 | 30 | 0.180 | 0.182 | 30 | 0.625 | 0.689 | 30 |
| trans-β-Carotene | 0.081 | 0.077 | 9 | 0.436 | 0.513 | 9 | 0.172 | 0.178 | 9 | 0.598 | 0.645 | 9 |
| Total cis-β-Carotene | 0.007 | 0.007 | 4 | 0.018 | 0.032 | 5 | 0.008 | 0.012 | 4 | 0.027 | 0.030 | 6 |
| Total α-Carotene | 0.013 | 0.014 | 22 | 0.153 | 0.229 | 25 | 0.235 | 0.289 | 25 | 0.077 | 0.079 | 25 |
| trans-Lycopene | 0.289 | 0.304 | 9 | 0.323 | 0.365 | 9 | 0.340 | 0.417 | 9 | 0.303 | 0.286 | 9 |
| Total β-Cryptoxanthin | 0.095 | 0.068 | 25 | 0.118 | 0.128 | 25 | 0.074 | 0.066 | 25 | 0.165 | 0.160 | 25 |
| Total Lutein&Zeaxanthin | 0.152 | 0.091 | 24 | 0.534 | 0.473 | 23 | 0.196 | 0.169 | 24 | 0.719 | 0.526 | 23 |

D2

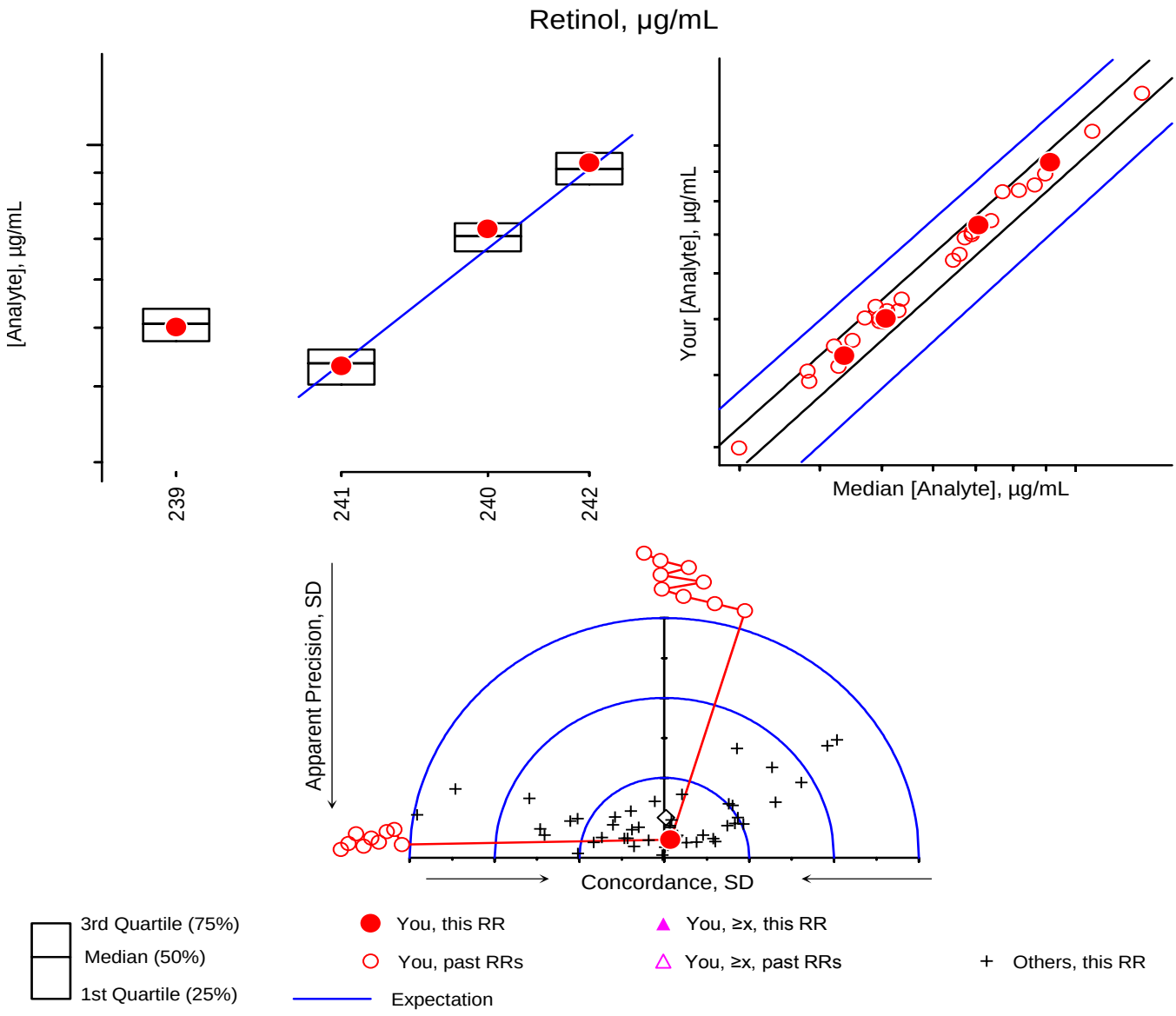
You : Your reported values for the listed analytes (micrograms/milliliter)
NAV : NIST Assigned Values, here equal to this RR's median
n : Number of non-NIST laboratories reporting quantitative values for this analyte in this serum

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

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

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

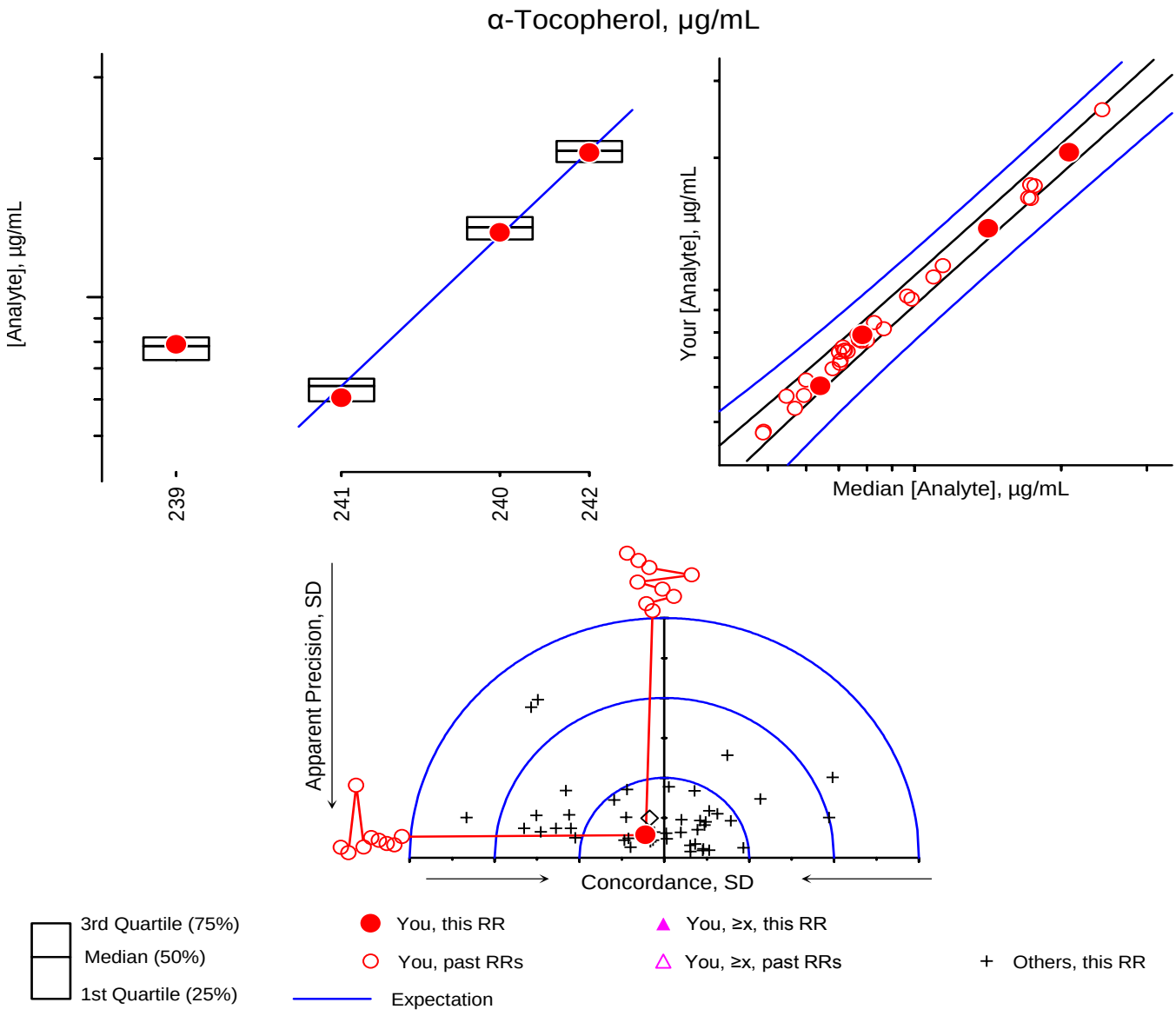
# Individualized RR XLII Report: FSV-BA




For the construction and interpretation of these plots, see the Final Report for "Round Robin" XL, August 1997

| Serum | Comments | History |
|---|---|---|
| #239 | Lyophilized, native, multi-donor | New |
| #241 | Lyophilized, prepared from the Serum 239 serum pool, augmented with: retinyl palmitate, γ- and δ-tocopherol, trans-α- and β-carotene, zeaxanthin, and trans-lycopene | New |
| #242 | Lyophilized, prepared from the Serum 239 serum pool, augmented with: retinol, retinyl palmitate, α- and δ-tocopherol, trans-α- and β-carotene, lutein, and trans-β-cryptoxanthin | New |
| #239 | Lyophilized, prepared as a 1;1 by volume blend of the Sera 241 and 242 serum pools | New |

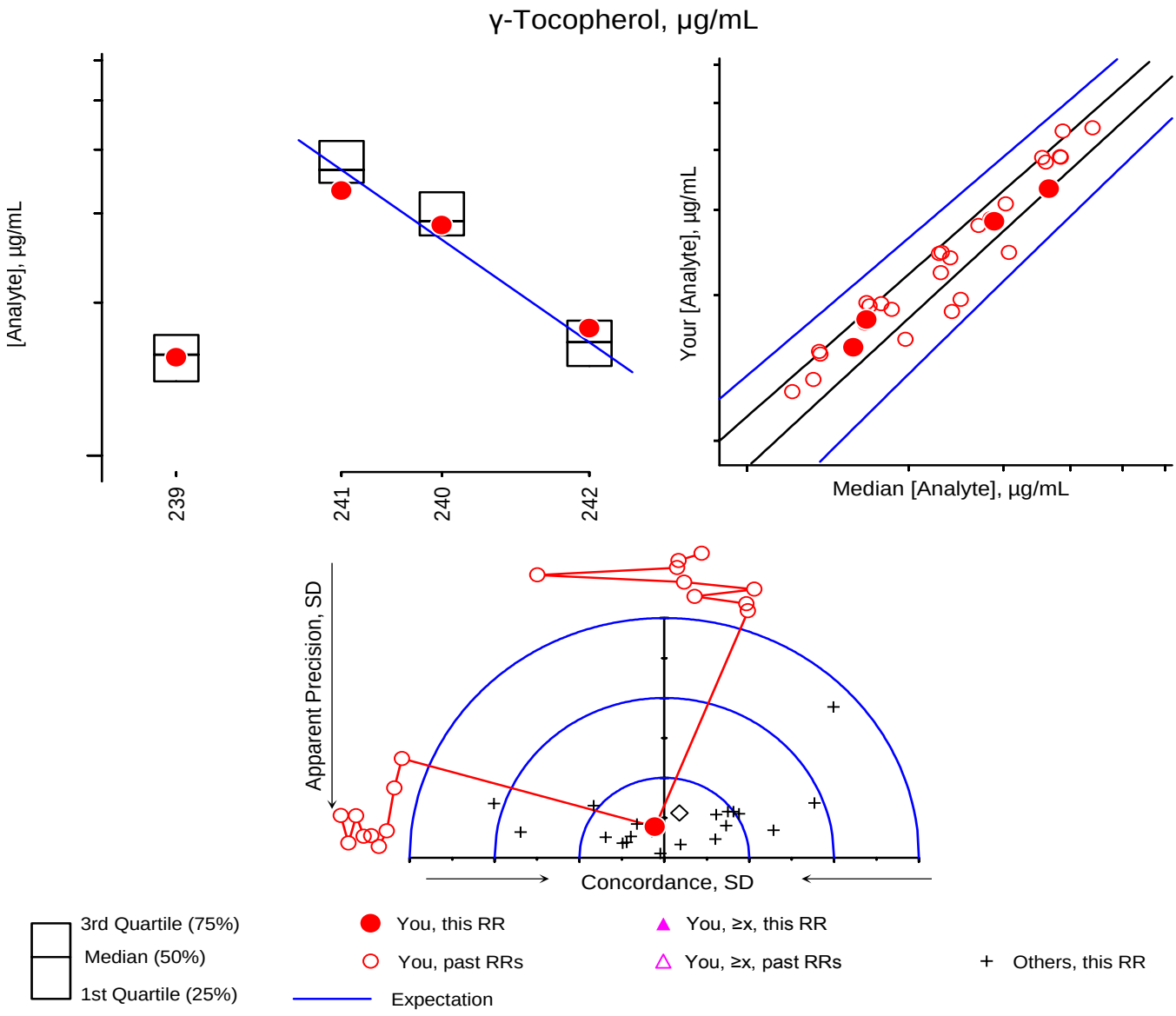
# Individualized RR XLII Report: FSV-BA




For the construction and interpretation of these plots, see the Final Report for "Round Robin" XL, August 1997

| Serum | Comments | History |
|---|---|---|
| #239 | Lyophilized, native, multi-donor | New |
| #241 | Lyophilized, prepared from the Serum 239 serum pool, augmented with: retinyl palmitate, γ- and δ-tocopherol, trans-α- and β-carotene, zeaxanthin, and trans-lycopene | New |
| #242 | Lyophilized, prepared from the Serum 239 serum pool, augmented with: retinol, retinyl palmitate, α- and δ-tocopherol, trans-α- and β-carotene, lutein, and trans-β-cryptoxanthin | New |
| #239 | Lyophilized, prepared as a 1;1 by volume blend of the Sera 241 and 242 serum pools | New |

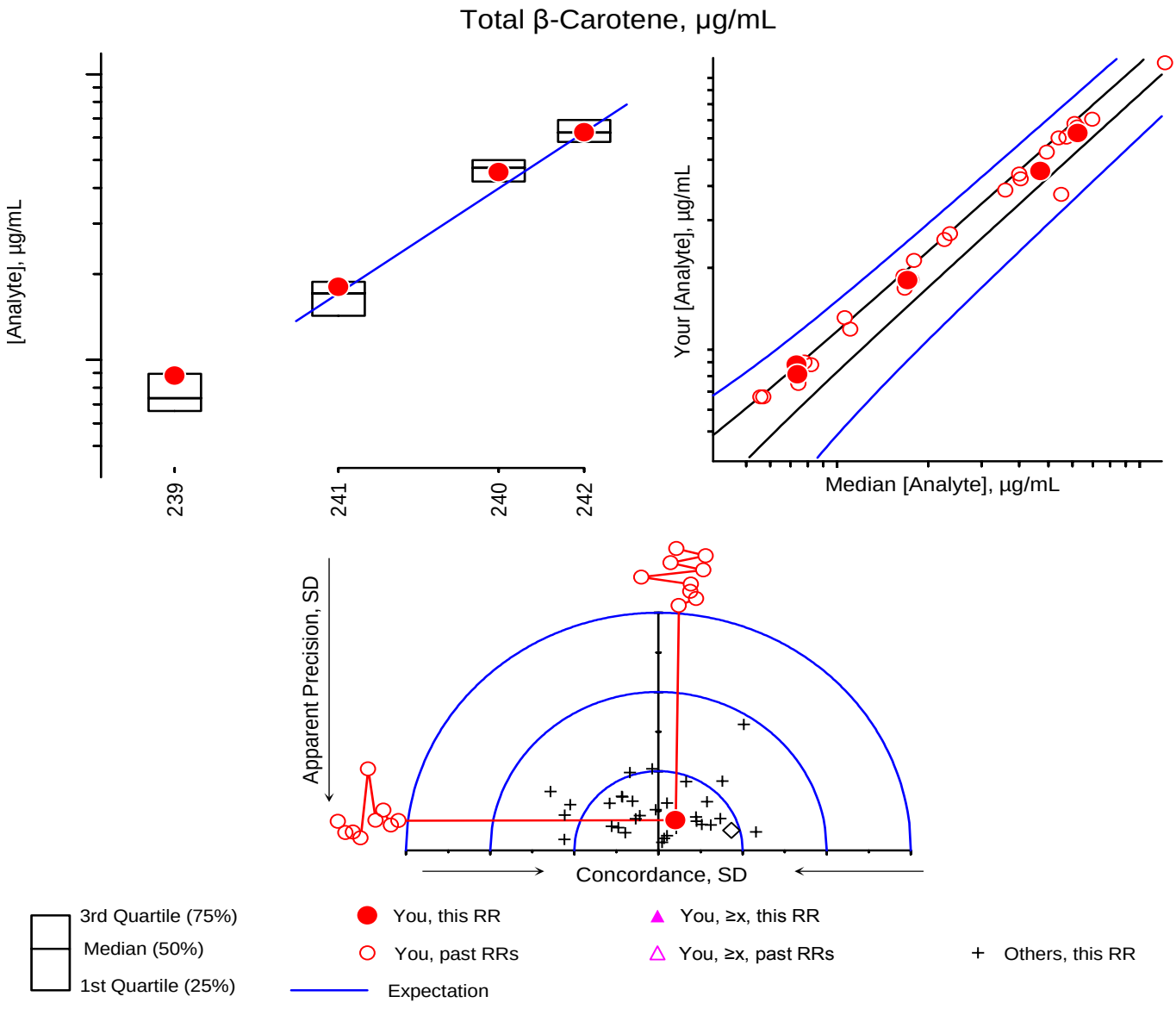
# Individualized RR XLII Report: FSV-BA




For the construction and interpretation of these plots, see the Final Report for "Round Robin" XL, August 1997

| Serum | Comments | History |
|---|---|---|
| #239 | Lyophilized, native, multi-donor | New |
| #241 | Lyophilized, prepared from the Serum 239 serum pool, augmented with: retinyl palmitate, γ- and δ-tocopherol, trans-α- and β-carotene, zeaxanthin, and trans-lycopene | New |
| #242 | Lyophilized, prepared from the Serum 239 serum pool, augmented with: retinol, retinyl palmitate, α- and δ-tocopherol, trans-α- and β-carotene, lutein, and trans-β-cryptoxanthin | New |
| #239 | Lyophilized, prepared as a 1;1 by volume blend of the Sera 241 and 242 serum pools | New |

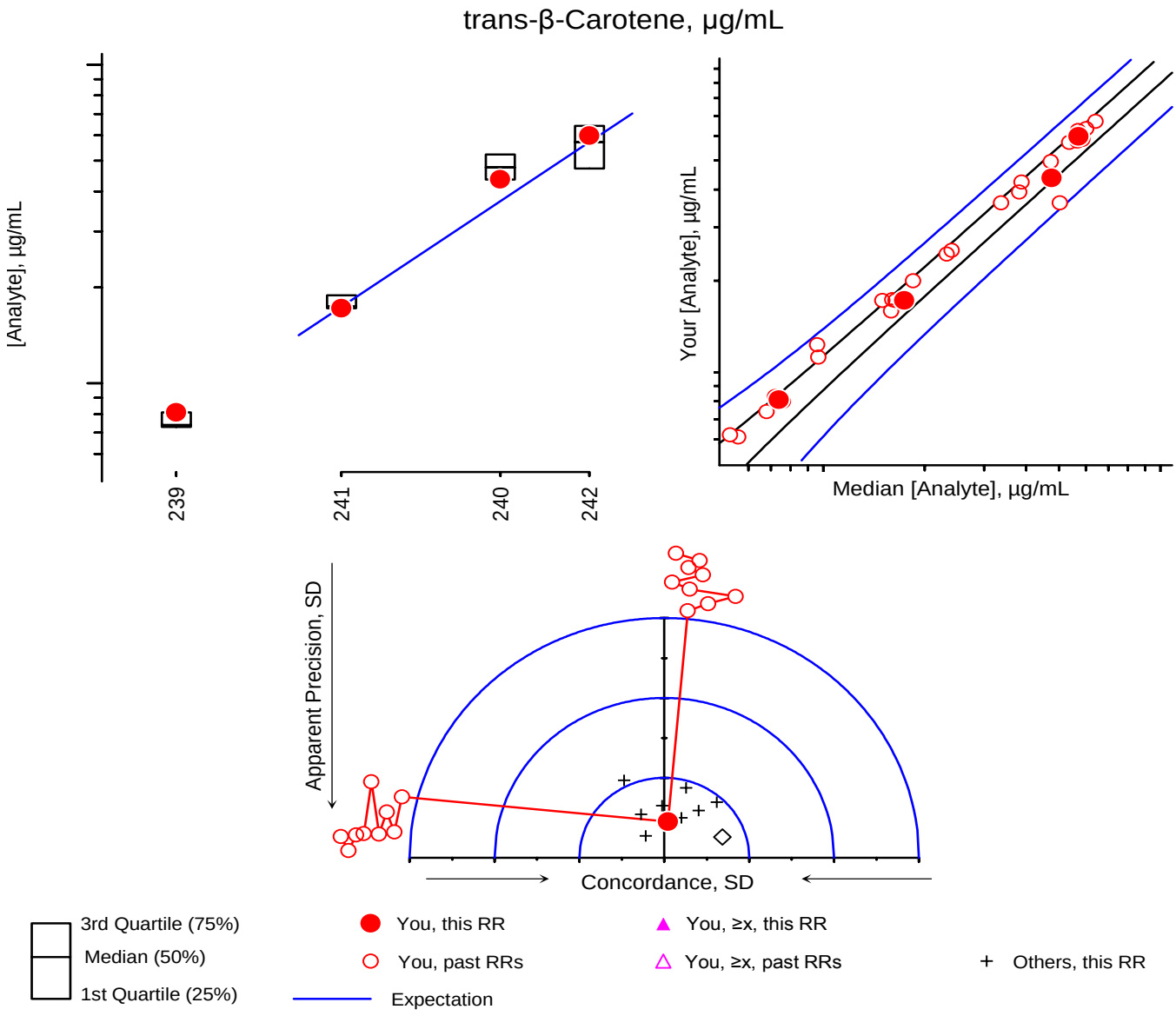
# Individualized RR XLII Report: FSV-BA




For the construction and interpretation of these plots, see the Final Report for "Round Robin" XL, August 1997

| Serum | Comments | History |
|---|---|---|
| #239 | Lyophilized, native, multi-donor | New |
| #241 | Lyophilized, prepared from the Serum 239 serum pool, augmented with: retinyl palmitate, γ- and δ-tocopherol, trans-α- and β-carotene, zeaxanthin, and trans-lycopene | New |
| #242 | Lyophilized, prepared from the Serum 239 serum pool, augmented with: retinol, retinyl palmitate, α- and δ-tocopherol, trans-α- and β-carotene, lutein, and trans-β-cryptoxanthin | New |
| #239 | Lyophilized, prepared as a 1;1 by volume blend of the Sera 241 and 242 serum pools | New |

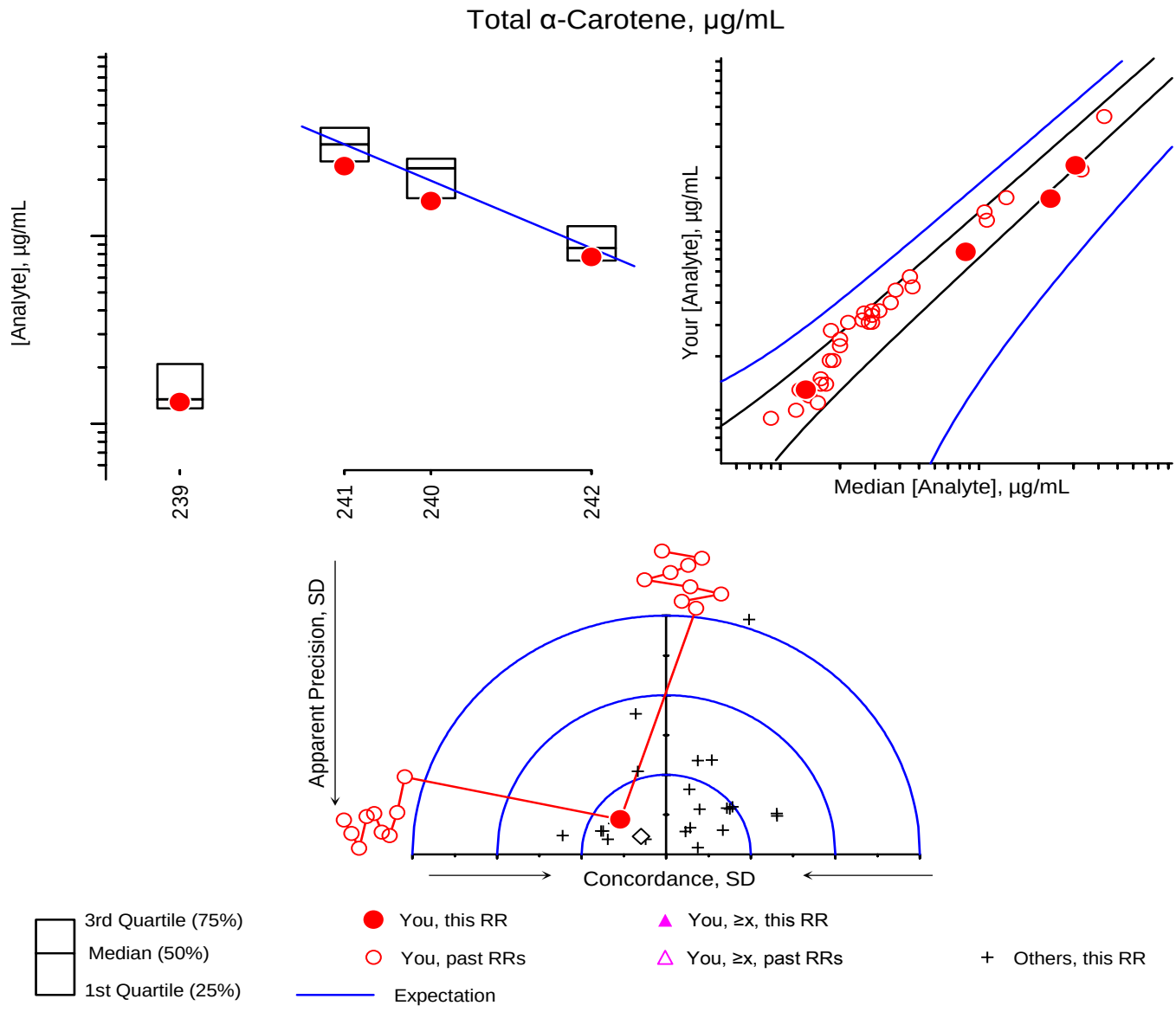
# Individualized RR XLII Report: FSV-BA




For the construction and interpretation of these plots, see the Final Report for "Round Robin" XL, August 1997

| Serum | Comments | History |
|---|---|---|
| #239 | Lyophilized, native, multi-donor | New |
| #241 | Lyophilized, prepared from the Serum 239 serum pool, augmented with: retinyl palmitate, γ- and δ-tocopherol, trans-α- and β-carotene, zeaxanthin, and trans-lycopene | New |
| #242 | Lyophilized, prepared from the Serum 239 serum pool, augmented with: retinol, retinyl palmitate, α- and δ-tocopherol, trans-α- and β-carotene, lutein, and trans-β-cryptoxanthin | New |
| #239 | Lyophilized, prepared as a 1;1 by volume blend of the Sera 241 and 242 serum pools | New |

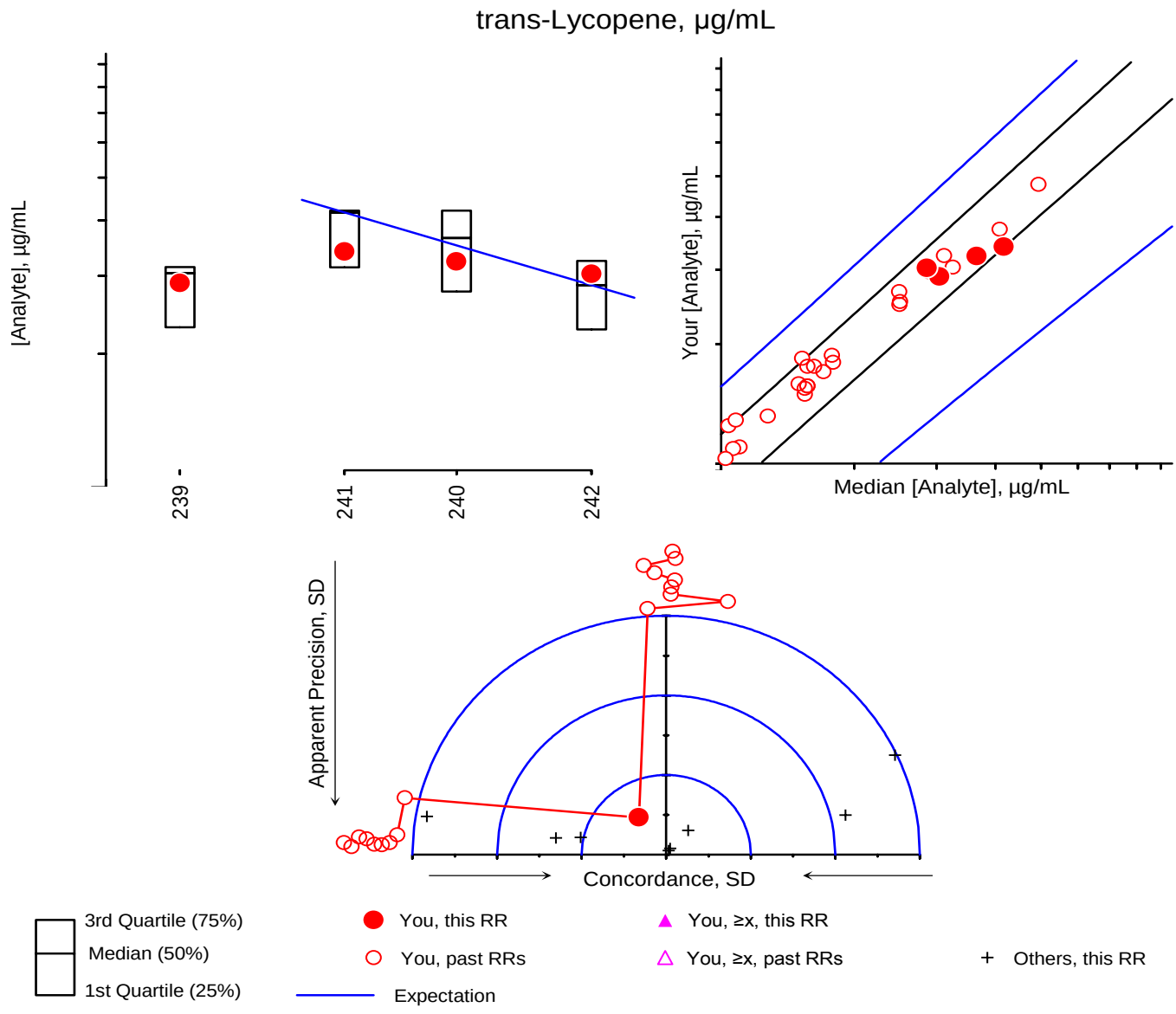
# Individualized RR XLII Report: FSV-BA




For the construction and interpretation of these plots, see the Final Report for "Round Robin" XL, August 1997

| Serum | Comments | History |
|---|---|---|
| #239 | Lyophilized, native, multi-donor | New |
| #241 | Lyophilized, prepared from the Serum 239 serum pool, augmented with: retinyl palmitate, γ- and δ-tocopherol, trans-α- and β-carotene, zeaxanthin, and trans-lycopene | New |
| #242 | Lyophilized, prepared from the Serum 239 serum pool, augmented with: retinol, retinyl palmitate, α- and δ-tocopherol, trans-α- and β-carotene, lutein, and trans-β-cryptoxanthin | New |
| #239 | Lyophilized, prepared as a 1;1 by volume blend of the Sera 241 and 242 serum pools | New |

# Individualized RR XLII Report: FSV-BA




For the construction and interpretation of these plots, see the Final Report for "Round Robin" XL, August 1997

| Serum | Comments | History |
|---|---|---|
| #239 | Lyophilized, native, multi-donor | New |
| #241 | Lyophilized, prepared from the Serum 239 serum pool, augmented with: retinyl palmitate, γ- and δ-tocopherol, trans-α- and β-carotene, zeaxanthin, and trans-lycopene | New |
| #242 | Lyophilized, prepared from the Serum 239 serum pool, augmented with: retinol, retinyl palmitate, α- and δ-tocopherol, trans-α- and β-carotene, lutein, and trans-β-cryptoxanthin | New |
| #239 | Lyophilized, prepared as a 1;1 by volume blend of the Sera 241 and 242 serum pools | New |

## Appendix E. Shipping Package Inserts for RR43

The following two items were included in each package shipped to an RR43 participant:

- Cover letter
- Datasheet

The cover letter and datasheet were enclosed in a sealed waterproof bag along with the samples themselves.

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

May 4, 1998

Dear Colleague:

Enclosed is the set of samples for the second quality assurance round robin exercise (Round Robin XLIII) for FY98. You will find one vial of each of four lyophilized serum samples for analysis along with a form for reporting your results. When reporting your results, please submit one value for each analyte for a given serum sample. If an obtained value is below your limit of quantitation, please indicate this result on the form by using NQ *(Not Quantitated)*. For analytes not measured, please leave a blank. Results are due to NIST by June 22, 1998. Results received two weeks after the due date will not be included in the summary report for this round robin study. Written feedback concerning the study will be provided around July 31.

Samples should be reconstituted with 1.0 mL of HPLC-grade water or equivalent. We recommend that dissolution be facilitated with 3 to 5 min agitation in an ultrasonic bath or at least 30 min at room temperature with intermittent swirling. (CAUTION: Vigorous shaking will cause foaming and possibly interfere with accurate measurement. The rubber stopper contains phthalate esters that will leach into the sample upon intermittent contact of the liquid sample with the stopper. These esters absorb strongly in the UV region and elute very 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.). For consistency, we request that laboratories use the following absorptivities (E 1% cm) in ethanol: retinol, 1843 at 325 nm; retinyl palmitate, 975 at 325 nm; α-tocopherol, 75.8 at 292 nm; γ-tocopherol, 91.4 at 298 nm; α-carotene, 2800 at 444 nm (in hexane); β-carotene, 2560 at 450 nm (in ethanol), 2592 at 452 nm (in hexane); lycopene, 3450 at 472 nm (in hexane).

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

Micronutrients Measurement Quality Assurance Program
NIST
Bldg. 222, Rm. B208
Gaithersburg, MD 20899
Fax: (301) 977-0685

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

Sincerely,

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

Enclosures

cc: S. Wise



NIST

*NIST*

# *Micronutrients Measurement Quality Assurance Program*

## Round Robin XLIII Results from Laboratory #_____

| | Serum | | | | |
|---|---|---|---|---|---|
| Analyte | 243 | 244 | 245 | 246 | Units* |
| retinol | | | | | |
| retinyl palmitate | | | | | |
| α-tocopherol | | | | | |
| γ-tocopherol | | | | | |
| δ-tocopherol | | | | | |
| total β-carotene | | | | | |
| trans-β-carotene | | | | | |
| total cis-β-carotene | | | | | |
| total α-carotene | | | | | |
| trans-α-carotene | | | | | |
| total lycopene | | | | | |
| trans-lycopene | | | | | |
| β-cryptoxanthin | | | | | |
| α-cryptoxanthin | | | | | |
| lutein | | | | | |
| zeaxanthin | | | | | |
| lutein&zeaxanthin | | | | | |
| Other Analytes? | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |

* We prefer mg/mL

Today's Date:

Comments?

Mail: MMQAP, 222/B208
NIST
Gaithersburg, MD 20899

Please return results on-or-before
June 22, 1998

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

## Appendix F. Final Report for RR43

The following 13 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 nature of the test samples and details any previous distributions
  - summarizes aspects of the study that we believe may be of interest to the participants

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

August 28, 1998

Dear Colleague:

Enclosed is the summary report of the results for Round Robin XLIII (RR43). Included in this report are: a summary of data for all laboratories; the measurement comparability summary for evaluating laboratory performance; a summary of individual laboratory performance for the past three years; a summary of the interlaboratory accuracy and precision over the same period of time for the measurement of retinol, α- and γ-tocopherol, and *trans*- and total β-carotene; and a graphical summary of the NIST assigned value (NAV) vs. your laboratory value for these analytes. As in previous reports, the NIST assigned values are derived from the equally weighted results from the analyses performed by NIST and the laboratories that participated in this interlaboratory comparison exercise. Since there were a number of corrections, we have also included a new "All Laboratory" report for RR42. Please note that there are no changes which impact any of the conclusions or value assignments previously made for RR42.

A detailed summary of the experimental design for this interlaboratory comparison exercise is provided in the attached report. All four serum samples (Sera 243-246) were distributed in two previous round robins. Each sample contained moderate to very low levels of the analytes of interest and were selected for redistribution to provide further documentation of 1) the stability of low-carotenoid sera and 2) the limits of quantitative interlaboratory comparison. Serum 243 is a native serum that was augmented with retinyl palmitate and originally distributed as Serum 181 (RR28) and later as Serum 222 (RR37). Sera 244, 245, and 246 are dilution series originally distributed as sera 195, 197, and 198 (RR31), respectively, and later as Serum 214, 211, and 212 (RR35), respectively. Serum 246, the high level of the series, is a native serum that was augmented with retinol, retinyl palmitate, and α-tocopherol. Serum 244 is a 50:50 volumetric mix of delipidized serum with the serum used for Serum 198. Serum 245 is a 75:25 volumetric mix of the same materials.

There were no significant changes in the observed median analyte concentration for any of the four sera. There is some evidence of increased measurement variability. With the possible exception of total a-carotene, none of the increased variability seems related to extremely low analyte concentrations. In contrast to the sera-blend experiment in RR42, both lutein and zeaxanthin show the expected decreases in the delipidized serum dilutions.

Data for evaluating laboratory performance in RR42 are provided in the comparability summary (Score Card) on page 6 of the "All Lab Report." The criteria used to evaluate laboratory performance are as follows: results rated 1 (within 1 SD of the assigned value) indicate EXCEPTIONAL performance, results rated 2 (within 2 SD) indicate ACCEPTABLE performance, results rated 3 (within 3 SD of the assigned value) are MARGINAL, and those rated 4 (>3 SD from the assigned value) indicate POOR performance relative to the current state-of-the-practice for these measurements.

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

Plans regarding the continuance of the QA program beyond FY 98 are still being discussed. We will keep you informed as decisions are finalized. Due to the lack of funding, we will not hold the "Fat-soluble Vitamin and Carotenoid Tutorial" session this fall. This tutorial has been held in past years primarily for new laboratories or personnel, and for those currently experiencing difficulties with their analysis. We plan to hold the next QA workshop as a pre-meeting in April at Experimental Biology '99 in Washington, DC if the program continues in FY 99.

Samples for RR44 were shipped during the second week of August. Results are due by October 9; feedback to labs will be provided by the second week of November.

For your information, please note the following related publications:

- Granado F, Olmedilla B, Gil-Martinez E, Blanco I, Millan I, Rojas-Hidalgo E. Carotenoids, retinol and tocopherols in patients with insulin-dependent diabetes mellitus and their immediate relatives. Clin Sci (Colch) 1998 Feb;94(2):189-195

- Granado F, Olmedilla B, Blanco I, Gil-Martinez E, Rojas-Hidalgo E. Variability in the intercomparison of food carotenoid content data: a user's point of view. Crit Rev Food Sci Nutr 1997 Nov;37(7): 621-633

Another source for clinical-related publications that we have found to be useful is via the worldwide web at: http://www.ncbi.nlm.nih.gov/pubmed/.

If you have any questions regarding this report, please contact me at 301/975-3120;
FAX: 301/977-0685; e-mail: jeanice.brownthomas@nist.gov.

Sincerely,

Jeanice Brown Thomas

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

cc: S. Wise

Enclosures

The attached NIST M2QAP Round Robin XLIII (RR43) feedback report includes:

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

| Page | "Individualized" Report Contents |
|---|---|
| 1 | Your values, our assigned values, and the % bias between the two |
| 2, 3 | "Comparison" plots for: retinol; α-, γ-tocopherol; total, trans-β-carotene; total α-carotene; total lycopene; β-cryptoxanthin; lutein; zeaxanthin; lutein & zeaxnthin |
| 4 | "Z-Score Concordance" plots for this RR's: retinol; α-, γ-tocopherol; total and trans-β-carotene; total α-carotene; total and trans-lycopene |
| 5 | % Bias Accuracy/Precision Summary of your last 3 years' results for: retinol; α-, γ-tocopherol; total β-carotene and trans-β-carotene |
| 6 | NIST assigned value versus your value scatterplots of your last 3 years' results for: retinol; α-, γ-tocopherol; and/or total and trans-β-carotene |
| 7-9 | % Bias bar chart of your last 3 year's results for: retinol, α-, γ-tocopherol, and/or total and trans-β-carotene |

# Analysis of NIST M²QAP Round Robin XLIII Results: Sera 243 to 246

**Background.** Four "old", twice-before-distributed samples were distributed in RR43.

Serum 243:a native serum augmented in retinyl palmitate. It was originally distributed as 181 (RR28) and later as 222 (RR37). This serum has an unusually low β-carotene level.

Serum 246: a native serum augmented in retinol, retinyl palmitate, and α-tocopherol. It was originally distributed as 198 (RR31) and later as 212 (RR35). This fairly "normal" serum is the high-level of a 2x-dilution series.

Serum 244: a 1:1 volumetric blend of the #198 native serum and delipidized serum, originally distributed as 195 (RR31) and later as 214 (RR35). This is the mid-level of a 2x-dilution series.

Serum 245: a 1:3 volumetric blend of the #198 native serum and delipidized serum, originally distributed as 197 (RR31) and later as 211 (RR35). This is the low-level of a 2x-dilution series.

## Qualitative Results.

Interferences: Five analysts reported a peak between retinol and the retinyl acetate or tocol internal standard in serum 243. Two analysts (not exactly overlapping the previous set) reported a small peak co-eluting with retinol in sera 244, 245, and 246. These peaks appear to again be plasticizer-related, suggesting that this may be a more general problem with commercially obtained sera than we previously thought. Alternatively, they could be related to the retinyl palmitate spike. We're adding this to our list of "things to worry about if we ever get the time/opportunity."

Solids: One analyst reported a small "lump" remaining in serum 245, the low concentration member of the dilution series, after extreme reconstitution effort. We have no clue as to why one of 200 vials can glitch like this; fortunately, the analysis results for this vial did not seem to be affected by the undissolved solids.

**Quantitative Results** The following NIST Data and Value/Uncertainty Assignments table presents all NIST data, summary statistics for the NIST data, summary results for RR43, and the NIST assigned values and uncertainties. The entries are defined as follows:

Individual NIST Analyst Data and Summary Statistics

A:1 to C:2 two aliquots ("1" and "2") of three vials (A, B, and C) of each serum were extracted and analyzed. Each analyst analyzed a separate set of three vials.

$n_x$ number of quantitative values for this analyte for this serum for this analyst

$Mean_x$ arithmetic average

$SD_x$ simple standard deviation

$SD_{repx}$ within-vial pooled standard deviation, reflecting variation in extraction, chromatography, peak integration, etc.

$SD_{betx}$ among-sample standard deviation, reflecting heterogeneity in preparing and reconstituting the serum samples

$SD_{NISTx}$ $\sqrt{SD_{repx}^2 + SD_{betx}^2}$, total standard deviation. This value is $\geq SD_x$, as sample replicates reduce the true degrees of freedom.

$CV_{NISTx}$ $100 \times SD_{NISTx}/Mean_x$

NIST Summary Statistics (for analytes reported by both NIST1 and NIST3)

n number of quantitative values for this analyte for this serum

Mean $(Mean_{NIST1} + Mean_{NIST3})/2$

$SD_{rep}$ within-vial pooled standard deviation

$SD_{het}$ among-sample standard deviation

$SD_{anl}$ between-analyst standard deviation. This is the residual standard deviation for regression of NIST3's $Mean_x$ values to NIST1's. The model used to determine $SD_{anl}$ is defined to the right of this block. Details include: model used, parameters and standard errors on the parameters, and $R^2$.

$SD_{NIST}$ $\sqrt{SD_{rep}^2 + SD_{het}^2 + SD_{anl}^2}$, total standard deviation for NIST analyses.

$CV_{NIST}$ $100 \times SD_{NIST}/Mean$

Summary Statistics for Previous Distributions

RR Round Robin in which this serum was first distributed

Serum Sample identification number of the initially distributed serum

$n_p$ number of non-NIST laboratories reporting quantitative values for this analyte for this serum in the initial distribution

$Median_p$ median of the reported values in the initial distribution

$eSD_p$ 0.741 × InterQuartile Range in the initial distribution

Summary Statistics for RR43

$n_n$ number of non-NIST laboratories reporting quantitative values for this analyte for this serum in this Round Robin

$Median_n$ median of the reported values in this Round Robin

$eSD_n$ 0.741 × InterQuartile Range in this Round Robin

P(n=p) $TDIST\left(\frac{|Median_n - Median_p|\sqrt{n_n + n_p - 2}}{\sqrt{\left((n_n-1)eSD_n^2 + (n_p-1)eSD_p^2\right)\left(\frac{1}{n_n}+\frac{1}{n_p}\right)}}, n_p+n_n-2, 2\text{-tail}\right)$

This is the approximate probability that the current median is the same as it was previously. Where the hypothesis that $Median_n = Median_p$ can be rejected with 95% confidence, the P(n=p) value is flagged with an "*". TDIST is Excel®'s student's t function.

P(n<p) $FDIST\left(\frac{eSD_n^2}{eSD_o^2}, n_n\text{-}1, n_p\text{-}1\right)$

This represents the approximate probability that the current interlaboratory

variance is the same as it was previously. Where the hypothesis that $eSD_n = eSD_p$ can be rejected with 95% confidence, the P(n<p) value is flagged with an "*". FDIST is Excel®'s F-distribution function.

$SD_{labs}$ $\sqrt{eSD_n^2 - SD_{NIST}^2}$, the residual non-NIST interlaboratory biases after correction for measurement-, sample-, and NIST-analyst-related sources of variance. When $SD_{NIST}$ is greater than $eSD_n$, $SD_{labs} = 0$.

$CV_{labs}$ $100 \times SD_{labs}/Median_n$

NIST Assigned Values and Uncertainties

NAV (Mean + $Median_n$) / 2, our best guess of the "true" analyte level

NAU Maximum$(0.05 \times NAV, \sqrt{SD_{NIST}^2 + SD_{labs}^2})$, our best guess for the "true" interlaboratory standard deviation characterizing measurement, sample heterogeneity, among-analyst, and among-laboratory sources of variation. When $SD_{labs}$ could not be determined, NAU is estimated as Maximum$(0.10 \times NAV, \sqrt{2\ SD_{NIST}^2})$.

CV $100 \times NAU / NAV$

**Measurement Performance Summary.**

Measurement Stability: There are no statistically significant changes in the median concentration observed for any analyte in any of the four sera. The β -cryptoxanthin levels in all four sera are somewhat reduced, but – judging from the among-RR pattern of changes – this may be more to changes in measurement procedure than to changes in analyte level.

There is some evidence for increased measurement variance, but the analyte/serum increases do not make an interpretable pattern: γ-tocopherol in serum 243, total β-carotene in serum 246, and *trans*-lycopene in serum 244. Only for total α-carotene in sera 244, 245, and 246 is the change systematic. We hypothesize that long-term storage may induce slow changes in the "background stuff" rather than in the analytes themselves, thus creating a variety of measurement-system-specific problems rather than a single obvious loss of signal. This, again, goes on our "to-worry-about-sometime" list.

Dilution series: All analytes show the expected two-fold (serum 244) and four-fold (serum 245) reduction from the level in the native serum 246, including zeaxanthin.

Dave Duewer
Research Chemist
David.Duewer@NIST.gov

Margaret Kline
Research Biologist
Margaret.Kline@NIST.gov

# NIST Data and Value/Uncertainty Assignments

## Retinol

| | NIST1 | | | | NIST3 | | | |
|---|---|---|---|---|---|---|---|---|
| | 243 | 244 | 245 | 246 | 243 | 244 | 245 | 246 |
| A:1 | 0.417 | 0.362 | 0.195 | 0.726 | 0.467 | 0.392 | 0.191 | 0.736 |
| A:2 | 0.462 | 0.338 | 0.180 | 0.786 | 0.454 | 0.395 | 0.192 | 0.743 |
| B:1 | 0.472 | 0.329 | 0.153 | 0.759 | 0.422 | 0.351 | 0.192 | 0.706 |
| B:2 | 0.412 | 0.355 | 0.182 | 0.736 | 0.431 | 0.347 | 0.193 | 0.689 |
| C:1 | 0.452 | 0.390 | 0.189 | 0.714 | 0.439 | 0.359 | | 0.682 |
| C:2 | 0.458 | 0.373 | 0.192 | 0.714 | 0.446 | 0.358 | 0.213 | 0.691 |
| $n_x$ | 6 | 6 | 6 | 6 | 6 | 6 | 5 | 6 |
| $Mean_x$ | 0.445 | 0.358 | 0.182 | 0.739 | 0.443 | 0.367 | 0.196 | 0.708 |
| $SD_x$ | 0.025 | 0.022 | 0.015 | 0.029 | 0.016 | 0.021 | 0.009 | 0.026 |
| $SD_{repx}$ | 0.031 | 0.016 | 0.013 | 0.026 | 0.007 | 0.002 | 0.001 | 0.008 |
| $SD_{betx}$ | 0.008 | 0.021 | 0.012 | 0.023 | 0.017 | 0.023 | 0.012 | 0.028 |
| $SD_{NISTx}$ | 0.032 | 0.026 | 0.018 | 0.035 | 0.018 | 0.024 | 0.012 | 0.029 |
| $CV_{NISTx}$ | 7.2 | 7.2 | 10 | 4.7 | 4.1 | 6.4 | 6.2 | 4.1 |

| | NIST | | | |
|---|---|---|---|---|
| n | 12 | 12 | 11 | 12 |
| Mean | 0.444 | 0.362 | 0.189 | 0.724 |
| $SD_{rep}$ | 0.022 | 0.011 | 0.009 | 0.019 |
| $SD_{bet}$ | 0.012 | 0.019 | 0.011 | 0.022 |
| $SD_{anl}$ | 0.005 | 0.005 | 0.005 | 0.005 |
| $SD_{NIST}$ | 0.026 | 0.023 | 0.015 | 0.030 |
| $CV_{NIST}$ | 5.8 | 6.3 | 8.0 | 4.1 |

NIST3=a+b*NIST1

a: 0.034 ±0.006
b: 0.914 ±0.013
$R^2$: 0.987

| | XXVIII | XXXI | XXXI | XXXI |
|---|---|---|---|---|
| | 181 | 195 | 197 | 198 |
| | 44 | 38 | 38 | 39 |
| | 0.483 | 0.378 | 0.195 | 0.725 |
| | 0.064 | 0.039 | 0.024 | 0.067 |

| | RRXLIII | | | | XXXVII | XXXV | XXXV | XXXV |
|---|---|---|---|---|---|---|---|---|
| | 243 | 244 | 245 | 246 | 222 | 214 | 211 | 212 |
| $n_n$ | 47 | 47 | 46 | 47 | 45 | 44 | 44 | 44 |
| $Median_n$ | 0.479 | 0.376 | 0.193 | 0.723 | 0.470 | 0.385 | 0.198 | 0.740 |
| $eSD_n$ | 0.035 | 0.040 | 0.016 | 0.066 | 0.050 | 0.034 | 0.021 | 0.069 |
| P(n=p) | 0.99 | 0.99 | 0.98 | 1.00 | | | | |
| P(n<p) | 1.00 | 0.47 | 0.99 | 0.59 | | | | |
| $SD_{labs}$ | 0.024 | 0.032 | 0.005 | 0.059 | | | | |
| $CV_{labs}$ | 5.0 | 8.6 | 2.8 | 8.2 | | | | |
| NAV | 0.462 | 0.369 | 0.191 | 0.723 | | | | |
| NAU | 0.035 | 0.040 | 0.016 | 0.066 | | | | |
| CV | 7.6 | 11 | 8.4 | 9.2 | | | | |

## α-Tocopherol

| | NIST1 | | | | NIST3 | | | |
|---|---|---|---|---|---|---|---|---|
| | 243 | 244 | 245 | 246 | 243 | 244 | 245 | 246 |
| A:1 | 7.06 | 7.13 | 3.79 | 14.2 | 6.10 | 7.21 | 2.89 | 14.1 |
| A:2 | 7.92 | 7.20 | 4.00 | 14.4 | 6.50 | 7.47 | 2.94 | 14.2 |
| B:1 | 7.47 | 6.73 | *nq* | 14.7 | 6.93 | 7.03 | 3.20 | 14.3 |
| B:2 | 7.05 | 7.54 | 3.56 | 14.3 | 6.65 | 6.93 | 3.19 | 14.1 |
| C:1 | 6.55 | 6.52 | 4.54 | 14.9 | 7.50 | 7.11 | | 14.8 |
| C:2 | 7.45 | 7.33 | 3.73 | 14.6 | 7.34 | 6.94 | 3.66 | 14.8 |
| $n_x$ | 6 | 6 | 5 | 6 | 6 | 6 | 5 | 6 |
| $Mean_x$ | 7.25 | 7.08 | 3.92 | 14.5 | 6.84 | 7.12 | 3.18 | 14.4 |
| $SD_x$ | 0.47 | 0.38 | 0.38 | 0.3 | 0.53 | 0.20 | 0.31 | 0.3 |
| $SD_{repx}$ | 0.54 | 0.47 | 0.34 | 0.2 | 0.21 | 0.13 | 0.02 | 0.1 |
| $SD_{betx}$ | 0.25 | 0.13 | 0.29 | 0.2 | 0.56 | 0.19 | 0.38 | 0.3 |
| $SD_{NISTx}$ | 0.59 | 0.49 | 0.45 | 0.3 | 0.60 | 0.24 | 0.38 | 0.3 |
| $CV_{NISTx}$ | 8.1 | 6.9 | 11 | 2.2 | 8.7 | 3.3 | 12 | 2.4 |

| | NIST | | | |
|---|---|---|---|---|
| | 12 | 12 | 10 | 12 |
| | 7.04 | 7.10 | 3.55 | 14.4 |
| | 0.41 | 0.35 | 0.24 | 0.2 |
| | 0.39 | 0.27 | 0.31 | 0.3 |
| | 0.34 | 0.34 | 0.34 | 0.3 |
| | 0.66 | 0.55 | 0.52 | 0.5 |
| | 9.4 | 7.8 | 15 | 3.2 |

NIST3=a+b*NIST1

a: -0.68 ±0.40
b: 1.04 ±0.04
$R^2$: 0.987

| | XXVIII | XXXI | XXXI | XXXI |
|---|---|---|---|---|
| | 181 | 195 | 197 | 198 |
| | 44 | 39 | 39 | 40 |
| | 7.11 | 7.18 | 3.63 | 14.2 |
| | 0.53 | 0.83 | 0.53 | 1.0 |

| | RRXLIII | | | | XXXVII | XXXV | XXXV | XXXV |
|---|---|---|---|---|---|---|---|---|
| | 243 | 244 | 245 | 246 | 222 | 214 | 211 | 212 |
| $n_n$ | 47 | 48 | 47 | 47 | 42 | 45 | 45 | 44 |
| $Median_n$ | 6.98 | 7.23 | 3.51 | 14.2 | 7.06 | 7.14 | 3.62 | 14.2 |
| $eSD_n$ | 0.59 | 0.53 | 0.49 | 1.3 | 0.60 | 0.82 | 0.59 | 1.4 |
| P(n=p) | 0.98 | 0.99 | 0.97 | 1.00 | | | | |
| P(n<p) | 0.54 | 1.00 | 0.91 | 0.60 | | | | |
| $SD_{labs}$ | 0 | 0 | 0 | 1.2 | | | | |
| $CV_{labs}$ | 0 | 0 | 0 | 8.7 | | | | |
| NAV | 7.01 | 7.16 | 3.53 | 14.3 | | | | |
| NAU | 0.66 | 0.55 | 0.52 | 1.3 | | | | |
| CV | 9.4 | 7.7 | 15 | 9.2 | | | | |

# NIST Data and Value/Uncertainty Assignments

## γ-Tocopherol

| | NIST1 | | | | NIST3 | | | |
|---|---|---|---|---|---|---|---|---|
| | 243 | 244 | 245 | 246 | 243 | 244 | 245 | 246 |
| A:1 | 2.59 | *nd* | *nd* | 2.67 | 2.11 | 1.290 | 0.626 | 2.52 |
| A:2 | 1.84 | *nd* | *nd* | 2.72 | 2.21 | 1.296 | 0.628 | 2.54 |
| B:1 | 2.05 | *nd* | *nd* | 2.79 | 2.25 | 1.289 | 0.650 | 2.53 |
| B:2 | 2.12 | *nd* | *nd* | 2.66 | 2.24 | 1.270 | 0.671 | 2.51 |
| C:1 | 2.11 | *nd* | *nd* | 2.71 | 2.29 | 1.226 | | 2.56 |
| C:2 | 2.34 | *nd* | *nd* | 2.71 | 2.33 | 1.216 | 0.746 | 2.59 |
| $n_x$ | 6 | 0 | 0 | 6 | 6 | 6 | 5 | 6 |
| $Mean_x$ | 2.18 | | | 2.71 | 2.24 | 1.265 | 0.664 | 2.54 |
| $SD_x$ | 0.26 | | | 0.05 | 0.08 | 0.035 | 0.049 | 0.03 |
| $SD_{rep}$ | 0.32 | | | 0.06 | 0.04 | 0.009 | 0.009 | 0.02 |
| $SD_{bet}$ | 0.08 | | | 0.01 | 0.08 | 0.038 | 0.062 | 0.03 |
| $SD_{NISTx}$ | 0.33 | | | 0.06 | 0.09 | 0.039 | 0.062 | 0.04 |
| $CV_{NISTx}$ | 15 | | | 2.2 | 3.9 | 3.1 | 9.4 | 1.4 |

| | NIST | | | |
|---|---|---|---|---|
| n | 12 | 6 | 5 | 12 |
| Mean | 2.21 | 1.265 | 0.664 | 2.63 |
| $SD_{rep}$ | 0.23 | 0.009 | 0.009 | 0.04 |
| $SD_{bet}$ | 0.08 | 0.038 | 0.062 | 0.09 |
| $SD_{wil}$ | | | | |
| $SD_{NIST}$ | 0.24 | 0.039 | 0.062 | 0.10 |
| $CV_{NIST}$ | 11 | 3.1 | 9.4 | 4.0 |

| | xxviii | xxxi | xxxi | xxxi |
|---|---|---|---|---|
| | 181 | 195 | 197 | 198 |
| | 16 | 15 | 15 | 16 |
| | 2.28 | 1.350 | 0.707 | 2.62 |
| | 0.16 | 0.184 | 0.149 | 0.14 |

| | RRXLIII | | | | xxxvii | xxxv | xxxv | xxxv |
|---|---|---|---|---|---|---|---|---|
| | 243 | 244 | 245 | 246 | 222 | 214 | 211 | 212 |
| $n_n$ | 23 | 23 | 22 | 22 | 22 | 21 | 22 | 21 |
| $Median_n$ | 2.31 | 1.280 | 0.685 | 2.51 | 2.31 | 1.330 | 0.711 | 2.70 |
| $eSD_n$ | 0.28 | 0.102 | 0.101 | 0.15 | 0.17 | 0.066 | 0.136 | 0.23 |
| P(n=p) | 1.00 | 0.87 | 0.95 | 0.82 | | | | |
| P(n<p) | *0.02 | 0.99 | 0.95 | 0.97 | | | | |
| $SD_{labs}$ | 0.13 | 0.094 | 0.079 | 0.11 | | | | |
| $CV_{labs}$ | 5.8 | 7.4 | 12 | 4.3 | | | | |
| NAV | 2.26 | 1.272 | 0.674 | 2.57 | | | | |
| NAU | 0.28 | 0.102 | 0.101 | 0.15 | | | | |
| CV | 12 | 8.0 | 15 | 5.8 | | | | |

## δ-Tocopherol

| | NIST1 | | | | NIST3 | | | |
|---|---|---|---|---|---|---|---|---|
| | 243 | 244 | 245 | 246 | 243 | 244 | 245 | 246 |
| $n_x$ | 0 | 0 | 0 | 0 | 6 | 6 | 5 | 6 |
| $Mean_x$ | | | | | 0.149 | 0.087 | 0.051 | 0.166 |
| $SD_x$ | | | | | 0.008 | 0.011 | 0.008 | 0.005 |
| $SD_{rep}$ | | | | | 0.009 | 0.011 | 0.004 | 0.003 |
| $SD_{bet}$ | | | | | 0.005 | 0.007 | 0.009 | 0.004 |
| $SD_{NISTx}$ | | | | | 0.011 | 0.013 | 0.010 | 0.005 |
| $CV_{NISTx}$ | | | | | 7.1 | 15 | 20 | 3.2 |

| | NIST | | | |
|---|---|---|---|---|
| | 6 | 6 | 5 | 6 |
| | 0.149 | 0.087 | 0.051 | 0.166 |
| | 0.009 | 0.011 | 0.004 | 0.003 |
| | 0.005 | 0.007 | 0.009 | 0.004 |
| | 0.011 | 0.013 | 0.010 | 0.005 |
| | 7.1 | 15 | 20 | 3.2 |

| | xxviii | xxxi | xxxi | xxxi |
|---|---|---|---|---|
| | 181 | 195 | 197 | 198 |
| | 0 | 0 | 0 | 0 |

| | RRXLIII | | | | xxxvii | xxxv | xxxv | xxxv |
|---|---|---|---|---|---|---|---|---|
| | 243 | 244 | 245 | 246 | 222 | 214 | 211 | 212 |
| $n_n$ | 3 | 3 | 3 | 3 | 5 | 1 | 1 | 1 |
| $Median_n$ | 0.238 | 0.152 | 0.074 | 0.285 | 0.170 | 0.015 | 0.047 | 0.330 |
| $eSD_n$ | 0.097 | 0.032 | 0.013 | 0.081 | 0.045 | | | |
| P(n=p) | 0.48 | | | | | | | |
| P(n<p) | 0.09 | | | | | | | |
| $SD_{labs}$ | 0.096 | 0.029 | 0.007 | 0.081 | | | | |
| $CV_{labs}$ | 40 | 19 | 10.0 | 28 | | | | |
| NAV | 0.193 | 0.119 | 0.063 | 0.226 | | | | |
| NAU | 0.097 | 0.032 | 0.013 | 0.081 | | | | |
| CV | 50 | 26 | 20 | 36 | | | | |

# NIST Data and Value/Uncertainty Assignments

## Total β-Carotene

| | NIST1 | | | | NIST3 | | | |
|---|---|---|---|---|---|---|---|---|
| | 243 | 244 | 245 | 246 | 243 | 244 | 245 | 246 |
| A:1 | 0.072 | 0.396 | 0.147 | 0.64 | *nq* | 0.389 | *nq* | 0.73 |
| A:2 | 0.079 | 0.380 | 0.139 | 0.64 | *nq* | 0.382 | *nq* | 0.77 |
| B:1 | 0.066 | 0.393 | 0.155 | 0.65 | *nq* | 0.360 | *nq* | 0.77 |
| B:2 | 0.078 | 0.402 | 0.152 | 0.62 | *nq* | 0.393 | *nq* | 0.75 |
| C:1 | 0.078 | 0.386 | 0.142 | 0.61 | *nq* | 0.354 | *nq* | 0.76 |
| C:2 | 0.080 | 0.394 | 0.153 | 0.60 | *nq* | 0.364 | *nq* | 0.77 |
| $n_x$ | 6 | 6 | 6 | 6 | 0 | 6 | 0 | 6 |
| $Mean_x$ | 0.076 | 0.392 | 0.148 | 0.63 | | 0.374 | | 0.76 |
| $SD_x$ | 0.006 | 0.008 | 0.006 | 0.02 | | 0.016 | | 0.02 |
| $SD_{rep}$ | 0.006 | 0.008 | 0.006 | 0.02 | | 0.014 | | 0.02 |
| $SD_{bet}$ | 0.003 | 0.005 | 0.005 | 0.02 | | 0.013 | | 0.01 |
| $SD_{NISTx}$ | 0.007 | 0.010 | 0.008 | 0.03 | | 0.020 | | 0.02 |
| $CV_{NISTx}$ | 9.1 | 2.4 | 5.2 | 4.0 | | 5.2 | | 2.7 |

| | NIST | | | |
|---|---|---|---|---|
| n | 6 | 12 | 6 | 12 |
| Mean | 0.076 | 0.383 | 0.148 | 0.69 |
| $SD_{rep}$ | 0.006 | 0.012 | 0.006 | 0.02 |
| $SD_{bet}$ | 0.003 | 0.013 | 0.005 | 0.07 |
| $SD_{anl}$ | | | | |
| $SD_{NIST}$ | 0.007 | 0.018 | 0.008 | 0.07 |
| $CV_{NIST}$ | 9.1 | 4.6 | 5.2 | 11 |

| | xxviii | xxxi | xxxi | xxxi |
|---|---|---|---|---|
| | 181 | 195 | 197 | 198 |
| | 33 | 30 | 30 | 31 |
| | 0.087 | 0.338 | 0.150 | 0.68 |
| | 0.022 | 0.047 | 0.029 | 0.08 |

| | RRXLIII | | | | xxxvii | xxxv | xxxv | xxxv |
|---|---|---|---|---|---|---|---|---|
| | 243 | 244 | 245 | 246 | 222 | 214 | 211 | 212 |
| $n_n$ | 32 | 32 | 31 | 31 | 28 | 34 | 33 | 33 |
| $Median_n$ | 0.073 | 0.336 | 0.144 | 0.66 | 0.077 | 0.360 | 0.167 | 0.70 |
| $eSD_n$ | 0.011 | 0.030 | 0.027 | 0.12 | 0.012 | 0.034 | 0.034 | 0.06 |
| P(n=p) | 0.92 | 0.99 | 0.96 | 0.96 | | | | |
| P(n<p) | 1.00 | 0.99 | 0.90 | *0.01 | | | | |
| $SD_{labs}$ | 0.009 | 0.024 | 0.026 | 0.09 | | | | |
| $CV_{labs}$ | 12 | 7.1 | 18 | 14 | | | | |
| NAV | 0.074 | 0.359 | 0.146 | 0.68 | | | | |
| NAU | 0.011 | 0.030 | 0.027 | 0.12 | | | | |
| CV | 15 | 8.3 | 19 | 18 | | | | |

## trans-β-Carotene

| | NIST1 | | | | NIST3 | | | |
|---|---|---|---|---|---|---|---|---|
| | 243 | 244 | 245 | 246 | 243 | 244 | 245 | 246 |
| A:1 | 0.070 | 0.348 | 0.140 | 0.593 | 0.059 | 0.345 | 0.073 | 0.663 |
| A:2 | 0.073 | 0.343 | 0.135 | 0.617 | 0.056 | 0.342 | 0.065 | 0.691 |
| B:1 | 0.060 | 0.331 | 0.140 | 0.629 | 0.074 | 0.324 | 0.120 | 0.693 |
| B:2 | 0.077 | 0.354 | 0.131 | 0.584 | 0.069 | 0.339 | 0.098 | 0.674 |
| C:1 | 0.073 | 0.333 | 0.118 | 0.590 | 0.072 | 0.321 | | 0.689 |
| C:2 | 0.078 | 0.342 | 0.132 | 0.572 | 0.068 | 0.322 | 0.120 | 0.697 |
| $n_x$ | 6 | 6 | 6 | 6 | 6 | 6 | 5 | 6 |
| $Mean_x$ | 0.072 | 0.342 | 0.133 | 0.598 | 0.066 | 0.332 | 0.095 | 0.685 |
| $SD_x$ | 0.007 | 0.009 | 0.008 | 0.021 | 0.007 | 0.011 | 0.026 | 0.013 |
| $SD_{rep}$ | 0.007 | 0.010 | 0.007 | 0.022 | 0.003 | 0.006 | 0.010 | 0.014 |
| $SD_{bet}$ | 0.003 | 0.004 | 0.007 | 0.014 | 0.008 | 0.011 | 0.027 | 0.008 |
| $SD_{NISTx}$ | 0.008 | 0.011 | 0.010 | 0.026 | 0.008 | 0.013 | 0.029 | 0.016 |
| $CV_{NISTx}$ | 11 | 3.2 | 7.4 | 4.4 | 12 | 3.8 | 30 | 2.4 |

| | NIST | | | |
|---|---|---|---|---|
| | 12 | 12 | 11 | 12 |
| | 0.069 | 0.337 | 0.114 | 0.641 |
| | 0.006 | 0.008 | 0.008 | 0.019 |
| | 0.016 | 0.018 | 0.019 | 0.015 |
| | 0.033 | 0.033 | 0.033 | 0.033 |
| | 0.037 | 0.038 | 0.039 | 0.041 |
| | 54 | 11 | 34 | 6.4 |

NIST3=a+b*NIST1

a: -0.048 ±0.028
b: 1.198 ±0.080
$R^2$: 0.987

| | xxviii | xxxi | xxxi | xxxi |
|---|---|---|---|---|
| | 181 | 195 | 197 | 198 |
| | 5 | 7 | 7 | 7 |
| | 0.085 | 0.332 | 0.160 | 0.628 |
| | 0.005 | 0.026 | 0.021 | 0.006 |

| | RRXLIII | | | | xxxvii | xxxv | xxxv | xxxv |
|---|---|---|---|---|---|---|---|---|
| | 243 | 244 | 245 | 246 | 222 | 214 | 211 | 212 |
| $n_n$ | 11 | 11 | 11 | 11 | 14 | 12 | 12 | 12 |
| $Median_n$ | 0.071 | 0.308 | 0.150 | 0.631 | 0.072 | 0.337 | 0.159 | 0.642 |
| $eSD_n$ | 0.010 | 0.022 | 0.017 | 0.038 | 0.010 | 0.034 | 0.021 | 0.066 |
| P(n=p) | 0.97 | 0.68 | 0.85 | 0.96 | | | | |
| P(n<p) | 0.60 | 0.92 | 0.74 | 0.95 | | | | |
| $SD_{labs}$ | 0 | 0 | 0 | 0 | | | | |
| $CV_{labs}$ | 0 | 0 | 0 | 0 | | | | |
| NAV | 0.070 | 0.323 | 0.132 | 0.636 | | | | |
| NAU | 0.037 | 0.038 | 0.039 | 0.041 | | | | |
| CV | 53 | 12 | 29 | 6.4 | | | | |

# NIST Data and Value/Uncertainty Assignments

## Total α-Carotene

| | NIST1 | | | | NIST3 | | | |
|---|---|---|---|---|---|---|---|---|
| | 243 | 244 | 245 | 246 | 243 | 244 | 245 | 246 |
| A:1 | *nd* | 0.026 | *nd* | 0.046 | *nq* | *nq* | *nq* | 0.044 |
| A:2 | *nd* | 0.027 | *nd* | 0.040 | *nq* | *nq* | *nq* | 0.052 |
| B:1 | *nd* | 0.029 | *nd* | 0.054 | *nq* | *nq* | *nq* | 0.053 |
| B:2 | *nd* | 0.030 | *nd* | 0.042 | *nq* | *nq* | *nq* | 0.049 |
| C:1 | *nd* | 0.026 | *nd* | 0.043 | *nq* | *nq* | *nq* | 0.050 |
| C:2 | *nd* | 0.023 | *nd* | 0.037 | *nq* | *nq* | *nq* | 0.051 |
| $n_x$ | 0 | 6 | 0 | 6 | 0 | 0 | 0 | 6 |
| $Mean_x$ | | 0.027 | | 0.044 | | | | 0.050 |
| $SD_x$ | | 0.002 | | 0.006 | | | | 0.003 |
| $SD_{repx}$ | | 0.001 | | 0.006 | | | | 0.004 |
| $SD_{betx}$ | | 0.002 | | 0.004 | | | | 0.001 |
| $SD_{NISTx}$ | | 0.003 | | 0.007 | | | | 0.004 |
| $CV_{NISTx}$ | | 9.9 | | 16 | | | | 7.6 |

| | NIST | | | |
|---|---|---|---|---|
| n | 0 | 6 | 0 | 12 |
| Mean | | 0.027 | | 0.047 |
| $SD_{rep}$ | | 0.001 | | 0.005 |
| $SD_{bet}$ | | 0.002 | | 0.004 |
| $SD_{anl}$ | | | | |
| $SD_{NIST}$ | | 0.003 | | 0.007 |
| $CV_{NIST}$ | | 9.9 | | 14 |

| | RRXLIII | | | | xxxvii | xxxv | xxxv | xxxv |
|---|---|---|---|---|---|---|---|---|
| | 243 | 244 | 245 | 246 | 222 | 214 | 211 | 212 |
| $n_n$ | 24 | 24 | 20 | 25 | 21 | 26 | 23 | 26 |
| $Median_n$ | 0.014 | 0.018 | 0.010 | 0.032 | 0.016 | 0.018 | 0.009 | 0.036 |
| $eSD_n$ | 0.003 | 0.008 | 0.004 | 0.012 | 0.005 | 0.004 | 0.003 | 0.008 |
| P(n=p) | 0.88 | 0.99 | 0.94 | 0.91 | | | | |
| P(n<p) | 0.99 | *0.00 | 0.06 | *0.01 | | | | |

| | xxviii | xxxi | xxxi | xxxi |
|---|---|---|---|---|
| | 181 | 195 | 197 | 198 |
| | 15 | | | |
| | 0.018 | | | |
| | 0.006 | | | |

| | 0.008 | 0.010 |
|---|---|---|
| $SD_{labs}$ | | |
| $CV_{labs}$ | 43 | 32 |

| | 0.022 | 0.039 |
|---|---|---|
| NAV | | |
| NAU | 0.008 | 0.012 |
| CV | 36 | 31 |

## trans-α-Carotene

| | NIST1 | | | | NIST3 | | | |
|---|---|---|---|---|---|---|---|---|
| | 243 | 244 | 245 | 246 | 243 | 244 | 245 | 246 |
| | | | | | 0.013 | 0.019 | *nq* | 0.030 |
| | | | | | 0.012 | 0.016 | *nq* | 0.032 |
| | | | | | 0.014 | 0.015 | *nq* | 0.033 |
| | | | | | 0.014 | 0.018 | *nq* | 0.029 |
| | | | | | 0.017 | 0.015 | *nq* | 0.033 |
| | | | | | 0.014 | 0.016 | *nq* | 0.033 |
| | 0 | 0 | 0 | 0 | 6 | 6 | 0 | 6 |
| | | | | | 0.014 | 0.016 | | 0.032 |
| | | | | | 0.001 | 0.001 | | 0.002 |
| | | | | | 0.001 | 0.002 | | 0.002 |
| | | | | | 0.001 | 0.001 | | 0.001 |
| | | | | | 0.002 | 0.002 | | 0.002 |
| | | | | | 13 | 11 | | 6.4 |

| | NIST | | | |
|---|---|---|---|---|
| | 6 | 6 | 0 | 6 |
| | 0.014 | 0.016 | | 0.032 |
| | 0.001 | 0.002 | | 0.002 |
| | 0.001 | 0.001 | | 0.001 |
| | | | | |
| | 0.002 | 0.002 | | 0.002 |
| | 13 | 11 | | 6.4 |

| | xxviii | xxxi | xxxi | xxxi |
|---|---|---|---|---|
| | 181 | 195 | 197 | 198 |
| | 0 | 0 | 0 | 0 |

| | RRXLIII | | | | xxxvii | xxxv | xxxv | xxxv |
|---|---|---|---|---|---|---|---|---|
| | 243 | 244 | 245 | 246 | 222 | 214 | 211 | 212 |
| $n_n$ | 1 | 1 | 0 | 1 | 0 | 0 | 0 | 0 |
| $Median_n$ | 0.014 | 0.014 | | 0.023 | | | | |
| $eSD_n$ | | | | | | | | |

| | | | |
|---|---|---|---|
| $SD_{labs}$ | | | |
| $CV_{labs}$ | | | |

| | 0.014 | 0.015 | 0.027 |
|---|---|---|---|
| NAV | | | |
| NAU | 0.002 | 0.003 | 0.003 |
| CV | 18 | 17 | 10 |

# NIST Data and Value/Uncertainty Assignments

## Total Lycopene

| | NIST1 | | | | NIST3 | | | |
|---|---|---|---|---|---|---|---|---|
| | 243 | 244 | 245 | 246 | 243 | 244 | 245 | 246 |
| A:1 | | | | | 0.46 | *nq* | *nq* | 0.184 |
| A:2 | | | | | 0.46 | *nq* | *nq* | 0.189 |
| B:1 | | | | | 0.55 | *nq* | *nq* | 0.209 |
| B:2 | | | | | 0.52 | *nq* | *nq* | 0.194 |
| C:1 | | | | | 0.56 | *nq* | *nq* | 0.227 |
| C:2 | | | | | 0.57 | *nq* | *nq* | 0.215 |
| $n_x$ | 0 | 0 | 0 | 0 | 6 | 0 | 0 | 6 |
| $Mean_x$ | | | | | 0.52 | | | 0.203 |
| $SD_x$ | | | | | 0.05 | | | 0.017 |
| $SD_{repx}$ | | | | | 0.01 | | | 0.008 |
| $SD_{betx}$ | | | | | 0.05 | | | 0.017 |
| $SD_{NISTx}$ | | | | | 0.06 | | | 0.019 |
| $CV_{NISTx}$ | | | | | 11 | | | 9.4 |

| | NIST | | | |
|---|---|---|---|---|
| n | 6 | 0 | 0 | 6 |
| Mean | 0.52 | | | 0.203 |
| $SD_{rep}$ | 0.01 | | | 0.008 |
| $SD_{bet}$ | 0.05 | | | 0.017 |
| $SD_{anl}$ | | | | |
| $SD_{NIST}$ | 0.06 | | | 0.019 |
| $CV_{NIST}$ | 11 | | | 9.4 |

| | XXVIII | XXXI | XXXI | XXXI |
|---|---|---|---|---|
| | 181 | 195 | 197 | 198 |
| | 16 | 20 | 19 | 22 |
| | 0.47 | 0.06 | 0.02 | 0.118 |
| | 0.10 | 0.02 | 0.01 | 0.049 |

| | RRXLIII | | | | XXXVII | XXXV | XXXV | XXXV |
|---|---|---|---|---|---|---|---|---|
| | 243 | 244 | 245 | 246 | 222 | 214 | 211 | 212 |
| $n_n$ | 29 | 28 | 23 | 27 | 24 | 23 | 23 | 24 |
| $Median_n$ | 0.43 | 0.07 | 0.03 | 0.122 | 0.45 | 0.07 | 0.03 | 0.142 |
| $eSD_n$ | 0.14 | 0.03 | 0.01 | 0.043 | 0.04 | 0.03 | 0.01 | 0.047 |
| P(n=p) | 0.95 | 0.98 | 0.96 | 0.98 | | | | |
| P(n<p) | 0.11 | 0.51 | 0.37 | 0.75 | | | | |
| $SD_{labs}$ | 0.12 | | | 0.038 | | | | |
| $CV_{labs}$ | 29 | | | 31 | | | | |
| NAV | 0.47 | | | 0.163 | | | | |
| NAU | 0.14 | | | 0.043 | | | | |
| CV | 29 | | | 26 | | | | |

## trans-Lycopene

| | NIST1 | | | | NIST3 | | | |
|---|---|---|---|---|---|---|---|---|
| | 243 | 244 | 245 | 246 | 243 | 244 | 245 | 246 |
| A:1 | | | | | 0.198 | 0.037 | *nq* | 0.069 |
| A:2 | | | | | 0.211 | 0.037 | *nq* | 0.075 |
| B:1 | | | | | 0.260 | 0.040 | *nq* | 0.075 |
| B:2 | | | | | 0.240 | 0.037 | *nq* | 0.076 |
| C:1 | | | | | 0.247 | 0.037 | *nq* | 0.075 |
| C:2 | | | | | 0.248 | 0.032 | *nq* | 0.080 |
| $n_x$ | 0 | 0 | 0 | 0 | 6 | 6 | 0 | 6 |
| $Mean_x$ | | | | | 0.234 | 0.037 | | 0.075 |
| $SD_x$ | | | | | 0.024 | 0.002 | | 0.003 |
| $SD_{repx}$ | | | | | 0.010 | 0.002 | | 0.003 |
| $SD_{betx}$ | | | | | 0.025 | 0.002 | | 0.003 |
| $SD_{NISTx}$ | | | | | 0.027 | 0.003 | | 0.004 |
| $CV_{NISTx}$ | | | | | 12 | 7.6 | | 5.2 |

| | NIST | | | |
|---|---|---|---|---|
| n | 6 | 6 | 0 | 6 |
| Mean | 0.234 | 0.037 | | 0.075 |
| $SD_{rep}$ | 0.010 | 0.002 | | 0.003 |
| $SD_{bet}$ | 0.025 | 0.002 | | 0.003 |
| $SD_{anl}$ | | | | |
| $SD_{NIST}$ | 0.027 | 0.003 | | 0.004 |
| $CV_{NIST}$ | 12 | 7.6 | | 5.2 |

| | XXVIII | XXXI | XXXI | XXXI |
|---|---|---|---|---|
| | 181 | 195 | 197 | 198 |
| | 0 | 1 | 1 | 1 |
| | | 0.041 | 0.015 | 0.070 |

| | RRXLIII | | | | XXXVII | XXXV | XXXV | XXXV |
|---|---|---|---|---|---|---|---|---|
| | 243 | 244 | 245 | 246 | 222 | 214 | 211 | 212 |
| $n_n$ | 9 | 9 | 9 | 9 | 7 | 6 | 6 | 6 |
| $Median_n$ | 0.292 | 0.035 | 0.017 | 0.067 | 0.311 | 0.046 | 0.024 | 0.087 |
| $eSD_n$ | 0.067 | 0.013 | 0.004 | 0.022 | 0.074 | 0.004 | 0.010 | 0.017 |
| P(n=p) | 0.89 | 0.58 | 0.60 | 0.61 | | | | |
| P(n<p) | 0.62 | *0.02 | 0.98 | 0.28 | | | | |
| $SD_{labs}$ | 0.061 | 0.012 | | 0.022 | | | | |
| $CV_{labs}$ | 21 | 35 | | 33 | | | | |
| NAV | 0.263 | 0.036 | | 0.071 | | | | |
| NAU | 0.067 | 0.013 | | 0.022 | | | | |
| CV | 25 | 35 | | 31 | | | | |

# NIST Data and Value/Uncertainty Assignments

## β-Cryptoxanthin

| | NIST1 | | | | NIST3 | | | |
|---|---|---|---|---|---|---|---|---|
| | 243 | 244 | 245 | 246 | 243 | 244 | 245 | 246 |
| A:1 | 0.047 | 0.015 | *nd* | 0.023 | 0.037 | 0.007 | *nd* | 0.016 |
| A:2 | 0.044 | 0.017 | *nd* | 0.024 | 0.043 | 0.006 | *nd* | 0.016 |
| B:1 | 0.043 | 0.017 | *nd* | 0.026 | 0.057 | 0.008 | *nd* | 0.016 |
| B:2 | 0.039 | 0.017 | *nd* | 0.023 | 0.046 | 0.007 | *nd* | 0.017 |
| C:1 | 0.040 | 0.017 | *nd* | 0.026 | 0.056 | 0.009 | *nd* | 0.016 |
| C:2 | 0.044 | 0.018 | *nd* | 0.027 | 0.046 | 0.007 | *nd* | 0.015 |
| $n_x$ | 6 | 6 | 0 | 6 | 6 | 6 | 0 | 6 |
| $Mean_x$ | 0.043 | 0.017 | | 0.025 | 0.047 | 0.007 | | 0.016 |
| $SD_x$ | 0.003 | 0.001 | | 0.002 | 0.008 | 0.001 | | 0.001 |
| $SD_{repx}$ | 0.002 | 0.001 | | 0.001 | 0.007 | 0.001 | | 0.001 |
| $SD_{betx}$ | 0.002 | 0.001 | | 0.002 | 0.007 | 0.001 | | 0.001 |
| $SD_{NISTx}$ | 0.003 | 0.001 | | 0.002 | 0.009 | 0.001 | | 0.001 |
| $CV_{NISTx}$ | 8.1 | 6.0 | | 8.5 | 20 | 17 | | 5.5 |

| | NIST | | | |
|---|---|---|---|---|
| n | 12 | 12 | 0 | 12 |
| Mean | 0.045 | 0.012 | | 0.020 |
| $SD_{rep}$ | 0.005 | 0.001 | | 0.001 |
| $SD_{bet}$ | 0.005 | 0.005 | | 0.005 |
| $SD_{ani}$ | | | | |
| $SD_{NIST}$ | 0.007 | 0.005 | | 0.005 |
| $CV_{NIST}$ | 16 | 44 | | 25 |

| | XXVIII | XXXI | XXXI | XXXI |
|---|---|---|---|---|
| | 181 | 195 | 197 | 198 |
| | 11 | 13 | 12 | 15 |
| | 0.070 | 0.013 | 0.008 | 0.024 |
| | 0.012 | 0.007 | 0.003 | 0.008 |

| | RRXLIII | | | | XXXVII | XXXV | XXXV | XXXV |
|---|---|---|---|---|---|---|---|---|
| | 243 | 244 | 245 | 246 | 222 | 214 | 211 | 212 |
| $n_n$ | 27 | 22 | 17 | 24 | 19 | 15 | 12 | 16 |
| $Median_n$ | 0.058 | 0.009 | 0.005 | 0.016 | 0.070 | 0.014 | 0.009 | 0.026 |
| $eSD_n$ | 0.016 | 0.002 | 0.002 | 0.004 | 0.019 | 0.005 | 0.004 | 0.007 |
| P(n=p) | 0.84 | 0.77 | 0.70 | 0.68 | | | | |
| P(n<p) | 0.79 | 1.00 | 1.00 | 0.99 | | | | |
| $SD_{labs}$ | 0.015 | 0 | | 0 | | | | |
| $CV_{labs}$ | 25 | 0 | | 0 | | | | |
| NAV | 0.052 | 0.011 | | 0.018 | | | | |
| NAU | 0.016 | 0.005 | | 0.005 | | | | |
| CV | 32 | 50 | | 28 | | | | |

## "Lutein"

| | NIST1 | | | | NIST3 | | | |
|---|---|---|---|---|---|---|---|---|
| | 243 | 244 | 245 | 246 | 243 | 244 | 245 | 246 |
| A:1 | 0.064 | 0.023 | 0.012 | 0.062 | 0.088 | 0.025 | 0.012 | 0.052 |
| A:2 | 0.069 | 0.021 | *nd* | 0.062 | 0.088 | 0.030 | 0.012 | 0.051 |
| B:1 | 0.068 | 0.017 | 0.012 | 0.063 | 0.093 | 0.028 | 0.012 | 0.056 |
| B:2 | 0.068 | 0.023 | *nd* | 0.068 | 0.087 | 0.027 | 0.015 | 0.055 |
| C:1 | 0.068 | 0.020 | *nd* | 0.066 | 0.090 | 0.025 | | 0.054 |
| C:2 | 0.062 | 0.021 | 0.012 | 0.063 | 0.091 | 0.024 | 0.016 | 0.055 |
| $n_x$ | 6 | 6 | 3 | 6 | 6 | 6 | 5 | 6 |
| $Mean_x$ | 0.067 | 0.021 | 0.012 | 0.064 | 0.090 | 0.026 | 0.013 | 0.054 |
| $SD_x$ | 0.003 | 0.002 | 0.000 | 0.002 | 0.002 | 0.002 | 0.002 | 0.002 |
| $SD_{repx}$ | 0.003 | 0.003 | | 0.002 | 0.002 | 0.002 | 0.001 | 0.001 |
| $SD_{betx}$ | 0.001 | 0.001 | | 0.002 | 0.001 | 0.002 | 0.002 | 0.002 |
| $SD_{NISTx}$ | 0.004 | 0.003 | | 0.003 | 0.003 | 0.003 | 0.002 | 0.002 |
| $CV_{NISTx}$ | 5.4 | 14 | | 4.6 | 3.2 | 11 | 15 | 4.0 |

| | NIST | | | |
|---|---|---|---|---|
| | 12 | 12 | 8 | 12 |
| | 0.078 | 0.024 | 0.013 | 0.059 |
| | 0.003 | 0.003 | 0.001 | 0.002 |
| | 0.009 | 0.002 | 0.001 | 0.010 |
| | 0.013 | 0.013 | 0.013 | 0.013 |
| | 0.016 | 0.014 | 0.014 | 0.017 |
| | 21 | 59 | 110 | 28 |

NIST3=a+b*NIST1

a: 0.000 ±
b: 1.113 ±0.141
$R^2$: 0.987

| | XXVIII | XXXI | XXXI | XXXI |
|---|---|---|---|---|
| | 181 | 195 | 197 | 198 |
| | 7 | 7 | 6 | 8 |
| | 0.068 | 0.020 | 0.011 | 0.057 |
| | 0.015 | 0.003 | 0.002 | 0.019 |

| | RRXLIII | | | | XXXVII | XXXV | XXXV | XXXV |
|---|---|---|---|---|---|---|---|---|
| | 243 | 244 | 245 | 246 | 222 | 214 | 211 | 212 |
| $n_n$ | 16 | 16 | 15 | 16 | 13 | 10 | 9 | 10 |
| $Median_n$ | 0.081 | 0.028 | 0.013 | 0.050 | 0.081 | 0.030 | 0.016 | 0.054 |
| $eSD_n$ | 0.014 | 0.005 | 0.002 | 0.008 | 0.019 | 0.004 | 0.005 | 0.007 |
| P(n=p) | 1.00 | 0.86 | 0.72 | 0.86 | | | | |
| P(n<p) | 0.86 | 0.32 | 1.00 | 1.00 | | | | |
| | 0 | 0 | 0 | 0 | | | | |
| | 0 | 0 | 0 | 0 | | | | |
| | 0.079 | 0.026 | 0.013 | 0.055 | | | | |
| | 0.016 | 0.014 | 0.014 | 0.017 | | | | |
| | 20 | 55 | 100 | 30 | | | | |

# NIST Data and Value/Uncertainty Assignments

## "Zeaxanthin"

| | NIST1 | | | | NIST3 | | | |
|---|---|---|---|---|---|---|---|---|
| | 243 | 244 | 245 | 246 | 243 | 244 | 245 | 246 |
| A:1 | 0.039 | *nd* | *nd* | 0.026 | 0.047 | 0.012 | 0.004 | 0.027 |
| A:2 | 0.034 | *nd* | *nd* | 0.021 | 0.047 | 0.015 | 0.005 | 0.025 |
| B:1 | 0.042 | *nd* | *nd* | 0.026 | 0.057 | 0.013 | 0.005 | 0.027 |
| B:2 | 0.042 | *nd* | *nd* | 0.023 | 0.054 | 0.012 | 0.007 | 0.027 |
| C:1 | 0.044 | *nd* | *nd* | 0.027 | 0.053 | 0.014 | | 0.026 |
| C:2 | 0.043 | *nd* | *nd* | 0.020 | 0.052 | 0.011 | 0.005 | 0.024 |
| $n_x$ | 6 | 0 | 0 | 6 | 6 | 6 | 5 | 6 |
| $Mean_x$ | 0.041 | | | 0.024 | 0.052 | 0.013 | 0.005 | 0.026 |
| $SD_x$ | 0.004 | | | 0.003 | 0.004 | 0.002 | 0.001 | 0.001 |
| $SD_{repx}$ | 0.002 | | | 0.004 | 0.002 | 0.002 | 0.001 | 0.001 |
| $SD_{betx}$ | 0.004 | | | 0.000 | 0.004 | 0.001 | 0.001 | 0.001 |
| $SD_{NISTx}$ | 0.004 | | | 0.004 | 0.004 | 0.002 | 0.001 | 0.002 |
| $CV_{NISTx}$ | 11 | | | 17 | 8.4 | 16 | 25 | 6.4 |

| | NIST | | | |
|---|---|---|---|---|
| n | 12 | 6 | 5 | 12 |
| Mean | 0.046 | 0.013 | 0.005 | 0.025 |
| $SD_{rep}$ | 0.002 | 0.002 | 0.001 | 0.003 |
| $SD_{bet}$ | 0.007 | 0.001 | 0.001 | 0.001 |
| $SD_{sml}$ | | | | |
| $SD_{NIST}$ | 0.007 | 0.002 | 0.001 | 0.003 |
| $CV_{NIST}$ | 16 | 16 | 25 | 13 |

| xxviii | xxxi | xxxi | xxxi |
|---|---|---|---|
| 181 | 195 | 197 | 198 |
| 3 | 4 | 4 | 4 |
| 0.026 | 0.017 | 0.009 | 0.025 |
| 0.004 | 0.019 | 0.005 | 0.013 |

| | RRXLIII | | | | xxxvii | xxxv | xxxv | xxxv |
|---|---|---|---|---|---|---|---|---|
| | 243 | 244 | 245 | 246 | 222 | 214 | 211 | 212 |
| $n_n$ | 14 | 14 | 12 | 14 | 10 | 6 | 6 | 6 |
| $Median_n$ | 0.031 | 0.008 | 0.005 | 0.017 | 0.039 | 0.012 | 0.006 | 0.019 |
| $eSD_n$ | 0.014 | 0.003 | 0.004 | 0.006 | 0.013 | 0.002 | 0.003 | 0.004 |
| P(n=p) | 0.82 | 0.59 | 0.88 | 0.83 | | | | |
| P(n<p) | 0.48 | 1.00 | 0.82 | 0.99 | | | | |
| $SD_{labs}$ | 0.012 | 0.002 | 0.003 | 0.005 | | | | |
| $CV_{labs}$ | 37 | 26 | 66 | 29 | | | | |
| NAV | 0.039 | 0.010 | 0.005 | 0.021 | | | | |
| NAU | 0.014 | 0.003 | 0.004 | 0.006 | | | | |
| CV | 35 | 28 | 68 | 28 | | | | |

## "Lutein&Zeaxanthin"

| | NIST1 | | | | NIST3 | | | |
|---|---|---|---|---|---|---|---|---|
| | 243 | 244 | 245 | 246 | 243 | 244 | 245 | 246 |
| A:1 | 0.103 | 0.023 | 0.012 | 0.088 | 0.135 | 0.037 | 0.016 | 0.079 |
| A:2 | 0.103 | 0.021 | *nd* | 0.083 | 0.136 | 0.046 | 0.017 | 0.076 |
| B:1 | 0.110 | 0.017 | 0.012 | 0.089 | 0.150 | 0.040 | 0.017 | 0.083 |
| B:2 | 0.111 | 0.023 | *nd* | 0.090 | 0.141 | 0.038 | 0.022 | 0.082 |
| C:1 | 0.112 | 0.020 | *nd* | 0.093 | 0.143 | 0.038 | | 0.080 |
| C:2 | 0.106 | 0.021 | 0.012 | 0.082 | 0.143 | 0.035 | 0.021 | 0.079 |
| $n_x$ | 6 | 6 | 3 | 6 | 6 | 6 | 5 | 6 |
| $Mean_x$ | 0.107 | 0.021 | 0.012 | 0.088 | 0.141 | 0.039 | 0.019 | 0.080 |
| $SD_x$ | 0.004 | 0.002 | 0.000 | 0.004 | 0.006 | 0.004 | 0.003 | 0.002 |
| $SD_{repx}$ | 0.003 | 0.003 | | 0.005 | 0.004 | 0.004 | 0.002 | 0.001 |
| $SD_{betx}$ | 0.004 | 0.001 | | 0.002 | 0.005 | 0.002 | 0.002 | 0.002 |
| $SD_{NISTx}$ | 0.005 | 0.003 | | 0.005 | 0.007 | 0.005 | 0.003 | 0.003 |
| $CV_{NISTx}$ | 4.4 | 14 | | 6.1 | 4.6 | 12 | 16 | 3.5 |

| | NIST | | | |
|---|---|---|---|---|
| n | 12 | 12 | 8 | 12 |
| Mean | 0.124 | 0.030 | 0.016 | 0.084 |
| $SD_{rep}$ | 0.003 | 0.003 | 0.001 | 0.004 |
| $SD_{bet}$ | 0.010 | 0.008 | 0.003 | 0.013 |
| $SD_{sml}$ | 0.018 | 0.018 | 0.018 | 0.018 |
| $SD_{NIST}$ | 0.021 | 0.020 | 0.019 | 0.023 |
| $CV_{NIST}$ | 17 | 68 | 120 | 27 |

| xxviii | xxxi | xxxi | xxxi |
|---|---|---|---|
| 181 | 195 | 197 | 198 |
| 7 | 12 | 12 | 13 |
| 0.109 | 0.047 | 0.027 | 0.081 |
| 0.020 | 0.011 | 0.016 | 0.013 |

| | RRXLIII | | | | xxxvii | xxxv | xxxv | xxxv |
|---|---|---|---|---|---|---|---|---|
| | 243 | 244 | 245 | 246 | 222 | 214 | 211 | 212 |
| $n_n$ | 27 | 27 | 24 | 26 | 17 | 16 | 15 | 16 |
| $Median_n$ | 0.117 | 0.033 | 0.018 | 0.067 | 0.101 | 0.040 | 0.020 | 0.074 |
| $eSD_n$ | 0.030 | 0.007 | 0.005 | 0.013 | 0.041 | 0.009 | 0.006 | 0.013 |
| P(n=p) | 0.90 | 0.78 | 0.88 | 0.87 | | | | |
| P(n<p) | 0.93 | 0.98 | 1.00 | 0.52 | | | | |
| $SD_{labs}$ | 0.021 | 0 | 0 | 0 | | | | |
| $CV_{labs}$ | 18 | 0 | 0 | 0 | | | | |
| NAV | 0.121 | 0.031 | 0.017 | 0.075 | | | | |
| NAU | 0.030 | 0.020 | 0.019 | 0.023 | | | | |
| CV | 25 | 65 | 110 | 30 | | | | |

## Appendix G. "All-Lab Report" for RR43

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

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

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

# Round Robin XLIII Laboratory Results

All Values in µg/mL

| | Retinol | | | | Retinyl Palmitate | | | | α-Tocopherol | | | | γ-Tocopherol | | | | δ-Tocopherol | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Lab | 243 | 244 | 245 | 246 | 243 | 244 | 245 | 246 | 243 | 244 | 245 | 246 | 243 | 244 | 245 | 246 | 243 | 244 | 245 | 246 |
| FSV-BA | 0.591 | 0.484 | 0.247 | 0.809 | 0.112 | 0.096 | 0.045 | 0.246 | 7.22 | 7.09 | 3.35 | 14.4 | 2.18 | 1.31 | 0.71 | 2.44 | | | | |
| FSV-BD | 0.436 | 0.409 | 0.151 | 0.727 | | | | | 8.31 | 7.76 | 4.38 | 16.1 | | | | | | | | |
| FSV-BE | 0.443 | 0.342 | 0.197 | 0.681 | | | | | 6.88 | 7.39 | 3.78 | 13.3 | 2.36 | 1.37 | 0.77 | 2.62 | | | | |
| FSV-BF | 0.495 | 0.401 | 0.189 | 0.791 | | | | | 6.98 | 7.53 | 3.32 | 14.6 | 2.32 | 1.27 | 0.67 | 2.53 | | | | |
| FSV-BG | 0.485 | 0.364 | 0.197 | 0.745 | 0.090 | 0.097 | 0.061 | 0.236 | 6.99 | 6.41 | 3.40 | 13.9 | 2.31 | 1.20 | 0.69 | 2.52 | | | | |
| FSV-BH | 0.464 | 0.347 | 0.183 | 0.715 | 0.081 | 0.109 | 0.039 | 0.251 | 7.58 | 7.86 | 3.87 | 15.2 | 2.71 | 1.56 | 0.83 | 3.05 | | | | |
| FSV-BI | 0.466 | 0.383 | 0.190 | 0.765 | 0.135 | 0.131 | 0.052 | 0.321 | 6.79 | 7.26 | 3.61 | 15.0 | 2.13 | 1.28 | 0.76 | 2.52 | | | | |
| FSV-BIa | 0.485 | 0.364 | 0.197 | 0.745 | | | | | | | | | | | | | | | | |
| FSV-BJ | 0.466 | 0.386 | 0.191 | 0.726 | 0.105 | 0.072 | *nd* | 0.283 | 7.44 | 8.71 | 4.46 | 15.4 | 2.68 | 1.60 | 0.80 | 2.81 | | | | |
| FSV-BK | 0.449 | 0.357 | 0.182 | 0.722 | | | | | 6.63 | 6.92 | 3.27 | 13.3 | | | | | | | | |
| FSV-BL | 0.400 | 0.200 | < 0.11 | 0.460 | | | | | 6.46 | 4.74 | 2.58 | 12.9 | | | | | | | | |
| FSV-BM | 0.434 | 0.350 | 0.140 | 0.696 | | | | | 7.80 | 7.20 | 3.40 | 14.4 | | | | | | | | |
| FSV-BN | 0.480 | 0.380 | 0.180 | 0.794 | 0.095 | 0.129 | 0.050 | 0.308 | 7.03 | 7.32 | 3.42 | 15.1 | 1.98 | 1.13 | 0.64 | 2.40 | 0.238 | 0.152 | 0.084 | 0.285 |
| FSV-BO | 0.630 | 0.513 | 0.246 | 0.897 | | | | | 5.69 | 5.81 | 2.84 | 11.8 | | | | | | | | |
| FSV-BQ | 0.488 | 0.435 | 0.237 | 0.777 | | | | | 7.38 | 7.49 | 3.92 | 14.4 | | | | | | | | |
| FSV-BR | 0.480 | 0.390 | 0.200 | 0.750 | | | | | | | | | | | | | | | | |
| FSV-BS | 0.516 | 0.427 | 0.212 | 0.718 | | | | | | | | | | | | | | | | |
| FSV-BT | 0.439 | 0.346 | 0.354 | 0.596 | 0.110 | 0.109 | 0.075 | 0.280 | 6.57 | 6.23 | 2.68 | 12.3 | 2.51 | 1.29 | 0.59 | 2.49 | 0.382 | 0.153 | 0.051 | 0.349 |
| FSV-BU | 0.516 | 0.415 | 0.188 | 0.765 | | | | | 6.88 | 7.15 | 3.91 | 13.5 | 2.99 | 1.55 | 1.10 | 2.69 | | | | |
| FSV-BV | 0.490 | 0.350 | 0.130 | 0.570 | | | | | 6.29 | 6.60 | 3.00 | 12.3 | 2.15 | 1.23 | 0.62 | 2.33 | | | | |
| FSV-BW | 0.488 | 0.373 | 0.191 | 0.710 | 0.134 | 0.153 | 0.052 | 0.400 | 6.40 | 6.80 | 3.42 | 13.3 | 2.27 | 1.25 | 0.68 | 2.48 | | | | |
| FSV-BX | 0.550 | 0.396 | 0.208 | 0.840 | | | | | 7.14 | 7.29 | 3.76 | 14.4 | 2.51 | 1.29 | 0.66 | 2.63 | | | | |
| FSV-BZ | | | | | | | | | 6.40 | 8.70 | 4.10 | 12.8 | 1.50 | 1.40 | 0.98 | 2.50 | | | | |
| FSV-CB | 0.575 | 0.453 | 0.224 | 0.807 | | | | | 7.50 | 7.86 | 3.66 | 14.9 | | | | | | | | |
| FSV-CC | 0.485 | 0.376 | 0.170 | 0.694 | | | | | 6.98 | 7.45 | 3.25 | 15.3 | | | | | | | | |
| FSV-CD | 0.446 | 0.360 | 0.194 | 0.611 | 0.161 | 0.162 | 0.082 | 0.401 | 7.57 | 7.50 | 3.81 | 14.1 | 2.32 | 1.24 | 0.62 | 2.31 | | | | |
| FSV-CE | 0.463 | 0.373 | 0.191 | 0.700 | | | | | 6.73 | 7.22 | 2.53 | 13.8 | | | | | | | | |
| FSV-CF | 0.479 | 0.380 | 0.197 | 0.723 | | | | | 7.90 | 7.70 | 3.80 | 15.6 | | | | | | | | |
| FSV-CH | 0.362 | 0.314 | 0.170 | 0.579 | | | | | 5.71 | 6.89 | 3.63 | 12.0 | 1.63 | 1.09 | 0.56 | 1.72 | | | | |
| FSV-CI | 0.510 | 0.420 | 0.190 | 0.790 | 0.220 | 0.160 | 0.050 | 0.350 | 5.90 | 6.20 | 2.70 | 11.8 | | | | | | | | |
| FSV-CK | 0.445 | 0.339 | 0.185 | 0.641 | | | | | 6.96 | 6.51 | 3.70 | 12.9 | 2.23 | 1.24 | 0.69 | 2.42 | | | | |
| FSV-CL | 0.368 | 0.334 | 0.157 | 0.685 | | | | | 8.06 | 8.28 | 4.38 | 16.5 | 2.14 | 1.26 | 0.65 | 2.63 | | | | |
| FSV-CM | | | | | | | | | 7.10 | 7.10 | 3.70 | 14.9 | | | | | | | | |
| FSV-CN | 0.463 | 0.363 | 0.205 | 0.729 | | | | | 6.95 | 7.09 | 3.25 | 14.2 | 1.78 | 0.94 | 0.44 | 2.06 | | | | |
| FSV-CP | | | | | | | | | 7.36 | 7.24 | !2.29 | !5.17 | 2.51 | 1.37 | !12.39 | !0.90 | | | | |
| FSV-CQ | 0.463 | 0.355 | 0.200 | 0.748 | | | | | 6.60 | 6.75 | 3.51 | 16.3 | | | | | | | | |
| FSV-CR | 0.490 | 0.420 | 0.210 | 0.790 | | | | | 7.10 | 8.60 | 4.10 | 15.4 | | | | | <0.3 | <0.3 | <0.3 | <0.3 |
| FSV-CS | 0.446 | 0.376 | 0.189 | 0.731 | | | | | 6.23 | 7.39 | 2.70 | 15.4 | 2.03 | 1.20 | 0.47 | 2.58 | | | | |
| FSV-CT | 0.519 | 0.427 | 0.217 | 0.881 | | | | | 8.09 | 8.83 | 4.48 | 16.0 | | | | | | | | |
| FSV-CU | 0.466 | 0.389 | 0.200 | 0.694 | 0.139 | 0.095 | 0.052 | 0.222 | 6.99 | 7.14 | 3.36 | 13.6 | | | | | | | | |
| FSV-CX | 0.440 | 0.380 | 0.210 | 0.690 | 0.090 | 0.060 | 0.040 | 0.230 | 6.91 | 7.36 | 3.95 | 13.9 | 2.54 | 1.52 | 0.74 | 2.95 | | | | |
| FSV-DA | 0.454 | 0.389 | 0.203 | 0.706 | 0.137 | 0.086 | 0.060 | 0.247 | 7.71 | 7.80 | 3.95 | 14.3 | 2.33 | 1.35 | 0.76 | 2.51 | 0.122 | 0.079 | 0.074 | 0.136 |
| FSV-DB | 0.500 | 0.369 | 0.195 | 0.734 | | | | | 6.79 | 6.95 | 3.50 | 13.0 | | | | | | | | |
| FSV-DF | 0.500 | 0.410 | 0.200 | 0.800 | | | | | | | | | | | | | | | | |
| FSV-DJ | 0.460 | 0.370 | 0.190 | 0.760 | | | | | 8.30 | 8.50 | 4.90 | 16.4 | | | | | | | | |
| FSV-DP | 0.474 | 0.369 | 0.175 | 0.697 | | | | | | | | | | | | | | | | |
| FSV-DR | 0.470 | 0.400 | 0.195 | 0.790 | | | | | 6.72 | 7.21 | 3.32 | 14.3 | | | | | | | | |
| FSV-DU | 0.490 | 0.410 | 0.190 | 0.810 | | | | | 7.34 | 7.54 | 4.39 | 14.9 | | | | | | | | |
| FSV-DW | 0.499 | 0.400 | 0.233 | 0.722 | | | | | 7.89 | 7.74 | 4.33 | 13.5 | | | | | | | | |
| FSV-EL | 0.560 | 0.370 | 0.140 | 0.550 | | | | | | | | | | | | | | | | |
| FSV-EM | 0.510 | 0.420 | 0.200 | 0.760 | | | | | 6.24 | 7.12 | 3.01 | 12.8 | | | | | | | | |
| FSV-ES | 0.450 | 0.355 | 0.184 | 0.691 | | | | | 6.83 | 6.59 | 3.46 | 13.9 | | | | | | | | |
| FSV-FG | 0.490 | 0.350 | 0.150 | 0.720 | | | | | 5.80 | 5.80 | 2.15 | 12.8 | | | | | | | | |
| FSV-FH | | | | | | | | | | 6.70 | 3.10 | 14.0 | | | | | | | | |
| n | 50 | 50 | 49 | 50 | 13 | 13 | 12 | 13 | 47 | 48 | 47 | 47 | 23 | 23 | 22 | 22 | 3 | 3 | 3 | 3 |
| Min | 0.362 | 0.200 | 0.130 | 0.460 | 0.081 | 0.060 | 0.039 | 0.222 | 5.69 | 4.74 | 2.15 | 11.8 | 1.50 | 0.94 | 0.44 | 1.72 | 0.122 | 0.079 | 0.051 | 0.136 |
| Median | 0.480 | 0.378 | 0.194 | 0.727 | 0.112 | 0.109 | 0.052 | 0.280 | 6.98 | 7.23 | 3.51 | 14.2 | 2.31 | 1.28 | 0.68 | 2.51 | 0.238 | 0.152 | 0.074 | 0.285 |
| Max | 0.630 | 0.513 | 0.354 | 0.897 | 0.220 | 0.162 | 0.082 | 0.401 | 8.31 | 8.83 | 4.90 | 16.5 | 2.99 | 1.60 | 1.10 | 3.05 | 0.382 | 0.153 | 0.084 | 0.349 |
| eSD | 0.030 | 0.034 | 0.015 | 0.054 | 0.033 | 0.033 | 0.011 | 0.060 | 0.59 | 0.57 | 0.53 | 1.3 | 0.29 | 0.12 | 0.11 | 0.16 | | | | |
| eCV | 6 | 9 | 8 | 7 | 29 | 31 | 21 | 22 | 8 | 8 | 15 | 9 | 13 | 9 | 15 | 6 | | | | |
| NISTa | 0.445 | 0.358 | 0.182 | 0.739 | | | | | 7.25 | 7.08 | 3.92 | 14.5 | 2.18 | *nd* | *nd* | 2.71 | | | | |
| NISTb | 0.443 | 0.367 | 0.196 | 0.708 | | | | | 6.84 | 7.12 | 3.18 | 14.4 | 2.24 | 1.26 | 0.66 | 2.54 | 0.149 | 0.087 | 0.051 | 0.166 |
| NAV | 0.462 | 0.370 | 0.192 | 0.725 | 0.112 | 0.109 | 0.052 | 0.280 | 7.01 | 7.16 | 3.53 | 14.3 | 2.26 | 1.27 | 0.68 | 2.57 | 0.149 | 0.087 | 0.053 | 0.166 |
| NAU | 0.047 | 0.038 | 0.020 | 0.061 | 0.033 | 0.033 | 0.011 | 0.060 | 0.66 | 0.63 | 0.62 | 1.3 | 0.29 | 0.16 | 0.10 | 0.27 | | | | |

# Round Robin XLIII Laboratory Results

All Values in µg/mL

| | Total β-Carotene | | | | trans-β-Carotene | | | | Total cis-β-Carotene | | | | Total α-Carotene | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Lab | 243 | 244 | 245 | 246 | 243 | 244 | 245 | 246 | 243 | 244 | 245 | 246 | 243 | 244 | 245 | 246 |
| FSV-BA | 0.085 | 0.337 | 0.131 | 0.70 | 0.079 | 0.315 | 0.123 | 0.669 | 0.006 | 0.022 | 0.008 | 0.034 | 0.013 | 0.013 | 0.008 | 0.041 |
| FSV-BD | 0.096 | 0.335 | 0.144 | 0.65 | | | | | | | | | | | | |
| FSV-BE | 0.081 | 0.357 | 0.094 | 0.76 | | | | | | | | | | | | |
| FSV-BF | 0.070 | 0.416 | 0.206 | 0.69 | | | | | | | | | 0.014 | 0.025 | 0.021 | 0.034 |
| FSV-BG | 0.071 | 0.311 | 0.117 | 0.66 | | | | | | | | | 0.016 | 0.018 | *nq* | 0.032 |
| FSV-BH | 0.071 | 0.340 | 0.169 | 0.68 | 0.071 | 0.315 | 0.155 | 0.633 | *nq* | 0.025 | 0.014 | 0.051 | 0.013 | 0.017 | 0.009 | 0.039 |
| FSV-BI | 0.069 | 0.338 | 0.138 | 0.66 | | | | | | | | | 0.012 | 0.014 | 0.007 | 0.032 |
| FSV-BIa | | | | | | | | | | | | | | | | |
| FSV-BJ | 0.063 | 0.326 | 0.074 | 0.88 | | | | | | | | | *nd* | 0.013 | *nd* | *nd* |
| FSV-BK | | | | | | | | | | | | | | | | |
| FSV-BL | | | | | | | | | | | | | | | | |
| FSV-BM | | | | | | | | | | | | | | | | |
| FSV-BN | 0.071 | 0.323 | 0.144 | 0.65 | 0.066 | 0.284 | 0.127 | 0.587 | 0.002 | 0.037 | 0.012 | 0.060 | 0.012 | 0.014 | 0.005 | 0.027 |
| FSV-BO | 0.080 | 0.321 | 0.132 | 0.56 | | | | | | | | | 0.013 | 0.013 | 0.011 | 0.025 |
| FSV-BQ | | | | | | | | | | | | | | | | |
| FSV-BR | | | | | | | | | | | | | | | | |
| FSV-BS | 0.091 | 0.320 | 0.140 | 0.48 | | | | | | | | | 0.038 | 0.040 | 0.032 | 0.043 |
| FSV-BT | 0.056 | 0.310 | 0.145 | 0.57 | 0.063 | 0.288 | 0.134 | 0.536 | 0.002 | 0.022 | 0.010 | 0.039 | 0.017 | 0.012 | 0.005 | 0.024 |
| FSV-BU | 0.069 | 0.323 | 0.155 | 0.65 | | | | | | | | | 0.021 | 0.026 | 0.165 | 0.048 |
| FSV-BV | 0.072 | 0.316 | 0.156 | 0.61 | | | | | | | | | 0.012 | 0.190 | 0.010 | 0.038 |
| FSV-BW | 0.087 | 0.358 | 0.126 | 0.74 | | | | | | | | | 0.013 | 0.011 | *nq* | 0.024 |
| FSV-BX | 0.073 | 0.145 | 0.144 | 0.21 | | | | | | | | | 0.015 | 0.010 | 0.010 | 0.015 |
| FSV-BZ | | | | | | | | | 0.005 | 0.009 | 0.003 | 0.020 | 0.020 | 0.020 | 0.015 | 0.028 |
| FSV-CB | 0.051 | 0.291 | 0.122 | 0.45 | | | | | | | | | 0.009 | 0.019 | 0.007 | 0.033 |
| FSV-CC | | | | | | | | | | | | | | | | |
| FSV-CD | 0.070 | 0.310 | 0.128 | 0.55 | | | | | | | | | 0.014 | 0.012 | 0.005 | 0.023 |
| FSV-CE | 0.096 | 0.388 | 0.060 | 0.90 | | | | | | | | | | | | |
| FSV-CF | | | | | | | | | | | | | | | | |
| FSV-CH | 0.104 | 0.322 | 0.201 | 0.55 | | | | | | | | | 0.016 | 0.023 | 0.031 | 0.024 |
| FSV-CI | | | | | | | | | | | | | | | | |
| FSV-CK | 0.113 | 0.445 | 0.174 | 0.87 | | | | | | | | | 0.021 | 0.024 | 0.010 | 0.044 |
| FSV-CL | 0.046 | 0.346 | 0.158 | 0.63 | | | | | | | | | 0.011 | 0.026 | 0.011 | 0.043 |
| FSV-CM | | | | | | | | | | | | | | | | |
| FSV-CN | >0.063 | >0.274 | >0.094 | >0.72 | 0.063 | 0.274 | 0.094 | 0.716 | | | | | *nq* | *nq* | *nq* | 0.029 |
| FSV-CP | 0.075 | 0.333 | !0.572 | !0.22 | | | | | | | | | 0.010 | 0.016 | !0.028 | !0.012 |
| FSV-CQ | 0.066 | 0.361 | 0.143 | 0.75 | | | | | | | | | | | | |
| FSV-CR | | | | | | | | | | | | | | | | |
| FSV-CS | 0.071 | 0.384 | 0.170 | 0.70 | 0.066 | 0.344 | 0.150 | 0.623 | *0.005* | *0.040* | *0.020* | *0.073* | 0.011 | 0.024 | 0.008 | 0.044 |
| FSV-CT | 0.076 | 0.399 | 0.163 | 0.05 | | | | | | | | | | | | |
| FSV-CU | >0.081 | >0.308 | >0.151 | >0.67 | 0.081 | 0.308 | 0.151 | 0.666 | | | | | | | | |
| FSV-CX | 0.070 | 0.300 | 0.160 | 0.56 | | | | | | | | | 0.010 | <0.01 | <0.01 | 0.030 |
| FSV-DA | 0.085 | 0.350 | 0.179 | 0.72 | 0.076 | 0.304 | 0.153 | 0.637 | 0.009 | 0.046 | 0.026 | 0.081 | 0.014 | 0.018 | 0.008 | 0.037 |
| FSV-DB | 0.079 | 0.401 | 0.161 | 0.86 | | | | | | | | | | | | |
| FSV-DF | | | | | | | | | | | | | | | | |
| FSV-DJ | | | | | | | | | | | | | | | | |
| FSV-DP | | | | | | | | | | | | | | | | |
| FSV-DR | 0.084 | 0.380 | 0.185 | 0.83 | | | | | | | | | | | | |
| FSV-DU | >0.075 | >0.270 | >0.140 | >0.28 | 0.075 | 0.270 | 0.140 | 0.280 | | | | | | | | |
| FSV-DW | 0.087 | 0.341 | 0.184 | 0.70 | 0.082 | 0.310 | 0.161 | 0.631 | 0.005 | 0.031 | 0.023 | 0.067 | 0.047 | 0.024 | 0.013 | 0.049 |
| FSV-EL | | | | | | | | | | | | | | | | |
| FSV-EM | 0.035 | 0.238 | 0.043 | 0.64 | | | | | | | | | <0.015 | <0.015 | <0.015 | 0.021 |
| FSV-ES | >0.050 | >0.318 | >0.156 | >0.61 | 0.050 | 0.318 | 0.156 | 0.613 | | | | | | | | |
| FSV-FG | | | | | | | | | | | | | | | | |
| FSV-FH | | | | | | | | | | | | | | | | |
| n | 32 | 32 | 31 | 31 | 11 | 11 | 11 | 11 | 7 | 8 | 8 | 8 | 24 | 24 | 20 | 25 |
| Min | 0.035 | 0.145 | 0.043 | 0.05 | 0.050 | 0.270 | 0.094 | 0.280 | 0.002 | 0.009 | 0.003 | 0.020 | 0.009 | 0.010 | 0.005 | 0.015 |
| Median | 0.073 | 0.336 | 0.144 | 0.66 | 0.071 | 0.308 | 0.150 | 0.631 | 0.005 | 0.028 | 0.013 | 0.056 | 0.014 | 0.018 | 0.010 | 0.032 |
| Max | 0.113 | 0.445 | 0.206 | 0.90 | 0.082 | 0.344 | 0.161 | 0.716 | 0.009 | 0.046 | 0.026 | 0.081 | 0.047 | 0.190 | 0.165 | 0.049 |
| eSD | 0.011 | 0.032 | 0.027 | 0.13 | 0.012 | 0.015 | 0.015 | 0.052 | 0.001 | 0.011 | 0.009 | 0.025 | 0.003 | 0.008 | 0.004 | 0.012 |
| eCV | 16 | 9 | 19 | 19 | 16 | 5 | 10 | 8 | 30 | 40 | 68 | 45 | 25 | 44 | 41 | 37 |
| NISTa | 0.076 | 0.392 | 0.148 | 0.63 | 0.072 | 0.342 | 0.133 | 0.598 | *0.004* | *0.050* | *0.015* | *0.031* | *nd* | 0.027 | *nd* | 0.044 |
| NISTb | >0.066 | 0.374 | >0.095 | 0.76 | 0.066 | 0.332 | 0.095 | 0.685 | *nq* | *0.041* | *nq* | *0.073* | >0.014 | >0.016 | *nq* | 0.050 |
| NAV | 0.072 | 0.359 | 0.133 | 0.68 | 0.070 | 0.323 | 0.133 | 0.636 | 0.004 | 0.037 | 0.014 | 0.054 | 0.014 | 0.020 | 0.010 | 0.039 |
| NAU | 0.015 | 0.060 | 0.032 | 0.12 | 0.011 | 0.040 | 0.040 | 0.066 | 0.003 | 0.017 | 0.010 | 0.032 | 0.005 | 0.009 | 0.005 | 0.016 |

# Round Robin XLIII Laboratory Results

All Values in µg/mL

| | trans-α-Carotene | | | | Total Lycopene | | | | trans-Lycopene | | | | Total β-Cryptoxanthin | | | | Total α-Cryptoxanthin | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Lab | 243 | 244 | 245 | 246 | 243 | 244 | 245 | 246 | 243 | 244 | 245 | 246 | 243 | 244 | 245 | 246 | 243 | 244 | 245 | 246 |
| FSV-BA | | | | | | | | | 0.312 | 0.047 | 0.014 | 0.080 | 0.074 | 0.029 | 0.068 | 0.031 | | | | |
| FSV-BD | | | | | 0.40 | 0.064 | 0.022 | 0.105 | | | | | 0.043 | 0.006 | 0.005 | 0.011 | | | | |
| FSV-BE | | | | | | | | | | | | | | | | | | | | |
| FSV-BF | | | | | 0.46 | 0.080 | 0.027 | 0.142 | | | | | 0.038 | 0.009 | 0.004 | 0.016 | | | | |
| FSV-BG | | | | | 0.48 | 0.071 | 0.037 | 0.123 | 0.274 | 0.030 | 0.020 | 0.058 | 0.050 | 0.009 | *nq* | 0.014 | | | | |
| FSV-BH | | | | | 0.59 | 0.076 | 0.035 | 0.159 | | | | | 0.071 | *nq* | *nq* | 0.025 | | | | |
| FSV-BI | | | | | 0.37 | 0.048 | 0.026 | 0.111 | | | | | 0.059 | 0.011 | 0.005 | 0.022 | | | | |
| FSV-BIa | | | | | | | | | | | | | | | | | | | | |
| FSV-BJ | | | | | 0.34 | 0.040 | *nd* | 0.095 | | | | | 0.062 | *nd* | *nd* | *nd* | | | | |
| FSV-BK | | | | | | | | | | | | | | | | | | | | |
| FSV-BL | | | | | | | | | | | | | | | | | | | | |
| FSV-BM | | | | | | | | | | | | | | | | | | | | |
| FSV-BN | | | | | 0.54 | 0.071 | 0.038 | 0.154 | 0.300 | 0.035 | 0.017 | 0.073 | 0.065 | 0.016 | 0.010 | 0.027 | 0.033 | 0.010 | 0.008 | 0.013 |
| FSV-BO | | | | | 0.59 | 0.071 | 0.034 | 0.108 | | | | | 0.047 | 0.010 | *nq* | 0.019 | | | | |
| FSV-BQ | | | | | | | | | | | | | | | | | | | | |
| FSV-BR | | | | | | | | | | | | | | | | | | | | |
| FSV-BS | | | | | 0.54 | 0.091 | 0.038 | 0.139 | 0.220 | 0.033 | 0.017 | 0.050 | 0.041 | 0.010 | 0.003 | 0.013 | | | | |
| FSV-BT | | | | | 0.43 | 0.066 | 0.023 | 0.125 | 0.348 | 0.049 | 0.020 | 0.091 | 0.069 | 0.014 | 0.005 | 0.025 | 0.040 | 0.006 | 0.004 | 0.012 |
| FSV-BU | | | | | 0.33 | 0.025 | 0.006 | 0.060 | | | | | 0.024 | 0.005 | 0.001 | 0.016 | | | | |
| FSV-BV | | | | | 0.05 | 0.070 | 0.033 | 0.136 | | | | | 0.042 | 0.005 | 0.002 | 0.011 | | | | |
| FSV-BW | | | | | 0.54 | 0.041 | *nq* | 0.114 | | | | | | | | | | | | |
| FSV-BX | | | | | 0.15 | 0.023 | 0.014 | 0.035 | | | | | 0.057 | 0.009 | 0.005 | 0.018 | | | | |
| FSV-BZ | | | | | 0.40 | 0.180 | *nd* | 0.210 | | | | | | | | | | | | |
| FSV-CB | | | | | 0.11 | 0.022 | 0.009 | 0.034 | | | | | 0.042 | 0.006 | 0.002 | 0.013 | | | | |
| FSV-CC | | | | | | | | | | | | | | | | | | | | |
| FSV-CD | | | | | 0.54 | 0.102 | 0.040 | 0.176 | | | | | 0.058 | 0.010 | 0.005 | 0.018 | 0.030 | 0.005 | 0.003 | 0.009 |
| FSV-CE | | | | | | | | | | | | | | | | | | | | |
| FSV-CF | | | | | | | | | | | | | | | | | | | | |
| FSV-CH | | | | | 0.40 | 0.079 | 0.044 | 0.092 | | | | | | | | | | | | |
| FSV-CI | | | | | | | | | | | | | | | | | | | | |
| FSV-CK | | | | | 0.61 | 0.093 | 0.035 | 0.178 | | | | | 0.058 | 0.009 | 0.006 | 0.019 | 0.049 | 0.007 | 0.005 | 0.015 |
| FSV-CL | | | | | 0.43 | 0.067 | 0.027 | 0.122 | | | | | 0.041 | 0.007 | 0.005 | 0.014 | 0.038 | 0.003 | *nd* | 0.012 |
| FSV-CM | | | | | | | | | | | | | | | | | | | | |
| FSV-CN | | | | | 0.42 | *nq* | *nq* | 0.099 | | | | | 0.059 | 0.005 | *nq* | 0.018 | | | | |
| FSV-CP | | | | | 0.31 | 0.039 | !0.067 | !0.025 | | | | | 0.064 | 0.009 | !0.018 | !0.001 | | | | |
| FSV-CQ | | | | | | | | | | | | | | | | | | | | |
| FSV-CR | | | | | | | | | | | | | | | | | | | | |
| FSV-CS | | | | | 0.48 | 0.087 | 0.030 | 0.155 | | | | | 0.052 | 0.008 | 0.003 | 0.016 | | | | |
| FSV-CT | | | | | | | | | 0.314 | 0.050 | 0.032 | 0.089 | | | | | | | | |
| FSV-CU | | | | | | | | | | | | | | | | | | | | |
| FSV-CX | | | | | 0.36 | 0.060 | 0.010 | 0.100 | | | | | 0.040 | <0.01 | <0.01 | 0.010 | | | | |
| FSV-DA | | | | | 0.56 | 0.081 | 0.032 | 0.161 | 0.292 | 0.035 | 0.017 | 0.067 | 0.065 | 0.010 | 0.004 | 0.015 | 0.024 | 0.008 | *nd* | 0.008 |
| FSV-DB | | | | | 0.60 | 0.101 | 0.021 | 0.159 | | | | | 0.060 | *nq* | *nq* | 0.014 | | | | |
| FSV-DF | | | | | | | | | | | | | | | | | | | | |
| FSV-DJ | | | | | | | | | | | | | | | | | | | | |
| FSV-DP | | | | | | | | | | | | | | | | | | | | |
| FSV-DR | | | | | | | | | | | | | | | | | | | | |
| FSV-DU | | | | | | | | | | | | | | | | | | | | |
| FSV-DW | | | | | 0.17 | 0.030 | 0.012 | 0.045 | 0.104 | 0.018 | 0.007 | 0.024 | 0.057 | 0.012 | 0.006 | 0.022 | | | | |
| FSV-EL | | | | | | | | | | | | | | | | | | | | |
| FSV-EM | | | | | 0.68 | 0.079 | <0.015 | <0.195 | | | | | 0.065 | <0.015 | <0.015 | <0.015 | | | | |
| FSV-ES | 0.014 | 0.014 | *nq* | 0.023 | 0.37 | 0.062 | 0.028 | 0.092 | 0.222 | 0.026 | 0.011 | 0.048 | 0.067 | 0.007 | *nq* | 0.017 | | | | |
| FSV-FG | | | | | | | | | | | | | | | | | | | | |
| FSV-FH | | | | | | | | | | | | | | | | | | | | |
| n | 1 | 1 | 0 | 1 | 29 | 28 | 23 | 27 | 9 | 9 | 9 | 9 | 27 | 22 | 17 | 24 | 6 | 6 | 4 | 6 |
| Min | | | | | 0.05 | 0.022 | 0.006 | 0.034 | 0.104 | 0.018 | 0.007 | 0.024 | 0.024 | 0.005 | 0.001 | 0.010 | 0.024 | 0.003 | 0.003 | 0.008 |
| Median | 0.014 | 0.014 | | 0.023 | 0.43 | 0.071 | 0.028 | 0.122 | 0.292 | 0.035 | 0.017 | 0.067 | 0.058 | 0.009 | 0.005 | 0.016 | 0.035 | 0.007 | 0.005 | 0.012 |
| Max | | | | | 0.68 | 0.180 | 0.044 | 0.210 | 0.348 | 0.050 | 0.032 | 0.091 | 0.074 | 0.029 | 0.068 | 0.031 | 0.049 | 0.010 | 0.008 | 0.015 |
| eSD | | | | | 0.16 | 0.020 | 0.010 | 0.044 | 0.033 | 0.013 | 0.004 | 0.025 | 0.012 | 0.003 | 0.001 | 0.004 | 0.008 | 0.002 | | 0.003 |
| eCV | | | | | 39 | 28 | 34 | 36 | 11 | 37 | 26 | 38 | 20 | 29 | 30 | 26 | 22 | 33 | | 25 |
| NISTa | | | | | | | | | | | | | 0.043 | 0.017 | *nd* | 0.025 | | | | |
| NISTb | 0.014 | 0.016 | *nq* | 0.032 | 0.52 | *nq* | *nq* | 0.203 | 0.234 | 0.037 | *nq* | 0.075 | 0.047 | 0.007 | *nq* | 0.016 | | | | |
| NAV | 0.014 | 0.016 | | 0.032 | 0.47 | 0.071 | 0.028 | 0.163 | 0.263 | 0.036 | 0.017 | 0.071 | 0.052 | 0.011 | 0.005 | 0.018 | 0.035 | 0.007 | | 0.012 |
| NAU | | | | | 0.15 | 0.025 | 0.010 | 0.071 | 0.078 | 0.013 | 0.006 | 0.023 | 0.019 | 0.007 | 0.003 | 0.007 | | | | |

# Round Robin XLIII Laboratory Results

All Values in µg/mL

| | Total Lutein | | | | Total Zeaxanthin | | | | Total Lutein&Zeaxanthin | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Lab | 243 | 244 | 245 | 246 | 243 | 244 | 245 | 246 | 243 | 244 | 245 | 246 |
| FSV-BA | | | | | | | | | 0.178 | 0.061 | 0.042 | 0.096 |
| FSV-BD | 0.076 | 0.024 | 0.013 | 0.050 | 0.031 | 0.008 | 0.002 | 0.016 | *0.107* | *0.032* | *0.015* | *0.066* |
| FSV-BE | | | | | | | | | | | | |
| FSV-BF | | | | | | | | | 0.110 | 0.041 | 0.018 | 0.078 |
| FSV-BG | 0.094 | 0.022 | 0.013 | 0.047 | 0.023 | 0.007 | 0.005 | 0.013 | 0.128 | 0.032 | 0.020 | 0.066 |
| FSV-BH | 0.061 | 0.023 | 0.012 | 0.046 | 0.024 | 0.007 | 0.004 | 0.014 | *0.085* | *0.030* | *0.016* | *0.060* |
| FSV-BI | 0.060 | 0.023 | 0.012 | 0.047 | 0.024 | 0.008 | 0.005 | 0.016 | *0.084* | *0.031* | *0.017* | *0.063* |
| FSV-BIa | | | | | | | | | | | | |
| FSV-BJ | | | | | | | | | 0.066 | 0.025 | 0.012 | 0.044 |
| FSV-BK | | | | | | | | | | | | |
| FSV-BL | | | | | | | | | | | | |
| FSV-BM | | | | | | | | | | | | |
| FSV-BN | 0.071 | 0.029 | 0.013 | 0.049 | 0.016 | 0.004 | 0.001 | 0.009 | 0.081 | 0.033 | 0.015 | 0.056 |
| FSV-BO | 0.121 | 0.037 | 0.020 | 0.063 | 0.044 | 0.013 | 0.010 | 0.023 | *0.166* | *0.050* | *0.030* | *0.086* |
| FSV-BQ | | | | | | | | | | | | |
| FSV-BR | | | | | | | | | | | | |
| FSV-BS | | | | | | | | | 0.105 | 0.036 | 0.011 | 0.054 |
| FSV-BT | 0.089 | 0.029 | 0.026 | 0.050 | 0.031 | 0.008 | 0.008 | 0.015 | 0.120 | 0.038 | 0.035 | 0.066 |
| FSV-BU | | | | | | | | | 0.117 | 0.031 | 0.022 | 0.068 |
| FSV-BV | | | | | | | | | 0.094 | 0.030 | 0.014 | 0.058 |
| FSV-BW | | | | | | | | | | | | |
| FSV-BX | 0.082 | 0.028 | 0.013 | 0.057 | 0.039 | 0.009 | 0.003 | 0.020 | 0.121 | 0.037 | 0.016 | 0.078 |
| FSV-BZ | 0.078 | 0.019 | 0.002 | 0.028 | | | | | | | | |
| FSV-CB | 0.086 | 0.028 | 0.007 | 0.059 | 0.035 | 0.009 | *nd* | 0.019 | *0.121* | *0.037* | *0.007* | *0.077* |
| FSV-CC | | | | | | | | | | | | |
| FSV-CD | | | | | | | | | 0.086 | 0.030 | 0.016 | 0.051 |
| FSV-CE | | | | | | | | | | | | |
| FSV-CF | | | | | | | | | | | | |
| FSV-CH | | | | | | | | | | | | |
| FSV-CI | | | | | | | | | | | | |
| FSV-CK | | | | | | | | | 0.138 | 0.047 | 0.025 | 0.073 |
| FSV-CL | | | | | | | | | 0.085 | 0.030 | 0.015 | 0.060 |
| FSV-CM | | | | | | | | | | | | |
| FSV-CN | 0.102 | 0.052 | 0.042 | 0.077 | 0.044 | 0.033 | 0.034 | 0.038 | 0.146 | 0.085 | 0.076 | 0.116 |
| FSV-CP | | | | | | | | | 0.139 | 0.039 | !0.071 | !0.029 |
| FSV-CQ | | | | | | | | | | | | |
| FSV-CR | | | | | | | | | | | | |
| FSV-CS | | | | | | | | | 0.132 | 0.033 | 0.018 | 0.069 |
| FSV-CT | 0.079 | 0.028 | 0.015 | 0.052 | 0.025 | 0.008 | 0.004 | 0.018 | *0.104* | *0.036* | *0.019* | *0.070* |
| FSV-CU | | | | | | | | | | | | |
| FSV-CX | 0.040 | 0.010 | <0.01 | 0.020 | <0.01 | <0.01 | <0.01 | <0.01 | | | | |
| FSV-DA | 0.086 | 0.027 | 0.013 | 0.059 | 0.048 | 0.012 | 0.005 | 0.025 | 0.134 | 0.039 | 0.018 | 0.084 |
| FSV-DB | | | | | | | | | 0.097 | 0.030 | 0.016 | 0.061 |
| FSV-DF | | | | | | | | | | | | |
| FSV-DJ | | | | | | | | | | | | |
| FSV-DP | | | | | | | | | | | | |
| FSV-DR | | | | | | | | | | | | |
| FSV-DU | | | | | | | | | | | | |
| FSV-DW | 0.090 | 0.030 | 0.015 | 0.057 | 0.047 | 0.012 | 0.010 | 0.023 | *0.137* | *0.042* | *0.025* | *0.080* |
| FSV-EL | | | | | | | | | | | | |
| FSV-EM | | | | | | | | | 0.152 | 0.022 | 0.017 | 0.105 |
| FSV-ES | 0.067 | 0.018 | 0.009 | 0.043 | 0.031 | 0.006 | *nq* | 0.014 | 0.098 | 0.024 | *nq* | 0.057 |
| FSV-FG | | | | | | | | | | | | |
| FSV-FH | | | | | | | | | | | | |
| n | 16 | 16 | 15 | 16 | 14 | 14 | 12 | 14 | 27 | 27 | 25 | 26 |
| Min | 0.040 | 0.010 | 0.002 | 0.020 | 0.016 | 0.004 | 0.001 | 0.009 | 0.066 | 0.022 | 0.007 | 0.044 |
| Median | 0.081 | 0.028 | 0.013 | 0.050 | 0.031 | 0.008 | 0.005 | 0.017 | 0.117 | 0.033 | 0.017 | 0.067 |
| Max | 0.121 | 0.052 | 0.042 | 0.077 | 0.048 | 0.033 | 0.034 | 0.038 | 0.178 | 0.085 | 0.076 | 0.116 |
| eSD | 0.014 | 0.006 | 0.003 | 0.010 | 0.011 | 0.002 | 0.003 | 0.005 | 0.030 | 0.006 | 0.003 | 0.015 |
| eCV | 17 | 22 | 23 | 21 | 35 | 20 | 68 | 28 | 25 | 18 | 17 | 22 |
| NISTa | 0.067 | 0.021 | 0.012 | 0.064 | 0.041 | *nd* | *nd* | 0.024 | *0.107* | *>0.021* | *>0.012* | *0.088* |
| NISTb | 0.090 | 0.026 | 0.013 | 0.054 | 0.052 | 0.013 | 0.005 | 0.026 | *0.141* | *0.039* | *0.019* | *0.080* |
| NAV | 0.079 | 0.026 | 0.013 | 0.055 | 0.039 | 0.010 | 0.005 | 0.021 | 0.121 | 0.032 | 0.016 | 0.075 |
| NAU | 0.018 | 0.007 | 0.003 | 0.013 | 0.017 | 0.005 | 0.004 | 0.008 | 0.030 | 0.008 | 0.005 | 0.019 |

# Round Robin XLIII Laboratory Results

## Analytes Reported By One Laboratory

Values in µg/mL

| Analyte | Code | 243 | 244 | 245 | 246 |
|---|---|---|---|---|---|
| Coenzyme Q10 | FSV-CH | 0.416 | 0.099 | 0.085 | 0.260 |
| Phytoene | FSV-CL | 0.105 | 0.126 | 0.059 | 0.199 |
| Phytofluene | FSV-CL | 0.053 | 0.027 | 0.005 | 0.039 |
| Total Carotenoids | FSV-EM | 1.28 | 0.54 | 0.20 | 1.16 |
| Total cis-β-Cryptoxanthin | FSV-BT | 0.027 | 0.005 | 0.001 | 0.008 |
| trans-anhydro-Lutein | FSV-BT | 0.054 | 0.013 | 0.009 | 0.025 |

## Legend

| Term | Definition |
|---|---|
| n | Number of (non-NIST) quantitative values reported for this analyte |
| Min | Minimum (non-NIST) quantitative value reported |
| Median | Median (non-NIST) quantitative value reported |
| Max | Maximum (non-NIST) quantitative value reported |
| eSD | Estimated standard deviation, calculated from the median absolute deviation from the median of the non-NIST results |
| eCV | Coefficient of Variation for (non-NIST) results: 100*eSD/Median |
| NAV | NIST Assigned Value, our estimate of the "true" analyte concentration |
| NAU | NIST Assigned Uncertainty, our estimate of the total (serum hetergeniety and inter- and intra-laboratory) standard deviation<br>For details on how we assign these quantities, see the "Analysis of Results." |
| *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 at least as large as x |
| ! | Discrepant value: heterogeneous serum, damaged sample, malfunction, etc. |
| *italics* | Not explicitly reported but calculated by NIST from reported values |

# Round Robin XLIII Laboratory Results

## Comparability Summary

| Lab | R | aT | gT | bC | tbC |
|---|---|---|---|---|---|
| FSV-BA | 1 | 1 | 1 | 1 | |
| FSV-BD | 1 | | | | |
| FSV-BE | 1 | 2 | 1 | 1 | |
| FSV-BF | 2 | 2 | | 2 | |
| FSV-BG | 2 | 1 | 1 | 3 | |
| FSV-BH | 1 | 2 | | 1 | |
| FSV-BI | 1 | 1 | 1 | 2 | |
| FSV-BIa | 3 | 1 | 1 | 1 | 1 |
| FSV-BJ | 1 | 2 | 2 | 2 | 1 |
| FSV-BK | 2 | 2 | 1 | 3 | |
| FSV-BL | 2 | 1 | 1 | 4 | |
| FSV-BM | | 3 | 3 | | |
| FSV-BN | | 1 | | | |
| FSV-BO | 4 | 4 | | | |
| FSV-BQ | 1 | 3 | 2 | 2 | |
| FSV-BR | 1 | 1 | | | |
| FSV-BS | 1 | 1 | | | 1 |
| FSV-BT | | 1 | 1 | 1 | |
| FSV-BU | 3 | 2 | 3 | 3 | |
| FSV-BV | 2 | 2 | 1 | 2 | |
| FSV-BW | 1 | 1 | 2 | 1 | |
| FSV-BX | 3 | 2 | | 2 | |
| FSV-BZ | 2 | 1 | 1 | 1 | 1 |
| FSV-CB | 2 | | | 2 | |
| FSV-CC | 3 | 3 | | 4 | |
| FSV-CD | 4 | 3 | | 1 | |
| FSV-CE | 4 | 2 | 1 | 2 | 2 |
| FSV-CF | 1 | | | | |
| FSV-CH | 3 | 1 | | | |
| FSV-CI | 1 | 3 | | | |
| FSV-CK | 3 | 2 | | | |
| FSV-CL | 2 | 3 | | | |
| FSV-CM | 2 | 1 | 1 | 2 | |
| FSV-CN | 1 | 1 | | 2 | |
| FSV-CP | 2 | 1 | 4 | 1 | |
| FSV-CQ | 1 | 2 | 1 | 2 | 1 |
| FSV-CR | 1 | | | | |
| FSV-CS | 3 | | | | |
| FSV-CT | 3 | 2 | 1 | 1 | |
| FSV-CU | 1 | 1 | 3 | | 2 |
| FSV-CX | 2 | 1 | | | |
| FSV-DA | 1 | 1 | 1 | 1 | |
| FSV-DB | 1 | 2 | | | |
| FSV-DF | 1 | 2 | 2 | 2 | 1 |
| FSV-DJ | 2 | 2 | | | 4 |
| FSV-DP | 2 | 1 | | 2 | |
| FSV-DR | 2 | 2 | | 3 | |
| FSV-DU | 2 | 2 | | | |
| FSV-DW | 1 | 2 | | 3 | |
| FSV-EL | 3 | 3 | | | |
| FSV-EM | 1 | 1 | | | 2 |
| FSV-ES | 2 | | | | |
| FSV-FG | 2 | 2 | | 2 | 2 |
| FSV-FH | | 1 | | | |
| NISTa | 1 | 1 | 1 | 1 | 1 |
| NISTb | 1 | 1 | 1 | 1 | 1 |
| n | 50 | 48 | 23 | 32 | 11 |

| | TR | aT | g/bT | bC | tbC |
|---|---|---|---|---|---|
| % 1 | 42 | 44 | 65 | 34 | 55 |
| % 2 | 34 | 40 | 17 | 44 | 36 |
| % 3 | 18 | 15 | 13 | 16 | 0 |
| % 4 | 6 | 2 | 4 | 6 | 9 |

| Label | Definition |
|---|---|
| Lab | Participant code |
| R | "Standard Score" for Retinol |
| aT | "Standard Score" for α-Tocopherol |
| gT | "Standard Score" for γ-Tocopherol |
| bC | "Standard Score" for Total β-Carotene |
| tbC | "Standard Score" for trans-β-Carotene |
| n | number of laboratories providing data for this analyte for at least one serum |

"Standard Score"

Given that our knowledge of the shapre, location, and width of the measurement distributions is approximate and that a limited number of laboratories are involved, we summarize comparability with the following 3-level "Standard Score" (StS)…

| StS | Definition | |
|---|---|---|
| A | All StV within ±*t* (1-0.683,n-1) | (i.e., ±1 SD) |
| B | All StV within ±*t* (1-0.954,n-1) | (i.e., ±2 SD) |
| C | At least one StV > ±*t* (1-0.954,n-1) | (i.e., ≥2 SD) |

where:

| | |
|---|---|
| StV | Standardized Value, the distance in standard deviation units your value is from the "true" concentration: StV = (your value - NAV) / NAU |
| NAV | NIST Assigned Value, our estimate of the "true" analyte concentration |
| NAU | NIST Assigned Uncertainty, our estimate of the total (serum heterogeneity and inter- and intra-laboratory measurement) standard deviation |
| *t* (1-α,n-1) | Two-tailed Student's t for coverage of ±1 and ±2 NAU about NAV assuming a normal population of size n |

| Expected | |
|---|---|
| 68.2 % | These are the observed and normal-population-expected proportions |
| 27.3 % | of each Standard Score (StS), based upon each laboratory's largest |
| 4.3 % | StV for the four sera. |
| 0.3 % | |

## Appendix H. Representative "Individualized Report" for RR43

Each participant in RR43 received an "Individualized Report" reflecting their reported results. Each report included a detailed analysis of the results they reported for the following analytes:

- Total Retinol
- α-Tocopherol
- γ-Tocopherol
- Total β-Carotene
- *trans*-β-Carotene
- Total α-Carotene
- Total Lycopene
- *trans*-Lycopene

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

**Note**

The design file and program used to generate the original RR43 "Individualized Reports" are no longer available. The following pages were produced using the 2013 version of the program (a linear descendant of that used in 1998) and a re-created design file.

# Individualized Round Robin XLIII Report: FSV-BA

## Summary

| | Serum 243 | | | Serum 244 | | | Serum 245 | | | Serum 246 | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Analyte | You | NAV | n | You | NAV | n | You | NAV | n | You | NAV | n |
| Retinol | 0.591 | 0.462 | 50 | 0.484 | 0.370 | 50 | 0.247 | 0.192 | 49 | 0.809 | 0.725 | 50 |
| Retinyl Palmitate | 0.112 | 0.112 | 13 | 0.096 | 0.109 | 13 | 0.045 | 0.052 | 12 | 0.246 | 0.280 | 13 |
| α-Tocopherol | 7.22 | 7.01 | 47 | 7.1 | 7.2 | 48 | 3.4 | 3.5 | 47 | 14.44 | 14.31 | 47 |
| γ-Tocopherol | 2.18 | 2.26 | 23 | 1.31 | 1.27 | 23 | 0.71 | 0.68 | 22 | 2.44 | 2.57 | 22 |
| Total β-Carotene | 0.085 | 0.072 | 32 | 0.337 | 0.359 | 32 | 0.131 | 0.133 | 31 | 0.703 | 0.676 | 31 |
| trans-β-Carotene | 0.079 | 0.070 | 11 | 0.315 | 0.323 | 11 | 0.123 | 0.133 | 11 | 0.669 | 0.636 | 11 |
| Total cis-β-Carotene | 0.006 | 0.004 | 7 | 0.022 | 0.037 | 8 | 0.008 | 0.014 | 8 | 0.034 | 0.054 | 8 |
| Total α-Carotene | 0.013 | 0.014 | 24 | 0.013 | 0.020 | 24 | 0.008 | 0.010 | 20 | 0.041 | 0.039 | 25 |
| trans-Lycopene | 0.312 | 0.263 | 9 | 0.047 | 0.036 | 9 | 0.014 | 0.017 | 9 | 0.080 | 0.071 | 9 |
| Total β-Cryptoxanthin | 0.074 | 0.052 | 27 | 0.029 | 0.011 | 22 | 0.068 | 0.005 | 17 | 0.031 | 0.018 | 24 |
| Total Lutein&Zeaxanthin | 0.178 | 0.121 | 27 | 0.061 | 0.032 | 27 | 0.042 | 0.016 | 25 | 0.096 | 0.075 | 26 |

H2

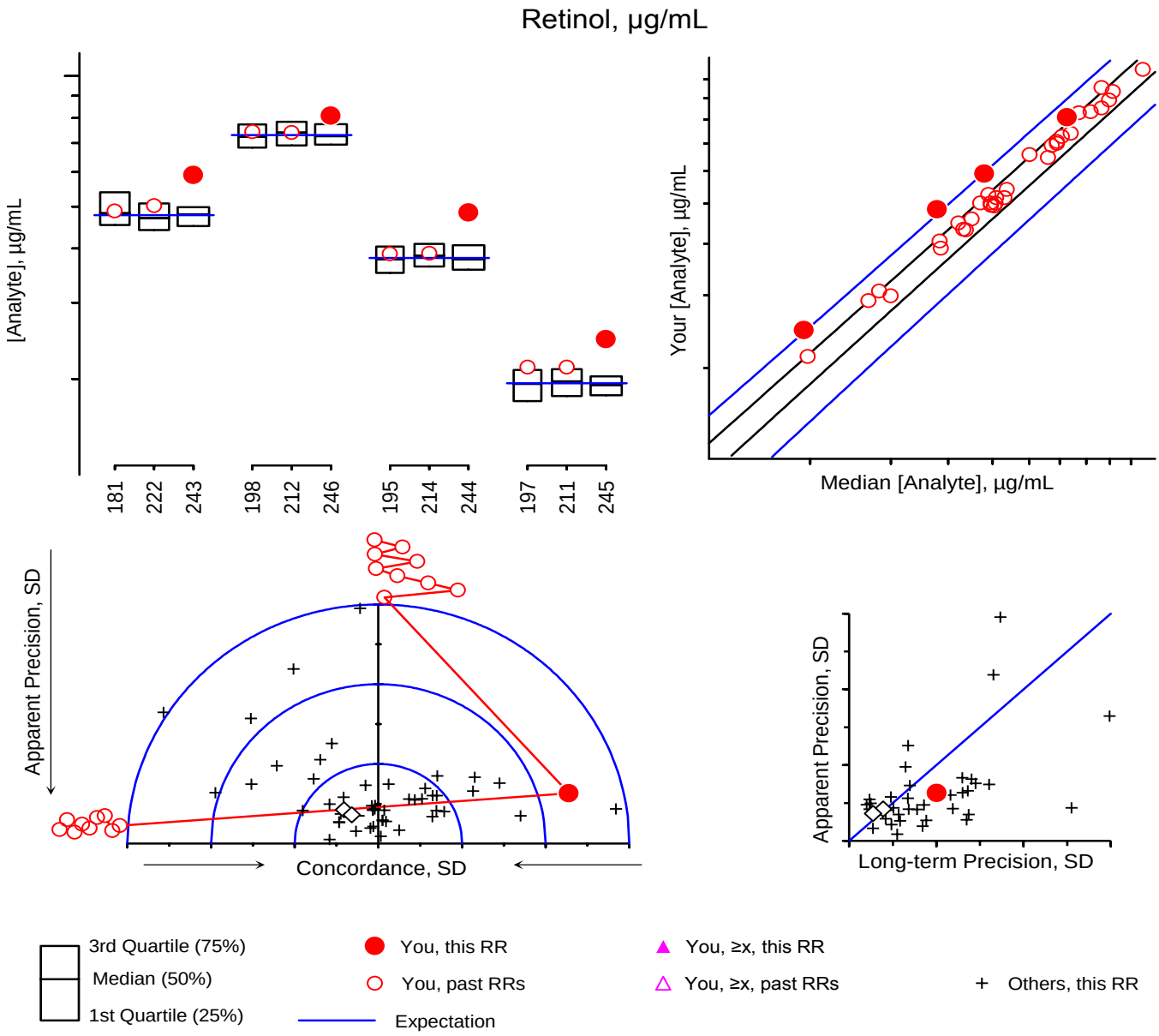
You : Your reported values for the listed analytes (micrograms/milliliter)
NAV : NIST Assigned Values, here equal to this RR's median
n : Number of non-NIST laboratories reporting quantitative values for this analyte in this serum

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

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

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

# Individualized RR XLIII Report: FSV-BA

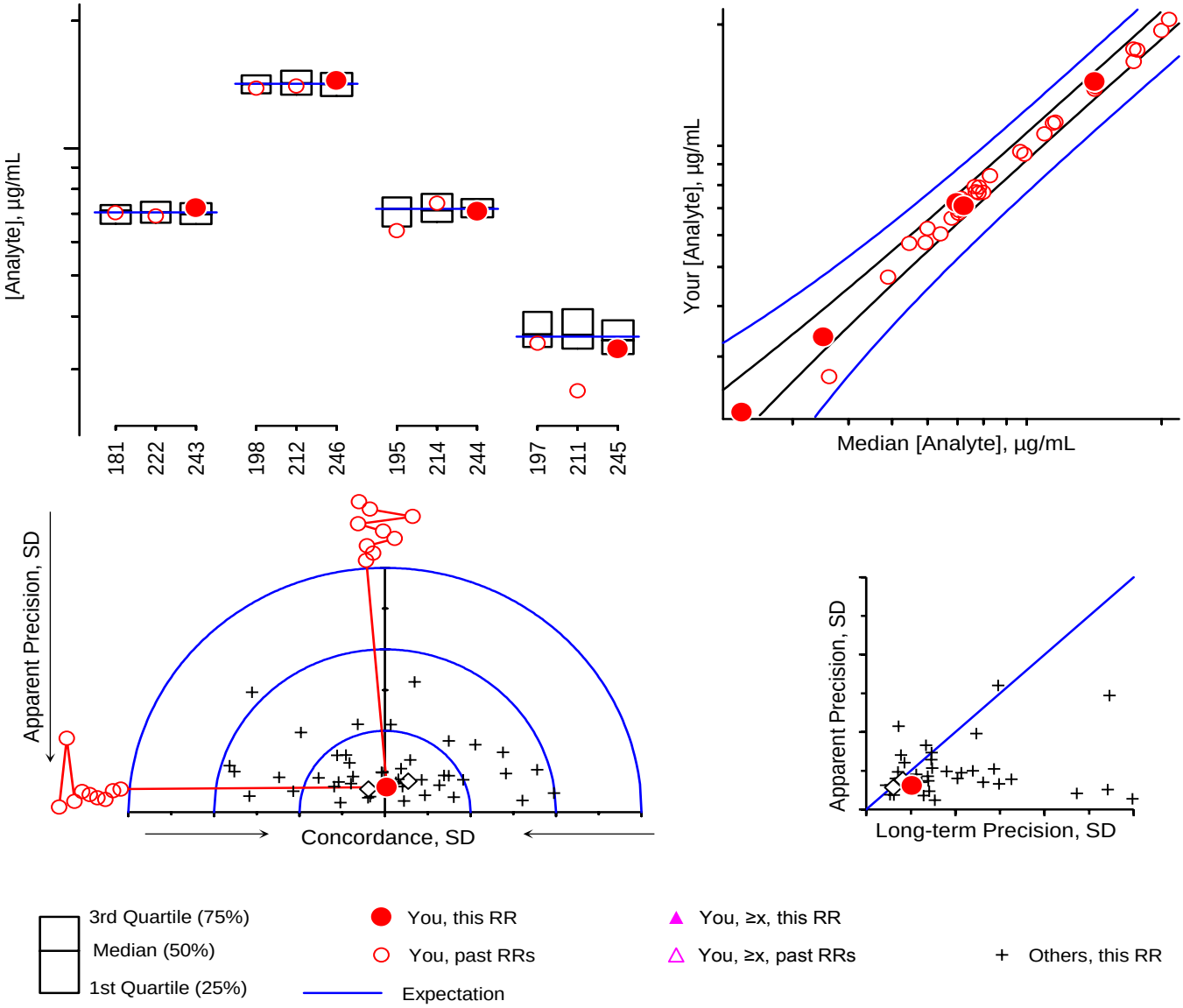



For the construction and interpretation of these plots, see the Final Report for "Round Robin" XL, August 1997

| Serum | Comments | History |
|---|---|---|
| #243 | Lyophilized, single-donor, augmented with retinyl palmitate | RR28:#181, RR37:#222 |
| #246 | Lyophilized, single donor, native | RR31:#198, RR35:#212 |
| #242 | Lyophilized, multi-donor, a 1:1 blend of the#241 pool and delipidized serum. | RR31:#195, RR35:#214 |
| #239 | Lyophilized, multi-donor, a 1:3 blend of the #241 pool and delipidized serum. | RR31:#197, RR35:#211 |

# Individualized RR XLIII Report: FSV-BA

α-Tocopherol, µg/mL

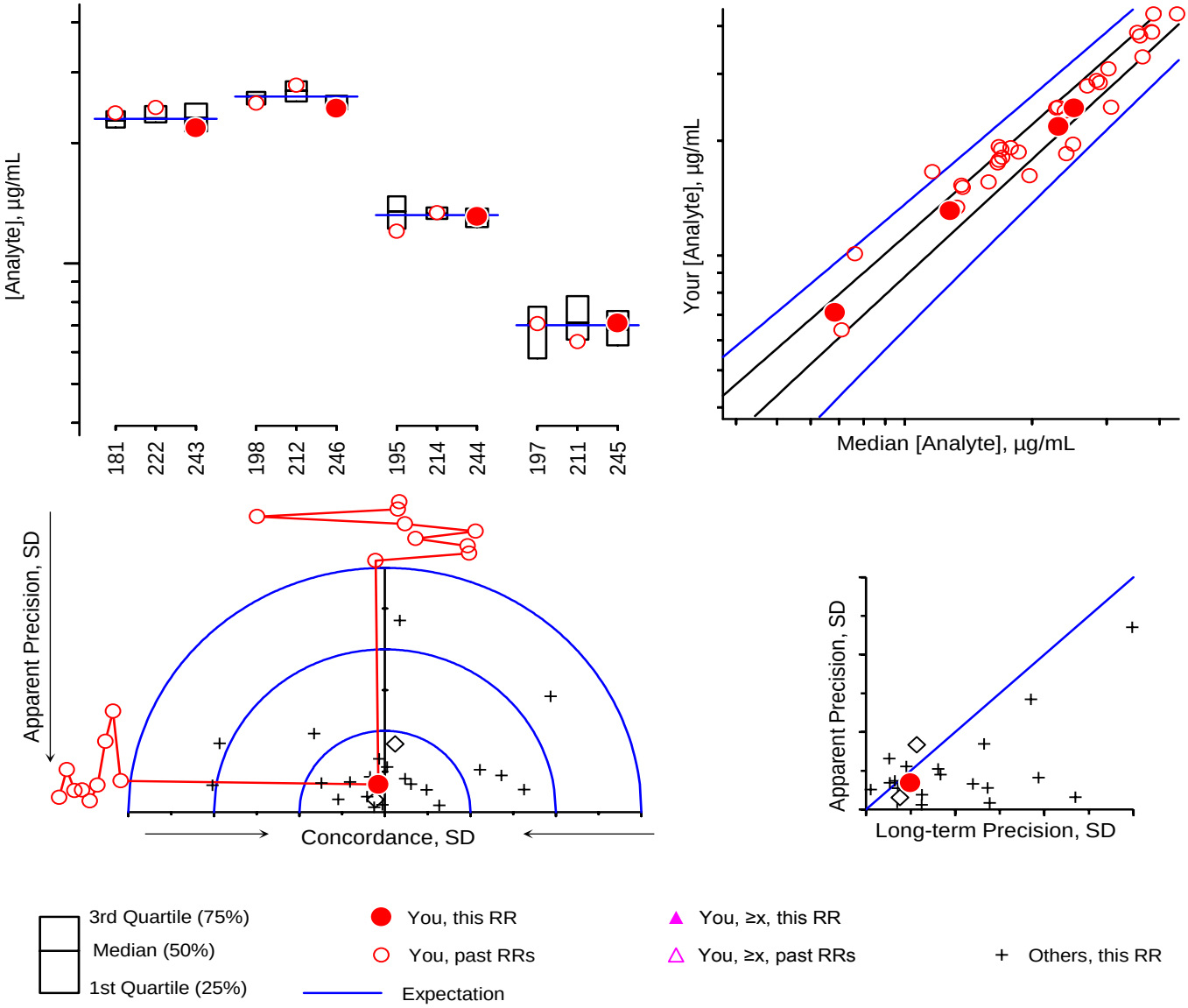



For the construction and interpretation of these plots, see the Final Report for "Round Robin" XL, August 1997

| Serum | Comments | History |
|---|---|---|
| #243 | Lyophilized, single-donor, augmented with retinyl palmitate | RR28:#181, RR37:#222 |
| #246 | Lyophilized, single donor, native | RR31:#198, RR35:#212 |
| #242 | Lyophilized, multi-donor, a 1:1 blend of the#241 pool and delipidized serum. | RR31:#195, RR35:#214 |
| #239 | Lyophilized, multi-donor, a 1:3 blend of the #241 pool and delipidized serum. | RR31:#197, RR35:#211 |

# Individualized RR XLIII Report: FSV-BA

γ-Tocopherol, µg/mL

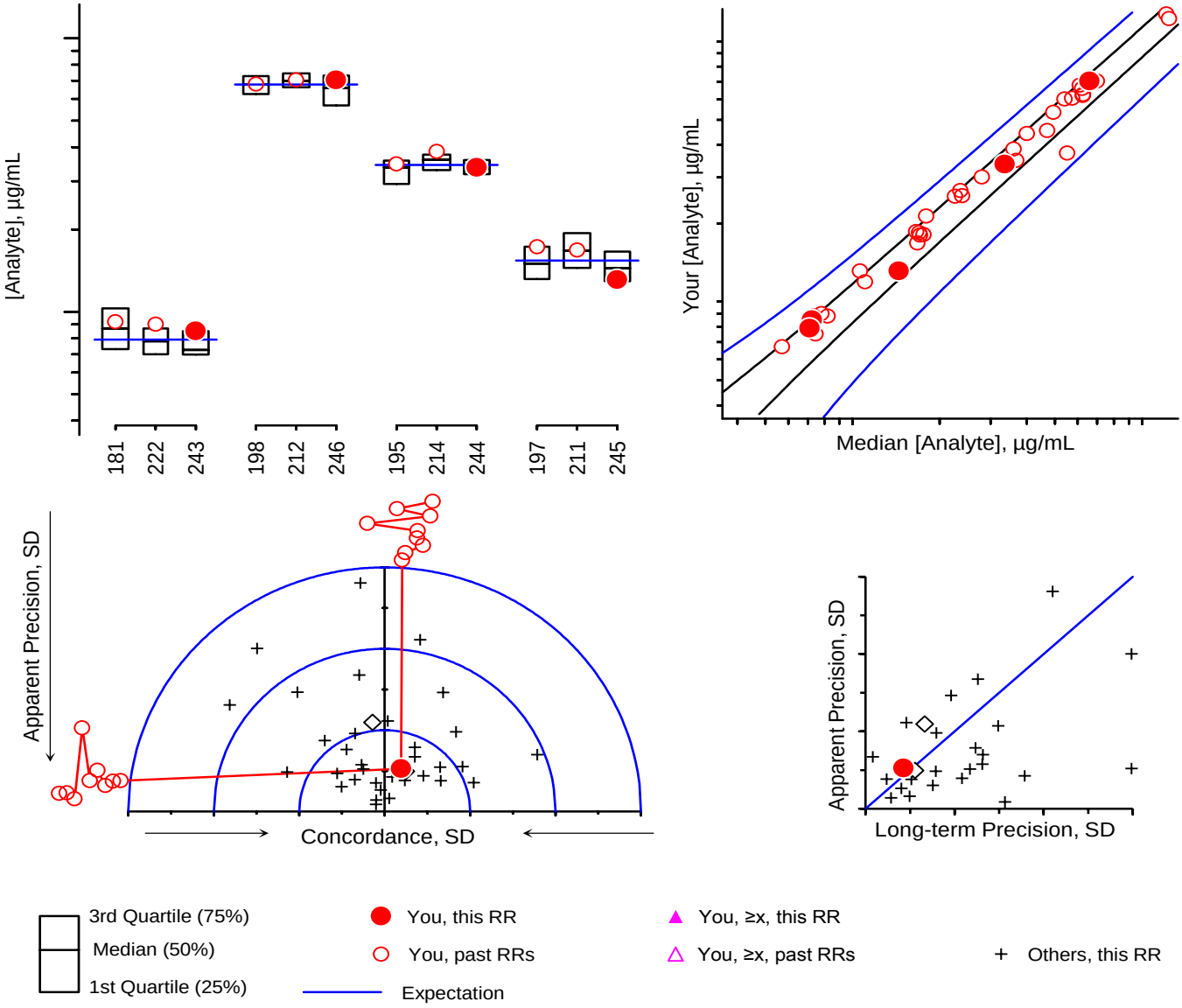



For the construction and interpretation of these plots, see the Final Report for "Round Robin" XL, August 1997

| Serum | Comments | History |
|---|---|---|
| #243 | Lyophilized, single-donor, augmented with retinyl palmitate | RR28:#181, RR37:#222 |
| #246 | Lyophilized, single donor, native | RR31:#198, RR35:#212 |
| #242 | Lyophilized, multi-donor, a 1:1 blend of the#241 pool and delipidized serum. | RR31:#195, RR35:#214 |
| #239 | Lyophilized, multi-donor, a 1:3 blend of the #241 pool and delipidized serum. | RR31:#197, RR35:#211 |

# Individualized RR XLIII Report: FSV-BA

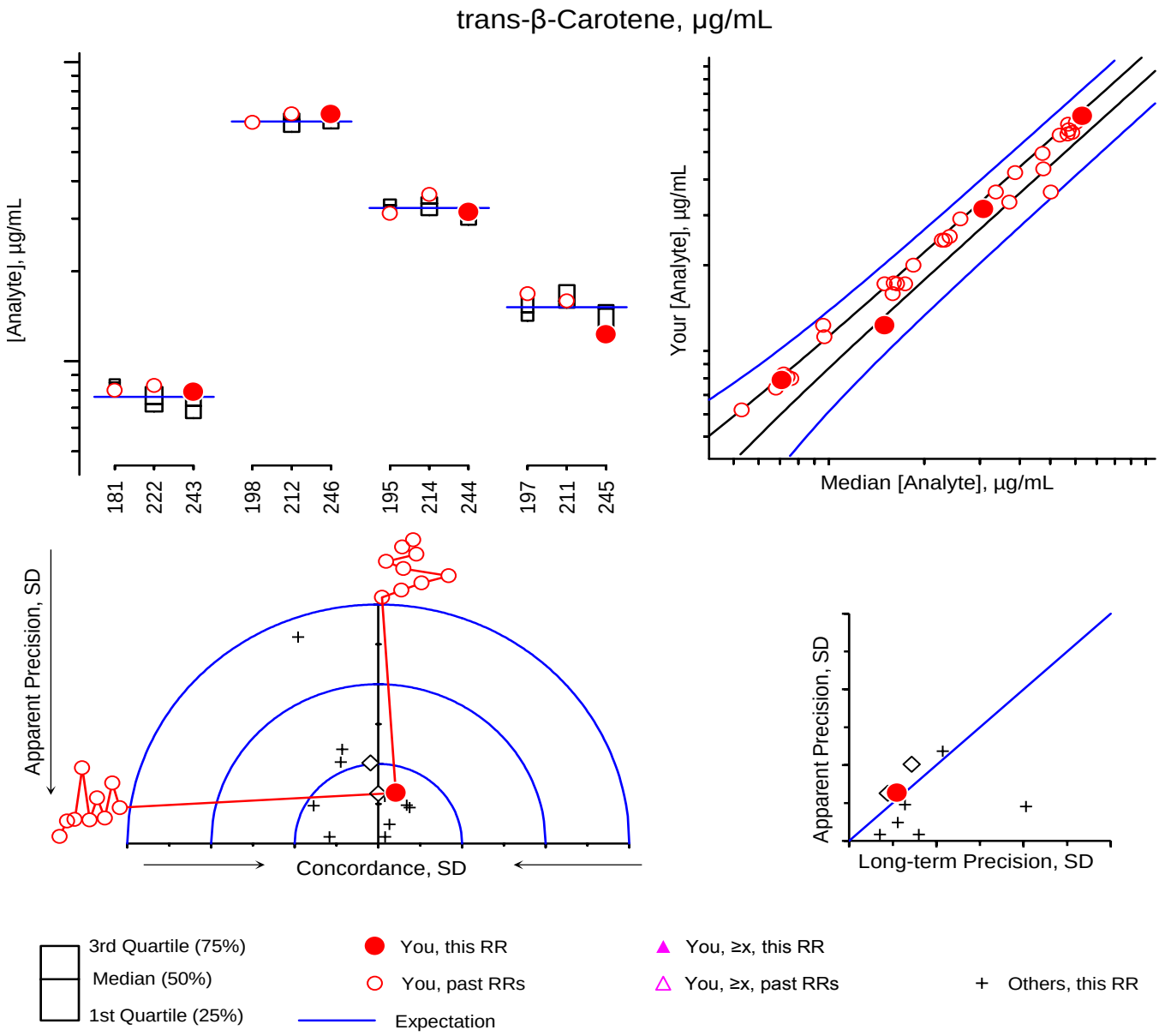
## Total β-Carotene, µg/mL




For the construction and interpretation of these plots, see the Final Report for "Round Robin" XL, August 1997

| Serum | Comments | History |
|---|---|---|
| #243 | Lyophilized, single-donor, augmented with retinyl palmitate | RR28:#181, RR37:#222 |
| #246 | Lyophilized, single donor, native | RR31:#198, RR35:#212 |
| #242 | Lyophilized, multi-donor, a 1:1 blend of the#241 pool and delipidized serum. | RR31:#195, RR35:#214 |
| #239 | Lyophilized, multi-donor, a 1:3 blend of the #241 pool and delipidized serum. | RR31:#197, RR35:#211 |

# Individualized RR XLIII Report: FSV-BA

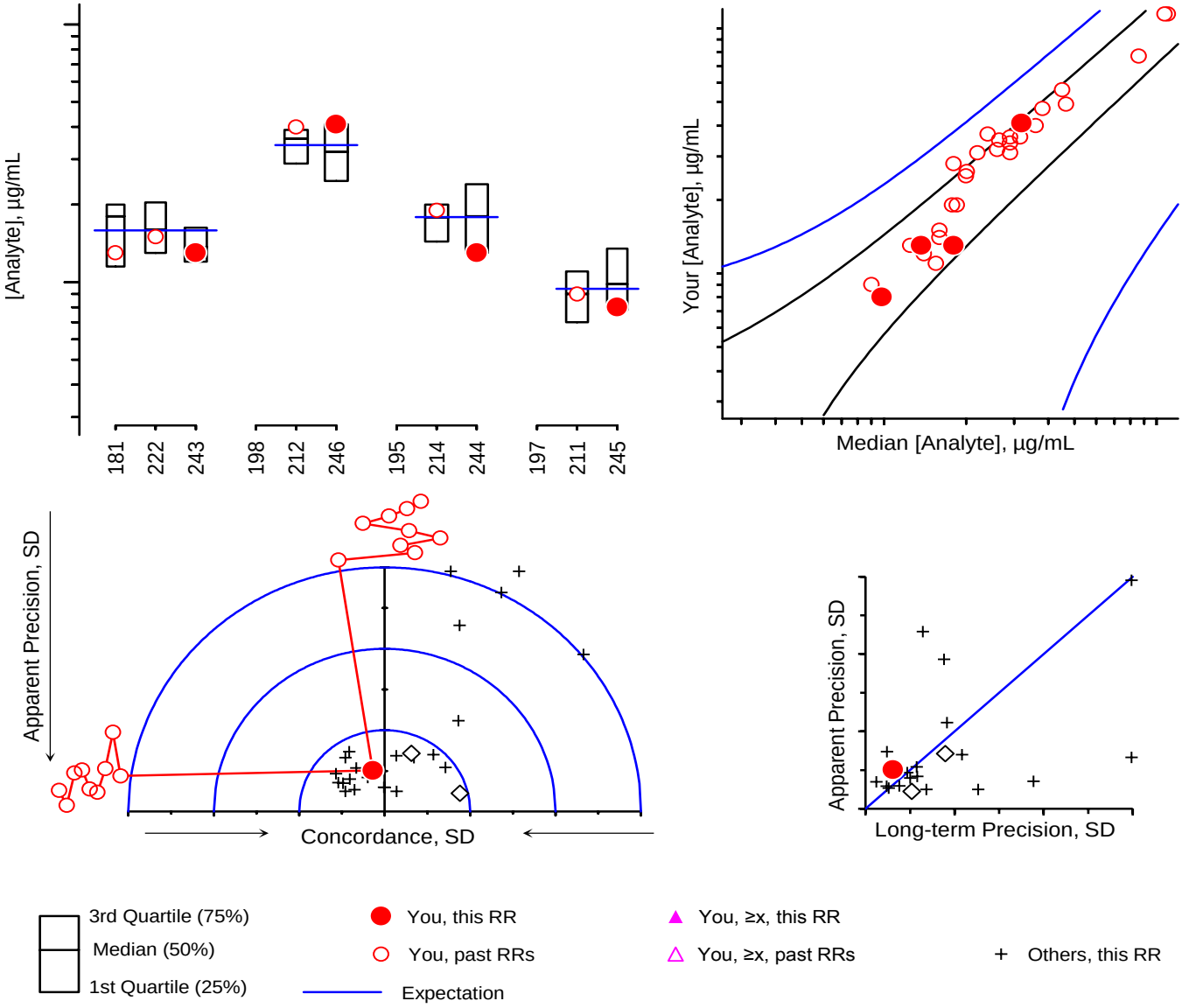



For the construction and interpretation of these plots, see the Final Report for "Round Robin" XL, August 1997

| Serum | Comments | History |
|---|---|---|
| #243 | Lyophilized, single-donor, augmented with retinyl palmitate | RR28:#181, RR37:#222 |
| #246 | Lyophilized, single donor, native | RR31:#198, RR35:#212 |
| #242 | Lyophilized, multi-donor, a 1:1 blend of the#241 pool and delipidized serum. | RR31:#195, RR35:#214 |
| #239 | Lyophilized, multi-donor, a 1:3 blend of the #241 pool and delipidized serum. | RR31:#197, RR35:#211 |

# Individualized RR XLIII Report: FSV-BA

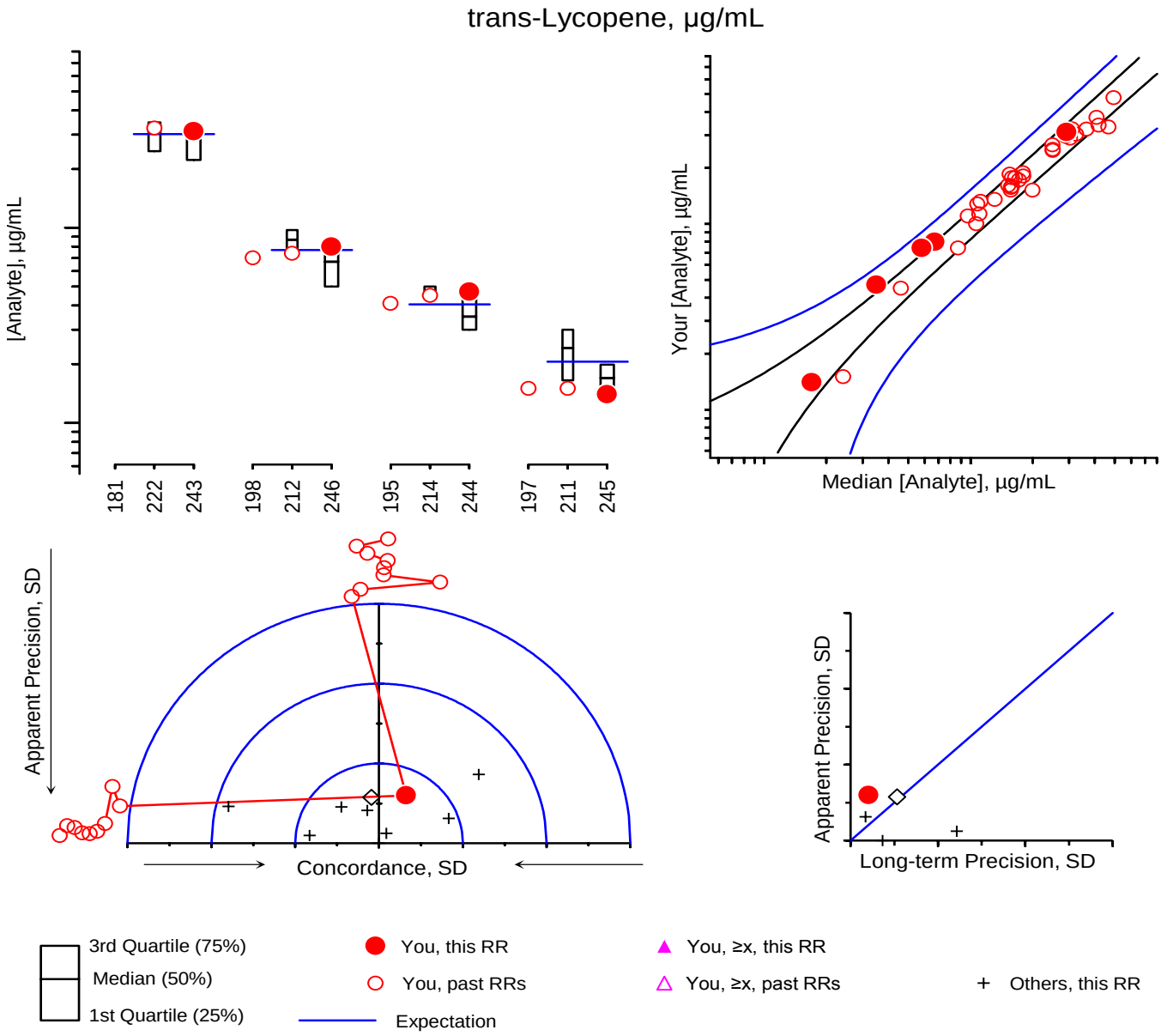
## Total α-Carotene, µg/mL




For the construction and interpretation of these plots, see the Final Report for "Round Robin" XL, August 1997

| Serum | Comments | History |
|---|---|---|
| #243 | Lyophilized, single-donor, augmented with retinyl palmitate | RR28:#181, RR37:#222 |
| #246 | Lyophilized, single donor, native | RR31:#198, RR35:#212 |
| #242 | Lyophilized, multi-donor, a 1:1 blend of the#241 pool and delipidized serum. | RR31:#195, RR35:#214 |
| #239 | Lyophilized, multi-donor, a 1:3 blend of the #241 pool and delipidized serum. | RR31:#197, RR35:#211 |

# Individualized RR XLIII Report: FSV-BA




For the construction and interpretation of these plots, see the Final Report for "Round Robin" XL, August 1997

| Serum | Comments | History |
|---|---|---|
| #243 | Lyophilized, single-donor, augmented with retinyl palmitate | RR28:#181, RR37:#222 |
| #246 | Lyophilized, single donor, native | RR31:#198, RR35:#212 |
| #242 | Lyophilized, multi-donor, a 1:1 blend of the#241 pool and delipidized serum. | RR31:#195, RR35:#214 |
| #239 | Lyophilized, multi-donor, a 1:3 blend of the #241 pool and delipidized serum. | RR31:#197, RR35:#211 |

## Appendix I. Shipping Package Inserts for RR44

The following two items were included in each package shipped to an RR44 participant:

- Cover letter
- Datasheet

The cover letter and datasheet were enclosed in a sealed waterproof bag along with the samples themselves.

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

October 30, 1998 Note: Actual date was sometime in August, 1998 -

Dear Colleague:

Enclosed is the set of samples for the third quality assurance round robin exercise (Round Robin XLIV) for FY98. You will find one vial of each of four lyophilized serum samples for analysis along with a form for reporting your results. When reporting your results, please submit one value for each analyte for a given serum sample. If an obtained value is below your limit of quantitation, please indicate this result on the form by using NQ *(Not Quantitated)*. For analytes not measured, please leave a blank. Results are due to NIST by October 9, 1998. Results received two weeks after the due date will not be included in the summary report for this round robin study. Written feedback concerning the study will be provided around November 13.

Samples should be reconstituted with 1.0 mL of HPLC-grade water or equivalent. We recommend that dissolution be facilitated with 3 to 5 min agitation in an ultrasonic bath or at least 30 min at room temperature with intermittent swirling. (CAUTION: Vigorous shaking will cause foaming and possibly interfere with accurate measurement. The rubber stopper contains phthalate esters that will leach into the sample upon intermittent contact of the liquid sample with the stopper. These esters absorb strongly in the UV region and elute very 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.). For consistency, we request that laboratories use the following absorptivities (E 1% cm) in ethanol: retinol, 1843 at 325 nm; retinyl palmitate, 975 at 325 nm; α-tocopherol, 75.8 at 292 nm; γ-tocopherol, 91.4 at 298 nm; α-carotene, 2800 at 444 nm (in hexane); β-carotene, 2560 at 450 nm (in ethanol), 2592 at 452 nm (in hexane); lycopene, 3450 at 472 nm (in hexane).

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

Micronutrients Measurement Quality Assurance Program
NIST
Bldg. 222, Rm. B208
Gaithersburg, MD 20899
Fax: (301) 977-0685

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

Sincerely,

Jeanice Brown Thomas

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

Enclosures

cc: S. Wise



NIST

*NIST*

# *Micronutrients Measurement Quality Assurance Program*

## Round Robin XLIV Results from Laboratory #_____

| | Serum | | | | |
|---|---|---|---|---|---|
| Analyte | 247 | 248 | 249 | 250 | Units* |
| retinol | | | | | |
| retinyl palmitate | | | | | |
| α-tocopherol | | | | | |
| γ-tocopherol | | | | | |
| δ-tocopherol | | | | | |
| total β-carotene | | | | | |
| trans-β-carotene | | | | | |
| total cis-β-carotene | | | | | |
| total α-carotene | | | | | |
| trans-α-carotene | | | | | |
| total lycopene | | | | | |
| trans-lycopene | | | | | |
| β-cryptoxanthin | | | | | |
| α-cryptoxanthin | | | | | |
| "lutein" | | | | | |
| "zeaxanthin" | | | | | |
| "lutein&zeaxanthin" | | | | | |
| Other Analytes? | | | | | |
| | | | | | |
| | | | | | |

* We prefer mg/mL

Today's Date:

Comments?

Mail: MMQAP, 222/B208
NIST
Gaithersburg, MD 20899

Please return results on-or-before
9-Oct-98

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

## Appendix J. Final Report for RR44

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

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

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

November 23, 1998

Dear Colleague:

Enclosed is the summary report of the results for Round Robin XLIV (RR44). Included in this report are: a summary of data for all laboratories; the measurement comparability summary for evaluating laboratory performance; a summary of individual laboratory performance and interlaboratory accuracy and precision; and a summary of the NIST assigned value (NAV) vs. your laboratory value for the analytes measured. As in previous reports, the NIST assigned values are derived from the equally weighted results from the analyses performed by NIST and the laboratories that participated in this interlaboratory comparison exercise.

In this round exercise, serum samples (Sera 248-249) were new distributions. Sera 248 and 249 are new serum pools to be used for candidate Standard Reference Material (SRM) 968c, Fat-Soluble Vitamins, Cholesterol, and Carotenoids in Human Serum. Serum 247 is a blend (1:1) of sera 248 and 249. Serum 250 was distributed in three previous round robins (round robins 30, 32, and 36 as Sera 192, 199, and 218, respectively). Per your request, a detailed (but brief) summary for this interlaboratory comparison exercise is provided in the attached report.

Data for evaluating laboratory performance in RR44 are provided in the comparability summary (Score Card) on page 6 of the "All Lab Report." The criteria used to evaluate laboratory performance are as follows: results rated 1 (within 1 SD of the assigned value) indicate EXCEPTIONAL performance, results rated 2 (within 2 SD) indicate ACCEPTABLE performance, results rated 3 (within 3 SD of the assigned value) are MARGINAL, and those rated 4 (>3 SD from the assigned value) indicate POOR performance relative to the current state-of-the-practice for these measurements.

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

Thank you for your responses regarding the continuance of the Micronutrients Measurement Quality Assurance (QA) Program. Due to the positive feedback, the QA program will continue through FY 99. The Intent-to-Participate form, which will provide us with formal notification of your intent to participate in the program for the upcoming year, has been mailed. Please contact us if you have not received your form.

As mentioned in your participation letter, the program will consist of two round robin studies for the fat-soluble vitamins and carotenoids and one study for vitamin C. The participation fee for the fat-soluble vitamins and carotenoids will be $665 for U.S. laboratories and $1020 for laboratories outside of the U.S. To participate in the vitamin C study, there will be a fee of $370 for U.S. laboratories and $530 for laboratories outside of the U.S.

Over the years, we have acquired a limited supply of well-characterized serum pools that were used in previous intercomparison exercises for the QA program. These materials can be used by laboratories as in-house control materials and will be made available to you at a nominal cost ($50 per vial). We will provide you with more details in a separate cover in January.



NIST

The next QA workshop will be held as a pre-meeting on Friday, April 16 at Experimental Biology '99 in Washington, DC. Details regarding the workshop, housing information, and registration forms will be mailed to you at the beginning of next year. If you would like details regarding the conference, please contact the secretariat for the American Institute of Nutrition at (301)530-7050; fax: (301)571-1892; e-mail: sec@asns.faseb.org.

Enclosed please find a reprint on the stability of vitamins in human serum and a draft of a manuscript, which has been submitted to Analytical Chemistry, on the use of interlaboratory comparison exercise results. If you have any questions regarding this report, please contact me at 301/975-3120; FAX: 301/977-0685; e-mail: jeanice.brownthomas@nist.gov.

Sincerely,

Jeanice Brown Thomas

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

cc: S. Wise

Enclosures

The NIST M²QAP Round Robin XLIV (RR44) report includes:

| Page | "All Lab" Report |
|---|---|
| 1-4 | A listing of all results and statistics for analytes reported by at least two laboratories |
| 5a | A list of results for five analytes reported by only one laboratory |
| 5b | A legend for the above two lists |
| 6 | A "Comparability Summary" or "Score Card" for retinol, α-tocopherol, γ-tocopherol, total and *trans*-β-carotene, total α-carotene, β-cryptoxanthin, and total lycopene. |

| Page | "Individualized" Report |
|---|---|
| 1 | A summary of your data and the NIST assigned values |
| 2, 3 | Summary box plots for retinol, α-tocopherol, γ-tocopherol, total and *trans*-β-carotene, total α-carotene, β-cryptoxanthin, total and *trans*-lycopene, lutein, and zeaxanthin |
| 4, 5 | Concordance/apparent precision plots for retinol, α-tocopherol, γ-tocopherol, total and *trans*-β-carotene, total α-carotene, β-cryptoxanthin, total and *trans*-lycopene, lutein, and zeaxanthin |
| 6 | Youden comparability plots for retinol, α-tocopherol,_γ-tocopherol, and total and *trans*-β-carotene |

All elements of this report should be familiar to you, although the names of the various elements have changed somewhat from previous versions. The enclosed preprint "M²QAP: Helping Participants Use Interlaboratory Comparison Exercise Results to Improve Their Long-term Measurement Performance" details the nomenclature, construction, and interpretation of the three plot types.

Please note that we no longer report "percent bias" (100[you-NAV]/NAV) but rather use z-scores ([you-NAV]/NAU) throughout, where NAV and NAU are our assignments of analyte value and total measurement uncertainty.

**Samples**. Three new and one "old" sera were distributed in RR44.

Sera **248** and **249** are new: they are the two components of candidate SRM® 968c. These two materials were prepared from well-characterized serum pools. All carotenoid levels are "native" but the sera were augmented with retinol, α-tocopherol, γ-tocopherol, and/or δ-tocopherol.

Serum **247** is new: it is a 1:1 blend of the 248 and 249 serum pools, with an experimental liposomic augmentation of *trans*-β-carotene and β-cryptoxanthin. Since this material was prepared from the "tag-ends" of the pools after all 248 and 249 production was complete, it provides an independent check for analyte degradation in the 248 and 249 pools.

Serum **250** was previously distributed as 192 in RR30, 199 in RR32, and 218 in RR36. This material was augmented with retinol and retinyl palmitate, but is natively high in most carotenoids.

**Qualitative Results**. One participant noted an integration problem with their retinol peak due to a small leading peak. In retrospect, we often see some early-elution "junk" in many of our lyophilized sera; differential resolution/integration of these interferents (esters?) may contribute to the remaining interlaboratory retinol differences. Add this to the list of research topics!

**Quantitative Results**. To preserve resources and sanity, we no longer report our detailed dissection of the NIST measurements. We do include the most relevant statistics of this analysis in the "All Laboratory" report. The following summary conclusions draw from both the NIST and RR44 analyses.

Sera **248** and **249**: these materials are more-than-adequately homogenous, reconstitute well, and contain no "unusual" interferents. The measured levels of all analytes are adequately close to the design levels. Systematic zeaxanthin differences between the two sets of NIST analyses suggest that our assays are measuring different sets of carotenoid components for this analyte. Differences in lycopene levels between NIST3 and the RR44 expected values suggest that our *trans*-lycopene may include some *cis*-lycopene component(s). The C30 column measurements should help resolve these issues.

Serum **247**: the measured levels of all analytes are very close to the expected (248 + 249)/2. This confirms that the composition of the serum pools used for sera 248 and 249 did not change during the production process. The experimental β-carotene and β-cryptoxanthin augmentation either did not "take" and/or hexane does not appreciably extract analyte from the liposomes.

Serum **250**: The composition of this material is apparently unchanged by its five years' storage at –80 °C, using both the formal statistical tests of level and variance and "Mark I eyeball" of the summary box plots. Almost all participants who reported discordant values for sera 247-249 reported a similarly discordant value for serum 250 (see the "target" plots in the Individualized Report). Since there is no compositional relationship between serum 250 and sera 247-249, these systematic differences are unlikely to be sample-related.

**Analyte nomenclature: total, *trans*-, and total *cis*-.** There is considerable variation in the reportage of "speciated" carotenoids, particularly when *cis*-isomers are detected but not quantitatively determinable. This is (currently) mostly a problem with β-carotene. We propose the following rules:

If your system does **not** routinely resolve the isomers, don't worry about it and just report "Total".

If your system **does** routinely resolve the isomers but one or more cannot be quantitatively measured, report just these isomers as "not detected (nd)" or "not quantified (nq)". Report any composite analyte as ">" (or "≥") the appropriate partial value. That is: a "total" normally calculated from *trans* + *cis* peak areas (whether or not separately integrated) should be reported as ">"*trans* if the *cis* is "nq". Reporting "nq" for the total in this situation is confusing if you don't typically report explicit *cis* values!

If you report any two of the "total = *trans* + *cis*" trio, we will calculate the third. These calculated values (like "lutein & zeaxanthin" from "lutein" + "zeaxanthin") are listed in italics in the "All Laboratory Report".

Submitted by:

David L. Duewer

## Appendix K. "All-Lab Report" for RR44

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

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

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

# Round Robin XLIV Laboratory Results

All Results in µg/mL

| | Retinol | | | | Retinyl Palmitate | | | | α-Tocopherol | | | | γ-Tocopherol | | | | δ-Tocopherol | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Lab | 247 | 248 | 249 | 250 | 247 | 248 | 249 | 250 | 247 | 248 | 249 | 250 | 247 | 248 | 249 | 250 | 247 | 248 | 249 | 250 |
| FSV-BA | 0.664 | 0.849 | 0.488 | 1.120 | 0.081 | 0.061 | 0.107 | 0.075 | 10.9 | 7.32 | 15.5 | 6.92 | 2.42 | 3.65 | 1.48 | 2.34 | | | | |
| FSV-BB | 0.711 | 0.886 | 0.519 | 1.130 | 0.052 | 0.022 | 0.077 | 0.040 | 10.9 | 6.90 | 15.7 | 6.55 | 3.09 | 4.48 | 1.86 | 2.60 | | | | |
| FSV-BBa | | | | | | | | | 12.6 | 7.95 | 17.6 | 7.11 | 2.59 | 3.96 | 1.49 | 2.28 | | | | |
| FSV-BD | 0.650 | 0.854 | 0.482 | 1.075 | | | | | 10.9 | 8.10 | 15.8 | 7.20 | | | | | | | | |
| FSV-BE | 0.660 | 0.860 | 0.480 | 1.060 | | | | | 10.3 | 6.80 | 14.7 | 6.30 | 2.70 | 4.00 | 1.60 | 2.50 | | | | |
| FSV-BF | 0.743 | 0.871 | 0.499 | 1.017 | | | | | 12.7 | 7.73 | 17.6 | 7.14 | 2.74 | 3.80 | 1.61 | 2.26 | | | | |
| FSV-BG | 0.658 | 0.855 | 0.473 | 1.122 | 0.051 | 0.029 | 0.070 | 0.060 | 12.2 | 7.99 | 17.6 | 7.33 | 2.58 | 3.81 | 1.56 | 2.36 | | | | |
| FSV-BH | 0.651 | 0.790 | 0.474 | 1.004 | 0.039 | 0.030 | 0.095 | *nq* | 11.6 | 7.37 | 17.0 | 6.97 | 2.67 | 3.90 | 1.61 | 2.35 | | | | |
| FSV-BI | 0.694 | 0.823 | 0.504 | 1.108 | 0.069 | 0.046 | 0.109 | *nd* | 11.0 | 6.96 | 16.1 | 6.98 | 2.40 | 3.56 | 1.54 | 2.20 | | | | |
| FSV-BJ | 0.742 | 0.920 | 0.518 | 1.168 | 0.045 | *nq* | 0.076 | *nq* | 12.6 | 8.22 | 17.3 | 7.61 | 2.95 | 4.13 | 1.63 | 2.54 | | | | |
| FSV-BK | 0.631 | 0.832 | 0.473 | 1.043 | | | | | 11.6 | 7.59 | 16.6 | 7.01 | | | | | | | | |
| FSV-BL | 0.340 | 0.460 | 0.260 | 0.600 | | | | | 7.3 | 5.17 | 10.3 | 4.74 | | | | | | | | |
| FSV-BM | 0.705 | 0.877 | 0.509 | 1.053 | | | | | 11.0 | 7.10 | 15.7 | 7.00 | | | | | | | | |
| FSV-BN | 0.636 | 0.851 | 0.499 | 1.061 | 0.041 | 0.028 | 0.077 | 0.023 | 11.9 | 7.44 | 18.1 | 7.17 | 2.33 | 3.41 | 1.49 | 2.01 | 0.389 | 0.164 | 0.581 | 0.163 |
| FSV-BO | 0.739 | 0.899 | 0.544 | 1.255 | | | | | 15.0 | 10.85 | 21.1 | 8.74 | | | | | | | | |
| FSV-BP | 0.600 | 0.952 | 0.546 | 1.214 | | | | | 11.8 | 7.99 | 17.8 | 7.67 | | | | | | | | |
| FSV-BQ | 0.748 | 0.917 | 0.544 | 1.163 | | | | | 10.7 | 6.65 | 15.2 | 6.29 | | | | | | | | |
| FSV-BR | 0.720 | 0.920 | 0.520 | 1.130 | | | | | | | | | | | | | | | | |
| FSV-BS | 0.672 | 0.944 | 0.566 | 1.098 | | | | | | | | | | | | | | | | |
| FSV-BT | 0.681 | 0.797 | 0.420 | 1.003 | 0.047 | 0.030 | 0.101 | 0.024 | 8.9 | 5.62 | 14.2 | 6.42 | 2.85 | 3.78 | 1.96 | 2.51 | 1.553 | 1.262 | 1.752 | 0.604 |
| FSV-BU | 0.642 | 0.882 | 0.494 | 1.092 | | | | | 10.6 | 7.04 | 15.6 | 6.84 | 2.73 | 3.30 | 1.43 | 2.03 | | | | |
| FSV-BV | 0.799 | 1.090 | 0.563 | 1.270 | | | | | 11.4 | 7.58 | 17.0 | 6.92 | 2.88 | 4.54 | 1.72 | 2.60 | | | | |
| FSV-BW | 0.700 | 0.903 | 0.490 | 1.008 | 0.060 | 0.033 | 0.103 | *nq* | 10.1 | 6.42 | 14.7 | 6.10 | 2.82 | 4.20 | 1.70 | 2.55 | | | | |
| FSV-BX | 0.649 | 0.838 | 0.457 | 1.062 | | | | | 11.8 | 7.70 | 16.4 | 6.91 | 2.65 | 3.87 | 1.63 | 2.36 | | | | |
| FSV-BZ | | | | | | | | | 12.1 | 4.90 | 14.3 | 5.50 | 3.60 | 3.30 | 1.90 | 2.30 | | | | |
| FSV-CB | 0.715 | 0.887 | 0.517 | 1.134 | | | | | 11.4 | 6.64 | 15.4 | 6.53 | | | | | | | | |
| FSV-CC | 0.609 | 0.816 | 0.452 | 0.969 | | | | | 12.1 | 7.62 | 16.9 | 7.42 | | | | | | | | |
| FSV-CD | 0.724 | 0.823 | 0.475 | 0.971 | 0.122 | 0.084 | 0.151 | 0.119 | 13.2 | 7.88 | 17.5 | 7.43 | 2.77 | 3.80 | 1.47 | 2.30 | | | | |
| FSV-CE | 0.591 | 0.712 | 0.473 | 1.030 | | | | | 12.0 | 7.35 | 18.3 | 7.57 | | | | | | | | |
| FSV-CF | 0.715 | 0.854 | 0.503 | 1.128 | | | | | 12.9 | 7.90 | 18.0 | 7.40 | | | | | | | | |
| FSV-CH | 0.616 | 0.849 | 0.450 | 1.020 | | | | | 10.4 | 7.68 | 16.7 | 6.89 | 2.18 | 3.43 | 1.38 | 1.86 | | | | |
| FSV-CI | 0.750 | 0.970 | 0.550 | 1.210 | 0.040 | 0.030 | 0.090 | 0.040 | 10.2 | 6.10 | 15.4 | 5.90 | | | | | | | | |
| FSV-CL | 0.625 | 0.843 | 0.434 | 0.998 | | | | | 16.0 | 12.16 | 21.2 | 10.69 | 3.02 | 4.80 | 1.60 | 2.72 | | | | |
| FSV-CM | | | | | | | | | 10.1 | 6.20 | 16.9 | 7.33 | | | | | | | | |
| FSV-CP | | | | | | | | | 11.8 | 7.97 | 16.7 | 7.38 | 2.78 | 4.08 | 1.67 | 2.53 | | | | |
| FSV-CR | 0.740 | 0.960 | 0.510 | 1.170 | | | | | 12.5 | 8.10 | 17.7 | 7.30 | | | | | 0.400 | <0.3 | 0.700 | <0.3 |
| FSV-CS | 0.636 | 0.823 | 0.448 | 1.073 | | | | | 11.5 | 6.70 | 15.2 | 6.11 | 3.06 | 4.17 | 1.65 | 2.46 | | | | |
| FSV-CU | 0.559 | 0.728 | 0.406 | 0.949 | 0.073 | 0.057 | 0.083 | 0.065 | 9.7 | 6.65 | 13.3 | 6.76 | | | | | | | | |
| FSV-CX | 0.700 | 0.830 | 0.480 | 1.140 | 0.010 | 0.010 | 0.050 | 0.060 | 11.6 | 7.12 | 16.5 | 7.32 | | | | | | | | |
| FSV-DA | 0.638 | 0.807 | 0.460 | 0.968 | *nd* | *nd* | 0.050 | *nd* | 11.4 | 7.20 | 16.3 | 6.75 | 2.76 | 3.97 | 1.66 | 2.32 | 0.403 | 0.183 | 0.658 | 0.176 |
| FSV-DB | 0.646 | 0.763 | 0.443 | 1.015 | | | | | 11.2 | 6.93 | 16.1 | 6.74 | | | | | | | | |
| FSV-DF | 0.680 | 0.850 | 0.470 | 1.130 | | | | | | | | | | | | | | | | |
| FSV-DJ | 0.660 | 0.860 | 0.480 | 1.020 | | | | | | | | | | | | | | | | |
| FSV-DK | 0.550 | 0.690 | 0.410 | 0.840 | 0.035 | 0.025 | 0.052 | 0.020 | 12.4 | 9.00 | 16.8 | 9.13 | 5.89 | 7.36 | 4.16 | 5.57 | | | | |
| FSV-DP | 0.681 | 0.871 | 0.478 | 1.122 | | | | | | | | | | | | | | | | |
| FSV-DR | 0.640 | 0.840 | 0.630 | 1.700 | | | | | 11.5 | 7.65 | 25.1 | 11.56 | | | | | | | | |
| FSV-DU | 0.710 | 0.900 | 0.520 | 1.090 | | | | | 13.2 | 8.02 | 18.1 | 7.80 | | | | | | | | |
| FSV-DW | 0.706 | 0.800 | 0.434 | 1.137 | | | | | 12.6 | 7.81 | 17.1 | 8.05 | | | | | | | | |
| FSV-ES | 0.656 | 0.859 | 0.504 | 1.117 | | | | | 12.9 | 8.15 | 18.8 | 8.06 | | | | | | | | |
| FSV-FG | 0.660 | 0.840 | 0.450 | 1.000 | | | | | 11.4 | 6.65 | 16.6 | 6.60 | | | | | | | | |
| FSV-FH | | | | | | | | | 11.4 | 6.80 | 16.4 | 6.90 | | | | | | | | |
| N | 46 | 46 | 46 | 46 | 14 | 13 | 15 | 10 | 46 | 46 | 46 | 46 | 23 | 23 | 23 | 23 | 4 | 3 | 4 | 3 |
| Min | 0.340 | 0.460 | 0.260 | 0.600 | 0.010 | 0.010 | 0.050 | 0.020 | 7.3 | 4.90 | 10.3 | 4.74 | 2.18 | 3.30 | 1.38 | 1.86 | 0.389 | 0.164 | 0.581 | 0.163 |
| Median | 0.662 | 0.853 | 0.481 | 1.083 | 0.049 | 0.030 | 0.083 | 0.050 | 11.5 | 7.40 | 16.6 | 7.01 | 2.76 | 3.90 | 1.61 | 2.36 | 0.402 | 0.183 | 0.679 | 0.176 |
| Max | 0.799 | 1.090 | 0.630 | 1.700 | 0.122 | 0.084 | 0.151 | 0.119 | 16.0 | 12.16 | 25.1 | 11.56 | 5.89 | 7.36 | 4.16 | 5.57 | 1.553 | 1.262 | 1.752 | 0.604 |
| eSD | 0.056 | 0.047 | 0.043 | 0.089 | 0.016 | 0.007 | 0.026 | 0.030 | 1.0 | 0.86 | 1.4 | 0.59 | 0.25 | 0.37 | 0.13 | 0.22 | | | | |
| eCV | 9 | 5 | 9 | 8 | 32 | 25 | 32 | 59 | 9 | 12 | 9 | 8 | 9 | 10 | 8 | 9 | | | | |
| $N_{past}$ | 0 | 0 | 0 | 44 | 0 | 0 | 0 | 7 | 0 | 0 | 0 | 44 | 0 | 0 | 0 | 20 | 0 | 0 | 0 | 6 |
| $Median_{past}$ | | | | 1.078 | | | | 0.054 | | | | 7.18 | | | | 2.47 | | | | 0.110 |
| $SD_{past}$ | | | | 0.112 | | | | 0.038 | | | | 0.57 | | | | 0.29 | | | | 0.020 |
| NISTa | 0.691 | 0.821 | 0.481 | 1.012 | | | | | 12.4 | 7.83 | 17.4 | 7.16 | 2.65 | 3.97 | 1.49 | 2.25 | 0.296 | 0.133 | 0.465 | *nd* |
| NISTb | 0.662 | 0.847 | 0.491 | 1.064 | | | | | 11.6 | 7.19 | 16.4 | 6.71 | 2.70 | 3.84 | 1.57 | 2.30 | 0.346 | 0.128 | 0.588 | 0.132 |
| $N_{NIST}$ | 5 | 22 | 22 | 6 | | | | | 5 | 22 | 22 | 6 | 5 | 22 | 22 | 6 | 5 | 22 | 22 | 3 |
| Mean | 0.677 | 0.834 | 0.486 | 1.038 | | | | | 12.0 | 7.51 | 16.9 | 6.94 | 2.68 | 3.90 | 1.53 | 2.27 | 0.321 | 0.131 | 0.526 | 0.132 |
| $S_{rep}$ | 0.047 | 0.014 | 0.007 | 0.028 | | | | | 0.4 | 0.21 | 0.2 | 0.15 | 0.17 | 0.07 | 0.04 | 0.03 | 0.006 | 0.007 | 0.015 | 0.004 |
| $S_{het}$ | 0.018 | 0.013 | 0.008 | 0.029 | | | | | 0.3 | 0.11 | 0.2 | 0.06 | 0.06 | 0.03 | 0.02 | 0.04 | 0.002 | 0.004 | 0.016 | 0.004 |
| $S_{anl}$ | 0.021 | 0.018 | 0.007 | 0.037 | | | | | 0.6 | 0.46 | 0.7 | 0.31 | 0.04 | 0.09 | 0.06 | 0.04 | 0.035 | 0.004 | 0.087 | |
| $S_{NIST}$ | 0.055 | 0.026 | 0.013 | 0.055 | | | | | 0.8 | 0.51 | 0.8 | 0.35 | 0.18 | 0.12 | 0.07 | 0.06 | 0.036 | 0.009 | 0.090 | 0.006 |
| NAV | 0.669 | 0.843 | 0.483 | 1.060 | 0.049 | 0.030 | 0.083 | 0.050 | 11.8 | 7.46 | 16.7 | 6.97 | 2.72 | 3.90 | 1.57 | 2.31 | | | | |
| NAU | 0.056 | 0.073 | 0.043 | 0.098 | 0.016 | 0.007 | 0.026 | 0.030 | 1.1 | 0.85 | 1.5 | 0.61 | 0.29 | 0.37 | 0.20 | 0.26 | | | | |

# Round Robin XLIV Laboratory Results

All Results in µg/mL

| | Total β-Carotene | | | | trans-β-Carotene | | | | Total cis-β-Carotene | | | | Total α-Carotene | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Lab | 247 | 248 | 249 | 250 | 247 | 248 | 249 | 250 | 247 | 248 | 249 | 250 | 247 | 248 | 249 | 250 |
| FSV-BA | 0.346 | 0.191 | 0.446 | 0.604 | 0.324 | 0.175 | 0.419 | 0.571 | 0.022 | 0.016 | 0.027 | 0.033 | 0.053 | 0.019 | 0.095 | 0.024 |
| FSV-BB | 0.355 | 0.203 | 0.478 | 0.605 | 0.331 | 0.188 | 0.447 | 0.567 | 0.024 | 0.015 | 0.031 | 0.038 | 0.047 | 0.010 | 0.083 | 0.013 |
| FSV-BBa | 0.297 | 0.159 | 0.392 | 0.517 | 0.279 | 0.149 | 0.367 | 0.487 | 0.019 | 0.010 | 0.025 | 0.030 | | | | |
| FSV-BD | 0.313 | 0.158 | 0.424 | 0.561 | | | | | | | | | | | | |
| FSV-BE | 0.384 | 0.208 | 0.500 | 0.690 | | | | | | | | | | | | |
| FSV-BF | 0.344 | 0.173 | 0.423 | 0.605 | | | | | | | | | 0.068 | 0.015 | 0.121 | 0.019 |
| FSV-BG | 0.347 | 0.165 | 0.451 | 0.594 | | | | | | | | | 0.054 | 0.016 | 0.100 | 0.019 |
| FSV-BH | 0.354 | 0.176 | 0.467 | 0.606 | 0.280 | 0.150 | 0.370 | 0.490 | 0.024 | 0.010 | 0.035 | 0.041 | 0.064 | 0.013 | 0.114 | 0.017 |
| FSV-BI | 0.301 | 0.163 | 0.409 | 0.554 | | | | | | | | | 0.050 | 0.016 | 0.090 | 0.014 |
| FSV-BJ | 0.317 | 0.162 | 0.411 | 0.588 | | | | | | | | | 0.067 | 0.017 | 0.117 | 0.016 |
| FSV-BK | | | | | | | | | | | | | | | | |
| FSV-BL | | | | | | | | | | | | | | | | |
| FSV-BM | | | | | | | | | | | | | | | | |
| FSV-BN | 0.326 | 0.181 | 0.462 | 0.544 | 0.296 | 0.163 | 0.413 | 0.504 | 0.030 | 0.018 | 0.044 | 0.040 | 0.048 | 0.014 | 0.091 | 0.012 |
| FSV-BO | 0.317 | 0.198 | 0.406 | 0.542 | | | | | | | | | 0.076 | 0.030 | 0.139 | 0.021 |
| FSV-BP | 0.179 | 0.101 | 0.250 | 0.366 | | | | | | | | | 0.053 | 0.014 | 0.077 | 0.021 |
| FSV-BQ | | | | | | | | | | | | | | | | |
| FSV-BR | | | | | | | | | | | | | | | | |
| FSV-BS | 0.305 | 0.186 | 0.429 | 0.528 | | | | | | | | | 0.084 | 0.049 | 0.131 | 0.052 |
| FSV-BT | 0.266 | 0.169 | 0.336 | 0.456 | 0.251 | 0.159 | 0.312 | 0.425 | 0.015 | 0.010 | 0.024 | 0.031 | 0.047 | 0.018 | 0.079 | 0.015 |
| FSV-BU | 0.355 | 0.186 | 0.445 | 0.578 | | | | | | | | | 0.070 | 0.021 | 0.112 | 0.024 |
| FSV-BV | 0.330 | 0.190 | 0.361 | 0.568 | | | | | | | | | 0.070 | 0.020 | 0.110 | 0.016 |
| FSV-BW | 0.388 | 0.203 | 0.495 | 0.653 | | | | | | | | | 0.063 | 0.019 | 0.105 | 0.013 |
| FSV-BX | >0.322 | >0.158 | >0.372 | >0.538 | 0.322 | 0.158 | 0.372 | 0.538 | | | | | 0.052 | 0.018 | 0.080 | 0.029 |
| FSV-BZ | >0.247 | >0.132 | >0.312 | >0.432 | 0.247 | 0.132 | 0.312 | 0.432 | 0.010 | *nd* | 0.020 | 0.030 | 0.054 | 0.038 | 0.070 | 0.041 |
| FSV-CB | 0.206 | 0.113 | 0.268 | 0.400 | | | | | | | | | 0.040 | 0.010 | 0.069 | 0.013 |
| FSV-CC | | | | | | | | | | | | | | | | |
| FSV-CD | 0.278 | 0.171 | 0.377 | 0.506 | | | | | | | | | 0.040 | 0.015 | 0.072 | 0.015 |
| FSV-CE | 0.430 | 0.206 | 0.629 | 0.799 | | | | | | | | | | | | |
| FSV-CF | | | | | | | | | | | | | | | | |
| FSV-CH | 0.295 | 0.180 | 0.355 | 0.543 | | | | | | | | | 0.039 | 0.013 | 0.072 | 0.015 |
| FSV-CI | | | | | | | | | | | | | | | | |
| FSV-CL | 0.291 | 0.155 | 0.398 | 0.533 | | | | | | | | | 0.069 | 0.016 | 0.013 | 0.019 |
| FSV-CM | | | | | | | | | | | | | | | | |
| FSV-CP | 0.394 | 0.210 | 0.495 | 0.677 | | | | | | | | | 0.054 | 0.014 | 0.093 | 0.015 |
| FSV-CR | | | | | | | | | | | | | | | | |
| FSV-CS | 0.356 | 0.181 | 0.402 | 0.537 | 0.320 | 0.163 | 0.359 | 0.487 | *0.036* | *0.018* | *0.043* | *0.050* | 0.070 | 0.017 | 0.108 | 0.016 |
| FSV-CU | >0.265 | >0.141 | >0.373 | >0.433 | 0.265 | 0.141 | 0.373 | 0.433 | | | | | | | | |
| FSV-CX | >0.300 | >0.150 | >0.360 | >0.490 | 0.300 | 0.150 | 0.360 | 0.490 | | | | | 0.050 | 0.010 | 0.080 | 0.020 |
| FSV-DA | 0.245 | 0.117 | 0.414 | 0.599 | 0.210 | 0.100 | 0.373 | 0.532 | 0.035 | 0.017 | 0.042 | 0.067 | 0.039 | 0.022 | 0.086 | 0.022 |
| FSV-DB | 0.305 | 0.170 | 0.382 | 0.544 | | | | | | | | | | | | |
| FSV-DF | | | | | | | | | | | | | | | | |
| FSV-DJ | | | | | | | | | | | | | | | | |
| FSV-DK | 0.196 | 0.113 | 0.228 | 0.399 | | | | | | | | | 0.032 | 0.007 | 0.049 | 0.013 |
| FSV-DP | | | | | | | | | | | | | | | | |
| FSV-DR | 0.290 | 0.235 | 0.410 | 1.080 | | | | | | | | | | | | |
| FSV-DU | 0.210 | 0.110 | 0.280 | 0.560 | | | | | | | | | | | | |
| FSV-DW | 0.355 | 0.185 | 0.441 | 0.671 | 0.324 | 0.170 | 0.399 | 0.614 | 0.031 | 0.015 | 0.040 | 0.056 | >0.046 | >0.014 | >0.086 | >0.017 |
| FSV-ES | >0.304 | >0.166 | >0.357 | >0.493 | 0.304 | 0.166 | 0.357 | 0.493 | | | | | >0.053 | >0.027 | >0.078 | >0.025 |
| FSV-FG | | | | | | | | | | | | | | | | |
| FSV-FH | | | | | | | | | | | | | | | | |
| N | 31 | 31 | 31 | 31 | 14 | 14 | 14 | 14 | 10 | 9 | 10 | 10 | 26 | 26 | 26 | 26 |
| Min | 0.179 | 0.101 | 0.228 | 0.366 | 0.210 | 0.100 | 0.312 | 0.425 | 0.010 | 0.010 | 0.020 | 0.030 | 0.032 | 0.007 | 0.013 | 0.012 |
| Median | 0.317 | 0.176 | 0.411 | 0.561 | 0.298 | 0.158 | 0.371 | 0.491 | 0.024 | 0.015 | 0.033 | 0.039 | 0.053 | 0.016 | 0.091 | 0.017 |
| Max | 0.430 | 0.235 | 0.629 | 1.080 | 0.331 | 0.188 | 0.447 | 0.614 | 0.036 | 0.018 | 0.044 | 0.067 | 0.084 | 0.049 | 0.139 | 0.052 |
| eSD | 0.055 | 0.022 | 0.052 | 0.064 | 0.037 | 0.013 | 0.019 | 0.065 | 0.010 | 0.004 | 0.013 | 0.013 | 0.018 | 0.004 | 0.027 | 0.005 |
| eCV | 17 | 13 | 13 | 11 | 12 | 8 | 5 | 13 | 40 | 30 | 39 | 33 | 33 | 28 | 29 | 31 |
| $N_{past}$ | 0 | 0 | 0 | 33 | 0 | 0 | 0 | 10 | 0 | 0 | 0 | 8 | 0 | 0 | 0 | 21 |
| $Median_{past}$ | | | | 0.589 | | | | 0.550 | | | | 0.042 | | | | 0.018 |
| $SD_{past}$ | | | | 0.085 | | | | 0.072 | | | | 0.030 | | | | 0.008 |
| NISTa | 0.358 | 0.179 | 0.444 | >0.475 | 0.348 | 0.170 | 0.431 | 0.475 | *0.010* | *0.009* | *0.013* | *nq* | 0.074 | 0.022 | 0.113 | *nd* |
| NISTb | 0.327 | >0.158 | 0.452 | 0.566 | 0.299 | 0.158 | 0.404 | 0.519 | *0.028* | *nq* | *0.048* | *0.047* | >0.054 | >0.017 | 0.111 | 0.029 |
| $N_{NIST}$ | 5 | 5 | 22 | 3 | 5 | 22 | 22 | 6 | 5 | 5 | 22 | 3 | 2 | 4 | 22 | 3 |
| Mean | 0.343 | 0.168 | 0.448 | 0.521 | 0.324 | 0.164 | 0.418 | 0.497 | 0.019 | 0.009 | 0.030 | 0.047 | 0.064 | 0.019 | 0.111 | 0.029 |
| $S_{rep}$ | 0.010 | 0.004 | 0.012 | 0.007 | 0.009 | 0.005 | 0.013 | 0.013 | 0.009 | 0.005 | 0.014 | 0.009 | 0.003 | 0.002 | 0.004 | 0.005 |
| $S_{het}$ | 0.022 | 0.004 | 0.019 | 0.008 | 0.015 | 0.004 | 0.014 | 0.011 | 0.008 | 0.004 | 0.012 | 0.004 | 0.003 | 0.002 | 0.007 | 0.002 |
| $S_{anl}$ | 0.022 | | 0.006 | | 0.035 | 0.008 | 0.019 | 0.031 | 0.013 | | 0.025 | | | | 0.000 | |
| $S_{NIST}$ | 0.033 | 0.006 | 0.023 | 0.011 | 0.039 | 0.010 | 0.027 | 0.035 | 0.018 | 0.006 | 0.031 | 0.010 | 0.005 | 0.003 | 0.008 | 0.005 |
| NAV | 0.330 | 0.172 | 0.429 | 0.541 | 0.311 | 0.161 | 0.394 | 0.494 | 0.022 | 0.012 | 0.032 | 0.043 | 0.059 | 0.018 | 0.101 | 0.023 |
| NAU | 0.051 | 0.030 | 0.065 | 0.083 | 0.043 | 0.019 | 0.052 | 0.052 | 0.018 | 0.008 | 0.031 | 0.014 | 0.017 | 0.006 | 0.029 | 0.011 |

# Round Robin XLIV Laboratory Results

All Results in µg/mL

| | trans-α-Carotene | | | | Total Lycopene | | | | trans-Lycopene | | | | Total β-Cryptoxanthin | | | | Total α-Cryptoxanthin | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Lab | 247 | 248 | 249 | 250 | 247 | 248 | 249 | 250 | 247 | 248 | 249 | 250 | 247 | 248 | 249 | 250 | 247 | 248 | 249 | 250 |
| FSV-BA | | | | | | | | | 0.195 | 0.167 | 0.215 | 0.364 | 0.076 | 0.094 | 0.045 | 0.063 | | | | |
| FSV-BB | | | | | 0.357 | 0.315 | 0.42 | 0.59 | 0.157 | 0.139 | 0.182 | 0.305 | 0.070 | 0.089 | 0.048 | 0.064 | 0.016 | 0.019 | 0.014 | 0.019 |
| FSV-BBa | | | | | | | | | | | | | 0.055 | 0.072 | 0.033 | 0.049 | | | | |
| FSV-BD | | | | | 0.271 | 0.207 | 0.34 | 0.48 | | | | | 0.046 | 0.062 | 0.021 | 0.040 | | | | |
| FSV-BE | | | | | | | | | | | | | | | | | | | | |
| FSV-BF | | | | | 0.407 | 0.364 | 0.46 | 0.72 | | | | | 0.053 | 0.072 | 0.028 | 0.045 | | | | |
| FSV-BG | | | | | 0.415 | 0.351 | 0.46 | 0.71 | 0.223 | 0.184 | 0.244 | 0.417 | 0.059 | 0.078 | 0.032 | 0.048 | | | | |
| FSV-BH | | | | | 0.372 | 0.323 | 0.43 | 0.63 | | | | | 0.079 | 0.097 | 0.047 | 0.063 | | | | |
| FSV-BI | | | | | 0.286 | 0.239 | 0.33 | 0.50 | | | | | 0.062 | 0.084 | 0.030 | 0.052 | | | | |
| FSV-BJ | | | | | 0.285 | 0.246 | 0.32 | 0.47 | | | | | 0.063 | 0.086 | 0.029 | 0.048 | | | | |
| FSV-BK | | | | | | | | | | | | | | | | | | | | |
| FSV-BL | | | | | | | | | | | | | | | | | | | | |
| FSV-BM | | | | | | | | | | | | | | | | | | | | |
| FSV-BN | | | | | 0.334 | 0.306 | 0.40 | 0.55 | 0.176 | 0.163 | 0.207 | 0.326 | 0.061 | 0.082 | 0.037 | 0.051 | 0.018 | 0.023 | 0.020 | 0.024 |
| FSV-BO | | | | | 0.564 | 0.507 | 0.62 | 0.97 | | | | | 0.050 | 0.076 | 0.032 | 0.047 | | | | |
| FSV-BP | | | | | 0.367 | 0.291 | 0.39 | 0.57 | | | | | 0.066 | 0.085 | 0.042 | 0.073 | | | | |
| FSV-BQ | | | | | | | | | | | | | | | | | | | | |
| FSV-BR | | | | | | | | | | | | | | | | | | | | |
| FSV-BS | | | | | 0.396 | 0.340 | 0.49 | 0.65 | 0.135 | 0.124 | 0.172 | 0.292 | 0.047 | 0.066 | 0.028 | 0.041 | | | | |
| FSV-BT | | | | | 0.266 | 0.289 | 0.31 | 0.45 | 0.219 | 0.236 | 0.249 | 0.379 | 0.070 | 0.083 | 0.044 | 0.058 | 0.027 | 0.029 | 0.027 | 0.034 |
| FSV-BU | | | | | 0.374 | 0.314 | 0.43 | 0.67 | | | | | 0.060 | 0.075 | 0.035 | 0.059 | | | | |
| FSV-BV | | | | | 0.390 | 0.362 | 0.42 | 0.66 | | | | | 0.031 | 0.047 | 0.014 | 0.024 | | | | |
| FSV-BW | | | | | 0.373 | 0.330 | 0.42 | 0.59 | | | | | | | | | | | | |
| FSV-BX | | | | | | | | | 0.177 | 0.150 | 0.173 | 0.338 | 0.045 | 0.061 | 0.026 | 0.042 | | | | |
| FSV-BZ | | | | | | | | | 0.160 | 0.080 | 0.180 | 0.400 | | | | | | | | |
| FSV-CB | | | | | 0.075 | 0.066 | 0.08 | 0.13 | | | | | 0.050 | 0.054 | 0.024 | 0.033 | | | | |
| FSV-CC | | | | | | | | | | | | | | | | | | | | |
| FSV-CD | | | | | 0.397 | 0.359 | 0.46 | 0.62 | | | | | 0.054 | 0.072 | 0.028 | 0.043 | 0.019 | 0.020 | 0.018 | 0.029 |
| FSV-CE | | | | | | | | | | | | | | | | | | | | |
| FSV-CF | | | | | | | | | | | | | | | | | | | | |
| FSV-CH | | | | | 0.600 | 0.615 | 0.74 | 1.11 | | | | | | | | | | | | |
| FSV-CI | | | | | | | | | | | | | | | | | | | | |
| FSV-CL | | | | | 0.376 | 0.330 | 0.46 | 0.65 | | | | | 0.054 | 0.073 | 0.035 | 0.057 | 0.015 | 0.017 | 0.040 | 0.083 |
| FSV-CM | | | | | | | | | | | | | | | | | | | | |
| FSV-CP | | | | | 0.212 | 0.186 | 0.23 | 0.39 | | | | | 0.068 | 0.089 | 0.035 | 0.057 | | | | |
| FSV-CR | | | | | | | | | | | | | | | | | | | | |
| FSV-CS | | | | | 0.369 | 0.304 | 0.37 | 0.52 | | | | | 0.058 | 0.074 | 0.031 | 0.044 | | | | |
| FSV-CU | | | | | | | | | | | | | | | | | | | | |
| FSV-CX | | | | | | | | | 0.350 | 0.290 | 0.380 | 0.560 | 0.070 | 0.080 | 0.050 | 0.070 | | | | |
| FSV-DA | *nq* | *nq* | *nq* | *nq* | 0.270 | 0.207 | 0.39 | 0.71 | 0.137 | 0.108 | 0.228 | 0.387 | 0.049 | 0.043 | 0.051 | 0.063 | 0.018 | 0.006 | 0.016 | 0.019 |
| FSV-DB | | | | | 0.361 | 0.328 | 0.39 | 0.65 | | | | | 0.051 | 0.081 | 0.025 | 0.046 | | | | |
| FSV-DF | | | | | | | | | | | | | | | | | | | | |
| FSV-DJ | | | | | | | | | | | | | | | | | | | | |
| FSV-DK | | | | | | | | | | | | | | | | | | | | |
| FSV-DP | | | | | | | | | | | | | | | | | | | | |
| FSV-DR | | | | | | | | | | | | | | | | | | | | |
| FSV-DU | | | | | | | | | | | | | | | | | | | | |
| FSV-DW | 0.046 | 0.014 | 0.086 | 0.017 | 0.222 | 0.196 | 0.25 | 0.47 | 0.141 | 0.126 | 0.155 | 0.319 | 0.059 | 0.081 | 0.032 | 0.059 | | | | |
| FSV-ES | 0.053 | 0.027 | 0.078 | 0.025 | 0.272 | 0.224 | 0.31 | 0.50 | 0.181 | 0.155 | 0.199 | 0.340 | 0.058 | 0.080 | 0.033 | 0.051 | | | | |
| FSV-FG | | | | | | | | | | | | | | | | | | | | |
| FSV-FH | | | | | | | | | | | | | | | | | | | | |
| N | 2 | 2 | 2 | 2 | 25 | 25 | 25 | 25 | 12 | 12 | 12 | 12 | 27 | 27 | 27 | 27 | 6 | 6 | 6 | 6 |
| Min | 0.046 | 0.014 | 0.078 | 0.017 | 0.075 | 0.066 | 0.08 | 0.13 | 0.135 | 0.080 | 0.155 | 0.292 | 0.031 | 0.043 | 0.014 | 0.024 | 0.015 | 0.006 | 0.014 | 0.019 |
| Median | 0.049 | 0.020 | 0.082 | 0.021 | 0.367 | 0.314 | 0.40 | 0.59 | 0.177 | 0.152 | 0.203 | 0.352 | 0.058 | 0.078 | 0.032 | 0.051 | 0.018 | 0.020 | 0.019 | 0.027 |
| Max | 0.053 | 0.027 | 0.086 | 0.025 | 0.600 | 0.615 | 0.74 | 1.11 | 0.350 | 0.290 | 0.380 | 0.560 | 0.079 | 0.097 | 0.051 | 0.073 | 0.027 | 0.029 | 0.040 | 0.083 |
| eSD | | | | | 0.059 | 0.067 | 0.09 | 0.13 | 0.041 | 0.040 | 0.040 | 0.050 | 0.012 | 0.009 | 0.006 | 0.012 | 0.002 | 0.004 | 0.006 | 0.011 |
| eCV | | | | | 16 | 21 | 23 | 21 | 23 | 27 | 20 | 14 | 20 | 11 | 19 | 23 | 12 | 23 | 31 | 42 |
| $N_{past}$ | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 24 | 0 | 0 | 0 | 9 | 0 | 0 | 0 | 18 | 0 | 0 | 0 | 0 |
| $Median_{past}$ | | | | | | | | 0.57 | | | | 0.409 | | | | 0.065 | | | | |
| $SD_{past}$ | | | | | | | | 0.14 | | | | 0.107 | | | | 0.018 | | | | |
| NISTa | | | | | | | | | | | | | 0.049 | 0.067 | 0.029 | 0.032 | | | | |
| NISTb | 0.054 | 0.017 | 0.098 | 0.017 | 0.400 | 0.342 | 0.48 | 0.68 | 0.188 | 0.158 | 0.213 | 0.336 | 0.057 | 0.079 | 0.035 | 0.050 | | | | |
| $N_{NIST}$ | 3 | 17 | 17 | 3 | 3 | 17 | 17 | 3 | 3 | 17 | 17 | 3 | 5 | 22 | 22 | 6 | | | | |
| Mean | 0.054 | 0.017 | 0.098 | 0.017 | 0.400 | 0.342 | 0.48 | 0.68 | 0.188 | 0.158 | 0.213 | 0.336 | 0.053 | 0.073 | 0.032 | 0.041 | | | | |
| $S_{rep}$ | 0.004 | 0.002 | 0.004 | 0.002 | 0.012 | 0.028 | 0.02 | 0.01 | 0.002 | 0.007 | 0.005 | 0.015 | 0.003 | 0.004 | 0.002 | 0.001 | | | | |
| $S_{het}$ | 0.001 | 0.002 | 0.004 | 0.002 | 0.006 | 0.014 | 0.02 | 0.00 | 0.015 | 0.005 | 0.005 | 0.008 | 0.003 | 0.004 | 0.003 | 0.002 | | | | |
| $S_{anl}$ | | | | | | | | | | | | | 0.006 | 0.009 | 0.004 | 0.013 | | | | |
| $S_{NIST}$ | 0.005 | 0.002 | 0.005 | 0.002 | 0.014 | 0.032 | 0.03 | 0.01 | 0.015 | 0.008 | 0.007 | 0.017 | 0.007 | 0.010 | 0.006 | 0.013 | | | | |
| NAV | | | | | 0.384 | 0.328 | 0.44 | 0.63 | 0.182 | 0.155 | 0.208 | 0.344 | 0.056 | 0.075 | 0.032 | 0.046 | | | | |
| NAU | | | | | 0.091 | 0.078 | 0.11 | 0.14 | 0.037 | 0.034 | 0.042 | 0.073 | 0.015 | 0.020 | 0.009 | 0.015 | | | | |

# Round Robin XLIV Laboratory Results

All Results in µg/mL

| | Total Lutein | | | | Total Zeaxanthin | | | | Total Lutein&Zeaxanthin | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Lab | 247 | 248 | 249 | 250 | 247 | 248 | 249 | 250 | 247 | 248 | 249 | 250 |
| FSV-BA | | | | | | | | | 0.175 | 0.167 | 0.179 | 0.182 |
| FSV-BB | 0.111 | 0.085 | 0.137 | 0.124 | 0.032 | 0.036 | 0.029 | 0.044 | 0.143 | 0.121 | 0.166 | 0.168 |
| FSV-BBa | 0.102 | 0.077 | 0.156 | 0.118 | 0.028 | 0.032 | 0.010 | 0.040 | 0.130 | 0.109 | 0.166 | 0.158 |
| FSV-BD | 0.072 | 0.055 | 0.086 | 0.083 | 0.026 | 0.032 | 0.031 | 0.036 | *0.098* | *0.087* | *0.117* | *0.119* |
| FSV-BE | | | | | | | | | | | | |
| FSV-BF | | | | | | | | | 0.096 | 0.086 | 0.109 | 0.093 |
| FSV-BG | 0.071 | 0.074 | 0.102 | 0.104 | 0.024 | 0.018 | 0.022 | 0.025 | 0.110 | 0.105 | 0.140 | 0.146 |
| FSV-BH | 0.050 | 0.037 | 0.063 | 0.051 | 0.022 | 0.025 | 0.017 | 0.026 | *0.072* | *0.062* | *0.080* | *0.077* |
| FSV-BI | 0.062 | 0.049 | 0.076 | 0.066 | 0.025 | 0.025 | 0.020 | 0.028 | | | | |
| FSV-BJ | | | | | | | | | 0.081 | 0.074 | 0.080 | 0.082 |
| FSV-BK | | | | | | | | | | | | |
| FSV-BL | | | | | | | | | | | | |
| FSV-BM | | | | | | | | | | | | |
| FSV-BN | 0.078 | 0.060 | 0.087 | 0.075 | 0.055 | 0.052 | 0.058 | 0.066 | 0.140 | 0.117 | 0.151 | 0.148 |
| FSV-BO | 0.111 | 0.094 | 0.135 | 0.118 | 0.044 | 0.047 | 0.034 | 0.048 | *0.155* | *0.142* | *0.169* | *0.166* |
| FSV-BP | | | | | | | | | | | | |
| FSV-BQ | | | | | | | | | | | | |
| FSV-BR | | | | | | | | | | | | |
| FSV-BS | 0.063 | 0.065 | 0.085 | 0.092 | 0.022 | 0.031 | 0.020 | 0.033 | 0.086 | 0.097 | 0.105 | 0.125 |
| FSV-BT | 0.100 | 0.088 | 0.085 | 0.093 | 0.038 | 0.040 | 0.030 | 0.029 | 0.138 | 0.127 | 0.114 | 0.122 |
| FSV-BU | | | | | | | | | 0.160 | 0.124 | 0.159 | 0.169 |
| FSV-BV | | | | | | | | | 0.090 | 0.089 | 0.095 | 0.098 |
| FSV-BW | | | | | | | | | | | | |
| FSV-BX | 0.006 | 0.041 | 0.072 | 0.062 | 0.024 | 0.022 | 0.022 | 0.027 | 0.030 | 0.063 | 0.094 | 0.089 |
| FSV-BZ | 0.073 | 0.070 | 0.080 | 0.072 | | | | | | | | |
| FSV-CB | | 0.050 | | 0.071 | | 0.030 | | 0.033 | 0.119 | | 0.122 | |
| FSV-CC | | | | | | | | | | | | |
| FSV-CD | | | | | | | | | 0.089 | 0.081 | 0.097 | 0.080 |
| FSV-CE | | | | | | | | | | | | |
| FSV-CF | | | | | | | | | | | | |
| FSV-CH | | | | | | | | | | | | |
| FSV-CI | | | | | | | | | | | | |
| FSV-CL | | | | | | | | | 0.096 | 0.094 | 0.099 | 0.096 |
| FSV-CM | | | | | | | | | | | | |
| FSV-CP | | | | | | | | | 0.112 | 0.104 | 0.118 | 0.130 |
| FSV-CR | | | | | | | | | | | | |
| FSV-CS | | | | | | | | | 0.106 | 0.095 | 0.102 | 0.096 |
| FSV-CU | | | | | | | | | | | | |
| FSV-CX | 0.060 | 0.040 | 0.070 | 0.070 | 0.040 | 0.050 | 0.050 | 0.070 | | | | |
| FSV-DA | 0.071 | 0.051 | 0.091 | 0.070 | 0.035 | 0.040 | 0.037 | 0.048 | 0.106 | 0.091 | 0.128 | 0.118 |
| FSV-DB | | | | | | | | | 0.095 | 0.095 | 0.095 | 0.097 |
| FSV-DF | | | | | | | | | | | | |
| FSV-DJ | | | | | | | | | | | | |
| FSV-DK | | | | | | | | | | | | |
| FSV-DP | | | | | | | | | | | | |
| FSV-DR | | | | | | | | | | | | |
| FSV-DU | | | | | | | | | | | | |
| FSV-DW | 0.049 | 0.036 | 0.061 | 0.061 | 0.025 | 0.028 | 0.024 | 0.040 | *0.074* | *0.064* | *0.085* | *0.101* |
| FSV-ES | 0.063 | 0.051 | 0.075 | 0.069 | 0.024 | 0.034 | 0.021 | 0.027 | *0.087* | *0.085* | *0.096* | *0.096* |
| FSV-FG | | | | | | | | | | | | |
| FSV-FH | | | | | | | | | | | | |
| N | 16 | 17 | 16 | 17 | 15 | 16 | 15 | 16 | 24 | 23 | 24 | 23 |
| Min | 0.006 | 0.036 | 0.061 | 0.051 | 0.022 | 0.018 | 0.010 | 0.025 | 0.030 | 0.062 | 0.080 | 0.077 |
| Median | 0.071 | 0.055 | 0.085 | 0.072 | 0.026 | 0.032 | 0.024 | 0.035 | 0.102 | 0.095 | 0.112 | 0.118 |
| Max | 0.111 | 0.094 | 0.156 | 0.124 | 0.055 | 0.052 | 0.058 | 0.070 | 0.175 | 0.167 | 0.179 | 0.182 |
| eSD | 0.015 | 0.022 | 0.017 | 0.016 | 0.005 | 0.010 | 0.008 | 0.011 | 0.024 | 0.021 | 0.025 | 0.037 |
| eCV | 21 | 40 | 20 | 23 | 21 | 32 | 35 | 32 | 24 | 22 | 22 | 31 |
| $N_{past}$ | 0 | 0 | 0 | 13 | 0 | 0 | 0 | 8 | 0 | 0 | 0 | 14 |
| $Median_{past}$ | | | | 0.087 | | | | 0.034 | | | | 0.116 |
| $SD_{past}$ | | | | 0.026 | | | | 0.011 | | | | 0.027 |
| NISTa | 0.077 | 0.059 | 0.090 | 0.084 | 0.020 | 0.020 | 0.013 | 0.041 | *0.097* | *0.079* | *0.102* | *0.125* |
| NISTb | 0.071 | 0.063 | 0.101 | 0.091 | 0.039 | 0.044 | 0.040 | 0.053 | *0.111* | *0.108* | *0.141* | *0.145* |
| $N_{NIST}$ | 5 | 22 | 22 | 6 | 5 | 22 | 22 | 6 | 5 | 22 | 22 | 6 |
| Mean | 0.074 | 0.061 | 0.095 | 0.088 | 0.030 | 0.032 | 0.026 | 0.047 | 0.104 | 0.093 | 0.122 | 0.135 |
| $S_{rep}$ | 0.002 | 0.003 | 0.003 | 0.001 | 0.002 | 0.002 | 0.002 | 0.001 | 0.003 | 0.004 | 0.005 | 0.002 |
| $S_{het}$ | 0.002 | 0.004 | 0.003 | 0.002 | 0.001 | 0.003 | 0.002 | 0.001 | 0.002 | 0.005 | 0.004 | 0.003 |
| $S_{anl}$ | 0.004 | 0.003 | 0.008 | 0.005 | 0.013 | 0.017 | 0.019 | 0.009 | 0.009 | 0.020 | 0.027 | 0.014 |
| $S_{NIST}$ | 0.005 | 0.006 | 0.009 | 0.006 | 0.013 | 0.018 | 0.019 | 0.009 | 0.010 | 0.021 | 0.028 | 0.014 |
| NAV | 0.073 | 0.058 | 0.090 | 0.080 | 0.028 | 0.032 | 0.025 | 0.041 | 0.103 | 0.094 | 0.117 | 0.126 |
| NAU | 0.016 | 0.019 | 0.020 | 0.021 | 0.014 | 0.018 | 0.020 | 0.015 | 0.032 | 0.021 | 0.036 | 0.040 |

# Round Robin XLIV Laboratory Results

## Analytes Reported By One Laboratory

Values in µg/mL

| Analyte | Code | 247 | 248 | 249 | 250 |
|---|---|---|---|---|---|
| Coenzyme Q10 | FSV-CH | 0.577 | 0.075 | 0.615 | 0.553 |
| Phytoene | FSV-CL | 0.074 | 0.066 | 0.088 | 0.084 |
| Phytofluene | FSV-CL | 0.074 | 0.047 | 0.066 | 0.071 |
| Total cis-β-Cryptoxanthin | FSV-BT | 0.019 | 0.024 | 0.016 | 0.029 |
| trans-anhydro-Lutein | FSV-BT | 0.040 | 0.035 | 0.045 | 0.046 |

## Legend

| Term | Definition |
|---|---|
| N | Number of (non-NIST) quantitative values reported for this analyte |
| Min | Minimum (non-NIST) quantitative value reported |
| Median | Median (non-NIST) quantitative value reported |
| Max | Maximum (non-NIST) quantitative value reported |
| eSD | Adjusted median absolute deviation from the median of the non-NIST results |
| eCV | 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 | 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 + Mean)/2 for analytes reported by NIST analyst(s)<br>= Median 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 and Mean. The expected long-term SD, eSD, is defined in: Duewer, et al. Anal Chem 1997;69(7):1406-1413. |
| *nd* | Not detected (i.e., no detectable peak for analyte) |
| *nq* | Detected but not quantitatively determined |
| <x | Concentration below the limit of quantification, x |
| >x | Concentration greater than x |
| *italics* | Not explicitly reported but calculated by NIST from reported values |

# Round Robin XLIV Laboratory Results

## Comparability Summary

| Lab | R | aT | gT | bC | tbC | aC | TLy | TbX |
|---|---|---|---|---|---|---|---|---|
| FSV-BA | 1 | 1 | 2 | 1 | 2 | 1 | 2 | 1 |
| FSV-BB | 1 | 1 | 2 | 2 | 2 | 1 | 1 | 1 |
| FSV-BBa | | 1 | 1 | 1 | 1 | | 2 | 2 |
| FSV-BD | 1 | 1 | | 1 | | 1 | 1 | 1 |
| FSV-BE | 1 | 2 | 1 | 2 | | | | |
| FSV-BF | 2 | 1 | 1 | 1 | | 1 | | 2 |
| FSV-BG | 1 | 1 | 1 | 1 | | 2 | 1 | 2 |
| FSV-BH | 1 | 1 | 1 | 1 | 1 | | | 1 |
| FSV-BI | 1 | 1 | 2 | 1 | | 1 | 1 | 2 |
| FSV-BJ | 2 | 2 | 1 | 1 | | 1 | | 1 |
| FSV-BK | 1 | 1 | | | | 3 | | |
| FSV-BL | 4 | 4 | | | | | | |
| FSV-BM | 1 | 1 | | | | | | |
| FSV-BN | 1 | 1 | 2 | 1 | 1 | 1 | 2 | 1 |
| FSV-BO | 2 | 4 | | 1 | | | | |
| FSV-BP | 2 | 2 | | 3 | | | | |
| FSV-BQ | 2 | 2 | | | | 1 | 2 | 1 |
| FSV-BR | 2 | | | | | 2 | 4 | |
| FSV-BS | 2 | | | 1 | | 3 | 1 | 1 |
| FSV-BT | 2 | 3 | 2 | 2 | 2 | 2 | | 2 |
| FSV-BU | 1 | 2 | 2 | 1 | | 2 | 4 | 2 |
| FSV-BV | 4 | 1 | 2 | 2 | | 1 | 1 | 2 |
| FSV-BW | 1 | 2 | 1 | 2 | | 1 | 1 | 1 |
| FSV-BX | 1 | 1 | 1 | | 1 | 4 | 1 | 1 |
| FSV-BZ | | 3 | 3 | | 2 | 2 | 3 | 1 |
| FSV-CB | 1 | 1 | | 3 | | 1 | 2 | 2 |
| FSV-CC | 2 | 1 | | | | | | |
| FSV-CD | 1 | 2 | 1 | 1 | | | | |
| FSV-CE | 2 | 2 | | 3 | | | | |
| FSV-CF | 1 | 2 | | | | | | |
| FSV-CH | 1 | 2 | 2 | 2 | | | | |
| FSV-CI | 2 | 2 | | | | 2 | 1 | 1 |
| FSV-CL | 2 | 4 | 3 | 1 | | 2 | | |
| FSV-CM | | 2 | | | | | 1 | 1 |
| FSV-CP | | 1 | 1 | 2 | | 1 | 1 | 1 |
| FSV-CR | 2 | 1 | | | | 2 | 2 | 3 |
| FSV-CS | 1 | 2 | 2 | 1 | 1 | | | |
| FSV-CU | 2 | 3 | | | 2 | 1 | 1 | 2 |
| FSV-CX | 1 | 1 | | | 1 | | | |
| FSV-DA | 1 | 1 | 1 | 2 | 3 | 1 | 1 | |
| FSV-DB | 2 | 1 | | 1 | | | | |
| FSV-DF | 1 | | | | | 1 | 1 | 1 |
| FSV-DJ | 1 | | | | | | | |
| FSV-DK | 3 | 4 | 4 | 3 | | | | |
| FSV-DP | 1 | | | | | | | |
| FSV-DR | 4 | 4 | | 4 | | | | |
| FSV-DU | 1 | 2 | | 3 | | | | |
| FSV-DW | 2 | 2 | | 2 | 3 | | 2 | 1 |
| FSV-ES | 1 | 2 | | | 1 | | | |
| FSV-FG | 1 | 1 | | | | | 2 | 1 |
| FSV-FH | | 1 | | | | | | |
| NISTa | 1 | 1 | 1 | 1 | 1 | 1 | | 1 |
| NISTb | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 |
| n | 47 | 47 | 24 | 32 | 15 | 27 | 25 | 28 |

| Label | Definition |
|---|---|
| Lab | laboratory number |
| R | "Standard Score" for Retinol |
| aT | "Standard Score" for $\alpha$-Tocopherol |
| gT | "Standard Score" for $\gamma$-Tocopherol |
| bC | "Standard Score" for Total $\beta$-Carotene |
| tbC | "Standard Score" for trans-$\beta$-Carotene |
| aC | "Standard Score" for Total $\alpha$-Carotene |
| Ly | "Standard Score" for Total Lycopene |
| bX | "Standard Score" for $\beta$-Cryptoxanthin |
| n | number of laboratories providing data for this analyte |

"Standard Score"

Given that our knowledge of the shape, location, and width of the measurement distributions is approximate and that a limited number of labs are involved, we summarize comparability with the following four-level "Standard Score" (StS)…

| StS | Definition | |
|---|---|---|
| 1 | All StV within ±*t* (1-0.683,n-1) | { ±1 SD} |
| 2 | All StV within ±*t* (1-0.954,n-1) | { ±2 SD} |
| 3 | All StV within ±*t* (1-0.997,n-1) | { ±3 SD} |
| 4 | At least one StV > ±*t* (1-0.997,n-1) | { >3 SD} |

| where: | |
|---|---|
| StV | Standardized Value, the distance in standard deviation units your value is from the "true" concentration: StV = (your value - NAV) / NAU |
| NAV | NIST Assigned Value, our estimate of the "true" analyte concentration |
| NAU | NIST Assigned Uncertainty, our estimate of the total measurement standard deviation (serum heterogeniety, analytical repeatibility, and among-laboratory reproducibility) |
| *t* (1-α,n-1) | Two-tailed Student's *t* for coverage of ±1, ±2, and ±3 NAU about NAV, assuming a normal population of size n |

| | TR | aT | g/bT | bC | tbC | aC | TLy | TbX | Expected |
|---|---|---|---|---|---|---|---|---|---|
| % 1 | 55 | 47 | 46 | 50 | 47 | 56 | 56 | 61 | 68.2 % |
| % 2 | 34 | 34 | 38 | 28 | 33 | 30 | 32 | 32 | 27.3 % |
| % 3 | 2 | 6 | 8 | 16 | 13 | 7 | 4 | 4 | 4.3 % |
| % 4 | 6 | 11 | 4 | 3 | 0 | 4 | 8 | 0 | 0.3 % |

These are the observed and normal-population-expected proportions of each Standard Score (StS), based upon each laboratory's largest StV for the four sera.

## Appendix L. Representative "Individualized Report" for RR44

Each participant in RR44 received an "Individualized Report" reflecting their reported results. Each report included a detailed analysis of the results they reported for the following analytes:

- Retinol
- α-Tocopherol
- γ-Tocopherol
- Total β-Carotene
- *trans*-β-Carotene
- Total α-Carotene
- Total Lycopene
- *trans*-Lycopene
- Total β-Cryptoxanthin
- Total Lutein
- Total Zeaxanthin

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

**Note**

The design file and program used to generate the original RR44 "Individualized Reports" are no longer available. The following pages were produced using the 2013 version of the program (a linear descendant of that used in 1998) and a re-created design file.

# Individualized Round Robin XLIV Report: FSV-BA

## Summary

| | Serum 247 | | | Serum 248 | | | Serum 249 | | | Serum 250 | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Analyte | You | NAV | n | You | NAV | n | You | NAV | n | You | NAV | n |
| Retinol | 0.664 | 0.669 | 46 | 0.849 | 0.843 | 46 | 0.488 | 0.483 | 46 | 1.120 | 1.060 | 46 |
| Retinyl Palmitate | 0.081 | 0.049 | 14 | 0.061 | 0.030 | 13 | 0.107 | 0.083 | 15 | 0.075 | 0.050 | 10 |
| α-Tocopherol | 10.92 | 11.77 | 46 | 7.3 | 7.5 | 46 | 15.5 | 16.7 | 46 | 6.92 | 6.97 | 46 |
| γ-Tocopherol | 2.42 | 2.72 | 23 | 3.65 | 3.90 | 23 | 1.48 | 1.57 | 23 | 2.34 | 2.31 | 23 |
| Total β-Carotene | 0.346 | 0.330 | 31 | 0.191 | 0.172 | 31 | 0.446 | 0.429 | 31 | 0.604 | 0.541 | 31 |
| trans-β-Carotene | 0.324 | 0.311 | 14 | 0.175 | 0.161 | 14 | 0.419 | 0.394 | 14 | 0.571 | 0.494 | 14 |
| Total cis-β-Carotene | 0.022 | 0.022 | 10 | 0.016 | 0.012 | 9 | 0.027 | 0.032 | 10 | 0.033 | 0.043 | 10 |
| Total α-Carotene | 0.053 | 0.059 | 26 | 0.019 | 0.018 | 26 | 0.095 | 0.101 | 26 | 0.024 | 0.023 | 26 |
| trans-Lycopene | 0.195 | 0.182 | 12 | 0.167 | 0.155 | 12 | 0.215 | 0.208 | 12 | 0.364 | 0.344 | 12 |
| Total β-Cryptoxanthin | 0.076 | 0.056 | 27 | 0.094 | 0.075 | 27 | 0.045 | 0.032 | 27 | 0.063 | 0.046 | 27 |
| Total Lutein&Zeaxanthin | 0.175 | 0.103 | 24 | 0.167 | 0.094 | 23 | 0.179 | 0.117 | 24 | 0.182 | 0.126 | 23 |

L2

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

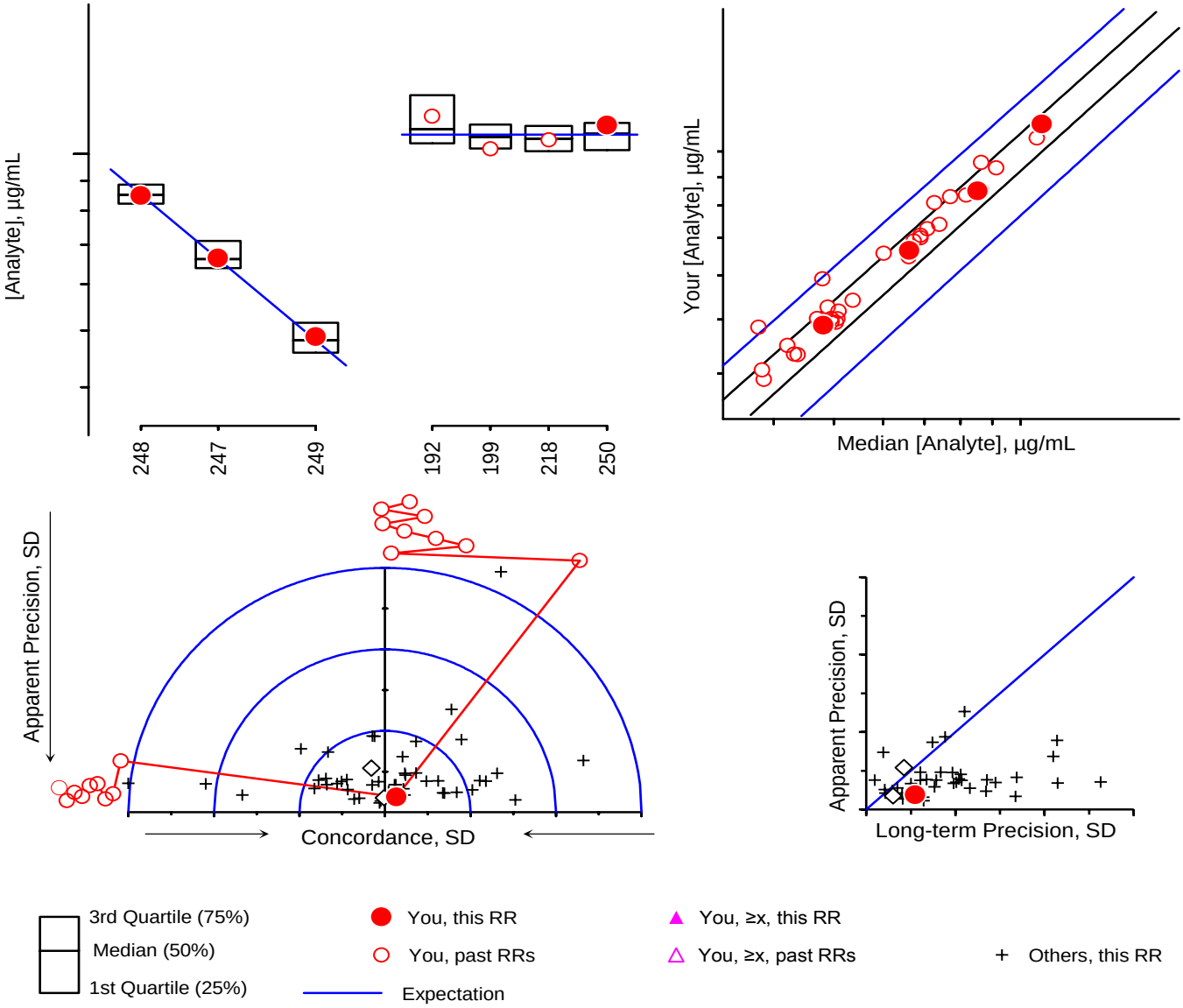
Please check our records against your records. Send corrections and/or updates to...

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

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

# Individualized RR XLIV Report: FSV-BA

Retinol, µg/mL

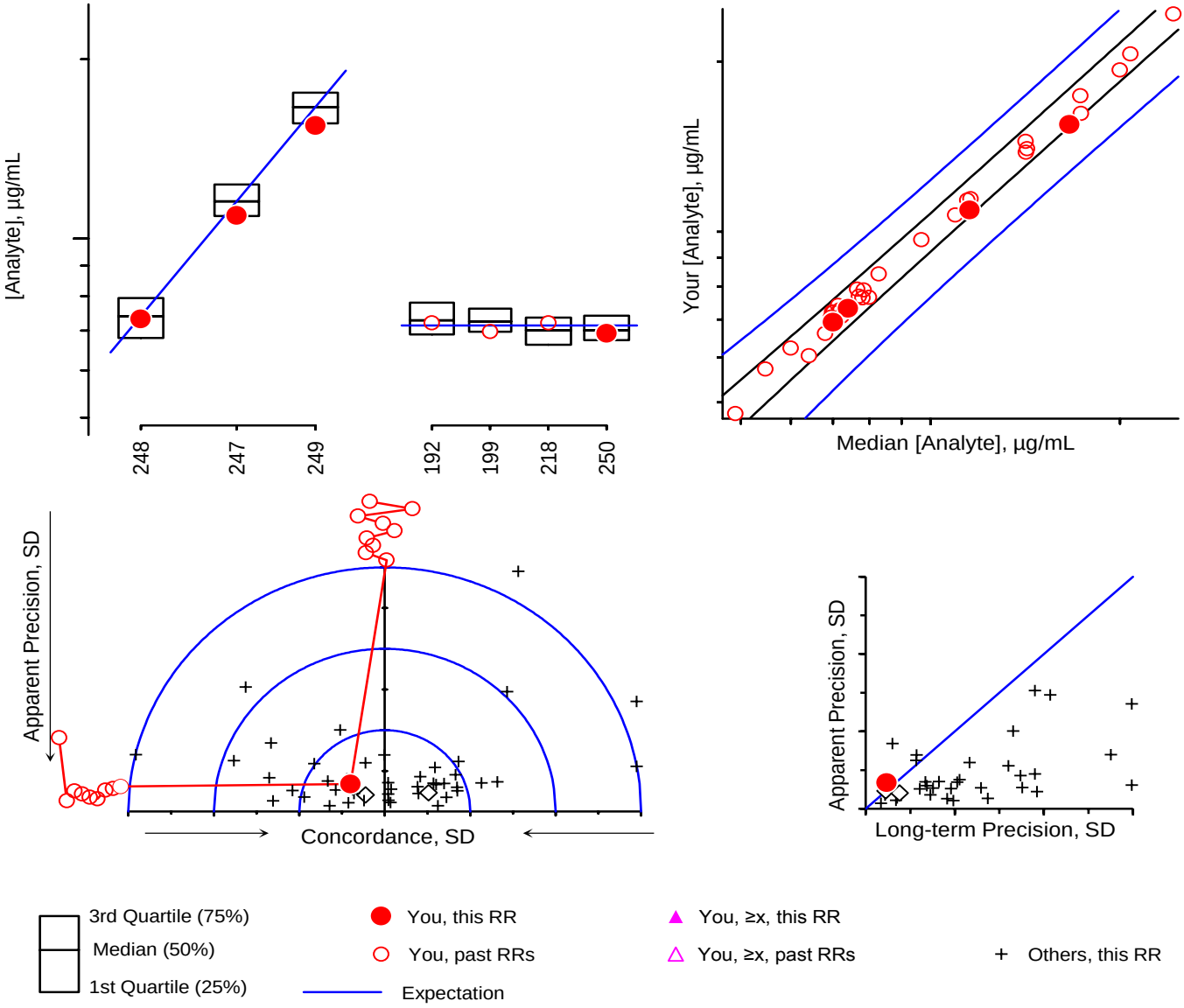



For the construction and interpretation of these plots, see the Final Report for "Round Robin" XL, August 1997

| Serum | Comments | History |
|---|---|---|
| #248 | Lyophilized, multi-donor, augmented with retinol, α- and γ-tocopherol | New: SRM 968c Lv 1 |
| #247 | Lyophilized, multi-donor, a 1:1 blend of the #248 and #249 pools. | New |
| #249 | Lyophilized, multi-donor, augmented with retinol, α- and γ-tocopherol | New: SRM 968c Lv 2 |
| #250 | Lyophilized, single-donor, augmented with retinol and retinyl palmitate | RR30:#192, RR32:#199, RR36:#218 |

# Individualized RR XLIV Report: FSV-BA

## α-Tocopherol, µg/mL

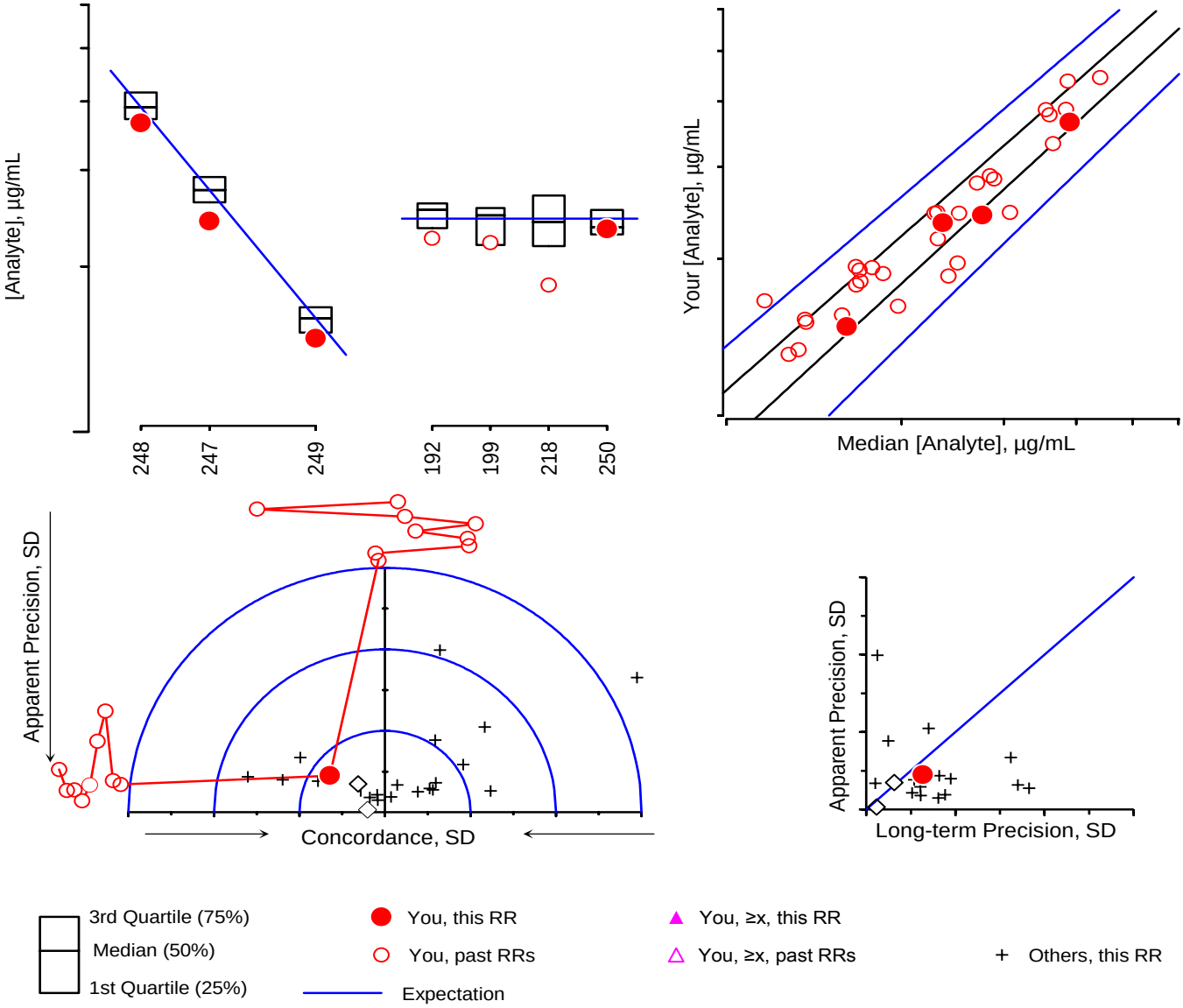



For the construction and interpretation of these plots, see the Final Report for "Round Robin" XL, August 1997

| Serum | Comments | History |
|---|---|---|
| #248 | Lyophilized, multi-donor, augmented with retinol, α- and γ-tocopherol | New: SRM 968c Lv 1 |
| #247 | Lyophilized, multi-donor, a 1:1 blend of the #248 and #249 pools. | New |
| #249 | Lyophilized, multi-donor, augmented with retinol, α- and γ-tocopherol | New: SRM 968c Lv 2 |
| #250 | Lyophilized, single-donor, augmented with retinol and retinyl palmitate | RR30:#192, RR32:#199, RR36:#218 |

# Individualized RR XLIV Report: FSV-BA

γ-Tocopherol, µg/mL

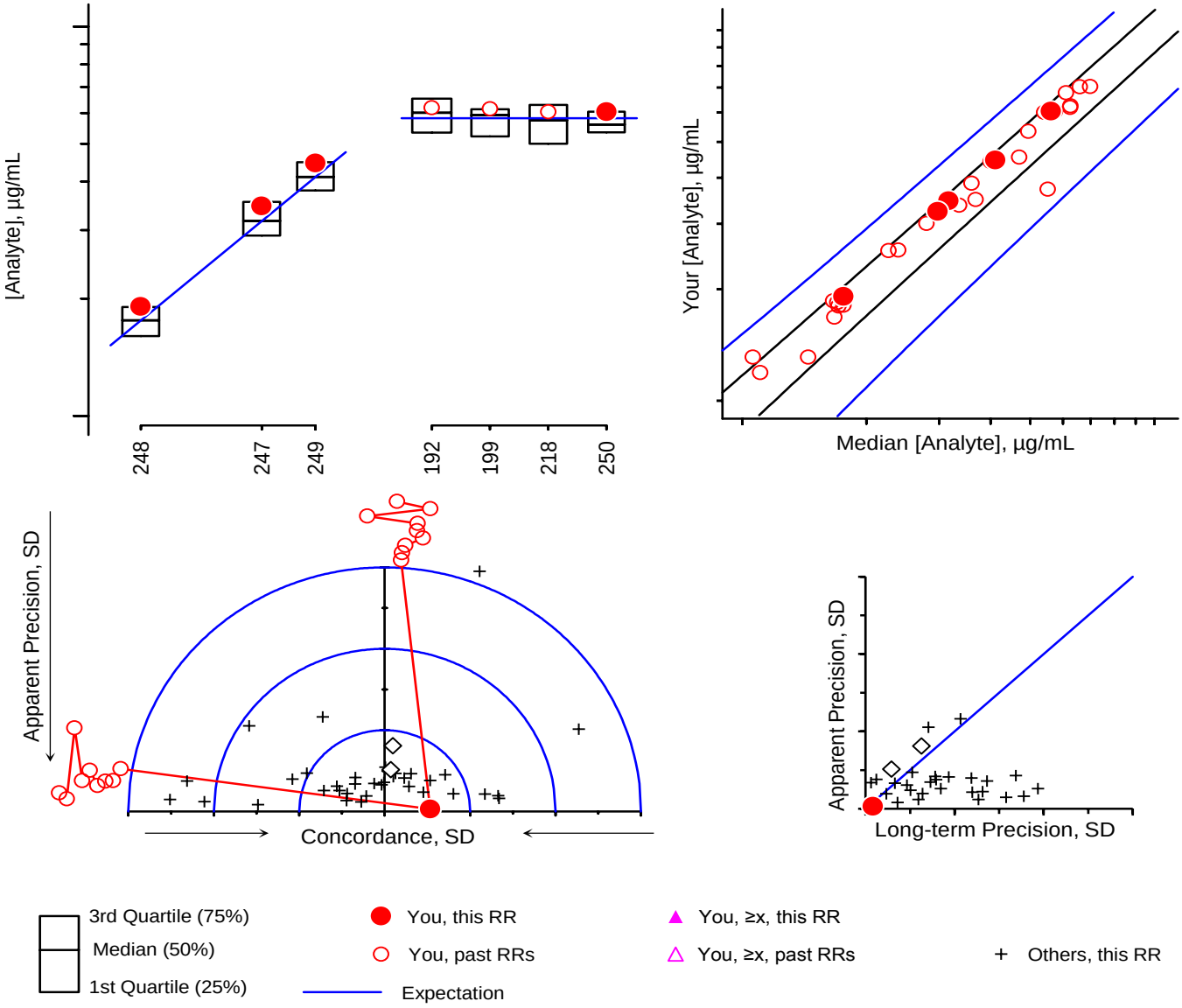



For the construction and interpretation of these plots, see the Final Report for "Round Robin" XL, August 1997

| Serum | Comments | History |
|---|---|---|
| #248 | Lyophilized, multi-donor, augmented with retinol, α- and γ-tocopherol | New: SRM 968c Lv 1 |
| #247 | Lyophilized, multi-donor, a 1:1 blend of the #248 and #249 pools. | New |
| #249 | Lyophilized, multi-donor, augmented with retinol, α- and γ-tocopherol | New: SRM 968c Lv 2 |
| #250 | Lyophilized, single-donor, augmented with retinol and retinyl palmitate | RR30:#192, RR32:#199, RR36:#218 |

# Individualized RR XLIV Report: FSV-BA

## Total β-Carotene, µg/mL

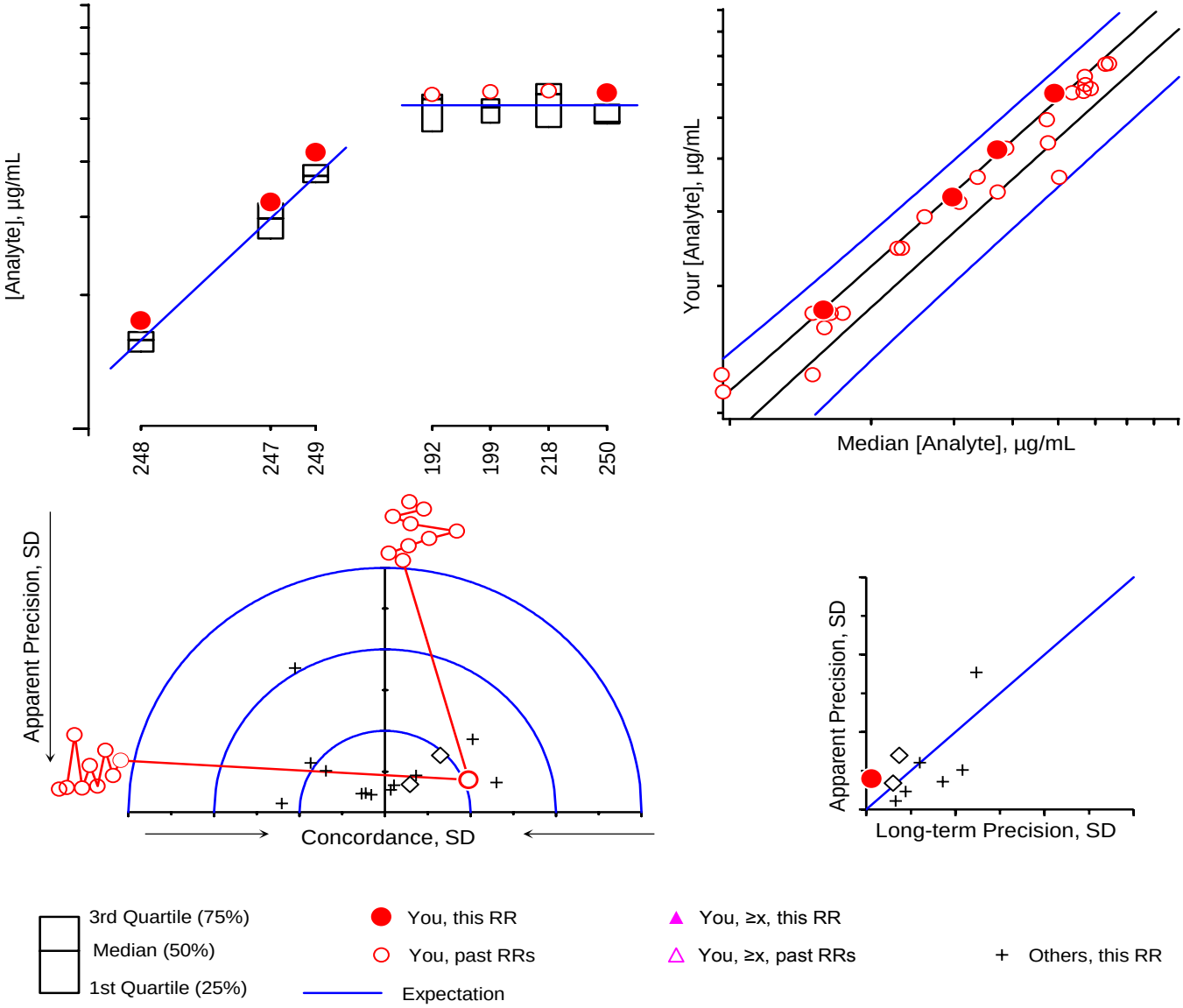



For the construction and interpretation of these plots, see the Final Report for "Round Robin" XL, August 1997

| Serum | Comments | History |
|---|---|---|
| #248 | Lyophilized, multi-donor, augmented with retinol, α- and γ-tocopherol | New: SRM 968c Lv 1 |
| #247 | Lyophilized, multi-donor, a 1:1 blend of the #248 and #249 pools. | New |
| #249 | Lyophilized, multi-donor, augmented with retinol, α- and γ-tocopherol | New: SRM 968c Lv 2 |
| #250 | Lyophilized, single-donor, augmented with retinol and retinyl palmitate | RR30:#192, RR32:#199, RR36:#218 |

# Individualized RR XLIV Report: FSV-BA

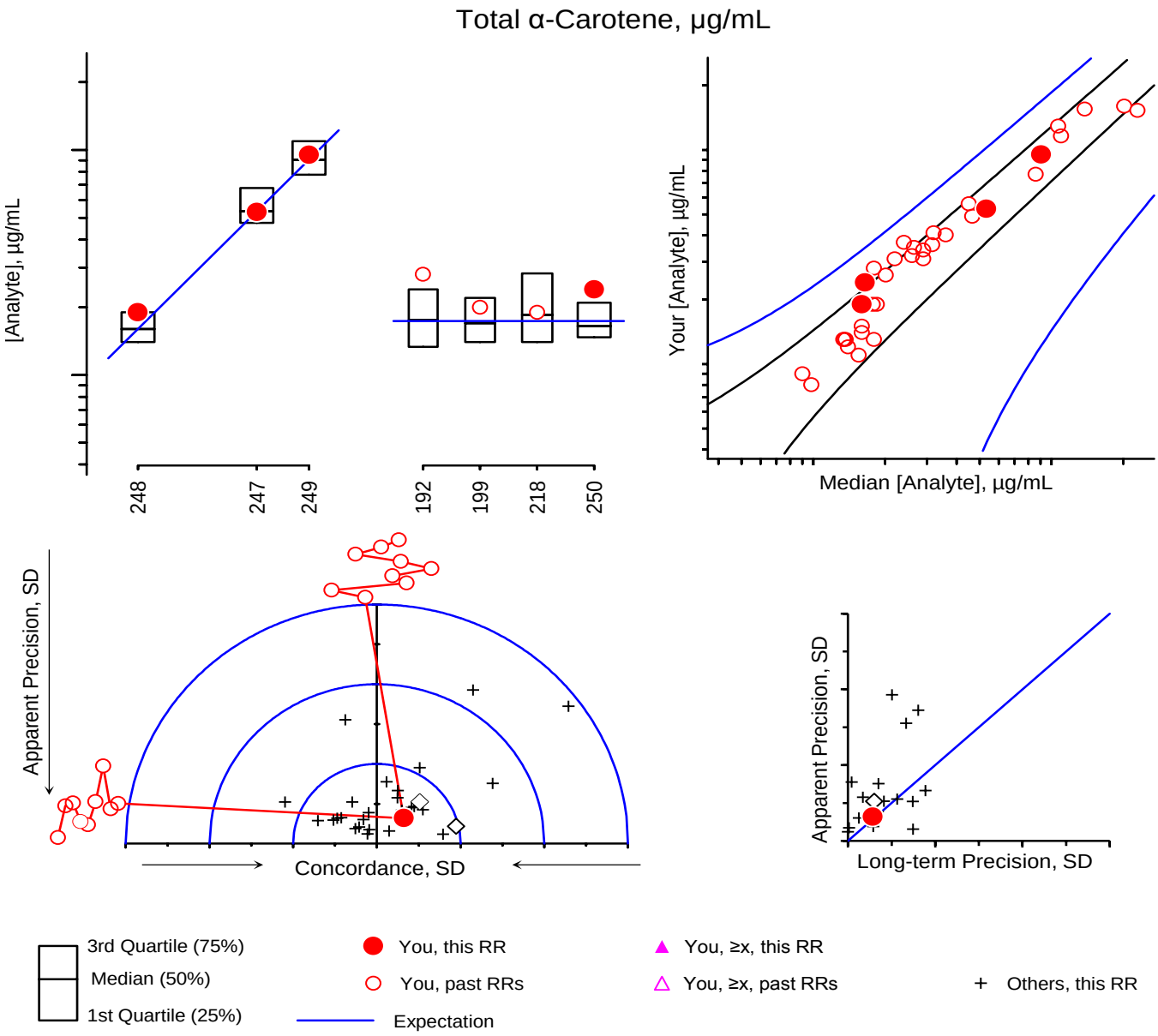
## trans-β-Carotene, µg/mL




For the construction and interpretation of these plots, see the Final Report for "Round Robin" XL, August 1997

| Serum | Comments | History |
|---|---|---|
| #248 | Lyophilized, multi-donor, augmented with retinol, α- and γ-tocopherol | New: SRM 968c Lv 1 |
| #247 | Lyophilized, multi-donor, a 1:1 blend of the #248 and #249 pools. | New |
| #249 | Lyophilized, multi-donor, augmented with retinol, α- and γ-tocopherol | New: SRM 968c Lv 2 |
| #250 | Lyophilized, single-donor, augmented with retinol and retinyl palmitate | RR30:#192, RR32:#199, RR36:#218 |

# Individualized RR XLIV Report: FSV-BA




For the construction and interpretation of these plots, see the Final Report for "Round Robin" XL, August 1997

| Serum | Comments | History |
|---|---|---|
| #248 | Lyophilized, multi-donor, augmented with retinol, α- and γ-tocopherol | New: SRM 968c Lv 1 |
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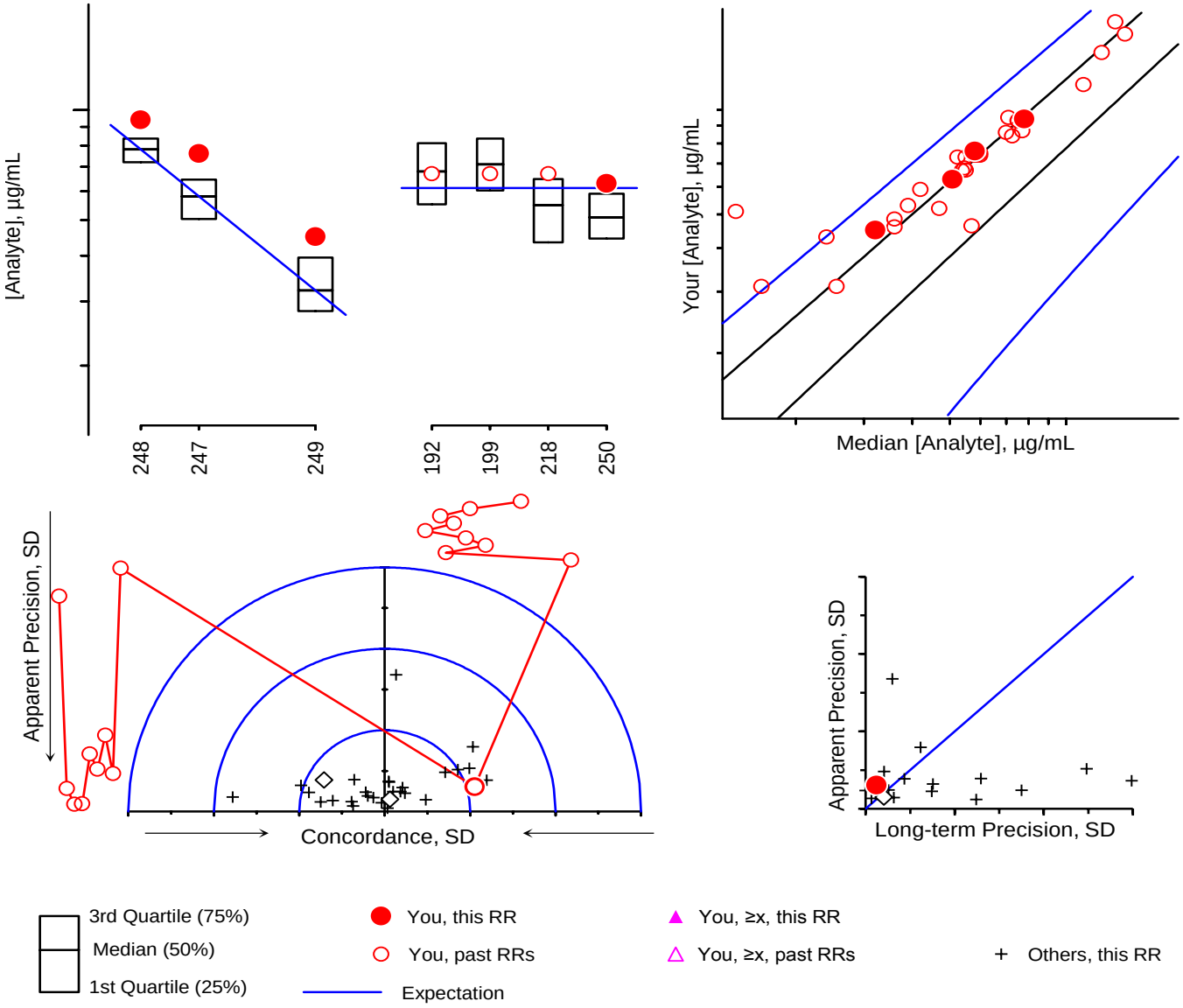
# Individualized RR XLIV Report: FSV-BA

## Total β-Cryptoxanthin, µg/mL

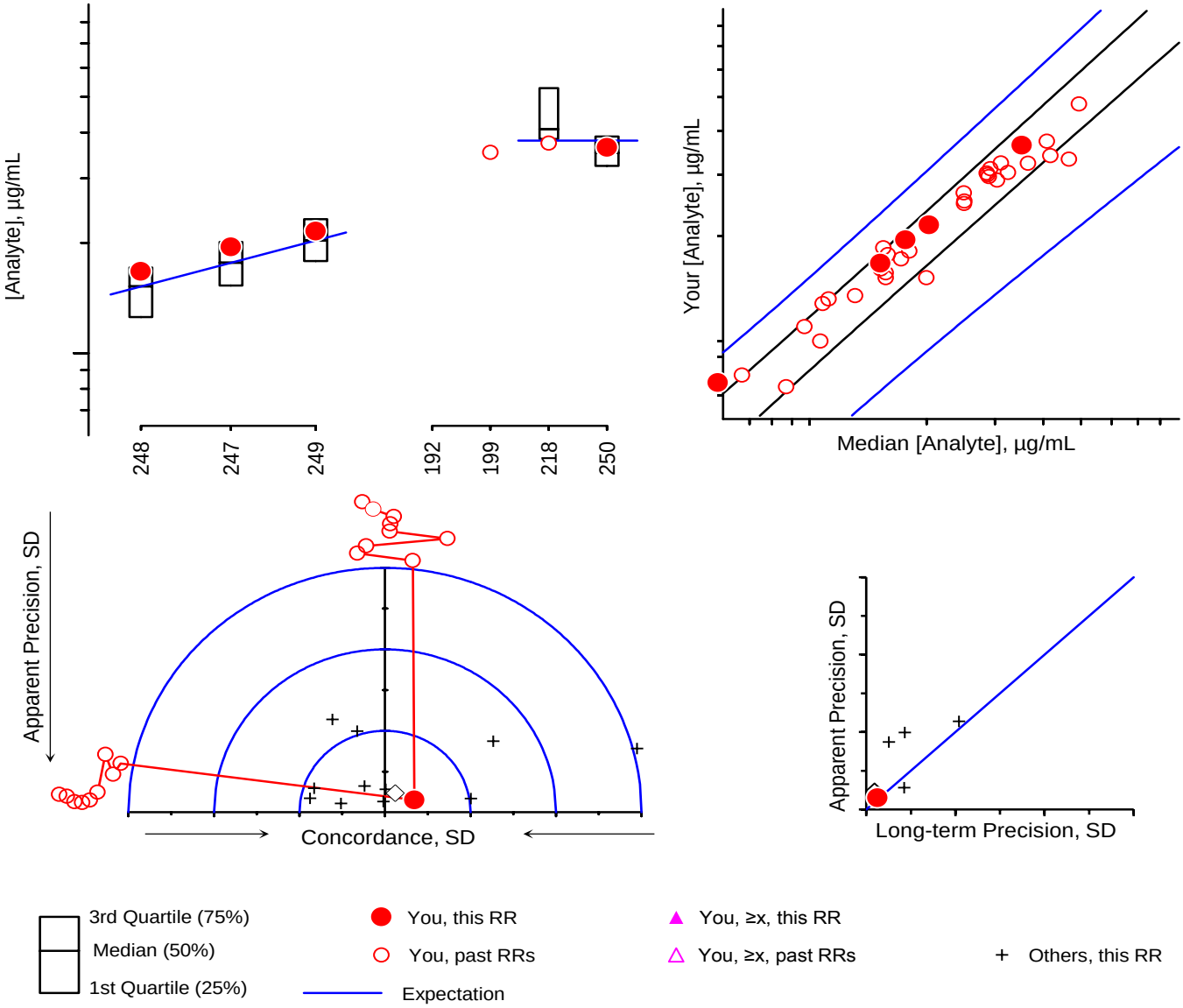



For the construction and interpretation of these plots, see the Final Report for "Round Robin" XL, August 1997

| Serum | Comments | History |
|---|---|---|
| #248 | Lyophilized, multi-donor, augmented with retinol, α- and γ-tocopherol | New: SRM 968c Lv 1 |
| #247 | Lyophilized, multi-donor, a 1:1 blend of the #248 and #249 pools. | New |
| #249 | Lyophilized, multi-donor, augmented with retinol, α- and γ-tocopherol | New: SRM 968c Lv 2 |
| #250 | Lyophilized, single-donor, augmented with retinol and retinyl palmitate | RR30:#192, RR32:#199, RR36:#218 |

# Individualized RR XLIV Report: FSV-BA

## trans-Lycopene, µg/mL




For the construction and interpretation of these plots, see the Final Report for "Round Robin" XL, August 1997

| Serum | Comments | History |
|---|---|---|
| #248 | Lyophilized, multi-donor, augmented with retinol, α- and γ-tocopherol | New: SRM 968c Lv 1 |
| #247 | Lyophilized, multi-donor, a 1:1 blend of the #248 and #249 pools. | New |
| #249 | Lyophilized, multi-donor, augmented with retinol, α- and γ-tocopherol | New: SRM 968c Lv 2 |
| #250 | Lyophilized, single-donor, augmented with retinol and retinyl palmitate | RR30:#192, RR32:#199, RR36:#218 |

## Appendix M. Shipping Package Inserts for RR11

The following three items were included in each package shipped to each RR11 participant:

- Cover letter
- Preparation of Stock Solution and Diluted Solution Datasheet
- Results Datasheet

The cover letter and datasheet were enclosed in a sealed waterproof bag along with the samples themselves.

September 10, 1998

«Name»
«Company»
«Address»

Dr. Margolis printed a separate cover letter for each participant. The words within the "«»" are parameters for a mail-merge macro routine.

Dear «Name»:

Thank you for agreeing to measure the ascorbic acid in the accompanying samples. Enclosed are two sets of samples, one set consisting of four ampules referred to as Test Samples (candidate SRM 970) and the second set consisting of a vial of solid ascorbic acid is the Control Sample.

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

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

2. Weigh out **180-220** mg of the solid ascorbic acid sample to 0.1 mg (if possible) and dissolve it in 100mLof5% MPA using a 100 mL volumetric flask. **Weigh the amount of MPA solution that was added.** This will be referred to as the Stock Solution.

3. Dilute the Stock Solution by **weighing** 0.5 mL of the stock solution into a 100 mL volumetric flask. Then add 5% MPA solution to 100 mL and **weigh the amount of MPA solution that was added.**

4. Record the ultraviolet spectrum of the diluted solution against 5% MPA solution as the blank using paired cuvettes.

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

6 Measure the concentration of the ascorbic acid in the **dilute solution** in duplicate along with the ampuled Test Samples.

The Test Samples are in sealed ampules and were prepared by adding equal volumes of spiked human serum to 10% metaphosphoric acid. We have checked them for stability and homogeneity and the ascorbic acid appears to be sufficiently stable.

Each ampule should contain between **20 and 120 µmol of ascorbic acid/L** of diluted serum and each ampule should be analyzed in duplicate by the method(s) used in your laboratory (preferably one measuring total ascorbic acid).

The Test Samples should be defrosted by warming at 20 °C for not more than 10 min otherwise some oxidation of ascorbic acid may occur.

A report form is attached and we would appreciate it if you would make your measurements and return the report to me by October 20, 1998. We also request that you send us a representative chromatogram from each lot and indicate whether you used the peak height or the peak area for calculating the concentration of ascorbic acid in your samples. Your results will be kept confidential. We will use these results in a study to demonstrate the comparative accuracy and the precision of the laboratories currently measuring ascorbic acid. However values will not be assigned to individual laboratories. If you wish to FAX your results to me, the Fax number is (301) 977- 0685. If you have any questions I can be reached at (301) 975-3137. Please let us know if the samples arrive defrosted and we will send you a duplicate set.

Thank you for your assistance.

Sincerely,

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

Attachment

# REPORT OF ANALYSIS

**NAME:**

**ADDRESS:**

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

**Method of Analysis:**

Please attach representative chromatograms.

Method used for calculating ascorbic acid concentration.

Peak height _____ Peak area _____

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

Date of Analysis:_____

## PREPARATION OF STOCK SOLUTION AND DILUTED SOLUTIONS

*STOCK SOLUTION*

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

*DILUTE SOLUTION*

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

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

# REPORT OF ANALYSIS

## RESULTS (µmol/L)

*CONTROL SAMPLE 1*

REPLICATE 1 _____µmol/L
REPLICATE 2 _____ µmol/L

*TEST SAMPLE HIGH VIAL #_____*

REPLICATE 1 _____µmol/L
REPLICATE 2 _____ µmol/L

*TEST SAMPLE HIGH VIAL #_____*

REPLICATE 1 _____µmol/L
REPLICATE 2 _____ µmol/L

*TEST SAMPLE LOW VIAL #_____*

REPLICATE 1 _____µmol/L
REPLICATE 2 _____ µmol/L

*TEST SAMPLE LOW VIAL #_____*

REPLICATE 1 _____µmol/L
REPLICATE 2 _____ µmol/L