NISTIR 7880-27

NIST Micronutrients Measurement Quality Assurance Program Winter, Spring, and Fall 1997 Comparability Studies

Results for Round Robin XXXIX, XL, and XLI Fat-Soluble Vitamins and Carotenoids in Human Serum and Round Robin 10 Ascorbic Acid in Human Serum

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July, 2013

U.S. Department of Commerce
Penny Pritzker, Secretary

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

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Abstract

The National Institute of Standards and Technology coordinates the Micronutrients Measurement Quality Assurance Program (MMQAP) for laboratories that measure fat- and water-soluble vitamins and carotenoids in human serum and plasma. This report describes the design of and results for the Winter, Spring and Fall 1997 MMQAP measurement comparability improvement studies: 1) Round Robin XXXIX Fat-Soluble Vitamins and Carotenoids in Human Serum, 2) Round Robin XL Fat-Soluble Vitamins and Carotenoids in Human Serum, 3) Round Robin XLI Fat-Soluble Vitamins and Carotenoids in Human Serum, and 4) Round Robin 10 Ascorbic Acid in Human Serum. The materials for Round Robin XXXIX were shipped to participants in January 1997; participants were requested to provide their measurement results by March 21, 1997. The materials for Round Robin XL were shipped to participants in April 1997; participants were requested to provide their measurement results by June 13, 1997. The materials for Round Robin XLI were shipped to participants in July 1997; participants were requested to provide their measurement results by September 19, 1997. The sample materials for Round Robin 10 were distributed in November 1996 with results due by January 15, 1997.

Keywords

Human Serum
Retinol, α-Tocopherol, γ-Tocopherol, Total and *Trans*-β-Carotene, SRM 968b
Ascorbic Acid

Table of Contents

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 XXXIX: Fat-Soluble Vitamins and Carotenoids in Human Serum

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin XXXIX comparability study (hereafter referred to as RR39) 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 January 1997. 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 RR39 consists of three documents:

- A cover letter and summary report for the current study that describes the samples, our analysis of the participants' results, and a detailed analysis of measurements made by NIST analysts. This cover letter and summary report 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 XL: Fat-Soluble Vitamins and Carotenoids in Human Serum

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin XL comparability study (hereafter referred to as RR40) 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 April 1997. 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 RR40 consists of three documents:

- A cover letter and summary report for the current study that describes the samples, our analysis of the participants' results, and a detailed analysis of measurements made by NIST analysts. This cover letter and summary report 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 XLI: Fat-Soluble Vitamins and Carotenoids in Human Serum

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin XLI comparability study (hereafter referred to as RR41) 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 July 1997. The communication materials included in the sample shipment are provided in Appendix I.

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 RR41 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 J.

- 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 K.

- 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 L.

Round Robin 10: Vitamin C in Human Serum

Participants in the MMQAP Vitamin C in Human Serum Round Robin 10 comparability study (hereafter referred to as RR10) received four frozen serum test samples and a solid ascorbic acid control material for analysis. These sample materials were shipped on dry ice to participants in September 1997. 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.

The final report delivered to all participants in RR10 consists of a cover letter and a series of Tables and Figures that summarize the results of the study. This report is reproduced as Appendix N.

While not distributed to the participants in RR10, Appendix O is a modified "All Lab Report" that lists the results for the test materials transformed into units of µmol/mL sample.

No "Individualized Report" was provided to the participants in RR10.

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 RR39

The following two items were included in each package shipped to an RR39 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-

March 4, 1997: (Most participants received samples in January, 1997)

Enclosed is the set of samples for the first quality assurance round robin exercise (Round Robin XXXIX) for FY97. 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 March 21, 1997. 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 18, 1997. If you joined the program after the scheduled distribution date, please submit your results as promptly as possible. You will be provided feedback within two weeks from receipt of your results.

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, 1850 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 XXXIX 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/NCI

Micronutrients Measurement Quality Assurance Program

Round Robin XXXIX Results from Laboratory #_____

	Serum				
Analyte	227	228	229	230	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					
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
March 15, 1997

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

Appendix B. Final Report for RR39

The following 15 pages are the final report as provided to all participants:

- Cover letter
- A "Report of Analysis" that:
  - 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
  - details the analysis of the NIST results

April 17, 1997

Dear Colleague:

Enclosed is the summary report of the results for Round Robin XXXIX (RR39). 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 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.

While the information in this report is much the same as in previous reports, the old "Report of (Meta)Analysis" and "Lies, Damned Lies, and Statistics" have been replaced with an "Analysis of Results" that more concisely describes the nature of the samples; the qualitative observations made by the participants and NIST analysts; actions taken in response to these observations; quantitative analysis for value and uncertainty assignments; and a summary of the overall laboratory performance drawn from the quantitative results.

The experimental design for this interlaboratory comparison exercise is summarized below. The four serum samples (Sera 227-230) distributed in RR39 address the following issues:

• **Serum and Measurement Stability.** Serum 227 is the high level component of SRM® 968b and was previously distributed as Sera 202 in RR32 and 210 in RR34. Reanalysis of such well-characterized materials documents both the continued stability of the material and of the "absolute accuracy" of the analyte level assignments. No significant changes in analyte stability were observed.

• **Measurement Precision and Concordance.** Serum 228 was previously distributed as the blind duplicate Sera 223 and 225 in RR38. Reanalysis of this material contributes to our on-going study of the sources of "long-term" within-laboratory measurement precision and among-laboratory measurement concordance. We will continue to explore within-laboratory measurement performance for discussion at our next quality assurance (QA) workshop.

• **Analyte Augmentation Techniques.** Sera 229 and 230 were prepared from the same "low normal" serum pool. We attempted to augment many of the commonly reported analytes to a "high" level in one of the two sera, with mixed success, ranging from nearly 100% of the intended levels for retinol, δ-, γ-, and α-tocopherol, lutein, and zeaxanthin to about 0% for β-carotene. We will continue to evaluate our augmentation techniques to determine why there was a complete loss of β-carotene and poor incorporation of α-carotene.

In this interlaboratory comparison exercise, the retinyl palmitate level was augmented in all four samples. While fairly high levels (about 0.05, 0.1, and 0.15 μg/mL) were achieved in the other three sera, measurement performance was acceptable only for the highest level (0.2 μg/mL) in Serum 227.

Serum 229 was augmented to a very high zeaxanthin level and Serum 230 was augmented to a very high lutein level. While most laboratories that separately report lutein and zeaxanthin did quite well, several laboratories misreported the high zeaxanthin in Serum 229 as lutein. If your laboratory had difficulty with this analyte pair, we recommend that you report a combined value for "lutein and zeaxanthin" rather than for the individual analytes.

Data for evaluating laboratory performance in RR39 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 or were rated "POOR" based on the criteria stated above, 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.

Samples for RR40 and the food Round Robin 6 (coordinated by Dr. Katherine Sharpless at 301/975-3121) will be shipped during the last week of April. Results for both exercises are due June 13; feedback to labs will be provided around July 25.

The Micronutrients Measurement QA Workshop will be held prior to the American Association for Clinical Chemistry meeting on **July 20, 1997** at the Best Western American Hotel in Atlanta, Georgia. You will be provided with further details about the workshop in about two weeks.

The QA homepage is now available! The web-site address for the homepage is: **http://www.cstl.nist.gov/nist839/839.02/qahome.htm**. Your comments and suggestions are welcomed.

Enclosed is a copy of our first publication on longitudinal QA analysis: **NIST/NCI Micronutrients Measurement Quality Assurance Program: Measurement Repeatabilities and Reproducibilities for Fat-Soluble Vitamin-Related Compounds in Human Sera,** Duewer, D.L., et al., *Anal Chem*, 69 (1997) 1406-1413.

The following are forthcoming publications:

- **Liquid Chromatographic Measurement of L-Ascorbic Acid and D-Ascorbic Acid in Biological Samples**, Margolis, S.A. and Schapira, R.M., *J. Chromatogr. B*, 690 (1997) 25-33.

- **Certification of Nutrients in Standard Reference Material 1846: Infant Formula**, Sharpless, K. E., et al., *J. AOAC*, in press.

- "**Vitamin A,**" In: Laboratory Medicine: A Scientific and Managerial Infobase, Sharpless, K.E., Brown Thomas, J., Turley, C.P., and Brewster, M.A., version 2.6, A. J. Pesce and L.A. Kaplan, editors, Pesce Kaplan Publishers, Cincinnati, OH, June 1997.

- "**Vitamin E,**" In: Laboratory Medicine: A Scientific and Managerial Infobase, Sharpless, K.E., Brown Thomas, J., Turley, C.P., and Brewster, M.A., version 2.6, A. J. Pesce and L.A. Kaplan, editors, Pesce Kaplan Publishers, Cincinnati, OH, June 1997.

Reprints of the above publications will be sent to you pending availability. If you have any questions, 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

Enclosures

cc: W. May
S. Wise

Analysis of N^2M^2QAP Round Robin XXXIX Results: Sera 227 to 230

Background: The following four samples were distributed in RR39

Serum 227: the "high level" component of Fat-Soluble Vitamins in Serum SRM® 968b. It was previously distributed as serum 202 in RR32 (9/94) and serum 210 in RR34 (6/95). This material was prepared from high-carotenoid serum pools and was augmented with retinol, retinyl palmitate (RP), and the tocopherols.

Serum 228: this was distributed as the blind duplicate sera 223 and 225 in RR38 (9/96). This material was prepared from a natural "normal" serum pool, lightly augmented with RP.

Serum 229: a new serum, prepared from a natural "low" serum pool. It was augmented with RP, γ- and δ-tocopherol, *trans*-lycopene, and lutein. (We *intended* to augment this serum with *trans*-β-carotene... and the notebook *says* we augmented it... but if we *actually* augmented it, it sure didn't go into solution!)

Serum 230: a new serum, prepared from the same natural "low" serum pool used for serum 229. It was augmented with retinol, RP, α- and δ-tocopherol, *trans*-α-carotene, *trans*-lycopene, β-cryptoxanthin, and zeaxanthin.

Qualitative Results: The following observations were noted in the RR39 reports:

Two participants reported a small unknown peak eluting in the retinol/δ-tocopherol region for both sera 229 and 230, with one participant suspecting the presence of *cis*-retinyl acetate. There is some evidence that these participants' results were degraded by this interferant. Analyst NIST3 confirms the presence of a small peak prior to the retinol peak that is compatible with the possible presence of a retinyl acetate isomer.

Action: We used a different source of RP for augmenting the RR40 samples. If this does not banish the interferant, we will need to more completely characterize all of the augmentation materials.

One participant noted the presence of a small amount of the freeze-dried material clinging to the vial stoppers of two samples. All results from this laboratory were within 2 SD of the median, with no correspondence between the magnitude of the measurement deviations and the suspect samples.

Action: None required. There is no evidence of contamination from the vial stoppers. (Analyst NIST3 notes that this is a fairly common occurance, and that you "just try to swoosh it down with the H_2O.")

One participant noted our use of clear vials while all the analytes are known to be photosensitive.

Action: We used 2.0 mL amber borosilicate vials for the RR40 samples instead of the old 3.5 mL flint glass vials. While the smaller volume of the new vials is of concern, only 2.0 mL and 5.0 mL amber vials are readily available. The smaller diameter and more regular shape of the 2.0 mL ambers does permit many more samples to be prepared in a single freeze-dryer batch. If the RR40 samples prove acceptable, we intend to use these amber vials for all future serum samples.

One participant noted with enthusiasm our success at achieving high lutein and zeaxanthin levels, and pointed to the need for higher α-carotene and β-cryptoxanthin levels.

Action: We're trying, we're trying...

Quantitative Results: Table 1, NIST Data and Value/Uncertainty Assignments, presents all NIST data, summary statistics for the NIST data, summary results for RR39, and the NIST assigned values and uncertainties. The assigned values and uncertainties for each analyte in each serum are summarized in Table 2, Summary of NIST Assigned Values/Uncertainties. The entries in Tables 1 and 2 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_{hetx} among-sample standard deviation, reflecting heterogeneity in preparing and reconstituting the serum samples

SD_{NISTx} $\sqrt{SD_{repx}^2 + SD_{hetx}^2}$, 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

n number of quantitative values for this analyte for this serum

Mean $(Mean_{NIST1} + Mean_{NIST3})/2$ or $Mean_{NIST3}$ for analytes that NIST1 did not report

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 or, for analytes that NIST1 did not report, to the interlaboratory Median (see below). 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} $100 \times SD_{NIST}/Mean$

Round Robin XXXIX 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)

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

This is the approximate probability that the current median is the same as it was in its initial distribution. 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 smaller than it was in its initial distribution. 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 $Maximum\left(0.05 \times NAV, \sqrt{SD_{NIST}^2 + SD_{labs}^2}\right)$, our best guess for the "true" interlaboratory standard deviation characterizing measurement, sample heterogeneity, interanalyst, and interlaboratory sources of variation. When SD_{labs} could not be determined, NAU is estimated as $Maximum\left(0.10 \times NAV, \sqrt{2\ SD_{NIST}^2}\right)$.

CV $100 \times NAU / NAV$

xCV $100 \times \sqrt{L_{qc}^2 + \left(\beta_0 \times NAV^{\beta_1}\right)^2} / NAV$, the CV we expect for a given NAV. L_{qc}, β_0, and β_1 are analyte-specific constants, determined from empirical analysis of the historical relationship between NAV and NAU for the specific analyte.

Measurement Performance Summary: The following is based on results presented in Table 1 of this report and the "Individualized Report" page 7 and 8 summary graphs:

Serum 227, high-level SRM 968b, stability: There is no significant difference in the level or measurement variability of any analyte among the three analyses of this material (sera 202, 210, and 227).

Action: Thankfully, none required.

Serum 228 stability: There is some evidence of increased measurement variability for total β-carotene, α-carotene, and zeaxanthin in the blind-duplicate material from RR38 (sera 223, 225, and 228). Measurement variability for these analytes is significantly higher when

compared to results from either serum 223 or serum 225 alone; however, the increase largely disappears when the results for the sera 223 and 225 blind duplicates are combined.

Action: It is quite plausible that augmented serum is less stable than the "native" sera we have previously studied. We must give serious thought as to the best use of our limited remaining stock of this material, but some extended analysis will be done! We will include the data from this trio of samples in any future extended data analysis of blind replicate samples.

Sera 229 and 230 homogeneity: None of the SD_{het} values are a much larger than expected component of SD_{NIST}. Thus, these sera are "homogenous" (rather, no analyte in either serum 229 or 230 is unusually heterogeneous). Further, the observed CV is no larger than expected (xCV) for retinol, RP, γ-tocopherol, total β-carotene, β-cryptoxanthin, and total lycopene. However, one or both sera have larger than expected CVs for α-tocopherol, *trans*-β-carotene, and total α-carotene. This may reflect interference from the (putative) retinyl acetate isomer (see the "qualitative" section) noted in these sera, some interaction among the tocopherols, or incorrect xCV parameters (see: Anal Chem 1997:69, 1406-1413).

Action: We will revisit the xCV parameters before July, 1997, paying particular attention to potential interactions among the tocopherols. We should have sufficient experience to obtain xCV parameters for a few more analytes, as well.

Retinyl palmitate: The CV for serum 227, NAV = 0.23 µg/mL, was a much better than expected 11%. The CVs for the next-highest level, serum 230 (NAV = 0.17 µg/mL) is expectedly high at 31%.

Action: RP remains analytically challenging. We need >0.25 µg/mL levels in several different samples before we can reliably identify the limit of quantitative comparison, L_{qc}, for this analyte. We'll try to find/make them!

"Lutein," "zeaxanthin," and "lutein&zeaxanthin": The CV_{labs} for lutein in serum 229 is very large (200%!). This results from several laboratories mis-identifying the large zeaxanthin component of this material. The zeaxanthin CVs are fairly small in both materials (~17%), and the variability of the combined "lutein&zeaxanthin" results appear quite acceptably small. The values for this combination "analyte" are, however, a composite of actual combined-measurements and simple additions of results for the individual analyte.

Action: While individual laboratories perform well, interlaboratory comparisons of lutein or zeaxanthin are not yet reliable. The combination of lutein and zeaxanthin levels is more analytically meaningful.

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Table 1
NIST Data and Value/Uncertainty Assignments

Retinol

	NIST1				NIST3			
	227	228	229	230	227	228	229	230
A:1	0.857	0.515	0.396	0.644	0.913	0.484	0.359	0.632
A:2	0.907	0.497	0.394	0.665	0.862	0.481	0.358	0.632
B:1	0.910	0.506	0.381	0.695	0.836	0.465	0.353	0.628
B:2	0.866	0.512	0.393	0.625	0.877	0.465	0.353	0.610
C:1	0.902	0.479	0.383	0.668	0.848	0.488	0.357	0.622
C:2	0.894	0.459	0.384	0.647	0.886	0.474	0.345	0.644
n_x	6	6	6	6	6	6	6	6
$Mean_x$	0.889	0.494	0.388	0.657	0.870	0.476	0.354	0.628
SD_x	0.023	0.022	0.006	0.024	0.028	0.010	0.005	0.011
SD_{repx}	0.028	0.011	0.005	0.031	0.031	0.006	0.005	0.011
SD_{betx}	0.008	0.022	0.006	0.003	0.016	0.010	0.004	0.008
SD_{NISTx}	0.029	0.025	0.008	0.031	0.035	0.011	0.006	0.014
CV_{NISTx}	3.2	5.0	1.9	4.7	4.0	2.4	1.7	2.2

Retinyl Palmitate

	NIST1				NIST3			
	227	228	229	230	227	228	229	230
A:1	0.246				0.160	0.035	0.078	0.149
A:2	0.261				0.178	0.034	0.092	0.157
B:1	0.258				0.217	0.063	0.078	0.147
B:2	0.225				0.180	0.050	0.085	0.144
C:1	0.245				0.150	0.041	0.060	0.172
C:2	0.251				0.204		0.072	0.145
n_x	6	0	0	0	6	5	6	6
$Mean_x$	0.248				0.181	0.045	0.078	0.152
SD_x	0.013				0.025	0.012	0.011	0.011
SD_{repx}	0.015				0.028	0.005	0.008	0.011
SD_{betx}	0.006				0.015	0.012	0.010	0.007
SD_{NISTx}	0.016				0.032	0.013	0.013	0.013
CV_{NISTx}	6.4				17	28	17	8.6

	NIST			
n	12	12	12	12
Mean	0.880	0.485	0.371	0.643
SD_{rep}	0.030	0.008	0.003	0.023
SD_{bet}	0.011	0.016	0.006	0.007
SD_{anl}	0.010	0.010	0.010	0.010
SD_{NIST}	0.033	0.021	0.012	0.026
CV_{NIST}	3.8	4.3	3.2	4.0

NIST3=a+b*NIST1

a: -0.036 ±0.017
b: 1.022 ±0.027
R^2: 0.999

	NIST			
	12	5	6	6
	0.215	0.045	0.078	0.152
	0.019	0.008	0.007	0.004
	0.015	0.012	0.010	0.007
	0.003	0.003	0.003	0.003
	0.024	0.014	0.013	0.008
	11	32	16	5.4

NIST3=a+b*Median

a: -0.028 ±0.004
b: 0.869 ±0.027
R^2: 0.998

RR	XXXIII	XXXVIII
Serum	202	223
n_p	40	46
$Median_p$	0.879	0.496
eSD_p	0.080	0.038

<—— Previous Results ——>

XXXIII	XXXVIII
202	223
9	15
0.263	0.084
0.028	0.047

RRXXXIX				
	227	228	229	230
n_n	47	48	48	48
$Median_n$	0.859	0.491	0.383	0.687
eSD_n	0.056	0.032	0.036	0.045
P(n=p)	0.95	0.98		
P(n<p)	0.99	0.86		
SD_{labs}	0.045	0.025	0.033	0.037
CV_{labs}	5.2	5.1	8.7	5.4

<— Current Results —>

RRXXXIX			
227	228	229	230
12	12	12	12
0.241	0.086	0.119	0.192
0.019	0.039	0.028	0.054
0.68	0.99		
0.90	0.75		
0	0.036	0.025	0.053
0	42	21	28

NAV	0.869	0.488	0.377	0.665
NAU	0.056	0.032	0.036	0.045
CV	6.4	6.7	9.4	6.8
xCV	8.9	8.8	9.1	8.7

<—— Assignments ——>

0.228	0.065	0.098	0.172
0.024	0.039	0.028	0.054
11	59	28	31
23	42	32	24

[39_dat]NISTtxt

Table 1
NIST Data and Value/Uncertainty Assignments

	α-Tocopherol								γ-Tocopherol							
	NIST1				NIST3				NIST1				NIST3			
	227	228	229	230	227	228	229	230	227	228	229	230	227	228	229	230
A:1	17.4	8.17	7.31	9.9	16.6	7.08	6.70	9.6	3.57	1.69	4.29	1.30	3.30	1.53	4.11	1.02
A:2	17.4	7.87	7.15	10.4	16.7	7.36	6.41	10.5	3.54	1.73	4.30	1.28	3.38	1.55	3.93	1.12
B:1	17.5	8.57	7.15	11.0	17.4	7.29	6.20	10.1	3.61	1.57	4.26	1.34	3.41	1.59	3.98	1.13
B:2	17.6	8.12	7.11	10.5	17.7	7.20	6.62	9.6	3.34	1.73	4.37	1.28	3.43	1.52	3.91	1.11
C:1	17.8	8.44	7.55	10.6	18.0	7.04	5.83	10.2	3.76	1.65	4.68	1.35	3.50	1.53	3.79	1.18
C:2	17.5	7.96	7.02	10.6	17.3	7.19	6.37	10.5	3.75	1.74	4.12	1.34	3.53	1.59	3.81	1.04
n_x	6	6	6	6	6	6	6	6	6	6	6	6	6	6	6	6
$Mean_x$	17.5	8.19	7.21	10.5	17.3	7.19	6.35	10.1	3.59	1.68	4.34	1.32	3.43	1.55	3.92	1.10
SD_x	0.1	0.27	0.19	0.3	0.5	0.12	0.32	0.4	0.15	0.07	0.19	0.03	0.08	0.03	0.12	0.06
SD_{repx}	0.1	0.30	0.23	0.3	0.3	0.14	0.31	0.4	0.11	0.08	0.23	0.03	0.04	0.04	0.08	0.07
SD_{betx}	0.1	0.16	0.08	0.3	0.6	0.07	0.23	0.2	0.14	0.03	0.05	0.03	0.08	0.01	0.11	0.03
SD_{NISTx}	0.2	0.34	0.24	0.4	0.6	0.15	0.38	0.5	0.18	0.08	0.24	0.04	0.09	0.04	0.14	0.08
CV_{NISTx}	1.0	4.1	3.4	3.9	3.6	2.1	6.1	4.6	5.0	4.9	5.5	3.0	2.7	2.6	3.5	6.9

	NIST				NIST3=a+b*NIST1	NIST				NIST3=a+b*NIST1
n	12	12	12	12		12	12	12	12	
Mean	17.4	7.69	6.78	10.3	a: 0	3.51	1.62	4.13	1.21	a: 0
SD_{rep}	0.1	0.23	0.25	0.4	b: 0.96 ±0.03	0.08	0.06	0.17	0.03	b: 0.92 ±0.02
SD_{bet}	0.5	0.36	0.33	0.2	R^2: 0.974	0.11	0.02	0.09	0.07	R^2: 0.988
SD_{anl}	0.7	0.69	0.69	0.7		0.10	0.10	0.10	0.10	
SD_{NIST}	0.8	0.81	0.81	0.8		0.17	0.12	0.22	0.12	
CV_{NIST}	4.8	11	12	7.9		4.8	7.1	5.3	10	

RR	XXXII	XXXVIII		XXXII	XXXVIII
Serum	202	223	<—— Previous Results ——>	202	223
n_p	42	44		18	22
$Median_p$	17.8	7.72		3.76	1.68
eSD_p	1.5	0.46		0.39	0.13

	RRXXXIX					RRXXXIX			
	227	228	229	230	<— Current Results —>	227	228	229	230
n_n	45	46	46	46		25	25	25	25
$Median_n$	17.0	7.54	7.11	11.4		3.86	1.78	4.36	1.36
eSD_n	1.4	0.55	0.74	1.2		0.25	0.09	0.30	0.14
P(n=p)	0.90	0.94				0.93	0.78		
P(n<p)	0.61	0.14				0.98	0.96		
SD_{labs}	1.1	0	0	0.8		0.19	0	0.21	0.07
CV_{labs}	6.5	0	0	7.3		4.9	0	4.7	5.3
NAV	17.2	7.61	6.95	10.8	<—— Assignments ——>	3.68	1.70	4.24	1.28
NAU	1.4	0.81	0.81	1.2		0.25	0.12	0.30	0.14
CV	8.1	11	12	11		6.9	6.8	7.1	11
xCV	7.4	7.7	8.0	7.2		7.5	9.1	7.4	10

[39_dat]NISTtxt

Table 1
NIST Data and Value/Uncertainty Assignments

δ-Tocopherol

	NIST1				NIST3			
	227	228	229	230	227	228	229	230
A:1							0.6	0.3
A:2							0.5	0.2
B:1							0.5	0.2
B:2							0.4	0.2
C:1							0.5	0.2
C:2							0.4	0.2
n_x	0	0	0	0	0	0	6	6
$Mean_x$							0.5	0.2
SD_x							0.1	0.0
SD_{repx}							0.1	0.0
SD_{betx}							0.1	0.0
SD_{NISTx}							0.1	0.0
CV_{NISTx}							22	18

Total β-Carotene

	NIST1				NIST3			
	227	228	229	230	227	228	229	230
A:1	1.22	0.179	0.112	0.110	1.14	0.143	0.076	0.079
A:2	1.31	0.181	0.114	0.105	1.19	0.146	0.087	0.079
B:1	1.27	0.182	0.114	0.114	1.17	0.145	0.078	0.074
B:2	1.34	0.178	0.113		1.22	0.143	0.082	0.072
C:1	1.30	0.171	0.111	0.110	1.22	0.144	0.080	0.073
C:2	1.18	0.180	0.109	0.116	1.21	0.142	0.083	0.083
n_x	6	6	6	5	6	6	6	6
$Mean_x$	1.27	0.179	0.112	0.111	1.19	0.144	0.081	0.077
SD_x	0.06	0.004	0.002	0.004	0.03	0.002	0.004	0.004
SD_{repx}	0.07	0.004	0.001	0.003	0.03	0.002	0.005	0.004
SD_{betx}	0.03	0.003	0.002	0.004	0.02	0.001	0.001	0.003
SD_{NISTx}	0.08	0.005	0.002	0.005	0.03	0.002	0.005	0.005
CV_{NISTx}	5.9	2.7	1.9	4.3	2.9	1.5	6.2	6.8

	NIST			
n	0	0	6	6
Mean			0.5	0.2
SD_{rep}			0.1	0.0
SD_{bet}			0.1	0.0
SD_{ani}				
SD_{NIST}				
CV_{NIST}				

	NIST			
n	12	12	12	11
Mean	1.23	0.161	0.097	0.094
SD_{rep}	0.05	0.003	0.004	0.002
SD_{bet}	0.03	0.002	0.001	0.004
SD_{ani}	0.00	0.001	0.001	0.001
SD_{NIST}	0.06	0.004	0.004	0.004
CV_{NIST}	4.7	2.3	4.1	4.6

NIST3=a+b*NIST1

a: -0.027 ±0.001
b: 0.961 ±0.001
R^2: 1.000

RR	XXXII	XXXVIII
Serum	202	223
n_p	1	5
$Median_p$	1.0	0.17
eSD_p		0.03

<——— Previous Results ———>

XXXII	XXXVIII
202	223
32	31
1.23	0.166
0.20	0.017

RRXXXIX	227	228	229	230
n_n	4	4	4	4
$Median_n$	0.2	0.15	0.5	0.4
eSD_n	0.0	0.03	0.1	0.1
P(n=p)		0.63		
P(n<p)		0.56		
SD_{labs}				
CV_{labs}				

<— Current Results —>

RRXXXIX	227	228	229	230
	33	33	32	33
	1.24	0.177	0.111	0.106
	0.13	0.027	0.014	0.017
	0.99	0.90		
	0.99	0.01*		
	0.12	0.026	0.014	0.016
	9.6	15	12	15

NAV			0.5	0.3
NAU				
CV				
xCV				

<—— Assignments ——>

1.24	0.169	0.104	0.100
0.13	0.027	0.014	0.017
11	16	14	17
12	19	21	22

[39_dat]NISTtxt

Table 1
NIST Data and Value/Uncertainty Assignments

trans-β-Carotene

	NIST1				NIST3			
	227	228	229	230	227	228	229	230
A:1	1.076	0.167	0.109	0.099	1.063	0.143	0.076	0.079
A:2	1.174	0.173	0.104	0.090	1.108	0.146	0.087	0.079
B:1	1.127	0.166	0.103	0.105	1.101	0.145	0.078	0.074
B:2	1.109	0.161	0.104		1.149	0.143	0.082	0.072
C:1	1.083	0.162	0.102	0.106	1.105	0.144	0.080	0.073
C:2	1.075	0.169	0.100	0.115	1.101	0.142	0.083	0.083
n_x	6	6	6	5	6	6	6	6
$Mean_x$	1.107	0.166	0.104	0.103	1.104	0.144	0.081	0.077
SD_x	0.039	0.004	0.003	0.009	0.027	0.002	0.004	0.004
SD_{repx}	0.041	0.004	0.002	0.005	0.027	0.002	0.005	0.004
SD_{hetx}	0.025	0.003	0.003	0.008	0.020	0.001	0.001	0.003
SD_{NISTx}	0.048	0.005	0.003	0.010	0.033	0.002	0.005	0.005
CV_{NISTx}	4.3	3.3	3.3	9.3	3.0	1.5	6.2	6.8

Total α-Carotene

	NIST1				NIST3			
	227	228	229	230	227	228	229	230
A:1	0.054			0.104	0.057	0.026	0.017	0.101
A:2	0.052			0.086	0.060	0.024	0.020	0.097
B:1	0.047			0.095	0.054	0.025	0.017	0.101
B:2	0.042				0.061	0.025	0.017	0.102
C:1	0.045			0.084	0.055	0.023	0.016	0.105
C:2	0.049			0.111	0.057	0.022	0.014	0.117
n_x	6	0	0	5	6	6	6	6
$Mean_x$	0.048			0.096	0.057	0.024	0.017	0.104
SD_x	0.005			0.011	0.003	0.001	0.002	0.007
SD_{repx}	0.003			0.013	0.003	0.001	0.001	0.005
SD_{hetx}	0.004			0.001	0.001	0.001	0.002	0.006
SD_{NISTx}	0.005			0.013	0.004	0.002	0.002	0.008
CV_{NISTx}	11			14	6.3	6.3	12	7.7

	NIST			
n	12	12	12	11
Mean	1.106	0.155	0.092	0.090
SD_{rep}	0.037	0.003	0.004	0.004
SD_{het}	0.020	0.002	0.002	0.006
SD_{anl}	0.001	0.001	0.001	0.001
SD_{NIST}	0.043	0.004	0.004	0.007
CV_{NIST}	3.8	2.7	4.7	7.9

NIST3=a+b*NIST1

a: -0.025 ±0.001
b: 1.020 ±0.001
R^2: 1.000

	NIST			
	12	6	6	11
	0.053	0.024	0.017	0.100
	0.004	0.001	0.001	0.009
	0.001	0.001	0.002	0.006
	0.003	0.003	0.003	0.003
	0.005	0.004	0.004	0.012
	9.6	15	22	12

NIST3=a+b*Median

a: -0.018 ±0.006
b: 1.664 ±0.176
R^2: 0.978

RR	XXXII	XXXVIII
Serum	202	223
n_p	8	12
$Median_p$	1.108	0.161
eSD_p	0.070	0.020

← Previous Results →

XXXII	XXXVIII
202	223
21	25
0.039	0.026
0.016	0.009

RRXXXIX				
	227	228	229	230
n_n	11	11	11	11
$Median_n$	1.164	0.150	0.097	0.096
eSD_n	0.064	0.019	0.010	0.020
P(n=p)	0.70	0.82		
P(n<p)	0.63	0.61		
SD_{labs}	0.048	0.018	0.009	0.019
CV_{labs}	4.1	12	8.8	20

← Current Results →

RRXXXIX			
227	228	229	230
27	27	27	28
0.045	0.027	0.020	0.141
0.014	0.015	0.012	0.033
0.91	0.98		
0.76	0.01*		
0.013	0.014	0.011	0.031
28	53	55	22

NAV	1.135	0.153	0.095	0.093
NAU	0.064	0.019	0.010	0.020
CV	5.6	12	10	22
xCV	9.0	9.8	11	11

← Assignments →

0.049	0.026	0.018	0.120
0.014	0.015	0.012	0.033
28	58	63	28
28	30	33	26

[39_dat]NISTtxt

Table 1
NIST Data and Value/Uncertainty Assignments

trans-α-Carotene

	NIST1				NIST3			
	227	228	229	230	227	228	229	230
A:1					0.03	0.03	0.017	0.10
A:2					0.04	0.02	0.020	0.10
B:1					0.03	0.02	0.017	0.10
B:2					0.04	0.02	0.017	0.10
C:1					0.03	0.02	0.016	0.11
C:2					0.03	0.02	0.014	0.12
n_x	0	0	0	0	6	6	6	6
$Mean_x$					0.03	0.02	0.017	0.10
SD_x					0.00	0.00	0.002	0.01
SD_{repx}					0.00	0.00	0.001	0.00
SD_{betx}					0.00	0.00	0.002	0.01
SD_{NISTx}					0.00	0.00	0.002	0.01
CV_{NISTx}					10	6.3	12	7.7

Total Lycopene

	NIST1				NIST3			
	227	228	229	230	227	228	229	230
A:1					0.298	0.454	0.56	0.360
A:2					0.335	0.443	0.57	0.389
B:1					0.340	0.448	0.57	0.358
B:2					0.340	0.488	0.58	0.358
C:1					0.337	0.474	0.57	0.345
C:2					0.300	0.454	0.52	0.400
n_x	0	0	0	0	6	6	6	6
$Mean_x$					0.325	0.460	0.56	0.368
SD_x					0.020	0.017	0.02	0.021
SD_{repx}					0.021	0.019	0.02	0.025
SD_{betx}					0.013	0.010	0.01	0.009
SD_{NISTx}					0.025	0.021	0.03	0.027
CV_{NISTx}					7.7	4.7	4.5	7.3

trans-α-Carotene

	NIST			
n	6	6	6	6
Mean	0.03	0.02	0.017	0.10
SD_{rep}	0.00	0.00	0.001	0.00
SD_{bet}	0.00	0.00	0.002	0.01
SD_{ani}				
SD_{NIST}				
CV_{NIST}				

Total Lycopene

NIST			
6	6	6	6
0.325	0.460	0.56	0.368
0.015	0.024	0.01	0.012
0.013	0.010	0.01	0.009
0.010	0.010	0.01	0.010
0.022	0.028	0.02	0.018
6.8	6.0	3.2	4.9

NIST3=a+b*Median

a: 0.044 ±0.025
b: 0.869 ±0.051
R^2: 0.993

RR	XXXII	XXXVIII
Serum	202	223
n_p	0	0
$Median_p$		
eSD_p		

<—— Previous Results ——>

XXXII	XXXVIII
202	223
22	27
0.314	0.463
0.068	0.084

trans-α-Carotene

RRXXXIX				
	227	228	229	230
n_n	0	0	0	0
$Median_n$				
eSD_n				
P(n=p)				
P(n<p)				
SD_{labs}				
CV_{labs}				

<— Current Results —>

Total Lycopene

RRXXXIX			
227	228	229	230
27	27	26	27
0.328	0.470	0.60	0.421
0.060	0.108	0.15	0.097
0.95	0.98		
0.74	0.10		
0.056	0.105	0.15	0.095
17	22	24	23

trans-α-Carotene

NAV	0.03	0.02	0.017	0.10
NAU				
CV				
xCV				

<—— Assignments ——>

Total Lycopene

0.326	0.465	0.58	0.395
0.060	0.108	0.15	0.097
18	23	25	25
25	23	23	24

[39_dat]NISTtxt

Table 1
NIST Data and Value/Uncertainty Assignments

trans-Lycopene

	NIST1				NIST3			
	227	228	229	230	227	228	229	230
A:1					0.138	0.183	0.333	0.179
A:2					0.151	0.189	0.349	0.188
B:1					0.155	0.191	0.352	0.179
B:2					0.153	0.197	0.356	0.175
C:1					0.151	0.191	0.364	0.179
C:2					0.151	0.188	0.328	0.199
n_x	0	0	0	0	6	6	6	6
$Mean_x$					0.150	0.190	0.347	0.183
SD_x					0.006	0.005	0.014	0.009
SD_{repx}					0.006	0.004	0.016	0.009
SD_{hetx}					0.005	0.004	0.007	0.006
SD_{NISTx}					0.007	0.005	0.018	0.011
CV_{NISTx}					4.9	2.9	5.1	6.1

	NIST			
n	6	6	6	6
Mean	0.150	0.190	0.347	0.183
SD_{rep}	0.006	0.004	0.007	0.005
SD_{het}	0.005	0.004	0.007	0.006
SD_{ani}	0.003	0.003	0.003	0.003
SD_{NIST}	0.008	0.006	0.010	0.008
CV_{NIST}	5.3	3.4	2.9	4.5

NIST3=a+b*Median

a: 0.034 ±0.004
b: 0.632 ±0.012
R^2: 0.999

RR	XXXII	XXXVIII
Serum	202	223
n_p	1	8
$Median_p$	0.178	0.251
eSD_p		0.042

← Previous Results →

XXXII	XXXVIII
202	223
16	19
0.049	0.055
0.016	0.014

RRXXXIX

	227	228	229	230
n_n	8	8	8	8
$Median_n$	0.181	0.250	0.494	0.290
eSD_n	0.024	0.029	0.038	0.044
P(n=p)		0.99		
P(n<p)		0.83		
SD_{labs}	0.023	0.028	0.037	0.043
CV_{labs}	13	11	7.4	15

← Current Results →

RRXXXIX

227	228	229	230
24	24	24	24
0.037	0.055	0.056	0.076
0.012	0.014	0.015	0.019
0.79	1.00		
0.90	0.46		
0.012	0.014	0.014	0.019
31	25	26	25

NAV	0.165	0.220	0.420	0.236
NAU	0.024	0.029	0.038	0.044
CV	15	13	9.0	19
xCV				

← Assignments →

0.034	0.047	0.047	0.065
0.012	0.014	0.015	0.019
34	31	31	29
30	28	28	26

β-Cryptoxanthin

	NIST1				NIST3			
	227	228	229	230	227	228	229	230
A:1					0.032	0.040	0.042	0.054
A:2					0.030	0.035	0.041	0.054
B:1					0.031	0.044	0.036	0.058
B:2					0.034	0.041	0.036	0.053
C:1					0.031	0.036	0.039	0.052
C:2					0.032	0.038	0.036	0.056
n_x	0	0	0	0	6	6	6	6
$Mean_x$					0.032	0.039	0.038	0.054
SD_x					0.001	0.003	0.003	0.002
SD_{repx}					0.002	0.003	0.001	0.003
SD_{hetx}					0.001	0.003	0.003	0.001
SD_{NISTx}					0.002	0.004	0.003	0.003
CV_{NISTx}					5.6	10	7.9	5.5

	NIST			
	6	6	6	6
	0.032	0.039	0.038	0.054
	0.002	0.003	0.001	0.003
	0.001	0.003	0.003	0.001
	0.001	0.001	0.001	0.001
	0.002	0.004	0.003	0.003
	6.9	11	7.6	6.2

NIST3=a+b*Median

a: 0.017 ±0.002
b: 0.396 ±0.040
R^2: 0.980

[39_dat]NISTtxt

Table 1
NIST Data and Value/Uncertainty Assignments

"Lutein"

	NIST1				NIST3			
	227	228	229	230	227	228	229	230
A:1					0.046	0.123	0.13	0.676
A:2					0.046	0.116	0.13	0.683
B:1					0.045	0.121	0.13	0.671
B:2					0.041	0.117	0.13	0.655
C:1					0.045	0.116	0.13	0.674
C:2					0.048	0.118	0.13	0.694
n_x	0	0	0	0	6	6	6	6
$Mean_x$					0.045	0.118	0.13	0.676
SD_x					0.002	0.003	0.00	0.013
SD_{repx}					0.002	0.003	0.00	0.011
SD_{betx}					0.002	0.001	0.00	0.011
SD_{NISTx}					0.002	0.004	0.00	0.016
CV_{NISTx}					5.4	3.0	2.5	2.3

"Zeaxanthin"

	NIST1				NIST3			
	227	228	229	230	227	228	229	230
A:1					0.023	0.034	0.74	0.081
A:2					0.024	0.025	0.76	0.079
B:1					0.023	0.024	0.74	0.078
B:2					0.021	0.033	0.74	0.071
C:1					0.023	0.019	0.75	0.076
C:2					0.024	0.029	0.73	0.081
n_x	0	0	0	0	6	6	6	6
$Mean_x$					0.023	0.027	0.75	0.078
SD_x					0.001	0.006	0.01	0.004
SD_{repx}					0.001	0.007	0.01	0.004
SD_{betx}					0.001	0.003	0.01	0.003
SD_{NISTx}					0.001	0.007	0.01	0.004
CV_{NISTx}					6.4	27	1.6	5.6

	NIST			
n	6	6	6	6
Mean	0.045	0.118	0.13	0.676
SD_{rep}	0.002	0.004	0.00	0.010
SD_{bet}	0.002	0.001	0.00	0.011
SD_{ani}	0.006	0.006	0.01	0.006
SD_{NIST}	0.007	0.007	0.01	0.016
CV_{NIST}	15	6.1	5.3	2.4

NIST3=a+b*Median

a: 0.015 ±0.008
b: 0.847 ±0.080
R^2: 0.983

	NIST			
	6	6	6	6
	0.023	0.027	0.75	0.078
	0.002	0.006	0.01	0.004
	0.001	0.003	0.01	0.003
	0.009	0.009	0.01	0.009
	0.009	0.011	0.01	0.010
	39	41	1.7	13

NIST3=a+b*Median

a: 0
b: 0.973 ±0.011
R^2: 0.999

RR	XXXII	XXXVIII
Serum	202	223
n_p	11	14
$Median_p$	0.036	0.125
eSD_p	0.015	0.010

<—— Previous Results ——>

XXXII	XXXVIII
202	223
6	11
0.014	0.029
0.012	0.010

	RRXXXIX			
	227	228	229	230
n_a	12	12	11	11
$Median_a$	0.037	0.116	0.14	0.788
eSD_a	0.009	0.017	0.28	0.082
P(n=p)	0.99	0.81		
P(n<p)	0.94	0.06		
SD_{labs}	0.006	0.015	0.28	0.080
CV_{labs}	17	13	200	10

<— Current Results —>

	RRXXXIX			
	227	228	229	230
	9	10	9	9
	0.014	0.037	0.77	0.087
	0.006	0.018	0.13	0.018
	1.00	0.82		
	0.96	0.04*		
	0	0.014	0.13	0.015
	0	38	17	17

NAV	0.041	0.117	0.13	0.732
NAU	0.009	0.017	0.28	0.082
CV	22	14	210	11
xCV				

<—— Assignments ——>

0.018	0.032	0.76	0.082
0.009	0.018	0.13	0.018
48	56	18	22

[39_dat]NISTtxt

Table 2
Summary of NIST Assigned Values & Uncertainties

	227			228			229			230		
Analyte	NAV	NAU	CV	NAV	NAU	CV	NAV	NAU	CV	NAV	NAU	CV
Retinol	0.869	0.056	6	0.488	0.032	7	0.377	0.036	9	0.665	0.045	7
Retinyl Palmitate	0.228	0.024	11	0.065	0.039	59	0.098	0.028	28	0.172	0.054	31
α-Tocopherol	17.2	1.4	8	7.61	0.81	11	6.95	0.81	12	10.8	1.2	11
γ-Tocopherol	3.68	0.25	7	1.70	0.12	7	4.24	0.30	7	1.28	0.14	11
δ-Tocopherol							0.5			0.3		
Total β-Carotene	1.24	0.13	11	0.169	0.027	16	0.104	0.014	14	0.100	0.017	17
trans-β-Carotene	1.135	0.064	6	0.153	0.019	12	0.095	0.010	10	0.093	0.020	22
Total α-Carotene	0.049	0.014	28	0.026	0.015	58	0.018	0.012	63	0.120	0.033	28
trans-α-Carotene	0.03			0.02			0.017			0.10		
Total Lycopene	0.326	0.060	18	0.465	0.108	23	0.58	0.15	25	0.395	0.097	25
trans-Lycopene	0.165	0.024	15	0.220	0.029	13	0.420	0.038	9	0.236	0.044	19
β-Cryptoxanthin	0.034	0.012	34	0.047	0.014	31	0.047	0.015	31	0.065	0.019	29
"Lutein"	0.041	0.009	22	0.117	0.017	14	0.13	0.28	210	0.732	0.082	11
"Zeaxanthin"	0.018	0.009	48	0.032	0.018	56	0.76	0.13	18	0.082	0.018	22

Appendix C. "All-Lab Report" for RR39

The following 6 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 XXXIX Laboratory Results

Values in µg/mL

	Retinol				Retinyl Palmitate				α-Tocopherol				γ-Tocopherol				δ-Tocopherol			
Lab	227	228	229	230	227	228	229	230	227	228	229	230	227	228	229	230	227	228	229	230
FSV-BA	0.853	0.502	0.406	0.707	0.243	0.104	0.095	0.161	17.4	7.92	7.28	11.4	3.86	1.92	4.44	1.51				
FSV-BD	0.830	0.478	0.361	0.662					16.6	7.50	7.00	10.9								
FSV-BE	0.925	0.531	0.388	0.752					17.7	7.99	7.92	12.7	3.74	1.71	4.65	1.32				
FSV-BF	0.880	0.530	0.430	0.780					17.6	7.80	7.30	12.1	3.60	1.70	4.10	1.30				
FSV-BG	0.997	0.555	0.439	0.799	0.236	0.079	0.121	0.195	17.8	8.04	7.79	12.5	3.89	1.78	4.74	1.37				
FSV-BGa	1.042	0.598	0.523	0.932	0.258	0.092	0.141	0.244	18.5	8.41	8.04	13.1	3.82	1.79	4.59	1.34				
FSV-BH	0.875	0.497	0.333	0.677	0.253	0.037	0.116	0.223	17.5	8.00	7.31	11.5	3.73	1.76	4.31	1.28				
FSV-BI	0.837	0.493	0.393	0.680	0.299	0.204	0.128	0.282	18.1	7.95	7.09	11.7	4.01	1.81	4.24	1.36				
FSV-BJ	0.864	0.521	0.387	0.719	0.234	0.078	0.144	0.189	16.0	7.79	7.11	11.8	3.73	1.81	4.50	1.45				
FSV-BK	0.858	0.483	0.384	0.687					19.5	8.72	7.83	12.8								
FSV-BL		0.487	0.372	0.687						5.17	6.46	10.3								
FSV-BM	0.859	0.520	0.497	0.696					17.7	7.10	6.50	10.0								
FSV-BN	0.820	0.470	0.370	0.710	0.230	0.060	0.100	0.230	16.3	7.32	6.99	11.5	3.96	1.86	4.74	1.36	0.220	0.21	0.56	0.46
FSV-BO	0.805	0.444	0.329	0.602					15.9	7.15	6.46	10.4								
FSV-BP	0.859	0.470	0.397	0.670					18.8	7.78	6.65	11.5								
FSV-BQ	0.740	0.550	0.390	0.680					17.6	7.44	6.95	9.8								
FSV-BR	0.910	0.560	0.390	0.690																
FSV-BS	0.880	0.420	0.310	0.620																
FSV-BT	0.899	0.483	0.405	0.788	0.240	0.095	0.122	0.262	17.9	8.25	7.78	11.4	4.18	1.83	4.64	1.43	0.218	0.13	0.75	0.46
FSV-BU	0.846	0.441	0.357	0.514					14.1	5.83	5.68	7.3	3.33	1.52	3.82	1.02				
FSV-BV	0.828	0.483	0.355	0.678					15.9	7.32	6.62	11.5	4.00	1.86	4.83	1.44				
FSV-BW	0.930	0.510	0.420	0.670	0.230	0.047	0.068	0.126	18.1	7.80	7.01	11.3	4.03	1.78	4.66	1.30				
FSV-BX	1.006	0.510	0.425	0.765					17.8	7.19	7.47	11.0	3.90	1.81	4.28	1.59				
FSV-BY	0.887	0.514	0.387	0.678	0.272	0.125	0.130	0.181	17.5	7.92	6.88	10.3	3.76	1.73	4.36	1.19	0.152	0.16	0.42	0.22
FSV-BZ									15.8	7.17	8.20	10.6	4.50	1.70	4.35	3.80				
FSV-CA	0.737	0.474	!0.302	!0.603					13.8	5.89	!4.32	!7.93								
FSV-CB	0.760	0.430	0.300	0.580					18.1	7.88	7.47	11.3								
FSV-CC	0.850	0.492	0.355	0.603					15.1	6.17	5.67	9.3								
FSV-CD	0.772	0.501	0.419	0.723	0.318	0.110	0.098	0.082	18.8	9.05	7.55	11.6	3.55	1.82	4.79	1.42				
FSV-CE	0.873	0.497	0.383	0.683					17.9	7.87	6.91	11.5								
FSV-CF	0.855	0.489	0.361	0.620					17.3	7.50	7.10	11.9								
FSV-CG	0.906	0.588	0.549	0.840					11.8	3.81	3.22	6.9	4.01	2.03	4.47	1.61				
FSV-CH	0.838	0.481	0.382	0.682					16.6	7.19	6.27	11.1	3.42	1.56	3.92	1.17				
FSV-CK	0.884	0.471	0.394	0.741					16.7	7.36	6.88	11.2	3.50	1.64	4.08	1.35				
FSV-CL	1.131	0.587	0.285	0.819					16.9	7.05	6.47	11.0	3.07	1.74	3.34	1.58				
FSV-CM									17.9	7.39	8.01	10.4								
FSV-CN	0.837	0.500	0.411	0.722					16.7	7.29	7.21	11.9	3.40	1.44	4.25	1.11				
FSV-CP									17.2	8.22	7.51	12.1	3.64	1.72	4.17	1.52				
FSV-CQ	0.718	0.507	0.349	0.696					16.3	7.93	7.54	12.1								
FSV-CR	0.886	0.513	0.387	0.699					18.6	8.20	7.68	12.3	7.06	3.23	8.32	2.25	0.103	*nd*	0.27	*nd*
FSV-CS	0.920	0.480	0.380	0.700																
FSV-CT	0.908	0.486	0.375	0.537					15.5	8.06	7.26	11.4								
FSV-CU	0.831	0.492	0.402	0.724	0.262	0.127	0.080	0.111	16.7	7.26	6.25	9.9								
FSV-CX	0.870	0.510	0.380	0.670	0.120	0.010	0.060	0.190	17.3	8.65	8.15	12.3	4.03	1.70	4.72	1.49				
FSV-DA	0.889	0.486	0.380	0.705	0.234	0.049	0.077	0.160	17.0	7.68	7.06	11.3	4.03	1.78	4.75	1.37	0.183	0.14	0.49	0.28
FSV-DB	0.920	0.530	0.280	0.690					16.9	7.83	6.08	11.7								
FSV-DJ	0.550	0.410	0.310	0.680					11.1	6.10	7.60	12.1								
FSV-DL	0.848	0.477	0.384	0.643					17.8	8.03	7.64	11.7	3.87	1.93	4.58	1.49				
FSV-DP	0.932	0.525	0.398	0.728																
FSV-DQ	0.820	0.440	0.360	0.690					18.6	9.47	8.43	14.3	4.03	1.88	4.28	1.63				
FSV-DR	0.851	0.459	0.354	0.673					16.6	7.50	7.29	11.9								
FSV-DS	0.990	0.500	0.330	0.550					15.3	7.62	7.23	10.2								
FSV-DU	1.243	0.478	0.417	0.908					23.2	7.57	8.06	12.7								
FSV-EI	0.763	0.404	0.310	0.527					14.8	6.54	6.25	10.3	3.67	1.71	4.12	1.31				
FSV-EL	0.930	0.520	0.430	0.760																
FSV-EM	0.850	0.540	0.320	0.660					14.7	6.40	4.20	7.7								
FSV-FN	0.891	0.515	0.382	0.693					18.8	8.35	7.26	12.0	3.76	1.81	4.13	1.36				
n	53	54	53	53	14	14	14	14	51	52	51	51	30	30	30	30	5	4	5	4
Min	0.550	0.404	0.280	0.514	0.120	0.010	0.060	0.082	11.1	3.81	3.22	6.9	3.07	1.44	3.34	1.02	0.103	0.133	0.27	0.220
Median	0.864	0.495	0.383	0.690	0.241	0.086	0.108	0.190	17.3	7.65	7.21	11.5	3.84	1.78	4.40	1.37	0.183	0.149	0.49	0.368
Max	1.243	0.598	0.549	0.932	0.318	0.204	0.144	0.282	23.2	9.47	8.43	14.3	7.06	3.23	8.32	3.80	0.220	0.210	0.75	0.465
eSD	0.052	0.029	0.034	0.043	0.017	0.046	0.031	0.055	1.1	0.57	0.64	0.9	0.27	0.11	0.38	0.12	0.052		0.11	
eCV	6	6	9	6	7	54	29	29	7	7	9	8	7	6	9	9	29		23	
NISTa	0.889	0.494	0.388	0.657	0.248	*nq*	*nq*	*nq*	17.5	8.19	7.21	10.5	3.59	1.68	4.34	1.32				
NISTb	0.870	0.476	0.354	0.628	0.181	0.045	0.078	0.152	17.3	7.19	6.35	10.1	3.43	1.55	3.92	1.10	*nd*	*nd*	0.48	0.22
NAV	0.872	0.490	0.377	0.666	0.241	0.086	0.108	0.190	17.4	7.67	7.00	10.9	3.67	1.70	4.26	1.29			0.48	
NAU	0.074	0.042	0.036	0.066	0.017	0.046	0.031	0.055	1.4	0.75	0.76	1.4	0.43	0.23	0.45	0.20			0.11	

Round Robin XXXIX Laboratory Results

Values in µg/mL

	Total β-Carotene				trans-β-Carotene				Total cis-β-Carotene				Total α-Carotene			
Lab	227	228	229	230	227	228	229	230	227	228	229	230	227	228	229	230
FSV-BA	1.22	0.181	0.119	0.131	1.17	0.172	0.112	0.123	0.058	0.009	0.007	0.008	0.056	0.035	0.028	0.155
FSV-BD	0.90	0.126	0.094	0.098												
FSV-BE	1.29	0.188	0.119	0.099												
FSV-BF	1.17	0.139	0.083	0.063									0.065	0.018	0.016	0.157
FSV-BG	1.19	0.185	0.111	0.112									0.045	0.025	0.020	0.135
FSV-BGa	1.23	0.180	0.111	0.114									0.051	0.048	0.039	0.120
FSV-BH	1.28	0.175	0.103	0.115	1.21	0.169	0.101	0.109	0.075	*nd*	*nd*	*nd*	0.048	0.026	0.018	0.179
FSV-BI	1.34	0.176	0.104	0.117									0.043	0.027	0.018	0.145
FSV-BJ	1.35	0.196	0.119	0.116									0.057	0.038	0.027	0.159
FSV-BK																
FSV-BL																
FSV-BM																
FSV-BN	1.28	0.160	0.110	0.110	1.20	0.150	0.097	0.101	*0.080*	*0.010*	*0.013*	*0.009*	0.040	0.020	0.015	0.170
FSV-BO	0.90	0.165	0.082	0.080									0.037	0.020	0.014	0.053
FSV-BP	1.18	0.101	0.093	0.102									0.042	0.013	0.014	0.097
FSV-BQ																
FSV-BR																
FSV-BS	1.24	0.200	0.120	0.120	1.18	0.170	0.120	0.120	0.055	0.032	*nd*	*nd*	0.059	0.047	0.038	0.133
FSV-BT	1.24	0.178	0.113	0.119	1.16	0.172	0.107	0.113	0.083	0.006	0.006	0.006	0.035	0.034	0.026	0.206
FSV-BU	1.12	0.146	0.100	0.101									0.031	0.016	0.013	0.095
FSV-BV	1.01	0.164	0.117	0.103									0.029	0.017	0.012	0.121
FSV-BW	1.40	0.211	0.112	0.103									0.027	0.027	0.016	0.107
FSV-BX	1.41	0.330		0.274									0.083	0.040	0.038	0.156
FSV-BY	1.28	0.177	0.100	0.092	1.16	0.165	0.090	0.084	0.117	0.012	0.011	0.008	0.050	0.040	0.029	0.092
FSV-BZ					0.90	0.140	0.080	0.083	*nq*	*nd*	*nd*	*nd*	0.070	0.050	0.033	0.102
FSV-CA																
FSV-CB																
FSV-CC																
FSV-CD	0.88	0.090	0.050	0.034									0.028	0.012	0.010	0.043
FSV-CE	1.10	0.172	0.107	0.138												
FSV-CF																
FSV-CG	1.52	0.216	0.137	0.142									0.055	0.040	0.033	0.156
FSV-CH	1.21	0.163	0.101	0.091									0.026	0.019	0.010	0.152
FSV-CK	1.22	0.188	0.134	0.147									0.055	0.045	0.038	0.178
FSV-CL	1.56	0.167	0.086	0.097									0.049	0.023	0.011	0.144
FSV-CM																
FSV-CN					0.53	0.073	0.039	0.051					*nd*	*nd*	*nd*	0.080
FSV-CP	1.14	0.145	0.102	0.104									0.036	0.018	0.014	0.129
FSV-CQ	1.78	0.239	0.075	0.052												
FSV-CR																
FSV-CS	1.29	0.159	0.102	0.098	1.16	0.145	0.092	0.088	*0.131*	*0.014*	*0.010*	*0.010*	0.052	0.026	0.016	0.144
FSV-CT	1.01	0.085	0.052	0.067												
FSV-CU																
FSV-CX	1.48	0.210	0.160	0.140									0.040	0.020	0.010	0.100
FSV-DA	1.13	0.156	0.116	0.106	1.04	0.144	0.101	0.096	0.090	0.012	0.015	0.010	0.045	0.021	0.017	0.138
FSV-DB	1.16	0.150	0.060	0.080												
FSV-DJ																
FSV-DL	1.47	0.192	0.120	0.126									0.054	0.033	0.024	0.145
FSV-DP																
FSV-DQ	1.30	0.224	0.141	0.135									0.043	0.084	0.053	0.350
FSV-DR	1.44	0.185	0.149	0.084												
FSV-DS	0.17	0.230	0.350	0.210												
FSV-DU	1.68	0.152	0.125	0.140												
FSV-EI					1.19	0.150	0.092	0.083					0.036	0.028	0.020	0.113
FSV-EL																
FSV-EM																
FSV-FN																
n	37	37	36	37	11	11	11	11	8	7	6	6	30	30	30	31
Min	0.17	0.085	0.050	0.034	0.53	0.073	0.039	0.051	0.055	0.006	0.006	0.006	0.026	0.012	0.010	0.043
Median	1.24	0.176	0.111	0.106	1.16	0.150	0.097	0.096	0.082	0.012	0.011	0.009	0.045	0.027	0.018	0.138
Max	1.78	0.330	0.350	0.274	1.21	0.172	0.120	0.123	0.131	0.032	0.015	0.010	0.083	0.084	0.053	0.350
eSD	0.16	0.030	0.015	0.021	0.06	0.022	0.010	0.019	0.024	0.003	0.005	0.001	0.014	0.013	0.010	0.031
eCV	13	17	13	20	5	15	10	20	29	25	44	17	30	47	54	23
NISTa	1.27	0.179	0.112	0.111	1.11	0.166	0.104	0.103	*0.162*	*0.012*	*0.009*	*0.008*	0.048	*nq*	*nq*	0.096
NISTb	1.19	>0.144	>0.081	>0.077	1.10	0.144	0.081	0.077	*0.087*	*nq*	*nq*	*nq*	0.057	0.024	0.017	0.104
NAV	1.24	0.169	0.104	0.100	1.13	0.153	0.095	0.093	0.103	0.012	0.010	0.008	0.049	0.025	0.017	0.119
NAU	0.16	0.031	0.023	0.023	0.12	0.019	0.017	0.021	0.078	0.005	0.005	0.006	0.015	0.015	0.011	0.047

Round Robin XXXIX Laboratory Results

Values in µg/mL

	Total Lycopene				trans-Lycopene				β-Cryptoxanthin				α-Cryptoxanthin			
Lab	227	228	229	230	227	228	229	230	227	228	229	230	227	228	229	230
FSV-BA					0.181	0.266	0.48	0.296	0.049	0.073	0.046	0.087				
FSV-BD	0.209	0.31	0.50	0.31					0.030	0.048	0.049	0.062				
FSV-BE																
FSV-BF	0.379	0.54	0.75	0.55												
FSV-BG	0.328	0.53	0.74	0.48	0.162	0.249	0.47	0.266	0.029	0.054	0.050	0.075				
FSV-BGa	0.369	0.54	0.73	0.49												
FSV-BH	0.358	0.55	0.76	0.52					0.048	0.074	0.074	0.098				
FSV-BI	0.407	0.56	0.86	0.57					0.046	0.062	0.067	0.100				
FSV-BJ	0.317	0.44	0.74	0.46												
FSV-BK																
FSV-BL																
FSV-BM																
FSV-BN	0.340	0.49	0.80	0.54	0.180	0.250	0.58	0.310	0.030	0.040	0.050	0.070	0.010	0.010	0.030	0.030
FSV-BO	0.357	0.50	0.57	0.45					0.027	0.044	0.050	0.069				
FSV-BP	0.263	0.09	0.45	0.33					0.077	0.117	0.045	0.062				
FSV-BQ																
FSV-BR																
FSV-BS	0.270	0.38	0.52	0.35					0.021	0.035	0.038	0.053				
FSV-BT	0.306	0.45	0.59	0.42	0.248	0.361	0.52	0.359	0.046	0.067	0.070	0.096	0.017	0.029	0.046	0.041
FSV-BU	0.320	0.38	0.54	0.45					0.042	0.079	0.103	0.100				
FSV-BV	0.243	0.34	0.49	0.30					0.011	0.019	0.019	0.026				
FSV-BW	0.300	0.47	0.59	0.39												
FSV-BX	0.389	0.29		0.20					0.016	0.029	0.031	0.044				
FSV-BY	0.326	0.50	0.61	0.38	0.181	0.237	0.44	0.221	0.045	0.049	0.050	0.063				
FSV-BZ					0.300	0.300	0.51	0.325								
FSV-CA																
FSV-CB																
FSV-CC																
FSV-CD	0.295	0.31	0.43	0.26					0.035	0.050	0.081	0.080				
FSV-CE																
FSV-CF																
FSV-CG	0.277	0.42	0.58	0.40					0.041	0.088	0.083	0.102				
FSV-CH	0.207	0.32	0.52	0.34												
FSV-CK	0.371	0.67	0.81	0.61					0.026	0.063	0.075	0.086	0.016	0.055	0.076	0.073
FSV-CL	0.333	0.50	0.63	0.45					0.032	0.036	0.035	0.043				
FSV-CM																
FSV-CN	0.179	0.25	0.41	0.25					0.030	0.046	0.048	0.075				
FSV-CP	0.289	0.39	0.57	0.41					0.039	0.064	0.074	0.093				
FSV-CQ																
FSV-CR																
FSV-CS	0.343	0.50	0.65	0.42					0.035	0.053	0.058	0.072				
FSV-CT																
FSV-CU																
FSV-CX	0.250	0.36	0.56	0.35					0.060	0.100	0.100	0.140				
FSV-DA	0.330	0.48	0.74	0.50	0.166	0.231	0.51	0.283	0.039	0.055	0.068	0.083	0.010	0.018	0.040	0.039
FSV-DB	0.382	0.52	0.64	0.41					0.054	0.066	0.057	0.095				
FSV-DJ																
FSV-DL	0.340	0.46	0.56	0.39					0.036	0.055	0.068	0.077				
FSV-DP																
FSV-DQ	0.374	0.61	0.85	0.53					0.039	0.065	0.068	0.081				
FSV-DR																
FSV-DS																
FSV-DU																
FSV-EI					0.135	0.173	0.34	0.150	0.032	0.047	0.054	0.073				
FSV-EL																
FSV-EM																
FSV-FN																
n	30	30	29	30	8	8	8	8	27	27	27	27	4	4	4	4
Min	0.179	0.09	0.41	0.20	0.135	0.173	0.34	0.150	0.011	0.019	0.019	0.026	0.010	0.010	0.030	0.030
Median	0.327	0.46	0.59	0.42	0.181	0.250	0.49	0.290	0.036	0.055	0.057	0.077	0.013	0.024	0.043	0.040
Max	0.407	0.67	0.86	0.61	0.300	0.361	0.58	0.359	0.077	0.117	0.103	0.140	0.017	0.055	0.076	0.073
eSD	0.060	0.11	0.14	0.10	0.024	0.026	0.04	0.044	0.010	0.016	0.016	0.022				
eCV	18	24	24	25	14	10	7	15	29	30	29	29				
NISTa																
NISTb	0.325	0.46	0.56	0.37	0.150	0.190	0.35	0.183	0.032	0.039	0.038	0.054				
NAV	0.326	0.46	0.58	0.39	0.165	0.220	0.42	0.236	0.034	0.047	0.048	0.066				
NAU	0.077	0.11	0.15	0.11	0.043	0.066	0.15	0.096	0.012	0.019	0.022	0.026				

Round Robin XXXIX Laboratory Results

Values in µg/mL

	Lutein				Zeaxanthin				Lutein&Zeaxanthin				Total Carotenoids			
Lab	227	228	229	230	227	228	229	230	227	228	229	230	227	228	229	230
FSV-BA									0.061	0.208	1.33	1.27				
FSV-BD	0.028	0.098	0.10	0.69	0.013	0.032	0.67	0.076	*0.041*	*0.130*	*0.77*	*0.77*				
FSV-BE																
FSV-BF																
FSV-BG	0.050	0.136	0.18	0.89	0.009	0.028	0.95	0.068	*0.059*	*0.164*	*1.13*	*0.96*				
FSV-BGa																
FSV-BH	0.027	0.103	0.09	0.74	*nd*	0.023	0.77	0.059	*0.027*	*0.126*	*0.86*	*0.80*				
FSV-BI	0.037	0.119	0.10	0.71	0.012	0.036	0.69	0.093	*0.049*	*0.155*	*0.79*	*0.80*				
FSV-BJ																
FSV-BK																
FSV-BL																
FSV-BM																
FSV-BN	0.018	0.100	0.80	0.72	0.010	0.050	0.04	0.100	*0.028*	*0.150*	*0.84*	*0.82*				
FSV-BO									0.027	0.102	0.64	0.60				
FSV-BP																
FSV-BQ																
FSV-BR																
FSV-BS	0.019	0.110	0.71	0.83												
FSV-BT	0.039	0.121			0.023	0.089	*nd*	*nd*	0.061	0.160	0.84	0.83	1.846	1.130	1.923	1.967
FSV-BU									0.037	0.139	0.74	0.72				
FSV-BV									0.040	0.127	0.65	0.60				
FSV-BW																
FSV-BX	0.045	0.137	0.14	0.91	0.014	0.034	0.83	0.077	*0.059*	*0.171*	*0.96*	*0.99*				
FSV-BY	0.037	0.126	1.02	0.82	0.020	0.037	0.06	0.136	0.057	0.163	1.11	1.09				
FSV-BZ	0.036	0.105	0.24	0.25												
FSV-CA																
FSV-CB																
FSV-CC																
FSV-CD									0.068	0.227	1.34	1.12				
FSV-CE																
FSV-CF																
FSV-CG									0.076	0.232	0.98	0.98				
FSV-CH																
FSV-CK									0.059	0.194	0.80	0.82				
FSV-CL									0.052	0.133	0.53	0.68				
FSV-CM																
FSV-CN									0.076	0.129	0.84	0.72				
FSV-CP									0.049	0.195	1.27	0.97				
FSV-CQ																
FSV-CR																
FSV-CS									0.052	0.151	0.77	0.82				
FSV-CT																
FSV-CU																
FSV-CX	0.040	0.210	0.19	0.55	0.010	0.040	0.41	0.100	*0.050*	*0.250*	*0.60*	*0.65*				
FSV-DA	0.032	0.113	0.10	0.81	0.019	0.059	0.92	0.087	0.051	0.172	1.02	0.91				
FSV-DB									0.052	0.160	0.64	0.70				
FSV-DJ																
FSV-DL									0.066	0.161	0.64	0.58				
FSV-DP																
FSV-DQ									0.052	0.138	0.58	0.81				
FSV-DR																
FSV-DS																
FSV-DU																
FSV-EI	0.044	0.129	0.14	0.79	0.043	0.079	0.85	0.135	*0.087*	*0.208*	*0.99*	*0.92*				
FSV-EL																
FSV-EM													1.890	1.160	1.620	1.650
FSV-FN																
n	13	13	12	12	10	11	10	10	25	25	25	25	2	2	2	2
Min	0.018	0.098	0.09	0.25	0.009	0.023	0.04	0.059	0.027	0.102	0.53	0.58	1.846	1.130	1.620	1.650
Median	0.037	0.119	0.16	0.76	0.013	0.037	0.73	0.090	0.052	0.160	0.84	0.82	1.868	1.145	1.772	1.808
Max	0.050	0.210	1.02	0.91	0.043	0.089	0.95	0.136	0.087	0.250	1.34	1.27	1.890	1.160	1.923	1.967
eSD	0.010	0.021	0.09	0.09	0.006	0.013	0.24	0.020	0.013	0.040	0.27	0.18				
eCV	28	17	55	12	42	36	33	22	26	25	33	22				
NISTa																
NISTb	0.045	0.118	0.13	0.68	0.023	0.027	0.75	0.078	*0.068*	*0.146*	*0.88*	*0.75*				
NAV	0.041	0.119	0.14	0.72	0.018	0.032	0.74	0.084	0.060	0.153	0.86	0.79				
NAU	0.011	0.027	0.19	0.19	0.010	0.017	0.27	0.028	0.016	0.043	0.25	0.19				

Round Robin XXXIX Laboratory

Analytes Reported By One Laboratory

Values in µg/mL

Analyte	Code	227	228	229	230
cis-Lutein&Zeaxanthin	FSV-BT	0.061	0.100	0.112	0.119
Coenzyme Q10	FSV-CH	0.249	0.227	0.288	0.269
trans-α-Carotene	NISTb	0.034	0.024	0.017	0.104

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 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 greater than or equal to x
!	Discrepant value: heterogeneous serum, damaged sample, malfunction, etc.
italics	Not explicitly reported but calculated by NIST from reported values

Round Robin XXXIX Laboratory Results

Comparability Summary

Lab	R	aT	gT	bC	tbC
FSV-BA	1	1	1	1	1
FSV-BD	1	1	1	1	
FSV-BE	4	2	1	1	
FSV-BF	2	2	2	1	
FSV-BG	2	3			
FSV-BGa	1	1		3	
FSV-BH	2	1	1	2	
FSV-BI	3	1		4	
FSV-BJ	2	2	1	1	
FSV-BK	1	1	2	2	2
FSV-BL	2	1	1	1	1
FSV-BM	2	1	1	2	
FSV-BN	2	1	2	4	
FSV-BO		2	4		2
FSV-BP		2			
FSV-BQ	1	4			
FSV-BR	1	1	1	1	
FSV-BS	1	2			
FSV-BT	1	1			
FSV-BU		1	2	1	
FSV-BV	1	1	1	2	
FSV-BW	1	1	1	1	
FSV-BX	4	1	2	3	
FSV-BY	1	2	1	3	
FSV-BZ	3	1			
FSV-CA	1	2		3	
FSV-CB	1	1	2	1	1
FSV-CC	3	2	1		1
FSV-CD	2			1	2
FSV-CE	2	2		3	
FSV-CF	2	2		3	
FSV-CG	2	2		4	
FSV-CH	2	2	2	1	1
FSV-CK	2				
FSV-CL	2	1			
FSV-CM	4	4			
FSV-CN	4	4	2	2	
FSV-CP	4	1			
FSV-CQ	1	2	4		
FSV-CR	2	2	2	3	
FSV-CS	3	2		2	
FSV-CT	3	3	2	1	
FSV-CU	1	1	2	1	1
FSV-CX	1				
FSV-DA	2				
FSV-DB	1	2	2	2	
FSV-DJ	1	1	2		4
FSV-DL	1	2			
FSV-DP	2	1	1	2	
FSV-DQ	1	1			
FSV-DR	1			1	1
FSV-DS	4	4		3	
FSV-DU	1	1		2	
FSV-EI	2	3	2	2	
FSV-EL	2	4			
FSV-EM	1	2	1		
FSV-FN	1	1		2	
NISTa	1	1	1	1	1
NISTb	1	1	1	1	1
n	54	52	30	37	11

Label	Definition
Lab	laboratory number
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 (non-NIST) 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 $\pm t$(1-0.683,n-1)	{i.e., ±1 SD}
2	All StV within $\pm t$(1-0.954,n-1)	{i.e., ±2 SD}
3	All StV within $\pm t$(1-0.997,n-1)	{i.e., ±3 SD}
4	At least one StV > $\pm t$(1-0.997,n-1)	{i.e., >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

StS	% Observed					Expected
1	61	72	78	54	69	68.2 %
2	29	15	16	33	23	27.3 %
3	5	11	0	8	0	4.3 %
4	5	2	6	5	8	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 D. Representative "Individualized Report" for RR39

Each participant in RR39 received an "Individualized Report" reflecting their reported results. Each report included a detailed analysis of the results they reported for some or all of the following analytes:

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

The software used to generate the original RR39 "Individualized Reports" is no longer available and we do not have hardcopy of the report as it was sent to any participant or NIST analyst. The following 11 pages were produced for participant FSV-BA using a descendant of the 1997 software. Three of the graphical tools used in the original reports ("Boxplot Comparisons", "Z-Score Concordance", and "NIST Assigned Values Vs Laboratory Values") have been retained and display the same information in much the same manner as in the original. However, the original report presented the same type of information for a number of analytes together on one or two pages rather than presenting all information for a given analyte on a single page. The modern software does not provide the "% RSD Bias and Precision History" table or the "% Difference" plots.

Individualized Round Robin XXXIX Report: FSV-BA

Summary

	Serum 227			Serum 228			Serum 229			Serum 230		
Analyte	You	NAV	n	You	NAV	n	You	NAV	n	You	NAV	n
Retinol	0.853	0.872	53	0.502	0.490	54	0.406	0.377	53	0.707	0.666	53
Retinyl Palmitate	0.243	0.241	14	0.104	0.086	14	0.095	0.108	14	0.161	0.190	14
α-Tocopherol	17.38	17.35	51	7.9	7.7	52	7.3	7.0	51	11.37	10.87	51
γ-Tocopherol	3.86	3.67	30	1.92	1.70	30	4.44	4.26	30	1.51	1.29	30
Total β-Carotene	1.224	1.235	37	0.181	0.169	37	0.119	0.104	36	0.131	0.100	37
trans-β-Carotene	1.166	1.135	11	0.172	0.153	11	0.112	0.095	11	0.123	0.093	11
Total cis-β-Carotene	0.058	0.103	8	0.009	0.012	7	0.007	0.010	6	0.008	0.008	6
Total α-Carotene	0.056	0.049	30	0.035	0.025	30	0.028	0.017	30	0.155	0.119	31
trans-Lycopene	0.181	0.165	8	0.266	0.220	8	0.478	0.421	8	0.296	0.236	8
β-Cryptoxanthin	0.049	0.034	27	0.073	0.047	27	0.046	0.048	27	0.087	0.066	27
Lutein&Zeaxanthin	0.061	0.060	25	0.208	0.153	25	1.330	0.855	25	1.270	0.786	25

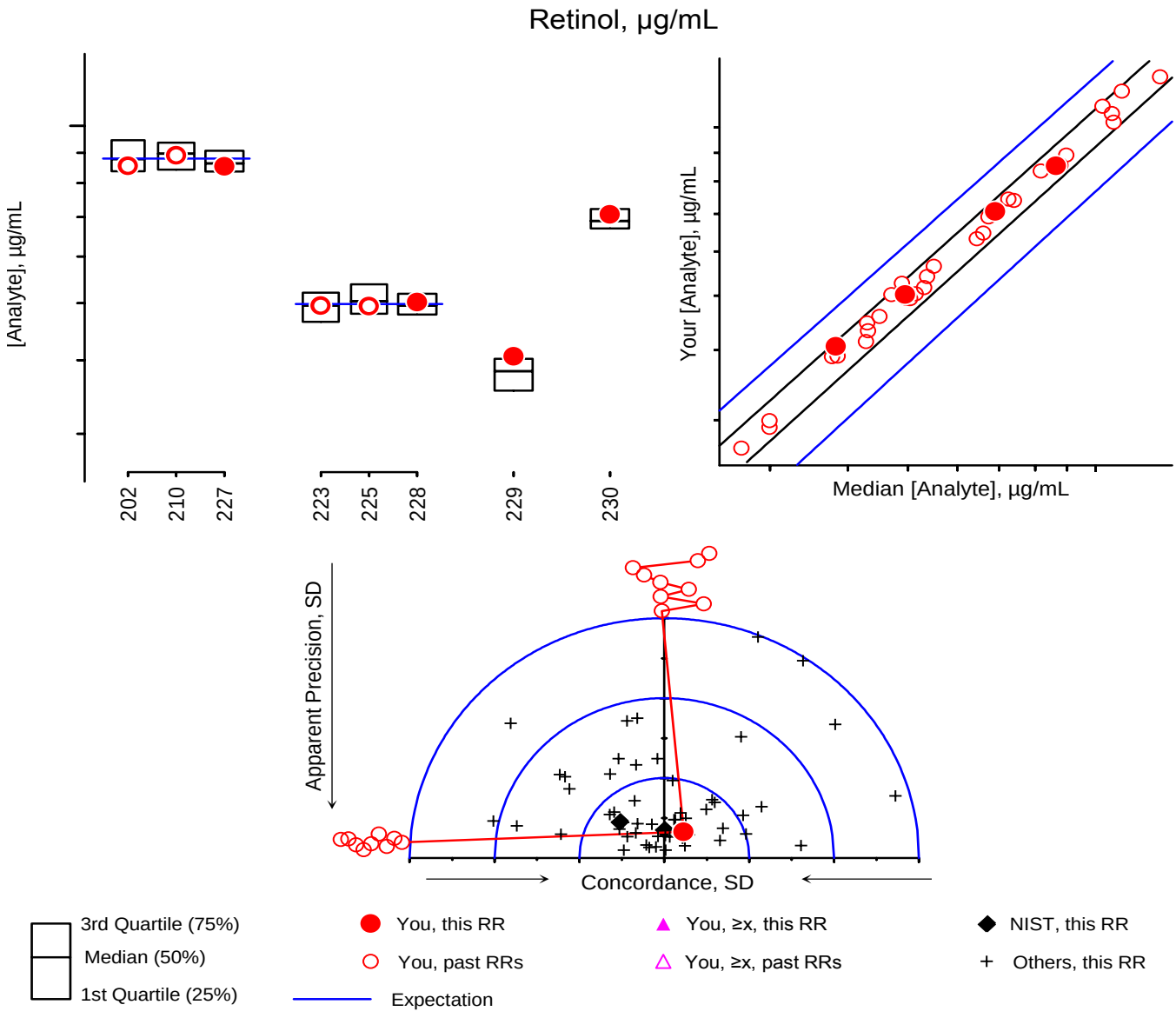
You : Your reported values for the listed analytes (micrograms/milliliter)
NAV : NIST Assigned Values, here equal to this RR's median
n : Number of non-NIST laboratories reporting quantitative values for this analyte in this serum

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

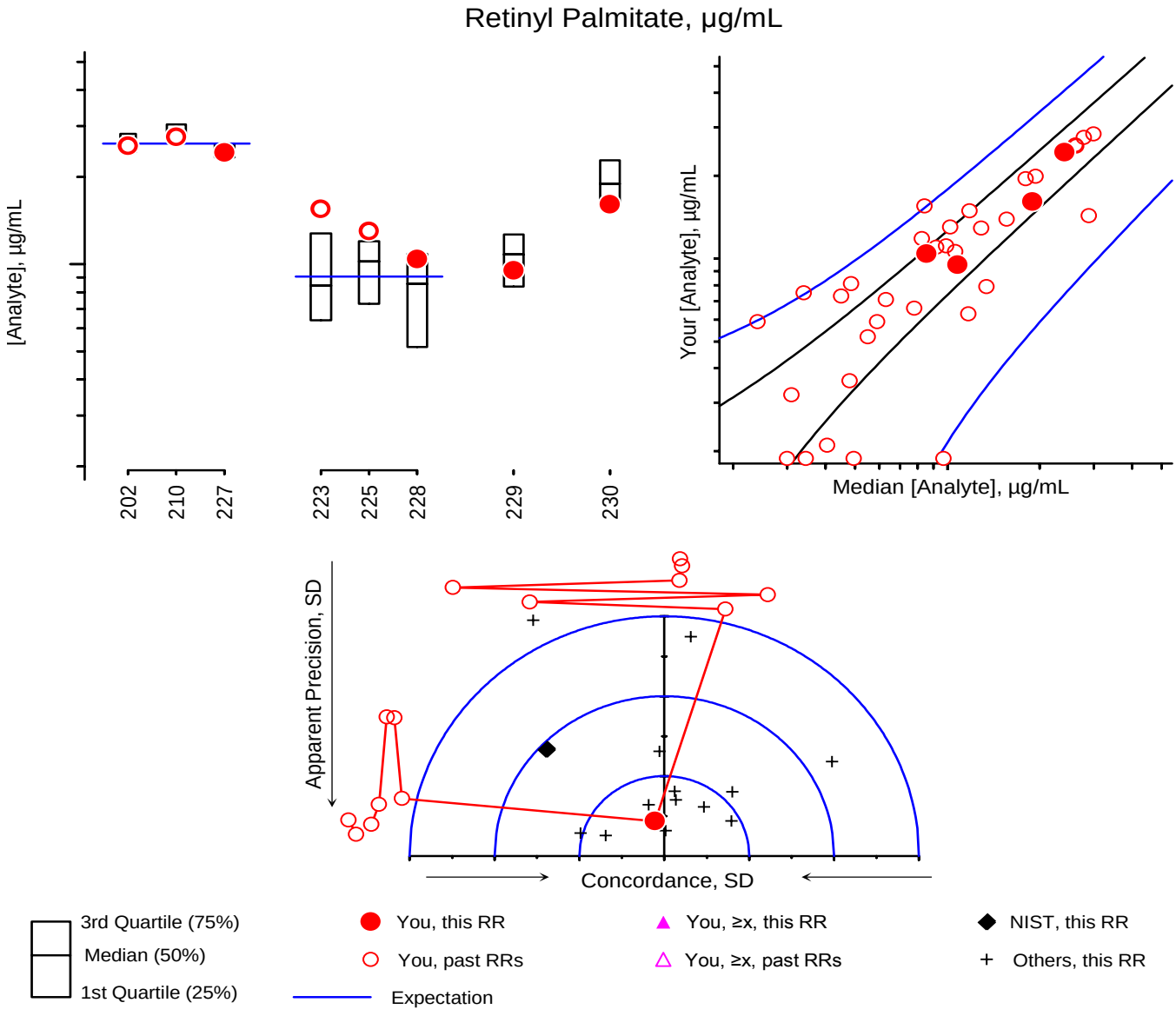
Individualized RR XXXIX Report: FSV-BA



The software that was used to produce the original "Individualized Reports" for this study is no longer available. The original reports provided the same graphical analyses but in different format. This sheet was generated using a descendent of the 1997 system. For details of the construction and interpretation of these plots, see: Duewer et al. Anal Chem 1999;71(9):1870-8.

Serum	Comments	History
#227	Lyophilized, multi-donor, native. This is the High level of SRM 968b.	RR32 #202, RR34 #210
#228	Lyophilized, single donor, augmented with retinyl palmitate.	RR38 #223 and #225
#229	Lyophilized, prepared a "low normal" serum pool, augmented with: retinyl palmitate, γ- and δ-tocopherol, lycopene, and lutein.	New
#230	Lyophilized, prepared a "low normal" serum pool, augmented with: retinol, retinyl palmitate, α- and γ-tocopherol, α-carotene, β-cryptoxanthin, lycopene, and zeaxanthin.	New

Individualized RR XXXIX Report: FSV-BA



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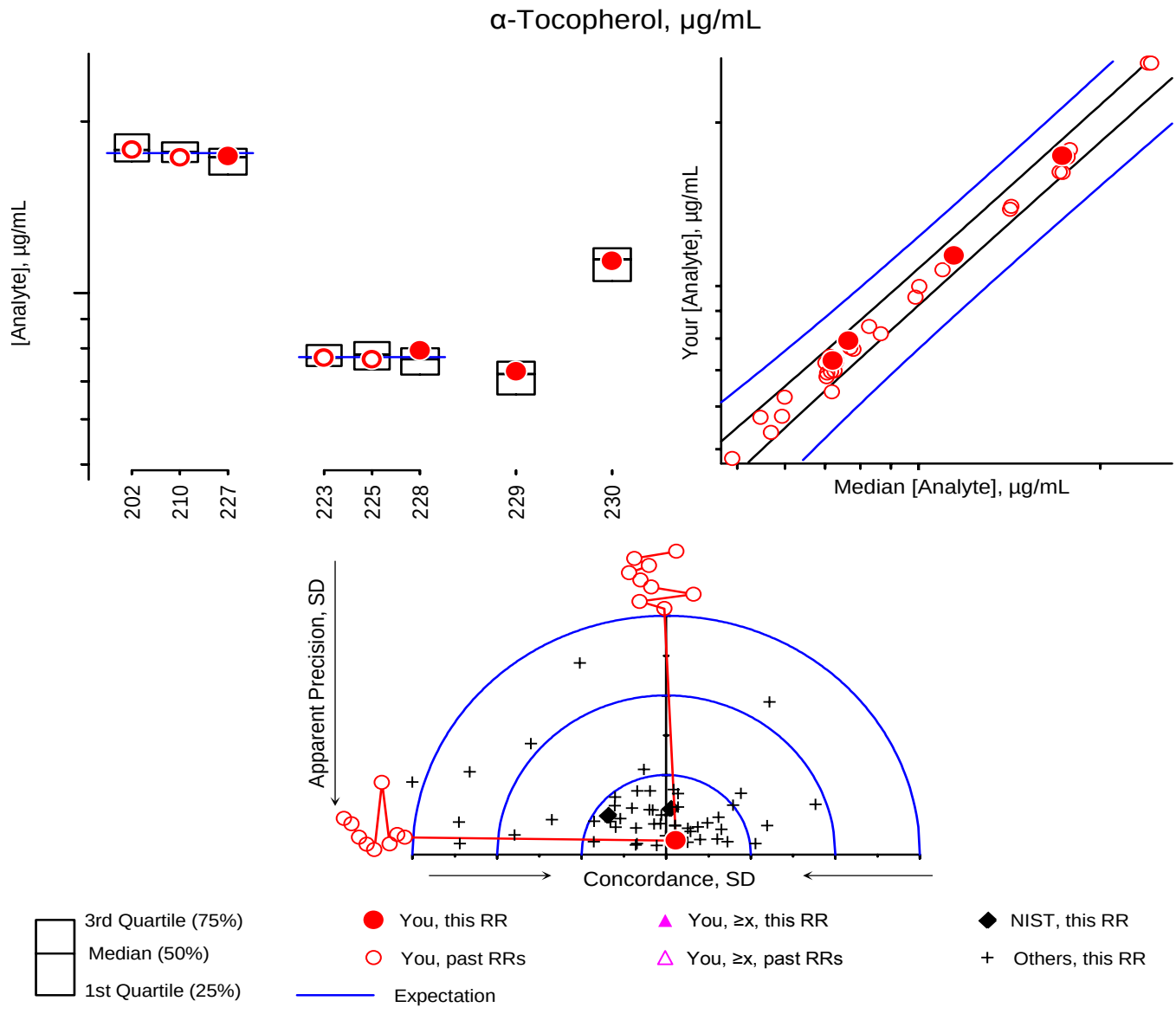

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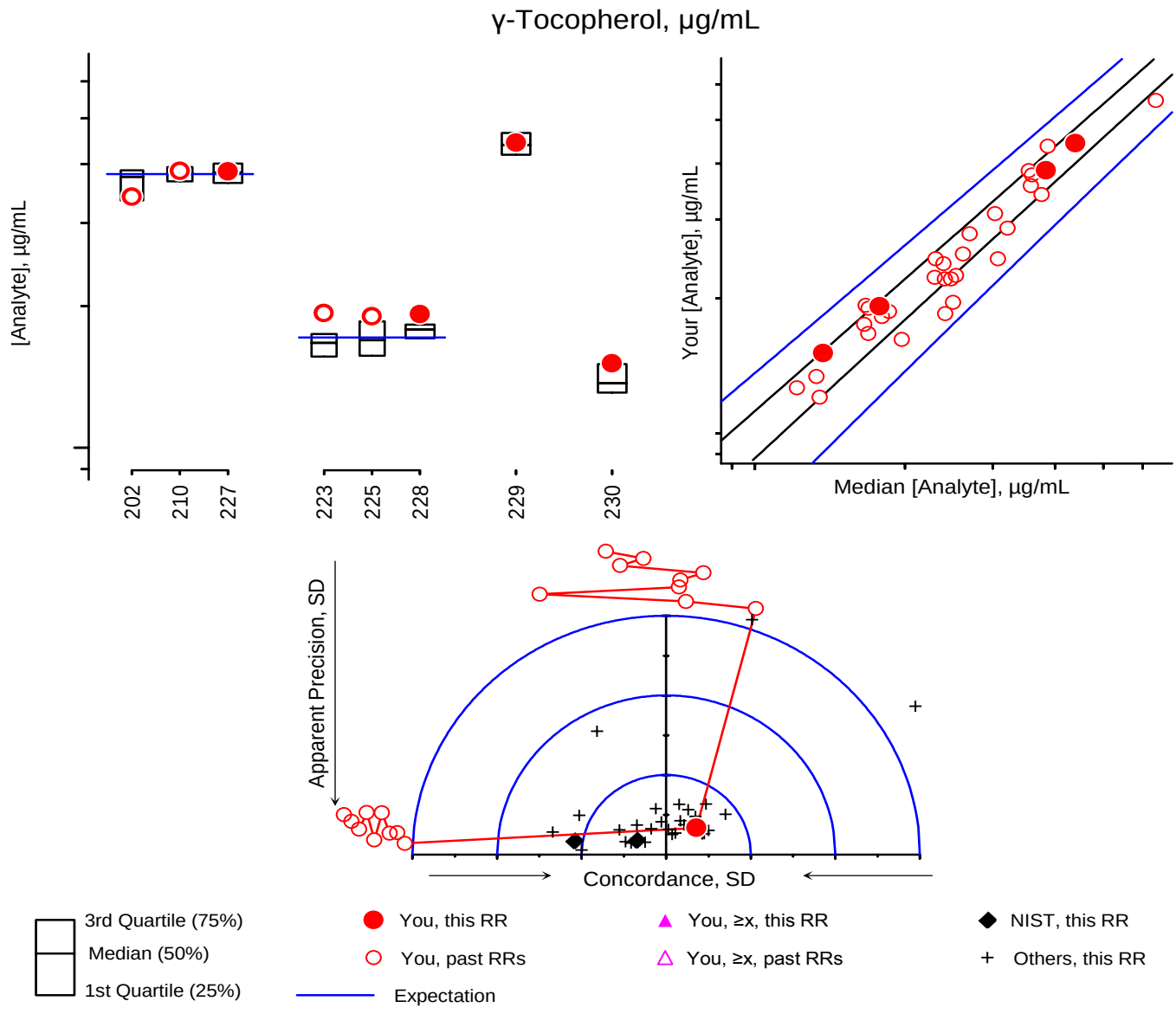

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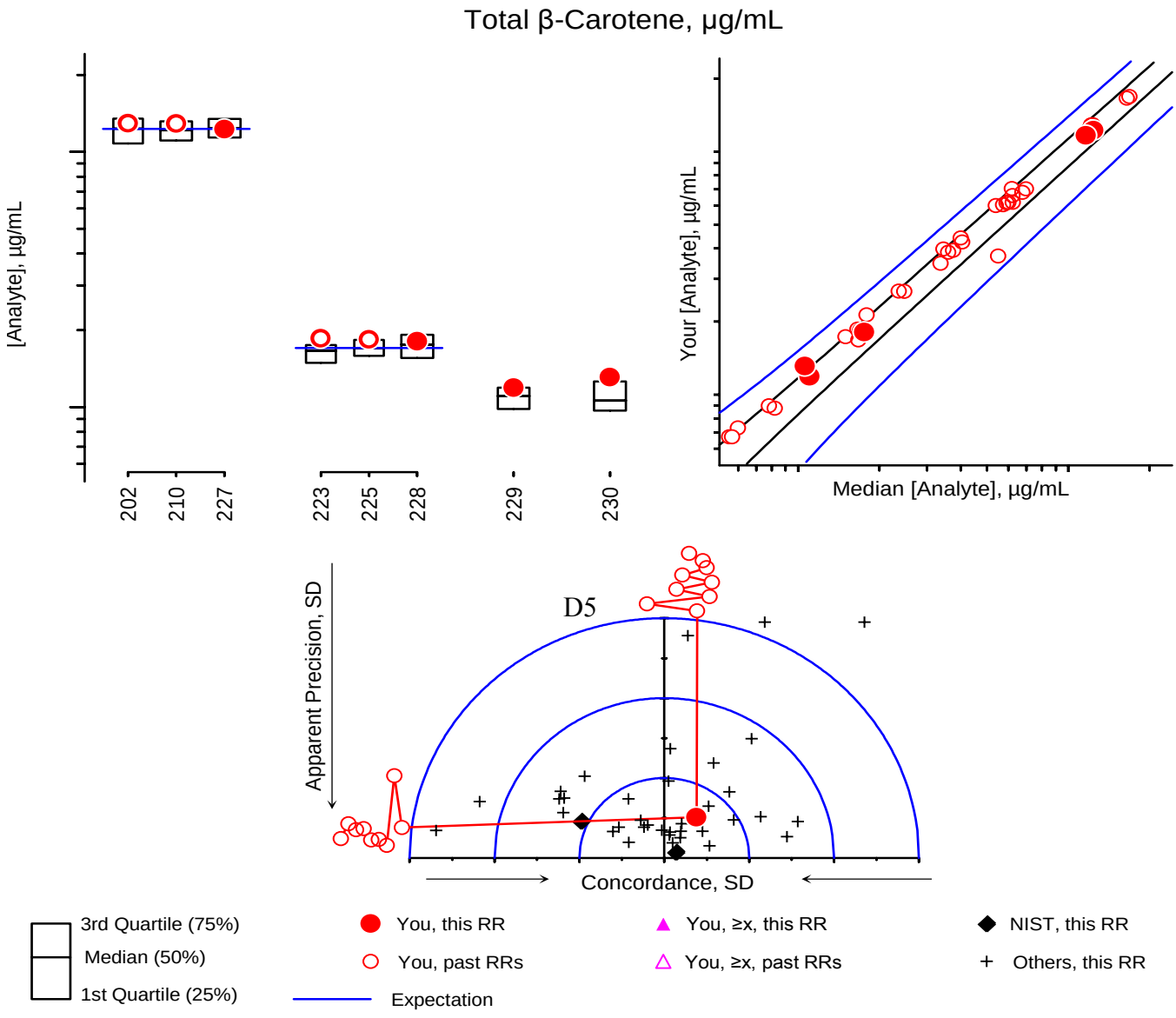

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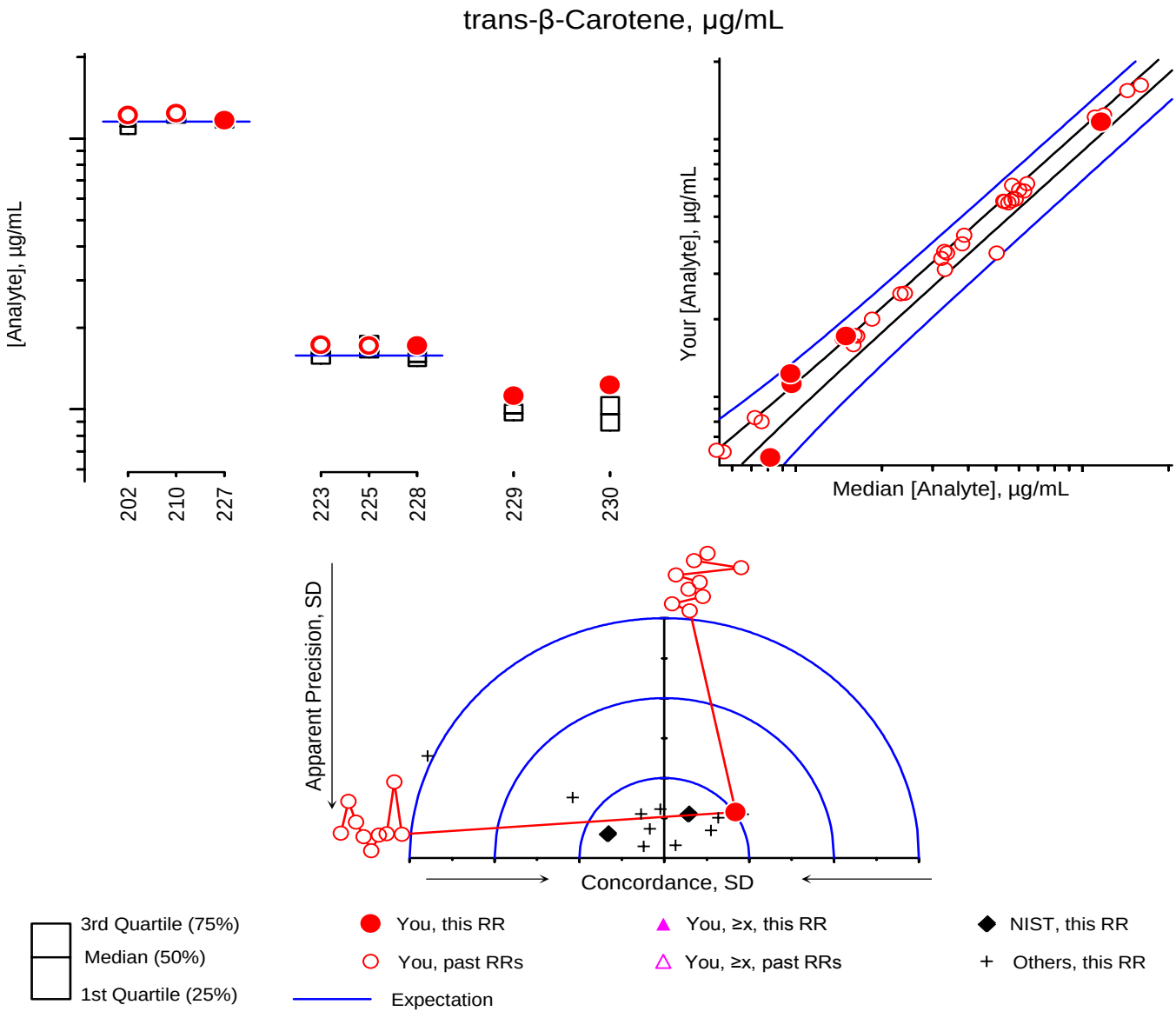

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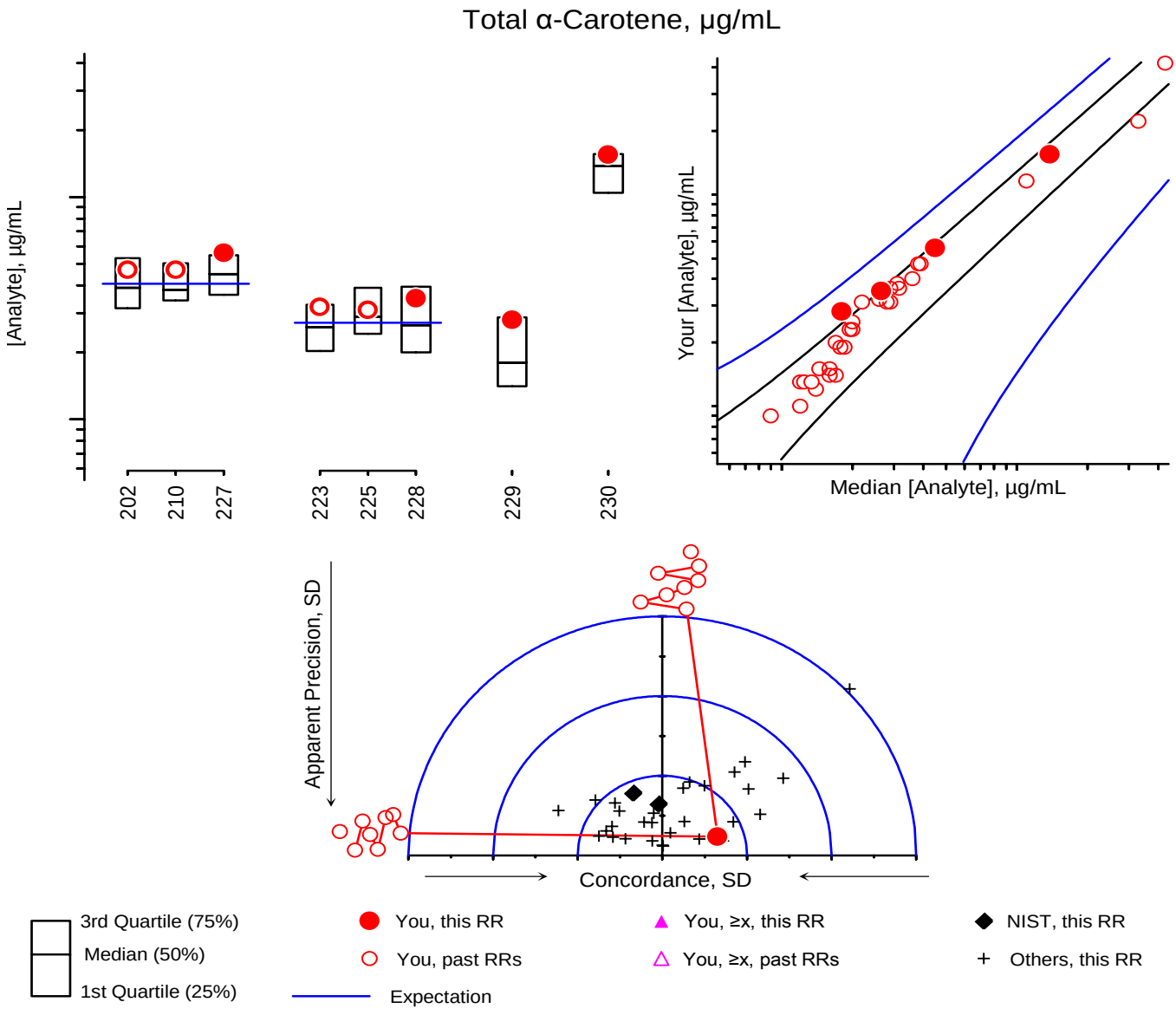

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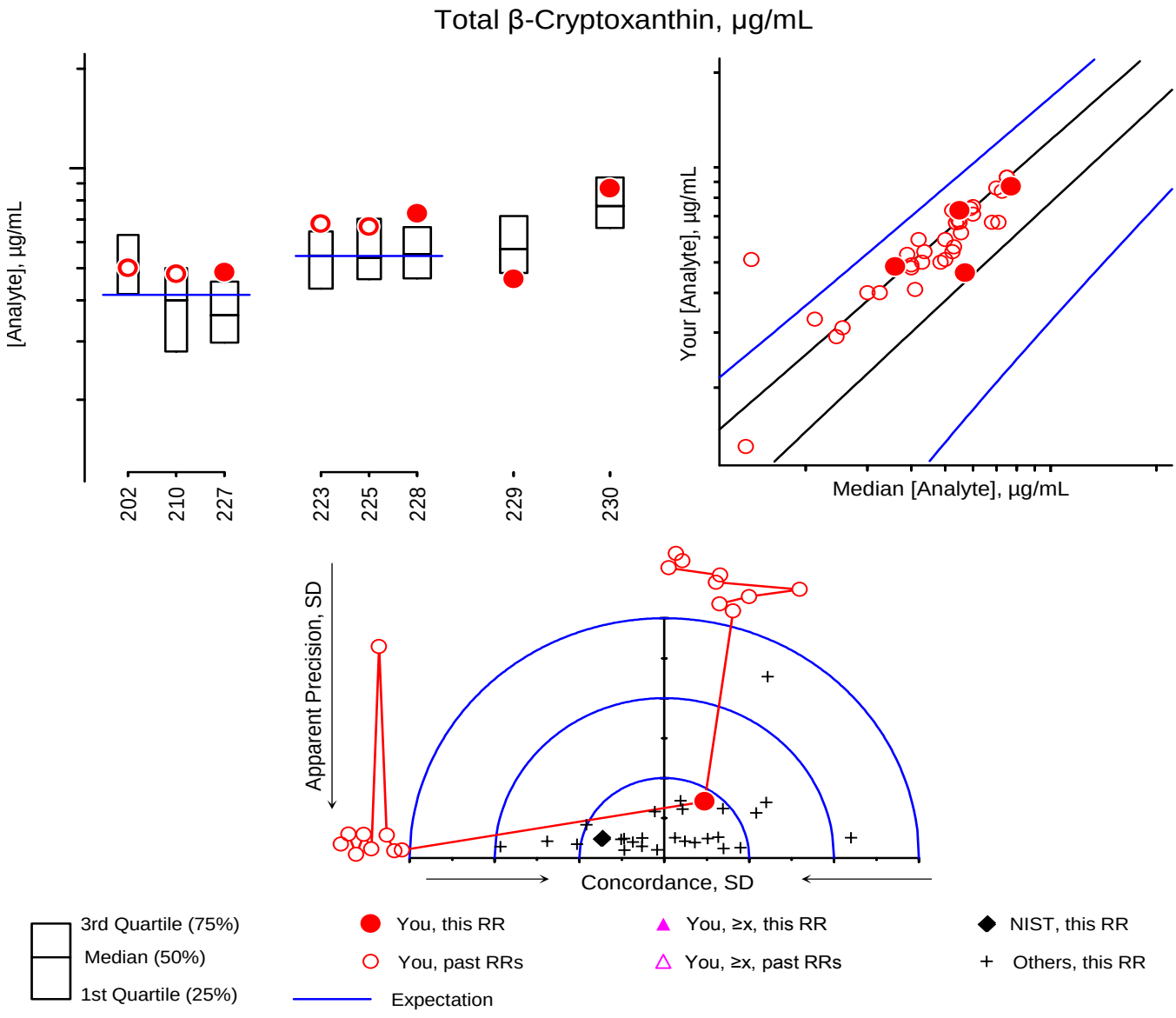

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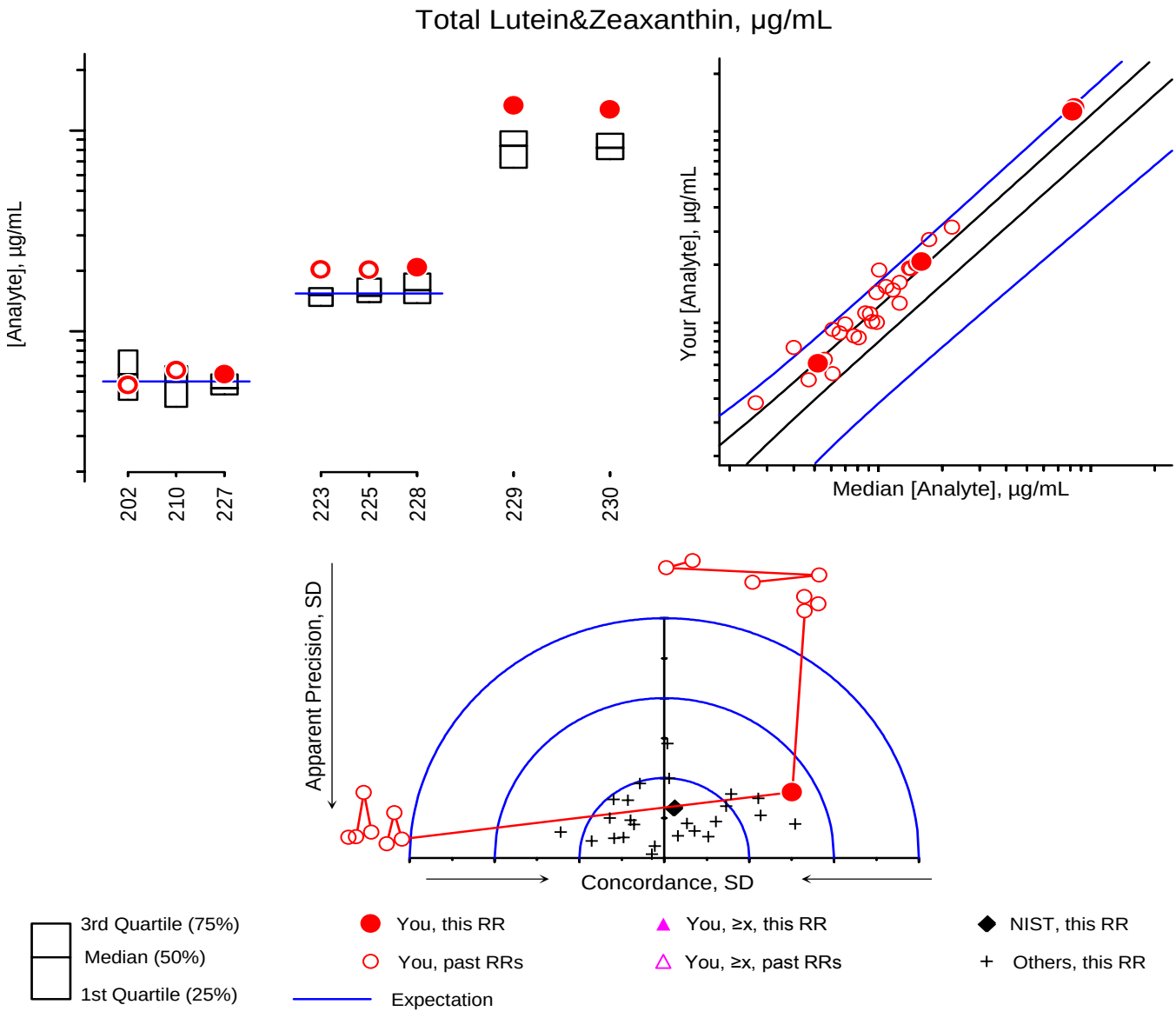

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Appendix E. Shipping Package Inserts for RR40

The following two items were included in each package shipped to an RR40 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-

April 7, 1997

Dear Colleague:

Enclosed is the set of samples for the second quality assurance round robin exercise (Round Robin XXXX) for FY97. 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 13, 1997. 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 25, 1997.

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, 1850 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 XXXX 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/NCI

Micronutrients Measurement Quality Assurance Program

Round Robin XL Results from Laboratory #_____

	Serum				
Analyte	231	232	233	234	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					
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 15, 1997

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

Appendix F. Final Report for RR40

The following 11 pages are the final report as provided to all participants:

- Cover letter and brief description of study design and overall results
- A "Report of Analysis" that:
  - 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
  - details the analysis of the NIST results

UNITED STATES DEPARTMENT OF COMMERC
National Institute of Standards and Technolog
Gaithersburg, Maryland 20899-0001

August 14, 1997

Dear Colleague:

Enclosed is the summary report of the results for Round Robin XL (RR40). 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.

The experimental design for this interlaboratory comparison exercise is summarized below. The four serum samples (Sera 231-234) distributed in RR40 address the following issues:

• **Serum and Measurement Stability.** Serum 234 is the mid level component of SRM® 968b. It was previously distributed as sera 201 and 209. Reanalysis of such well-characterized materials documents both the continued stability of the material and the "absolute accuracy" of the analyte level assignments. No significant changes in analyte concentrations were observed.

• **Analyte Augmentation Techniques.** Sera 231, 232, and 233 were prepared from the same serum pool as models for the proposed Serum 968c. We again had mixed success, achieving nearly our target levels with many of the minor analytes, but completely missing the mark with α-tocopherol. We will re-evaluate our augmentation techniques and continue to explore ways to achieve sufficient levels of the analytes of interest. We apologize for any inconvenience caused by our aggressive (and sometimes unsuccessful) augmentation experiments.

• **Measurement Precision and Concordance.** For several years now, we have been working toward a different evaluation/presentation of measurement quality. We now introduce the "Z-Score Concordance" plots. These are experimental and may be significantly changed/deleted, but they were received with some interest at the QA Workshop in Atlanta.

The concepts necessary for "properly" evaluating measurement quality are debatable. While we are still struggling with both concepts and words, here is a synopsis. The common basic concept is "accuracy" – usually interpreted as "closeness to truth" (or at least to a knowable "limiting mean"). Through repeated measurements over time on the same material(s), two different aspects of measurement accuracy can be identified: "bias" and "precision." "Bias" can be interpreted as the average difference from "truth; "precision" can be interpreted as the amount of scatter in the individual differences. We've used these words in the "Individualized Report," estimating "accuracy" as the average

$$\% \text{ Bias} = 100*(\text{NAV} - \text{your value}) / \text{NAV}$$

taken over all the sera in the Round Robin and estimating "precision" as the standard deviation of the % bias values.

The primary goal of the program is to get all participants to measure usefully similar values for different types of serum samples. Thus, for our purposes, concordance is how well the laboratories agree among themselves. As with accuracy, concordance also has two components. Rather than invent new words, we use "Average Concordance" for the "on average, how close?" component and "Concordance Consistency" for the "how much scatter?" component.

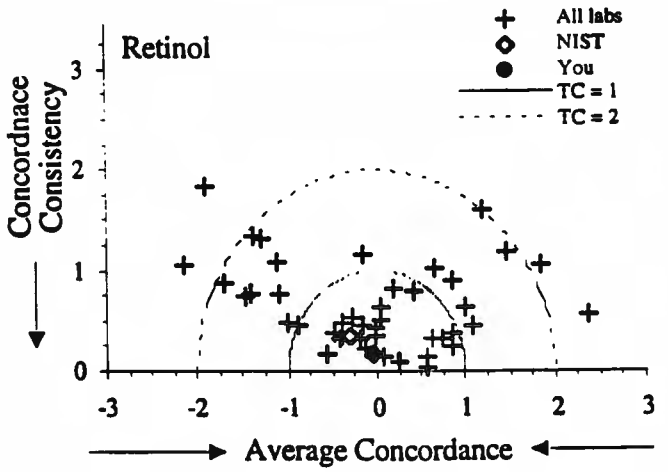
The estimated expected uncertainties are used to assign the NIST Assigned Uncertainty or "NAU" values; the NAU values are used in calculating the "Comparability Summary" scores. The "comparability" scores are based on your standardized values (StV):

$$StV = (\text{Your value} - NAV)/NAU$$

This is a specific case of "Z-score" standardization. We have chosen to define the "Z-Score Concordance" values as:

$$ZSC = (\text{Your Value} - \text{Median})/NAU$$

to remove any "NIST bias." The "Average Concordance" is the average of the ZSC values over all the sera analyzed, and the "Concordance Consistency" is the standard deviation of the ZSC values. As a minor complication, we average across related analytes (total and *trans* β-carotene and lycopene) to allow more information to be compressed onto a single page.



Each of the analyte (or analyte group) plots shows the total "Z-Score Concordance" values for all participants in RR40. Your values are shown as a black square, and the two NIST analysts' values are shown as open circles. Ideally, everyone should cluster very close to the "0" value: Average Concordance of zero, Concordance Consistency of zero. As a rough guide, we believe that the "good" values fall inside the inner semicircle.

$$TC = \text{Total Concordance} = \sqrt{(\text{AverageConcordance})^2 + (\text{ConcordanceConsistency})^2} = 1$$

However, while there is a tendency for consistency to decrease as the average moves away from zero, there are some laboratories that are very consistent yet rather far away from the median. Since this is clearly better than being high on one sample and low on another, we are still considering how best to "grade" the Total Concordance values.

Data for evaluating laboratory performance in RR40 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 or were rated "POOR" based on the criteria stated above, 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.

The Micronutrients Measurement QA Workshop was held prior to the American Association Clinical Chemistry (AACC) meeting on July 20 at the Best Western American Hotel in Atlanta, Georgia. At the workshop there was some discussion regarding fundamental changes for future presentation of results and the identification of sources of bias among the laboratories. Regarding the latter, program participants will be asked to provide updated information on methodology, techniques, and other chromatographic parameters more routinely to help refine our database. Participants also expressed their interests in having materials characterized for folates, phytoestrogens, homocysteine, polyamines, and curcumin. Primary standards for compounds typically measured in the program (such as lycopene) were also on the "wish" list.

Based on the workshop attendance, it appears that most of the veteran QA participants generally do not attend the AACC meeting. Since there was greater attendance when the workshop was held with the Experimental Biology meeting in 1996, it was suggested that our next workshop be held in conjunction with the 1999 Experimental Biology meeting in Washington, DC. We will keep you informed of the workshop plans as they are finalized.

We also plan to hold the **Fat-Soluble Vitamin and Carotenoid Analysis Tutorial** again this fall at NIST, provided there is adequate interest. The scheduled date for the session is **October 27, 1997.** This tutorial is intended primarily for new laboratories, new laboratory personnel, or those currently experiencing difficulties with their analysis. As in past years, this session will include a discussion of calibration, sample preparation, and chromatographic techniques for measuring fat-soluble vitamins and carotenoids in serum.

Enclosed are copies of the following publications:

- **Liquid Chromatographic Measurement of L-Ascorbic Acid and D-Ascorbic Acid in Biological Samples**, Margolis, S.A. and Schapira, R.M., *J. Chromatogr. B*, 690 (1997) 25-33.

- **Certification of Nutrients in Standard Reference Material 1846: Infant Formula**, Sharpless, K. E., Schiller, S.B., Margolis, S.A., Brown Thomas, J., Iyengar, G.V., Gills, T.E., Wise, S.A., Tanner, J.T., and Wolf, W.R., *J. AOAC*, 80 (1997) 611-21.

Other recent publications of note are:

- **Reference values for retinol, tocopherol, and main carotenoids in serum of control and insulin-dependent diabetic Spanish subjects.** Olmedilla B., Granado F., Gil-Martinez E., Blanco I., and Rojas-Hidalgo E. *Clin. Chem.* 1997:43(6);1066-1071. Prof. Olmedilla can be contacted at: bolmed@nutr.cph.es or Servicio de Nutriciòn, Clinica Puerta de Hierro, 28035 Madrid, Spain.

- **NHANES III, Reference Manuals and Reports CD-ROM** (October 1996), National Center for Health Statistics, Centers for Disease Control and Prevention, Hyattsville, MD 20782, Phone: (301) 436-8500, FAX: (301) 436-4258; e-mail: SETS@nch10a.em.cdc.gov.

Samples for RR41 were shipped during the last week of July. Results are due September 19; feedback to labs will be provided around October 27.

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 N^2M^2QAP Round Robin XL (RR40) Feedback includes the standard "All Lab" report:

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

The order of many components of the "Individualized" report has been changed to allow easy deletion of "empty" %bias plots. Also, we've added an experimental set of "concordance" charts to help you evaluate the performance characteristics of your measurements.

Page	"Individualized" Report Contents
1	Your values, our assigned values, and the %bias between the two
2, 3	"Comparisons" plots for: retinol, retinyl palmitate, α- and γ-tocopherol, total and *trans*-β-carotene, total α-carotene, total lycopene, β-cryptoxanthin, lutein, and lutein&zeaxanthin
4	"Z-Score Concordance" plots for this RR's: retinol, α-tocopherol, γ-tocopherol, total and *trans*-β-carotene, total α-carotene, and total and *trans*-lycopene.
5	% Bias Accuracy/Precision Summary for your last 3 years' results
6	Our assigned value vs. your value scatterplots for retinol, α- and γ-tocopherol, and total and *trans*-β-carotene, also for your last 3 years' results
7-9	% Bias barchart for retinol, α- and γ-tocopherol, and/or total and *trans*-β-carotene for your last 3 years' results. Only plots showing some data are included!

Analysis of N^2M^2QAP Round Robin XL Results: Sera ~~227 to 230~~ 231 to 234 -

Background: The following four samples were distributed in RR40

Sera 231-233: experimental sera, prepared from a single "low" serum pool and augmented with the various analytes to (try to) hit 10%, 50%, and 90% population levels. We did with some, not with others. We also over-spiked retinyl palmitate, to *try* to get a level that everyone could measure well.

Serum 234: the "middle level" component of SRM® 968b: Fat-Soluble Vitamins and Cholesterol in Human Serum. It was previously distributed as serum 201 in RR32 (9/94) and serum 209 in RR34 (6/95).

Qualitative Results: The following observations were noted in the RR40 reports:

Several participants obtained consistently low retinol values for the experimental sera, 231-233, while getting the mid-level 968b serum dead on. This suggests a systematic method-related bias. One of the workshop attendee's suggested that this may reflect differences in protein denaturation.

Action: This is a very important observation! We will re-examine old data, now that we know what to look for. But, to figure out the mechanism, we need your help! If you all are willing, we'd like to collect details on your total analytical method. We'll try to figure out the least painful way to do this, both for you and for us, and get started on this in early Fall.

One participant noted that the amber vials are too small.

Action: Sigh. We've only been able to locate these 2.0 and 5.0 mL ambers, and the 5.0's are just to big for our production methods. Sorry. If someone can locate 2.5 to 3.0 mL ambers for a reasonable price, we'll consider changing...

One participant noted with enthusiasm our success at achieving a high β-cryptoxanthin levels.

Action: We're trying, we're trying...

Quantitative Results The following NIST Data and Value/Uncertainty Assignments table presents all NIST data, summary statistics for the NIST data, summary results for RR40, 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 ≥ SD_x, as sample replicates reduce the true degrees of freedom.

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

NIST Summary Statistics

n number of quantitative values for this analyte for this serum

Mean $(Mean_{NIST1} + Mean_{NIST3})/2$ or $Mean_{NIST3}$ for analytes that NIST1 did not report

SD_{rep} within-vial pooled standard deviation

SD_{bet} 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 or, for analytes that NIST1 did not report, to the interlaboratory Median (see below). 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} $\sqrt{SD_{rep}^2 + SD_{bet}^2 + SD_{anl}^2}$, total standard deviation for NIST analyses.

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

Round Robin XL 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)

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

This is the approximate probability that the current median is the same as it was in its initial distribution. 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 smaller than it was in its initial distribution. 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 $\text{Maximum}(0.05 \times \text{NAV}, \sqrt{\text{SD}_{\text{NIST}}^2 + \text{SD}_{\text{labs}}^2})$, our best guess for the "true" interlaboratory standard deviation characterizing measurement, sample heterogeneity, interanalyst, and interlaboratory sources of variation. When SD_{labs} could not be determined, NAU is estimated as $\text{Maximum}(0.10 \times \text{NAV}, \sqrt{2\ \text{SD}_{\text{NIST}}^2})$.

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

Measurement Performance Summary: The following is based on results presented in the NIST Data and Value/Uncertainty Assignments table of this report and the "Individualized Report" page 2 and 3 summary graphs:

Serum 234, mid-level SRM 968b, stability: There is no significant difference in the level or measurement variability of any analyte among the three analyses of this material (sera 201, 209, and 234).

Action: Thankfully, none required.

Sera 231 - 233 homogeneity: It is probable that these materials are less homogenous than desired, particularly for α-tocopherol where considerably less was observed than was spiked!

Action: This is an ongoing problem and major concern. We are considering revising the various "grade sheets" for this RR40, perhaps just to reflect the SRM 968b values. More on this in the RR41 package! Only enough of these sera were produced for this one RR, so you won't see them again.

Retinyl palmitate: The CVs for *all* sera are 20-30%, nearly independent of analyte level.

Action: RP remains analytically challenging! The community needs to develop better techniques if this analyte is of importance. For now, don't believe any RP numbers!

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NIST Data and Value/Uncertainty Assignments

Retinol

	NIST1				NIST3			
	231	232	233	234	231	232	233	234
A:1	0.251	0.349	0.807	0.467	0.250	0.421	0.783	0.506
A:2	0.275	0.333	0.786	0.481	0.263	0.443	0.812	
B:1	0.261	0.351	0.748	0.488	0.263	0.425	0.789	0.504
B:2	0.250	0.495	0.738	0.517	0.256	0.419	0.784	0.495
C:1	0.243	0.401	0.808	0.475	0.264	0.417	0.775	0.499
C:2	0.224	0.489	0.794	0.483	0.258	0.403	0.734	0.496
n_x	6	6	6	6	6	6	6	5
$Mean_x$	0.251	0.403	0.780	0.485	0.259	0.421	0.779	0.500
SD_x	0.017	0.073	0.030	0.017	0.005	0.013	0.025	0.005
SD_{repx}	0.013	0.069	0.011	0.013	0.006	0.011	0.021	0.004
SD_{betx}	0.015	0.055	0.032	0.015	0.002	0.011	0.022	0.004
SD_{NISTx}	0.021	0.088	0.034	0.020	0.007	0.015	0.030	0.006
CV_{NISTx}	8.2	22	4.4	4.2	2.7	3.6	3.9	1.2

Retinyl Palmitate

	NIST1				NIST3			
	231	232	233	234	231	232	233	234
					0.84	1.31	0.213	0.161
					0.88	1.28	0.151	0.155
					1.07	1.38	0.160	0.141
					1.24	1.45	0.143	0.169
					1.07	1.48	0.224	0.154
						1.50	0.199	0.128
n_x	0	0	0	0	5	6	6	6
$Mean_x$					1.02	1.40	0.182	0.151
SD_x					0.16	0.09	0.034	0.015
SD_{repx}					0.07	0.03	0.028	0.016
SD_{betx}					0.15	0.10	0.030	0.009
SD_{NISTx}					0.16	0.10	0.041	0.018
CV_{NISTx}					16	7.4	22	12

	NIST			
n	12	12	12	11
Mean	0.255	0.412	0.780	0.493
SD_{rep}	0.011	0.049	0.012	0.010
SD_{bet}	0.010	0.035	0.024	0.011
SD_{mi}	0.010	0.010	0.010	0.010
SD_{NIST}	0.018	0.061	0.029	0.018
CV_{NIST}	7.0	15	3.7	3.6

NIST3=a+b*NIST1

a: 0.020 ±0.014
b: 0.977 ±0.026
R^2: 0.999

	NIST			
	5	6	6	6
	1.02	1.40	0.182	0.151
	0.10	0.04	0.027	0.016
	0.15	0.10	0.030	0.009
	0.03	0.03	0.034	0.034
	0.18	0.11	0.053	0.039
	18	8.1	29	26

NIST3=a+b*Median

a: -0.065 ±0.041
b: 1.379 ±0.053
R^2: 0.997

RR	XXXIV
Serum	209
n_p	48
$Median_p$	0.530
eSD_p	0.050

	XXXIV
	209
	13
	0.19
	0.02

	RRXL			
	231	232	233	234
n_a	42	43	43	43
$Median_a$	0.265	0.421	0.770	0.509
eSD_a	0.034	0.048	0.058	0.033
P(n=p)				0.92
P(n<p)				1.00
SD_{labs}	0.029	0	0.050	0.028
CV_{labs}	11	0	6.5	5.6
NAV	0.260	0.417	0.775	0.501
NAU	0.034	0.061	0.058	0.033
CV	13	15	7.5	6.7
xCV	10	9.0	8.8	8.8

<— Current Results —>

<— Assignments —>

	RRXL			
	231	232	233	234
	14	14	14	14
	0.77	1.07	0.186	0.179
	0.15	0.32	0.043	0.025
				0.79
				0.07
	0	0.30	0	0
	0	28	0	0
	0.89	1.24	0.184	0.165
	0.18	0.32	0.053	0.039
	20	26	29	23
	20	20	24	25

NIST Data and Value/Uncertainty Assignments

α-Tocopherol

	NIST1				NIST3			
	231	232	233	234	231	232	233	234
A:1	4.55	5.73	8.53	9.41	4.46	6.51	7.39	9.35
A:2	5.43	5.87	7.94	9.32	4.31	6.02	7.46	
B:1	5.00	5.91	8.05	9.48	4.94	7.05	8.20	10.21
B:2	4.62	8.14	7.88	9.95	4.93	7.01	7.91	10.05
C:1	4.56	6.78	7.48	9.45	4.89	6.68	7.94	9.80
C:2	4.16	7.47	8.08	9.44	4.94	6.68	7.30	9.78
n_x	6	6	6	6	6	6	6	5
$Mean_x$	4.72	6.65	8.00	9.51	4.74	6.66	7.70	9.84
SD_x	0.44	0.99	0.34	0.22	0.28	0.38	0.36	0.32
SD_{rep}	0.42	0.95	0.35	0.20	0.06	0.20	0.29	0.06
SD_{bet}	0.32	0.74	0.23	0.18	0.31	0.38	0.32	0.39
SD_{NISTx}	0.53	1.20	0.42	0.27	0.32	0.43	0.43	0.39
CV_{NISTx}	11	18	5.3	2.8	6.7	6.5	5.6	4.0

	NIST			
n	12	12	12	11
Mean	4.73	6.65	7.85	9.67
SD_{rep}	0.30	0.69	0.28	0.15
SD_{bet}	0.27	0.49	0.24	0.43
SD_{mi}	0.14	0.14	0.14	0.14
SD_{NIST}	0.43	0.86	0.39	0.48
CV_{NIST}	9.1	13	5.0	4.9

NIST3=a+b*NIST1

a: 0.50 ±0.39
b: 0.91 ±0.06
R^2: 0.991

RR	XXXIV
Serum	209
n_p	47
$Median_p$	9.87
eSD_p	0.85

	RRXL			
	231	232	233	234
n_q	39	39	39	39
$Median_q$	4.87	6.80	7.89	9.68
eSD_q	0.68	0.58	0.86	0.71
P(n=p)				0.96
P(n<p)				0.88
SD_{labs}	0.53	0	0.76	0.52
CV_{labs}	11	0	9.7	5.4
NAV	4.80	6.73	7.87	9.67
NAU	0.68	0.86	0.86	0.71
CV	14	13	11	7.3
xCV	9.8	8.1	7.6	7.3

γ-Tocopherol

	NIST1				NIST3			
	231	232	233	234	231	232	233	234
A:1	1.46	2.33	0.722	2.08	1.41	2.64	0.689	2.25
A:2	1.46	2.36	0.703	2.04	1.39	2.57	0.725	
B:1	1.46	2.41	0.707	2.17	1.47	2.76	0.764	2.34
B:2	1.39	3.32	0.725	2.34	1.46	2.75	0.740	2.31
C:1	1.34	2.83	0.747	2.15	1.46	2.57	0.767	2.23
C:2	1.45	3.01	0.772	2.15	1.45	2.58	0.703	2.26
n_x	6	6	6	6	6	6	6	5
$Mean_x$	1.43	2.71	0.729	2.16	1.44	2.65	0.731	2.28
SD_x	0.05	0.41	0.026	0.10	0.03	0.09	0.032	0.05
SD_{rep}	0.05	0.38	0.015	0.07	0.01	0.03	0.031	0.02
SD_{bet}	0.03	0.32	0.026	0.10	0.04	0.10	0.023	0.04
SD_{NISTx}	0.06	0.50	0.030	0.12	0.04	0.10	0.039	0.05
CV_{NISTx}	4.4	18	4.1	5.7	2.6	3.8	5.3	2.1

	NIST			
	12	12	12	11
	1.43	2.68	0.730	2.22
	0.04	0.27	0.018	0.05
	0.03	0.20	0.023	0.11
	0.03	0.03	0.029	0.03
	0.06	0.34	0.041	0.12
	4.1	13	5.7	5.6

NIST3=a+b*NIST1

a: 0.044 ±0.038
b: 0.963 ±0.021
R^2: 0.999

	XXXIV
	209
	23
	2.39
	0.24

	RRXL			
	231	232	233	234
	23	23	23	23
	1.36	2.85	0.770	2.29
	0.15	0.31	0.105	0.22
				0.90
				0.65
	0.14	0	0.096	0.19
	10	0	12	8.2
	1.40	2.77	0.750	2.25
	0.15	0.34	0.105	0.22
	11	12	14	9.9
	10	7.9	15	8.3

<—— Current Results ——>

<—— Assignments ——>

[40_dat]NISTtxt

NIST Data and Value/Uncertainty Assignments

δ-Tocopherol

	NIST1				NIST3			
	231	232	233	234	231	232	233	234
A:1	1.10				1.13	0.213	0.209	0.162
A:2	1.18				1.12	0.196	0.235	
B:1	1.15				1.16	0.259	0.265	0.181
B:2	1.13				1.15	0.242	0.238	0.182
C:1	1.15				1.16	0.230	0.247	0.167
C:2					1.16	0.237	0.242	0.170
n_x	5	0	0	0	6	6	6	5
$Mean_x$	1.14				1.15	0.229	0.239	0.172
SD_x	0.03				0.02	0.022	0.018	0.009
SD_{rep_x}	0.03				0.00	0.010	0.015	0.001
SD_{het_x}	0.01				0.02	0.023	0.015	0.010
SD_{NIST_x}	0.03				0.02	0.025	0.022	0.010
CV_{NIST_x}	3.0				1.8	11	9.0	5.8

	NIST			
n	11	6	6	5
Mean	1.15	0.229	0.239	0.172
SD_{rep}	0.02	0.012	0.019	0.000
SD_{het}	0.02	0.023	0.015	0.010
SD_{lab}	0.00	0.003	0.003	0.003
SD_{NIST}	0.03	0.026	0.024	0.010
CV_{NIST}	2.7	11	10	6.0

NIST3=a+b*Median

a: 0.050 ±0.003
b: 1.040 ±0.004
R^2: 1.000

RR	XXXIV
Serum	209
n_p	3
$Median_p$	0.21
eSD_p	

	RRXL			
	231	232	233	234
n_a	4	3	3	3
$Median_a$	1.06	0.170	0.184	0.116
eSD_a	0.12	0.006	0.011	0.031
P(n=p)				
P(n<p)				
SD_{labs}	0.11	0	0	0.030
CV_{labs}	11	0	0	26

NAV	1.10	0.200	0.212	0.144
NAU	0.12	0.026	0.024	0.031
CV	11	13	12	22
xCV				

Total β-Carotene

	NIST1				NIST3			
	231	232	233	234	231	232	233	234
A:1	0.076	0.185	0.453	0.643	0.071	0.202	0.392	0.606
A:2	0.076	0.269	0.446	0.589	0.067	0.211	0.405	
B:1	0.072	0.230	0.445	0.677	0.086	0.236	0.472	0.648
B:2	0.069	0.224	0.460	0.657	0.083	0.255	0.463	0.652
C:1	0.071	0.231	0.414	0.641	0.078	0.204	0.434	0.642
C:2	0.079	0.243	0.466	0.642	0.075	0.235	0.441	0.642
n_x	6	6	6	6	6	6	6	5
$Mean_x$	0.074	0.230	0.447	0.642	0.077	0.224	0.434	0.638
SD_x	0.004	0.027	0.018	0.029	0.007	0.021	0.032	0.019
SD_{rep_x}	0.004	0.035	0.022	0.024	0.003	0.015	0.007	0.001
SD_{het_x}	0.003	0.006	0.007	0.025	0.008	0.020	0.035	0.024
SD_{NIST_x}	0.005	0.035	0.023	0.035	0.008	0.025	0.035	0.024
CV_{NIST_x}	6.2	15	5.2	5.4	11	11	8.1	3.7

	NIST			
	12	12	12	11
	0.075	0.227	0.441	0.640
	0.003	0.026	0.016	0.017
	0.005	0.013	0.022	0.023
	0.002	0.002	0.002	0.002
	0.007	0.029	0.028	0.028
	8.7	13	6.3	4.4

NIST3=a+b*NIST1

a: 0.005 ±0.003
b: 0.959 ±0.009
R^2: 1.000

	XXXIV
	209
	37
	0.617
	0.103

	RRXL			
	231	232	233	234
	25	25	25	25
	0.074	0.219	0.494	0.610
	0.013	0.050	0.083	0.080
				0.98
				0.90
	0.011	0.040	0.078	0.075
	15	18	16	12

	0.075	0.223	0.467	0.625
	0.013	0.050	0.083	0.080
	17	22	18	13
	24	18	15	14

NIST Data and Value/Uncertainty Assignments

trans-β-Carotene

	NIST1				NIST3			
	231	232	233	234	231	232	233	234
A:1	0.071	0.182	0.437	0.512	0.060	0.185	0.357	0.557
A:2	0.074	0.258	0.377	0.539	0.056	0.182	0.367	
B:1	0.070	0.186	0.339	0.587	0.072	0.216	0.431	0.599
B:2	0.065	0.212	0.371	0.524	0.073	0.230	0.428	0.589
C:1	0.062	0.210	0.355	0.538	0.061	0.190	0.404	0.592
C:2	0.076	0.187	0.358	0.553	0.069	0.212	0.404	0.593
n_x	6	6	6	6	6	6	6	5
$Mean_x$	0.070	0.206	0.373	0.542	0.065	0.202	0.399	0.586
SD_x	0.005	0.029	0.034	0.026	0.007	0.020	0.031	0.017
SD_{rep}	0.006	0.034	0.028	0.028	0.004	0.011	0.004	0.004
SD_{het}	0.003	0.012	0.029	0.015	0.007	0.020	0.034	0.021
SD_{NISTx}	0.007	0.036	0.041	0.032	0.008	0.022	0.034	0.022
CV_{NISTx}	9.8	18	11	5.9	13	11	8.6	3.7

	NIST			
n	12	12	12	11
Mean	0.067	0.204	0.386	0.564
SD_{rep}	0.005	0.025	0.020	0.021
SD_{het}	0.006	0.016	0.030	0.017
SD_{sal}	0.010	0.010	0.010	0.010
SD_{NIST}	0.013	0.031	0.037	0.029
CV_{NIST}	19	15	9.7	5.1

NIST3=a+b*NIST1

a: -0.016 ±0.012
b: 1.102 ±0.048
R^2: 0.996

RR	XXXIV
Serum	209
n_p	11
$Median_p$	0.603
eSD_p	0.046

	RRXL			
	231	232	233	234
n_a	10	10	10	10
$Median_a$	0.068	0.233	0.473	0.570
eSD_a	0.009	0.059	0.062	0.049
P(n=p)				0.77
P(n<p)				0.42
SD_{labs}	0	0.050	0.050	0.040
CV_{labs}	0	22	10	7.1
NAV	0.068	0.218	0.429	0.567
NAU	0.013	0.059	0.062	0.049
CV	18	27	14	8.7
xCV	13	9.4	9.1	9.1

Total α-Carotene

	NIST1				NIST3			
	231	232	233	234	231	232	233	234
A:1	0.052	0.024	0.107	0.043	0.045	0.018	0.129	0.039
A:2	0.050	0.025	0.104	0.046	0.041	0.023	0.117	
B:1	0.052	0.025	0.097	0.034	0.057	0.020	0.123	0.045
B:2	0.053	0.026	0.108	0.036	0.056	0.024	0.112	0.052
C:1	0.050	0.027	0.104	0.035	0.059	0.020	0.107	0.042
C:2	0.047	0.025	0.082	0.032	0.054	0.026	0.113	0.049
n_x	6	6	6	6	6	6	6	5
$Mean_x$	0.051	0.026	0.100	0.038	0.052	0.022	0.117	0.046
SD_x	0.002	0.001	0.010	0.005	0.007	0.003	0.008	0.005
SD_{rep}	0.002	0.001	0.010	0.002	0.003	0.004	0.007	0.004
SD_{het}	0.002	0.001	0.007	0.006	0.008	0.001	0.007	0.005
SD_{NISTx}	0.003	0.001	0.012	0.006	0.008	0.004	0.010	0.006
CV_{NISTx}	5.3	5.2	12	16	15	18	8.3	14

	NIST			
	12	12	12	11
	0.051	0.024	0.108	0.042
	0.002	0.003	0.009	0.003
	0.005	0.001	0.007	0.007
	0.002	0.002	0.002	0.002
	0.006	0.003	0.011	0.008
	11	14	11	19

NIST3=a+b*NIST1

a: -0.012 ±0.002
b: 1.277 ±0.030
R^2: 0.999

	XXXIV
	209
	23
	0.028
	0.011

	RRXL			
	231	232	233	234
	26	24	26	26
	0.047	0.015	0.107	0.029
	0.014	0.005	0.029	0.008
				0.98
				0.94
	0.013	0.004	0.027	0
	28	29	25	0
	0.049	0.019	0.108	0.035
	0.014	0.005	0.029	0.008
	29	28	27	22
	28	32	26	29

<— Current Results —>

<—— Assignments ——>

[40_dat]NISTtxt

NIST Data and Value/Uncertainty Assignments

trans-α-Carotene

	NIST1				NIST3			
	231	232	233	234	231	232	233	234
A:1					0.04	0.015	0.12	0.02
A:2					0.04	0.014	0.10	
B:1					0.05	0.016	0.11	0.03
B:2					0.05	0.018	0.10	0.03
C:1					0.04	0.017	0.10	0.03
C:2					0.05	0.019	0.10	0.03
n_x	0	0	0	0	6	6	6	5
$Mean_x$					0.05	0.016	0.11	0.03
SD_x					0.01	0.002	0.01	0.00
SD_{rep_x}					0.00	0.001	0.01	0.00
SD_{bet_x}					0.01	0.002	0.01	0.00
SD_{NIST_x}					0.01	0.002	0.01	0.00
CV_{NIST_x}					14	15	8.8	16

Total Lycopene

	NIST1				NIST3			
	231	232	233	234	231	232	233	234
A:1					0.198	0.218	0.176	0.311
A:2					0.192	0.205	0.192	
B:1					0.260	0.270	0.244	0.357
B:2					0.268	0.295	0.233	0.350
C:1					0.257	0.225	0.186	0.313
C:2					0.244	0.237	0.222	0.333
n_x	0	0	0	0	6	6	6	5
$Mean_x$					0.236	0.242	0.209	0.333
SD_x					0.033	0.034	0.028	0.021
SD_{rep_x}					0.007	0.012	0.017	0.009
SD_{bet_x}					0.037	0.037	0.028	0.022
SD_{NIST_x}					0.037	0.039	0.032	0.024
CV_{NIST_x}					16	16	15	7.1

NIST

n	6	6	6	5
Mean	0.05	0.016	0.11	0.03
SD_{rep}	0.00	0.001	0.01	0.00
SD_{bet}	0.01	0.002	0.01	0.00
SD_{tot}				
SD_{NIST}				
CV_{NIST}				

NIST

	6	6	6	5
	0.236	0.242	0.209	0.333
	0.005	0.015	0.009	0.004
	0.037	0.037	0.028	0.022
	0.019	0.019	0.019	0.019
	0.042	0.044	0.035	0.030
	18	18	17	8.9

NIST3=a+b*Median

a: 0
b: 1.107 ±0.054
R^2: 0.046

RR	XXXIV
Serum	209
n_p	2
$Median_p$	
eSD_p	

XXXIV
209
24
0.276
0.059

RRXL

	231	232	233	234
n_n	0	0	0	0
$Median_n$				
eSD_n				
P(n=p)				
P(n<p)				
SD_{labs}				
CV_{labs}				

<— Current Results —>

RRXL

231	232	233	234
26	26	26	26
0.193	0.231	0.194	0.272
0.050	0.060	0.049	0.077
			0.99
			0.10
0.027	0.040	0.034	0.072
14	17	18	26

NAV	0.05	0.016	0.11	0.03
NAU				
CV				
xCV				

<— Assignments —>

0.215	0.236	0.201	0.302
0.050	0.060	0.049	0.077
23	25	24	26
26	26	26	25

NIST Data and Value/Uncertainty Assignments

trans-Lycopene

	NIST1				NIST3			
	231	232	233	234	231	232	233	234
A:1					0.087	0.105	0.081	0.136
A:2					0.084	0.102	0.089	
B:1					0.108	0.136	0.103	0.158
B:2					0.111	0.148	0.105	0.155
C:1					0.100	0.112	0.092	0.148
C:2					0.103	0.119	0.097	0.147
n_x	0	0	0	0	6	6	6	5
$Mean_x$					0.099	0.120	0.094	0.149
SD_x					0.011	0.018	0.009	0.009
SD_{repx}					0.002	0.006	0.004	0.001
SD_{betx}					0.012	0.020	0.010	0.010
SD_{NISTx}					0.012	0.021	0.011	0.010
CV_{NISTx}					12	17	11	6.9

	NIST			
n	6	6	6	5
Mean	0.099	0.120	0.094	0.149
SD_{rep}	0.002	0.007	0.004	0.002
SD_{bet}	0.012	0.020	0.010	0.010
SD_{sal}	0.006	0.006	0.006	0.006
SD_{NIST}	0.014	0.022	0.012	0.012
CV_{NIST}	14	18	13	8.0

NIST3=a+b*Median

a: 0
b: 0.903 ±0.029
R^2: 0.670

RR	XXXIV
Serum	209
n_p	6
$Median_p$	0.177
eSD_p	0.047

	RRXL			
	231	232	233	234
n_e	7	7	7	7
$Median_n$	0.108	0.128	0.112	0.148
eSD_n	0.014	0.043	0.019	0.038
P(n=p)				0.71
P(n<p)				0.69
SD_{labs}	0.002	0.037	0.015	0.036
CV_{labs}	2.2	29	13	25
NAV	0.103	0.124	0.103	0.148
NAU	0.014	0.043	0.019	0.038
CV	13	34	18	26
xCV				

<— Current Results —>

<—— Assignments ——>

β-Cryptoxanthin

	NIST1				NIST3			
	231	232	233	234	231	232	233	234
A:1					0.032	0.096	0.225	0.023
A:2					0.030	0.090	0.241	
B:1					0.037	0.105	0.261	0.027
B:2					0.036	0.112	0.256	0.031
C:1					0.039	0.102	0.239	0.025
C:2					0.036	0.106	0.225	0.029
n_x	0	0	0	0	6	6	6	5
$Mean_x$					0.035	0.102	0.241	0.027
SD_x					0.003	0.008	0.015	0.003
SD_{repx}					0.001	0.004	0.009	0.002
SD_{betx}					0.003	0.008	0.015	0.003
SD_{NISTx}					0.004	0.009	0.018	0.003
CV_{NISTx}					11	8.9	7.4	13

	NIST			
	6	6	6	5
	0.035	0.102	0.241	0.027
	0.001	0.005	0.007	0.002
	0.003	0.008	0.015	0.003
	0.014	0.014	0.014	0.014
	0.014	0.017	0.022	0.014
	41	16	9.1	53

NIST3=a+b*Median

a: -0.016 ±0.016
b: 0.936 ±0.088
R^2: 0.983

	XXXIV
	209
	17
	0.043
	0.013

	RRXL			
	231	232	233	234
	22	22	22	22
	0.047	0.138	0.270	0.034
	0.008	0.026	0.045	0.013
				0.83
				0.57
	0	0.019	0.039	0
	0	14	15	0
	0.041	0.120	0.255	0.030
	0.014	0.026	0.045	0.014
	35	21	18	47
	29	23	20	31

NIST Data and Value/Uncertainty Assignments

"Lutein"

	NIST1				NIST3			
	231	232	233	234	231	232	233	234
A:1					0.143	0.240	0.052	0.067
A:2					0.142	0.242	0.056	
B:1					0.146	0.249	0.054	0.062
B:2					0.143	0.244	0.053	0.066
C:1					0.145	0.231	0.056	0.062
C:2					0.146	0.236	0.058	0.066
n_x	0	0	0	0	6	6	6	5
$Mean_x$					0.144	0.240	0.055	0.065
SD_x					0.002	0.006	0.002	0.002
SD_{repx}					0.001	0.003	0.002	0.002
SD_{betx}					0.002	0.006	0.002	0.001
SD_{NISTx}					0.002	0.007	0.003	0.002
CV_{NISTx}					1.3	2.9	4.7	3.8

	NIST			
n	6	6	6	5
Mean	0.144	0.240	0.055	0.065
SD_{rep}	0.001	0.003	0.002	0.002
SD_{bet}	0.002	0.006	0.002	0.001
SD_{anl}	0.001	0.001	0.001	0.001
SD_{NIST}	0.002	0.007	0.003	0.003
CV_{NIST}	1.5	2.9	4.6	4.1

NIST3=a+b*Median

a: 0
b: 0.933 ±0.002
R^2: 1.000

RR	XXXIV
Serum	209
n_p	10
$Median_p$	0.067
eSD_p	0.010

	RRXL			
	231	232	233	234
n_n	13	12	13	13
$Median_n$	0.154	0.258	0.059	0.062
eSD_n	0.079	0.065	0.010	0.019
P(n=p)				0.89
P(n<p)				0.04*
SD_{bbs}	0.079	0.065	0.010	0.018
CV_{bbs}	51	25	17	30
NAV	0.149	0.249	0.057	0.063
NAU	0.079	0.065	0.010	0.019
CV	53	26	18	29
xCV				

<— Current Results —>

<—— Assignments ——>

"Zeaxanthin"

	NIST1				NIST3			
	231	232	233	234	231	232	233	234
A:1					0.261	0.026	0.028	0.035
A:2					0.258	0.027	0.030	
B:1					0.270	0.030	0.031	0.033
B:2					0.263	0.030	0.029	0.036
C:1					0.266	0.027	0.029	0.029
C:2					0.266	0.028	0.029	0.033
n_x	0	0	0	0	6	6	6	5
$Mean_x$					0.264	0.028	0.029	0.033
SD_x					0.004	0.002	0.001	0.003
SD_{repx}					0.003	0.001	0.001	0.002
SD_{betx}					0.004	0.002	0.001	0.002
SD_{NISTx}					0.005	0.002	0.002	0.003
CV_{NISTx}					1.8	6.3	5.2	9.5

	NIST			
	6	6	6	5
	0.264	0.028	0.029	0.033
	0.004	0.001	0.001	0.002
	0.004	0.002	0.001	0.002
	0.001	0.001	0.001	0.001
	0.006	0.002	0.002	0.003
	2.2	8.0	7.5	9.6

NIST3=a+b*Median

a: -0.003 ±0.001
b: 1.114 ±0.008
R^2: 1.000

	XXXIV
	209
	6
	0.027
	0.001

	RRXL			
	231	232	233	234
	10	7	9	9
	0.240	0.027	0.030	0.030
	0.025	0.009	0.008	0.009
				0.83
				0.00*
	0.024	0.009	0.008	0.008
	10	32	26	28
	0.252	0.028	0.030	0.032
	0.025	0.009	0.008	0.009
	9.9	32	27	28

[40_dat]NISTxls

NIST Data and Value/Uncertainty Assignments

"Lutein&Zeaxanthin"

	NIST1				NIST3			
	231	232	233	234	231	232	233	234
A:1					0.404	0.267	0.080	0.102
A:2					0.400	0.269	0.086	
B:1					0.415	0.278	0.086	0.095
B:2					0.406	0.274	0.082	0.102
C:1					0.411	0.258	0.084	0.091
C:2					0.412	0.264	0.086	0.099
n_x	0	0	0	0	6	6	6	5
$Mean_x$					0.408	0.268	0.084	0.098
SD_x					0.006	0.007	0.003	0.005
SD_{repx}					0.004	0.003	0.003	0.004
SD_{betx}					0.005	0.008	0.001	0.003
SD_{NISTx}					0.007	0.008	0.003	0.005
CV_{NISTx}					1.6	3.1	3.8	5.5

	NIST			
n	6	6	6	5
Mean	0.408	0.268	0.084	0.098
SD_{rep}	0.006	0.003	0.003	0.004
SD_{bet}	0.005	0.008	0.001	0.003
SD_{mat}	0.021	0.021	0.021	0.021
SD_{NIST}	0.022	0.023	0.021	0.022
CV_{NIST}	5.5	8.4	25	22

NIST3=a+b*Median

a: 0

b: 1.101 ±0.047

R^2: 0.966

RR	XXXIV
Serum	209
n_p	17
$Median_p$	0.094
eSD_p	0.021

RRXL

	231	232	233	234
n_a	20	20	20	20
$Median_a$	0.355	0.266	0.077	0.086
eSD_a	0.079	0.085	0.019	0.017
P(n=p)				0.88
P(n<p)				0.78
SD_{bias}	0.075	0.082	0	0
CV_{bias}	21	31	0	0
NAV	0.381	0.267	0.081	0.092
NAU	0.079	0.085	0.021	0.022
CV	21	32	27	24
xCV				

Appendix G. "All-Lab Report" for RR40

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 XL Laboratory Results

Values in µg/mL

	Retinol				Retinyl Palmitate				α-Tocopherol				γ-Tocopherol				δ-Tocopherol			
Lab	231	232	233	234	231	232	233	234	231	232	233	234	231	232	233	234	231	232	233	234
FSV-BA	0.291	0.449	0.830	0.517	0.67	0.97	0.161	0.216	4.72	6.61	7.66	9.68	1.53	2.88	1.01	2.44				
FSV-BD	0.258	0.388	0.792	0.519					4.00	6.80	8.30	9.90								
FSV-BE		0.414	0.790	0.493						6.77	8.33	9.76		2.85	0.79	2.41				
FSV-BF	0.293	0.454	0.839	0.537					5.26	7.48	8.87	10.19	1.39	2.87	0.77	2.35				
FSV-BG	0.276	0.488	0.803	0.540	0.65	0.92	0.140	0.157	4.78	7.14	7.50	9.53	1.34	2.83	0.73	2.27				
FSV-BGa	0.329	0.536	0.964	0.600	0.85	1.09	0.166	0.159	5.06	7.34	8.54	10.24	1.33	2.78	0.78	2.31				
FSV-BH	0.166	0.261	0.620	0.490	0.71	1.47	0.227	0.199	5.18	7.10	8.31	10.32	1.38	2.29	0.77	2.07				
FSV-BI	0.274	0.430	0.770	0.491	0.92	1.36	0.232	0.188	4.60	6.62	7.82	9.28	1.35	2.62	0.70	2.13				
FSV-BJ	0.285	0.452	0.806	0.527	0.69	1.06	0.191	0.155	6.02	7.95	9.24	9.22	1.65	3.17	0.89	2.12				
FSV-BK	0.280	0.446	0.815	0.527					4.72	6.73	7.81	9.64								
FSV-BL	<0.11	0.229	0.315	0.516					2.58	4.31	5.17	8.61								
FSV-BM	0.273	0.309	0.478	0.476					5.50	7.90	7.30	10.90								
FSV-BN	0.251	0.414	0.780	0.502	0.84	1.32	0.180	0.185	4.82	7.48	7.89	9.59	1.45	3.00	0.72	2.62	1.11	0.16	0.18	0.12
FSV-BO	0.298	0.441	0.842	0.539					5.38	7.11	9.26	9.99								
FSV-BP	0.251	0.415	0.758	0.478					5.07	7.05	8.36	10.45								
FSV-BQ	0.291	0.462	0.867	0.548					5.40	7.40	8.90	11.30								
FSV-BR	0.280	0.410	0.760	0.560																
FSV-BS	0.250	0.390	0.720	0.490																
FSV-BU	0.297	0.461	0.796	0.572					4.01	5.81	7.15	9.38	1.19	2.00	0.61	1.90				
FSV-BV	0.231	0.394	0.734	0.458					4.08	6.31	7.55	8.60	1.30	2.90	0.73	2.21				
FSV-BW	0.255	0.378	0.725	0.418	1.69	2.74	0.375	0.320	4.35	6.06	8.04	9.46	1.22	2.85	0.79	2.44				
FSV-BX	0.252	0.288	0.742	0.517					5.25	6.81	6.97	8.33	1.39	2.68	0.74	2.17				
FSV-BY	0.264	0.529	0.855	0.514	0.14	0.22	0.199	0.178	5.16	8.82	9.22	10.11	1.48	3.42	0.91	2.30	0.82	0.17	0.17	0.10
FSV-BZ									5.90	7.30	8.75	10.10	3.30	3.60	1.20	2.65				
FSV-CA	0.254	0.410	0.667	0.390					3.97	5.97	7.02	7.43								
FSV-CB	0.235	0.370	0.685	0.425					3.88	6.64	7.98	9.68								
FSV-CC	0.250	0.387	0.840	0.527					4.87	6.78	8.28	9.70								
FSV-CD	0.270	0.380	0.631	0.432	0.83	1.23	0.266	0.142	4.68	6.89	9.00	9.65	1.28	2.42	0.68	2.00				
FSV-CE	0.277	0.422	0.730	0.518					4.93	7.32	8.05	9.97								
FSV-CF	0.246	0.421	0.745	0.483					4.60	7.80	7.40	9.10								
FSV-CH	0.234	0.376	0.653	0.434					4.21	6.17	7.14	8.70	1.00	2.16	0.54	1.73				
FSV-CK	0.213	0.386	0.750	0.443					3.91	5.75	7.49	7.83	1.24	2.44	0.63	2.04				
FSV-CL	0.235	0.467	0.901	0.598					5.71	6.28	9.22	8.39	1.10	3.03	2.80	2.36				
FSV-CM									<4.0	6.63	8.00	10.35								
FSV-CN	0.233	0.384	0.744	0.509					4.05	6.13	7.10	8.79	1.07	2.24	0.41	1.87				
FSV-CQ	0.263	0.428	0.778	0.481					5.22	7.15	7.33	10.08								
FSV-CR	0.270	0.430	0.790	0.520					5.10	7.10	8.40	10.30					1.00	<0.3	<0.3	<0.3
FSV-CS	0.231	0.385	0.779	0.504																
FSV-CU	0.268	0.428	0.749	0.474	0.50	0.72	0.163	0.190	4.30	6.12	7.00	9.38								
FSV-CX	0.200	0.450	0.810	0.510	0.82	1.47	0.250	0.180	7.06	8.18	9.35	10.16	1.31	3.14	0.79	2.22				
FSV-DA	0.266	0.408	0.770	0.500	1.12	1.22	0.198	0.172	5.48	7.03	8.67	9.75	1.54	2.65	0.93	2.34	1.19	0.22	0.20	0.16
FSV-DB	0.293	0.427	0.769	0.483					5.19	6.70	8.19	9.77								
FSV-DJ	0.330	0.460	0.940	0.520					5.00	6.70	9.00	8.80								
FSV-DK	0.298	0.476	0.915	0.616	0.42	0.65	0.118	0.113	4.42	6.61	7.55	9.35	1.51	2.93	0.71	2.51				
FSV-DP	0.269	0.425	0.783	0.504	0.87	1.37	0.152	0.155												
FSV-DQ	0.337	0.457	0.754	0.514					5.17	6.82	7.52	10.11	1.44	2.59	0.72	2.30				
FSV-DR	0.325	0.507	0.948	0.563					6.14	7.48	8.62	10.17								
FSV-EI	0.223	0.358	0.676	0.404					4.45	6.62	7.33	7.72	1.47	3.07	0.82	2.07				
FSV-EL	0.220	0.380	0.680	0.490																
FSV-EM	0.280	0.440	0.770	0.480					4.14	6.35	7.26	8.85								
FSV-FN	0.278	0.409	0.735	0.525					5.29	7.19	7.74	11.47	1.36	2.60	0.76	2.29				
n	47	49	49	49	15	15	15	15	44	46	46	46	25	26	26	26	4	3	3	3
Min	0.166	0.229	0.315	0.390	0.14	0.22	0.118	0.113	2.58	4.31	5.17	7.43	1.00	2.00	0.41	1.73	0.821	0.158	0.171	0.100
Median	0.268	0.421	0.770	0.509	0.82	1.22	0.191	0.178	4.90	6.80	7.99	9.68	1.36	2.84	0.76	2.28	1.056	0.174	0.184	0.116
Max	0.337	0.536	0.964	0.616	1.69	2.74	0.375	0.320	7.06	8.82	9.35	11.47	3.30	3.60	2.80	2.65	1.190	0.222	0.200	0.155
eSD	0.026	0.047	0.056	0.034	0.15	0.31	0.050	0.024	0.68	0.49	0.87	0.73	0.14	0.29	0.07	0.20				
eCV	10	11	7	7	18	25	26	14	14	7	11	8	10	10	10	9				
NISTa	0.251	0.403	0.780	0.485					4.72	6.65	8.00	9.51	1.43	2.71	0.73	2.16	1.14	*nd*	*nd*	*nd*
NISTb	0.259	0.421	0.779	0.500	1.02	1.40	0.182	0.151	4.74	6.66	7.70	9.84	1.44	2.65	0.73	2.28	1.15	0.23	0.24	0.17
NAV	0.261	0.417	0.775	0.501	0.82	1.22	0.191	0.178	4.82	6.73	7.92	9.61	1.40	2.76	0.75	2.25				
NAU	0.027	0.065	0.065	0.044	0.15	0.31	0.050	0.024	0.69	0.91	0.88	0.77	0.17	0.38	0.11	0.25				

Round Robin XL Laboratory Results

Values in µg/mL

	Total β-Carotene				trans-β-Carotene				Total cis-β-Carotene				Total α-Carotene			
Lab	231	232	233	234	231	232	233	234	231	232	233	234	231	232	233	234
FSV-BA	0.075	0.254	0.533	0.678	0.074	0.245	0.494	0.626	0.001	0.009	0.039	0.052	0.049	0.011	0.129	0.034
FSV-BD	0.074	0.217	0.447	0.523												
FSV-BE		0.227	0.544	0.663												
FSV-BF	0.062	0.253	0.569	0.573									0.036	0.015	0.107	0.023
FSV-BG	0.068	0.195	0.433	0.625									0.042	0.020	0.081	0.024
FSV-BGa	0.063	0.196	0.492	0.659									0.037	0.016	0.082	0.047
FSV-BH	0.070	0.213	0.446	0.603	0.070	0.213	0.430	0.570	<0.01	<0.01	0.016	0.033	0.053	0.013	0.107	0.029
FSV-BI	0.066	0.211	0.457	0.562									0.045	0.012	0.103	0.025
FSV-BJ	0.076	0.219	0.463	0.610									0.068	0.027	0.173	0.046
FSV-BK																
FSV-BL																
FSV-BM																
FSV-BN	0.078	0.268	0.608	0.706	0.065	0.248	0.569	0.637	*nq*	*nq*	0.012	0.041	0.048	0.015	0.111	0.028
FSV-BO	0.064	0.211	0.484	0.533									0.037	0.014	0.097	0.027
FSV-BP	0.068	0.296	0.651	0.614									0.056	0.022	0.151	0.031
FSV-BQ																
FSV-BR																
FSV-BS					0.060	0.220	0.410	0.570					0.030	*nq*	0.070	0.020
FSV-BU	0.077	0.226	0.496	0.616									0.056	0.014	0.107	0.029
FSV-BV	0.101	0.280	0.544	0.551									0.037	0.011	0.087	0.021
FSV-BW	0.125	0.393	0.778	0.968									0.066	0.016	0.144	0.021
FSV-BX	0.207	0.602	0.103	0.053									0.079	0.077	0.146	0.045
FSV-BY	0.083	0.295	0.535	0.650	0.073	0.266	0.489	0.569	0.011	0.030	0.046	0.081	0.044	0.027	0.093	0.038
FSV-BZ	*0.040*	*0.136*	*0.334*	*0.548*	0.040	0.136	0.330	0.511	*nd*	*nd*	0.004	0.037	0.039	0.027	0.072	0.036
FSV-CA																
FSV-CB																
FSV-CC																
FSV-CD	0.068	0.216	0.471	0.530									0.036	0.009	0.079	0.022
FSV-CE	0.082	0.243	0.567	0.544												
FSV-CF																
FSV-CH	0.047	0.196	0.399	0.561									0.028	0.006	0.079	0.020
FSV-CK	0.083	0.213	0.494	0.548									0.059	0.105	0.123	0.038
FSV-CL	0.065	0.209	0.494	0.587									0.059	0.012	0.119	0.029
FSV-CM																
FSV-CN					0.047	0.160	0.438	0.544					0.033	*nq*	0.078	0.013
FSV-CQ	0.356	0.572	0.457	0.475												
FSV-CR																
FSV-CS	0.092	0.276	0.569	0.660	0.083	0.255	0.523	0.591	*0.009*	*0.021*	*0.046*	*0.069*	0.062	0.016	0.133	0.033
FSV-CU																
FSV-CX	0.070	0.340	0.820	0.860									0.040	0.010	0.110	0.040
FSV-DA	0.072	0.216	0.498	0.620	0.066	0.200	0.457	0.578	0.006	0.016	0.041	0.042	0.051	0.021	0.093	0.023
FSV-DB	0.055	0.167	0.378	0.542												
FSV-DJ																
FSV-DK	0.091	0.263	0.591	0.790									0.053	0.017	0.116	0.031
FSV-DP																
FSV-DQ	0.184	0.161	0.138	0.620									0.012	0.015	0.037	0.030
FSV-DR	0.098	0.320	0.661	0.690												
FSV-EI					0.082	0.290	0.660	0.502					0.056	0.020	0.125	0.024
FSV-EL																
FSV-EM																
FSV-FN																
n	30	31	31	31	10	10	10	10	4	4	7	7	28	26	28	28
Min	0.040	0.136	0.103	0.053	0.040	0.136	0.330	0.502	0.001	0.009	0.004	0.033	0.012	0.006	0.037	0.013
Median	0.075	0.226	0.494	0.610	0.068	0.233	0.473	0.570	0.008	0.019	0.039	0.042	0.047	0.016	0.107	0.029
Max	0.356	0.602	0.820	0.968	0.083	0.290	0.660	0.637	0.011	0.030	0.046	0.081	0.079	0.105	0.173	0.047
eSD	0.017	0.050	0.086	0.083	0.009	0.037	0.062	0.029			0.022	0.016	0.014	0.006	0.031	0.009
eCV	22	22	17	14	14	16	13	5			56	38	30	41	29	29
NISTa	0.074	0.230	0.447	0.642	0.070	0.206	0.373	0.542	*0.004*	*0.025*	*0.074*	*0.099*	0.051	0.026	0.100	0.038
NISTb	0.077	0.224	0.434	0.638	0.065	0.202	0.399	0.586	*0.011*	*0.021*	*0.036*	*0.052*	0.052	0.022	0.117	0.046
NAV	0.075	0.227	0.467	0.624	0.068	0.218	0.429	0.566			0.047	0.059	0.049	0.020	0.108	0.035
NAU	0.017	0.050	0.094	0.086	0.010	0.042	0.088	0.059			0.042	0.050	0.015	0.009	0.031	0.013

Round Robin XL Laboratory Results

Values in µg/mL

	Total Lycopene				trans-Lycopene				β-Cryptoxanthin				α-Cryptoxanthin			
Lab	231	232	233	234	231	232	233	234	231	232	233	234	231	232	233	234
FSV-BA					0.128	0.177	0.132	0.185	0.052	0.179	0.365	0.046				
FSV-BD	0.158	0.192	0.144	0.195					0.039	0.134	0.256	0.026				
FSV-BE																
FSV-BF	0.205	0.262	0.200	0.264					0.036	0.122	0.285	0.027				
FSV-BG	0.203	0.219	0.164	0.278	0.102	0.128	0.086	0.148	0.038	0.013	0.028	0.021				
FSV-BGa	0.173	0.190	0.184	0.334												
FSV-BH	0.241	0.261	0.224	0.298					0.053	0.141	0.292	0.046				
FSV-BI	0.196	0.241	0.187	0.279					0.050	0.157	0.355	0.039				
FSV-BJ	0.215	0.322	0.220	0.328												
FSV-BK																
FSV-BL																
FSV-BM																
FSV-BN	0.245	0.312	0.257	0.322	0.120	0.196	0.134	0.165	0.043	0.146	0.321	0.032	0.008	0.009	0.017	0.013
FSV-BO	0.228	0.251	0.286	0.341					0.044	0.117	0.260	0.033				
FSV-BP	0.086	0.283	0.322	0.429					0.071	0.116	0.236	0.068				
FSV-BQ																
FSV-BR																
FSV-BS	0.180	0.190	0.120	0.220					0.050	0.160	0.300	0.040				
FSV-BU	0.269	0.265	0.209	0.324					0.045	0.127	0.271	0.035				
FSV-BV	0.191	0.254	0.198	0.234					0.075	0.141	0.307	0.027				
FSV-BW	0.480	0.563	0.398	0.598												
FSV-BX	0.192	0.271	0.995	0.726												
FSV-BY	0.190	0.275	0.204	0.263	0.107	0.169	0.114	0.155	0.041	0.145	0.259	0.030				
FSV-BZ	0.195	0.220	0.190	0.242												
FSV-CA																
FSV-CB																
FSV-CC																
FSV-CD	0.228	0.246	0.199	0.268					0.045	0.123	0.270	0.038				
FSV-CE																
FSV-CF																
FSV-CH	0.085	0.125	0.077	0.144												
FSV-CK	0.153	0.168	0.155	0.218	0.078	0.104	0.082	0.106	0.050	0.123	0.268	0.042	0.018	0.019	0.024	0.034
FSV-CL	0.180	0.184	0.125	0.183					0.041	0.083	0.177	0.028				
FSV-CM																
FSV-CN	0.150	0.115	0.087	0.218					0.033	0.119	0.265	0.025				
FSV-CQ																
FSV-CR																
FSV-CS	0.271	0.274	0.256	0.328					0.044	0.131	0.251	0.031				
FSV-CU																
FSV-CX	0.140	0.220	0.170	0.230					0.080	0.280	0.630	0.060				
FSV-DA	0.259	0.268	0.209	0.337	0.126	0.159	0.112	0.176	0.047	0.131	0.265	0.038	0.012	0.010	0.018	0.013
FSV-DB	0.170	0.199	0.152	0.320					0.052	0.146	0.341	0.047				
FSV-DJ																
FSV-DK	0.332	0.375	0.310	0.478												
FSV-DP																
FSV-DQ	0.177	0.221	0.184	0.265					0.078	0.181	0.184	0.047				
FSV-DR																
FSV-EI					0.108	0.111	0.109	0.111	0.047	0.171	0.323	0.036				
FSV-EL																
FSV-EM																
FSV-FN																
n	28	28	28	28	7	7	7	7	23	23	23	23	3	3	3	3
Min	0.085	0.115	0.077	0.144	0.078	0.104	0.082	0.106	0.033	0.013	0.028	0.021	0.008	0.009	0.017	0.013
Median	0.194	0.249	0.199	0.279	0.108	0.159	0.112	0.155	0.047	0.134	0.270	0.036	0.012	0.010	0.018	0.013
Max	0.480	0.563	0.995	0.726	0.128	0.196	0.134	0.185	0.080	0.280	0.630	0.068	0.018	0.019	0.024	0.034
eSD	0.044	0.055	0.052	0.072	0.014	0.040	0.019	0.030	0.007	0.022	0.042	0.011				
eCV	23	22	26	26	13	25	17	20	16	16	16	31				
NISTa																
NISTb	0.236	0.242	0.209	0.333	0.099	0.120	0.094	0.149	0.035	0.102	0.241	0.027				
NAV	0.215	0.245	0.204	0.304	0.103	0.140	0.103	0.151	0.041	0.118	0.256	0.031				
NAU	0.058	0.061	0.053	0.080	0.023	0.048	0.026	0.032	0.015	0.039	0.062	0.013				

Round Robin XL Laboratory Results

Values in µg/mL

	Lutein				Zeaxanthin				Lutein&Zeaxanthin			
Lab	231	232	233	234	231	232	233	234	231	232	233	234
FSV-BA									0.683	0.529	0.152	0.124
FSV-BD	0.144	0.361	0.059	0.059	0.244	0.035	0.031	0.023	*0.388*	*0.396*	*0.090*	*0.082*
FSV-BE												
FSV-BF									0.294	0.236	0.071	0.086
FSV-BG	0.154	0.285	0.048	0.055	0.098	0.019	0.011	0.030	*0.252*	*0.304*	*0.059*	*0.085*
FSV-BGa												
FSV-BH	0.110	0.202	0.063	0.043	0.230	<0.02	<0.02	<0.02	*0.340*	*0.202*	*0.063*	*0.043*
FSV-BI	0.116	0.212	0.040	0.042	0.236	0.024	0.025	0.015	*0.352*	*0.236*	*0.065*	*0.057*
FSV-BJ												
FSV-BK												
FSV-BL												
FSV-BM												
FSV-BN	0.124	0.244	0.038	0.043	0.243	0.027	0.019	0.019	*0.367*	*0.271*	*0.057*	*0.062*
FSV-BO									0.219	0.200	0.050	0.051
FSV-BP												
FSV-BQ												
FSV-BR												
FSV-BS									0.430	0.260	0.060	0.100
FSV-BU									0.594	0.295	0.087	0.110
FSV-BV									0.305	0.236	0.071	0.076
FSV-BW												
FSV-BX	0.152	0.224	0.063	0.077	0.179	0.020	0.013	0.014	*0.331*	*0.244*	*0.076*	*0.091*
FSV-BY	0.155	0.362	0.057	0.062	0.202	*nq*	0.022	0.020	0.357	0.362	0.079	0.082
FSV-BZ	0.238	0.231	0.080	0.087								
FSV-CA												
FSV-CB												
FSV-CC												
FSV-CD									0.312	0.207	0.064	0.080
FSV-CE												
FSV-CF												
FSV-CH												
FSV-CK	0.346	0.257	0.087	0.100								
FSV-CL									0.226	0.177	0.065	0.073
FSV-CM												
FSV-CN	0.109	*nd*	0.057	0.058	0.222	*nd*	0.037	0.034	0.331	0.213	0.094	0.092
FSV-CQ												
FSV-CR												
FSV-CS									0.375	0.315	0.082	0.086
FSV-CU												
FSV-CX	0.230	0.390	0.100	0.080	0.270	0.040	0.030	0.030	*0.500*	*0.430*	*0.130*	*0.110*
FSV-DA	0.159	0.260	0.059	0.071	0.302	0.030	0.033	0.036	*0.461*	*0.290*	*0.092*	*0.107*
FSV-DB									0.319	0.209	0.075	0.079
FSV-DJ												
FSV-DK	0.318	0.235	0.067	0.084								
FSV-DP												
FSV-DQ									0.107	0.141	0.133	0.093
FSV-DR												
FSV-EI	0.139	0.309	0.053	0.062	0.262	0.060	0.037	0.036	*0.401*	*0.369*	*0.090*	*0.098*
FSV-EL												
FSV-EM												
FSV-FN												
n	14	13	14	14	11	8	10	10	22	22	22	22
Min	0.109	0.202	0.038	0.042	0.098	0.019	0.011	0.014	0.107	0.141	0.050	0.043
Median	0.153	0.257	0.059	0.062	0.236	0.029	0.028	0.027	0.346	0.252	0.076	0.086
Max	0.346	0.390	0.100	0.100	0.302	0.060	0.037	0.036	0.683	0.529	0.152	0.124
eSD	0.063	0.058	0.009	0.017	0.030	0.010	0.009	0.010	0.068	0.076	0.019	0.015
eCV	41	23	15	28	13	35	34	39	20	30	25	17
NISTa												
NISTb	0.144	0.240	0.055	0.065	0.264	0.028	0.029	0.033	*0.408*	*0.268*	*0.084*	*0.098*
NAV	0.149	0.248	0.057	0.064	0.250	0.028	0.028	0.030	0.377	0.260	0.080	0.092
NAU	0.063	0.059	0.013	0.018	0.075	0.010	0.010	0.011	0.088	0.077	0.020	0.021

Round Robin XL Laboratory Results

Analytes Reported By One Laboratory

Values in µg/mL

Analyte	Code	231	232	233	234
Coenzyme Q10	FSV-CH	0.247	0.216	0.166	1.014
Total Carotenoids	FSV-EM	0.800	0.780	1.100	1.150
trans-α-Carotene	NISTb	0.047	0.016	0.107	0.030

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 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 less than x, the limit of detection
italics	Not explicitly reported but calculated by NIST from reported values

Round Robin XL Laboratory Results

Comparability Summary

Lab	R	aT	gT	bC	tbC
FSV-BA	2	3	2	2	2
FSV-BD	1	1	1	1	
FSV-BE	3	1	1	1	
FSV-BF	2	1	1	1	
FSV-BG	3	3			
FSV-BGa	1	2		2	
FSV-BH	2	2	1	2	
FSV-BI	1	1		4	
FSV-BJ	1	1	1	1	
FSV-BK	2	1	3	1	1
FSV-BL	4	1	2	1	1
FSV-BM	2	3	2	1	
FSV-BN	2	2	1	4	
FSV-BO		2	4	2	3
FSV-BP		1			
FSV-BQ	4	4			
FSV-BR	1	2	2	1	
FSV-BS	1	1			
FSV-BU	1	2			
FSV-BV	2	2	3	2	
FSV-BW	3	2	4	1	
FSV-BX	3	4	1	4	
FSV-BY	2	2			
FSV-BZ	1	2		2	
FSV-CA	1	1	2	2	2
FSV-CB	3	3	1		3
FSV-CC	1				1
FSV-CD	2	2		2	
FSV-CE	2				
FSV-CF	2	3			
FSV-CH	3	2			
FSV-CK	4	2			
FSV-CL	1	1			
FSV-CM	3	2	1	2	
FSV-CN	3	1	2	2	
FSV-CQ	2	1		2	
FSV-CR	2	2	2	1	
FSV-CS	1	1	2	1	1
FSV-CU	1				
FSV-CX	2				
FSV-DA	2	2	1	2	
FSV-DB	2	2	3		2
FSV-DJ	1	1			
FSV-DK	2	1	2	4	
FSV-DP	1	2			
FSV-DQ	2			2	2
FSV-DR	3	2		2	
FSV-EI	3	1	1	4	
FSV-EL	1	1			
FSV-EM	1	3	1		
FSV-FN	1	1		2	
NISTa	1	1	1	1	1
NISTb	1	1	1	1	1
n	49	46	26	31	10

Label	Definition
Lab	laboratory number
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 (non-NIST) 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)	{i.e., ±1 SD}
2	All StV within ±*t* (1-0.954,n-1)	{i.e., ±2 SD}
3	All StV within ±*t* (1-0.997,n-1)	{i.e., ±3 SD}
4	At least one StV > ±*t* (1-0.997,n-1)	{i.e., >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 heterogeneity, 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

Lab	% Observed					Expected
1	57	65	68	64	75	68.2 %
2	35	33	18	21	17	27.3 %
3	6	2	7	3	8	4.3 %
4	2	0	7	12	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 H. Representative "Individualized Report" for RR40

Each participant in RR40 received an "Individualized Report" reflecting their reported results. Each report included a detailed analysis of the results they reported for the following analytes:

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

The software used to generate the original RR40 "Individualized Reports" is no longer available and we do not have a hardcopy of the report as it was sent to any participant or NIST analyst. The following 11 pages were produced for participant FSV-BA using a descendant of the 1997 software. Three of the graphical tools used in the original reports ("Boxplot Comparisons", "Z-Score Concordance", and "NIST Assigned Values Vs Laboratory Values") have been retained and display the same information in much the same manner as in the original. However, the original report presented the same type of information for a number of analytes together on one or two pages rather than presenting all information for a given analyte on a single page. The modern software does not provide the "% RSD Bias and Precision History" table or the "% Difference" plots.

Individualized Round Robin XL Report: FSV-BA

Summary

	Serum 231			Serum 232			Serum 233			Serum 234		
Analyte	You	NAV	n	You	NAV	n	You	NAV	n	You	NAV	n
Retinol	0.291	0.261	47	0.449	0.417	49	0.830	0.775	49	0.517	0.501	49
Retinyl Palmitate	0.668	0.820	15	0.973	1.220	15	0.161	0.191	15	0.216	0.178	15
α-Tocopherol	4.72	4.82	44	6.6	6.7	46	7.7	7.9	46	9.68	9.61	46
γ-Tocopherol	1.53	1.40	25	2.88	2.76	26	1.01	0.75	26	2.44	2.25	26
Total β-Carotene	0.075	0.075	30	0.254	0.227	31	0.533	0.467	31	0.678	0.624	31
trans-β-Carotene	0.074	0.068	10	0.245	0.218	10	0.494	0.429	10	0.626	0.566	10
Total cis-β-Carotene	0.001		4	0.009		4	0.039	0.047	7	0.052	0.059	7
Total α-Carotene	0.049	0.049	28	0.011	0.020	26	0.129	0.108	28	0.034	0.035	28
trans-Lycopene	0.128	0.103	7	0.177	0.140	7	0.132	0.103	7	0.185	0.151	7
β-Cryptoxanthin	0.052	0.041	23	0.179	0.118	23	0.365	0.256	23	0.046	0.031	23
Lutein&Zeaxanthin	0.683	0.377	22	0.529	0.260	22	0.152	0.080	22	0.124	0.092	22

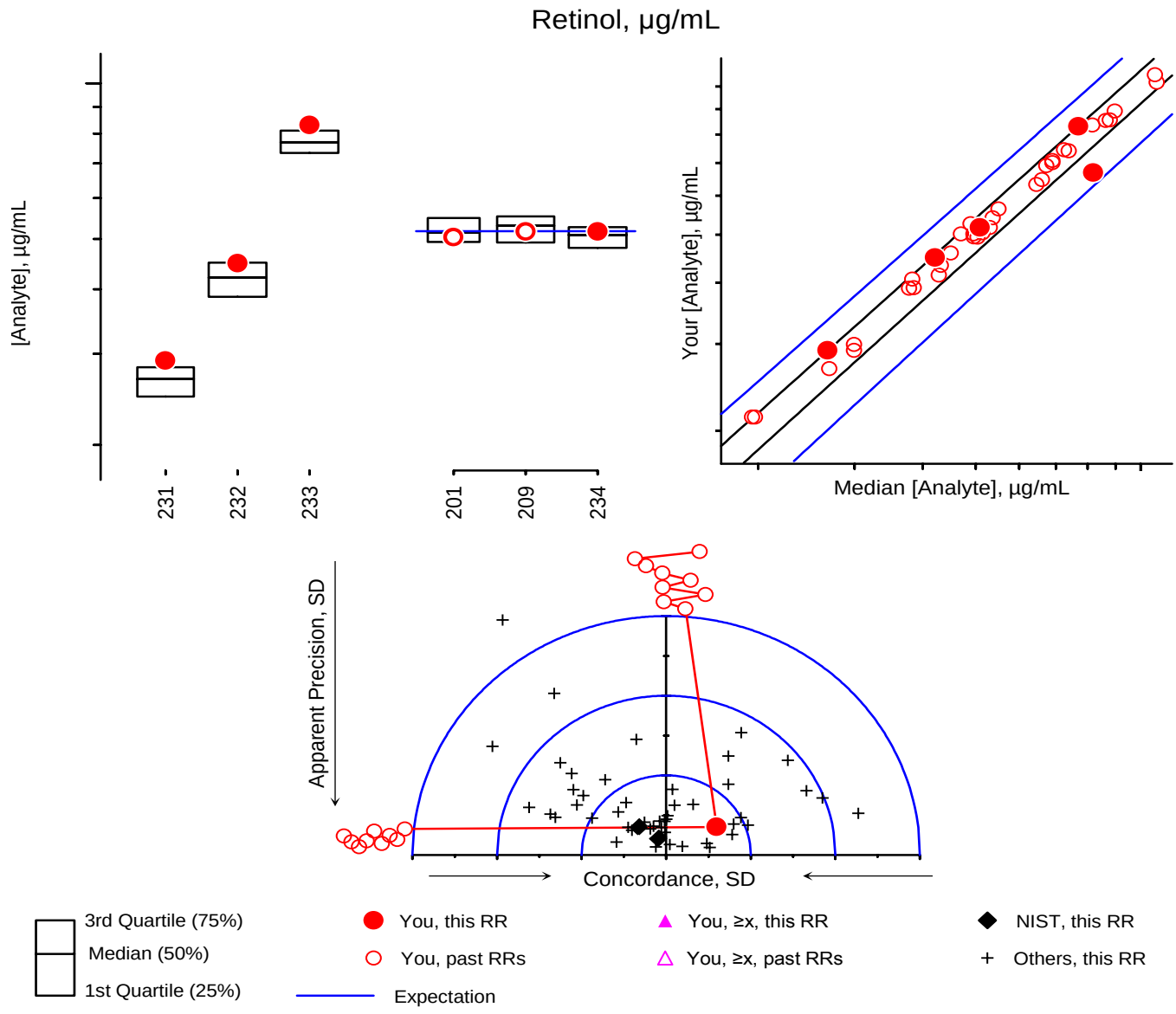
You : Your reported values for the listed analytes (micrograms/milliliter)
NAV : NIST Assigned Values, here equal to this RR's median
n : Number of non-NIST laboratories reporting quantitative values for this analyte in this serum

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

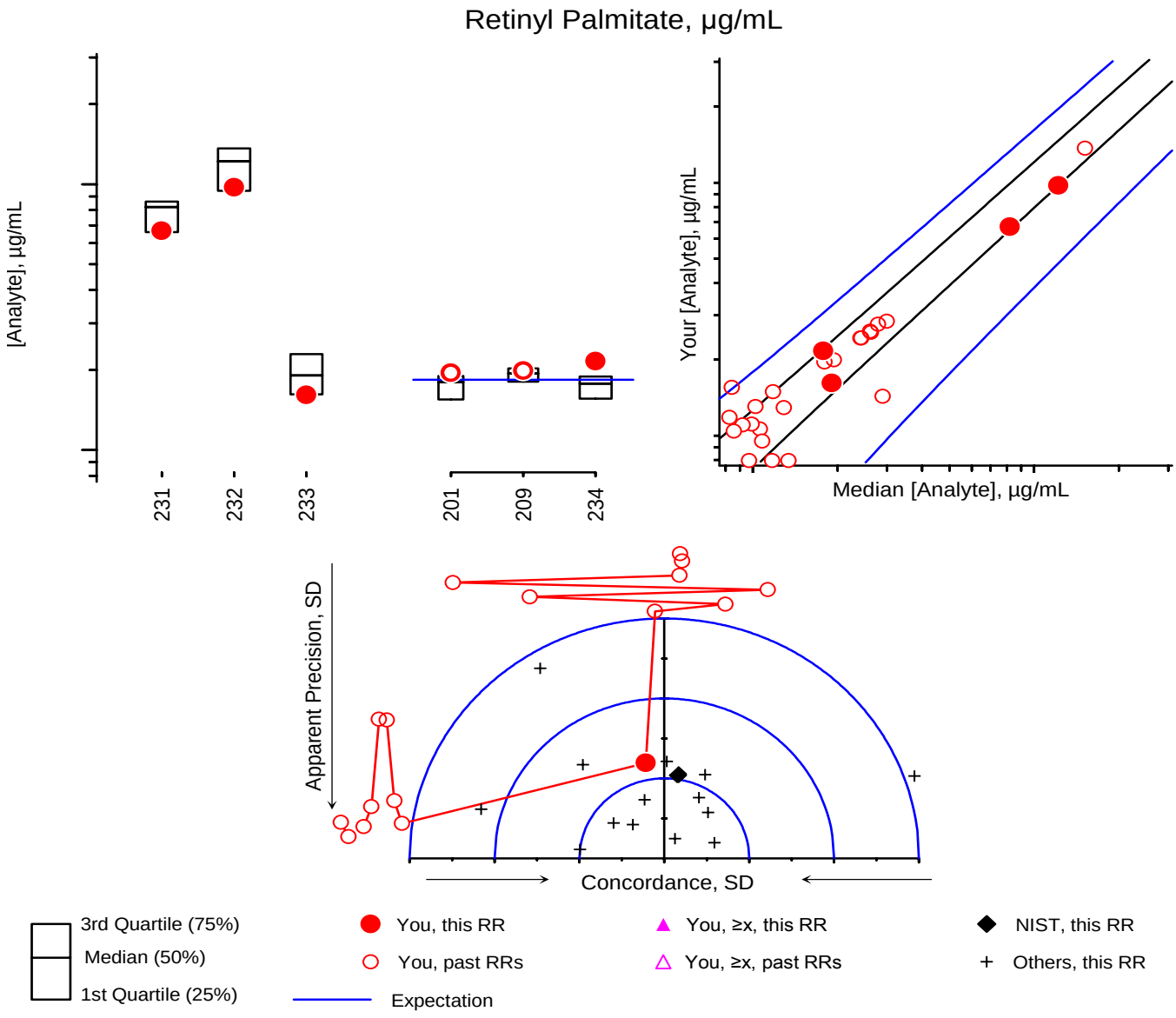
Individualized RR XL Report: FSV-BA



The software that was used to produce the original "Individualized Reports" for this study is no longer available. The original reports provided the same graphical analyses but in different format. This sheet was generated using a descendent of the 1997 system. For details of the construction and interpretation of these plots, see: Duewer et al. Anal Chem 1999;71(9):1870-8.

Serum	Comments	History
#231	Lyophilized, prepared from a "low" single donor pool augmented with many anayltes to ≈10% of normal population level.	New
#232	Lyophilized, prepared from a "low" single donor pool augmented with many anayltes to ≈50% of normal population level.	New
#233	Lyophilized, prepared from a "low" single donor pool augmented with many anayltes to ≈90% of normal population level.	New
#234	Lyophilized, multi-donor, native. This is the Mid level of SRM 968b.	RR32 #201, RR34 #209

Individualized RR XL Report: FSV-BA



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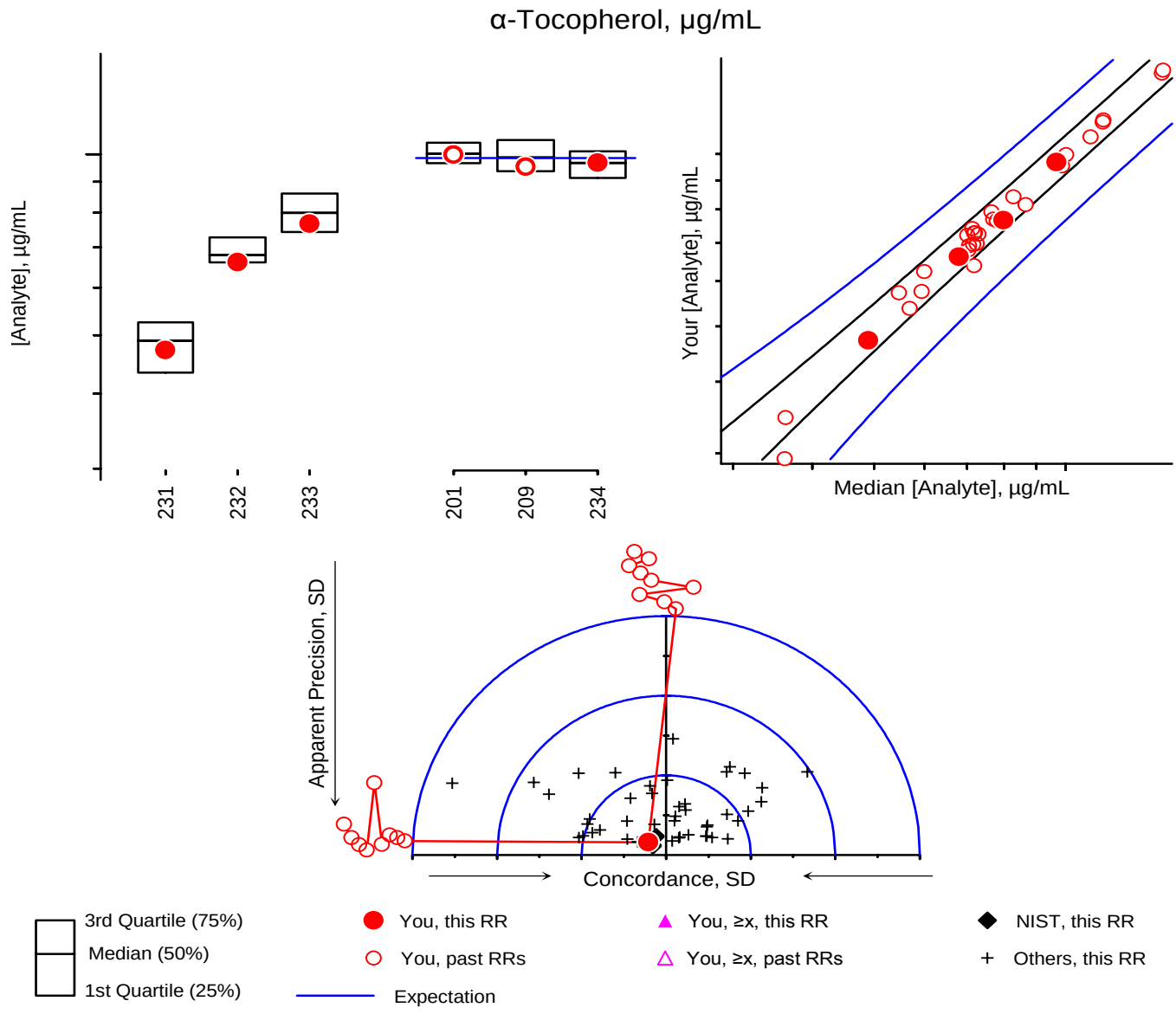
Individualized RR XL Report: FSV-BA



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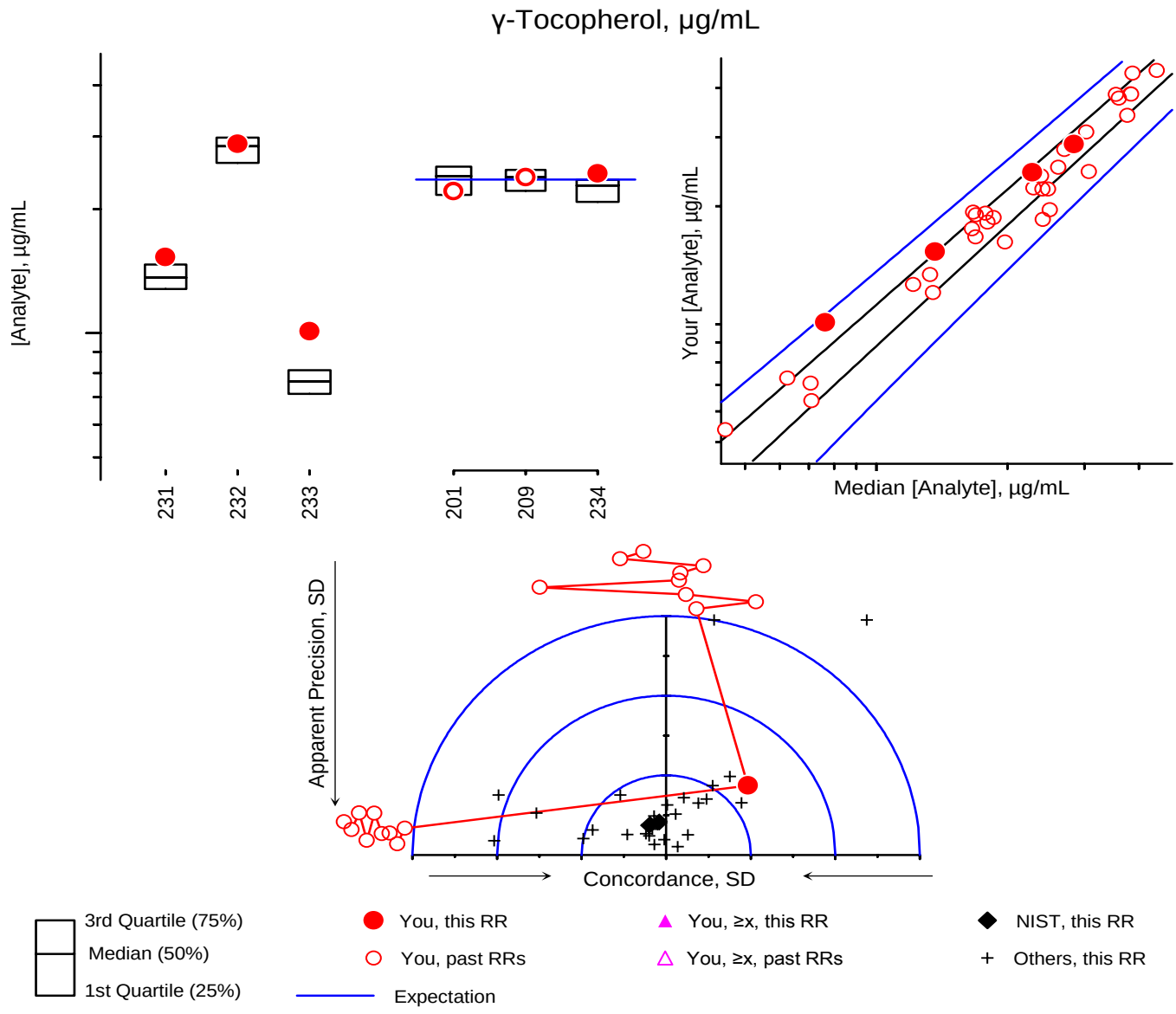
Individualized RR XL Report: FSV-BA



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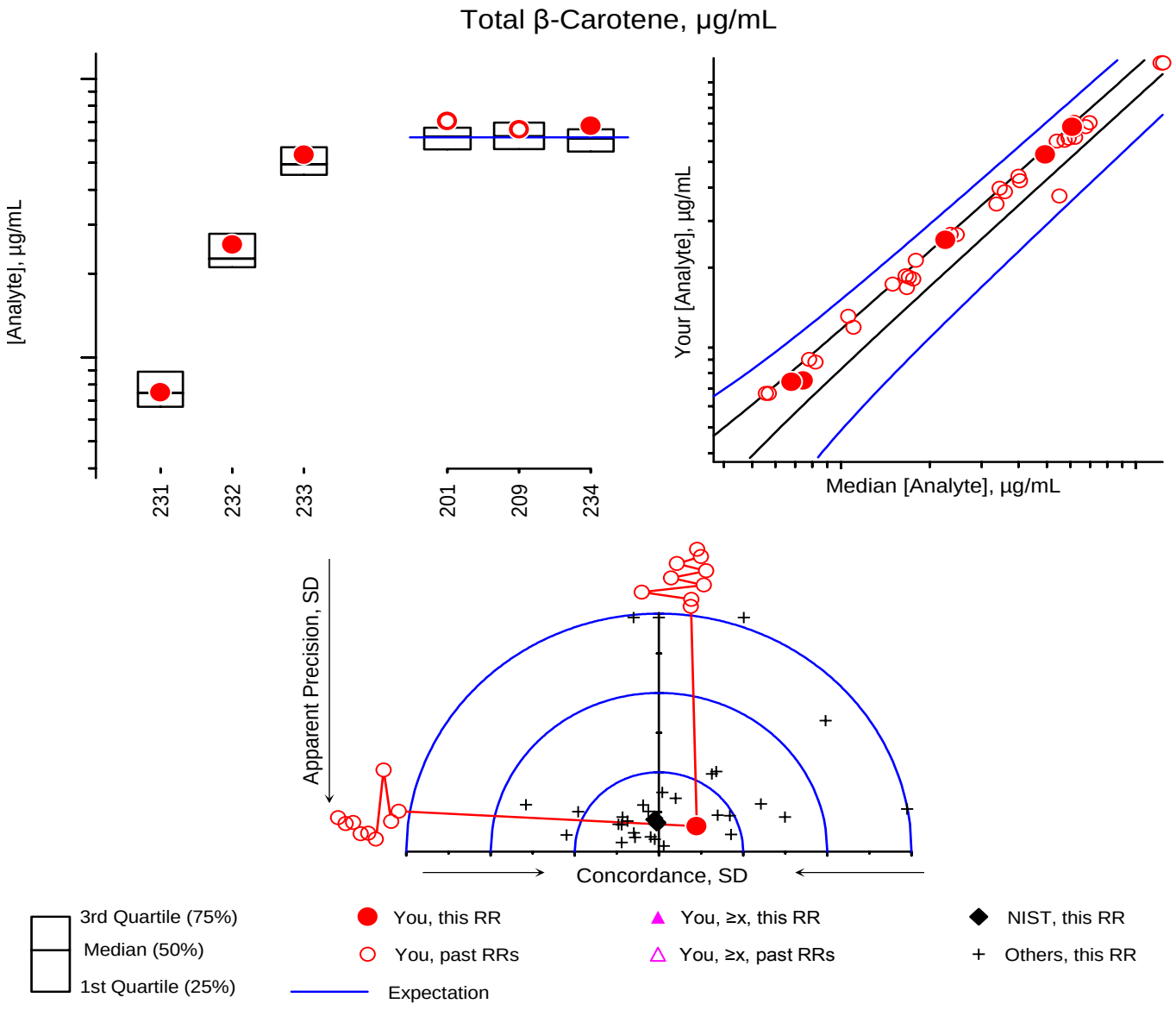
Individualized RR XL Report: FSV-BA



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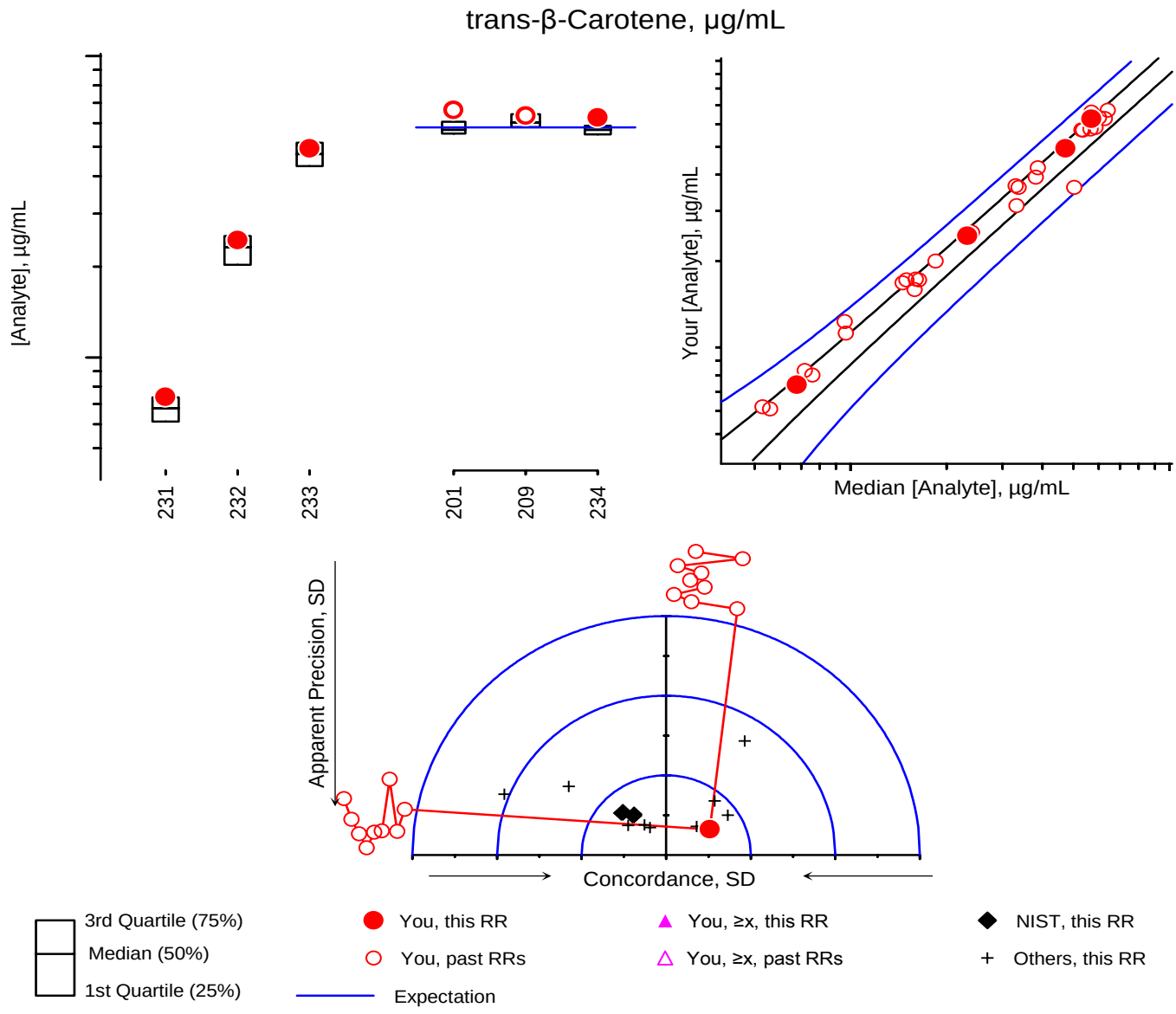

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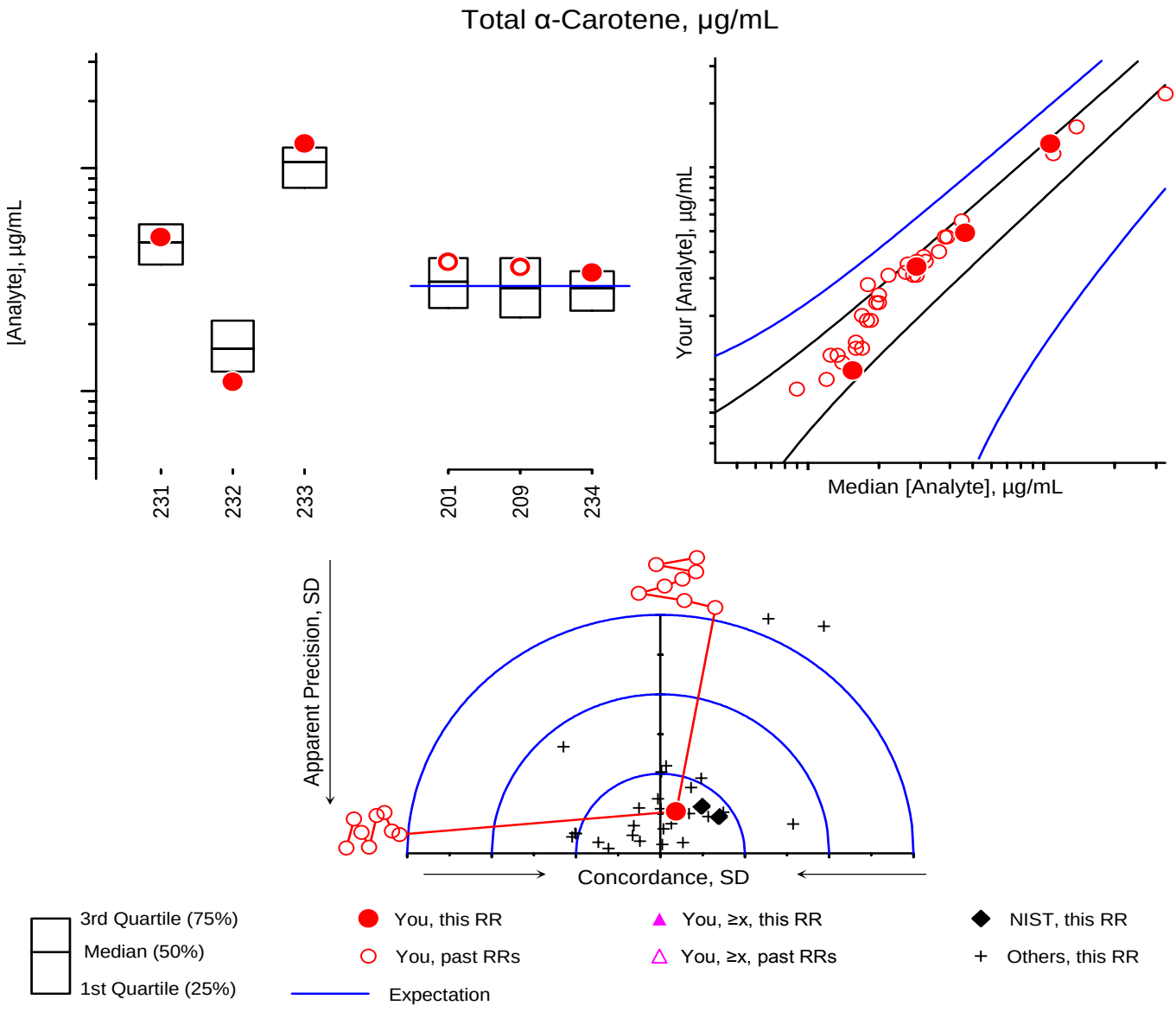

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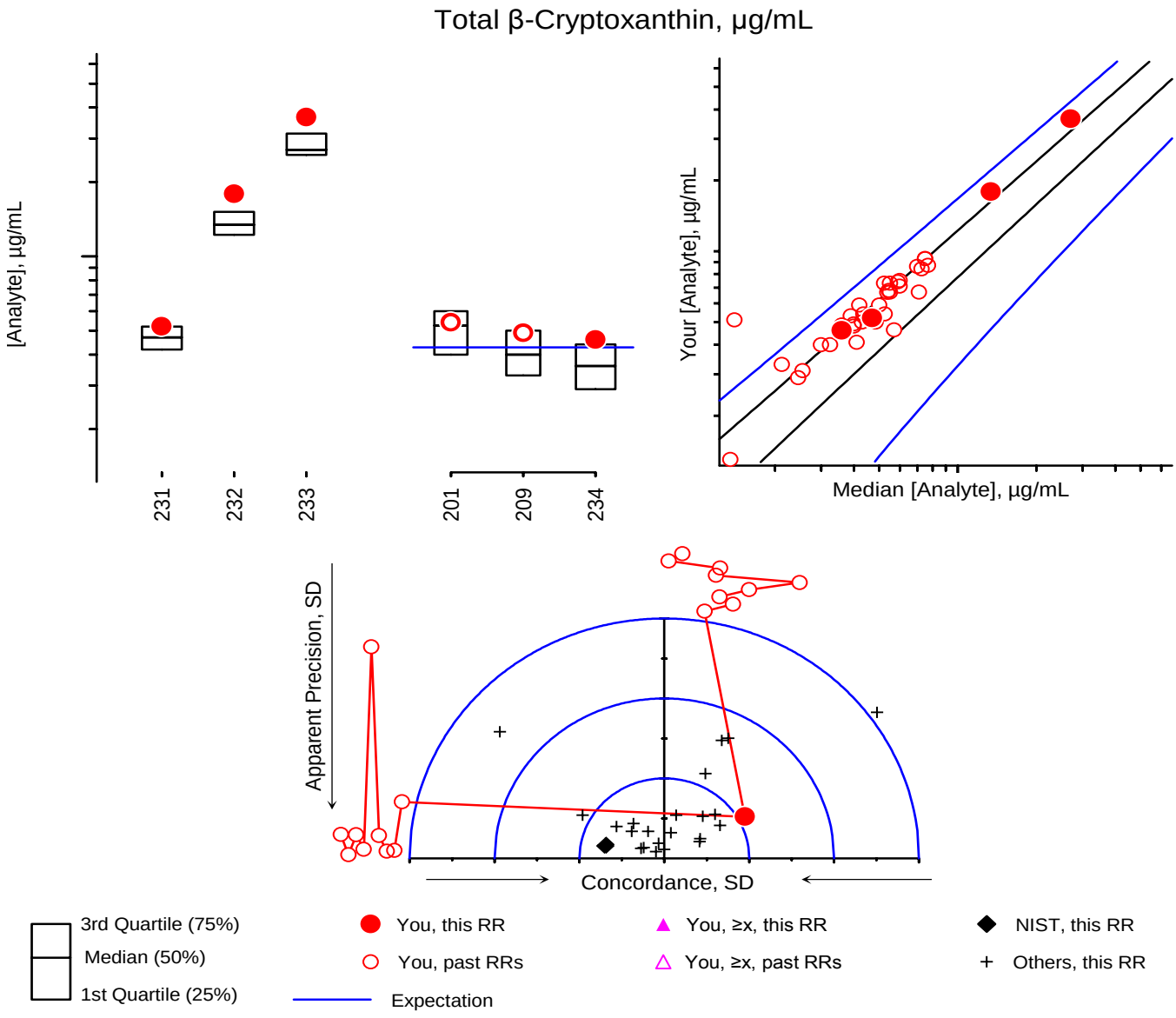

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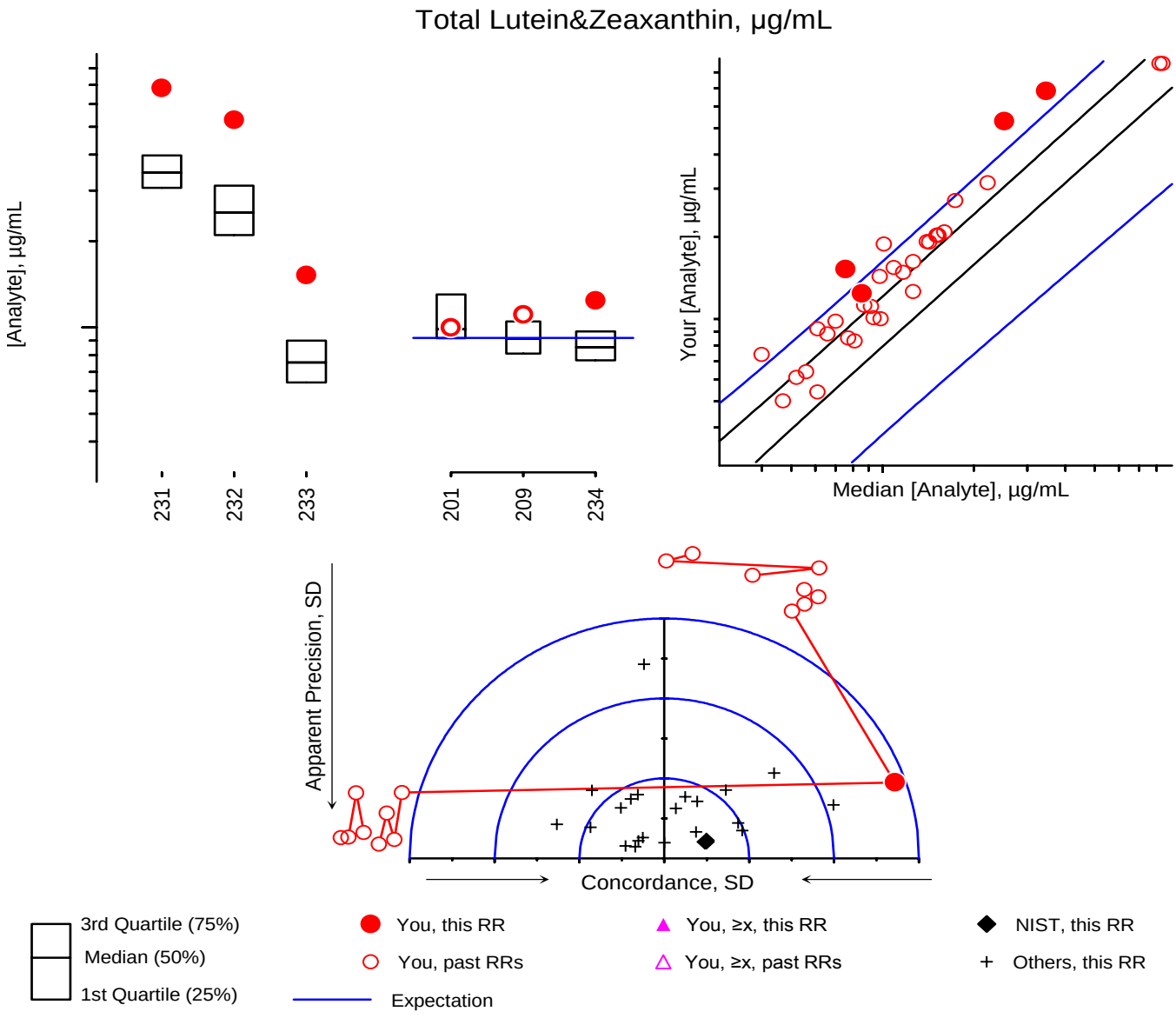

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#234	Lyophilized, multi-donor, native. This is the Mid level of SRM 968b.	RR32 #201, RR34 #209

Appendix I. Shipping Package Inserts for RR41

The following two items were included in each package shipped to an RR41 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-

July 7, 1997

Dear Colleague:

Enclosed is the set of samples for the second quality assurance round robin exercise (Round Robin XLI) for FY97. 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 September 19, 1997. 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 October 27, 1997.

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, 1850 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 XLI 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/NCI

Micronutrients Measurement Quality Assurance Program

Round Robin XLI Results from Laboratory #_____

	Serum				
Analyte	235	236	237	238	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					
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
September 22, 1997

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

Appendix J. Final Report for RR41

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

- Cover letter
- A "Report of Analysis" that:
  - 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
  - details the analysis of the NIST results
- A description of the contents of the "All-Lab" report

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

November 13, 1997

Dear Colleague:

This report summarizes both overall and individual laboratory performance for the three round robin exercises conducted during 1997. Included in this report are: tabular summaries of data for Round Robins (RR) XXXIX, XL, and XLI; a graphical summary of the interlaboratory median vs. individualized laboratory data for retinol, α- and γ-tocopherol, and total and *trans*-β-carotene; percent bias charts for retinol, α- and γ-tocopherol, and *trans*- and total β-carotene; and a summary of individual laboratory performance for the past three years. Tabular data only are provided for α-carotene, β-cryptoxanthin, lutein, lycopene, retinyl palmitate, and zeaxanthin. Over the past three years the overall interlaboratory precision has remained at an average estimated coefficient of variation (eCV) of <10% for retinol and α-tocopherol measurements and approximately 21% for β-carotene during the same period of time. The eCV for γ-tocopherol has remained at about 10% over the past three years.

A special "Standard Reference Material (SRM) 968b Report" is also enclosed as our "gift" to you for supporting our efforts to create a renewal material of the SRM. One of the three components of SRM 968b was distributed in each of the round robin exercises during the 1997 Quality Assurance (QA) Program. This report summarizes your results, the all-participant distributions, and the certified or noncertified values for these materials.

Serum 227 (the high level of SRM 968b) from RRXXXIX (Sera 227-230) was previously distributed as Sera 202 in RRXXXII and 210 in RRXXXIV. No significant changes in stability were observed. Serum 228 was previously distributed as blind duplicates (Sera 223 and 225) in RRXXXVIII. This material was reanalyzed to determine long-term within-laboratory measurement precision and among-laboratory measurement concordance. Sera 229 and 230 were new sera augmented with many of the commonly reported analytes (i.e., retinol, δ-, γ-, and α-tocopherol, lutein, and zeaxanthin.

RRXL (Sera 231-234) consisted of three new sera and the mid-level SRM 968b (Serum 234) which was previously distributed as Sera 201 and 209. Sera 231-233 were prepared from a serum pool as models for the proposed renewal material SRM 968c. The overall laboratory performance for retinol, γ- and α-tocopherol, and β-carotene for this exercise was comparable to that of the overall interlaboratory performance over the past three years.

The experimental design for Round Robin XLI (Sera 235-238) is summarized below. The four serum samples addressed the following issues:

• **Serum and Measurement Stability.** Serum 235 is the low-level component of SRM 968b, Fat-Soluble Vitamins and Cholesterol in Human Serum. It was previously distributed as Sera 200 (RRXXXII) and 207 (RRXXXIV). Reanalysis of such well-characterized samples documents the long-term stability of both materials and measurement systems. No statistically significant changes were observed in this material.

• **Analyte augmentation techniques.** Sera 236, 237, and 238 were again prepared as models for the high-, mid-, and low-level sera of the proposed renewal material SRM 968c. We intend to have the three "major" analytes (retinol, α-tocopherol, and β-carotene) at the same relative levels in SRM 968b, while having the "minor" analytes (α-carotene, lycopene, β-cryptoxanthin, lutein, and zeaxanthin) at different relative levels in the three sera. This design will provide significant added value to laboratories interested in carotenoid speciation. We were successful at achieving our target levels in the high- and mid-level samples (Sera 236 and 237, respectively). However, the augmentation technique did not work as well for Serum 238. Due to extreme difficulties in reconstituting this serum sample, results for Serum 238 were not used in the interlaboratory data analyses.

• **Measurement Precision and Concordance.** We have modified the "NIST assigned value versus your value scatter plots" on page 6 of your "Individualized Data" Report to better represent what we expect the measurement uncertainties to be. The plots now present ±1 eSD and ±3 eSD contours, instead of ±15% and ±50% measurement intervals.

Data for evaluating your laboratory's performance in Round Robin XLI are provided in the comparability summary on page 6 of the "All Lab" Report. The criteria used to summarize laboratory performance are as follows: results rated **1** (within ± 1 SD of the assigned value) indicate **EXCEPTIONAL** performance, those rated **2** (within ± 2 SD) indicate **ACCEPTABLE** performance, a rating of **3** (within ± 3 SD of the assigned value) is **MARGINAL** performance, and **4** (>3 SD from the assigned value) indicates **POOR** performance relative to the current state-of-the-practice for these measurements.

If you have concerns regarding your laboratory performance or were rated "**POOR**" based on the criteria stated above, 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 method modifications, your measured values do not agree with the certified values, we suggest that you contact us for consultation.

We anticipate that the 1998 QA Program will consist of three round robin exercises for the analysis of fat-soluble vitamins and carotenoids in serum and one exercise for the analysis of ascorbic acid in serum. The first set of samples for the fat-soluble vitamins in serum analysis for FY 98 will be distributed during the week of February 9. Results are due by April 3; written feedback will be provided to labs around April 30. The second set of samples will be shipped the week of May 4 with results due by June 26 and feedback to labs by July 31. The third set of samples will be shipped the week of August 10. Results will be due by September 25. Feedback will be provided to the laboratories around October 30.

The first set of samples for the measurement of ascorbic acid in serum will be distributed in early March 1998. This round robin study is being coordinated by Dr. Sam Margolis (301/975-3137).

The Fat-Soluble Vitamins and Carotenoid Analysis Tutorial session was held on October 27 at NIST. The session was well-attended and provided a great opportunity for new laboratories to discuss in detail their measurement techniques.

Certificates of participation in the FY 97 QA Program will be distributed in January 1998. If you have any questions, please feel free to contact me at: 301/975-3120; fax: 301/977-0685; or e-mail: jeanice.brownthomas@nist.gov.

Sincerely,

Jeanice Brown Thomas

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

Enclosures

cc: W.E. May
S. A. Wise

Analysis of N^2M^2QAP Round Robin XLI Results: Sera 235 to 238

Background: Four samples were distributed in RR41

Serum 235: the "low level" component of SRM® 968b: Fat-Soluble Vitamins and Cholesterol in Human Serum. It was previously distributed as Serum 200 in RR32 (9/94) and Serum 207 in RR34 (6/95).

Sera 236-238: experimental sera, prepared from a single "low" serum pool and augmented with the various analytes to (try to) hit 10%, 50%, and 90% population levels. The organic solvent-free (HDL, LDL) liposome technique suggested by the RR40 results was used in the preparation of all three sera.

Qualitative Results: The following observations were noted in the RR41 reports:

Nearly everyone reported that Serum 238 did not reconstitute, often describing a "large viscous globule" remaining in the vial.

Action: Sorry, our mistake and Serum 238 should not have been shipped. We will confirm the "reconstitutability" of all new sera prior to shipping.

No data for Serum 238 will be used in any analysis that reflects on your laboratory's performance. The data is of interest to us from the "what does a known heterogeneous material look like?" standpoint, so we do report the all-participant boxplots on pages 2 and 3. (We echo your data for Serum 238 on page 1 of the individualized report, but flag them as "non-quantitative values".)

We tried to notify all of you about this problem as soon as it was drawn to our attention. We thank all of you who provided us information on this material!

Note: If you did NOT observe a reconstitution problem with Serum 238, you probably SHOULD have. You may want to review your sample-evaluation procedures.

A few participants noted small amounts of suspended solid in Sera 237 and/or 236.

Action: We are continuing our analyte augmentation research. We will attempt to reduce or eliminate this problem. We will check all new sera for the presence of suspended solids on reconstitution.

Quantitative Results The following NIST Data and Value/Uncertainty Assignments table presents all NIST data, summary statistics for the NIST data, summary results for RR41, 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_{bet} 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} $\sqrt{SD_{rep}^2 + SD_{bet}^2 + SD_{anl}^2}$, total standard deviation for NIST analyses.

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

Initial Distribution (if any) Summary Statistics

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

Round Robin XLI 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 in this Round Robin

eSD_n 0.741 × InterQuartile Range in this Round Robin

P(n=p) $\text{TDIST}\left(\dfrac{\left|\text{Median}_n - \text{Median}_p\right|\sqrt{n_n + n_p - 2}}{\sqrt{\left((n_n - 1)\text{eSD}_n^2 + (n_p - 1)\text{eSD}_p^2\right)\left(\dfrac{1}{n_n} + \dfrac{1}{n_p}\right)}}, n_p+n_n\text{-}2, \text{2-tail}\right)$

This is the approximate probability that the current median is the same as it was in its initial distribution. Where the hypothesis that $\text{Median}_n = \text{Median}_p$ 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) $\text{FDIST}\left(\dfrac{\text{eSD}_n^2}{\text{eSD}_o^2}, n_n\text{-}1, n_p\text{-}1\right)$

This represents the approximate probability that the current interlaboratory variance is smaller than it was in its initial distribution. Where the hypothesis that $\text{eSD}_n < \text{eSD}_p$ can be rejected with 95% confidence, the P(n<p) value is flagged with an "*". FDIST is Excel®'s F-distribution function.

SD_{labs} $\sqrt{\text{eSD}_n^2 - \text{SD}_{\text{NIST}}^2}$, 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, $\text{SD}_{\text{labs}} = 0$.

CV_{labs} $100 \times \text{SD}_{\text{labs}}/\text{Median}_n$

NIST Assigned Values and Uncertainties

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

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

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

xCV CV "expected" for the given NAV analyte level. This calculation is detailed in: 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.

Measurement Performance Summary: The following is based on results presented in the NIST Data and Value/Uncertainty Assignments table of this report and the "Individualized Report" page 2 and 3 summary graphs:

Serum 235, low-level SRM 968b, stability: There is no significant difference in the level or measurement variability of any analyte among the three analyses of this material (Sera 200, 207, and 235).

Serum 236 homogeneity: Although there were a few reports of incomplete dissolution for this serum, the observed measurement variabilities were about as expected for homogeneous materials. The observed analyte levels were in excellent accord with the design levels.

Serum 237 homogeneity: There were several reports of incomplete dissolution for this serum. While most analytes were reasonably well behaved, the observed β-carotene level was lower than expected and the observed measurement variability was somewhat higher than expected. We suspect that some of the high-level augmented analytes are being non-specifically bound to various proteins.

Action: We continue to refine our augmentation techniques.

Serum 238 heterogeneity: The analytes in this serum reconstituted quite differentially. The median levels and observed variability were only somewhat greater than expected for the relatively hydrophilic retinol and γ-tocopherol. The carotenoids levels were much lower than designed (and the variability greater than expected for those levels).

Action: We have added an explicit "reconstitutability" requirement to our sample preparation QA/QC. We will continue to refine our augmentation techniques.

Retinyl palmitate: The CVs for *all* sera are greater than 30%.

Action: The current SRM 968b "certified" retinyl palmitate levels will be downgraded to to "non-certified" (or "reference", if we can upgrade the nomenclature!) values. If we can get the funding, we will evaluate the various analytical methodologies used or proposed for RP analysis.

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NIST Data and Value/Uncertainty Assignments

Retinol

	NIST1				NIST3			
	235	236	237	238	235	236	237	238
A:1	0.308	0.640	0.810	0.254	0.319	0.601	0.896	0.227
A:2	0.362	0.599	0.775	0.214	0.308	0.613	0.910	0.234
B:1	0.293	0.626	0.923	0.192	0.285	0.569	0.908	0.285
B:2	0.278	0.567	0.937	0.221	0.283	0.571	0.863	0.279
C:1	0.275	0.538	0.942	0.230	0.295	0.596	0.896	0.211
C:2	0.293	0.562	0.913	0.231	0.295	0.598	0.890	0.218
n_x	6	6	6	6	6	6	6	6
$Mean_x$	0.301	0.589	0.883	0.224	0.298	0.591	0.894	0.242
SD_x	0.032	0.040	0.072	0.021	0.014	0.018	0.017	0.032
SD_{repx}	0.024	0.031	0.019	0.020	0.005	0.005	0.019	0.005
SD_{betx}	0.029	0.036	0.079	0.015	0.015	0.019	0.009	0.035
SD_{NISTx}	0.038	0.047	0.081	0.025	0.016	0.020	0.021	0.036
CV_{NISTx}	12	8.0	9.2	11	5.2	3.3	2.4	15

	NIST			
n	12	12	12	12
Mean	0.299	0.590	0.889	0.233
SD_{rep}	0.017	0.022	0.019	0.015
SD_{bet}	0.021	0.026	0.051	0.028
SD_{anl}	0.000	0.000	0.000	0.000
SD_{NIST}	0.027	0.034	0.055	0.031
CV_{NIST}	9.1	5.8	6.2	14

NIST3=a+b*NIST1

a: -0.012 ±0.001
b: 1.025 ±0.001
R^2: 1.000

RR	XXXII
Serum	200
n_p	40
$Median_p$	0.300
eSD_p	0.026

	RRXLI			
	235	236	237	238
n_n	46	46	46	38
$Median_n$	0.284	0.602	0.872	0.303
eSD_n	0.018	0.044	0.088	0.035
P(n=p)	0.88			
P(n<p)	0.98			
SD_{labs}	0	0.027	0.069	0.015
CV_{labs}	0	4.5	7.9	4.8
NAV	0.291	0.596	0.880	0.268
NAU	0.027	0.044	0.088	0.035
CV	9.3	7.3	10	13
xCV	9.9	8.7	8.9	10

<— Current Results —>

<—— Assignments ——>

α-Tocopherol

	NIST1				NIST3			
	235	236	237	238	235	236	237	238
A:1	8.52	13.35	20.8	2.8	7.36	11.39	20.3	2.7
A:2	8.70	12.62	21.4	2.9	7.36	11.33	20.2	2.7
B:1	7.18	12.62	20.4	2.5	6.71	10.96	20.6	2.9
B:2	7.27	12.16	21.4	2.9	6.61	10.97	19.6	2.9
C:1	6.44	11.39	20.8	3.0	6.87	11.43	20.2	2.4
C:2	7.27	11.31	20.5	3.0	6.94	11.56	20.7	2.5
n_x	6	6	6	6	6	6	6	6
$Mean_x$	7.56	12.24	20.9	2.9	6.97	11.27	20.3	2.7
SD_x	0.87	0.79	0.4	0.2	0.32	0.25	0.4	0.2
SD_{repx}	0.35	0.36	0.5	0.2	0.05	0.06	0.5	0.0
SD_{betx}	0.92	0.83	0.2	0.1	0.35	0.27	0.2	0.2
SD_{NISTx}	0.99	0.90	0.6	0.2	0.36	0.28	0.5	0.2
CV_{NISTx}	13	7.4	2.7	8.0	5.1	2.5	2.4	8.5

	NIST			
n	12	12	12	12
Mean	7.27	11.76	20.6	2.8
SD_{rep}	0.25	0.26	0.5	0.1
SD_{bet}	0.63	0.57	0.2	0.4
SD_{anl}	0.30	0.30	0.3	0.3
SD_{NIST}	0.75	0.69	0.6	0.5
CV_{NIST}	10	5.9	2.9	18

NIST3=a+b*NIST1

a: -0.79 ±0.47
b: 1.01 ±0.03
R^2: 0.998

XXXII
200
42
7.16
0.52

	RRXLI			
	235	236	237	238
n_n	44	44	44	34
$Median_n$	6.91	11.53	20.0	4.1
eSD_n	0.55	0.74	1.2	1.2
P(n=p)	0.92			
P(n<p)	0.38			
SD_{labs}	0	0.24	1.0	1.1
CV_{labs}	0	2.1	5.0	27
NAV	7.09	11.64	20.3	3.4
NAU	0.75	0.74	1.2	1.2
CV	11	6.3	5.7	35
xCV	7.9	7.2	7.7	13

[41_dat]NISTtxt

NIST Data and Value/Uncertainty Assignments

γ-Tocopherol

	NIST1				NIST3			
	235	236	237	238	235	236	237	238
A:1	2.15	7.37	1.15	1.17	1.66	5.80	1.07	1.02
A:2	2.18	7.05	1.12	1.19	1.66	5.78	1.08	1.07
B:1	1.70	7.18	1.04	1.07	1.51	5.55	1.08	1.17
B:2	1.73	7.93	1.14	1.08	1.51	5.51	1.03	1.16
C:1	1.67	6.11	1.18	1.27	1.54	5.68	1.03	0.93
C:2	1.70	5.79	1.27	1.24	1.55	5.74	1.06	0.95
n_x	6	6	6	6	6	6	6	6
$Mean_x$	1.86	6.91	1.15	1.17	1.57	5.67	1.06	1.05
SD_x	0.24	0.81	0.07	0.08	0.07	0.12	0.02	0.10
SD_{repx}	0.02	0.36	0.05	0.02	0.00	0.03	0.02	0.02
SD_{betx}	0.27	0.84	0.07	0.09	0.08	0.13	0.01	0.11
SD_{NISTx}	0.27	0.92	0.09	0.09	0.08	0.14	0.03	0.11
CV_{NISTx}	15	13	7.5	7.7	5.1	2.4	2.6	11

	NIST			
n	12	12	12	12
Mean	1.71	6.29	1.10	1.11
SD_{rep}	0.02	0.26	0.04	0.02
SD_{bet}	0.15	0.44	0.04	0.08
SD_{anl}	0.04	0.04	0.04	0.04
SD_{NIST}	0.15	0.51	0.07	0.09
CV_{NIST}	9.0	8.1	6.4	8.5

NIST3=a+b*NIST1

a: 0.11 ±0.04
b: 0.81 ±0.01
R^2: 1.000

RR	XXXII
Serum	200
n_p	18
$Median_p$	1.69
eSD_p	0.23

	RRXLI			
	235	236	237	238
n_a	23	23	23	18
$Median_a$	1.70	6.28	1.18	1.51
eSD_a	0.26	0.39	0.19	0.31
P(n=p)	0.99			
P(n<p)	0.33			
SD_{labs}	0.21	0	0.18	0.29
CV_{labs}	12	0	15	19
NAV	1.71	6.28	1.14	1.31
NAU	0.26	0.51	0.19	0.31
CV	15	8.1	17	23
xCV	9.1	7.2	11	10

<— Current Results —>

<—— Assignments ——>

δ-Tocopherol

	NIST1				NIST3			
	235	236	237	238	235	236	237	238
		0.456	0.612	0.613	0.095	0.330	0.543	0.609
		0.418	0.600	0.639	0.101	0.353	0.536	0.609
		0.414	0.549	0.545	0.087	0.315	0.543	0.701
		0.485	0.641	0.567	0.084	0.311	0.517	0.709
		0.367	0.538	0.622	0.085	0.323	0.502	0.556
		0.380	0.504	0.617	0.094	0.326	0.532	0.561
0	6	6	6	6	6	6	6	6
	0.420	0.574	0.601	0.091	0.326	0.529	0.624	
	0.045	0.052	0.037	0.007	0.015	0.016	0.067	
	0.033	0.040	0.014	0.005	0.010	0.016	0.004	
	0.041	0.046	0.039	0.006	0.014	0.011	0.074	
	0.052	0.062	0.041	0.008	0.017	0.020	0.075	
	12	11	6.9	8.6	5.3	3.8	12	

	NIST			
	6	12	12	12
	0.091	0.373	0.551	0.612
	0.005	0.024	0.031	0.010
	0.006	0.032	0.031	0.076
	0.049	0.049	0.049	0.049
	0.050	0.064	0.066	0.091
	54	17	12	15

NIST3=a+b*NIST1

a: 0.000 ±
b: 0.871 ±0.069
R^2: 0.780

	XXXII
	200
	1
	0.866

	RRXLI			
	235	236	237	238
	5	6	6	6
	0.090	0.375	0.575	0.915
	0.030	0.052	0.058	0.068
	0	0	0	0
	0	0	0	0
	0.091	0.374	0.563	0.764
	0.050	0.064	0.066	0.091
	55	17	12	12

[41_dat]NISTtxt

NIST Data and Value/Uncertainty Assignments

Total β-Carotene

	NIST1				NIST3			
	235	236	237	238	235	236	237	238
A:1	0.256	0.303	0.311	0.048	0.243	0.254	0.340	0.043
A:2	0.226	0.310	0.420	0.060	0.247	0.267	0.332	0.041
B:1	0.262	0.317	0.273	0.045	0.228	0.254	0.376	0.047
B:2	0.242	0.319	0.455	0.049	0.231	0.258	0.349	0.050
C:1	0.239	0.290	0.342	0.047	0.241	0.269	0.414	0.040
C:2	0.245	0.283	0.392	0.055	0.234	0.265	0.403	0.043
n_x	6	6	6	6	6	6	6	6
$Mean_x$	0.245	0.304	0.366	0.051	0.237	0.261	0.369	0.044
SD_x	0.013	0.015	0.069	0.006	0.007	0.007	0.034	0.004
SD_{repx}	0.015	0.004	0.089	0.006	0.004	0.006	0.012	0.002
SD_{betx}	0.006	0.016	0.002	0.004	0.008	0.006	0.037	0.004
SD_{NISTx}	0.016	0.017	0.089	0.007	0.009	0.008	0.039	0.004
CV_{NISTx}	6.6	5.5	24	14	3.6	3.1	10	9.9

	NIST			
n	12	12	12	12
Mean	0.241	0.282	0.367	0.047
SD_{rep}	0.011	0.005	0.064	0.005
SD_{bet}	0.007	0.019	0.026	0.004
SD_{anl}	0.025	0.025	0.025	0.025
SD_{NIST}	0.028	0.031	0.073	0.026
CV_{NIST}	12	11	20	54

NIST3=a+b*NIST1

a: 0.000 ±

b: 0.953 ±0.047

R^2: 0.764

RR	XXXII
Serum	200
n_p	32
$Median_p$	0.248
eSD_p	0.040

	RRXLI				
	235	236	237	238	<— Current Results —>
n_n	27	27	27	22	
$Median_n$	0.242	0.280	0.376	0.085	
eSD_n	0.039	0.038	0.098	0.025	
P(n=p)	0.97				
P(n<p)	0.56				
SD_{labs}	0.027	0.021	0.064	0	
CV_{labs}	11	7.4	17	0	
NAV	0.242	0.281	0.372	0.066	<—— Assignments ——>
NAU	0.039	0.038	0.098	0.026	
CV	16	13	26	39	
xCV	17	17	16	25	

trans β-Carotene

	NIST1				NIST3			
	235	236	237	238	235	236	237	238
A:1	0.213	0.205	0.308	0.038	0.229	0.239	0.331	0.043
A:2	0.217	0.216	0.340	0.048	0.231	0.249	0.325	0.041
B:1	0.202	0.211	0.268	0.044	0.213	0.241	0.373	0.047
B:2	0.222	0.210	0.325	0.042	0.213	0.238	0.332	0.050
C:1	0.204	0.193	0.265	0.041	0.217	0.257	0.399	0.040
C:2	0.225	0.197	0.281	0.044	0.222	0.275	0.390	0.043
n_x	6	6	6	6	6	6	6	6
$Mean_x$	0.214	0.205	0.298	0.043	0.221	0.250	0.358	0.044
SD_x	0.009	0.009	0.032	0.003	0.008	0.014	0.033	0.004
SD_{repx}	0.012	0.005	0.027	0.004	0.002	0.008	0.017	0.002
SD_{betx}	0.001	0.009	0.026	0.000	0.009	0.014	0.034	0.004
SD_{NISTx}	0.012	0.010	0.038	0.004	0.009	0.017	0.038	0.004
CV_{NISTx}	5.6	5.0	13	10	4.0	6.6	11	9.9

	NIST			
	12	12	12	12
	0.217	0.228	0.328	0.043
	0.008	0.005	0.026	0.003
	0.016	0.013	0.029	0.004
	0.023	0.023	0.023	0.023
	0.029	0.027	0.045	0.023
	13	12	14	54

NIST3=a+b*NIST1

a: 0.000 ±

b: 1.162 ±0.054

R^2: 0.812

XXXII
200
8
0.232
0.022

RRXLI			
235	236	237	238
10	11	11	10
0.228	0.260	0.372	0.080
0.023	0.018	0.030	0.008
0.92			
0.49			
0	0	0	0
0	0	0	0
0.222	0.244	0.350	0.062
0.029	0.027	0.045	0.023
13	11	13	38
9.4	9.3	9.2	13

[41_dat]NISTtxt

NIST Data and Value/Uncertainty Assignments

	Total α-Carotene								Total Lycopene				trans-Lycopene			
	NIST1				NIST3				NIST3				NIST3			
	235	236	237	238	235	236	237	238	235	236	237	238	235	236	237	238
A:1			0.155		0.022	0.032	0.203	0.005	0.189	0.43	0.23	0.018	0.089	0.34	0.143	0.012
A:2			0.161		0.029	0.042	0.198	0.004	0.196	0.48	0.20	0.023	0.088	0.35	0.132	0.014
B:1			0.160		0.024	0.039	0.215	0.005	0.190	0.42	0.26	0.021	0.083	0.31	0.169	0.014
B:2			0.173		0.028	0.035	0.207	0.006	0.189	0.41	0.23	0.017	0.083	0.32	0.141	0.012
C:1			0.162		0.028	0.030	0.223		0.211	0.47	0.26	0.015	0.091	0.36	0.181	0.011
C:2			0.152		0.028	0.041	0.216	0.005	0.190	0.51	0.26	0.013	0.085	0.39	0.172	0.011
n_x	0	0	6	0	6	6	6	5	6	6	6	6	6	6	6	6
$Mean_x$			0.160		0.027	0.037	0.210	0.005	0.194	0.45	0.24	0.018	0.087	0.34	0.156	0.012
SD_x			0.007		0.003	0.005	0.009	0.001	0.009	0.04	0.02	0.004	0.003	0.03	0.020	0.001
SD_{repx}			0.007		0.003	0.006	0.005	0.001	0.009	0.02	0.02	0.003	0.002	0.01	0.013	0.001
SD_{betx}			0.005		0.001	0.001	0.010	0.001	0.006	0.04	0.02	0.003	0.003	0.03	0.020	0.001
SD_{NISTx}			0.009		0.004	0.006	0.011	0.001	0.011	0.04	0.03	0.004	0.004	0.03	0.023	0.002
CV_{NISTx}			5.5		13	17	5.1	15	5.5	9.8	12	24	4.5	9.6	15	13

	NIST			
n	6	6	12	5
Mean	0.027	0.037	0.185	0.005
SD_{rep}	0.004	0.005	0.006	0.001
SD_{bet}	0.001	0.001	0.010	0.001
SD_{anl}				
SD_{NIST}	0.004	0.005	0.011	0.001
CV_{NIST}	15	13	6.1	17

RR	XXXII
Serum	200
n_p	22
$Median_p$	0.020
eSD_p	0.009

XXXII
200
22
0.179
0.031

XXXII
200
1
0.103

	RRXLI					RRXLI				RRXLI			
	235	236	237	238	<— Current Results —>	235	236	237	238	235	236	237	238
n_n	23	25	26	17		25	26	26	20	7	7	7	5
$Median_n$	0.021	0.024	0.206	0.011		0.183	0.48	0.24	0.057	0.097	0.47	0.200	0.029
eSD_n	0.005	0.006	0.047	0.007		0.043	0.21	0.11	0.018	0.012	0.20	0.066	0.010
P(n=p)	0.95					0.97							
P(n<p)	0.99					0.07							
SD_{labs}	0.003	0.004	0.046	0.007		0.042	0.20	0.11	0.017	0.011	0.20	0.062	0.010
CV_{labs}	14	15	22	67		23	43	44	31	12	43	31	33
NAV	0.024	0.030	0.196	0.008	<— Assignments —>	0.189	0.47	0.24	0.037	0.092	0.40	0.178	0.021
NAU	0.005	0.006	0.047	0.007		0.043	0.21	0.11	0.018	0.012	0.20	0.066	0.010
CV	20	20	24	93		23	45	46	48	13	51	37	47
xCV	31	29	26	47		27	23	26	33				

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NIST Data and Value/Uncertainty Assignments

	β-Cryptoxanthin				"Lutein"				"Zeaxanthin"				"Lutein&Zeaxanthin"			
	NIST3				NIST3				NIST3				NIST3			
	235	236	237	238	235	236	237	238	235	236	237	238	235	236	237	238
A:1	0.021	0.096	0.345	0.007	0.060	0.441	0.065	0.04	0.025	0.051	0.062	0.027	0.085	0.492	0.127	0.067
A:2	0.022	0.099	0.334	0.008	0.059	0.447	0.073	0.04	0.023	0.051	0.066	0.025	0.082	0.498	0.139	0.065
B:1	0.021	0.093	0.381	0.008	0.054	0.412	0.067	0.05	0.022	0.049	0.061	0.029	0.076	0.461	0.128	0.076
B:2	0.020	0.093	0.333	0.007	0.051	0.408	0.062	0.05	0.022	0.048	0.059	0.029	0.073	0.456	0.121	0.076
C:1	0.022	0.101	0.384	0.006	0.055	0.433	0.065	0.04	0.026	0.050	0.064	0.029	0.081	0.483	0.129	0.068
C:2	0.021	0.100	0.384	0.007	0.055	0.433	0.068	0.04	0.026	0.050	0.063	0.026	0.081	0.483	0.131	0.063
n_x	6	6	6	6	6	6	6	6	6	6	6	6	6	6	6	6
$Mean_x$	0.021	0.097	0.360	0.007	0.056	0.429	0.067	0.04	0.024	0.050	0.063	0.028	0.080	0.479	0.129	0.069
SD_x	0.001	0.004	0.025	0.001	0.003	0.016	0.004	0.00	0.002	0.001	0.002	0.002	0.004	0.017	0.006	0.006
SD_{repx}	0.001	0.001	0.020	0.001	0.001	0.003	0.004	0.00	0.001	0.000	0.002	0.001	0.002	0.003	0.006	0.002
SD_{betx}	0.001	0.004	0.022	0.001	0.004	0.017	0.002	0.00	0.002	0.001	0.002	0.001	0.005	0.019	0.004	0.006
SD_{NISTx}	0.001	0.004	0.030	0.001	0.004	0.018	0.005	0.00	0.002	0.001	0.003	0.002	0.005	0.019	0.007	0.006
CV_{NISTx}	4.3	4.1	8.4	13	6.8	4.1	6.9	12	9.0	2.7	4.6	7.6	6.2	3.9	5.6	9.1

n	
Mean	
SD_{rep}	
SD_{bet}	
SD_{ani}	
SD_{NIST}	
CV_{NIST}	

RR	XXXII	XXXII	XXXII	XXXII
Serum	200	200	200	200
n_p	16	11	6	12
$Median_p$	0.041	0.064	0.020	0.094
eSD_p	0.016	0.014	0.005	0.027

	RRXLI				RRXLI				RRXLI				RRXLI			
	235	236	237	238	235	236	237	238	235	236	237	238	235	236	237	238
n_n	22	23	23	17	12	11	12	11	9	9	10	9	20	22	22	16
$Median_n$	0.026	0.125	0.419	0.018	0.070	0.502	0.079	0.08	0.023	0.063	0.059	0.044	0.087	0.575	0.145	0.111
eSD_n	0.008	0.035	0.083	0.010	0.013	0.049	0.030	0.07	0.008	0.010	0.022	0.026	0.019	0.100	0.025	0.046
P(n=p)	0.67				0.87				0.83				0.92			
P(n<p)	1.00				0.64				0.14				0.91			
SD_{labs}	0.008	0.035	0.078	0.010	0.012	0.046	0.029	0.07	0.008	0.010	0.022	0.026	0.018	0.098	0.024	0.045
CV_{labs}	32	28	19	53	17	9.2	37	84	35	16	37	59	21	17	16	41
NAV	0.023	0.111	0.389	0.013	0.063	0.466	0.073	0.06	0.024	0.056	0.061	0.036	0.083	0.527	0.137	0.090
NAU	0.008	0.035	0.083	0.010	0.013	0.049	0.030	0.07	0.008	0.010	0.022	0.026	0.019	0.100	0.025	0.046
CV	35	32	21	77	20	11	41	110	35	18	36	73	23	19	18	51
xCV	33	23	18	39												

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The attached N^2M^2QAP Round Robin XLI (RR41) Feedback includes:

Page	"All Lab" Report
1-4	A listing of all results for analytes reported by at least two laboratories, plus essential summary statistics
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
1	Your values, our assigned values, and the %bias between the two.
2, 3	"Comparisons" plots for: retinol, retinyl palmitate, α- and γ-tocopherol, total and *trans*-β-carotene, total α-carotene, total lycopene, β-cryptoxanthin, lutein, and lutein & zeaxanthin. (There are insufficient zeaxanthin data to justify a separate plot, given that we need space for a Legend... We do sum all individually reported lutein and zeaxanthin values into the "lutein & zeaxanthin" composite.)
4	"Z-Score Concordance" plots for this RR's: retinol, α-tocopherol, γ-tocopherol, total and *trans*-β-carotene, total α-carotene, and total and *trans*-lycopene.
5	%Bias Accuracy/Precision Summary of your last 3 years' results for: retinol, α-tocopherol, 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-tocopherol, γ-tocopherol, and/or total and *trans*-β-carotene.
7-9	%Bias barchart of your last 3 years' results for: retinol, α- and γ-tocopherol, and/or total and *trans*-β-carotene.

Page	"SRM 968b" Report
1, 2	"Comparisons" plots summarizing your RR32, RR34, RR39, RR40, and RR41 results for the low-, mid-, and high-level components of SRM 968b with analytes: retinol, retinyl palmitate, α- and γ-tocopherol, total and *trans*-β-carotene, total α-carotene, total lycopene, β-cryptoxanthin, lutein, and lutein & zeaxanthin. (Again, there is insufficient zeaxanthin data to justify an individual analyte plot.)

Appendix K. "All-Lab Report" for RR41

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 XLI Laboratory Results

Values in µg/mL

	Retinol				Retinyl Palmitate				α-Tocopherol				γ-Tocopherol				δ-Tocopherol			
Lab	235	236	237	238	235	236	237	238	235	236	237	238	235	236	237	238	235	236	237	238
FSV-BA	0.307	0.657	0.956	!0.323					7.07	11.44	19.3	!3.23	1.81	6.5	1.66	!1.42				
FSV-BD	0.283	0.620	0.889	!0.312					7.05	13.18	22.2	!4.96								
FSV-BE	0.266								7.17				1.70							
FSV-BF	0.310	0.647	0.941	!0.232					7.11	12.45	21.9	!2.06	1.70	6.2	1.12	!0.91				
FSV-BG	0.298	0.638	0.977	!0.303	0.082	0.30	0.092	!0.059	7.03	11.96	21.8	!3.71	1.72	6.5	1.29	!1.46				
FSV-BH	0.294	0.625	0.899	!0.319	0.056	0.26	0.064	!0.21	7.27	12.35	21.2	!5.9	1.49	5.6	1.10	!1.76	0.090	0.310	0.490	!1.04
FSV-BI	0.287	0.612	0.889	!0.304	0.075	0.36	0.118	!0.155	6.64	11.79	20.1	!4.42	1.59	6.3	1.12	!1.64				
FSV-BJ	0.273	0.593	0.791	!0.322	0.109	0.16	0.116	!0.199	7.67	12.48	23.0	!6.21	2.34	8.7	0.38	!2.81				
FSV-BK	0.311	0.654	0.930	!0.325					7.89	12.47	21.2	!5.03								
FSV-BL	0.290	0.460	0.740	!0.23					7.75	11.63	19.8	!5.6								
FSV-BM	0.287	0.593	0.864	!0.3					6.80	11.70	20.6	!3.8								
FSV-BN	0.274	0.612	0.934	!0.307	0.118	0.20	0.082	!0.178	6.88	10.99	19.2	!4.42	1.79	6.9	1.35	!1.9	0.310	0.379	0.651	!0.98
FSV-BO	0.267	0.570	0.773						6.50	11.03	19.6									
FSV-BP	0.276	0.578	0.825	!0.302					6.76	11.85	19.8									
FSV-BQ	0.324	0.711	0.966	!0.397					7.00	13.00	21.9	!5.5								
FSV-BR	0.280	0.610	0.890	!0.32																
FSV-BS																				
FSV-BT	0.364	0.589	0.859	!0.408	0.089	0.34	0.091	!0.196	5.76	10.96	19.3	!3.7	1.46	5.9	0.98	!1.31	0.130	0.431	0.627	!0.93
FSV-BU	0.351	0.587	0.851	!0.396					7.92	11.05	20.9	!5.76	2.19	5.3	1.83	!1.97				
FSV-BV	0.283	0.540	0.857						5.94	9.68	19.1		1.75	6.4	1.23					
FSV-BW	0.276	0.623	0.863	!0.303	0.063	0.14	0.044	!0.047	6.80	11.00	19.6	!3.52	1.96	6.9	1.25	!1.73				
FSV-BZ									6.60	11.80	21.2	!2.1	1.40	6.4	1.30	!0.95				
FSV-CA	0.234	0.504	0.735						5.63	8.79	16.1									
FSV-CB	0.250	0.560	0.790	!0.27					7.39	12.36	21.7	!4.55								
FSV-CC	0.294	0.646	0.931						6.41	11.17	19.5									
FSV-CD	0.278	0.519	0.804		0.109	0.20	0.093		7.34	11.90	19.9		1.53	6.5	1.03					
FSV-CE	0.283	0.635	0.841	!0.238					7.01	11.99	19.5	!2.84								
FSV-CF	0.275	!0.506	0.722	!0.289					6.40	!7.2	20.0	!3								
FSV-CG	0.237	0.605	0.894						5.48	9.03	15.3		1.83	6.0	1.29					
FSV-CH	0.248	0.544	0.760	!0.273					8.08	11.54	18.5	!5.21	1.24	4.7	0.79	!1.21				
FSV-CK	0.248	0.589	0.861						5.74	10.87	19.4		1.49	6.3	1.08					
FSV-CL	0.284	0.499	0.679	!0.249					12.19	15.83	21.6	!7	2.13	6.3	0.79	!1.43				
FSV-CM									7.20	10.90	20.7	<4.0								
FSV-CN	0.302	0.587	0.842	!0.282					7.11	12.12	20.8	!3.34	1.58	5.9	0.93	!1.17				
FSV-CQ	0.300	0.641	0.947	!0.331					7.62	12.31	20.9	!4								
FSV-CR	0.290	0.610	0.910	!0.26					6.80	11.30	20.4	!1.4					<0.3	0.400	0.600	!0.5
FSV-CS	0.267	0.595	0.888	!0.29																
FSV-CU	0.290	0.624	0.940	!0.322	0.056	0.19	0.051	!0.04	6.40	10.43	17.3	!2.49								
FSV-CX	0.260	0.550	0.810	!0.28	0.100	0.34	0.100	!0.1	6.91	11.52	20.7	!4.15	1.62	6.1	1.19	!1.55	0.090	0.170	0.550	!0.9
FSV-DA	0.276	0.626	0.954	!0.326	0.086	0.16	0.044	!0.035	5.98	10.98	20.4	!3.9	1.41	5.9	1.15	!1.53	0.076	0.371	0.540	!0.87
FSV-DB	0.285	0.635	0.880	!0.31					7.19	11.84	20.0	!4.52								
FSV-DJ	0.300	0.610	0.960	!0.29					6.50	11.80	20.7	!4								
FSV-DK	0.271	0.529	0.855	!0.26	0.037	0.11	0.032	!0.054	7.40	11.35	19.6	!4.57	2.00	6.9	1.19	!2.06				
FSV-DP	0.297	0.647	0.978	!0.318																
FSV-DQ	0.400	0.650	0.790						6.57	11.81	17.4		1.32	6.3	0.54					
FSV-DR	0.268	0.580	0.807	!0.258					6.91	11.74	20.0	!4.55								
FSV-DU	0.291	0.599	0.977	!0.329					7.48	10.47	19.3	!5.34								
FSV-EI	0.258	0.553	0.811						6.00	9.86	17.0		1.86	7.2	1.37					
FSV-EL	0.290	0.630	1.020	!0.3																
FSV-EM	0.240	0.530	0.830	!0.23					7.16	11.01	19.0	!2.71								
FSV-FN	0.279	0.571	0.884	!0.315					6.37	10.50	19.4	!4.04	1.60	5.8	1.18	!1.5				
n	48	46	47	38	12	12	12	11	46	44	45	34	25	24	24	18	5	6	6	6
Min	0.234	0.460	0.679	0.230	0.037	0.11	0.032	0.04	5.48	8.79	15.3	1.4	1.24	4.7	0.38	0.91	0.076	0.170	0.490	0.50
Median	0.283	0.602	0.864	0.303	0.084	0.20	0.086	0.10	6.96	11.58	20.0	4.1	1.70	6.3	1.16	1.52	0.090	0.375	0.575	0.92
Max	0.400	0.711	1.020	0.408	0.118	0.36	0.118	0.21	12.19	15.83	23.0	7.0	2.34	8.7	1.83	2.81	0.310	0.431	0.651	1.04
eSD	0.019	0.047	0.089	0.030	0.034	0.09	0.039	0.09	0.61	0.86	1.1	1.3	0.25	0.4	0.19	0.34	0.021	0.060	0.064	0.08
eCV	7	8	10	10	41	45	45	89	9	7	5	31	15	7	17	23	23	16	11	9
NISTa	0.301	0.589	0.883	!0.224					7.56	12.24	20.9	!2.86	1.86	6.9	1.15	!1.17	*nd*	0.420	0.574	!0.6
NISTb	0.298	0.591	0.894	!0.242					6.97	11.27	20.3	!2.71	1.57	5.7	1.06	!1.05	0.091	0.326	0.529	!0.62
NAV	0.291	0.596	0.876	0.268	0.084	0.20	0.086	0.10	7.11	11.67	20.3	3.4	1.71	6.3	1.13	1.31	0.091	0.374	0.563	0.76
NAU	0.031	0.051	0.092	0.060	0.034	0.09	0.039	0.09	0.88	0.96	1.7	1.5	0.28	1.1	0.21	0.42	0.030	0.077	0.060	0.22

Round Robin XLI Laboratory Results

Values in µg/mL

	Total β-Carotene				trans-β-Carotene				Total cis-β-Carotene				Total α-Carotene			
Lab	235	236	237	238	235	236	237	238	235	236	237	238	235	236	237	238
FSV-BA	0.255	0.301	0.35	!0.085	0.245	0.291	0.333	!0.082	0.010	0.010	0.016	!0.003	0.026	0.037	0.160	!0.01
FSV-BD	0.261	0.303	0.44	!0.11												
FSV-BE	0.239															
FSV-BF	0.239	0.304	0.43	!0.036									0.027	0.034	0.325	!0.01
FSV-BG	0.236	0.304	0.43	!0.074									0.021	0.028	0.219	!0.009
FSV-BH	0.242	0.292	0.43	!0.105	0.229	0.277	0.407	!0.105	0.013	0.015	0.020	<0.01	0.015	0.024	0.266	!0.063
FSV-BI	0.226	0.280	0.35	!0.077									0.018	0.020	0.184	!0.013
FSV-BJ	0.229	0.268	0.31	!0.105									0.021	0.033	0.153	!0.027
FSV-BK																
FSV-BL																
FSV-BM																
FSV-BN	0.243	0.280	0.46	!0.112	0.217	0.255	0.421	!0.092	0.015	0.015	0.025	!0.008	0.017	0.024	0.268	!0.011
FSV-BO	0.212	0.255	0.30										0.021	0.027	0.180	
FSV-BP	0.470	0.488	0.69	!0.212									0.042	0.045	0.336	!0.036
FSV-BQ																
FSV-BR																
FSV-BS		>0.260	>0.290			0.260	0.290	!0.079						0.024	0.160	*nq*
FSV-BT	0.262	0.267	0.38	!0.084	0.245	0.255	0.357	!0.079	0.017	0.012	0.019	!0.005	0.019	0.022	0.214	!0.026
FSV-BU	0.270	0.333	0.45	!0.121									0.015	0.024	0.230	!0.02
FSV-BV	0.251	0.294	0.40										0.014	0.021	0.202	
FSV-BW	0.278	0.365	0.46	!0.086									*nq*	0.017	0.223	*nq*
FSV-BZ	>0.246	>0.290	>0.370		0.246	0.290	0.370	!0.08	0.010	0.009	0.009	*nd*	0.030	0.030	0.140	!0.02
FSV-CA																
FSV-CB																
FSV-CC																
FSV-CD	0.145	0.274	0.28										0.011	0.016	0.161	
FSV-CE	0.220	0.260	0.49	!0.062												
FSV-CF																
FSV-CG	0.288	0.327	0.48										0.035	0.041	0.210	
FSV-CH	0.206	0.248	0.36	!0.078									0.009	0.013	0.192	!0.008
FSV-CK	0.200	0.210	0.29										0.022	0.024	0.190	
FSV-CL	0.204	0.185	0.22	!0.052									0.021	0.028	0.152	!0.009
FSV-CM																
FSV-CN	>0.213	>0.259	>0.361		0.213	0.259	0.361	!0.051					*nq*	*nq*	0.222	*nq*
FSV-CQ	0.741	0.245	0.32	!0.012												
FSV-CR																
FSV-CS	0.277	0.290	0.41	!0.088	0.248	0.263	0.372	!0.08	*0.029*	*0.027*	*0.035*		0.028	0.030	0.268	!0.014
FSV-CU																
FSV-CX	0.160	0.230	0.27	!0.02									0.010	0.010	0.120	!0.01
FSV-DA	0.214	0.261	0.41	!0.066	0.189	0.233	0.380	!0.064	0.025	0.028	0.033	!0.002	0.019	0.038	0.217	!0.009
FSV-DB	0.211	0.223	0.28	!0.076												
FSV-DJ																
FSV-DK	0.250	0.300	0.36	!0.085									0.019	0.027	0.196	!0.011
FSV-DP																
FSV-DQ	0.290	0.320	0.29										0.021	0.022	0.096	
FSV-DR	0.213	0.251	0.26	!0.09												
FSV-DU	>0.188	>0.282	>0.392		0.188	0.282	0.392	!0.122								
FSV-EI	>0.226	>0.252	>0.435		0.226	0.252	0.435						0.018	0.023	0.248	
FSV-EL																
FSV-EM																
FSV-FN																
n	29	28	28	22	10	11	11	10	7	7	7	6	24	26	27	17
Min	0.145	0.185	0.22	0.012	0.188	0.233	0.290	0.051	0.010	0.009	0.009	0.002	0.009	0.010	0.096	0.008
Median	0.239	0.280	0.37	0.085	0.228	0.260	0.372	0.080	0.015	0.015	0.020	0.004	0.020	0.024	0.202	0.011
Max	0.741	0.488	0.69	0.212	0.248	0.291	0.435	0.122	0.029	0.028	0.035	0.008	0.042	0.045	0.336	0.063
eSD	0.039	0.036	0.10	0.029	0.026	0.012	0.030	0.010	0.007	0.007	0.007		0.006	0.006	0.061	0.003
eCV	16	13	27	34	11	5	8	13	49	49	37		30	25	30	27
NISTa	0.245	0.304	0.37	!0.051	0.214	0.205	0.298	!0.043	*0.031*	*0.099*	*0.068*		*nd*	*nd*	0.160	*nd*
NISTb	0.237	0.261	0.37	!0.044	0.221	0.250	0.358	!0.044	*0.017*	*0.011*	*0.011*		0.027	0.037	0.210	!0.005
NAV	0.240	0.282	0.37	0.066	0.222	0.243	0.350	0.062	0.019	0.036	0.030		0.023	0.030	0.194	0.008
NAU	0.038	0.043	0.10	0.036	0.027	0.040	0.065	0.028	0.021	0.066	0.064		0.008	0.012	0.056	0.009

Round Robin XLI Laboratory Results

Values in µg/mL

	Total Lycopene				trans-Lycopene				β-Cryptoxanthin				α-Cryptoxanthin			
Lab	235	236	237	238	235	236	237	238	235	236	237	238	235	236	237	238
FSV-BA					0.110	0.33	0.152	!0.015	0.043	0.146	0.486	!0.017				
FSV-BD	0.140	0.38	0.18	!0.062					0.022	0.093	0.344	!0.02				
FSV-BE																
FSV-BF	0.183	0.80	0.39	!0.036					0.019	0.116	0.438	!0.011				
FSV-BG	0.220	0.60	0.34	!0.071	0.100	0.47	0.222	!0.028	0.021	0.013	0.056	!0.018				
FSV-BH	0.198	0.82	0.35	!0.192					0.035	0.146	0.492	!0.05				
FSV-BI	0.138	0.33	0.17	!0.042					0.031	0.147	0.529	!0.024				
FSV-BJ	0.122	0.33	0.18	!0.065												
FSV-BK																
FSV-BL																
FSV-BM																
FSV-BN	0.183	0.86	0.38	!0.054	0.092	0.74	0.288	!0.041	0.031	0.154	0.600	!0.025	0.016	0.017	0.031	!0.007
FSV-BO	0.273	0.61	0.31						0.027	0.122	0.395					
FSV-BP	0.305	0.86	0.42	!0.085					0.017	0.067	0.221	!0.007				
FSV-BQ																
FSV-BR																
FSV-BS		0.60	0.23	!0.07						0.150	0.460	!0.023				
FSV-BT	0.187	0.43	0.21	!0.061	0.148	0.39	0.180	!0.054	0.036	0.131	0.419	!0.033	0.020	0.015	0.014	!0.005
FSV-BU	0.205	0.59	0.27	!0.042					0.031	0.150	0.463	!0.034				
FSV-BV	0.170	0.69	0.26						0.018	0.115	0.429					
FSV-BW	0.265	0.76	0.36	!0.054												
FSV-BZ	0.230	0.56	0.33	!0.14												
FSV-CA																
FSV-CB																
FSV-CC																
FSV-CD	0.117	0.28	0.14						0.023	0.103	0.257					
FSV-CE																
FSV-CF																
FSV-CG	0.146	0.46	0.23						0.029	0.125	0.417					
FSV-CH	0.083	0.34	0.17	!0.029												
FSV-CK	0.119	0.32	0.16		0.001	0.00	0.002		0.028	0.115	0.373		0.020	0.028	0.030	
FSV-CL	0.197	0.44	0.26	!0.06					0.020	0.099	0.269	!0.01				
FSV-CM																
FSV-CN	0.095	0.46	0.19	*nq*					0.019	0.136	0.478	!0.009				
FSV-CQ																
FSV-CR																
FSV-CS	0.184	0.36	0.22	!0.053					0.024	0.098	0.356	!0.017				
FSV-CU																
FSV-CX	0.120	0.45	0.12	!0.01					0.020	0.170	0.360	!0.01				
FSV-DA	0.191	0.81	0.43	!0.049	0.097	0.71	0.336	!0.029	0.024	0.079	0.398	!0.013	0.015	0.011	0.023	!0.005
FSV-DB	0.198	0.42	0.22	!0.062					0.033	0.140	0.458	!0.023				
FSV-DJ																
FSV-DK	0.159	0.50	0.25	!0.033												
FSV-DP																
FSV-DQ	0.167	0.17	0.15						0.020	0.072	0.235					
FSV-DR																
FSV-DU																
FSV-EI					0.086	0.56	0.200		0.027	0.134	0.485					
FSV-EL																
FSV-EM																
FSV-FN																
n	26	27	27	20	7	7	7	5	23	24	24	17	4	4	4	3
Min	0.083	0.17	0.12	0.010	0.001	0.00	0.002	0.015	0.017	0.013	0.056	0.007	0.015	0.011	0.014	0.005
Median	0.183	0.46	0.23	0.057	0.097	0.47	0.200	0.029	0.024	0.124	0.418	0.018	0.018	0.016	0.027	0.005
Max	0.305	0.86	0.43	0.192	0.148	0.74	0.336	0.054	0.043	0.170	0.600	0.050	0.020	0.028	0.031	0.007
eSD	0.055	0.19	0.10	0.020	0.016	0.20	0.071	0.018	0.007	0.034	0.090	0.010				
eCV	30	42	42	35	17	43	36	61	31	27	22	58				
NISTa																
NISTb	0.194	0.45	0.24	!0.018	0.087	0.34	0.156	!0.012	0.021	0.097	0.360	!0.007				
NAV	0.189	0.46	0.24	0.037	0.092	0.40	0.178	0.021	0.023	0.110	0.389	0.013				
NAU	0.048	0.21	0.11	0.033	0.022	0.22	0.073	0.015	0.008	0.040	0.095	0.012				

Round Robin XLI Laboratory Results

Values in µg/mL

	Lutein				Zeaxanthin				Lutein&Zeaxanthin				Total Carotenoids			
Lab	235	236	237	238	235	236	237	238	235	236	237	238	235	236	237	238
FSV-BA									0.125	0.87	0.217	!0.235				
FSV-BD	0.070	0.47	0.064	!0.079	0.023	0.051	0.055	!0.057	*0.093*	*0.52*	*0.119*	*!0.136*				
FSV-BE																
FSV-BF									0.102	0.60	0.163	!0.08				
FSV-BG	0.071	0.53	0.130	!0.081	0.015	0.063	0.024	!0.013	*0.086*	*0.59*	*0.154*	*!0.094*				
FSV-BH	0.054	0.46	0.069	!0.17	<0.02	0.046	0.063	!0.31	*<0.07*	*0.51*	*0.132*	*!0.48*				
FSV-BI	0.054	0.47	0.058	!0.064	0.020	0.053	0.048	!0.044	*0.074*	*0.52*	*0.106*	*!0.108*				
FSV-BJ																
FSV-BK																
FSV-BL																
FSV-BM																
FSV-BN	0.062	0.54	0.076	!0.066	0.023	0.098	0.091	!0.063	0.074	0.58	0.136	!0.106				
FSV-BO									0.043	0.37	0.063					
FSV-BP																
FSV-BQ																
FSV-BR																
FSV-BS										0.67	0.150	!0.19				
FSV-BT	0.088	0.44	0.107	!0.243	0.031	0.019	0.027	!0.022	0.119	0.46	0.134	!0.265				
FSV-BU									0.086	0.66	0.165	!0.136				
FSV-BV									0.090	0.58	0.155					
FSV-BW																
FSV-BZ	0.080	0.97	0.120	!0.54												
FSV-CA																
FSV-CB																
FSV-CC																
FSV-CD									0.075	0.53	0.124					
FSV-CE																
FSV-CF																
FSV-CG									0.091	0.63	0.175					
FSV-CH																
FSV-CK									0.088	0.49	0.144					
FSV-CL									0.080	0.38	0.086	!0.072				
FSV-CM																
FSV-CN	0.062	0.44	0.069	!0.063	0.037	0.065	0.078	!0.05	0.099	0.50	0.147	!0.113				
FSV-CQ																
FSV-CR																
FSV-CS									0.077	0.46	0.129	!0.15				
FSV-CU																
FSV-CX	0.090		0.100	!0.08	0.020		0.050	!0.02	*0.110*	*1.13*	*0.150*	*!0.1*				
FSV-DA	0.046	0.53	0.075	!0.057	0.025	0.063	0.078	!0.044	*0.071*	*0.59*	*0.153*	*!0.101*				
FSV-DB									0.072	0.45	0.117	!0.08				
FSV-DJ																
FSV-DK	0.076	0.50	0.114	!0.141												
FSV-DP																
FSV-DQ									0.072	0.62	0.072					
FSV-DR																
FSV-DU																
FSV-EI	0.069	0.55	0.082		0.033	0.085	0.086		*0.102*	*0.64*	*0.168*					
FSV-EL																
FSV-EM													0.70	1.44	1.40	!0.27
FSV-FN																
n	12	11	12	11	9	9	10	9	21	23	23	16	1	1	1	1
Min	0.046	0.44	0.058	0.057	0.015	0.019	0.024	0.013	0.043	0.37	0.063	0.072				
Median	0.070	0.50	0.079	0.080	0.023	0.063	0.059	0.044	0.086	0.58	0.144	0.111	0.70	1.44	1.40	0.27
Max	0.090	0.97	0.130	0.540	0.037	0.098	0.091	0.310	0.125	1.13	0.217	0.480				
eSD	0.013	0.05	0.027	0.025	0.004	0.018	0.028	0.028	0.018	0.10	0.028	0.042				
eCV	19	10	34	32	19	28	48	64	21	18	20	38				
NISTa																
NISTb	0.056	0.43	0.067	!0.042	0.024	0.050	0.063	!0.028	*0.080*	*0.48*	*0.129*		0.59	1.30	1.35	!0.169
NAV	0.063	0.47	0.073	0.061	0.024	0.056	0.061	0.036	0.083	0.53	0.136	0.090				
NAU	0.018	0.13	0.031	0.072	0.008	0.020	0.022	0.028	0.019	0.14	0.033	0.054				

Round Robin XLI Laboratory Results

Analytes Reported By One Laboratory

Values in µg/mL

Analyte	Code	235	236	237	238
Coenzyme Q10	FSV-CH	0.243	0.322	0.326	!0.109
Total cis-β-Cryptoxanthin	FSV-BT	0.012	0.007	0.008	!0.005
trans-Anhydro-Lutein	FSV-BT	0.040	0.046	0.041	!0.017
trans-α-Carotene	NISTb	0.019	0.024	0.197	!0.005

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 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 than or equal to x
!	Discrepant value: heterogeneous serum, damaged sample, malfunction, etc.
italics	Not explicitly reported but calculated by NIST from reported values

Round Robin XLI Laboratory Results

Comparability Summary

Lab	R	aT	gT	bC	tbC
FSV-BA	1	1	1	1	
FSV-BD	2	1	1	1	
FSV-BE	2	3			
FSV-BF	1	2		1	
FSV-BG	1	1	1	1	
FSV-BH	1	1		4	
FSV-BI	1	1	1	1	
FSV-BJ	2	1	3	1	2
FSV-BK	1	1	1	1	1
FSV-BL	2	2	1	2	
FSV-BM		1	2		2
FSV-BN		1			
FSV-BO	3	1			
FSV-BP	1	2	4	1	
FSV-BQ	2	1			
FSV-BR	1	2			
FSV-BS	2	2	2	1	
FSV-BT	3	4	2	3	
FSV-BU	1	1	1	3	
FSV-BV	2	1			
FSV-BW	1	1		4	
FSV-BZ	1	1	2	1	2
FSV-CA	2	2	2		2
FSV-CB					1
FSV-CC	2	1		1	
FSV-CD	3	2	1	1	1
FSV-CE	1				
FSV-CF	3	2			
FSV-CG	1	1			
FSV-CH	2	3	1	2	
FSV-CK	1	1			
FSV-CL	1	1			
FSV-CM	2	1	1	3	
FSV-CN	2	1	2	1	
FSV-CQ	1	1		2	
FSV-CR	2	1	4	2	
FSV-CS	1	2	2	1	2
FSV-CU	2				
FSV-CX	2				
FSV-DA	2	3	1	1	
FSV-DB	1	1	1		1
FSV-DJ	1	1			
FSV-DK	1	1	1	2	
FSV-DP	2	1			
FSV-DQ	1			1	1
FSV-DR	2	2			2
FSV-DU	1	1		2	
FSV-EI	4	2	3	2	
FSV-EL	2	1			
FSV-EM	1	2	1		
FSV-FN	1	1		2	
NISTa	1	1	1	1	1
NISTb	1	1	1	1	1
n	48	46	25	29	11

Label	Definition
Lab	laboratory number
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 (non-NIST) 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)	{i.e., ±1 SD}
2	All StV within ±*t* (1-0.954,n-1)	{i.e., ±2 SD}
3	All StV within ±*t* (1-0.997,n-1)	{i.e., ±3 SD}
4	At least one StV > ±*t* (1-0.997,n-1)	{i.e., >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

Lab	% Observed					Expected
1	73	77	77	71	69	68.2 %
2	22	17	12	23	31	27.3 %
3	4	4	8	0	0	4.3 %
4	0	2	4	6	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 RR41

Each participant in RR41 received an "Individualized Report" reflecting their reported results. Each report included a detailed analysis of the results they reported for the following analytes:

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

In addition, each participant in RR41 received an "SRM 968b Report" that summarized their results for the three components of SRM 968b in RR32, RR34, RR39, RR40, as well as in RR41. This summary addressed all of the above analytes except *trans*-Lycopene.

The software systems used to generate the original "Individualized Report" and the "SRM 968b Report" are no longer available and we do not have hardcopy of the report sent to participant FSV-BA. We do, however, have hardcopy for a NIST analyst; pages L2 to L10 are the "Individualized Report" and pages L11 and L12 are the "SRM 968b Report" provided to analyst NISTb.

Pages L13 to L22 were produced for the results of participant FSV-BA using a descendant of the 1997 software. Although the form of the presentation has changed radically, three of the graphical tools used in the original reports ("Boxplot Comparisons", "Z-Score Concordance", and "NIST Assigned Values Vs Laboratory Values") have been retained and display the same information in much the same manner as in the original. The current software does not provide the "% RSD Bias and Precision History" table nor the "% Difference" plots.

Individualized Round Robin XLI Report: NISTb

Your Data, NIST Assigned Values, and %Differences

	Serum 235				Serum 236				Serum 237				Serum 238			
Analyte	You	NAV	%Δ	n	You	NAV	%Δ	n	You	NAV	%Δ	n	You	NAV	%Δ	n
Retinol	0.298	0.291	2	46	0.591	0.596	-1	46	0.894	0.880	2	46	*0.242			38
α-Tocopherol	6.97	7.09	-2	44	11.3	11.6	-3	44	20.3	20.3	0	44	*2.71			34
γ-Tocopherol	1.57	1.71	-8	23	5.67	6.28	-10	23	1.06	1.14	-7	23	*1.05			18
δ-Tocopherol	0.091	0.091	1	5	0.326	0.374	-13	6	0.529	0.563	-6	6	*0.62			6
Total β-Carotene	0.237	0.242	-2	27	0.261	0.281	-7	27	0.369	0.372	-1	27	*0.044			22
trans-β-Carotene	0.221	0.222	-1	10	0.250	0.244	2	11	0.358	0.350	2	11	*0.044			10
Total α-Carotene	0.027	0.024	12	23	0.037	0.030	21	25	0.210	0.196	7	26	*0.005			17
trans-α-Carotene	0.019				0.024				0.197				*0.005			
Total Lycopene	0.194	0.189	3	25	0.453	0.467	-3	26	0.240	0.242	-1	26	*0.018			20
trans-Lycopene	0.087	0.092	-6	7	0.342	0.404	-15	7	0.156	0.178	-12	7	*0.012			5
β-Cryptoxanthin	0.021	0.023	-9	22	0.097	0.111	-13	23	0.360	0.389	-8	23	*0.007			17
Total Carotenoids	0.586			1	1.30			1	1.35			1	*0.169			1
"Lutein&Zeaxanthin"	0.080	0.083	-5	20	0.479	0.527	-9	22	0.129	0.137	-6	22	*0.069			16
"Lutein"	0.056	0.063	-11	12	0.429	0.466	-8	11	0.067	0.073	-8	12	*0.042			11
"Zeaxanthin"	0.024	0.024	2	9	0.050	0.056	-12	9	0.063	0.061	3	10	*0.028			9

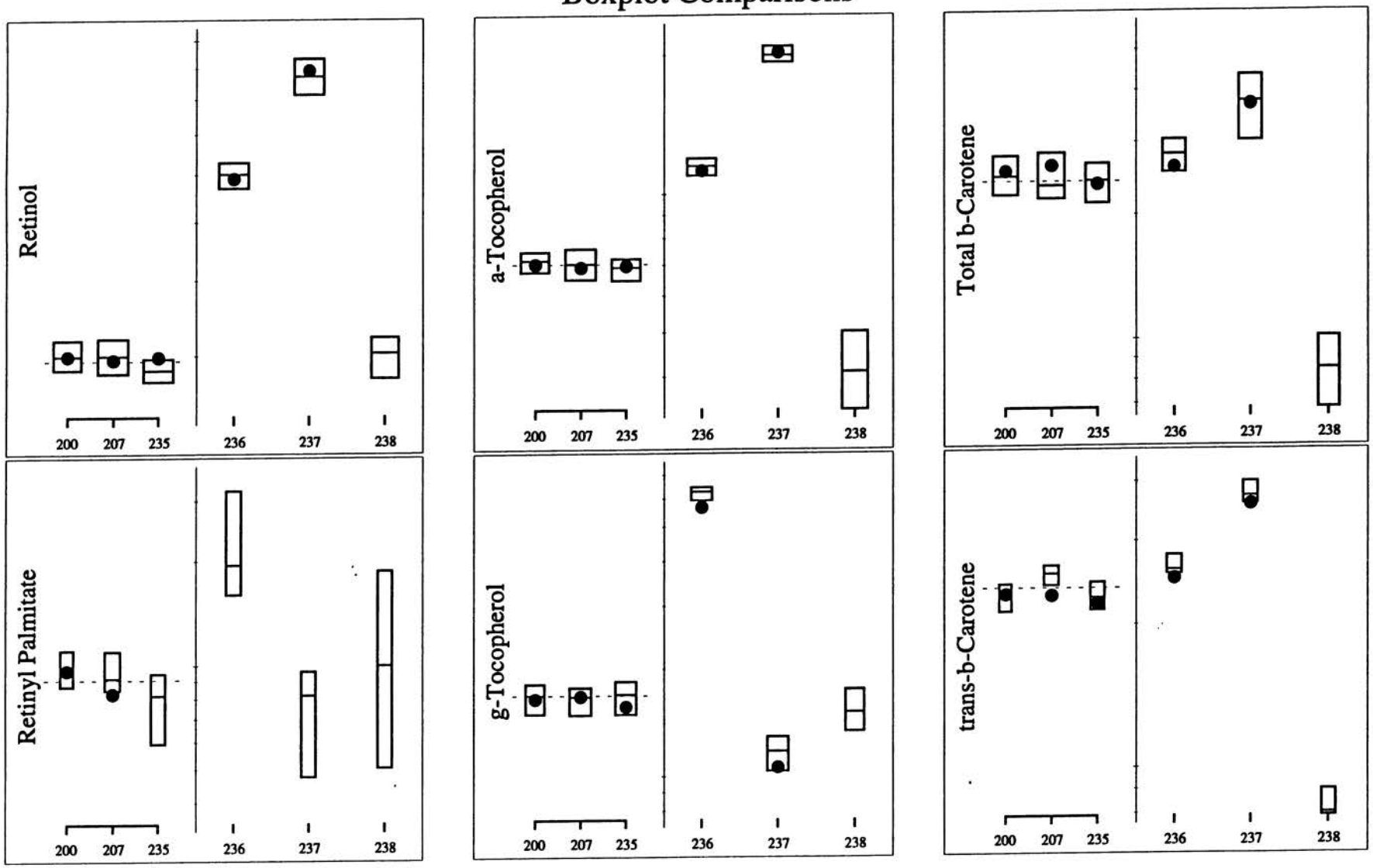
L2

You : Your reported values for the listed analytes (micrograms/milliliter)
NAV : NIST Assigned Values, equal to (NIST's average-of-averages + this Round Robin's median) / 2
%Δ : Percent difference between your value and the NAV
* : Non-quantitative value: heterogeneous serum, damaged sample, procedural error, etc.

Please check our recorded values against your records.
Send corrections to: N^2M^2QAP 222/B208, NIST, Gaithersburg, MD 20899; fax 301-977-0685; email David.Duewer@NIST.gov

Individualized Round Robin XLI Report: NISTb

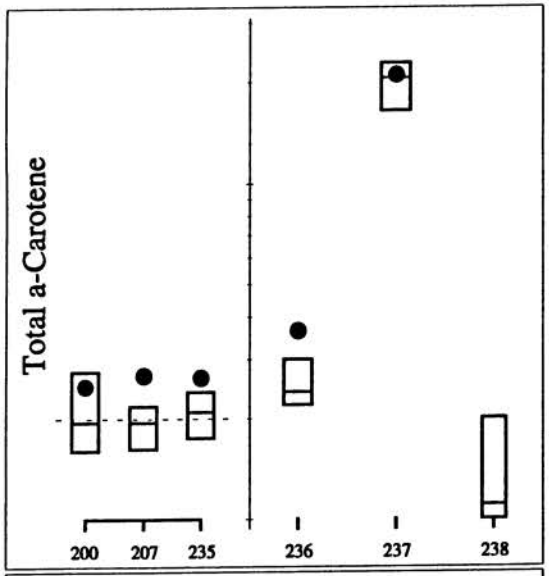
Boxplot Comparisons

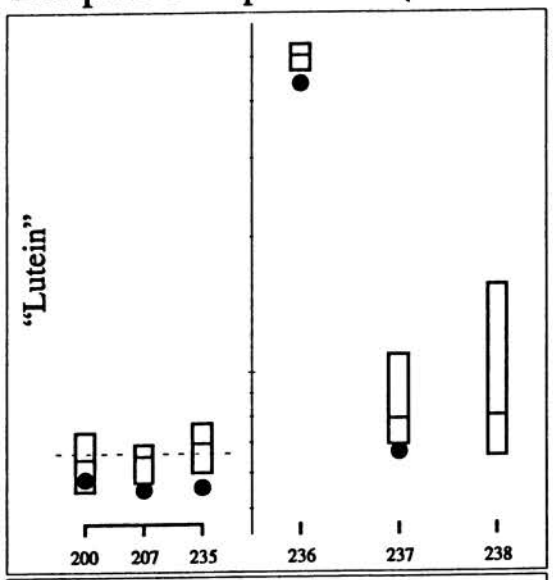


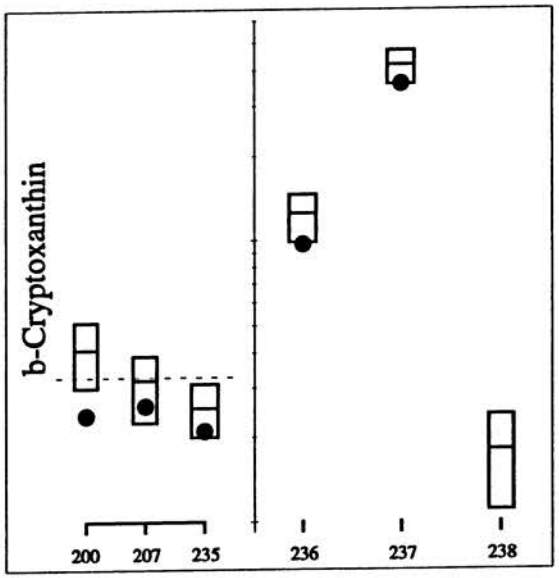
L3

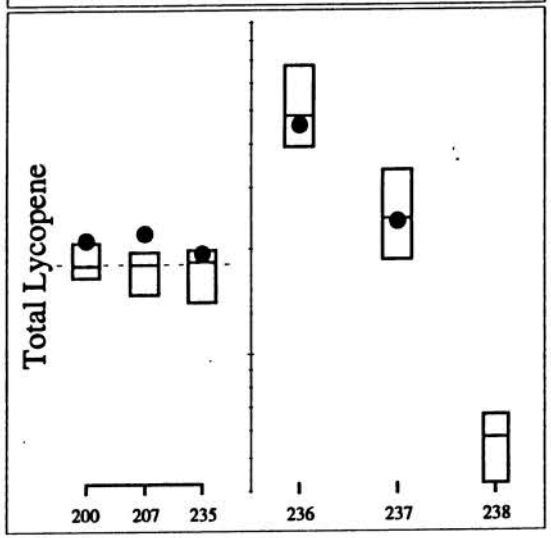
Individualized Round Robin XLI Report: NISTb

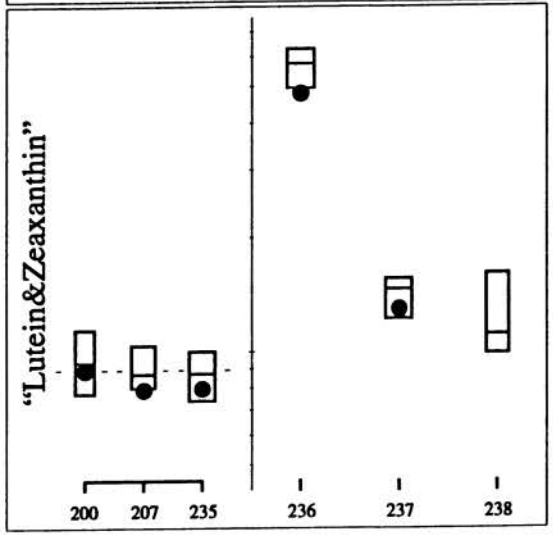
Boxplot Comparisons (Continued)











Legend

Solid circles: Your data

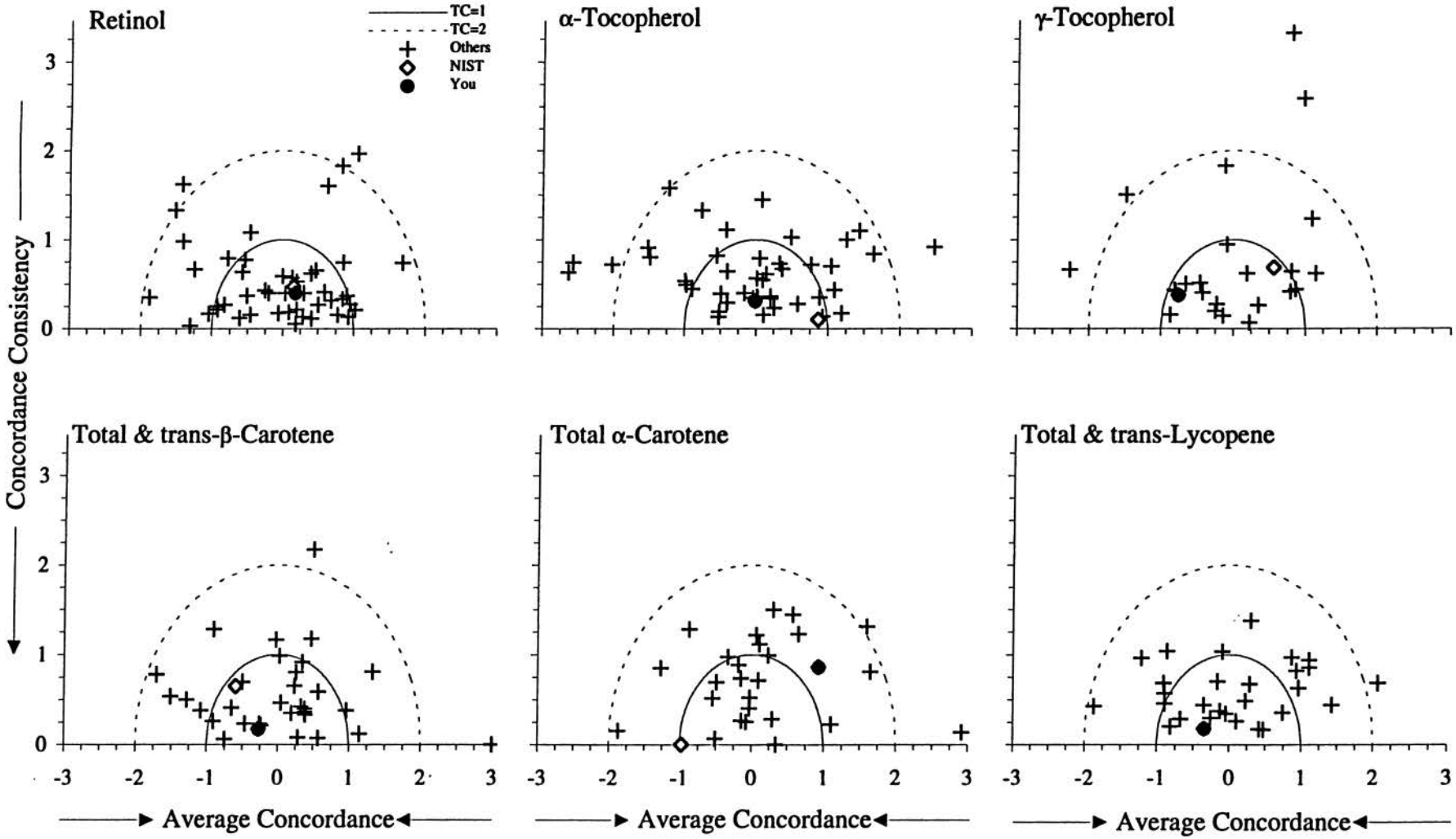
Box plots: Top - 75% of values smaller than this
Bottom - 75% of values greater than this
Middle - Median value
Width - Proportional to number of values

200,207,235: Low-level of SRM 968b
236: New serum
237: New serum
238: New, poorly reconstitutable serum

L4

Individualized Round Robin XLI Report: NISTb

Z-Score Concordance



L5

Individualized Round Robin XLI Report: NISTb

%RSD Bias and Precision History

	Retinol		α-Tocopherol		γ-Tocopherol		β-Carotene Total		β-Carotene *trans*	
RR	mΔ	vΔ	mΔ	vΔ	mΔ	vΔ	mΔ	vΔ	mΔ	vΔ
XXXII	0	1	0	0	1	1	2	1	3	0
XXXIII	4	3	-8	2	-2	5	-28	16	-26	13
XXXIV	1	3	-1	0	2	2	3	1	-1	3
XXXV	-3	4	-7	6	-11	4	-3	4	-4	4
XXXVI	1	1	3	1	2	3	-2	3	0	3
XXXVII	-3	3	1	4	2	4	-6	6	-3	6
XXXVIII	-4	3	1	3	5	2	1	7	4	6
XXXIX	-4	3	-6	4	-9	3	-16	9	-10	7
XL	0	1	-1	2	-1	3	0	4	-4	5
XLI	1	2	-2	2	-8	1	-3	3	1	2

L6

%Δ was traditionally evaluated

%Δ	Evaluation
0-5%	Exceptional
6-10%	Acceptable
11-20%	Marginal
> 20%	Poor

However, there is no single set of criteria that is appropriate for all analytes, at all levels, for all purposes.

Interpretation

Precision and bias are separate aspects of measurement comparison.
Precision can be estimated as vΔ, the standard deviation of %Δ, and bias as mΔ, the average %Δ, for all (valid) sera of a Round Robin (RR).

%Δ Percent relative difference from Nist Assigned Value (NAV).
%Δ = 100(Your value - NAV) / NAV

NAV NIST Assigned Value, our best estimate of analyte concentration
NAV = (NIST's average-of-averages + Round Robin median) / 2

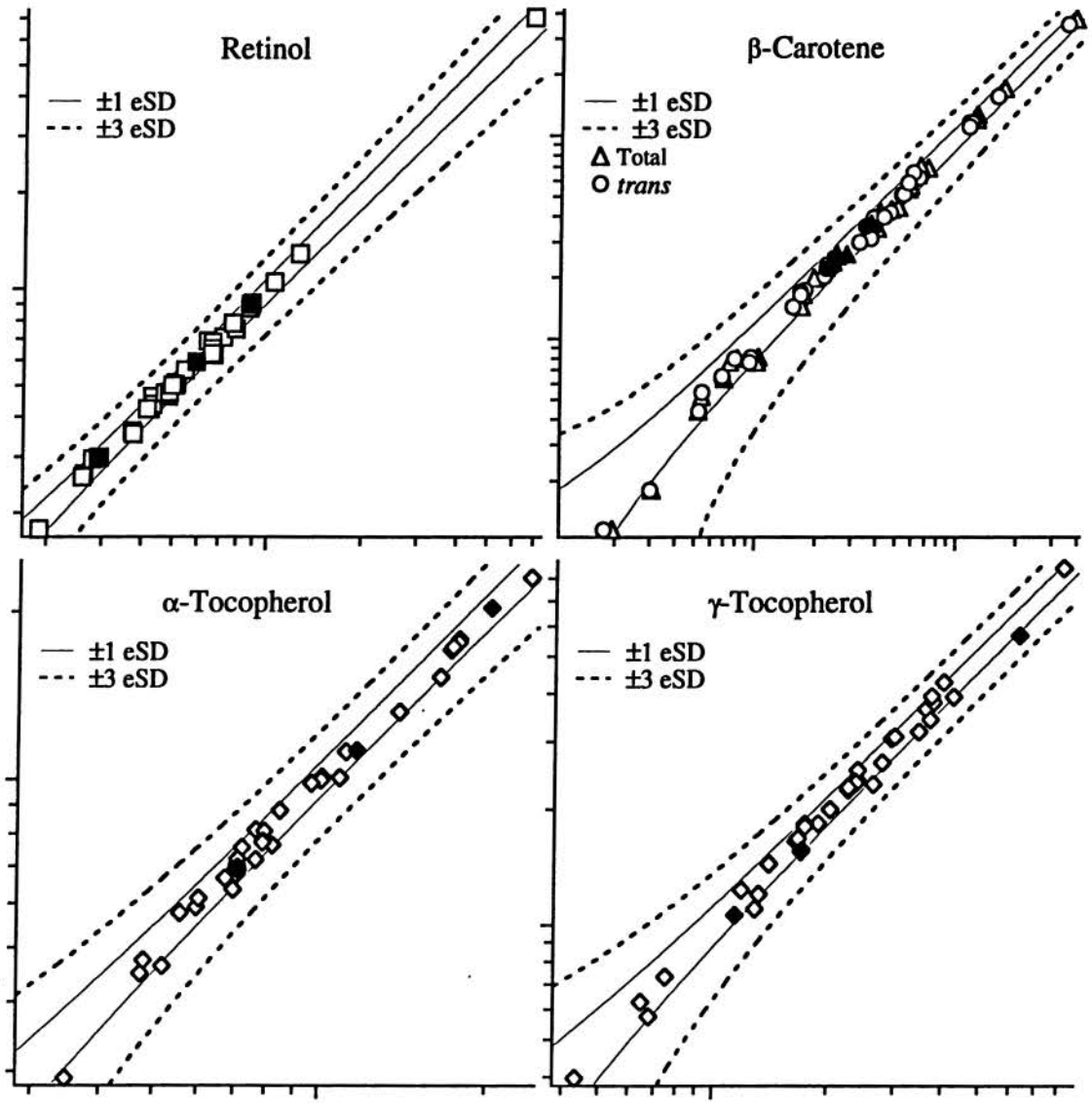
mΔ Mean difference, the average %Δ for all RR's sera

vΔ Difference variability, one standard deviation of %Δ for all RR's sera

It's best to be unbiased and precise (small mΔ, small vΔ)!
Good precision (small vΔ) with bias (large mΔ) is better than the converse: such values are internally consistent and may be related to others' values once the relative biases are known.
Poor precision (large vΔ) suggests that your measurement system is not in adequate control for the analyte levels examined.

Individualized Round Robin XLI Report: NISTb

NIST Assigned Values Vs Laboratory NIST3 Values



Legend

Open Symbols: RR XXXII-XL
Black Symbols: RR XLI

Abscissa (X-axis): NIST Assigned Value
Ordinate (Y-axis): Your reported value

Interpretation

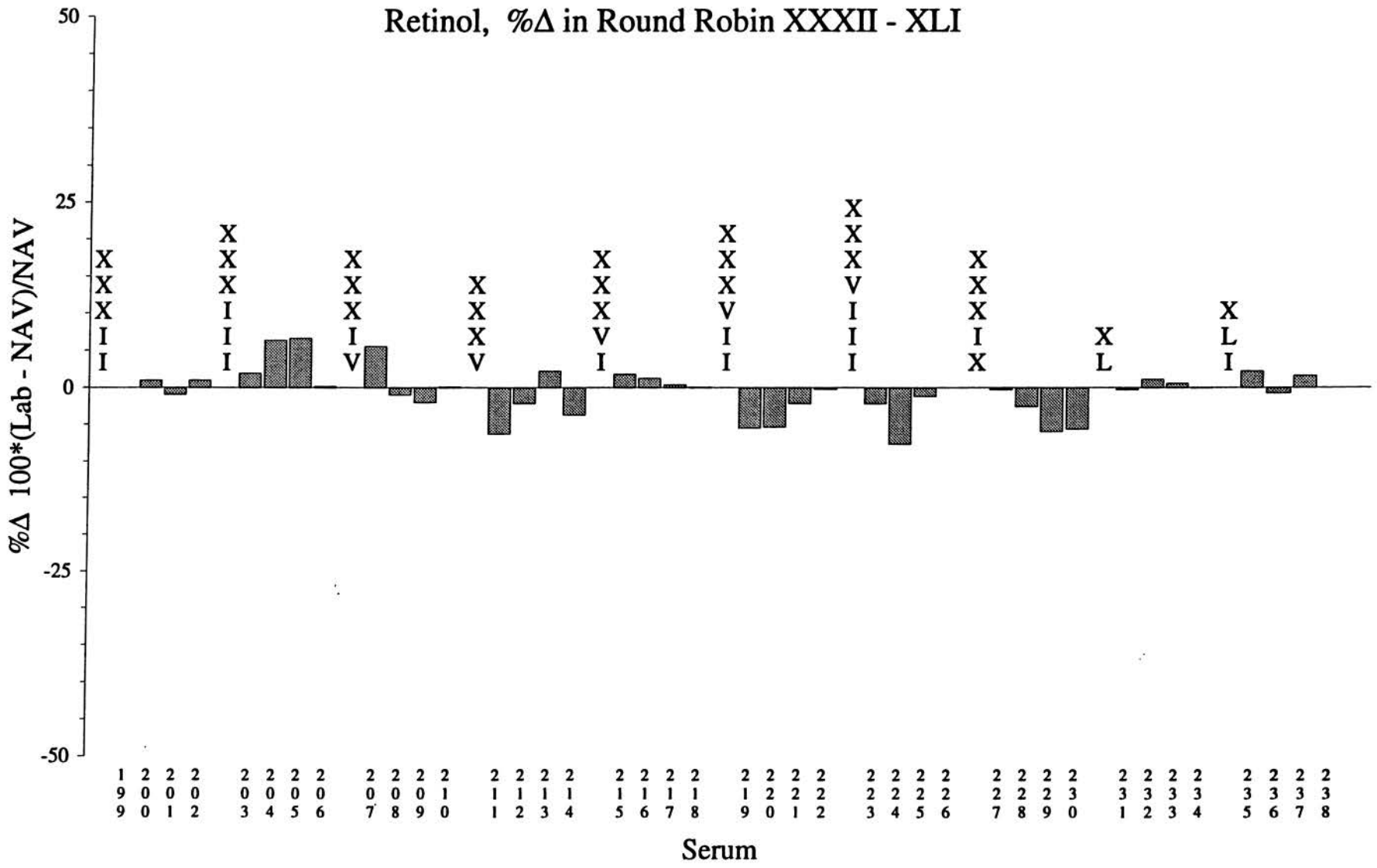
If much of your data is scattered outside the ±3 eSD lines, your measurement system is inadequately precise.

If your data are systematically higher or lower the NIST Assigned Value, your measurement system is biased relative to that of the majority of N^2M^2QAP participants.

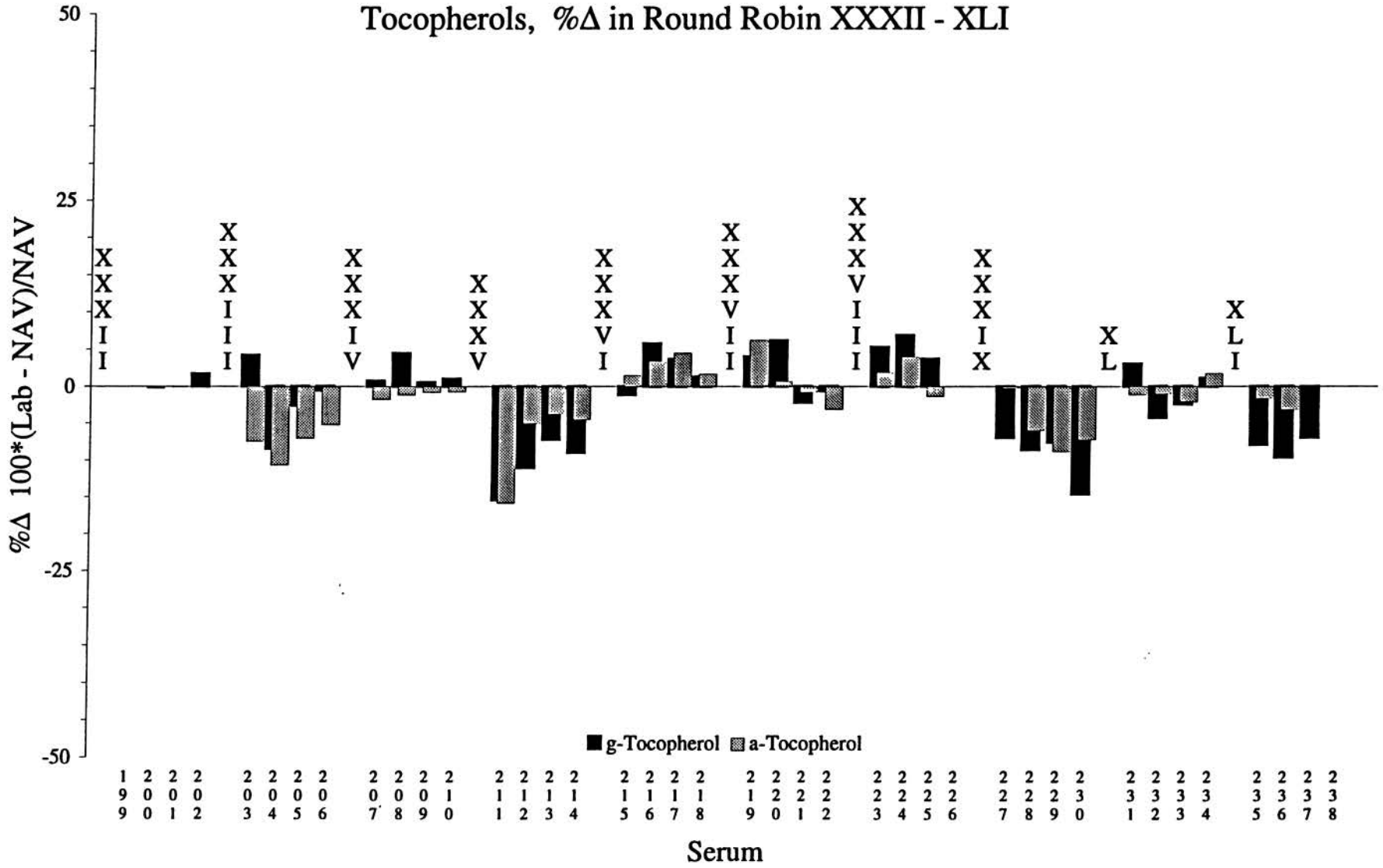
If there are one or two "wild" outliers, they may be calculation or transcription errors. Please check your submitted values against the original data.

The "eSD" calculations are described in: Duewer, *et al* . Analytical Chemistry 1997 69(7):1406-1413.

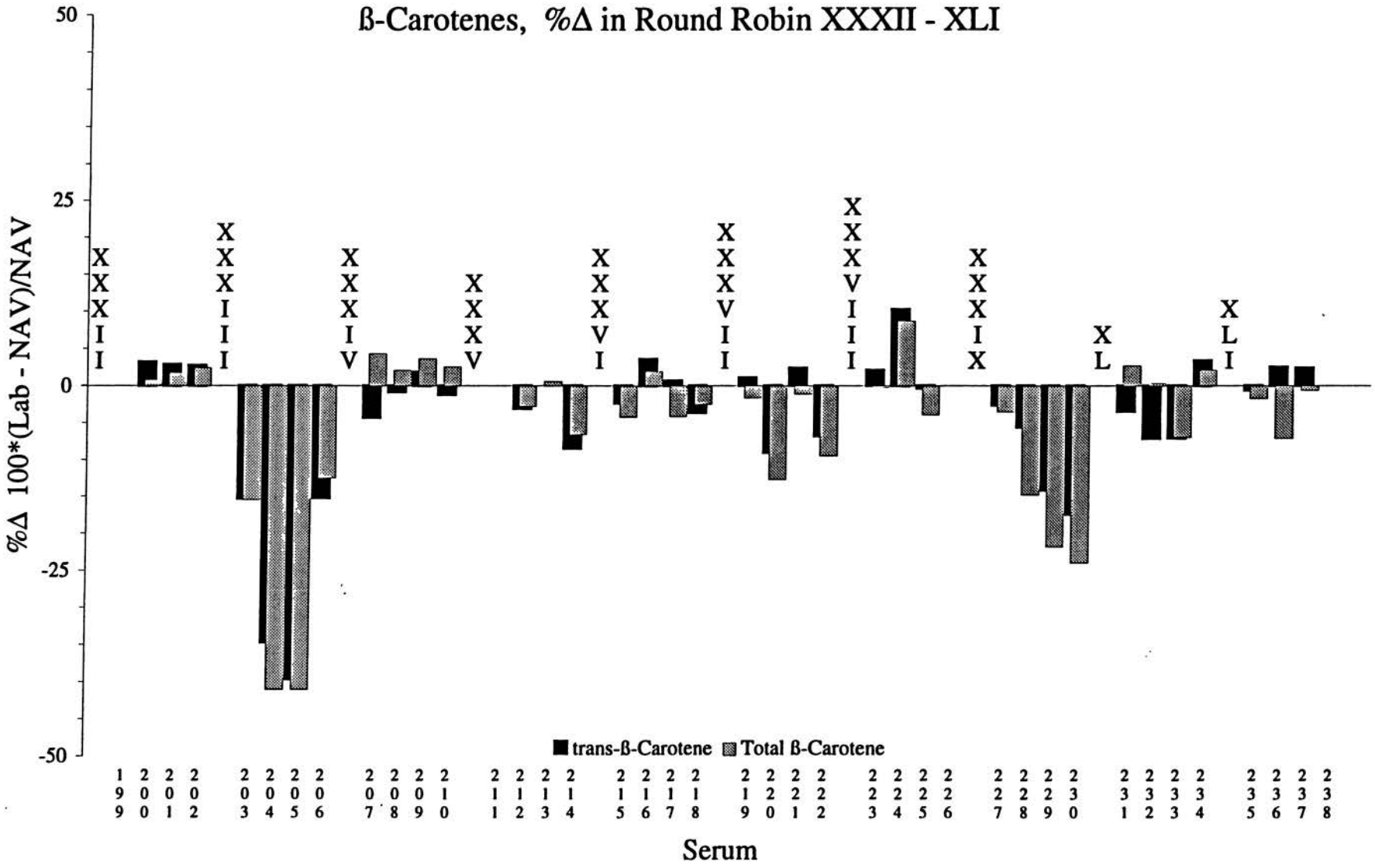
Individualized Round Robin XLI Report: NISTb



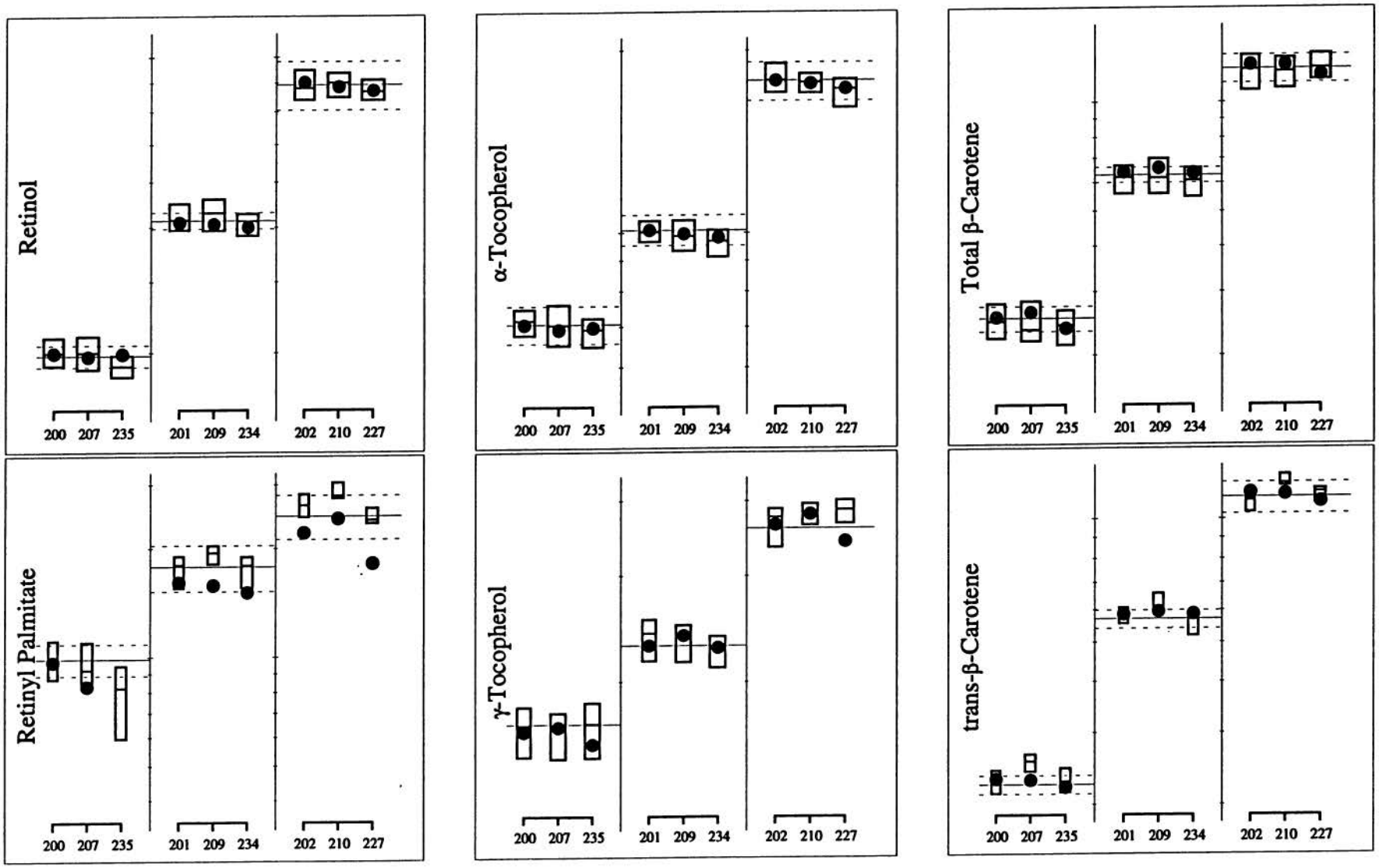
Individualized Round Robin XLI Report: NISTb



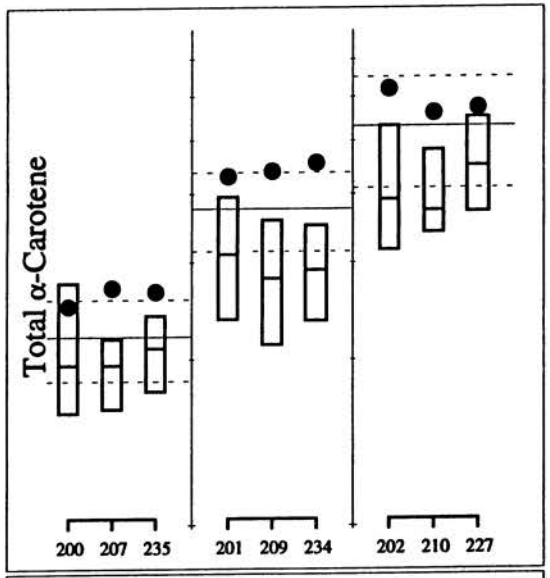
Individualized Round Robin XLI Report: NISTb

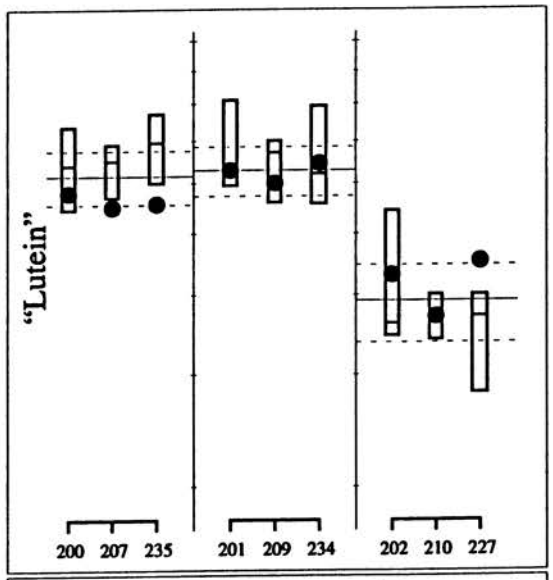


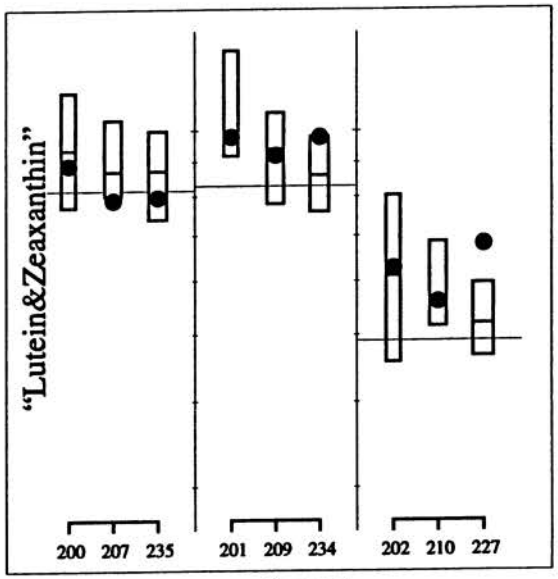
SRM 968b Report: Laboratory NISTb

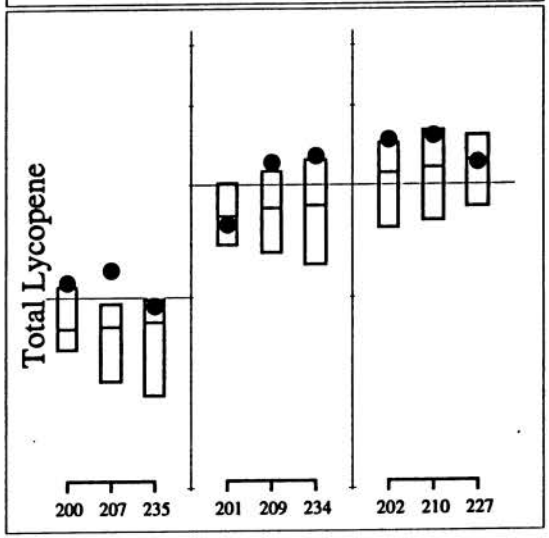


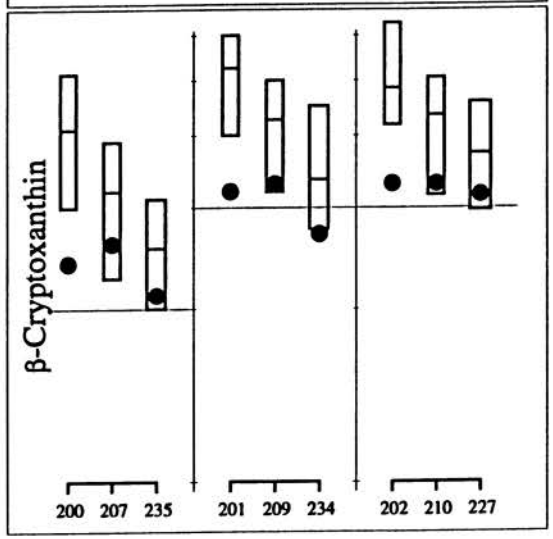
SRM 968b Report: Laboratory NISTb











Legend

Solid circles: Your data

Box plots: Top - 75% of values smaller than this
Bottom - 75% of values greater than this
Middle - Median value
Width - Proportional to number of values

Light lines: Solid - Certified/"Non-certified" values
Dotted - 95% Confidence intervals

200,207,235: SRM 968b "low-level" sera
201,209,234: SRM 968b "mid-level" sera
202,210,227: SRM 968b "high-level" sera

L12

Individualized Round Robin XLI Report: FSV-BA

Summary

	Serum 235			Serum 236			Serum 237			Serum 238		
Analyte	You	NAV	n	You	NAV	n	You	NAV	n	You	NAV	n
Retinol	0.307	0.291	48	0.657	0.596	46	0.956	0.876	47	!0.323	0.268	38
α-Tocopherol	7.07	7.11	46	11.4	11.7	44	19.3	20.3	45	!3.23	3.44	34
γ-Tocopherol	1.81	1.71	25	6.45	6.28	24	1.66	1.13	24	!1.42	1.31	18
Total β-Carotene	0.255	0.240	29	0.301	0.282	28	0.349	0.368	28	!0.085	0.066	22
trans-β-Carotene	0.245	0.222	10	0.291	0.243	11	0.333	0.350	11	!0.082	0.062	10
Total cis-β-Carotene	0.010	0.019	7	0.010	0.036	7	0.016	0.030	7	!0.003		6
Total α-Carotene	0.026	0.023	24	0.037	0.030	26	0.160	0.194	27	!0.01	0.008	17
trans-Lycopene	0.110	0.092	7	0.332	0.404	7	0.152	0.178	7	!0.015	0.021	5
β-Cryptoxanthin	0.043	0.023	23	0.146	0.110	24	0.486	0.389	24	!0.017	0.013	17
Lutein&Zeaxanthin	0.125	0.083	21	0.874	0.527	23	0.217	0.136	23	!0.235	0.090	16

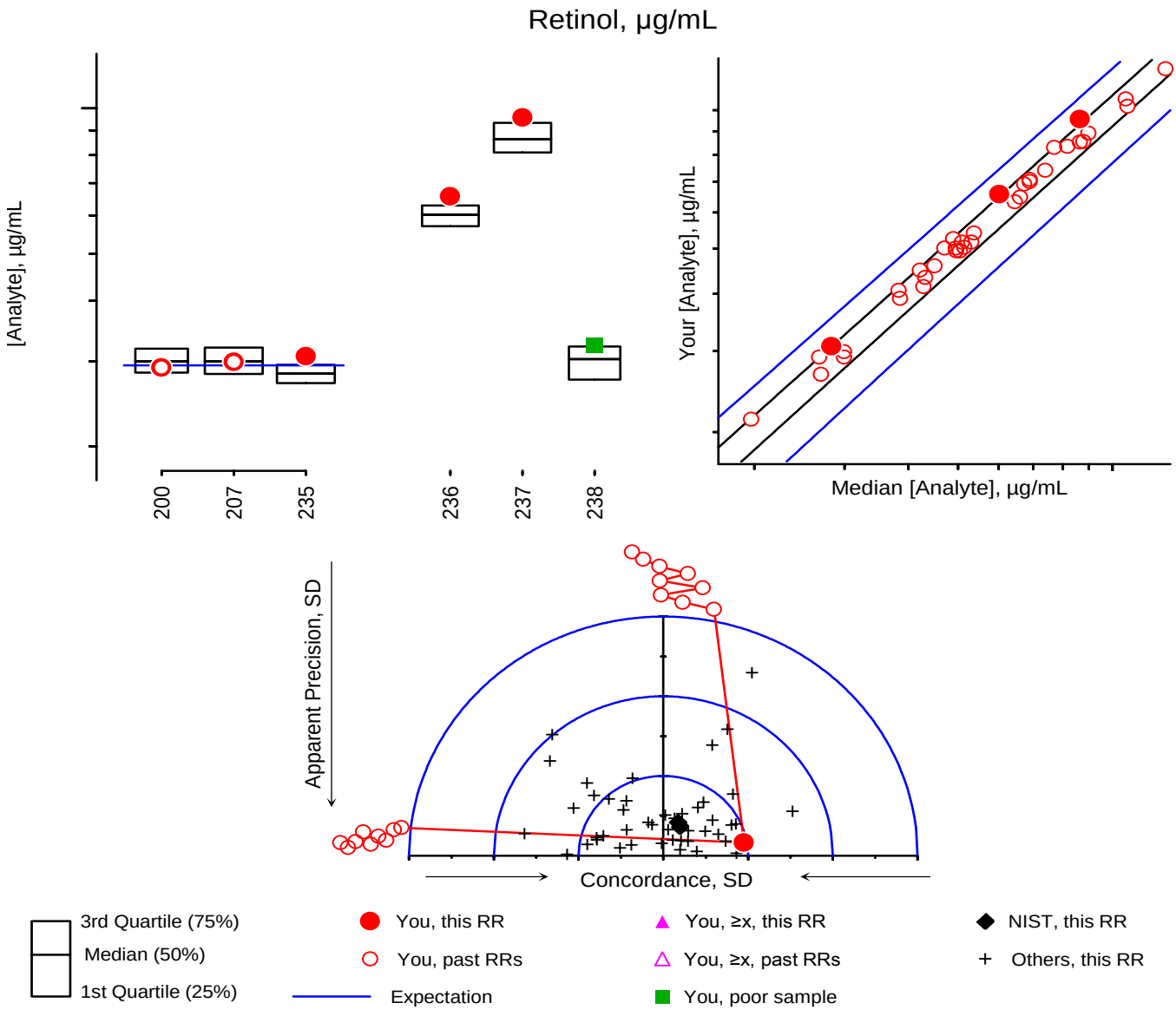
You : Your reported values for the listed analytes (micrograms/milliliter)
NAV : NIST Assigned Values, here equal to this RR's median
n : Number of non-NIST laboratories reporting quantitative values for this analyte in this serum

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

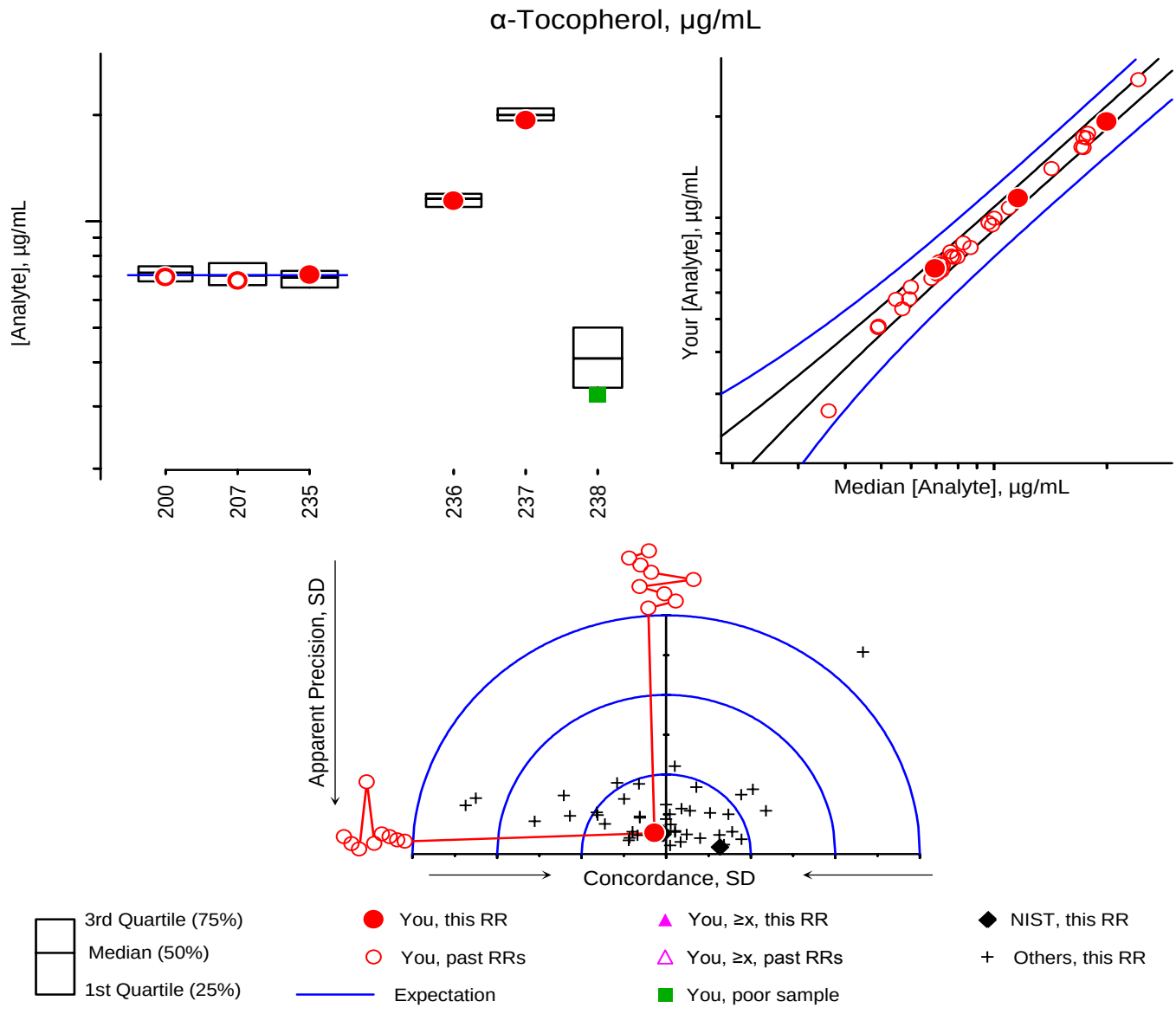
Individualized RR XLI Report: FSV-BA



The software that was used to produce the original "Individualized Reports" for this study is no longer available. The original reports provided the same graphical analyses but in different format. This sheet was generated using a descendent of the 1997 system. For details of the construction and interpretation of these plots, see: Duewer et al. Anal Chem 1999;71(9):1870-8.

Serum	Comments	History
#235	Lyophilized, multi-donor, native. This is the Low level of SRM 968b.	RR32 #201, RR34 #209
#236	Lyophilized, prepared from a "low" single donor pool augmented with many anayltes to ≈10% of normal population level.	New
#237	Lyophilized, prepared from a "low" single donor pool augmented with many anayltes to ≈50% of normal population level.	New
#238	Lyophilized, prepared from a "low" single donor pool augmented with many anayltes to ≈90% of normal population level. This material is heterogeneous; results for this material are not used to access performance.	New

Individualized RR XLI Report: FSV-BA



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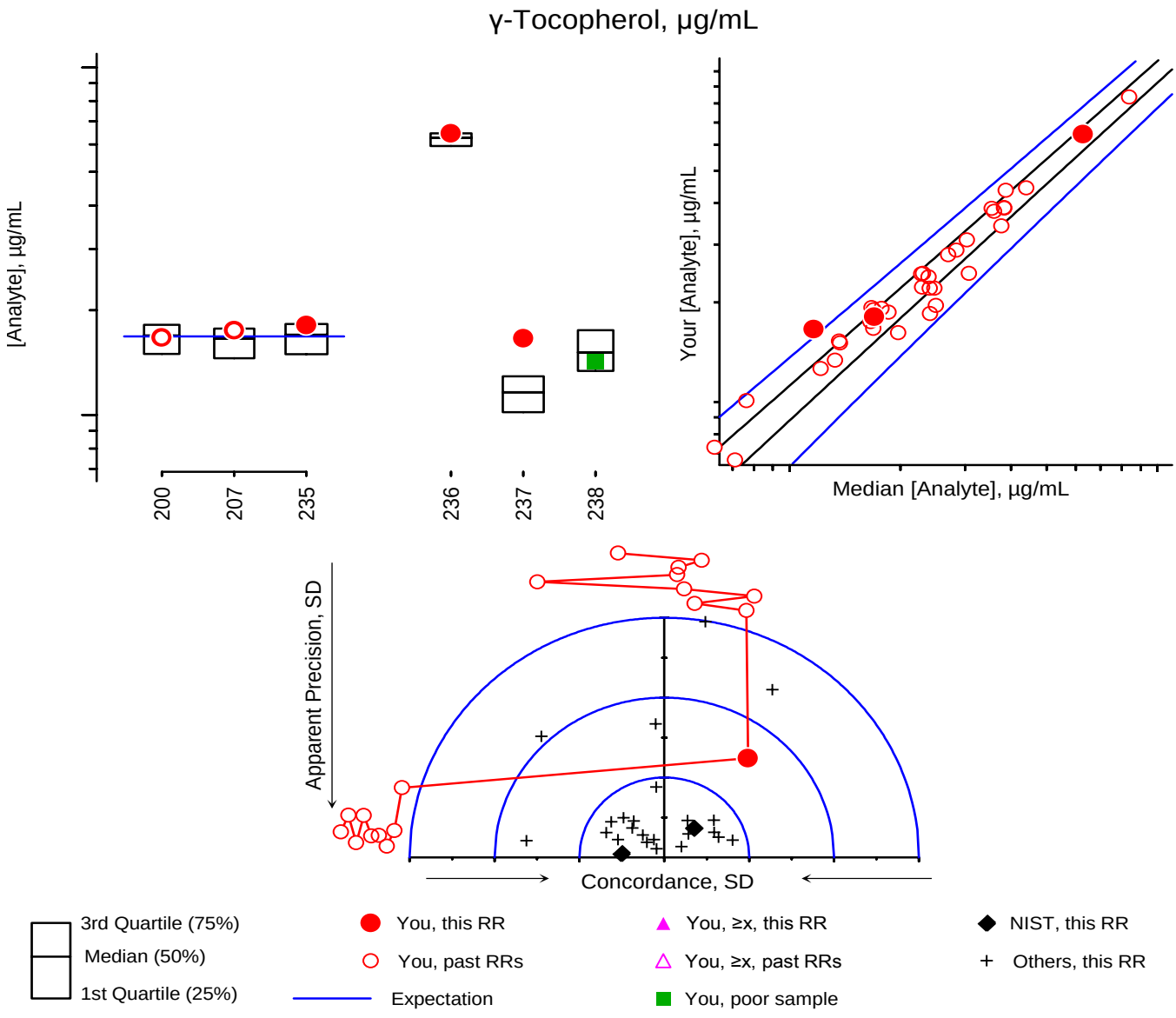
Individualized RR XLI Report: FSV-BA



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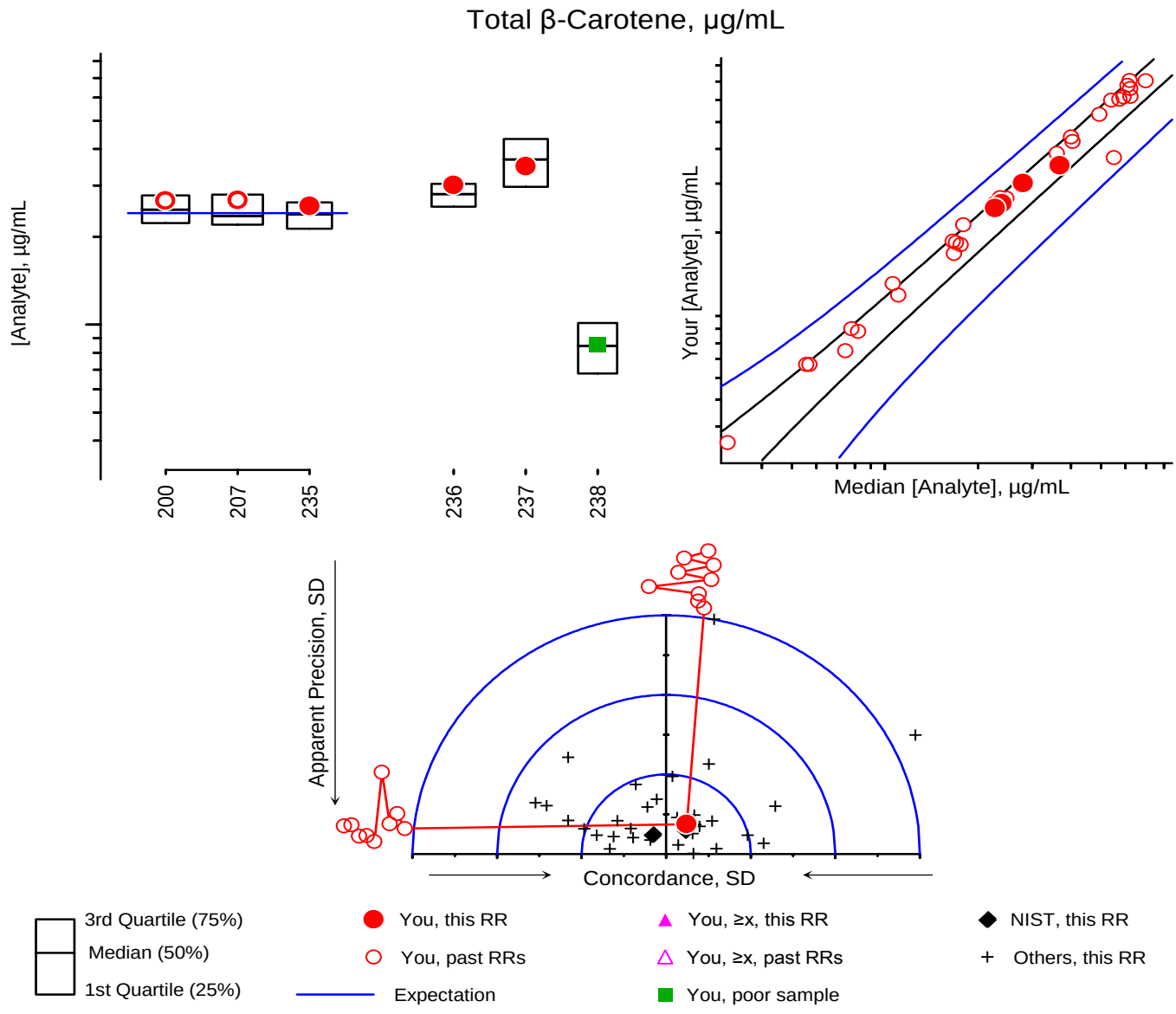
Individualized RR XLI Report: FSV-BA



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Serum	Comments	History
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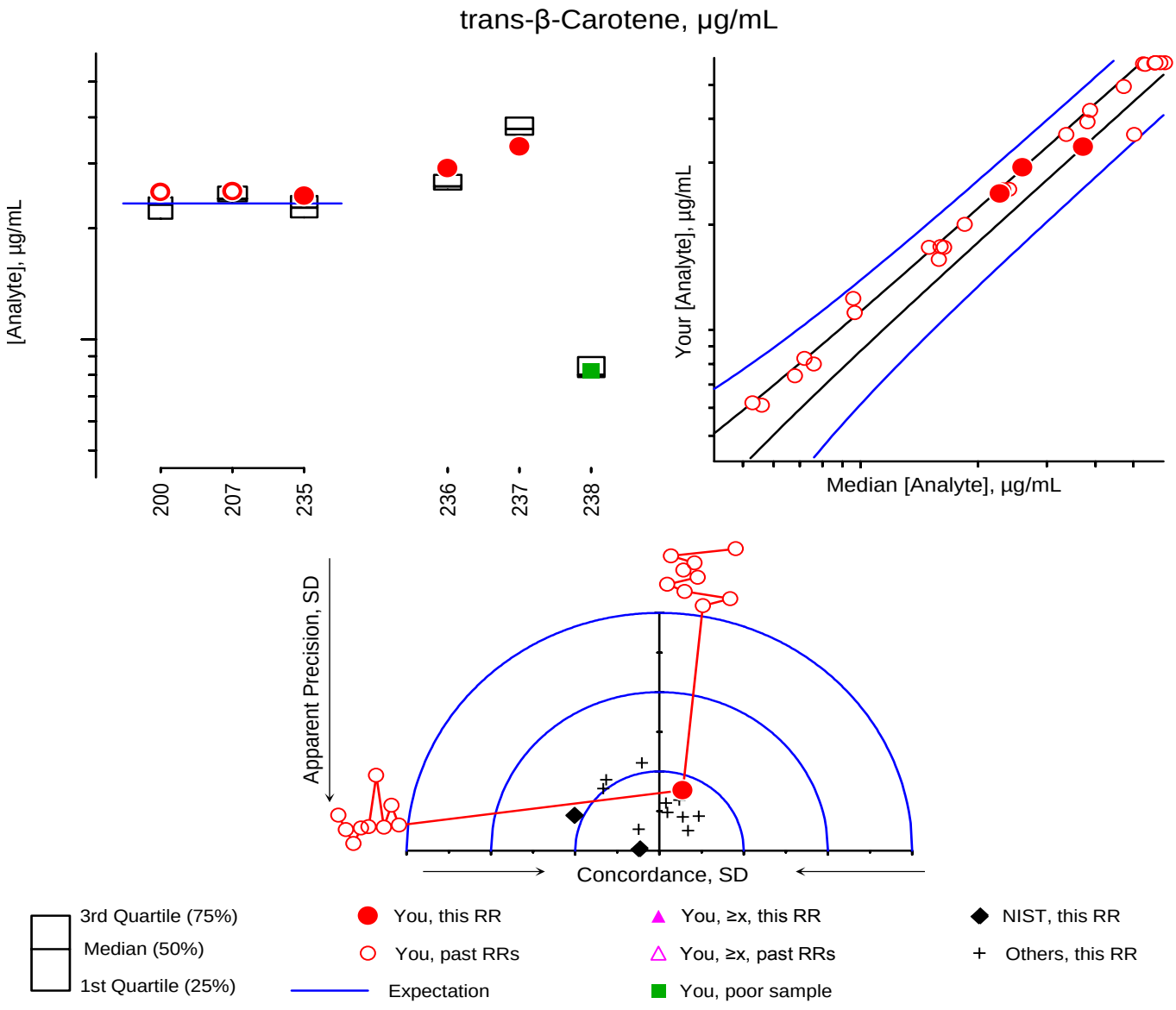
Individualized RR XLI Report: FSV-BA



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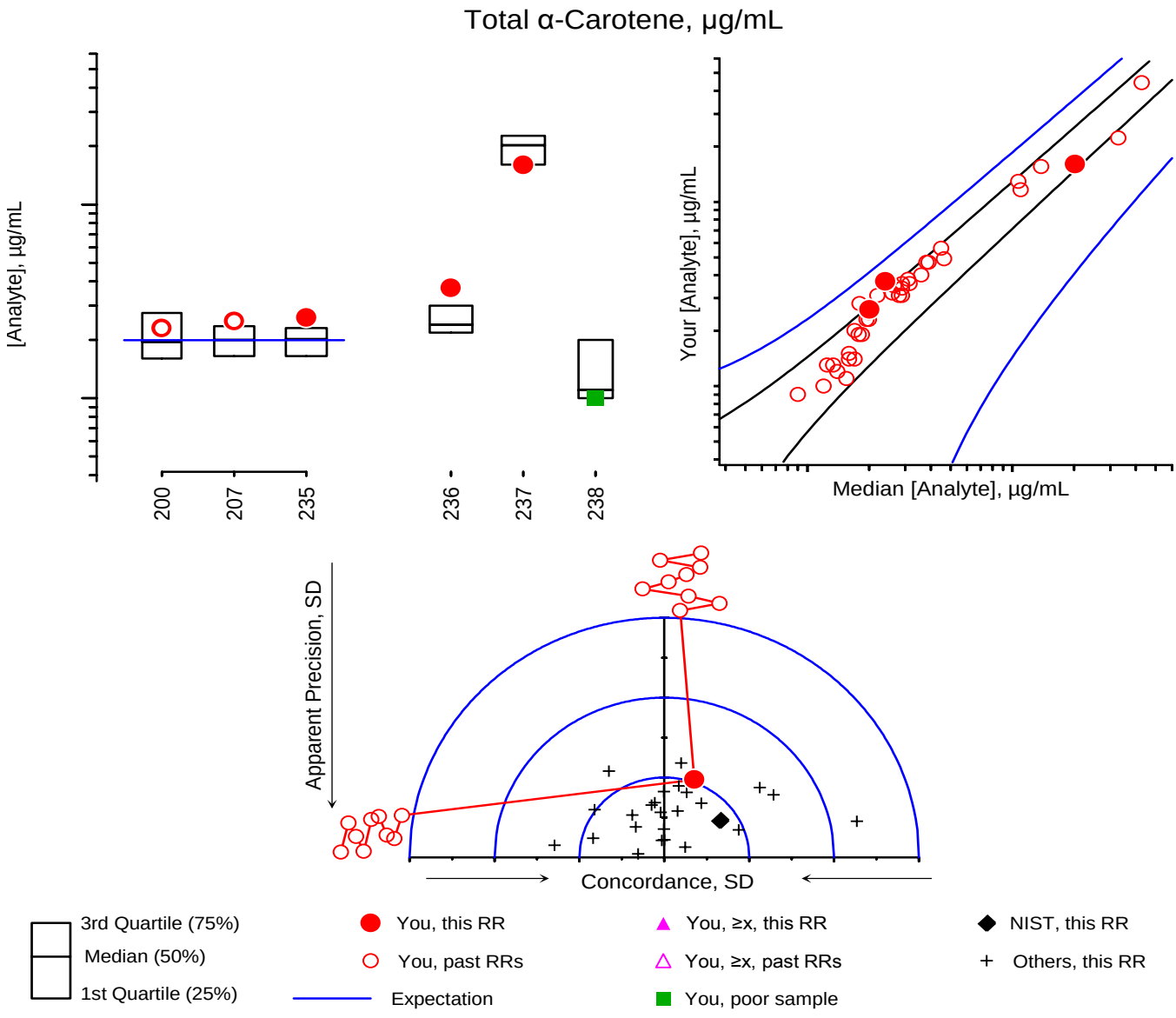
Individualized RR XLI Report: FSV-BA



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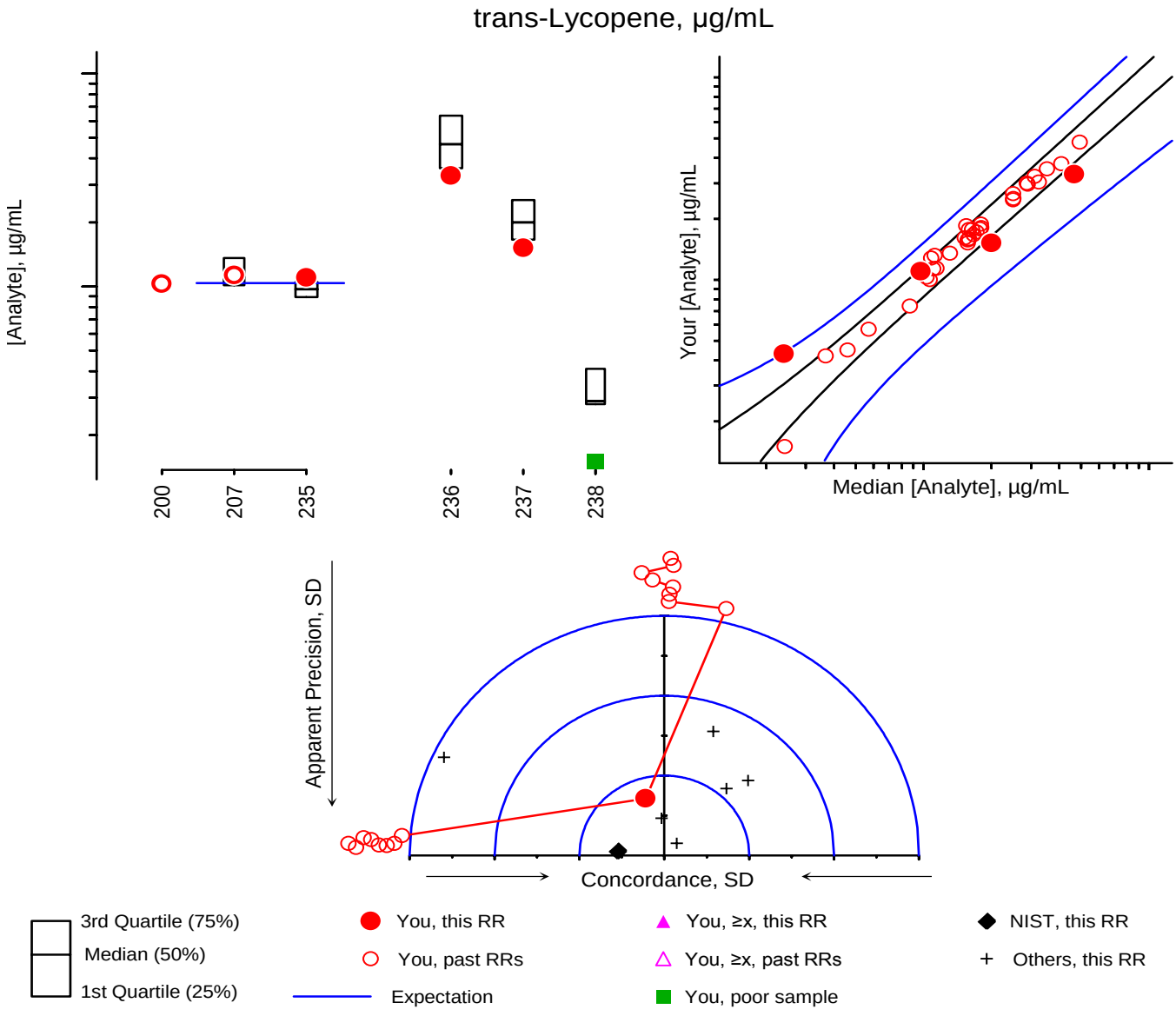
Individualized RR XLI Report: FSV-BA



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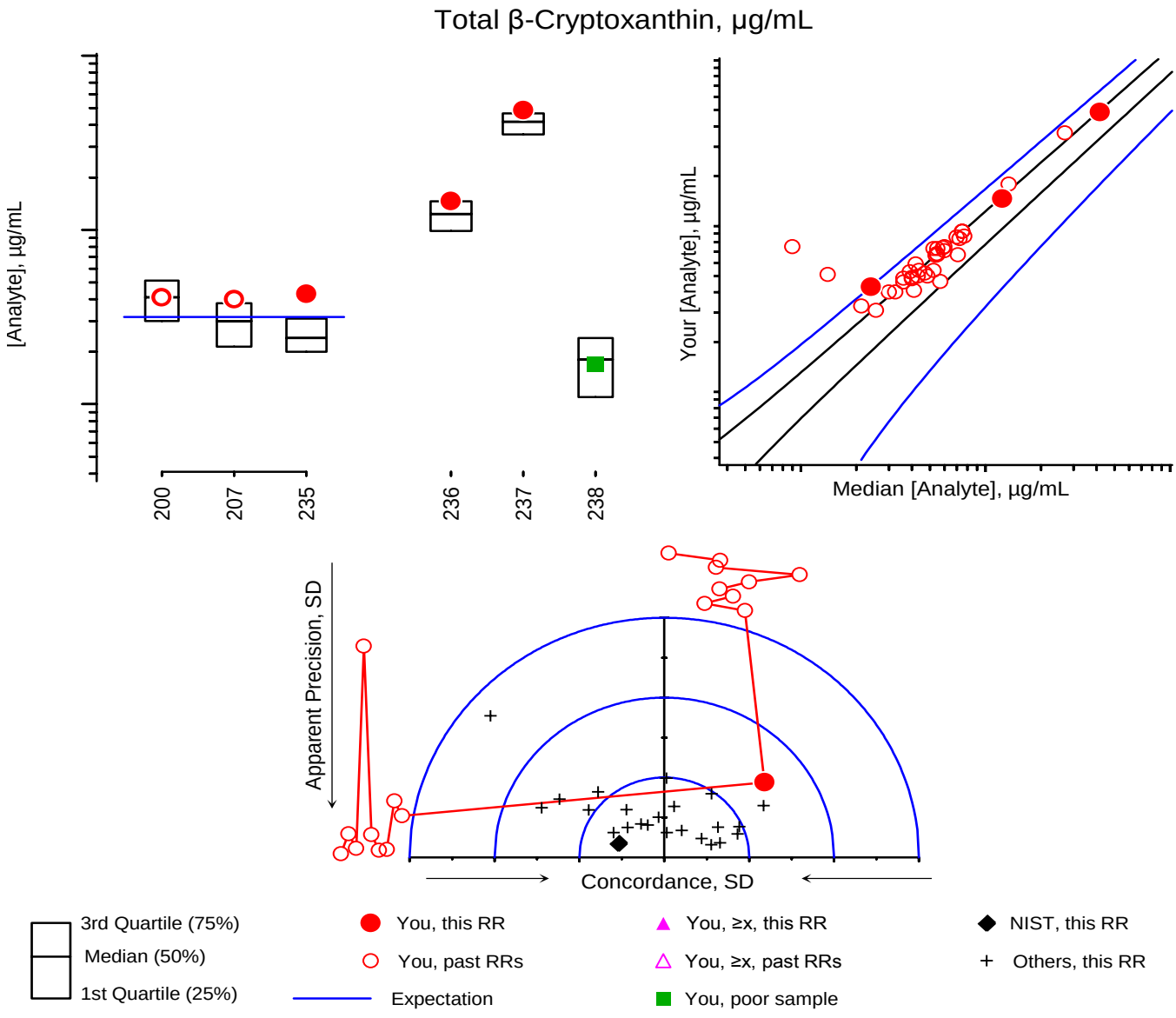
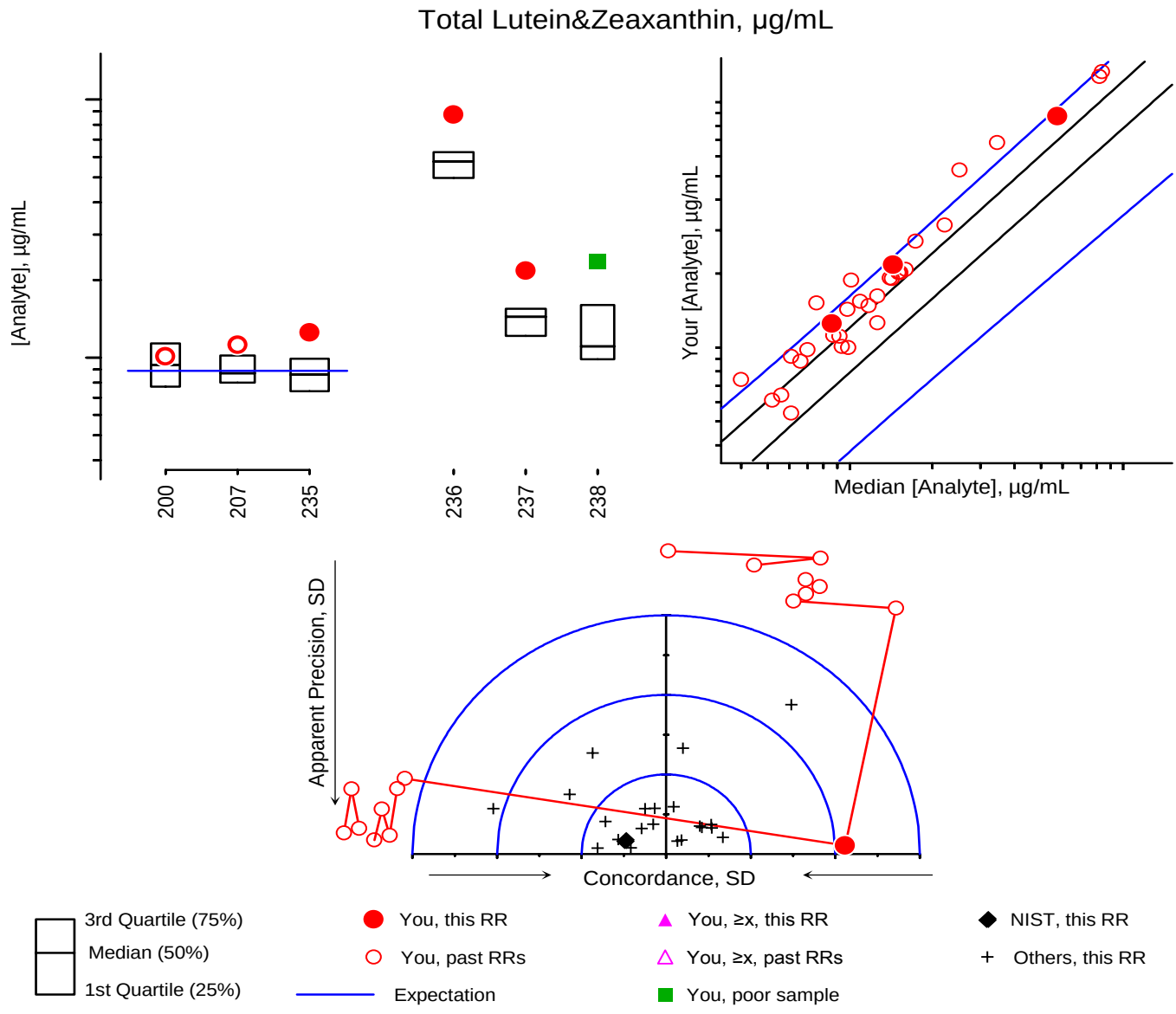
Individualized RR XLI Report: FSV-BA



The software that was used to produce the original "Individualized Reports" for this study is no longer available. The original reports provided the same graphical analyses but in different format. This sheet was generated using a descendent of the 1997 system. For details of the construction and interpretation of these plots, see: Duewer et al. Anal Chem 1999;71(9):1870-8.

Serum	Comments	History
#235	Lyophilized, multi-donor, native. This is the Low level of SRM 968b.	RR32 #201, RR34 #209
#236	Lyophilized, prepared from a "low" single donor pool augmented with many anayltes to ≈10% of normal population level.	New
#237	Lyophilized, prepared from a "low" single donor pool augmented with many anayltes to ≈50% of normal population level.	New
#238	Lyophilized, prepared from a "low" single donor pool augmented with many anayltes to ≈90% of normal population level. This material is heterogeneous; results for this material are not used to access performance.	New

Individualized RR XLI Report: FSV-BA



The software that was used to produce the original "Individualized Reports" for this study is no longer available. The original reports provided the same graphical analyses but in different format. This sheet was generated using a descendent of the 1997 system. For details of the construction and interpretation of these plots, see: Duewer et al. Anal Chem 1999;71(9):1870-8.

Serum	Comments	History
#235	Lyophilized, multi-donor, native. This is the Low level of SRM 968b.	RR32 #201, RR34 #209
#236	Lyophilized, prepared from a "low" single donor pool augmented with many anayltes to ≈10% of normal population level.	New
#237	Lyophilized, prepared from a "low" single donor pool augmented with many anayltes to ≈50% of normal population level.	New
#238	Lyophilized, prepared from a "low" single donor pool augmented with many anayltes to ≈90% of normal population level. This material is heterogeneous; results for this material are not used to access performance.	New

Appendix M. Shipping Package Inserts for RR10

The following two items were included in each package shipped to a RR10 participant:

- Cover letter and instructions for preparing the Control Sample
- Report of Analysis datasheets for the preparation of the Control Sample and for the analysis of the Control and Serum Samples

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

November 13, 1996

FIELD(Title) FIELD(First) FIELD(Last)
FIELD(Company)
FIELD(Address)

Dr. Margolis printed a separate cover letter for each participant. The "FIELD( )" statements are commands for a mail-merge macro routine.

Dear FIELD(Title) FIELD(Last):

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 are Test Samples 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:

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

2. Weigh out 180-220 mg to 0.1 mg (if possible) and dissolve it in 100 mL of 5% MPA using a 100 mL volumetric flask. 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 **nm** and 244 nm.

6. Measure the concentration of 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. All samples have been stored at -70 °C and should be kept at this temperature. We have checked them for stability and the ascorbic acid appears sufficiently stable.

Each ampule contains 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 your report to me by **January 15, 1997.** We also request that you send us a representative chromatogram for each lot and indicate whether you used the peak area or the peak height 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 precision of the laboratories currently measuring ascorbic acid. However, values will not be assigned to individual labs. 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.

Thank you for your assistance.

Sincerely,

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

Enclosures

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 standards. __________________

Date of Analysis: ____________

~~~~~~

PREPARATION OF STOCK SOLUTION AND DILUTED SOLUTION

STOCK SOLUTION

Weight of ascorbic acid in the Stock Solution __________ mg

DILUTE SOLUTION

Weight of added Stock Solution (0.5 mL) __________ mg
Weight of 5% MPA added to 100 mL volumetric Flask __________ g

Absorbance of Dilute Solution at **243 nm** __________
Absorbance of Dilute Solution at **244 nm** __________
~~~~~~

REPORT OF ANALYSIS

RESULTS (µmol/L)

CONTROL SAMPLE

REPLICATE 1 _____ µmol/L
REPLICATE 2 _____ µmol/L

SERUM 682B, VIAL # _____

REPLICATE 1 _____ µmol/L
REPLICATE 2 _____ µmol/L

SERUM 682B, VIAL # _____

REPLICATE 1 _____ µmol/L
REPLICATE 2 _____ µmol/L

SERUM 682A, VIAL # _____

REPLICATE 1 _____ µmol/L
REPLICATE 2 _____ µmol/L

SERUM 682A, VIAL # _____

REPLICATE 1 _____ µmol/L
REPLICATE 2 _____ µmol/L

Appendix N. Final Report for RR10

The following 11 pages are the final report as provided to all participants. This report contains:

- Cover letter and analysis of results
- Table 1 "Results of Round Robin RR10 for the Measurement of AA in Human Serum"
- Table 2 "Results of Round Robin RR10 for the Measurement of AA using NIST AA Sample"
- Legend for the following four Figures
- Figure 1 "Tukey Box Plot of the Round Robin RR09 Results"
- Figure 2 "Tukey Box Plot of the Round Robin RR10 Results"
- Figure 3 "Distribution of Round Robin RR10 Results for lots 682a and 682b"
- Figure 4 "Distribution of Round Robin RR10 Calculated and Measured Results for the Ascorbic Acid Standard"

A number of the results reported in Tables 1 and 2 were later revised to correct for miscommunication of the reporting units. Since the listed results do not necessarily represent measurement performance, the Lab identifiers used by Dr. Margolis have been redacted from these Tables rather than re-coded. The reporting unit confusion impacts some of the conclusions discussed in the cover letter. However, the results discussed in the Dr. Margolis's text have **not** been updated or corrected.

The "All Lab Report" in Appendix O lists the corrected results and provides more extensive statistical summaries.

November 4, 1997

FIELD(Title) FIELD(First) FIELD(Last)
FIELD(Company)
FIELD(Address)

Dr. Margolis printed a separate letter for each participant. The "FIELD( )" statements are commands for a mail-merge macro routine.

Dear FIELD(Title) FIELD(Last):

This report describes both the overall-group and your laboratory performance in Round Robin X for the measurement of ascorbic acid (AA) in human plasma. This study involved the duplicate analyses of two unknown samples, 682a and 682b and a solid ascorbic acid sample as a standard. Your results are designated as Lab. No. ~ in the tables and figures.

Table 1 provides a summary of the data submitted by the participating laboratories (the NIST data were not included in the statistical analysis). Two laboratories submitted two sets of measurements, each done by a different method. As shown in Table 1 the percent Relative Standard Deviation (%RSD) for both lots was 15.1 and 14.5. The intralaboratory %RSD varied from 0.4-5.1 with one exception.

These results indicate that the intralaboratory variation remains essentially unchanged from the two previous round robins (0.3-4.0). However, the interlaboratory %RSD is similar to that of RR IX (13.9 and 11.8) and has increased slightly. The box plots in Figures 1 and 2 graphically summarize the results, the highest and lowest 10% of the measurements for each lot are plotted as small open circles, the two simple lines each span the next 15% intervals, and the center box contains the values from the remaining data sets. The NIST mean value for the total ascorbic acid + dehydroascorbic acid is represented by a solid circle. The horizontal line in the 50% boxes represents the median interlaboratory values which are nearly identical to those of NIST serum ascorbic acid concentrations.

In RR IX we asked each laboratory to make up a solution from solid ascorbic acid, measure its UV absorbance, and assay the ascorbic acid content. Unfortunately, the concentration was low except for several laboratories who increased the concentration 10 fold. The purpose of this segment of the study was to try to evaluate the role that your standards and your measurement technique might be playing in the accuracy and precision of your measurement process. In RR X we asked you to perform the same measurements on ascorbic acid solutions that were ten times more concentrated. These results are summarized in Table 2. The ascorbic acid concentrations were calculated from the weights that you reported, and the volume of metaphosphoric acid (MPA) was calculated by using a density of 1.004 g/L for 5% MPA. The analysis of the weights of a 500 µL aliquot of the AA stock solution gave a mean of 513 mg, SD 10.2, %RSD 2.0 (range 490-527 mg). The analysis of the results of the conversion of the weighed amount of 100 mL of MPA in to the 100 mL volumetric flask was a mean of 102.5 g, SD 0.96, %RSD 0.94 (range 99-103 g). These data indicate that the laboratories are able to accurately weigh samples between 0.500 and 100 g. It also indicates that the pipettes used to measure sub mL volumes is biased

high by 2.5% at 500 µL. An important question that is not answered in this study is whether this is a constant bias or proportional to the volume being measured. However, it strongly suggests that each laboratory should calibrate its micropipettes over the range that they are used. One way to do this is to weigh a series of aliquots (5-10) of a liquid such as water, convert the weights to volumes using the density of water, and calculate the accuracy and precision of the pipette and pipetting technique. Using the data that you submitted to us, we calculated the concentration of the AA in the standard solution (column 1, Table 2) which each of you made and assumed that the error in concentration was no greater than the error in weighing 0.5 to 100 g. The amount of AA that you actually measured in your assay of the standard solution is listed in column 2, Table 2. To compare the measurements on the standard solutions, we normalized all of the data obtained by assaying the standard solution to a starting concentration of 50 µmol/L using the equation:

$$\frac{\textit{assayed [AA]}}{\textit{weighed [AA]}} = \frac{\textit{normalized [AA]}}{50}$$

The results of this calculation are summarized in column 3, Table 2. The mean of these data is 49.1, SD 4.9, %RSD 10.1 (range 39.3 - 54 µmol/L). If the estimated error in weighing and in filling the volumetric flasks is small (1 - 2%), then the major source of error is in the assay itself. This would include the accuracy of the pipets, the accuracy of the standards particularly if they are diluted, the accuracy of the volume of the sample standard and/or serum) delivered to the assay mixture, and the accuracy of any constants used in the calculation of the concentration of the analyte from the results of the assay. These calculations exclude the data from laboratory 1 which made up its stock solution 1/10 that of the desired value, laboratory 13 which made an additional 1:20 dilution of its diluted stock solution and laboratory 30 whose data concerning the standard solution we could not interpret. Finally we asked each of you to measure the absorbance of your standard solution at 243 and 244 nm. Every laboratory obtained similar values at each wave length indicating that the wavelength was correct; however, the mean $E^{1\%}$ for a 1 cm cell was 555 AU/mole/cm, SD 25, %RSD 4.5. At NIST the $E^{1\%}$ was determined for Fisher and Sigma samples of AA and the values were 529 and 533 AU/mole/cm respectively. The reported values varied from 501 to 606 AU/mole/cm excluding laboratory 1 which made up its stock solution 1/10 of the desired value and laboratory 30 whose data concerning the standard solution we could not interpret. These results without the excluded data indicate that there is a need among some laboratories to calibrate their spectrophotometers with absorbance standards such as SRM 2031.

In conclusion we can identify the following sources of systematic bias in the measurements.

1. The spectroscopic error in the measurement of the absorbance of a standard solution (*%RSD = 4.5*)
2. The pipetting of aqueous solutions mean *2.5% above expected value*
3. The weighing of samples 0.5-100 g (*%RSD of 1 and 2% respectively*)
4. The measurement of the concentration of a *50 µmol/L* standard solution (*mean = 49.1 µmol/L, %RSD = 10.1*). The mean value is close to the expected value therefore the error probably lies in the accuracy of the measurement of the sample or the calculation constants.
5. The measurement of the serum AA (%RSD = 14.5 and 15.1). This could either reflect

an error in delivering the total sample to the assay because of the viscosity of the sample or in the constants used in calculating the AA concentration.

If your values differed from those of NIST by more than 5%, we suggest that you evaluate whether you accurately deliver the correct sample volume that can vary either as a function of the sample viscosity or the accuracy of the pipet. Alternatively, we suggest that you evaluate the accuracy of the constants that you use in converting the assay results to a final AA concentration in the serum. If your results deviate significantly from the assigned values, we would suggest that you reexamine your methods for possible systematic errors. The distribution of the results of each laboratory are graphically illustrated in Figures 3-7.

The next set of samples (RR XI) will be shipped during November, 1997. If you have any questions concerning the previous round robins please contact me at 301/975-3137 or by e-mail at sam.margolis@nist.gov.

Sincerely,

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

Enclosures

Table 1 Results of Round Robin X for the Measurement of AA in Human Serum

Lab	Method[b]	Ascorbic Acid (μmol/L Serum)[a] Lot 682b	Lot 682a
[redacted]	LC	106.0 ± 2.0	74.5 ± 1.5
	DNPH	119.5 ± 3.9	85.9 ± 1.3
	LC-EC		
	ENZ	125.0 ± 0.0	89.5 ± 0.6
	DNPH		
	DCIP	113.8 ± 4.3	71.0 ± 5.1
	DCIP	105.4 ± 0.7	82.2 ± 1.8
	LC-EC	140.6 ± 0.4	100.3 ± 2.1
	LC-EC	90.8 ± 0.6	66.1 ± 3.2
	LC		
	LC-EC	110.8 ± 1.3	79.0 ± 0.3
	LC	[c]	86.4 ± 0.7
	LC-EC		
		91.7 ± 1.4	63.9 ± 2.0
	AUTOAN	124.6 ± 2.3	89.1 ± 2.3
	LC-OPD	121.4 ± 0.7	85.2 ± 0.5
	LC-EC	87.6 ± 0.6	63.4 ± 0.6
	LC-OPD	112.2 ± 10.5	84.3 ± 4.2
	LC	136.8 ± 2.4	92.8 ± 1.5
	LC-EC		
		94.5 ± 0.1	72.5 ± 2.0
	DNPH	139.6 ± 1.0	101.1 ± 1.1
	ENZ	120.7 ± 1.1	85.9 ± 5.1
	LC	108.0 ± 2.9	80.2 ± 1.2
MEAN		112.5	80.9
SD		17.0	11.7
%RSD		15.1	14.5
NIST AA + DHAA	LC-EC	115.4 ± 2.7[d]	83.2 ± 5.5[d]
NIST AA	LC-EC	100.1 ± 1.6[d]	70.7 ± 1.8[d]

[a] Values represent the mean and SD of replicate measurements on two samples (total of 4 measurements).

[b] The abbreviations in this column are as folows: LC, liquid chromatography; DNPH, dinitrophenylhydrazine; EC, electrochemical detector; ENZ, enzymatic assay; DCIP, dichloroindophenol; AUTOAN, autoanalyzer; OPD, orthophenlyenediamine.

[c] The wrong sample was sent and therefore the results are not included.
[d] Values represent the mean and SD of replicate measurements on five samples (total of 10 measurements).

Table 2 Results of Round Robin X for the Measurement of AA using NIST AA Sample

Lab	Method	Ascorbic Acid (μmol/L MPA)			$E^{1\%}$ [a]
		Calculated	Measured	Normalized[b]	
[redacted]	LC	5.81	5.80	49.9	790
	DNPH				
	LC-EC				
	ENZ	59.2	62.8	53.0	552
	DNPH				
	DCIP	51.1	46.3	45.2	533
	DCIP	55.6	55.1	49.1	569
	LC-EC	56.9	61.5	54.0	553
	LC-EC	57.9	62.6	54.0	561
	LC				
	LC-EC	29.0	26.2	45.2	555
	LC	29.4	28.7	48.9	606
	LC-EC				
		59.7	46.9	39.3	556
	AUTOAN	59.2	57.5	48.5	546
	LC-OPD	57.7	59.6	51.7	556
	LC-EC	54.0	43.7	40.4	603
	LC-OPD	56.2	59.2	52.6	576
	LC	58.2	61.8	53.2	541
	LC-EC				
		56.5	45.4	40.2	528
	DNPH	35.9	35.7	49.7	501
	ENZ	63.5	68.4	53.9	544
	LC		55.9		
Mean				49.1	555[c]
SD				4.9	26
%RSD				10.1	4.7
NIST Sigma (5% MPA)					533
NIST Fisher (5% MPA)[d]					529
Lit. (aqueous solution)					560

[a] Absorptivity of a 1% solution in a 1 cm cell.

[b] The standard values for each laboratory were dependent on the amount of AA weighed by each lab. In order to compare the measured AA concentrations of each laboratory, the measured values were normalized to correspond to a weighed value of 50 mmol/L.

[c] This value does not include the results of laboratory 1.

[d] This sample was obtained from the Center for Disease Control.

Figure 1. Tukey Box Plot of the Round Robin X Results.

The open circles represent the highest and lowest 10% of the measurements. The two simple lines each span the next 15% intervals and the center box contains the values from the remaining data sets. The NIST mean value for the total ascorbic acid (AA + DHAA) is represented by a solid circle. The horizontal line in the 50% box represents the median inter-laboratory values which are slightly different from the NIST values.

Figure 2. Tukey Box Plot of the Round Robin X Results.

The open circles represent the highest and lowest 10% of the measurements. The two simple lines each span the next 15% intervals and the center box contains the values from the remaining data sets. The NIST mean value for the total ascorbic acid (AA + DHAA) is represented by a solid circle. The horizontal line in the 50% box represents the median inter-laboratory values which are slightly different from the NIST values.

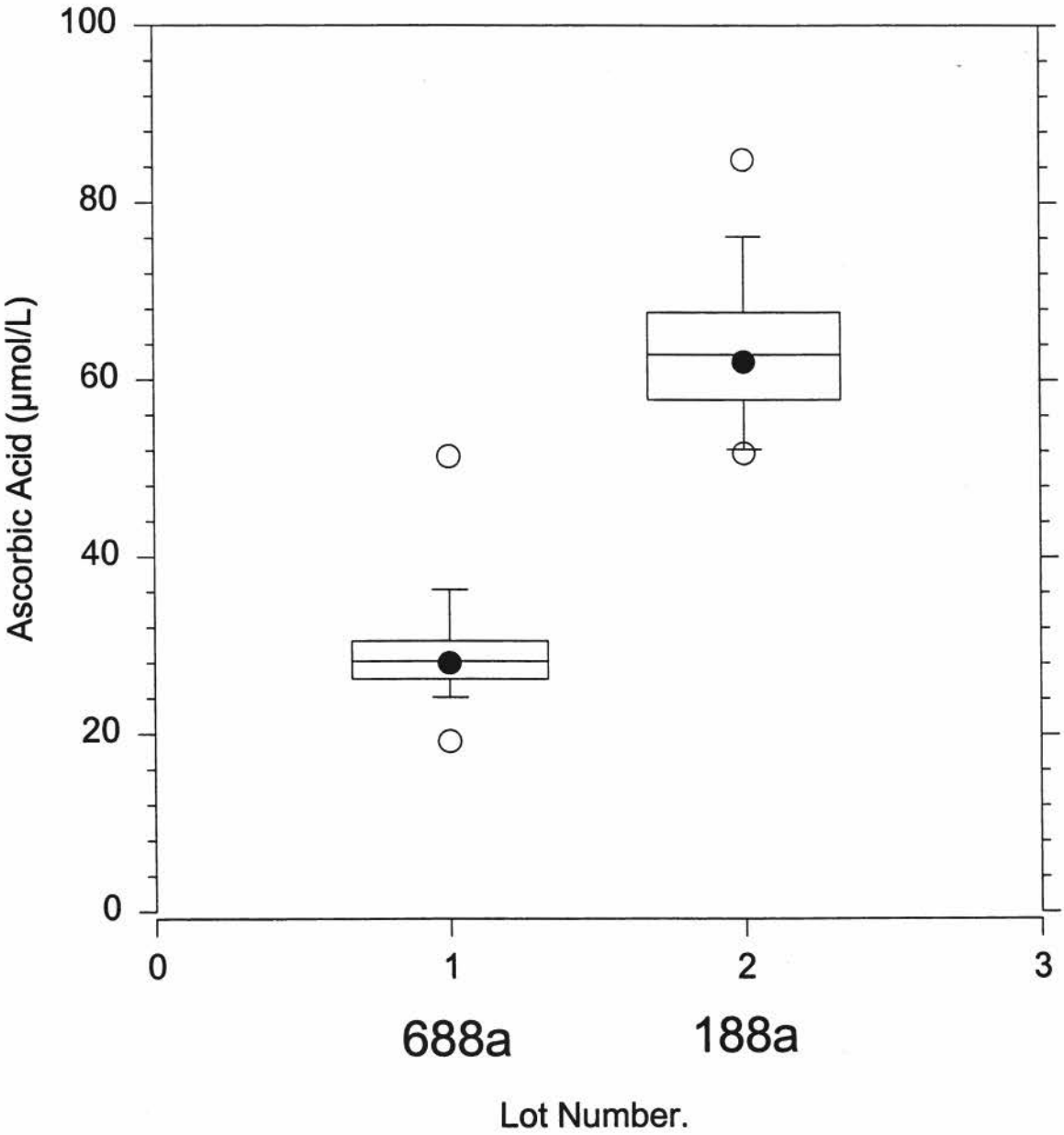
Figure 3. Distribution of Round Robin X Results for lots 682a and 682b.

◇ First Vial; First Measurement
□ First Vial; Second Measurement
△ Second Vial; First Measurement
○ Second Vial; Second Measurement
The solid points are for Lot 682a
The open points are for Lot 682b

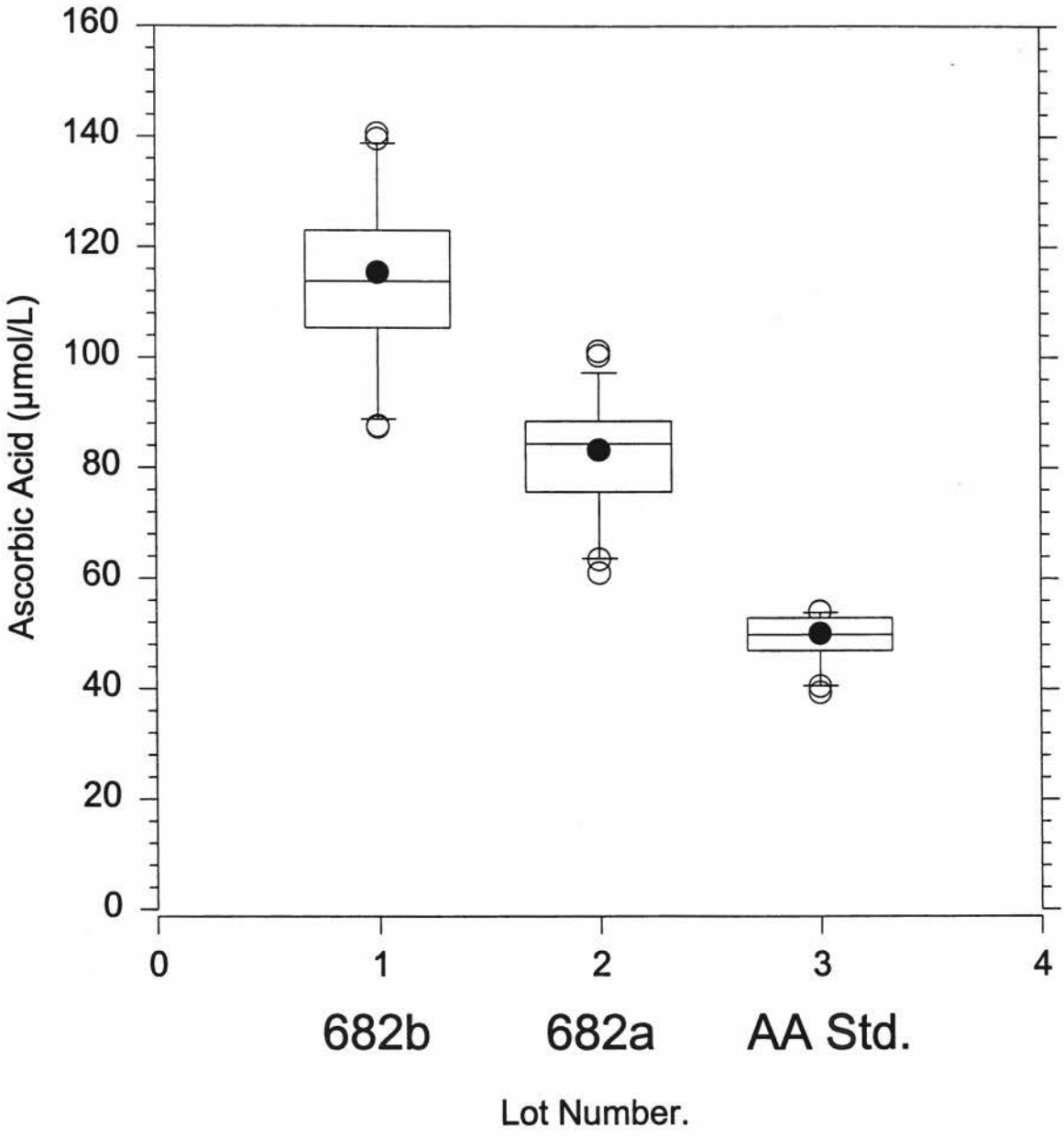
Figure 4. Distribution of Round Robin X Calculated and Measured Results for The Ascorbic Acid Standard.

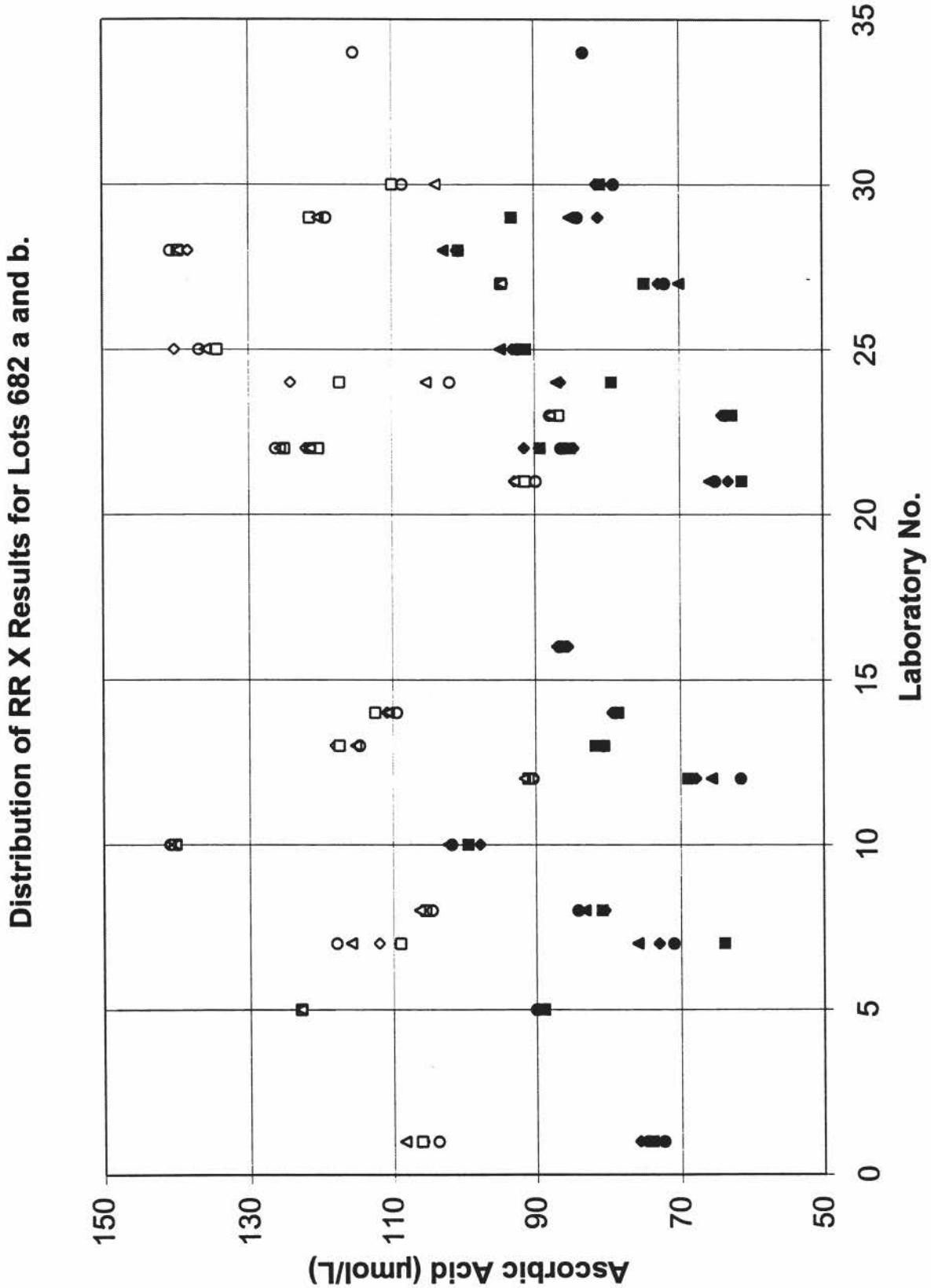
The open squares represent the measured AA concentration.
The solid diamonds represent the calculated AA concentration.

Round Robin IX

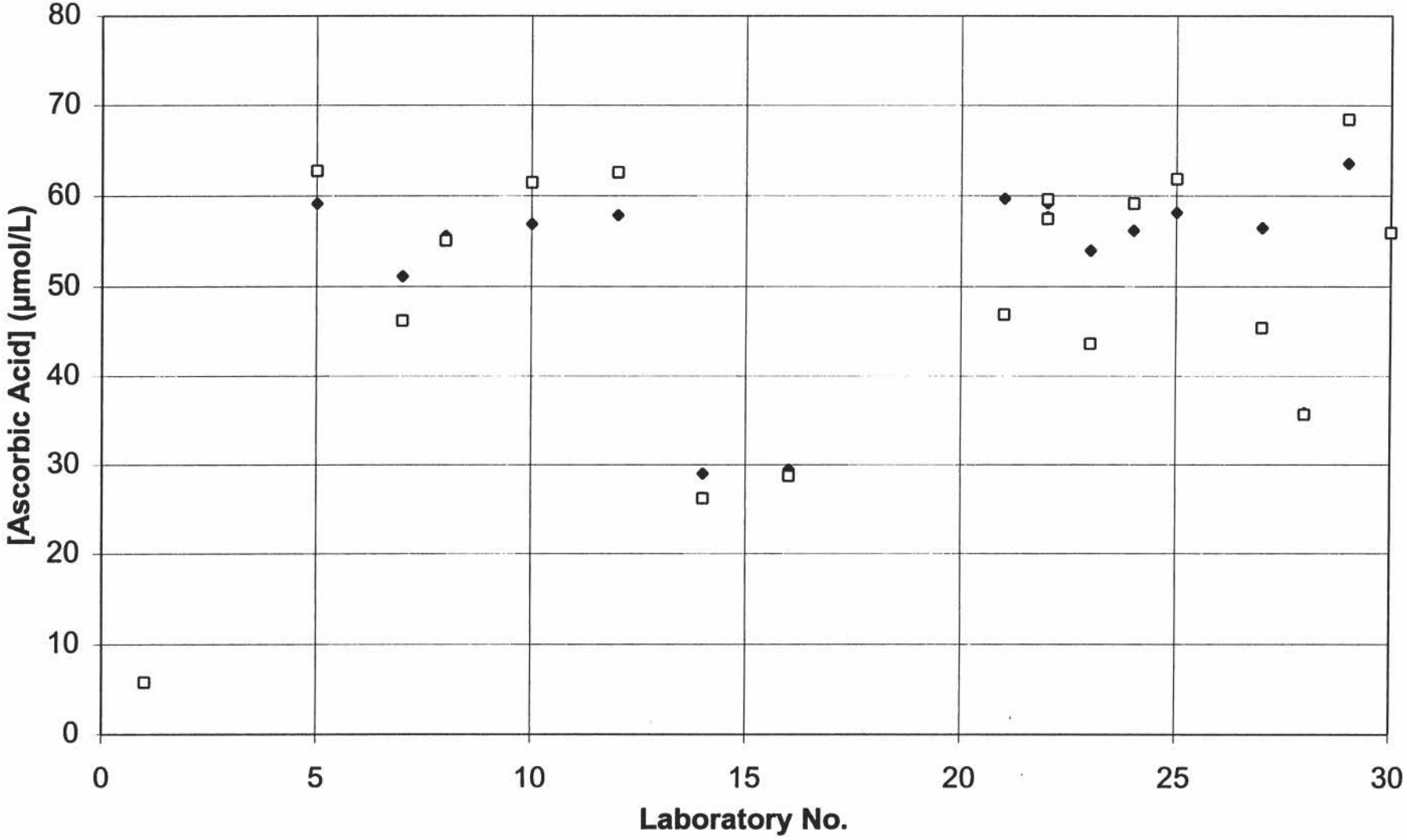


Round Robin X




Distribution of RR X Results for Lots 682 a and b.
Ascorbic Acid (µmol/L)
150
130
110
90
70
50
0
5
10
15
20
25
30
35
Laboratory No.

Distribution of RR X Results on the AA Standard



Appendix O. "All-Lab Report" for VC-RR10

The following is a single page "All-Lab Report" that contains the same information as originally provided to all participants, with the following exceptions:

- the participant identifiers (Lab) have been altered to ensure confidentiality of identification codes assigned to laboratories..
- the order in which the participant results are listed has been altered.
- results for the Serum Samples have been corrected and transformed to have units of µmol/mL sample.
- results for the Control and Serum Samples have been consolidated.
- additional summary statistics have been included.

Vitamin C Round Robin 10

		Control Solution					Ascorbic Acid, mmol/mL											
		$E_{1\%}$	AA, µmol/mL			Ratio	682B					682A					Calibrated	
Lab	Method	$^{dL}/_{gcm}$	Cal	Obs	S_{dup}	$^{Obs}/_{Cal}$	Mean	S_{dup}	S_{rep}	S_{het}	S_{tot}	Mean	S_{dup}	S_{rep}	S_{het}	S_{tot}	682B	682A
VC-MA	HPLC-EC	565	54.3	2.3	0.0	-	58.2	1.0	0.2	1.0	1.0	40.5	0.3	0.1	0.3	0.3		
VC-MB	AO-OPD	564	58.5	62.8	0.4	1.07	61.5	0.0	0.0	0.0	0.0	44.8	0.0	0.4	0.0	0.4	57.4	41.8
VC-MC	HPLC-EC	802	5.8	5.8	0.2	1.01	53.0	0.0	1.2	0.0	1.2	37.2	0.3	0.9	0.0	0.9	52.7	37.0
VC-MD	24DNPH						59.8	0.5	2.3	0.0	2.3	42.9	0.7	0.4	0.6	0.7		
VC-ME	HPLC-UV	565	57.1	57.5	1.4	1.01	62.3	1.0	1.0	0.7	1.2	44.6	0.9	0.9	0.6	1.1	61.9	44.2
VC-MF	HPLC-EC	552	58.6	59.6	0.8	1.02	60.7	0.1	0.4	0.0	0.4	42.6	0.2	0.2	0.1	0.2	59.6	41.8
VC-MG	HPLC-EC	594	54.9	61.8	0.4	1.13	68.4	0.3	1.5	0.0	1.5	46.4	0.5	0.7	0.0	0.7	60.7	41.2
VC-ML	24DNPH	562	59.1	46.9	0.9	0.79	45.9	0.2	0.8	0.0	0.8	32.0	1.1	0.5	1.1	1.2	57.7	40.2
VC-MO	HPLC-EC			55.9	0.2		54.0	1.3	1.2	1.0	1.5	40.1	0.7	0.2	0.7	0.7		
VC-MQ	HPLC-UV	578	55.0	55.1	1.3	1.00	52.7	0.0	0.4	0.0	0.4	41.1	1.1	0.2	1.1	1.1	52.6	41.1
VC-MS	AutoAnal	533	55.9	45.4	1.1	0.81	47.2	0.0	0.1	0.0	0.1	36.2	1.0	0.7	0.9	1.1	58.1	44.6
VC-MV	HPLC-OPD	560	56.3	61.5	0.6	1.09	70.5	0.3	0.3	0.2	0.4	50.1	1.2	0.4	1.2	1.2	64.6	45.9
VC-MW	24DNPH	507	35.5	35.7	0.2	1.00	69.8	0.4	0.4	0.3	0.5	50.6	0.3	0.6	0.0	0.6	69.5	50.3
VC-MX	HPLC-EC	611	29.2	28.7	0.2	0.99						43.2	0.4	0.2	0.3	0.4		43.8
VC-MZ	HPLC-EC	560	28.7	26.2	0.1	0.91	55.4	0.6	0.5	0.5	0.7	39.5	0.0	0.2	0.0	0.2	60.7	43.2
VC-NB	HPLC-UV	609	53.4	43.7	0.2	0.82	43.8	0.2	0.3	0.0	0.3	31.7	0.0	0.4	0.0	0.4	53.6	38.8
VC-ND	HPLC-EC	583	55.6	59.2	0.5	1.06	56.1	6.1	1.9	6.0	6.3	42.5	1.4	2.5	0.0	2.5	52.7	39.9
VC-NG	HPLC-EC	566	57.2	62.6		1.09	45.4	0.1	0.3	0.0	0.3	33.0	1.7	1.1	1.5	1.8	41.5	30.2
VC-NJ	AO	551	62.8	68.4	1.2	1.09	60.3	0.6	0.3	0.6	0.6	42.9	0.9	3.0	0.0	3.0	55.4	39.4
VC-NO	HPLC-UV	538	50.6	46.3	4.6	0.91	56.9	2.3	0.9	2.2	2.4	35.5	1.8	2.6	0.0	2.6	62.2	38.8
	N	18	18	19		20	19					20					16	17
	Mean	578	49.4	46.6		0.99	56.9					40.9					57.6	41.3
	SD	61	15	19		0.10	8					5					6	4
	CV	11	30	41		11	14					13					11	10
	Min	507	5.8	2.3	0.0	0.79	43.8	0.0	0.0	0.0	0.0	31.7	0.0	0.1	0.0	0.2	41.5	30.2
	Median	564	55.3	55.1	0.5	1.00	56.9	0.3	0.4	0.0	0.7	41.8	0.7	0.5	0.2	0.8	57.9	41.2
	Max	802	62.8	68.4	4.6	1.13	70.5	6.1	2.3	6.0	6.3	50.6	1.8	3.0	1.5	3.0	69.5	50.3
	eSD	20	4	12		0.10	6					4					6	3
	eCV	4	7	22		10	11					10					11	8

02