NISTIR 7880-28

NIST Micronutrients Measurement Quality Assurance Program Winter, Spring, and Fall 1996 Comparability Studies

Results for Round Robin XXXVI, XXXVII, and XXXVIII Fat-Soluble Vitamins and Carotenoids in Human Serum and Round Robin 9 Ascorbic Acid in Human Serum

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

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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 1996 MMQAP measurement comparability improvement studies: 1) Round Robin XXXVI Fat-Soluble Vitamins and Carotenoids in Human Serum, 2) Round Robin XXXVII Fat-Soluble Vitamins and Carotenoids in Human Serum, 3) Round Robin XXXVIII Fat-Soluble Vitamins and Carotenoids in Human Serum, and 4) Round Robin 9 Ascorbic Acid in Human Serum. The materials for Round Robin XXXVI were shipped to participants in January 1996; participants were requested to provide their measurement results by March 15, 1996. The materials for Round Robin XXXVII were shipped to participants in April 1996; participants were requested to provide their measurement results by June 14, 1996. The materials for Round Robin XXXVIII were shipped to participants in July 1996; participants were requested to provide their measurement results by September 20, 1996. The sample materials for Round Robin 9 were distributed in May 1996 with results due by July 2, 1996.

Keywords

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

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin XXXVI comparability study (hereafter referred to as RR36) 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 1996. 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 RR36 has three sections:

- A cover letter, a "Lies, Damned Lies, and Statistics" summary report that describes the samples and our analysis of the participants' results, and a "Report of (Meta)Analysis" that presents a detailed analysis of measurements made by NIST analysts. This cover letter and associated reports are 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 XXXVII: Fat-Soluble Vitamins and Carotenoids in Human Serum

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin XXXVII comparability study (hereafter referred to as RR37) 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 1996. 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 RR37 has three sections:

- A cover letter, a "Lies, Damned Lies, and Statistics" summary report that describes the samples and our analysis of the participants' results, and a "Report of (Meta)Analysis" that presents a detailed analysis of measurements made by NIST analysts. This cover letter and associated reports are 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 XXXVIII: Fat-Soluble Vitamins and Carotenoids in Human Serum

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin XXXVIII comparability study (hereafter referred to as RR38) 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 1996. 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 RR38 has three sections:

- A cover letter, a "Lies, Damned Lies, and Statistics" summary report that describes the samples and our analysis of the participants' results, and a "Report of (Meta)Analysis" that presents a detailed analysis of measurements made by NIST analysts. This cover letter and associated reports are 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 9: Vitamin C in Human Serum

Participants in the MMQAP Vitamin C in Human Serum Round Robin 9 comparability study (hereafter referred to as RR09) 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 May 1996. 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 RR09 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 RR09, 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 RR09.

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 1996;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 RR36

The following two items were included in each package shipped to an RR36 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-

January 17, 1996

Dear Colleague:

Enclosed is the set of samples for the first QA round robin exercise (Round Robin XXXVI) of FY96 . You will find one vial 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 detection limit, please indicate this result on the form by using ND *(Not Detected)*. For analytes not measured, please leave a blank. Results are due to NIST by March 15. Results received two weeks after the due date will not be included in the summary report for this round robin study. Results will be discussed at the QA workshop which will be held Saturday, April 13, as a pre-meeting in conjunction with Experimental Biology '96 in Washington, DC. Around mid-April written feedback concerning the study will be provided to participants who cannot attend the workshop.

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 which 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 HPLC systems creating analytical problems.) Pipette a known volume of serum from the vial for analysis since 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; β-carotene, 2560 at 450 nm; lycopene (in hexane), 3450 at 472 nm.

Please mail or FAX your results for Round Robin XXXVI to:

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

Also enclosed are housing information and instructions for those who plan to attend the workshop. **There is no registration fee for the pre-meeting**. However, if you plan on attending the Experimental Biology conference on April 14-17, a registration fee is required. Please contact the American Institute of Nutrition secretariat at (301)530-7050; FAX: (301)571-1892 for details regarding the conference. We will continue to update you with the details of the workshop as plans are finalized. If you have questions regarding this round robin exercise or the workshop, please call me at (301) 975-3120 or mail/FAX queries to the above address.

Sincerely,

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

Enclosures

cc: W. May
S. Wise

NIST/NCI

Micronutrients Measurement Quality Assurance Program

Round Robin **XXXVI** Results from Laboratory #_____

	Serum				
Analyte	215	216	217	218	Units*
retinol					
retinyl palmitate					
α-tocopherol					
γ-tocopherol					
δ-tocopherol					
total β-carotene					
trans-β-carotene					
total cis-β-carotene					
total 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, 1996

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

Appendix B. Final Report for RR36

The following 20 pages are the final report for RR36 as provided to all participants:

- Cover letter
- A discussion entitled “Lies, Damn Lies, and Statistics” 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
- A “Report of (Meta)Analysis” that details the analysis of NIST measurements

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

April 23, 1996

Dear Colleague:

Enclosed is the summary report of the results for Round Robin XXXVI (Sera 215-218). 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 β-carotene; and a graphical summary of the NIST assigned value vs. laboratory value for these analytes. The NIST assigned values are derived from the equally weighted results from the analyses performed by NIST and the laboratories that participated in this round robin exercise.

In this round robin exercise, all serum samples were previously distributed. Serum 215 was distributed as Serum 170 in Round Robin XXVI; Serum 216 was distributed as Serum 182 in Round Robin XXVIII; Serum 217 as Serum 194 in Round Robin XXX; and Serum 218 as Serum 199 in Round Robin XXXII.

The overall laboratory performance for retinol, α- and γ-tocopherol, and β-carotene for this round robin exercise is comparable to that of the overall interlaboratory performance over the past two years. In this round robin, the average estimated coefficient of variation (eCV) is about 8% for both retinol and α-tocopherol, and about 14% for γ-tocopherol. The overall interlaboratory precision of retinol and α-tocopherol measurements for the past two years averaged around 10%. The interlaboratory variation for total β-carotene is about 19% for this round robin, as compared to the average eCV of around 21% over the past two years, provided that the levels are above the limit of quantification. Over the past years, in exercises where the levels of β-carotene were significantly lower (≤100 ng/mL), a higher variation was obtained due to the difficulty of making measurements at levels ≤100 ng/mL that appear to be at or below the limit of quantification for most laboratories. Similar findings are reported for retinyl palmitate, which was also at low concentrations in this exercise.

Interlaboratory precision for total α-carotene, β-cryptoxanthin, total lycopene, lutein, and zeaxanthin continues to improve (provided that the levels of these analytes are above the limit of quantification). About 48% of the reporting laboratories submitted results for total lycopene with an average eCV of about 24%. Values are also reported for δ-tocopherol, canthaxanthin, total carotenoids, *cis*-lutein and zeaxanthin, and coenzyme Q10. Laboratories are encouraged to report values for as many quantifiable analytes as possible.

Data for evaluating laboratory performance in Round Robin XXXVI are provided in the comparability summary on page 6 of the 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, 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.

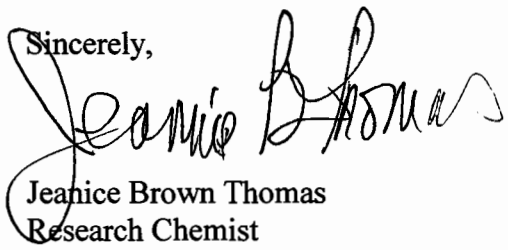
If you have concerns regarding your laboratory performance or were rated "POOR" based on the criteria stated above, we suggest that you obtain a unit of SRM 968b, Fat-Soluble Vitamins and Cholesterol in Human Serum, and analyze all three levels. If with minor modifications, your measured values do not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

Samples for Round Robin XXXVII and the food Round Robin IV (coordinated by Dr. Katherine Sharpless at 301/975-3121) will be shipped during the last week of April. The food round robin will be shipped only to the laboratories that indicate that they want samples. Results for both round robin exercises are due June 14; feedback to labs will be provided by July 26.

The home page for the QA program is still under construction. Laboratories will be updated as to when it becomes available. Reprints are now available for the publication: **Population Distributions and Intralaboratory Reproducibility for Fat-Soluble Vitamin-Related Compounds in Human Serum**, Sharpless and Duewer, Anal Chem, vol. 67, pp. 4416-4422, 1995. The following are forthcoming publications: **Certification of Fat-Soluble Vitamins, Carotenoids, and Cholesterol in Human Serum: Standard Reference Material 968b**, Brown Thomas *et al.*, Fresenius J Anal Chem, 1996, **Liquid Chromatographic Determination of Carotenoids in Human Serum Using an Engineered C_{30} Stationary Phase**, Sharpless *et al.*, J Chromatogr B:, 1996, and **The Measurement of Ascorbic Acid in Human Plasma and Serum: Stability, Intralaboratory Repeatability, and Interlaboratory Reproducibility**, Margolis and Duewer, Clin Chem, 1996. Please send reprint requests to:

Micronutrients Measurement Quality Assurance Program
National Institute of Standards and Technology
Chemistry, B208
Gaithersburg, MD 20899
Fax: (301)977-0685

Sincerely,



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

cc: W. May

"Lies, Damned Lies, and Statistics"

Mark Twain

The attached N^2M^2QAP Round Robin XXXVI Report has the same form as RR XXXV's. It includes the "All Lab" listing of everyone's results, your "Individualized" results, and our "(Meta)Analysis" of value and uncertainty assignments.

The "All Lab" report has the following elements:

Page	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 four analytes reported by only one laboratory.
5b	A Legend for the above two lists.
6	The "Measurement Comparability Summary" (or "Score card")

Your "Individualized" report has the following elements:

Page	Contents
1	Your values, our assigned values, and the %bias between you and us
2	%Bias barchart for retinol for your last 3 years' results
3	%Bias barchart for α- and γ-tocopherol for your last 3 years' results
4	%Bias barchart for total and *trans*-β-carotene for your last 3 years' results
5	Our assigned value vs. your value scatterplots for retinol, α- and γ-tocopherol, and total and *trans*-β-carotene, again for your last 3 years' results
6	Accuracy/Precision Summary, yet again for your last 3 years' results
7	Comparison-to-Prior-Analyses plots for retinol, retinyl palmitate, α- and γ-tocopherol, and total and *trans*-β-carotene
8	Comparison-to-Prior-Analyses plots for total α-carotene, total lycopene, β-cryptoxanthin, lutein, zeaxanthin, and lutein & zeaxanthin

The Comparison-to-Prior-Analyses plots on pages 7 and 8 show your individual results and a box-plot summary of the group's results, plotted against results from prior Round Robins:

Serum 215 was distributed as 170 and 172 in RR XXVI.
Serum 216 was distributed as 182 and 185 in RR XXVIII.
Serum 217 was distributed as 194 in RR XXX.
Serum 218 was distributed as 192 in RR XXX and 199 in RR XXXII.
The horizontal lines represent the mean value over all the distributions for each serum.

Note that sera 215 and 216 were previously distributed as same-exercise blind replicates. It is well worth your while to critically examine these plots. Differences between samples 170 and 72 and between 182 and 185 speak to how well you can repeat your analyses when they are

performed within a very short time period (minutes to hours); this is *short-term repeatability*. Differences between/among 170, 172 and 215; 182, 185 and 216; 194 and 217; and 192, 199, and 218 speak to how well you can repeat your analyses when separated by a rather long time period (months to years); this is *long-term repeatability*. You will be hearing more about this.

We are currently analyzing the past twelve years (!) of M^2N^2QAP interlaboratory comparison results. One aspect of the analysis of considerable interest is the relationship between interlaboratory variation (reproducibility eSD) and analyte concentration (median). The attached Table and Figures summarize the analysis. Note that: 1) the eSD has a low-concentration asymptote; 2) while all the high-concentration relationships appear linear, the slopes are different among the analytes; and 3) the "Coefficient of Variation" is ~constant over the analytical range of clinical interest only for retinol and α-tocopherol. We are currently modifying our analysis of interlaboratory results for very low analyte concentrations. We also are examining our quality criteria for the different analytes.

In our report for RR XXXV, we provided reference to some recent literature on the concentration of various fat-soluble vitamin-related compounds of interest. We apologize for switching the values for References 5 (Olmedilla, 1992) and 6 (Ascherio, 1992) in the associated table of summary values. Dr. Olmedilla provides an additional reference:

> Olmedilla B, Granado F, Blanco I, Rojas-Hidalgo E. Seasonal and sex-related variations in six serum carotenoids, retinol, and α-tocopherol. *Am J Clin Nutr* 1994; 60:106-10.

We would be most grateful if you would forward to us any pre/reprints you may have that are relevant to defining the analytical range for compounds of interest. We will provide a complete and updated summary with the RR XXXVIII report.

Your comments and suggestions are welcome. If you discover any errors in our recording or interpretation of your data, please let us know!

Dave Duewer
Research Chemometrician
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Margaret Kline
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Long-term Reproducibility as a Function of Analyte Concentration[a]

$$eSD = \sqrt{L_{qc}^2 + (\beta_0 \times [Analyte]^{\beta_1})^2}$$

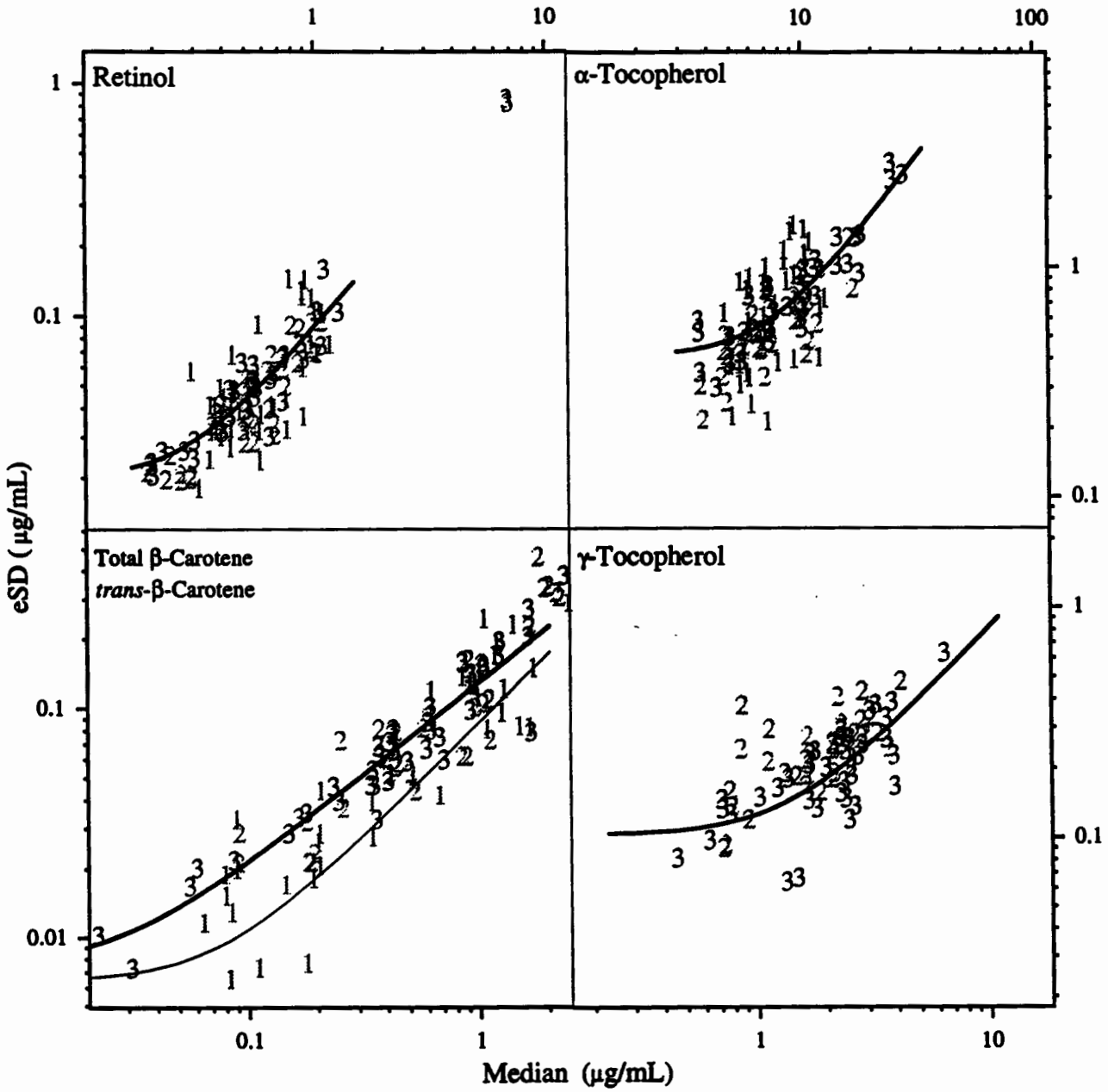
Analyte	Code	L_{qc}	β_o	β_1	Training[b] n	Training[b] ÷SD[d]	Evaluation[c] n	Evaluation[c] ÷SD[d]
Retinol	R	0.020 (0.003)	0.088 (0.005)	1.17 (0.07)	39	1.2	76	1.4
α-Tocopherol	αT	0.41 (0.06)	0.03 (0.02)	1.3 (0.2)	39	1.3	76	1.5
γ-Tocopherol	γT	0.10 (0.03)	0.07 (0.03)	1.0 (0.3)	39	1.4	36	1.7
Total β-Carotene	TβC	0.007 (0.003)	0.129 (0.008)	0.80 (0.07)	39	1.3	76	1.6
trans-β-Carotene	*t*βC	0.006 (0.003)	0.09 (0.01)	1.0 (0.2)	38	1.8	32	3.2
Retinyl Palmitate	RP	0.024 (0.003)	0.2 (0.5)	1 (2)	34	1.6	15	3.5
Total α-Carotene	αC	0.003 (0.002)	0.24 (0.01)	0.96 (0.02)	39	1.4	20	1.9
β-Cryptoxanthin	βCr	0.002 (0.003)	0.149 (0.006)	0.8 (0.1)	39	1.3	20	2.1
Total Lycopene	Ly	0	0.21 (0.02)	0.86 (0.05)	39	1.3	36	1.6

a) Concentrations in μg/mL

b) Results for samples distributed in RR XXVII through RR XXXV were used to "train" the least-squares regression.

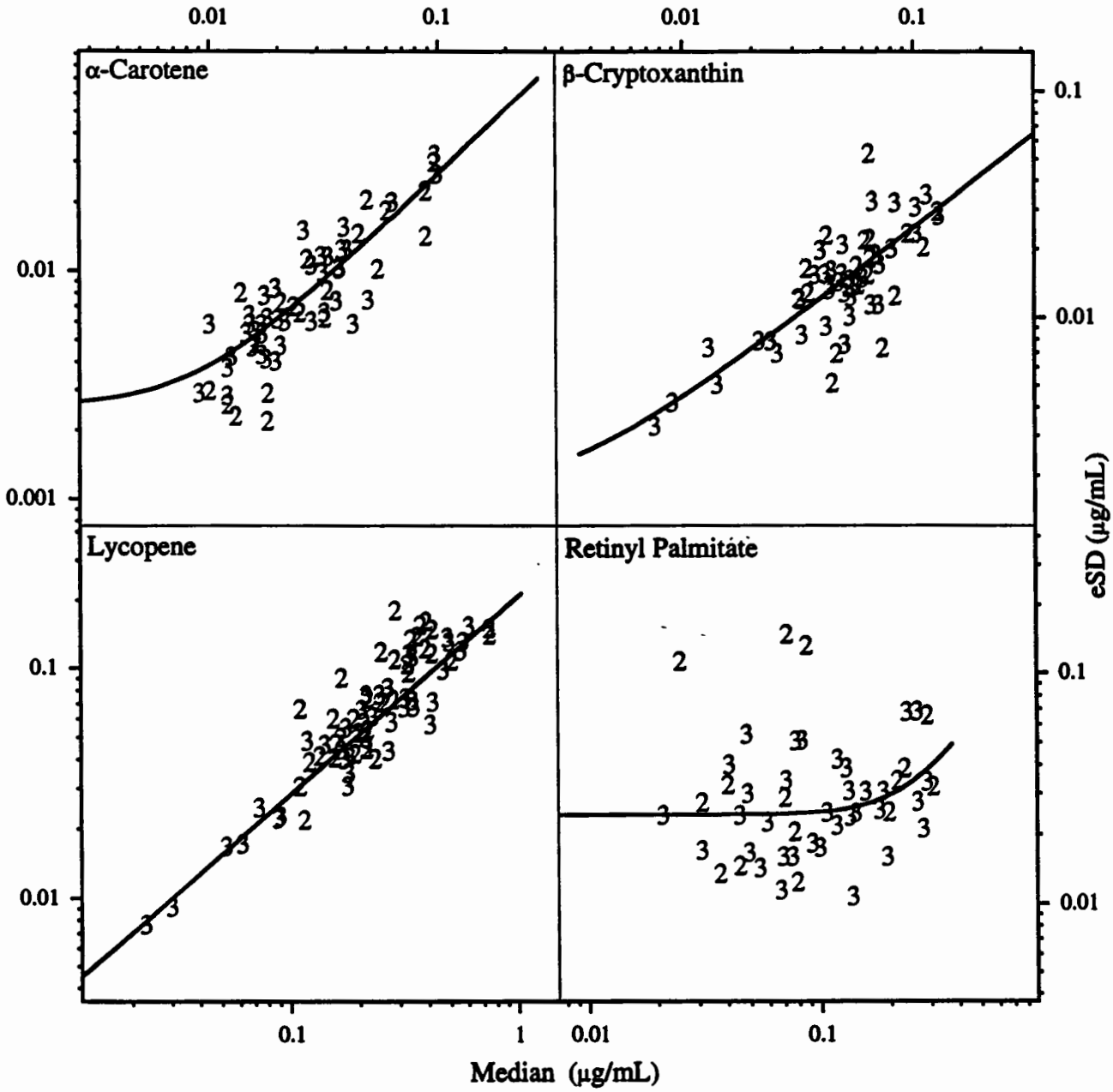
c) Results for samples distributed in RR VIII through RR XXVI we used to evaluate the predictive utility of the regression models.

d) The concentration-space "standard deviation" is an asymmetric factor, with ÷SD spanning the region from (concentration / SD) to (concentration × SD).

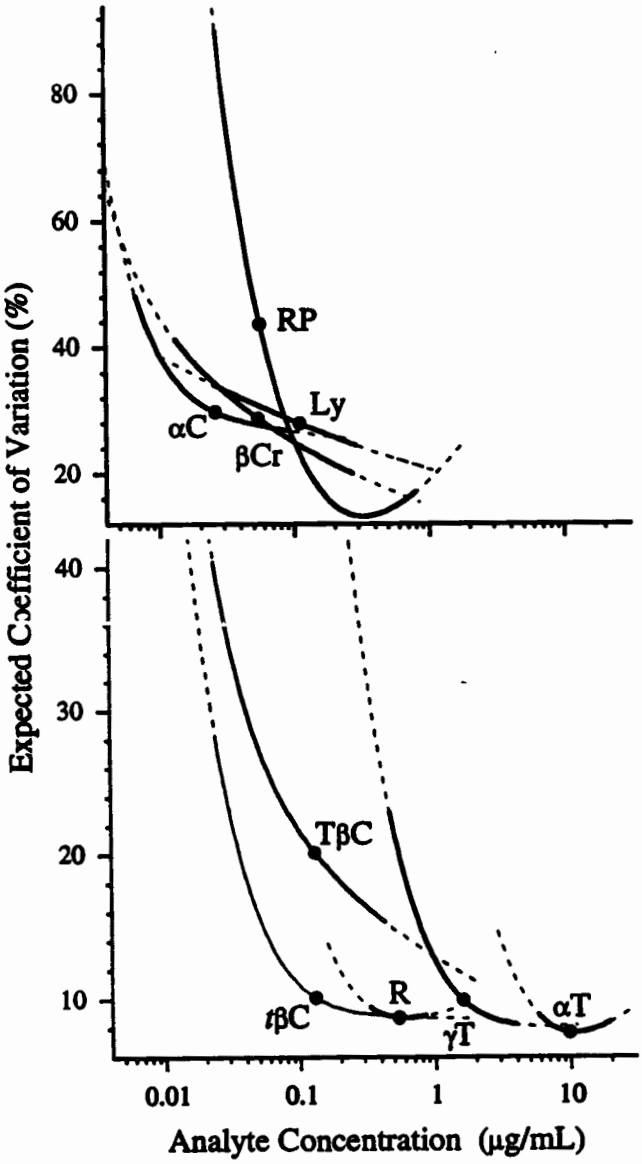


Dependence of Dispersion on Location: eSD as a Function of Concentration.

These plots display the dependencies of eSD (0.741 × interquartile range) on the median estimate of analyte concentration. The data have been divided into three approximately equal-sized groups by: all data taken during 1986–89 ("1"), 1990–92 ("2"), and 1993–95 ("3"). The lines represent least squares regression to the model: $eSD = \sqrt{L_{qc}^2 + (\beta_0 \times Median^{\beta_1})^2}$, using just the most recent third of the data. Each axis of each scattergram spans the same 120-fold relative concentration range.



Dependence of Dispersion on Location: eSD as a Function of Concentration.



Dependence of Expected Coefficient of Variation on Analyte Concentration.

Curves are displayed for eCV as calculated from all eSD regression models shown in the previous Figures. Dashed light lines span the entire range of analyte concentrations encountered during analysis of U.S. adult human sera; the solid dark lines span the central 90% of these measurements; the median is denoted as "●".

From: David L. Duewer Date: April 12, 1996

To: Jeanice Brown Thomas, Margaret Kline, Willie E. May, Katherine E. Sharpless
cc: Gary W. Kramer, Dennis J. Reeder, Stephen A. Wise

Re: Value and Uncertainty Assignment for the NIST/NCI Micronutrient Measurement Quality Assurance Program's Round Robin XXXVI (RR36) Sera: 215 to 218.

Background: Four sera were distributed in RR36. Serum 215 was distributed as 170 and 172 in RR26, serum 216 was distributed as 182 and 185 in RR28, serum 217 was distributed as 194 in RR30, and serum 218 was distributed as 192 in RR30 and 199 in RR32. Note that both sera 216 and 217 were originally distributed as same-exercise blind replicates.

Analysts NIST1 and NIST3 independently extracted and analyzed two aliquots of three vials of each serum using normal procedures. Both analysts reported quantitative results for retinol, α-tocopherol, γ-tocopherol, total β-carotene, and *trans*-β-carotene. Analyst NIST3 also reported results for retinyl palmitate, δ-tocopherol, total α-carotene, *trans*-α-carotene, total lycopene, *trans*-lycopene, β-cryptoxanthin, "lutein", and "zeaxanthin". (NIST3 is uncertain about the isomeric composition of the lutein and zeaxanthin peaks.)

Results: Table 1 presents the NIST data, summary statistics for the NIST data, summary results for the most recent previous exercise for each serum, summary results for RR36, and the NIST assigned values and uncertainties. Table 2 presents a compact summary of the assigned values and uncertainties for each analyte in each serum.

The entries in Table 1 are 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 ≥ 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 estimated as the residual standard deviation for the linear regression of NIST1's $Mean_x$ values onto NIST3's. For analytes that NIST1 did not report, SD_{anl} is estimated from the regression of the RR35 interlaboratory Median (see below) onto NIST3's $Mean_x$ values. The

regression model used to determine SD_{anl} is defined to the right of this block. Details include: the model used, the parameters and standard errors on the parameters, and R^2 for the regression.

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

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

- Previous Round Robin Summary Statistics

RR identifier of the Round Robin in which this serum was distributed

Serum identifier of the serum

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

$Median_p$ median of the reported values. The median is a robust location estimate

eSD_p 0.741 × InterQuartile Range (IQR). The IQR is a robust dispersion estimate. The scale factor 0.741 is the expected ratio between SD and IQR for normal distributions.

- Round Robin XXXVI 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) $TDIST\left(\frac{|Median_n - Median_p|\sqrt{n_n + n_p - 2}}{\sqrt{\left((n_n - 1)eSD_n^2 + (n_p - 1)eSD_p^2\right)\left(\frac{1}{n_n} + \frac{1}{n_p}\right)}}, n_p+n_n-2, 2\ tail\right)$

This is the approximate probability that the median in RR35 is the same as it was in the previous RR. Where the hypothesis that $Median_n = Median_p$ could be rejected with 95% confidence, the P(n=p) value would be 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 interlaboratory variance in RR36 is smaller than it was in the previous RR. 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, inter-analyst, 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 $100 \times NAU / NAV$

Conclusions: All median values from RR36 ($Median_n$) are statistically indistinguishable at the 95% confidence level from the corresponding values ($Median_p$) determined in previous exercises. Nearly all of the interlaboratory standard deviations from RR36 (eSD_n) are likewise indistinguishable at the 95% confidence level from their previous values (eSD_p). The few apparent increases are scattered across analytes and sera: there is no evidence for degradation of any of the sera during storage.

The two sets of sera that were originally distributed as same-exercise blind duplicates provide a mechanism for examining both short- and long-term repeatability. While strictly a property of individual laboratories, we can easily evaluate the repeatability characteristic of an "average" participant. The following example is for retinol, with the first set of sera: 170 and 172 distributed in RR26 and 215 distributed in RR36.

	Same-Exercise ("Short-Term")					Among-Exercise ("Long-Term")					
Lab	170	172	$Mean_A$	Δ_A		Lab	170	172	215	$Mean_B$	Δ_B	
	0.69	0.68	0.68	0.00			0.69	0.68	0.67	0.67	0.02	
	0.61	0.62	0.61	-0.01			0.61	0.62	0.59	0.60	0.02	
	0.67	0.69	0.68	-0.02			0.67	0.69	0.66	0.67	0.02	
	0.65	0.67	0.66	-0.01								
	0.60	0.60	0.60	0.01			0.60	0.60	0.60	0.60	0.00	
	0.63	0.63	0.63	0.01								
	0.67	0.67	0.67	-0.01			0.67	0.67	0.62	0.65	0.04	
	0.62	0.64	0.63	-0.02			0.62	0.64	0.63	0.63	0.00	
	0.76	0.68	0.72	0.09			0.76	0.68	0.58	0.65	0.14	
	0.71	0.69	0.70	0.02			0.71	0.69	0.66	0.68	0.03	
	0.64	0.67	0.65	-0.02								
	0.65	0.65	0.65	0.00			0.65	0.65	0.65	0.65	0.00	
	0.67	0.67	0.67	0.00			0.67	0.67	0.47	0.57	0.20	
	0.65	0.65	0.65	-0.01			0.65	0.65	0.65	0.65	0.00	
	0.61	0.62	0.62	-0.01								
	0.70	0.72	0.71	-0.03								
	0.68	0.74	0.71	-0.05			0.68	0.74	0.69	0.70	0.02	
	0.65	0.60	0.62	0.05			0.65	0.60	0.63	0.63	-0.01	
	0.69	0.68	0.69	0.01			0.69	0.68	0.66	0.67	0.03	
	0.67	0.67	0.67	0.00								
	0.59	0.60	0.60	-0.01			0.59	0.60	0.60	0.60	0.00	
	0.66	0.67	0.66	-0.01								
	0.69	0.69	0.69	0.00								
	0.72	0.68	0.70	0.04			0.72	0.68	0.55	0.62	0.15	
	0.68	0.71	0.70	-0.03								
	0.77	0.79	0.78	-0.02			0.77	0.79	0.63	0.71	0.15	
	0.77	0.77	0.77	0.00			0.77	0.77	0.62	0.69	0.15	
	0.70	0.72	0.71	-0.02			0.70	0.72	0.66	0.68	0.05	
	0.68	0.66	0.67	0.02			0.68	0.66	0.74	0.71	-0.07	
	0.64	0.67	0.66	-0.03			0.64	0.67	0.75	0.70	-0.09	
	0.68	0.65	0.67	0.03			0.68	0.65	0.68	0.67	-0.02	
	0.61	0.61	0.61	0.00			0.61	0.61	0.55	0.58	0.06	
n	32	32	32	32	Ratio		23	23	23	23	23	Ratio
Median	0.668	0.666	0.667	-0.004	-0.01		0.670	0.671	0.630	0.652	0.021	0.03
IQR	0.047	0.045	0.053	0.026	0.49		0.049	0.045	0.061	0.055	0.061	1.11

Where: $Mean_A = (X_{170}+X_{172})/2$, $\Delta_A = X_{170}-X_{172}$, $Mean_B = (Mean_A+X_{215})/2$, and $\Delta_B = Mean_A-X_{215}$. The short- and long-term bias are more conveniently expressed relative to the sample concentration: (median Δ) / (median Mean). The corresponding repeatabilities are more conveniently expressed relative to the interlaboratory reproducibility: (IQR Δ) / (IQR Mean).

In this example, the short-term repeatability is about one-half (0.49) of the interlaboratory reproducibility; the long-term repeatability is slightly LARGER (1.11) than the reproducibility. The following summarizes the fractional biases and repeatabilities for a number of analytes, where the short-term values have been pooled over the two same-exercise sets and the long-term have been pooled over the six different-exercise pairs.

		Bias		Repeatability	
Analyte	N	Short	Long	Short	Long
α-Tocopherol	40	0.001	0.040	0.29	1.46
Retinol	38	-0.003	0.046	0.42	1.33
Total β-Carotene	28	0.007	0.043	0.48	1.48
γ-Tocopherol	16	-0.007	0.017	0.50	1.55
Total α-Carotene	16	0.022	-0.023	0.35	0.79
Total Lycopene	16	-0.006	0.030	0.26	1.76
β-Cryptoxanthin	11	-0.007	-0.007	0.29	0.98
Retinyl palmitate	8	0.045	0.015	0.37	0.77
trans-β-Carotene	6	0.022	0.002	0.37	2.30

Where "N" is the average number of laboratories contributing data, the short- and long-term biases are (median Δ) / (median Mean), and the repeatabilities are (IQR Δ) / (IQR Mean). The long-term biases tend to be a little larger than the short-term, but this is at the "noise level" of the data. ALL short-term repeatabilities are much smaller than the reproducibility. ALL long-term repeatabilities are much larger than their short-term cousins; long-term repeatabilities for the data-rich analytes are considerably larger than the reproducibilities.

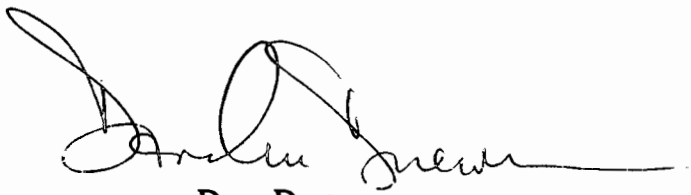
The basic conclusion is that our "average" participant is as or more variable over time as the "in control" group of laboratories taken as a whole. Individual participants, of course, will have their own characteristic repeatabilities... some much better and some rather worse than this "average" analysis.

⇒ *We could "rank" individual laboratories by their long-term repeatabilities.* ⇐

Is this desirable?

Systematic constant and/or proportional biases are again apparent between the NIST1 and NIST3 analyses for many analytes. These biases are statistically significant when the standard errors for the intercept "a" and/or slope "b" regression parameters are small relative to the differences between the observed and expected 0.0 (intercept) and 1.0 (slope) values.

This memo will be included as part of the Round Robin XXXVI report.



Dave Duewer
Reseach Busybody

Table 1
NIST Data and Calculations

Retinol

	NIST1				NIST3			
	215	216	217	218	215	216	217	218
A:1	0.707	0.548	0.425	1.071	0.661	0.539	0.433	1.048
A:2	0.685	0.610	0.444	1.048	0.660	0.540	0.443	1.075
B:1	0.674	0.554	0.445	1.021	0.660	0.564	0.431	1.049
B:2	0.688	0.537	0.442	1.032	0.702	0.562	0.424	1.035
C:1	0.675	0.547	0.445	0.993	0.724	0.567	0.439	1.050
C:2		0.611	0.448	1.036	0.686	0.565	0.443	1.049
n_x	5	6	6	6	6	6	6	6
$Mean_x$	0.686	0.568	0.442	1.034	0.682	0.556	0.436	1.051
SD_x	0.013	0.033	0.008	0.026	0.027	0.013	0.008	0.013
SD_{repx}	0.011	0.037	0.008	0.020	0.023	0.001	0.005	0.012
SD_{hetx}	0.011	0.019	0.006	0.023	0.022	0.015	0.007	0.010
SD_{NISTx}	0.015	0.042	0.010	0.031	0.032	0.015	0.009	0.016
CV_{NISTx}	2.2	7.4	2.3	3.0	4.7	2.7	2.1	1.5

Retinyl Palmitate

	NIST1				NIST3			
	215	216	217	218	215	216	217	218
A:1	0.248				0.226			
A:2	0.228				0.247			
B:1	0.279							
B:2	0.249							
C:1	0.249							
C:2	0.230							
n_x	6	0	0	0	2	0	0	0
$Mean_x$	0.247				0.237			
SD_x	0.018				0.015			
SD_{repx}	0.017				0.015			
SD_{hetx}	0.015							
SD_{NISTx}	0.022							
CV_{NISTx}	9.0							

	NIST			
n	11	12	12	12
Mean	0.684	0.562	0.439	1.042
SD_{rep}	0.019	0.026	0.007	0.017
SDhet	0.016	0.016	0.007	0.017
SD_{anl}	0.006	0.006	0.006	0.006
SD_{NIST}	0.025	0.031	0.012	0.025
CV_{NIST}	3.7	5.6	2.7	2.4

NIST3=a+b*NIST1

a: -0.032 ±0.010
b: 1.046 ±0.015
R^2: 0.999

	NIST			
n	8	0	0	0
Mean	0.242			
SD_{rep}	0.016			
SDhet	0.015			
SD_{anl}				
SD_{NIST}	0.022			
CV_{NIST}	9.1			

RR	XXVI	XXVIII	XXX	XXXII
Serum	170	182	194	199
n_p	33	43	43	39
$Median_p$	0.668	0.550	0.430	1.070
eSD_p	0.035	0.051	0.037	0.075

	XXVI	XXVIII	XXX	XXXII
	170	182	194	199
	8	8	6	6
	0.307	0.08	0.04	0.05
	0.033	0.02	0.04	0.03

	RRXXXVI			
	215	216	217	218
n_n	45	45	45	45
$Median_n$	0.657	0.536	0.425	1.059
eSD_n	0.055	0.039	0.037	0.082
P(n=p)	0.96	0.95	0.98	0.94
P(n<p)	0.00*	0.96	0.56	1.00
SD_{labs}	0.049	0.022	0.035	0.078
CV_{labs}	7.4	4.2	8.2	7.4

<— Current Results —>

	RRXXXVI			
	215	216	217	218
	14	13	8	10
	0.300	0.06	0.02	0.03
	0.062	0.02	0.03	0.03
	0.95	0.77	0.79	0.56
	0.05*	0.30	0.86	0.96
	0.058			
	19			

NAV	0.670	0.549	0.432	1.051
NAU	0.055	0.039	0.037	0.082
CV	8.2	7.0	8.5	7.8

<— Assignments —>

	0.271
	0.062
	23

Table 1
NIST Data and Calculations

α-Tocopherol

	NIST1				NIST3			
	215	216	217	218	215	216	217	218
A:1	6.03	5.67	7.09	7.09	6.06	5.71	7.57	7.26
A:2	6.43	5.93	6.77	6.99	6.19	5.71	7.28	6.97
B:1	5.96	5.61	7.13	7.13	5.92	5.86	7.84	7.19
B:2	5.79	5.25	6.95	7.23	6.05	5.83	7.45	7.06
C:1	6.27	5.81	6.99	7.45	5.98	5.83	7.59	7.24
C:2		5.83	6.80	6.89	6.57	5.73	7.56	7.51
n_x	5	6	6	6	6	6	6	6
$Mean_x$	6.10	5.68	6.95	7.13	6.13	5.78	7.55	7.21
SD_x	0.25	0.24	0.15	0.20	0.23	0.07	0.18	0.19
SD_{repx}	0.17	0.18	0.17	0.24	0.25	0.04	0.20	0.17
SD_{betx}	0.22	0.22	0.07	0.08	0.14	0.07	0.11	0.15
SD_{NISTx}	0.28	0.29	0.18	0.25	0.29	0.08	0.23	0.22
CV_{NISTx}	4.6	5.0	2.6	3.5	4.7	1.4	3.0	3.1

γ-Tocopherol

	NIST1				NIST3			
	215	216	217	218	215	216	217	218
A:1	2.39	2.02	2.47	2.55	2.00	2.53	3.10	2.40
A:2	2.36	2.25	2.77	2.27	2.01	2.52	3.02	2.34
B:1	2.23	2.05	2.68	2.02	1.95	2.57	3.14	2.37
B:2	2.00	2.10	3.16	2.22	1.99	2.52	3.07	2.37
C:1	2.05	2.05	2.94	2.39	1.96	2.51	3.07	2.35
C:2		2.13	2.59	2.15	2.07	2.51	3.12	2.39
n_x	5	6	6	6	6	6	6	6
$Mean_x$	2.21	2.10	2.77	2.27	2.00	2.53	3.09	2.37
SD_x	0.18	0.08	0.25	0.19	0.04	0.02	0.04	0.02
SD_{repx}	0.09	0.10	0.27	0.17	0.05	0.02	0.05	0.03
SD_{betx}	0.17	0.03	0.15	0.15	0.02	0.02	0.02	0.00
SD_{NISTx}	0.20	0.11	0.31	0.23	0.05	0.03	0.05	0.03
CV_{NISTx}	8.9	5.0	11	10.0	2.6	1.2	1.7	1.3

	NIST			
n	11	12	12	12
Mean	6.11	5.73	7.25	7.17
SD_{rep}	0.14	0.13	0.22	0.19
SDhet	0.20	0.16	0.22	0.13
SD_{anl}	0.26	0.26	0.26	0.26
SD_{NIST}	0.35	0.33	0.40	0.35
CV_{NIST}	5.8	5.7	5.5	4.9

NIST3=a+b*NIST1

a: 0
b: 1.03 ±0.02
R^2: 0.826

	NIST			
	11	12	12	12
	2.10	2.31	2.93	2.32
	0.07	0.08	0.20	0.12
	0.22	0.16	0.12	0.10
	0.28	0.28	0.28	0.28
	0.36	0.33	0.36	0.32
	17	14	12	14

NIST3=a+b*NIST1

a: 0
b: 1.07 ±0.06
R^2: 0.391

RR	XXVI	XXVIII	XXX	XXXII
Serum	170	182	194	199
n_p	35	43	43	41
$Median_p$	6.15	5.48	7.30	7.25
eSD_p	0.63	0.40	0.55	0.48

<— Previous Results —>

	XXVI	XXVIII	XXX	XXXII
	170	182	194	199
	12	16	20	18
	2.10	2.48	3.22	2.48
	0.26	0.12	0.38	0.27

	RRXXXVI			
	215	216	217	218
n_a	43	43	43	43
$Median_a$	5.97	5.47	7.18	7.01
eSD_a	0.44	0.37	0.67	0.56
P(n=p)	0.94	1.00	0.97	0.93
P(n<p)	0.99	0.69	0.10	0.88
SD_{labs}	0.26	0.17	0.54	0.44
CV_{labs}	4.3	3.1	7.5	6.3

<— Current Results —>

	RRXXXVI			
	215	216	217	218
	21	21	21	21
	1.97	2.50	3.08	2.41
	0.29	0.28	0.33	0.35
	0.88	0.97	0.90	0.89
	0.39	0.00*	0.73	0.01*
	0	0	0	0.14
	0	0	0	5.6

NAV	6.04	5.60	7.22	7.09
NAU	0.44	0.37	0.67	0.56
CV	7.2	6.6	9.3	7.9

<— Assignments —>

2.04	2.41	3.00	2.36
0.36	0.33	0.36	0.35
18	14	12	15

Table 1
NIST Data and Calculations

trans-Lycopene

	NIST1				NIST3			
	215	216	217	218	215	216	217	218
A:1					0.113	0.129	0.277	0.312
A:2					0.124	0.138	0.275	0.324
B:1					0.113	0.138	0.275	0.323
B:2					0.117	0.141	0.278	0.330
C:1					0.111	0.143	0.280	0.316
C:2					0.119	0.134	0.267	0.332
n_x	0	0	0	0	6	6	6	6
$Mean_x$					0.116	0.137	0.275	0.323
SD_x					0.005	0.005	0.005	0.008
SD_{repx}					0.006	0.005	0.006	0.009
SD_{hetx}					0.002	0.003	0.002	0.004
SD_{NISTx}					0.006	0.006	0.006	0.010
CV_{NISTx}					5.3	4.5	2.1	3.0

	NIST			
n	6	6	6	6
Mean	0.116	0.137	0.275	0.323
SD_{rep}	0.005	0.004	0.002	0.006
SDhet	0.002	0.003	0.002	0.004
SD_{anl}	0.009	0.009	0.009	0.009
SD_{NIST}	0.010	0.010	0.009	0.012
CV_{NIST}	8.9	7.5	3.3	3.6

NIST3=a+b*Median

a: 0.018 ±0.016
b: 0.772 ±0.050
R^2: 0.992

RR	XXVI	XXVIII	XXX	XXXII
Serum	170	182	194	199
n_p	1	0		1
$Median_p$	0.134			0.353
eSD_p				

<— Previous Results —>

	RRXXXVI			
	215	216	217	218
n_n	7	7	7	7
$Median_n$	0.134	0.157	0.324	0.401
eSD_n	0.026	0.013	0.014	0.075
P(n=p)				
P(n<p)				
SD_{labs}	0.024	0.008	0.010	0.074
CV_{labs}	18	5.3	3.1	18

<— Current Results —>

NAV	0.125	0.147	0.300	0.362
NAU	0.026	0.013	0.015	0.075
CV	21	9.0	5.0	21

<—— Assignments ——>

β-Cryptoxanthin

	NIST1				NIST3			
	215	216	217	218	215	216	217	218
A:1					0.048	0.038	0.037	0.041
A:2					0.049	0.034	0.034	0.046
B:1					0.057	0.033	0.034	0.040
B:2					0.048	0.038	0.035	0.042
C:1					0.041	0.032	0.037	0.050
C:2					0.049	0.031	0.028	0.046
n_x	0	0	0	0	6	6	6	6
$Mean_x$					0.049	0.034	0.034	0.044
SD_x					0.005	0.003	0.003	0.004
SD_{repx}					0.005	0.002	0.004	0.003
SD_{hetx}					0.003	0.002	0.001	0.004
SD_{NISTx}					0.006	0.003	0.004	0.004
CV_{NISTx}					12	9.9	12	10

	NIST			
	6	6	6	6
	0.049	0.034	0.034	0.044
	0.005	0.003	0.001	0.002
	0.003	0.002	0.001	0.004
	0.002	0.002	0.002	0.002
	0.007	0.004	0.003	0.005
	13	12	7.6	10

NIST3=a+b*Median

a: 0.008 ±0.006
b: 0.649 ±0.135
R^2: 0.918

	XXVI	XXVIII	XXX	XXXII
	170	182	194	199
	9	11	18	16
	0.073	0.042	0.056	0.071
	0.007	0.009	0.014	0.017

	RRXXXVI			
	215	216	217	218
	17	17	17	17
	0.059	0.039	0.042	0.055
	0.016	0.012	0.014	0.019
	0.67	0.93	0.75	0.82
	0.02*	0.22	0.47	0.55
	0.014	0.011	0.014	0.018
	24	28	33	33

	0.054	0.037	0.038	0.050
	0.016	0.012	0.014	0.019
	29	32	37	37

Table 1
NIST Data and Calculations

δ-Tocopherol

	NIST1				NIST3			
	215	216	217	218	215	216	217	218
A:1					0.08	0.12	0.13	0.13
A:2					0.08	0.14	0.11	0.14
B:1					0.07	0.13	0.12	0.14
B:2					0.08	0.14	0.11	0.12
C:1					0.09	0.12	0.10	0.12
C:2					0.07	0.13	0.12	0.13
n_x	0	0	0	0	6	6	6	6
$Mean_x$					0.08	0.13	0.12	0.13
SD_x					0.01	0.01	0.01	0.01
SD_{repx}					0.01	0.01	0.01	0.01
SD_{hetx}					0.00	0.01	0.00	0.01
SD_{NISTx}					0.01	0.01	0.01	0.01
CV_{NISTx}					9.7	7.2	8.3	9.8

Total β-Carotene

	NIST1				NIST3			
	215	216	217	218	215	216	217	218
A:1	0.665	0.385		0.635	0.498	0.405	0.048	0.557
A:2	0.598	0.450		0.613	0.600	0.407	0.047	0.578
B:1	0.674	0.431		0.598	0.521	0.425	0.056	0.568
B:2	0.666	0.447		0.585	0.555	0.435	0.057	0.562
C:1	0.693	0.394		0.669	0.525	0.423	0.049	0.579
C:2		0.443		0.571	0.583	0.420	0.052	0.561
n_x	5	6	0	6	6	6	6	6
$Mean_x$	0.659	0.425		0.612	0.547	0.419	0.052	0.567
SD_x	0.036	0.028		0.036	0.039	0.012	0.004	0.009
SD_{repx}	0.028	0.034		0.041	0.050	0.004	0.002	0.012
SD_{hetx}	0.031	0.012		0.018	0.008	0.012	0.005	0.002
SD_{NISTx}	0.042	0.036		0.045	0.051	0.013	0.005	0.012
CV_{NISTx}	6.3	8.5		7.4	9.3	3.1	9.3	2.1

δ-Tocopherol

	NIST			
n	6	6	6	6
Mean	0.08	0.13	0.12	0.13
SD_{rep}	0.01	0.01	0.01	0.01
SDhet	0.00	0.01	0.00	0.01
SD_{anl}	0.03	0.03	0.03	0.03
SD_{NIST}	0.03	0.03	0.03	0.03
CV_{NIST}	38	24	27	25

NIST3=a+b*Median

a: 0.11 ±0.07
b: 0.02 ±0.47
R^2: 0.000

Total β-Carotene

	NIST			
n	11	12	6	12
Mean	0.603	0.422	0.052	0.590
SD_{rep}	0.038	0.024	0.001	0.030
SDhet	0.008	0.012	0.005	0.002
SD_{anl}	0.016	0.016	0.016	0.016
SD_{NIST}	0.042	0.032	0.017	0.034
CV_{NIST}	7.0	7.5	32	5.8

NIST3=a+b*Median

a: 0
b: 1.026 ±0.023
R^2: 0.993

RR	XXVI	XXVIII	XXX	XXXII
Serum	170	182	194	199
n_p		0	0	1
$Median_p$				0.81
eSD_p				

<— Previous Results —>

	XXVI	XXVIII	XXX	XXXII
	170	182	194	199
	30	33	32	32
	0.602	0.430	0.060	0.593
	0.093	0.067	0.021	0.068

δ-Tocopherol

	RRXXXVI			
	215	216	217	218
n_n	4	4	4	4
$Median_n$	0.15	0.19	0.11	0.12
eSD_n	0.21	0.17	0.16	0.15
P(n=p)				
P(n<p)				
SD_{labs}	0.21	0.17	0.16	0.15
CV_{labs}	141	86	144	124
NAV	0.12	0.16	0.11	0.13
NAU	0.21	0.17	0.16	0.15
CV	190	100	140	120

<— Current Results —>

<—— Assignments ——>

Total β-Carotene

	RRXXXVI			
	215	216	217	218
	31	30	31	30
	0.551	0.391	0.056	0.565
	0.090	0.071	0.014	0.098
	0.89	0.89	0.95	0.92
	0.58	0.37	0.98	0.29
	0.079	0.063	0	0.092
	14	16	0	16
	0.577	0.406	0.054	0.577
	0.090	0.071	0.017	0.098
	16	17	31	17

Table 1
NIST Data and Calculations

trans-β-Carotene

	NIST1				NIST3			
	215	216	217	218	215	216	217	218
A:1	0.539	0.344		0.598	0.480	0.393	0.052	0.509
A:2	0.520	0.365		0.488	0.551	0.389	0.053	0.530
B:1	0.515	0.343		0.488	0.495	0.390	0.060	0.517
B:2	0.508	0.331		0.486	0.514	0.395	0.059	0.519
C:1	0.548	0.381		0.528	0.505	0.399	0.052	0.507
C:2		0.398		0.487	0.536	0.401	0.050	0.514
n_x	5	6	0	6	6	6	6	6
$Mean_x$	0.526	0.360		0.513	0.514	0.395	0.054	0.516
SD_x	0.017	0.026		0.045	0.026	0.005	0.004	0.008
SD_{repx}	0.008	0.012		0.048	0.033	0.003	0.001	0.009
SD_{hetx}	0.018	0.027		0.028	0.008	0.005	0.005	0.005
SD_{NISTx}	0.020	0.029		0.056	0.034	0.006	0.005	0.010
CV_{NISTx}	3.8	8.1		11	6.5	1.4	8.6	2.0

Total α-Carotene

	NIST1				NIST3			
	215	216	217	218	215	216	217	218
					0.019	0.021	0.014	0.042
					0.043	0.021	0.014	0.064
					0.020	0.017	0.011	0.028
					0.032	0.022	0.010	0.029
					0.023	0.029	0.013	0.040
					0.029	0.020	0.014	0.030
	0	0	0	0	6	6	6	6
					0.028	0.022	0.013	0.039
					0.009	0.004	0.002	0.014
					0.011	0.004	0.000	0.010
					0.003	0.003	0.002	0.013
					0.012	0.005	0.002	0.016
					41	23	15	41

trans-β-Carotene

	NIST			
n	11	12	6	12
Mean	0.520	0.377	0.054	0.514
SD_{rep}	0.023	0.009	0.001	0.034
SDhet	0.008	0.005	0.005	0.005
SD_{anl}	0.024	0.024	0.024	0.024
SD_{NIST}	0.034	0.026	0.025	0.042
CV_{NIST}	6.6	7.0	46	8.3

NIST3=a+b*Median

a: 0
b: 0.928 ±0.035
R^2: 0.979

RR	XXVI	XXVIII	XXX	XXXII
Serum	170	182	194	199
n_p	4	5	9	7
$Median_p$	0.553	0.397	0.049	0.531
eSD_p		0.020	0.016	0.047

<— Previous Results —>

RRXXXVI				
	215	216	217	218
n_n	11	11	11	11
$Median_n$	0.532	0.395	0.053	0.577
eSD_n	0.079	0.041	0.015	0.081
P(n=p)		0.98	0.91	0.89
P(n<p)		0.09	0.58	0.39
SD_{labs}	0.072	0.032	0	0.069
CV_{labs}	13	8.1	0	12

<— Current Results —>

NAV	0.526	0.386	0.054	0.546
NAU	0.079	0.041	0.025	0.081
CV	15	11	46	15

<—— Assignments ——>

Total α-Carotene

	NIST			
	6	6	6	6
	0.028	0.022	0.013	0.039
	0.012	0.003	0.000	0.009
	0.003	0.003	0.002	0.013
	0.001	0.001	0.001	0.001
	0.012	0.004	0.002	0.015
	44	20	19	40

NIST3=a+b*Median

a: -0.061 ±0.007
b: 5.247 ±0.398
R^2: 0.989

	XXVI	XXVIII	XXX	XXXII
	170	182	194	199
	16	15	21	21
	0.024	0.016	0.012	0.017
	0.007	0.006	0.003	0.006

RRXXXVI				
	215	216	217	218
	23	21	21	21
	0.022	0.016	0.014	0.019
	0.008	0.004	0.007	0.011
	0.95	1.00	0.91	0.96
	0.34	0.86	0.00*	0.07
	0	0	0.007	0
	0	0	50	0

	0.025	0.019	0.013	0.029
	0.012	0.004	0.007	0.015
	50	23	55	53

Table 1
NIST Data and Calculations

trans-α-Carotene

	NIST1				NIST3			
	215	216	217	218	215	216	217	218
A:1					0.02	0.02	0.017	0.02
A:2					0.03	0.02	0.021	0.04
B:1					0.02	0.01	0.016	0.02
B:2					0.03	0.02	0.017	0.02
C:1					0.03	0.03	0.017	0.02
C:2					0.02	0.02	0.014	0.02
n_x	0	0	0	0	6	6	6	6
$Mean_x$					0.03	0.02	0.017	0.02
SD_x					0.00	0.00	0.002	0.01
SD_{repx}					0.01	0.01	0.002	0.01
SD_{hetx}					0.00	0.00	0.002	0.01
SD_{NISTx}					0.01	0.01	0.003	0.01
CV_{NISTx}					23	27	16	46

Total Lycopene

	NIST1				NIST3			
	215	216	217	218	215	216	217	218
A:1					0.191	0.225	0.63	0.63
A:2					0.216	0.274	0.59	0.68
B:1					0.217	0.255	0.58	0.63
B:2					0.243	0.246	0.59	0.67
C:1					0.261	0.269	0.59	0.68
C:2					0.207	0.211	0.58	0.65
n_x	0	0	0	0	6	6	6	6
$Mean_x$					0.222	0.247	0.59	0.66
SD_x					0.025	0.025	0.02	0.02
SD_{repx}					0.027	0.031	0.02	0.03
SD_{hetx}					0.016	0.006	0.01	0.01
SD_{NISTx}					0.031	0.032	0.02	0.03
CV_{NISTx}					14	13	3.7	4.3

	NIST			
n	6	6	6	6
Mean	0.03	0.02	0.017	0.02
SD_{rep}	0.01	0.01	0.002	0.01
SDhet	0.00	0.00	0.002	0.01
SD_{anl}				
SD_{NIST}				
CV_{NIST}				

	NIST			
	6	6	6	6
	0.222	0.247	0.59	0.66
	0.018	0.021	0.02	0.03
	0.016	0.006	0.01	0.01
	0.019	0.019	0.02	0.02
	0.031	0.029	0.03	0.04
	14	12	4.8	5.6

NIST3=a+b*Median

a: 0

b: 1.263 ±0.026

R^2: 0.985

RR	XXVI	XXVIII	XXX	XXXII
Serum	170	182	194	199
n_p		0	0	0
$Median_p$				
eSD_p				

<— Previous Results —>

XXVI	XXVIII	XXX	XXXII
170	182	194	199
16	16	23	22
0.190	0.215	0.50	0.57
0.061	0.051	0.13	0.13

	RRXXXVI			
	215	216	217	218
n_n	0	0	0	0
$Median_n$				
eSD_n				
P(n=p)				
P(n<p)				
SD_{labs}				
CV_{labs}				

<— Current Results —>

RRXXXVI			
215	216	217	218
23	23	23	23
0.186	0.191	0.46	0.53
0.047	0.043	0.13	0.12
0.98	0.87	0.93	0.88
0.88	0.75	0.55	0.86
0.035	0.033	0.13	0.12
19	17	28	22

NAV	0.03	0.02	0.017	0.02
NAU				
CV				

<— Assignments —>

0.204	0.219	0.52	0.60
0.047	0.043	0.13	0.12
23	20	25	21

Table 1
NIST Data and Calculations

"Lutein"

	NIST1				NIST3			
	215	216	217	218	215	216	217	218
A:1					0.111	0.044	0.076	0.082
A:2					0.107	0.042	0.077	0.083
B:1					0.102	0.041	0.079	0.082
B:2					0.108	0.043	0.071	0.080
C:1					0.105	0.038	0.073	0.076
C:2					0.112	0.042	0.068	0.076
n_x	0	0	0	0	6	6	6	6
$Mean_x$					0.108	0.042	0.074	0.080
SD_x					0.004	0.002	0.004	0.003
SD_{repx}					0.004	0.002	0.004	0.001
SD_{hetx}					0.002	0.002	0.003	0.003
SD_{NISTx}					0.005	0.002	0.005	0.003
CV_{NISTx}					4.4	5.8	6.5	4.1

	NIST				NIST3=a+b*Median
n	6	6	6	6	
Mean	0.108	0.042	0.074	0.080	a: 0.010 ±0.006
SD_{rep}	0.004	0.001	0.004	0.001	b: 0.917 ±0.082
SDhet	0.002	0.002	0.003	0.003	R^2: 0.968
SD_{anl}	0.004	0.004	0.004	0.004	
SD_{NIST}	0.006	0.005	0.007	0.005	
CV_{NIST}	5.7	11	9.1	6.6	

RR	XXVI	XXVIII	XXX	XXXII
Serum	170	182	194	199
n_p	4	7	14	11
$Median_p$	0.109	0.040	0.092	0.090
eSD_p		0.003	0.036	0.025

<— Previous Results —>

	RRXXXVI			
	215	216	217	218
n_n	12	12	12	12
$Median_n$	0.109	0.038	0.067	0.073
eSD_n	0.014	0.005	0.009	0.012
P(n=p)		0.83	0.72	0.74
P(n<p)		0.17	1.00	1.00
SD_{labs}	0.012	0.002	0.006	0.010
CV_{labs}	11	5.2	9.5	14

<— Current Results —>

NAV	0.108	0.040	0.070	0.076
NAU	0.014	0.005	0.009	0.012
CV	13	13	13	15

<—— Assignments ——>

"Zeaxanthin"

	NIST1				NIST3			
	215	216	217	218	215	216	217	218
A:1					0.058	0.035	0.047	0.057
A:2					0.053	0.031	0.048	0.056
B:1					0.046	0.029	0.050	0.055
B:2					0.052	0.030	0.049	0.053
C:1					0.050	0.027	0.042	0.051
C:2					0.055	0.032	0.028	0.052
n_x	0	0	0	0	6	6	6	6
$Mean_x$					0.052	0.030	0.044	0.054
SD_x					0.004	0.003	0.008	0.002
SD_{repx}					0.004	0.003	0.006	0.001
SD_{hetx}					0.003	0.002	0.008	0.002
SD_{NISTx}					0.005	0.003	0.010	0.003
CV_{NISTx}					9.0	11	22	4.9

	NIST				NIST3=a+b*Median
n	6	6	6	6	
Mean	0.052	0.030	0.044	0.054	a: -0.049 ±0.008
SD_{rep}	0.004	0.002	0.001	0.001	b: 2.956 ±0.255
SDhet	0.003	0.002	0.008	0.002	R^2: 0.985
SD_{anl}	0.004	0.004	0.004	0.004	
SD_{NIST}	0.006	0.005	0.009	0.005	
CV_{NIST}	12	16	20	8.9	

RR	XXVI	XXVIII	XXX	XXXII
Serum	170	182	194	199
n_p	3	3	6	7
$Median_p$	0.040	0.030	0.034	0.030
eSD_p			0.010	0.011

	RRXXXVI			
	215	216	217	218
n_n	8	8	8	8
$Median_n$	0.048	0.026	0.030	0.035
eSD_n	0.015	0.003	0.007	0.012
P(n=p)			0.77	0.96
P(n<p)			0.80	0.31
SD_{labs}	0.013	0	0	0.011
CV_{labs}	28	0	0	33

NAV	0.050	0.028	0.037	0.045
NAU	0.015	0.005	0.009	0.012
CV	30	17	24	28

Table 2
Summary of Assigned Values and Uncertainties

	215			216			217			218		
Analyte	NAV	NAU	CV	NAV	NAU	CV	NAV	NAU	CV	NAV	NAU	CV
Retinol	0.670	0.055	8.2	0.549	0.039	7.0	0.432	0.037	8.5	1.051	0.082	7.8
Retinyl Palmitate	0.271	0.062	23									
α-Tocopherol	6.04	0.44	7.2	5.60	0.37	6.6	7.22	0.67	9.3	7.09	0.56	7.9
γ-Tocopherol	2.04	0.36	18	2.41	0.33	14	3.00	0.36	12	2.36	0.35	15
δ-Tocopherol	0.12	0.21	190	0.16	0.17	100	0.11	0.16	140	0.13	0.15	120
Total β-Carotene	0.577	0.090	16	0.406	0.071	17	0.054	0.017	31	0.577	0.098	17
trans-β-Carotene	0.526	0.079	15	0.386	0.041	11	0.054	0.025	46	0.546	0.081	15
Total α-Carotene	0.025	0.012	50	0.019	0.004	23	0.013	0.007	55	0.029	0.015	53
trans-α-Carotene	0.03			0.02			0.017			0.02		
Total Lycopene	0.204	0.047	23	0.219	0.043	20	0.52	0.13	25	0.60	0.12	21
trans-Lycopene	0.125	0.026	21	0.147	0.013	9.0	0.300	0.015	5.0	0.362	0.075	21
β-Cryptoxanthin	0.054	0.016	29	0.037	0.012	32	0.038	0.014	37	0.050	0.019	37
"Lutein"	0.108	0.014	13	0.040	0.005	13	0.070	0.009	13	0.076	0.012	15
"Zeaxanthin"	0.050	0.015	30	0.028	0.005	17	0.037	0.009	24	0.045	0.012	28

Appendix C. "All-Lab Report" for RR36

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 XXXVI Laboratory Results

Values in µg/mL

	Total Retinol				Retinyl Palmitate				α-Tocopherol				γ-Tocopherol			
Lab	215	216	217	218	215	216	217	218	215	216	217	218	215	216	217	218
FSV-BA	0.647	0.541	0.433	1.057	0.283	0.071	0.059	0.075	6.23	5.72	7.28	7.21	1.62	1.96	2.45	1.85
FSV-BD	0.624	0.530	0.415	1.054					5.65	5.00	6.74	6.31				
FSV-BE	0.661	0.544	0.440	1.073					6.50	5.86	7.71	7.51	1.97	2.65	3.25	2.47
FSV-BF	0.630	0.505	0.439	0.959					6.26	5.45	6.77	7.01	1.83	2.33	2.78	2.19
FSV-BG	0.660	0.530	0.470	1.150	0.309	0.050	0.014	0.034	6.06	5.52	7.39	7.18				
FSV-BH	0.666	0.499	0.416	1.065	0.357	0.048	*nd*	*nd*	6.32	5.75	7.44	7.38	2.22	2.71	3.26	2.56
FSV-BI	0.591	0.501	0.396	0.981	0.352	0.043	*nd*	*nd*	5.85	5.27	7.01	6.94	1.90	2.35	2.88	2.24
FSV-BJ	0.629	0.522	0.418	1.021	0.313	*nd*	*nd*	*nd*	5.94	5.23	7.07	6.87	2.04	2.42	3.08	2.20
FSV-BK	0.657	0.584	0.462	1.194					5.89	5.26	6.90	6.92				
FSV-BL	0.687	0.544	0.458	1.088					5.94	5.43	7.32	7.15				
FSV-BM	0.688	0.530	0.410	0.950					5.90	5.70	8.60	8.80				
FSV-BN	0.740	0.621	0.509	1.240	0.520	0.083	*nd*	*nd*	6.63	6.10	7.95	7.64	2.42	3.25	3.62	2.78
FSV-BO	0.748	0.564	0.470	1.106					6.42	5.47	6.46	6.59				
FSV-BP	0.656	0.533	0.435	0.961					5.93	5.72	7.05	7.01				
FSV-BQ	0.670	0.580	0.440	1.030					4.90	3.90	5.40	5.50				
FSV-BR	0.740	0.580	0.430	1.060												
FSV-BS	0.630	0.460	0.400	0.810												
FSV-BT	0.547	0.537	0.422	1.051	0.286	0.065	0.013	0.032	5.60	5.71	7.21	7.14	1.80	2.66	3.18	2.45
FSV-BU	0.640	0.547	0.511	1.381					6.01	5.24	7.35	6.17	1.90	2.38	3.26	2.78
FSV-BW	0.750	0.640	0.510	1.290	0.360	0.043	*nd*	0.034	6.40	5.50	7.30	6.90	1.60	1.80	2.20	1.70
FSV-BX	0.653	0.522	0.447	1.028					5.87	5.25	6.66	6.52	1.92	2.36	2.86	2.16
FSV-BY	0.665	0.523	0.417	1.054	0.322	0.068	0.011	0.025	6.18	5.57	7.18	7.24	2.12	2.52	3.00	2.41
FSV-BZ									6.06	5.68	7.58	7.47	1.87	2.05	2.81	2.22
FSV-CA	0.597	0.476	0.351	0.890					5.18	4.91	5.85	6.21				
FSV-CB	0.615	0.534	0.412	1.059					5.79	5.17	6.60	6.75				
FSV-CC	0.722	0.556	0.434	1.072					5.04	4.57	5.74	5.74				
FSV-CD	0.657	0.541	0.425	1.090	0.249	0.078	0.034	0.083	8.14	7.42	9.68	9.09	2.75	3.33	4.11	3.09
FSV-CF	0.705	0.580	0.463	1.141					6.90	6.10	8.20	8.00				
FSV-CG	0.628	0.498	0.405	1.021					2.92	2.30	2.30	3.39	2.19	2.70	3.16	2.55
FSV-CH	0.550	0.450	0.340	0.820					5.97	5.09	6.22	6.24	2.10	2.50	2.90	2.32
FSV-CK	0.680	0.550	0.450	1.110					6.16	5.75	7.18	7.30	2.12	2.72	3.24	2.83
FSV-CM									5.30	6.20	7.70	6.50				
FSV-CN	0.689	0.544	0.453	1.009					6.50	5.42	7.74	6.92	1.79	2.19	2.81	2.01
FSV-CQ	0.577	0.454	0.389	0.931					6.11	5.03	7.03	7.28				
FSV-CR	0.710	0.590	0.470	1.190					6.00	5.60	7.00	7.00				
FSV-CS	0.678	0.562	0.424	1.120												
FSV-CT	0.693	0.573	0.416	1.119					6.25	5.66	7.43	7.34				
FSV-CU	0.602	0.508	0.409	0.995	0.253	0.063	0.047	0.089	5.94	5.20	6.44	6.76				
FSV-CX	0.700	0.560	0.480	1.170	0.290	0.030	0.050	0.040	6.30	5.53	7.20	7.05	2.24	2.72	3.54	2.66
FSV-DA	0.723	0.660	0.487	1.100	0.220	0.070	0.010	0.030	6.80	6.58	8.51	7.95	2.28	3.01	3.60	2.73
FSV-DB	0.735	0.610	0.475	1.155					6.43	6.03	7.72	7.60				
FSV-DJ	0.694	0.535	0.436	1.101					6.20	5.20	7.60	8.40				
FSV-DK	0.550	0.510	0.390	1.090	0.140	0.024	*nd*	0.012	5.59	5.29	6.46	6.67				
FSV-DM	0.630	0.536	0.425	1.074					5.46	5.27	6.61	7.04				
FSV-DP	0.684	0.545	0.448	1.093												
FSV-DS	0.680	0.520	0.350	0.880					8.78	7.89	7.79	8.66				
FSV-DU	1.079	0.619	0.481	1.272					11.74	6.48	9.07	8.52				
FSV-DX	0.612	0.526	0.402	1.019					5.96	5.76	7.25	7.18				
FSV-EA	0.570	0.500	0.400	0.940					5.10	4.80	6.10	6.10	2.50	3.10	3.60	3.00
FSV-EH	0.570	0.520	0.430	1.060					5.27	5.19	7.04	6.76	1.70	2.22	2.83	2.17
FSV-EK	0.578	0.504	0.407	0.994					5.34	5.22	6.18	6.43	1.49	2.05	2.43	1.89
FSV-EL	0.760	0.650	0.500	1.170												
n	50	50	50	50	14	13	8	10	47	47	47	47	23	23	23	23
Min	0.547	0.450	0.340	0.810	0.140	0.024	0.010	0.012	2.92	2.30	2.30	3.39	1.49	1.80	2.20	1.70
Median	0.661	0.537	0.432	1.063	0.300	0.063	0.024	0.034	6.00	5.47	7.18	7.01	1.97	2.50	3.08	2.41
Max	1.079	0.660	0.511	1.381	0.520	0.083	0.059	0.089	11.74	7.89	9.68	9.09	2.75	3.33	4.11	3.09
eSD	0.052	0.039	0.035	0.082	0.072	0.022	0.020	0.011	0.47	0.40	0.77	0.55	0.27	0.33	0.36	0.37
eCV	8	7	8	8	24	35	83	33	8	7	11	8	14	13	12	15
NISTa	0.686	0.568	0.442	1.034	0.247	*nd*	*nd*	*nd*	6.10	5.68	6.95	7.13	2.21	2.10	2.77	2.27
NISTb	0.682	0.556	0.436	1.051	0.237	*nd*	*nd*	*nd*	6.13	5.78	7.55	7.21	2.00	2.53	3.09	2.37
NAV	0.672	0.549	0.435	1.052	0.300	0.063	0.024	0.034	6.06	5.60	7.22	7.09	2.03	2.41	3.00	2.36
NAU	0.057	0.048	0.038	0.092	0.072	0.022	0.020	0.011	0.57	0.57	0.71	0.62	0.30	0.35	0.34	0.39

Round Robin XXXVI Laboratory Results

Values in µg/mL

	δ-Tocopherol				Total β-Carotene				trans-β-Carotene				Total cis-β-Carotene			
Lab	215	216	217	218	215	216	217	218	215	216	217	218	215	216	217	218
FSV-BA					0.600	0.442	0.067	0.604	0.572	0.423	0.062	0.577	*0.028*	*0.019*	*0.005*	*0.027*
FSV-BD					0.458	0.331	0.048	0.468								
FSV-BE					0.656	0.458	0.066	0.668								
FSV-BF					0.566	0.419	0.068	0.570								
FSV-BG					0.636		0.082									
FSV-BH					0.578	0.401	0.051	0.590	0.549	0.385	0.051	0.557	0.029	0.016	*nd*	0.033
FSV-BI					0.537	0.377	0.057	0.553								
FSV-BJ					0.662	0.473	0.063	0.637								
FSV-BK																
FSV-BL																
FSV-BM																
FSV-BN	0.118	0.183	0.082	0.108	0.619	0.462	0.054	0.644	0.590	0.440	0.054	0.610	0.029	0.022	*nd*	0.034
FSV-BO					0.406	0.298	0.052	0.404								
FSV-BP					0.385	0.257	0.044	0.407								
FSV-BQ																
FSV-BR																
FSV-BS					0.630	0.447	0.110	0.625	0.603	0.416	0.080	0.598	0.027	0.031	0.030	0.027
FSV-BT	0.706	0.605	0.538	0.521	0.469	0.376	0.049	0.541	0.444	0.362	0.048	0.509	0.025	0.014	0.001	0.032
FSV-BU					0.495	0.345	0.051	0.514								
FSV-BW					0.480	0.330	0.050	0.500								
FSV-BX					0.442	0.366	0.086	0.549								
FSV-BY	0.098	0.098	0.073	0.076	0.571	0.424	0.056	0.628	0.540	0.395	0.053	0.597	0.031	0.029	0.003	0.031
FSV-BZ					*0.459*	*0.366*	*0.003*	*0.488*	0.451	0.365	0.002	0.468	0.008	0.001	0.001	0.020
FSV-CA																
FSV-CB																
FSV-CC																
FSV-CD					>0.460	>0.315	>0.034	>0.398	0.460	0.315	0.034	0.398				
FSV-CF																
FSV-CG					0.673	0.471	0.061	0.671								
FSV-CH					0.526	0.337	0.045	0.484								
FSV-CK					0.600	0.410	0.060	0.590								
FSV-CM																
FSV-CN					>0.566	>0.365	>0.029	>0.467	0.566	0.365	0.029	0.467				
FSV-CQ					0.465	0.326	0.025	0.442								
FSV-CR																
FSV-CS																
FSV-CT					0.591	0.417	0.055	0.633								
FSV-CU					0.584	0.435	0.079	0.636	0.532	0.398	0.068	0.580	*0.052*	*0.037*	*0.011*	*0.056*
FSV-CX					0.680	0.480	0.070	0.780								
FSV-DA	0.183	0.203	0.142	0.135	0.488	0.555	0.110	0.735	0.431	0.503	0.088	0.667	0.057	0.052	0.022	0.068
FSV-DB					0.570	0.440	0.050	0.620								
FSV-DJ																
FSV-DK					0.510	0.350	0.040	0.500								
FSV-DM					0.551	0.412	0.058	0.578								
FSV-DP																
FSV-DS					0.410	0.380	0.100	0.540								
FSV-DU					>0.630	>0.390	*nd*	>0.650	0.630	0.390	*nd*	0.650				
FSV-DX					>0.456	>0.340	>0.041	>0.444	0.456	0.340	0.041	0.444				
FSV-EA					0.540	0.340	0.050	0.560								
FSV-EH	0.073	0.100	0.094	0.110	0.506	0.400	0.085	0.580	0.470	0.376	0.085	0.524	0.036	0.024	*nd*	0.056
FSV-EK					0.447	0.354	0.088	0.500								
FSV-EL																
n	5	5	5	5	33	32	33	32	14	14	13	14	10	10	7	10
Min	0.073	0.098	0.073	0.076	0.385	0.257	0.003	0.404	0.431	0.315	0.002	0.398	0.008	0.001	0.001	0.020
Median	0.118	0.183	0.094	0.110	0.540	0.401	0.057	0.574	0.536	0.388	0.053	0.567	0.029	0.023	0.005	0.033
Max	0.706	0.605	0.538	0.521	0.680	0.555	0.110	0.780	0.630	0.503	0.088	0.667	0.057	0.052	0.030	0.068
eSD	0.067	0.123	0.031	0.037	0.089	0.069	0.013	0.091	0.099	0.035	0.022	0.075	0.004	0.011	0.006	0.008
eCV	57	67	33	34	16	17	23	16	18	9	42	13	15	48	119	25
NISTa					0.659	0.425	*nd*	0.612	0.526	0.360	*nd*	0.513	*0.133*	*0.065*	*nd*	*0.099*
NISTb	0.080	0.130	0.117	0.132	0.547	0.419	0.052	0.567	0.514	0.395	0.054	0.516	*0.033*	*0.024*	*-0.003*	*0.051*
NAV	0.099	0.157	0.105	0.121	0.573	0.411	0.056	0.582	0.529	0.382	0.052	0.541	0.057	0.034	0.004	0.054
NAU	0.069	0.085	0.047	0.025	0.108	0.070	0.015	0.097	0.085	0.042	0.020	0.096	0.087	0.046	0.011	0.057

Round Robin XXXVI Laboratory Results

Values in µg/mL

	Total α-Carotene				trans-α-Carotene				Total Lycopene				trans-Lycopene				β-Cryptoxanthin			
Lab	215	216	217	218	215	216	217	218	215	216	217	218	215	216	217	218	215	216	217	218
FSV-BA	0.031	0.014	0.012	0.019									0.135	0.152	0.304	0.37	0.074	0.053	0.059	0.067
FSV-BD									0.144	0.155	0.36	0.44					0.045	0.030	0.034	0.040
FSV-BE																				
FSV-BF	0.029	0.018	0.022	0.014					0.218	0.227	0.51	0.64								
FSV-BG	0.026	0.017	0.039	0.037					0.213	0.179	0.63	0.65								
FSV-BH	0.025	0.014	0.010	0.017					0.228	0.235	0.57	0.66					0.086	0.053	0.065	0.078
FSV-BI	0.022	0.012	0.011	0.014					0.120	0.130	0.31	0.35					0.070	0.044	0.043	0.055
FSV-BJ	0.035	0.026	0.020	0.026					0.124	0.132	0.28	0.33								
FSV-BK																				
FSV-BL																				
FSV-BM																				
FSV-BN	0.026	0.016	0.013	0.015					0.267	0.276	0.63	0.71	0.135	0.159	0.324	0.39	0.056	0.039	0.041	0.045
FSV-BO	0.019	0.011	0.009	0.012					0.279	0.229	0.70	0.88					0.058	0.034	0.040	0.050
FSV-BP	0.019	0.015	0.013	0.029					0.175	0.188	0.39	0.50					0.071	0.050	0.059	0.080
FSV-BQ																				
FSV-BR																				
FSV-BS	0.034	0.023	0.020	0.032					0.161	0.162	0.37	0.44					0.054	0.034	0.033	0.042
FSV-BT	0.019	0.013	0.016	0.018					0.154	0.178	0.42	0.50	0.128	0.148	0.335	0.42	0.063	0.048	0.051	0.063
FSV-BU																				
FSV-BW	0.030	0.010	0.010	0.020					0.150	0.150	0.40	0.48								
FSV-BX	0.021	0.017	0.016	0.017					0.129	0.327	0.64	0.74					0.033	0.033	0.051	0.055
FSV-BY	0.026	0.019	0.023	0.032					0.204	0.249	0.50	0.62	0.134	0.177	0.325	0.40	0.070	0.050	0.056	0.067
FSV-BZ	0.022	0.019	0.014	0.032									0.019	0.157	0.255	0.55				
FSV-CA																				
FSV-CB																				
FSV-CC																				
FSV-CD	0.015	0.010	0.006	0.007					0.190	0.191	0.44	0.48					0.049	0.035	0.030	0.039
FSV-CF																				
FSV-CG	0.035	0.027	0.024	0.031					0.170	0.174	0.42	0.48					0.048	0.031	0.037	0.042
FSV-CH	0.017	0.009	0.008	0.009					0.204	0.189	0.46	0.53								
FSV-CK					0.03	0.02	0.02	0.03	0.175	0.177	0.40	0.46					0.064	0.041	0.044	0.058
FSV-CM																				
FSV-CN	*nd*	*nd*	*nd*	*nd*					0.164	0.165	0.46	0.51					0.048	0.024	0.027	0.031
FSV-CQ																				
FSV-CR																				
FSV-CS																				
FSV-CT																				
FSV-CU																				
FSV-CX	0.010	<0.01	0.010	<0.01					0.210	0.210	0.53	0.62					0.060	0.040	0.040	0.050
FSV-DA	0.015	0.015	0.018	0.021					0.118	0.213	0.59	0.58	0.071	0.130	0.320	0.33	0.059	0.037	0.042	0.056
FSV-DB	*nq*	*nq*	*nq*	*nq*					0.220	0.250	0.57	0.65					0.084	0.055	0.065	0.076
FSV-DJ																				
FSV-DK	0.015	*nd*	*nd*	0.003																
FSV-DM	0.031	0.028	0.031	0.026					0.217	0.204	0.45	0.62								
FSV-DP																				
FSV-DS																				
FSV-DU																				
FSV-DX													0.188	0.210	0.448	0.55				
FSV-EA	0.020	0.021	*nd*	*nd*																
FSV-EH	0.010	*nd*	*nd*	0.010					0.203	0.251	0.72	0.82	0.110	0.157	0.453	0.52	0.048	0.032	0.039	0.045
FSV-EK									0.186	0.191	0.33	0.43								
FSV-EL																				
n	24	21	21	22	1	1	1	1	25	25	25	25	8	8	8	8	19	19	19	19
Min	0.010	0.009	0.006	0.003					0.118	0.130	0.28	0.33	0.019	0.130	0.255	0.33	0.033	0.024	0.027	0.031
Median	0.022	0.016	0.014	0.019	0.031	0.018	0.015	0.025	0.186	0.191	0.46	0.53	0.131	0.157	0.325	0.41	0.059	0.039	0.042	0.055
Max	0.035	0.028	0.039	0.037					0.279	0.327	0.72	0.88	0.188	0.210	0.453	0.55	0.086	0.055	0.065	0.080
eSD	0.009	0.004	0.006	0.011					0.046	0.043	0.12	0.13	0.019	0.010	0.023	0.09	0.016	0.010	0.013	0.018
eCV	40	28	42	60					25	23	27	25	14	6	7	21	28	27	31	32
NISTa					*nd*	*nd*	*nd*	*nd*												
NISTb	0.028	0.022	0.013	0.039	0.027	0.020	0.017	0.024	0.222	0.247	0.59	0.66	0.116	0.137	0.275	0.32	0.049	0.034	0.034	0.044
NAV	0.025	0.019	0.013	0.029					0.204	0.219	0.52	0.60	0.124	0.147	0.300	0.37	0.054	0.037	0.038	0.050
NAU	0.012	0.007	0.007	0.022					0.055	0.063	0.16	0.16	0.029	0.035	0.075	0.12	0.018	0.012	0.013	0.018

Round Robin XXXVI Laboratory Results

Values in µg/mL

	α-Cryptoxanthin				Lutein				Zeaxanthin				Lutein&Zeaxanthin			
Lab	215	216	217	218	215	216	217	218	215	216	217	218	215	216	217	218
FSV-BA													0.202	0.098	0.143	0.154
FSV-BD					0.106	0.037	0.058	0.064	0.042	0.021	0.021	0.024	*0.148*	*0.058*	*0.079*	*0.088*
FSV-BE																
FSV-BF																
FSV-BG																
FSV-BH					0.108	0.039	0.057	0.070	0.049	0.027	0.020	0.022	*0.157*	*0.066*	*0.077*	*0.092*
FSV-BI					0.106	0.037	0.067	0.069	0.047	0.023	0.028	0.035	0.153	0.067	0.098	0.106
FSV-BJ																
FSV-BK																
FSV-BL																
FSV-BM																
FSV-BN	0.028	0.010	0.017	0.016	0.111	0.037	0.068	0.075	0.073	0.030	0.043	0.047	0.184	0.067	0.111	0.122
FSV-BO													0.102	0.044	0.071	0.073
FSV-BP																
FSV-BQ																
FSV-BR																
FSV-BS					0.110	0.030	0.075	0.114								
FSV-BT	0.033	0.020	0.036	0.033	0.136	0.064	0.097	0.090	0.016	0.027	0.032	0.029	0.152	0.091	0.129	0.119
FSV-BU																
FSV-BW																
FSV-BX					0.161	0.027	0.064	0.076	0.095	0.026	0.026	0.044				
FSV-BY					0.108	0.040	0.066	0.089	0.035	0.025	0.031	0.035	*0.143*	*0.065*	*0.097*	*0.124*
FSV-BZ					0.086	0.047	0.059	0.061								
FSV-CA																
FSV-CB																
FSV-CC																
FSV-CD													0.230	0.102	0.147	0.145
FSV-CF																
FSV-CG													0.127	0.074	0.103	0.109
FSV-CH																
FSV-CK	0.043	0.026	0.044	0.051	0.184	0.088	0.132	0.138								
FSV-CM																
FSV-CN					0.124	0.049	0.075	0.066								
FSV-CQ																
FSV-CR																
FSV-CS																
FSV-CT					0.099	0.035	0.065	0.070								
FSV-CU																
FSV-CX													0.160	0.080	0.110	0.120
FSV-DA					0.126	0.042	0.078	0.082	0.056	0.035	0.041	0.052	0.182	0.077	0.119	0.134
FSV-DB													0.140	0.070	0.096	0.100
FSV-DJ																
FSV-DK																
FSV-DM													0.139	0.061	0.095	0.099
FSV-DP																
FSV-DS																
FSV-DU																
FSV-DX													0.158	0.060	0.092	0.104
FSV-EA																
FSV-EH					0.090	0.046	0.064	0.073	0.051	0.025	0.028	0.034	*0.142*	*0.071*	*0.092*	*0.107*
FSV-EK													0.153	0.087	0.110	0.116
FSV-EL																
n	3	3	3	3	14	14	14	14	9	9	9	9	17	17	17	17
Min	0.028	0.010	0.017	0.016	0.086	0.027	0.057	0.061	0.016	0.021	0.020	0.022	0.102	0.044	0.071	0.073
Median	0.033	0.020	0.036	0.033	0.109	0.040	0.067	0.074	0.049	0.026	0.028	0.035	0.153	0.070	0.098	0.109
Max	0.043	0.026	0.044	0.051	0.184	0.088	0.132	0.138	0.095	0.035	0.043	0.052	0.230	0.102	0.147	0.154
eSD					0.019	0.008	0.012	0.012	0.010	0.003	0.006	0.013	0.017	0.013	0.018	0.016
eCV					17	21	18	17	21	10	21	37	11	19	18	15
NISTa																
NISTb					0.108	0.042	0.074	0.080	0.052	0.030	0.044	0.054	*0.160*	*0.072*	*0.118*	*0.134*
NAV					0.108	0.041	0.070	0.077	0.051	0.028	0.036	0.045	0.156	0.071	0.108	0.121
NAU					0.024	0.009	0.016	0.017	0.014	0.008	0.015	0.017	0.033	0.015	0.025	0.029

Round Robin XXXVI Laboratory Results

Analytes Reported By One Laboratory

Values in µg/mL

Analyte	Code	215	216	217	218
cis-(Lutein&Zeaxanthin)	FSV-BT	0.058	0.035	0.097	0.099
Coenzyme Q10	FSV-CH	0.340	0.343	0.566	0.419
Canthaxanthin	FSV-CD	*nd*	*nd*	0.053	0.050
Total Carotenoids	FSV-BT	1.031	0.813	0.916	1.485

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 "Report of (Meta)Analysis."
nd	Not detected (i.e., no detectable peak for analyte)
nq	Detected but not quantitatively determined
>x	Concentration greater than or equal to x
<x	Concentration at or below the limit of detection, x
italics	Not explicitly reported but calculated by NIST from reported values

Round Robin XXXVI Laboratory Results

Comparability Summary

Lab	R	aT	gT	bC	tbC
FSV-BA	1	1	1	1	1
FSV-BD	1	2	1	1	
FSV-BE	1	1		2	
FSV-BF	2	1			
FSV-BG	2	1		2	
FSV-BH	2	1	1	1	
FSV-BI	2	1		2	
FSV-BJ	1	1	1	1	
FSV-BK	2	1	2	1	1
FSV-BL	1	1	1	1	1
FSV-BM	2	3	2	1	
FSV-BN	2	2	1	2	
FSV-BO	2	2	1	4	3
FSV-BP	1	1			
FSV-BQ	1	3			
FSV-BR	2		1	1	
FSV-BS	3				
FSV-BT	3	1		2	1
FSV-BU	4	2	1	2	
FSV-BW	3	1		1	
FSV-BX	1	1	2	2	
FSV-BY	1	1	2	2	
FSV-BZ		1			
FSV-CA	3	2		3	
FSV-CB	1	1	3	1	2
FSV-CC	1	3		4	2
FSV-CD	1	4		1	
FSV-CF	1	2		2	
FSV-CG	2	4		3	
FSV-CH	3	2	1	1	1
FSV-CK	1	1			
FSV-CM		2			
FSV-CN	1	1			
FSV-CQ	2	1	1	1	
FSV-CR	2	1			
FSV-CS	1				
FSV-CT	1	1			1
FSV-CU	2	2	2	3	
FSV-CX	2	1	3		2
FSV-DA	3	2		2	
FSV-DB	2	1		1	
FSV-DJ	1	3	2	1	
FSV-DK	3	2	2	4	3
FSV-DM	1	2			
FSV-DP	1				
FSV-DS	3	4	2	2	2
FSV-DU	4	4	1		2
FSV-DX	2	1			
FSV-EA	2	2	3	2	
FSV-EH	2	2			
FSV-EK	2	2			
FSV-EL	3				2
NISTa	1	1	1	1	1
NISTb	1	1	1	1	1
n	46	44	22	32	13

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 (serum heterogeniety and iter- and intra-laboratory) measurement standard deviation.
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

For details on the NIST Assigned quantities, see this Round Robin's "Report of (Meta)Analysis."

StS	% Observed					Expected
1	43	55	55	47	46	68.2 %
2	41	34	36	38	46	27.3 %
3	20	9	14	9	15	4.3 %
4	4	9	0	9	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 D. Representative "Individualized Report" for RR36

Each participant in RR36 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
- Zeaxanthin
- Lutein & Zeaxanthin

The following 8 pages are the "Individualized Report" for the analytes evaluated by participant FSV-BA.

Individualized Round Robin XXXVI Report to: FSV-BA

Your Data, NIST Assigned Values, and %Differences

	Serum 215				Serum 216				Serum 217				Serum 218			
Analyte	You	NAV	%Δ	n	You	NAV	%Δ	n	You	NAV	%Δ	n	You	NAV	%Δ	n
Retinol	.65	.67	-3	44	.54	.55	-1	44	.43	.43	0	44	1.06	1.05	1	44
Retinyl Palmitate	.28	.27	4	14	.07			13	.06			8	.08			10
α-Tocopherol	6.23	6.05	3	42	5.72	5.61	2	42	7.28	7.22	1	42	7.21	7.09	2	42
γ-Tocopherol	1.62	2.05	-21	20	1.96	2.41	-19	20	2.45	3.02	-19	20	1.85	2.38	-22	20
Total β-Carotene	.60	.58	3	30	.44	.41	8	29	.07	.05	24	30	.60	.58	4	29
trans-β-Carotene	.57	.53	9	11	.42	.39	10	11	.06	.05	15	11	.58	.55	6	11
Total α-Carotene	.03	.03	19	22	.01	.02	-26	20	.01	.01	-8	20	.02	.03	-34	20
trans-Lycopene	.14	.13	8	7	.15	.15	3	7	.30	.30	1	7	.37	.36	3	7
β-Cryptoxanthin	.07	.05	37	16	.05	.04	43	16	.06	.04	55	16	.07	.05	40	16
"Lutein&Zeaxanthin"	.20			16	.10			16	.14			16	.15			16

D2

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

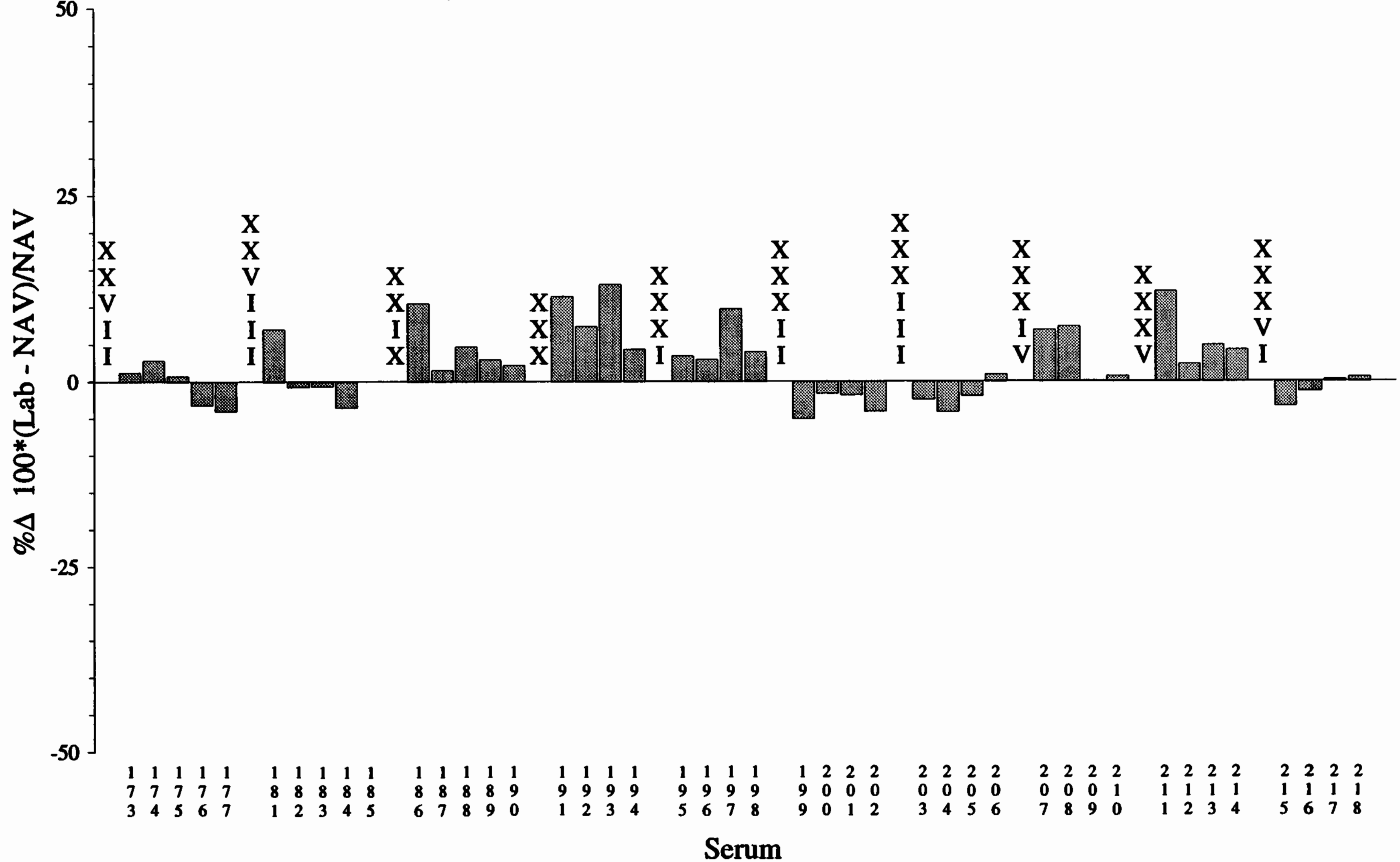
n : Number of non-NIST laboratories reporting quantitative values for this analyte in this serum

Please check our recorded values against your records.

Send corrections to: NNMMQAP 222/B208, NIST, Gaithersburg, MD 20899; fax 301-977-0685; email David.Duewer@NIST.gov

Individualized Round Robin XXXVI Report to: FSV-BA

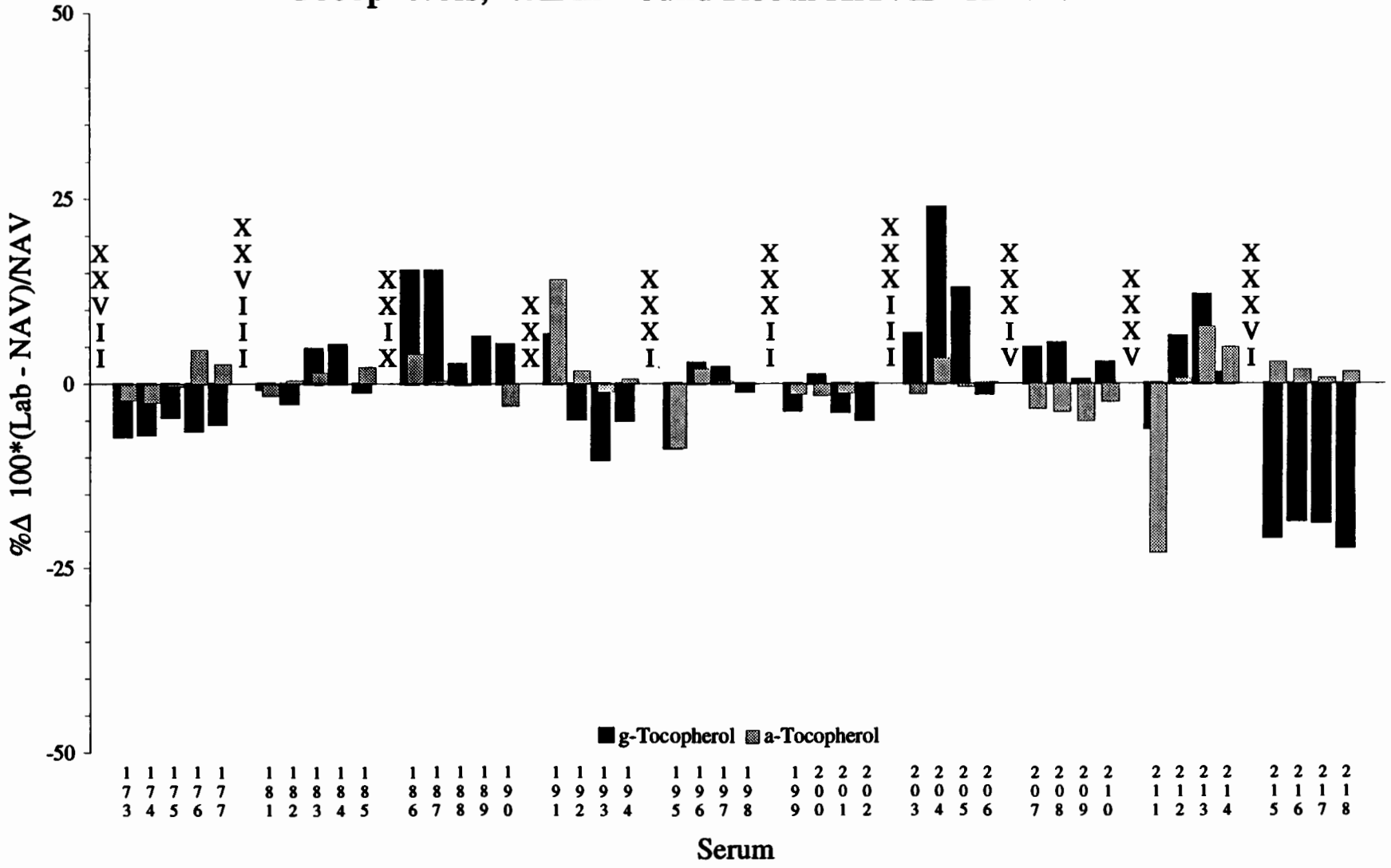
Retinol, %Δ in Round Robin XXVII - XXXVI



D3

Individualized Round Robin XXXVI Report to: FSV-BA

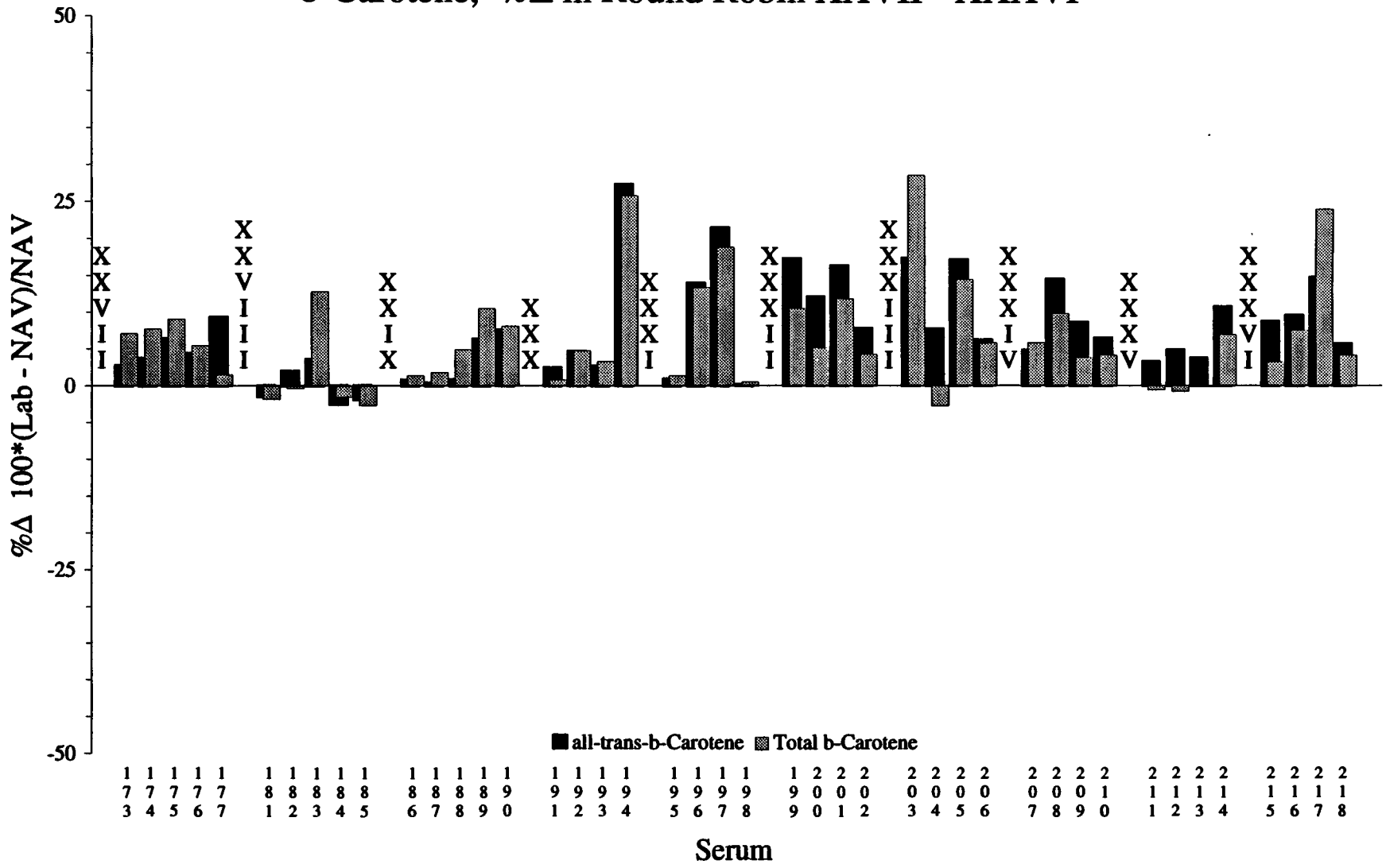
Tocopherols, %Δ in Round Robin XXVII - XXXVI



D4

Individualized Round Robin XXXVI Report to: FSV-BA

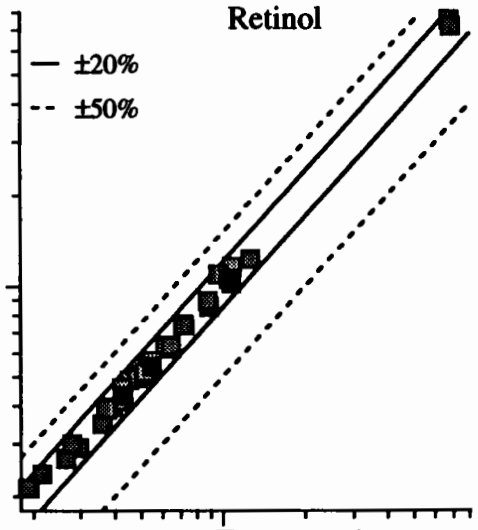
b-Carotene, %Δ in Round Robin XXVII - XXXVI

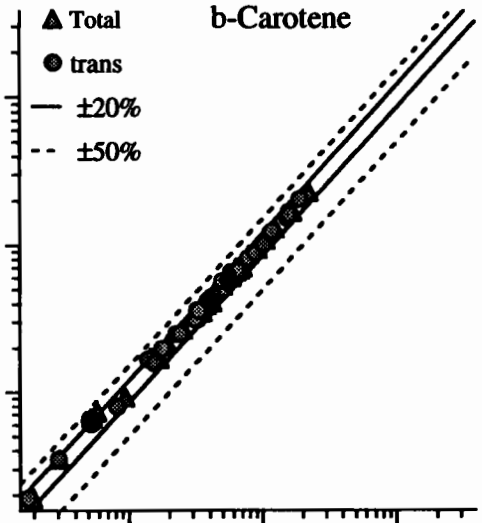


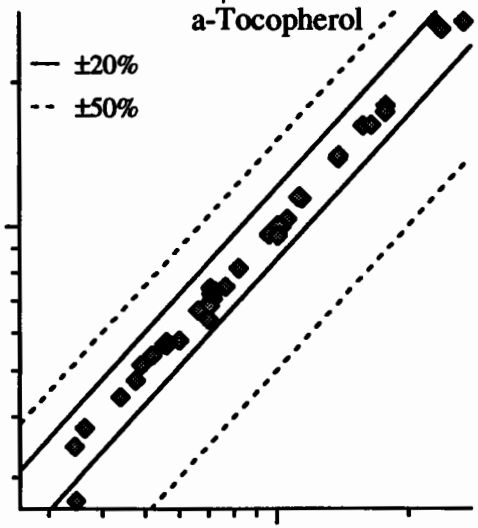
D5

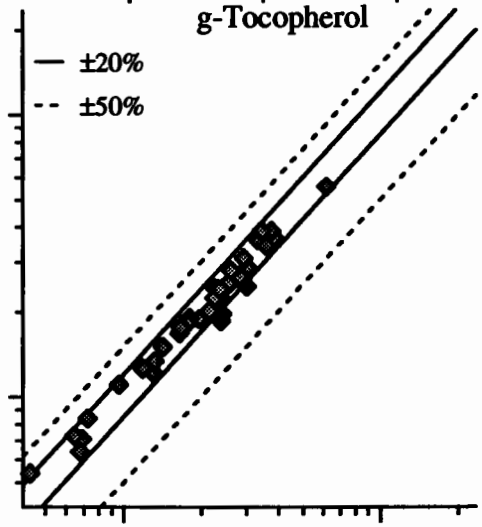
Individualized Round Robin XXXVI Report to: FSV-BA

NIST Assigned Values Vs Laboratory Values









Legend

Shaded Symbols: Round Robin XXVII-XXXV
Black Symbols: Round Robin XXXVI

Ordinate: NIST Assigned Value
Abscissa: Your reported concentration

Interpretation

Adequately intercomparable data are within ±20% lines. If you have data scattered outside ±50% lines, your measurement system is not consistent with those of most participating laboratories. If your data are systematically higher or lower than the NAV, your system may be consistent but your results are biased.

If your data show increased scatter at low concentrations, your "limit of quantification" may not be what you think it is.

If there are one or two "wild" outliers, they might be calculation or transcription errors. We would appreciate hearing from you about any such problems.

D6

Individualized Round Robin XXXVI Report to: FSV-BA

Accuracy/Precision Summary

	Ret		aToc		gToc		Total		trans	
	mΔ	vΔ	mΔ	vΔ	mΔ	vΔ	mΔ	vΔ	mΔ	vΔ
XXVII	-1	3	0	3	-6	1	6	3	5	3
XXVIII	0	4	1	2	1	4	1	7	0	3
XXIX	4	4	0	3	9	6	5	4	3	3
XXX	9	4	4	7	-3	7	9	12	9	12
XXXI	5	3	-2	5	-1	5	9	9	9	10
XXXII	-3	2	-1	1	-3	3	8	4	13	4
XXXIII	-2	2	0	2	11	11	12	13	12	6
XXXIV	4	4	-4	1	3	2	6	3	9	4
XXXV	6	4	-2	14	3	8	1	4	6	3
XXXVI	-1	2	2	1	-20	2	10	10	10	4

Legend

Ret	Retinol
aToc	a-Tocopherol
gToc	g-Tocopherol
Total	Total b-Carotene
trans	all-trans-b-Carotene
mΔ	Mean difference, the average %Δ for all sera of a given RR, where %Δ = 100(Your value - NAV) / NAV
vΔ	Difference variability, one standard deviation of %Δ for all sera of a RR
NAV	NIST Assigned Value, our best estimate of analyte concentration... NAV = (NIST's average-of-averages + Round Robin median) / 2

(Traditional) Performance Criteria

The absolute value of %Δ of every measurement has traditionally been evaluated as follows...

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

More representative criteria need to be established, factoring in each serum's analyte level and the analyte distribution in adult human populations. Stay tuned, we're working on it...

Interpretation

Accuracy and precision are separate but kindred aspects of measurement comparison. We estimate accuracy as mΔ, the average %Δ, and precision as vΔ, the standard deviation of %Δ, for all sera of a Round Robin.

It's best to be accurate and precise (small mΔ, small vΔ)!

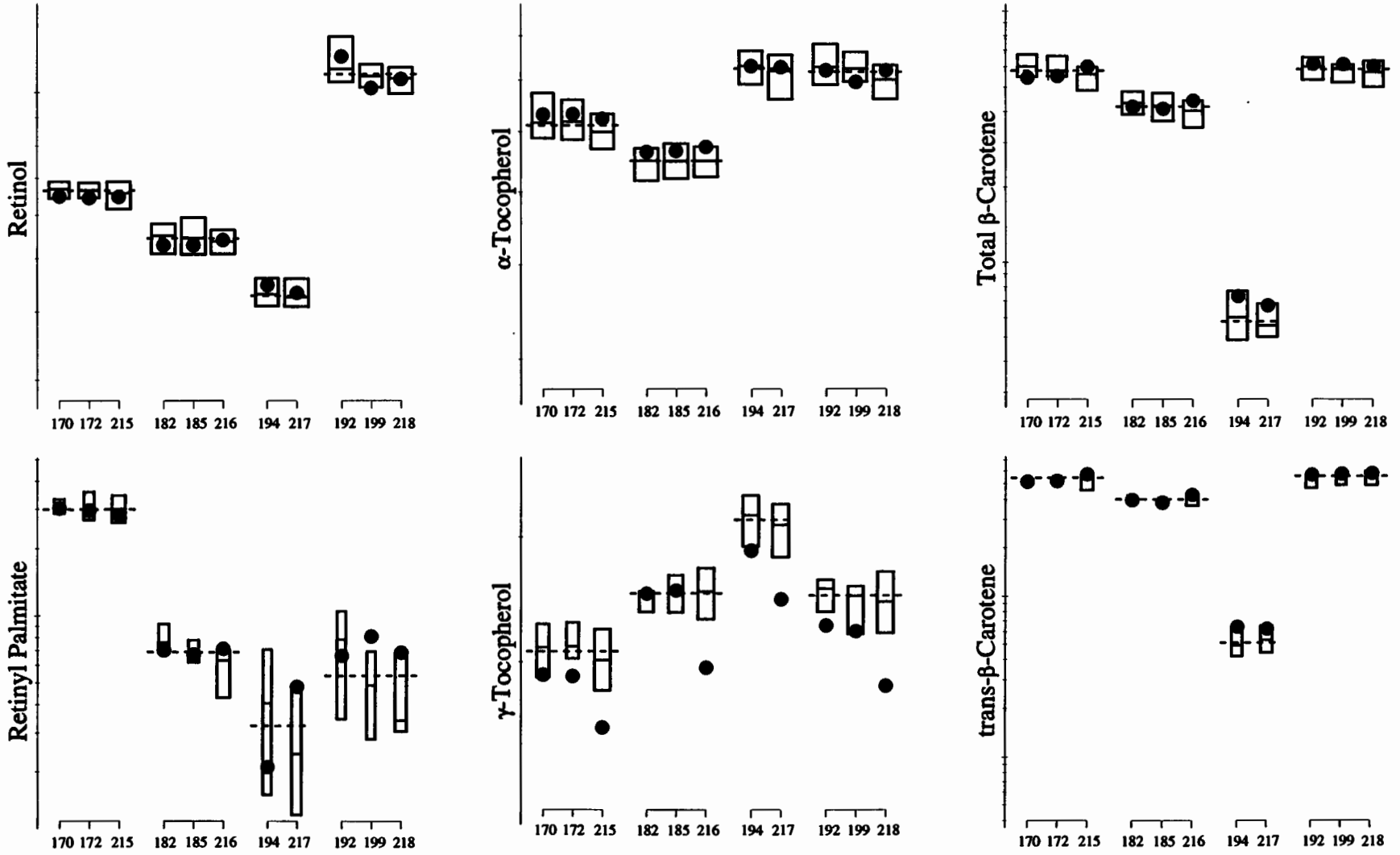
Good precision (small vΔ) with poor accuracy (large mΔ) is better than the converse: at least such values are internally consistent and may be relatable to others' values once the relative biases have been determined.

Poor precision (large vΔ) suggests that your measurement system is not in adequate control for the analyte levels examined.

D7

Individualized Round Robin XXXVI Report to: FSV-BA

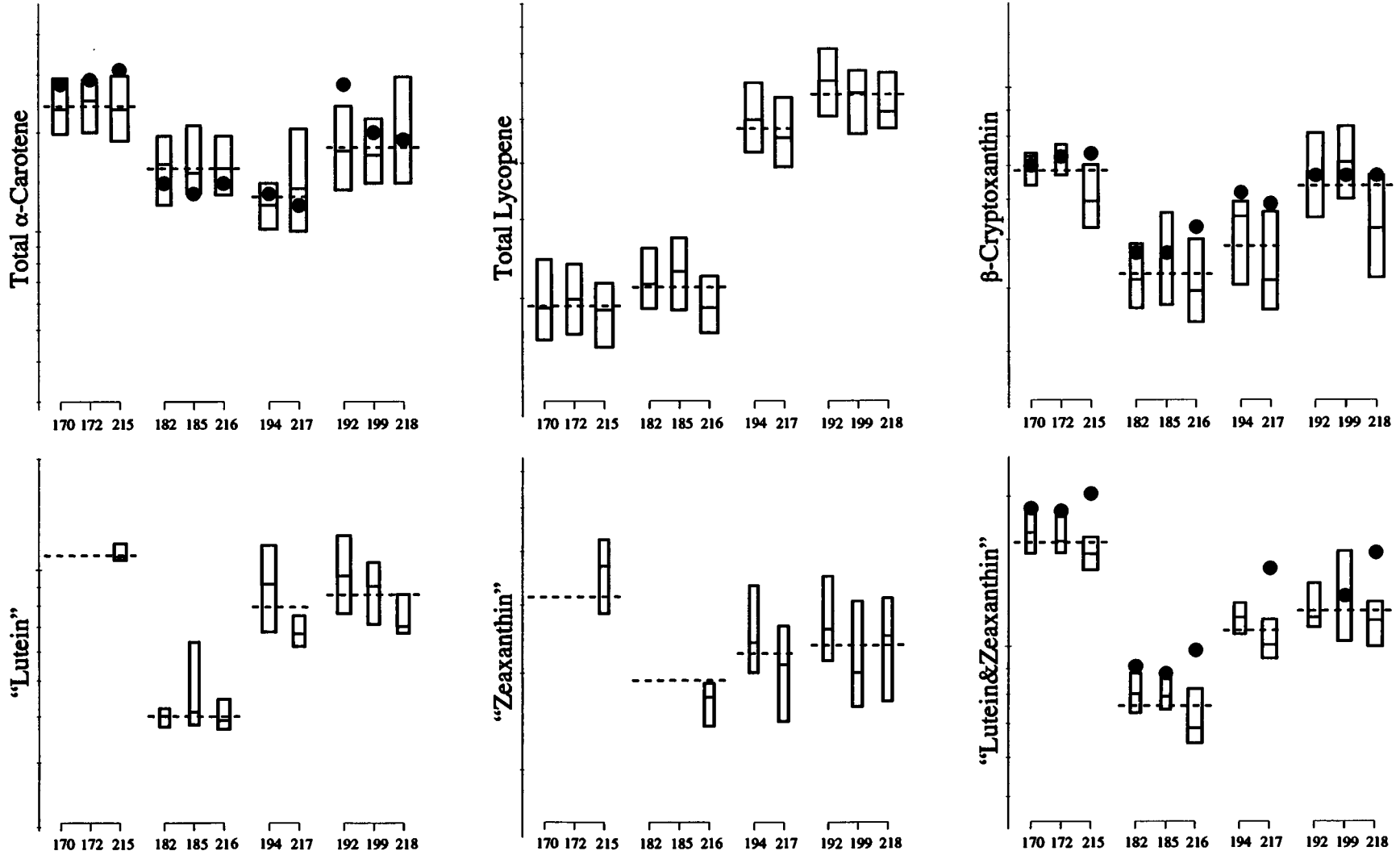
Comparisons to Prior Analyses



D8

Individualized Round Robin XXXVI Report to: FSV-BA

Comparisons to Prior Analyses (Continued)



D9

Appendix E. Shipping Package Inserts for RR37

The following two items were included in each package shipped to an RR37 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 15, 1996

Dear Colleague:

Enclosed is the set of samples for round robin exercise (Round Robin XXXVII) . You will find one vial 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 detection limit, please indicate this result on the form by using ND *(Not Detected)*. For analytes not measured, please leave a blank. Results are due to NIST by June 14. 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 26.

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 which 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 HPLC systems creating analytical problems.) Pipette a known volume of serum from the vial for analysis since 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; β-carotene, 2560 at 450 nm; lycopene (in hexane), 3450 at 472 nm.

Please mail or FAX your results for Round Robin XXXVII 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: jeanice.brown-thomas@nist.gov; or mail/FAX queries to the above address.

Sincerely,

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

cc: W. May
S. Wise

NIST/NCI

Micronutrients Measurement Quality Assurance Program

Round Robin **XXXVII** Results from Laboratory #_____

	Serum				
Analyte	219	220	221	222	Units*
retinol					
retinyl palmitate					
α-tocopherol					
γ-tocopherol					
δ-tocopherol					
total β-carotene					
trans-β-carotene					
total cis-β-carotene					
total 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 14, 1996

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

Appendix F. Final Report for RR37

The following 15 pages are the final report for RR37 as provided to all participants:

- Cover letter
- A discussion entitled “Lies, Damn Lies, and Statistics” 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
- A “Report of (Meta)Analysis” that details the analysis of NIST measurements

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

July 25, 1996

Dear Colleague:

Enclosed is the summary report of the results for Round Robin XXXVII (Sera 219-222). 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 round robin exercise.

There were three new sera (Serum 219-221) in this round robin exercise. Serum 222 was previously distributed as Serum 181 in Round Robin XXVIII. The new sera contained augmented levels of retinol, retinyl palmitate, and/or δ-, γ-, α-tocopherol. Specifically, the levels of δ- and γ-tocopherol and β-carotene were augmented in Serum 219 through the use of a commercial dietary supplement. The levels of δ-tocopherol, α-carotene, and lutein were augmented in Serum 220. Serum 221 contained augmented levels of α-tocopherol and retinyl palmitate.

In this round robin exercise, the average estimated coefficient of variation (eCV) is about 9% for retinol and α- and γ-tocopherol. This is comparable to the overall laboratory performance (approximately 10%) for these analytes over the past two years. The interlaboratory variation for total β-carotene is about 13% for this round robin, as compared to the average eCV of about 19% for Round Robin XXXVI, and 21% over the past two years. In previous exercises where the levels of β-carotene were significantly lower (≤100 ng/mL), a higher variation was obtained due to the difficulty of making measurements at levels that appear to be at or below the limit of quantification for most laboratories. In this exercise, the variation for β-carotene in the sera with augmented levels of β-carotene appear to be similar to that in the low level sera. The same observations hold true for retinyl palmitate. These observations will be further evaluated in future studies.

Approximately 10% of the laboratories reported values for δ-tocopherol with an eCV of about 18%. The eCVs for total α-carotene and lutein were 34% and 21%, respectively. Values for two additional analytes (phytofluene and α-cryptoxanthin) were reported in this study. We continue to encourage laboratories to report values for as many quantifiable analytes as possible.

Data for evaluating laboratory performance in Round Robin XXXVII are provided in the comparability summary on page 6 of the 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 Round Robin XXXVIII and the food Round Robin 5 (coordinated by Dr. Katherine Sharpless at 301/975-3121) were shipped during mid-July. Results for both round robin exercises are due September 20; feedback to labs will be provided around October 25.

Based on the positive feedback that we received at the NIST/NCI Micronutrients Measurement Quality Assurance (QA) Workshop held this past April, we will try to continue to hold our workshops in conjunction with national clinical meetings. We are currently looking into hosting our next QA workshop at the American Association for Clinical Chemistry meeting, which will be held in Atlanta, Georgia during July 20-24, 1997. 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 25, 1996.** 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.

Please complete the enclosed tutorial registration form and mail/fax it to: NIST/NCI Micronutrients Measurement Quality Assurance Program, NIST, Chem B208, Gaithersburg, MD 20899; FAX#: 301/977-0685 by **October 4, 1996**.

If you have questions, please contact me at 301/975-3120; FAX: 301/977-0685; e-mail: jeanice.brown-thomas@nist.gov.

Sincerely,

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

cc: W. May
S. Wise

"Lies, Damned Lies, and Statistics"

Mark Twain

The attached N^2M^2QAP Round Robin XXXVII Report includes the "All Lab" listing of everyone's results, your "Individualized" results, and our "(Meta)Analysis" of value and uncertainty assignments.

The "All Lab" report has the following elements:

Page	Contents
1-4	A listing of all results for analytes reported by at least two laboratories, plus essential summary statistics.
5top	A list of results for the four analytes reported by only one laboratory.
5bot	A Legend for the above two lists.
6	The "Measurement Comparability Summary" (or "Score card")

Your "Individualized" report has the following elements:

Page	Contents
1	Your values, our assigned values, and the %bias between you and us
2-4	%Bias barchart for retinol, α- and γ-tocopherol, and total and *trans*-β-carotene for your last 3 years' results
5	Our assigned value vs. your value scatterplots for retinol, α- and γ-tocopherol, and total and *trans*-β-carotene, again for your last 3 years' results
6	Accuracy/Precision Summary, yet again for your last 3 years' results
7&8	"Comparisons" plots for retinol, retinyl palmitate, α- and γ-tocopherol, total and *trans*-β-carotene, total α-carotene, total lycopene, β-cryptoxanthin, lutein, zeaxanthin, and lutein & zeaxanthin

The "Comparisons" plots on pages 7 and 8 show your individual results relative to a box-plot summary of the group's results. Serum 222 was distributed as #181 in RR XXVIII. The horizontal line on the left side of each plot is the average of the RR XXVIII and XXXVII interlaboratory medians.

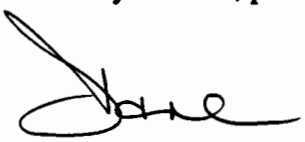
Sera 219, 220, and 221 are experiments in analyte augmentation. They started life as the same (rather boring) low-carotenoid serum pool. All three were augmented to various levels of retinol, retinyl palmitate, and/or α-, γ-, and δ-tocopherol. Serum 219 was also augmented with an extract *very* rich in β-carotene and Serum 220 with an extract rich in α-carotene and lutein. None of the analytes in any of these sera showed "excess" heterogeneity. Only one of you commented on the presence of "odd" extra peaks. Given better characterized sources for the various analytes, we believe we can now (affordably) create more interesting and analytically challenging samples.

Retinyl palmitate remains a difficult analyte: both the very high 1.5 µg/mL level in serum 221 and the reliably detected 0.10 µg/mL in serum 222 had CVs of about 20%. The low levels (0.03-0.04 µg/mL) levels in sera 219 and 220 are below the group's limit of reliable detection, let alone quantification.

All of you recognized the exceptionally high β-carotene level of serum 219, with most of you quite close to the 3.7 µg/mL median. Several of you commented that this was above the linear part of your calibration. However, there were a few values much above the median that were reported without comment. Because the β-carotene level for serum 219 was so *very* high with this serum, we have excluded it from the "Score Card" calculations. We encourage you to report values as "greater-than-or-equal to (≥)" when you realize that you are above your calibration limits: "3.7" and "≥ 3" are more obviously related than are "3.7" and "7.9". In the future, only labs that appropriately flag their suspect data and/or note the problem in the "Comments" space will be excused from strict application of the Score Card's rules!

The high δ-tocopherol levels in sera 219 and 220 did not adversely affect quantification of α- and/or γ-tocopherol. Excellent! We are a bit surprised that none of you commented on the unusual tocopherol patterns... are such oddities routine in practice or have you become blasé about the N^2M^2QAP weirdnesses? More qualitative feedback on "things you find odd about the samples" would be appreciated!

Your comments and suggestions are welcome. If you discover any errors in our recording or interpretation of your data, please let us know!



Dave Duewer
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Margaret Kline
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From: David L. Duewer Date: July 22, 1996

To: Jeanice Brown Thomas, Margaret Kline, Willie E. May, Katherine E. Sharpless
cc: Gary W. Kramer, Dennis J. Reeder, Stephen A. Wise

Re: Value and Uncertainty Assignment for the NIST/NCI Micronutrient Measurement Quality Assurance Program's Round Robin XXXVII (RR37) Sera: 219 to 222.

Background: Four sera were distributed in RR37. Serum 222 was distributed as #181 in RR28. Sera 219, 220, and 221 were prepared for this exercise from the same low-carotenoid serum pool. All three sera were augmented to various levels of retinol, retinyl palmitate, and/or α-, γ-, or δ-tocopherol. Serum 219 was augmented to a very high *trans*-β-carotene level with an extract of a commercial dietary supplement. Sera 220 and 221 were augmented with plant extracts, with serum 220 achieving desired α-carotene and lutien levels. A different augmentation technique was evaluated with each of these three sera.

Analysts NIST1 and NIST3 independently extracted and analyzed aliquots of three vials of each serum using normal procedures. Both analysts reported quantitative results for retinol, α-tocopherol, γ-tocopherol, total β-carotene, and *trans*-β-carotene. Analyst NIST3 also reported results for retinyl palmitate, δ-tocopherol, total α-carotene, *trans*-α-carotene, total lycopene, *trans*-lycopene, β-cryptoxanthin, "lutein", and "zeaxanthin". Due to detector malfunction, NIST3 had to analyse three or four aliquots of several vials to fully characterize each sample. When more than two values for an analyte were reported for a given vial, only the minimum and maximum of the reported values have been used in this data analysis.

Results: Table 1 presents the NIST data, summary statistics for the NIST data, summary results for the most recent previous exercise for each serum, summary results for RR37, and the NIST assigned values and uncertainties. Table 2 presents a compact summary of the assigned values and uncertainties for each analyte in each serum. 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 the linear regression of NIST1's $Mean_x$ values to NIST3's or, for analytes that NIST1 did not report, to the interlaboratory Median (see below). The regression model used to determine SD_{anl} is defined to the right of this block. Details include: the model used, the parameters and standard errors on the parameters, and R^2 for the regression.

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

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

- Previous Round Robin Summary Statistics

RR identifier of the Round Robin in which this serum was distributed

Serum identifier of the serum

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

$Median_p$ median of the reported values. The median is a robust location estimate.

eSD_p 0.741 × InterQuartile Range (IQR). The IQR is a robust dispersion estimate. The scale factor 0.741 is the expected ratio between SD and IQR for normal distributions.

- Round Robin XXXVI 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) $$TDIST\left(\frac{\left|Median_n - Median_p\right|\sqrt{n_n + n_p - 2}}{\sqrt{\left((n_n - 1)eSD_n^2 + (n_p - 1)eSD_p^2\right)\left(\frac{1}{n_n} + \frac{1}{n_p}\right)}}, n_p+n_n-2, 2\ tail\right)$$

This is the approximate probability that the current median is the same as it was in the previous RR. Where the hypothesis that $Median_n = Median_p$ could be rejected with 95% confidence, the P(n=p) value would be 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 the previous RR. Where the hypothesis that $eSD_n < eSD_p$ can be rejected with 95% confidence, the P(n<p) value is flagged with an "*". FDIST is Excel®'s F-distribution function.

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

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

- NIST Assigned Values and Uncertainties

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

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

  CV $100 \times NAU / NAV$

Conclusions: The current interlaboratory median ($Median_n$) and standard deviations (eSD_n) for serum 222 are statistically indistinguishable at the 95% confidence level from those of RR28 ($Median_p$ and eSD_p). Thus there is no evidence of degradation during the past three years storage at -75 °C.

The ratio between the repeatability (SD_{rep}) and heterogeneity (SD_{het}) components of variation in the NIST measurements are roughly comparable for all analytes in all sera. The augmentation techniques used with sera 219-221 seem not to have compromised sample homogeneity.

This memo will be included as part of the Round Robin XXXVII report.

Dave
Reseach Busybody

Table 1
NIST Data and Calculations

Retinol

	NIST1				NIST3			
	219	220	221	222	219	220	221	222
A:1	0.782	0.584	0.622	0.448	0.705	0.459	0.644	0.450
A:2	0.821	0.479	0.670	0.474	0.852	0.472	0.666	0.495
B:1	0.791	0.529	0.644	0.497	0.732	0.444	0.638	0.466
B:2	0.744	0.511	0.710	0.513	0.693	0.473	0.633	0.471
C:1	0.769	0.506	0.695	0.502	0.801	0.465	0.661	0.478
C:2	0.810	0.474	0.655	0.475	0.705	0.457	0.665	0.481
n_x	6	6	6	6	6	6	6	6
$Mean_x$	0.786	0.514	0.666	0.485	0.748	0.462	0.651	0.474
SD_x	0.028	0.040	0.033	0.024	0.064	0.011	0.015	0.015
SD_{repx}	0.030	0.045	0.037	0.017	0.073	0.013	0.009	0.019
SD_{betx}	0.017	0.021	0.017	0.022	0.033	0.004	0.014	0.006
SD_{NISTx}	0.035	0.050	0.041	0.028	0.081	0.014	0.017	0.019
CV_{NISTx}	4.4	9.8	6.2	5.7	11	3.0	2.6	4.1

Retinyl Palmitate

	NIST1				NIST3			
	219	220	221	222	219	220	221	222
			1.03	0.114			1.73	
			1.12	0.112			1.93	0.109
			1.02	0.116				0.109
			1.35	0.114			1.72	0.151
			1.24	0.112				0.118
			1.05	0.110				0.103
	0	0	6	6	0	0	3	5
			1.14	0.113			1.79	0.118
			0.13	0.002			0.12	0.019
			0.16	0.001			0.10	0.018
			0.05	0.002				0.012
			0.17	0.002				0.022
			15	2.2				18

	NIST			
n	12	12	12	12
Mean	0.767	0.488	0.659	0.479
SD_{rep}	0.050	0.034	0.027	0.018
SD_{bet}	0.024	0.020	0.016	0.015
SD_{anl}	0.020	0.020	0.020	0.020
SD_{NIST}	0.059	0.045	0.037	0.031
CV_{NIST}	7.7	9.2	5.7	6.4

NIST3=a+b*NIST1

a: 0

b: 0.954 ±0.016

R^2: 0.960

NIST			
0	0	9	11
		1.46	0.116
		0.14	0.017
		0.08	0.012
		0.16	0.021
		11	18

RR	XXVIII
Serum	181
n_p	44
$Median_p$	0.483
eSD_p	0.064

<— Previous Results —>

XXVIII
181
8
0.13
0.03

	RRXXXVII			
	219	220	221	222
n_n	45	45	44	45
$Median_n$	0.817	0.489	0.673	0.470
eSD_n	0.068	0.040	0.063	0.050
P(n=p)				0.96
P(n<p)				0.95
SD_{labs}	0.033	0	0.051	0.039
CV_{labs}	4.1	0	7.6	8.3
NAV	0.792	0.488	0.666	0.475
NAU	0.068	0.045	0.063	0.050
CV	8.5	9.1	9.5	10

<— Current Results —>

<—— Assignments ——>

RRXXXVII			
219	220	221	222
10	11	13	14
0.03	0.03	1.52	0.097
0.01	0.02	0.31	0.019
			0.53
			0.95
		0.27	0
		18	0
		1.49	0.106
		0.31	0.021
		21	20

Table 1
NIST Data and Calculations

α-Tocopherol

	NIST1				NIST3			
	219	220	221	222	219	220	221	222
A:1	7.86	11.44	17.1	7.69	8.32	10.83	16.3	6.21
A:2	7.43	11.33	16.7	7.62	8.29	11.64	17.7	6.56
B:1	7.27	11.74	16.9	7.33	7.69	11.50	16.9	6.90
B:2	7.63	11.16	17.1	7.70	8.50	11.72	17.6	7.30
C:1	8.09	11.49	16.9	7.15	8.15	11.06	17.3	7.19
C:2	8.92	11.44	17.3	7.18	7.81	10.51	16.6	7.15
n_x	6	6	6	6	6	6	6	6
$Mean_x$	7.87	11.43	17.0	7.44	8.13	11.21	17.1	6.89
SD_x	0.59	0.19	0.2	0.25	0.32	0.48	0.6	0.43
SD_{repx}	0.41	0.24	0.2	0.15	0.36	0.41	0.7	0.22
SD_{betx}	0.56	0.04	0.1	0.25	0.16	0.41	0.2	0.44
SD_{NISTx}	0.69	0.25	0.3	0.29	0.40	0.58	0.7	0.49
CV_{NISTx}	8.8	2.2	1.5	3.9	4.9	5.2	4.3	7.1

	NIST			
n	12	12	12	12
Mean	8.00	11.32	17.0	7.17
SD_{rep}	0.44	0.30	0.5	0.22
SD_{bet}	0.41	0.27	0.2	0.42
SD_{anl}	0.36	0.36	0.4	0.36
SD_{NIST}	0.70	0.55	0.7	0.60
CV_{NIST}	8.8	4.8	3.9	8.3

NIST3=a+b*NIST1

a: 0
b: 0.99 ±0.02
R^2: 0.987

RR	XXVIII
Serum	181
n_p	44
$Median_p$	7.11
eSD_p	0.53

<— Previous Results —>

	RRXXXVII			
	219	220	221	222
n_a	42	42	41	42
$Median_a$	7.30	10.96	17.3	7.06
eSD_a	0.58	0.79	1.7	0.60
P(n=p)				0.98
P(n<p)				0.21
SD_{labs}	0	0.58	1.6	0
CV_{labs}	0	5.3	9.2	0

<— Current Results —>

NAV	7.65	11.14	17.2	7.11
NAU	0.70	0.79	1.7	0.60
CV	9.2	7.1	10	8.4

<— Assignments —>

γ-Tocopherol

	NIST1				NIST3			
	219	220	221	222	219	220	221	222
A:1	7.12	4.46	1.88	2.22	8.76	3.77	1.79	2.22
A:2	7.79	3.18	1.97	2.32	8.70	3.96	1.85	2.32
B:1	8.02	3.52	1.97	2.62	8.41	4.02	1.82	2.38
B:2	7.11	3.47	2.29	2.58	8.62	4.03	1.86	2.36
C:1	7.15	4.12	1.87	2.59	8.42	3.99	1.86	2.38
C:2	7.59	3.65	1.84	2.51	8.12	3.92	1.84	2.39
n_x	6	6	6	6	6	6	6	6
$Mean_x$	7.46	3.74	1.97	2.48	8.50	3.95	1.84	2.34
SD_x	0.40	0.47	0.17	0.16	0.24	0.10	0.03	0.07
SD_{repx}	0.50	0.56	0.14	0.05	0.15	0.08	0.03	0.04
SD_{betx}	0.10	0.21	0.14	0.18	0.23	0.08	0.01	0.06
SD_{NISTx}	0.51	0.60	0.20	0.18	0.27	0.12	0.04	0.08
CV_{NISTx}	6.8	16	10	7.4	3.2	2.9	1.9	3.3

	NIST			
	12	12	12	12
	7.98	3.84	1.90	2.41
	0.36	0.40	0.10	0.05
	0.16	0.17	0.11	0.15
	0.06	0.06	0.06	0.06
	0.40	0.44	0.16	0.16
	5.0	11	8.5	6.8

NIST3=a+b*NIST1

a: -0.62 ±0.06
b: 1.22 ±0.01
R^2: 1.000

	XXVIII
	181
	16
	2.28
	0.16

	RRXXXVII			
	22	22	21	22
	8.39	3.60	1.86	2.31
	0.61	0.33	0.26	0.17
				0.96
				0.46
	0.46	0	0.20	0.02
	5.5	0	11	1.0

	8.18	3.72	1.88	2.36
	0.61	0.44	0.26	0.17
	7.5	12	14	7.0

Table 1
NIST Data and Calculations

δ-Tocopherol

	NIST1				NIST3			
	219	220	221	222	219	220	221	222
A:1	3.51	7.44	0.89		3.37	9.86	0.88	0.156
A:2	3.66	7.48	0.93		3.32	10.20	0.95	0.194
B:1	3.48	8.71	0.85		3.26	10.17	0.87	0.197
B:2	3.57	7.41	0.92		3.33	10.10	0.93	0.207
C:1	3.09	8.78	0.81		3.28	10.00	1.10	0.199
C:2	3.16	7.88	0.82		3.25	9.77	0.98	0.219
n_x	6	6	6	0	6	6	6	6
$Mean_x$	3.41	7.95	0.87		3.30	10.02	0.95	0.195
SD_x	0.23	0.64	0.05		0.05	0.17	0.08	0.021
SD_{repx}	0.08	0.65	0.03		0.04	0.17	0.06	0.018
SD_{betx}	0.25	0.45	0.05		0.04	0.13	0.08	0.018
SD_{NISTx}	0.26	0.79	0.06		0.06	0.21	0.09	0.025
CV_{NISTx}	7.7	9.9	6.9		1.7	2.1	10.0	13

	NIST			
n	12	12	12	6
Mean	3.36	8.98	0.91	0.195
SD_{rep}	0.06	0.47	0.04	0.017
SD_{bet}	0.04	0.13	0.08	0.018
SD_{anl}	0.04	0.04	0.04	0.038
SD_{NIST}	0.08	0.49	0.09	0.046
CV_{NIST}	2.5	5.4	10	23

NIST3=a+b*Median

a: -0.032 ±0.026
b: 1.235 ±0.006
R^2: 1.000

	XXVIII
RR Serum	181
n_p	0
$Median_p$	
eSD_p	

<— Previous Results —>

	RRXXXVII			
	219	220	221	222
n_n	5	5	4	5
$Median_n$	2.74	8.13	0.78	0.170
eSD_n	0.44	0.96	0.34	0.045
P(n=p)				
P(n<p)				
SD_{labs}	0.44	0.82	0.33	0
CV_{labs}	16	10	42	0

<— Current Results —>

NAV	3.05	8.55	0.85	0.183
NAU	0.44	0.96	0.34	0.046
CV	15	11	40	25

<—— Assignments ——>

Total β-Carotene

	NIST1				NIST3			
	219	220	221	222	219	220	221	222
A:1	3.70	0.559			3.46	0.422	0.076	0.055
A:2	4.09	0.489			3.82	0.397	0.080	0.062
B:1	3.60	0.491			3.81	0.395	0.073	0.065
B:2	3.87	0.494				0.433	0.073	0.066
C:1	3.54	0.496				0.538	0.091	0.066
C:2	3.77	0.509			3.82	0.475	0.086	0.067
n_x	6	6	0	0	4	6	6	6
$Mean_x$	3.76	0.506			3.73	0.443	0.080	0.063
SD_x	0.20	0.027			0.18	0.055	0.007	0.005
SD_{repx}	0.21	0.029			0.15	0.032	0.003	0.003
SD_{betx}	0.12	0.016				0.055	0.008	0.004
SD_{NISTx}	0.25	0.033				0.063	0.008	0.005
CV_{NISTx}	6.5	6.6				14	11	8.4

	NIST			
n	10	12	6	6
Mean	3.74	0.475	0.080	0.063
SD_{rep}	0.18	0.027	0.002	0.003
SD_{bet}	0.10	0.055	0.008	0.004
SD_{anl}	0.05	0.046	0.046	0.046
SD_{NIST}	0.22	0.076	0.046	0.046
CV_{NIST}	5.8	16	58	73

NIST3=a+b*Median

a: -0.029 ±0.028
b: 0.975 ±0.014
R^2: 0.999

	XXVIII
RR Serum	181
n_p	33
$Median_p$	0.087
eSD_p	0.022

	RRXXXVII			
	219	220	221	222
n_n	28	28	27	28
$Median_n$	3.85	0.541	0.082	0.077
eSD_n	0.30	0.101	0.009	0.012
P(n=p)				0.89
P(n<p)				1.00
SD_{labs}	0.21	0.066	0	0
CV_{labs}	5.4	12	0	0

NAV	3.79	0.508	0.081	0.070
NAU	0.30	0.101	0.046	0.046
CV	7.9	20	57	66

Table 1
NIST Data and Calculations

trans-β-Carotene

	NIST1				NIST3			
	219	220	221	222	219	220	221	222
A:1	3.27	0.452			3.24	0.401	0.076	0.055
A:2	3.57	0.412			3.52	0.381	0.080	0.062
B:1	3.11	0.410			3.52	0.370	0.073	0.065
B:2	3.35	0.397				0.407	0.073	0.066
C:1	3.22	0.420			3.69	0.503	0.091	0.066
C:2	3.37	0.422			3.57	0.451	0.086	0.067
n_x	6	6	0	0	5	6	6	6
$Mean_x$	3.31	0.419			3.51	0.419	0.080	0.064
SD_x	0.16	0.019			0.16	0.050	0.007	0.005
SD_{repx}	0.17	0.017			0.12	0.027	0.003	0.003
SD_{betx}	0.10	0.014			0.12	0.050	0.008	0.004
SD_{NISTx}	0.19	0.022			0.18	0.057	0.008	0.005
CV_{NISTx}	5.8	5.3			5.0	14	10	8.3

	NIST			
n	11	12	6	6
Mean	3.41	0.419	0.080	0.064
SD_{rep}	0.14	0.020	0.002	0.003
SD_{bet}	0.12	0.050	0.008	0.004
SD_{anl}	0.05	0.047	0.047	0.047
SD_{NIST}	0.19	0.072	0.047	0.047
CV_{NIST}	5.7	17	59	74

NIST3=a+b*Median

a: 0
b: 0.990 ±0.013
R^2: 0.998

RR	XXVIII
Serum	181
n_p	5
$Median_p$	0.085
eSD_p	0.005

<— Previous Results —>

	RRXXXVII			
	219	220	221	222
n_s	14	14	13	14
$Median_s$	3.53	0.503	0.076	0.072
eSD_s	0.27	0.094	0.008	0.010
P(n=p)				0.48
P(n<p)				0.10
SD_{labs}	0.19	0.060	0	0
CV_{labs}	5.4	12	0	0

<— Current Results —>

NAV	3.47	0.461	0.078	0.068
NAU	0.27	0.094	0.047	0.047
CV	7.9	20	61	69

<—— Assignments ——>

Total α-Carotene

	NIST1				NIST3			
	219	220	221	222	219	220	221	222
A:1	0.133	0.274			0.092	0.245	0.011	0.011
A:2	0.146	0.235			0.129	0.239	0.012	0.014
B:1	0.145	0.233			0.122	0.223	0.011	0.012
B:2	0.138	0.212				0.249	0.013	0.015
C:1	0.148	0.238			0.114	0.264	0.015	0.013
C:2	0.135	0.226			0.104	0.231	0.015	0.014
n_x	6	6	0	0	5	6	6	6
$Mean_x$	0.141	0.236			0.112	0.242	0.013	0.013
SD_x	0.006	0.021			0.015	0.014	0.002	0.001
SD_{repx}	0.008	0.019			0.016	0.017	0.001	0.002
SD_{betx}	0.001	0.016			0.007	0.006	0.002	0.001
SD_{NISTx}	0.008	0.025			0.017	0.018	0.002	0.002
CV_{NISTx}	5.8	11			15	7.6	16	13

	NIST			
	11	12	6	6
	0.127	0.239	0.013	0.013
	0.012	0.017	0.001	0.002
	0.007	0.006	0.002	0.001
	0.017	0.017	0.017	0.017
	0.022	0.025	0.017	0.017
	18	10	130	130

NIST3=a+b*Median

a: 0
b: 0.764 ±0.050
R^2: 0.950

	XXVIII
	181
	15
	0.018
	0.006

	RRXXXVII			
	219	220	221	222
	24	24	21	21
	0.110	0.329	0.013	0.016
	0.023	0.084	0.007	0.005
				0.91
				0.72
	0.006	0.080	0	0
	5.8	24	0	0

	0.118	0.284	0.013	0.014
	0.023	0.084	0.017	0.017
	20	30	130	120

Table 1
NIST Data and Calculations

trans-α-Carotene

	NIST1				NIST3			
	219	220	221	222	219	220	221	222
A:1					0.08	0.2	0.011	0.011
A:2					0.10	0.2	0.014	0.014
B:1					0.10	0.2	0.012	0.012
B:2						0.2	0.013	0.015
C:1					0.09	0.3	0.015	0.013
C:2					0.09	0.2	0.015	0.014
n_x	0	0	0	0	5	6	6	6
$Mean_x$					0.09	0.2	0.013	0.013
SD_x					0.01	0.0	0.002	0.001
SD_{repx}					0.01	0.0	0.001	0.002
SD_{betx}					0.00	0.0	0.001	0.001
SD_{NISTx}					0.01	0.0	0.002	0.002
CV_{NISTx}					9.4	6.5	15	14

	NIST			
n	5	6	6	6
Mean	0.09	0.2	0.013	0.013
SD_{rep}	0.01	0.0	0.001	0.002
SD_{bet}	0.00	0.0	0.001	0.001
SD_{anl}				
SD_{NIST}				
CV_{NIST}				

RR	XXVIII	
Serum	181	<— Previous Results —>
n_p	0	
$Median_p$		
eSD_p		

	RRXXXVII				
	219	220	221	222	<— Current Results —>
n_a	0	0	0	0	
$Median_a$					
eSD_a					
P(n=p)					
P(n<p)					
SD_{labs}					
CV_{labs}					
NAV	0.09	0.2	0.013	0.013	<— Assignments —>
NAU					
CV					

Total Lycopene

	NIST1				NIST3			
	219	220	221	222	219	220	221	222
A:1					0.351	0.329	0.313	0.450
A:2					0.367	0.340	0.323	0.491
B:1					0.370	0.337	0.315	0.526
B:2					0.366	0.353	0.328	0.536
C:1					0.372	0.360	0.334	0.532
C:2					0.349	0.343	0.325	0.536
n_x	0	0	0	0	6	6	6	6
$Mean_x$					0.363	0.343	0.323	0.512
SD_x					0.010	0.011	0.008	0.035
SD_{repx}					0.012	0.010	0.008	0.017
SD_{betx}					0.005	0.009	0.006	0.036
SD_{NISTx}					0.013	0.013	0.010	0.040
CV_{NISTx}					3.5	3.9	3.0	7.8

	NIST			
	6	6	6	6
	0.363	0.343	0.323	0.512
	0.007	0.010	0.008	0.018
	0.005	0.009	0.006	0.036
	0.002	0.002	0.002	0.002
	0.009	0.013	0.010	0.040
	2.4	3.9	3.2	7.8

XXVIII
181
16
0.468
0.100

	RRXXXVII			
	219	220	221	222
	24	24	23	24
	0.280	0.256	0.237	0.448
	0.052	0.048	0.043	0.040
				0.93
				1.00
	0.051	0.046	0.042	0.002
	18	18	18	0.4
	0.321	0.300	0.280	0.480
	0.052	0.048	0.043	0.040
	16	16	15	8.4

NIST3=a+b*Median

a: 0.114 ±0.004
b: 0.888 ±0.012
R^2: 0.999

Table 1
NIST Data and Calculations

trans-Lycopene

	NIST1				NIST3			
	219	220	221	222	219	220	221	222
A:1					0.152	0.145	0.148	0.201
A:2					0.159	0.151	0.158	0.221
B:1					0.150	0.144	0.148	0.237
B:2					0.151	0.155	0.152	0.242
C:1					0.165	0.154	0.159	0.247
C:2					0.155	0.152	0.153	0.245
n_x	0	0	0	0	6	6	6	6
$Mean_x$					0.155	0.150	0.153	0.232
SD_x					0.006	0.005	0.005	0.018
SD_{repx}					0.005	0.005	0.005	0.008
SD_{betx}					0.005	0.003	0.003	0.019
SD_{NISTx}					0.007	0.006	0.006	0.020
CV_{NISTx}					4.4	3.8	3.8	8.8

	NIST			
n	6	6	6	6
Mean	0.155	0.150	0.153	0.232
SD_{rep}	0.003	0.007	0.005	0.009
SD_{het}	0.005	0.003	0.003	0.019
SD_{ani}	0.008	0.008	0.008	0.008
SD_{NIST}	0.009	0.010	0.009	0.022
CV_{NIST}	6.0	7.0	6.1	9.4

NIST3=a+b*Median

a: 0.070 ±0.012
b: 0.518 ±0.057
R^2: 0.953

RR	XXVIII
Serum	181
n_p	0
$Median_p$	
eSD_p	

<— Previous Results —>

	RRXXXVII			
	219	220	221	222
n_n	7	7	6	7
$Median_n$	0.157	0.172	0.153	0.311
eSD_n	0.028	0.032	0.027	0.074
P(n=p)				
P(n<p)				
SD_{labs}	0.026	0.030	0.025	0.071
CV_{labs}	17	17	16	23

<— Current Results —>

NAV	0.156	0.161	0.153	0.272
NAU	0.028	0.032	0.027	0.074
CV	18	20	17	27

<—— Assignments ——>

β-Cryptoxanthin

	NIST1				NIST3			
	219	220	221	222	219	220	221	222
A:1					0.065	0.058	0.060	0.040
A:2					0.067	0.060	0.061	0.047
B:1					0.062	0.060	0.060	0.049
B:2					0.072	0.061	0.060	0.054
C:1					0.066	0.060	0.060	0.050
C:2					0.058	0.056	0.058	0.052
n_x	0	0	0	0	6	6	6	6
$Mean_x$					0.065	0.059	0.060	0.049
SD_x					0.005	0.002	0.001	0.005
SD_{repx}					0.005	0.002	0.001	0.004
SD_{betx}					0.003	0.001	0.001	0.004
SD_{NISTx}					0.006	0.002	0.001	0.006
CV_{NISTx}					9.1	3.8	2.0	12

	NIST			
n	6	6	6	6
Mean	0.065	0.059	0.060	0.049
SD_{rep}	0.006	0.001	0.000	0.004
SD_{het}	0.003	0.001	0.001	0.004
SD_{ani}	0.004	0.004	0.004	0.004
SD_{NIST}	0.007	0.004	0.004	0.007
CV_{NIST}	12	7.1	6.6	15

NIST3=a+b*Median

a: -0.128 ±0.068
b: 2.545 ±0.933
R^2: 0.621

	XXVIII
	181
	11
	0.070
	0.012

	RRXXXVII			
	219	220	221	222
	19	19	18	19
	0.075	0.075	0.073	0.070
	0.025	0.022	0.020	0.019
				0.99
				0.05
	0.023	0.022	0.019	0.018
	31	29	27	25

	0.070	0.067	0.066	0.059
	0.025	0.022	0.020	0.019
	35	33	30	32

Table 1
NIST Data and Calculations

"Lutein"

	NIST1				NIST3			
	219	220	221	222	219	220	221	222
A:1					0.084	0.119	0.082	0.081
A:2					0.086	0.120	0.086	0.086
B:1					0.080	0.120	0.082	0.085
B:2					0.081	0.124	0.082	0.085
C:1					0.086	0.126	0.086	0.090
C:2					0.084	0.123	0.084	0.085
n_x	0	0	0	0	6	6	6	6
$Mean_x$					0.084	0.122	0.084	0.085
SD_x					0.003	0.003	0.002	0.003
SD_{repx}					0.001	0.002	0.002	0.003
SD_{betx}					0.003	0.002	0.002	0.002
SD_{NISTx}					0.003	0.003	0.002	0.004
CV_{NISTx}					3.4	2.7	2.8	4.1

	NIST			
n	6	6	6	6
Mean	0.084	0.122	0.084	0.085
SD_{rep}	0.001	0.002	0.002	0.002
SD_{bet}	0.003	0.002	0.002	0.002
SD_{anl}	0.002	0.002	0.002	0.002
SD_{NIST}	0.004	0.004	0.003	0.004
CV_{NIST}	4.2	3.3	3.7	4.2

NIST3=a+b*Median

a: 0
b: 1.024 ±0.012
R^2: 0.973

RR	XXVIII
Serum	181
n_p	7
$Median_p$	0.068
eSD_p	0.015

<— Previous Results —>

	RRXXXVII			
	219	220	221	222
n_a	13	13	12	13
$Median_a$	0.080	0.120	0.084	0.081
eSD_a	0.017	0.025	0.016	0.019
P(n=p)				0.74
P(n<p)				0.27
SD_{labs}	0.017	0.025	0.016	0.019
CV_{labs}	21	21	19	23

<— Current Results —>

NAV	0.082	0.121	0.084	0.083
NAU	0.017	0.025	0.016	0.019
CV	21	21	19	23

<—— Assignments ——>

"Zeaxanthin"

	NIST1				NIST3			
	219	220	221	222	219	220	221	222
A:1					0.046	0.052	0.048	0.044
A:2					0.048	0.049	0.052	0.047
B:1					0.045	0.051	0.050	0.046
B:2					0.043	0.053	0.047	0.047
C:1					0.047	0.052	0.048	0.049
C:2					0.045	0.051	0.050	0.047
n_x	0	0	0	0	6	6	6	6
$Mean_x$					0.046	0.051	0.049	0.047
SD_x					0.002	0.001	0.002	0.002
SD_{repx}					0.001	0.002	0.002	0.002
SD_{betx}					0.002	0.001	0.001	0.001
SD_{NISTx}					0.002	0.002	0.002	0.002
CV_{NISTx}					4.6	3.3	4.7	4.2

	NIST			
	6	6	6	6
	0.046	0.051	0.049	0.047
	0.001	0.002	0.002	0.001
	0.002	0.001	0.001	0.001
	0.003	0.003	0.003	0.003
	0.003	0.003	0.004	0.003
	7.4	6.3	7.4	6.9

NIST3=a+b*Median

a: 0.031 ±0.020
b: 0.401 ±0.457
R^2: 0.077

XXVIII
181
3
0.026

RRXXXVII			
219	220	221	222
11	11	10	10
0.044	0.045	0.046	0.039
0.022	0.021	0.023	0.013
0.022	0.020	0.022	0.013
49	45	49	33

0.045	0.048	0.048	0.043
0.022	0.021	0.023	0.013
49	43	48	31

Table 2
Summary of NIST Assigned Values and Uncertainties

	219			220			221			222		
Analyte	NAV	NAU	CV	NAV	NAU	CV	NAV	NAU	CV	NAV	NAU	CV
Retinol	0.792	0.068	8.5	0.488	0.045	9.1	0.666	0.063	9.5	0.475	0.050	10
Retinyl Palmitate							1.49	0.31	21	0.106	0.021	20
α-Tocopherol	7.65	0.70	9.2	11.14	0.79	7.1	17.2	1.7	10	7.11	0.60	8.4
γ-Tocopherol	8.18	0.61	7.5	3.72	0.44	12	1.88	0.26	14	2.36	0.17	7.0
δ-Tocopherol	3.05	0.44	15	8.55	0.96	11	0.85	0.34	40	0.183	0.046	25
Total β-Carotene	3.79	0.30	7.9	0.508	0.101	20	0.081	0.046	57	0.070	0.046	66
trans-β-Carotene	3.47	0.27	7.9	0.461	0.094	20	0.078	0.047	61	0.068	0.047	69
Total α-Carotene	0.118	0.023	20	0.284	0.084	30	0.013	0.017	130	0.014	0.017	120
trans-α-Carotene	0.09			0.2			0.013			0.013		
Total Lycopene	0.321	0.052	16	0.300	0.048	16	0.280	0.043	15	0.480	0.040	8.4
trans-Lycopene	0.156	0.028	18	0.161	0.032	20	0.153	0.027	17	0.272	0.074	27
β-Cryptoxanthin	0.070	0.025	35	0.067	0.022	33	0.066	0.020	30	0.059	0.019	32
"Lutein"	0.082	0.017	21	0.121	0.025	21	0.084	0.016	19	0.083	0.019	23
"Zeaxanthin"	0.045	0.022	49	0.048	0.021	43	0.048	0.023	48	0.043	0.013	31

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Appendix G. "All-Lab Report" for RR37

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 XXXVII Laboratory Results

Values in µg/mL

	Retinol				Retinyl Palmitate				α-Tocopherol				γ-Tocopherol			
Lab	219	220	221	222	219	220	221	222	219	220	221	222	219	220	221	222
FSV-BA	0.835	0.526	0.691	0.502	0.009	0.008	1.37	0.012	7.24	10.71	16.2	6.91	8.37	3.77	1.87	2.45
FSV-BD	0.782	0.486	0.674	0.481					6.82	9.62	15.9	6.88				
FSV-BE	0.872	0.521	0.682	0.456					7.26	10.76	16.8	6.78	8.02	3.35	1.60	2.27
FSV-BG	0.860	0.510	0.710	0.520	0.031	0.020	1.52	0.085	7.38	11.21	17.4	7.52	7.34	3.31	1.63	2.29
FSV-BH	0.702	0.384	0.524	0.440	0.038	0.030	1.18	0.113	7.75	11.46	17.9	7.65	8.71	3.81	1.94	2.59
FSV-BI	0.796	0.459	0.628	0.419	*nd*	*nd*	1.82	0.127	7.47	10.74	15.8	6.32	7.70	3.38	1.47	2.05
FSV-BJ	0.825	0.499	0.692	0.478	*nd*	*nd*	1.25	0.095	7.98	11.60	18.3	7.20	9.34	4.08	2.16	2.53
FSV-BK	0.806	0.522	0.623	0.545					6.03	9.94	15.9	5.84				
FSV-BL	0.773	0.458	0.687	0.487					7.75	12.06	19.0	7.75				
FSV-BM	0.658	0.355	0.556	0.416					8.30	14.30	19.2	7.30				
FSV-BN	0.847	0.473	0.653	0.463	0.029	0.011	1.67	0.085	6.95	10.38	16.2	6.67	7.90	3.32	1.77	2.26
FSV-BO	0.725	0.430	0.653	0.464					7.19	11.37	18.6	7.28				
FSV-BP	0.769	0.439	0.541	0.416					8.39	13.10	19.4	8.86				
FSV-BQ	0.760	0.520	0.670	0.510					8.01	11.70	18.1	8.00				
FSV-BR	0.880	0.540	0.754	0.508												
FSV-BS	0.920	0.520	0.720	0.470												
FSV-BT	0.888	0.495	0.689	0.612	0.042	0.051	1.67	0.097	8.18	11.39	17.3	6.66	8.82	3.94	2.10	2.43
FSV-BU	0.843	0.502	0.733	0.539												
FSV-BW	0.760	0.420	0.620	0.430	0.030	0.022	1.44	0.097	7.28	11.16	17.9	6.60	8.83	3.80	2.00	2.51
FSV-BX	1.169	0.573	1.005	0.639					12.10	17.53	26.6	10.81	11.86	5.07	2.59	3.26
FSV-BY	0.784	0.489	0.666	0.457	0.021	0.012	1.75	0.120	7.49	10.71	17.5	7.07	8.11	3.35	1.74	2.32
FSV-BZ									6.60	10.00	14.9	6.05	7.10	3.58	2.11	1.92
FSV-CA	0.701	0.402	0.510	0.391					6.60	9.86	14.0	6.45				
FSV-CB	0.723	0.419	0.571	0.407					6.28	10.42	18.0	6.29				
FSV-CC	0.892	0.471	0.690	0.452					7.89	12.91	20.1	7.65				
FSV-CF	0.700	0.456	0.641	0.441					7.50	11.80	18.7	7.60				
FSV-CH	0.700	0.410	0.590	0.400					6.32	9.61	16.0	6.79	7.66	3.43	1.75	2.25
FSV-CK	0.860	0.502	0.707	0.453					7.29	11.16	16.3	7.34	8.42	4.00	2.17	2.48
FSV-CM									7.00	11.40	18.7	7.10				
FSV-CN	0.790	0.480	0.640	0.480					7.65	11.46	17.8	6.83	8.48	3.40	1.51	2.25
FSV-CP									8.13	12.11	19.6	8.16	7.85	3.82	1.78	2.42
FSV-CQ	0.841	0.546	0.736	0.501					6.95	10.26	15.7	6.49				
FSV-CR	0.830	0.480	0.710	0.520					7.30	11.40	17.2	7.10				
FSV-CS	0.832	0.508	0.715	0.485												
FSV-CU	0.811	0.485	0.640	0.459	0.284	0.073	1.05	0.111	7.16	10.65	15.2	7.07				
FSV-CX	0.830	0.490	0.680	0.490	0.392	0.047	1.52	0.123	7.54	11.06	19.6	7.47	7.82	3.13	1.78	1.98
FSV-DA	0.810	0.510	0.660	0.470	*nd*	0.090	1.60	0.100	7.16	11.00	16.7	7.05	8.41	3.74	1.86	2.51
FSV-DB	0.930	0.560	0.760	0.530					7.86	10.91	18.2	7.93				
FSV-DJ	1.070	0.600	0.800	0.570					9.68	13.16	18.5	7.44				
FSV-DK	0.880	0.500	0.700	0.540	*nd*	*nd*	0.70	0.053	7.30	9.90	17.1	7.10	8.40	3.50	1.80	2.10
FSV-DM	0.817	0.515	0.672	0.482					6.99	10.69	17.1	6.94				
FSV-DP	0.774	0.462	0.609	0.448												
FSV-DS	0.639	0.390	0.532	0.380					6.40	9.02	13.5	6.48				
FSV-DX	0.884	0.509	0.717	0.469					6.37	10.13	14.7	5.90				
FSV-EH	0.840	0.470		0.530	0.046	0.035		0.097	7.08	10.22		6.23	6.53	3.25		2.09
FSV-EI	0.701	0.438	0.611	0.397					7.48	10.85	16.8	7.05	8.72	3.76	2.41	2.29
FSV-EL	0.770	0.440	0.570	0.430												
FSV-FY	0.829	0.501	0.682	0.488					6.97	10.71	16.8	6.79	8.41	3.62	1.92	2.43
n	45	45	44	45	10	11	13	14	42	42	41	42	22	22	21	22
Min	0.639	0.355	0.510	0.380	0.009	0.008	0.70	0.012	6.03	9.02	13.5	5.84	6.53	3.13	1.47	1.92
Median	0.817	0.489	0.673	0.470	0.035	0.030	1.52	0.097	7.30	10.96	17.3	7.06	8.39	3.60	1.86	2.31
Max	1.169	0.600	1.005	0.639	0.392	0.090	1.82	0.127	12.10	17.53	26.6	10.81	11.86	5.07	2.59	3.26
eSD	0.070	0.046	0.059	0.047	0.014	0.027	0.23	0.019	0.52	0.82	1.7	0.60	0.65	0.33	0.21	0.24
eCV	9	9	9	10	40	89	15	20	7	8	10	9	8	9	11	10
NISTa	0.786	0.514	0.666	0.485	*nd*	*nd*	1.14	0.113	7.87	11.43	17.0	7.44	7.46	3.74	1.97	2.48
NISTb	0.748	0.462	0.651	0.474	*nd*	*nd*	1.79	0.118	8.13	11.21	17.1	6.89	8.50	3.95	1.84	2.34
NAV	0.792	0.488	0.666	0.475	0.035	0.030	1.52	0.097	7.65	11.14	17.2	7.11	8.18	3.72	1.88	2.36
NAU	0.077	0.052	0.064	0.050	0.014	0.027	0.23	0.019	0.80	0.89	1.7	0.62	0.89	0.49	0.26	0.26

Round Robin XXXVII Laboratory Results

Values in µg/mL

	δ-Tocopherol				Total β-Carotene				trans-β-Carotene				Total cis-β-Carotene			
Lab	219	220	221	222	219	220	221	222	219	220	221	222	219	220	221	222
FSV-BA					3.76	0.37	0.088	0.090	3.66	0.36	0.080	0.083	0.10	0.011	0.008	0.007
FSV-BD					3.87	0.49	0.083	0.066								
FSV-BE					2.98	0.37	0.082	0.073								
FSV-BG					3.82	0.33	0.075	0.078								
FSV-BH					3.82	0.61	0.076	0.073	3.70	0.60	0.076	0.073	0.12	0.011	*nd*	*nd*
FSV-BI					4.10	0.49	0.081	0.071								
FSV-BJ					3.54	0.53	0.096	0.087								
FSV-BK																
FSV-BL																
FSV-BM																
FSV-BN	2.74	8.1	0.718	0.109	3.73	0.60	0.077	0.080	3.46	0.58	0.067	0.069	0.27	0.020	0.010	0.011
FSV-BO					2.94	0.28	0.055	0.053								
FSV-BP					3.97	0.69	0.085	0.070								
FSV-BQ																
FSV-BR																
FSV-BS					4.07	0.63	0.140	0.170	3.88	0.59	0.120	0.110	0.19	0.040	0.020	0.060
FSV-BT	4.38	10.9	1.666	0.494	3.71	0.52	0.081	0.076	3.60	0.51	0.078	0.074	0.11	0.009	0.004	0.002
FSV-BU					4.67	0.60	0.087	0.082								
FSV-BW					6.05	0.71	0.089	0.085								
FSV-BX					7.98	1.19	0.220	0.317								
FSV-BY	3.17	9.2	0.851	0.180	3.95	0.34	0.084	0.078	3.63	0.32	0.073	0.068	0.31	0.013	0.011	0.011
FSV-BZ					*3.48*	*0.66*	*>0.150*	*0.152*	3.18	0.64	0.150	0.126	0.30	0.021	*nd*	0.026
FSV-CA																
FSV-CB																
FSV-CC																
FSV-CF																
FSV-CH					3.65	0.59	0.074	0.070								
FSV-CK					4.58	0.59	0.087	0.083								
FSV-CM																
FSV-CN					>4.59	>0.496	>0.081	>0.068	4.59	0.50	0.081	0.068				
FSV-CP					3.88	0.62	0.082	0.063								
FSV-CQ					6.02	0.64	0.040	0.051								
FSV-CR																
FSV-CS																
FSV-CU					>4.04	>0.434	>0.087	>0.079	4.04	0.43	0.087	0.079				
FSV-CX					3.76	0.60	0.110	0.090								
FSV-DA	2.57	7.9	0.690	0.120	3.32	0.53	0.078	0.071	3.04	0.51	0.070	0.071	0.28	0.020	0.008	*nd*
FSV-DB					5.84	0.58	0.010	0.080								
FSV-DJ																
FSV-DK					3.90	0.42	0.090	0.090								
FSV-DM					4.01	0.49	0.072	0.073								
FSV-DP																
FSV-DS					2.64	0.36	0.075	0.059								
FSV-DX					>3.31	>0.365	>0.071	>0.046	3.31	0.37	0.071	0.046				
FSV-EH	2.46	7.5		0.170	3.64	0.50		0.110	3.34	0.47		0.097	0.30	0.030		0.010
FSV-EI					3.68	0.55	0.068	0.068	3.39	0.54	0.066	0.064	*0.29*	*0.012*	*0.002*	*0.004*
FSV-EL																
FSV-FY					>3.21	>0.494	>0.063	>0.054	3.21	0.49	0.063	0.054				
n	5	5	4	5	29	29	27	29	14	14	13	14	10	10	7	8
Min	2.46	7.5	0.690	0.109	2.64	0.28	0.010	0.051	3.04	0.32	0.063	0.046	0.10	0.009	0.002	0.002
Median	2.74	8.1	0.785	0.170	3.82	0.55	0.082	0.078	3.53	0.50	0.076	0.072	0.28	0.017	0.008	0.011
Max	4.38	10.9	1.666	0.494	7.98	1.19	0.220	0.317	4.59	0.64	0.150	0.126	0.31	0.040	0.020	0.060
eSD	0.41	1.0		0.074	0.28	0.09	0.010	0.012	0.30	0.11	0.009	0.011	0.05	0.007	0.004	0.007
eCV	15	12		44	7	17	13	15	8	21	12	15	18	45	56	71
NISTa	3.41	7.9	0.871	*nd*	3.76	0.51	*nd*	*nd*	3.31	0.42	*nd*	*nd*	*0.45*	*0.088*	*nd*	*nd*
NISTb	3.30	10.0	0.952	0.195	3.73	0.44	0.080	0.063	3.51	0.42	0.080	0.063	*0.22*	*0.024*	*0.000*	*0.000*
NAV	3.05	8.6		0.183	3.79	0.51	0.081	0.071	3.47	0.46	0.078	0.068	0.32	0.036		
NAU	0.62	1.7		0.048	0.42	0.11	0.017	0.019	0.33	0.11	0.011	0.012	0.16	0.054		

Round Robin XXXVII Laboratory Results

Values in µg/mL

	Total α-Carotene				Total Lycopene				trans-Lycopene				β-Cryptoxanthin			
Lab	219	220	221	222	219	220	221	222	219	220	221	222	219	220	221	222
FSV-BA	0.116	0.22	0.013	0.015					0.157	0.172	0.161	0.324	0.093	0.093	0.084	0.086
FSV-BD					0.204	0.183	0.204	0.34					0.056	0.056	0.057	0.056
FSV-BE																
FSV-BG	0.090	0.13	0.021	0.034	0.279	0.178	0.230	0.43								
FSV-BH	0.128	0.44	0.011	0.013	0.302	0.304	0.290	0.56					0.095	0.092	0.092	0.096
FSV-BI	0.101	0.30	0.012	0.013	0.215	0.218	0.221	0.38					0.082	0.078	0.074	0.059
FSV-BJ	0.122	0.37	0.031	0.020	0.140	0.164	0.154	0.28								
FSV-BK																
FSV-BL																
FSV-BM																
FSV-BN	0.093	0.40	0.010	0.009	0.272	0.237	0.236	0.44	0.126	0.131	0.133	0.237	0.062	0.057	0.058	0.053
FSV-BO	0.108	0.18	0.013	0.009	0.397	0.274	0.253	0.45					0.054	0.056	0.054	0.053
FSV-BP	0.132	0.47	0.029	0.016	0.313	0.286	0.284	0.61					0.080	0.078	0.074	0.079
FSV-BQ																
FSV-BR																
FSV-BS	0.140	0.39	0.040	0.040	0.100	0.100	0.110	0.45					0.120	0.090	0.090	0.090
FSV-BT	0.078	0.31	0.016	0.020	0.244	0.251	0.237	0.46	0.183	0.192	0.185	0.370	0.098	0.094	0.095	0.084
FSV-BU																
FSV-BW	0.082	0.31	0.011	0.014												
FSV-BX	0.118	0.42	0.081	0.064	1.385	0.376	0.107	0.29					0.105	0.105	0.077	0.122
FSV-BY	0.076	0.14	0.010	0.018	0.307	0.287	0.298	0.52	0.168	0.175	0.182	0.311	0.063	0.064	0.066	0.066
FSV-BZ	0.114	0.24	0.028	0.025	0.312	0.294	0.300	0.42								
FSV-CA																
FSV-CB																
FSV-CC																
FSV-CF																
FSV-CH	0.107	0.36	0.010	0.017	0.229	0.223	0.236	0.49								
FSV-CK	0.150	0.43	0.017	0.019	0.276	0.229	0.217	0.38					0.075	0.073	0.068	0.072
FSV-CM																
FSV-CN	0.132	0.33	*nd*	*nd*	0.266	0.263	0.262	0.45					0.072	0.071	0.065	0.064
FSV-CP	0.110	0.35	0.018	0.030	0.131	0.139	0.141	0.25					0.068	0.075	0.071	0.061
FSV-CQ																
FSV-CR																
FSV-CS																
FSV-CU																
FSV-CX	0.060	0.28	<0.01	<0.01	0.310	0.240	0.270	0.44					0.130	0.110	0.120	0.080
FSV-DA	0.095	0.33	0.009	0.011	0.280	0.260	0.260	0.46	0.130	0.140	0.140	0.240	0.060	0.060	0.060	0.070
FSV-DB	*nq*	*nq*	*nq*	*nq*	0.326	0.273	0.356	0.46					0.117	0.106	0.102	0.111
FSV-DJ																
FSV-DK	0.110	0.22	0.009	0.010												
FSV-DM	0.117	0.32	0.016	0.014	0.290	0.269	0.227	0.46								
FSV-DP																
FSV-DS																
FSV-DX					0.258	0.250	0.262	0.44								
FSV-EH	0.130	0.38		*nd*	0.430	0.450		0.76	0.280	0.290		0.470	0.066	0.064		0.057
FSV-EI	0.081	0.33	0.012	0.015	0.306	0.308	0.296	0.57	0.146	0.142	0.144	0.253	0.064	0.061	0.061	0.059
FSV-EL																
FSV-FY																
n	24	24	21	21	24	24	23	24	7	7	6	7	19	19	18	19
Min	0.060	0.13	0.009	0.009	0.100	0.100	0.107	0.25	0.126	0.131	0.133	0.237	0.054	0.056	0.054	0.053
Median	0.110	0.33	0.013	0.016	0.280	0.256	0.237	0.45	0.157	0.172	0.153	0.311	0.075	0.075	0.073	0.070
Max	0.150	0.47	0.081	0.064	1.385	0.450	0.356	0.76	0.280	0.290	0.185	0.470	0.130	0.110	0.120	0.122
eSD	0.026	0.08	0.005	0.006	0.049	0.047	0.049	0.05	0.039	0.044	0.024	0.087	0.022	0.022	0.018	0.019
eCV	24	25	38	37	18	19	21	10	25	26	16	28	30	30	25	28
NISTa	0.141	0.24	*nd*	*nd*												
NISTb	0.112	0.24	0.013	0.013	0.363	0.343	0.323	0.51	0.155	0.150	0.153	0.232	0.065	0.059	0.060	0.049
NAV	0.119	0.28	0.013	0.015	0.321	0.300	0.280	0.48	0.156	0.161	0.153	0.272	0.070	0.067	0.066	0.059
NAU	0.033	0.11	0.007	0.006	0.089	0.088	0.085	0.11	0.032	0.039	0.031	0.093	0.026	0.025	0.022	0.024

Round Robin XXXVII Laboratory Results

Values in µg/mL

	Lutein				Zeaxanthin				Lutein&Zeaxanthin				Coenzyme Q10			
Lab	219	220	221	222	219	220	221	222	219	220	221	222	219	220	221	222
FSV-BA									0.192	0.271	0.191	0.188				
FSV-BD	0.096	0.139	0.095	0.081	0.044	0.042	0.050	0.041	*0.140*	*0.181*	*0.145*	*0.081*				
FSV-BE																
FSV-BG																
FSV-BH	0.074	0.105	0.074	0.064	0.031	0.035	0.040	*nd*	*0.105*	*0.140*	*0.114*	*0.064*				
FSV-BI	0.074	0.106	0.068	0.055	0.032	0.038	0.036	0.025	*0.106*	*0.144*	*0.104*	*0.055*				
FSV-BJ																
FSV-BK																
FSV-BL																
FSV-BM																
FSV-BN	0.052	0.087	0.052	0.055	0.020	0.022	0.025	0.019	0.072	0.109	0.077	0.075				
FSV-BO									0.116	0.159	0.116	0.085				
FSV-BP																
FSV-BQ																
FSV-BR																
FSV-BS	0.070	0.160	0.090	0.080												
FSV-BT	0.136	0.171	0.131	0.130	0.029	0.025	0.028	0.023	0.165	0.196	0.160	0.152				
FSV-BU																
FSV-BW																
FSV-BX	0.160	0.310	0.200	0.160	0.080	0.068	0.080	0.046	*0.240*	*0.378*	*0.280*	*0.160*				
FSV-BY	0.073	0.121	0.074	0.063	0.042	0.050	0.042	0.031	*0.115*	*0.171*	*0.116*	*0.063*				
FSV-BZ	0.096	0.108	0.088	0.085												
FSV-CA																
FSV-CB																
FSV-CC																
FSV-CF																
FSV-CH													0.950	1.080	1.060	0.760
FSV-CK									0.169	0.218	0.174	0.139				
FSV-CM																
FSV-CN	0.071	0.112	0.073	0.064												
FSV-CP									0.143	0.192	0.146	0.113				
FSV-CQ																
FSV-CR																
FSV-CS																
FSV-CU																
FSV-CX									0.140	0.160	0.140	0.070				
FSV-DA	0.080	0.120	0.080	0.090	0.050	0.060	0.060	0.040	0.130	0.180	0.140	0.130				
FSV-DB									0.141	0.174	0.147	0.106				
FSV-DJ																
FSV-DK																
FSV-DM					0.158	0.194	0.157	0.120								
FSV-DP																
FSV-DS																
FSV-DX									0.130	0.159	0.128	0.101				
FSV-EH	0.080	0.120		0.083	0.046	0.045		0.037	*0.126*	*0.165*		*0.083*	1.200	1.200		0.910
FSV-EI	0.103	0.142	0.098	0.101	0.072	0.072	0.070	0.064	*0.175*	*0.214*	*0.168*	*0.101*				
FSV-EL																
FSV-FY																
n	13	13	12	13	11	11	10	10	17	17	16	17	2	2	1	2
Min	0.052	0.087	0.052	0.055	0.020	0.022	0.025	0.019	0.072	0.109	0.077	0.055	0.95	1.08		0.76
Median	0.080	0.120	0.084	0.081	0.044	0.045	0.046	0.039	0.140	0.174	0.143	0.101	1.075	1.140	1.060	0.835
Max	0.160	0.310	0.200	0.160	0.158	0.194	0.157	0.120	0.240	0.378	0.280	0.188	1.200	1.200		0.910
eSD	0.015	0.022	0.016	0.025	0.019	0.022	0.023	0.016	0.037	0.026	0.039	0.043				
eCV	19	19	19	31	44	49	51	40	26	15	27	43				
NISTa																
NISTb	0.084	0.122	0.084	0.085	0.046	0.051	0.049	0.047	*0.129*	*0.173*	*0.133*	*0.085*				
NAV	0.082	0.121	0.084	0.083	0.045	0.048	0.048	0.043	0.135	0.174	0.138	0.117				
NAU	0.018	0.027	0.019	0.020	0.022	0.021	0.023	0.014	0.037	0.038	0.035	0.046				

Round Robin XXXVII Laboratory Results

Analytes Reported By One Laboratory

Values in µg/mL

Analyte	Code	219	220	221	222
Phytofluene	FSV-DA	0.040	0.060	0.050	0.040
α-Cryptoxanthin	FSV-BN	0.043	0.046	0.042	0.025
trans-α-Carotene	NISTb	0.090	0.237	0.013	0.013

Legend

Term	Definition
n	Number of (non-NIST) quantitative values reported for this analyte
Min	Minimum (non-NIST) quantitative value reported
Median	Median (non-NIST) quantitative value reported
Max	Maximum (non-NIST) quantitative value reported
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 "Report of (Meta)Analysis."
nd	Not detected (i.e., no detectable peak for analyte)
nq	Detected but not quantitatively determined
>x	Concentration greater than or equal to x
<x	Concentration at or below the limit of quantification, x
italics	Not explicitly reported but calculated by NIST from reported values

Round Robin XXXVII Laboratory Results

Comparability Summary

Lab	R	aT	gT	bC*	tbC*
FSV-BA	1	1	1	2	2
FSV-BD	1	2	2	1	
FSV-BE	2	1	1	2	
FSV-BG	1	1			
FSV-BH	3	1		1	
FSV-BI	2	2		3	
FSV-BJ	1	1	2	2	
FSV-BK	2	2	1	2	2
FSV-BL	1	2	1	1	2
FSV-BM	3	4	2	1	
FSV-BN	1	1	4	4	
FSV-BO	2	1	2	4	4
FSV-BP	2	3			
FSV-BQ	1	2			
FSV-BR	2		2	1	
FSV-BS	2				
FSV-BT	3	1			1
FSV-BU	2		1	1	
FSV-BW	2	1	1	1	
FSV-BX	4	4		1	
FSV-BY	1	1	2	2	
FSV-BZ		2			
FSV-CA	3	2		2	
FSV-CB	2	2	1	1	2
FSV-CC	2	2	2	1	2
FSV-CF	2	1		4	3
FSV-CH	2	2		3	
FSV-CK	1	1		2	
FSV-CM		1	1	1	1
FSV-CN	1	1			
FSV-CP		2			
FSV-CQ	2	1			
FSV-CR	1	1			
FSV-CS	1				
FSV-CU	1	2			2
FSV-CX	1	2	1	1	
FSV-DA	1	1		4	
FSV-DB	2	2		1	
FSV-DJ	4	3	1	1	1
FSV-DK	2	2			
FSV-DM	1	1			
FSV-DP	1		1		2
FSV-DS	3	3	2	2	3
FSV-DX	2	2	2		1
FSV-EH	2	2			
FSV-EI	2	1	1	2	
FSV-EL	2				
FSV-FY	1	1			
NISTa	1	1	1	1	1
NISTb	1	1	1	1	1
n	45	42	22	29	14

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

* Ratings based on sera 220-222 only, due to the extremely high trans-β-carotene concentration of serum 219.

"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 (serum heterogeniety and iter- and intra-laboratory) measurement standard deviation.
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

For details on the NIST Assigned quantities, see this Round Robin's "Report of (Meta)Analysis."

StS	% Observed					Expected
1	40	48	55	48	29	68.2 %
2	44	40	41	31	50	27.3 %
3	11	7	0	7	14	4.3 %
4	4	5	5	14	7	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 RR37

Each participant in RR37 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
- Zeaxanthin
- Lutein & Zeaxanthin

The following 8 pages are the "Individualized Report" for the analytes evaluated by participant FSV-BA.

Individualized Round Robin XXXVII Report to: FSV-BA

Your Data, NIST Assigned Values, and %Differences

	Serum 219				Serum 220				Serum 221				Serum 222			
Analyte	You	NAV	%Δ	n	You	NAV	%Δ	n	You	NAV	%Δ	n	You	NAV	%Δ	n
Retinol	.84	.79	5	45	.53	.49	8	45	.69	.67	4	44	.50	.48	6	45
Retinyl Palmitate	.01			10	.01			11	1.37	1.49	-8	13	.01	.11	-89	14
α-Tocopherol	7.24	7.65	-5	42	10.71	11.14	-4	42	16.18	17.20	-6	41	6.91	7.11	-3	42
γ-Tocopherol	8.37	8.18	2	22	3.77	3.72	1	22	1.87	1.88	-1	21	2.45	2.36	4	22
Total β-Carotene	3.76	3.79	-1	28	.37	.51	-27	28	.09	.08	9	27	.09	.07	29	28
trans-β-Carotene	3.66	3.47	5	14	.36	.46	-22	14	.08	.08	3	13	.08	.07	22	14
Total cis-β-Carotene	.10			9	.01			9	.01			6	.01			7
Total α-Carotene	.12	.12	-2	24	.22	.28	-22	24	.01	.01	0	21	.02	.01	7	21
trans-Lycopene	.16	.16	1	7	.17	.16	7	7	.16	.15	5	6	.32	.27	19	7
β-Cryptoxanthin	.09	.07	33	19	.09	.07	39	19	.08	.07	27	18	.09	.06	46	19
"Lutein&Zeaxanthin"	.19			17	.27			17	.19			16	.19			17

H2

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

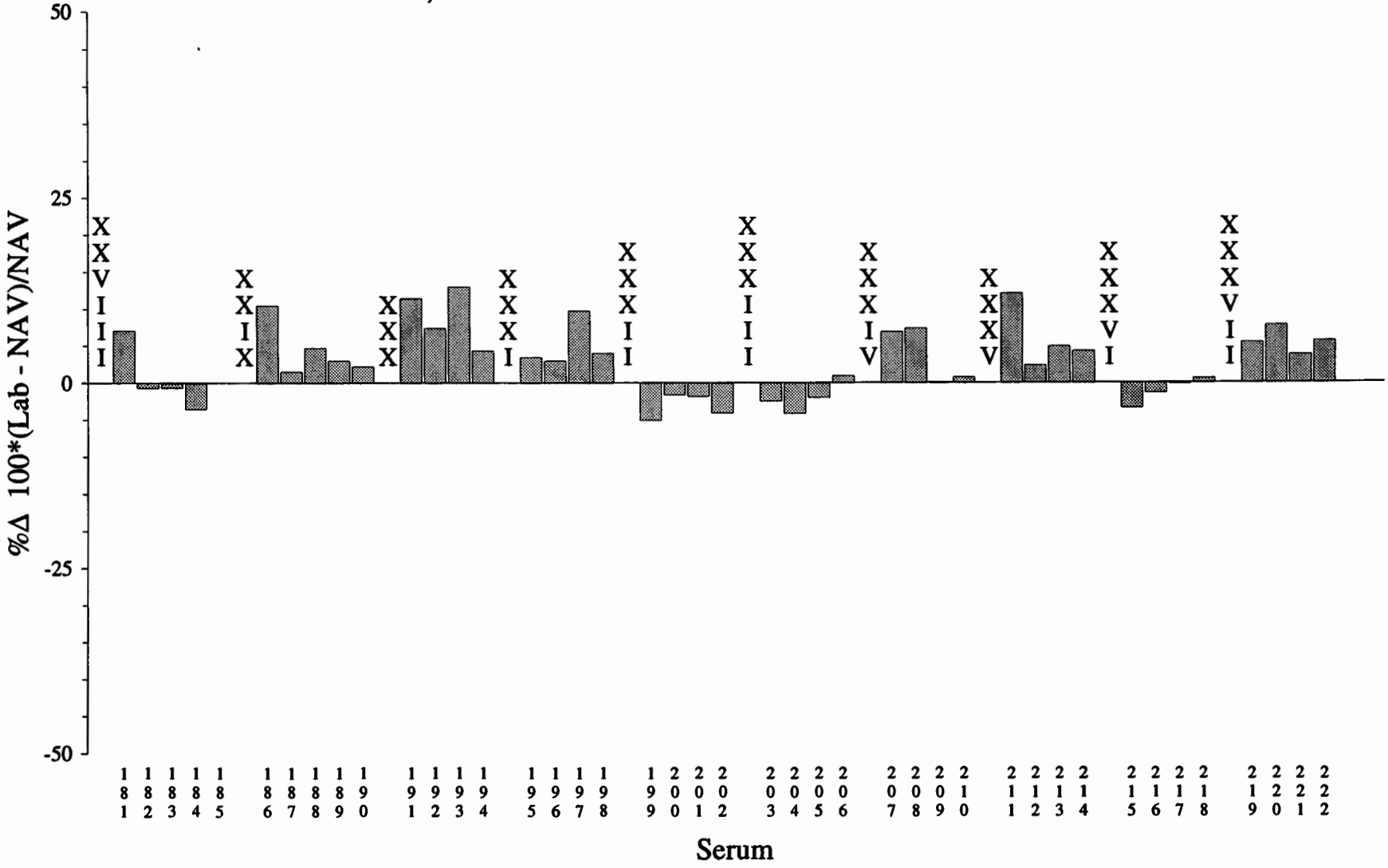
n : Number of non-NIST laboratories reporting quantitative values for this analyte in this serum

Please check our recorded values against your records.

Send corrections to: NNMMQAP 222/B208, NIST, Gaithersburg, MD 20899; fax 301-977-0685; email David.Duewer@NIST.gov

Individualized Round Robin XXXVII Report to: FSV-BA

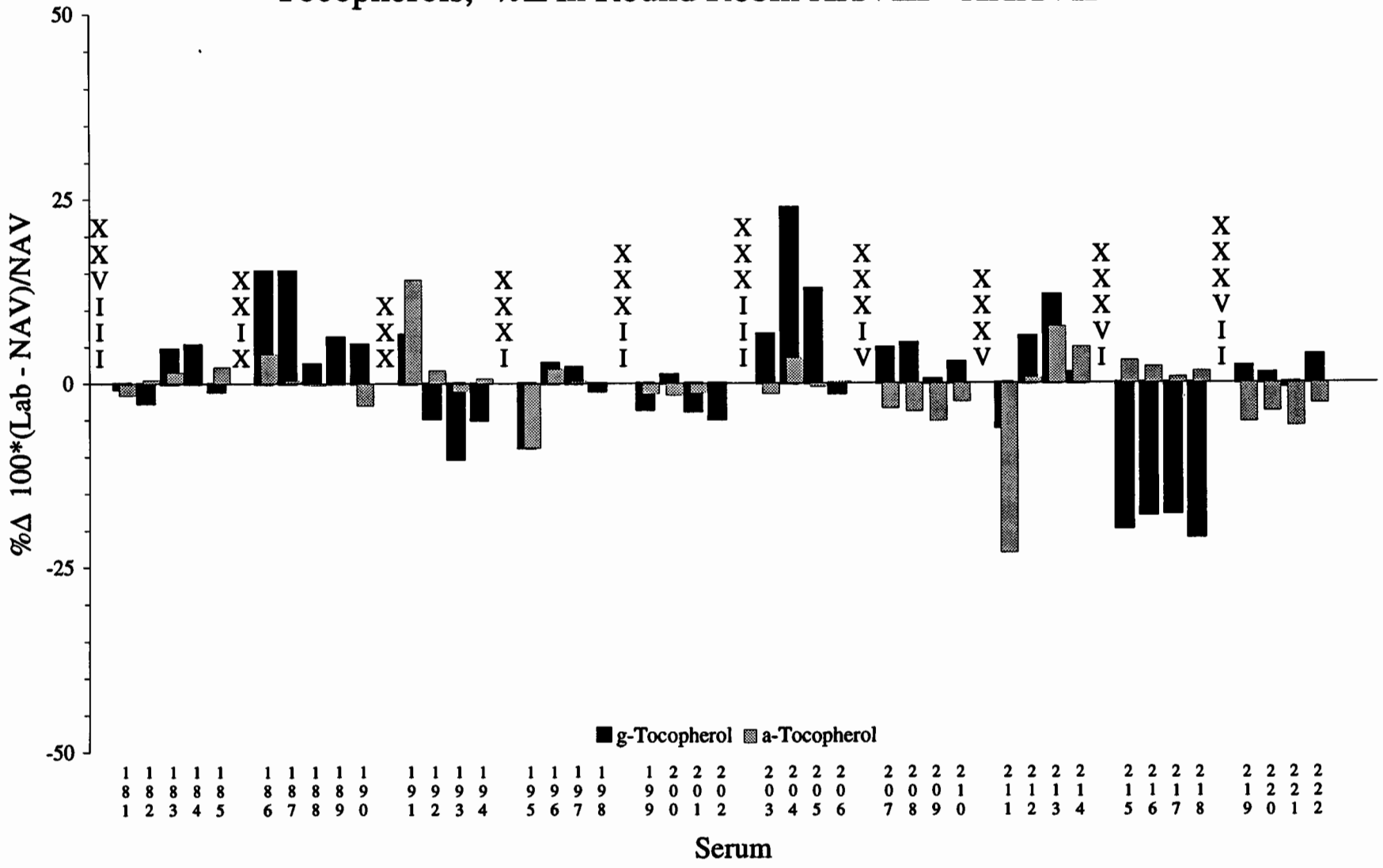
Retinol, %Δ in Round Robin XXVIII - XXXVII



H3

Individualized Round Robin XXXVII Report to: FSV-BA

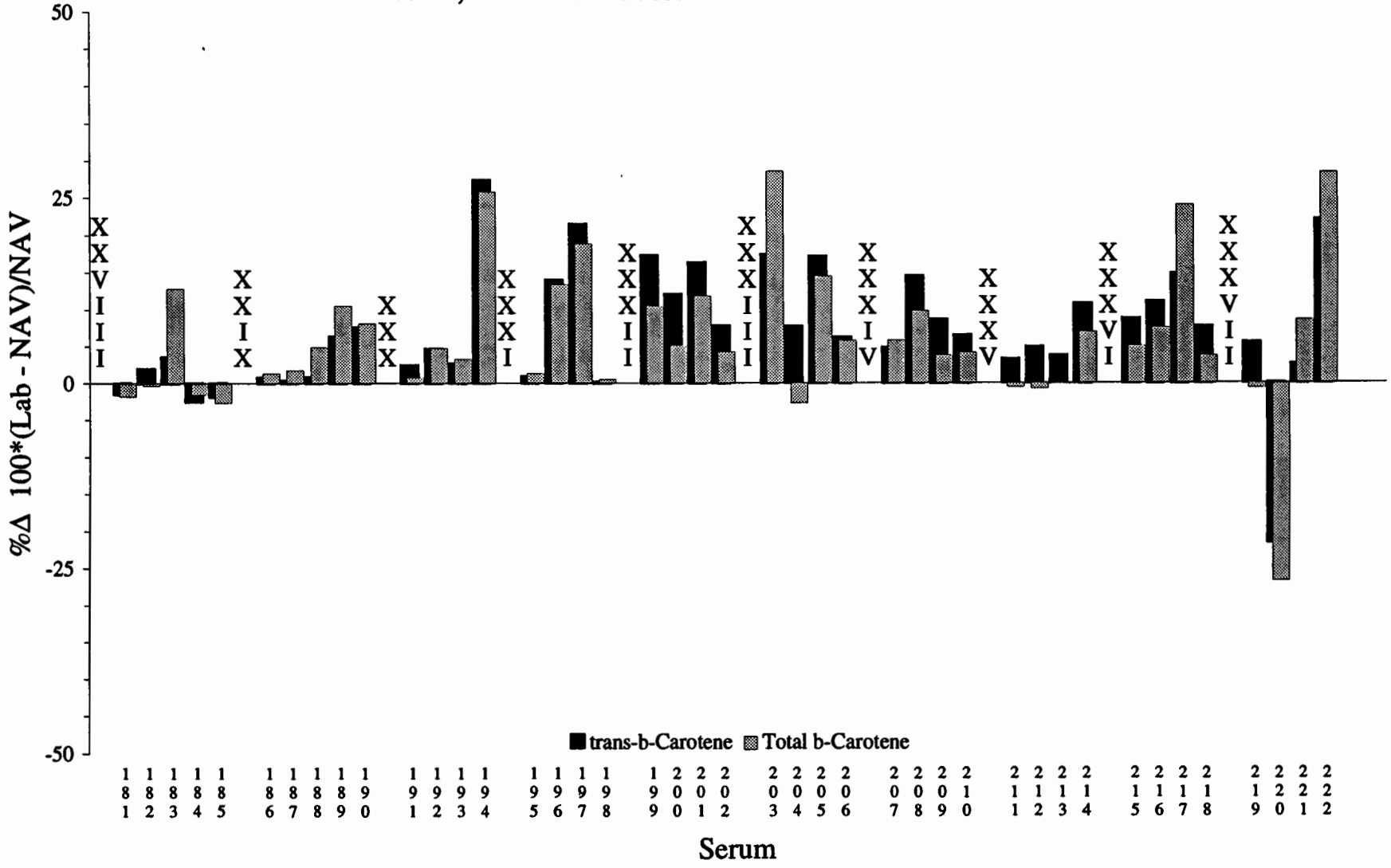
Tocopherols, %Δ in Round Robin XXVIII - XXXVII



H4

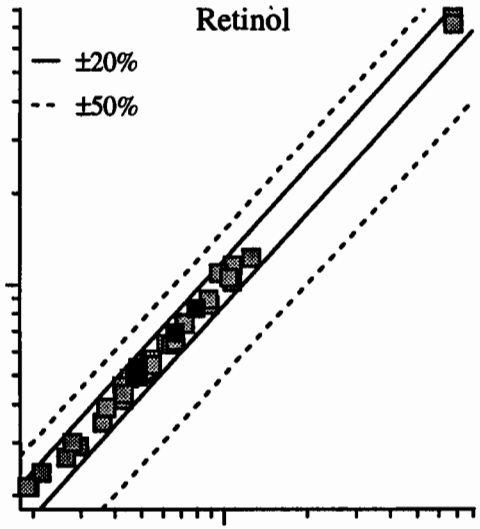
Individualized Round Robin XXXVII Report to: FSV-BA

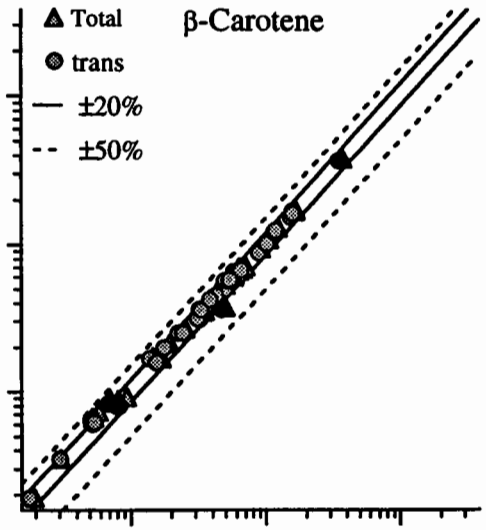
b-Carotene, %Δ in Round Robin XXVIII - XXXVII

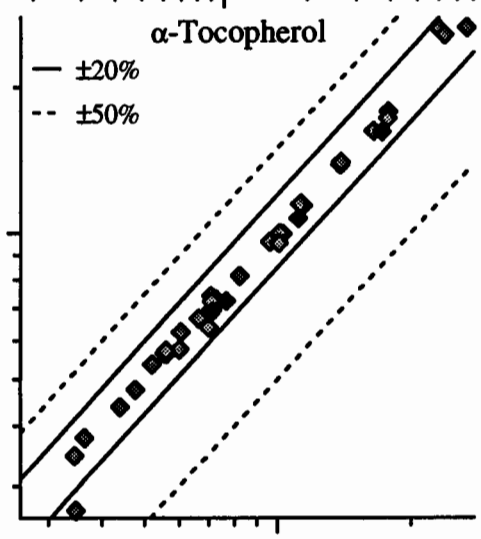


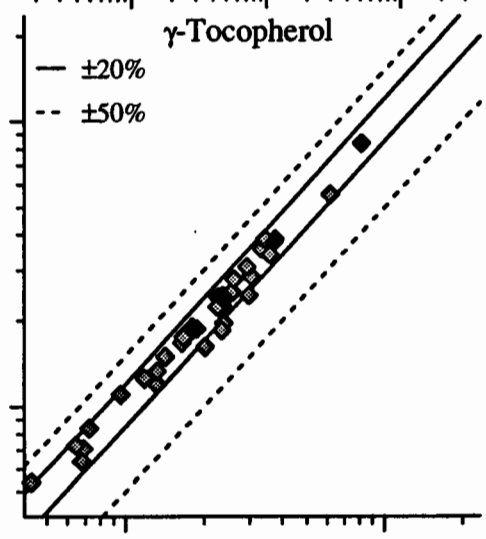
Individualized Round Robin XXXVII Report to: FSV-BA

NIST Assigned Values Vs Laboratory Values









Legend

Shaded Symbols: RR XXVIII-XXXVI
Black Symbols: RR XXXVII

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

Interpretation

Adequately intercomparable data are within ±20% lines. If you have data scattered outside ±50% lines, your measurement system is not consistent with those of most participating laboratories. If your data are systematically higher or lower than the NAV, your system may be consistent but your results are biased.

If your data show increased scatter at low concentrations, your "limit of quantification" may not be what you think it is.

If there are one or two "wild" outliers, they might be calculation or transcription errors. We would appreciate hearing from you about any such problems.

9H

Individualized Round Robin XXXVII Report to: FSV-BA

Accuracy/Precision Summary

	Ret		aToc		gToc		Total		trans	
	mΔ	vΔ	mΔ	vΔ	mΔ	vΔ	mΔ	vΔ	mΔ	vΔ
XXVIII	0	4	1	2	1	4	1	7	0	3
XXIX	4	4	0	3	9	6	5	4	3	3
XXX	9	4	4	7	-3	7	9	12	9	12
XXXI	5	3	-2	5	-1	5	9	9	9	10
XXXII	-3	2	-1	1	-3	3	8	4	13	4
XXXIII	-2	2	0	2	11	11	12	13	12	6
XXXIV	4	4	-4	1	3	2	6	3	9	4
XXXV	6	4	-2	14	3	8	1	4	6	3
XXXVI	-1	2	2	1	-19	2	10	9	11	3
XXXVII	6	2	-4	1	2	2	2	23	2	18

Legend

Ret	Retinol
aToc	α-Tocopherol
gToc	γ-Tocopherol
Total	Total β-Carotene
trans	trans-β-Carotene
mΔ	Mean difference, the average %Δ for all sera of a given RR, where %Δ = 100(Your value - NAV) / NAV
vΔ	Difference variability, one standard deviation of %Δ for all sera of a RR
NAV	NIST Assigned Value, our best estimate of analyte concentration... NAV = (NIST's average-of-averages + Round Robin median) / 2

(Traditional) Performance Criteria

The absolute value of %Δ of every measurement has traditionally been evaluated as follows...

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

More representative criteria need to be established, factoring in each serum's analyte level and the analyte distribution in adult human populations. Stay tuned, we're working on it...

Interpretation

Accuracy and precision are separate but kindred aspects of measurement comparison. We estimate accuracy as mΔ, the average %Δ, and precision as vΔ, the standard deviation of %Δ, for all sera of a Round Robin.

It's best to be accurate and precise (small mΔ, small vΔ)!

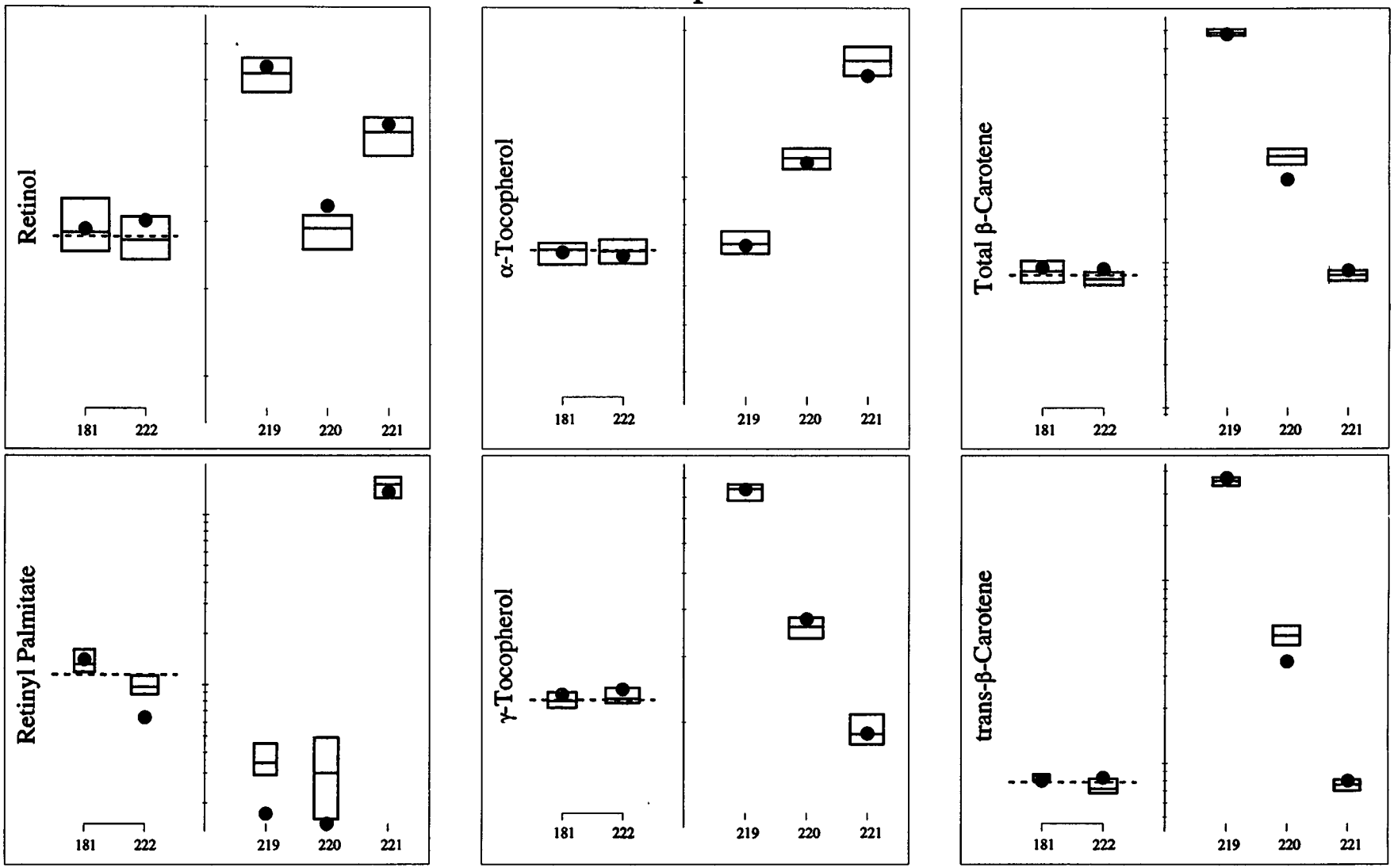
Good precision (small vΔ) with poor accuracy (large mΔ) is better than the converse: at least such values are internally consistent and may be relatable to others' values once the relative biases have been determined.

Poor precision (large vΔ) suggests that your measurement system is not in adequate control for the analyte levels examined.

H7

Individualized Round Robin XXXVII Report to: FSV-BA

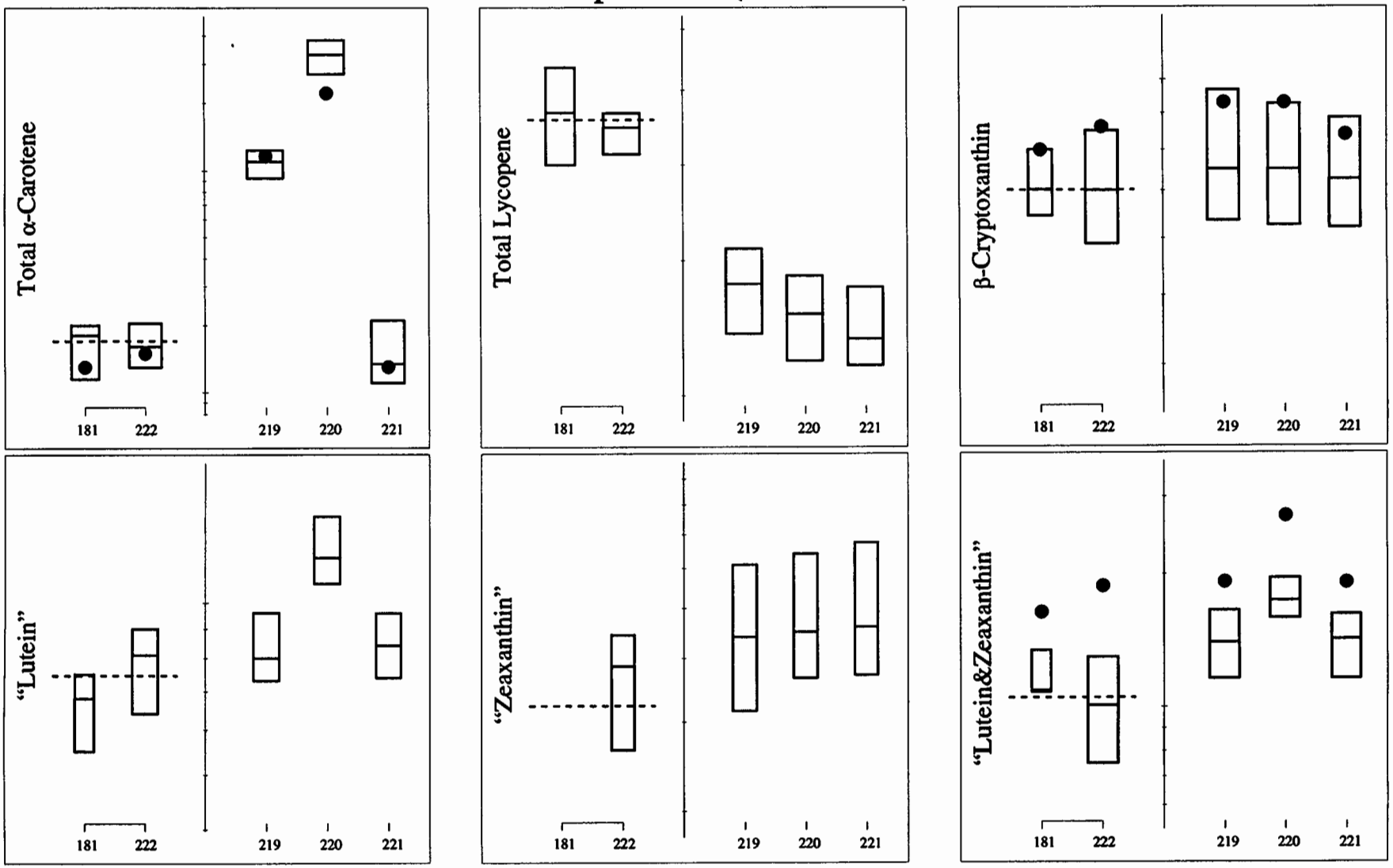
Comparisons



8H

Individualized Round Robin XXXVII Report to: FSV-BA

Comparisons (Continued)



6H

Appendix I. Shipping Package Inserts for RR38

The following two items were included in each package shipped to an RR38 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 9, 1996

Dear Colleague:

Enclosed is the set of samples for Round Robin XXXVIII. You will find one vial 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 detection limit, please indicate this result on the form by using ND *(Not Detected)*. For analytes not measured, please leave a blank. Results are due to NIST by September 20. 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 25.

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 which 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 HPLC systems creating analytical problems.) Pipette a known volume of serum from the vial for analysis since 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; β-carotene, 2560 at 450 nm; lycopene (in hexane), 3450 at 472 nm.

Please mail or FAX your results for Round Robin XXXVIII 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: jeanice.brown-thomas@nist.gov; or mail/FAX queries to the above address.

Sincerely,

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

cc: S. Wise

NIST

NIST/NCI

Micronutrients Measurement Quality Assurance Program

Round Robin **XXXVIII** Results from Laboratory #_____

	Serum				
Analyte	223	224	225	226	Units*
retinol					
retinyl palmitate					
α-tocopherol					
γ-tocopherol					
δ-tocopherol					
total β-carotene					
trans-β-carotene					
total cis-β-carotene					
total 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 20, 1996

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

Appendix J. Final Report for RR38

The following 19 pages are the final report for RR38 as provided to all participants:

- Cover letter
- A discussion entitled “Lies, Damn Lies, and Statistics” 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
- A “Report of (Meta)Analysis” that details the analysis of NIST measurements

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

November 6, 1996

Dear Colleague:

This report summarizes both overall and individual laboratory performance for the three round robin exercises conducted during 1996. Included in this report are: tabular summaries of data for Round Robins XXXVI, XXXVII, and XXXVIII; 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 about 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 two years.

In Round Robin XXXVI (Sera 215-218), all serum samples were previously distributed. Serum 215 was distributed as Serum 170 in Round Robin XXVI; Serum 216 was distributed as Serum 182 in Round Robin XXVIII; Serum 217 as Serum 194 in Round Robin XXX; and Serum 218 as Serum 199 in Round Robin XXXII. The overall laboratory performance for retinol, α- and γ-tocopherol, and β-carotene for this exercise was comparable to that of the overall interlaboratory performance over the past three years.

Round Robin XXXVII (Sera 219-222) consisted of three new sera. Serum 222 was previously distributed as Serum 181 in Round Robin XXVIII. Sera 219-222 contained augmented levels of retinol, retinyl palmitate, and/or δ-, γ-, and α-tocopherol. Results from this exercise indicated that the variation for β-carotene and retinyl palmitate in the sera with augmented levels of these analytes appeared to be similar to that of sera with low levels of β-carotene. In previous exercises where the levels of β-carotene were significantly lower (≤100 ng/mL), a higher variation was observed due to the difficulty of making measurements at levels that appeared to be at or below the limit of quantification for most laboratories.

Round Robin XXXVIII (Sera 223-226) consisted of all new sera. Sera 223 and 225 are blind duplicates of a serum pool with augmented levels of retinyl palmitate. Serum sample 224 (obtained from the same pool used to make Sera 223 and 225) contained augmented levels of retinol, retinyl palmitate, α- and γ-tocopherol, *trans*-α- and β-carotene, *trans*-lycopene, β-cryptoxanthin, lutein, and zeaxanthin. Serum 226 was a newly designed serum sample from a delipidized serum pool augmented with normal-to-high levels of all routinely measured analytes. This serum pool also contained trace amounts of dimethyl sulfoxide and cholesterol, which probably accounted for some reports of a gel-like consistency of the serum as well as observed particulate matter during extraction. Due to these observations, the results from this serum sample were not included in the overall laboratory performance for Round Robin XXXVIII or in the comparability summary (scorecard) in the attached report.

In Round Robin XXXVIII, the interlaboratory precision (excluding the results from Serum 226) was about 8% for retinol, 10% for γ-tocopherol, and about 6% for α-tocopherol. The average estimated coefficient of variation (eCV) for β-carotene was about 14%. The overall laboratory precision for retinyl palmitate and lycopene was about 39% and 19%, respectively. We will continue to evaluate ways to improve the interlaboratory comparability for these analytes in future studies.

Data for evaluating your laboratory's performance in Round Robin XXXVIII are provided in the comparability summary on page 6 of the 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.

As mentioned in a previous mailing, the participation fees for the 1997 QA program for non-NCI funded laboratories will be $400 for US labs and $900 for non-US labs. An invoice to that effect will be mailed in January. Also, as a reminder, to participate in the 1997 program your intent-to-participate form should be returned to us no later than **November 29, 1996.**

The 1997 QA Program will consist of three round robin exercises for the analysis of fat-soluble vitamins and carotenoids in serum, two exercises for the analysis of ascorbic acid in serum, and two exercises for the analysis of fat-soluble vitamins and carotenoids in food. The FY97 program thrusts will continue to focus on methods development for the measurement of difluoromethylornithine, epigallocatechin gallate, and 4-hydroxyphenylretinamide in serum. Other program thrusts will be addressed upon NCI's request.

The first set of samples for the fat-soluble vitamins in serum analysis will be distributed during the week of January 27. Results are due by March 21; written feedback will be provided to labs by April 18. The second set of samples will be shipped the week of April 28 with results due by June 13 and feedback to labs by July 25. The third set of samples will be shipped the week of July 28. Results will be due by September 19. Feedback will be provided to the laboratories around October 27.

The round robin studies for the measurement of fat-soluble vitamins and carotenoids in food will also be scheduled in April and July to coincide with the second and third serum round robin studies. The coordinator of these exercises is Dr. Katherine Sharpless (301/975-3121).

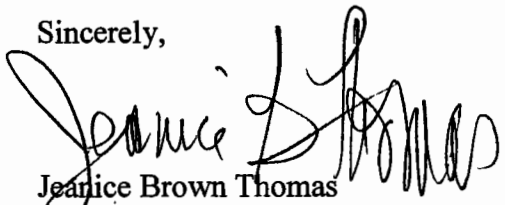
The first set of samples for the measurement of ascorbic acid in serum will be distributed in November 1996. The second set will be distributed in May. These round robin studies are being coordinated by Dr. Sam Margolis (301/975-3137).

The Fat-Soluble Vitamins and Carotenoid Analysis Tutorial session was held on October 25, 1996 at NIST. As in past years, the session was well-attended and provided a great opportunity for new laboratories to discuss in detail their measurement techniques.

Plans are still underway for hosting our next QA workshop at the American Association for Clinical Chemistry meeting, which will be held in Atlanta, Georgia during July 20-24, 1997. We will keep you informed of the workshop plans as they are finalized.

Certificates of participation in the FY96 QA Program will be distributed in January 1997. The homepage section for the QA program is still under construction. We anticipate that it will become available in January 1997. Lastly, for your convenience, we have enclosed reprints from some of our most recent publications. Please feel free to contact us for additional copies.

Sincerely,



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

Enclosures

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

"Lies, Damned Lies, and Statistics"

Benjamin Disraeli

The attached N^2M^2QAP Round Robin XXXVIII Report includes the standard "All Lab" listing of everyone's results, your "Individualized" results, and our "(Meta)Analysis" of value and uncertainty assignments.

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 four analytes reported by only one laboratory.
5b	A legend for the above two lists
6	The "Measurement Comparability Summary" (or "Score Card")

Page	"Individualized" Report Contents
1	Your values, our assigned values, and the %bias between the two
2-4	%Bias barchart for retinol, α- and γ-tocopherol, and total and *trans*-β-carotene for your last 3 years' results
5	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
6	Accuracy/Precision Summary, yet again for your last 3 years' results
7-8	"Comparisons" plots for retinol, retinyl palmitate, α- and γ-tocopherol, total and *trans*-β-carotene, total α-carotene, total lycopene, β-cryptoxanthin, lutein, zeaxanthin, and lutein & zeaxanthin

All samples distributed in this exercise were newly prepared. The samples labeled as sera #223 and #225 are blind duplicates, prepared from a retinyl palmitate-augmented but otherwise "normal" serum pool. Serum #224 started off as a rather boring low-carotenoid pool and was augmented with retinol, retinyl palmitate, α- and γ-tocopherol, *trans*-α- and *trans*-β-carotene, *trans*- lycopene, β-cryptoxanthin, lutein, and zeaxanthin!

Serum #226... sigh... was an attempt to create a totally "known" material starting from delipidized serum. While we did achieve (nearly) the intended *average* level for all analytes, we introduced considerable vial-to-vial heterogeneity. Many of you reported suspended solids: we suspect that the apparently incorporated cholesterol used as an analyte-carrier sometimes "unincorporated" during freeze-drying.... Maybe there was a little dimethyl sulfoxide left in some of the vials, too.... We learned a lot from the experiment, but we apologize for the troubles it caused you! While we report all the data for this serum, none of the serum #226 results are used in our various performance calculations.

We believe that the results for serum #224 document our ability to "build" a normal serum pool to rather high carotenoid levels. Using the graphical analysis previously described in the RRXXXVII report, Figures 1 and 2 document the estimated standard deviation for the RRXXXVIII sera relative to their expected values for retinol, total β-carotene, α-tocopherol, γ-tocopherol, total α-carotene, total lycopene, β-cryptoxanthin, and retinyl palmitate. In these figures, results from the first 4 years ('86-'89) of the N^2M^2QAP exercises are denoted "1", those from the '90-'92 exercises are denoted "2", and '93-'95 are denoted "3". The lines denote the best-fit sigmoidal curve to the past three years' data. The solid diamonds are samples #223, 224, and 225. With the exception of retinyl palmitate, the observed eSDs for these samples are about as expected from the historical relationships. The solid circles denote the sample #226 data: the excess eSD from sample heterogeneity is apparent for all analytes.

Note that the observed eSD for retinyl palmitate depends slightly, if at all, on the observed median concentration. While it *may* be possible to qualitatively determine whether or not an individual has taken retinyl palmitate supplements using current measurement techniques, the values reported for this analyte should not be regarded as even "semi-quantitative."

Why did we distribute the blind replicates, samples #223 and 225? Several reasons, but mostly that this is our way of getting the same type of "complete replicate measurements" from all participants. It also provides you a "pure" mechanism for demonstrating your same-day/instrument/analyst analytical precision (repeatability). (Contrast this with the "Score Card", where "accuracy" - or at least agreement with the rest of the participants - is the dominant consideration.) No matter what the actual analyte concentrations may be, you should get very similar results for two random samples drawn from the same (hopefully) homogenous pool. For a single analyte, $|value_1 - value_2|$ is an adequate metric. However, the standard deviation

$$SD_{12} = \sqrt{\frac{\sum_{i=1}^{2}\left(value_i - (value_1 + value_2)/2\right)^2}{2-1}} = \frac{|value_1 - value_2|}{\sqrt{2}}$$

is equally good and facilitates generalization to more than two replicates. When comparing across two or more analytes, it is convenient to express any *intra*laboratory measurement differences as a fraction of the observed *inter*laboratory eSD: $F = \frac{SD_{12}}{eSD}$.

Table 1 lists all the reported quantitative values for samples #223 and 225 for retinol, α-tocopherol, γ-tocopherol, and total β-carotene. (OK, I cheat: I've lumped some *trans*-β-carotene values in with the total β-carotene.) Table 1 also presents the individual analyte F values and a bounded average F for each participant: $F_{avg} = \sum_{i}^{n} \min(F_i, 4) \Big/ n$. The "min(F_i,4)"

insures that a single very unlikely difference will have the same influence (four interlaboratory eSDs) no matter *how* "very unlikely" the difference. The F_{avg} values are sorted from "best" to "least good". The number of individual analyte F values (n) contributing to F_{avg} is also listed. (A corollary to Murphy's Law - "Things that can go wrong, will go wrong." - is "The more things that can go wrong, the more things that will go wrong." Thus, a good F_{avg} with four analytes is "better" than the same F_{avg} with only one or two analytes.)

More than 80% of the F_{avg} values are 0.5 or smaller, indicating that the very large majority of you have excellent short-term control over your measurement processes. We believe that data transcription errors may account for some of the the larger F_{avg} values. In any case, the two largest values represent two quite distinct situations. Laboratory 62 has excellent repeatability for all analytes except total β-carotene, for which very different values were reported (0.16 and 0.40); since all analytes (except retinyl palmitate) were "native" in this serum, we believe the 0.40 value to be an isolated error. In contrast, all of the analyte values reported by Laboratory 113 for sample #225 are considerably larger than those reported for #223; this indicates a sample-wide problem such as reconstitution volume or internal standard quantification.

Of course, it is always possible (ahem) that a few of our samples are unusually different from the rest. Hence, you should neither take undue pride in a single very good F_{avg} value nor be unduly alarmed by a single "less good" F_{avg}. We will continue to periodically include such pairs as part of the N^2M^2QAP and we encourage you to use truly "blind" replicates as part of your internal quality control efforts. (And we are developing methods for indirectly estimating both short-term and long-term repeatabilities without necessarily using blind replicates.)

Oh, yes. You may have noted listings for "Lab 17.1". This participant submitted results for two methods, one established ("17") and one experimental ("17.1"). Both performed well, with the edge to the experimental! We're happy to help you evaluate new methods, but we do ask that you share the nature of the different submitted data sets with us. (If we don't have this information, we average the values to give a single result.)

Your comments and suggestions are welcome. If you discover any errors in our recording or interpretation of your data, please let us know!

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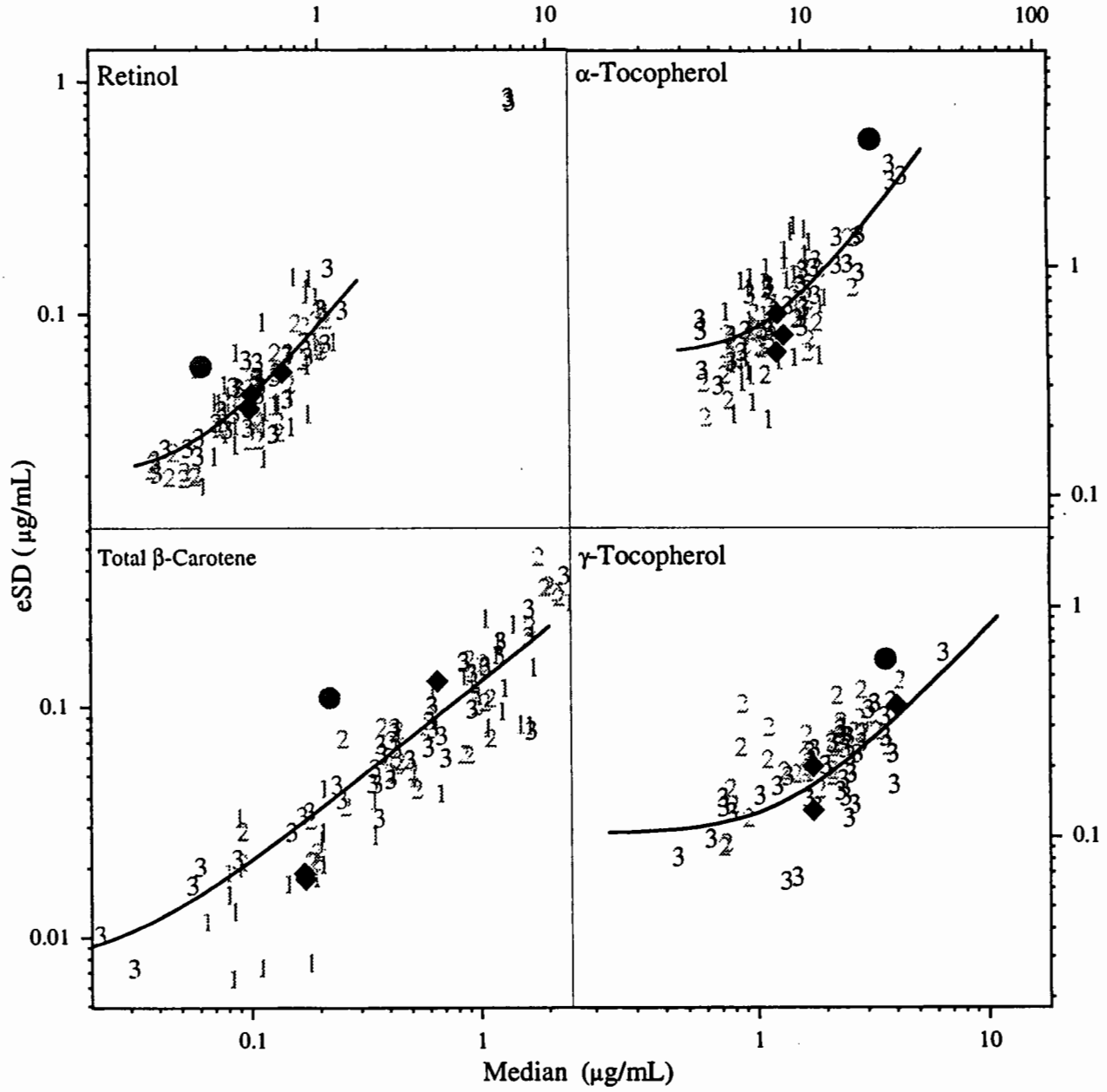


Figure 1

Observed RR XXXVIII eSDs Relative to Their Expected Values for Retinol, Total β-Carotene, α-Tocopherol, and γ-Tocopherol

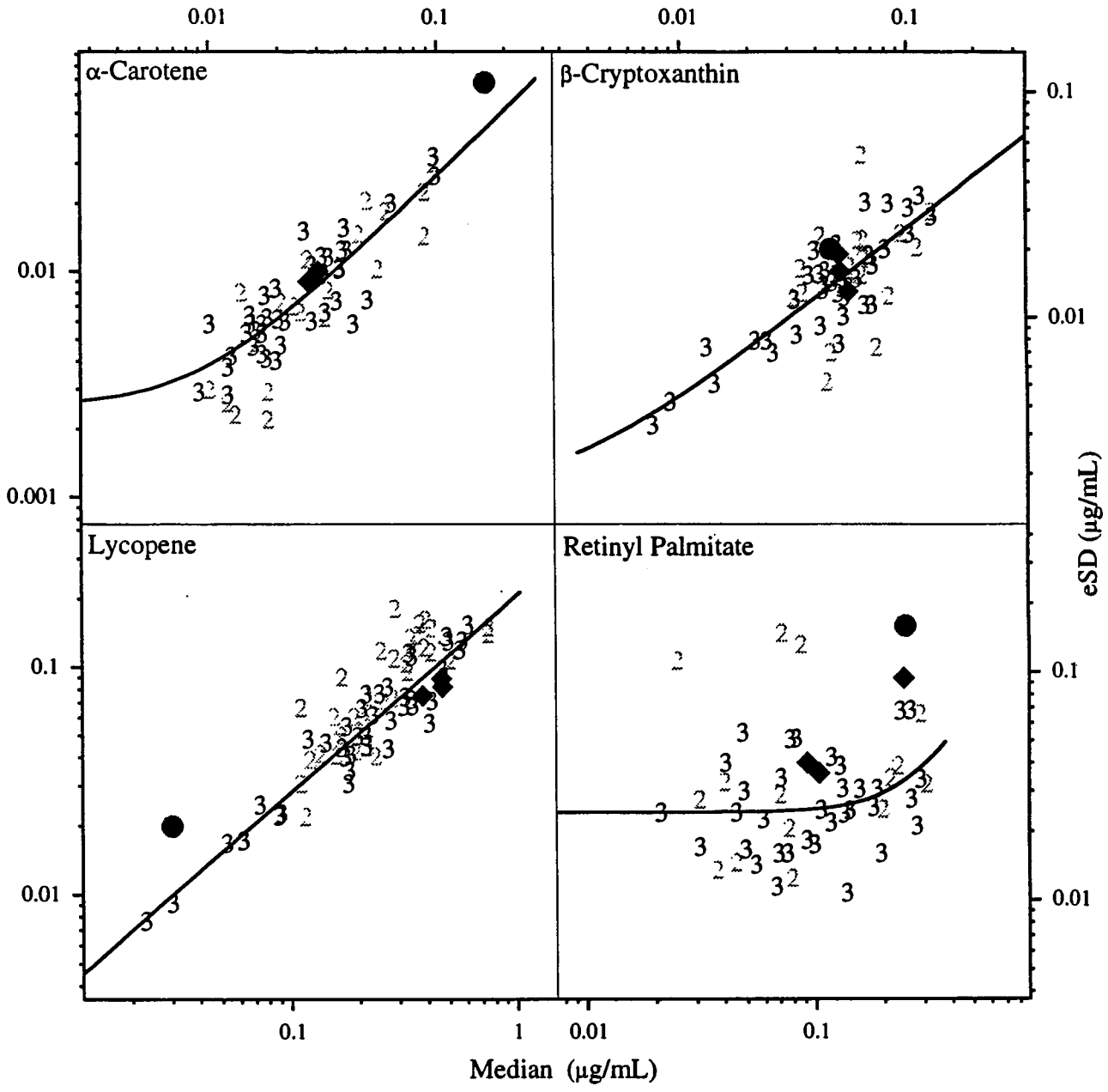


Figure 2

Observed RR XXXVIII eSDs Relative to Their Expected Values
for Total a-Carotene, Total Lycopene, b-Cryptoxanthin, and Retinyl Palmitate

Table 1. Short-Term Repeatabilities from Sample #223 and 225 Blind Replicates

Lab	Retinol			α-Tocopherol			γ-Tocopherol			Total β-Carotene		
	223	225	F	223	225	F	223	225	F	223	225	F
	0.53	0.53	0.00	7.65	7.51	0.16	1.67	1.65	0.09	0.175	0.170	0.19
	0.52	0.50	0.22	8.14	7.94	0.25	1.64	1.58	0.25	0.193	0.183	0.38
	0.54	0.54	0.02	7.91	7.79	0.15	1.72	1.69	0.10	0.197	0.194	0.11
	0.49	0.48	0.13	7.34	7.59	0.29						
	0.49	0.49	0.00	8.00	7.80	0.24				0.173	0.178	0.19
	0.56	0.55	0.17	7.97	7.80	0.20	1.68	1.74	0.26	0.169	0.156	0.50
	0.44	0.44	0.08	7.31	7.30	0.01				0.087	0.067	0.76
	0.50	0.49	0.02	7.69	7.64	0.06	1.93	1.90	0.13	0.186	0.184	0.08
	0.44	0.49	0.79	8.00	7.85	0.18	1.76	1.70	0.26	0.170	0.171	0.04
	0.41	0.43	0.37	5.94	6.00	0.07	1.52	1.56	0.17	0.141	0.161	0.77
	0.56	0.55	0.17	7.91	7.88	0.04	1.58	1.58	0.00	0.160	0.400	4.00
				7.70	7.34	0.42	1.50	1.20	1.29	0.180	0.186	0.23
				7.81	7.89	0.09						
	0.52	0.54	0.48	9.04	9.91	1.02						
	0.50	0.55	0.86	6.87	7.70	0.98	1.57	1.81	0.99	0.155	0.173	0.69
	0.55	0.54	0.02	8.67	8.86	0.22						
	0.48	0.48	0.03	7.64	7.60	0.05				0.090	0.085	0.19
	0.46	0.50	0.67	8.89	8.77	0.14	1.75	1.88	0.56	0.139	0.153	0.54
	0.49	0.50	0.13	8.30	8.42	0.14				0.171	0.167	0.15
	0.35	0.39	0.74	7.34	7.35	0.01	1.37	1.55	0.77	0.130	0.170	1.53
	0.51	0.53	0.34	7.97	8.00	0.04	1.55	1.59	0.17	0.230	0.220	0.38
	0.45	0.43	0.24	7.37	7.07	0.35						
	0.60	0.62	0.24	8.64	8.85	0.25				0.166	0.169	0.11
	0.55	0.55	0.00	7.74	7.70	0.05	1.62	1.59	0.13	0.150	0.150	0.00
	0.45	0.63	3.03	6.74	9.45	3.19	1.64	2.28	2.74	0.146	0.203	2.18
	0.52	0.59	1.13							0.210	0.210	0.00
	0.53	0.53	0.07	7.39	7.12	0.32				0.166	0.178	0.46
	0.53	0.52	0.19	8.01	8.14	0.15				0.117	0.124	0.27
	0.33	0.30	0.37	6.08	6.26	0.21				0.145	0.136	0.34
	0.50	0.52	0.45	7.64	8.18	0.64	1.69	1.83	0.61	0.156	0.163	0.25
	0.52	0.53	0.17									
	0.52	0.54	0.34	7.50	7.10	0.47						
	0.77	0.75	0.34	9.80	10.60	0.94						
	0.44	0.47	0.47	8.10	8.30	0.24						
	0.49	0.50	0.17	7.50	7.50	0.00						
	0.51	0.43	1.26	7.59	7.14	0.54				0.155	0.155	0.00
	0.48	0.48	0.00	7.54	8.50	1.13	1.03	0.96	0.30	0.157	0.160	0.11
										0.175	0.176	0.03
	0.51	0.51	0.00	8.00	8.10	0.12	1.95	2.04	0.39	0.172	0.177	0.19
	0.51	0.51	0.03									
	0.50	0.51	0.17									
	0.42	0.40	0.37	8.50	7.34	1.37	1.74	1.54	0.86	0.158	0.150	0.31
	0.44	0.45	0.10	9.24	8.99	0.29	1.71	1.71	0.00	0.228	0.212	0.61
	0.49	0.50	0.12	7.24	7.41	0.20						
	0.47	0.48	0.17	7.58	7.14	0.52	2.05	2.00	0.21	0.220	0.210	0.38
	0.47	0.47	0.03	8.30	8.20	0.12						
	0.50	0.50	0.07									
	0.53	0.55	0.34	7.44	7.34	0.12				0.190	0.190	0.00
	0.49	0.53	0.66	7.57	7.91	0.41				0.169	0.163	0.23
			Avg			Avg			Avg			Avg
NSV	0.51	0.51	0.51	7.93	7.88	7.91	1.74	1.74	1.74	0.170	0.172	0.171
NSU	0.04	0.05	0.04	0.58	0.62	0.60	0.13	0.20	0.17	0.019	0.018	0.019

Combined		
Lab	n	F_{avg}
	1	0.03
	1	0.03
	4	0.04
	1	0.07
	4	0.07
	2	0.08
	2	0.08
	3	0.09
	1	0.09
	4	0.10
	4	0.11
	2	0.12
	3	0.14
	3	0.14
	3	0.15
	2	0.16
	1	0.17
	1	0.17
	4	0.17
	3	0.20
	3	0.20
	2	0.21
	4	0.23
	4	0.25
	4	0.27
	3	0.28
	4	0.28
	3	0.29
	2	0.29
	3	0.31
	4	0.32
	4	0.32
	4	0.34
	2	0.35
	4	0.39
	2	0.40
	3	0.43
	4	0.48
	4	0.48
	2	0.57
	3	0.60
	2	0.64
	3	0.65
	4	0.73
	2	0.75
	4	0.76
	4	0.88
	4	1.05
	4	2.79

From: David L. Duewer Date: October 30, 1996

To: Jeanice Brown Thomas, Margaret Kline, Willie E. May, Katherine E. Sharpless, Gary W. Kramer, Dennis J. Reeder, Stephen A. Wise

Re: Value and Uncertainty Assignment for the NIST/NCI Micronutrient Measurement Quality Assurance Program's Round Robin XXXVIII (RR38) Sera: 223 to 226.

Background: Four samples representing three newly prepared sera were distributed in RR38. (While distributing at least one "old" sera in each RR is desirable, we have insufficient quantity of "interesting" materials to be compulsive about it.) All three of the new sera were designed to test one or more aspects of our "designer serum" program for the cost-effective construction of the next generation of SRM® 968.

The samples labeled #223 and #225 are blind duplicates from a single preparation of a retinyl palmitate-augmented serum. Serum #224 was constructed from the "normal" serum pool used for the RR37 sera (#219, 220, and 221), augmented with retinol, retinyl palmitate, α-tocopherol, γ-tocopherol, δ-tocopherol, β-carotene,α-carotene, lycopene, zeaxanthin, and lutein. Serum #226 is a "completely artificial" construct, created from a lipid-stripped serum pool augmented with the above commonly reported analytes.

Both NIST1 and NIST3 analysts reported residual solids in one-or-more vials of serum, as did many RR38 participants. Two participants reported a gel-like consistency for the reconstituted serum. Several analyte carriers were used in the construction of serum #226, including dimethyl sulfoxide and cholesterol.

Results: Table 1 presents the NIST data, summary statistics for the NIST data, summary results for RR38, and the NIST assigned values and uncertainties. Table 2 presents a compact summary of the assigned values and uncertainties for each analyte in each serum. Because of presumptive heterogeneity, all values for serum #226 are presented in *italics*. 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 XXXVIII 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 $0.741 \times$ InterQuartile Range (IQR)
  - SD_{labs} $\sqrt{eSD_n^2 - SD_{NIST}^2}$, the residual non-NIST interlaboratory biases after correction for measurement-, sample-, and NIST-analyst-related sources of variance. When SD_{NIST} is greater than eSD_n, $SD_{labs} = 0$.
  - CV_{labs} $100 \times SD_{labs}/Median_n$

- NIST Assigned Values and Uncertainties
  - NAV (Mean + $Median_n$) / 2, our best guess of the "true" analyte level
  - NAU Maximum$(0.05 \times NAV, \sqrt{SD_{NIST}^2 + SD_{labs}^2})$, our best guess for the "true" interlaboratory standard deviation characterizing measurement, sample heterogeneity, interanalyst, and interlaboratory sources of variation. When SD_{labs} could not be determined, NAU is estimated as Maximum$(0.10 \times NAV, \sqrt{2\ SD_{NIST}^2})$.
  - CV $100 \times NAU / NAV$

Conclusions: The blind replicate samples, labeled as sera #223 and #225, have nearly identical NAVs - the expected result for blind duplicates! The NAUs for the two samples are also similar, although those for #225 are generally slightly larger: this may indicate an analysis-order effect. The historical data may be able to shed some light on this possibility. In any case, the NAUs for sera #223/225 and #224 are typical of those previously observed for the NAV levels of each analyte. (The "Lies, Damn Lies, and Statistics" report for this RR will include discussions of short-term repeatability and the historical relationship between analyte level and eSD.)

The NAUs for sera #226 are considerably larger than expected, with a large heterogeneity component. This is doubtless related to the reported "undissolved solids" (cholesterol carrier that escaped filtration?) None of the reported values for this serum will be used in any of the laboratory performance metrics. [As a statistical aside, the values for serum #226 were also not used in evaluating the between-analyst standard deviation, SD_{anl}. Since samples #223 and #225 are duplicates of the same serum, the regression-determined values for this variance component are not as well determined as is typical. The low R^2 value for α-tocopherol is a consequence of the similar levels for this analyte in serum #223/225 and #224.] Serum #226 should NOT be distributed again! The remaining vials should be discarded.

Although it does not appear that we can construct a completely "known concentration" serum, the successful augmentation of various carotenoids in sera #223/225 and #224 indicates that we do now have the technology to convert "routine" to "high" carotenoid levels in our serum pools.

This memo will be included as part of the Round Robin XXXVIII report.

Table 1
NIST Data and Calculations

Retinol

	NIST1				NIST3			
	223	224	225	226	223	224	225	226
A:1	0.522	0.694	0.475	*0.319*	0.496	0.606		*0.314*
A:2	0.521	0.663	0.553	*0.339*	0.512	0.613	0.502	*0.326*
B:1	0.542	0.661	0.535	*0.380*	0.514	0.660	0.518	*0.325*
B:2	0.581	0.699	0.506	*0.350*	0.491	0.642	0.510	*0.317*
C:1	0.553	0.717	0.562	*0.332*	0.489	0.611	0.497	*0.319*
C:2	0.573	0.730	0.529	*0.355*	0.495	0.607	0.491	*0.315*
n_x	6	6	6	*6*	6	6	5	*6*
$Mean_x$	0.549	0.694	0.527	*0.346*	0.500	0.623	0.503	*0.319*
SD_x	0.025	0.028	0.032	*0.021*	0.011	0.023	0.011	*0.005*
SD_{repx}	0.018	0.021	0.037	*0.017*	0.012	0.008	0.004	*0.006*
SD_{betx}	0.024	0.026	0.017	*0.018*	0.006	0.024	0.010	*0.002*
SD_{NISTx}	0.030	0.033	0.040	*0.025*	0.013	0.026	0.011	*0.006*
CV_{NISTx}	5.4	4.7	7.6	*7.3*	2.7	4.1	2.2	*1.9*

	NIST			
n	12	12	11	*12*
Mean	0.524	0.658	0.515	*0.333*
SD_{rep}	0.016	0.017	0.026	*0.013*
SD_{bet}	0.013	0.020	0.011	*0.022*
SD_{anl}	0.015	0.015	0.015	*0.015*
SD_{NIST}	0.026	0.030	0.032	*0.030*
CV_{NIST}	4.9	4.5	6.2	*8.9*

NIST3=a+b*NIST1

a: 0.092 ±0.067
b: 0.762 ±0.114
R^2: 0.957

	RRXXXVIII			
	223	224	225	226
n_n	44	44	44	*43*
$Median_n$	0.498	0.691	0.506	*0.308*
eSD_n	0.039	0.056	0.045	*0.059*
SD_{labs}	0.030	0.047	0.031	*0.051*
CV_{labs}	6.0	6.8	6.2	*16*
NAV	0.511	0.675	0.510	*0.320*
NAU	0.039	0.056	0.045	*0.059*
CV	7.7	8.3	8.8	*18*

<— Current Results —>

<—— Assignments ——>

Retinyl Palmitate

	NIST1				NIST3			
	223	224	225	226	223	224	225	226
A:1	0.149	0.308	0.147	*0.33*				
A:2	0.153	0.245	0.146	*0.31*				
B:1	0.154	0.274	0.142	*0.30*			0.161	*0.08*
B:2	0.152	0.304	0.145	*0.35*	0.155	0.316	0.134	*0.08*
C:1	0.147	0.292	0.151	*0.34*	0.153	0.342	0.166	*0.13*
C:2	0.146	0.297	0.149	*0.34*	0.153	0.320	0.150	*0.11*
n_x	6	6	6	*6*	3	3	4	*4*
$Mean_x$	0.150	0.287	0.147	*0.33*	0.153	0.326	0.153	*0.10*
SD_x	0.003	0.024	0.003	*0.02*	0.001	0.014	0.014	*0.03*
SD_{repx}	0.002	0.029	0.002	*0.02*	0.000	0.011	0.016	*0.01*
SD_{betx}	0.003	0.009	0.003	*0.01*			0.007	*0.03*
SD_{NISTx}	0.004	0.030	0.004	*0.02*			0.017	*0.03*
CV_{NISTx}	2.5	10	2.5	*7.1*			11	*31*

	NIST			
	9	9	10	*10*
	0.152	0.306	0.150	*0.21*
	0.001	0.022	0.012	*0.02*
	0.003	0.010	0.005	*0.15*
	0.003	0.003	0.003	*0.00*
	0.004	0.024	0.013	*0.15*
	2.9	8.0	8.9	*72*

NIST3=a+b*NIST1

a: -0.032 ±0.005
b: 1.249 ±0.024
R^2: 0.999

	RRXXXVIII			
	223	224	225	226
	14	14	14	*14*
	0.092	0.244	0.104	*0.25*
	0.040	0.094	0.036	*0.16*
	0.040	0.091	0.034	*0.06*
	43	37	32	*23*
	0.122	0.275	0.127	*0.23*
	0.040	0.094	0.036	*0.16*
	33	34	29	*71*

Table 1
NIST Data and Calculations

α-Tocopherol

	NIST1				NIST3			
	223	224	225	*226*	223	224	225	*226*
A:1	8.76	8.19	7.89	*18.5*	8.13	8.71		*19.7*
A:2	8.37	8.17	8.95	*19.2*	7.50	8.63	7.39	*17.4*
B:1	7.88	8.55	7.82	*20.1*	8.20	8.89	7.82	*18.3*
B:2	8.26	8.84	7.92	*18.7*	7.92	8.70	7.60	*18.3*
C:1	8.67	8.75	8.31	*16.7*	8.50	9.09	8.04	*19.4*
C:2	7.62	8.67	8.08	*19.5*	8.21	8.82	8.00	*19.1*
n_x	6	6	6	*6*	6	6	5	*6*
$Mean_x$	8.26	8.53	8.16	*18.8*	8.08	8.81	7.77	*18.7*
SD_x	0.44	0.29	0.42	*1.2*	0.34	0.17	0.27	*0.8*
SD_{repx}	0.48	0.12	0.44	*1.3*	0.30	0.14	0.09	*0.9*
SD_{hetx}	0.27	0.30	0.28	*0.6*	0.27	0.14	0.31	*0.5*
SD_{NISTx}	0.55	0.33	0.52	*1.5*	0.41	0.20	0.33	*1.1*
CV_{NISTx}	6.7	3.8	6.4	*7.7*	5.0	2.3	4.2	*5.6*

	NIST			
n	12	12	11	*12*
Mean	8.17	8.67	7.97	*18.8*
SD_{rep}	0.40	0.12	0.33	*1.1*
SD_{het}	0.24	0.29	0.33	*0.5*
SD_{anl}	0.34	0.34	0.34	*0.3*
SD_{NIST}	0.58	0.47	0.58	*1.3*
CV_{NIST}	7.1	5.4	7.3	*6.9*

NIST3=a+b*NIST1

a: 0
b: 0.99 ±0.02
R^2: 0.338

	RRXXXVIII			
	223	224	225	*226*
n_n	41	41	41	*40*
$Median_n$	7.70	8.28	7.79	*19.7*
eSD_n	0.42	0.50	0.62	*3.6*
SD_{labs}	0	0.16	0.23	*3.3*
CV_{labs}	0	2.0	3.0	*17*
NAV	7.93	8.47	7.88	*19.2*
NAU	0.58	0.50	0.62	*3.6*
CV	7.4	5.9	7.9	*19*

<— Current Results —>

<—— Assignments ——>

γ-Tocopherol

	NIST1				NIST3			
	223	224	225	*226*	223	224	225	*226*
A:1	1.71	3.79	1.77	*4.05*	1.83	4.32		*3.63*
A:2	1.74	3.72	1.90	*4.30*	1.79	4.33	1.76	*3.38*
B:1	1.83	3.67	1.74	*3.92*	1.80	4.25	1.82	*3.39*
B:2	1.82	4.06	1.84	*4.48*	1.85	4.27	1.79	*3.38*
C:1	1.81	4.15	1.77	*3.75*	1.86	4.27	1.82	*3.58*
C:2	1.88	4.39	1.84	*3.75*	1.85	4.23	1.82	*3.57*
n_x	6	6	6	*6*	6	6	5	*6*
$Mean_x$	1.80	3.96	1.81	*4.04*	1.83	4.28	1.80	*3.49*
SD_x	0.06	0.28	0.06	*0.30*	0.03	0.04	0.03	*0.12*
SD_{repx}	0.03	0.19	0.07	*0.25*	0.03	0.02	0.01	*0.10*
SD_{hetx}	0.06	0.27	0.02	*0.25*	0.03	0.04	0.03	*0.10*
SD_{NISTx}	0.07	0.33	0.08	*0.36*	0.04	0.05	0.03	*0.14*
CV_{NISTx}	4.1	8.4	4.2	*8.8*	2.0	1.1	1.8	*4.1*

	NIST			
	12	12	11	*12*
	1.81	4.12	1.81	*3.76*
	0.03	0.13	0.05	*0.19*
	0.05	0.20	0.03	*0.52*
	0.03	0.03	0.03	*0.03*
	0.07	0.24	0.07	*0.55*
	3.7	5.9	3.7	*15*

NIST3=a+b*NIST1

a: -0.24 ±0.05
b: 1.14 ±0.02
R^2: 1.000

RRXXXVIII			
223	224	225	*226*
20	20	20	*19*
1.66	3.89	1.67	*3.38*
0.13	0.37	0.20	*0.59*
0.11	0.28	0.19	*0.22*
6.7	7.1	11	*6.4*
1.74	4.00	1.74	*3.57*
0.13	0.37	0.20	*0.59*
7.5	9.2	11	*17*

Table 1
NIST Data and Calculations

δ-Tocopherol

	NIST1				NIST3			
	223	224	225	*226*	223	224	225	*226*
A:1		0.840			0.203	0.801		*0.44*
A:2		0.784			0.196	0.845	0.223	*0.40*
B:1		0.826			0.168	0.808	0.188	*0.41*
B:2		0.798			0.176	0.824	0.172	*0.44*
C:1		0.791			0.209	0.870	0.212	*0.48*
C:2		0.802			0.202	0.842	0.196	*0.47*
n_x	0	6	0	*0*	6	6	5	*6*
$Mean_x$		0.807			0.192	0.832	0.198	*0.44*
SD_x		0.022			0.016	0.026	0.020	*0.03*
SD_{repx}		0.026			0.005	0.022	0.009	*0.02*
SD_{hetx}		0.009			0.018	0.021	0.021	*0.03*
SD_{NISTx}		0.027			0.018	0.031	0.023	*0.04*
CV_{NISTx}		3.4			9.6	3.7	12	*8.2*

Total β-Carotene

	NIST1				NIST3			
	223	224	225	*226*	223	224	225	*226*
A:1	0.180	0.59	0.186	*0.29*	0.174	0.74		*0.11*
A:2	0.179	0.64	0.180	*0.30*	0.169	0.68	0.164	*0.12*
B:1	0.182	0.67	0.184	*0.32*	0.168	0.72	0.163	*0.10*
B:2	0.180	0.65	0.181	*0.29*	0.167	0.70	0.163	*0.12*
C:1	0.180	0.60	0.178	*0.27*	0.170		0.169	*0.09*
C:2	0.181	0.62	0.175	*0.29*	0.168	0.69	0.167	*0.08*
n_x	6	6	6	*6*	6	5	5	*6*
$Mean_x$	0.180	0.63	0.181	*0.29*	0.169	0.71	0.165	*0.10*
SD_x	0.001	0.03	0.004	*0.02*	0.003	0.03	0.003	*0.02*
SD_{repx}	0.001	0.02	0.003	*0.02*	0.002	0.03	0.001	*0.01*
SD_{hetx}	0.001	0.03	0.004	*0.01*	0.002	0.01	0.003	*0.02*
SD_{NISTx}	0.001	0.03	0.005	*0.02*	0.003	0.03	0.003	*0.02*
CV_{NISTx}	0.7	5.5	2.6	*6.9*	1.9	4.2	1.8	*18*

δ-Tocopherol

	NIST			
n	6	12	5	*6*
Mean	0.192	0.819	0.198	*0.44*
SD_{rep}	0.005	0.023	0.009	*0.02*
SD_{het}	0.018	0.021	0.021	*0.03*
SD_{anl}	0.007	0.007	0.007	*0.01*
SD_{NIST}	0.020	0.033	0.024	*0.04*
CV_{NIST}	10	4.0	12	*8.8*

NIST3=a+b*Median

a: 0
b: 1.105 ±0.009
R^2: 0.999

Total β-Carotene

	NIST			
	12	11	11	*12*
	0.175	0.67	0.173	*0.20*
	0.002	0.03	0.002	*0.01*
	0.002	0.02	0.004	*0.11*
	0.003	0.00	0.003	*0.00*
	0.004	0.03	0.005	*0.11*
	2.4	5.1	3.0	*57*

NIST3=a+b*NIST1

a: -0.050 ±0.003
b: 1.204 ±0.009
R^2: 1.000

δ-Tocopherol

	RRXXXVIII			
	223	224	225	*226*
n_n	4	4	4	*4*
$Median_n$	0.175	0.755	0.170	*0.45*
eSD_n	0.103	0.098	0.102	*0.12*
SD_{labs}	0.101	0.093	0.099	*0.11*
CV_{labs}	58	12	58	*25*
NAV	0.184	0.787	0.184	*0.44*
NAU	0.103	0.098	0.102	*0.12*
CV	56	12	55	*27*

<— Current Results —>

<—— Assignments ——>

Total β-Carotene

	RRXXXVIII			
	223	224	225	*226*
	28	28	28	*27*
	0.166	0.63	0.171	*0.24*
	0.019	0.13	0.018	*0.08*
	0.019	0.12	0.017	*0*
	11	19	10	*0*
	0.170	0.65	0.172	*0.22*
	0.019	0.13	0.018	*0.11*
	11	19	10	*51*

Table 1
NIST Data and Calculations

trans-β-Carotene

	NIST1				NIST3			
	223	224	225	*226*	223	224	225	*226*
A:1	0.168	0.504	0.175	*0.19*	0.174	0.675		*0.11*
A:2	0.172	0.551	0.170	*0.19*	0.169	0.643	0.164	*0.12*
B:1	0.176	0.574	0.175	*0.22*	0.168	0.683	0.163	*0.10*
B:2	0.176	0.584	0.177	*0.22*	0.167	0.674	0.163	*0.12*
C:1	0.172	0.577	0.166	*0.25*	0.170	0.641	0.169	*0.09*
C:2	0.174	0.525	0.164	*0.25*	0.168	0.633	0.167	*0.08*
n_x	6	6	6	*6*	6	6	5	*6*
$Mean_x$	0.173	0.553	0.171	*0.22*	0.169	0.658	0.165	*0.10*
SD_x	0.003	0.032	0.005	*0.03*	0.003	0.022	0.003	*0.02*
SD_{repx}	0.002	0.029	0.002	*0.00*	0.002	0.014	0.001	*0.01*
SD_{betx}	0.003	0.026	0.006	*0.03*	0.002	0.021	0.003	*0.02*
SD_{NISTx}	0.004	0.039	0.006	*0.03*	0.003	0.025	0.003	*0.02*
CV_{NISTx}	2.0	7.0	3.6	*15*	1.9	3.8	1.8	*18*

	NIST			
n	12	12	11	*12*
Mean	0.171	0.605	0.168	*0.16*
SD_{rep}	0.002	0.023	0.002	*0.01*
SD_{het}	0.003	0.025	0.005	*0.07*
SD_{anl}	0.001	0.001	0.001	*0.00*
SD_{NIST}	0.004	0.034	0.005	*0.07*
CV_{NIST}	2.2	5.6	3.2	*46*

NIST3=a+b*NIST1

a: -0.055 ±0.001
b: 1.291 ±0.004
R^2: 1.000

	RRXXXVIII			
	223	224	225	*226*
n_n	11	11	11	*11*
$Median_n$	0.160	0.588	0.163	*0.28*
eSD_n	0.017	0.091	0.018	*0.12*
SD_{labs}	0.016	0.085	0.017	*0.09*
CV_{labs}	10	14	10	*32*
NAV	0.166	0.597	0.166	*0.22*
NAU	0.017	0.091	0.018	*0.12*
CV	10	15	11	*53*

<— Current Results —>

<—— Assignments ——>

Total α-Carotene

	NIST1				NIST3			
	223	224	225	*226*	223	224	225	*226*
		0.397		*0.200*	0.030	0.496		*0.065*
		0.454		*0.188*	0.033	0.467	0.051	*0.053*
		0.449		*0.192*	0.031	0.488	0.037	*0.056*
		0.423		*0.186*	0.032	0.474	0.025	*0.067*
		0.395		*0.180*	0.026	0.475		*0.051*
		0.401		*0.197*	0.033	0.480	0.036	*0.050*
	0	6	0	*6*	6	6	4	*6*
		0.420		*0.191*	0.031	0.480	0.037	*0.057*
		0.027		*0.007*	0.003	0.010	0.011	*0.007*
		0.026		*0.009*	0.003	0.013	0.005	*0.007*
		0.020		*0.003*	0.001	0.002		*0.006*
		0.032		*0.009*	0.004	0.013		*0.009*
		7.7		*4.9*	11	2.8		*15*

	NIST			
	6	12	4	*12*
	0.031	0.450	0.037	*0.124*
	0.001	0.021	0.007	*0.009*
	0.001	0.002	0.011	*0.006*
	0.002	0.002	0.002	*0.002*
	0.003	0.021	0.013	*0.010*
	8.9	4.7	34	*8.4*

NIST3=a+b*Median

a: 0.002 ±0.002
b: 1.114 ±0.006
R^2: 1.000

	RRXXXVIII			
	223	224	225	*226*
	23	25	23	*24*
	0.027	0.429	0.030	*0.164*
	0.009	0.080	0.010	*0.068*
	0.009	0.077	0	*0.068*
	32	18	0	*41*
	0.029	0.439	0.034	*0.144*
	0.009	0.080	0.013	*0.068*
	32	18	38	*48*

Table 1
NIST Data and Calculations

trans-α-Carotene

	NIST1				NIST3			
	223	224	225	226	223	224	225	226
A:1					0.03	0.5		*0.07*
A:2					0.03	0.5	0.03	*0.05*
B:1					0.03	0.5	0.02	*0.06*
B:2					0.03	0.5	0.03	*0.07*
C:1					0.03	0.5	0.03	*0.05*
C:2					0.03	0.5	0.03	*0.05*
n_x	0	0	0	*0*	6	6	5	*6*
$Mean_x$					0.03	0.5	0.03	*0.06*
SD_x					0.00	0.0	0.00	*0.01*
SD_{repx}					0.00	0.0	0.00	*0.01*
SD_{hetx}					0.00	0.0	0.00	*0.01*
SD_{NISTx}					0.00	0.0	0.00	*0.01*
CV_{NISTx}					11	2.4	17	*15*

Total Lycopene

	NIST1				NIST3			
	223	224	225	226	223	224	225	226
A:1					0.611	0.497		
A:2					0.573	0.495	0.624	
B:1					0.632	0.543	0.596	
B:2					0.574	0.509	0.535	
C:1					0.569	0.482	0.590	
C:2					0.580	0.494	0.590	
n_x	0	0	0	*0*	6	6	5	*0*
$Mean_x$					0.590	0.503	0.587	
SD_x					0.026	0.021	0.032	
SD_{repx}					0.029	0.014	0.025	
SD_{hetx}					0.014	0.020	0.029	
SD_{NISTx}					0.032	0.025	0.038	
CV_{NISTx}					5.4	4.9	6.5	

	NIST			
n	6	6	5	*6*
Mean	0.03	0.5	0.03	*0.06*
SD_{rep}	0.00	0.0	0.00	*0.01*
SD_{het}	0.00	0.0	0.00	*0.01*
SD_{anl}				
SD_{NIST}				
CV_{NIST}				

	NIST			
	6	6	5	*0*
	0.590	0.503	0.587	
	0.037	0.019	0.035	
	0.014	0.020	0.029	
	0.002	0.002	0.002	
	0.040	0.028	0.046	
	6.7	5.6	7.8	

NIST3=a+b*Median

a: 0.123 ±0.015
b: 1.003 ±0.035
R^2: 0.998

	RRXXXVIII					RRXXXVIII			
	223	224	225	226	<— Current Results —>	223	224	225	226
n_n	0	0	0	*0*		26	26	26	*22*
$Median_n$						0.467	0.380	0.461	*0.03*
eSD_n						0.082	0.075	0.089	*0.02*
SD_{labs}						0.072	0.069	0.076	
CV_{labs}						15	18	16	
NAV	0.03	0.5	0.03	*0.06*	<—— Assignments ——>	0.528	0.441	0.524	
NAU						0.082	0.075	0.089	
CV						16	17	17	

Table 1
NIST Data and Calculations

trans-Lycopene

	NIST1				NIST3			
	223	224	225	*226*	223	224	225	*226*
A:1					0.234	0.321		
A:2					0.221	0.306	0.221	
B:1					0.233	0.330	0.233	
B:2					0.233	0.322	0.228	
C:1					0.237	0.313	0.239	
C:2					0.225	0.310	0.229	
n_x	0	0	0	*0*	6	6	5	*0*
$Mean_x$					0.231	0.317	0.230	
SD_x					0.006	0.009	0.007	
SD_{repx}					0.007	0.007	0.005	
SD_{hetx}					0.003	0.008	0.007	
SD_{NISTx}					0.008	0.011	0.008	
CV_{NISTx}					3.3	3.4	3.6	

	NIST			
n	6	6	5	*0*
Mean	0.231	0.317	0.230	
SD_{rep}	0.005	0.008	0.003	
SD_{het}	0.003	0.008	0.007	
SD_{anl}	0.000	0.000	0.000	
SD_{NIST}	0.006	0.011	0.007	
CV_{NIST}	2.6	3.5	3.2	

NIST3=a+b*Median

a: -0.225 ±0.003
b: 1.808 ±0.009
R^2: 1.000

	RRXXXVIII			
	223	224	225	*226*
n_n	7	7	7	*7*
$Median_n$	0.252	0.300	0.252	*0.04*
eSD_n	0.053	0.046	0.039	*0.02*
SD_{labs}	0.052	0.045	0.038	
CV_{labs}	21	15	15	
NAV	0.241	0.309	0.241	
NAU	0.053	0.046	0.039	
CV	22	15	16	

<— Current Results —>

<—— Assignments ——>

β-Cryptoxanthin

	NIST1				NIST3			
	223	224	225	*226*	223	224	225	*226*
A:1					0.051	0.052		*0.028*
A:2					0.051	0.048	0.047	*0.023*
B:1					0.049	0.053	0.049	*0.024*
B:2					0.048	0.053	0.048	*0.029*
C:1					0.046	0.049	0.048	*0.023*
C:2					0.046	0.049	0.050	*0.028*
n_x	0	0	0	*0*	6	6	5	*6*
$Mean_x$					0.049	0.050	0.049	*0.026*
SD_x					0.002	0.002	0.001	*0.003*
SD_{repx}					0.000	0.002	0.001	*0.003*
SD_{hetx}					0.003	0.002	0.001	*0.001*
SD_{NISTx}					0.003	0.003	0.001	*0.003*
CV_{NISTx}					5.3	5.2	2.0	*13*

	NIST			
	6	6	5	*6*
	0.049	0.050	0.049	*0.026*
	0.000	0.002	0.000	*0.003*
	0.003	0.002	0.001	*0.001*
	0.000	0.000	0.000	*0.000*
	0.003	0.003	0.001	*0.003*
	5.3	5.2	2.0	*13*

NIST3=a+b*Median

a: 0.035 ±0.003
b: 0.260 ±0.059
R^2: 0.905

	RRXXXVIII			
	223	224	225	*226*
	17	17	17	*16*
	0.054	0.060	0.052	*0.067*
	0.016	0.013	0.019	*0.020*
	0.016	0.012	0.019	*0.020*
	30	21	36	*29*
	0.051	0.055	0.050	*0.046*
	0.016	0.013	0.019	*0.020*
	32	23	37	*43*

Table 1
NIST Data and Calculations

"Lutein"

	NIST1				NIST3			
	223	224	225	*226*	223	224	225	*226*
A:1					0.117	0.137		*0.139*
A:2					0.125	0.133	0.118	*0.135*
B:1					0.117	0.137	0.116	*0.131*
B:2					0.120	0.137	0.117	*0.131*
C:1					0.117	0.134	0.117	*0.138*
C:2					0.115	0.135	0.117	*0.141*
n_x	0	0	0	*0*	6	6	5	*6*
$Mean_x$					0.118	0.136	0.117	*0.136*
SD_x					0.004	0.002	0.001	*0.004*
SD_{repx}					0.004	0.002	0.001	*0.002*
SD_{hetx}					0.003	0.001	0.001	*0.005*
SD_{NISTx}					0.005	0.002	0.001	*0.005*
CV_{NISTx}					3.8	1.5	0.8	*3.7*

"Zeaxanthin"

	NIST1				NIST3			
	223	224	225	*226*	223	224	225	*226*
A:1					0.048	0.093		*0.092*
A:2					0.051	0.092	0.045	*0.090*
B:1					0.046	0.093	0.045	*0.089*
B:2					0.049	0.095	0.046	*0.091*
C:1					0.045	0.090	0.044	*0.090*
C:2					0.041	0.092	0.044	*0.095*
n_x	0	0	0	*0*	6	6	5	*6*
$Mean_x$					0.047	0.092	0.045	*0.091*
SD_x					0.003	0.002	0.001	*0.002*
SD_{repx}					0.003	0.001	0.001	*0.003*
SD_{hetx}					0.003	0.002	0.001	*0.001*
SD_{NISTx}					0.004	0.002	0.001	*0.003*
CV_{NISTx}					8.7	2.1	2.1	*3.0*

	NIST			
n	6	6	5	*6*
Mean	0.118	0.136	0.117	*0.136*
SD_{rep}	0.004	0.002	0.001	*0.002*
SD_{het}	0.003	0.001	0.001	*0.005*
SD_{anl}	0.004	0.004	0.004	*0.004*
SD_{NIST}	0.006	0.004	0.004	*0.006*
CV_{NIST}	5.0	3.2	3.3	*4.5*

NIST3=a+b*Median

a: 0
b: 0.919 ±0.016
R^2: 0.753

	NIST			
	6	6	5	*6*
	0.047	0.092	0.045	*0.091*
	0.002	0.001	0.001	*0.001*
	0.003	0.002	0.001	*0.001*
	0.002	0.002	0.002	*0.002*
	0.005	0.003	0.003	*0.003*
	10	3.4	6.2	*3.3*

NIST3=a+b*Median

a: 0.021 ±0.003
b: 0.878 ±0.058
R^2: 0.991

	RRXXXVIII			
	223	224	225	*226*
n_n	13	13	13	*12*
$Median_n$	0.125	0.147	0.132	*0.135*
eSD_n	0.012	0.030	0.014	*0.051*
SD_{labs}	0.010	0.029	0.014	*0.051*
CV_{labs}	8.2	20	10	*38*
NAV	0.122	0.141	0.124	*0.135*
NAU	0.012	0.030	0.014	*0.051*
CV	9.8	21	11	*38*

<— Current Results —>

<—— Assignments ——>

	RRXXXVIII			
	223	224	225	*226*
	10	10	10	*9*
	0.028	0.082	0.030	*0.080*
	0.012	0.009	0.016	*0.031*
	0.011	0.008	0.015	*0.031*
	40	10	52	*39*
	0.037	0.087	0.037	*0.086*
	0.012	0.009	0.016	*0.031*
	32	10	42	*36*

Table 2
Summary of NIST Assigned Values and Uncertainties

	Blind Replicates											
	223			225			224			*226*		
Analyte	NAV	NAU	CV	NAV	NAU	CV	NAV	NAU	CV	*NAV*	*NAU*	*CV*
Retinol	0.511	0.039	7.7	0.510	0.045	8.8	0.675	0.056	8.3	*0.320*	*0.059*	*18*
Retinyl Palmitate	0.122	0.040	33	0.127	0.036	29	0.275	0.094	34	*0.23*	*0.16*	*71*
α-Tocopherol	7.93	0.58	7.4	7.88	0.62	7.9	8.47	0.50	5.9	*19.2*	*3.6*	*19*
γ-Tocopherol	1.74	0.13	7.5	1.74	0.20	11	4.00	0.37	9.2	*3.57*	*0.59*	*17*
δ-Tocopherol	0.184	0.103	56	0.184	0.102	55	0.787	0.098	12	*0.44*	*0.12*	*27*
Total β-Carotene	0.170	0.019	11	0.172	0.018	10	0.65	0.13	19	*0.22*	*0.11*	*51*
trans-β-Carotene	0.166	0.017	10	0.166	0.018	11	0.597	0.091	15	*0.22*	*0.12*	*53*
Total α-Carotene	0.029	0.009	32	0.034	0.013	38	0.439	0.080	18	*0.144*	*0.068*	*48*
trans-α-Carotene	0.03			0.03			0.5			*0.06*		
Total Lycopene	0.528	0.082	16	0.524	0.089	17	0.441	0.075	17			
trans-Lycopene	0.241	0.053	22	0.241	0.039	16	0.309	0.046	15			
β-Cryptoxanthin	0.051	0.016	32	0.050	0.019	37	0.055	0.013	23	*0.046*	*0.020*	*43*
"Lutein"	0.122	0.012	9.8	0.124	0.014	11	0.141	0.030	21	*0.135*	*0.051*	*38*
"Zeaxanthin"	0.037	0.012	32	0.037	0.016	42	0.087	0.009	10	*0.086*	*0.031*	*36*

Appendix K. "All-Lab Report" for RR38

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 XXXVIII Laboratory Results

Values in µg/mL

	Total Retinol				Retinyl Palmitate				α-Tocopherol				γ-Tocopherol			
Lab	223	224	225	226	223	224	225	226	223	224	225	226	223	224	225	226
FSV-BA	0.495	0.700	0.494	!0.342	0.155	0.24	0.130	!0.169	7.69	8.42	7.64	!19.26	1.93	4.37	1.90	!3.7
FSV-BD	0.490	0.620	0.490	!0.301					8.00	8.10	7.80	!19.8				
FSV-BF	0.563	0.766	0.553	!0.332					7.97	8.37	7.80	!17.26	1.68	3.87	1.74	!3.02
FSV-BG	0.537	0.715	0.536	!0.363	0.073	0.24	0.070	!0.218	7.91	8.50	7.79	!23.09	1.72	4.06	1.69	!3.96
FSV-BGa	0.517	0.697	0.504	!0.362	0.077	0.27	0.078	!0.244	8.14	8.61	7.94	!23.24	1.64	3.62	1.58	!3.51
FSV-BH	0.439	0.623	0.486	!0.233	0.051	0.22	0.053	!0.322	8.00	8.54	7.85	!20.58	1.76	3.98	1.70	!3.56
FSV-BI	0.526	0.726	0.526	!0.272	0.128	0.36	0.121	!0.299	7.65	8.24	7.51	!16.66	1.67	3.75	1.65	!2.84
FSV-BJ	0.501	0.730	0.552	!0.39	0.082	0.20	0.076	!0.166	6.87	8.17	7.70	!25.27	1.57	4.02	1.81	!4.70
FSV-BK	0.545	0.743	0.544	!0.231					8.67	9.54	8.86	!17.73				
FSV-BL	0.516	0.687	0.544	!0.345					9.04	9.91	9.91	!22.83				
FSV-BM	0.441	0.542	0.469	!0.285					8.10	10.00	8.30	!21.7				
FSV-BN	0.550	0.760	0.550	!0.35	0.100	0.33	0.110	!0.49	7.74	8.21	7.70	!21.27	1.62	3.75	1.59	!3.27
FSV-BO	0.533	0.730	0.521	!0.235					8.01	8.27	8.14	!12.10				
FSV-BP	0.601	0.838	0.615	!0.407					8.64	9.54	8.85	!24.28				
FSV-BQ	0.520	0.700	0.540	!0.33					7.50	7.70	7.10	!16				
FSV-BR	0.520	0.720	0.530	!0.26												
FSV-BS	0.522	0.668	0.589	!0.269												
FSV-BT	0.496	0.728	0.522	!0.640	0.127	0.37	0.119	!0.633	7.64	8.65	8.18	!16.22	1.69	3.92	1.83	!2.84
FSV-BU																
FSV-BV	0.420	0.523	0.398	!0.232					8.50	8.39	7.34	!20.69	1.74	3.91	1.54	!3.38
FSV-BW	0.470	0.650	0.480	!0.27	0.035	0.13	0.039	!0.066	7.58	8.02	7.14	!17.58	2.05	4.75	2.00	!3.64
FSV-BX	0.560	0.740	0.550						7.91	8.40	7.88		1.58	3.49	1.58	
FSV-BZ									7.70	8.30	7.34	!21.7	1.50	3.40	1.20	!2.9
FSV-CA	0.489	0.655	0.481	!0.141					7.34	8.01	7.59	!19.56				
FSV-CB	0.448	0.607	0.434	!0.268					7.37	7.64	7.07	!22.89				
FSV-CC	0.493	0.527	0.500	!0.149					7.24	7.85	7.41	!9.22				
FSV-CF	0.470	0.735	0.472	!0.284					8.30	8.90	8.20	!16.9				
FSV-CH	0.460	0.690	0.500	!0.3					8.89	9.36	8.77	!19.77	1.75	4.29	1.88	!3.43
FSV-CK	0.407	0.547	0.428	!0.236	0.084	0.15	0.102	!0.073	5.94	6.17	6.00	!14.62	1.52	3.14	1.56	!2.72
FSV-CL	0.304	0.467	0.285	!0.178					3.44	4.20	3.37	!9.645	0.97	2.13	0.99	!1.81
FSV-CM									7.81	8.28	7.89	!21.26				
FSV-CP									8.74	9.41	8.95	!18.31	1.89	4.49	1.97	!3.37
FSV-CQ	0.438	0.574	0.443	!0.304					7.31	7.94	7.30	!18.82				
FSV-CR	0.490	0.690	0.500	!0.31					7.50	8.30	7.50	!21.6				
FSV-CS	0.496	0.720	0.500	!0.311												
FSV-CT	0.529	0.692	0.533	!0.131					7.39	7.90	7.12	!17.07				
FSV-CU	0.480	0.686	0.482	!0.339	0.129	0.18	0.129	!0.08	7.64	7.90	7.60	!15.28				
FSV-CX	0.510	0.700	0.530	!0.45	0.150	0.33	0.130	!0.25	7.97	8.55	8.00	!26.13	1.55	3.46	1.59	!4.05
FSV-DA	0.510	0.670	0.510	!0.31	0.102	0.25	0.106	!0.438	8.00	8.65	8.10	!20.7	1.95	4.43	2.04	!3.75
FSV-DJ	0.770	0.890	0.750	!0.39					9.80	11.50	10.60	!28.4				
FSV-DK	0.480	0.690	0.480	!0.34	0.040	0.12	0.040	!0.41	7.54	8.20	8.50	!23.9	1.03	2.80	0.96	!2.8
FSV-DM	0.487	0.663	0.495	!0.351					8.30	8.62	8.42	!20.74				
FSV-DP	0.505	0.684	0.507	!0.305												
FSV-DR	0.494	0.673	0.533						7.57	7.80	7.91					
FSV-DS	0.326	0.432	0.304	!0.322					6.08	6.39	6.26	!16.78				
FSV-DU	0.530	0.790	0.550	!0.38					7.44	7.87	7.34	!16.81				
FSV-DX	0.505	0.701	0.430	!0.308					7.59	7.89	7.14	!17.95				
FSV-EA	0.350	0.522	0.394	!0.192					7.34	8.00	7.35	!18.2	1.37	3.84	1.55	!3.05
FSV-EH	0.440	0.685	0.446	!0.553	0.055	0.10	0.092	!0.087	9.24	8.69	8.99	!20.33	1.71	3.56	1.71	!3.12
FSV-EI	0.450	0.675	0.630	!0.186					6.74	7.63	9.45	!10.48	1.64	4.14	2.28	!2.00
FSV-EL	0.500	0.740	0.510	!0.31												
n	47	47	47	0	15	15	15	0	45	45	45	0	23	23	23	0
Min	0.304	0.432	0.285		0.035	0.10	0.039		3.44	4.20	3.37		0.97	2.13	0.96	
Median	0.496	0.690	0.504		0.084	0.24	0.102		7.70	8.28	7.80		1.67	3.87	1.69	
Max	0.770	0.890	0.750		0.155	0.37	0.130		9.80	11.50	10.60		2.05	4.75	2.28	
eSD	0.039	0.057	0.047		0.049	0.13	0.039		0.47	0.55	0.68		0.13	0.46	0.20	
eCV	8	8	9		58	53	38		6	7	9		8	12	12	
NISTa	0.549	0.694	0.527	!0.345	0.150	0.29	0.147	!0.327	8.26	8.53	8.16	!18.79	1.80	3.96	1.81	!4.04
NISTb	0.500	0.623	0.503	!0.319	0.153	0.33	0.153	!0.099	8.08	8.81	7.77	!18.72	1.83	4.28	1.80	!3.49
NAV	0.510	0.674	0.510		0.084	0.24	0.102		7.93	8.47	7.87		1.74	4.00	1.75	
NAU	0.046	0.062	0.044		0.049	0.13	0.039		0.73	0.73	0.66		0.22	0.46	0.23	

Round Robin XXXVIII Laboratory Results

Values in µg/mL

	δ-Tocopherol				Total β-Carotene				trans-β-Carotene				Total cis-β-Carotene			
Lab	223	224	225	226	223	224	225	226	223	224	225	226	223	224	225	226
FSV-BA					0.186	0.619	0.184	!0.894	0.173	0.585	0.172	!0.84	0.013	0.034	0.012	!0.054
FSV-BD					0.173	0.710	0.178	!0.242								
FSV-BF					0.169	0.511	0.156	!0.241								
FSV-BG					0.197	0.714	0.194	!0.249								
FSV-BGa					0.193	0.676	0.183	!0.227								
FSV-BH					0.170	0.692	0.171	!0.307	0.162	0.677	0.163	!0.299	*nd*	0.015	*nd*	*nd*
FSV-BI					0.175	0.694	0.170	!0.237								
FSV-BJ					0.155	0.458	0.173	!0.155								
FSV-BK																
FSV-BL																
FSV-BM																
FSV-BN	0.180	0.750	0.160	!0.44	0.150	0.660	0.150	!0.37	0.140	0.650	0.150	!0.35	0.004	0.014	0.002	!0.021
FSV-BO					0.117	0.360	0.124	!0.082								
FSV-BP					0.166	0.374	0.169	!0.113								
FSV-BQ																
FSV-BR																
FSV-BS					0.210	0.490	0.210	!0.13	0.160	0.470	0.160	!0.13	0.050	0.020	0.050	*nd*
FSV-BT	0.049	0.525	0.019	!0.164	0.156	0.672	0.163	!0.414	0.150	0.662	0.155	!0.402	0.006	0.010	0.007	!0.012
FSV-BU					0.175	0.693	0.176	!0.253								
FSV-BV					0.158	0.612	0.150	!0.256								
FSV-BW					0.220	0.770	0.210	!0.11								
FSV-BX					0.160	0.360	0.400									
FSV-BZ					>0.180	>0.633	>0.186		0.180	0.633	0.186	!0.265	*nd*	*nd*	0.015	*nd*
FSV-CA																
FSV-CB																
FSV-CC																
FSV-CF																
FSV-CH					0.139	0.645	0.153	!0.247								
FSV-CK					0.141	0.510	0.161	!0.083								
FSV-CL					0.096	0.339	0.093	!0.092								
FSV-CM																
FSV-CP					0.158	0.651	0.161	!0.203								
FSV-CQ					0.087	0.606	0.067	!0.111								
FSV-CR																
FSV-CS																
FSV-CT					0.166	0.585	0.178	!0.186								
FSV-CU					>0.090	>0.287	>0.085		0.090	0.287	0.085	!0.486				
FSV-CX					0.230	0.850	0.220	!0.2								
FSV-DA	0.170	0.760	0.180	!0.45	0.172	0.656	0.177	!0.288	0.161	0.615	0.165	!0.265	0.011	0.041	0.012	!0.037
FSV-DJ																
FSV-DK					0.157	0.560	0.160	!0.32								
FSV-DM					0.171	0.629	0.167	!0.209								
FSV-DP																
FSV-DR					0.169	0.396	0.163									
FSV-DS					0.145	0.469	0.136	!0.142								
FSV-DU					>0.190	>0.460	>0.190		0.190	0.460	0.190	!0.28				
FSV-DX					>0.155	>0.567	>0.155		0.155	0.567	0.155	!0.173				
FSV-EA	0.384	0.830	0.354	!0.526	0.130	0.630	0.170	!0.27								
FSV-EH	0.140	0.610	0.140	!0.33	0.228	0.472	0.212	!0.163	0.205	0.432	0.196	!0.15	*0.023*	*0.040*	*0.016*	
FSV-EI					0.146	0.636	0.203	!0.171	0.134	0.588	0.190	!0.165	*0.012*	*0.048*	*0.013*	
FSV-EL																
n	5	5	5	0	32	32	32	0	12	12	12	0	7	8	8	0
Min	0.049	0.525	0.019		0.087	0.339	0.067		0.090	0.287	0.085		0.004	0.010	0.002	
Median	0.170	0.750	0.160		0.166	0.624	0.170		0.161	0.587	0.164		0.012	0.027	0.013	
Max	0.384	0.830	0.354		0.230	0.850	0.400		0.205	0.677	0.196		0.050	0.048	0.050	
eSD	0.044	0.119	0.030		0.020	0.103	0.020		0.024	0.103	0.017		0.009	0.019	0.004	
eCV	26	16	19		12	16	12		15	18	10		72	71	36	
NISTa	*nd*	0.807	*nd*	*nd*	0.180	0.628	0.181	!0.293	0.173	0.553	0.171	!0.217	*0.007*	*0.076*	*0.009*	
NISTb	0.192	0.832	0.198	!0.439	>0.169	0.706	>0.165	>0.103	0.169	0.658	0.165	!0.101	*nq*	*0.048*	*nq*	
NAV	0.181	0.785	0.181		0.170	0.645	0.171		0.166	0.596	0.166		0.010	0.044	0.011	
NAU	0.034	0.122	0.042		0.028	0.142	0.028		0.022	0.127	0.024		0.008	0.043	0.005	

Round Robin XXXVIII Laboratory Results

Values in µg/mL

	Total α-Carotene				Total Lycopene				trans-Lycopene				β-Cryptoxanthin			
Lab	223	224	225	226	223	224	225	226	223	224	225	226	223	224	225	226
FSV-BA	0.032	0.442	0.031	!0.308	0.427	0.386	0.415	!0.063	0.252	0.300	0.248	!0.069	0.068	0.075	0.067	!0.204
FSV-BD					0.400	0.379	0.413	!0.017					0.052	0.060	0.051	!0.061
FSV-BF	0.033	0.482	0.025	!0.291	0.460	0.372	0.462	!0.035								
FSV-BG	0.040	0.427	0.039	!0.148	0.565	0.490	0.561	!0.029	0.273	0.334	0.262	!0.017	0.054	0.055	0.045	!0.047
FSV-BGa	0.055	0.320	0.050	!0.11	0.533	0.415	0.522	!0.015								
FSV-BH	0.024	0.527	0.025	!0.214	0.517	0.462	0.534	!0.028					0.071	0.067	0.072	!0.074
FSV-BI	0.026	0.479	0.026	!0.202	0.481	0.481	0.460	!0.022					0.059	0.070	0.059	!0.068
FSV-BJ	0.033	0.531	0.044	!0.187	0.220	0.250	0.261	*nd*								
FSV-BK																
FSV-BL																
FSV-BM																
FSV-BN	0.014	0.540	0.027	!0.29	0.650	0.580	0.640	!0.047	0.300	0.420	0.330	!0.04	0.044	0.053	0.051	!0.071
FSV-BO	*nd*	0.192	*nd*	!0.039	0.474	0.361	0.427	!0.031					0.041	0.047	0.044	!0.049
FSV-BP	0.024	0.244	0.026	!0.077	0.491	0.369	0.535	!0.015					0.131	0.109	0.118	!0.069
FSV-BQ																
FSV-BR																
FSV-BS	0.060	0.430	0.060	!0.11	0.090	0.190	0.085	!0.01					0.060	0.070	0.070	!0.08
FSV-BT	0.030	0.420	0.033	!0.203	0.392	0.316	0.405	!0.050	0.309	0.275	0.314	!0.039	0.066	0.079	0.072	!0.092
FSV-BU	0.021	0.456	0.021	!0.198	0.520	0.472	0.524	!0.031					0.074	0.084	0.072	!0.079
FSV-BV	0.019	0.374	0.018	!0.143	0.424	0.355	0.400	!0.016								
FSV-BW	0.029	0.500	0.029	!0.069	0.550	0.480	0.590	*nd*								
FSV-BX	0.016	0.180	0.060		0.370	0.270	0.610						0.030	0.030	0.030	
FSV-BZ	0.038	0.371	0.036	!0.123	0.246	0.323	0.256	*nd*								
FSV-CA																
FSV-CB																
FSV-CC																
FSV-CF																
FSV-CH	0.017	0.454	0.019	!0.2	0.357	0.361	0.409	!0.014								
FSV-CK	0.031	0.350	0.049	!0.081	0.420	0.376	0.415	!0.039	0.200	0.249	0.197	!0.021	0.048	0.050	0.047	!0.048
FSV-CL	0.014	0.269	0.012	!0.065	0.262	0.240	0.261	*nd*					0.027	0.034	0.027	!0.031
FSV-CM																
FSV-CP	0.021	0.402	0.022	!0.130	0.338	0.308	0.339	!0.012					0.056	0.061	0.056	!0.065
FSV-CQ																
FSV-CR																
FSV-CS																
FSV-CT																
FSV-CU																
FSV-CX	0.020	0.520	0.030	!0.08	0.540	0.470	0.530	!0.02					0.040	0.060	0.050	!0.05
FSV-DA	0.025	0.429	0.029	!0.253	0.479	0.461	0.476	!0.052	0.231	0.315	0.224	!0.038	0.055	0.055	0.052	!0.065
FSV-DJ																
FSV-DK	0.020	0.350	0.020	!0.17												
FSV-DM	0.033	0.453	0.039	!0.157	0.409	0.380	0.431	!0.067								
FSV-DP																
FSV-DR																
FSV-DS																
FSV-DU																
FSV-DX					0.463	0.373	0.457	!0.024								
FSV-EA	*nd*	0.420	*nd*	!0.183	0.501	0.420	0.500	!0.02					0.028	0.042	0.026	!0.031
FSV-EH	0.026	0.505	0.024	!0.112					0.249	0.240	0.238	*nd*	0.064	0.066	0.056	!0.06
FSV-EI	0.027	0.408	0.039	!0.135	0.471	0.402	0.674	!0.039	0.180	0.242	0.252	!0.017	0.052	0.057	0.072	!0.038
FSV-EL																
n	26	28	26	0	28	28	28	0	8	8	8	0	20	20	20	0
Min	0.014	0.180	0.012		0.090	0.190	0.085		0.180	0.240	0.197		0.027	0.030	0.026	
Median	0.026	0.428	0.029		0.462	0.377	0.459		0.251	0.288	0.250		0.055	0.060	0.054	
Max	0.060	0.540	0.060		0.650	0.580	0.674		0.309	0.420	0.330		0.131	0.109	0.118	
eSD	0.009	0.082	0.011		0.089	0.086	0.096		0.053	0.063	0.028		0.016	0.015	0.017	
eCV	34	19	37		19	23	21		21	22	11		30	25	31	
NISTa	*nd*	0.420	*nd*	!0.191												
NISTb	0.031	0.480	0.037	!0.057	0.590	0.503	0.587	*nd*	0.231	0.317	0.230	*nd*	0.049	0.050	0.049	!0.026
NAV	0.028	0.439	0.034		0.526	0.440	0.526		0.241	0.302	0.239		0.052	0.055	0.051	
NAU	0.010	0.114	0.015		0.137	0.124	0.140		0.053	0.063	0.054		0.016	0.017	0.018	

Round Robin XXXVIII Laboratory Results

Values in µg/mL

	α-Cryptoxanthin				Lutein				Zeaxanthin				Lutein&Zeaxanthin			
Lab	223	224	225	226	223	224	225	226	223	224	225	226	223	224	225	226
FSV-BA													0.203	0.315	0.202	!0.112
FSV-BD					0.125	0.179	0.128	!0.223	0.020	0.086	0.022	!0.117	*0.145*	*0.265*	*0.150*	
FSV-BF																
FSV-BG					0.134	0.149	0.138	!0.183	0.026	0.075	0.024	!0.097	*0.160*	*0.224*	*0.162*	
FSV-BGa																
FSV-BH					0.102	0.113	0.096	!0.174	0.024	0.082	0.028	!0.08	*0.126*	*0.195*	*0.124*	
FSV-BI					0.124	0.147	0.123	!0.124	0.033	0.081	0.033	!0.067	*0.157*	*0.228*	*0.156*	
FSV-BJ																
FSV-BK																
FSV-BL																
FSV-BM																
FSV-BN	0.011	0.038	0.016	!0.011	0.110	0.120	0.110	!0.14	0.029	0.073	0.031	!0.1	0.130	0.190	0.140	!0.25
FSV-BO													0.113	0.151	0.113	!0.192
FSV-BP																
FSV-BQ																
FSV-BR																
FSV-BS					0.140	0.130	0.170	!0.13								
FSV-BT	0.027	0.047	0.026	!0.013	0.127	0.218	0.132	!0.429	0.026	0.029	0.027	!0.018	0.153	0.247	0.159	!0.447
FSV-BU													0.177	0.273	0.199	!0.455
FSV-BV													0.151	0.201	0.141	!0.237
FSV-BW																
FSV-BX					0.160	0.170	0.150		0.050	0.090	0.050		*0.210*	*0.260*	*0.200*	
FSV-BZ					0.122	0.211	0.216	!0.259								
FSV-CA																
FSV-CB																
FSV-CC																
FSV-CF																
FSV-CH																
FSV-CK	0.033	0.046	0.033	!0.015									0.092	0.114	0.094	!0.138
FSV-CL													0.084	0.114	0.076	!0.113
FSV-CM																
FSV-CP													0.180	0.272	0.180	!0.352
FSV-CQ																
FSV-CR																
FSV-CS																
FSV-CT					0.115	0.149	0.117	!0.123								
FSV-CU																
FSV-CX					0.130	0.140	0.130	!0.12	0.020	0.060	0.020	!0.05	*0.150*	*0.200*	*0.150*	
FSV-DA	0.016	0.038	0.017	!0.01	0.131	0.135	0.136	!0.13	0.050	0.094	0.056	!0.09	0.181	0.229	0.192	!0.22
FSV-DJ																
FSV-DK																
FSV-DM													0.156	0.199	0.142	!0.235
FSV-DP																
FSV-DR																
FSV-DS																
FSV-DU																
FSV-DX													0.135	0.220	0.138	!0.214
FSV-EA																
FSV-EH					0.122	0.162	0.118	!0.265	0.034	0.108	0.033	!0.199	*0.156*	*0.270*	*0.151*	
FSV-EI					0.102	0.126	0.142	!0.079	0.043	0.084	0.060	!0.055	*0.145*	*0.210*	*0.202*	
FSV-EL																
n	4	4	4	0	14	14	14	0	11	11	11	0	20	20	20	0
Min	0.011	0.038	0.016		0.102	0.113	0.096		0.020	0.029	0.020		0.084	0.114	0.076	
Median	0.021	0.042	0.022		0.125	0.148	0.131		0.029	0.082	0.031		0.152	0.222	0.151	
Max	0.033	0.047	0.033		0.160	0.218	0.216		0.050	0.108	0.060		0.210	0.315	0.202	
eSD	0.012	0.006	0.008		0.012	0.030	0.018		0.007	0.012	0.010		0.029	0.044	0.029	
eCV					10	20	14		26	14	33		19	20	19	
NISTa																
NISTb					0.118	0.136	0.117	!0.136	0.047	0.092	0.045	!0.091	*0.165*	*0.228*	*0.162*	
NAV					0.122	0.142	0.124		0.038	0.087	0.038		0.159	0.225	0.156	
NAU					0.028	0.034	0.031		0.016	0.025	0.015		0.034	0.048	0.033	

Round Robin XXXVIII Laboratory Results

Analytes Reported By One Laboratory

Values in µg/mL

Analyte	Code	223	224	225	226
Coenzyme Q10	FSV-CH	0.190	0.330	0.210	!0.04
trans-α-Carotene	NISTb	0.031	0.463	0.027	!0.057

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 "Report of (Meta)Analysis."
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 XXXVIII Laboratory Results

Comparability Summary

Lab	R	aT	gT	bC	tbC
FSV-BA	1	1	1	1	
FSV-BD	1	1	1	1	
FSV-BF	2	1	1	1	
FSV-BG	1	1			
FSV-BGa	1	1		1	
FSV-BH	2	1	1	1	
FSV-BI	1	1		4	
FSV-BJ	1	2	1	1	1
FSV-BK	2	2	1	1	1
FSV-BL	1	3	2	2	
FSV-BM	3	3	2	4	
FSV-BN	2	1	3		1
FSV-BO	1	1			
FSV-BP	3	2			
FSV-BQ	1	2	1	2	
FSV-BR	1				
FSV-BS	2				3
FSV-BT	1	1	2	1	
FSV-BU			1	2	
FSV-BV	3	1	4	3	
FSV-BW	1	2		1	
FSV-BX	2	1	2	2	
FSV-BZ		1	2	3	
FSV-CA	1	1			
FSV-CB	2	2		2	
FSV-CC	3	1	1	1	2
FSV-CF	1	1	3	2	2
FSV-CH	2	2		2	1
FSV-CK	3	3		1	
FSV-CL	4	4		2	
FSV-CM		1		2	
FSV-CP		2	1	1	1
FSV-CQ	2	1			
FSV-CR	1	1			
FSV-CS	1				
FSV-CT	1	2			
FSV-CU	1	1			
FSV-CX	1	1			1
FSV-DA	1	1	4	1	
FSV-DJ	4	4		1	
FSV-DK	1	1	2	1	1
FSV-DM	1	1			
FSV-DP	1				
FSV-DR	1	1	1	1	
FSV-DS	4	3	1	2	2
FSV-DU	2	1			
FSV-DX	2	2	2	2	
FSV-EA	4	1			
FSV-EH	2	2			
FSV-EI	3	3			2
FSV-EL	2			2	
NISTa	1	1	1	1	1
NISTb	1	1	1	1	1
n	47	45	23	32	12

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 (serum heterogeniety and iter- and intra-laboratory) measurement standard deviation.
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

For details on the NIST Assigned quantities, see this Round Robin's "Report of (Meta)Analysis."

StS	% Observed					Expected
1	51	60	52	50	58	68.2 %
2	28	24	30	38	33	27.3 %
3	13	11	9	6	8	4.3 %
4	9	4	9	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 RR38

Each participant in RR38 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
- Zeaxanthin
- Lutein & Zeaxanthin

The following 8 pages are the "Individualized Report" for the analytes evaluated by participant FSV-BA.

Individualized Round Robin XXXVIII Report to: FSV-BA

Your Data, NIST Assigned Values, and %Differences

	Serum 223				Serum 224				Serum 225				Serum 226			
Analyte	You	NAV	%Δ	n	You	NAV	%Δ	n	You	NAV	%Δ	n	You	NAV	%Δ	n
Retinol	.50	.51	-3	45	.70	.68	4	45	.49	.51	-3	45	.34	.32	7	44
Retinyl Palmitate	.16	.12	27	15	.24	.28	-12	15	.13	.13	2	15	.17	.23	-27	15
α-Tocopherol	7.69	7.93	-3	42	8.42	8.47	-1	42	7.64	7.88	-3	42	19.26	19.20	0	41
γ-Tocopherol	1.93	1.74	11	21	4.37	4.00	9	21	1.90	1.74	9	21	3.70	3.57	4	20
Total β-Carotene	.19	.17	9	29	.62	.65	-5	29	.18	.17	7	29	.89	.22	306	28
trans-β-Carotene	.17	.17	4	12	.59	.60	-2	12	.17	.17	4	12	.84	.22	282	12
Total cis-β-Carotene	.01			5	.03			6	.01			6	.05			4
Total α-Carotene	.03	.03	10	24	.44	.44	1	26	.03	.03	-9	24	.31	.14	114	25
Total Lycopene	.43	.53	-19	26	.39	.44	-12	26	.42	.52	-21	26	.06			22
trans-Lycopene	.25	.24	5	8	.30	.31	-3	8	.25	.24	3	8	.07			7
β-Cryptoxanthin	.07	.05	33	18	.08	.06	36	18	.07	.05	33	18	.20	.05	343	17
"Lutein&Zeaxanthin"	.20			17	.32			17	.20			17	.11			16

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

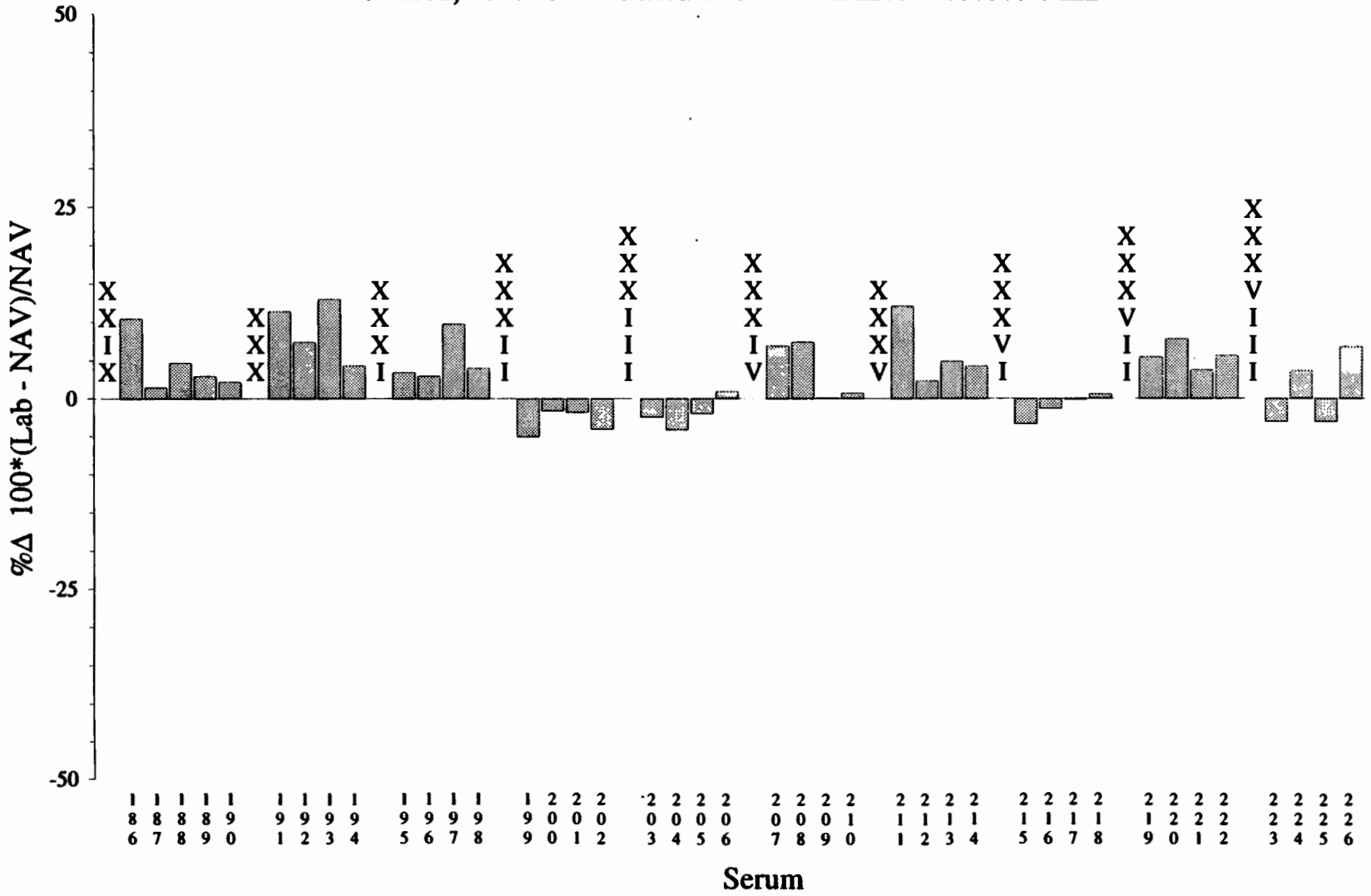
n : Number of non-NIST laboratories reporting quantitative values for this analyte in this serum

Please check our recorded values against your records.

Send corrections to: NNMMQAP 222/B208, NIST, Gaithersburg, MD 20899; fax 301-977-0685; email David.Duewer@NIST.gov

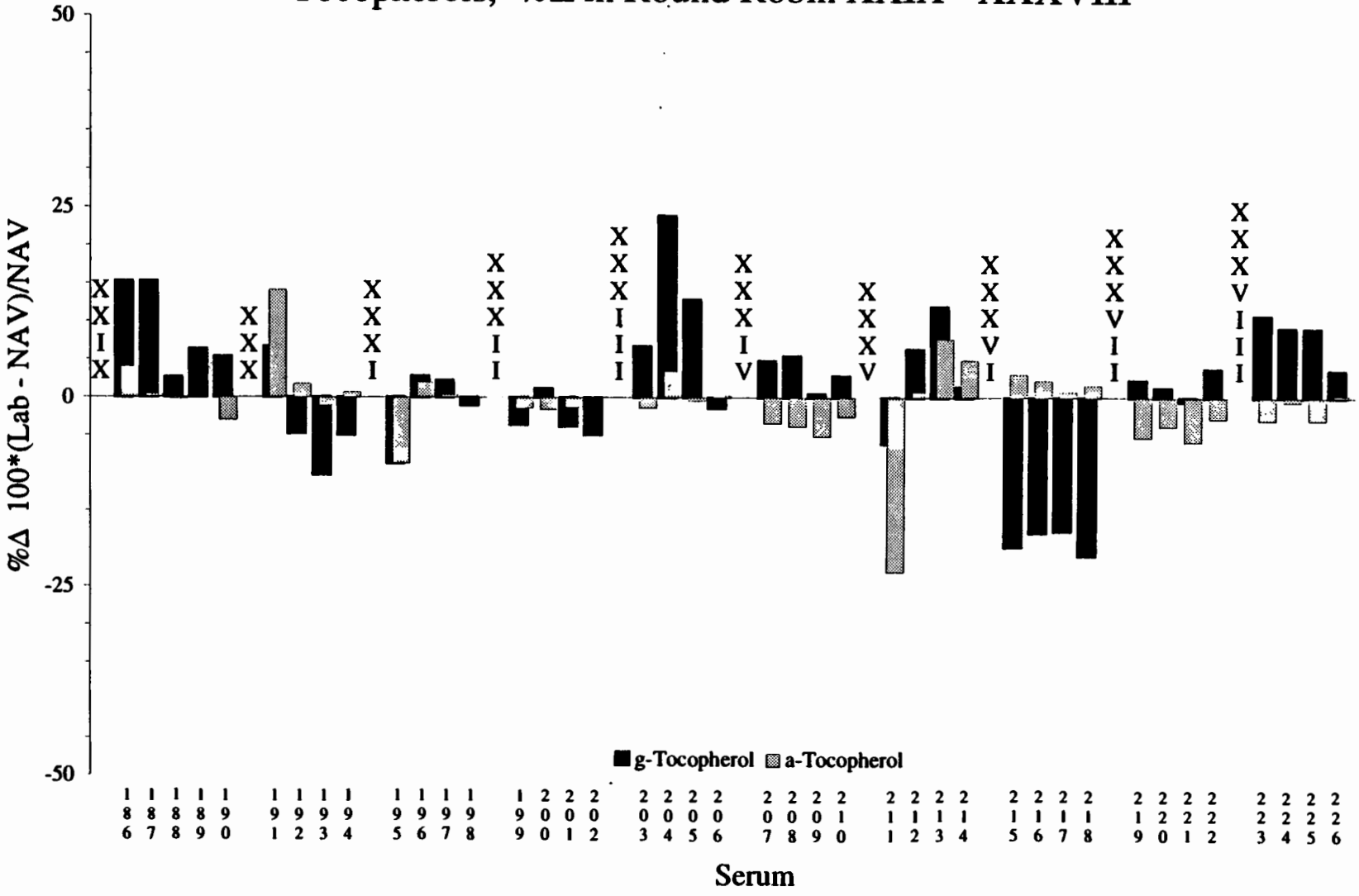
Individualized Round Robin XXXVIII Report to: FSV-BA

Retinol, %Δ in Round Robin XXIX - XXXVIII



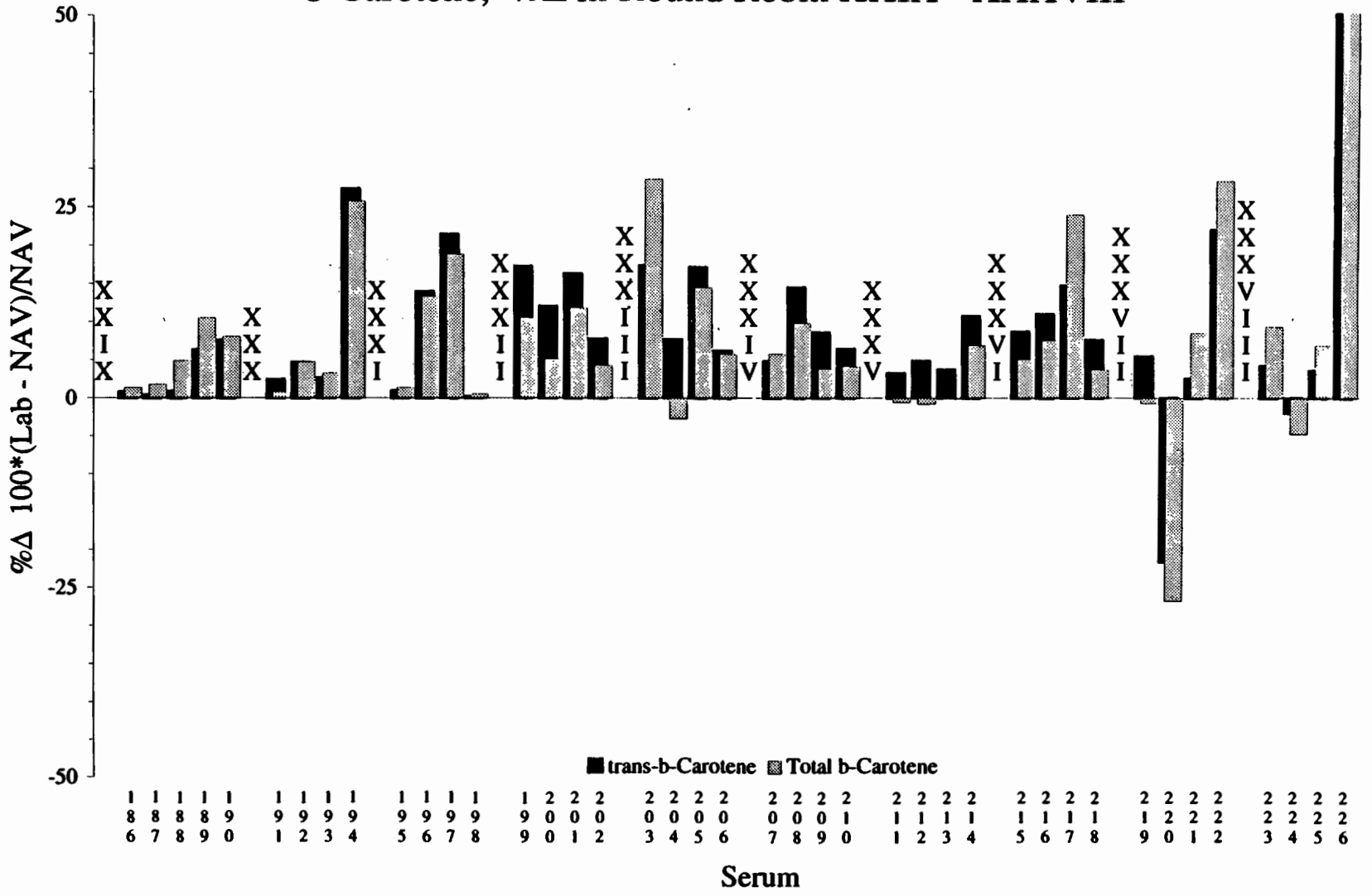
Individualized Round Robin XXXVIII Report to: FSV-BA

Tocopherols, %Δ in Round Robin XXIX - XXXVIII



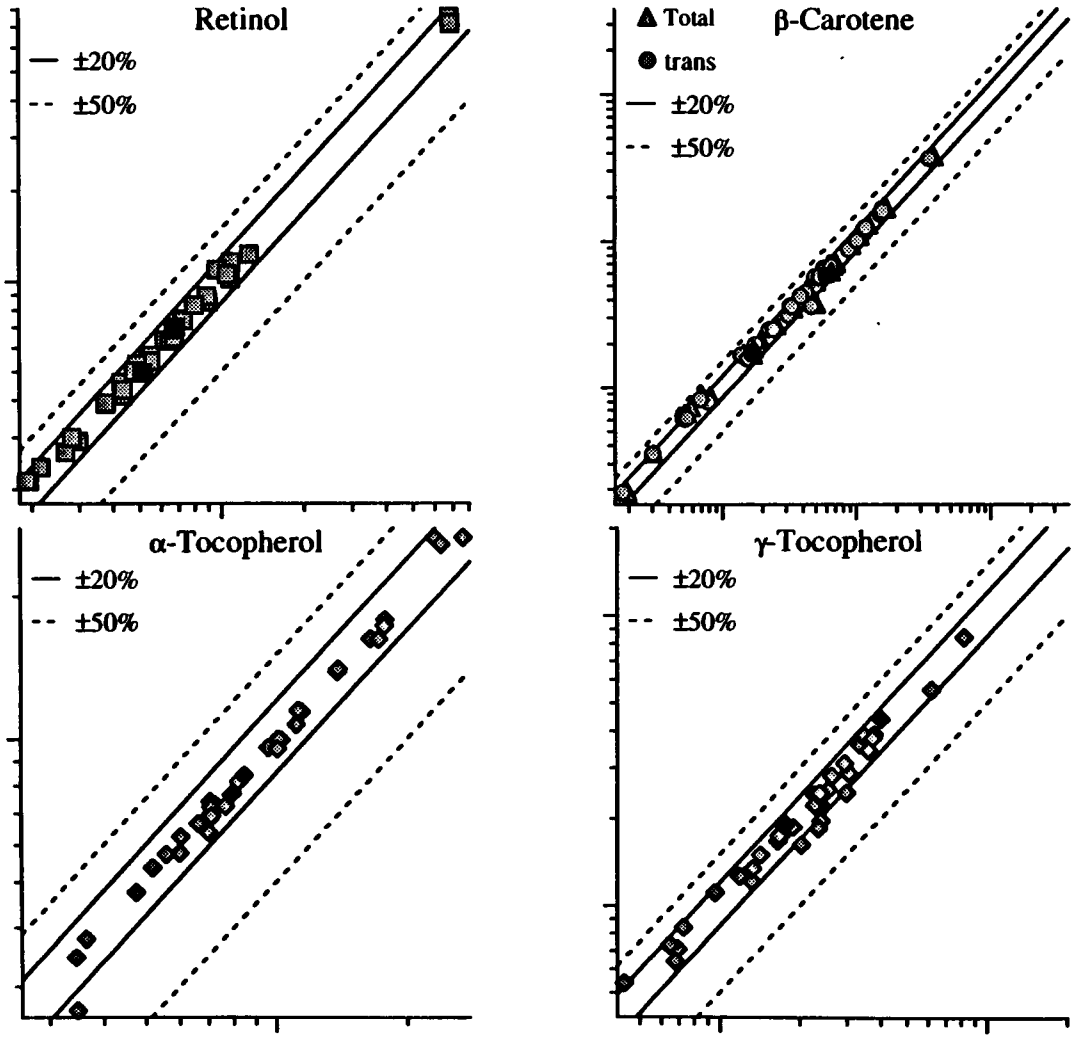
Individualized Round Robin XXXVIII Report to: FSV-BA

b-Carotene, %Δ in Round Robin XXIX - XXXVIII



Individualized Round Robin XXXVIII Report to: FSV-BA

NIST Assigned Values Vs Laboratory Values



Legend

Shaded Symbols: RR XXIX-XXXVII
Black Symbols: RR XXXVIII

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

Interpretation

Adequately intercomparable data are within ±20% lines. If you have data scattered outside ±50% lines, your measurement system is not consistent with those of most participating laboratories. If your data are systematically higher or lower than the NAV, your system may be consistent but your results are biased.

If your data show increased scatter at low concentrations, your "limit of quantification" may not be what you think it is.

If there are one or two "wild" outliers, they might be calculation or transcription errors. We would appreciate hearing from you about any such problems.

Individualized Round Robin XXXVIII Report to: FSV-BA

Accuracy/Precision Summary

	Ret		aToc		gToc		Total		trans	
	mΔ	vΔ	mΔ	vΔ	mΔ	vΔ	mΔ	vΔ	mΔ	vΔ
XXIX	4	4	0	3	9	6	5	4	3	3
XXX	9	4	4	7	-3	7	9	12	9	12
XXXI	5	3	-2	5	-1	5	9	9	9	10
XXXII	-3	2	-1	1	-3	3	8	4	13	4
XXXIII	-2	2	0	2	11	11	12	13	12	6
XXXIV	4	4	-4	1	3	2	6	3	9	4
XXXV	6	4	-2	14	3	8	1	4	6	3
XXXVI	-1	2	2	1	-19	2	10	9	11	3
XXXVII	6	2	-4	1	2	2	2	23	2	18
XXXVIII	-1	4	-2	1	10	1	4	8	2	3

Legend

Ret	Retinol
aToc	α-Tocopherol
gToc	γ-Tocopherol
Total	Total β-Carotene
trans	trans-β-Carotene
mΔ	Mean difference, the average %Δ for all sera of a given RR, where %Δ = 100(Your value - NAV) / NAV
vΔ	Difference variability, one standard deviation of %Δ for all sera of a RR
NAV	NIST Assigned Value, our best estimate of analyte concentration... NAV = (NIST's average-of-averages + Round Robin median) / 2

(Traditional) Performance Criteria

The absolute value of %Δ of every measurement has traditionally been evaluated as follows...

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

More representative criteria need to be established, factoring in each serum's analyte level and the analyte distribution in adult human populations. Stay tuned, we're working on it...

Interpretation

Accuracy and precision are separate but kindred aspects of measurement comparison. We estimate accuracy as mΔ, the average %Δ, and precision as vΔ, the standard deviation of %Δ, for all sera of a Round Robin.

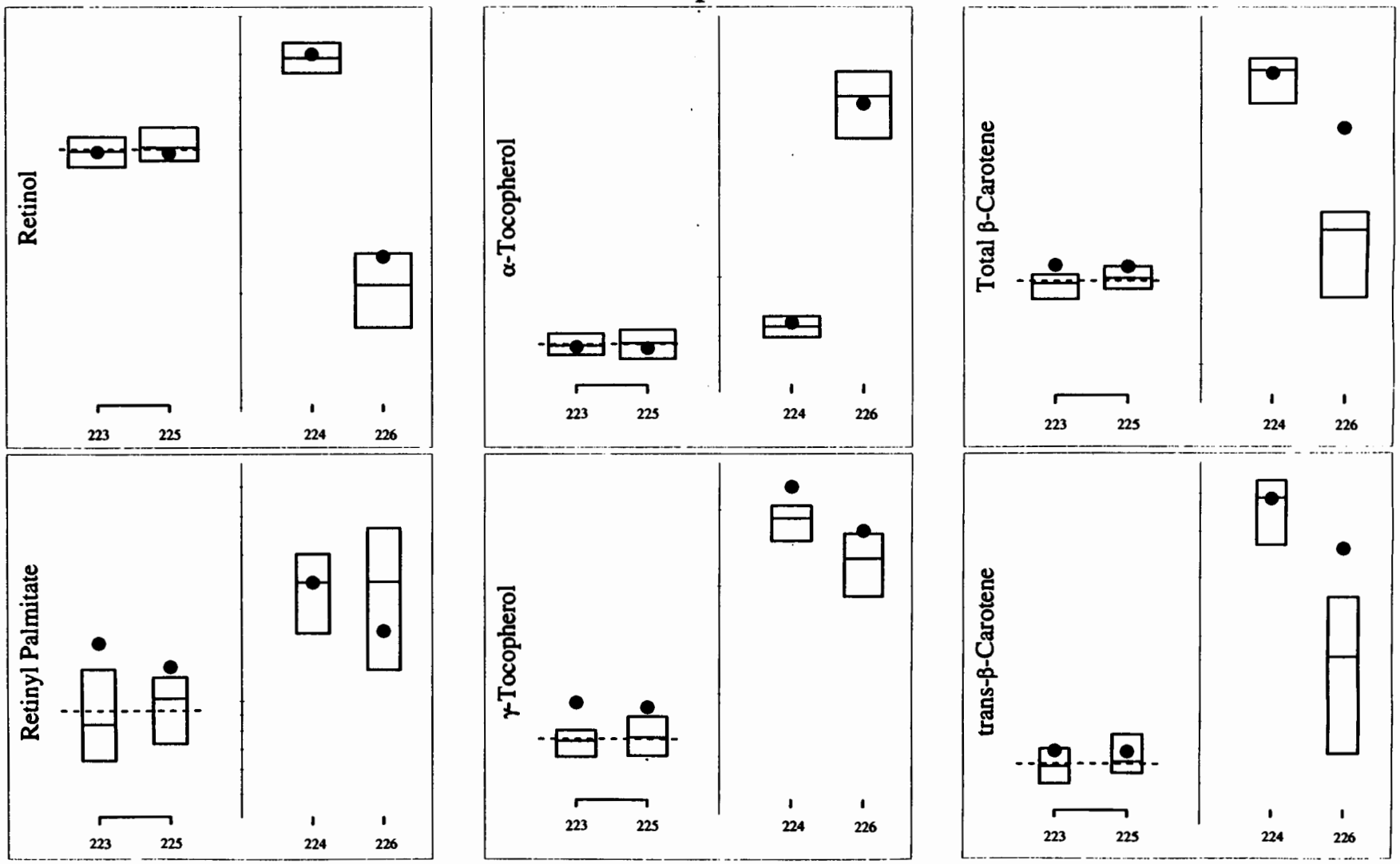
It's best to be accurate and precise (small mΔ, small vΔ)!

Good precision (small vΔ) with poor accuracy (large mΔ) is better than the converse: at least such values are internally consistent and may be relatable to others' values once the relative biases have been determined.

Poor precision (large vΔ) suggests that your measurement system is not in adequate control for the analyte levels examined.

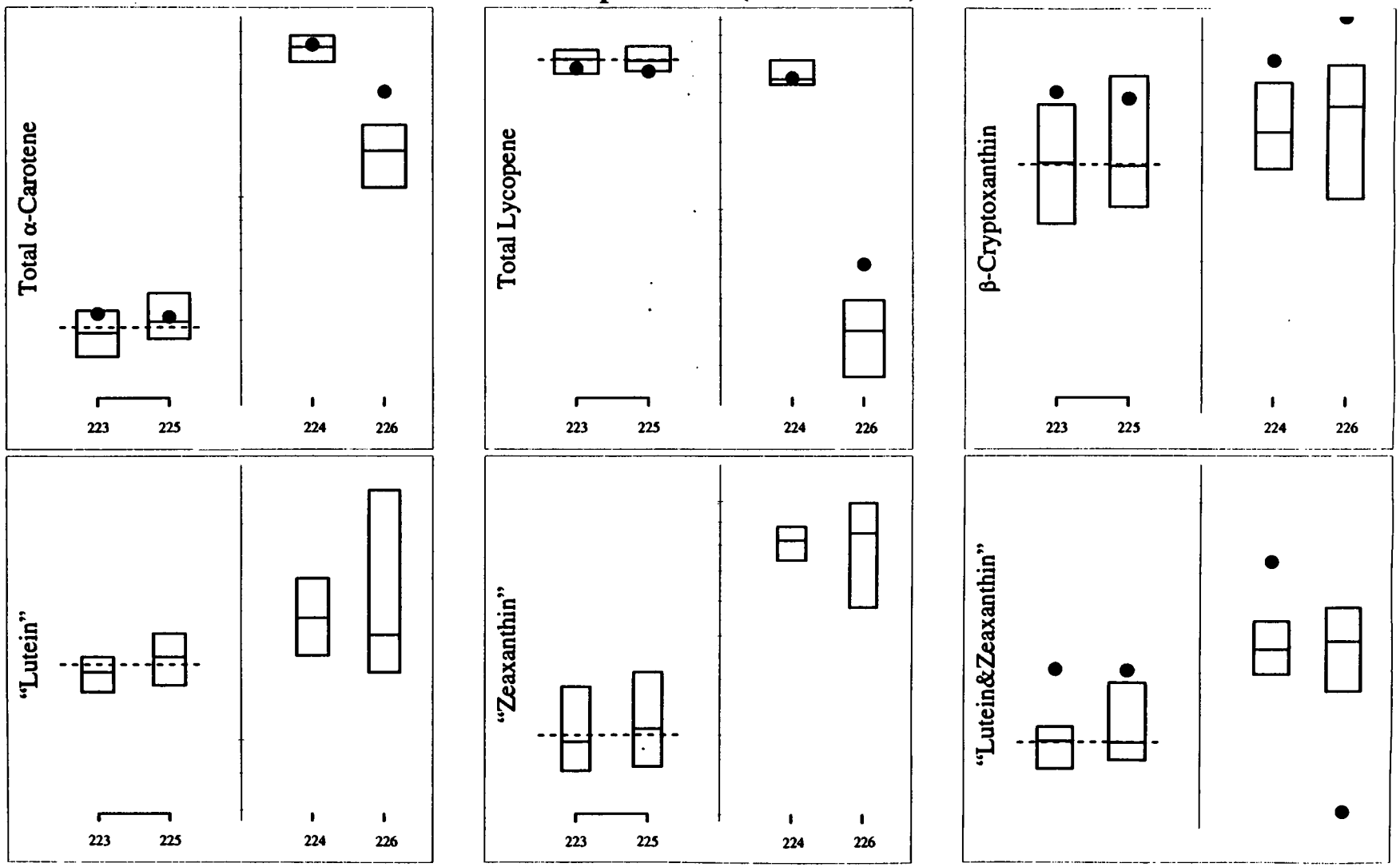
Individualized Round Robin XXXVIII Report to: FSV-BA

Comparisons



Individualized Round Robin XXXVIII Report to: FSV-BA

Comparisons (Continued)



Appendix M. Shipping Package Inserts for RR09

The following two items were included in each package shipped to a RR09 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.

May 20, 1996

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

Dr. Margolis printed a separate cover letter for each participant. The "FIELD()" macro commands were part of a mail-merge system.

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 10-20 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 **July 2, 1996** 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 688A, VIAL # _____

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

SERUM 688A, VIAL # _____

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

SERUM 188A, VIAL # _____

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

SERUM 188A, VIAL # _____

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

Appendix N. Final Report for RR09

The following seven pages are the final report for RR08 and RR09 as provided to all participants. This report contains:

- Cover letter and analysis of results.
- Table 1 "Results of Round Robin RR08 Measurement of Ascorbic Acid in Human Serum".
- Table 2 "Results of Round Robin RR09 Measurement of Ascorbic Acid in Human Serum".
- Table 2 "Results of Round Robin RR09 Measurement of AA using NIST Ascorbic Acid Sample".
- Figure 1 "Tukey Box Plot of the Round Robin RR08 Results"
- Figure 2 "Tukey Box Plot of the Round Robin RR09 Results"

A number of the results reported in the Tables 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 12, 1996

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

Dr. Margolis printed a separate cover letter for each participant. The "FIELD()" macro commands were part of a mail-merge system.

Dear FIELD(Title) FIELD(Last):

This report describes both the overall-group and your laboratory performance in Round Robin VIII and Round Robin IX for the measurement of ascorbic acid in human plasma. The studies involve the duplicate analyses of four unknown samples [two from Lots 688a and 682a (RR VIII); 688a and 188a (RR IX)]. RR VIII also included a control sample from Lot 688b and RR IX included solid ascorbic acid as a standard. Specifically your data evaluation package contains tabular presentations of all of the data submitted for ascorbic acid Round Robins VIII and IX. Your results are designated as FIELD(Lab No.) in the tables and figures.

Tables 1 and 2 provide 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 Tables 1 and 2 the percent Relative Standard Deviation (%RSD) for both lots ranged from 4.7 - 13.9. The intralaboratory SD varied from 0.3 - 5.8 with the exception of one laboratory in each round robin. These results indicate that the intralaboratory variation remains essentially unchanged from the two previous round robins (SD = 0.3 - 4.0). However, the interlaboratory %RSD has decreased by approximately 50%. This occurred with the improved accuracy of several laboratories in the last two round robins and coincides with the introduction of the control material into RR VIII with the requirement that the control measurement must be within 10% of the assigned value. The Tukey 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 except for Lot 682a which is 8.7% higher and which represents the upper end of the range of serum ascorbic acid concentrations.

In RR IX we asked each lab 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 which increased the concentration 10 fold. The purpose of this segment of the study was to try to evaluate the role that your standards might be playing in the accuracy and precision of your measurement process. Because of the low ascorbic acid concentration, the data (Table 3) are not very meaningful except to indicate that there was a large degree of variability that may have been related to the inability to do accurate spectrophotometry at the designated analyte concentration. In RR X we will ask you to do the same measurements on ascorbic acid solutions that are ten times more concentrated.

The improvement in the interlaboratory precision of these round robins is encouraging and we hope that the next round robin will assess the role of the standards in the measurement accuracy and their contribution to the interlaboratory variability. If your results deviated widely from the assigned values, we recommend 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 X) will be shipped around October 28, 1996. If you have any questions concerning the previous round robins, please contact me at (301)975-3137.

Sincerely,

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

Enclosures

Table 1: **Results of Round Robin VIII Measurement of Ascorbic Acid in Human Serum.**

		Ascorbic Acid (µmol/L Serum)[a]		
Lab	**Method**	**Lot 688b**	**Lot 688a**	**Lot 682a**
	DNPH	57.8 ± 2.5	28.0 ± 2.5	92.2 ± 2.3
	LC	57.2 ± 2.4	26.7 ± 1.2	90.4 ± 1.3
	LC-EC			
	ENZ	60.7 ± 0.9	31.5 ± 0.4	91.7 ± 0.6
	DNPH	62.1 ± 1.4	32.0 ± 0.2	92.0 ± 0.3
	DCIP			
	DCIP	57.8 ± 2.5	28.0 ± 2.5	92.2 ± 2.3
	LC-EC	61.6 ± 2.0	34.2 ± 1.2	98.7 ± 1.3
	LC-EC	56.8 ± 1.8	26.9 ± 7.8	99.3 ±10.7
	LC			
	LC-EC	57.3 ± 0.6	29.4 ± 0.4	89.7 ± 0.9
	LC			
	LC-EC	58.3 ± 1.5	25.9 ± 1.2	90.0 ± 1.7
	AUTOAN	58.9 ± 1.7	28.8 ± 1.3	87.3 ± 2.9
	LC-OPD	54.2 ± 0.3	27.3 ± 0	84.2 ± 0.7
	LC-EC	67.7 ± 1.0	32.9 ± 0.2	103.2 ± 1.5
	LC-OPD	56.9 ± 0.7	26.9 ± 3.3	78.0 ± 1.4
	LC			
	LC-EC	51.6 ± 1.2	26.2 ± 1.2	81.8 ± 0.6
	LC-Coul	na	32.6 ± 0.8	65.1 ± 0.5
MEAN		57.7	28.3	90.5
SD		2.7	2.5	6.1
%RSD		4.7	8.7	6.7
NIST AA + DHAA	LC-EC	56.8 ± 1.6[b]	27.4 ± 3.2[b]	83.2 ± 5.5[b]
NIST AA	LC-EC	43.2 ± 3.3[b]	6.5 ± 1.4[b]	70.7 ± 1.8[b]

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

[b] Values represent the mean and SD of replicate measurements on five samples (total of 10 measurements).

Table 2: Results of Round Robin IX Measurement of Ascorbic Acid in Human Serum.

		Ascorbic Acid (µmol/L Serum)[a]	
Lab	**Method**	**Lot 688a**	**Lot 188a**
	LC	28.9 ± 0.6	64.1 ± 0.8
	DNPH	25.5 ± 2.7	56.8 ± 4.6
	LC-EC	26.4 ± 2.1	58.6 ± 5.7
	ENZ	30.5 ± 0.5	62.5 ± 1.3
	DNPH		
	DCIP		
	DCIP	28.3 ± 3.2	67.7 ± 5.8
	LC-EC	34.7 ± 0.5	70.9 ± 2.3
	LC-EC		
	LC		
	LC-EC	24.7 ± 2.7	65.2 ± 0.8
	LC		
	LC-EC	19.2 ± 0.6	52.2 ± 3.5
	AUTOAN	28.1 ± 1.0	57.8 ± 1.2
	LC-OPD	28.1 ± 0.6	61.3 ± 0.7
	LC-EC		
	LC-OPD	34.6 ± 2.6	75.2 ± 2.8
	LC	28.7 ± 2.2	63.3 ± 2.2
	LC-EC	25.9 ± 0.9	51.7 ± 0.6
	LC-Coul	32.3 ± 1.1	64.3 ± 0.4
	DNPH	51.3 ± 27.4	84.8 ± 24.3
	ENZ	26.2 ± 1.6	56.0 ± 1.1
	LC	25.1 ± 1.4	50.2 ± 3.0
MEAN		27.7	60.9
SD		3.8	7.2
%RSD		13.9	11.8
NIST AA + DHAA	LC-EC	27.4 ± 0.3[A]	64.2 ± 1.8[d]
NIST AA	LC-EC	6.5 ± 1.4[A]	40.2 ± 6.6[d]

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

[b] These samples were nor included in the mean because of their very large SD.

[c] Values represent the mean and SD of replicate measurements on five samples (total of 10 measurements).

Table 3: Results of Round Robin IX Measurement of AA using NIST Ascorbic Acid Sample[a].

Lab	Method	Ascorbic Acid (µmol/L MPA) Calculated	Measured	$E^{1\%}$[b]	AA Source
	LC DNPH	2.84	2.84	745	Sigma
	LC-EC	5.14	[a]		Sigma
	ENZ DNPH DCIP	5.32	15.4	445	Sigma
	DCIP	3.96	8.75	775	Sigma
	LC-EC	5.75	8.0	576	Sigma
	LC-EC LC				
	LC-EC	5.73	5.6	615	Aldrich
	LC LC-EC	4.13	2.85	510	Merck
	AUTOAN	4.95	4.80	598	na
	LC-OPD	5.40	5.42	583	na
	LC-EC	5.09	13.0	900	BDH
	LC-OPD	4.72	4.50	791	Sigma
	LC	3.46	3.02	522	BDH
	LC-EC	50.2	48.1	585	Sigma
	DNPH	5.49	3.85	643	Sigma
	ENZ	4.31	4.26	702	Sigma
	LC	20.6	6.05	199	Merck
NIST (5% MPA)				570	Sigma
Literature Value. (aqueous)				←	560
NIST[b]				513	Sigma

[a] Used NIST AA to prepare standards for assay.

[b] 1%= Absorbance at 20°C of a solution of 1g/100mL measured in a cuvette with a path of 1 cm.

[c] Solvent: water : acetonitrile, 1:3 + DTT 1 mg/mL.

Figure 1

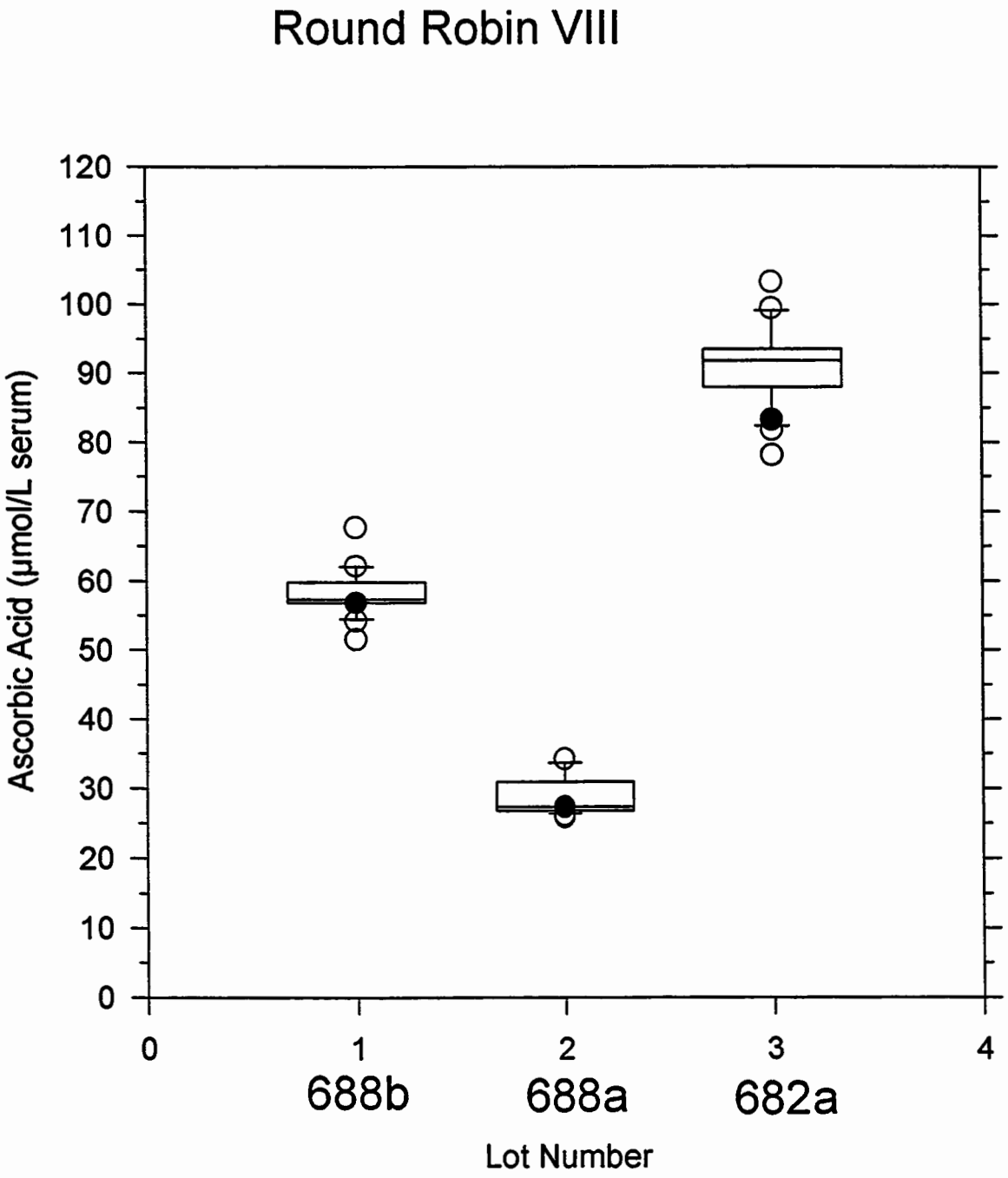
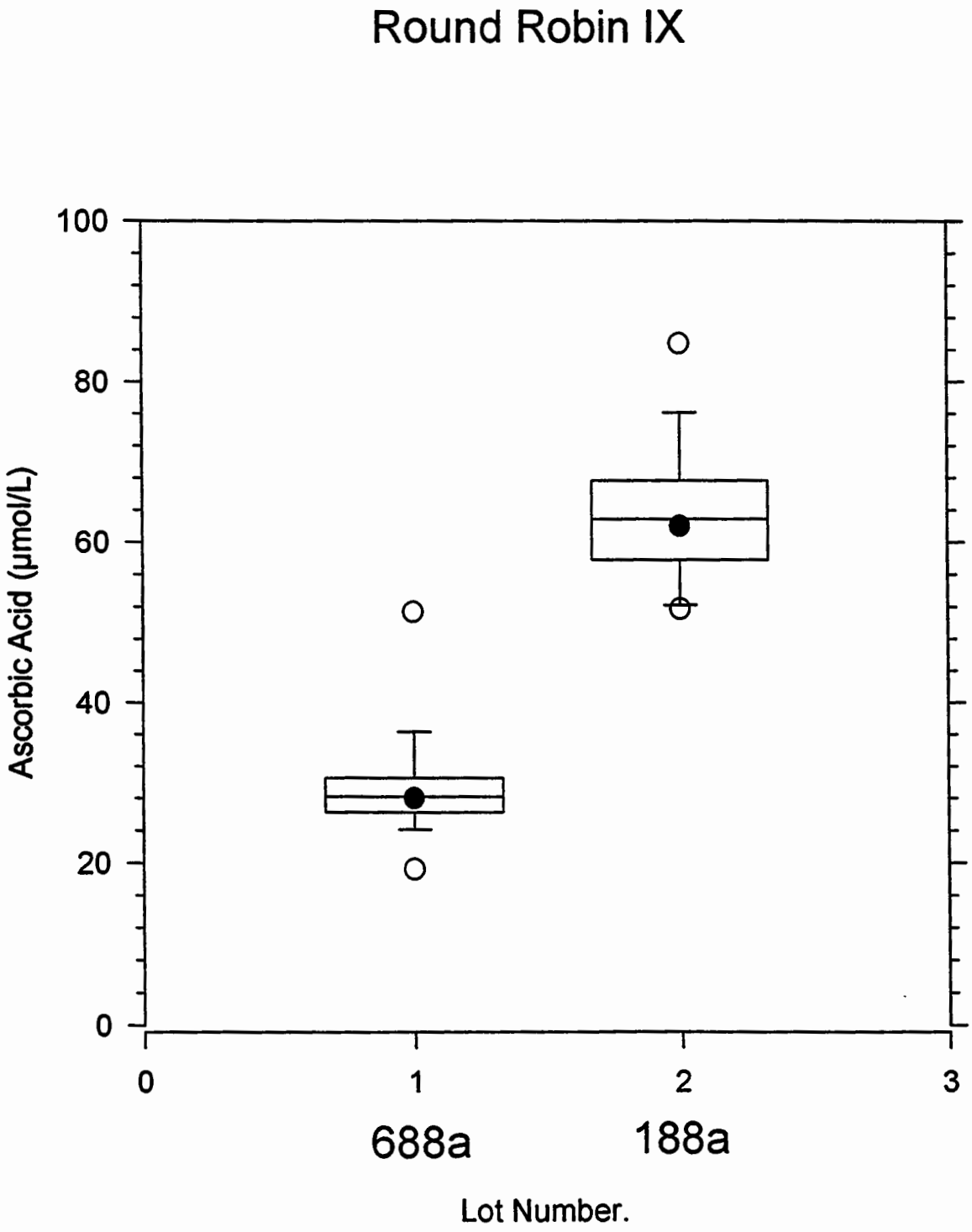

Round Robin VIII
Ascorbic Acid (µmol/L serum)
120
110
100
90
80
70
60
50
40
30
20
10
0
0
1
2
3
4
688b
688a
682a
Lot Number

Figure 2


Round Robin IX
100
80
60
40
20
0
Ascorbic Acid (µmol/L)
0
1
2
3
688a
188a
Lot Number.

Appendix O. "All-Lab Report" for VC-RR09

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 09

[Total Ascorbic Acid], mmol/mL

		AA Control		688A					188A				
Lab	Method	Av	S_{rep}	Av	S_{dup}	S_{rep}	S_{het}	S_{tot}	Av	S_{dup}	S_{rep}	S_{het}	S_{tot}
VC-MA	HPLC-UV	1.42	0.00	13.2	1.9	1.9	1.3	2.3	25.7	0.2	0.3	0.0	0.3
VC-MB	AO-OPD	7.70	0.00	15.3	0.2	0.3	0.0	0.3	31.2	0.7	0.4	0.7	0.8
VC-MC	HPLC-EC	2.84	0.00	14.4	0.3	0.0	0.0	0.3	31.9	0.2	0.0	0.0	0.2
VC-MD	24DNPH			12.8	1.3	1.1	1.1	1.5	28.2	2.0	1.9	1.5	2.4
VC-ME	24DNPH	2.71	0.02	14.1	0.0	0.4	0.0	0.3	30.7	0.3	0.3	0.2	0.4
VC-MF	HPLC-EC	2.40	0.02	14.1	0.4	0.4	0.2	0.5	28.9	0.5	0.5	0.3	0.6
VC-MG	HPLC-UV	1.51	0.04	14.4	1.4	0.2	1.3	1.4	31.6	1.2	0.7	1.1	1.3
VC-MO	AutoAnal	3.02	0.10	12.5	0.3	0.8	0.0	0.6	25.1	1.2	1.3	0.8	1.6
VC-MQ	HPLC-EC	4.31	0.00	13.4	1.0	2.7	0.0	2.2	33.8	2.8	2.1	2.4	3.2
VC-MV	24DNPH	4.00	0.00	17.3	0.2	0.2	0.2	0.2	35.5	1.3	0.5	1.3	1.4
VC-MX	HPLC-UV	1.93	0.11	14.7		1.0			32.6		0.2		
VC-MZ	24DNPH	2.85	0.06	12.6	1.3	0.5	1.3	1.4	32.6	0.3	0.4	0.2	0.4
VC-NA	HPLC-UV	1.43	0.04	9.6	0.2	0.3	0.0	0.3	26.1	0.1	2.1	0.0	1.5
VC-ND	HPLC-EC	4.50	0.28	17.3	1.6	0.2	1.6	1.6	37.6	1.6	0.6	1.6	1.7
VC-NJ	AO	2.13	0.20	13.1	0.6	0.8	0.3	0.8	28.0	0.6	0.3	0.6	0.6
VC-NL	HPLC-EC	24.04	0.00	13.1		0.0			25.8		0.4		
VC-NR	HPLC-UV			13.0	1.1	0.5	1.0	1.1	30.6	1.0	5.9	0.0	4.3
	N	15		17					17				
	Min	1.4	0.1	9.6	0.0	0.0	0.0	0.2	25.1	0.1	0.0	0.0	0.2
	Median	2.8		13.4	0.6	0.4	0.2	0.8	30.7	0.7	0.5	0.6	1.3
	Max	24.0		17.3	1.9	2.7	1.6	2.3	37.6	2.8	5.9	2.4	4.3
	eSD	1.7		1.2					3.6				
	eCV	61		9					12				

AO	Ascorbate oxidase
AutoAnal	Auto Analyzer
24DNPH	2,4-Dinitrophenylhydrazine
EC	Electrochemical detector
HPLC	Liquid chromatography
OPD	Orthophenlyenediamine
UV	Ultraviolet absorbance