NISTIR 7880-30

NIST Micronutrients Measurement Quality Assurance Program Winter, Spring, and Fall 1994 Comparability Studies

Results for Round Robins XXX, XXXI, and XXXII
Fat-Soluble Vitamins and Carotenoids in Human Serum
and Round Robins 5 and 6 Ascorbic Acid in Human Serum

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This publication is available free of charge from:
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National Institute of
Standards and Technology
U.S. Department of Commerce

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November 2014

U.S. Department of Commerce
Penny Pritzker, Secretary

National Institute of Standards and Technology
Willie E. May, Acting Under Secretary of Commerce for Standards and Technology and Acting 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 1994 MMQAP measurement comparability improvement studies: 1) Round Robin XXX Fat-Soluble Vitamins and Carotenoids in Human Serum, 2) Round Robin XXXI Fat-Soluble Vitamins and Carotenoids in Human Serum, 3) Round Robin XXXII Fat-Soluble Vitamins and Carotenoids in Human Serum, 4) Round Robin 5 Ascorbic Acid in Human Serum, and 5) Round Robin 6 Ascorbic Acid in Human Serum. The materials for Round Robin XXX were shipped to participants in January 1994; participants were requested to provide their measurement results by March 19, 1994. The materials for Round Robin XXXI were shipped to participants in April 1994; participants were requested to provide their measurement results by July 25, 1994. The materials for Round Robin XXXII were shipped to participants in July 1994; participants were requested to provide their measurement results by August 30, 1994. The sample materials for Round Robin 5 were distributed in April 1994 with results due by May 31, 1994. The sample materials for Round Robin 6 were distributed in August 1994 with results due by September 30, 1994.

Keywords

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

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Introduction

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

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin XXX comparability study (hereafter referred to as RR30) 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 1994. 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 some participants report values for non-target analytes.

The final report delivered to every participant in RR30 has three sections:

- A cover letter and a "Lies, Damned Lies, and Statistics" summary report that describes the samples and our analysis of the participants' results. This cover letter and summary report are reproduced as Appendix B.

- The "All-Lab Report" that lists all of the reported measurement results and a number of consensus statistics for analytes reported by more than one participant. 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. An example "Individualized Report" is reproduced as Appendix D.

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

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin XXXI comparability study (hereafter referred to as RR31) 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 1994. 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 some participants report values for non-target analytes.

The final report delivered to every participant in RR31 has three sections:

- A cover letter and a "Lies, Damned Lies, and Statistics" summary report that describes the samples and our analysis of the participants' results. This cover letter and summary report are reproduced as Appendix F.

- The "All-Lab Report" that lists all of the reported measurement results and a number of consensus statistics for analytes reported by more than one participant. 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. An example "Individualized Report" is reproduced as Appendix H.

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

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin XXXII comparability study (hereafter referred to as RR32) 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 1994. 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 some participants report values for non-target analytes.

A preliminary report for RR32 was mailed to all participants shortly before the Micronutrients QA Workshop held on October 15, 1994. Based upon feedback from the Workshop participants, the format of the "All-Lab Report" section of the Final report was considerably modified. The revised final report delivered to every participant in RR32 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 listing of summary statistics for Total Retinol, α-Tocopherol and Total β-Carotene in all sera that had been distributed more

than once from RR01 through RR32. This cover letter and associated reports are reproduced as Appendix J.

- The "All-Lab Report" that lists all of the reported measurement results and a number of consensus statistics for analytes reported by more than one participant. 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. An example "Individualized Report" is reproduced as Appendix L.

Round Robin 5: Vitamin C in Human Serum

Participants in the MMQAP Vitamin C in Human Serum Round Robin 5 comparability study (hereafter referred to as RR05) received four frozen serum test samples, two ampoules of each of two different materials. These samples were shipped on dry ice to participants in January 1994. The available 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.

The final report delivered to all participants in RR05 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 RR05, Appendix O is a revised "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 RR05.

Round Robin 6: Vitamin C in Human Serum

Participants in the MMQAP Vitamin C in Human Serum Round Robin 6 comparability study (hereafter referred to as RR06) received four frozen serum test samples and four frozen serum bottles of control materials for analysis. Both the test and control materials consisted of two containers each of two different materials. These materials were shipped on dry ice to participants in August 1994. The communication materials included in the sample shipment are provided in Appendix P.

The test and control 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 analyze the control materials and evaluate their results before analyzing the test materials, but were not asked to report the values that they obtained for the control materials. Participants were asked to provide two results for each vial of the test samples.

As described in NISTIR 7880-29, the data and results for RR06 were reported August 1995 in combination with the data and results for RR07. While not distributed to the participants in RR06,

Appendix Q 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 RR06.

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 1994;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 1994;42(8):1257-1262.

Appendix A. Shipping Package Inserts for RR30

The following two items were included in each package shipped to an RR30 participant:

- Cover letter
- Datasheet

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

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

January 5, 1994

Dear Colleague:

The 1994 Micronutrient Quality Assurance Program will include three fat-soluble vitamins and carotenoid compounds in serum studies. The core analytes in the program will be: retinol, retinyl palmitate, α-tocopherol, γ-tocopherol, and total β-carotene. Data will also be collected and analyzed for α-carotene, trans β-carotene, β-cryptoxanthin, lutein, total lycopene, trans-lycopene, and zeaxanthin.

Enclosed is the set of samples for the first 1994 Round Robin exercise (Round Robin XXX). You will find duplicate vials of four lyophilized sera 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 the detection limit, please indicate this result on the form by using ND *(Not Detected)*. For values not obtained, please leave a blank for the given analyte. Results will be due to NIST by March 19, 1994. Written feedback concerning the study will be provided to you by April 25, 1994.

Samples should be reconstituted with 1.0 mL of HPLC-Grade water or equivalent. We recommend that dissolution be facilitated with 3 to 5 minutes agitation in an ultrasonic bath or at least 30 min at room temperature with intermittent swirling. (CAUTION: Vigorous shaking will cause foaming an 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, 3450 at 472 nm.

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

Ms. Donna Sirk
NIST
Bldg. 222, Rm. B156
Gaithersburg, MD 20899
FAX: (301) 926-8671

If you have questions regarding this round robin exercise, please call me at (301) 975-3120 or mail/FAX queries to the above address.

Sincerely,

Jeanice Brown Thomas

Jeanice Brown Thomas
Clinical Coordinator
Organic Analytical Research Division
Chemical Science and Technology Laboratory

Attachment

cc: W. May

ROUND ROBIN XXX RESULTS FROM LABORATORY # _____
DATE OF ANALYSIS _____
RESULTS IN ug/mL

SAMPLE NUMBER	ANALYTE	RESULT
SERUM 191	RETINOL	_____
	ALPHA-TOCOPHEROL	_____
VIAL # ___	BETA-CAROTENE	_____
SERUM 192	RETINOL	_____
	ALPHA-TOCOPHEROL	_____
VIAL # ___	BETA-CAROTENE	_____
SERUM 193	RETINOL	_____
	ALPHA-TOCOPHEROL	_____
VIAL # ___	BETA-CAROTENE	_____
SERUM 194	RETINOL	_____
	ALPHA-TOCOPHEROL	_____
VIAL # ___	BETA-CAROTENE	_____
SERUM	RETINOL	_____
	ALPHA-TOCOPHEROL	_____
VIAL # ___	BETA-CAROTENE	_____

OPTIONAL ANALYTES: SUPPLY ONE RESULT IF AVAILABLE

SERUM #	191	192	193	194
TRANS-BETA CAROTENE				
ALPHA-CAROTENE				
RETINYL PALMITATE				
GAMMA-TOCOPHEROL				
LYCOPENE (TOTAL)				
9-CIS-BETA CAROTENE				
13-CIS-BETA-CAROTENE				
LUTEIN				
ZEAXANTHIN				
BETA-CRYPTOXANTHIN				

DIRECTIONS: Reconstitute with 1 mL distilled water.

RESULTS DUE BY: March 19, 1994
FAX RESULTS TO 301/926-8671

Appendix B. Final Report for RR30

The following five pages are the final report for RR30 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

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

April 21, 1994

Dear Colleague:

Enclosed is the summary report of the results for Round Robin XXX (Sera 191-194). You will find the data presentation in this report somewhat different from that in the past. We hope that you will find it more useful and convenient for assessing and tracking your laboratory's performance. I would appreciate your comments.

The changes incorporated in this data presentation are explained in detail in the attached report from Dave Duewer and Margaret Kline. Please note that in the new data presentation, the **median** (the value for which 50% of the data are larger and 50% are smaller) is used to assign the consensus laboratory value. The use of the median helps eliminate data biases against new laboratories. After evaluation of all previous round robin data, we find that the median is in excellent agreement with the trimmed core average, which was used for value assignment in the past.

Tables 1 and 2 provide a summary of data for all laboratories. The overall laboratory performance for retinol, and γ- and α-tocopherol is good. The average estimated coefficient of variation (eCV) is about 11% for retinol, 9.5% for α-tocopherol, and about 9% for γ-tocopherol. The eCV for total β-carotene is approximately 21%. The high variation for β-carotene is again partly due to the difficulty of making measurements at the parts-per-billion (ppb) range. This is indicated in Serum 194 where the concentration of total β-carotene is <100 ppb. For most of the reporting laboratories, this level is at the limit of quantification.

The concentrations of the remaining analytes in Tables 2 and 3 (lycopene, retinyl palmitate, and other carotenoid compounds) appear to be too low for a fair assessment of either overall or individual measurement capabilities. However, since the number of laboratories that measure these analytes seems to be increasing, we will evaluate the possibility of enriching the sera with selected analytes for future round robin studies.

Data for evaluating your laboratory's performance relative to the other participants is provided in Table 4. Your laboratory's individual laboratory performance is provided in pages 1-6 of the attached report and includes a graphical summary of your laboratory's past 3 years' performance and a tabular summary of your inter-laboratory accuracy and precision.

The performance criteria previously established for past round robins is used to summarize laboratory performance for Round Robin XXX. By convention, 0-5% bias from the assigned value represents **EXCEPTIONAL** performance, 6-10% **ACCEPTABLE** performance, 11-20% **MARGINAL** performance and >20% **POOR** performance relative to the current state-of-the-practice for these measurements.

If you have concerns regarding your performance or are a lab whose performance would be rate "U" based on the convention stated above, we suggest that you obtain a unit of SRM 968a and analyze all three levels. If, with minor method modifications your measured values do not agree with the certified values, please contact us for consultation. SRM 968a can be obtained through the NIST Standard Reference Materials Program (301/975-6776 or FAX: 301/948-3730).

Round Robin XXXI samples will be shipped during the week of April 25. If you do not receive these samples, please call Ms. Donna Sirk at 301/975-3174. Results are due by June 14. We expect to provide you feedback concerning your performance by July 25.

We have been notified by NCI that their interests will be focused on new agents undergoing Phase I clinical trials. Therefore, we are currently evaluating options (such as pricing and round robin distributions) for maintaining the quality of service that you have received in the past. We will provide you with further details within the next few months.

If you have questions, please contact me at 301/975-3120.

Sincerely,

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

Enclosures

cc: W. May

"Lies, Damned Lies, and Statistics"

Mark Twain

Brace yourselves, the attached Round Robin XXX Report differs from its predecessors.

The most obvious changes are in the format of the "All Lab" data list and the major analyte summary charts for each laboratory. We've added an individualized listing of your submitted data and three new summaries: a rank-ordering of all laboratories based on similarity to the consensus values, individualized scatterplots of your data against the consensus values, and an individualized "precision and accuracy" breakdown. Less obviously, we've made major philosophical changes to our statistical analysis.

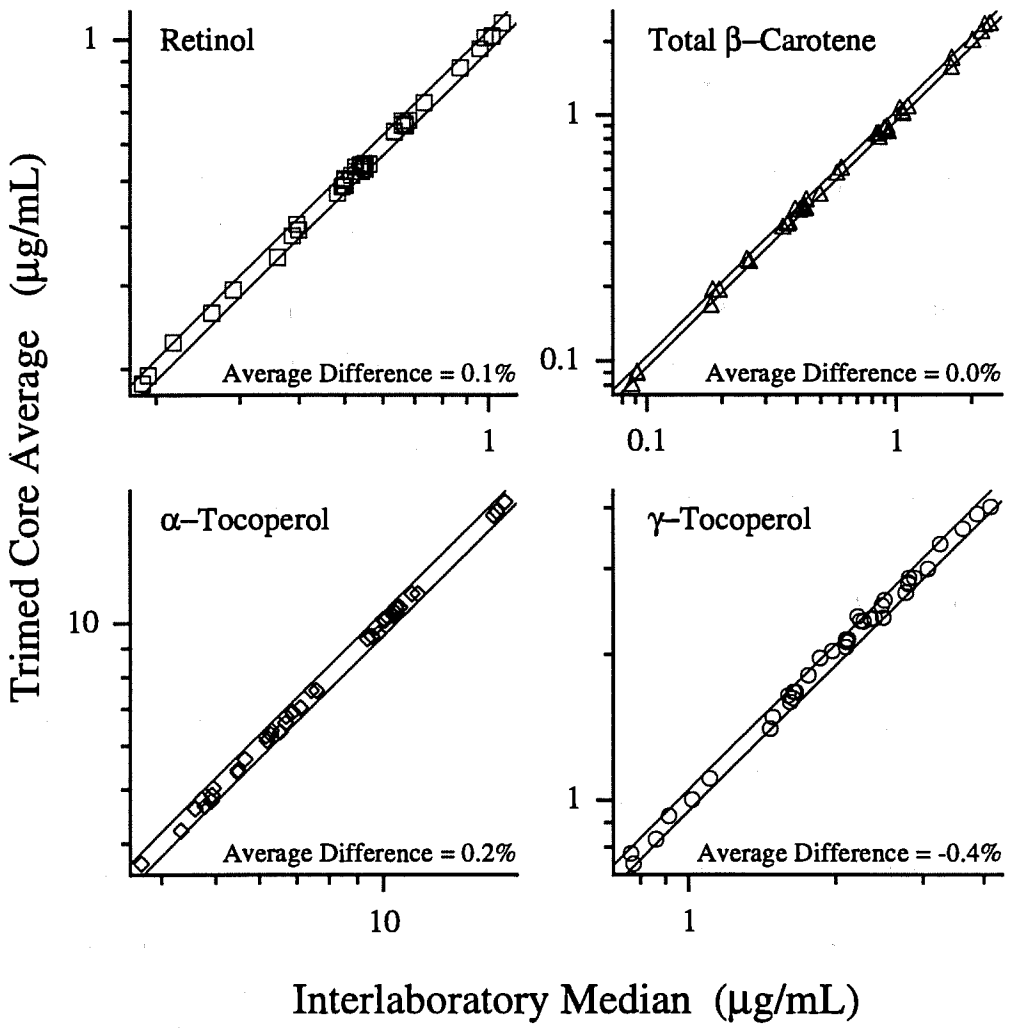
Earlier Reports used "core groups" of experienced laboratories to define the consensus inter-laboratory average and standard deviation values. In statistical jargon, this is "trimming" or "censoring" the data. While a routine and accepted practice, effective trimming requires considerable time and agony on the part of the data analyst - decisions are always subjective and somewhat arbitrary. After study of all previous Round Robin data, we find that robust percentile statistics give easy-to-automate results which are in excellent agreement with the past trimmed values (see overleaf).

This change is less scary than it sounds. Our new "best value" statistic - the median (the value for which 50% of the data are larger, 50% smaller) - is doubtless familiar to you. It has the wonderful property of ignoring weird values, as long as there is a good fistful of data with about equal numbers of high and low values. In the ideal case, where all data define a nice symmetric distribution, the average and the median give the same value.

Our new "inter-laboratory variation" statistic - the interquartile range or IQR (the span of the central 50% of the data) - may not be as familiar, but it is a well established measure of dispersion. Like the median, the IQR is resistant to a few weird values. For an ideal normal distribution, 0.741(IQR) is equal to the standard deviation. We're also in the process of rethinking how to compare variation over analytes, concentration ranges, and time. We will discuss this in the Round Robin XXXI report.

The take-home message for now is that our philosophical changes give the same practical results. Of course, our NIST results continue to provide you with independent assessments of the analyte concentrations. With the exceptions of the minor analytes α-Carotene and β-Cryptoxanthin, NIST results continue in good accord with the inter-laboratory consensus values.

Inter-laboratory Median versus Trimmed Core Average
for Major Analytes of Round Robin XXI - XXIX



The parallel lines in each graphical segment show ±5% difference from equality, where the % difference is defined as: $100 \frac{\text{(Trimmed Core Average - median)}}{\text{median}}$.

There is excellent agreement between the old and the new consensus "best-value" statistics for the past three years (Round Robin XXI to XXIX), with no apparent systematic offsets. Even the extreme differences (smallest negative, largest positive) are modest: Retinol (-4.6%, 4.4%); α-Tocopherol (-1.8%, 3.0%), γ-Tocopherol (-8.1%, 4.7%), and total β-Carotene (-6.3%, 6.9%).

This Round Robin XXX Report includes the following:

- The "All Laboratory Data Report"

  Table 1: "Major" Analytes (2 pages) and Table 2: "Minor" Analytes (2 pages).
  Each laboratory's data on a single line, the uncensored mean and standard deviation, and the percentile-based consensus values. (Ok, so α-Carotene isn't a "major" analyte by concentration, number of reporting laboratories, or known importance... It's in Table 1 to save paper...)

  Table 3: Analytes Reported By Only One Laboratory (1 page)
  Information values for all analytes reported by only one laboratory. The legend for Tables 1 and 2 is also presented on this page.

- Relative Performance and Experience Factors (1 page):

  Table 4: Relative Laboratory Rankings
  The consensus statistics come from the central 50% of the data. This presents one way of telling whether you are part of the data-defined center!

  Table 5: Experience Factors Estimated by Number of Round Robin Participations.
  We also try to evaluate whether performance is related to the number of Round Robin participations. We believe it does, particularly when analyzing all three major analytes.

- Your Laboratory's "Individualized Report" (6 pages).

  Pg. 1: Your data and the consensus values.

  Pg. 2-4: Graphical summaries of your past 3 years' performance, as measured by the percent difference from the median for the major analytes: Retinol, α- and γ-Tocopherol, and Total- and all-trans-β-Carotene.

  Pg. 5: Graphical summary of your past 3 years' performance, as measured by the scatterplot of your data vs. the consensus values for the major analytes.

  Pg. 6: Tabular summary of your inter-laboratory comparison "accuracy" and "precision" for your past 3 years' performance.

We would appreciate hearing from you about the report: the new formats, the new summaries, and the statistical changes. Corrections, additions, deletions, strokes, and screams-of-rage are all solicited. Please let us know if <u>any</u> of our recorded data doesn't accurately reflect your real measurements!

Dave Duewer
Research Chemometrician
301-975-3935
DLDuewer@enh.NIST.gov

Margaret Kline
Research Biologist

Appendix C. "All-Lab Report" for RR30

The following 6 pages are the "All-Lab" and the associated "Relative Performance and Experience Factors" reports for RR30 as provided to all participants, with three exceptions:

- the participant identifiers (Lab) have been altered.
- the order in which the participant results are listed has been altered.
- the robust coefficient of variation has been recalculated using a more efficient robust estimator of the standard deviation: the adjusted median absolute deviation from the median (MADe) rather than the adjusted interquartile range (IQR).

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.

All Laboratory Data Report for Round Robin XXX

Table 1: "Major" Analytes (micrograms/milliliter)

	Total Retinol				α-Tocopherol				γ/β-Tocopherol				Total β-Carotene				trans-β-Carotene				Total α-Carotene			
Lab	191	192	193	194	191	192	193	194	191	192	193	194	191	192	193	194	191	192	193	194	191	192	193	194
FSV-BA	7.60	1.16	1.09	0.447	26.5	7.21	26.6	7.30	3.57	2.25	5.53	2.87	1.66	0.619	0.393	0.073	1.54	0.566	0.344	0.064	0.036	0.028	0.015	0.013
FSV-BD	8.15	1.22	1.13	0.446	21.4	6.60	25.4	7.20																
FSV-BE	2.26	1.06	0.97	0.399	22.1	7.40	26.8	7.50	3.49	2.55	6.05	3.27	1.92	0.712	0.454	0.069					0.011	0.008	0.008	0.007
FSV-BF	7.14	1.15	1.06	0.460	24.0	8.00	26.4	7.30	3.50	2.60	6.60	3.10	1.67	0.661	0.440	0.076					0.014	0.010	0.012	0.014
FSV-BH	6.51	1.01	0.89	0.432	24.0	7.50	28.0	7.59	2.78	1.98	5.06	2.54	1.59	0.579	0.369	0.048	1.50	0.553	0.345	0.048	0.026	0.012	0.009	0.008
FSV-BI	6.84	1.26	1.09	0.409	27.2	7.38	27.1	7.46	3.77	2.54	6.60	3.25	1.56	0.590	0.375	0.059					0.024	0.014	0.014	0.012
FSV-BJ	6.67	1.31	1.11	0.439	22.7	6.99	25.1	7.45	3.68	2.57	6.36	3.40	1.72	0.653	0.423	0.075					0.038	0.024	0.022	0.014
FSV-BK	6.63	1.05	0.99	0.427	26.5	6.91	26.6	6.81																
FSV-BL	10.20	1.31	1.10	0.520	21.7	7.74	30.0	8.10																
FSV-BM	7.90	0.98	0.62	0.415	26.2	7.60	30.0	7.80																
FSV-BN	5.43	0.95	0.92		24.7	8.51	28.2		3.64	2.43	5.53	2.92	1.32	0.434	0.330	0.057	1.27		0.317	0.042	0.040	0.022	0.013	0.011
FSV-BO	4.36	1.31	1.12	0.433	22.1	7.14	28.3	7.24					1.93	0.654	0.392	0.065					0.019	0.011	0.008	0.005
FSV-BP	6.34	1.06	0.99	0.430	22.4	8.65	29.7	8.50					2.20	0.690	0.468	0.158					0.035	0.023	0.021	0.020
FSV-BQ	5.41	1.05	1.01	0.418	22.7	6.40	21.2	6.60					1.47	0.642	0.448	0.078								
FSV-BR	7.09	1.08	1.07	0.434																				
FSV-BS													>1.04	>0.381	>0.273	>0.141	1.04	0.381	0.273	0.141				
FSV-BT	6.27	1.04	0.93	0.390	23.6	6.74	24.3	6.61	3.59	2.32	5.78	2.88	1.57	0.569	0.361	0.050	1.47	0.534	0.328	0.049	0.021	0.017	0.015	0.016
FSV-BX	8.87	1.40	1.28	0.492	29.8	8.69	34.2	8.52	3.85	2.49	6.72	3.26												
FSV-BY	7.31	1.08	0.86	0.413	24.0	7.28	27.8	7.29	3.66	2.43	6.49	3.01	1.85	0.660	0.334	0.049					0.039	0.018	0.014	0.012
FSV-BZ					26.3	8.30	29.5	6.10	3.33	2.27	5.71	2.30	1.78	0.510	0.280	0.120					0.080	0.050	0.050	0.050
FSV-CA	6.82	1.19	1.03	0.466	25.9	6.79	30.5	6.59																
FSV-CB	6.95	1.19	1.18	0.510	24.2	7.94	28.6	7.76					>1.40	>0.600	>0.190	>0.040	1.40	0.600	0.190	0.040	0.010	0.010	*nq*	*nq*
FSV-CG	15.04	2.59	2.41	1.095	8.2	5.80	14.7	7.22	8.23	5.80	14.70	7.22	3.00	1.104	0.712	0.108					0.085	0.071	0.062	0.058
FSV-CH	6.58	0.95	0.94	0.366	22.6	6.02	23.9	5.81	2.74	1.67	4.41	2.13	1.23	0.439	0.284	0.036					0.017	*nq*	0.004	0.003
FSV-CJ	6.45	1.05	1.08	0.436	22.8	6.88	26.4	7.45					1.33	0.487	0.344	0.053					*nq*	*nq*	*nq*	0.014
FSV-CK	7.13	1.27	1.16	0.425	20.9	7.18	27.7	7.17	3.46	2.62	6.57	3.29	1.91	0.676	0.396	0.062					0.040	0.024	0.018	0.019
FSV-CL	7.57	1.30	1.32	0.580	27.7	8.93	34.9	8.01	4.32	3.01	8.25	3.60	1.51	0.590	0.410	0.100								
FSV-CM					27.3	9.07	36.3	8.59																
FSV-CO	6.83	1.04	1.06	0.403	23.7	6.97	26.9	6.85					1.71	0.601	0.400	0.069	1.65	0.553	0.354	0.051	0.042	0.022	0.017	0.014
FSV-CP	5.22	0.88	0.82	0.325	14.6	4.33	17.5	5.33					1.19	0.459	0.276	0.036					0.026	0.015	0.013	0.010
FSV-CQ	6.86	1.06	1.02	0.420	26.6	7.36	28.0	7.48					1.62	0.750	1.040	*nq*								
FSV-CR	6.42	1.10	1.05	0.475	23.2	7.23	27.1	7.39																
FSV-CT	9.30	1.43	0.82	0.472	42.8	8.41	41.7	9.64					1.76	0.620	0.452	0.060	1.39	0.468	0.305					
FSV-CU	6.71	1.04	1.01	0.405	24.7	6.80	23.5	6.16					1.90	0.669	0.428	0.112	1.62	0.612	0.367	0.078				
FSV-CV	7.23	1.21	1.06	0.460	25.0	7.80	27.7	7.65	3.44	2.53	6.40	3.14	1.69	0.600	0.410	0.045					0.012	0.050	0.050	0.010
FSV-CX	6.54	0.96	0.95	0.400	21.4	6.90	25.0	7.12	3.36	2.36	5.90	3.18	1.27	0.380	0.220	0.030					<0.01	<0.01	<0.01	<0.01
FSV-CY		1.00	1.01	0.410	24.0	6.95	27.3	7.10					1.66	0.650	0.380	<0.08								
FSV-DJ		1.16	0.97	0.460		7.80	29.4	7.90																
FSV-DL	4.51	0.77	0.69	0.379	14.8	4.64	17.6	5.98	3.20	2.56	6.27	4.26	1.54	0.606	0.364	0.057					0.021	0.014	0.010	0.011
FSV-DM	7.35	1.16	1.08	0.433	21.5	5.60	22.6	5.46					1.67	0.558	0.357	0.047								
FSV-DS	5.17	1.05	0.74	0.390	20.3	7.73	23.2	7.64					1.49	0.642	0.235	0.060								

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All Laboratory Data Report for Round Robin XXX

Table 1: "Major" Analytes (micrograms/milliliter)

	Total Retinol				α-Tocopherol				γ/β-Tocopherol				Total β-Carotene				trans-β-Carotene				Total α-Carotene			
Lab	191	192	193	194	191	192	193	194	191	192	193	194	191	192	193	194	191	192	193	194	191	192	193	194
FSV-DX	8.18	1.11	1.00	0.422	22.4	7.41	26.9	8.73					1.28	0.463	0.296	0.043	0.02	0.016	0.012	0.010				
FSV-EA	8.53	1.17	1.06	0.430	26.0	7.40	27.2	7.00	4.30	3.00	7.10	3.60	1.59	0.570	0.370	0.570					0.043	0.016	0.016	0.012
FSV-EC	8.41	1.29	1.00	0.429	24.1	7.15	25.5	6.97					1.21	0.438	0.257	0.047								
FSV-EK	7.92	1.28	1.18	0.488	27.2	7.85	30.6	7.60	4.10	2.67	7.38	3.50	1.14	0.466	0.312	*nq*								
FSV-EX	5.86	0.94	0.86	0.370	24.1	7.28	28.5	7.43					1.76	0.610	0.400	0.050								
FSV-FF		1.27	1.08	0.434									1.65	0.573	0.362	0.081								
n	41	44	44	43	43	44	44	43	20	20	20	20	35	35	35	32	10	9	10	9	21	20	20	21
Min	2.26	0.77	0.62	0.325	8.2	4.33	14.7	5.33	2.74	1.67	4.41	2.13	1.14	0.380	0.220	0.030	0.02	0.016	0.012	0.010	0.010	0.008	0.004	0.003
Mean	7.04	1.16	1.04	0.449	23.8	7.26	27.1	7.29	3.80	2.63	6.67	3.34	1.64	0.598	0.393	0.083	1.29	0.476	0.284	0.058	0.032	0.023	0.020	0.016
Max	15.04	2.59	2.41	1.095	42.8	9.07	41.7	9.64	8.23	5.80	14.70	7.22	3.00	1.104	1.040	0.570	1.65	0.612	0.367	0.141	0.085	0.071	0.062	0.058
SD	1.90	0.26	0.25	0.110	4.8	0.98	4.7	0.86	1.12	0.80	2.06	1.03	0.34	0.126	0.142	0.093	0.48	0.186	0.108	0.036	0.020	0.016	0.016	0.013
CV	27	23	24	25	20	13	17	12	29	30	31	31	21	21	36	113	37	39	38	62	62	70	80	85
NIST1	6.99	1.12	0.93	0.443	22.0	6.88	26.0	7.26	2.89	2.05	5.78	2.57	1.62	0.550	0.367	0.054	1.59	0.501	0.345	0.053				
NIST3	6.62	1.00	0.88	0.411	22.7	6.89	27.3	7.14	3.34	2.34	6.14	3.09	1.67	0.612	0.404	0.058	1.55	0.557	0.351	0.050	0.059	0.034	0.026	0.015
Median	6.84	1.10	1.03	0.430	24.0	7.28	27.1	7.30	3.58	2.54	6.38	3.22	1.65	0.601	0.375	0.060	1.44	0.553	0.323	0.049	0.026	0.018	0.015	0.012
eCV	12	13	10	9	12	10	10	7	8	7	13	13	13	13	18	33	14	13	12	27	68	51	42	25

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All Laboratory Data Report for Round Robin XXX

Table 2: "Minor" Analytes (micrograms/milliliter)

	Total Lycopene				Total β-Cryptoxanthin				Total Lutein				Total Zeaxanthin				Total Lutein&Zeaxanthin				Retinyl Palmitate			
Lab	191	192	193	194	191	192	193	194	191	192	193	194	191	192	193	194	191	192	193	194	191	192	193	194
FSV-BA					0.051	0.067	0.056	0.062													0.139	0.066	0.036	0.021
FSV-BD																								
FSV-BE																								
FSV-BF	0.274	0.81	0.81	0.65																				
FSV-BH	0.134	0.51	0.41	0.43	0.052	0.072	0.062	0.057	0.047	0.072	0.046	0.056	*nq*	0.036	*nq*	*nq*		*0.108*			0.160	*nq*	*nq*	*nq*
FSV-BI	0.152	0.57	0.45	0.50	0.049	0.056	0.048	0.050	0.024	0.064	0.050	0.065	0.011	0.035	0.030	0.035	*0.035*	*0.099*	*0.080*	*0.100*	0.186	*nq*	*nq*	*nq*
FSV-BJ	0.229	0.87	0.67	0.79																	0.185	*nq*	*nq*	*nq*
FSV-BK																								
FSV-BL																								
FSV-BM																								
FSV-BN	0.129	0.36	0.26		0.035	0.041	0.041	0.035	0.011	0.033	0.029	0.036	0.005	0.017	0.011	0.018	*0.016*	*0.050*	*0.040*	*0.054*	0.136	*nq*	*nq*	*nq*
FSV-BO	0.208	0.50	0.43	0.41	0.050	0.063	0.052	0.052	0.048	0.128	0.101	0.130												
FSV-BP	0.287	0.72	0.62	0.61	0.030	0.069	0.035	0.028																
FSV-BQ																								
FSV-BR																								
FSV-BS																								
FSV-BT	0.117	0.44	0.34	0.34	0.045	0.055	0.045	0.043	0.023	0.082	0.058	0.079	0.007	0.028	0.023	0.029	*0.030*	*0.110*	*0.081*	*0.108*	0.041	0.020	0.009	0.006
FSV-BX																								
FSV-BY	0.167	0.62	0.51	0.48	0.056	0.075	0.056	0.057	0.023	0.074	0.053	0.063	0.010	0.039	0.030	0.033	0.035	0.114	0.095	0.105	0.126	0.024	0.015	0.014
FSV-BZ	0.690	0.84	0.75	0.68					0.104	0.104	0.120	0.100												
FSV-CA																								
FSV-CB	0.150	0.65	0.27	0.28	0.030	0.040	0.030	0.030									0.100	0.190	0.210	0.230				
FSV-CG	0.186	0.71	0.59	0.59	0.121	0.170	0.141	0.142													0.432	0.391	0.382	0.363
FSV-CH	0.103	0.39	0.31	0.32																				
FSV-CJ	0.155	0.54	0.47	0.47	0.043	0.070	0.054	0.056									0.040	0.124	0.104	0.120				
FSV-CK	0.193	0.71	0.55	0.63	0.035	0.476	0.048	0.122									0.045	0.128	0.105	0.122				
FSV-CL	0.150	0.63	0.54	0.54	0.070	0.090	0.070	0.060	0.070	0.160	0.150	0.120												
FSV-CM																								
FSV-CO	0.144	0.48	0.39	0.39	0.052	0.088	0.058	0.061	0.035	0.106	0.099	0.106	0.020	0.107	0.087	0.081	*0.055*	*0.213*	*0.186*	*0.187*				
FSV-CP	0.164	0.59	0.51	0.47	0.041	0.048	0.041	0.040									0.057	0.110	0.092	0.107				
FSV-CQ																								
FSV-CR																								
FSV-CT									0.031	0.088	0.060	0.076	0.018	0.051	0.043	0.046	*0.049*	*0.139*	*0.103*	*0.122*				
FSV-CU																					0.152	0.109	0.106	0.074
FSV-CV	0.265	0.78	0.66	0.58																				
FSV-CX	0.170	0.62	0.48	0.51	0.050	0.050	0.030	0.030	0.040	0.110	0.070	0.100									0.160	0.090	0.060	0.060
FSV-CY																								
FSV-DJ																								
FSV-DL	0.095	0.32	0.29	0.39	0.055	0.083	0.061	0.057																
FSV-DM																								
FSV-DS																								

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All Laboratory Data Report for Round Robin XXX

Table 2: "Minor" Analytes (micrograms/milliliter)

	Total Lycopene				Total β-Cryptoxanthin				Total Lutein				Total Zeaxanthin				Total Lutein&Zeaxanthin				Retinyl Palmitate			
Lab	191	192	193	194	191	192	193	194	191	192	193	194	191	192	193	194	191	192	193	194	191	192	193	194
FSV-DX	0.207	0.72	0.62	0.65					0.029	0.083	0.075	0.083												
FSV-EA	0.170	0.55	0.46	0.48	0.050	0.059	0.059	0.055	0.052	0.201	0.173	0.191												
FSV-EC																								
FSV-EK	*nq*	0.60	0.50	0.51					*nq*	0.153	0.129	0.146												
FSV-EX																								
FSV-FF																								
n	23	24	24	23	18	18	18	18	13	14	14	14	6	7	6	6	10	11	10	10	10	6	6	6
Min	0.095	0.32	0.26	0.28	0.030	0.040	0.030	0.028	0.011	0.033	0.029	0.036	0.005	0.017	0.011	0.018	0.016	0.050	0.040	0.054	0.041	0.020	0.009	0.006
Mean	0.197	0.61	0.50	0.51	0.051	0.093	0.055	0.058	0.041	0.104	0.087	0.097	0.012	0.045	0.037	0.040	0.046	0.126	0.110	0.126	0.172	0.117	0.101	0.090
Max	0.690	0.87	0.81	0.79	0.121	0.476	0.141	0.142	0.104	0.201	0.173	0.191	0.020	0.107	0.087	0.081	0.100	0.213	0.210	0.230	0.432	0.391	0.382	0.363
SD	0.119	0.15	0.15	0.13	0.020	0.100	0.024	0.030	0.024	0.044	0.043	0.041	0.006	0.029	0.026	0.022	0.023	0.044	0.051	0.049	0.100	0.139	0.142	0.137
CV	60	25	30	25	40	107	44	51	59	42	50	42	51	66	71	54	49	35	46	39	58	119	140	152
NIST1																								
NIST3	0.204	0.63	0.55	0.53	0.036	0.051	0.041	0.041	0.032	0.099	0.077	0.082	0.011	0.039	0.034	0.030	*0.043*	*0.138*	*0.111*	*0.112*	0.116	*nq*	*nq*	*nq*
Median	0.167	0.61	0.49	0.50	0.050	0.068	0.053	0.056	0.035	0.096	0.073	0.092	0.011	0.036	0.030	0.034	0.042	0.114	0.099	0.114	0.156	0.078	0.048	0.041
eCV	29	26	30	27	16	31	22	16	51	36	54	45	64	33	49	37	35	18	18	11	23	81	111	110

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All Laboratory Data Report for Round Robin XXX

Table 3: Analytes Reported By Only One laboratory

All concentrations in micrograms/milliliter

Analyte	Code	191	192	193	194
δ-Tocopherol	NISTb	0.22	0.14	0.47	0.13
9-&13-cis-β-Carotene	FSV-BT	0.03	0.09	0.08	0.10
trans-α-Carotene	NISTb	0.025	0.021	0.018	0.015
dihydro-Lycopene	FSV-BT	0.033	0.086	0.083	0.095

Legend for Tables 1 and 2

nq Not quantitatively determined (i.e., reported as '0', 'trace', 'not determined', etc.)

<x Concentration at or below "limit of quantification" x.

Statistics (NIST's values are excluded from all calculations)

n Number of laboratories reporting quantitative values for this analyte for this serum.

Min Minimum reported value.

Mean Average over all reported values.

Max Maximum reported value.

SD Standard deviation over all values.

CV Coefficient of Variation (% relative standard deviation): for (non-NIST) results: 100*SD/Mean

Median Median over all reported values (i.e., 50% of values are larger, 50% are smaller.)

eCV Estimated CV, based on the adjusted median absolute deviation from the median (MADe).
The original report estimated eCV from the interquartile range (IQR).

Relative Performance and Experience Factors

Table 4

Relative Laboratory Rankings

Lab	N	Ret	aToc	bCar	Overall	gToc
FSV-BA	21	24	17	3	0 *	63
FSV-BD	26	55	55			
FSV-BE	24	69	24	76	66	0
FSV-BF	26	5	29	45	16 *	21
FSV-BH	20	52	14	12	13 *	89
FSV-BI	26	38	31	0	6 *	11
FSV-BJ	12	60	21	30	28 *	26
FSV-BK	11	12	40			
FSV-BL	13	88	67			
FSV-BM	10	76	60			
FSV-BN	8	81	52	64	75	53
FSV-BO	6	74	26	48	53	
FSV-BP	8	14	79	94	69	
FSV-BQ	3	40	71	42	59	
FSV-BR	3	0				
FSV-BS	5			92		
FSV-BT	4	67	45	15	44 *	47
FSV-BX	7	93	93			42
FSV-BY	26	43	2	58	25 *	16
FSV-BZ	19		74	67		74
FSV-CA	25	10	69			
FSV-CB	7	62	43			
FSV-CG	10	100	83	97	97	95
FSV-CH	11	79	76	85	94	95
FSV-CJ	26	26	19	39	19 *	
FSV-CK	11	45	38	61	50 *	32
FSV-CL	1	83	88	27	78	84
FSV-CM	15		95			
FSV-CO	24	33	7	9	3 *	
FSV-CP	8	95	100	91	100	
FSV-CQ	13	2	36	82	34 *	
FSV-CR	3	31	0			
FSV-CT	7	90	98	36	88	
FSV-CU	8	36	62	70	63	
FSV-CV	12	57	33	33	41 *	5
FSV-CX	3	64	50	100	84	37
FSV-CY	17	48	5	18	9 *	
FSV-DJ	3	17	57			
FSV-DL	10	98	90	6	72	58
FSV-DM	9	7	81	24	31 *	
FSV-DS	6	86	64	55	81	
FSV-DX	3	19	48	73	47 *	
FSV-EA	5	29	10	52	22 *	79
FSV-EC	6	50	12	88	56	
FSV-EK	3	71	86	79	91	68
FSV-EX	3	74	19	29	38 *	
FSV-FF	3	21		21		

Legend

Lab	Laboratory code
N	Number of prior Round Robin participations (any analyte)
Ret	Relative Rank for: Retinol
aToc	Relative Rank for: a-Tocopherol
bCar	Relative Rank for: Total b-Carotene
gToc	Relative Rank for: g/b-Tocopherol
Relative Rank	An approximate measure of "closeness" to the interlaboratory medians in Round Robin XXX, expressed on a scale of 0 (closest) to 100 (least close). Each of the listed ranks composites individual rankings for the four sera. Labs with Relative Rank of 0 to 50 define the "Inner" group for a given analyte. Labs ranking 51 to 100 define the "Outer" group.
Overall	An approximate measure of "overall closeness" to the interlaboratory medians in Round Robin XXX, based on averaging the Relative Ranks for Retinol, a-Tocopherol, total b-Carotene assays. Only labs that reported all three analytes in Round Robin XXX have an "Overall" rank.
*	Laboratories with an Overall rank of 0 to

Table 5

Average Number of Round Robin participations in the last "n" Years

	n=10		n=3		n=1	
	In	Out	In	Out	In	Out
Ret	12.7	9.2	6.7	6.0	2.9	2.4
aToc	13.1	8.7	7.2	5.9	2.8	2.5
bCar	13.2	7.4	7.1	5.6	2.7	2.6
Overall	14.5	5.1	7.6	4.6	2.8	2.3
gToc	7.4	3.6	5.8	3.4	2.3	1.7

Does Experience Count?

Looking at the average number of Round Robin participations for labs with Relative Ranks of 0 to 50 for a given analyte (the "Inner" group) versus those with Relative Ranks of 51 to 100 (the "Outer" group), the "Inner" laboratories are consistently enriched in experience. The enrichment is more pronounced for the Overall three-analyte composite than it is for individual analytes. Experience seems to count more as you do more.

Appendix D. Representative "Individualized Report" for RR30

Each participant in RR30 received an "Individualized Report" reflecting their reported results. Each report included a detailed analysis of the results they reported for the following analytes:

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

The following six pages are the "Individualized Report" for the analytes evaluated by participant FSV-NIST1 (listed as FSV-NISTa in the "All Lab Report").

Individualized Round Robin XXX Report to: NIST1

Your Data, Inter-laboratory Medians, and %Differences

	Serum 191				Serum 192				Serum 193				Serum 194			
Analyte	You	Med	%Δ	n	You	Med	%Δ	n	You	Med	%Δ	n	You	Med	%Δ	n
Retinol	6.99	6.85	2	40	1.12	1.11	1	43	0.93	1.03	-10	43	0.44	0.43	3	42
a-Tocopherol	21.99	23.98	-8	42	6.88	7.28	-6	43	26.02	27.06	-4	43	7.26	7.29	0	42
g-Tocopherol	2.89	3.58	-19	20	2.05	2.54	-19	20	5.78	6.38	-9	20	2.57	3.22	-20	20
Total b-Carotene	1.62	1.64	-1	34	0.55	0.60	-8	34	0.37	0.37	-1	34	0.05	0.06	-10	31
trans-b-Carotene	1.59	1.44	11	10	0.50	0.55	-9	9	0.35	0.32	7	10	0.05	0.05	8	9

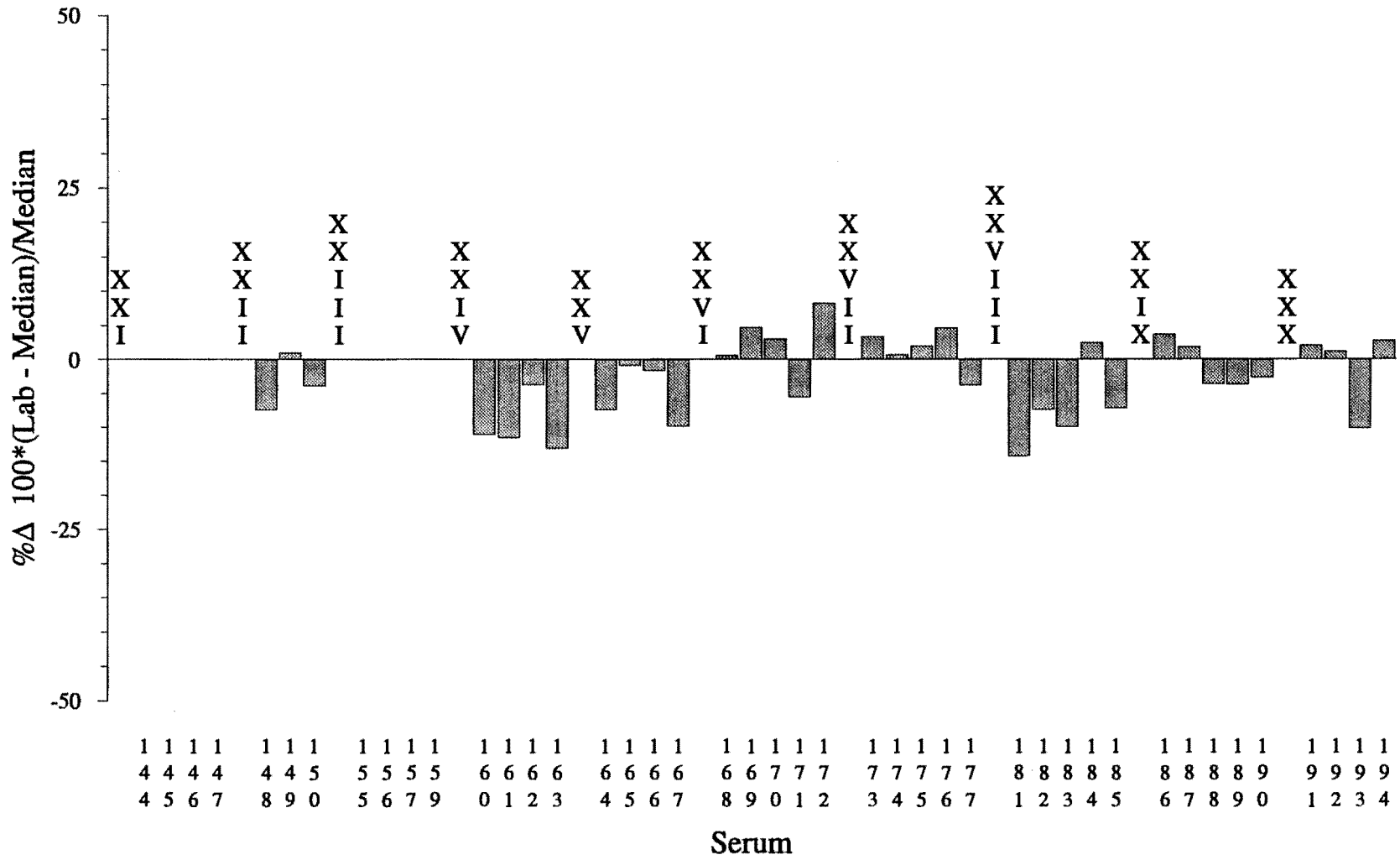
D2

You : Your reported values for the listed analytes (micrograms/milliliter)
Med : The median of the values reported by all participating laboratories (excluding NIST.)
%Δ : The percent difference between your value and the median (reported for main analytes only.)
n : The number of laboratories reporting quantitative values for this analyte in this serum (excluding NIST.)

Please check our recorded values against your records.
Fax any corrections to: (301) 926-8671, c/o Donna Sirk, 222/B156, NIST, Gaithersburg, MD 20899

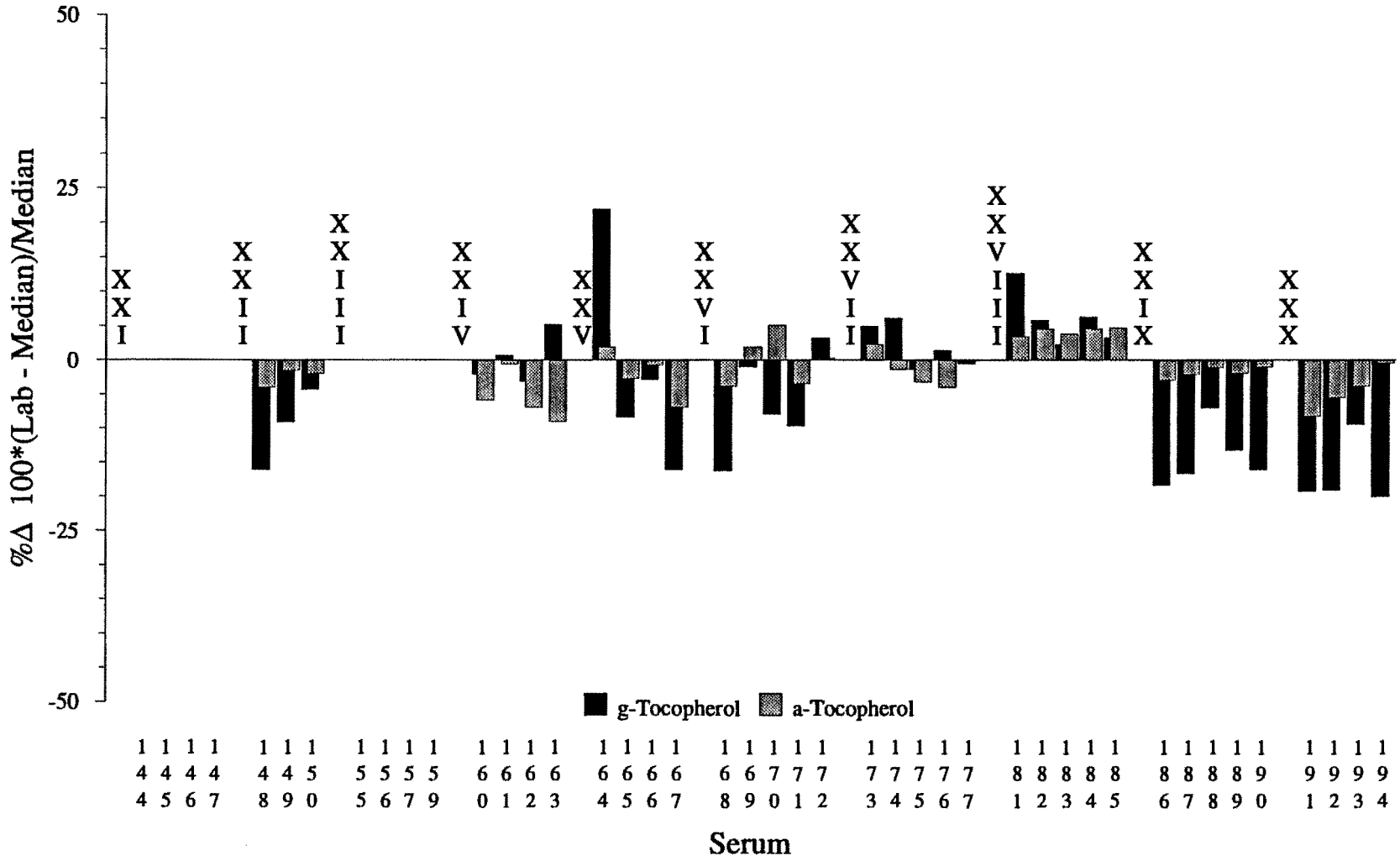
Individualized Round Robin XXX Report to: NIST1

Retinol, %Δ in Round Robin XXI - XXIX



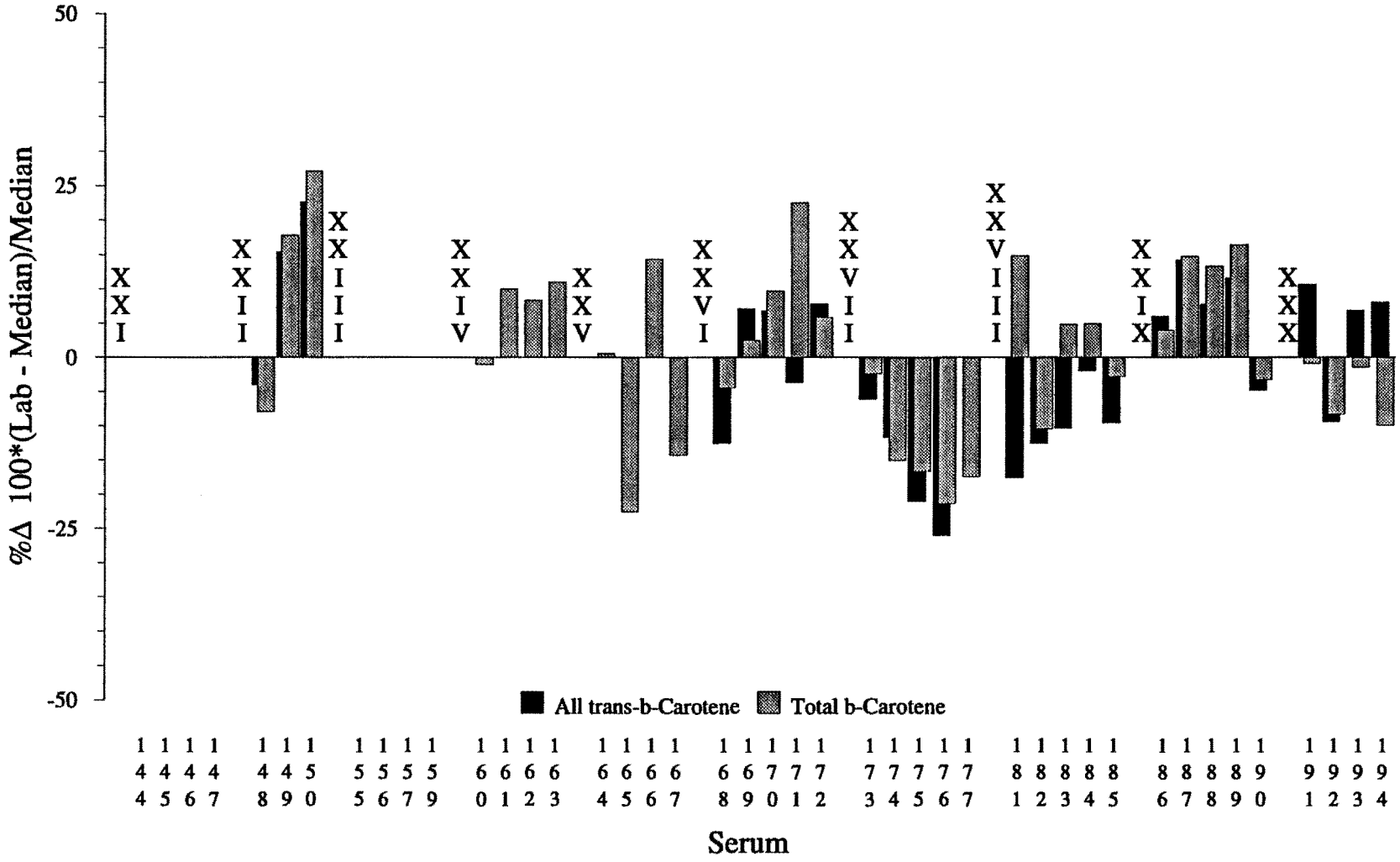
Individualized Round Robin XXX Report to: NIST1

Tocopherol, %Δ in Round Robin XXI - XXIX



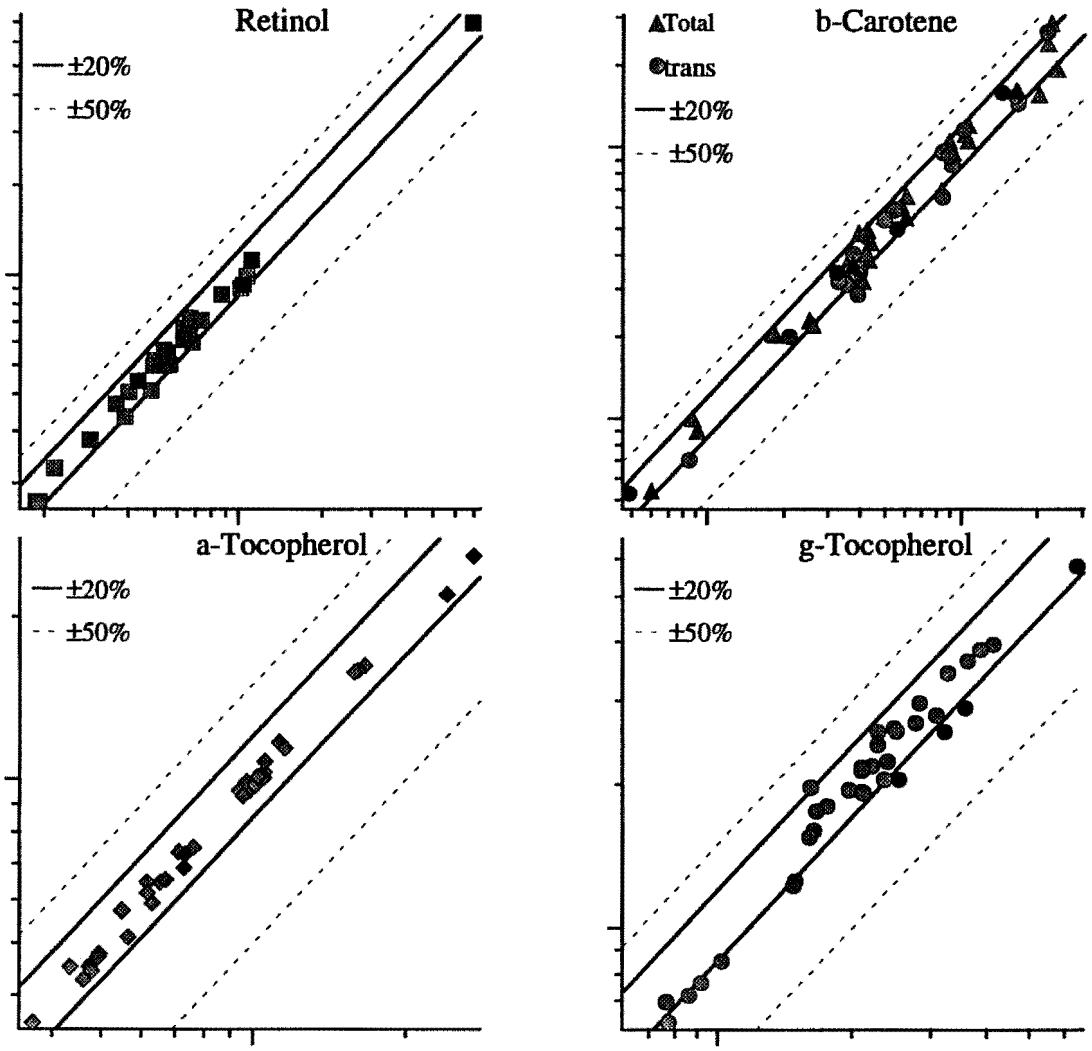
Individualized Round Robin XXX Report to: NIST1

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



Individualized Round Robin XXX Report to: NIST1

Inter-laboratory Median Vs Laboratory NIST1 Values



Legend

Shaded Symbols: Round Robin XXI-XXIX
Black Symbols: Round Robin XXX

Ordinate: Median analyte concentration
Abscissa: Your reported concentration

Interpretation

Adequately intercomparible data are within the ±20% lines. If you have data scattered outside the ±50% lines, your measurement system is not consistent with that of most participating laboratories. If your data are systematically higher or lower than the median, 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 XXX Report to: NIST1

(Provisional) Performance Summary

	Ret		aToc		gToc		Total		trans	
	mΔ	vΔ	mΔ	vΔ	mΔ	vΔ	mΔ	vΔ	mΔ	vΔ
XXI										
XXII	-3	4	-3	1	-10	6	12	18	11	14
XXIII										
XXIV	-10	4	-6	4	0	4	7	6		
XXV	-5	4	-2	4	-1	16	-5	16		
XXVI	2	5	0	4	-6	8	7	10	1	9
XXVII	1	3	-1	3	2	3	-15	7	-16	9
XXVIII	-7	6	4	1	6	4	2	10	-10	6
XXIX	-1	3	-2	1	-14	4	9	8	7	7
XXX	-1	6	-5	3	-17	5	-5	5	4	9

(Provisional) Performance Criteria

In past Round Robins,the absolute value of the %Δ of each measurement was evaluated as follows:

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

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 - median) / median
vΔ : Difference variability, one standard deviation of %Δ for all sera of a RR

Rational

There are no definitive methods for any of these fat-soluble vitamin-related analytes in serum. You can't judge how well you are performing in any absolute sense. However, you can tell how your measurements agree with the group's consensus values - and to ours here at NIST. Just as with absolute comparisons, there are two seperate-but-kindred aspects to this relative comparison. The first, related to accuracy, can be estimated as the "mean difference." This is the average %Δ [100(your value - median)/median] over all sera of a given Round Robin. The second, related to precision, can be estimated as the "difference variability." This is the one standard deviation of the %Δ over all sera of a given Round Robin.

It's obviously best to be accurate and precise (small mΔ, small vΔ.) In some sense, good precision (small vΔ) with poor accuracy (large mΔ) is better than the converse: at least such values are internally consistent and can be related to the group consensus values - IF the mΔ is CONSTANT over time. Poor precision (large vΔ) suggests that your measurement system is qualitatively different from that used by NIST and the majority of participating laboratories.

This all assumes that the expected "%Δ" does not depend on analyte level. This IS NOT true near or below your "limit of quantification," and may not be true enough elsewhere.
More on this later...

Appendix E. Shipping Package Inserts for RR31

The following two or three items were included in each package shipped to an RR31 participant:

- Cover letter
- Datasheet for RR31 serum materials
- Datasheet for soy oil and candidate SRM 2383: Fat-Soluble Vitamins and Carotenoids in Food materials. Only those participants who had previously agreed to evaluate these materials received this datasheet. It is included here because it is discussed in the RR31 cover letter.

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

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

April 19, 1994

Dear Colleague:

Enclosed is the set of samples for Round Robin XXXI. You will find duplicate vials of four lyophilized sera 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 the detection limit, please indicate this result on the form by using ND *(Not Detected)*. For values not obtained, please leave a blank for the given analyte. Results are due to NIST by June 14, 1994. Written feedback concerning the study will be provided to you by July 25, 1994.

Samples should be reconstituted with 1.0 mL of HPLC-grade water or equivalent. We recommend that dissolution be facilitated with 3 to 5 minutes 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, 97.5 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, 3450 at 472 nm.

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

Ms. Donna Sirk
NIST
Bldg. 222, Rm. B156
Gaithersburg, MD 20899
FAX: (301) 926-8671

If you have questions regarding this round robin exercise, please call me at (301) 975-3120 or mail/FAX queries to the above address. The results from Round Robin XXX will be forwarded to you within a week.

For those of you who requested them, samples of the soy oil containing isomers of β-carotene and Candidate SRM 2383: Fat-Soluble Vitamins and Carotenoids in Food are included with your Round Robin XXXI shipment. The food should be refrigerated upon receipt. The soy oil can be stored at room temperature.

Analyze the food using the sample preparation and HPLC methods that you would normally use for such a material. If possible, please analyze one sample from each bottle. Report results (in μg/g) on the attached sheet, and return them with your serum Round Robin results. As in the serum Round Robin, if you do not measure something, please leave that space on

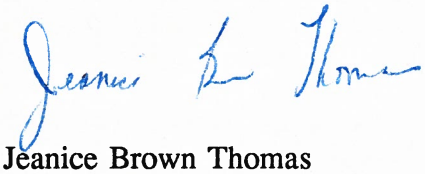
your report form blank; if you are capable of measuring something, but do not see that compound in your sample, please enter "ND."

The isomers-in-soy-oil sample is being provided to see how well your HPLC system separates the isomers of β-carotene. Tetrahydrofuran (THF) was added to the sample to dissolve the β-carotene crystals before it was pipetted into the sample vials. If crystals have precipitated during shipping, it may be necessary to add a few more drops of THF. This material is very concentrated, and you need to dilute the sample in a solvent appropriate for injection into your HPLC system. Because you will be reporting peak areas (thereby making the final concentrations unimportant), you can judge by eye how much solvent to add as you dilute the sample; use enough solvent so that the peaks remain on scale, but not so much that the peaks are below your limit of detection.

On the attached sheet, please report PEAK AREA for the peaks in this material that you are able to identify. Also report the total peak area for the peaks you would normally classify as "Total β-carotene;" if you normally report only total β-carotene when you participate in the serum Round Robins and you think you do not separate any β-carotene isomers, then report only the area of that peak that you would call your total β-carotene. Also provide the area under *all* peaks. From your results, we will determine the percentages of various cis isomers that your method is capable of separating. Please also send us a chromatogram of this sample with identified peaks labeled.

If you have any questions regarding the food or the soy oil round robin exercises, please contact Katherine Sharpless at (301)975-3121 or mail/FAX questions to the above address.

Sincerely,



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

Attachments

cc: W. May

bc: K. Sharpless
S. Wise

ROUND ROBIN XXXI RESULTS FROM LABORATORY # _____
DATE OF ANALYSIS _____
RESULTS IN ug/mL

SAMPLE NUMBER	ANALYTE	RESULT
SERUM 195	RETINOL	_____
	ALPHA-TOCOPHEROL	_____
VIAL # ___	BETA-CAROTENE	_____
SERUM 196	RETINOL	_____
	ALPHA-TOCOPHEROL	_____
VIAL # ___	BETA-CAROTENE	_____
SERUM 197	RETINOL	_____
	ALPHA-TOCOPHEROL	_____
VIAL # ___	BETA-CAROTENE	_____
SERUM 198	RETINOL	_____
	ALPHA-TOCOPHEROL	_____
VIAL # ___	BETA-CAROTENE	_____
SERUM	RETINOL	_____
	ALPHA-TOCOPHEROL	_____
VIAL # ___	BETA-CAROTENE	_____

OPTIONAL ANALYTES: SUPPLY ONE RESULT IF AVAILABLE

SERUM #	195	196	197	198	
TRANS-BETA CAROTENE					
ALPHA-CAROTENE					
RETINYL PALMITATE					
GAMMA-TOCOPHEROL					
LYCOPENE (TOTAL)					
9-CIS-BETA CAROTENE					
13-CIS-BETA-CAROTENE					
LUTEIN					
ZEAXANTHIN					
BETA-CRYPTOXANTHIN					

DIRECTIONS:

RESULTS DUE BY: June 14, 1994
FAX RESULTS TO 301/926-8671

Candidate SRM 2383: Fat-Soluble Vitamins and Carotenoids in Food - Round Robin 1							
			CONCENTRATION (ug/g)				
			Trial 1	Trial 2		Mean	
Vitamin A							
	retinol						
	retinyl palmitate						
Vitamin E							
	d-tocopherol						
	g-tocopherol						
	a-tocopherol						
Carotenoids							
	lutein						
	zeaxanthin						
	B-cryptoxanthin						
	lycopene						
		total					
		all-trans					
	a-carotene						
		total					
		all-trans					
	B-carotene						
		total					
		all-trans					
B-Carotene Isomers in Soy Oil - Round Robin 1							
					PEAK AREA		
	trans-B-carotene						
	9-cis-B-carotene						
	13-cis-B-carotene						
	15-cis-B-carotene						
	total-B-carotene						
	total all peaks						
Other peaks you are able to identify (please list)							

Appendix F. Final Report for RR31

The following six pages are the final report for RR31 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

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

August 8, 1994

Dear Colleague:

Enclosed is the summary report of the results for Round Robin XXXI (Serum 195-198). In this round robin exercise, serum 195 is a 50:50 volumetric mix of serum 198 and stripped serum; serum 197 is a 25:75 volumetric mix. Serum 196 was previously distributed in Round Robin XXIX as serum 183.

Tables 1-4 provide a summary of data for all laboratories. As for Round Robin XXX, the overall laboratory performance for retinol and γ- and α-tocopherol for this round robin exercise is good. The average estimated coefficient of variation (eCV) is about 11% for retinol 10.5% for α-tocopherol, and about 11 % for γ-tocopherol. The overall laboratory performance for total β-carotene improved somewhat from the last round robin exercise. The interlaboratory variation for total β-carotene in Round Robin XXX was approximately 21%; for this round robin study, the variation is about 16%. The higher interlaboratory variation was perhaps due to the difficulty of making measurements at much lower levels (≤100 ng/mL vs ≥300 ng/mL).

The concentration of retinyl palmitate was enriched in all samples used in this exercise. The average eCV for retinyl palmitate was about 26%. We will continue to enrich the levels of retinyl palmitate in future round robin samples in an effort to better assess laboratory performance for this analyte.

Data for evaluating your laboratory's performance relative to the other participants' is provided in Table 5. Your laboratory's individual laboratory performance is provided in pages 1-8 of the attached report and includes a graphical summary of laboratory's past performance for Round Robins XXII-XXXI and a tabular summary of your interlaboratory accuracy and precision.

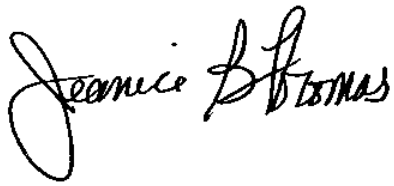
The performance criteria that was previously established for past round robins is used to summarize laboratory performance for Round Robin XXXI. By convention, 0-5% bias from the assigned value represents **EXCEPTIONAL** performance, 6-10% **ACCEPTABLE** performance, 11-20% **MARGINAL** performance and >20% **POOR** performance relative to the current state-of-the-practice for these measurements.

If you have concerns regarding your performance or are a lab whose performance would be rated "**POOR**" based on the convention stated above, we suggest that you obtain a unit of SRM 968a and analyze all three levels. If, with minor method modifications your measured values do not agree with the certified values, feel free to contact us for consultation. SRM 968a can be obtained through the NIST Standard Reference Materials Program (301/975-6776 or FAX: 301/948-3730)

Round Robin XXXII samples were shipped during the last week of July. If you have not received these samples, please call Ms. Donna Sirk at 301/975-3174. Results are due by August 30. The results from this study will be discussed at the Micronutrients QA Workshop, which will be held on Saturday, October 15, 1994 at the Crystal Gateway Marriot Hotel in Arlington, VA.

If you have questions, please contact me at 301/975-3120.

Sincerely,



Jeanice Brown Thomas
Research Chemist
Organic Analytical Research Division
Chemical Science and technology Laboratory

Enclosures

"Lies, Damned Lies, and Statistics"
Mark Twain

For better or worse, the attached Round Robin XXXI Report is not *much* different from XXX's... Actually, just a few cosmetic refinements and two IMPORTANT pages of plots specific to the RR XXXI samples.

Refinements: The "All Lab" report has been broken into five chunks:

(1) Retinoids and Tocopherols (retinol, retinyl palmitate, α- and γ-tocopherol).

(2) Carotenes (total and *trans*-β-carotene, total α-carotene, total and *trans*-lycopene).

(3) Xanthophylls (β-cryptoxanthin, lutein, zeaxanthin, lutein & zeaxanthin).
We're cheating on the "lutein & zeaxanthin" data... some of you report lutein and zeaxanthin separately, some report only the combined value, some report both. Since we've got a data column for the combined analytes, we've decided to fill it in as best we can by adding together all separately reported lutein and zeaxanthin pairs.

(4) Analytes reported by only single laboratories.

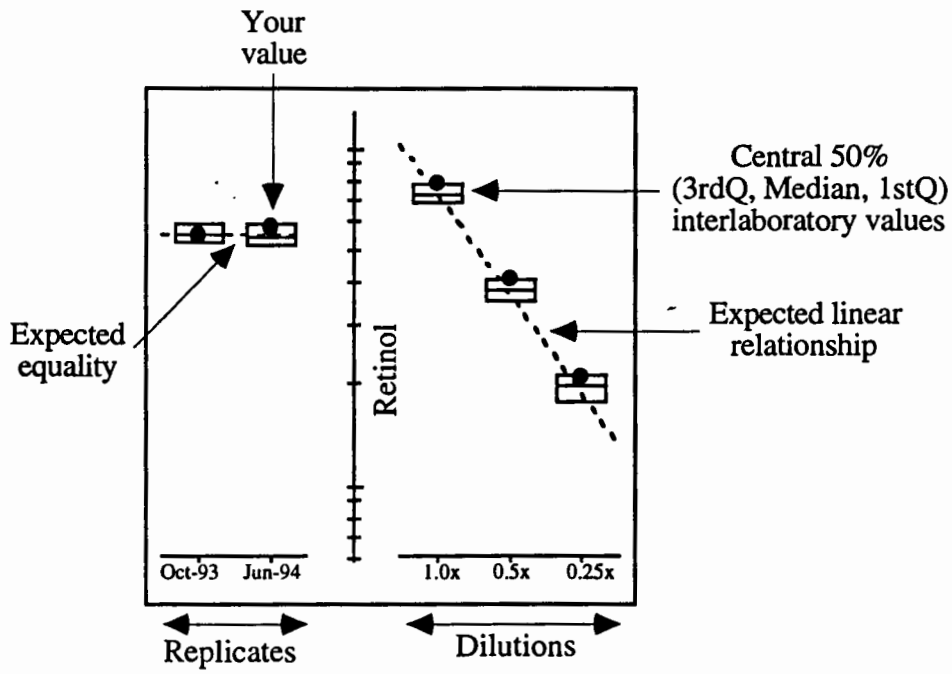
(5) Relative Rankings.
This time, we've scaled the Rankings from 1 (among the 25% of reporting labs closest to the interlaboratory medians) to 4 (among the 25% of reporting labs farthest from the medians). We've also changed how we average across sera (by ranks rather than pooled CV), but the results don't change noticeably at 1-4 resolution.

Same-as-before: The "Individualized" report's first six pages are identical to those of Round Robin XXX (except that RRs XXII through XXX are now the reference period for the graphs).

Pg. 1 Data table
Pg. 2 %Bias barchart for retinol
Pg. 3 %Bias barchart for α- and γ- tocopherol
Pg. 4 %Bias barchart for total and trans-β-carotene
Pg. 5 Interlaboratory median vs. your value scatterplots for retinol, α- and γ- tocopherol, total and *trans*-β-carotene
Pg. 6 (Provisional) Performance Summary

New & IMPORTANT

The "Individualized" report's pages 7 and 8 present information that may help you assess your absolute measurement performance. Serum 196 was a replicate of serum 183 (from RR XXIX). Serum 195 was a 50:50 volumetric mix of serum 198 and delipidized serum; serum 197 was a 25:75 volumetric mix. Your values, the central 50% of the interlaboratory results, and the expected relationships among the sera are displayed for all analytes for which at least five laboratories reported values. The displays are as follows:



How should you interpret these little graphs, and why should you care? If you analyze for one or more of the above analytes, the left-hand side of each graph lets you quickly spot how stable your assay has been over the last year. If the black dots aren't pretty near each other, your assay is not stable.

The right-hand side of each graph plots the dilution series and draws the expected linear relation. If the black dots aren't parallel to the line, your assay is not linear (or, if the lowest concentration is too high, your assay was pushed past its limit of quantification.) If the black dots aren't in the right order (high, middle, low), you need to examine the plots for all analytes that you report. Is the pattern the same for all analytes? Then you may have a problem in your extraction/sample prep/sample identification system. If it's just in one analyte, then you may need to critically examine your peak-recognition and quantification method.

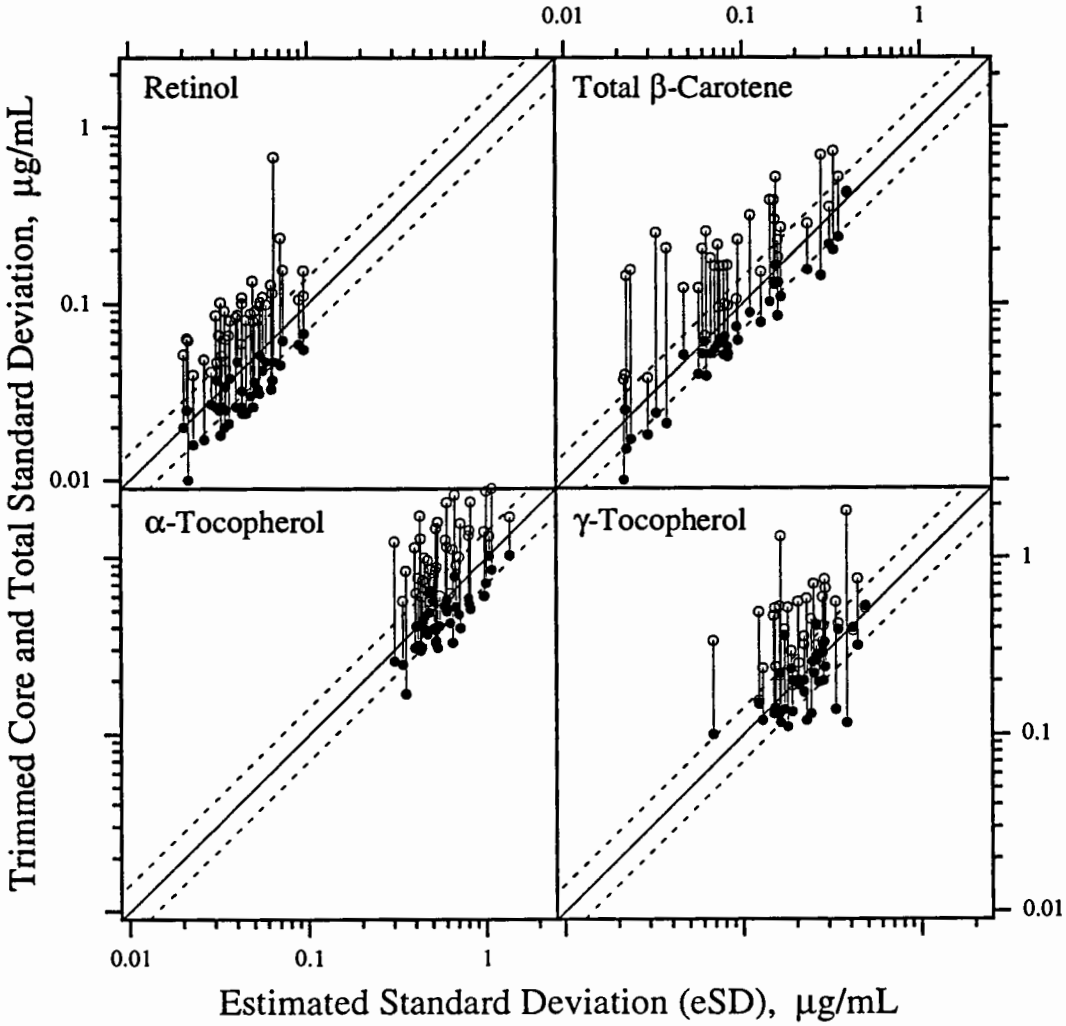
The central 50% boxes (showing the 3rd quartile, median, and 1st quartile values) give you another look at how comparable your values are with those of other laboratories. More importantly, the discord between the boxes for replicate sera and/or deviation from linearity in the dilution series highlight "analytical opportunities" with the analytes: retinyl palmitate, lutein, and total lycopene.

We promised to discuss "how to compare variation over analytes, concentration ranges, and time" in Round Robin XXX's report. That's too big a bite for one sitting; we'll start with

the relationships between the previously reported total standard deviation (SD) and "Trimmed Core" standard deviation (TC-SD) and our "new" dispersion statistic: the estimated Standard Deviation (eSD). The eSD is defined as 0.741 times the interquartile range (IQR) - our old friend, the central 50% of the data. The factor 0.741 is the number of "standard deviations" (z) required to span the central 50% of a normal distribution:

$$\frac{1}{z_{+25\%} - z_{-25\%}} \approx \frac{1}{0.674 + 0.674} = \frac{1}{1.349} = 0.741$$

Round Robin XXI - XXIX Inter-laboratory eSD versus Total and "Trimmed Core" SD for Retinol, Total β-Carotene, and α- and γ-Tocopherol



○ Total (all reported values) SD
● Trimmed Core SD
—— SD = eSD
---- SD = eSD/$2^{1/2}$, eSD*$2^{1/2}$

Unlike the location statistics (median vs. "Trimmed Core") compared in the Round Robin XXX report, eSD is not exactly equal to any previously reported dispersion estimate. There are many plausible dispersion estimates, perhaps the largest being the SD (open circles) and the smallest being TC-SD. This is a consequence of the "Trimmed Core" selection process, which insured that the estimated dispersion would be lower for the subset than for the whole.

The eSD is closely proportional to TC-SD for all four analytes for (nearly) all sera, although it generally is about a factor of $\sqrt{2}$ larger. Whether eSD is a tad large or the TC-SD values were a tad small really can't be resolved: they clearly show the same trends for retinol, α-tocopherol and total β-carotene. The relationship between eSD and TC-SD for γ-tocopherol appears somewhat different than for the other three: this actually reflects a differential "trimming" process used with this "semi-major" analyte and inclusion of the NIST laboratories into the reported SD and TC-SD. We could cheat and redo the trimming and calculations to be in better accord with the other three analytes, but choose rather to just use the eSD and go forward.

The Round Robin XXXII Report will discuss how to actually use the median and eSD statistics to monitor analytical variation.

Once again, 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
301-975-3935
DLDuewer@enh.NIST.gov

Margaret Kline
Research Biologist

Appendix G. "All-Lab Report" for RR31

The following eight pages are the "All-Lab Report" for RR31 as provided to all participants, with three exceptions:

- the participant identifiers (Lab) have been altered.
- the order in which the participant results are listed has been altered.
- the robust coefficient of variation has been recalculated using a more efficient robust estimator of the standard deviation: the adjusted median absolute deviation from the median (MADe) rather than the adjusted interquartile range (IQR).

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.

All Laboratory Report for Round Robin XXXI

Table 1: Retinoids and Tocopherols (micrograms/milliliter)

	Total Retinol				Retinyl Palmitate				α-Tocopherol				γ/β-Tocopherol			
Lab	195	196	197	198	195	196	197	198	195	196	197	198	195	196	197	198
FSV-BA	0.389	0.564	0.213	0.744	0.106	0.149	0.052	0.258	6.38	11.5	3.46	13.8	1.20	1.82	0.707	2.51
FSV-BD	0.383	0.552	0.200	0.751					6.90	11.0	3.70	12.6				
FSV-BE	0.355	0.522	0.184	0.702					7.26	11.2	3.68	13.3	1.46	2.00	0.780	2.70
FSV-BF	0.415	0.597	0.198	0.780					7.78	12.2	3.82	15.5	1.34	1.93	0.730	2.61
FSV-BH	0.273	0.494	0.142	0.611					8.08	12.1	4.09	15.6	1.07	1.38	0.550	2.09
FSV-BI	0.346	0.491	0.166	0.671	0.158	0.149	0.074	0.352	7.79	12.1	3.86	14.8	1.44	1.88	0.710	2.76
FSV-BJ	0.412	0.588	0.210	0.811	0.090	0.100	*nq*	0.190	7.50	10.8	3.91	13.1	1.56	1.88	0.892	2.62
FSV-BK	0.367	0.537	0.200	0.721					6.44	10.3	3.40	12.9				
FSV-BL	0.430	0.601	0.229	0.802					9.30	14.1	4.69	16.1				
FSV-BM	0.363	0.591	0.162	0.772					8.40	12.2	4.20	15.1				
FSV-BN				0.570				0.243				14.2				2.44
FSV-BO	0.352	0.581	0.197	0.713					6.09	11.0	3.58	12.7				
FSV-BP	0.410	0.590	0.210	0.800					7.53	11.4	4.24	15.2				
FSV-BQ	0.319	0.513	0.147	0.728					6.12	10.4	1.95	13.5				
FSV-BR	0.401	0.622	0.195	0.789												
FSV-BS																
FSV-BT	0.365	0.544	0.261	0.691	0.133	0.151	0.058	0.321	6.91	10.8	3.41	13.8	1.30	1.70	0.608	2.57
FSV-BY	0.348	0.517	0.176	0.608	0.111	0.082	0.037	0.265	7.30	10.8	3.37	13.8	1.39	1.69	0.684	2.68
FSV-BZ									6.97	11.5	3.23	13.3	1.56	1.75	0.462	2.28
FSV-CA	0.417	0.614	0.250	0.757					7.82	12.4	4.12	18.7				
FSV-CB	0.420	0.650	0.200	0.810					8.41	10.8	4.37	14.9				
FSV-CG	0.116	0.168	0.063	0.231					1.40	2.4	0.68	3.0				
FSV-CH	0.243	0.502	0.156	0.598					5.68	11.9	2.95	13.1	0.80	1.57	0.495	1.90
FSV-CJ	0.390	0.525	0.226	0.745					8.07	11.2	3.76	14.9				
FSV-CK	0.318	0.493	0.186	0.682					6.59	11.0	4.26	13.6	1.35	1.80	0.910	2.60
FSV-CO	0.375	0.545	0.192	0.736					6.36	11.6	2.68	13.7				
FSV-CP									6.45	11.0	4.72	14.2				
FSV-CQ	0.310	0.450	0.170	0.630					7.55	13.4	3.79	15.6				
FSV-CR	0.390	0.570	0.200	0.770					7.20	11.3	3.60	14.5				
FSV-CU	0.395	0.539	0.201	0.685	0.086	0.118	0.074	0.172	6.63	10.9	3.08	12.3				
FSV-CV	0.370	0.560	0.195	0.723					7.20	11.0	3.38	14.4				
FSV-CX	0.390	0.600	0.210	0.730	0.030	0.060	0.050	0.330	7.61	11.7	4.10	14.4	1.48	2.05	0.780	2.81

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All Laboratory Report for Round Robin XXXI

Table 1: Retinoids and Tocopherols (micrograms/milliliter)

	Total Retinol				Retinyl Palmitate				α-Tocopherol				γ/β-Tocopherol			
Lab	195	196	197	198	195	196	197	198	195	196	197	198	195	196	197	198
FSV-CY	0.383	0.533	0.195	0.694					6.46	11.5	2.75	14.6				
FSV-DJ	0.450	0.710	0.260	0.840					8.30	13.5	4.10	16.1				
FSV-DK	0.410	0.620	0.190	0.810					6.90	10.3	4.40	13.0				
FSV-DL	0.366	0.548	0.194	0.710					6.83	11.8	3.51	14.3	1.24	1.76	0.660	2.66
FSV-DS	0.380	0.640	0.240	0.800					7.00	11.9	4.32	14.3				
FSV-DX	0.405	0.570	0.226	0.721					7.18	12.3	3.63	14.1				
FSV-EA	0.311	0.485	0.157	0.637					6.80	11.7	3.40	14.1	1.08	1.50	0.520	2.65
FSV-EC	0.351	0.545	0.170	0.725					7.53	11.8	3.56	15.3				
FSV-EK	0.413	0.552	0.194	0.775					5.74	13.3	2.21	13.9	1.54	1.82	0.820	2.81
FSV-EX	0.360	0.460	0.190	0.660					7.69	11.2	3.54	14.8				
n	38	38	38	39	7	7	6	8	39	39	39	40	15	15	15	16
Min	0.116	0.168	0.063	0.231	0.030	0.060	0.037	0.172	1.40	2.4	0.68	3.0	0.80	1.38	0.462	1.90
Mean	0.366	0.547	0.194	0.711	0.102	0.116	0.058	0.266	7.03	11.4	3.58	14.0	1.32	1.77	0.687	2.54
Max	0.450	0.710	0.261	0.840	0.158	0.151	0.074	0.352	9.30	14.1	4.72	18.7	1.56	2.05	0.910	2.81
SD	0.060	0.083	0.036	0.103	0.040	0.036	0.015	0.065	1.21	1.7	0.77	2.1	0.21	0.18	0.139	0.25
CV	16	15	18	14	40	32	25	24	17	15	22	15	16	10	20	10
NISTa	0.380	0.558	0.192	0.710					6.55	10.9	3.36	13.5	1.25	1.75	0.723	2.42
NISTb	0.369	0.534	0.194	0.702	nq	0.130	0.036	0.208	7.09	11.1	3.17	13.6	1.33	1.74	0.629	2.51
NISTc	0.411	0.582	0.206	0.784	0.085	0.141	0.037	0.251	8.04	12.6	3.42	15.7	1.47	1.96	0.697	2.87
Median	0.378	0.550	0.195	0.725	0.106	0.118	0.055	0.262	7.18	11.5	3.63	14.2	1.35	1.80	0.707	2.62
eCV	11	11	11	10	28	39	31	36	12	7	16	7	14	8	21	5

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All Laboratory Report for Round Robin XXXI

Table 2: Carotenes and Lycopene (micrograms/milliliter)

	Total β-Carotene				trans-β-Carotene				Total α-Carotene				Total Lycopene				trans-Lycopene			
Lab	195	196	197	198	195	196	197	198	195	196	197	198	195	196	197	198	195	196	197	198
FSV-BA	0.347	0.398	0.173	0.680	0.312	0.366	0.168	0.628	0.014	0.046	0.010	0.037					0.041	0.151	0.015	0.070
FSV-BD																				
FSV-BE	0.366	0.390	0.144	0.749					0.007	0.027	0.004	0.015								
FSV-BF	0.536	0.289	0.249	0.784					0.043	0.055	0.010	0.072	0.077	0.307	0.015	0.211				
FSV-BH	0.340	0.352	0.160	0.678	0.312	0.325	0.146	0.626	0.016	0.040	*nd*	0.032	0.053	0.194	0.023	0.111				
FSV-BI	0.366	0.391	0.155	0.722					0.018	0.047	0.009	0.030	0.060	0.231	0.026	0.121				
FSV-BJ	0.329	0.336	0.145	0.684					0.024	0.049	0.020	0.046	0.072	0.320	0.034	0.018				
FSV-BK																				
FSV-BL																				
FSV-BM																				
FSV-BN				0.301								0.042				0.102				
FSV-BO	0.355	0.352	0.178	0.745					0.015	0.021	0.014	0.047	0.073	0.187	0.023	0.120				
FSV-BP	0.293	0.289	0.146	0.619					0.022	0.036	0.015	0.030	0.129	0.294	0.074	0.200				
FSV-BQ	0.330	0.360	0.130	0.630																
FSV-BR																				
FSV-BS					0.353	0.260	0.160	0.511												
FSV-BT	0.358	0.317	0.150	0.682	0.332	0.297	0.138	0.631	0.016	0.038	0.005	0.028	0.076	0.194	0.028	0.134				
FSV-BY	0.355	0.260	0.137	0.633					0.020	0.030	0.012	0.039	0.062	0.216	0.014	0.124				
FSV-BZ	0.380	0.400	0.194	0.588					0.034	0.040	0.032	0.040	0.030	0.020	*nq*	0.010				
FSV-CA																				
FSV-CB	0.276	0.301	0.087	0.626					0.015	0.022	0.004	0.018	0.030	0.132	0.011	0.073				
FSV-CG	0.366	0.384	0.163	0.747																
FSV-CH	0.218	0.333	0.119	0.594					0.009	0.027	0.010	0.013	0.031	0.214	0.014	0.077				
FSV-CJ									0.020	0.036	0.002	0.032	0.112	0.312	0.045	0.217				
FSV-CK	0.335	0.309	0.089	0.649					0.021	0.046	0.006	0.050	0.071	0.129	0.017	0.189				
FSV-CO	0.356	0.385	0.173	0.734	0.342	0.330	0.135	0.623	0.042	0.054	0.027	0.063	0.056	0.191	0.028	0.106				
FSV-CP	0.296	0.327	0.139	0.638					0.016	0.043	0.005	0.039	0.003	0.229	0.027	0.004				
FSV-CQ	0.242	0.277	0.073	0.516																
FSV-CR																				
FSV-CU	0.568	0.579	0.287	0.973	0.508	0.521	0.270	0.922												
FSV-CV	0.335	0.395	0.153	0.667									0.103	0.410	0.020	0.228				
FSV-CX	0.320	0.190	0.180	0.530					0.010	0.020	<0.01	0.020	0.070	0.190	0.040	0.140				

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All Laboratory Report for Round Robin XXXI

Table 2: Carotenes and Lycopene (micrograms/milliliter)

	Total β-Carotene				trans-β-Carotene				Total α-Carotene				Total Lycopene				trans-Lycopene			
Lab	195	196	197	198	195	196	197	198	195	196	197	198	195	196	197	198	195	196	197	198
FSV-CY	0.233	0.233	0.114	0.817																
FSV-DJ																				
FSV-DK																				
FSV-DL	0.281	0.320	0.124	0.671					0.019	0.043	0.010	0.045	0.044	0.148	0.018	0.104				
FSV-DS	0.393	0.452	0.250	0.753																
FSV-DX	0.240	0.345	0.092	0.712	0.228	0.335	0.092	0.635	0.017	0.067	*nd*	0.030	0.061	0.306	0.024	0.178				
FSV-EA	0.330	0.345	0.150	0.596					0.016	0.053	0.009	0.037	0.052	0.193	0.020	0.117				
FSV-EC	0.346	0.376	0.160	0.680																
FSV-EK	0.287	0.347	0.189	0.625										0.235		0.111				
FSV-EX	0.340	0.350	0.150	0.680																
n	30	30	30	31	7	7	7	7	21	21	18	22	20	21	19	22	1	1	1	1
Min	0.218	0.190	0.073	0.301	0.228	0.260	0.092	0.511	0.007	0.020	0.002	0.013	0.003	0.020	0.011	0.004				
Mean	0.337	0.346	0.155	0.668	0.341	0.348	0.158	0.654	0.020	0.040	0.011	0.037	0.063	0.222	0.026	0.122				
Max	0.568	0.579	0.287	0.973	0.508	0.521	0.270	0.922	0.043	0.067	0.032	0.072	0.129	0.410	0.074	0.228				
SD	0.074	0.070	0.047	0.111	0.084	0.083	0.055	0.126	0.009	0.012	0.008	0.014	0.029	0.084	0.015	0.064				
CV	22	20	31	17	25	24	35	19	48	31	71	39	47	38	55	52				
NISTa	0.361	0.364	0.164	0.690	0.295	0.307	0.131	0.661												
NISTb	0.333	0.348	0.118	0.659	0.278	0.318	0.103	0.592					0.106	0.291	0.050	0.192	0.035	0.119	0.028	0.064
NISTc	0.346	0.392	0.116	0.732	0.304	0.357	0.095	0.655	0.022	0.050	0.009	0.052	0.110	0.329	0.021	0.235	0.037	0.152	0.012	0.082
Median	0.338	0.346	0.150	0.678	0.332	0.330	0.146	0.628	0.017	0.040	0.010	0.037	0.062	0.214	0.023	0.118				
eCV	13	16	23	12	9	15	14	1	26	33	66	30	31	19	32	39				

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All Laboratory Report for Round Robin XXXI

Table 3: Xanthophylls (micrograms/milliliter)

	Total β-Cryptoxanthin				Total Lutein				Total Zeaxanthin				Total Lutein&Zeaxanthin			
Lab	195	196	197	198	195	196	197	198	195	196	197	198	195	196	197	198
FSV-BA	0.013	0.059	0.008	0.029									0.050	0.085	0.038	0.083
FSV-BD																
FSV-BE																
FSV-BF																
FSV-BH	*nq*	0.056	*nq*	0.025	*nq*	0.047	*nq*	0.063	*nq*	*nq*	*nq*	*nq*				
FSV-BI	0.014	0.057	0.007	0.023	0.024	0.045	0.013	0.045	0.009	0.020	0.005	0.015	0.033	0.065	0.018	0.065
FSV-BJ																
FSV-BK																
FSV-BL																
FSV-BM																
FSV-BN				0.015												0.040
FSV-BO	0.008	0.040	*nq*	0.014									0.037	0.068	0.018	0.069
FSV-BP	0.004	0.040	0.002	0.017												
FSV-BQ																
FSV-BR																
FSV-BS																
FSV-BT	0.014	0.050	0.008	0.030	0.027	0.052	0.020	0.055	0.013	0.024	0.009	0.019	0.064	0.123	0.048	0.123
FSV-BY	0.020	0.045	0.009	0.031	0.020	0.036	0.010	0.041	0.010	0.014	0.006	0.017	0.030	0.050	0.016	0.058
FSV-BZ					0.020	0.070	0.010	0.070								
FSV-CA																
FSV-CB	0.006	0.025	0.003	0.011									0.050	0.080	0.029	0.085
FSV-CG																
FSV-CH																
FSV-CJ	0.012	0.050	0.004	0.029									0.047	0.078	0.025	0.092
FSV-CK	0.013	0.051	0.005	0.025									0.042	0.079	0.028	0.081
FSV-CO	0.034	0.083	0.013	0.036	0.182	0.073	0.028	0.097	0.072	0.039	0.017	0.042	*0.254*	*0.112*	*0.045*	*0.139*
FSV-CP	0.061	0.042	0.061	0.020									0.052	0.066	0.095	0.068
FSV-CQ																
FSV-CR																
FSV-CU																
FSV-CV																
FSV-CX	0.010	0.030	0.010	0.020	0.020	0.080	0.010	0.050								

All Laboratory Report for Round Robin XXXI

Table 3: Xanthophylls (micrograms/milliliter)

	Total β-Cryptoxanthin				Total Lutein				Total Zeaxanthin				Total Lutein&Zeaxanthin			
Lab	195	196	197	198	195	196	197	198	195	196	197	198	195	196	197	198
FSV-CY																
FSV-DJ																
FSV-DK																
FSV-DL	0.020	0.099	0.006	0.312									0.021	0.076	0.010	0.086
FSV-DS																
FSV-DX					0.024	0.033	0.012	0.048	0.023	0.029	0.011	0.032	*0.047*	*0.062*	*0.023*	*0.080*
FSV-EA	0.014	0.041	0.008	0.024												
FSV-EC																
FSV-EK		0.112			0.020	0.072		0.080								
FSV-EX																
n	14	16	13	16	8	9	7	9	5	5	5	5	12	12	12	13
Min	0.004	0.025	0.002	0.011	0.020	0.033	0.010	0.041	0.009	0.014	0.005	0.015	0.021	0.050	0.010	0.040
Mean	0.017	0.055	0.011	0.041	0.042	0.056	0.015	0.061	0.025	0.025	0.010	0.025	0.061	0.079	0.033	0.082
Max	0.061	0.112	0.061	0.312	0.182	0.080	0.028	0.097	0.072	0.039	0.017	0.042	0.254	0.123	0.095	0.139
SD	0.015	0.024	0.015	0.073	0.057	0.018	0.007	0.018	0.027	0.009	0.005	0.012	0.062	0.021	0.023	0.026
CV	84	43	138	176	134	31	47	30	105	38	50	46	102	26	70	31
NISTa																
NISTb	0.010	0.035	0.005	0.014	0.024	0.037	0.014	0.046	0.016	0.021	0.005	0.022	*0.040*	*0.058*	*0.019*	*0.068*
NISTc	0.009	0.041	0.004	0.018	0.026	0.045	0.012	0.051	0.007	0.020	0.004	0.013	0.033	0.065	0.016	0.064
Median	0.014	0.050	0.008	0.025	0.022	0.052	0.012	0.055	0.013	0.024	0.009	0.019	0.047	0.077	0.027	0.081
eCV	52	27	37	31	13	51	25	27	46	31	49	31	24	20	53	21

All Laboratory Report for Round Robin XXXI

Table 4: Analytes Reported By Only One Laboratory

Analytes Reported By One Laboratory

Values in µg/mL

Analyte	Code	195	196	197	198
Total cis-β-Carotene	FSV-BT	0.027	0.020	0.012	0.051
13-cis-β-Carotene	FSV-DX	0.012	0.010	*nq*	0.076
Total Carotenoids	FSV-BT	0.566	0.804	0.261	1.063
trans-α-Carotene	NISTb	0.013	0.040	0.004	0.030
δ-Tocopherol	NISTb	0.095	0.107	0.037	0.181

Legend for Tables 1, 2, and 3

nd	Not detected or reported as '0'
nq	Not quantitatively determined (i.e., reported as 'trace', 'not determined', etc.)
<x	Concentration at or below "limit of quantification" x.

Statistics (NIST's values are excluded from all calculations)

n	Number of laboratories reporting quantitative values for this analyte for this serum.
Min	Minimum reported value.
Mean	Average over all reported values.
Max	Maximum reported value.
SD	Standard deviation over all values.
CV	Coefficient of Variation (% relative standard deviation): for (non-NIST) results: 100*SD/Mean
Median	Median over all reported values (i.e., 50% of values are larger, 50% are smaller.)
eCV	Estimated CV, based on the adjusted median absolute deviation from the median (MADe). The original report estimated eCV from the interquartile range (IQR).

All Laboratory Report for Round Robin XXXI

Table 5: Relative Rankings

Lab	Ret	aToc	bCar	OA	gToc
FSV-BA	1	1	2	1	1
FSV-BD	1	2			
FSV-BE	2	1	2	1	3
FSV-BF	2	3	4	4	1
FSV-BH	4	4	1	3	4
FSV-BI	3	2	2	3	2
FSV-BJ	3	3	1	2	2
FSV-BK	1	3			
FSV-BL	4	4			
FSV-BM	3	4			
FSV-BN	4	1	4	4	3
FSV-BO	1	3	2	2	
FSV-BP	3	2	3	3	
FSV-BQ	3	4	1	3	
FSV-BR	2				
FSV-BS					
FSV-BT	2	2	1	1	2
FSV-BY	3	2	3	3	2
FSV-BZ		1	4		4
FSV-CA	4	4			
FSV-CB	4	4	3	4	
FSV-CG	4	4	3	4	
FSV-CH	4	4	3	4	4
FSV-CJ	2	2			
FSV-CK	3	3	2	2	1
FSV-CO	1	3	3	2	
FSV-CP		3	2		
FSV-CQ	4	3	4	4	
FSV-CR	1	1			
FSV-CU	2	3	4	4	
FSV-CV	1	1	1	1	
FSV-CX	2	1	4	2	3
FSV-CY	1	2	4	2	
FSV-DJ	4	4			
FSV-DK	3	3			
FSV-DL	1	1	2	1	1
FSV-DS	3	2	4	3	
FSV-DX	2	1	3	2	
FSV-EA	4	1	1	1	4
FSV-EC	1	2	1	1	
FSV-EK	2	4	2	3	3
FSV-EX	2	1	1	1	

Legend

- **Lab** Laboratory code
- **Ret** Relative Rank for: Retinol
- **aToc** Relative Rank for: a-Tocopherol
- **bCar** Relative Rank for: Total b-Carotene
- **OA** Overall Relative rank for labs that report Ret, aToc, and bCar.
- **gToc** Relative Rank for: g/b-Tocopherol

Relative Rank

An approximate measure of "closeness" to the interlaboratory medians for a given analyte in Round Robin XXXI. It is calculated as the PERCENTRANK of ABS(value-median)/median over all reported laboratory values for a given serum, averaged over all sera. The resultant average is itself ranked and scored as:

1: among the 25% of reporting labs closest to the median
2: not in the closest 25%, but in the closest 50%
3: not in the closest 50%, but in the closest 75%
4: among the 25% of reporting labs farthest from the median

The "Overall" Relative Rank is calculated from the sum of the Relative Ranks of Retinol, a-Tocopherol, and total b-Carotene. The sum itself is ranked and scored as above.

["PERCENTRANK" is an excel spreadsheet function. It returns the percentage standing of a given value among a set of values.]

Note: "Relative Rank" does NOT measure "correctness, "goodness," etc. It reflects only a laboratory's relative agreement among its peers. Ideally, all analyses from all participating laboratories should agree within acceptable limits: the absolute variance evaluation for all labs could then be "Excellent" ... but the Relative Ranks would still go from "Closest" to "Farthest."

Appendix H. Representative "Individualized Report" for RR31

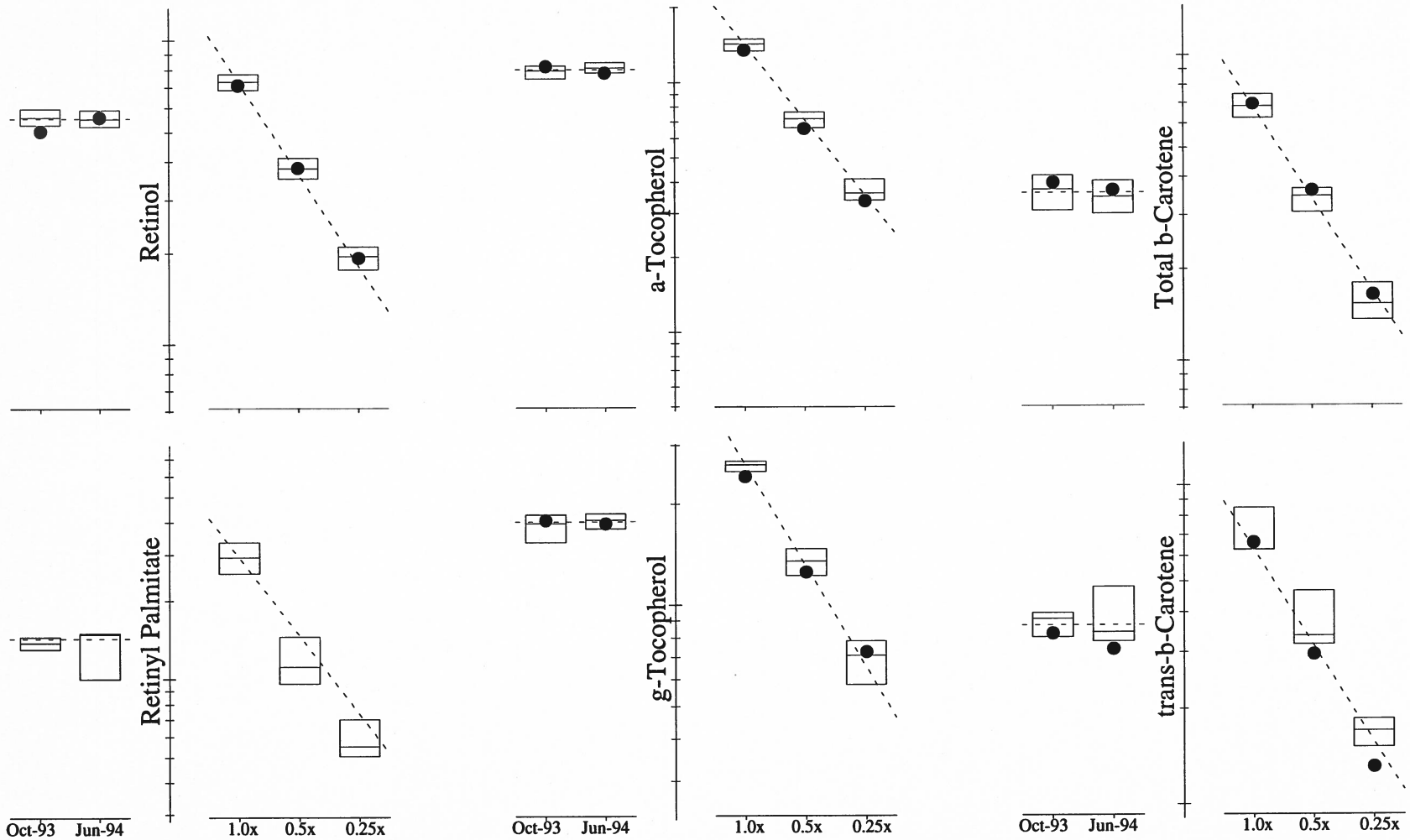
Each participant in RR31 received an "Individualized Report" reflecting their reported results. Each report included a detailed analysis of the results they reported for the following analytes:

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

The following two pages are the "Individualized Report" for the analytes evaluated by participant FSV-NIST1 (listed as FSV-NISTa in the "All Lab Report").

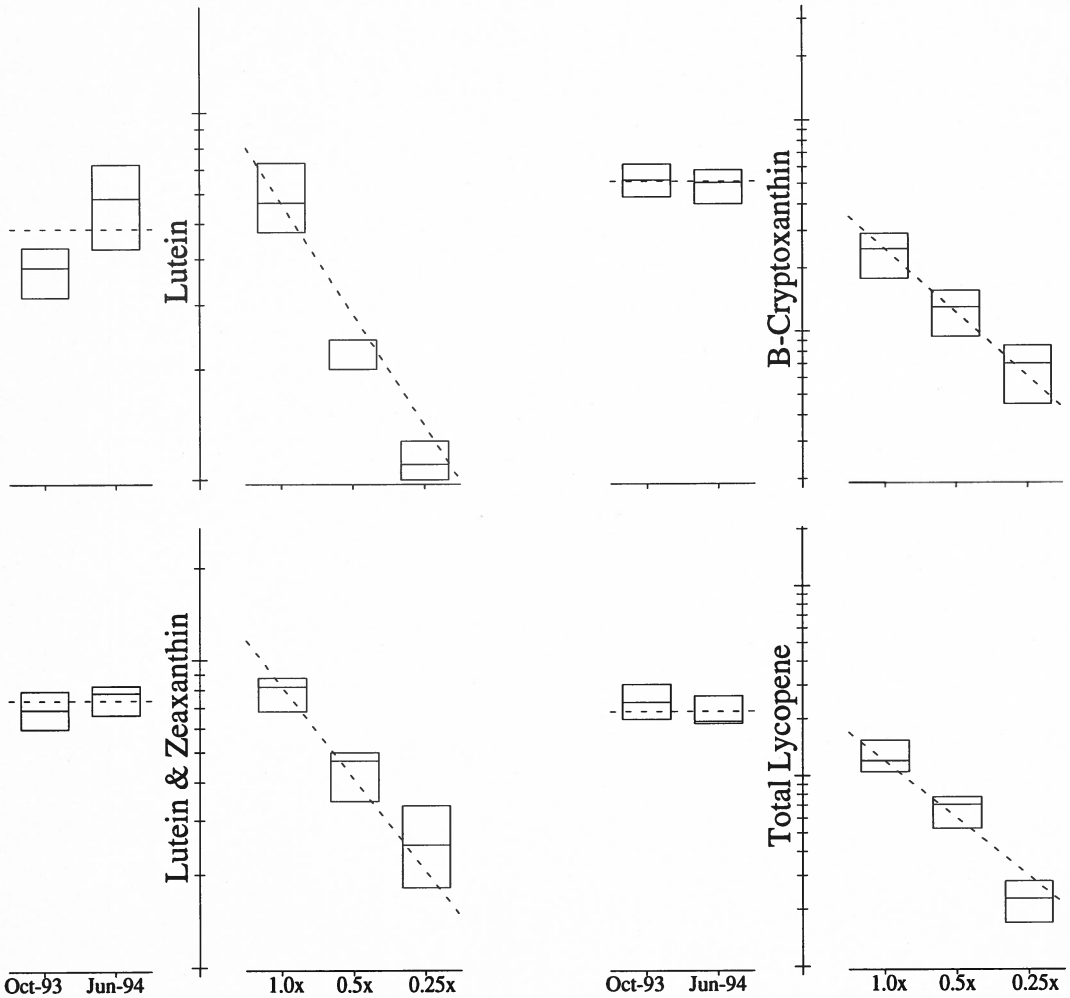
Individualized Round Robin XXXI Report to: NIST1

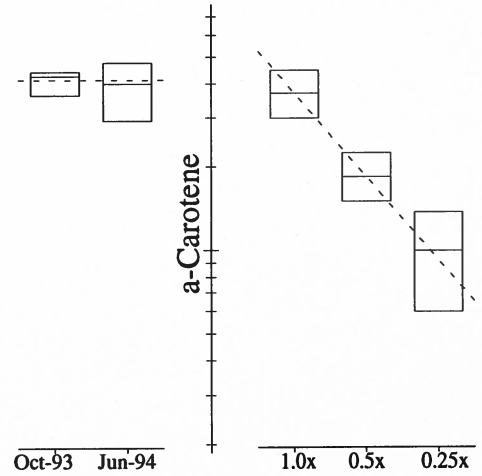
Comparisons to Known Values



Individualized Round Robin XXXI Report to: NIST1

Comparisons to Known Values





Legend

Solid circles are your lab's values.

Boxes bound central 50% of interlaboratory values, with center-bar denoting the median.

Lefthand values are duplicate series: sera 183 & 196.
Righthand values are dilution series: 198, 195, & 197.

Dotted lines are the known relationships…
Left: Average of serum 183 and 196.
Right: 1x, 0.5x, & 0.25x median value of serum 198.

H3

Appendix I. Shipping Package Inserts for RR32

The following two items were included in each package shipped to an RR32 participant:

- Cover letter
- Datasheet

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

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

July 18, 1994

Dear Colleague:

Enclosed is the set of samples for Round Robin XXXII. You will find duplicate vials of four lyophilized sera 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 the detection limit, please indicate this result on the form by using ND *(Not Detected)*. For values not obtained, please leave a blank for the given analyte. Results are due to NIST by August 30, 1994. The results from this study will be discussed at the Micronutrients QA Workshop, which will be held on Saturday, October 15, 1994 at the Crystal Gateway Marriott Hotel in Arlington, VA. (The details of the workshop were mailed to you last month).

Samples should be reconstituted with 1.0 mL of HPLC-grade water or equivalent. We recommend that dissolution be facilitated with 3 to 5 minutes 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, 97.5 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, 3450 at 472 nm.

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

Ms. Donna Sirk
NIST
Bldg. 222, Rm. B156
Gaithersburg, MD 20899
FAX: (301) 926-8671

If you have questions regarding this round robin exercise; or have not received the information regarding the Micronutrient QA Workshop, please call me at (301) 975-3120 or mail/FAX queries to the above address. The results from Round Robin XXXI will be forwarded to you within two weeks. The results from the studies for β-carotene in soy oil and the fat-soluble vitamins in food will be forwarded during that time, as well.

Sincerely,

Jeanice Brown Thomas

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

Attachments

ROUND ROBIN XXXII RESULTS FROM LABORATORY # _____
DATE OF ANALYSIS _____
RESULTS IN ug/mL

SAMPLE NUMBER	ANALYTE	RESULT
SERUM 199	RETINOL	_____
	ALPHA-TOCOPHEROL	_____
VIAL # ___	BETA-CAROTENE	_____
SERUM 200	RETINOL	_____
	ALPHA-TOCOPHEROL	_____
VIAL # ___	BETA-CAROTENE	_____
SERUM 201	RETINOL	_____
	ALPHA-TOCOPHEROL	_____
VIAL # ___	BETA-CAROTENE	_____
SERUM 202	RETINOL	_____
	ALPHA-TOCOPHEROL	_____
VIAL # ___	BETA-CAROTENE	_____

OPTIONAL ANALYTES: SUPPLY ONE RESULT IF AVAILABLE

SERUM #	199	200	201	202	
TRANS-BETA CAROTENE					
ALPHA-CAROTENE					
RETINYL PALMITATE					
GAMMA-TOCOPHEROL					
LYCOPENE (TOTAL)					
9-CIS-BETA CAROTENE					
13-CIS-BETA-CAROTENE					
LUTEIN					
ZEAXANTHIN					
BETA-CRYPTOXANTHIN					

DIRECTIONS:

RESULTS DUE BY: August 30, 1994
FAX RESULTS TO 301/926-8671

Appendix J. Final Report for RR32

A preliminary report for RR32 was sent to all participants shortly before the QA Workshop held on October 15, 1994. Based upon feedback from the Workshop participants, the format used in the "All Lab Report" for RR32 was significantly revised before the Final Report was distributed.

The following 15 pages include:

- The cover letter sent with the preliminary report. Since the preliminary report itself did not reflect all RR32 results, it is not reproduced.
- A reconstruction of the Cover letter sent with the Final Report. The original text of this letter has been lost; this version is based on an extant year-end summary report, statements in the "Lies, Damn Lies, and Statistic" document for RR32, and the report format used in RR33.
- 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 listing and graphical presentation of (Total) Retinol, α-Tocopherol, and Total β-Carotene summary statistics for all samples that were distributed more than once from RR01 through RR32. This material was presented at the QA Workshop and included (without further discussion) in the documentation packet sent to all participants. Figures 3 to 5 in this material were originally distributed in grayscale; the colored originals are reproduced here.

The "Lies, Damn Lies, and Statistics" states that the documentation packet for RR32 contained revised format "All Lab Reports" for RR30 and RR31. As the data in the reformatted reports is identical to that in the versions provided in Appendices C and G of this document, the revised "All Lab Reports" for RR30 and RR31 are not reproduced.

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

October 4, 1994

Dear Colleague:

Enclosed are the preliminary results for Round Robin XXXII (Sera 199-202). These results are being mailed to you to provide feedback in a timely manner and to afford those who will be attending the QA workshop the opportunity to preview their results. Individualized laboratory performance and NIST values for this round robin exercise will be included in the final report. The final results for Round Robin XXXII, as well as a summary of results from past QA round robin activities will be discussed at the QA workshop on October 15. Written feedback regarding the final results will be provided to all participants, thereafter.

If you have any questions, please contact me at (301)975-3120 or Fax (301)926-8671.

Sincerely,

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

cc: W. May

Enclosures

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

October 28, 1994

The original text of this letter has been lost. This version has been prepared from an extant year-end summary report.

Dear Colleague:

Enclosed is the summary report of the results for Round Robin XXXII (Serum 199-202). Serum 199 was previously distributed in Round Robin XXX. Sera 200-202 are from serum pools for a renewal material, Standard Reference Material 968b, Fat-Soluble Vitamins and Cholesterol in Human Serum, which will be available next year.

The average estimated coefficient of variation (eCV) for Round Robin XXXII is about 9 % for retinol, 7 % for α-tocopherol, 10% for γ-tocopherol, and about 14 % for total β-carotene. The average eCV for retinyl palmitate is approximately 27 %. This variation is due to the difficulty of making measurements at levels that are <100 parts-per-billion, as indicated in Serum 199 where the eCV is 63 % and 66 % in Round Robin XXX. We will continue to improve the measurement quality of retinyl palmitate and the major carotenoids as well.

Data for evaluating your laboratory's performance in Round Robin XXXII are provided on page 5 of the attached Round Robin XXXII report. The criteria used to summarize laboratory performance is as follows: results rated **1** (within ± 1 SD of the median value) indicate **EXCEPTIONAL** performance, those rated **2** (within ± 2 SD) indicate **ACCEPTABLE** performance, a rating of **3** (within ± 3 SD) is **MARGINAL** performance and **4** (>3 SD of the median) indicates **POOR** performance relative to the current state-of-the-practice.

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

As last year, the fees for non-NCI funded laboratories will be $300 for US labs and $600 for non-US labs. An invoice to that effect will be mailed to those laboratories.

The 1995 program will consist of three round robin exercises for the analysis of fat-soluble vitamins and carotenoids in serum, three studies for the analysis of ascorbic acid in serum, and two exercises for the analysis of fat-soluble vitamins and carotenoids in food. The first set of samples for the fat-soluble vitamins in serum analysis will be distributed the week of January 23. Results are due march 20; written feedback will be provided to labs by April 24. The second set of samples will be shipped the week of April 24 with results due by June 16 and feedback to labs by July 24. The third set of samples will be shipped the week of July 24 with results due by September 15 and feedback by October 20.

The round robin studies for the analysis of fat-soluble vitamins and carotenoids in food will also be scheduled in April and July to coincide with the second and third fat-soluble vitamins in

serum round robin studies. The coordinator of these exercises is Dr. Katherine Sharpless (301-975-3121).

The first set of samples for the analysis of ascorbic acid in serum will be distributed in January. The second and third sets will be distributed in April and July, respectively. These round robin studies are being coordinated by Dr. Sam Margolis (301-975-3137).

As a result of our efforts to provide consultation services in methods development and refinement a methods manual, Methods for Analysis of Cancer Chemopreventive Agents in Human Serum, has been completed and will be available upon request in the coming year. Also, at the past QA workshop it was suggested that a certificate of participation for the QA program be distributed to the laboratories. We are currently working on this and will distribute certificates in December or early January. Another topic of discussion at the workshop was the need to broaden the scope of the program to include a larger segment of the clinical community. In response, we will explore the possibility of holding future QA workshops at national meetings to include a broader audience.

If you have questions, please contact me at 301-975-3120.

Sincerely,

Jeanice Brown Thomas
Research Chemist
Organic Analytical Research Division
Chemical Science and technology Laboratory

Enclosures

"Lies, Damned Lies, and Statistics"
Mark Twain

The attached Round Robin XXXII Report is very similar to RR XXXI's, with some format simplification in the "All Lab" data summary and a major change in (what was) the "Relative Performance Summary" page...

Simplification: The "All Lab" report now presents all data for each analyte on a single page, with as many analytes on a page as will conveniently fit. Page 4 lists the analytes reported by only a single (non-NIST) lab, as well as the Legend.

Major change: Due to popular request, the ranking method used in the "Relative Performance Summary" (i.e., $\frac{1}{4}$ of you getting a "1", $\frac{1}{4}$ a "4") has been replaced with a "standard score" related to how many standard deviations away from the median you are. It's now *possible* for everyone to score well. The new name for this page 5 of the "All Lab Report" is the "Interlaboratory Measurement Comparability Summary." Details of the calculation are provided on page 5, just to the right of the scores.

Freebie: Complete "All Lab Reports" for RRs XXX and XXXI analogous to the new RR XXXII report are included in your packet.

Same-as-before: The "Individualized" report's first six pages are identical to those of Round Robin XXXI (except that RRs XXIII - XXXI are now the reference period).

Page	Contents
1	Your lab's data table
2	%Bias barchart for retinol
3	%Bias barchart for α- and γ- tocopherol
4	%Bias barchart for total and *trans*-β-carotene
5	Interlaboratory median vs. your value scatterplots for retinol, α- and γ-tocopherol, total and *trans*-β-carotene
6	(Provisional) Performance Summary
7	Comparison plots for retinol, retinyl palmitate, α- and γ- tocopherol, total and *trans*-β-carotene.
8	Comparison plots for total α-carotene, β-cryptoxanthin, lycopene, lutein, zeaxanthin, and lutein & zeaxanthin

The "Comparison to Known Relationships" on pages 7 and 8 cheat somewhat, as only serum 199 was a replicate (serum 192 from RR XXX). Sera 200 to 202 are new, with no established relationships among them: they're included in the graphs mostly because it was easy to do so... and maybe the per-serum comparison of your data to the group's is interesting?

In RR XXX's report, the interlaboratory median was shown to be very similar to the "Trimmed Core" mean values used in prior years. In RR XXXI's report, the "estimated standard deviation (eSD)" - defined as 0.74*(interquartile range) - was shown to be midway between the simple SD and the "Trimmed Core" SD. As the simple SD is too big (some of you *are* outliers...) and the "Trimmed Core" SD is too small (throw away enough data and there is no variation), being midway is ok.

Now "to compare variation over analytes, concentration ranges, and time..." First, let's look at reproducibility over concentration. Figure 1 plots Median *vs* eSD for retinol, α-tocopherol, and total β-carotene over three time periods: RR I-XIII, RR XIV-XXI, and RR XXII-XXXII. Measurements were clearly less reproducible across laboratories during the first period for all three of these analytes, with no real difference between the second and third periods. (No data is available for γ-tocopherol for the first period.) Interestingly, the slope of the regression lines for retinol, α-tocopherol, and total β-carotene for the combined second and third periods are all about the same:

$$\log_{10}(\text{eSD}) = \text{Constant}_a + 0.83*\log_{10}(\text{Median}) \quad \text{or}$$
$$\text{eSD} = 10^{(\text{Constant}_a)}(\text{Median}^{0.83})$$

where the "Constant_a" is specific to each analyte. This relationship is shown as a dotted line in Figure 1. Note that the slope is 0.83, <u>not</u> 1.00: the eCV (estimated Coefficient of Variance, 100*eSD/Median) is <u>not</u> constant for all concentrations. The eCV is larger at low concentrations and smaller at high concentrations.

This really isn't surprising - why should CV be constant over all concentrations? But it does make estimating average interlaboratory reproducibility over time more difficult: the analyte levels of the sera affect the expected reproducibility for a given RR. To correct for this, we must adjust the observed eCV at its observed median to its expected value at some fixed median, using the above relation for each analyte. A convenient and fairly representative fixed median is the average of all observed medians.

Figure 2 plots a highly smoothed time series of adjutsed eCV values over all Round Robins, with some interesting timepoints noted. Bottom line? Interlaboratory reproducibility for RR samples was best when common control sera were being used (but how representative of real day-to-day performance was this?) Reproducibility has declined slightly since that time, but is clearly much better than the first few years. Perhaps a reproducibility plateau has been reached for all four analytes in the past year or so.

The Round Robin XXXIII Report will present an approach to documenting measurement consistency over time, not just reproducibility.

Once again, 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
301-975-3935
DLDuewer@enh.NIST.gov

Margaret Kline
Research Biologist

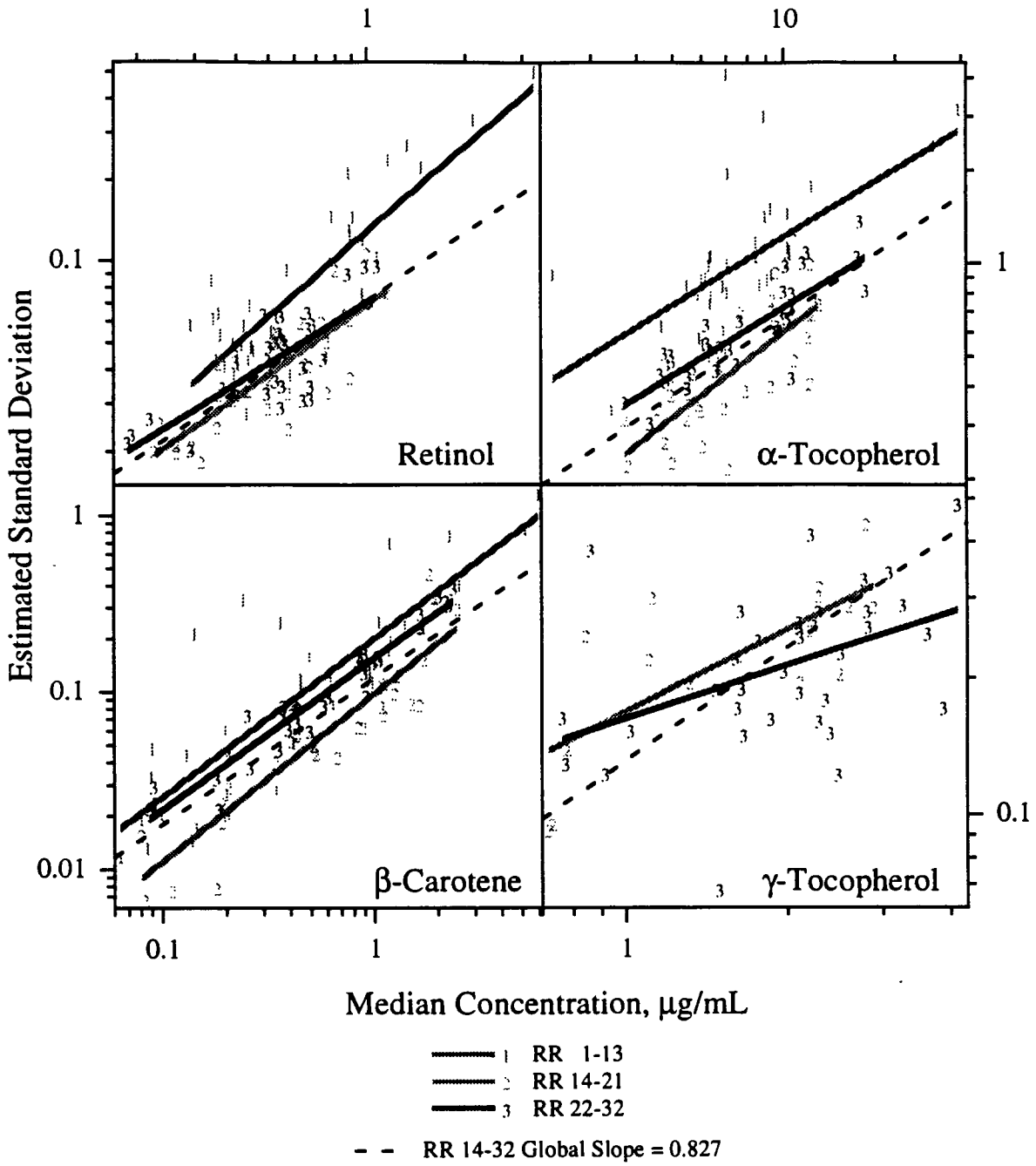


Figure 1

Interlaboratory Median *vs* eSD
for Retinol, Total β-Carotene, and α- and γ-Tocopherol

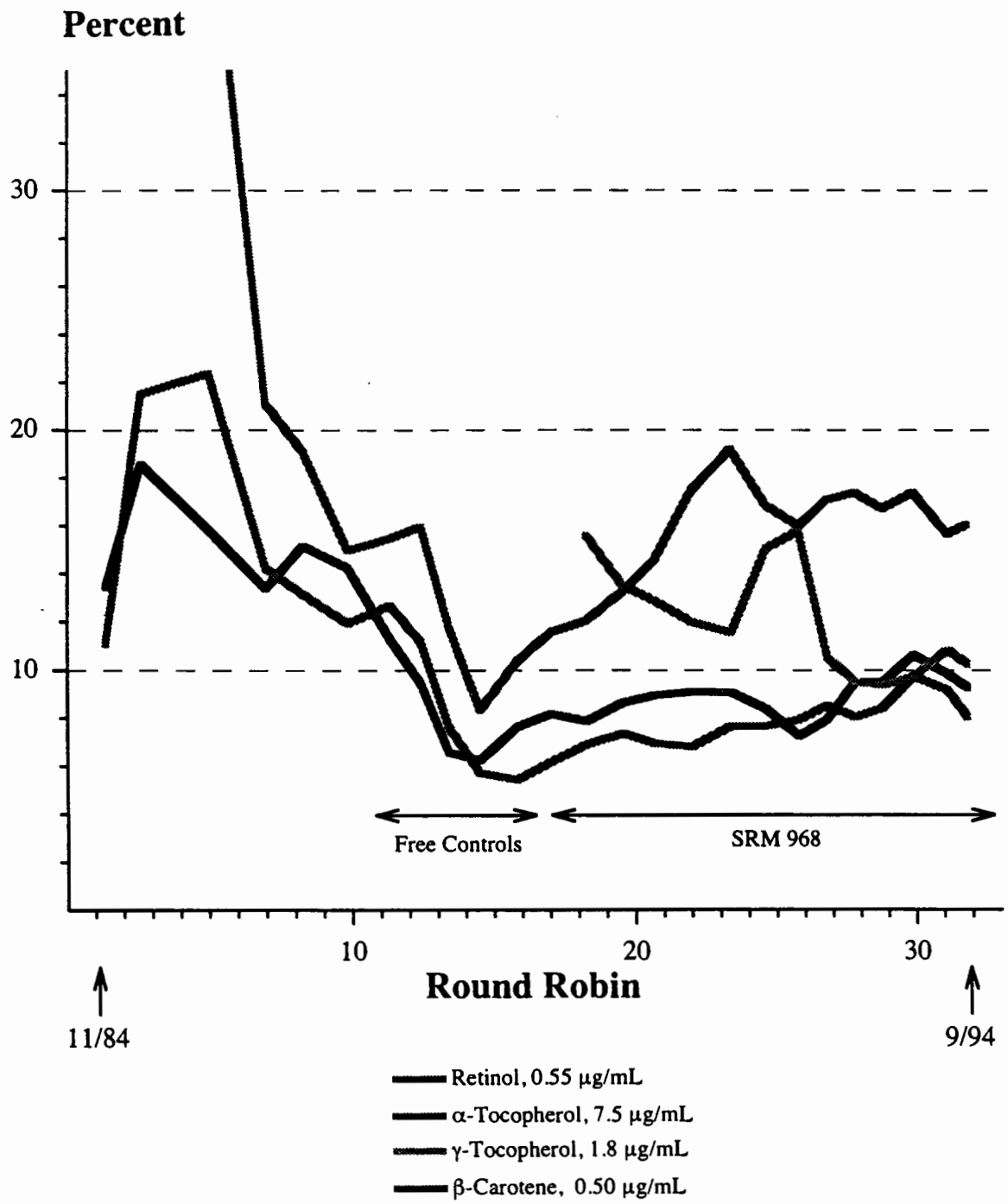


Figure 2

Interlaboratory Precision over Time
for Retinol, Total β-Carotene, and α- and γ-Tocopherol

Summary Results for all Sera
Round Robin Intercomparisons I - XXXII

			Retinol						a-Tocopherol						Total b-Carotene					
RR	Date	Serum	n	Mean	CV	NIST	Med	eCV	n	Mean	CV	NIST	Med	eCV	n	Mean	CV	NIST	Med	eCV
01	10/1/84	5a	16	1.01	56	0.90	0.85	8	11	9.1	12	10.0	9.0	10	12	1.53	42	1.16	1.19	59
		6a	16	2.40	38	2.16	2.11	16	11	4.1	60	3.4	3.4	10	12	5.51	25	5.06	5.19	16
		7a	16	3.82	44	3.75	3.25	16	11	25.8	34	25.1	25.3	10	12	6.20	21	4.30	6.00	22
02	1/30/85	18a	18	0.54	32	0.51	0.53	12	12	6.3	12	6.3	6.3	8	13	0.70	83	0.53	0.45	26
		19a	18	1.57	21	1.52	1.47	15	12	12.6	12	12.7	12.2	6	13	2.72	37	2.83	2.28	34
03	4/1/85	6	18	1.54	40	1.35	1.33	20	12	11.8	63	11.0	8.9	35	10	0.37	76	0.09	0.36	69
		7	18	1.04	46	0.89	0.87	24	12	8.6	62	7.1	7.1	28	12	0.21	88	0.40	0.14	150
		8	18	0.38	40	0.37	0.34	25	12	3.1	64	2.6	2.4	39	5	0.10	101	0.13	0.09	54
		9	18	1.33	51	1.25	1.16	21	12	9.1	64	7.2	7.0	60	11	0.28	99	0.14	0.24	139
05	10/1/85	19	21	0.52	28	0.44	0.51	11	15	30.4	27	33.8	29.6	11	18	0.50	41	0.39	0.49	32
		20	23	0.48	25	0.43	0.46	9	16	7.0	27	7.7	7.0	12	19	0.14	41	0.12	0.13	34
		21	23	0.44	28	0.38	0.42	14	16	6.4	31	7.6	6.3	17	19	0.41	33	0.34	0.42	21
07	6/1/86	31	24	0.75	23	0.77	0.77	13	20	11.2	18	11.9	11.9	15	17	0.19	103	0.12	0.14	11
		32 21	24	0.41	24	0.43	0.41	12	20	6.3	15	6.1	6.3	12	17	0.46	51	0.40	0.45	24
		33	27	0.35	32	0.36	0.35	13	23	6.3	24	6.6	6.3	17	20	0.16	133	0.07	0.10	21
		34	28	0.36	30	0.35	0.35	18	23	6.3	17	6.7	6.1	15	20	0.26	74	0.16	0.20	23
		35 34	27	0.35	30	0.36	0.34	18	23	6.2	24	6.7	6.3	13	20	0.25	72	0.16	0.20	31
		44 33	28	0.37	31	0.35	0.36	15	23	6.2	20	6.7	6.3	16	20	0.14	129	0.06	0.09	26
08	10/1/86	50	26	0.41	41	0.37	0.39	13	22	9.3	26	8.6	8.4	14	18	0.09	40	0.07	0.09	38
		51	26	0.46	34	0.44	0.43	10	22	7.2	22	7.1	7.0	14	18	1.17	34	1.04	1.05	24
		52	26	0.94	34	0.94	0.91	16	22	10.7	21	10.6	10.3	14	18	0.64	29	0.56	0.62	19
09	3/1/87	62	27	0.30	25	0.33	0.29	20	24	10.9	14	11.1	10.4	11	19	0.15	27	0.14	0.15	12
		63	27	0.88	21	0.92	0.88	15	24	7.1	16	6.7	6.8	13	19	0.91	25	0.88	0.92	14
		64	27	0.57	20	0.60	0.57	16	24	5.0	24	4.8	4.7	14	19	0.07	22	0.07	0.06	19
		65	27	0.46	19	0.48	0.44	16	24	9.5	12	9.3	9.3	10	19	0.40	21	0.38	0.41	13
		66	27	0.80	24	0.81	0.78	19	24	8.9	14	8.1	8.4	13	19	1.37	28	1.21	1.41	17
11	10/1/87	72 63	23	0.87	17	0.92	0.89	14	20	6.9	19	6.8	6.6	7	16	1.07	37	0.86	0.98	12
		73 52	23	0.97	17	0.97	0.97	8	20	11.3	22	10.9	10.7	12	16	0.69	37	0.55	0.64	13
		74 65	23	0.46	19	0.47	0.44	11	20	9.2	21	9.4	8.9	16	16	0.47	38	0.36	0.44	17
		75 50	23	0.40	18	0.39	0.40	10	20	9.1	22	9.0	8.6	10	16	0.09	38	0.07	0.08	16
		76 51	23	0.45	20	0.44	0.45	11	20	7.3	21	7.3	6.9	9	16	1.30	37	1.01	1.17	15

Summary Results for all Sera
Round Robin Intercomparisons I - XXXII

				Retinol						a-Tocopherol						Total b-Carotene					
RR	Date	Serum		n	Mean	CV	NIST	Med	eCV	n	Mean	CV	NIST	Med	eCV	n	Mean	CV	NIST	Med	eCV
12	2/29/88	77	D	31	1.05	21	1.01	1.07	10	28	9.1	16	9.5	9.2	17	20	0.99	23	1.05	1.00	15
		78	A	31	0.40	16	0.36	0.40	11	28	5.5	15	5.7	5.5	16	20	0.20	19	0.20	0.21	10
		79		31	0.38	23	0.34	0.37	11	28	5.8	15	6.1	5.8	14	20	2.58	24	2.54	2.51	16
		80	C	31	0.96	23	0.90	0.97	12	28	10.2	14	10.7	10.3	11	20	0.85	20	0.77	0.85	16
		81	B	31	0.45	17	0.41	0.44	11	28	6.0	15	5.7	6.0	15	20	0.20	25	0.22	0.21	22
13	6/1/88	90		28	0.40	9	0.38	0.39	10	26	7.0	9	7.0	6.9	8	18	0.09	30	0.09	0.08	24
		91	D	28	1.03	8	1.00	1.02	7	26	9.4	9	9.4	9.3	8	18	1.00	15	1.12	0.96	13
		92		28	0.59	11	0.57	0.58	7	26	6.0	11	5.9	6.0	9	18	1.24	17	1.33	1.19	16
		93		28	0.75	8	0.70	0.75	6	26	4.9	11	4.6	4.9	10	18	0.35	17	0.33	0.34	8
		94	B	28	0.44	13	0.41	0.43	6	26	6.0	10	5.9	5.9	10	18	0.21	19	0.22	0.20	13
14	10/1/88	95	92	23	0.59	12		0.57	6	21	5.8	24		5.7	8	15	1.28	10		1.28	10
		96	66*1.2	23	0.91	8		0.89	4	21	9.3	7		9.2	4	15	1.70	7		1.72	9
		97	62*1.2	23	0.36	19		0.35	7	21	11.7	9		11.8	3	15	0.18	7		0.18	4
		98	92	23	0.58	9		0.58	4	21	6.0	20		5.8	6	15	1.28	10		1.25	8
		99	64*1.2	23	0.64	13		0.65	6	21	5.4	12		5.4	7	15	0.09	21		0.08	8
15	3/1/89	100		28	0.31	7	0.31	0.31	6	26	5.0	10	4.9	4.9	8	16	0.11	13	0.14	0.11	7
		101		28	0.47	8	0.47	0.47	8	26	7.7	10	7.3	7.9	5	16	0.66	7	0.66	0.67	6
		102		28	1.18	9	1.17	1.16	7	26	12.3	8	11.9	12.5	6	16	1.48	7	1.40	1.50	6
16	6/1/89	109	90	27	0.41	23	0.39	0.39	8	25	7.0	7	6.8	7.1	3	17	0.08	28	0.09	0.08	19
		110	79	27	0.40	43	0.33	0.36	9	25	6.0	11	5.6	5.9	6	17	2.57	22	2.45	2.47	12
		111	93	27	0.79	26	0.73	0.76	4	25	4.9	9	4.4	5.0	5	17	0.37	24	0.30	0.34	12
		112	B	27	0.46	26	0.38	0.43	8	24	6.0	7	5.4	6.0	4	17	0.21	26	0.18	0.21	14
17	10/1/89	115	62*1.2	30	0.39	31	0.36	0.35	12	25	11.5	14	12.0	11.9	6	18	0.21	36	0.15	0.19	10
		116		30	1.07	22	1.06	1.00	11	25	7.6	10	7.5	7.7	9	18	1.11	10	0.97	1.08	8
		117	64*1.2	30	0.69	20	0.66	0.66	6	25	5.4	7	5.3	5.4	6	18	0.11	80	0.07	0.09	23
		118		30	0.54	15	0.49	0.51	10	24	10.1	14	10.1	10.3	6	18	0.52	14	0.40	0.51	10
		119	66*1.2	29	0.93	19	0.98	0.89	7	24	9.3	12	9.2	9.4	7	18	1.68	8	1.36	1.65	5
18	3/1/90	120		30	0.26	11	0.28	0.26	8	29	4.8	7	5.0	4.8	6	21	0.41	12	0.49	0.41	13
		121		30	0.50	16	0.52	0.50	6	29	6.7	13	7.2	6.6	7	21	1.00	20	0.63	1.05	10
		122		30	0.89	23	0.91	0.85	7	30	9.4	13	9.2	9.3	8	21	1.96	18	1.60	1.94	18

Summary Results for all Sera
Round Robin Intercomparisons I - XXXII

			Retinol						a-Tocopherol						Total b-Carotene					
RR	Date	Serum	n	Mean	CV	NIST	Med	eCV	n	Mean	CV	NIST	Med	eCV	n	Mean	CV	NIST	Med	eCV
19	6/1/90	130	36	0.24	21	0.21	0.23	11	34	4.0	45	3.3	3.8	6	25	1.00	25	0.98	0.96	11
		131	37	0.52	22	0.52	0.51	10	34	6.3	23	6.8	6.6	7	24	0.23	66	0.21	0.19	12
		132	37	0.30	17	0.31	0.29	7	34	4.4	24	4.4	4.6	7	25	1.14	29	1.11	1.11	7
		133	37	0.76	17	0.82	0.75	7	34	11.2	21	11.5	11.9	8	25	0.55	30	0.50	0.53	8
20	10/22/90	138	28	0.54	17	0.58	0.55	9	30	10.4	12	10.6	10.3	10	21	1.70	31	1.97	1.67	14
		139	28	0.62	15	0.67	0.63	9	30	9.7	8	10.1	9.7	6	21	0.44	12	0.70	0.44	13
		140 133	29	0.72	13	0.83	0.73	8	31	11.6	11	11.4	11.6	5	22	0.52	15	0.51	0.50	11
		141 122	29	0.80	17	0.90	0.80	12	31	9.5	14	8.9	9.6	10	22	1.90	30	1.58	1.82	26
		142 130	29	0.22	12	0.23	0.23	9	31	3.9	16	3.3	3.7	8	22	0.91	20	0.95	0.89	7
21	3/1/91	144 C	34	0.96	16	0.92	0.96	8	35	10.7	8	10.4	10.7	5	24	0.84	30	0.80	0.84	7
		145 121	34	0.50	16	0.49	0.50	7	35	6.9	8	6.6	6.9	5	24	1.14	28	1.04	1.11	10
		146 131	34	0.54	19	0.52	0.52	11	35	6.6	11	6.3	6.7	7	24	0.24	66	0.20	0.19	12
		147 120	34	0.28	23	0.25	0.26	8	35	5.0	13	4.6	4.9	8	24	0.45	27	0.40	0.43	13
22	7/9/91	148 968aL	45	0.20	31	0.17	0.19	11	45	4.7	17	4.5	4.7	11	34	0.27	35	0.23	0.25	29
		149 968aM	45	0.49	20	0.50	0.50	9	45	10.2	17	10.3	10.5	4	34	0.88	17	1.05	0.89	14
		150 968aH	45	0.75	91	0.63	0.66	10	45	16.0	13	16.2	16.6	5	34	2.31	15	2.86	2.25	14
23	9/1/91	155	44	0.53	15	0.57	0.53	9	44	7.5	13	7.6	7.4	6	30	0.35	28	0.42	0.37	22
		156	44	0.40	15	0.41	0.40	11	44	10.0	14	9.8	10.0	8	31	0.79	23	0.85	0.85	19
		157	44	0.98	16	0.99	0.98	10	44	6.3	14	6.4	6.3	8	31	0.09	42	0.09	0.09	32
		159 968aM	43	0.50	18	0.52	0.50	10	43	10.5	15	10.4	10.5	7	30	0.86	31	0.79	0.90	18
24	3/6/92	160 157	43	1.01	23	0.91	1.02	7	41	6.3	13	5.9	6.3	8	33	0.10	38	0.09	0.09	24
		161 968aH	44	0.68	14	0.60	0.68	9	42	15.9	11	15.9	16.0	9	34	2.19	33	2.40	2.18	15
		162 132	44	0.29	18	0.28	0.29	7	42	4.6	13	4.3	4.6	9	34	1.02	22	1.12	1.03	15
		163 A	44	0.39	13	0.34	0.39	9	42	5.7	13	5.1	5.6	8	34	0.18	22	0.20	0.18	12
25	6/26/92	164 D	31	1.07	10	0.99	1.07	9	33	9.7	13	9.6	9.4	6	29	1.09	35	1.06	1.05	14
		165 122	31	0.89	12	0.86	0.87	10	33	9.8	9	9.5	9.8	7	28	2.09	25	1.56	2.02	17
		166 131	31	0.54	12	0.52	0.53	7	33	6.7	15	6.5	6.5	7	29	0.22	113	0.21	0.18	18
		167 968aL	31	0.20	20	0.17	0.19	12	33	4.8	16	4.5	4.8	9	28	0.30	68	0.22	0.26	15
26	10/16/92	168 138	34	0.55	11	0.55	0.55	6	36	10.5	10	10.1	10.5	7	31	1.66	17	1.59	1.66	14
		169 139	34	0.64	14	0.66	0.63	6	36	9.6	12	9.9	9.7	6	31	0.43	23	0.45	0.44	18
		170	35	0.67	7	0.69	0.67	5	37	6.2	10	6.5	6.2	10	32	0.62	17	0.66	0.60	15
		171	35	0.55	8	0.51	0.54	5	37	4.9	11	4.8	5.0	10	32	0.40	16	0.48	0.39	16
		172 170	35	0.67	7	0.72	0.67	5	37	6.1	10	6.2	6.2	9	32	0.61	16	0.61	0.58	14

Summary Results for all Sera
Round Robin Intercomparisons I - XXXII

				Retinol						a-Tocopherol						Total b-Carotene					
RR	Date	Serum		n	Mean	CV	NIST	Med	eCV	n	Mean	CV	NIST	Med	eCV	n	Mean	CV	NIST	Med	eCV
27	3/1/93	173	968aM	46	0.50	17	0.52	0.50	8	42	10.6	13	10.8	10.5	8	38	0.91	25	0.89	0.91	10
		174	155	46	0.54	18	0.54	0.54	10	42	7.4	15	7.5	7.6	9	39	0.40	53	0.32	0.37	20
		175	156	46	0.41	16	0.41	0.40	8	42	10.1	14	9.8	10.1	10	39	0.85	35	0.69	0.83	18
		176	171	46	0.55	15	0.56	0.54	10	42	4.9	15	4.7	4.9	10	39	0.42	39	0.32	0.41	18
		177	968aH	26	0.67	16	0.63	0.66	9	24	15.7	8	15.8	15.8	7	23	2.37	18	1.95	2.36	16
28	6/1/93	181		44	0.50	23	0.41	0.48	13	44	6.7	22	7.4	7.1	7	33	0.14	105	0.10	0.09	26
		182		43	0.54	17	0.51	0.55	9	43	5.3	22	5.7	5.5	7	33	0.46	39	0.39	0.43	16
		183		44	0.57	24	0.50	0.56	9	44	10.7	23	11.7	11.2	9	33	0.39	42	0.39	0.37	19
		184		44	0.37	25	0.37	0.36	10	44	4.3	29	4.5	4.3	7	32	0.37	33	0.37	0.35	13
		185	182	44	0.56	23	0.50	0.55	11	44	5.3	25	5.7	5.5	8	33	0.45	36	0.41	0.42	19
29	9/1/93	186	130	46	0.22	22	0.23	0.22	12	47	3.5	24	3.6	3.7	10	38	0.93	42	0.96	0.93	15
		187	121	45	0.50	20	0.50	0.49	7	46	6.5	24	6.6	6.7	8	38	1.07	49	1.21	1.05	15
		188	133	46	0.72	15	0.70	0.73	6	47	11.2	22	11.4	11.5	9	38	0.50	41	0.56	0.49	12
		189	139	46	0.63	14	0.61	0.63	5	47	9.3	22	9.3	9.5	6	38	0.43	38	0.50	0.43	19
		190	138	46	0.54	15	0.54	0.55	8	47	10.1	23	10.1	10.2	7	38	1.64	43	1.60	1.65	17
30	3/19/94	191		40	7.07	27	6.99	6.85	13	42	23.8	20	22.0	24.0	12	34	1.63	21	1.62	1.64	12
		192		43	1.16	23	1.12	1.11	15	43	7.3	14	6.9	7.3	9	34	0.60	21	0.55	0.60	16
		193		43	1.05	24	0.93	1.03	9	43	27.1	17	26.0	27.1	10	34	0.39	37	0.37	0.37	18
		194		42	0.45	25	0.44	0.43	8	42	7.3	12	7.3	7.3	8	31	0.08	113	0.05	0.06	35
31	6/14/94	195	198/2	37	0.37	17	0.38	0.38	11	38	7.0	17	6.6	7.1	12	29	0.34	22	0.36	0.34	14
		196	183	37	0.55	15	0.56	0.55	9	38	11.4	15	10.9	11.5	7	29	0.35	21	0.36	0.35	16
		197	198/4	37	0.19	19	0.19	0.20	13	38	3.6	22	3.4	3.7	15	29	0.15	31	0.16	0.15	21
		198		38	0.71	15	0.71	0.73	9	39	14.0	16	13.5	14.2	8	30	0.67	17	0.69	0.67	12
32	8/30/94	199	192	36	1.08	11		1.06	8	38	7.1	16		7.2	6	29	0.57	17		0.59	12
		200	968bL	36	0.31	16		0.30	9	38	7.0	19		7.1	8	29	0.25	24		0.25	16
		201	968bM	36	0.52	10		0.51	8	38	9.8	16		10.0	6	29	0.60	19		0.61	12
		202	968bH	36	0.89	12		0.88	9	38	17.4	16		17.7	8	29	1.20	19		1.23	16

Figure 3
Interlaboratory Retinol Measurement of Replicate Samples

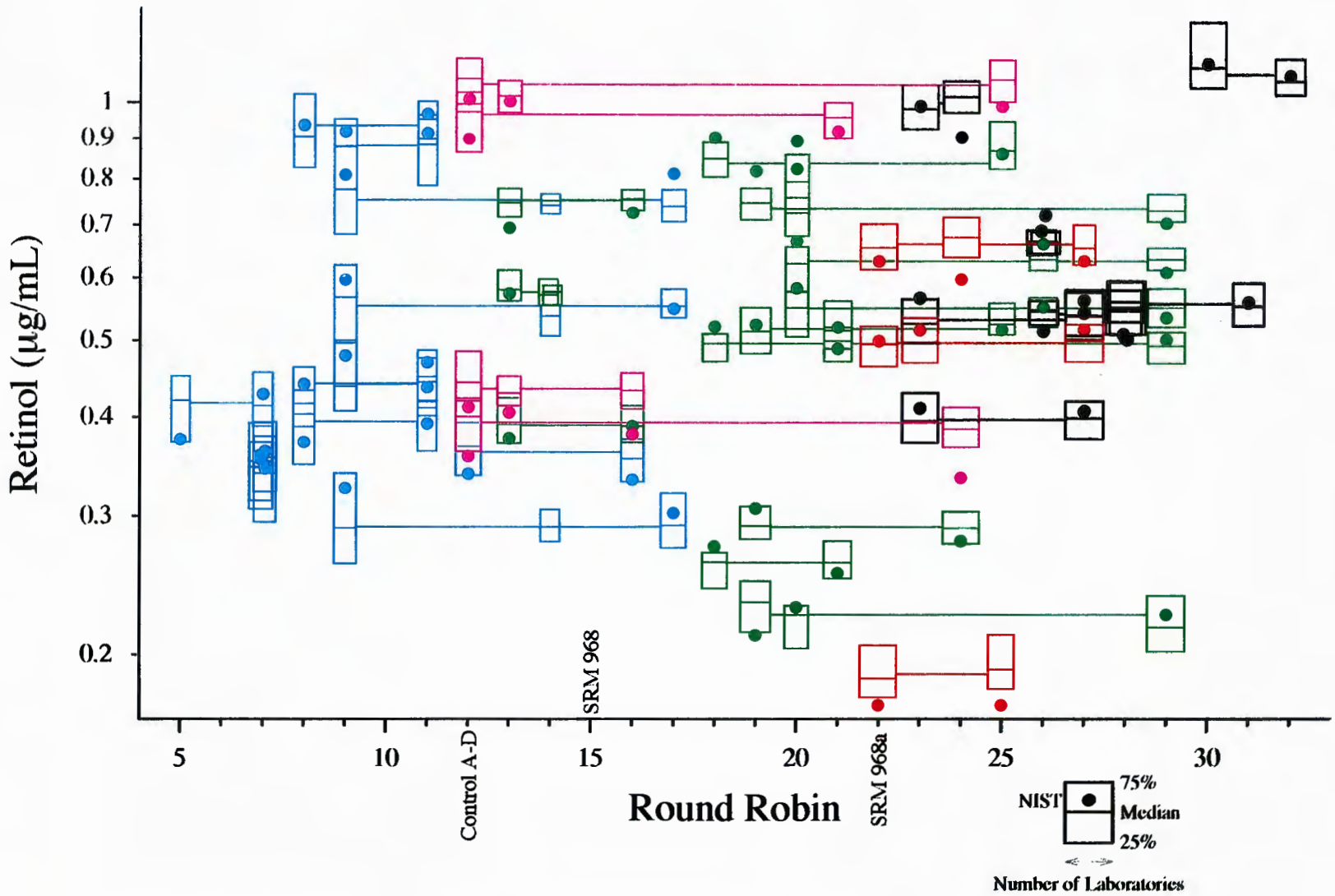


Figure 4
Interlaboratory α-Tocopherol Measurement of Replicate Samples

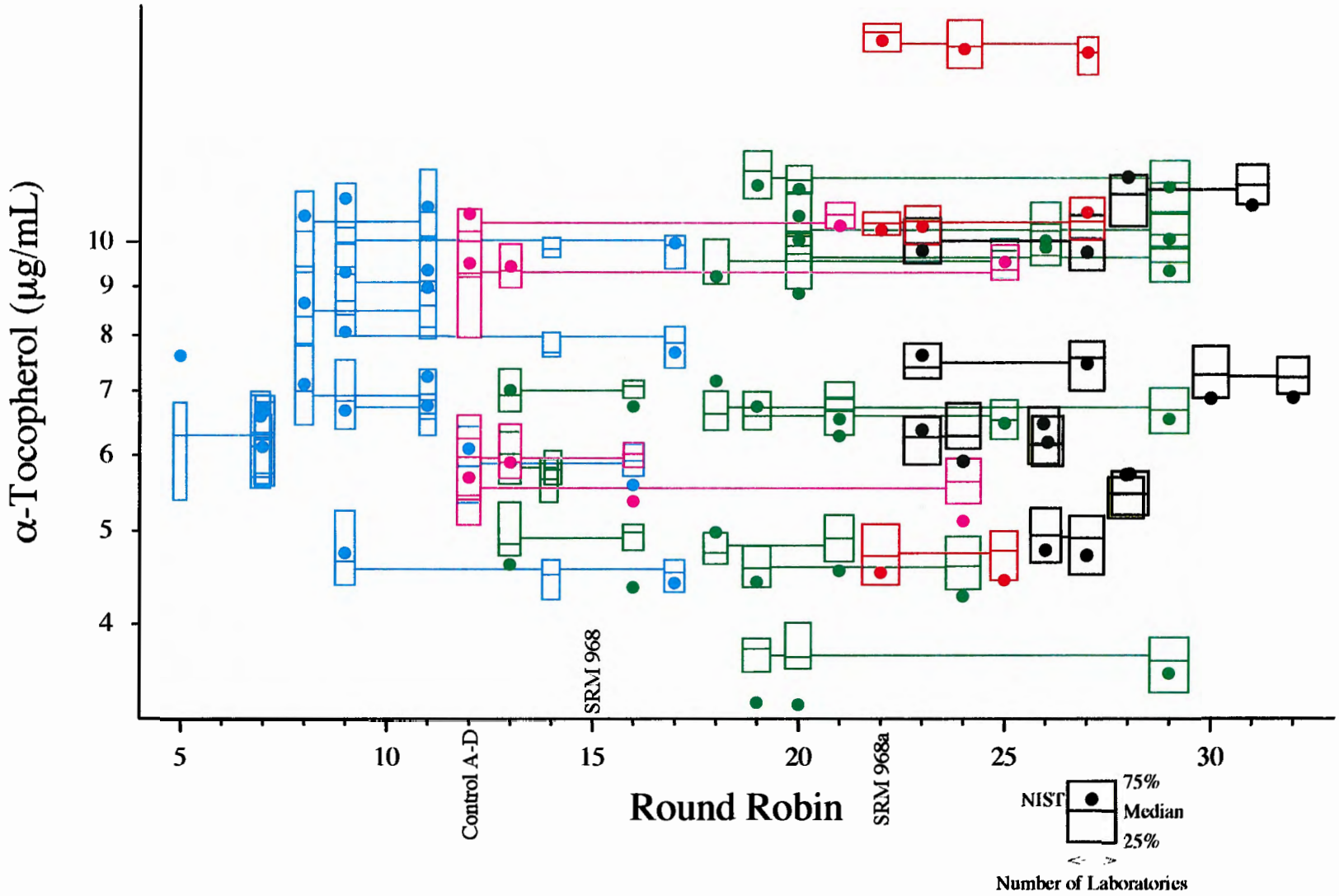
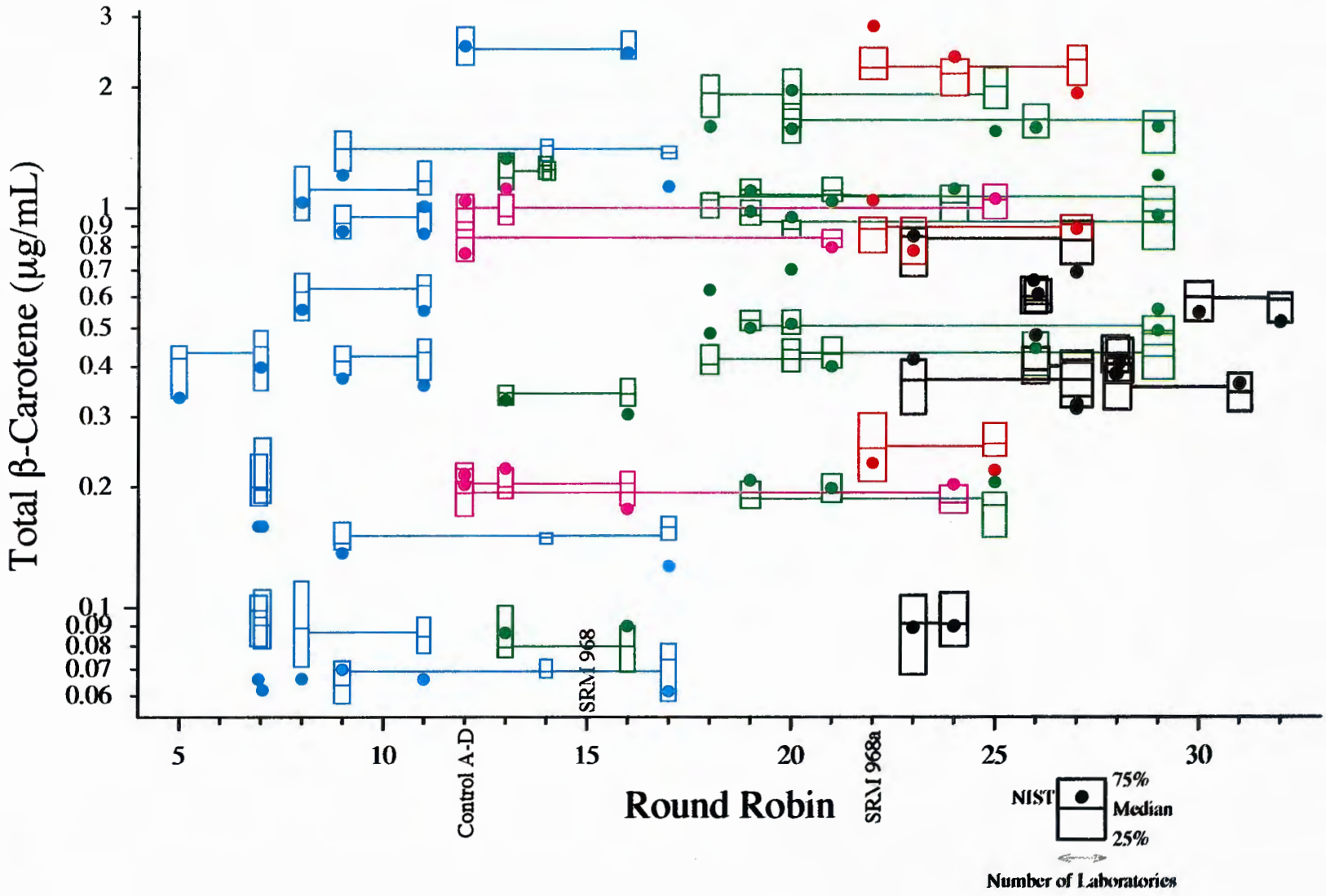


Figure 5
Interlaboratory Total β-Carotene Measurement of Replicate Samples



Appendix K. "All-Lab Report" for RR32

The following five pages are the "All-Lab Report" for RR32 as provided to all participants, with the following exceptions:

- the participant identifiers (Lab) have been altered.
- the order in which the participant results are listed has been altered.
- the robust standard deviation (eSD) has been recalculated using a more efficient robust estimator: the adjusted median absolute deviation from the median (MADe) rather than the adjusted interquartile range (IQR).
- the robust coefficient of variation (eCV) has been recalculated using the recalculated eSD.
- The "Interlaboratory Measurement Comparability Summary" scores have been recalculated using the recalculated eSD

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 XXXII Laboratory Results
Results in ug/mL

	Total Retinol				Retinyl Palmitate				α-Tocopherol				γ/β-Tocopherol				δ-Tocopherol			
Lab	199	200	201	202	199	200	201	202	199	200	201	202	199	200	201	202	199	200	201	202
FSV-BA	1.020	0.291	0.504	0.855	0.081	0.111	0.195	0.256	6.97	6.95	9.98	17.8	2.21	1.67	2.21	3.41				
FSV-BD	1.137	0.308	0.512	0.878					6.60	6.20	9.10	16.1								
FSV-BE	1.155	0.301	0.554	0.945					7.69	7.22	10.39	18.0	2.53	1.75	2.53	3.81				
FSV-BF	1.098	0.311	0.540	0.890					7.40	7.08	10.25	18.1	2.56	1.82	2.57	4.05				
FSV-BH	0.899	0.301	0.505	0.776	*nq*	0.073	0.108	0.227	7.19	7.25	10.17	17.8	2.03	1.45	2.02	3.11				
FSV-BI	1.027	0.281	0.507	0.873	*nq*	0.134	0.219	0.282	7.43	7.14	10.07	17.3	2.53	1.75	2.49	3.80				
FSV-BJ	1.049	0.285	0.513	0.861	*nq*	0.091	0.181	0.283	7.62	7.57	10.48	18.3	2.67	1.92	2.60	4.23				
FSV-BK	1.056	0.277	0.494	0.853					6.51	6.26	9.02	16.4								
FSV-BL	1.088	0.401	0.601	0.974					9.05	9.05	11.20	21.5								
FSV-BM	1.100	0.299	0.547	0.906					7.00	6.60	9.20	17.5								
FSV-BN	1.057	0.317	0.530	0.866	0.031	0.087	0.155	0.244	9.76	9.71	12.24	19.5	2.55	1.99	2.54	3.66				
FSV-BO	1.305	0.281	0.565	1.057					6.45	5.94	8.92	16.3								
FSV-BP	1.220	0.315	0.582	1.109					7.75	7.49	10.05	17.1								
FSV-BQ	0.945	0.355	0.521	0.853					5.55	4.61	9.65	15.3								
FSV-BT	1.041	0.270	0.472	0.807	0.027	0.099	0.180	0.263	7.22	6.71	9.54	16.7	2.50	1.65	2.41	3.82	0.81	0.87	1.00	0.95
FSV-BX																				
FSV-BY	1.100	0.327	0.549	0.907	0.012	0.081	0.173	0.283	7.21	7.07	9.81	17.0	2.35	1.64	2.31	3.73				
FSV-BZ									7.92	7.97	10.50	19.0	2.21	1.71	2.15	3.26				
FSV-CA	1.023	0.336	0.538	0.879					7.09	7.20	9.86	17.9								
FSV-CB	1.415	0.380	0.620	1.125					7.69	7.56	10.33	18.7								
FSV-CH	1.090	0.291	0.502	0.889					7.72	7.43	10.90	20.0	2.05	1.44	2.05	3.27				
FSV-CK	0.910	0.310	0.520	0.920					6.81	7.11	9.52	17.1	2.46	1.80	2.43	3.80				
FSV-CL	0.913	0.219	0.420	0.657					6.32	4.83	7.92	13.0								
FSV-CM									7.30	7.20	10.10	22.3								
FSV-CO	0.970	0.260	0.464	0.804					6.75	6.31	9.35	17.0								
FSV-CQ	1.079	0.265	0.469	0.751					7.73	7.29	12.13	18.8								
FSV-CR	1.020	0.290	0.510	0.870					7.30	7.00	9.90	17.7								
FSV-CT	1.135	0.275	0.493	0.889					7.29	6.98	9.90	17.0	2.18	1.47	2.27	3.33				
FSV-CU	1.051	0.285	0.490	0.809	0.066	0.108	0.147	0.184	6.38	6.13	9.00	15.8								
FSV-CV	1.240	0.310	0.490	0.870					7.50	7.20	9.70	18.9	2.60	1.60	2.40	3.90				
FSV-CX	0.970	0.250	0.440	0.730	0.070	0.120	0.190	0.270	7.25	7.17	10.29	18.6	2.18	1.43	2.07	3.55				
FSV-CY	1.070	0.287	0.522	0.848					7.16	6.95	10.00	20.2								
FSV-DJ	1.090	0.300	0.550	0.880					7.50	7.30	10.30	20.1								
FSV-DK	0.890	0.220	0.410	0.690					7.15	8.44	11.60	19.2								
FSV-DL	1.111	0.294	0.523	0.764					6.96	6.63	10.63	15.6	5.66	3.76	6.22	8.17				
FSV-DS	1.030	0.480	0.510	0.950					7.37	8.88	9.88	17.6								
FSV-DU		0.332	0.621	1.368						7.77	11.40	19.1								
FSV-DX	1.220	0.316	0.568	0.962					8.45	7.15	10.60	19.4								
FSV-EA	1.269	0.357	0.621	1.088					7.87	7.55	10.70	19.0	2.61	1.98	2.63	3.91				
FSV-EC	1.075	0.323	0.509	0.961					7.34	7.64	9.96	16.6								
FSV-EK	1.260	0.370	0.600	1.030					6.11	5.74	6.69	15.5	1.74	1.19	1.72	2.84				
FSV-EX	0.950	0.290	0.440	0.760					7.21	7.18	10.04	18.2								
FSV-FD	1.039	0.295	0.516	0.903					7.17	7.03	9.93	17.1								
n	39	40	40	40	6	9	9	9	41	42	42	42	18	18	18	18	1	1	1	1
Min	0.890	0.219	0.410	0.657	0.012	0.073	0.108	0.184	5.55	4.61	6.69	13.0	1.74	1.19	1.72	2.84				
Mean	1.080	0.306	0.521	0.895	0.048	0.100	0.172	0.255	7.29	7.11	10.03	17.9	2.53	1.78	2.53	3.87	0.81	0.87	1.00	0.95
Max	1.415	0.480	0.621	1.368	0.081	0.134	0.219	0.283	9.76	9.71	12.24	22.3	5.66	3.76	6.22	8.17				
SD	0.117	0.047	0.052	0.130	0.028	0.020	0.032	0.033	0.73	0.94	0.98	1.7	0.82	0.54	0.95	1.13				
CV	11	15	10	15	59	20	19	13	10	13	10	10	32	30	38	29				
NISTa	1.080	0.303	0.539	0.923					6.90	6.82	9.39	17.2	2.11	1.43	2.04	3.25				
NISTb		0.299	0.509	0.901		0.090	0.161	0.221		7.05	10.10	17.8		1.65	2.30	3.65		0.09	0.13	0.18
Median	1.070	0.300	0.515	0.879	0.049	0.099	0.180	0.263	7.25	7.16	10.02	17.8	2.48	1.69	2.41	3.76				
eCV	7	9	7	11	66	18	12	11	6	7	6	9	10	16	11	10				

K2

Round Robin XXXII Laboratory Results
Results in ug/mL

	Total β-Carotene				trans-β-Carotene				Total cis-β-Carotene				9-cis-β-Carotene			
Lab	199	200	201	202	199	200	201	202	199	200	201	202	199	200	201	202
FSV-BA	0.615	0.266	0.705	1.29	0.574	0.251	0.662	1.22	0.041	0.015	0.043	0.075				
FSV-BD																
FSV-BE	0.578	0.239	0.624	1.21												
FSV-BF	0.571	0.319	0.587	1.23												
FSV-BH	0.594	0.223	0.654	1.25	0.564	0.213	0.623	1.19	*0.030*	*0.010*	*0.031*	*0.059*				
FSV-BI	0.591	0.244	0.614	1.10												
FSV-BJ	0.602	0.277	0.673	1.21												
FSV-BK																
FSV-BL																
FSV-BM																
FSV-BN	0.542	0.218	0.449	1.00	0.485	0.196	0.415	0.90	*0.057*	*0.022*	*0.034*	*0.098*	0.031	*nq*	*nq*	0.049
FSV-BO	0.766	0.369	0.782	1.45												
FSV-BP	0.463	0.237	0.496	0.86												
FSV-BQ	0.661	0.328	0.399	1.45												
FSV-BT	0.524	0.246	0.598	1.21	0.493	0.231	0.560	1.12	0.032	0.015	0.038	0.089				
FSV-BX	>0.540	>0.240	>0.575	>1.099		0.240	0.575	1.10								
FSV-BY	0.626	0.306	0.763	1.54												
FSV-BZ	0.470	0.298	0.609	0.95												
FSV-CA																
FSV-CB	0.595	0.226	0.594	1.14												
FSV-CH	0.604	0.249	0.667	1.41												
FSV-CK	0.706	0.278	0.749	1.44												
FSV-CL	0.363	0.136	0.356	0.73												
FSV-CM																
FSV-CO	0.611	0.269	0.670	1.28	0.531	0.234	0.565	1.12	*0.081*	*0.035*	*0.106*	*0.163*	0.063	<0.014	0.057	0.120
FSV-CQ	0.376	0.120	0.428	0.87												
FSV-CR																
FSV-CT	0.613	0.264	0.634	1.28												
FSV-CU	0.517	0.218	0.562	1.03	0.480	0.211	0.532	0.98	*0.037*	*0.007*	*0.030*	*0.049*				
FSV-CV	0.610	0.275	0.640	1.27												
FSV-CX	0.440	0.200	0.480	0.85												
FSV-CY	0.500	0.223	0.557	1.59												
FSV-DJ																
FSV-DK																
FSV-DL	0.468	0.193	0.558	0.92												
FSV-DS	0.666	0.440	0.742	1.33												
FSV-DU		>0.320	>0.600	>1.060		0.320	0.600	1.06								
FSV-DX	0.556	0.239	0.633	1.24												
FSV-EA	0.839	0.353	0.885	1.69												
FSV-EC	0.596	0.258	0.654	1.42												
FSV-EK	0.680	0.260	0.540	1.15												
FSV-EX	0.560	0.180	0.590	1.15												
FSV-FD	0.585	0.241	0.656	1.24												
n	32	32	32	32	6	8	8	8	6	6	6	6	2	0	1	2
Min	0.363	0.120	0.356	0.73	0.480	0.196	0.415	0.90	0.030	0.007	0.030	0.049	0.031			0.049
Mean	0.578	0.256	0.611	1.21	0.521	0.237	0.566	1.08	0.046	0.017	0.047	0.089	0.047		0.057	0.085
Max	0.839	0.440	0.885	1.69	0.574	0.320	0.662	1.22	0.081	0.035	0.106	0.163	0.063			0.120
SD	0.100	0.063	0.114	0.23	0.041	0.038	0.073	0.10	0.019	0.010	0.029	0.041				
CV	17	25	19	19	8	16	13	10	42	58	62	46				
NISTa	0.520	0.240	0.624	1.05	0.448	0.209	0.558	0.98	*0.072*	*0.031*	*0.066*	*0.072*				
NISTb		0.255	0.641	1.27		0.231	0.585	1.16		*0.024*	*0.056*	*0.110*				
Median	0.593	0.248	0.619	1.23	0.512	0.232	0.570	1.11	0.039	0.015	0.036	0.082				
eCV	11	17	13	19	8	12	9	9	30	59	23	35				

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Round Robin XXXII Laboratory Results
Results in ug/mL

	Total α-Carotene				Total Lycopene				trans-Lycopene				Total β-Cryptoxanthin				Total Lutein			
Lab	199	200	201	202	199	200	201	202	199	200	201	202	199	200	201	202	199	200	201	202
FSV-BA	0.020	0.023	0.038	0.047					0.35	0.10	0.17	0.18	0.067	0.041	0.054	0.050				
FSV-BD																				
FSV-BE	0.018	0.029	0.038	0.043																
FSV-BF	0.029	0.033	0.057	0.076	0.59	0.251	0.310	0.391												
FSV-BH	0.017	0.017	0.032	0.044	0.61	0.165	0.300	0.322					0.078	0.040	0.055	0.052	0.070	0.055	0.062	0.051
FSV-BI	0.014	0.016	0.027	0.035	0.52	0.152	0.240	0.250					0.062	0.028	0.040	0.038	0.070	0.054	0.057	0.035
FSV-BJ	0.022	0.030	0.040	0.053	0.55	0.177	0.288	0.277												
FSV-BK																				
FSV-BL																				
FSV-BM																				
FSV-BN	0.015	0.016	0.026	0.037	0.71	0.250	0.302	0.358					0.023	0.020	0.024	0.022	0.040	0.022	0.026	0.016
FSV-BO	0.010	0.017	0.024	0.032	0.50	0.171	0.252	0.291					0.109	0.102	0.109	0.110				
FSV-BP	0.017	0.017	0.023	0.026	0.65	0.201	0.258	0.360					0.154	0.081	0.105	0.095				
FSV-BQ																				
FSV-BT	0.019	0.020	0.030	0.039	0.48	0.180	0.265	0.305					0.061	0.032	0.044	0.043	0.090	0.064	0.068	0.036
FSV-BX	0.003	0.011	0.014	0.015	0.87	0.293	0.389	0.337					0.075	0.049	0.058	0.047	0.073	0.052	0.056	0.031
FSV-BY	0.018	0.020	0.044	0.068	0.63	0.183	0.286	0.327					0.075	0.042	0.051	0.053	0.096	0.067	0.062	0.034
FSV-BZ	0.042	0.032	0.059	0.053	0.38	0.175	0.229	0.243									0.072	0.061	0.064	0.036
FSV-CA																				
FSV-CB	0.015	0.014	0.024	0.032	0.42	0.108	0.180	0.223					0.056	0.026	0.037	0.038				
FSV-CH	0.008	0.008	0.010	0.020	0.63	0.170	0.270	0.314												
FSV-CK	0.024	0.023	0.038	0.055	0.77	0.219	0.192	0.433					0.066	0.030	0.043	0.047				
FSV-CL	0.010	0.011	0.016	0.020	0.59	0.167	0.266	0.313					0.164	0.133	0.143	0.138				
FSV-CM																				
FSV-CO	0.034	0.035	0.052	0.076	0.45	0.156	0.240	0.250					0.084	0.058	0.063	0.063	0.135	0.129	0.102	0.058
FSV-CQ																				
FSV-CR																				
FSV-CT																				
FSV-CU																				
FSV-CV					0.56	0.185	0.285	0.300												
FSV-CX	0.010	0.020	0.020	0.030	0.66	0.210	0.320	0.330					0.050	0.030	0.040	0.030	0.110	0.080	0.090	0.050
FSV-CY																				
FSV-DJ																				
FSV-DK																				
FSV-DL	0.015	0.016	0.032	0.039	0.46	0.134	0.247	0.226					0.058	0.041	0.037	0.045				
FSV-DS																				
FSV-DU																				
FSV-DX	*nq*	0.019	0.027	*nq*	0.46	0.184	0.308	0.353									0.099	0.067	0.072	0.059
FSV-EA	0.032	0.033	0.048	0.066	0.76	0.240	0.326	0.435					0.084	0.048	0.059	0.063				
FSV-EC																				
FSV-EK					0.35	0.110	0.180	0.200									0.130	0.090	0.090	0.810
FSV-EX																				
FSV-FD																				
n	21	22	22	21	22	22	22	22	1	1	1	1	16	16	16	16	11	11	11	11
Min	0.003	0.008	0.010	0.015	0.35	0.108	0.180	0.200					0.023	0.020	0.024	0.022	0.040	0.022	0.026	0.016
Mean	0.019	0.021	0.033	0.043	0.57	0.185	0.270	0.311	0.35	0.10	0.17	0.18	0.079	0.050	0.060	0.058	0.090	0.067	0.068	0.111
Max	0.042	0.035	0.059	0.076	0.87	0.293	0.389	0.435					0.164	0.133	0.143	0.138	0.135	0.129	0.102	0.810
SD	0.009	0.008	0.013	0.018	0.13	0.045	0.050	0.064					0.036	0.031	0.032	0.031	0.028	0.027	0.021	0.232
CV	50	38	41	42	23	24	19	21					46	61	53	53	32	40	30	210
NISTa	0.024	0.019	0.037	0.039																
NISTb		0.025	0.043	0.062		0.235	0.359	0.406		0.09	0.15	0.16		0.024	0.032	0.033		0.058	0.063	0.043
Median	0.017	0.020	0.031	0.039	0.57	0.179	0.268	0.314					0.071	0.041	0.053	0.049	0.090	0.064	0.064	0.036
eCV	44	27	37	49	23	19	17	20					27	40	32	32	33	23	19	58

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Round Robin XXXII Laboratory Results
Results in ug/mL

Lab	Total Zeaxanthin 199	200	201	202	Total Lutein&Zeaxanthin 199	200	201	202
FSV-BA					0.126	0.101	0.100	0.054
FSV-BD								
FSV-BE								
FSV-BF								
FSV-BH	0.025	*nq*	*nq*	*nq*	0.095			
FSV-BI	0.030	0.015	0.018	0.010	0.102	0.071	0.080	0.045
FSV-BJ								
FSV-BK								
FSV-BL								
FSV-BM								
FSV-BN	0.027	0.014	0.070	0.004	*0.067*	*0.036*	*0.096*	*0.020*
FSV-BO					0.181	0.115	0.147	0.087
FSV-BP								
FSV-BQ								
FSV-BT	0.025	0.018	0.027	0.013	0.220	0.129	0.164	0.114
FSV-BX	0.043	0.023	0.029	0.015	*0.116*	*0.075*	*0.085*	*0.046*
FSV-BY	0.038	0.022	0.023	0.031	*0.134*	*0.089*	*0.085*	*0.065*
FSV-BZ								
FSV-CA								
FSV-CB					0.155	0.113	0.125	0.078
FSV-CH								
FSV-CK					0.119	0.090	0.094	0.058
FSV-CL					0.127	0.097	0.112	0.064
FSV-CM								
FSV-CO	0.087	0.059	0.056	0.041	*0.222*	*0.187*	*0.158*	*0.098*
FSV-CQ								
FSV-CR								
FSV-CT								
FSV-CU								
FSV-CV								
FSV-CX								
FSV-CY								
FSV-DJ								
FSV-DK								
FSV-DL					0.097	0.078	0.097	0.045
FSV-DS								
FSV-DU								
FSV-DX								
FSV-EA								
FSV-EC								
FSV-EK								
FSV-EX								
FSV-FD								
n	7	6	6	6	13	12	12	12
Min	0.025	0.014	0.018	0.004	0.067	0.036	0.080	0.020
Mean	0.039	0.025	0.037	0.019	0.135	0.098	0.112	0.065
Max	0.087	0.059	0.070	0.041	0.222	0.187	0.164	0.114
SD	0.022	0.017	0.021	0.014	0.047	0.037	0.030	0.026
CV	56	67	56	74	35	38	26	41
NISTa								
NISTb		0.031	0.042	0.032		*0.089*	*0.105*	*0.075*
Median	0.030	0.020	0.028	0.014	0.126	0.094	0.099	0.061
eCV	25	28	40	74	34	31	20	39

Analytes Reported By One Laboratory Only
Values in µg/mL

Analyte	Code	199	200	201	202
13-cis-β-Carotene	FSV-BN	0.04	0.02	0.03	0.07
Total Carotenoids	FSV-BT	1.45	0.69	1.21	1.80
trans-α-Carotene	NISTb		0.02	0.03	0.04

Term	Definition
n	Number quantitative values reported
Min	Minimum value reported
Mean	Mean of values reported
Max	Maximum value reported
SD	Standard deviation of values reported
CV	Coefficient of variation, 100*SD/Mean
Median	Median of values reported
eCV	Robust estimate of CV, 100*eSD/Median
eSD	Robust estimate of standard deviation
nq	Not quantitatively determined, i.e., reported as "0", 'trace', 'not determined, etc.
<x	Concentration at or below the limit of quantification (LOQ) x.
italics	Calculated from reported values

The original report estimated eSD and eCV from the interquartile range (IQR).

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Round Robin XXXII Laboratory Report

Interlaboratory Measurement Comparability Summary

Lab	R	aT	gT	bC	tbC
FSV-BA	1	1	2	2	2
FSV-BD	1	2			
FSV-BE	2	2	1	1	
FSV-BG	1	1	1	2	
FSV-BH	3	1	2	1	1
FSV-BI	1	1	1	1	
FSV-BJ	1	1	2	1	
FSV-BK	1	2			
FSV-BM	4	4			
FSV-BN	1	2			
FSV-BO	1	4	2	2	3
FSV-BP	3	3		3	
FSV-BQ	3	2		2	
FSV-BR	3	4		3	
FSV-BS	2	1	1	2	1
FSV-BT				1	1
FSV-BU	2	1	1	2	
FSV-BV		2	2	2	
FSV-BX	2	1			
FSV-BY	4	1		1	
FSV-CA	1	2	2	1	
FSV-CB	3	2	1	2	
FSV-CD	3	4		4	
FSV-CH		3			
FSV-CJ	2	2		1	1
FSV-CK	2	4		4	
FSV-CL	1	1			
FSV-CM	1	1	2	1	
FSV-CN	1	2		2	2
FSV-CP	3	1	1	1	
FSV-CR	2	1	2	3	
FSV-CT	1	2		2	
FSV-CU	1	2			
FSV-CV	3	3			
FSV-CX	2	2	4	2	
FSV-CY	4	4		4	
FSV-DA	4	3		2	3
FSV-DB	2	3		1	
FSV-DJ	3	2	2	4	
FSV-DK	1	1		1	
FSV-DL	3	4	3	2	
FSV-DM	2	1		2	
FSV-DP	1	1		1	

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

where:	
StV	Standardized Value, the distance in standard deviation units your value is from the "true" concentration: StV = (your value - Median) / eSD
Median	Median of values reported
eSD	Robust estimate of standard deviation
$t(1\text{-}\alpha,n\text{-}1)$	Two-tailed Student's t for coverage of ±1, ±2, and ±3 eSD about Median, assuming a normal population of size n

K6

Appendix H. Representative "Individualized Report" for RR32

Each participant in RR32 received an "Individualized Report" reflecting their reported results. Each report included a detailed analysis of the results they reported for the following analytes:

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

The following eight pages are the "Individualized Report" for the analytes evaluated by participant FSV-NIST1 (listed as FSV-NISTa in the "All Lab Report").

Individualized Round Robin XXXII Report to: NIST1

Your Data, Inter-laboratory Medians, and %Differences

	Serum 199				Serum 200				Serum 201				Serum 202			
Analyte	You	Med	%Δ	n	You	Med	%Δ	n	You	Med	%Δ	n	You	Med	%Δ	n
Retinol	1.08	1.06	2	37	0.30	0.30	1	37	0.54	0.51	5	37	0.92	0.87	6	37
a-Tocopherol	6.90	7.22	-4	39	6.82	7.14	-4	39	9.39	9.98	-6	39	17.17	17.70	-3	39
g-Tocopherol	2.11	2.50	-15	17	1.43	1.71	-16	17	2.04	2.41	-15	17	3.25	3.80	-14	17
Total b-Carotene	.520	.588	-12	30	.240	.245	-2	30	.624	.612	2	30	1.050	1.219	-14	30
trans-b-Carotene	.448	.535	-16	8	.209	.232	-10	8	.558	.570	-2	8	.978	1.117	-12	8
Total a-Carotene	.024	.018	37	20	.019	.020	-5	21	.037	.032	16	21	.039	.041	-5	20

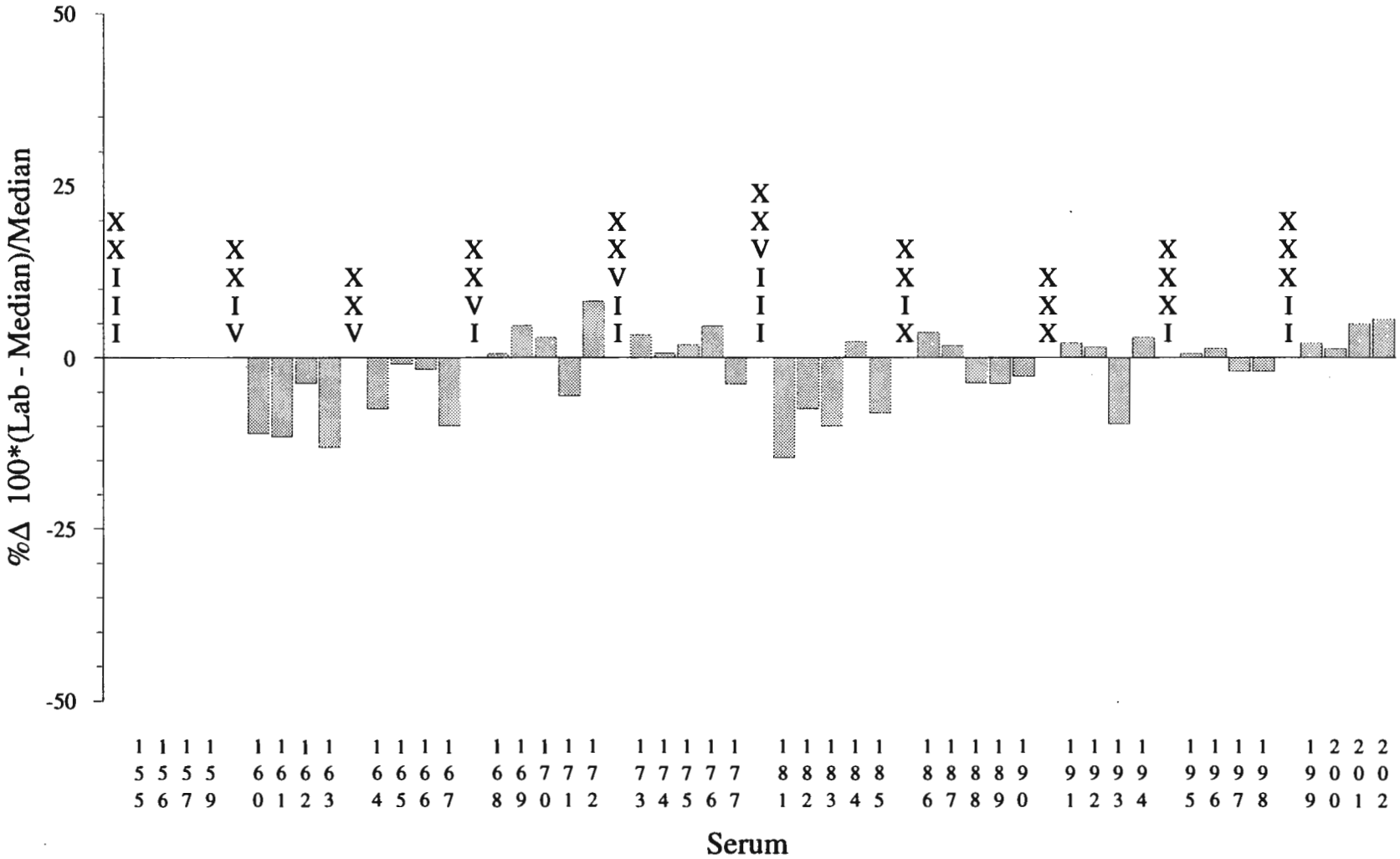
L2

You : Your reported values for the listed analytes (micrograms/milliliter)
Med : The median of the values reported by all participating laboratories (excluding NIST.)
%Δ : The percent difference between your value and the median (reported for main analytes only.)
n : The number of laboratories reporting quantitative values for this analyte in this serum (excluding NIST.)

Please check our recorded values against your records.
Fax any corrections to: (301) 926-8671, c/o Donna Sirk, 222/B156, NIST, Gaithersburg, MD 20899

Individualized Round Robin XXXII Report to: NIST1

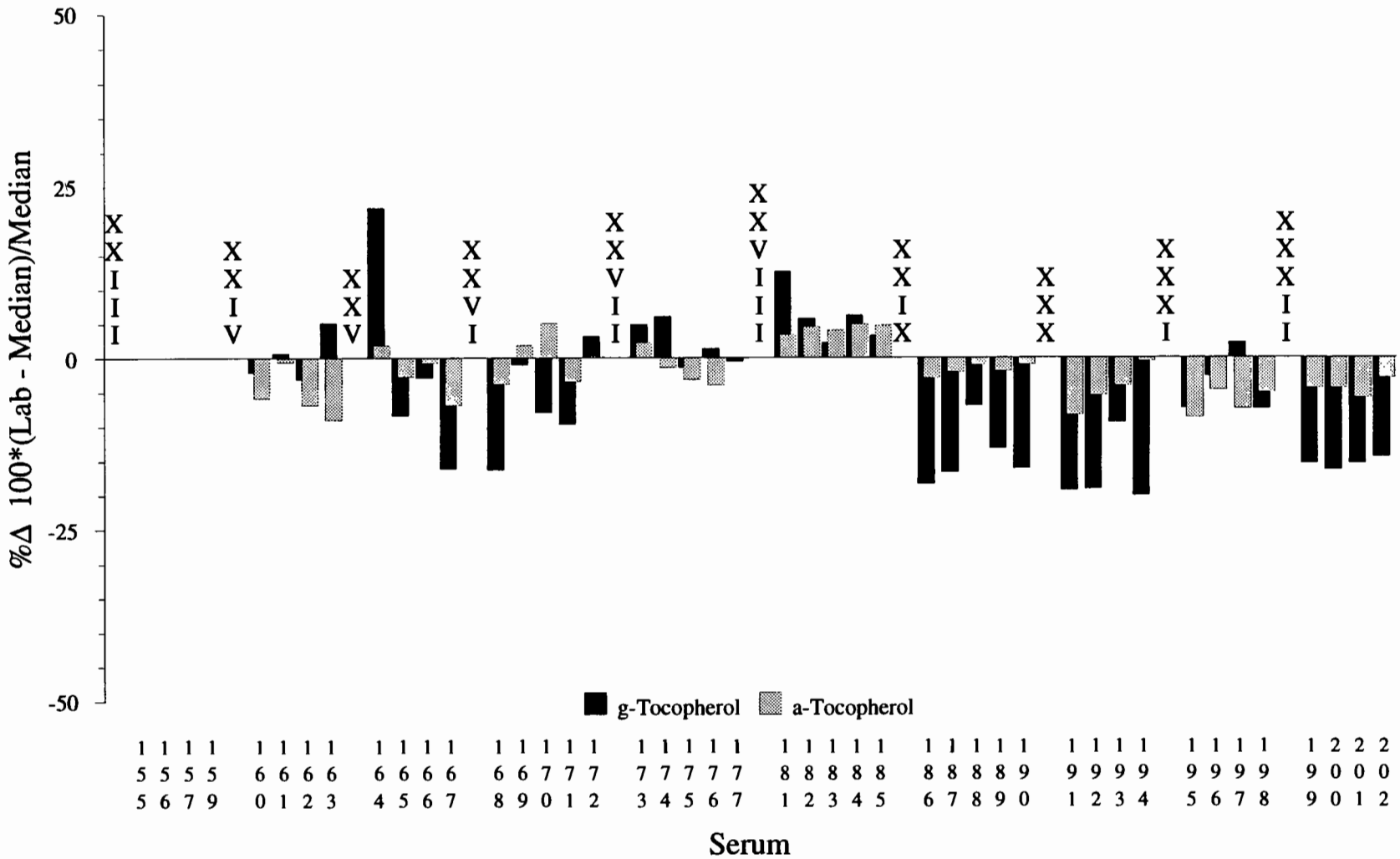
Retinol, %Δ in Round Robin XXIII - XXXII



L3

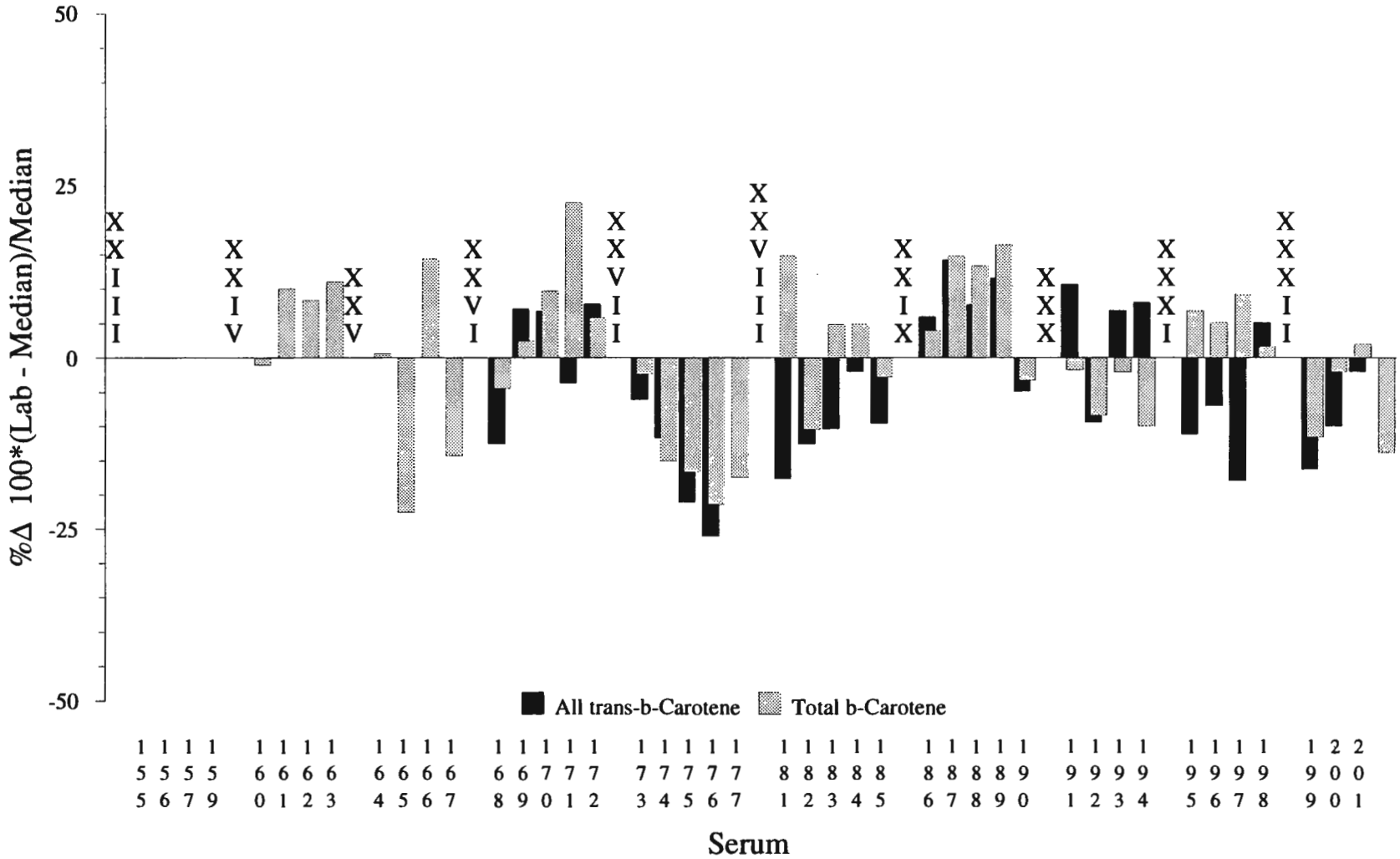
Individualized Round Robin XXXII Report to: NIST1

Tocopherol, %Δ in Round Robin XXIII - XXXII



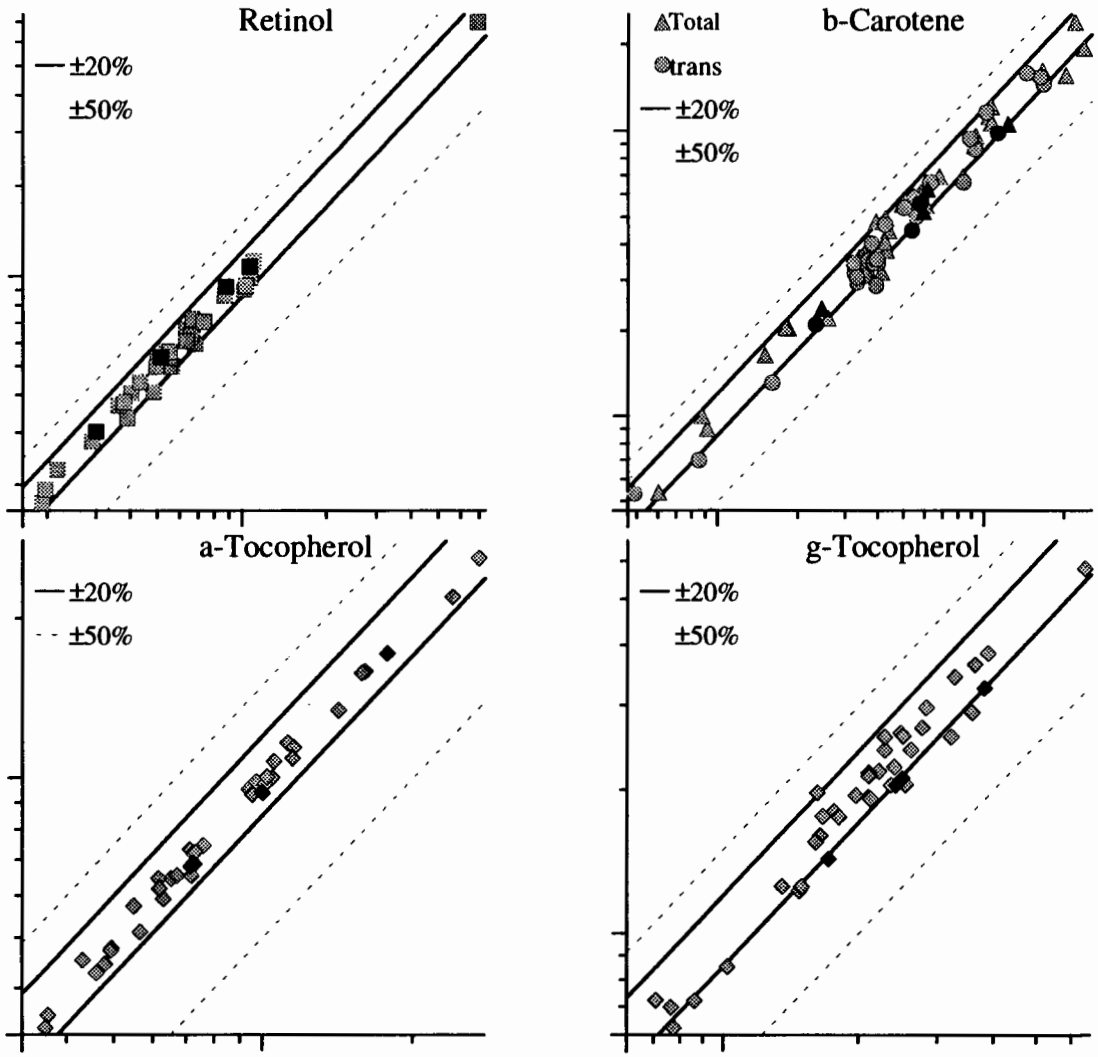
Individualized Round Robin XXXII Report to: NIST1

b-Carotene, %Δ in Round Robin XXIII - XXXII



Individualized Round Robin XXXII Report to: NIST1

Inter-laboratory Median Vs Laboratory NIST1 Values



Legend

Shaded Symbols: Round Robin XXIII-XXXI
Black Symbols: Round Robin XXXII

Ordinate: Median analyte concentration
Abscissa: Your reported concentration

Interpretation

Adequately intercomparible data are within the ±20% lines. If you have data scattered outside the ±50% lines, your measurement system is not consistent with that of most participating laboratories. If your data are systematically higher or lower than the median, 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 XXXII Report to: NIST1

(Provisional) Performance Summary

	Ret		aToc		gToc		Total		trans	
	mΔ	vΔ	mΔ	vΔ	mΔ	vΔ	mΔ	vΔ	mΔ	vΔ
XXIII										
XXIV	-10	4	-6	4	0	4	7	6		
XXV	-5	4	-2	4	-1	16	-5	16		
XXVI	2	5	0	4	-6	8	7	10	1	9
XXVII	1	3	-1	3	2	3	-15	7	-16	9
XXVIII	-8	6	4	1	6	4	2	10	-10	6
XXIX	-1	3	-2	1	-14	4	9	8	7	7
XXX	-1	6	-5	3	-17	5	-6	4	4	9
XXXI	0	2	-6	2	-4	5	6	3	-8	10
XXXII	4	2	-4	1	-15	1	-6	8	-10	6

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 - median) / median
vΔ: Difference variability, one standard deviation of %Δ for all sera of a RR

(Provisional) Performance Criteria

In past Round Robins, the absolute value of the %Δ of each measurement was evaluated
0-5%: Exceptional
6-10%: Acceptable
11-20%: Marginal
> 20%: Poor

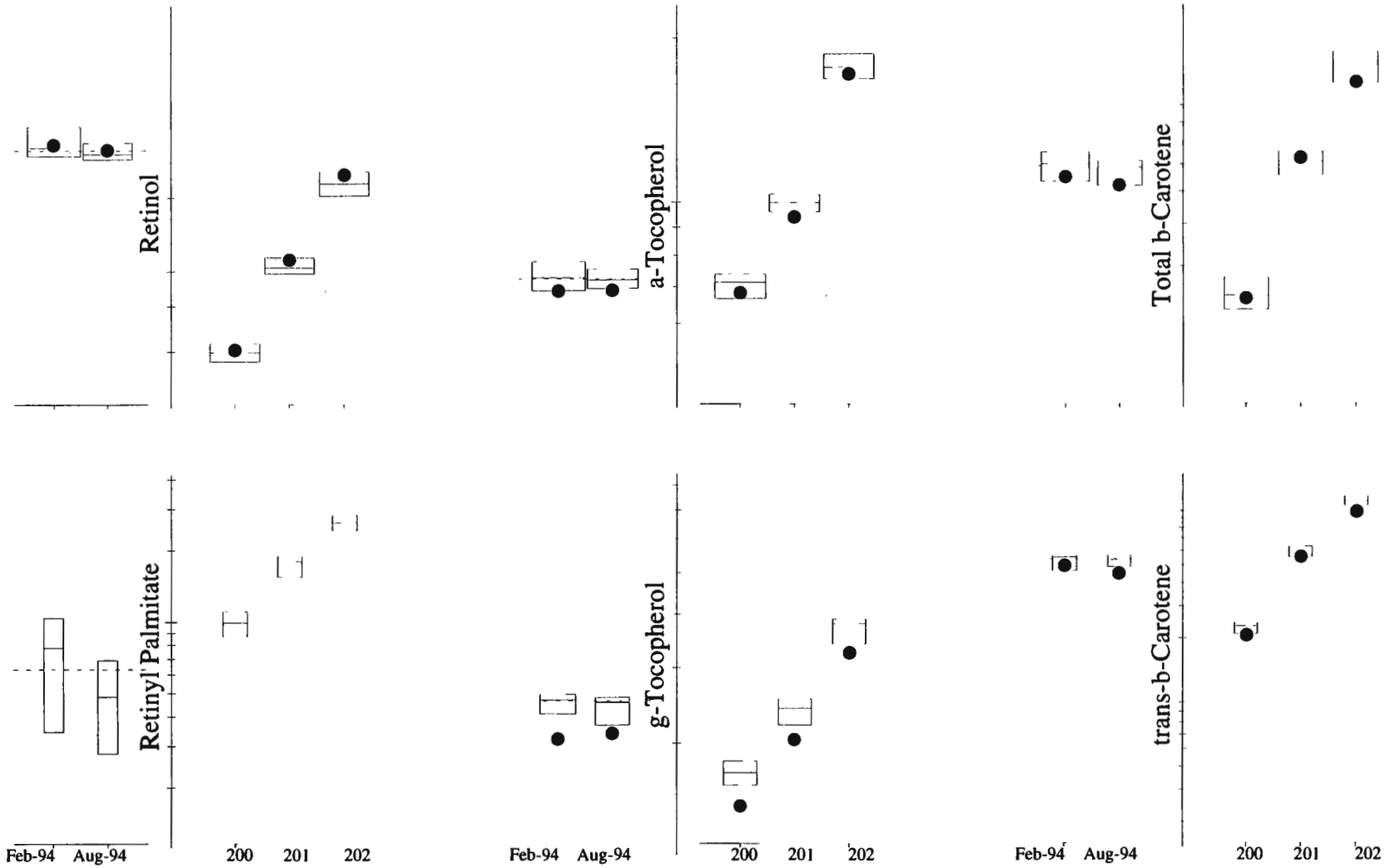
Rational

There are no definitive methods for fat-soluble vitamin-related analytes in serum. It's difficult to judge your absolute performance. However, you can compare your measurements to the group's consensus values - and to ours here at NIST. There are two kindred aspects to this relative comparison: accuracy and precision. Accuracy can be estimated as average %Δ [100(your value - median)/median] over all sera of a given Round Robin. Precision can be estimated as one standard deviation of the %Δ over all sera of a given Round Robin.

It's best to be accurate and precise (small mΔ, small vΔ.) In some sense, good precision (small vΔ) with poor accuracy (large mΔ) is better than the converse: such values are internally consistent and may be relatable to the group's values. Poor precision (large vΔ) suggests that your measurement system is qualitatively different from that used by NIST and the majority of participating laboratories.

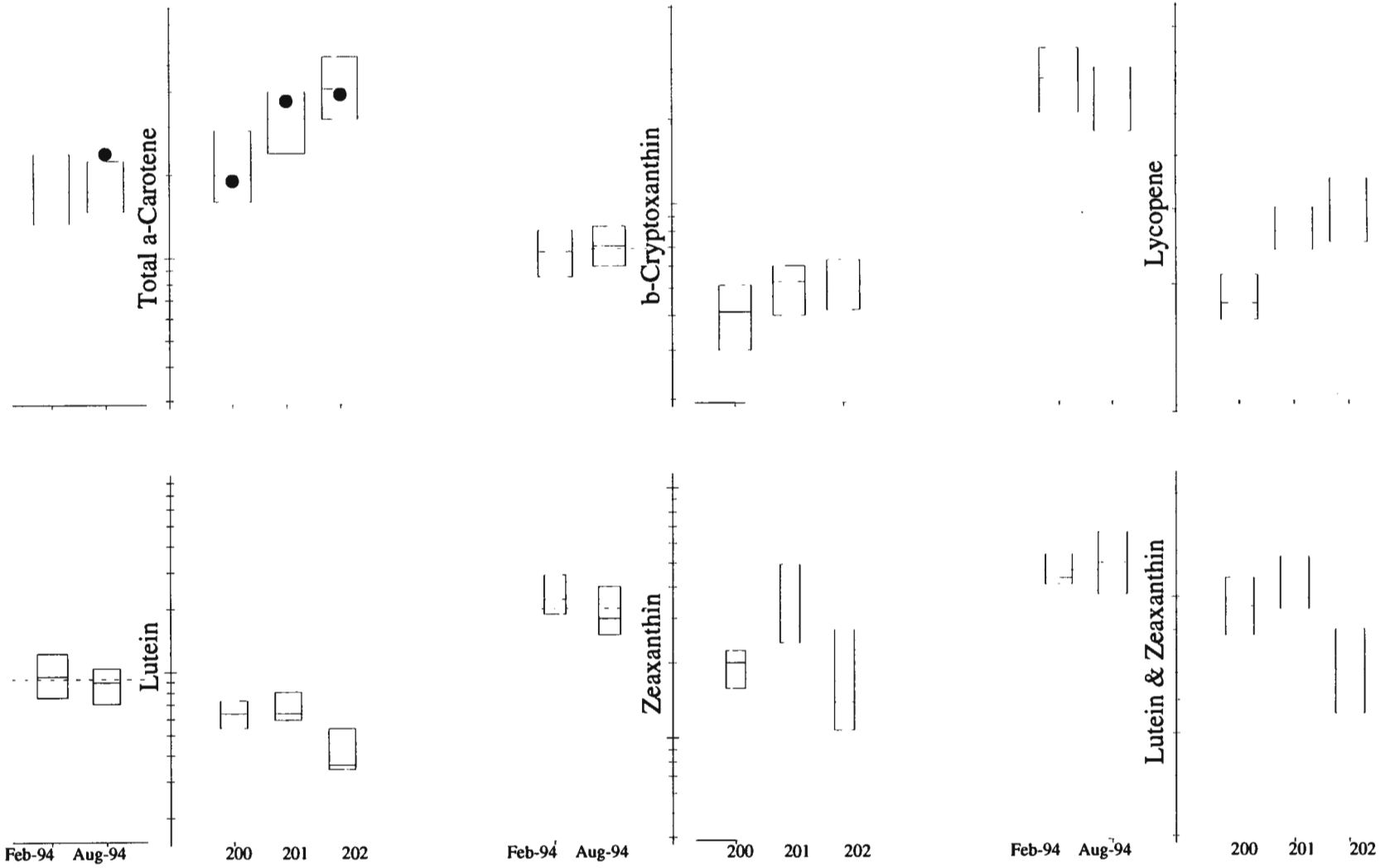
Individualized Round Robin XXXII Report to: NIST1

Comparisons to Known Relationships



Individualized Round Robin XXXII Report to: NIST1

Comparisons to Known Relationships (Continued)



Appendix M. Shipping Package Inserts for RR05

The following two items were included in each package shipped to a RR05 participant:

- Cover letter
- Report of Analysis datasheet

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

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

April 14, 1994

1~

2~

Dr. Margolis sent individual letters to invited study participants. The "~1" and "~2" were mail-merge commands for inserting a participant's name and address. This page was prepared from the original working draft.

Thank you for agreeing to measure the ascorbic acid in the accompanying samples. The samples which are in sealed ampoules were prepared by adding equal volumes of spiked human serum to 10% metaphosphoric acid (MPA). All samples have been stored at -70 °C and should be kept at this temperature. I have checked them for stability and the ascorbic acid appears sufficiently stable.

You should find four ampoules in the shipping container. Each ampoule contains between 0.2 and 0.8 mg of ascorbic acid/dL of serum and each ampoule should be analyzed in duplicate by the method(s) used in your laboratory (preferably one measuring total ascorbic acid).

The 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 I would appreciate it if you would make your measurements and return your results to me by **May 31, 1994**. 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, the number is (301) 926-8671. 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
Organic Analytical Research Division
Chemical Science and Technology Laboratory

REPORT OF ANALYSIS

NAME:

Name and mailing address were specified on sheet set to each participant

ADDRESS:

Telephone Number: _____

FAX Number: _____

RESULTS (µg/mL)

SERUM 178, VIAL# _____

REPLICATE 1 _____ µg/mL
REPLICATE 2 _____

SERUM 178, VIAL# _____
REPLICATE 1 _____ µg/mL
REPLICATE 2 _____

SERUM 179, VIAL# _____

REPLICATE 1 _____ µg/mL
REPLICATE 2 _____

SERUM 179, VIAL# _____

REPLICATE 1 _____ µg/mL
REPLICATE 2 _____

Appendix N. Final Report for RR05

The following eight pages are the final report for RR05 as provided to all participants. This report contains:

- Cover letter and analysis of results.
- Table 1 "Results of the Round Robin Measurement of Ascorbic Acid in Human Plasma".
- Table 2 "NIST Results for the Measurement of AA in Human Plasma".
- Figure 1 "Box Plot of the Round Robin Results"
- Figure 2 "Distribution of the Round Robin Results for Lot 178"
- Figure 3 "Distribution of the Round Robin Results for Lot 179"
- A page intended to facilitate the participants' return of comments

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.

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

August 1, 1994

The letter as distributed to participants has been lost. This version has been prepared from an extant draft.

Dear Colleague:

This report describes both the overall-group and your laboratory's performance in Round Robin V for the measurement of ascorbic acid in human plasma. The study involve the duplicate analysis of four samples (two each from lots 178 and 179). Specifically, your package contains tabular presentations of all data submitted for ascorbic acid for Round Robin V. Your results are designated "Data Set_#" in the tables and figures.

Table 1 provides a summary of the data submitted by sixteen Laboratories (the NIST data were not included in the statistical analysis). Two laboratories submitted three sets of measurements, each done by a different method. As shown in Table 1, the percent Relative Standard Deviation (RSD) for both lots ranged from 17.9 to 26.8. The intralaboratory %RSD ranged from 0.7 to 8.5 except for one measurement with a %RSD of 15.7 indicating that the major source of variation was the interlaboratory variation. The box plot in Figure 1 is a graphic summary of the results. The highest and lowest 10 % of the measurements for each lot are plotted as small solid circles, the two simple lines each span the next 15 % intervals and the center box contains the values from ten data sets. The NIST mean value for ascorbic acid (AA) + dehydro-AA are represented by a circle with a T inside and the NIST mean for reduced AA is represented by an R inside a circle. The horizontal line in the 50% boxes represents the median interlaboratory values which are slightly below the NIST values and the X represents the mean which is essentially identical to the NIST values. Finaly, the results of the previous round robin for lot 178 are compared to the results of this round robin and show a similar distribution of results.

Figures 2 and 3 are plots of the distribution of the data points submitted by each laboratory for each sample lot. From these plots and data in Table 1, it is apparent that the results obtained with the dinitrophenylhydrazine (DNPH) method (data sets 2 to 4), except for data set 3 lot 179, fall into a group of values which are higher than the NIST mean by approximately 10 to 25 %. Two sets of LC data (sets 6 and 7) which were high were reported by the same laboratory that made measurements by the DNPH method. The majority of the sets of data obtained by LC methods are 5 to 15 % below the NIST mean. The high average mean of the DNPH measurements is not significant because of the wide distribution of values but it is consistent with previous round robins and suggest that the DNPH method may be positively biased. The basis of the distribution of the majority of the LC measurements below the NIST mean requires further evaluation along with the large variation in the results (%RSD = 18 to 26 %). The observation that all of the gravimetrically added AA was accounted for (Table 2) also supports the need to continue these round robins in order to reduce the measurement variation.

If you have any questions concerning Round Robin V please indicate it on the enclose sheet and return it to us via mail (Donna Sirk, NIST, Chemistry B156, Gaithersburg, MD 20899) or FAX (301-926-8671), or contact me at 301-975-3137.

The next set of samples, Round Robin VI, will be shipped around the August 15.

Sincerely,

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

Enclosures

Table 1. Results of the Round Robin Measurement of Ascorbic Acid in Human Plasma.

Lab	Data Set[b]	Method	Ascorbic Acid (mmol/L Plasma)[a] Lot 178	Lot 179
	2	DNPH	39.8 ± 0.7	53.8 ± 0.7
	6	LC	33.2 ± 2.8	55.4 ± 1.2
	7	LC	35.2 ± 1.6	58.8 ± 2.8
	12	LC-EC	20.1 ± 2.2	46.0 ± 7.2
	3	DNPH	33.5 ± 8.1	43.2 ± 0
	5	ENZ	26.7 ± 2.0	50.3 ± 3.6
	4	DNPH	38.5 ± 0.3	56.2 ± 1.0
	1	DCIP	30.5 ± 2.0	45.4 ± 0.7
	13	LC-EC	19.5 ± 2.9	43.9 ± 3.6
	14	LC-EC	28.4 ± 0	45.4 ± 0
	15	LC-EC	22.6 ± 2.0	45.1 ± 2.9
	11	LC		
	8	LC-EC+d	20.1 ± 0.8	32.9 ± 0.4
	9	LC-EC+p	18.3 ± 0.9	36.2 ± 0.3
	10	LC-EC-p	14.9 ± 0.6	31.8 ± 0.3
	18	LC	22.4 ± 0.3	39.8 ± 0
	16	LC-EC	21.8 ± 0.2	40.4 ±0.4
	19		23.0 ± 1.3	38.0 ±1.3
	20		30.9 ± 0.4	36.9 ± 3.2
	21		21.9 ± 0.3	40.3 ± 0
	17	LC-EC	22.8 ± 1.9	41.1 ± 0.6
MEAN			26.6	44.1
SD			7.1	7.9
%RSD			28.8	17.9
NIST AA + DHAA		LC-EC	27.0 ± 0.5[A]	43.5 ± 1.5[d]
NIST AA		LC-EC	10.2 ± 0.4[A]	12.1 ± 3.9[d]

[a] Values represent the mean and SD of replicate measurements on two samples (total of 4 measurements).
[b] The Data Set numbers correspond to those in the figures 2-4.
[c] Values represent the mean and SD of replicate measurements on five samples (total of 10 measurements).

Table 2. NIST Results for the Measurement of AA in Human Plasma.

Lot No.	Ascorbic Acid (mmol/L) without DTT (AA)	with DTT (AA+DHAA)	Supplemented Amount
Plasma 1	5.1 ± 1.1 (4)[a]	11.5 ± 0.2 (4)	
178	10.2 ± 0.4 (4)	27.0 ± 0.5 (4)	15.8
179	12.1 ± 3.9 (10)	43.5 ± 1.5 (10)	32.1
180	15.1 ± 0.4 (4)	42.7 ± 0.3 (4)	31.6
Plasma 2	18.9 ± 6.5 (4)	52.2 ± 1.6 (4)	
179B	67.2 ± 26.4 (5)	154.4 ± 1.1 (10)	101.3

[a] The value in parentheses is the number of measurements made.

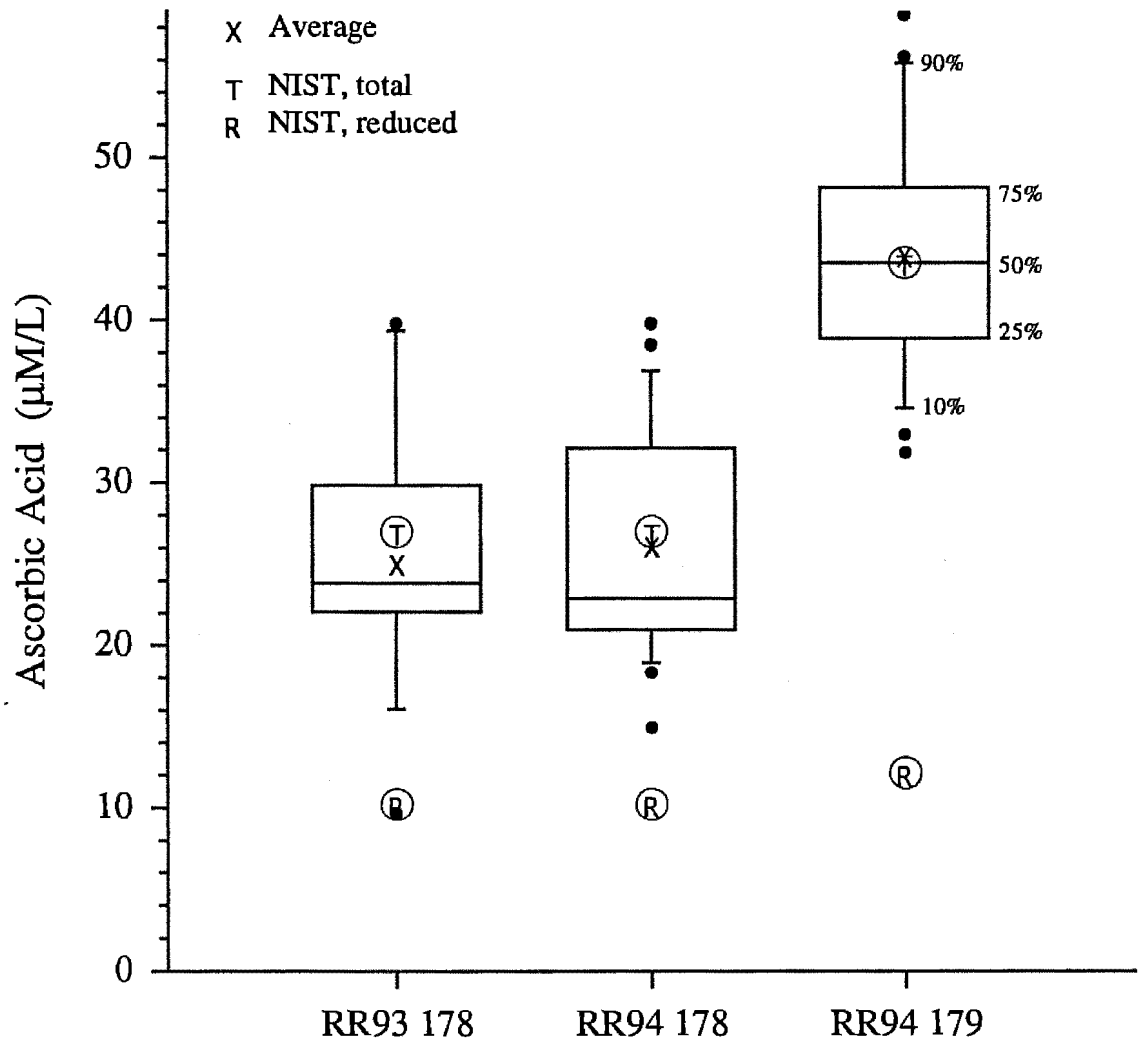
Figure 1. Box Plot of the Round Robin Results

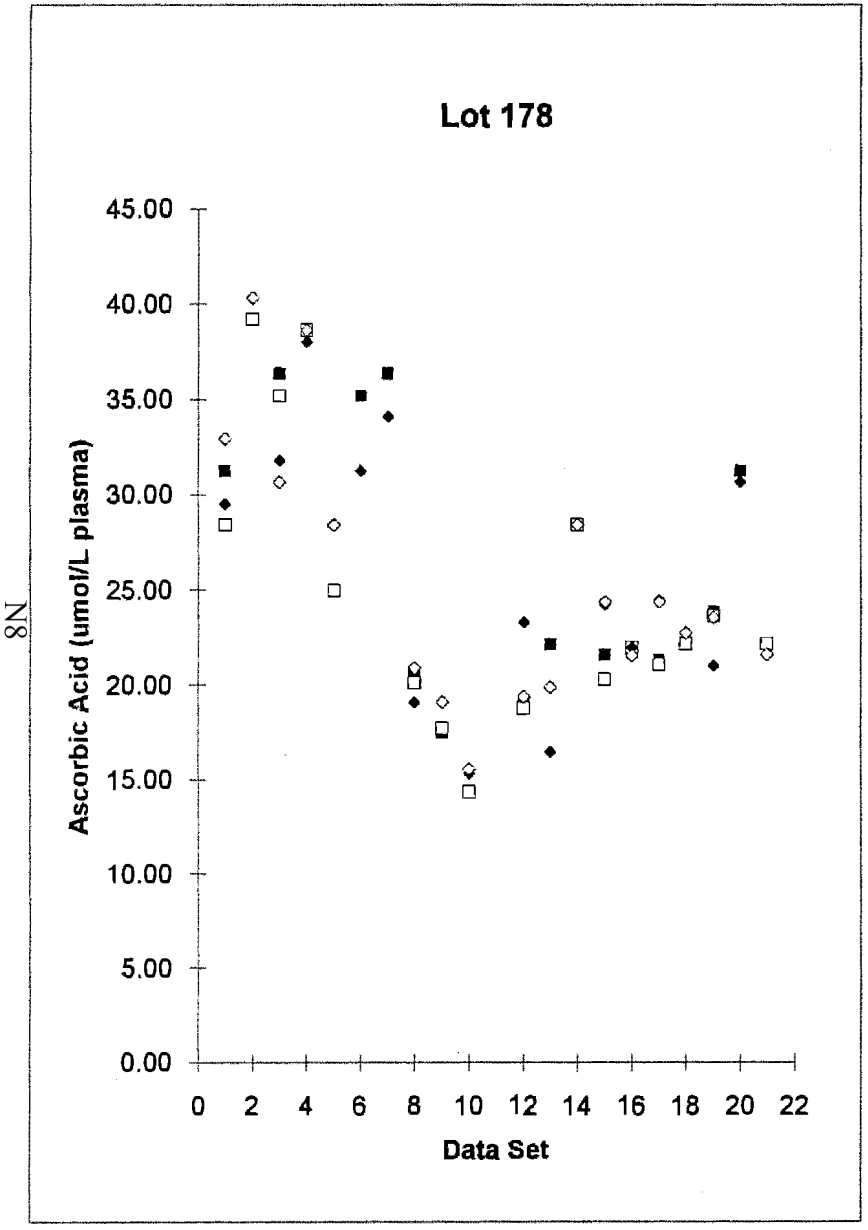
Figure 2. Distribution of Round Robin Results for Lot 178

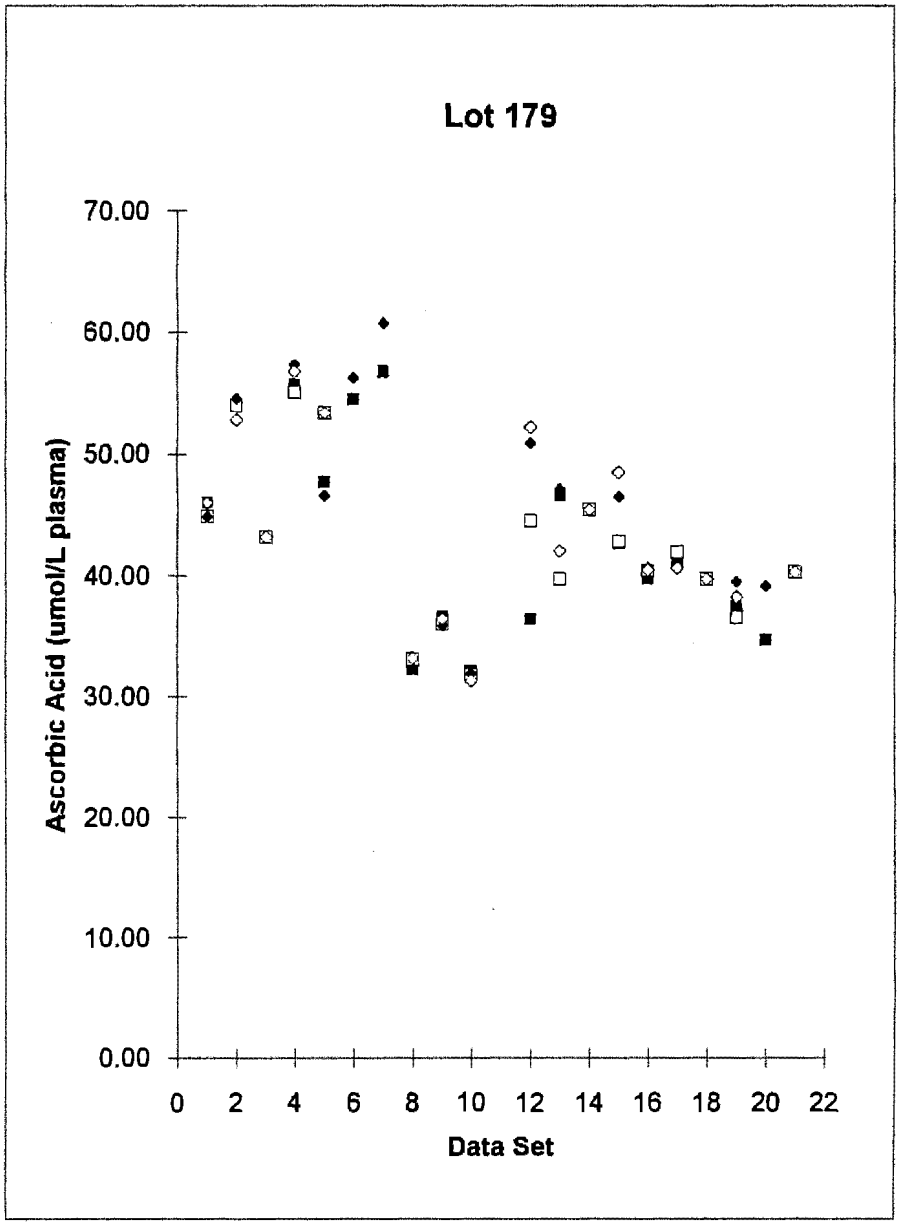
Figure 3. Distribution of Round Robin Results for Lot 179

Key for Figures 2 and 3:

■ First Vial; First Measurement
♦ Second Vial; First Measurement
□ First Vial: Second Measurement
◊ Second Vial;Second Measurement


X Average
T NIST, total
R NIST, reduced
90%
75%
50%
25%
10%
Ascorbic Acid (µM/L)
0
10
20
30
40
50
RR93 178
RR94 178
RR94 179


Lot 178
Ascorbic Acid (umol/L plasma)
45.00
40.00
35.00
30.00
25.00
20.00
15.00
10.00
5.00
0.00
0 2 4 6 8 10 12 14 16 18 20 22
Data Set


Lot 179
Ascorbic Acid (umol/L plasma)
70.00
60.00
50.00
40.00
30.00
20.00
10.00
0.00
0 2 4 6 8 10 12 14 16 18 20 22
Data Set

N8

NCI QUALITY ASSURANCE PROGRAM

title
company
address

Appendix O. "All-Lab Report" for VC-RR05

The following two pages a revised the "All-Lab Report" for RR05. This report 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 have been corrected and transformed to have units of µmol/L sample.
- additional summary statistics have been included.
- a Legend page has been added

Vitamin C Round Robin 5

			178, [TAA] µmol/L					179, [TAA] µmol/L				
Lab	Date	Method	Mean	S_{dup}	S_{rep}	S_{het}	S_{tot}	Mean	S_{dup}	S_{rep}	S_{het}	S_{tot}
VC-MA	30-Oct-94	HPLC-EC	10.79	0.00	0.00	0.00	0.00	18.10	0.30	0.43	0.00	0.37
VC-MB	13-May-94	AO	13.63	0.80	0.57	0.70	0.70	25.12	0.20	2.22	0.00	1.57
VC-MC	16-May-94	24DNPH	19.87	0.40	0.00	0.40	0.28	26.90	0.10	0.43	0.00	0.31
VC-MD	16-May-94	HPLC-EC	17.10	1.10	0.76	0.96	0.95	28.53	1.20	1.08	0.93	1.14
VC-ME	1-Jun-94	HPLC-EC	10.93	0.20	0.00	0.20	0.14	20.16	0.00	0.00	0.00	0.00
VC-ML	31-May-94	HPLC-UV	15.47	0.20			0.14	18.45	1.61			1.14
VC-MV	10-Jun-94	HPLC-EC	14.19	0.00	0.00	0.00	0.00	22.71	0.00	0.00	0.00	0.00
VC-MX	24-May-94	HPLC-EC	11.21	0.20	0.00	0.20	0.14	19.87	0.00	0.00	0.00	0.00
VC-MZ	26-May-94	HPLC-EC	10.06	0.10	0.46	0.00	0.33	16.45	0.18	0.20	0.11	0.19
VC-NA	25-May-94	HPLC-EC	11.55	0.45	0.56	0.22	0.51	18.98	0.66	0.39	0.60	0.54
VC-NB	1-Jun-94	HPLC-EC	11.40	1.14	0.06	1.14	0.81	20.55	0.28	0.20	0.24	0.24
VC-NC	13-Jul-94	24DNPH	19.22	0.18	0.22	0.09	0.20	27.86	0.45	0.60	0.16	0.53
VC-NG	6-Jun-94	HPLC-EC	11.31	1.19	0.33	1.17	0.88	22.55	1.69	0.50	1.65	1.24
VC-NN	15-May-94	24DNPH	16.75	1.61	0.40	1.58	1.17	21.57	0.00	0.00	0.00	0.00
VC-NP	13-May-94	HPLC-EC	10.88	0.03	0.11	0.00	0.08	20.18	0.17	0.17	0.12	0.17
		N	15					15				
		Min	10.06	0.00	0.00	0.00	0.00	16.45	0.00	0.00	0.00	0.00
		Median	11.55	0.20	0.17	0.21	0.28	20.55	0.20	0.29	0.06	0.31
		Max	19.87	1.61	0.76	1.58	1.17	28.53	1.69	2.22	1.65	1.57
		eSD	2.20					3.11				
		eCV	19.0					15.2				

O2

Datasets

Each participant typically reported two replicate measurements for each of two duplicate vials for each test sample. However, occasionally only one vial of each pair was evaluated or a single result was reported for each of the duplicate vials.

Legend

Lab	Laboratory Code
Date	Date that the results were received at NIST
Method	Type of assay
	AO — Ascorbate oxidase
	24DNPH — 2,4-Dinitrophenylhydrazine
	EC — Electrochemical detector
	HPLC — Liquid chromatography
	OPD — Orthophenlyenediamine
	UV — Ultraviolet absorbance
Mean	Mean of duplicate means
S_{dup}	Standard deviation of duplicate means
S_{rep}	Pooled standard deviation of replicates
S_{het}	Estimated sample heterogeneity, $\sqrt{\mathrm{MAX}\left(0, S_{dup}^2 - S_{het}^2\right)}$
S_{tot}	Estimated standard deviation of the mean, $\sqrt{\left(S_{dup}^2 + S_{rep}^2\right)/n}$, where n is the number of vials evaluated and is typically 2.
N	The number of participants
Min	Minimum value in the column
Median	Median value in the column
Max	Maximum value in the column
eSD	Adjusted median absolute deviation from the median (MADe)
eCV	Estimated coefficient of variation, 100*eSD/Median

Appendix P. Shipping Package Inserts for RR06

The following two items were included in each package shipped to a RR06 participant:

- Cover letter
- Report of Analysis datasheet

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

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

August 22, 1994

Dr. Margolis sent individual letters to invited study participants. The name and greeting for a participant have been redacted from this image.

Dear

Thank you for agreeing to measure the ascorbic acid in the accompanying samples. Enclosed are two sets of samples; one set consisting of four serum bottles are controls and the second set consisting of four ampules are Test Samples.

The control materials consist of two serum bottles of sample 107 (0.48 mg ascorbic acid/dL) and two of sample 108 (1.18 mg ascorbic acid/dL). These samples have been lyophilized and contain dithiothreitol (DTT) (1 mg/mL). Vials should be reconstituted with one mL of water. Sample volumes after reconstitution will therefore be somewhat less than 1.1 mL.

We ask that you use these control materials to test your method before you analyze the round robin (RR) samples. We request that the following testing pattern be used.

- Analyze single vials of samples 107 and 108. If these results are within 10% of the assigned values, proceed with the analysis of the unknown samples.
- If either of your controls are biased by 10-30%, we suggest that you review your method, make adjustments as necessary, then analyze the second pair of controls.
- If your results for either of the controls exceed 30%, we ask that you thoroughly examine your procedures and make necessary adjustments, and then analyze the second set of Controls. If your data remains significantly biased, feel free to contact us to discuss possible solutions to your measurement problems.

Please note that the control materials contain the reducing agent DTT which may interfere with the your method of analysis if you are using a colorimetric method or if your HPLC method does not resolve DTT and ascorbic acid. If this is the case just analyze the samples in the sealed ampules as described below.

The samples that are in sealed ampules were prepared by adding equal volumes of spiked human serum to 10% metaphosphoric acid (MPA). 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 0.1 and 1.5 mg of ascorbic acid/dL of sample 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 results to me by **September 30, 1994**. 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 number is (301)926-8671. 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
Organic Analytical Research Division
Chemical Science and Technology Laboratory

Enclosures

REPORT OF ANALYSIS

Name and mailing address specified on sheet set to each participant

LAB NO.: *Assigned code number*

PHONE NO.:

FAX NO.:

Method of Analysis:

Date of Analysis:

RESULTS (µg/dL)

SERUM 179, VIAL# _____

REPLICATE 1 _____ mg/dL
REPLICATE 2 _____ mg/dL

SERUM 179, VIAL# _____

REPLICATE 1 _____ mg/dL
REPLICATE 2 _____ mg/dL

SERUM 180, VIAL# _____

REPLICATE 1 _____ mg/dL
REPLICATE 2 _____ mg/dL

SERUM 180, VIAL# _____

REPLICATE 1 _____ mg/dL
REPLICATE 2 _____ mg/dL

Appendix Q. "All-Lab Report" for VC-RR06

The following two pages a revised the "All-Lab Report" for RR06. This report 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 have been corrected and transformed to have units of µmol/L sample.
- additional summary statistics have been included.
- a Legend page has been added

Vitamin C Round Robin 6

			179, [TAA] µmol/L					180, [TAA] µmol/L				
Lab	Date	Method	Mean	S_{dup}	S_{rep}	S_{het}	S_{tot}	Mean	S_{dup}	S_{rep}	S_{het}	S_{tot}
VC-MA	25-May-95	HPLC-EC	19.66	0.10	0.14	0.00	0.12	19.16	0.20	0.40	0.00	0.32
VC-MB	19-Sep-94	AO	20.44	0.80	2.34	0.00	1.75	20.30	0.60	0.63	0.40	0.62
VC-MC	22-Sep-94	24DNPH	27.39	0.20	0.63	0.00	0.47	26.40	0.80	0.40	0.75	0.63
VC-MD	26-Sep-94	HPLC-EC	24.27	0.60	1.02	0.00	0.84	23.70	1.00	0.63	0.90	0.84
VC-ME	22-Aug-94	HPLC-EC	21.08	0.10	0.14	0.00	0.12	20.16	0.60	0.45	0.51	0.53
VC-MF	20-Sep-94	AutoAnal	20.72	0.40	0.28	0.35	0.35	19.59	1.00	0.45	0.95	0.78
VC-ML	23-Sep-94	HPLC-UV	29.04		2.37		2.37	19.99	1.53	0.80	1.42	1.22
VC-MV	23-Sep-94	HPLC-EC	19.80	0.30	0.71	0.00	0.55	19.94	0.70	0.76	0.45	0.73
VC-MX	28-Sep-94	HPLC-EC	19.59	0.80	0.00	0.80	0.57	18.81	0.50	0.14	0.49	0.37
VC-NA	28-Sep-94	HPLC-EC	14.07	0.21	0.49	0.00	0.38	13.84	0.38	0.54	0.00	0.47
VC-NG	17-Oct-94	HPLC-EC	22.78	0.30	0.14	0.28	0.24	22.00	0.00	0.20	0.00	0.14
		N	11					11				
		Min	14.07	0.10	0.00	0.00	0.12	13.84	0.00	0.14	0.00	0.14
		Median	20.72	0.30	0.49	0.00	0.47	19.99	0.60	0.45	0.49	0.62
		Max	29.04	0.80	2.37	0.80	2.37	26.40	1.53	0.80	1.42	1.22
		eSD	1.68					1.22				
		eCV	8.1					6.1				

Q2

Datasets

Each participant typically reported two replicate measurements for each of two duplicate vials for each test sample. However, occasionally only one vial of each pair was evaluated or a single result was reported for each of the duplicate vials.

Legend

Lab	Laboratory Code
Date	Date that the results were received at NIST
Method	Type of assay
	AO — Ascorbate oxidase
	24DNPH — 2,4-Dinitrophenylhydrazine
	EC — Electrochemical detector
	HPLC — Liquid chromatography
	OPD — Orthophenlyenediamine
	UV — Ultraviolet absorbance
Mean	Mean of duplicate means
S_{dup}	Standard deviation of duplicate means
S_{rep}	Pooled standard deviation of replicates
S_{het}	Estimated sample heterogeneity, $\sqrt{\mathrm{MAX}\left(0, S_{dup}^2 - S_{het}^2\right)}$
S_{tot}	Estimated standard deviation of the mean, $\sqrt{\left(S_{dup}^2 + S_{rep}^2\right)/n}$, where n is the number of vials evaluated and is typically 2.
N	The number of participants
Min	Minimum value in the column
Median	Median value in the column
Max	Maximum value in the column
eSD	Adjusted median absolute deviation from the median (MADe)
eCV	Estimated coefficient of variation, 100*eSD/Median