

NISTIR 7880-32

**NIST Micronutrients Measurement
Quality Assurance Program
Winter, Spring, and Fall 1992
Comparability Studies**

Results for Round Robins XXIV, XXV, and XXVI
Fat-Soluble Vitamins and Carotenoids in Human Serum
and Round Robin 3 Ascorbic Acid in Human Serum

Neal E. Craft (Former Employee)
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December, 2014



U.S. Department of Commerce
Penny Pritzker, Secretary

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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 1992 MMQAP measurement comparability improvement studies: 1) Round Robin XXIV Fat-Soluble Vitamins and Carotenoids in Human Serum, 2) Round Robin XXV Fat-Soluble Vitamins and Carotenoids in Human Serum, 3) Round Robin XXVI Fat-Soluble Vitamins and Carotenoids in Human Serum, and 4) Round Robin 3 Ascorbic Acid in Human Serum. The materials for Round Robin XXIV were shipped to participants in January 1992; participants were requested to provide their measurement results by March 6, 1992. The materials for Round Robin XXV were shipped to participants in May 1992; participants were requested to provide their measurement results by June 26, 1992. The materials for Round Robin XXVI were shipped to participants in August 1992; participants were requested to provide their measurement results by October 16, 1992. The sample materials for Round Robin 3 were distributed in August 1992 with results due by September 26, 1992.

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 XXIV: Fat-Soluble Vitamins and Carotenoids in Human Serum

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin XXIV comparability study (hereafter referred to as RR24) 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 1992. The communication materials included in the sample shipment are described 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 RR24 is reproduced as Appendix B. This report included:

- Our analysis of the participants' results.
- Tabular presentations of all participant results

Each participant also received an "Individualized Report" that graphs their results for selected analytes. An example "Individualized Report" is reproduced as Appendix C.

Appendix D lists all of the measurement results reported for RR24 in a more accessible format.

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

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin XXV comparability study (hereafter referred to as RR25) 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 May 1992. The communication materials included in the sample shipment are described 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 RR25 is reproduced as Appendix F. This report included:

- Our analysis of the participants' results.
- Tabular presentations of all participant results

Each participant also received an "Individualized Report" that graphs their results for selected analytes. This report included graphical summaries of the participant's results relative to the assigned values over the previous five years and the consistency of their results for blind replicate samples over the previous four years. While there is no longer an example of a RR25 Individualized Report, its design would have been similar to the RR24 example in Appendix C.

Appendix G lists all of the measurement results reported for RR25 in a more accessible format.

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

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin XXVI comparability study (hereafter referred to as RR26) received three lyophilized and two liquid-frozen human serum test samples for analysis. Unless multiple vials were previously requested, participants received one vial of each material. These sample materials were shipped on dry ice to participants in August 1992. The communication materials included in the sample shipment are described 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.

It is probable that a preliminary report for RR26 was mailed to all participants shortly before the November 14, 1992 NIST/NCI Micronutrients Analysis Workshop, however, no version of either the letter or the preliminary report is available. The "Summary of 1992 Round Robin Activities" was mailed to every participant in the 1992 program in November 1992. This summary is reproduced as Appendix I.

Each participant also received an "Individualized Report" that graphically analyzes their results for selected analytes. This report included graphical summaries of the participant's results relative to the assigned values over the previous five years and the consistency of their results for blind

replicate samples over the previous four years. While there is no longer an example of a RR26 Individualized Report, its design would have been similar to the RR24 example in Appendix C. Appendix J lists all of the measurement results reported for RR26 in a more accessible format.

Round Robin 3: Vitamin C in Human Serum

Participants in the MMQAP Vitamin C in Human Serum Round Robin 3 comparability study (hereafter referred to as RR03) received two frozen serum samples of the same material. These samples were shipped on dry ice to participants in August 1992. The available communication materials included in the sample shipment are provided in Appendix K.

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.

No copy of a final report for RR03 as delivered to the participants is available. However, a NIST-internal Report of Analysis that presents and discusses the RR03 results was prepared. Appendix L presents the relevant content from this document.

While not distributed to the participants in RR03, Appendix O presents a revised “All-Lab Report” that lists the results for the test materials transformed into units of $\mu\text{mol/mL}$ sample.

No “Individualized Report” was provided to the participants in RR03.

References

- 1 Duewer DL, Brown Thomas J, Kline MC, MacCrehan WA, Schaffer R, Sharpless KE, May WE, Crowell JA. NIST/NCI Micronutrients Measurement Quality Assurance Program: Measurement Repeatabilities and Reproducibilities for Fat-Soluble Vitamin-Related Compounds in Human Sera. *Anal Chem* 1997;69(7):1406-1413.
- 2 Margolis SA, Duewer DL. Measurement Of Ascorbic Acid in Human Plasma and Serum: Stability, Intralaboratory Repeatability, and Interlaboratory Reproducibility. *Clin Chem* 1996;42(8):1257-1262.

Appendix A. Shipping Package Inserts for RR24

Two items were included in each package shipped to an RR24 participant:

- **Cover letter.** The original letter has been lost. It likely would have described the four sample materials (sera 160 to 163), given guidance on reconstituting these lyophilized samples, stated that results were due March 6, 1992 and to whom they should be sent, and who to contact with technical questions.
- **Datasheet.** The following page reproduces the form.

These items were attached to the shipping box.

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

SAMPLE #	ANALYTE	RESULT
SERUM 160 VIAL # ____	RETINOL	_____
	ALPHA-TOCOPHEROL	_____
	BETA-CAROTENE (TOTAL)	_____
SERUM 161 VIAL # ____	RETINOL	_____
	ALPHA-TOCOPHEROL	_____
	BETA-CAROTENE (TOTAL)	_____
SERUM 162 VIAL # ____	RETINOL	_____
	ALPHA-TOCOPHEROL	_____
	BETA-CAROTENE (TOTAL)	_____
SERUM 163 VIAL # ____	RETINOL	_____
	ALPHA-TOCOPHEROL	_____
	BETA-CAROTENE (TOTAL)	_____

OPTIONAL ANALYTES : SUPPLY ONE RESULT IF AVAILABLE

SERUM #	160	161	162	163
TRANS-BETA-CAROTENE	_____	_____	_____	_____
ALPHA-CAROTENE	_____	_____	_____	_____
RETINYL PALMITATE	_____	_____	_____	_____
GAMMA-TOCOPHEROL	_____	_____	_____	_____
LYCOPENE (TOTAL)	_____	_____	_____	_____
9-CIS-BETA-CAROTENE	_____	_____	_____	_____
13-CIS-BETA-CAROTENE	_____	_____	_____	_____
LUTEIN	_____	_____	_____	_____
ZEAXANTHIN	_____	_____	_____	_____
BETA-CRYPTOXANTHIN	_____	_____	_____	_____

RECONSTITUTE SERUM SAMPLES WITH 1.0 mL OF WATER.
 NIST FAX # 301-926 8671

RESULTS DUE BACK MARCH 6, 1992

Appendix B. Final Report for RR24

The following eight pages are the final report for RR24 as provided to all participants. This report consists of:

- A cover letter and discussion.
- Tables 1 to 3 and 7 that list the results and various summary values for total retinol, α -tocopherol, and total β -carotene, and γ/β -tocopherol.
- Tables 4 to 6 and 8 to 13 that list the results and simple summary statistics for *trans*- β -carotene, total α -carotene, retinyl palmitate, total lutein, total lycopene, total zeaxanthin, total lutein & zeaxanthin, total β -cryptoxanthin, and total *cis*- β -carotene.

Due to the complex formatting used in the Tables, the originally listed laboratory codes have been deleted without replacement. However, Appendix D provides a complete listing of the RR24 results where the original codes have been altered to ensure confidentiality. Appendix D also provides more relevant summary statistics.



June 2, 1993

NIST

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

Dear Colleague:

This report describes both overall-group and your laboratory's performance in Round Robin XXIV. Specifically, your packet contains, for retinol, alpha-tocopherol, and beta-carotene, respectively: tabular presentations of all data submitted for Round-Robin Study XXIV; a Blind Control Chart, representing a summary of your laboratory's data vs the assigned values for the past five years; and a graphical presentation of data from your laboratory's analysis of blind duplicate samples. Tabular data only is provided for alpha-carotene, all-*trans*-beta-carotene, beta-cryptoxanthin, lutein, lycopene, retinyl palmitate, gamma-tocopherol and zeaxanthin.

Table 1 provides a summary of data submitted for retinol. Forty-three labs submitted data: 29 "Core Labs" (program participants for one year or more) and 13 "New Labs"- 10 participating for the third time and 3 participating in our QA Program round-robin study for the first time; one lab submitted results after the closing date. Their data is included in the table and not considered in statistical evaluations. The interlaboratory CV for the 10 three-study "New Labs" was reduced from an initial average of 65% in RRXXII to an average of 23% for both RRXXIII and this study. The mean CV for the 3 first-study "New Labs" was 36%. The average interlaboratory CV for the 29 Core Labs was 8.5%. Each of the RRXXIV serum pools had been used in a previous study. The trimmed Core Lab average retinol value for the four RRXXIV serum pools was in good agreement with both the NIST values and the previous trimmed Core Lab Average for these pools.

Table 2 provides a summary of data submitted for alpha-tocopherol. Forty-one laboratories submitted data: 30 Core Labs; 8 three-study New Labs; 3 first-study New Labs. The interlaboratory CV for the three-study new labs has improved steadily, from 55% in RRXXII to 23% in RRXXIII to 15% in this study. The mean CV for the 3 first-study New Labs and 30 Core Labs was 32% and 10% respectively in this study. The trimmed Core Lab average alpha-tocopherol value for the four RRXXIV serum pools was in excellent agreement with both the NIST values and the previous trimmed Core Lab Average for these pools.

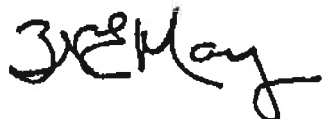
Table 3 provides a summary of data submitted for total beta-carotene. Thirty-three laboratories submitted data: 21 Core Labs; 9 three-study New Labs; 2 first-study New Labs; one late lab. As in recent studies, the overall quality of the beta-carotene data is somewhat poorer than for retinol and alpha-tocopherol. The average CV for measurements made by the three-study New Labs has gone from 25% in RRXXII to 70% in RRXXIII to 40% in this study. The Core Lab average CV's have gotten progressively worse: 21%, 25% and 28% over the past three studies. The agreement between the two first-study New Labs is rather good, however they appear to be biased based on both the NIST and Core Lab trimmed averages (current and past). A series of experiments for use in RRXXVI is being designed to address this growing concern.

Tables 4-12 provide summaries of RRXXIV data submitted for additional fat-soluble vitamin and carotenoid compounds. Please note that analyses for gamma-tocopherol have improved to the point that its data treatment is comparable to that for alpha-tocopherol. Except for *trans* beta-carotene and lycopene, the concentrations of the remaining analytes appear to be too low for a fair assessment of either individual or interlaboratory measurement capabilities. Pools with elevated levels of most analytes will be available for use in RRXXVI.

Data for your use in evaluating your laboratory's individual performance in RRXXIV is provided on the right side of Tables 1-3 and 7. The "Core Lab" Trimmed Values were used as the assigned values. 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 art for these measurements. If you have concerns regarding your performance or are a lab whose performance would be rated "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, feel free to contact us for consultation. SRM 968a can be obtained through the NIST Standard Reference Materials Program (301/975-6776) at a cost of ~215/unit including shipping. A copy of the Certificate of Analysis for SRM 968a is enclosed for your information.

You should have received RRXXV samples by now. Results will be due by June 26 and we expect to provide you with feedback concerning your performance by August 7. Results from all three 1992 Interlaboratory Studies will be discussed at our Annual QA Workshop in November. The actual date has not been set. However, as last year, we will attempt to honor your requests and convene the meeting on a Saturday. Contact us ASAP if you have any strong preferences. Dr. Neal Craft will begin putting the program together within the next month. Feel free to contact him in writing (at this address) or by phone at 301/975-3111.

Sincerely,



Willie E. May, Ph.D.
Chief
Organic Analytical Research Division
Chemical Science and Technology Laboratory

Enclosures

cc: N. Craft
W. Malone

Table 1. Round Robin XXIV
Retinol Results

					% Bias from Trimmed Core Lab Average				
Lab #	Serum # 160	Serum # 161	Serum # 162	Serum # 163	Lab #	Serum # 160	Serum # 161	Serum # 162	Serum # 163
	1.057	0.661	0.296	0.362					
	0.977	0.645	0.283	0.383		3.8	-1.9	1.2	-5.5
	0.969	0.618	0.273	0.359		-4.1	-4.2	-3.3	0.0
	1.085	+0.806	0.300	+0.443		-4.9	-8.3	-6.6	-6.2
	1.078	0.697	0.306	0.389		6.5	19.7	2.5	15.7
	0.973	0.639	0.276	0.368		5.8	3.5	4.6	1.6
	0.995	0.704	0.277	0.392		-4.5	-5.1	-5.7	-4.0
	+1.140	0.690	0.330	0.410		-2.3	4.5	-5.3	2.4
	+0.840	0.582	0.252	0.330		11.9	2.5	12.8	7.1
	0.980	0.634	0.255	0.342		-17.5	-13.6	-13.9	-13.8
	1.073	0.691	0.302	0.407		-3.8	-5.9	-12.8	-10.7
	1.010	0.653	0.288	0.383		5.3	2.6	3.2	6.3
	0.957	0.631	0.257	0.334		-0.8	-3.0	-1.6	0.0
	+1.374	0.749	0.300	0.367		-6.0	-6.3	-12.2	-12.8
	1.020	0.738	0.310	0.426		34.9	11.2	2.5	-4.1
	+0.890	0.610	0.300	0.380		0.1	9.6	6.0	11.3
	0.944	0.622	0.289	0.379		-12.6	-9.4	2.5	-0.7
	0.990	0.715	0.301	0.428		-7.3	-7.6	-1.2	-1.0
	1.070	0.700	0.295	0.394		-2.8	6.2	2.9	11.8
	0.970	0.648	0.289	0.381		5.0	3.9	0.8	2.9
	1.018	0.646	0.293	0.376		-4.8	-3.8	-1.2	-0.5
	1.033	0.682	0.304	0.385		-0.1	-4.1	0.1	-1.8
	1.094	0.682	0.332	0.398		1.4	1.3	3.8	0.6
	1.010	0.673	0.287	0.390		7.4	1.3	13.5	4.0
	1.063	0.726	0.293	0.390		-0.8	-0.1	-1.9	1.9
	0.978	0.616	0.281	0.360		4.4	7.8	0.1	1.9
	1.014	0.698	0.287	0.405		-4.0	-8.5	-4.0	-6.0
	1.018	0.658	0.269	0.400		-0.5	3.6	-1.9	5.8
	+0.675	0.767	0.335	0.418		-0.1	-2.3	-8.1	4.5
	1.040	0.715	0.291	+0.461		-33.7	13.9	14.3	9.2
	1.056	0.753	0.363	0.425		2.1	6.2	-0.5	20.4
	1.213	0.898	0.359	0.487		3.7	11.8	24.1	11.0
	1.039	0.650	0.289	0.383		19.1	33.3	22.7	27.2
	0.851	0.538	0.234	0.276		2.0	-3.5	-1.2	0.0
	1.022	0.731	0.383	0.496		-16.5	-20.1	-20.0	-27.9
	0.109	0.735	0.305	0.375		0.3	8.5	30.9	29.5
	0.983	0.640	0.271	0.368		-89.3	9.1	4.2	-2.1
	0.961	0.638	0.233	0.360		-3.5	-5.0	-7.4	-3.9
	1.313	1.052	0.378	0.476		-5.7	-5.3	-20.4	-6.0
	1.870	0.713	0.044	0.441		28.9	56.2	29.1	24.2
	1.230	0.830	0.320	0.470		83.6	5.9	-85.0	15.2
	0.763	0.590	0.287	0.335		20.8	23.2	9.4	22.8
	0.490	0.380	0.200	0.220		-25.1	-12.4	-2.1	-12.5
						-51.9	-43.6	-31.6	-42.5
AVG (42)	1.002	0.681	0.288	0.387					
SD	0.242	0.102	0.052	0.052					
RSD	24.2	15.0	18.2	13.6					
Core Labs (29;15-97)									
AVG	1.012	0.678	0.293	0.388	Core Labs (29;15-97)	1.019	0.673	0.293	0.383
SD	0.114	0.052	0.020	0.031	TRIM AVG	1.019	0.673	0.293	0.383
RSD	11.2	7.7	7.0	7.9	SD	0.045	0.047	0.020	0.026
					RSD	4.4	6.9	7.0	6.7
(a)New Labs (10;98-115)					NIST				
AVG	1.042	0.735	0.286	0.409	AVG	0.945	0.604	0.273	0.355
SD	0.435	0.146	0.102	0.069	SD	0.048	0.041	0.019	0.030
RSD	41.8	19.9	35.7	16.9	PREVIOUS VALUE	1.010	0.659	0.291	0.369
(b)New Labs (3;116-118)									
AVG	0.828	0.600	0.269	0.342					
SD	0.374	0.225	0.062	0.125					
RSD	45.2	37.5	23.0	36.6					

+ = Value removed for Core Lab Trimmed Average.

L= Late results not included in the statistics.
(a) = participating for the third time.
(b) = participating for the first time.

Table 2. Round Robin XXIV
Alpha-Tocopherol Results

Lab #	Serum # 160	Serum # 161	Serum # 162	Serum # 163
	6.28	16.55	4.62	5.72
	5.74	16.05	4.36	5.56
	+5.23	14.27	3.99	4.94
	6.80	17.20	4.60	5.99
	+7.04	17.63	4.95	5.88
	6.21	15.97	4.45	5.63
	6.40	15.20	4.28	5.56
	6.10	16.20	4.80	5.80
	+7.33	17.95	4.78	6.51
	6.45	16.15	4.52	5.52
	+4.68	13.97	+3.23	+4.40
	6.70	16.17	5.13	6.16
	6.27	16.30	4.76	5.81
	+5.40	14.08	4.00	4.98
	+8.09	+18.56	4.52	5.15
	6.29	15.77	4.58	5.34
	6.21	16.24	4.83	5.56
	6.57	16.35	+6.62	6.37
	6.01	15.68	4.79	5.81
	6.98	16.25	4.91	+7.07
	6.10	16.10	4.50	5.60
	6.28	14.74	4.36	5.70
	6.41	15.69	4.57	5.58
	6.76	17.25	5.07	6.37
	6.16	15.20	4.34	5.36
	6.83	17.62	4.82	5.75
	6.09	15.47	4.78	5.65
	6.21	15.54	4.48	5.48
	+3.13	+18.46	5.04	5.42
	6.19	15.08	4.10	5.19
	6.66	17.23	4.96	5.85
	4.91	11.87	3.57	4.74
	7.20	15.50	5.00	6.70
	6.31	15.29	4.35	5.18
	7.38	20.43	5.55	6.71
	7.20	18.25	5.27	6.03
	6.59	14.62	4.60	5.73
	5.50	14.26	3.81	4.72
	7.90	17.52	5.66	7.30
	5.30	12.67	0.82	4.83
	3.46	11.87	3.56	3.30
AVG (41)	6.23	15.95	4.61	5.64
SD	0.97	1.72	0.60	0.73
RSD	15.6	10.8	13.1	12.9
Core Labs (30;15-97)				
AVG	6.24	16.12	4.63	5.66
SD	0.87	1.20	0.55	0.52
RSD	14.0	7.4	11.9	9.2
(a)New Labs (8;98-113)				
AVG	6.47	15.93	4.64	5.71
SD	0.87	2.64	0.69	0.78
RSD	13.5	16.6	15.0	13.7
(b)New Labs (3;116-118)				
AVG	5.55	14.02	4.35	5.14
SD	2.23	3.06	1.14	2.02
RSD	40.2	21.8	26.3	39.3

% Bias from Trimmed Core Lab Average

Lab #	Serum # 160	Serum # 161	Serum # 162	Serum # 163
	-1.7	3.8	0.3	1.2
	-10.2	0.6	-5.4	-1.7
	-18.2	-10.5	-13.5	-12.7
	6.4	7.9	-0.2	5.9
	10.2	10.6	7.4	3.9
	-2.8	0.1	-3.5	-0.5
	0.2	-4.7	-7.1	-1.7
	-4.5	1.6	4.2	2.5
	14.7	12.6	3.7	15.0
	0.9	1.3	-1.9	-2.5
	-26.8	-12.4	-29.9	-22.2
	4.8	1.4	11.3	8.9
	-1.9	2.2	3.3	2.7
	-15.5	-11.7	-13.2	-12.0
	26.6	16.4	-2.0	-9.0
	-1.6	-1.1	-0.6	-5.6
	-2.8	1.8	4.8	-1.7
	2.8	2.5	43.7	12.6
	-6.0	-1.7	4.0	2.7
	9.2	1.9	6.6	24.9
	-4.5	1.0	-2.3	-1.0
	-1.7	-7.5	-5.5	0.8
	0.3	-1.6	-0.8	-1.4
	5.8	8.2	10.0	12.6
	-3.6	-4.7	-5.8	-5.3
	6.9	10.5	4.6	1.6
	-4.7	-3.0	3.7	-0.2
	-2.8	-2.6	-2.8	-3.2
	-51.1	15.8	9.4	-4.3
	-3.1	-5.4	-11.0	-8.3
	4.2	8.0	7.6	3.4
	-23.2	-25.6	-22.5	-16.2
	12.7	-2.8	8.5	18.4
	-1.3	-4.1	-5.6	-8.5
	15.5	28.1	20.4	18.6
	12.7	14.4	14.3	6.5
	3.1	-8.3	-0.3	1.3
	-13.9	-10.6	-17.3	-16.6
	23.6	9.9	22.8	29.0
	-17.1	-20.6	-17.1	-14.7
	-45.9	-25.6	-22.7	-41.7
Core Labs (30;15-97)				
TRIN AVG	6.39	15.95	4.61	5.66
SD	0.33	1.04	0.31	0.39
RSD	5.1	6.5	6.7	7.0
NIST				
AVG	6.42	16.45	4.72	5.74
SD	0.44	0.59	0.39	0.61
PREVIOUS VALUE	6.28	16.59	4.62	5.64

+ = Value removed for Core Lab Trimmed Average.

L = Late results not included in the statistics.

(a) = participating for the third time.

(b) = participating for the first time.

Table 3. Round Robin XXIV
Total Beta-Carotene Results

Lab #	Serum # 160	Serum # 161	Serum # 162	Serum # 163
	0.094	2.539	1.193	0.203
	0.078	2.180	0.988	0.178
	+0.043	1.979	1.074	0.181
	0.092	2.294	1.001	0.182
	0.111	2.414	1.089	0.211
	+0.130	2.341	1.203	0.199
	0.086	+3.900	+1.760	0.255
	0.096	+2.750	1.212	0.173
	0.092	2.300	1.030	0.190
	0.080	2.457	1.069	0.190
	+0.052	1.856	+0.779	0.183
	0.084	2.300	1.026	0.183
	0.084	1.910	0.959	0.223
	0.079	2.160	+0.851	0.153
	0.109	2.293	1.153	0.225
	0.091	+1.540	1.060	0.241
	0.088	2.271	1.035	0.189
	0.080	1.953	0.939	0.156
	0.104	2.146	1.165	0.201
	0.095	2.070	0.895	0.182
	+0.305	+2.873	+1.301	0.186
	0.090	+0.235	1.040	0.190
	0.081	1.949	0.762	0.132
	0.166	1.788	0.958	0.173
	0.141	2.364	1.083	0.229
	0.034	0.890	0.450	0.069
	0.114	1.965	0.921	0.184
	0.106	2.234	0.939	0.178
	0.038	1.682	0.651	0.104
	0.154	4.635	1.137	0.126
	0.221	2.600	1.140	0.244
	0.073	2.180	0.710	0.150
	0.066	1.630	0.715	0.132
AVG (33)	0.100	2.205	1.016	0.183
SD	0.051	0.725	0.224	0.039
RSD	51.2	32.9	22.0	21.5
Core Labs (21;15-97)				
AVG	0.098	2.223	1.092	0.195
SD	0.051	0.653	0.196	0.025
RSD	51.6	29.4	17.9	12.9
(a)New Labs (9;98-115)				
AVG	0.117	2.234	0.893	0.160
SD	0.061	1.024	0.233	0.057
RSD	52.0	45.8	26.1	35.8
(b)New Labs (2;116-117)				
AVG	0.070	1.905	0.713	0.141
SD	0.005	0.389	0.004	0.013
RSD	7.1	20.4	0.5	9.0

% Bias from Trimmed Core Lab Average

Lab #	Serum # 160	Serum # 161	Serum # 162	Serum # 163
	3.9	14.8	11.2	4.2
	-13.7	-1.5	-7.9	-8.7
	-53.0	-10.5	0.1	-7.1
	1.7	3.7	-6.7	-6.6
	22.2	9.1	1.5	8.0
	43.8	5.8	12.1	2.1
	-4.9	76.3	64.1	30.9
	6.2	24.3	13.0	-11.2
	1.7	4.0	-4.0	-2.5
	-11.5	11.1	-0.3	-2.5
	-42.5	-16.1	-27.4	-6.1
	-7.1	4.0	-4.4	-6.1
	-7.1	-13.7	-10.6	14.4
	-12.6	-2.4	-20.7	-21.5
	20.5	3.7	7.5	15.5
	0.6	-30.4	-1.2	23.7
	-2.7	2.7	-3.5	-3.0
	-11.7	-11.7	-12.4	-19.7
	15.0	-3.0	8.6	3.1
	5.1	-6.4	-16.6	-6.6
	237.3	29.9	21.3	-4.5
	-0.5	-89.4	-3.1	-2.5
	-10.4	-11.9	-29.0	-32.3
	83.6	-19.2	-10.7	-11.2
	55.9	6.9	1.0	17.5
	-62.4	-59.8	-58.1	-64.6
	26.1	-11.2	-14.1	-5.6
	17.2	1.0	-12.5	-8.7
	-58.0	-24.0	-39.3	-46.6
	69.9	109.5	5.9	-35.4
	144.4	17.5	6.3	25.2
	-19.3	-1.5	-33.8	-23.0
	-27.0	-26.3	-33.3	-32.3
Core Labs (21;15-97)				
TRIM AVG	0.090	2.212	1.073	0.195
SD	0.010	0.201	0.085	0.025
RSD	11.0	9.1	7.9	12.9
NIST				
AVG	0.078	2.179	1.049	0.191
SD	0.013	0.222	0.062	0.013
PREVIOUS VALUE	0.090	2.338	1.145	0.202

+ = Value removed for Core Lab Trimmed Average.

L = Late results not included in the statistics.
(a) = participating for the third time.
(b) = participating for the first time.

Table 4. Round Robin XXIV
Trans-Beta-Carotene Results

Lab #	Serum # 160	Serum # 161	Serum # 162	Serum # 163
	0.083	2.352	1.089	0.185
	0.086	2.140	0.980	0.180
	0.080	2.276	1.009	0.180
	0.043	1.554	0.672	0.139
	0.080	2.093	0.975	0.179
	0.073	1.690	0.670	0.120
	0.030	0.727	0.399	0.062
	0.075	1.798	0.987	0.163
	0.113	3.883	0.941	0.126
NIST	0.072	1.900	0.947	0.181
AVG	0.073	2.041	0.867	0.151
SD	0.023	0.797	0.215	0.040
RSD	31.2	39.1	24.8	26.3

Table 6. Round Robin XXIV
Retinyl Palmitate

Lab #	Serum # 160	Serum # 161	Serum # 162	Serum # 163
	0.010	0.024		
	0.020	0.035	0.008	0.031
	0.101	0.081	0.026	0.030
		0.040		0.026
	0.115	0.080	0.041	0.064
AVG	0.062	0.052	0.025	0.038
SD	0.054	0.027	0.017	0.018
RSD	88.0	51.2	67.0	46.5

Table 7. Round Robin XXIV
Gamma-Tocopherol

Lab #	Serum # 160	Serum # 161	Serum # 162	Serum # 163
	1.70	4.09	1.64	3.59
	1.60	3.80	1.60	3.40
	1.30	3.60	1.60	3.20
	1.57	3.61	1.56	3.24
	1.96	3.85	1.95	3.71
	1.67	3.64	1.64	3.28
	1.45	3.45	1.51	3.07
	*2.36	*4.47	1.82	3.41
	1.75	3.73	2.04	3.24
	1.88	3.75	1.77	3.67
	1.78	3.68	1.73	3.45
	1.77	3.28	1.44	3.81
	*0.53	*1.18	*0.54	*1.04
	1.40	3.38	1.45	3.10
	1.38	3.44	1.28	3.07
	*1.04	*2.91	*1.08	*2.27
NIST	1.51	3.43	1.52	3.32
AVG	1.57	3.49	1.54	3.17
SD	0.40	0.69	0.35	0.65
RSD	25.4	19.6	22.5	20.5
TRIMMED				
AVG	1.62	3.62	1.64	3.37
SD	0.20	0.22	0.20	0.24
RSD	12.2	6.0	12.2	7.0

Table 5. Round Robin XXIV
Alpha-Carotene

Lab #	Serum # 160	Serum # 161	Serum # 162	Serum # 163
	0.026	0.101	0.038	0.012
	0.017	0.088	0.032	0.010
	0.017		0.021	
	0.049	0.280	0.077	
	0.003	0.042	0.012	
	0.026	0.100	0.036	0.009
	0.016	0.096	0.032	0.010
	0.020	0.074	0.029	0.013
	0.013	0.083	0.032	0.014
	0.015	0.086	0.018	0.005
	0.023	0.074	0.031	0.012
	0.014	0.121	0.054	0.010
	0.196	0.091	0.034	0.009
	0.025	0.084	0.030	0.010
	0.033	0.121	0.046	0.038
	0.021	0.093	0.028	0.009
	0.039	0.086	0.029	0.053
		0.038	0.017	0.005
	0.016	0.079	0.037	0.019
		0.229	0.046	
NIST	0.018	0.091	0.034	0.010
AVG	0.031	0.103	0.034	0.015
SD	0.041	0.056	0.014	0.012
RSD	133.6	54.7	41.0	84.4

% Bias from Trimmed Lab Average

Lab #	Serum # 160	Serum # 161	Serum # 162	Serum # 163
	4.8	12.9	0.1	6.5
	-1.4	4.9	-2.2	0.8
	-19.9	-0.6	-2.2	-5.1
	-3.3	-0.4	-4.7	-3.9
	20.8	6.3	19.2	10.0
	2.9	0.5	0.2	-2.7
	-10.6	-4.8	-7.7	-8.9
	60.2	23.4	11.2	1.1
	7.8	2.9	24.7	-3.9
	15.8	3.4	8.2	8.9
	9.7	1.6	5.7	2.3
	9.1	-9.5	-12.0	13.0
	-67.3	-67.4	-67.0	-69.2
	-13.8	-6.8	-11.7	-7.9
	-14.9	-5.1	-21.7	-8.9
	-35.9	-19.7	-34.0	-32.7

* = Value removed for the TRIMMED average.

Table 8. Round Robin XXIV
Lutein

Lab #	Serum # 160	Serum # 161	Serum # 162	Serum # 163
	0.067	0.086	0.053	0.045
	0.055	0.079	0.045	0.039
	0.109	0.138	0.081	0.068
	0.073	0.093	0.047	0.049
	0.174	0.196	0.107	0.152
	0.062	0.081	0.048	0.045
	0.091	0.147	0.082	0.093
	0.045	0.043	0.026	0.027
	0.030	0.045	0.029	0.020
		0.182	0.114	0.310
NIST	0.072	0.079	0.049	0.027
AVG	0.078	0.106	0.062	0.080
SD	0.040	0.052	0.030	0.085
RSD	51.9	48.8	48.0	107.1

Table 9. Round Robin XXIV
Lycopene

Lab #	Serum # 160	Serum # 161	Serum # 162	Serum # 163
	0.824	0.339	0.205	0.168
	0.754	0.347	0.200	0.166
	0.845	0.372	0.198	0.142
	1.066	0.362	0.219	0.168
	1.175	0.563	0.344	0.265
	0.484	0.210	0.120	0.102
	0.792	0.351	0.181	0.161
	0.183	0.146	0.064	0.068
	1.235	0.416	0.195	0.204
	0.727	0.290	0.143	0.127
	0.416	0.175	0.111	0.091
	0.910	0.486	0.341	0.293
	0.747	0.279	0.165	0.128
	0.642	0.217	0.102	0.114
	0.806	0.484	0.237	0.167
	0.646	0.218	0.122	0.100
	0.621	0.281	0.134	0.121
	0.315	0.106	0.077	0.052
	0.638	0.272	0.136	0.125
		0.182	0.114	0.310
NIST	0.779	0.316	0.182	0.139
AVG	0.730	0.305	0.171	0.153
SD	0.261	0.118	0.074	0.068
RSD	35.8	38.8	43.3	44.4

Table 10. Round Robin XXIV
Zeaxanthin

Lab #	Serum # 160	Serum # 161	Serum # 162	Serum # 163
	0.010	0.022	0.013	0.012
	0.019	0.027	0.019	0.021
	0.015	0.021	0.015	0.015
	0.011	0.021	0.015	0.011
		0.070	0.040	0.091
NIST	0.038	0.036	0.026	0.025
AVG	0.019	0.033	0.021	0.029
SD	0.011	0.019	0.010	0.031
RSD	61.4	58.2	48.0	105.5

Table 11. Round Robin XXIV
Lutein + Zeaxanthin

Lab #	Serum # 160	Serum # 161	Serum # 162	Serum # 163
	0.080	0.110	0.074	0.065
	0.127	0.166	0.098	0.085
	0.095	0.131	0.077	0.067
	0.057	0.076	0.050	0.050
AVG	0.090	0.121	0.075	0.067
SD	0.029	0.038	0.020	0.014
RSD	32.7	31.3	26.3	21.5

Table 12. Round Robin XXIV
Beta-Cryptoxanthin

Lab #	Serum # 160	Serum # 161	Serum # 162	Serum # 163
	0.057	0.061	0.042	0.034
	0.046	0.063	0.042	0.031
	0.124	0.111	0.056	0.038
	0.062	0.065	0.041	0.035
	0.031	0.035	0.022	0.021
	0.065	0.066	0.031	0.031
	0.165	0.086	0.067	0.063
	0.085	0.110	0.066	0.052
	0.037	0.058	0.030	0.026
	0.094	0.062	0.035	0.034
	0.117	0.090	0.068	0.060
	0.239	0.155	0.103	0.065
	0.031	0.027	0.018	0.015
	0.037	0.053	0.041	0.037
NIST	0.038	0.037	0.025	0.020
AVG	0.082	0.072	0.046	0.037
SD	0.059	0.034	0.023	0.016
RSD	71.9	47.0	49.5	42.0

Table 13. Round Robin XXIV
9+13 Cis-Beta-Carotene

Lab #	Serum # 160	Serum # 161	Serum # 162	Serum # 163
	0.008	0.163	0.051	0.014

Appendix C. Representative “Individualized Report” for RR24

Each participant in RR24 received graphical summaries of their own measurement performance for total retinol, α -tocopherol, and/or total β -carotene. In RR24, two sets of graphs were prepared:

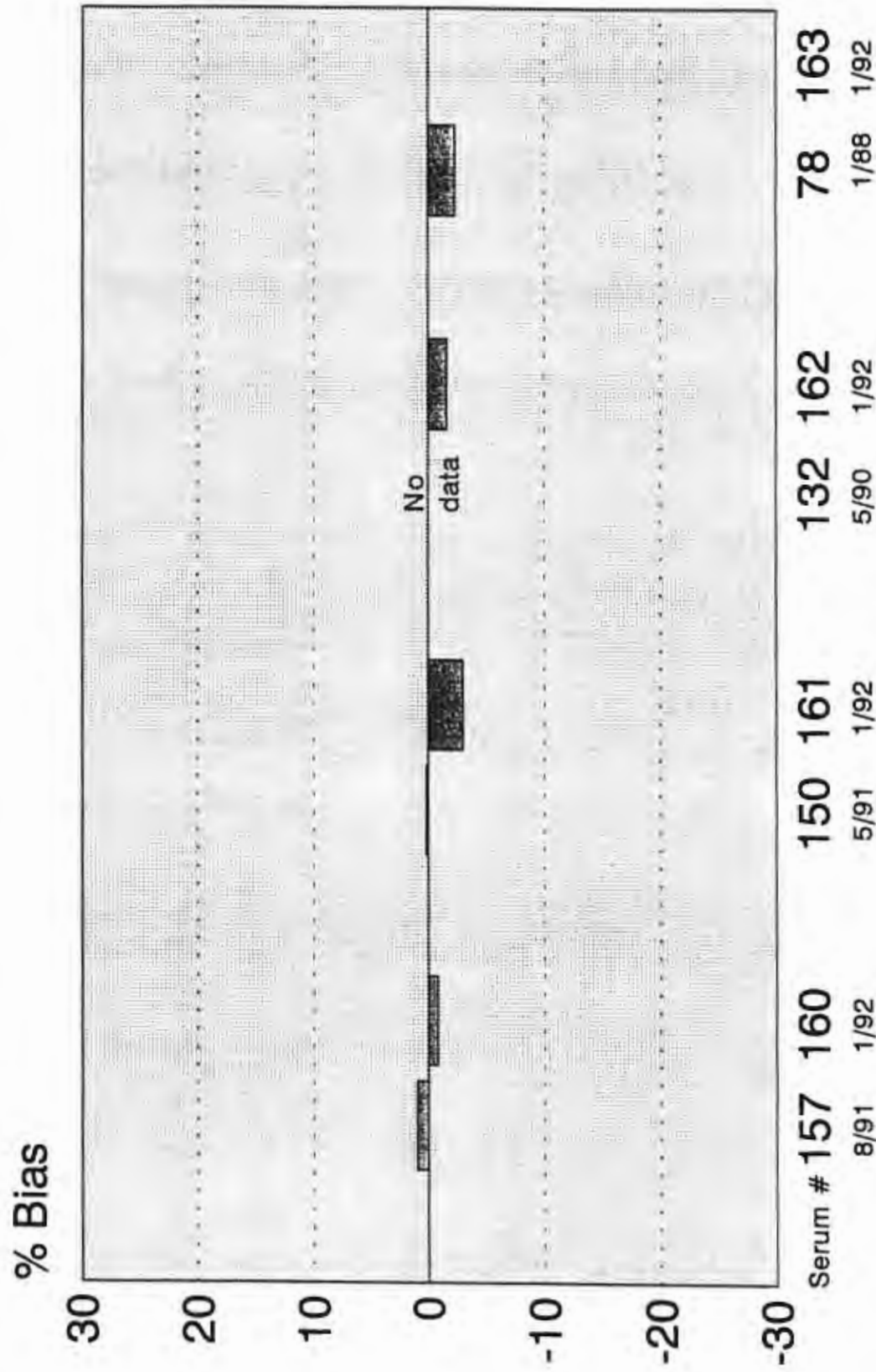
- “Percent Bias” relative to the “Trimmed Core Lab Average” for of the serum-based samples distributed from 7/1987 through 1/92.
- “Blind Duplicate Performance”, documenting the history of the % Bias values for just the sera distributed in RR24.

The following six pages constitute the individualized report for participant FSV-BA.

Laboratory FSV-BA

Blind Duplicate Performance

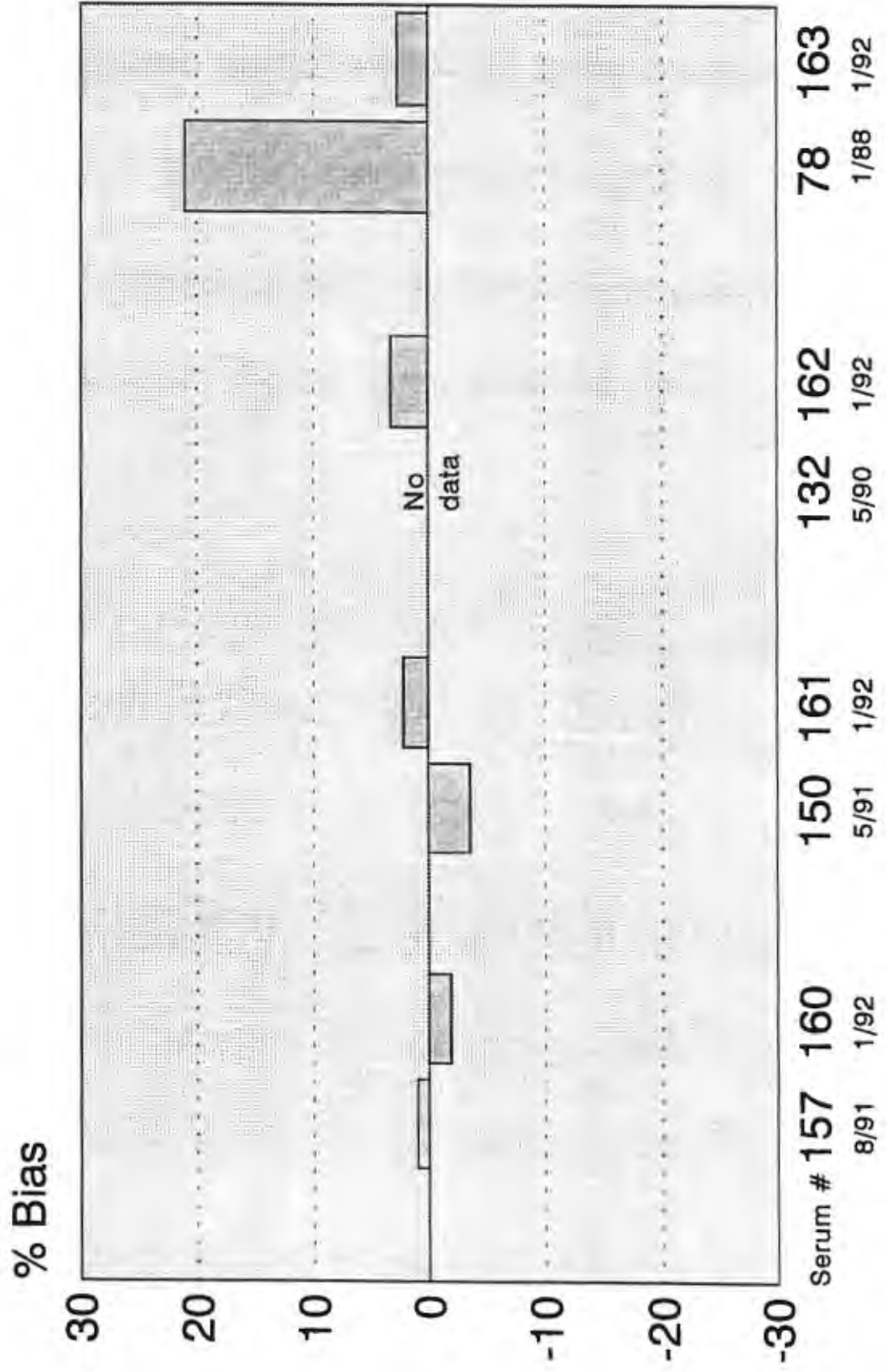
Retinol



Laboratory FSV-BA

Blind Duplicate Performance

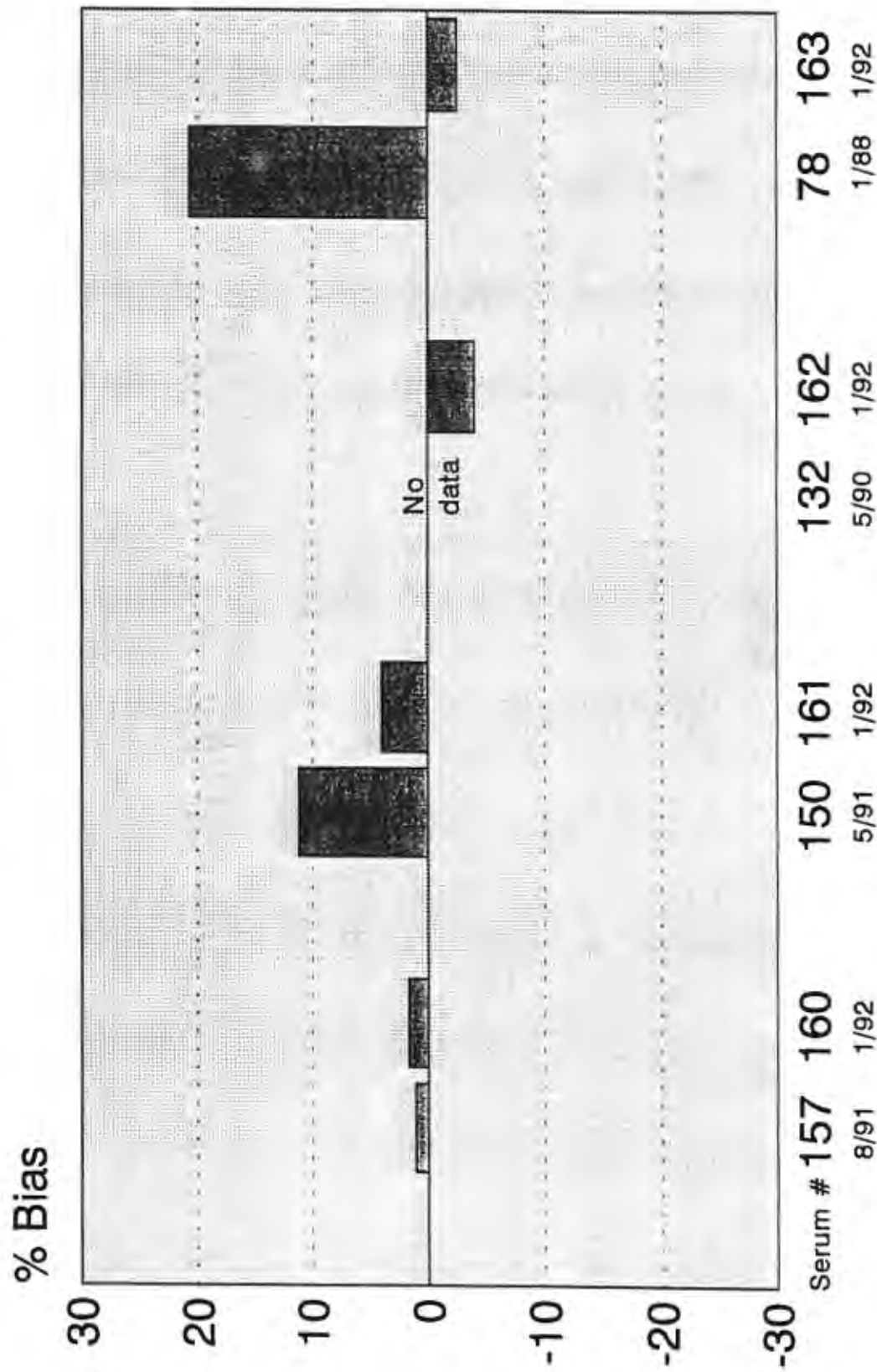
Alpha-Tocopherol



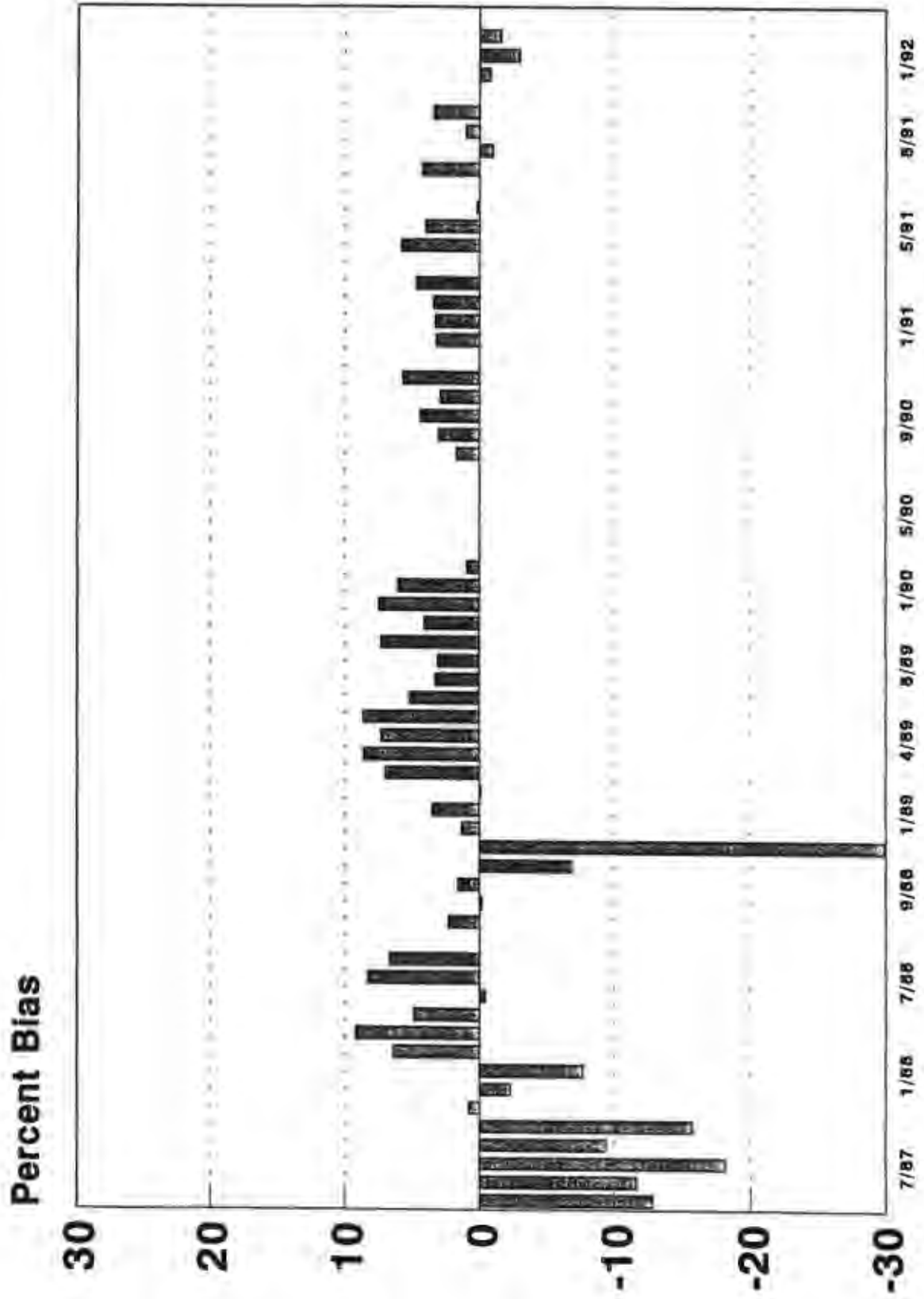
Laboratory FSV-BA

Blind Duplicate Performance

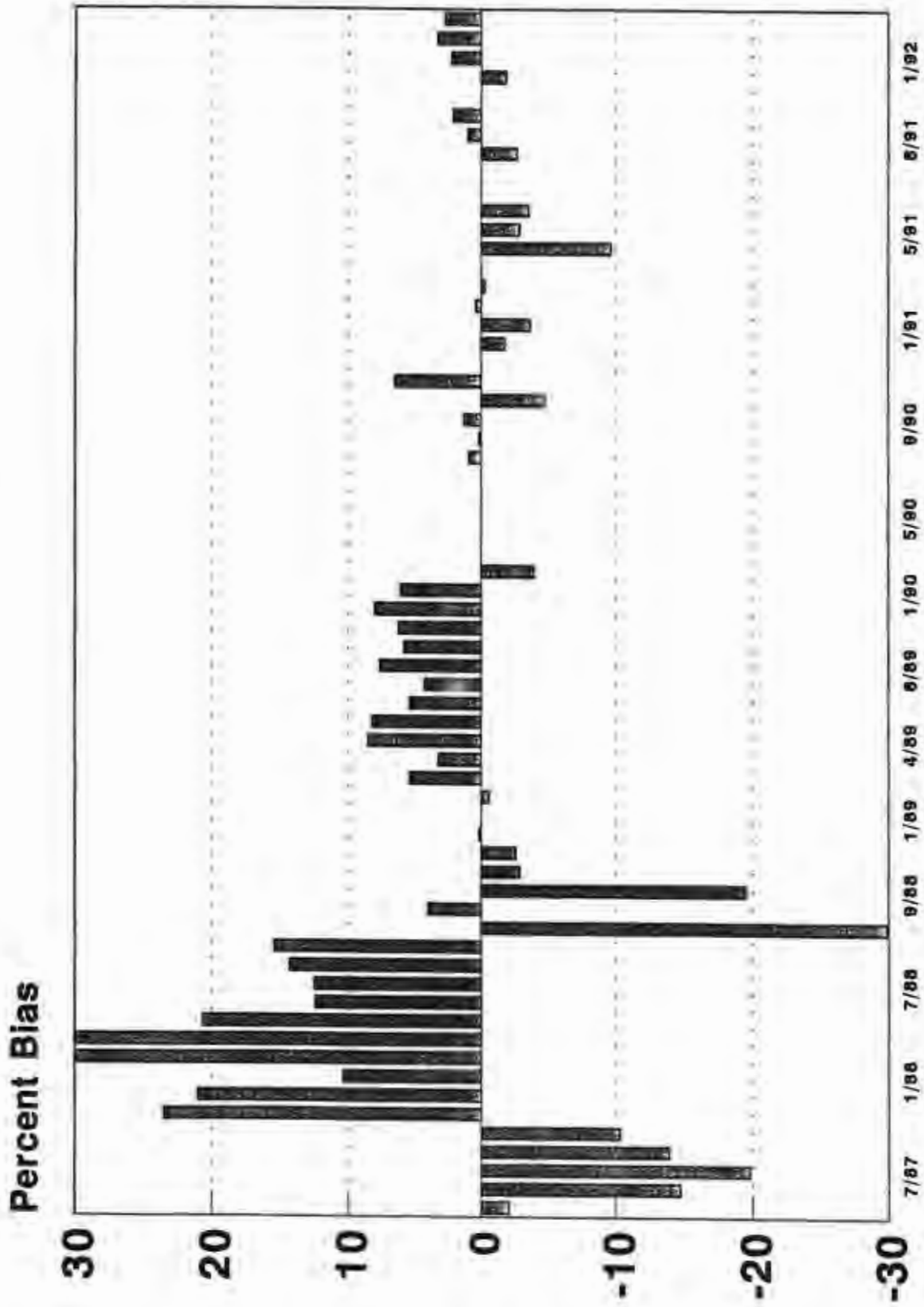
Total Beta-Carotene



Laboratory FSV-BA Retinol

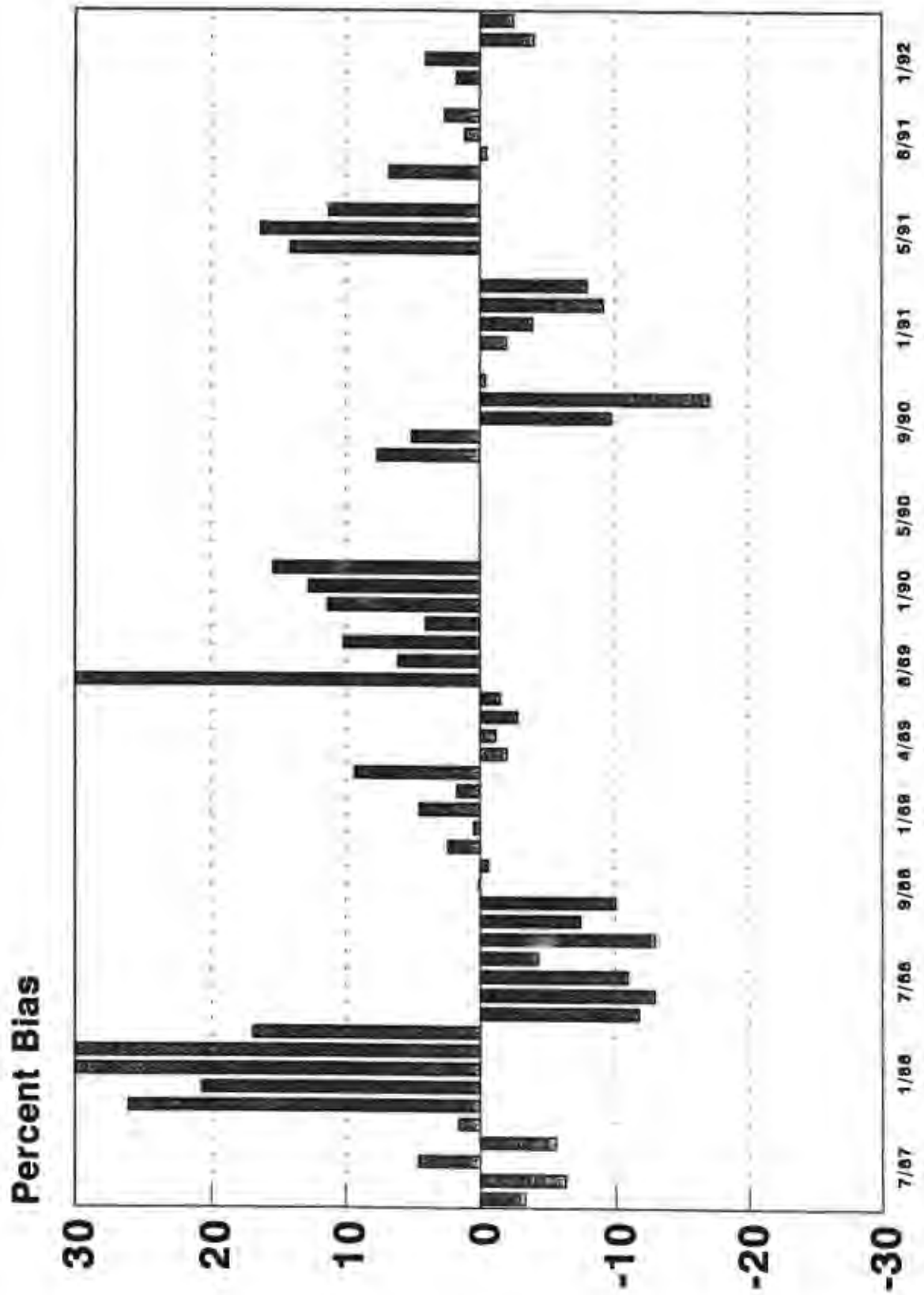


Laboratory FSV-BA Alpha-Tocopherol



% Bias from Assigned Values

Laboratory FSV-BA Total Beta-Carotene



% Bias from Assigned Values

Appendix D. Updated “All-Lab Report” for RR24

The following six pages are a modernized version of an “All-Lab” report for RR24. This report has three parts:

- pages 1 thru 4 list results for all analytes reported by at least twice, counting both participants and NIST analysts.
- page 5 provides a legend for pages 1 thru 4.
- page 6 summarizes each participants’ performance for total retinol, α - and γ/β -tocopherol, and total β -carotene. These summaries are compatible with the percent bias evaluation advice given in the RR24 Report. However, the current bias summaries are estimated relative to the median of all reported values for each analyte in each serum rather than to the “Trimmed Core Lab Average” used in the original and detailed in Appendix B. These original reference values were estimated from on-time results of the more experienced participants, with subjective exclusion of results deemed non-representative.

To ensure confidentiality, the laboratory identifiers used in this “All-Lab Report” have been altered from those used in RR24. 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.

Note: The “NIST” values in the Tables described in Appendix B are the average of the NISTa, NISTb, and NISTc results.

Round Robin XXIV Laboratory Results

Lab	Total Retinol, µg/mL				Retinyl Palmitate, µg/mL				α-Tocopherol, µg/mL				γ/β-Tocopherol, µg/mL			
	160	161	162	163	160	161	162	163	160	161	162	163	160	161	162	163
FSV-BA	1.01	0.653	0.288	0.383	0.101	0.081	0.026	0.030	6.27	16.3	4.76	5.81	1.67	3.64	1.64	3.28
FSV-BD	1.00	0.704	0.277	0.392					6.40	15.2	4.28	5.56				
FSV-BE	0.98	0.634	0.255	0.342					4.68	14.0	3.23	4.40				
FSV-BF	1.14	0.690	0.330	0.410					6.10	16.2	4.80	5.80	1.30	3.60	1.60	3.20
FSV-BG	0.97	0.618	0.273	0.359	0.020	0.035	0.008	0.031	5.23	14.3	3.99	4.94				
FSV-BH	0.96	0.631	0.257	0.334	nd	0.040	nd	0.026	5.40	14.1	4.00	4.98	1.45	3.45	1.51	3.07
FSV-BI	0.98	0.645	0.283	0.383	nd	nd	nd	nd	5.74	16.1	4.36	5.56	1.60	3.80	1.60	3.40
FSV-BJ	1.02	0.646	0.293	0.376	nd	nd	nd	nd	6.28	14.7	4.36	5.70	1.88	3.75	1.77	3.67
FSV-BK	1.03	0.682	0.304	0.385					6.41	15.7	4.57	5.58				
FSV-BL	0.99	0.715	0.301	0.428					6.98	16.3	4.91	7.07				
FSV-BM	0.97	0.648	0.289	0.381					6.10	16.1	4.50	5.60				
FSV-BN	1.09	0.735	0.305	0.375					7.20	18.2	5.27	6.03	1.40	3.38	1.45	3.10
FSV-BO	1.23	0.830	0.320	0.470					7.90	17.5	5.66	7.30				
FSV-BP	1.02	0.731	0.383	0.496					7.38	20.4	5.55	6.71				
FSV-BS	1.31	1.052	0.378	0.476												
FSV-BX																
FSV-BY	1.06	0.661	0.296	0.362	0.010	0.024	nd	nd	6.28	16.5	4.62	5.72	1.70	4.09	1.64	3.59
FSV-CA	1.08	0.697	0.306	0.389					7.04	17.6	4.95	5.88				
FSV-CB	0.85	0.538	0.234	0.276					6.31	15.3	4.35	5.18				
FSV-CH	1.25	0.767	0.335	0.418					10.70	18.5	5.04	5.42				
FSV-CJ	1.09	0.806	0.300	0.443					6.80	17.2	4.60	5.99				
FSV-CK	1.37	0.749	0.300	0.367					8.09	18.6	4.52	5.15	2.36	4.47	1.82	3.41
FSV-CL	1.06	0.753	0.363	0.425					6.66	17.2	4.96	5.85	0.53	1.18	0.54	1.04
FSV-CM									6.01	15.7	4.79	5.81				
FSV-CO	0.97	0.639	0.276	0.368					6.21	16.0	4.45	5.63				
FSV-CP																
FSV-CQ	0.84	0.582	0.252	0.330					7.33	18.0	4.78	6.51				
FSV-CS	1.07	0.700	0.295	0.394												
FSV-CT	1.87	0.713	0.044	0.441												
FSV-CU	0.98	0.616	0.281	0.360	0.115	0.080	0.041	0.064	6.09	15.5	4.78	5.65				
FSV-CV	1.01	0.673	0.287	0.390					6.16	15.2	4.34	5.36	1.78	3.68	1.73	3.45
FSV-CY	1.02	0.738	0.310	0.426					6.29	15.8	4.58	5.34				
FSV-DC	1.07	0.691	0.302	0.407					6.70	16.2	5.13	6.16	1.96	3.85	1.95	3.71
FSV-DE									6.45	16.2	4.52	5.52	1.57	3.61	1.56	3.24
FSV-DL	1.02	0.658	0.269	0.400												
FSV-DM	1.21	0.898	0.359	0.487					4.91	11.9	3.57	4.74				
FSV-DS	0.76	0.590	0.287	0.335					5.30	12.7	3.82	4.83				
FSV-DW																
FSV-DY	0.94	0.622	0.289	0.379					6.57	16.4	6.62	6.37				
FSV-EA	1.04	0.650	0.289	0.383					7.20	15.5	5.00	6.70				
FSV-EB	1.01	0.698	0.287	0.405					6.21	15.5	4.48	5.48				
FSV-EC	1.04	0.646	0.286	0.395					6.80	15.9	5.15	6.57				
FSV-EF	0.89	0.610	0.300	0.380					6.21	16.2	4.83	5.56	1.75	3.73	2.04	3.24
FSV-EI	0.96	0.638	0.233	0.360					5.50	14.3	3.81	4.72	1.38	3.44	1.28	3.07
FSV-EJ	1.04	0.715	0.291	0.461					6.19	15.1	4.10	5.19	1.77	3.28	1.44	3.81
FSV-EN	0.49	0.380	0.200	0.220					3.46	11.9	3.56	3.30	1.04	2.91	1.08	2.27
FSV-EV	1.06	0.726	0.293	0.390					6.83	17.6	4.82	5.75				
FSV-EY	1.09	0.682	0.332	0.398					6.76	17.3	5.07	6.37				
FSV-FM	0.98	0.640	0.271	0.368					6.59	14.6	4.60	5.73				
n	43	44	44	44	4	5	3	4	41	42	42	42	16	16	16	16
Min	0.490	0.380	0.044	0.220	0.010	0.024	0.008	0.026	3.46	11.9	3.23	3.30	0.53	1.18	0.54	1.04
Mean	1.037	0.684	0.289	0.390	0.062	0.052	0.025	0.038	6.32	15.9	4.62	5.66	1.57	3.49	1.54	3.16
Max	1.870	1.052	0.383	0.496	0.115	0.081	0.041	0.064	8.09	20.4	6.62	7.30	2.36	4.47	2.04	3.81
SD	0.189	0.099	0.052	0.051	0.054	0.027	0.017	0.018	0.85	1.7	0.60	0.73	0.41	0.71	0.36	0.67
CV	10	9	13	10	47	33	41	27	11	8	9	10	17	16	17	18
NISTa	0.905	0.599	0.279	0.336					5.92	15.9	4.28	5.13	1.60	3.65	1.55	3.43
NISTb	0.932	0.565	0.252	0.339					6.58	17.1	5.01	6.35	1.61	3.60	1.65	3.40
NISTc	0.998	0.647	0.288	0.389					6.76	16.4	4.87	5.75	1.32	3.05	1.36	3.14
Median	1.018	0.678	0.290	0.387	0.040				6.29	16.01	4.60	5.64	1.64	3.63	1.60	3.26
eSD	0.071	0.058	0.020	0.032	0.024				0.61	1.29	0.42	0.48	0.31	0.27	0.23	0.28
eCV	7	9	7	8	59				10	8	9	9	19	7	15	9

Round Robin XXIV Laboratory Results

Lab	Total β -Carotene, $\mu\text{g/mL}$				trans- β -Carotene, $\mu\text{g/mL}$				Total cis- β -Carotene, $\mu\text{g/mL}$			
	160	161	162	163	160	161	162	163	160	161	162	163
FSV-BA	0.092	2.30	1.03	0.190	0.086	2.14	0.98	0.180	0.006	0.16	0.05	0.010
FSV-BD												
FSV-BE												
FSV-BF	0.130	2.34	1.20	0.199								
FSV-BG	0.043	1.98	1.07	0.181								
FSV-BH	0.080	2.46	1.07	0.190	0.080	2.28	1.01	0.180	0.000	0.18	0.06	0.010
FSV-BI	0.078	2.18	0.99	0.178								
FSV-BJ	0.109	2.29	1.15	0.225								
FSV-BK												
FSV-BL												
FSV-BM												
FSV-BN	0.114	1.97	0.99	0.184	0.075	1.80	0.92	0.163	0.039	0.17	0.07	0.021
FSV-BO	0.073	2.18	0.71	0.150								
FSV-BP	0.141	2.36	1.08	0.229								
FSV-BS	0.154	4.64	1.14	0.126	0.113	3.88	0.94	0.126	0.042	0.75	0.20	nd
FSV-BX	0.084	2.30	1.03	0.183								
FSV-BY	0.094	2.54	1.19	0.203	0.083	2.35	1.09	0.185	0.011	0.19	0.10	0.018
FSV-CA												
FSV-CB												
FSV-CH	0.31	2.87	1.30	0.186								
FSV-CJ	0.092	2.29	1.00	0.182								
FSV-CK	0.052	1.86	0.78	0.183	0.043	1.55	0.67	0.139	0.009	0.30	0.11	0.044
FSV-CL	0.081	1.95	0.76	0.132	0.073	1.69	0.67	0.120	0.008	0.26	0.09	0.012
FSV-CM												
FSV-CO	0.111	2.41	1.09	0.211								
FSV-CP	0.080	1.95	0.94	0.156								
FSV-CQ	0.086	3.90	1.76	0.255								
FSV-CS												
FSV-CT	0.221	2.60	1.14	0.244								
FSV-CU	0.088	2.27	1.04	0.189	0.080	2.09	0.98	0.179	0.008	0.18	0.06	0.010
FSV-CV	0.091	1.54	1.06	0.241								
FSV-CY	0.084	1.91	0.96	0.223								
FSV-DC	0.096	2.75	1.21	0.173								
FSV-DE												
FSV-DL	0.095	2.07	0.90	0.182								
FSV-DM	0.166	1.79	0.96	0.173								
FSV-DS	0.066	1.63	0.72	0.132								
FSV-DW	0.034	0.89	0.45	0.069	0.030	0.73	0.40	0.062	0.004	0.16	0.05	0.007
FSV-DY	0.079	2.16	0.85	0.153								
FSV-EA												
FSV-EB	0.104	2.15	1.17	0.201								
FSV-EC	0.112	1.65	1.26	0.217								
FSV-EF												
FSV-EI	0.038	1.68	0.65	0.104								
FSV-EJ	0.090	0.24	1.04	0.190								
FSV-EN												
FSV-EV												
FSV-EY												
FSV-FM	0.106	2.23	0.94	0.178								
n	33	34	34	34	9	9	9	9	9	9	9	8
Min	0.034	0.24	0.45	0.069	0.030	0.73	0.40	0.062	0.000	0.16	0.05	0.007
Mean	0.096	2.19	1.02	0.183	0.074	2.06	0.85	0.148	0.014	0.26	0.09	0.017
Max	0.221	4.64	1.76	0.255	0.113	3.88	1.09	0.185	0.042	0.75	0.20	0.044
SD	0.037	0.73	0.23	0.039	0.024	0.84	0.22	0.041	0.015	0.19	0.05	0.012
CV	17	16	13	15	22	22	20	22	37	25	24	27
NISTa	0.090	2.40	1.12	0.204								
NISTb	0.065	1.96	1.02	0.190	0.063	1.62	0.86	0.189	0.002	0.34	0.15	0.001
NISTc	0.080	2.18	1.01	0.179	0.081	2.18	1.03	0.172	nd	nd	nd	0.007
Median	0.091	2.180	1.033	0.184	0.080	2.093	0.941	0.163	0.008	0.181	0.066	0.011
eSD	0.019	0.345	0.157	0.027	0.009	0.437	0.101	0.033	0.004	0.027	0.024	0.004
eCV	21	16	15	15	11	21	11	20	56	15	36	34

D3

Round Robin XXIV Laboratory Results

Lab	Total α -Carotene, $\mu\text{g/mL}$				Total Lycopene, $\mu\text{g/mL}$				Total β -Cryptoxanthin, $\mu\text{g/mL}$			
	160	161	162	163	160	161	162	163	160	161	162	163
FSV-BA	0.026	0.100	0.036	0.009	0.48	0.21	0.120	0.102	0.062	0.065	0.041	0.035
FSV-BD												
FSV-BE												
FSV-BF	0.049	0.280	0.077	nd								
FSV-BG												
FSV-BH	0.016	0.096	0.032	0.010	0.79	0.35	0.181	0.161	0.031	0.035	0.022	0.021
FSV-BI	0.017	0.088	0.032	0.010	0.75	0.35	0.200	0.166	0.046	0.063	0.042	0.031
FSV-BJ	0.023	0.074	0.031	0.012	0.42	0.18	0.111	0.091				
FSV-BK												
FSV-BL												
FSV-BM												
FSV-BN	0.016	0.079	0.037	0.019	0.64	0.27	0.136	0.125	0.037	0.053	0.041	0.037
FSV-BO												
FSV-BP	0.039	0.086	0.029	0.053	0.62	0.28	0.134	0.121	0.239	0.155	0.103	0.065
FSV-BS	nd	0.229	0.046	nd								
FSV-BX	0.013	0.083	0.032	0.014	1.24	0.42	0.195	0.204	0.165	0.086	0.067	0.063
FSV-BY	0.026	0.101	0.038	0.012	0.82	0.34	0.205	0.168	0.057	0.061	0.042	0.034
FSV-CA					1.07	0.36	0.219	0.168				
FSV-CB												
FSV-CH												
FSV-CJ	0.017	nd	0.021	nd	0.85	0.37	0.198	0.142	0.124	0.111	0.056	0.038
FSV-CK	0.020	0.074	0.029	0.013	0.18	0.15	0.064	0.068	0.065	0.066	0.031	0.031
FSV-CL	0.021	0.093	0.028	0.009	0.65	0.22	0.122	0.100	0.117	0.090	0.068	0.060
FSV-CM												
FSV-CO												
FSV-CP	0.196	0.091	0.034	0.009	0.75	0.28	0.165	0.128	0.037	0.058	0.030	0.026
FSV-CQ												
FSV-CS												
FSV-CT												
FSV-CU												
FSV-CV	0.014	0.121	0.054	0.010	0.91	0.49	0.341	0.293				
FSV-CY												
FSV-DC	0.003	0.042	0.012	nd	1.18	0.56	0.344	0.265				
FSV-DE												
FSV-DL	0.025	0.084	0.030	0.010	0.64	0.22	0.102	0.114	0.094	0.062	0.035	0.034
FSV-DM												
FSV-DS												
FSV-DW		0.038	0.017	0.005	0.32	0.11	0.077	0.052	0.031	0.027	0.018	0.015
FSV-DY	0.015	0.086	0.018	<0.005	0.73	0.29	0.143	0.127	0.085	0.110	0.066	0.052
FSV-EA												
FSV-EB												
FSV-EC												
FSV-EF												
FSV-EI												
FSV-EJ	0.033	0.121	0.046	0.038	0.81	0.48	0.237	0.167				
FSV-EN												
FSV-EV												
FSV-EY												
FSV-FM												
n	18	19	20	15	19	19	19	19	14	14	14	14
Min	0.003	0.038	0.012	0.005	0.18	0.11	0.064	0.052	0.031	0.027	0.018	0.015
Mean	0.032	0.103	0.034	0.016	0.73	0.31	0.173	0.145	0.085	0.074	0.047	0.039
Max	0.196	0.280	0.077	0.053	1.24	0.56	0.344	0.293	0.239	0.155	0.103	0.065
SD	0.042	0.058	0.014	0.013	0.27	0.12	0.077	0.061	0.060	0.034	0.023	0.015
CV	22	21	19	24	22	22	22	21	25	22	22	24
NISTa												
NISTb	0.019	0.106	0.035	0.012	0.88	0.31	0.178	0.162	0.043	0.038	0.024	0.020
NISTc	0.017	0.076	0.034	0.009	0.68	0.32	0.185	0.116	0.033	0.037	0.026	0.019
Median	0.021	0.088	0.032	0.010	0.747	0.290	0.165	0.128	0.064	0.064	0.042	0.035
eSD	0.008	0.018	0.007	0.003	0.162	0.108	0.064	0.055	0.042	0.024	0.020	0.009
eCV	40	20	21	30	22	37	39	43	67	38	47	25

D4

Round Robin XXIV Laboratory Results

Lab	Total Lutein, µg/mL				Total Zeaxanthin, µg/mL				Total Lutein&Zeaxanthin, µg/mL			
	160	161	162	163	160	161	162	163	160	161	162	163
FSV-BA									0.127	0.166	0.098	0.085
FSV-BD												
FSV-BE												
FSV-BF												
FSV-BG												
FSV-BH	0.073	0.093	0.047	0.049	0.019	0.027	0.019	0.021	0.092	0.120	0.066	0.070
FSV-BI	0.055	0.079	0.045	0.039	0.010	0.022	0.013	0.012	0.080	0.110	0.074	0.065
FSV-BJ												
FSV-BK												
FSV-BL												
FSV-BM												
FSV-BN	0.030	0.045	0.029	0.020	0.011	0.021	0.015	0.011	0.057	0.076	0.050	0.050
FSV-BO												
FSV-BP												
FSV-BS												
FSV-BX	0.062	0.081	0.048	0.045	0.015	0.021	0.015	0.015	0.077	0.102	0.063	0.060
FSV-BY	0.067	0.086	0.053	0.045								
FSV-CA												
FSV-CB												
FSV-CH												
FSV-CJ	0.109	0.138	0.081	0.068								
FSV-CK	0.174	0.196	0.107	0.152								
FSV-CL												
FSV-CM												
FSV-CO												
FSV-CP									0.095	0.131	0.077	0.067
FSV-CQ												
FSV-CS												
FSV-CT	nd	0.182	0.114	0.310	nd	0.070	0.040	0.091	nd	0.252	0.154	0.401
FSV-CU												
FSV-CV												
FSV-CY												
FSV-DC												
FSV-DE												
FSV-DL												
FSV-DM												
FSV-DS												
FSV-DW	0.045	0.043	0.026	0.027								
FSV-DY	0.091	0.147	0.082	0.093								
FSV-EA												
FSV-EB												
FSV-EC												
FSV-EF												
FSV-EI												
FSV-EJ												
FSV-EN												
FSV-EV												
FSV-EY												
FSV-FM												
n	9	10	10	10	4	5	5	5	6	7	7	7
Min	0.030	0.043	0.026	0.020	0.010	0.021	0.013	0.011	0.057	0.076	0.050	0.050
Mean	0.078	0.109	0.063	0.085	0.014	0.032	0.020	0.030	0.088	0.137	0.083	0.114
Max	0.174	0.196	0.114	0.310	0.019	0.070	0.040	0.091	0.127	0.252	0.154	0.401
SD	0.043	0.054	0.031	0.088	0.004	0.021	0.011	0.034	0.023	0.058	0.035	0.127
CV	25	27	27	28	22	30	28	38	18	23	22	32
NISTa												
NISTb	0.078	0.080	0.051	0.043	0.053	0.041	0.033	0.033	0.131	0.121	0.084	0.076
NISTc	0.067	0.078	0.047	0.039	0.023	0.030	0.019	0.017	0.090	0.108	0.066	0.056
Median	0.067	0.090	0.051	0.047	0.013	0.022	0.015	0.015	0.086	0.120	0.074	0.067
eSD	0.033	0.067	0.034	0.030	0.004	0.001	0.003	0.006	0.014	0.027	0.016	0.010
eCV	49	75	68	65	29	7	20	40	16	22	22	15

D5

Round Robin XXIV Laboratory Results

Legend

- nd* Not detected (i.e., not reported or reported as '0', 'not determined', etc.)
<x Value less than x
! Invalid sample, reason unknown
italics Value calculated from reported results
- n Number of non-NIST laboratories reporting quantitative results
Min Minimum non-NIST reported value.
Mean Average over all non-NIST reported values.
Max Maximum non-NIST reported value.
SD Standard deviation over all non-NIST values.
CV Coefficient of Variation (% relative standard deviation): $100 \cdot \text{SD} / \text{Mean}$
- Median Median over all non-NIST reported values
eSD Robust estimate of SD based on the adjusted median absolute difference from the median (MADe)
eCV Robust estimate of CV, $100 \cdot \text{eSD} / \text{Median}$

Round Robin XXIV Laboratory Results

% Bias Summary

Lab	TR	aT	g/bT	bC
FSV-BA	-2±1	2±2	1±1	2±3
FSV-BD	0±4	-3±4		
FSV-BE	-8±4	-23±7		
FSV-BF	8±6	1±3	-6±10	19±17
FSV-BG	-7±2	-13±3		-15±26
FSV-BH	-9±4	-13±1	-7±3	2±10
FSV-BI	-3±2	-4±4	2±3	-5±6
FSV-BJ	-2±3	-3±4	10±5	15±8
FSV-BK	2±2	0±2		
FSV-BL	4±6	11±10		
FSV-BM	-3±2	-1±2		
FSV-BN	4±5	12±4	-9±4	3±16
FSV-BO	19±6	22±9		-17±13
FSV-BP	17±15	21±5		23±23
FSV-BS	34±14			40±64
FSV-BX				-1±5
FSV-BY	-1±5	1±2	7±5	11±6
FSV-CA	4±3	8±3		
FSV-CB	-21±5	-4±4		
FSV-CH	12±4	7±10		20±16
FSV-CJ	11±7	5±4		1±4
FSV-CK	11±17	9±17	22±17	-21±18
FSV-CL	12±9	6±2	-67±1	-19±9
FSV-CM		0±4		
FSV-CO	-5±1	-1±1		13±7
FSV-CP				-12±2
FSV-CQ	-15±2	12±6		46±38
FSV-CS	3±2			
FSV-CT	5±69			51±62
FSV-CU	-6±3	-1±3		1±3
FSV-CV	0±1	-4±2	6±3	1±25
FSV-CY	7±4	-2±2		-1±15
FSV-DC	4±2	7±5	15±7	11±14
FSV-DE		0±2	-2±2	
FSV-DL	-2±4			-4±7
FSV-DM	25±6	-22±4		13±47
FSV-DS	-13±10	-17±3		-28±2
FSV-DW				-60±3
FSV-DY	-4±4	16±19		-12±8
FSV-EA	-1±3	10±10		
FSV-EB	2±3	-2±1		9±7
FSV-EC	0±3	9±7		10±23
FSV-EF	-5±7	1±3	9±13	
FSV-EI	-10±7	-14±3	-12±7	-40±15
FSV-EJ	7±8	-7±4	1±13	-22±45
FSV-EN	-42±9	-34±11	-30±7	
FSV-EV	3±3	6±4		
FSV-EY	6±6	10±3		
FSV-FM	-5±1	-1±6		2±11
NISTa	-10±4	-6±4	0±4	7±6
NISTb	-13±3	8±3	1±3	-9±14
NISTc	-2±2	4±3	-13±7	-4±5

Label	Definition
Lab	Participant code
TR	Total Retinol
aT	a-Tocopherol
g/bT	g/b-Tocopherol
bC	Total b-Carotene
% Bias	(Mean ± SD) of individual serum biases
Mean	Average of $(x_i - \text{Median}_i) / \text{Median}_i$
SD	Standard deviation of $(x_i - \text{Median}_i) / \text{Median}_i$
x_i	Result for analyte in serum _i
Median _i	Median of non-NIST results in serum _i

The original analysis listed % Bias for each result for each serum calculated relative to the "Trimmed Core Lab Average" of that analyte in the serum. The summary values reported here are the (arithmetic mean ± standard deviation) of each laboratory's reported results for the analyte estimated relative to each serum's median value.

Appendix E. Shipping Package Inserts for RR25

Two items were included in each package shipped to an RR25 participant:

- **Cover letter.** The original letter has been lost. It likely would have described the four sample materials (sera 164 thru 167), given guidance on reconstituting these lyophilized samples, stated that results were due June 26, 1992 and to whom they should be sent, and who to contact with technical or programmatic questions.
- **Datasheet.** The following page reproduces the form.

These items were attached to the shipping box.

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

SAMPLE #	ANALYTE	RESULT
SERUM 164 VIAL # ____	RETINOL	_____
	ALPHA-TOCOPHEROL	_____
	BETA-CAROTENE (TOTAL)	_____
SERUM 165 VIAL # ____	RETINOL	_____
	ALPHA-TOCOPHEROL	_____
	BETA-CAROTENE (TOTAL)	_____
SERUM 166 VIAL # ____	RETINOL	_____
	ALPHA-TOCOPHEROL	_____
	BETA-CAROTENE (TOTAL)	_____
SERUM 167 VIAL # ____	RETINOL	_____
	ALPHA-TOCOPHEROL	_____
	BETA-CAROTENE (TOTAL)	_____

OPTIONAL ANALYTES : SUPPLY ONE RESULT IF AVAILABLE

SERUM #	164	165	166	167
TRANS-BETA-CAROTENE	_____	_____	_____	_____
ALPHA-CAROTENE	_____	_____	_____	_____
RETINYL PALMITATE	_____	_____	_____	_____
GAMMA-TOCOPHEROL	_____	_____	_____	_____
LYCOPENE (TOTAL)	_____	_____	_____	_____
9-CIS-BETA-CAROTENE	_____	_____	_____	_____
13-CIS-BETA-CAROTENE	_____	_____	_____	_____
LUTEIN	_____	_____	_____	_____
ZEAXANTHIN	_____	_____	_____	_____
BETA-CRYPTOXANTHIN	_____	_____	_____	_____

RECONSTITUTE SERUM SAMPLES WITH 1.0 mL OF WATER.
 RESULTS DUE BY JUNE 26, 1992
 NIST FAX # 301-926 8671

Appendix F. Final Report for RR25

The following seven pages are the final report for RR25 as provided to all participants:

- A cover letter and discussion.
- Tables 1 thru 4 that list the results and various summary values for total retinol, α -tocopherol, total β -carotene, and γ/β -tocopherol.
- Tables 5 thru 13 that list the results and simple summary statistics for *trans*- β -carotene, total α -carotene, retinyl palmitate, total lutein, total lycopene, total zeaxanthin, total lutein & zeaxanthin, total β -cryptoxanthin, and *trans*-lycopene,.

Due to the complex formatting used in the Tables, the originally listed laboratory codes have been deleted without replacement. However, Appendix G provides a complete listing of the RR25 results where the original codes have been altered to ensure confidentiality. Appendix G also provides more relevant summary statistics.



NIST

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

August 3, 1992

Dear Colleague:

This report describes both overall-group and your laboratory's performance in Round Robin XXV. Specifically, your packet contains, for retinol, α -tocopherol, and β -carotene respectively: tabular presentations of all data submitted for Round-Robin XXV; a Blind Control Chart, representing a summary of your laboratory's data vs the assigned values for the past five years; a graphical presentation of data from your laboratory's analysis of eight blind replicate samples over the past four years. Tabular data only is provided for γ -tocopherol, α -carotene, trans- β carotene, β cryptoxanthin, lutein, lycopene, retinyl palmitate, and zeaxanthin.

Table 1 provides a summary of the data submitted for retinol in Round Robin XXV. Twenty-eight labs submitted data (42 in RR XXIV): 23 "Core Labs" (program participants for at least five round-robin studies); and 5 "New Labs"- 3 participating for the fourth time and 2 participating in our round-robin study for the second time. As shown in Table 1, the relative standard deviation (RSD) for all retinol measurements approached 10%, with little distinction among the "Core" and "New" Labs, for samples 164, 165 and 166. The concentration for sample 167 was ~200 ppb, and the RSD for "All Labs" was a factor of two higher than for the other three samples where there appeared to be no correlation between concentration and RSD. The Core Lab RSD (15%) was significantly less than that for the New Labs and, slightly improved from one year ago, when you analyzed this same sample (18.5%). The Core Lab RSD for retinol has ranged from 8.5-12.5 over the past two years.

Table 2 provides a summary of data submitted for the determination of α -tocopherol in Round Robin XXV. Thirty laboratories submitted data (44 in RR XXIV): 24 Core Labs; 3 four-study New Labs; 3 two-study New Labs. There was no correlation between concentration and RSD. The overall RSD for the Core Labs has remained in the 9-11% range over the past two years. The New labs performed reasonably well; their overall performance was adversely influenced by one laboratory only.

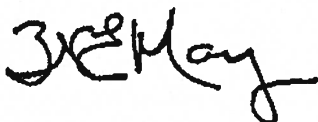
Table 3 provides a summary of data submitted for total beta-carotene. Twenty-seven laboratories submitted data: 21 Core Labs; 3 four-study New Labs; 3 two-study New Labs. As in recent studies, the overall quality of the β -carotene data is somewhat poorer than for retinol and α -tocopherol. In this study, like others over the past several years, the RSD for "All Labs" was rather high and substantially influenced by a few labs. The Core Lab RSD was 16% in the study compared to > 20% over the past two years. There is no obvious correlation between RSD and concentration above 100 ppb. For concentrations less than 100 ppb, RSD appears to increase dramatically.

Tables 4-13 provide summaries of RRRXXV data submitted for other fat-soluble vitamin and carotenoid compounds. Except for trans β -carotene, lutein and lycopene, the concentrations of the analytes in the matrix are perhaps too low to fairly assess either individual or interlaboratory measurement capabilities. Pools with elevated levels of most analytes will be available for use in RRRXXVI.

Data for your use in evaluating your laboratory's individual performance in RRXXV is provided on the right side of Tables 1-4. The "Core Lab" Trimmed Values were used as the assigned values. 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 art for these measurements. If you have concerns regarding your performance or are a lab whose performance would be rated as 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).

Round-Robin samples will be shipped during the week of August 24. Results from all three 1992 Interlaboratory Studies will be discussed at our Annual QA Workshop in early November. The actual date has not been set yet. However, as last year, we will attempt to honor your requests and convene the meeting on a Saturday. Contact us ASAP if you have any strong preferences. The date will be provided in the correspondence that accompanies RR XXVI samples. Please contact Dr. Neal Craft at 301/975-3111 if you have suggestions for Workshop topics or activities. As for the past two years, we are planning a one-day pre-workshop hands-on Fat-Soluble Vitamin and Carotenoids Analysis Session for QA Program participants. Contact Neal if you're interested.

Sincerely,



Willie E. May, Ph.D.
Chief
Organic Analytical Research Division
Chemical Science and Technology Laboratory

Appendix G. Updated “All-Lab Report” for RR25

The following four pages are an updated version of an “All-Lab” report for RR25. This report has three parts:

- pages 1 and 2 list results for all analytes reported by at least twice, counting both participants and NIST analysts.
- page 3 provides a legend for pages 1 and 2.
- page 4 summarizes each participants’ performance for total retinol, α - and γ/β -tocopherol, and total β -carotene. These summaries are compatible with the percent bias evaluation advice given in the RR25 Report. However, the current bias summaries are estimated relative to the median of all reported values for each analyte in each serum rather than to the “Trimmed Core Lab Average” used in the original and detailed in Appendix F. These original reference values were estimated from on-time results of the more experienced participants, with subjective exclusion of results deemed non-representative.

To ensure confidentiality, the laboratory identifiers used in this “All-Lab Report” have been altered from those used in RR25. 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.

Note: The analyst designated NISTa in this updated All-Lab report is designated “NIST” in the Tables described in Appendix F.

Round Robin XXV Laboratory Results

Lab	Total Retinol			Retinyl Palmitate			α-Tocopherol			γ/β-Tocopherol			Total β-Carotene			trans-β-Carotene			Total cis-β-Carotene					
	164	165	167	164	165	166	167	164	165	166	167	164	165	166	167	164	165	166	167	164	165	166	167	
FSV-BA	0.999	0.824	0.506	0.186	0.999	0.824	0.506	0.186	1.60	0.78	2.54	0.83	0.999	2.050	0.197	0.240	0.935	1.920	0.187	0.222	0.064	0.130	0.010	0.018
FSV-BD	1.114	0.910	0.516	0.190	0.083	0.065	0.033	0.003	1.45	0.90	2.50	0.85	1.105	1.736	0.146	0.249								
FSV-BE	1.047	0.968	0.511	0.188					1.50	0.70	2.40	0.70	1.124	2.491	0.157	0.334								
FSV-BF	1.155	0.901	0.532	0.226	0.071	nd	nd	nd	1.63	0.71	2.73	0.84	1.148	1.742	0.180	0.261								
FSV-BG	1.088	0.867	0.525	0.176	0.060	0.023	nd	nd	1.73	0.76	2.81	0.87	1.055	1.984	0.185	0.273								
FSV-BH	0.876	0.679	0.427	0.181	0.092	nd	nd	nd	1.75	1.01	2.91	1.13	1.085	2.001	0.186	0.264								
FSV-BI	1.136	0.914	0.557	0.193	0.089	0.042	nd	nd	1.35	0.61	2.48	0.78	1.119	1.980	0.197	0.251								
FSV-BJ	1.053	0.764	0.520	0.182	0.057	nd	nd	nd	1.54	0.77	2.90	0.94	0.955	2.035	0.174	0.275								
FSV-BK	1.069	1.047	0.549	0.208	0.042	0.010	0.010	0.010	1.418	2.077	0.186	0.343	1.418	2.077	0.186	0.343								
FSV-BL	0.905	0.797	0.484	0.166					1.612	1.502	0.386	0.294	1.612	1.502	0.386	0.294								
FSV-BM	1.217	0.879	0.559	0.166	0.042	0.010	0.010	0.010	1.057	1.947	0.192	0.289	1.057	1.947	0.192	0.289								
FSV-BN	1.108	0.936	0.620	0.283																				
FSV-BO	1.108	0.936	0.620	0.283																				
FSV-BP	1.055	0.851	0.506	0.184																				
FSV-BQ	1.055	0.851	0.506	0.184																				
FSV-BR	0.998	0.795	0.496	0.194																				
FSV-BS	0.899	0.716	0.436	0.151																				
FSV-BT	1.009	0.832	0.500	0.188																				
FSV-BU	1.255	1.124	0.559	0.219																				
FSV-BV	0.990	0.787	0.514	0.165																				
FSV-BW	1.162	0.921	0.584	0.217																				
FSV-BX	1.140	0.855	0.512	0.199																				
FSV-CU	1.024	0.853	0.507	0.197	0.087	0.037	0.040	nd	9.40	8.70	6.76	4.45	0.976	1.743	0.194	0.225								
FSV-CV	1.080	0.874	0.514	0.180					9.02	9.80	5.58	4.26	0.981	2.210	0.124	0.255								
FSV-CY	1.130	0.870	0.540	0.180					8.91	8.83	5.52	4.78	1.160	2.380	0.250	0.400								
FSV-DC	0.953	0.794	0.513	0.192					9.37	9.13	6.67	5.69	1.053	2.133	0.149	0.230								
FSV-DL	1.120	0.970	0.564	0.201					9.14	10.08	5.97	4.78	1.053	2.133	0.149	0.230								
FSV-DM	1.376	1.156	0.720	0.302					9.41	9.89	6.46	3.91	0.495	2.030	0.128	0.195								
FSV-DS	1.058	1.007	0.703	0.331					9.19	10.53	7.15	5.49	0.775	1.958	0.150	0.241								
FSV-DY	1.073	0.827	0.535	0.221					10.04	10.05	6.62	4.57	0.755	1.382	<0.05	0.150								
FSV-EB	1.070	0.957	0.547	0.218					11.20	11.42	7.56	5.96	0.885	1.960	0.169	0.232								
FSV-EC	1.179	0.961	0.556	0.201					10.29	9.45	6.32	4.15	0.866	1.408	0.180	0.231								
FSV-EJ	0.858	0.861	0.697	0.148					10.69	10.87	7.64	7.68	1.316	2.882	0.218	0.357								
FSV-EN									10.69	10.87	7.64	7.68	1.316	2.882	0.218	0.357								
n	31	31	31	31	8	5	3	2	33	33	33	33	12	12	12	12	29	29	28	29	4	4	4	4
Min	0.858	0.679	0.427	0.148	0.042	0.010	0.003	0.003	8.28	8.21	5.24	3.21	1.35	0.61	2.38	0.70	0.50	1.21	0.124	0.150	0.842	1.628	0.162	0.210
Mean	1.071	0.887	0.542	0.202	0.073	0.035	0.028	0.007	9.70	9.82	6.67	4.83	1.73	1.23	2.85	1.67	1.09	2.06	0.230	0.299	0.921	1.805	0.176	0.232
Max	1.376	1.156	0.720	0.331	0.092	0.065	0.040	0.010	15.74	12.94	10.87	7.68	2.85	5.31	4.44	7.05	2.75	4.13	1.500	1.320	0.991	1.920	0.187	0.257
SD	0.111	0.106	0.066	0.040	0.018	0.021	0.016	0.005	1.27	0.91	1.01	0.77	0.39	1.32	0.56	1.84	0.38	0.54	0.254	0.203	0.062	0.126	0.010	0.020
CV	8	9	9	12	20	32	39	49	8	7	9	10	14	25	13	26	14	13	17	15	6	7	6	8
NISTa	0.99	0.862	0.516	0.173	0.955	9.53	6.48	4.45	1.97	0.70	2.69	0.72	1.06	1.56	0.206	0.221	0.961	1.350	0.183	0.198	0.099	0.210	0.023	0.023
Median	1.070	0.870	0.525	0.192	0.077	0.037	0.033	0.007	9.37	9.80	6.53	4.78	1.62	0.76	2.77	0.86	1.05	2.00	0.180	0.261	0.064	0.127	0.011	0.017
eSD	0.098	0.098	0.036	0.018	0.020	0.021			0.55	0.89	0.47	0.49	0.19	0.13	0.40	0.12	0.16	0.38	0.025	0.042				
eCV	9	11	7	9	26	56			6	9	7	10	12	18	14	14	15	19	14	14				

Round Robin XXV Laboratory Results

Lab	Total α-Carotene			Total Lycopene			Total β-Cryptoxanthin			Total Lutein			Total Zeaxanthin			Total Lutein&Zeaxanthin					
	164	165	166	167	164	165	166	167	164	165	166	167	164	165	166	167	164	165	166	167	
FSV-BA	0.038	0.063	0.012	0.011	0.303	0.135	0.258	0.162	0.055	0.048	0.046	0.340					0.134	0.129	0.154	0.087	
FSV-BD																					
FSV-BE	0.020	0.040	0.010	0.010	0.210	0.050	0.150	0.080													
FSV-BF	0.039	0.088	nd	0.032	0.330	0.162	0.256	0.218													
FSV-BG	0.034	0.026	nd	nd	0.512	0.106	0.342	0.226													
FSV-BH	0.031	0.057	0.009	0.012	0.333	0.136	0.272	0.180	0.096	0.058	0.084	0.061					0.111	0.071	0.112	0.055	
FSV-BI	0.032	0.063	0.016	0.016	0.246	0.113	0.201	0.140	0.067	0.046	0.056	0.034					0.127	0.083	0.140	0.055	
FSV-BJ	0.023	0.039	0.012	0.015	0.163	0.070	0.142	0.085													
FSV-BK																					
FSV-BN	0.019	0.047	nd	0.016	0.161	0.111	0.160	0.122	0.037	0.034	0.029	0.017					0.094	0.076	0.113	0.056	
FSV-BO	0.038	0.068	nd	nd	0.183	0.115	0.262	0.262	0.095	0.049	0.073	0.040					0.131	0.083	0.146	0.055	
FSV-BP	0.049	0.037	0.013	0.019	0.242	0.137	0.217	0.156													
FSV-BY	0.037	0.062	0.014	0.020	0.268	0.107	0.239	0.172	0.056	0.041	0.037	0.030									
FSV-BZ																					
FSV-CA																					
FSV-CB																					
FSV-CH																					
FSV-CJ	0.024	0.029	0.008	nd	0.282	0.119	0.227	0.124	0.065	0.037	0.047	0.024					0.130	0.107	0.145	0.064	
FSV-CM																					
FSV-CO																					
FSV-CP	0.039	0.074	0.013	0.017	0.258	0.118	0.215	0.165	0.043	0.028	0.036	0.022					0.093	0.067	0.108	0.047	
FSV-CQ																					
FSV-CT																					
FSV-CU																					
FSV-CV	0.028	0.064	0.010	0.018	0.206	0.100	0.182	0.157													
FSV-CY																					
FSV-DC																					
FSV-DL	0.028	0.048	0.011	0.011	0.126	0.215	0.130	0.075	0.050	0.033	0.066	0.026					0.115	0.075	0.138	0.059	
FSV-DM																					
FSV-DS																					
FSV-DY					0.227	0.102	0.199	0.131													
FSV-EB																					
FSV-EC																					
FSV-EJ	0.041	0.069	0.016	0.020	0.338	0.215	0.220	0.206													
FSV-FEN																					

n	Total α-Carotene			Total Lycopene			Total β-Cryptoxanthin			Total Lutein			Total Zeaxanthin			Total Lutein&Zeaxanthin								
	16	16	16	17	17	17	17	17	9	9	9	9	4	4	4	3	3	3	3	3	3			
Min	0.019	0.026	0.008	0.010	0.126	0.050	0.130	0.075	0.037	0.028	0.029	0.017	0.055	0.033	0.080	0.035	0.016	0.014	0.018	0.014	0.093	0.067	0.108	0.047
Mean	0.033	0.055	0.012	0.017	0.258	0.124	0.216	0.157	0.063	0.042	0.053	0.066	0.073	0.048	0.091	0.040	0.030	0.016	0.028	0.018	0.117	0.086	0.132	0.060
Max	0.049	0.088	0.016	0.032	0.512	0.215	0.342	0.262	0.096	0.058	0.084	0.340	0.092	0.056	0.101	0.044	0.039	0.019	0.037	0.022	0.134	0.129	0.154	0.087
SD	0.008	0.017	0.003	0.006	0.091	0.043	0.054	0.052	0.021	0.010	0.019	0.104	0.015	0.010	0.011	0.005	0.012	0.003	0.010	0.006	0.016	0.021	0.018	0.012
CV	17	20	16	18	18	20	16	20	22	16	22	30	17	19	11	10	31	14	26	26	12	16	12	14

NISTA

Median	0.033	0.060	0.012	0.016	0.246	0.115	0.217	0.157	0.056	0.041	0.047	0.030	0.073	0.051	0.092	0.041	0.034	0.015	0.028	0.018	0.121	0.080	0.139	0.056
eSD	0.008	0.018	0.003	0.006	0.085	0.022	0.058	0.049	0.016	0.010	0.016	0.012									0.015	0.010	0.016	0.003
eCV	25	30	25	37	34	19	27	31	29	25	35	40									12	12	12	5

Round Robin XXV Laboratory Results

Analytes Reported By One Laboratory

Values in µg/mL

Analyte	Code	164	165	166	167
trans-Lycopene	FSV-BA	0.157	0.076	0.136	0.078

Legend

nd Not detected (i.e., not reported or reported as '0', 'not determined', etc.)

italics Value calculated from reported results

n Number of non-NIST laboratories reporting quantitative results

Min Minimum non-NIST reported value.

Mean Average over all non-NIST reported values.

Max Maximum non-NIST reported value.

SD Standard deviation over all non-NIST values.

CV Coefficient of Variation (% relative standard deviation): $100 \cdot \text{SD} / \text{Mean}$

Median Median over all non-NIST reported values

eSD Robust estimate of SD based on the adjusted median absolute difference from the median (MADe)

eCV Robust estimate of CV, $100 \cdot \text{eSD} / \text{Median}$

Round Robin XXV Laboratory Results

% Bias Summary

Lab	TR	aT	g/bT	bC
FSV-BA	-5±2	1±4	-3±4	-1±8
FSV-BD	1±3	-2±1		
FSV-BE	1±7	-2±8	-1±13	-8±11
FSV-BF	8±7	6±4	-12±5	11±19
FSV-BG	-2±4	4±2		-1±9
FSV-BH	-16±7	-4±4	-2±3	2±3
FSV-BI	4±3	1±6	2±3	2±2
FSV-BJ	-5±5	2±8	19±14	3±6
FSV-BK	8±9	2±3		
FSV-BN	-8±6	5±5	-14±5	-2±6
FSV-BO	2±12	2±4		18±17
FSV-BP	19±20	46±25		39±60
FSV-BY	-3±1	-2±2	2±6	4±6
FSV-BZ		10±18		
FSV-CA	-5±4	7±6		
FSV-CB	-18±2	-7±5		
FSV-CH	-4±1	-6±4		-5±4
FSV-CJ	17±9	-19±10		-29±11
FSV-CM		-6±6		
FSV-CO	-8±5	-3±5		4±7
FSV-CP	10±3	-14±3		-14±6
FSV-CQ	1±4	0±5		3±18
FSV-CT				3±11
FSV-CU	-2±3	-4±7		-7±10
FSV-CV	-2±3	-7±7	-5±6	-8±17
FSV-CY	1±5	-8±7		30±20
FSV-DC	-5±5	4±11	37±65	-6±11
FSV-DL	7±3	-2±5		-27±22
FSV-DM	39±13	-5±9		-13±10
FSV-DS	30±32	7±7		-34±7
FSV-DY	3±9			-9±6
FSV-EB	7±6	2±5		-15±13
FSV-EC	8±3	19±4		31±10
FSV-EJ	-3±26	-3±9	334±377	12±10
FSV-EN		26±23	135±95	351±286
NISTa	-5±4	-2±4	-1±16	-6±17

Label	Definition
Lab	Participant code
TR	Total Retinol
aT	a-Tocopherol
g/bT	g/b-Tocopherol
bC	Total b-Carotene
% Bias	(Mean ± SD) of individual serum biases
Mean	Average of $(x_i - \text{Median}_i) / \text{Median}_i$
SD	Standard deviation of $(x_i - \text{Median}_i) / \text{Median}_i$
x_i	Result for analyte in serum _i
Median _i	Median of non-NIST results in serum _i

The original analysis listed % Bias for each result for each serum calculated relative to the "Trimmed Core Lab Average" of that analyte in the serum. The summary values reported here are the (arithmetic mean ± standard deviation) of each laboratory's reported results for the analyte estimated relative to each serum's median value.

Appendix H. Shipping Package Inserts for RR26

Two items were included in each package shipped to an RR26 participant:

- **Cover letter.** The original letter has been lost. It likely would have described the three lyophilized (sera 168 thru 170) and two liquid-frozen (sera 171 and 172) sample materials, given guidance on reconstituting the lyophilized samples, stated that results were due October 16, 1992 and to whom they should be sent, and who to contact with technical questions.
- **Datasheet.** The following page reproduces the form.

These items were attached to the shipping box.

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

SAMPLE #	ANALYTE	RESULT
SERUM 168	RETINOL	_____
	ALPHA-TOCOPHEROL	_____
VIAL # ____	BETA-CAROTENE (TOTAL)	_____
SERUM 169	RETINOL	_____
	ALPHA-TOCOPHEROL	_____
VIAL # ____	BETA-CAROTENE (TOTAL)	_____
SERUM 170	RETINOL	_____
	ALPHA-TOCOPHEROL	_____
VIAL # ____	BETA-CAROTENE (TOTAL)	_____
SERUM 171	RETINOL	_____
	ALPHA-TOCOPHEROL	_____
VIAL # ____	BETA-CAROTENE (TOTAL)	_____
SERUM 172	RETINOL	_____
	ALPHA-TOCOPHEROL	_____
VIAL # ____	BETA-CAROTENE (TOTAL)	_____

OPTIONAL ANALYTES : SUPPLY ONE RESULT IF AVAILABLE

SERUM #	168	169	170	171	172
TRANS-BETA-CAROTENE	_____	_____	_____	_____	_____
ALPHA-CAROTENE	_____	_____	_____	_____	_____
RETINYL PALMITATE	_____	_____	_____	_____	_____
GAMMA-TOCOPHEROL	_____	_____	_____	_____	_____
LYCOPENE (TOTAL)	_____	_____	_____	_____	_____
9-CIS-BETA-CAROTENE	_____	_____	_____	_____	_____
13-CIS-BETA-CAROTENE	_____	_____	_____	_____	_____
LUTEIN	_____	_____	_____	_____	_____
ZEAXANTHIN	_____	_____	_____	_____	_____
BETA-CRYPTOXANTHIN	_____	_____	_____	_____	_____

RECONSTITUTE SERUM SAMPLE NUMBERS 170, 171, 172 WITH 1.0 mL OF WATER.
 RESULTS DUE BY OCTOBER 16, 1992.
 NIST FAX # 301-926 8671

Appendix I. Final Report for RR26

The following 16 pages are the final report for RR26 as provided to all participants:

- The “Summary of 1992 Round Robin Activities” that was sent to participants in November 1992.
- Tables 1a and 1b that summarize results for total retinol, α -tocopherol, and total β -carotene for the six Round Robin studies conducted from 1991 through 1992.
- Tables 4 thru 7 that list the results and various summary values for total retinol, α -tocopherol, total β -carotene, and γ/β -tocopherol. Note: Tables 2 and 3 reiterated the results provided in the RR24 and RR25 Final Reports and so are not included here.
- Tables 8 thru 16 that list the results and simple summary statistics for *trans*- β -carotene, total α -carotene, retinyl palmitate, total lutein, total lycopene, total zeaxanthin, total lutein & zeaxanthin, total β -cryptoxanthin, and *trans*-lycopene.
- Six graphical presentations of “Interlaboratory Precision vs Time” for total retinol, α -tocopherol, and total β -carotene. Three of the Figures display the precision estimated from the “core laboratory” (laboratories that participated in previous round robin studies) measurements; the other three display precision estimated after trimming results considered to be distributional outliers.

Due to the complex formatting used in the Tables 4 thru 16, the originally listed laboratory codes have been deleted without replacement. However, Appendix J provides a complete listing of the RR26 results where the original codes have been altered to ensure confidentiality. Appendix J also provides more relevant summary statistics.

SUMMARY OF 1992 ROUND ROBIN ACTIVITIES

This report describes both overall-group and individual laboratory performance in the three Round-Robin exercises conducted during 1992. Specifically, this section contains, for retinol, α -tocopherol, and β -carotene, respectively: both core and trimmed graphical information concerning Interlaboratory Precision vs Time over the past 6 1/2 years; for historical purposes, a summary table is also provided for data collected over the past two years; tabular presentations of both individual laboratory and summary statistical data for Round Robins XXIV, XXV and XXVI; a Blind Control Chart representing a summary of each laboratory's data vs assigned values for the past five years; a graphical presentation of data from each laboratory's analysis of blind replicate samples over the past two years. Tabular data only are provided for α -carotene, trans β -carotene, β -cryptoxanthin, lutein, lycopene, retinyl palmitate, γ -tocopherol and zeaxanthin.

Table 1 provides a summary of interlaboratory data for retinol, α -tocopherol and β -carotene over the past two years. The mean relative standard deviation (RSD; standard deviation expressed as a %) for retinol Core Laboratories (Laboratories that had been involved in the Quality Assurance (QA) program for at least one year prior to Round-Robin XXI) has averaged 10.7% over the past six Round Robin exercises with a range of 8.7-12.7%. New Laboratory RSD values have decreased steadily from approximately 65% in Round Robin XXII to values indistinguishable from Core Lab values in the past two studies.

The mean RSD for α -tocopherol of Core Laboratories has averaged 9.6% with a range of 8.6-10.6% over the past six Round Robin exercises. During the same period, New Laboratory RSD values have decreased from 54% in Round Robin XXII to approximately 20% in Round Robin's XXIII-XV, and to less than 10% in Round Robin XXVI.

The mean RSD for β -carotene of Core Laboratories (using data for all sera with concentrations higher than 200 ng/mL) averaged 24% during 1991 and was reduced to 18% during 1992. The New Laboratory mean RSD during this period was 41% with a range between 17% (for RR XXVI) and 82% showing no particular trend.

Tables 2 and 3 provide a listing and statistical summary of data submitted in Round Robins XXIV and XXV respectively, for retinol (42 labs), α -tocopherol (41 labs) and β -carotene (33 labs). The "Core Lab Trimmed Averages" were used as Assigned Values for these samples.

Five Serum samples (168-172) were distributed for analysis in Round Robin XXVI. Samples 168 and 169 were liquid samples and 170-172 were lyophilized. Sera 170 and 172 were blind duplicates. Table 4 provides a summary of data submitted for retinol. Thirty-three labs submitted data: 26 "Core Labs" and 7 "New Labs" -- 3 participating for the fifth time and 4 participating for the third time. Data for use in evaluating laboratory performance is provided to the right side of the Table. The "Core Lab" Trimmed Values were used as the assigned values. 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 art.

Tables 5 and 6 provide Round Robin XXVI data for α -tocopherol and β -total carotene respectively. Thirty-five labs submitted data for α -tocopherol; 28 "Core Labs" and 7 "New Labs". Thirty labs submitted data for total β -carotene; 23 "Core Labs" and 7 "New Labs". The erratic nature of the data being reported for β -carotene continues to cause us concern. Therefore we have initiated extensive laboratory investigations focused on problems associated with the extraction and HPLC analysis of carotenoid compounds, with special emphasis on β -carotene. The results from some of those studies are presented later in this report.

Tables 7-16 provide summaries of data reported in Round Robin XXVI for other fat-soluble vitamin and carotenoid compounds. During the coming year, special efforts will be made to improve the measurement quality of retinyl palmitate and additional carotenoid compounds.

If labs have concerns regarding their performance or a lab was rated "U" based on the convention stated above, we suggest that they obtain a unit of SRM 968a and analyze all three levels. If, with minor method modifications, their measured values do not agree with the certified values, they contact Dr. Neal E. Craft at 301/975-3111 for consultation. We are willing to provide in-house consultation (at the labs expense) if need be, but have found that most problems can be solved via telephone conversations.

For non-NCI funded labs, fees of \$300 for US labs and \$600 for non-US labs will be assessed for participating in the "Fat-Soluble Vitamins QA 1993" program. An invoice to that effect will be mailed to those laboratories.

The 1993 QA Program will consist of three Round Robin exercises. The first set of samples will be distributed the week of January 25 with results due March 19; written feed back will be provided to labs by April 26. The second set of samples will be shipped the week of April 26 with results due by June 14 and feedback to labs by July 26. The third set of samples will be shipped the week of July 26. Results will be due by August 30 so that we can discuss all three exercises at the Annual QA Workshop that is being planned for late September or early October. The actual date has not been set yet. Feedback to participants who are not able to attend the QA Workshop will be provided by November 15.

Table 1b

		ROUND ROBIN XXXIV RETINOL			ROUND ROBIN XXV RETINOL			ROUND ROBIN XXVI RETINOL		
	SAMPLE #	SAMPLE #	SAMPLE #	SAMPLE #	SAMPLE #	SAMPLE #	SAMPLE #	SAMPLE #	SAMPLE #	SAMPLE #
	160	161	162	163	164	165	166	167	168	170
GRAND (42) RSD	1,002 24.2	0,681 15.0	0,268 16.2	0,387 13.6	1,074 10.2	0,685 11.5	0,546 12.2	0,202 20.2	0,549 10.6	0,644 10.7
CORE (28) RSD	1,012 11.2	0,678 7.7	0,293 7.0	0,388 7.9	1,078 10.2	0,685 12.2	0,539 11.5	0,187 14.9	0,548 11.6	0,644 12.1
NEW (13) RSD	1,069 30.2	0,703 23.8	0,282 32.7	0,393 21.4	1,037 13.1	0,865 13.2	0,560 19.0	0,224 35.2	0,559 7.9	0,648 4.7
ASSIGNED RSD	1,019 4.4	0,673 6.9	0,293 7.0	0,383 8.7	1,085 6.2	0,872 8.8	0,525 4.1	0,194 8.3	0,546 4.6	0,639 7.3
PREVIOUS DATE	10/10 08/91	0,659 05/91	0,281 05/90	0,368 01/88	1,022 07/88	0,848 01/80	0,515 05/80	0,184 05/81	0,553 10/80	0,827 10/80
a - TOCOPHEROL										
GRAND (41) RSD	6,23 15.6	15,95 10.8	4,61 13.1	5,64 12.9	9,70 13.3	9,82 9.0	4,61 13.1	5,64 12.9	10,59 9.6	9,82 8.3
CORE (30) RSD	6,24 14.0	16,12 7.4	4,63 11.9	5,66 9.2	9,45 8.8	9,59 8.8	4,63 11.9	5,66 9.2	10,41 8.7	9,85 8.8
NEW (11) RSD	6,21 21.0	15,41 17.8	4,48 32.0	5,55 20.6	10,81 24.4	10,86 11.6	7,60 22.2	5,51 21.8	11,24 11.6	9,70 6.6
ASSIGNED RSD	6,39 5.1	15,95 6.5	4,61 6.7	5,66 7.0	9,38 5.6	9,59 5.5	6,36 7.2	4,66 6.8	10,33 4.6	9,83 5.1
PREVIOUS DATE	08/81	05/91	05/80	01/88	07/88	01/80	05/80	05/81	10/80	10/80
b - CAROTENE										
GRAND (32) RSD	0,100 51.2	2,205 32.9	1,016 22.0	0,183 21.5	1,087 35.7	2,038 25.2	0,226 113	0,300 89.7	1,662 17.8	0,439 22.2
CORE (21) RSD	0,098 51.6	2,223 29.4	1,082 17.9	0,185 12.9	0,998 17.3	1,989 13.5	0,172 16.8	0,264 16.6	1,710 15.6	0,447 23.9
NEW (12) RSD	6,21 21.0	15,41 17.8	4,48 32.0	5,55 20.6	1,405 52.2	2,247 44.2	0,415 131	0,435 101	1,518 23.9	0,415 14.5
ASSIGNED RSD	0,090 11.0	2,212 9.1	1,073 7.8	0,185 12.9	1,023 12.9	2,030 11.7	0,169 14.0	0,256 8.2	1,716 9.0	0,456 14.3
PREVIOUS DATE	08/81	05/81	05/80	01/88	07/88	01/80	05/80	05/81	10/80	10/80
a - TOCOPHEROL										
GRAND (35) RSD	10,59 9.6	9,82 8.3	6,25 10.1	4,96 11.5	10,59 9.6	9,82 8.3	6,25 10.1	4,96 11.5	10,59 9.6	9,82 8.3
CORE (28) RSD	10,41 8.7	9,85 8.8	6,21 10.3	4,93 12.1	10,41 8.7	9,85 8.8	6,21 10.3	4,93 12.1	10,41 8.7	9,85 8.8
NEW (7) RSD	11,24 11.6	9,70 6.6	6,39 9.7	5,08 9.5	11,24 11.6	9,70 6.6	6,39 9.7	5,08 9.5	11,24 11.6	9,70 6.6
ASSIGNED RSD	10,33 4.6	9,83 5.1	6,23 6.9	6,03 9.7	10,33 4.6	9,83 5.1	6,23 6.9	6,03 9.7	10,33 4.6	9,83 5.1
PREVIOUS DATE	10/80	10/80	10/80	10/80	10/80	10/80	10/80	10/80	10/80	10/80
b - CAROTENE										
GRAND (30) RSD	1,662 17.8	0,439 22.2	0,621 17.5	0,400 18.8	1,662 17.8	0,439 22.2	0,621 17.5	0,400 18.8	1,662 17.8	0,439 22.2
CORE (23) RSD	1,710 15.6	0,447 17.4	0,642 14.6	0,418 15.3	1,710 15.6	0,447 17.4	0,642 14.6	0,418 15.3	1,710 15.6	0,447 17.4
NEW (7) RSD	1,518 23.9	0,415 14.5	0,553 17.3	0,348 19.3	1,518 23.9	0,415 14.5	0,553 17.3	0,348 19.3	1,518 23.9	0,415 14.5
ASSIGNED RSD	1,716 9.0	0,456 14.3	0,617 11.9	0,416 14.6	1,716 9.0	0,456 14.3	0,617 11.9	0,416 14.6	1,716 9.0	0,456 14.3
PREVIOUS DATE	10/80	10/80	10/80	10/80	10/80	10/80	10/80	10/80	10/80	10/80

Table 4

Round Robin XXVI Retinol Results						% Bias from Trimmed Core Lab Average.					
Lab#	Serum# 168	Serum# 169	Serum# 170	Serum# 171	Serum# 172	Lab#	Serum# 168	Serum# 169	Serum# 170	Serum# 171	Serum# 172
	0.551	0.638	0.685	0.562	0.684		0.9	-0.2	4.2	4.5	3.3
	0.517	0.601	0.606	0.505	0.617		-5.3	-6.0	-7.8	-6.1	-6.8
	0.576	0.661	0.670	* 0.609	0.692		5.5	3.4	1.9	13.3	4.5
	0.541	0.652	0.652	0.572	0.665		-0.9	2.0	-0.8	6.4	0.4
*	0.480	0.573	0.602	0.504	0.596		-12.1	-10.4	-8.4	-6.3	-10.0
	0.513	0.591	0.634	0.508	0.627		-6.0	-7.5	-3.6	-5.6	-5.4
	0.554	0.633	0.666	0.536	0.671		1.5	-1.0	1.3	-0.3	1.3
*	0.463	0.544	0.618	0.491	0.637		-15.2	-14.9	-6.0	-8.7	-3.8
	0.565	0.705	* 0.762	0.593	0.676		3.5	10.3	15.9	10.3	2.1
	0.596	0.733	0.705	0.578	0.687		9.2	14.7	7.2	7.5	3.8
	0.495	0.621	0.643	0.523	0.665		-9.3	-2.9	-2.2	-2.7	0.4
	0.539	0.629	0.649	0.533	0.645		-1.3	-1.6	-1.3	-0.9	-2.6
	0.570	0.616	0.668	0.550	0.666		4.4	-3.6	1.6	2.3	0.6
	0.574	0.662	0.647	0.526	0.653		5.2	3.6	-1.6	-2.2	-1.4
	0.550	0.620	0.610	0.550	0.620		0.8	-3.0	-7.2	2.3	-6.4
*	0.409	* 0.482	0.695	0.534	0.723		-25.1	-24.6	5.7	-0.7	9.2
	0.525	0.606	0.680	0.542	0.735		-3.8	-5.2	3.4	0.8	11.0
	0.556	0.632	0.648	0.548	0.599		1.9	1.1	-1.4	1.9	-9.5
*	0.613	0.707	0.691	0.545	0.684		12.3	10.6	5.0	1.3	3.2
	0.516	0.635	0.668	0.557	0.665		-5.5	-0.7	1.6	3.6	0.4
	0.533	0.618	0.594	0.493	0.603		-2.4	-3.3	-9.6	-8.3	-8.9
	0.555	0.711	0.655	0.521	0.666		1.7	11.2	-0.4	-3.1	0.6
			0.690	0.552	0.692				5.0	2.7	4.5
	0.521	* 0.336	0.719	* 0.617	0.677		-4.6	-47.4	9.4	14.7	2.3
	0.545	0.675	0.683	0.546	0.709		-0.2	5.6	3.9	1.5	7.1
*	0.759	* 0.901	* 0.769	* 0.632	* 0.793		39.1	41.0	17.0	17.5	19.8
	0.630	0.678	0.770	0.630	0.767		15.4	6.1	17.1	17.2	15.8
	0.586	0.666	0.697	0.591	0.721		7.4	4.2	6.0	9.9	8.9
	0.556	0.621	0.683	0.555	0.661		1.9	-2.9	3.9	3.2	-0.2
	0.525	0.638	0.642	0.496	0.674		-3.8	-0.2	-2.3	-7.9	1.7
	0.540	0.600	0.680	0.530	0.650		-1.1	-6.1	3.4	-1.4	-1.8
	0.512	0.649	0.613	0.510	0.609		-6.2	1.5	-6.8	-5.2	-8.0
	0.566	0.681	0.673	0.524	0.679		3.7	6.5	2.4	-2.6	2.5
NIST	0.551	0.664	0.688	0.514	0.721						
All Labs AVG (33)	0.549	0.644	0.669	0.547	0.670	Core Labs Trimmed (15-101;26)					
SD	0.058	0.069	0.045	0.038	0.045	AVG	0.546	0.639	0.657	0.538	0.662
RSD	10.4	10.7	6.7	7.0	6.8	SD	0.025	0.047	0.034	0.027	0.037
Core Labs (15-101;26)						RSD	4.6	7.3	5.2	4.9	5.6
AVG	0.546	0.644	0.666	0.547	0.667	PREVIOUS					
SD	0.063	0.078	0.044	0.037	0.045	VALUE	0.553	0.627			
RSD	11.6	12.1	6.6	6.7	6.7						
(a) New Labs(105-110;3)											
AVG	0.591	0.695	0.717	0.592	0.716	* = Value removed for Core Lab Trimmed Average.					
SD	0.037	0.030	0.047	0.038	0.053	(a) = participating for the fifth time.					
RSD	6.3	4.6	6.5	6.3	7.4	(b) = participating for the third time.					
(b) New Labs(116-120;4)											
AVG	0.536	0.642	0.652	0.515	0.653	Samples 170 and 172 are the same Sers.					
SD	0.023	0.033	0.031	0.015	0.032						
RSD	4.3	5.2	4.7	3.0	4.9						

Table 5

Round Robin XXVI Alpha-Tocopherol						% Bias from Trimmed Core Lab Average					
Lab#	Serum# 168	Serum# 169	Serum# 170	Serum# 171	Serum# 172	Lab#	Serum# 168	Serum# 169	Serum# 170	Serum# 171	Serum# 172
	10.04	9.42	6.12	5.06	6.17		-2.8	-4.2	-1.7	0.5	0.6
	10.05	9.49	5.93	4.82	5.94		-2.7	-3.5	-4.8	-4.3	-3.2
	11.02	10.09	6.35	5.44	6.55		6.7	2.7	2.0	8.1	6.7
	10.41	9.64	6.74	5.96	6.72		0.8	-1.9	8.2	18.4	9.5
	9.47	9.17	6.08	5.00	5.96		-8.3	-6.7	-2.4	-0.7	-2.8
	9.98	9.34	5.53	4.55	5.37		-3.4	-5.0	-11.3	-9.7	-12.5
	9.80	10.50	6.70	5.30	6.40		-5.1	6.8	7.6	5.3	4.3
	10.48	10.06	7.02	5.31	6.78		1.5	2.3	12.7	5.5	10.5
	10.53	10.39	6.94	4.90	6.21		2.0	5.7	11.5	-2.7	1.2
	10.59	9.92	5.92	4.40	5.53		2.5	0.9	-4.9	-12.6	-9.9
	10.10	9.59	* 7.55	6.03	* 7.15		-2.2	-2.4	21.3	19.8	16.6
	10.39	10.00	6.31	5.20	6.32		0.6	1.7	1.3	3.3	3.0
	10.92	9.15	6.57	4.95	6.08		5.7	-6.9	2.3	-1.7	-0.9
	11.07	10.55	6.36	4.88	6.28		7.2	7.3	2.1	-3.1	2.4
	* 11.50	* 11.00	7.00	5.20	6.90		11.4	11.9	12.4	3.3	12.5
	9.37	8.84	5.91	4.87	5.78		-9.3	-10.1	-5.1	-3.3	-5.8
	10.42	9.73	5.81	4.16	6.10		0.9	-1.0	-6.7	-17.4	-0.6
	* 12.20	10.34	* 7.24	5.95	* 7.33		18.1	5.2	16.3	18.2	19.5
	10.91	10.56	6.15	5.05	6.25		5.6	7.5	-1.2	0.2	1.9
	10.25	9.87	5.85	4.61	5.70		-0.8	0.4	-6.1	-8.5	-7.1
	10.60	9.56	6.74	5.50	6.74		2.6	-2.7	8.2	9.2	9.9
	10.26	9.60	5.97	4.99	5.92		-0.6	-2.4	-4.1	-1.0	-3.4
	* 7.64	* 7.13	* 4.54	* 3.79	* 4.66		-26.0	-27.5	-27.0	-24.8	-24.1
	10.80	10.61	6.06	4.68	6.04		4.6	8.0	-2.8	-7.1	-1.6
			5.71	4.54	5.64				-8.3	-9.8	-8.1
	9.84	* 4.92	6.15	5.25	6.24		-4.7	-49.9	-1.2	4.3	1.7
	* 12.24	* 11.95	5.73	* 3.49	5.62		18.5	21.6	-8.0	-30.6	-8.4
	9.74	9.66	* 5.14	4.31	* 5.14		-5.7	-1.8	-17.4	-14.3	-16.2
	12.20	9.60	6.19	4.87	6.63		18.1	-2.3	-0.6	-3.3	8.1
	11.24	9.62	6.72	5.28	6.28		8.8	-2.1	7.9	4.9	2.4
	12.20	10.83	7.17	5.74	7.14		18.1	10.2	15.1	14.0	16.4
	12.90	9.44	7.11	5.68	7.11		24.9	-3.8	14.1	12.8	15.9
	9.13	8.65	5.68	4.58	5.70		-11.6	-12.0	-8.8	-9.0	-7.1
	10.41	9.86	6.09	4.71	5.91		0.8	0.3	-2.2	-6.5	-3.6
	10.58	9.89	5.74	4.71	5.94		2.5	0.6	-7.9	-6.5	-3.2
NIST	10.05	9.88	6.47	4.78	6.19						
All Labs											
AVG (35)	10.59	9.82	6.25	4.96	6.18	Core Labs Trimmed (15-101;28)					
SD	1.03	0.82	0.63	0.57	0.59	AVG	10.33	9.83	6.23	5.03	6.13
RSD	9.8	8.3	10.1	11.5	9.6	SD	0.48	0.50	0.43	0.49	0.41
Core Labs (15-101;28)						RSD	4.6	5.1	6.9	9.7	6.8
AVG	10.41	9.85	6.21	4.93	6.13	PREVIOUS					
SD	0.90	0.86	0.64	0.60	0.60	VALUE	10.46	9.65			
RSD	8.7	8.8	10.3	12.1	9.7						
(a) New Labs (105-110;3)											
AVG	11.88	10.02	6.69	5.30	6.68						
SD	0.55	0.70	0.49	0.44	0.43						
RSD	4.7	7.0	7.3	8.2	6.5						
(b) New Labs (116-120;4)											
AVG	10.76	9.46	6.15	4.92	6.17						
SD	1.57	0.58	0.66	0.51	0.64						
RSD	14.6	6.1	10.7	10.4	10.4						

* = Value removed for Core Lab Trimmed Average.

(a) = participating for the fifth time.

(b) = participating for the third time.

Samples 170 and 172 are the same Sers.

Table 6

Round Robin XXVI Total Beta-Carotene						% Bias from Trimmed Core Lab Average.					
Lab#	Serum# 168	Serum# 169	Serum# 170	Serum# 171	Serum# 172	Lab#	Serum# 168	Serum# 169	Serum# 170	Serum# 171	Serum# 172
	1.664	0.436	0.668	0.461	0.684		-3.0	-4.4	8.3	10.8	16.4
	1.633	0.412	0.540	0.361	0.549		-4.8	-9.7	-12.5	-13.2	-6.6
	1.420	0.473	0.711	0.504	0.565		-17.2	3.7	15.3	21.2	-3.9
	1.819	0.500	0.626	0.389	0.391		6.0	9.6	1.5	-6.5	0.6
	1.727	0.453	0.593	0.408	0.583		0.7	-0.7	-3.9	-1.9	-0.8
	* 2.221	0.548	* 0.996	0.525	* 0.886		29.4	20.1	61.5	26.2	50.8
	1.490	* 0.102	0.724	0.374	0.699		-13.2	-77.6	17.4	-10.1	18.9
	1.984	* 0.623	0.692	0.480	* 0.713		15.6	36.6	12.2	15.4	21.3
	1.797	0.464	0.617	0.399	0.568		4.7	1.7	0.0	-4.1	-3.4
	1.570	0.425	0.565	0.376	0.552		-8.5	-6.8	-11.6	-9.6	-6.1
	1.760	0.381	0.626	0.408	0.602		2.6	-16.5	1.5	-1.9	2.4
	1.830	0.554	0.736	0.471	0.621		6.7	21.5	19.3	13.2	5.7
	1.890	0.470	0.580	0.360	0.550		10.2	3.0	-6.0	-13.5	-6.4
	* 1.087	0.336	0.517	0.379	0.538		-36.6	-26.3	-16.2	-8.9	-6.5
	1.876	0.489	0.658	0.425	0.651		9.3	7.2	6.7	2.2	10.8
	1.770	0.514	* 0.806	0.522	* 0.817		3.2	12.7	30.7	25.5	39.0
	1.654	0.417	0.545	0.378	0.498		-3.6	-8.6	-11.6	-9.1	-15.3
	1.505	0.373	0.550	0.342	0.567		-12.3	-18.2	-10.9	-17.8	-3.5
	* 1.276	0.371	0.673	0.455	0.651		-25.6	-18.7	9.1	9.4	10.8
			0.493	0.312	0.545				-20.1	-25.0	-7.3
	1.580	* 0.230	0.586	0.411	0.579		-7.9	-49.6	-5.0	-1.3	-1.5
	1.780	0.484	0.564	0.343	0.573		3.7	6.1	-8.6	-17.5	-2.5
	* 2.149	0.566	0.710	0.485	* 0.736		25.2	24.1	15.1	16.6	25.2
	1.152	0.346	0.461	0.296	0.499		-32.9	-24.1	-25.3	-28.8	-15.1
	1.813	0.433	0.623	0.424	0.666		5.7	-5.1	1.0	1.9	13.3
	2.108	0.467	0.608	0.287	0.722		22.9	2.3	-1.5	-31.1	22.8
	1.085	0.495	0.541	0.377	0.549		-36.8	8.5	-12.3	-9.5	-6.7
	1.550	0.396	0.597	0.381	0.570		-9.7	-13.2	-3.2	-8.4	-3.0
	1.576	0.432	0.580	0.397	0.550		-8.1	-5.3	-6.0	-4.6	-6.4
	1.345	0.333	0.459	0.275	0.389		-21.6	-27.0	-25.6	-33.9	-33.8
NIST	1.590	0.447	0.661	0.482	0.610						
All Labs						Core Labs Trimmed (15-101;23)					
AVG (30)	1.662	0.439	0.621	0.400	0.609	AVG	1.716	0.456	0.617	0.416	0.588
SD	0.298	0.097	0.109	0.066	0.100	SD	0.155	0.065	0.074	0.061	0.052
RSD	17.9	22.2	17.5	16.6	16.4	RSD	9.0	14.3	11.9	14.6	8.9
Core Labs (15-101;23)						PREVIOUS VALUE					
AVG	1.710	0.447	0.642	0.416	0.623	VALUE	1.618	0.441			
SD	0.266	0.107	0.112	0.061	0.095						
RSD	15.6	23.9	17.4	14.6	15.3						
(a) New Labs (108-114;3)						* = Value removed for Core Lab Trimmed Average.					
AVG	1.691	0.415	0.564	0.336	0.629	(a) = participating for the fifth time.					
SD	0.490	0.062	0.089	0.077	0.116	(b) = participating for the third time.					
RSD	29.0	15.0	15.9	22.9	18.4	Samples 170 and 172 are the same Sera.					
(b) New Labs (116-120;4)											
AVG	1.389	0.414	0.544	0.357	0.514						
SD	0.227	0.068	0.061	0.056	0.084						
RSD	16.4	16.4	11.3	15.6	16.4						

Table 7 Round Robin XXVI

Gamma-Tocopherol					
LAB#	Serum# 168	Serum# 169	Serum# 170	Serum# 171	Serum# 172
	1.53	2.20	2.25	2.28	2.27
	1.48	2.23	2.05	2.08	2.07
	1.30	2.11	2.14	2.00	2.06
	1.62	2.55	2.36	2.40	2.28
	1.22	2.91	2.30	2.41	2.10
	1.46	2.16	1.92	1.91	1.91
	1.29	1.85	1.79	1.79	1.74
	1.34	2.09	1.83	1.83	* 2.76
	1.50	2.58	2.05	2.20	2.11
	* 1.96	2.96	* 2.64	2.64	2.62
	* 1.81	3.03	2.16	2.15	2.22
	1.27	2.03	1.85	2.00	1.91
AVG	1.482	2.392	2.112	2.140	2.170
SD	0.226	0.401	0.251	0.257	0.291
RSD	15.2	16.8	11.9	12.0	13.4
NIST 1	1.23	2.19	1.93	1.91	2.17
NIST 3	1.51	2.24	2.02	2.03	1.88
Trimmed			*****	*****	*****
AVG	1.401	2.392	2.064	2.140	2.116
SD	0.133	0.401	0.197	0.257	0.234
RSD	9.5	16.8	9.5	12.0	11.1

* = Value removed for Trimmed Lab Average.

% Bias from Trimmed Lab Average.

LAB#	168	169	170	171	172
	9.1	-8.2	8.9	6.3	7.2
	5.6	-6.8	-0.7	-2.8	-2.2
	-7.2	-11.8	3.7	-6.6	-2.7
	15.6	6.6	14.3	12.1	7.7
	-12.9	21.7	11.4	12.6	-0.8
	4.2	19.7	-7.0	-10.8	-9.7
	-7.9	-22.6	-13.3	-16.4	-17.8
	-4.4	-12.6	-11.3	-14.5	30.4
	7.1	7.9	-0.5	2.8	-0.5
	39.9	23.8	27.9	23.3	23.8
	29.2	26.7	4.7	0.4	4.9
	-9.1	-15.0	-10.2	-6.6	-10.0

Samples 170 and 172 are the same Sera.

Table 8 Round Robin XXVI

Trans-Beta-Carotene					
LAB#	Serum# 168	Serum# 169	Serum# 170	Serum# 171	Serum# 172
56	1.510	0.392	0.512	0.346	0.516
60	1.694	0.365	0.592	0.385	0.570
92	1.625	0.383	0.514	0.345	0.471
110	1.848	0.369	0.620	0.429	0.600
AVG	1.669	0.377	0.560	0.376	0.539
SD	0.141	0.012	0.055	0.040	0.057
RSD	8.5	3.3	9.8	10.6	10.6
NIST 1	1.450	0.403	0.591	0.352	0.586
NIST 3	1.56	0.388	0.567	0.382	0.518
PREVIOUS VALUE	1.490	0.401			

Table 9 Round Robin XXVI

Alpha-Carotene					
LAB#	Serum# 168	Serum# 169	Serum# 170	Serum# 171	Serum# 172
15	0.109	0.062	0.024	0.015	0.025
16	0.088	0.058	0.021	0.013	0.020
19	0.063	0.042	0.020	0.013	0.019
17	0.027	0.026			
32	0.115	0.079	0.052		0.033
46	0.088	0.052	0.044	0.017	0.029
56	0.099	0.062	0.028	0.013	0.029
60	0.098	0.049	0.022	0.013	0.022
73	0.092	0.059	0.032	0.021	0.029
86	0.072	0.048	0.018	0.012	0.018
89	0.086	0.056	0.026	0.016	0.026
93	0.125	0.071	0.028	0.016	0.029
95			0.019	0.011	0.020
97	0.114	0.088	0.034	0.039	0.035
108	0.066	0.047	0.016	0.013	0.020
110	0.078	0.048	0.023	0.012	0.025
116	0.067	0.053	0.015		0.023
AVG	0.087	0.056	0.026	0.016	0.025
SD	0.025	0.015	0.010	0.007	0.005
RSD	28.3	26.1	38.3	44.4	20.7
NIST 1	0.102	0.057	0.037	0.017	0.025
NIST 3	0.110	0.066	0.034	0.027	0.035

Table 10 Round Robin XXVI
Retinyl Palmitate

LAB#	Serum# 168	Serum# 169	Serum# 170	Serum# 171	Serum# 172
	0.166	0.044	0.259	0.136	0.262
	0.297	0.085	0.372	0.207	0.374
	0.223	0.045	0.312	0.201	0.270
	0.220	0.076	0.302	0.184	0.296
	0.160	0.038	0.227	0.154	0.240
	0.247	0.084	0.296	0.193	0.282
	0.375	0.158	0.474	0.330	0.471
	0.237	0.064	0.317	0.222	0.352
AVG	0.241	0.074	0.320	0.203	0.318
SD	0.070	0.039	0.075	0.058	0.077
RSD	29.0	52.0	23.6	28.7	24.0
NIST 3	.217	.059	.297	.185	.261
PREVIOUS VALUE	0.192	0.069			

Table 11 Round Robin XXVI
Lutein

LAB#	Serum# 168	Serum# 169	Serum# 170	Serum# 171	Serum# 172
	0.129	0.109	0.109	0.047	0.111
	0.157	0.137	0.112	0.046	0.111
	0.151	0.102	0.109	0.045	0.115
	0.095	0.081	0.075	0.031	0.073
AVG	0.133	0.107	0.101	0.042	0.103
SD	0.028	0.023	0.018	0.008	0.020
RSD	21.1	21.6	17.3	17.9	19.3
NIST 3	.145	.112	.110	.046	.105

Table 12 Round Robin XXVI

Lycopene					
LAB#	Serum# 168	Serum# 169	Serum# 170	Serum# 171	Serum# 172
	0.340	0.381	0.238	0.202	0.242
	0.294	0.290	0.169	0.140	0.170
	0.398	0.392	0.264	0.334	0.221
	0.270	0.265	0.188	0.158	0.190
	0.472	0.522	0.342	0.246	0.304
	0.440	0.450	0.300	0.230	0.270
	0.338	0.276	0.192	0.156	0.183
	0.215	0.249	0.183	0.175	0.199
	0.187	0.186	0.123	0.101	0.124
	0.362	0.384	0.230	0.196	0.222
	0.436	0.466	0.269	0.220	0.286
			0.124	0.101	0.143
	0.394	0.332	0.233	0.196	0.237
	0.202	0.200	0.139	0.132	0.155
	0.183	0.170	0.140	0.110	0.124
	0.209	0.329	0.182	0.159	0.199
AVG	0.316	0.326	0.207	0.179	0.204
SD	0.101	0.106	0.065	0.061	0.055
RSD	31.8	32.5	31.1	34.2	26.7
NIST 3	.451	.422	.250	.225	.234

Table 13 Round Robin XXVI

Zeaxanthin					
LAB#	Serum# 168	Serum# 169	Serum# 170	Serum# 171	Serum# 172
	0.038	0.036	0.043	0.023	0.043
	0.034	0.033	0.040	0.027	0.037
	0.017	0.018	0.023	0.012	0.020
AVG	0.030	0.029	0.035	0.021	0.033
SD	0.011	0.010	0.011	0.008	0.012
RSD	37.6	33.3	30.5	37.6	35.8
NIST 3	.041	.040	.051	.026	.048

Table 14 Round Robin XXVI

Lutein + Zeaxanthin					
LAB#	Serum# 168	Serum# 169	Serum# 170	Serum# 171	Serum# 172
	0.186	0.159	0.149	0.071	0.151
	0.212	0.182	0.182	0.080	0.189
	0.227	0.198	0.189	0.083	0.187
	0.341	0.263	0.169	0.079	0.157
	0.126	0.107	0.101	0.044	0.097
	0.128	0.249	0.230	0.092	0.233
AVG	0.203	0.193	0.168	0.074	0.168
SD	0.079	0.058	0.040	0.015	0.042
RSD	39.1	30.0	23.6	20.4	24.9

Table 15 Round Robin XXVI

Beta-Cryptoxanthin					
LAB#	Serum# 168	Serum# 169	Serum# 170	Serum# 171	Serum# 172
	0.118	0.099	0.064	0.041	0.067
	0.179	0.146	0.086	0.051	0.087
	0.124	0.107	0.074	0.045	0.072
	0.149	0.120	0.070	0.044	0.073
	0.130	0.113	0.062	0.036	0.062
			0.073	0.048	0.077
	0.200	0.150	0.100	0.058	0.097
	0.111	0.097	0.073	0.043	0.074
	0.108	0.085	0.055	0.032	0.059
	0.067	0.177	0.065		
AVG	0.132	0.122	0.072	0.044	0.074
SD	0.040	0.030	0.013	0.008	0.012
RSD	30.1	24.6	17.8	17.5	16.0
MIST 3	.109	.087	.060	.034	0.054

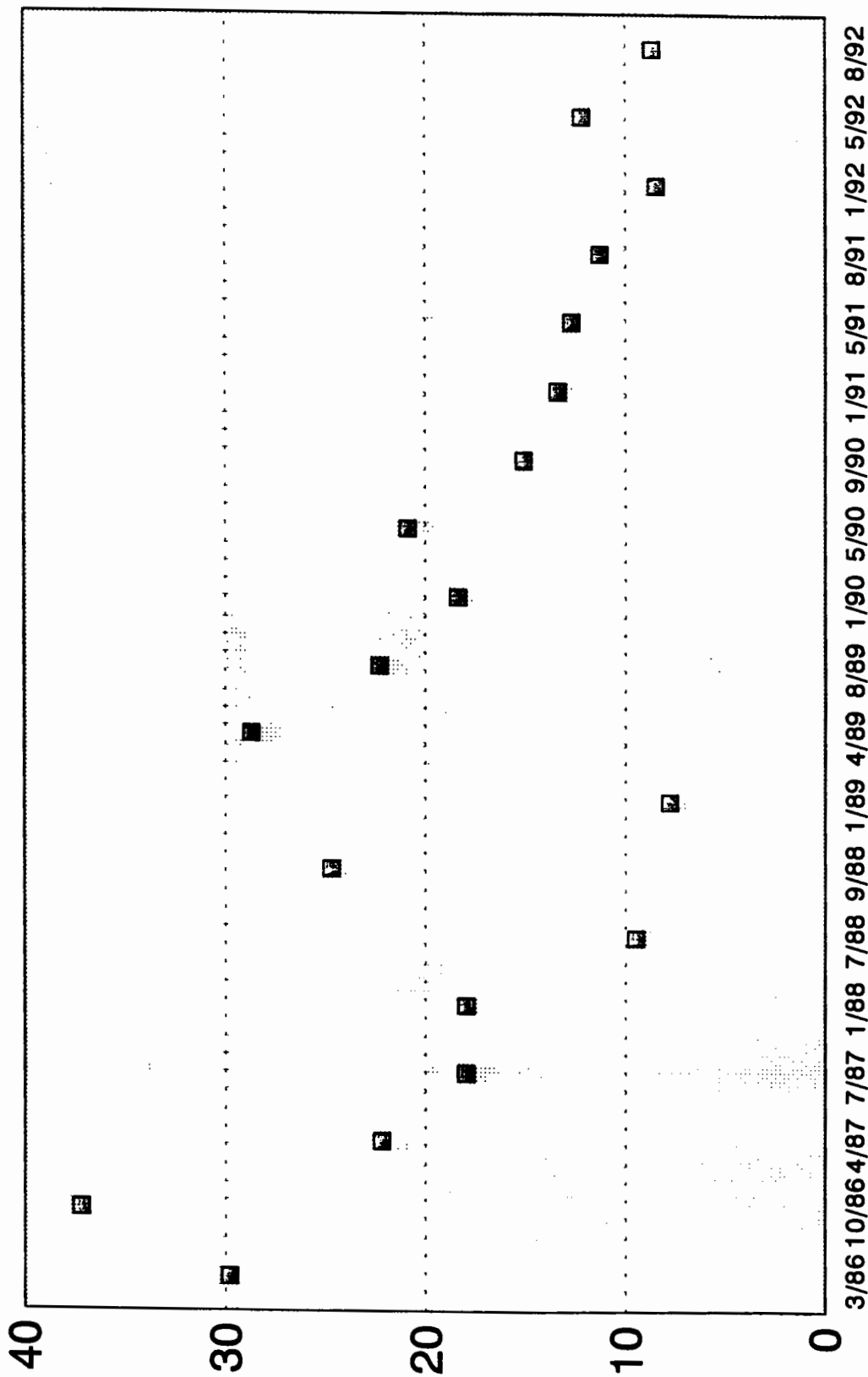
Table 16 Round Robin XXVI

Lycopene (TRANS)					
LAB#	Serum# 168	Serum# 169	Serum# 170	Serum# 171	Serum# 172
	0.214	0.213	0.134	0.117	0.134
MIST 3	.180	.166	.124	.106	.112

Interlaboratory Precision vs Time

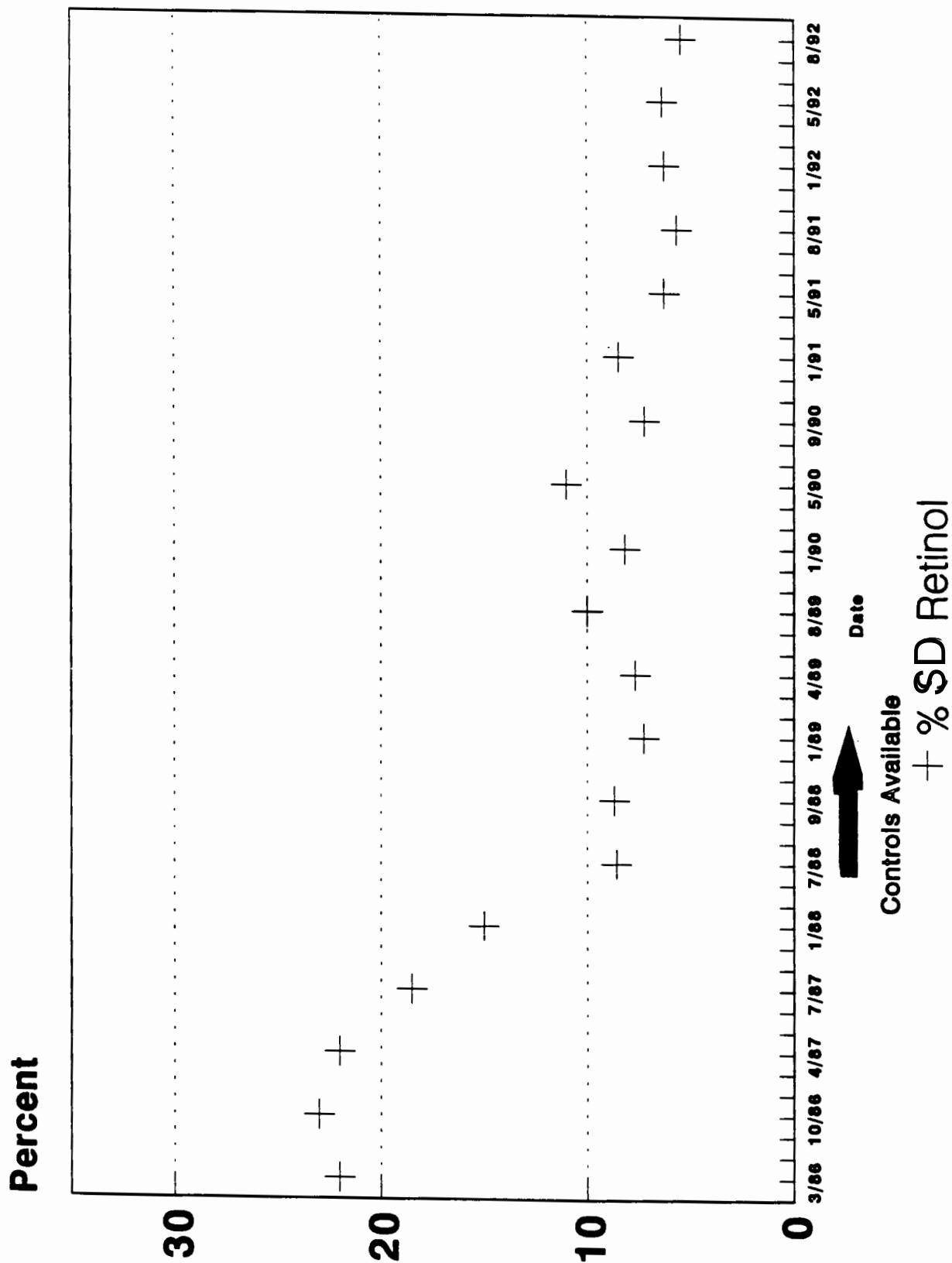
Core Laboratories

Retinol



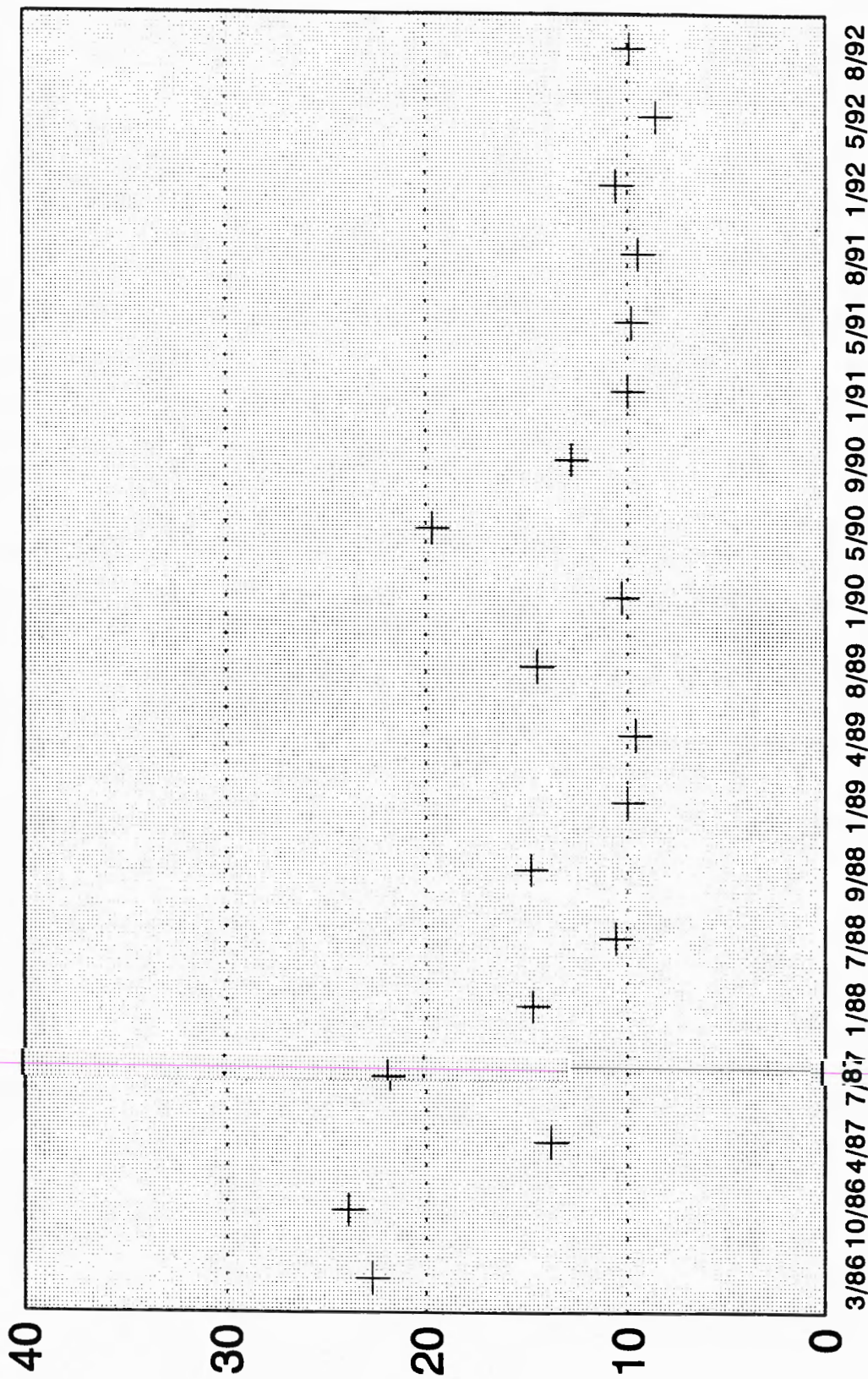
Interlaboratory Precision vs Time Retinol

Core Labs Trimmed



Interlaboratory Precision vs Time

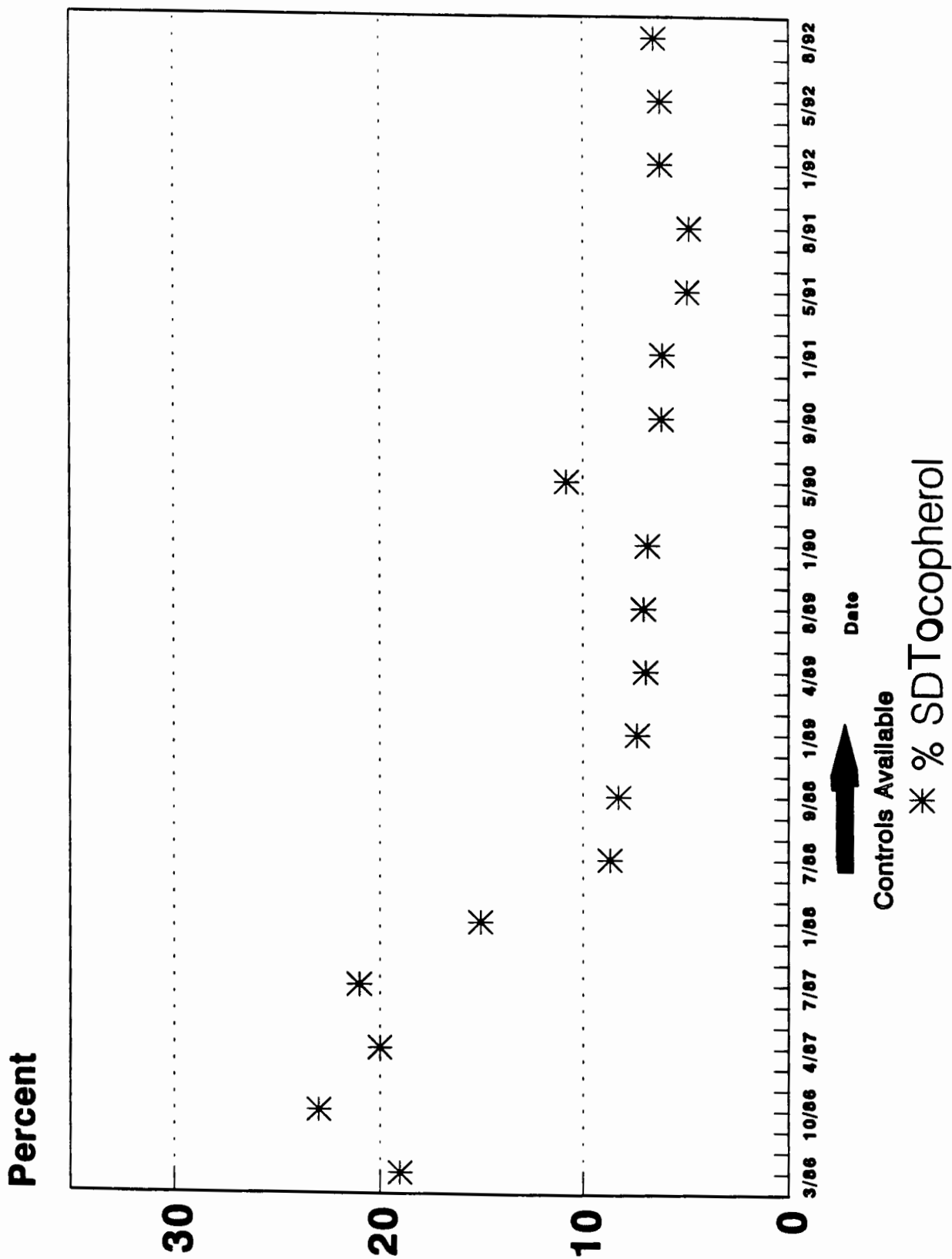
Core Laboratories
Alpha-Tocopherol



Interlaboratory Precision vs Time

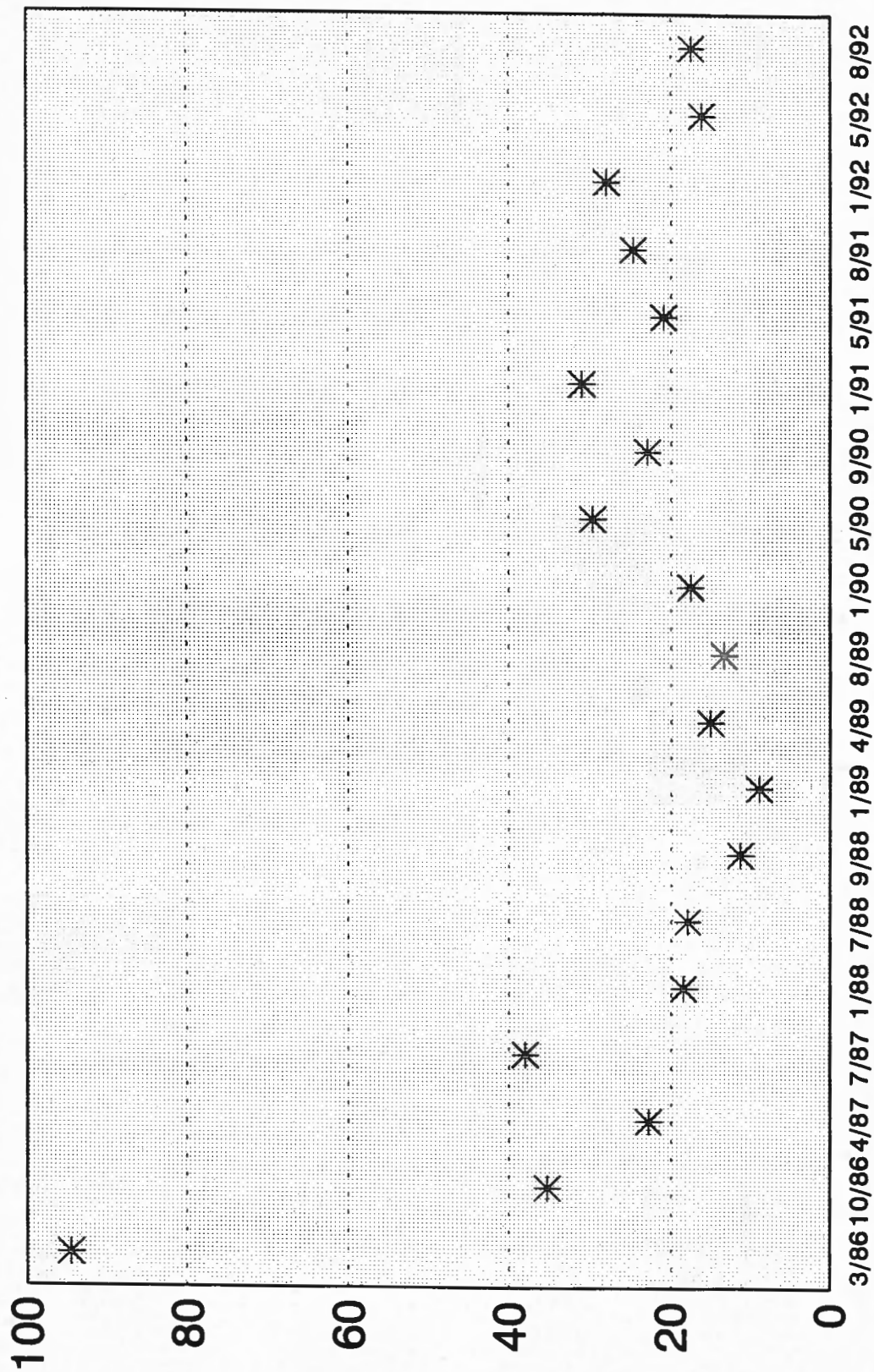
Alpha-Tocopherol

Core Labs Trimmed



Interlaboratory Precision vs Time

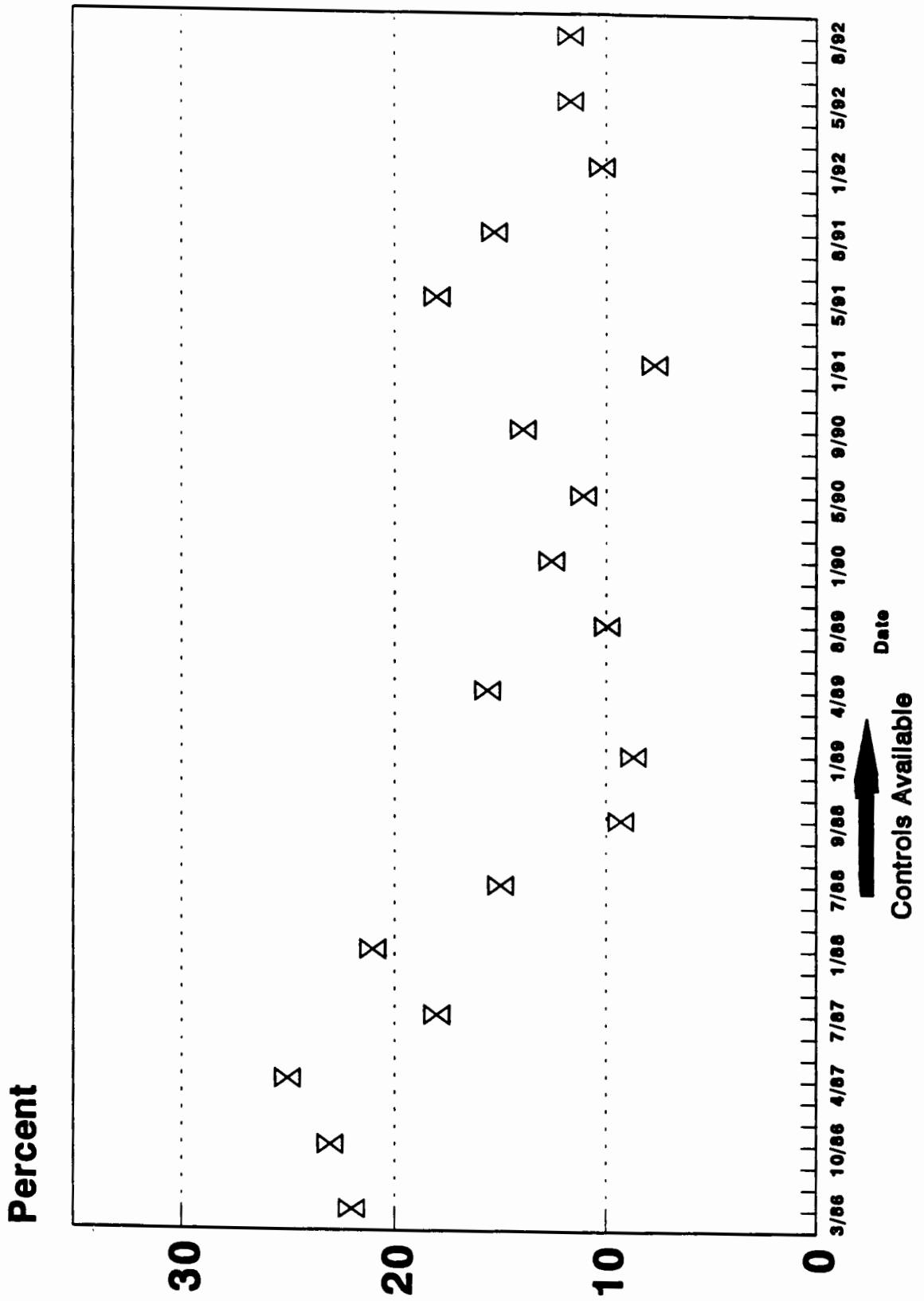
Core Laboratories
Total Beta-Carotene



Interlaboratory Precision vs Time

Beta-Carotene

Core Labs Trimmed



X % SD Beta-Carotene

Appendix J. Updated “All-Lab Report” for RR26

The following five pages are an updated version of an “All-Lab” report for RR26. This report has three parts:

- pages 1 thru 3 list results for all analytes reported by at least twice, counting both participants and NIST analysts.
- page 4 lists values for all analytes reported by only once. This page also provides a legend for pages 1 thru 3.
- page 5 summarizes each participants’ performance for total retinol, α - and γ/β -tocopherol, and total β -carotene. These summaries are compatible with the percent bias evaluation advice given in the RR26 Report. However, the current bias summaries are estimated relative to the median of all reported values for each analyte in each serum rather than to the “Trimmed Core Lab Average” used in the original and detailed in Appendix J. These original reference values were estimated from on-time results of the more experienced participants, with subjective exclusion of results deemed non-representative.

To ensure confidentiality, the laboratory identifiers used in this “All-Lab Report” have been altered from those used in RR26. 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.

Note: The analysts designated as NISTa and NISTb in this updated All-Lab report are designated as “NIST 1” and “NIST 3” in the Tables described in Appendix I.

Round Robin XXVI Laboratory Results

Lab	Total Retinol					Retinyl Palmitate					α-Tocopherol					γ/β-Tocopherol					Total β-Carotene					trans-β-Carotene				
	168	169	170	171	172	168	169	170	171	172	168	169	170	171	172	1.46	2.16	1.92	1.91	1.91	168	169	170	171	172	168	169	170	171	172
FSV-BA	0.539	0.629	0.649	0.533	0.645	0.220	0.076	0.302	0.184	0.296	10.39	10.00	6.31	5.20	6.32	1.46	2.16	1.92	1.91	1.91	1.570	0.425	0.545	0.376	0.552	1.510	0.392	0.512	0.346	0.516
FSV-BD	0.554	0.633	0.666	0.536	0.671						9.80	10.50	6.70	5.30	6.40	1.62	2.55	2.36	2.40	2.28	1.984	0.623	0.692	0.480	0.713					
FSV-BE	0.596	0.733	0.705	0.578	0.687						10.59	9.92	5.92	4.40	5.53	1.30	2.11	2.14	2.00	2.06	2.221	0.548	0.996	0.525	0.886					
FSV-BF	0.463	0.544	0.618	0.491	0.637						10.48	10.06	7.02	5.31	6.78	1.29	1.85	1.79	1.79	1.74	1.420	0.473	0.711	0.504	0.565					
FSV-BG	0.576	0.661	0.670	0.609	0.692	0.223	0.045	0.312	0.201	0.270	11.02	10.09	6.35	5.44	6.55	1.48	2.23	2.05	2.08	2.07	1.760	0.381	0.626	0.408	0.602	1.694	0.365	0.592	0.385	0.570
FSV-BH	0.570	0.616	0.668	0.550	0.666	0.160	0.038	0.227	0.154	0.240	10.92	9.15	6.37	4.95	6.08	1.50	2.58	2.05	2.20	2.11	1.633	0.412	0.540	0.361	0.549					
FSV-BI	0.517	0.601	0.606	0.505	0.617	0.297	0.085	0.372	0.207	0.374	10.05	9.49	5.93	4.82	5.94						1.876	0.489	0.658	0.425	0.651					
FSV-BJ	0.556	0.632	0.648	0.548	0.599	0.247	0.084	0.296	0.193	0.282	10.25	9.87	5.85	4.61	5.70															
FSV-BK	0.613	0.707	0.691	0.545	0.684						12.20	10.34	7.24	5.95	7.33	1.27	2.03	1.85	2.00	1.91	1.848	0.433	0.623	0.429	0.666	1.813	0.369	0.620	0.424	0.600
FSV-BL	0.525	0.606	0.680	0.542	0.735	0.237	0.064	0.317	0.222	0.352	12.20	10.83	7.17	5.74	7.14						1.085	0.495	0.541	0.377	0.549					
FSV-BN	0.556	0.621	0.683	0.555	0.661						12.90	9.46	7.11	5.68	7.11	1.52	0.346	0.461	0.296	0.499	1.152	0.346	0.461	0.296	0.499					
FSV-BO	0.525	0.638	0.642	0.496	0.674						11.24	9.62	6.72	5.28	6.28	2.108	0.467	0.608	0.287	0.722	2.108	0.467	0.608	0.287	0.722					
FSV-BP	0.586	0.666	0.697	0.591	0.721											1.576	0.432	0.580	0.397	0.550	1.576	0.432	0.580	0.397	0.550					
FSV-BS																														
FSV-BT	0.512	0.649	0.613	0.510	0.609						10.41	9.86	6.09	4.71	5.91	1.34	2.09	1.83	1.83	2.76	1.664	0.436	0.668	0.461	0.684					
FSV-BX	0.574	0.662	0.647	0.526	0.653	0.166	0.044	0.259	0.136	0.262	11.07	10.55	6.36	4.88	6.28	1.53	2.20	2.25	2.28	2.27	1.830	0.554	0.736	0.471	0.621					
FSV-BY	0.551	0.638	0.685	0.562	0.684						10.04	9.42	6.12	5.06	6.17						1.580	0.230	0.586	0.411	0.579					
FSV-BZ											11.50	11.00	7.00	5.20	6.90						1.819	0.500	0.626	0.389	0.591					
FSV-CA	0.480	0.573	0.602	0.504	0.596						9.47	9.17	6.08	5.00	5.96						1.727	0.453	0.593	0.408	0.583					
FSV-CB	0.630	0.678	0.770	0.630	0.767						12.20	9.60	6.19	4.87	6.63						1.505	0.373	0.550	0.342	0.567					
FSV-CH	0.521	0.336	0.719	0.617	0.677						9.84	4.92	6.15	5.25	6.24						1.490	0.102	0.724	0.374	0.699					
FSV-CJ	0.541	0.652	0.652	0.572	0.665						10.41	9.64	6.74	5.96	6.72						1.654	0.417	0.545	0.378	0.498					
FSV-CM											10.42	9.73	5.81	4.16	6.10	1.96	2.96	2.64	2.64	2.62	1.770	0.514	0.806	0.522	0.817	1.625	0.383	0.514	0.345	0.471
FSV-CO	0.513	0.591	0.634	0.508	0.627	0.375	0.158	0.474	0.330	0.471	9.98	9.34	5.53	4.55	5.37						1.890	0.470	0.580	0.360	0.550					
FSV-CP											9.37	8.84	5.91	4.87	5.78						1.797	0.464	0.617	0.399	0.568					
FSV-CQ	0.565	0.705	0.762	0.593	0.676						na	na	5.71	4.54	5.64	1.22	2.91	2.30	2.41	2.10	na	na	0.493	0.312	0.545					
FSV-CU	0.533	0.618	0.594	0.493	0.603						7.64	7.13	4.54	3.79	4.66						2.149	0.566	0.710	0.485	0.736					
FSV-CV	0.516	0.635	0.668	0.557	0.665						10.53	10.39	6.94	4.90	6.21						1.550	0.396	0.597	0.381	0.570					
FSV-CY	0.550	0.620	0.610	0.550	0.620						10.26	9.60	5.97	4.99	5.92						1.087	0.336	0.517	0.379	0.538					
FSV-DC	0.495	0.621	0.643	0.523	0.665						10.60	9.56	6.74	5.50	6.74						1.276	0.371	0.673	0.455	0.651					
FSV-DL	na	na	0.690	0.552	0.692						9.37	8.84	5.91	4.87	5.78						1.345	0.333	0.459	0.275	0.389					
FSV-DM	0.759	0.901	0.769	0.632	0.793						na	na	5.71	4.54	5.64	1.81	3.03	2.16	2.15	2.22	1.780	0.484	0.564	0.343	0.573					
FSV-DS	0.540	0.600	0.680	0.530	0.650						10.10	9.59	7.55	6.03	7.15															
FSV-DY	0.409	0.482	0.695	0.534	0.723						9.74	9.66	5.14	4.31	5.14															
FSV-EB	0.555	0.711	0.655	0.521	0.666						9.13	8.65	5.68	4.58	5.70															
FSV-EC	0.566	0.681	0.673	0.524	0.679						10.80	10.61	6.06	4.68	6.04															
FSV-EJ	0.545	0.675	0.683	0.546	0.709						10.58	9.89	5.74	4.71	5.94															
n	32	32	33	33	33	8	8	8	8	8	12.24	11.95	5.73	3.49	5.62															
Min	0.409	0.336	0.594	0.491	0.596	0.160	0.038	0.227	0.136	0.240	34	34	35	35	35	1.22	1.85	1.79	1.79	1.74	1.085	0.102	0.459	0.275	0.389	1.510	0.365	0.512	0.345	0.471
Mean	0.548	0.635	0.669	0.547	0.670	0.241	0.074	0.320	0.203	0.318	7.64	4.92	4.54	3.49	4.66	1.48	2.39	2.11	2.14	2.17	1.660	0.432	0.621	0.400	0.609	1.661	0.377	0.560	0.375	0.539
Max	0.759	0.901	0.770	0.632	0.793	0.375	0.158	0.474	0.330	0.471	10.57	9.68	6.25	4.96	6.18	1.96	3.03	2.64	2.64	2.76	2.221	0.623	0.996	0.525	0.886	1.813	0.392	0.620	0.424	0.600
SD	0.058	0.087	0.045	0.038	0.045	0.070	0.039	0.075	0.058	0.077	1.03	1.16	0.63	0.57	0.59	0.23	0.40	0.25	0.26	0.29	0.294	0.103	0.109	0.067	0.100	0.127	0.013	0.055	0.038	0.057
CV	11	14	7	7	7	29	52	24	29	24	10	12	10	12	10	15	17	12	12	13	18	24	18	17	16	8	3	10	10	11
NISTa	0.551	0.664	0.688	0.514	0.721	0.244	0.065	0.328	0.204	0.288	10.05	9.88	6.47	4.78	6.19	1.23	2.19	1.93	1.91	2.17	1.590	0.447	0.661	0.482	0.610	1.450	0.403	0.591	0.352	0.586
NISTb	0.719	0.738	0.785	0.647	0.747						9.60	8.25	5.40	4.31	5.01	1.36	2.03	1.85	1.85	1.71	1.660	0.415	0.606	0.417	0.560					
Median	0.548	0.634	0.668	0.545	0.666	0.230	0.070	0.307	0.197	0.289	10.45	9.69	6.15	4.95	6.17	1.47	2.21	2.10	2.12	2.10	1.664	0.436	0.602	0.393	0.576	1.660	0.376	0.553	0.366	0.543
eSD	0.036	0.044	0.033	0.032	0.031	0.060	0.030	0.044	0.028	0.056	0.70	0.53	0.62	0.51	0.58	0.24	0.38	0.28	0.27	0.26	0.246	0.082	0.088	0.051	0.051					
eCV	7	7	5	6	5	26	42	14	14	19	7	6	10	10	9	16	17	13	13	12	15	19	15	15	13	15	19	15	15	13

Round Robin XXVI Laboratory Results

Lab	Total cis-β-Carotene				Total α-Carotene				Total Lycopene				trans-Lycopene				Total β-Cryptoxanthin			
	168	169	170	171	172	168	169	170	171	172	168	169	170	171	172	168	169	170	171	172
FSV-BA	0.060	0.033	0.033	0.030	0.036	0.099	0.062	0.028	0.013	0.029	0.440	0.450	0.300	0.230	0.270	0.149	0.120	0.070	0.044	0.073
FSV-BD						0.088	0.052	0.044	0.017	0.029	0.472	0.522	0.342	0.246	0.304					
FSV-BE						0.115	0.079	0.052	nd	0.033	0.398	0.392	0.264	0.334	0.221					
FSV-BF						0.027	0.026	nd	nd	nd	0.338	0.276	0.192	0.156	0.183					
FSV-BG						0.098	0.049	0.022	0.013	0.022	0.294	0.290	0.169	0.140	0.170	0.200	0.150	0.100	0.058	0.097
FSV-BH	0.066	0.016	0.034	0.023	0.032	0.088	0.058	0.021	0.013	0.020	0.187	0.186	0.123	0.101	0.124	0.179	0.146	0.086	0.051	0.087
FSV-BI						0.072	0.048	0.018	0.012	0.018										
FSV-BJ																				
FSV-BK																				
FSV-BL																				
FSV-BN	0.035	0.064	0.003	0.005	0.066	0.078	0.048	0.023	0.012	0.025	0.183	0.170	0.140	0.110	0.124	0.108	0.085	0.055	0.032	0.059
FSV-BO						0.067	0.053	0.015	nd	0.023	0.209	0.329	0.182	0.159	0.199	0.067	0.177	0.065	nd	nd
FSV-BP						0.066	0.047	0.016	0.013	0.020	0.202	0.200	0.139	0.132	0.155					
FSV-BS																				
FSV-BT																				
FSV-BX																				
FSV-BY						0.109	0.062	0.024	0.015	0.025	0.340	0.381	0.238	0.202	0.242	0.118	0.099	0.064	0.041	0.067
FSV-BZ																				
FSV-CA																				
FSV-CB																				
FSV-CH																				
FSV-CJ						0.063	0.042	0.020	0.013	0.019	0.270	0.265	0.188	0.158	0.190	0.124	0.107	0.074	0.045	0.072
FSV-CM																				
FSV-CO																				
FSV-CP						0.125	0.071	0.028	0.016	0.029	0.436	0.466	0.269	0.220	0.286	0.130	0.113	0.062	0.036	0.062
FSV-CQ																				
FSV-CU	0.029	0.034	0.031	0.033	0.027	0.086	0.056	0.026	0.016	0.026	0.362	0.384	0.230	0.196	0.222	na	na	0.073	0.048	0.077
FSV-CV																				
FSV-CY						na	na	0.019	0.011	0.020	na	na	0.124	0.101	0.145	na	na	0.073	0.048	0.077
FSV-DC																				
FSV-DL						0.092	0.059	0.032	0.021	0.029	0.215	0.249	0.183	0.175	0.199	0.111	0.097	0.073	0.043	0.074
FSV-DM																				
FSV-DS																				
FSV-DY																				
FSV-EB																				
FSV-EC																				
FSV-EJ						0.114	0.088	0.034	0.039	0.035	0.394	0.332	0.233	0.196	0.237	0.118	0.099	0.064	0.041	0.067
n	4	4	4	4	4	16	16	16	14	16	15	15	16	16	16	1	1	1	1	1
Min	0.029	0.016	0.003	0.005	0.027	0.027	0.026	0.015	0.011	0.018	0.183	0.170	0.123	0.101	0.124	0.067	0.085	0.055	0.032	0.059
Mean	0.048	0.037	0.025	0.023	0.040	0.087	0.056	0.026	0.016	0.025	0.316	0.326	0.207	0.179	0.204	0.132	0.122	0.072	0.044	0.074
Max	0.066	0.064	0.034	0.033	0.066	0.125	0.088	0.052	0.039	0.035	0.472	0.522	0.342	0.334	0.304	0.200	0.177	0.100	0.058	0.097
SD	0.018	0.020	0.015	0.013	0.018	0.025	0.015	0.010	0.007	0.005	0.101	0.106	0.065	0.061	0.055	0.040	0.030	0.013	0.008	0.012
CV	38	54	59	55	44	28	26	38	44	21	32	33	31	34	27	30	25	18	18	16
NISTa	0.140	0.044	0.070	0.130	0.024	0.102	0.057	0.037	0.017	0.025	0.502	0.457	0.270	0.244	0.253	0.112	0.087	0.059	0.034	0.054
NISTb						0.079	0.053	0.022	0.015	0.021	0.201	0.179	0.134	0.115	0.121	0.124	0.113	0.072	0.044	0.073
Median	0.048	0.034	0.032	0.027	0.034	0.088	0.055	0.024	0.013	0.025	0.338	0.329	0.190	0.167	0.199	0.024	0.024	0.010	0.006	0.009
eSD						0.027	0.010	0.007	0.002	0.006	0.145	0.095	0.073	0.052	0.060	0.024	0.024	0.010	0.006	0.009
eCV						31	19	30	17	25	43	29	38	31	30	19	21	15	13	12

Round Robin XXVI Laboratory Results

Lab	Total Lutein				Total Zeaxanthin				Total Lutein&Zeaxanthin						
	168	169	170	171	172	168	169	170	171	172	168	169	170	171	172
FSV-BA															
FSV-BD															
FSV-BE															
FSV-BF															
FSV-BG															
FSV-BH	0.151	0.102	0.109	0.045	0.115	0.034	0.033	0.040	0.027	0.037	0.185	0.135	0.149	0.072	0.152
FSV-BI	0.157	0.137	0.112	0.046	0.111	0.038	0.036	0.043	0.023	0.043	0.186	0.159	0.149	0.071	0.151
FSV-BJ															
FSV-BK															
FSV-BL															
FSV-BN	0.095	0.081	0.075	0.031	0.073	0.017	0.018	0.023	0.012	0.020	0.126	0.107	0.101	0.044	0.097
FSV-BO															
FSV-BP															
FSV-BS															
FSV-BT															
FSV-BX															
FSV-BY	0.129	0.109	0.109	0.047	0.111										
FSV-BZ															
FSV-CA															
FSV-CB															
FSV-CH															
FSV-CJ															
FSV-CM															
FSV-CO															
FSV-CP															
FSV-CQ															
FSV-CU															
FSV-CV															
FSV-CY															
FSV-DC															
FSV-DL															
FSV-DM															
FSV-DS															
FSV-DY															
FSV-EB															
FSV-EC															
FSV-EJ															
n	4	4	4	4	4	3	3	3	3	3	7	7	8	8	8
Min	0.095	0.081	0.075	0.031	0.073	0.017	0.018	0.023	0.012	0.020	0.126	0.107	0.101	0.044	0.097
Mean	0.133	0.107	0.101	0.042	0.103	0.030	0.029	0.035	0.021	0.033	0.201	0.185	0.166	0.074	0.166
Max	0.157	0.137	0.112	0.047	0.115	0.038	0.036	0.043	0.027	0.043	0.341	0.263	0.230	0.092	0.233
SD	0.028	0.023	0.018	0.008	0.020	0.011	0.010	0.011	0.008	0.012	0.073	0.057	0.037	0.014	0.039
CV	21	22	17	18	19	38	33	31	38	36	36	31	23	19	24
NISTa															
NISTb	0.156	0.115	0.114	0.047	0.108	0.043	0.041	0.052	0.027	0.049	0.199	0.156	0.166	0.074	0.157
Median	0.140	0.106	0.109	0.046	0.111	0.034	0.033	0.040	0.023	0.037	0.186	0.182	0.164	0.076	0.160
eSD											0.061	0.070	0.024	0.007	0.027
eCV						33	38	15	9	17					

Round Robin XXVI Laboratory Results

Legend

na Sample not available for analysis

nd Not detected (i.e., not reported or reported as '0', 'not determined', etc.)

italics Value calculated from reported results

n Number of non-NIST laboratories reporting quantitative results

Min Minimum non-NIST reported value.

Mean Average over all non-NIST reported values.

Max Maximum non-NIST reported value.

SD Standard deviation over all non-NIST values.

CV Coefficient of Variation (% relative standard deviation): $100 \cdot \text{SD} / \text{Mean}$

Median Median over all non-NIST reported values

eSD Robust estimate of SD based on the adjusted median absolute difference from the median (MADe)

eCV Robust estimate of CV, $100 \cdot \text{eSD} / \text{Median}$

% Bias Summary

Lab	TR	aT	g/bT	bC
FSV-BA	-2±1	3±2	-6±4	-5±3
FSV-BD	0±1	4±6		
FSV-BE	8±5	-4±6	12±3	25±11
FSV-BF	-10±5	7±5	-4±5	42±17
FSV-BG	5±4	6±3		8±17
FSV-BH	0±2	0±4	-15±2	1±8
FSV-BI	-7±2	-3±1	-1±1	-6±3
FSV-BJ	-2±5	3±4	4±7	11±2
FSV-BK	6±5	-4±4		
FSV-BL	1±6	16±5		
FSV-BN	1±2	15±2	-10±3	8±6
FSV-BO	-3±4	13±10		-8±17
FSV-BP	7±2	5±4		-23±6
FSV-BS				7±22
FSV-BT	-5±4	-2±3		-3±3
FSV-BX	0±4	4±4	-2±19	
FSV-BY	2±1	-1±2	5±4	9±9
FSV-BZ		11±4		17±8
FSV-CA	-10±2	-4±4		
FSV-CB	14±4	4±8		
FSV-CH	-6±24	-10±23		-10±21
FSV-CJ	1±3	8±9		6±6
FSV-CM		-5±7		
FSV-CO	-6±1	-8±4		2±2
FSV-CP		-25±1		-9±5
FSV-CQ	8±5	4±6		-10±40
FSV-CU	-7±4	-2±2		-6±5
FSV-CV	-1±3	6±6	28±5	27±14
FSV-CY	-3±4	-6±4		1±9
FSV-DC	-4±4	11±13	8±18	3±4
FSV-DL	3±1	-8±1		-15±8
FSV-DM	26±13	-11±7		26±5
FSV-DS	-2±3	-9±2		-4±4
FSV-DY	-8±16			-16±13
FSV-EB	1±6	1±6		0±18
FSV-EC	2±4	-2±4		-26±5
FSV-EJ	3±3	-1±21	14±15	0±10
NISTa	2±5	0±4	-6±8	7±10
NISTb	19±7	-13±4	-12±4	0±4

Label	Definition
Lab	Participant code
TR	Total Retinol
aT	a-Tocopherol
g/bT	g/b-Tocopherol
bC	Total b-Carotene
% Bias	(Mean ± SD) of individual serum biases
Mean _i	Average of (x _i -Median _i)/Median _i
SD	Standard deviation of (x _i -Median _i)/Median _i
x _i	Result for analyte in serum _i
Median _i	Median of non-NIST results in serum _i

The original analysis listed % Bias for each result for each serum calculated relative to the "Trimmed Core Lab Average" of that analyte in the serum. The summary values reported here are the (arithmetic mean ± standard deviation) of each laboratory's reported results for the analyte estimated relative to each serum's median value.

Appendix K. Shipping Package Inserts for RR03

The following two items were included in each package shipped to a RR03 participant:

- Cover letter
- Report of Analysis datasheet

These items were attached to the shipping box.



NIST

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

August 17, 1992

1~

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 a working draft.

2~

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 two ampoules in the shipping container. Each ampoule should be analyzed in duplicate by the method(s) used in your laboratory. 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 **September 25, 1992**. 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 Telefax your results to me, the number is (301) 926-8671.

Thank you for your assistance.

Sincerely,

Sam A. Margolis, Ph. D.
Research Chemist
Organic Analytical Research Division
Center for Analytical Chemistry

REPORT OF ANALYSIS

Name: _____

Address: _____

Telephone Number: _____

FAX Number: _____

METHOD of ANALYSIS: _____

DATE of ANALYSIS:: _____

RESULTS (µg/mL)

SERUM 103, VIAL# _____

REPLICATE 1 _____ µg/mL

REPLICATE 2 _____ µg/mL

SERUM 103, VIAL# _____

REPLICATE 1 _____ µg/mL

REPLICATE 2 _____ µg/mL

Appendix L. Final Report for RR03

There is no extant version of a final report as sent to the RR03 participants. The following discussion and table have been extracted from a NIST-internal Report of Analysis that Dr. Margolis prepared in late 1993.

The round robin data are summarized in Table 1. ... The results of the ... six participants [who received unfrozen samples and who returned results] show a wide degree of variation even though the mean is relatively close to that measured by NIST. The LC measurements of laboratories 1 and 4 are similar to our value for [ascorbic acid (AA)] and probably reflect the fact that these methods are not measuring the [dehydroascorbic acid (DHAA)] that is present. The results of laboratory 6 are approximately 10% higher than those of NIST. These are consistent with earlier results from this laboratory using the dinitrophenyl-hydrazine (DNPH) method. The values reported by the other three laboratories probably reflect the variation in the accuracy of the various methods. The SD of the round robin study is approximately 13 times that of NIST and suggests that the data must be differentiated with respect to AA and AA + DHAA also with respect to the type of method used for analysis.

Table 1. Results of the Round Robin Measurement of Ascorbic acid in Human Serum

<u>Laboratory</u>	<u>Method</u>	<u>AA (mmol/L)</u>	
		<u>Replicate #1</u>	<u>Replicate #2</u>
1	LC-EC	54.4	54.6
		56.7	56.8
2	LC-EC	70.4	68.1
		42.0	44.3
3	DNPH	42.6	45.3
		65.3	63.0
4	LC-EC	48.8	48.6
		53.4	52.2
5	LC-EC	61.8	
		61.2	
6	DNPH	72.7	72.1
		72.7	73.3
Lab Mean \pm SD		58.2 \pm 10.6 (n=22)	
NIST Mean \pm SD	LC-EC	AA	50.4 \pm 3.2 (n=5)
		AA+DHAA	64.8 \pm 0.8 (n=5)

The "All Lab Report" in Appendix M provides more extensive statistical summaries.

Appendix M. Updated “All-Lab Report” for VC-RR03

The following page is the updated “All-Lab Report” for RR03.

Vitamin C Round Robin 3

Lab	Date	Analyte	Method	103, [AA or TAA] mmol/mL				
				Mean	S _{dup}	S _{rep}	S _{het}	S _{tot}
VC-MB	24/02/93		HPLC-EC	61.5	0.4			0.4
VC-MC	17/09/92		HPLC-EC	55.6	1.6	0.1	1.6	1.6
VC-NC	25/09/92	TAA	24DNPH	72.7	0.4	0.4	0.3	0.5
VC-NN	26/09/92		24DNPH	54.0	14.3	1.8	14.2	14.3
VC-NR	25/09/92	AA	HPLC-EC	56.2	18.5	1.6	18.4	18.5
VC-NS	21/08/92		HPLC-EC	50.2	3.6	1.3	3.5	3.7
NIST		AA	HPLC-EC	50.4				3.2
NIST		TAA	HPLC-EC	64.8				0.8
			N	6				
			Min	50.2	0.4	0.1	0.3	0.4
			Median	55.9	2.6	1.3	3.5	2.6
			Max	72.7	18.5	1.8	18.4	18.5
			eSD	5.5				
			eCV	10				

Legend

Lab Laboratory Code
Date Date that the results were received at NIST
Analyte Analyte specified by the laboratory
Method Type of assay
 24DNPH 2,4-Dinitrophenylhydrazine
 HPLC-EC Liquid chromatography with electrochemical detection

Mean Mean of duplicate means

S_{dup} Standard deviation of duplicate means

S_{rep} Pooled standard deviation of replicates

S_{het} Estimated sample heterogeneity, $\sqrt{\text{MAX}(0, S_{\text{dup}}^2 - S_{\text{het}}^2)}$

S_{tot} Estimated standard deviation of the mean, $\sqrt{(S_{\text{dup}}^2 + S_{\text{rep}}^2)/n}$
 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