

NISTIR 7880-33

**NIST Micronutrients Measurement
Quality Assurance Program
Winter, Spring, and Fall 1991
Comparability Studies**

Results for Round Robins XXI, XXII, and XXIII
Fat-Soluble Vitamins and Carotenoids in Human Serum

Neal E. Craft (Former Employee)

David L. Duewer

Margaret C. Kline

Sam A. Margolis (Retired)

Willie E. May

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June 2015



U.S. Department of Commerce
Penny Pritzker, Secretary

National Institute of Standards and Technology

Willie E. May, Under Secretary of Commerce for Standards and Technology and Director

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Abstract

The National Institute of Standards and Technology coordinates the Micronutrients Measurement Quality Assurance Program (MMQAP) for laboratories that measure fat- and water-soluble vitamins and carotenoids in human serum and plasma. This report describes the design of and results for the Winter, Spring and Fall 1991 MMQAP measurement comparability improvement studies: 1) Round Robin XXI Fat-Soluble Vitamins and Carotenoids in Human Serum, 2) Round Robin XXII Fat-Soluble Vitamins and Carotenoids in Human Serum, and 3) Round Robin XXIII Fat-Soluble Vitamins and Carotenoids in Human Serum. The materials for Round Robin XXI were shipped to participants in January 1991; participants were requested to provide their measurement results by March 1, 1991. The materials for Round Robin XXII were shipped to participants in May 1991; participants were requested to provide their measurement results by July 9, 1991. The materials for Round Robin XXIII were shipped to participants in August 1991; participants were requested to provide their measurement results by September 13, 1991.

Keywords

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

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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]

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

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin XXI comparability study (hereafter referred to as RR21) 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 1991. 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 RR21 is reproduced as Appendix B. This report included:

- Our analysis of the participants' results.
- Tabular presentations of all participant results.
- Graphical presentations of interlaboratory precision over time. These figures are no longer available, but would have been very similar to those in Appendix E.

Each participant also received an "Individualized Report" that graphed their results for selected analytes. These figures are no longer available, but would have been very similar in format to those in Appendix F.

Appendix C lists all of the measurement results reported for RR21 in a more accessible format.

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

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin XXII comparability study (hereafter referred to as RR22) received three 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 1991. The communication materials included in the sample shipment are described in Appendix D.

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 RR22 is reproduced as Appendix E. This report included:

- Our analysis of the participants' results.
- Tabular presentations of all participant results.
- Graphical presentations of interlaboratory precision over time.

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. An example of a RR22 Individualized Report is given in Appendix F.

Appendix G lists all of the measurement results reported for RR22 in a more accessible format.

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

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin XXIII comparability study (hereafter referred to as RR23) received four lyophilized human sera and one human serum extract in hexane 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 1991. The communication materials included in the sample shipment are described in Appendix H.

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 RR23 was mailed to all participants shortly before the October 19, 1991 NIST/NCI Micronutrients Analysis Workshop, however, no version of either the letter or the preliminary report is available. Likewise, only the tabular parts of the Final Report for RR23 is available; these tables are reproduced as Appendix I.

It is probable that each participant also received an "Individualized Report" that graphically analyzes their results for selected analytes. While there is no longer an example of a RR23 Individualized Report, its design would have been similar to the RR22 example in Appendix F.

Appendix J lists all of the measurement results reported for RR23 in a more accessible format.

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.

Appendix A. Shipping Package Inserts for RR21

Two items were included in each package shipped to an RR21 participant:

- **Cover letter.** The original letter has been lost. It likely would have described the four lyophilized sample materials (sera 144 to 147), given guidance on reconstituting these samples, stated that results were due March 1, 1991 and to whom they should be sent, and who to contact with technical questions.
- **Datasheets.** The following two pages reproduce the form. The first page of the form is for reporting the primary-focus analytes: retinol, α -tocopherol, and total and *trans*- β -carotene. The second page is for reporting three “optional” analytes: retinyl palmitate, γ -tocopherol, and lycopene; one participant also reported α -carotene. In the absence of other information, the lycopene and α -carotene results are assumed to report total lycopene and total α -carotene.

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

RR XXI JANUARY 1991

REPORT ON NIST/NCI SAMPLES FROM LABORATORY # _____

DATE OF ANALYSIS _____

RESULTS IN $\mu\text{g/mL}$

SAMPLE #	ANALYTE	Trans	Total
SERUM 144	RETINOL		
VIAL # _____	B-CAROTENE		
	A-TOCOPHEROL		
SERUM 144	RETINOL		
VIAL # _____	B-CAROTENE		
	A-TOCOPHEROL		
SERUM 144	RETINOL		
VIAL # _____	B-CAROTENE		
	A-TOCOPHEROL		
SERUM 144	RETINOL		
VIAL # _____	B-CAROTENE		
	A-TOCOPHEROL		

Add 1.0 mL of Distilled water to the Serum Samples

RR XXI JANUARY 1991

REPORT ON NIST/NCI SAMPLES FROM LABORATORY # _____

OPTIONAL ANALYTES

DATE OF ANALYSIS _____

RESULTS IN $\mu\text{g/mL}$

SAMPLE #	ANALYTE	Trans	Total
SERUM 144	RETINYL PALMITATE		
VIAL # _____	LYCOPENE		
	GAMMA-TOCOPHEROL		
SERUM 144	RETINYL PALMITATE		
VIAL # _____	LYCOPENE		
	GAMMA-TOCOPHEROL		
SERUM 144	RETINYL PALMITATE		
VIAL # _____	LYCOPENE		
	GAMMA-TOCOPHEROL		
SERUM 144	RETINYL PALMITATE		
VIAL # _____	LYCOPENE		
	GAMMA-TOCOPHEROL		

Appendix B. Final Report for RR21

The following six pages are the available components of the final report for RR21 as provided to all participants. The original report consisted of:

- A cover letter and discussion.
- Tables 1 to 3 that list results and various summary values for retinol, α -tocopherol, and total β -carotene.
- Tables 4 to 7 that list results and simple summary statistics for *trans*- β -carotene, retinyl palmitate, lycopene, and γ -tocopherol.
- Graphical presentations of interlaboratory precision over time for retinol, α -tocopherol, and total β -carotene.

When the isomeric form of an analyte is not specified, it is likely that most participants reported “total” (the sum of all isomers.) Since resolution of γ -tocopherol and β -tocopherol is challenging, the results reported as γ -tocopherol can be confidently assumed to be γ/β -tocopherol.

The graphical presentations for RR21 are no longer available; however, they would have been very similar to the graphs in the Final Report for RR22 in Appendix E.

Due to the complex formatting used in the Tables, the originally listed laboratory codes have been deleted without replacement. However, Appendix C provides a complete listing of the RR21 results where the original codes have been altered to ensure confidentiality. Appendix C also provides more relevant summary statistics.



NIST

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

May 1, 1991

Dear Colleague:

Enclosed is a packet describing both overall and your labs performance in NIST/NCI Fat-Soluble in Serum Analysis Round-Robin XXI. Specifically, the packet contains for retinol, α -tocopherol, and β -carotene, respectively: a tabular presentation of all data received; a graphical presentation of the improvement in Interlaboratory Precision vs. Time; a Control Chart representing your labs performance vs. the assigned value for all samples over the past three years; a graphical presentation of your analyses of blind-duplicate samples over the past three years. Tabular data only are presented for trans- β -carotene, retinyl palmitate, lycopene and γ -tocopherol.

Data reported for retinol, α -tocopherol and β -carotene are given in Tables 1-3. All values reported for the study are shown on the left side of section "A" of the Tables along with the Grand Average for all data reported. The middle or "B" section of Tables 1-3 provides the trimmed average, standard deviation, and an identification of the values dropped from the pool during the trimming process (indicated by an asterisk). Simply stated, the trimmed average is determined by iteratively dropping all values outside two standard deviations from the floating average. An NIST value is also provided in the "B" section of Tables 1-3 along with the trimmed average determined when each sample was analyzed previously. The bias (%) relative to the assigned value for each sample is given in the "C" section of each Table.

As in past studies, the trimmed average is used as the Assigned Pool Value for all three core analytes (retinol, α -tocopherol, and total β -carotene). The relatively close agreement between the NIST and the trimmed average allows us to continue the convention of 0-5% bias from the assigned value representing EXCEPTIONAL performance, 6-10% ACCEPTABLE performance, 11-20% MARGINAL performance, and >20% UNACCEPTABLE with respect to the current state-of-the-practice for measurement of these analytes in serum/plasma.

We encourage labs that receive "U" ratings for any of the core analytes to contact us for discussions concerning possible sources of bias in your methodology. Given our limited resources, we will have to consider dropping labs that receive unacceptable ratings for three consecutive studies.

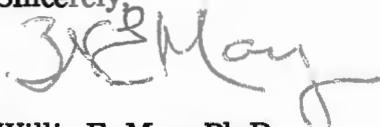
Data for non-core analytes reported for RRXXI are provided in Tables 4-7. For trans- β -carotene (Table 4), it is very encouraging that the RSD of the five reported values is less than 10% for all four samples. Retinyl palmitate levels are very low (Table 5) for the samples used in this study. For the sample with the highest concentration (144), the agreement among the data reported by four of the five labs, RSD 22%, is quite commendable for the 50 ppb level.

May 1, 1991
Page 2

Twelve laboratories reported data for lycopene and γ -tocopherol. The RSD for γ -tocopherol is approaching the level that we expect for the core analytes ($\leq 10\%$). For lycopene, we suspect that the scatter in the data may, in part, be due to partial separation of lycopene isomers and/or instability of lycopene reference standard solutions. We will carefully monitor results for these analytes and make recommendations to you at this years workshop (Saturday, October 26) regarding strategies for improving interlaboratory measurement capabilities/data.

Round-Robin XXII samples will be shipped to you on May 14. Results are required by July 9.

Sincerely,



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



Neal E. Craft
Biologist
Organic Analytical Research Division
Chemical Science Technology Laboratory

Enclosures

Table 1.

A

Round Robin XXI

Retinol Results

Lab #	Serum #		Serum #		Serum #		Serum #		Serum #		Serum #		Serum #		Serum #		Serum #		Serum #		Percent Bias from Assigned Pool Value.	
	144	145	146	147	144	145	146	147	144	145	146	147	144	145	146	147	144	145	146	147		
0.934	0.483	0.468	0.253		0.934	0.483	0.468		0.253		-2.5	-1.4	-8.9	-3.3								
0.995	0.525	0.544	0.273		0.995	0.525	0.544		0.273		3.9	7.2	5.9	4.3								
1.086	0.518	0.533	0.279		1.086	0.518	0.533		0.279		13.4	5.8	3.8	6.6								
1.062	0.521	0.592	0.299		1.062	0.521	0.592		0.299		10.9	6.4	15.3	14.2								
0.960	0.507	0.529	0.277		0.960	0.507	0.529		0.277		0.2	3.4	3.0	5.8								
0.932	0.507	0.552	0.273		0.932	0.507	0.552		0.273		-2.7	3.5	7.5	4.3								
1.060	0.480	0.523	0.259		1.060	0.480	0.523		0.259		10.7	-2.0	1.8	-1.1								
0.892	0.470	0.505	0.226		0.892	0.470	0.505		0.226		-6.9	-4.0	-1.7	-13.7								
0.910	0.494	0.492	0.258		0.910	0.494	0.492		0.258		-5.0	0.9	-4.2	-1.4								
0.951	0.468	0.500	0.263		0.951	0.468	0.500		0.263		-0.7	-4.4	-2.6	0.5								
0.920	0.510	0.500	0.260		0.920	0.510	0.500		0.260		-4.0	4.1	-2.6	-0.7								
0.955	0.499	0.561	0.281		0.955	0.499	0.561		0.281		-0.3	1.9	9.2	7.3								
0.989	0.506	0.531	0.274		0.989	0.506	0.531		0.274		3.2	3.3	3.4	4.7								
1.115	0.421	0.412	0.214		*1.115	0.421	0.412		0.214		16.4	-14.1	-19.7	-18.3								
0.890	0.483	0.472	0.261		0.890	0.483	0.472		0.261		-7.1	-1.4	-8.1	-0.3								
0.868	0.446	0.435	0.224		0.868	0.446	0.435		0.224		-9.4	-8.9	-15.3	-14.4								
0.960	0.500	0.500	0.250		0.960	0.500	0.500		0.250		0.2	2.1	-2.6	-4.5								
0.770	0.430	0.440	0.210		*0.770	0.430	0.440		0.210		-19.6	-12.2	-14.3	-19.8								
1.145	0.599	0.610	0.315		*1.145	*0.599	0.610		0.315		19.5	22.3	18.8	20.3	U							
0.759	0.422	0.440	0.202		*0.759	0.422	0.440		*0.202		-20.8	-13.8	-14.3	-22.8	U							
0.902	0.425	0.446	0.236		0.902	0.425	0.446		0.236		-5.8	-13.2	-13.1	-9.8								
0.965	0.492	0.499	0.261		0.965	0.492	0.499		0.261		0.7	0.5	-2.8	-0.3								
0.978	0.311	0.565	0.240		0.978	*0.311	0.565		*0.560		2.1	-36.5	10.0	106.3	U							
1.014	0.547	0.562	0.278		1.014	0.547	0.562		0.278		5.9	11.7	9.4	6.2								
0.904	0.463	0.507	0.252		0.904	0.463	0.507		0.252		-5.6	-5.5	-1.3	-3.7								
1.049	0.602	0.601	0.299		1.049	*0.602	0.601		0.299		9.5	22.9	17.0	14.2								
1.003	0.572	0.554	0.295		1.003	0.572	0.554		0.295		4.7	16.8	7.9	12.7								
0.988	0.550	0.489	0.268		0.988	0.550	0.489		0.268		3.1	12.3	-4.8	-5.3								
0.968	0.565	0.639	0.302		0.968	0.565	0.639		0.302		1.1	15.4	24.4	15.4								
0.959	0.509	0.584	0.237		0.959	0.509	0.584		0.237		0.1	3.9	13.7	-9.5								
0.840	0.472	0.485	0.277		0.840	0.472	0.485		0.277		-12.3	-3.6	-5.6	5.6								
1.580	0.830	0.860	0.460		*1.580	*0.830	*0.860		*0.460		64.9	69.5	67.5	75.7	U							
	0.897	0.494	0.488	0.260		NIST	0.897	0.494	0.488	0.260	-6.4	-6.4	-0.9	-0.7								
Grand AVG	0.976	0.504	0.528	0.276	TRIMMED AVG	0.958	0.490	0.514	0.262													
SD	0.138	0.081	0.065	0.062	SD	0.062	0.038	0.051	0.025													
RSD	14.2	16.1	23.4	6.5	RSD	6.5	7.8	10.0	9.7													
					PREV TRIMMED AVG	0.921	0.485	0.515	0.259													
					DATE	1/88	1/90	5/90	1/90													

Grand Avg = all values included.

L = Late results not included in the statistics TRIMMED AVG = the mean of the values that fall within + or - 2 standard deviations.

All concentrations are reported in ug/mL.

* = Value found to be outside + or - 2 standard deviations of the average.

Table 2.

B

C

A Round Robin XXI Alpha-Tocopherol Results

Lab #	Serum # 144	Serum # 145	Serum # 146	Serum # 147	Lab #	Serum # 144	Serum # 145	Serum # 146	Serum # 147	Round Robin XXI Alpha-Tocopherol Results
10.54	6.72	6.68	4.84		10.54	6.72	6.68	4.84		
10.72	6.51	6.21	4.52		10.72	6.51	6.21	4.52		
10.91	7.22	6.90	5.10		10.91	7.22	6.90	5.10		
11.81	7.20	7.95	5.51		11.81	7.20	*7.95	5.51		
11.23	7.09	6.71	4.99		11.23	7.09	6.71	4.99		
11.10	6.80	6.90	5.20		11.10	6.80	6.90	5.20		
11.00	6.70	6.60	5.10		11.00	6.70	6.60	5.10		
10.53	6.80	6.69	4.35		10.53	6.80	6.69	4.35		
8.95	5.85	5.65	4.15		*8.95	*5.85	5.65	4.15		
9.84	6.84	6.55	4.89		9.84	6.84	6.55	4.89		
10.37	6.87	6.14	4.79		10.37	6.87	6.14	4.79		
10.99	7.33	6.49	5.16		10.99	7.33	6.49	5.16		
10.30	6.70	6.63	4.75		10.30	6.70	6.63	4.75		
10.49	6.65	6.62	4.87		10.49	6.65	6.62	4.87		
12.86	6.71	3.96	3.75		12.86	6.71	*3.96	*3.75		
10.92	7.03	6.79	5.23		10.92	7.03	6.79	5.23		
11.47	7.43	6.66	4.51		11.47	7.43	6.66	4.51		
9.86	6.73	6.93	4.99		9.86	6.73	6.93	4.99		
10.70	6.89	6.44	4.92		10.70	6.89	6.44	4.92		
10.16	7.63	6.95	5.67		10.16	*7.63	6.95	5.67		
11.00	7.17	6.82	5.50		11.00	7.17	6.82	5.50		
13.48	7.90	7.43	4.62		*13.48	*7.90	7.43	4.62		
10.53	7.10	7.28	5.57		10.53	7.10	7.28	5.57		
10.69	7.14	6.71	5.10		10.69	7.14	6.71	5.10		
10.50	5.90	7.50	7.00		10.50	*5.00	7.50	*7.00		
10.92	6.70	6.38	4.70		10.92	6.70	6.38	4.70		
10.10	6.13	5.96	4.76		10.10	*6.13	5.96	4.76		
10.70	6.90	5.20			10.70	6.90	5.20			
10.32	7.77	5.94	4.06		10.32	*7.77	5.94	4.06		
12.06	7.85	8.62	6.24		12.06	7.85	8.62	6.24		
10.00	7.11	6.77	5.03		10.00	7.11	6.77	5.03		
10.50	6.63	6.17	4.47		10.50	6.63	6.17	4.47		
9.30	5.97	5.79	4.19		9.30	*5.97	5.79	4.19		
10.71	6.60	6.23	4.69		10.71	6.60	6.23	4.69		
				NIST	10.40	6.55	6.29	4.55		
Grand AVG	10.751	6.872	6.616	4.953	TRIMMED AVG	10.679	6.906	6.594	4.884	
SD	0.872	0.574	0.752	0.615	SD	0.645	0.249	0.440	0.411	
RSD	8.1	8.4	11.4	12.4	RSD	6.0	3.6	6.7	8.4	

Grand AVG = all values included.

L = Late results not included in the statistics

All concentrations are reported in ug/ml.

PREV TRIMMED AVG

DATE

TRIMMED AVG = the mean of the values that fall within + or - 2 standard deviations of the average.

DATE

1/88

1/90

SERUM #

5/90

1/90

Percent Bias from Assigned Pool Value.

Table 3.

A

Round Robin XXI Total Beta-Carotene Results

Lab #	Serum #		Serum #		Serum #		Serum #		Serum #		Serum #		Serum #		Serum #	
	144	145	146	147	144	145	146	147	144	145	146	147	144	145	146	147
0.855	1.077	0.190	0.422		0.855	1.077	0.190	0.422	0.8	1.1	-2.5	-0.1				
0.786	0.921	0.172	0.373		0.786	0.921	0.172	0.373	-7.3	-15.4	-11.7	-11.7				
0.826	1.004	0.186	0.435		0.826	1.004	0.186	0.435	-2.6	-7.8	-4.5	3.0				
0.895	1.223	0.209	0.477		0.895	1.223	0.209	0.477	5.5	12.3	7.3	13.0				
1.130	1.516	0.259	0.550		*1.130	*1.516	*0.259	*0.550	33.3	39.3	32.9	30.3	U			
0.802	1.163	0.204	0.474		0.802	1.163	0.204	0.474	-5.4	6.8	4.7	12.3				
0.882	1.138	0.136	0.384		0.882	1.138	*0.136	0.384	4.0	4.5	-30.2	-9.1				
0.880	1.070	0.200	0.430		0.880	1.070	0.200	0.430	3.8	-1.7	2.7	1.8				
0.895	1.027	0.191	0.434		0.895	1.027	0.191	0.434	5.5	-5.7	-2.0	2.8				
0.831	1.046	0.177	0.389		0.831	1.046	0.177	0.389	-2.0	-3.9	-9.1	-7.9				
0.795	1.107	0.176	0.442		0.795	1.107	0.176	0.442	-6.2	1.7	-9.7	4.7				
0.818	1.188	0.193	0.443		0.818	1.188	0.193	0.443	-3.5	9.1	-0.9	4.9				
0.860	1.020	0.230	0.430		0.860	1.020	0.230	0.430	1.4	-6.3	18.1	1.8				
0.738	0.911	0.186	0.351		*0.738	0.911	0.186	0.351	-13.0	-16.3	-4.5	-16.9				
0.870	1.204	0.204	0.448		0.870	1.204	0.204	0.448	2.6	10.6	4.7	6.1				
0.853	1.110	0.214	0.558		0.833	1.110	0.214	*0.558	-1.8	2.0	9.8	32.2				
0.372	0.467	0.082	0.199		*0.372	*0.467	*0.082	*0.199	-56.1	-57.1	-57.9	-52.9	U			
0.980	1.130	0.221	0.495		*0.980	1.130	0.221	0.495	15.6	3.8	13.4	17.2				
0.918	1.200	0.168	0.403		0.918	1.200	0.168	0.403	8.3	10.2	-13.8	-4.6				
0.601	1.290	0.296	0.561		0.601	1.290	0.296	0.561	-29.1	18.5	51.9	32.9	U			
1.650	2.360	0.527	0.905		*1.650	*2.360	*0.527	*0.905	94.6	116.8	170.5	114.3	U			
0.854	1.065	0.196	0.401		0.854	1.065	0.196	0.401	0.7	-2.2	0.5	-5.0				
0.816	1.079	0.190	0.369		0.816	1.079	0.190	0.369	-3.8	-0.9	-2.5	-12.6				
					NIST	0.801	1.040	0.199	0.402							

Grand AVG = all values included.
L = Late results not included in the statistics
All concentrations are reported in ug/ml.

Grand AVG 0.865 1.144 0.209 0.451
SD 0.219 0.323 0.080 0.125
RSD 25.4 28.2 38.3 27.7

TRIMMED AVG SD RSD

PREV TRIMMED AVG DATE

1/88 1/90 5/90 1/90

L = Late results not included in the statistics
All concentrations are reported in ug/ml.
TRIMMED AVG = the mean of the values that fall within + or - 2 standard deviations of the average.
* = Value found to be outside + or - 2 standard deviations of the average.

Appendix C. Updated “All-Lab Report” for RR21

The following five pages are a revised version of an “All-Lab” report for RR21. This report has three parts:

- pages 1 to 3 list results for all analytes reported at least twice, counting both participants and NIST analysts.
- page 4 provides a legend for pages 1 thru 3.
- page 5 summarizes each participant’s performance for retinol, α -tocopherol, and total β -carotene. These summaries are compatible with the percent bias evaluation advice given in the RR21 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 Average” used in the original and detailed in Appendix B. These original reference values were estimated from on-time results, 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 RR21. 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 results designated NISTc in this updated All-Lab report are designated as NIST in the Tables of Appendix B.

Round Robin XXI Laboratory Results

Lab	Total Retinol				Retinyl Palmitate				α-Tocopherol				γ/β-Tocopherol			
	144	145	146	147	144	145	146	147	144	145	146	147	144	145	146	147
FSV-BA	0.989	0.506	0.531	0.274	0.038	0.027	0.010	0.012	10.49	6.65	6.62	4.87	5.21	2.02	5.22	4.20
FSV-BD	0.932	0.507	0.552	0.273					11.10	6.80	6.90	5.20				
FSV-BE	0.951	0.468	0.500	0.263					10.37	6.87	6.14	4.79				
FSV-BF	0.892	0.470	0.505	0.226					10.53	6.80	6.69	4.35	2.40	0.90	2.50	1.90
FSV-BH	0.890	0.483	0.472	0.261	0.039	nd	nd	nd	10.92	7.03	6.79	5.23	2.50	0.91	2.51	2.22
FSV-BI	0.995	0.525	0.544	0.273	0.059	nd	nd	nd	10.72	6.51	6.21	4.52	2.87	1.23	2.90	2.50
FSV-BJ	0.965	0.492	0.499	0.261					10.69	7.14	6.71	5.10	3.11	1.11	2.92	2.53
FSV-BK	0.978	0.311	0.565	0.540					10.50	5.00	7.50	7.00				
FSV-BL	1.145	0.599	0.610	0.315					11.00	7.17	6.82	5.50				
FSV-BM	0.902	0.425	0.446	0.236					10.53	7.10	7.28	5.57				
FSV-BY	0.934	0.483	0.468	0.253	0.045	nd	0.014	nd	10.54	6.72	6.68	4.84	2.99	1.20	3.00	2.53
FSV-BZ									9.86	6.73	6.93	4.99				
FSV-CA	1.062	0.521	0.592	0.299					11.81	7.20	7.95	5.51				
FSV-CH	0.968	0.565	0.639	0.302					12.06	7.85	8.62	6.24				
FSV-CJ	1.086	0.518	0.533	0.279					10.91	7.22	6.90	5.10				
FSV-CK	0.868	0.446	0.435	0.224					11.47	7.43	6.66	4.51				
FSV-CM									10.50	7.10	7.30	5.90				
FSV-CO	0.960	0.507	0.529	0.277					11.23	7.09	6.71	4.99				
FSV-CP																
FSV-CQ	0.910	0.494	0.492	0.258					8.95	5.85	5.65	4.15				
FSV-CV	0.904	0.463	0.507	0.252					10.10	6.13	5.96	4.76	2.57	1.38	2.65	2.44
FSV-CY	0.960	0.500	0.500	0.250					10.70	6.89	6.44	4.92				
FSV-DC	0.955	0.499	0.561	0.281					10.30	6.70	6.63	4.75	2.75	0.98	2.68	2.26
FSV-DE									9.84	6.84	6.55	4.89	2.51	1.01	2.66	2.24
FSV-DG	0.920	0.510	0.500	0.260					10.99	7.33	6.49	5.16				
FSV-DH	0.759	0.422	0.440	0.202	0.166	0.172	0.068	0.097	13.48	7.90	7.43	4.62	3.29	1.00	3.18	2.49
FSV-DL	0.988	0.550	0.489	0.248												
FSV-DM	1.580	0.830	0.860	0.460					9.30	5.97	5.79	4.19				
FSV-DN	1.060	0.480	0.523	0.259					11.00	6.70	6.60	5.10				
FSV-DY	0.540	0.514	0.907	0.279												
FSV-DZ	1.115	0.421	0.412	0.214					12.86	6.71	3.96	3.75				
FSV-EB	1.003	0.572	0.554	0.295					10.32	7.77	5.94	4.06				
FSV-EF	0.770	0.430	0.440	0.210					10.16	7.63	6.95	5.67	2.60	1.33	2.87	2.53
FSV-EJ	0.957	0.509	0.584	0.237					10.00	7.11	6.77	5.03	3.25	na	3.64	2.52
FSV-EV	1.049	0.602	0.601	0.299					10.70	6.90	6.90	5.20				
FSV-EY	1.014	0.547	0.562	0.278					10.92	6.70	6.38	4.70				
FSV-FL	0.897	0.494	0.488	0.260					10.71	6.60	6.23	4.69				
FSV-FR	0.840	0.472	0.485	0.277					10.50	6.63	6.17	4.47				
n	34	34	34	34	5	2	3	2	35	35	35	35	12	11	12	12
Min	0.540	0.311	0.412	0.202	0.038	0.027	0.010	0.012	8.95	5.00	3.96	3.75	2.40	0.90	2.50	1.90
Mean	0.963	0.504	0.539	0.276	0.069	0.099	0.031	0.055	10.74	6.88	6.64	4.98	3.00	1.19	3.06	2.53
Max	1.580	0.830	0.907	0.540	0.166	0.172	0.068	0.097	13.48	7.90	8.62	7.00	5.21	2.02	5.22	4.20
SD	0.155	0.080	0.103	0.064	0.055	0.103	0.032	0.060	0.86	0.57	0.75	0.63	0.76	0.32	0.75	0.56
CV	10	10	11	12	33	60	48	62	6	7	9	9	15	16	14	13
NISTc	0.921	0.488	0.520	0.254					10.40	6.55	6.29	4.55	2.58	0.79	2.65	2.01
Median	0.958	0.500	0.515	0.262	0.045				10.69	6.87	6.68	4.92	2.81	1.11	2.89	2.50
eSD	0.082	0.039	0.061	0.024	0.011				0.46	0.36	0.38	0.42	0.44	0.20	0.34	0.07
eCV	9	8	12	9	24				4	5	6	8	16	18	12	3

Round Robin XXI Laboratory Results

Lab	Total β -Carotene				trans- β -Carotene				Total cis- β -Carotene			
	144	145	146	147	144	145	146	147	144	145	146	147
FSV-BA	0.831	1.046	0.177	0.389	0.779	0.986	0.169	0.363	0.052	0.060	0.008	0.026
FSV-BD												
FSV-BE												
FSV-BF	0.802	1.163	0.204	0.474								
FSV-BH	0.795	1.107	0.176	0.442	0.744	1.045	0.170	0.415	0.051	0.062	0.006	0.027
FSV-BI	0.786	0.921	0.172	0.373								
FSV-BJ	0.870	1.204	0.204	0.448								
FSV-BK												
FSV-BL												
FSV-BM												
FSV-BY	0.855	1.077	0.190	0.422								
FSV-BZ												
FSV-CA												
FSV-CH	0.601	1.290	0.296	0.561								
FSV-CJ	0.826	1.004	0.186	0.435								
FSV-CK	0.818	1.188	0.193	0.443	0.730	1.075	0.172	0.396	0.088	0.113	0.021	0.047
FSV-CM												
FSV-CO	0.895	1.223	0.209	0.477								
FSV-CP	0.372	0.467	0.082	0.199								
FSV-CQ	0.882	1.138	0.136	0.384								
FSV-CV	0.833	1.110	0.214	0.558								
FSV-CY	0.860	1.020	0.230	0.430								
FSV-DC	0.895	1.027	0.191	0.434								
FSV-DE												
FSV-DG	0.880	1.070	0.200	0.430								
FSV-DH	0.738	0.911	0.186	0.351	0.687	0.849	0.177	0.325	0.051	0.062	0.009	0.026
FSV-DL	0.918	1.200	0.168	0.403								
FSV-DM												
FSV-DN	1.130	1.516	0.259	0.550								
FSV-DY	0.864	1.131	0.188	0.419								
FSV-DZ												
FSV-EB	0.980	1.130	0.221	0.495								
FSV-EF												
FSV-EJ	1.650	2.360	0.527	0.905								
FSV-EV												
FSV-EY												
FSV-FL	0.816	1.079	0.190	0.369								
FSV-FR	0.854	1.065	0.196	0.401								
n	24	24	24	24	4	4	4	4	4	4	4	4
Min	0.372	0.467	0.082	0.199	0.687	0.849	0.169	0.325	0.051	0.060	0.006	0.026
Mean	0.865	1.144	0.208	0.450	0.735	0.989	0.172	0.375	0.061	0.074	0.011	0.032
Max	1.650	2.360	0.527	0.905	0.779	1.075	0.177	0.415	0.088	0.113	0.021	0.047
SD	0.214	0.316	0.078	0.123	0.038	0.100	0.004	0.040	0.018	0.026	0.007	0.010
CV	13	13	15	14	5	9	2	10	21	23	32	22
NISTc	0.801	1.040	0.199	0.402	0.723	0.950	0.179	0.360	0.078	0.090	0.020	0.042
Median	0.855	1.109	0.192	0.432								
eSD	0.058	0.119	0.023	0.063								
eCV	7	11	12	15								

C3

Round Robin XXI Laboratory Results

Lab	Total Lycopene			
	144	145	146	147
FSV-BA	0.140	0.218	0.133	0.154
FSV-BD				
FSV-BE				
FSV-BF	0.256	0.360	0.211	0.270
FSV-BH	0.279	0.447	0.243	0.341
FSV-BI	0.178	0.247	0.146	0.193
FSV-BJ	0.197	0.267	0.166	0.215
FSV-BK				
FSV-BL				
FSV-BM				
FSV-BY	0.275	0.388	0.247	0.281
FSV-BZ				
FSV-CA				
FSV-CH				
FSV-CJ				
FSV-CK	0.259	0.420	0.217	0.307
FSV-CM				
FSV-CO				
FSV-CP	0.128	0.178	0.120	0.154
FSV-CQ				
FSV-CV	0.360	0.515	0.317	0.450
FSV-CY				
FSV-DC	0.192	0.258	0.120	0.282
FSV-DE				
FSV-DG				
FSV-DH	0.317	0.449	0.283	0.315
FSV-DL				
FSV-DM				
FSV-DN				
FSV-DY	0.182	0.301	0.231	0.231
FSV-DZ				
FSV-EB				
FSV-EF				
FSV-EJ	1.660	1.570	1.670	1.630
FSV-EV				
FSV-EY				
FSV-FL				
FSV-FR				

n 13 13 13 13

Min 0.128 0.178 0.120 0.154

Mean 0.340 0.432 0.316 0.371

Max 1.660 1.570 1.670 1.630

SD 0.402 0.357 0.412 0.387

CV 24 23 25 24

NISTc

Median 0.256 0.360 0.217 0.281

eSD 0.095 0.138 0.098 0.089

eCV 37 38 45 32

Round Robin XXI Laboratory Results

Analytes Reported By One Laboratory

Values in $\mu\text{g/mL}$

Analyte	Code	144	145	146	147
Total α -Carotene	FSV-BJ	0.028	0.026	0.011	0.016

Table Legend

Symbol	Interpretation
<i>na</i>	Not available (not reported by participant)
<i>nd</i>	Not detected (not reported or reported as '0', 'not determined', etc.)
<i>italics</i>	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): $100 * \text{SD}/\text{Mean}$
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, $100 * \text{eSD}/\text{Median}$

Round Robin XXI Laboratory Results

%Bias Summary

Lab	TR	aT	bC	Label	Definition
FSV-BA	3±1	-2±1	-7±3	Lab	Participant code
FSV-BD	3±4	3±3		TR	Total Retinol
FSV-BE	-2±3	-3±3		aT	a-Tocopherol
FSV-BF	-7±5	-3±5	4±7	bC	Total or trans-b-Carotene
FSV-BH	-5±4	3±2	-3±5	% Bias	(Mean ± SD) of individual serum biases
FSV-BI	5±1	-5±4	-12±4		
FSV-BJ	-1±2	2±2	5±3	Mean	Average of $(x_i - \text{Median}_i)/\text{Median}_i$
FSV-BK	20±61	6±29		SD	Standard deviation of $(x_i - \text{Median}_i)/\text{Median}_i$
FSV-BL	20±1	5±4		x_i	Result for analyte in serum _i
FSV-BM	-11±4	6±6		Median _i	Median of non-NIST results in serum _i
FSV-BY	-5±3	-1±1	-2±1		
FSV-BZ		-1±5			
FSV-CA	11±5	12±6			
FSV-CH	13±10	21±8	18±35		
FSV-CJ	7±5	4±1	-4±4		
FSV-CK	-13±3	2±8	1±5		
FSV-CM		8±9			
FSV-CO	2±2	3±2	9±3		
FSV-CP			-56±2		
FSV-CQ	-3±2	-16±1	-9±15		
FSV-CV	-5±2	-8±4	10±14		
FSV-CY	-2±2	-1±2	3±12		
FSV-DC	4±5	-3±1	-1±5		
FSV-DE		-3±4			
FSV-DG	-1±3	3±4	1±3		
FSV-DH	-18±4	12±13	-13±7		
FSV-DL	1±7		-1±10		
FSV-DM	68±5	-14±1			
FSV-DN	2±6	1±3	33±4		
FSV-DY	10±49		0±2		
FSV-DZ	-9±17	-12±26			
FSV-EB	10±5	-5±13	12±6		
FSV-EF	-17±3	6±9			
FSV-EJ	1±9	0±4	122±36		
FSV-EV	15±5	2±3			
FSV-EY	8±2	-2±3			
FSV-FL	-3±3	-4±3	-6±6		
FSV-FR	-5±7	-6±3	-2±4		
NISTc	-2±2	-5±2	-4±5		

Appendix D. Shipping Package Inserts for RR22

Two items were included in each package shipped to an RR22 participant:

- **Cover letter.** The original letter has been lost. It likely would have described the three lyophilized sample materials (sera 148 to 150), given guidance on reconstituting these lyophilized samples, stated that results were due July 9, 1991 and to whom they should be sent, and who to contact with technical questions.
- **Datasheet.** The following page reproduces the form.

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

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

SAMPLE #	ANALYTE	RESULT		
SERUM 148 VIAL # _____	RETINOL ALPHA-TOCOPHEROL BETA-CAROTENE (TOTAL)	_____ _____ _____		
SERUM 149 VIAL # _____	RETINOL ALPHA-TOCOPHEROL BETA-CAROTENE (TOTAL)	_____ _____ _____		
SERUM 150 VIAL # _____	RETINOL ALPHA-TOCOPHEROL BETA-CAROTENE (TOTAL)	_____ _____ _____		
OPTIONAL ANALYTES : SUPPLY ONE RESULT IF AVAILABLE				
	SERUM #	148	149	150
TRANS-BETA-CAROTENE		_____	_____	_____
ALPHA-CAROTENE		_____	_____	_____
RETINYL PALMITATE		_____	_____	_____
GAMMA-TOCOPHEROL		_____	_____	_____
LYCOPENE (TOTAL)		_____	_____	_____
9-CIS-BETA-CAROTENE		_____	_____	_____
13-CIS-BETA-CAROTENE		_____	_____	_____
LUTEIN		_____	_____	_____
ZEAXANTHIN		_____	_____	_____
BETA-CRYPTOXANTHIN		_____	_____	_____

RECONSTITUTE WITH 1.0 mL of WATER.

Appendix E. Final Report for RR22

The following nine pages are the final report for RR22 as provided to all participants:

- A cover letter and discussion.
- Tables 1 to 3 that list the results and various summary values for retinol, α -tocopherol, and total β -carotene.
- A page of unnumbered tables that list the results and simple summary statistics for *trans*- β -carotene, α -carotene, retinyl palmitate, γ -tocopherol, total lycopene, lutein, zeaxanthin, lutein & zeaxanthin, and β -cryptoxanthin.
- Graphical presentations of interlaboratory precision over time for retinol, α -tocopherol, and total β -carotene.

When the isomeric form of an analyte is not specified, it is likely that most participants reported “total” (the sum of all isomers.) Since resolution of γ -tocopherol and β -tocopherol is challenging, the results reported as γ -tocopherol can be confidently assumed to be γ/β -tocopherol.

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 RR22 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

July 31, 1991

Dear Colleague:

Enclosed is a packet describing both overall and your labs performance in NIST/NCI Fat-Soluble Vitamins in Serum Round-Robin XXII. Specifically, the packet contains for retinol, α -tocopherol and β -carotene, respectively: a tabular presentation of all data received; a graphical presentation of interlaboratory precision as a function of time; a Control Chart representing your labs performance vs. the assigned value for all samples analyzed over the past four years. Tabular data only are presented for α -carotene, all trans- β -carotene, β -cryptoxanthin, lutein, lycopene, retinyl palmitate, γ -tocopherol and zeaxanthin.

Data for retinol, α -tocopherol and β -carotene are provided in Tables 1-3. All values reported for the study are provided in section "A" of each respective Table. Section "B" of Tables 1-3 provides a "Trimmed Average", standard deviation, and identification of values dropped from the pool during the trimming process (indicated by an asterisk). Simply stated, "Trimmed Average" is determined by iteratively dropping all values outside two standard deviations of the floating average. The "C" sections of Tables 1-3 provide the bias, relative to the "Trimmed Average" for your analysis of each of the three samples that comprised Round-Robin XXII. The three serum samples used in Round-Robin XXII were taken from pools that are currently being analyzed for issuance as SRM 968a (a replacement for SRM 968). Certification will be based on data from three independent sets of NIST measurements along with data from seven additional labs. The preliminary data from their analyses are also provided in Tables 1-3 under "PRE SRM".

As in past studies, the "Trimmed Average" is used as the Assigned Pool Value for all three core analytes (retinol, α -tocopherol and β -carotene). For retinol and α -tocopherol, the agreement among the "Trimmed Average", NIST value and the "SRM Labs Average" is excellent and allows us to continue the convention of 0-5% bias from the assigned value representing EXCEPTIONAL performance, 6-10% ACCEPTABLE performance, 11-20% MARGINAL performance, and >20% UNACCEPTABLE with respect to the current state-of-the-practice for measurement of these analytes in serum/plasma. For β -carotene, the agreement among the three values is not as good and we suggest that, for the time being, any values that are within \pm 20% of the "Trimmed Average" be deemed acceptable. We see this as only an interim measure and intend to resume our normal data quality expectations for β -carotene in the next Round Robin.

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 968 and analyze all three levels. SRM 968 can be obtained from the NIST Standard Reference Materials Program (301/975-6776) at a cost of \$154/unit plus shipping. If, with minor method modifications, your measured values do not agree with the certified values, feel free to contact us for consultation. We are willing to provide in-house consultation (at your expense) for you or one of your staff if need be, but have found that most problems can be solved via telephone conversations. Please contact Dr. Neal E. Craft at 301/975-3111. As will be discussed later, we are also willing to arrange a hands-on laboratory tutorial session in conjunction with our QA workshop this fall. Sustained poor performance can result in suspension from the program.

July 31, 1991
Page 2

Fifteen new labs joined the program during the past year, most of which have considerable experience in performing fat-soluble vitamins in serum measurements. Their joining gives us another opportunity to gauge the impact that the NIST/NCI QA program is having on improving interlaboratory precision for fat-soluble vitamin/serum measurements. As indicated by the tabular data for retinol, and α -tocopherol, the interlaboratory precision for the core labs (participants for at least one-year) is a factor of five better than the new labs and is in better agreement with the preliminary SRM certified value. Though not as dramatic, the β -carotene values from the core labs are also more harmonious than those from the new labs.

The QA group has expanded by 20% during the past year and all indications are that this rate of growth will continue for the next few years. Therefore, beginning January 1991, we will begin charging a service fee to help defray the cost of sample shipments. The fee shall be \$300 annually for US labs and \$600 annually for non-US labs. Details concerning the billing structure will be provided to you shortly.

Our annual QA Workshop will be held on Saturday, October 19 here at NIST. Among other things, the agenda will include: a review of 1990 Round-Robin results; an update on NIST fat-soluble vitamin analysis automation activities; a discussion of QA needs for new analytes; discussion of long-term QA program structure. More detailed information concerning the program agenda will be forthcoming. We invite your suggestions for discussion topics. Depending on interest/need, we are willing to arrange a Friday hands-on laboratory session for new labs and those experiencing chronic measurement problems. Please indicate your interest on the enclosed form.

Samples for Round-Robin XXIII will be shipped on Tuesday, August 6. Four serum samples and an extract are provided. One mL of the extract corresponds to 1 mL of serum. Therefore, for those who do quantitation based on externals, simply provide the concentrations of retinol and α -tocopherol in the solution provided. Retinyl acetate (1.0 μ g/mL) and α -tocopherol acetate (40.0) and tocol (20.7) have been added for those who base their quantitation on internal standards. Process the hexane extract provided as you would any serum/plasma extract routinely analyzed in your lab. Report concentrations of retinol and α -tocopherol relative to the most appropriate of the internal standards that we have added.

Sincerely

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

Enclosure

cc: N. E. Craft

Table 1.

A

B

C

Round Robin XXII Retinol Results				Round Robin XXII Retinol Results				Round Robin XXII Percent Bias from Trimmed Average.		
Lab #	Serum # 148	Serum # 149	Serum # 150	Lab #	Serum # 148	Serum # 149	Serum # 150	Serum # 148	Serum # 149	Serum # 150
	0.183	0.472	0.636		0.183	0.472	0.636	-1.7	-3.4	-3.0
	0.179	0.488	0.650		0.179	0.488	0.650	-3.6	-0.2	-1.0
	0.185	0.495	0.678		0.185	0.495	0.678	-0.6	1.3	3.4
	0.192	0.444	0.554		0.192	0.444	0.554	3.2	-9.1	-15.5
	0.202	0.578	0.753		0.202	*0.578	0.753	8.5	18.3	14.8
	0.175	0.468	0.616		0.175	0.468	0.616	-5.8	-4.2	-6.2
	0.204	0.522	0.704		0.204	0.522	0.704	9.6	6.9	7.3
	0.198	0.538	0.726		0.198	0.538	0.726	6.4	10.2	10.7
	0.144	0.411	0.581		*0.144	*0.411	0.581	-22.6	-15.9	-11.4
	0.193	0.500	0.679		0.193	0.500	0.679	3.7	2.4	3.5
	0.211	0.487	0.645		*0.211	0.487	0.645	13.5	-0.2	-1.6
	0.196	0.514	0.697		0.196	0.514	0.697	5.3	5.2	6.3
	0.197	0.508	0.657		0.197	0.508	0.657	5.8	4.0	0.2
	0.185	0.489	0.664		0.185	0.489	0.664	-0.7	0.2	1.2
	0.178	0.478	0.638		0.178	0.478	0.638	-4.6	-2.2	-2.8
	0.154	0.441	0.577		*0.154	0.441	0.577	-17.3	-9.7	-12.0
	0.170	0.490	0.650		0.170	0.490	0.650	-8.7	0.3	-0.9
	0.190	0.510	0.650		0.190	0.510	0.650	2.1	4.4	-0.9
	0.232	0.522	0.675		*0.232	0.522	0.675	24.7	6.9	2.9
	0.230	0.557	0.703		*0.230	*0.557	0.703	23.6	14.0	7.2
	0.171	0.477	0.536		0.171	0.477	*0.536	-8.1	-2.3	-18.3
	0.190	0.497	0.658		0.190	0.497	0.658	2.1	1.8	0.3
	0.184	0.481	0.665		0.184	0.481	0.665	-1.1	-1.5	1.4
	0.272	0.570	0.693		*0.272	*0.570	0.693	46.1	16.7	5.6
	0.218	0.616	0.805		*0.218	*0.616	*0.805	17.1	26.1	22.7
	0.187	0.500	0.687		0.187	0.500	0.687	0.5	2.4	4.7
	0.186	0.509	0.646		0.186	0.509	0.646	-0.1	4.2	-1.5
	0.348	0.649	0.829		*0.348	*0.649	*0.829	87.0	32.9	26.4
	0.182	0.457	0.585		0.182	0.457	0.585	-2.2	-6.4	-10.8
	0.187	0.464	0.595		0.187	0.464	0.595	0.5	-5.0	-9.3
	0.218	0.554	0.740		*0.218	*0.554	0.740	17.1	13.4	12.8
	0.169	0.504	0.604		0.169	0.504	0.604	-9.2	3.2	-7.9
	0.230	0.660	0.870		*0.230	*0.660	*0.870	23.6	35.1	32.6
	0.206	0.498	0.646		0.206	0.498	0.646	10.7	2.0	-1.5
	0.170	0.460	0.630		0.170	0.460	0.630	-8.7	-5.8	-4.0
	0.133	0.289	0.357		*0.133	*0.289	*0.357	-28.5	-40.8	-45.6
	0.180	0.480	0.680		0.180	0.480	0.680	-3.3	-1.7	3.7
	0.506	0.690	0.719		*0.506	*0.690	0.719	171.9	41.3	9.6
	0.199	0.530	0.628		0.199	0.530	0.628	6.9	8.4	-4.3
	0.120	0.064	0.263		*0.120	*0.064	*0.263	-35.5	-86.9	-59.9
	0.122	0.351	0.436		*0.122	*0.351	*0.436	-34.5	-28.1	-33.5
	0.638	1.396	2.216		*0.638	*1.396	*2.216	242.8	185.8	237.8
	0.258	0.377	0.552		*0.258	*0.377	0.552	38.5	-22.7	-15.8
	0.177	0.452	0.750		0.177	0.452	0.750	-4.9	-7.5	14.3
NIST-1	0.173	0.500	0.631							
NIST-2	0.183	0.510	0.668							
NIST-3	0.199	0.500	0.738							
ALL LABS	(44)			ALL LABS	TRIMMED			PRELIMINARY SRM VALUES		
GRAND AVG	0.210	0.510	0.680	AVG	0.186	0.488	0.656	Avg	0.184	0.485
SD	0.090	0.168	0.260	SD	0.011	0.025	0.052	SD	0.016	0.015
RSD	42.6	33.0	38.2	RSD	5.8	5.1	8.0	RSD	8.6	3.0
a CORE LABS	(31; 15-97)			CORE LABS TRIMMED						
AVERAGE	0.197	0.506	0.662	AVERAGE	0.186	0.489	0.659			
SD	0.036	0.050	0.066	SD	0.010	0.024	0.047			
RSD	18.5	9.8	9.9	RSD	5.4	5.0	7.1			
b NEW LABS	(12; 101-115)			NEW LABS TRIMMED						
AVERAGE	0.245	0.521	0.729	AVERAGE	0.179	0.479	0.594			
SD	0.161	0.321	0.499	SD	0.046	0.127	0.179			
RSD	65.7	61.7	68.4	RSD	25.5	26.4	30.2			

* = Value found to be outside + or - 2 standard deviations of the Trimmed average.

L= Late data used only for All Lab Average.

a= Involved in program for at least one-year.

b= Joined program within the last 12 months.

Table 2.

A

B

C

Round Robin XXII Alpha-Tocopherol Results			Round Robin XXII Alpha-Tocopherol Results			Round Robin XXII Percent Bias from Trimmed Average.					
Lab #	Serum # 148	Serum # 149	Serum # 150	Lab #	Serum # 148	Serum # 149	Serum # 150	Serum # 148	Serum # 149	Serum # 150	
	4.89	10.25	16.68		4.89	10.25	16.68	2.3	-2.3	1.3	
	4.47	10.23	16.96		4.47	10.23	16.96	-6.3	-2.4	2.9	
	4.95	10.34	17.48		4.95	10.34	17.48	3.7	-1.4	6.1	
	5.42	8.98	13.69		5.42	*8.98	*13.69	13.5	-14.4	-16.9	
	4.37	10.33	16.39		4.37	10.33	16.39	-8.4	-1.5	-0.5	
	4.18	9.97	15.82		4.18	9.97	15.82	-12.5	-4.9	-3.9	
	5.15	10.90	17.11		5.15	10.90	17.11	7.9	3.9	3.9	
	4.58	10.73	16.68		4.58	10.73	16.68	-4.0	2.3	1.2	
	4.19	10.47	16.27		4.19	10.47	16.27	-12.2	-0.2	-1.2	
	4.67	10.66	16.90		4.67	10.66	16.90	-2.1	1.6	2.6	
	4.44	10.60	16.68		4.44	10.60	16.68	-7.0	1.1	1.3	
	5.04	10.88	17.42		5.04	10.88	17.42	5.7	3.7	5.8	
	5.10	9.92	14.90		5.10	9.92	*14.90	6.8	-5.4	-9.5	
	4.31	10.18	15.88		4.31	10.18	15.88	-9.7	-2.9	-3.6	
	4.44	10.75	16.55		4.44	10.75	16.55	-7.0	2.5	0.5	
	4.76	10.38	16.11		4.76	10.38	16.11	-0.2	-1.0	-2.2	
	4.05	10.33	15.66		4.05	10.33	15.66	-15.2	-1.5	-4.9	
	4.89	6.02	11.10		4.89	*6.02	*11.10	2.4	-42.6	-32.6	
	4.85	10.60	16.60		4.85	10.60	16.60	1.6	1.1	0.8	
	5.24	10.47	15.56		5.24	10.47	15.56	9.8	-0.2	-5.5	
	4.59	10.04	16.20		4.59	10.04	16.20	-3.8	-4.3	-1.7	
	5.34	10.86	16.90		5.34	10.86	16.90	11.9	3.5	2.6	
	4.79	9.28	13.88		4.79	*9.28	*13.88	0.4	-11.5	-15.8	
	4.60	10.40	16.60		4.60	10.40	16.60	-3.6	-0.9	0.8	
	5.15	10.69	16.34		5.15	10.69	16.34	7.9	2.0	-0.8	
	5.05	10.27	16.03		5.05	10.27	16.03	5.8	-2.1	-2.7	
	5.60	12.37	18.84		*5.60	*12.37	*18.84	17.4	17.9	14.3	
	5.83	11.93	17.40		*5.83	*11.93	17.40	22.1	13.7	5.6	
	5.50	11.10	17.30		5.50	11.10	17.30	15.2	5.8	5.0	
	3.31	9.74	16.08		*3.31	*9.74	16.08	-30.7	-7.1	-2.4	
	4.69	10.93	18.71		4.69	10.93	*18.71	-1.7	4.2	13.6	
	4.72	9.58	14.61		4.72	9.58	14.61	-1.1	-8.7	-11.3	
	5.29	10.66	18.13		5.29	10.66	*18.13	10.7	1.6	10.1	
	4.85	11.27	16.66		4.85	*11.27	16.66	1.5	7.4	1.1	
	3.66	10.82	17.85		*3.66	10.82	*17.85	-23.3	3.2	8.4	
	4.65	10.50	15.80		4.65	10.50	15.80	-2.6	0.1	-4.1	
	4.52	10.29	16.54		4.52	10.29	16.54	-5.3	-1.9	0.4	
	2.80	8.06	9.34		*2.80	*8.06	*9.34	-41.3	-23.2	-43.3	
	5.08	10.24	16.81		5.08	10.24	16.81	6.4	-2.4	2.0	
	7.81	14.49	19.71		*7.81	*14.49	*19.71	63.6	38.1	19.7	
	5.09	11.05	16.67		5.09	11.05	16.67	6.6	5.4	1.2	
	0.30	0.20	0.95		*0.30	*0.20	*0.95	-93.7	-98.1	-94.2	
	4.40	10.71	15.53		4.40	10.71	15.53	-7.8	2.1	-5.7	
	9.91	24.38	38.41		*9.91	*24.38	*38.41	107.6	132.4	133.2	
NIST-1	4.53	10.30	16.22								
NIST-2	4.29	10.43	15.71								
NIST-3	3.96	9.18	15.98								
ALL LABS	(44)			ALL LABS	TRIMMED			PRELIMINARY SRM VALUES			
GRAND AVG	4.807	10.519	16.403	AVG	4.773	10.489	16.473	Avg	4.52	10.39	16.41
SD	1.271	2.873	4.464	SD	0.367	0.351	0.624	SD	0.30	0.36	0.53
RSD	26.4	27.3	27.2	RSD	7.7	3.4	3.8	RSD	6.6	3.5	3.2
a CORE LABS	(34; 15-97)			CORE LABS	TRIMMED						
AVERAGE	4.790	10.353	16.320	AVERAGE	4.804	10.499	16.588				
SD	0.503	1.017	1.464	SD	0.401	0.318	0.516				
RSD	10.5	9.8	9.0	RSD	8.3	3.0	3.1				
b NEW LABS	(10; 101-113)			NEW LABS	TRIMMED						
AVERAGE	4.822	11.074	16.761	AVERAGE	4.315	10.602	16.987				
SD	2.606	5.943	9.346	SD	0.824	0.316	1.416				
RSD	54.0	53.7	55.8	RSD	19.1	3.0	8.3				

* = Value found to be outside + or - 2 standard deviations of the Trimmed average.

L= Late data used only for All Lab Average.

a= Involved in program for at least one-year.

b= Joined program within the last 12 months.

Table 3.

A

Round Robin XXII
Total Beta-Carotene Results

Lab #	Serum # 148	Serum # 149	Serum # 150
	0.309	0.979	2.557
	0.225	0.767	2.024
	0.531	0.744	2.220
	0.139	0.645	1.627
	0.294	0.950	2.446
	0.361	0.937	2.606
	0.224	0.818	2.131
	0.278	0.946	2.501
	0.282	0.868	2.277
	0.288	0.991	2.580
	0.309	0.931	2.494
	0.322	1.111	2.931
	0.245	0.949	2.644
	0.330	0.900	2.530
	0.239	0.842	2.177
	0.479	0.906	2.064
	0.300	0.951	2.381
	0.503	0.948	2.100
	0.195	0.909	2.340
	0.215	0.822	2.397
	0.188	0.747	1.950
	0.169	0.685	1.784
	0.284	1.139	3.036
	0.180	0.810	2.190
	0.260	1.040	2.890
	0.230	0.840	2.140
	0.350	1.180	3.700
	0.133	0.640	1.673
	0.253	0.832	2.129
	0.205	0.965	2.510
	0.362	1.319	2.894
	0.168	0.666	2.193
	0.234	0.691	1.830
NIST-1	0.231	1.050	2.860
NIST-2	0.203	0.960	2.620

Round Robin XXII
Total Beta-Carotene Results

Lab #	Serum # 148	Serum # 149	Serum # 150
	0.309	0.979	2.557
	0.225	0.767	2.024
	*0.531	0.744	2.220
	0.139	0.645	1.627
	0.294	0.950	2.446
	0.361	0.937	2.606
	0.224	0.818	2.131
	0.278	0.946	2.501
	0.282	0.868	2.277
	0.288	0.991	2.580
	0.309	0.931	2.494
	0.322	*1.111	2.931
	0.245	0.949	2.644
	0.330	0.900	2.530
	0.239	0.842	2.177
	*0.479	0.906	2.064
	0.300	0.951	2.381
	*0.503	0.948	2.100
	0.195	0.909	2.340
	0.215	0.822	2.397
	0.188	0.747	1.950
	0.169	0.685	1.784
	0.284	1.139	3.036
	0.180	0.810	2.190
	0.260	1.040	2.890
	0.230	0.840	2.140
	0.350	1.180	3.700
	0.133	0.640	1.673
	0.253	0.832	2.129
	0.205	0.965	2.510
	0.362	1.319	2.894
	0.168	0.666	2.193
	0.234	0.691	1.830

Round Robin XXII
Percent Bias from Trimmed Average.

	Serum # 148	Serum # 149	Serum # 150
	22.4	14.9	10.2
	-10.8	-10.1	-12.8
	110.4	-12.7	-4.3
	-44.9	-24.3	-29.9
	16.5	11.4	5.4
	43.2	10.0	12.3
	-11.2	-4.0	-8.2
	10.2	11.0	7.8
	11.7	1.8	-1.9
	14.1	16.3	11.2
	22.4	9.2	7.5
	27.4	30.3	26.3
	-2.8	11.4	14.0
	30.8	5.6	9.0
	-5.3	-1.2	-6.2
	89.8	6.3	-11.0
	18.9	11.6	2.6
	99.3	11.2	-9.5
	-22.7	6.6	0.9
	-14.8	-3.6	3.3
	-25.5	-12.4	-16.0
	-33.0	-19.7	-23.1
	12.5	33.6	30.9
	-28.7	-5.0	-5.6
	3.0	22.0	24.6
	-8.9	-1.4	-7.8
	38.7	38.4	59.5
	-47.3	-24.9	-27.9
	0.2	-2.4	-8.3
	-18.8	13.2	8.2
	43.4	54.8	24.7
	-33.4	-21.9	-5.5
	-7.3	-18.9	-21.1

ALL LABS	(33)	ALL LABS	TRIMMED	PRELIMINARY SRM VALUES		
GRAND AVG	0.275	0.894	2.367	Avg	0.252	0.852
SD	0.096	0.154	0.419	SD	0.064	0.114
RSD	34.8	17.2	17.7	RSD	25.3	13.4

a CORE LABS	(23; 15-97)	CORE LABS	TRIMMED
AVERAGE	0.292	0.891	2.339
SD	0.101	0.121	0.337
RSD	34.6	13.6	14.4

b NEW LABS	(10; 101-115)	NEW LABS	TRIMMED
AVERAGE	0.238	0.898	2.415
SD	0.074	0.226	0.602
RSD	31.1	25.2	24.9

* = Value found to be outside + or - 2 standard deviations of the Trimmed average.

L= Late data used only for All Lab Average.

a= Involved in program for at least one-year.

b= Joined program within the last 12 months.

Trans-Beta-Carotene

Alpha-Carotene

Retinyl Palmitate

Lab#	Serum # 148	Serum # 149	Serum # 150	Lab#	Serum # 148	Serum # 149	Serum # 150	Lab#	Serum # 148	Serum # 149	Serum # 150
NIST-1	0.201	0.963	2.650	NIST-1	0.026	0.062	0.124		0.009	0.019	0.023
NIST-3	0.193	0.785	2.398	NIST-2	0.010	0.051	0.099		0.003	0.032	0.034
*0.273	0.925	2.420		NIST-3	0.011	0.043	0.096		*0.305	*0.509	0.042
*0.304	1.050	2.749			0.013	0.044	0.092		0.025	0.087	0.284
0.217	0.841	2.295					0.074				0.101
0.185	0.828	2.180				0.035	0.065				0.288
0.200	0.840	2.140			0.025	0.054	0.118				
0.200	0.650	1.650			0.011	0.059	0.119				
0.219	0.701	1.799			0.013	0.046	0.104	AVERAGE	0.050	0.088	0.129
AVERAGE	0.202	0.843	2.253		0.013	0.037	0.074	SD	0.076	0.088	0.125
SD	0.012	0.125	0.361		0.018	0.066	0.130	RSD	151.8	100.7	97.0
RSD	6.0	14.8	16.0			0.052	0.082				
					0.009	0.036	0.078				
					*0.082	0.129	*0.200				
					0.030	0.070	0.130				
					0.043	0.080	0.097				
					0.014	0.037	0.056				
					0.014	0.041	0.096				
				AVERAGE	0.019	0.057	0.101				
				SD	0.010	0.024	0.023				
				RSD	52.3	42.3	22.8				

Gamma-Tocopherol

Total Lycopene

Lutein

Lab#	Serum # 148	Serum # 149	Serum # 150	Lab#	Serum # 148	Serum # 149	Serum # 150	Lab#	Serum # 148	Serum # 149	Serum # 150
NIST-1	0.77	2.80	3.95	NIST-2	0.124	0.390	0.382	NIST-3	0.032	0.086	0.079
NIST-2	0.88	2.84	3.82	NIST-3	0.062	0.183	0.202		0.035	0.122	0.089
NIST-3	0.99	3.26	5.06		0.162	0.660	0.319		0.073	0.201	0.185
	1.08	3.18	4.44		0.117	0.290	0.272		0.045	0.110	0.083
	0.88	2.89	4.13		0.172		0.329		0.021	0.060	0.043
	0.91	3.17	4.33		0.101	0.360	0.312		*0.336	0.102	0.093
	0.78	2.56	3.62		0.172	0.488	0.421				
	1.29	3.17	4.32		0.167	0.431	0.371	AVERAGE	0.040	0.113	0.095
	0.86	2.72	3.73		0.141	0.388	0.398	SD	0.023	0.048	0.047
	0.94	3.12	4.33		0.216	0.420	0.351	RSD	56.2	42.3	49.9
	0.76	2.82	3.91		0.070	0.147	0.132				
	0.79	2.70	3.58		0.338	0.655	0.568				
	1.16	3.84	5.09		0.073	0.239	0.207				
	0.80	2.43	3.02		0.203	0.576	1.139				
	0.96	3.08	4.20		0.155	0.392	0.437				
	0.96	3.75	4.26		0.112	0.294	0.056				
	0.75	3.08	3.98		0.151	0.366	0.299				
	0.95	2.40	3.26								
AVERAGE	0.917	2.989	4.057	AVERAGE	0.149	0.392	0.364				
SD	0.147	0.392	0.533	SD	0.066	0.149	0.233				
RSD	16.0	13.1	13.1	RSD	44.3	38.1	64.0				

Zeaxanthin

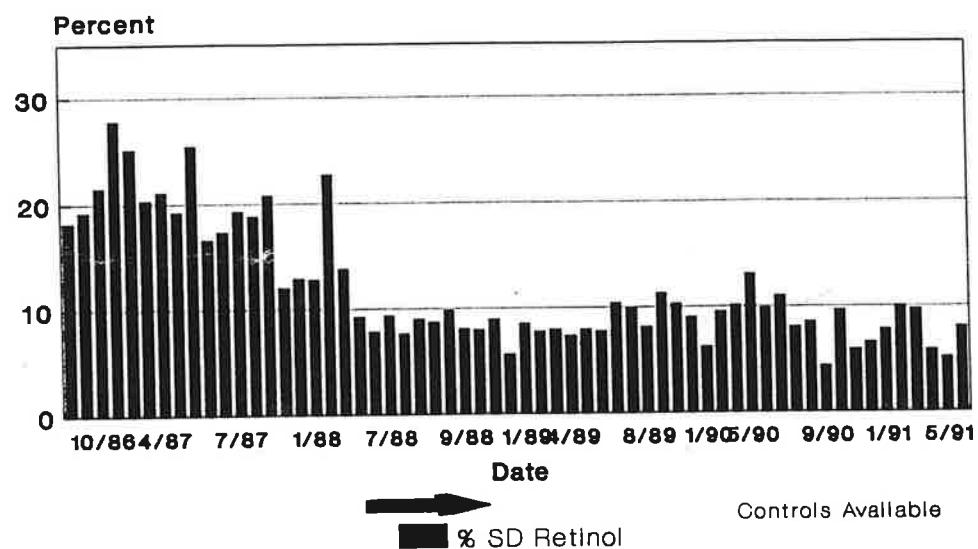
LUTEIN/ZEAXANTHIN

Beta-Cryptoxanthin

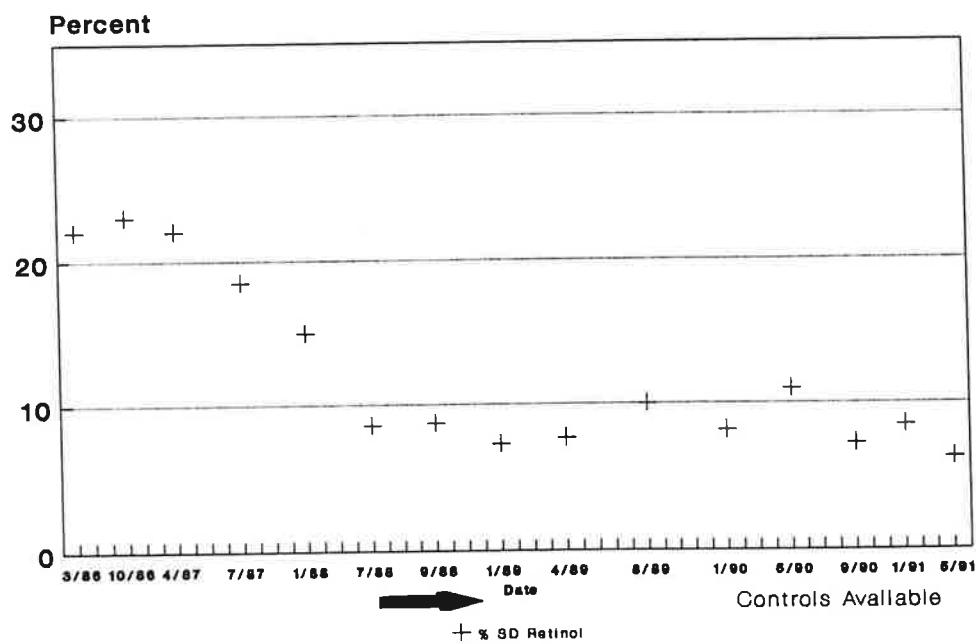
Lab#	Serum # 148	Serum # 149	Serum # 150	Lab#	Serum # 148	Serum # 149	Serum # 150	Lab#	Serum # 148	Serum # 149	Serum # 150
NIST-3	0.013	0.040	0.037		0.051	0.145	0.126	NIST-3	0.018	0.058	0.047
	*0.112	*0.224	*0.179		0.060	0.178	0.130		0.032	0.093	0.066
	0.013	0.035	0.029		0.112	0.224	0.179		0.024	0.097	0.074
	0.016	0.035	0.021						0.039	0.095	0.063
	0.017	0.041	0.016	AVERAGE	0.074	0.182	0.145		0.042	0.127	0.075
AVERAGE	0.015	0.038	0.026	SD	0.033	0.040	0.030		0.057	0.103	0.088
SD	0.002	0.003	0.009	RSD	44.3	21.8	20.4		0.031	0.089	0.061
RSD	14.4	8.6	36.3						0.055	*0.208	*0.193
									0.021	0.061	0.037
									0.022	0.063	0.047

* = Value removed before statistical analysis.

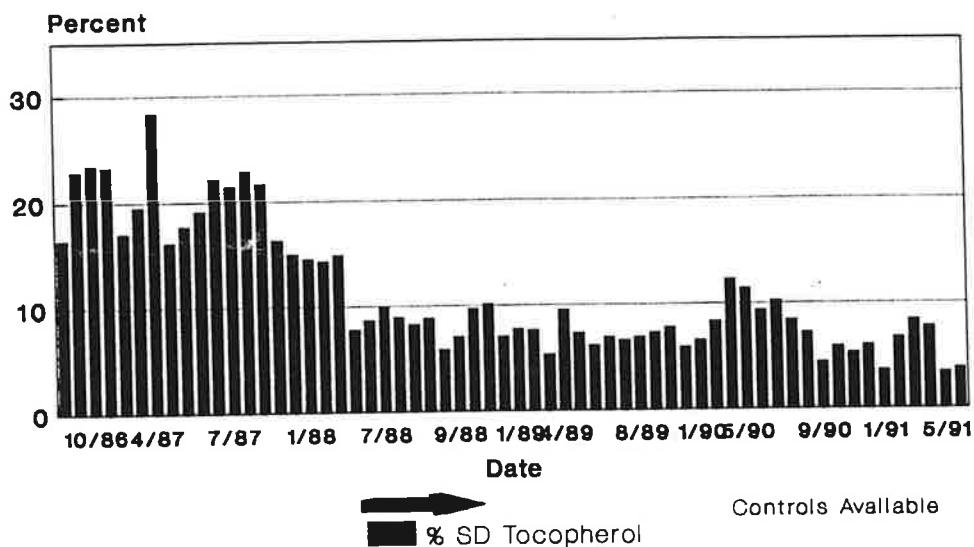
Interlaboratory Precision vs Time Retinol



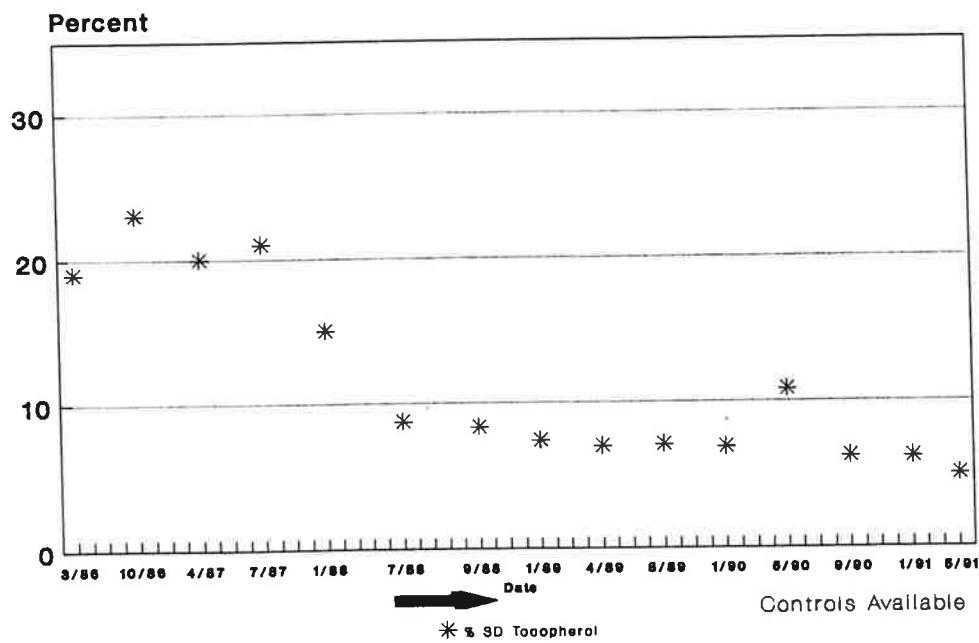
Interlaboratory Precision vs Time Retinol



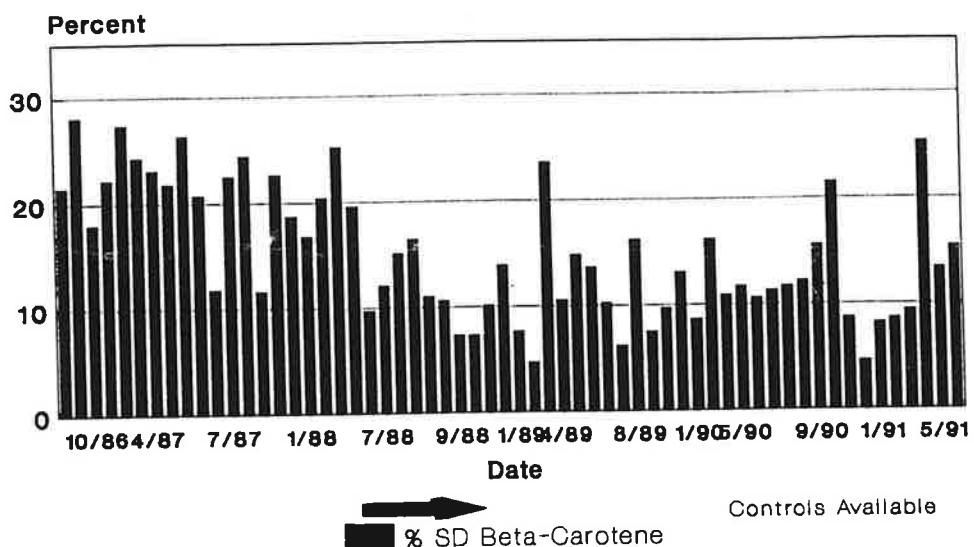
Interlaboratory Precision vs Time Alpha-Tocopherol



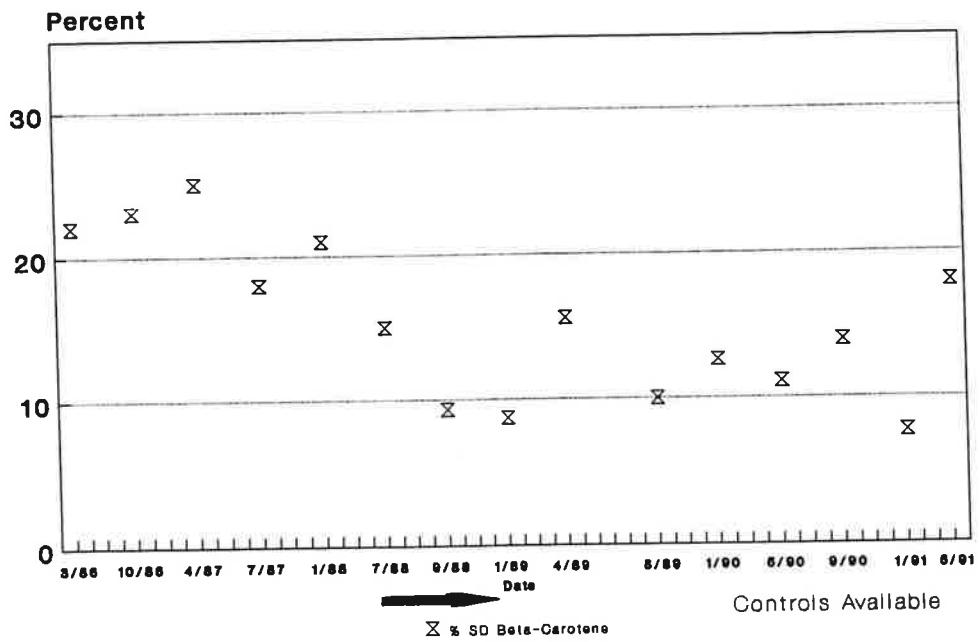
Interlaboratory Precision vs Time Alpha-Tocopherol



Interlaboratory Precision vs Time Beta-Carotene



Interlaboratory Precision vs Time Beta-Carotene



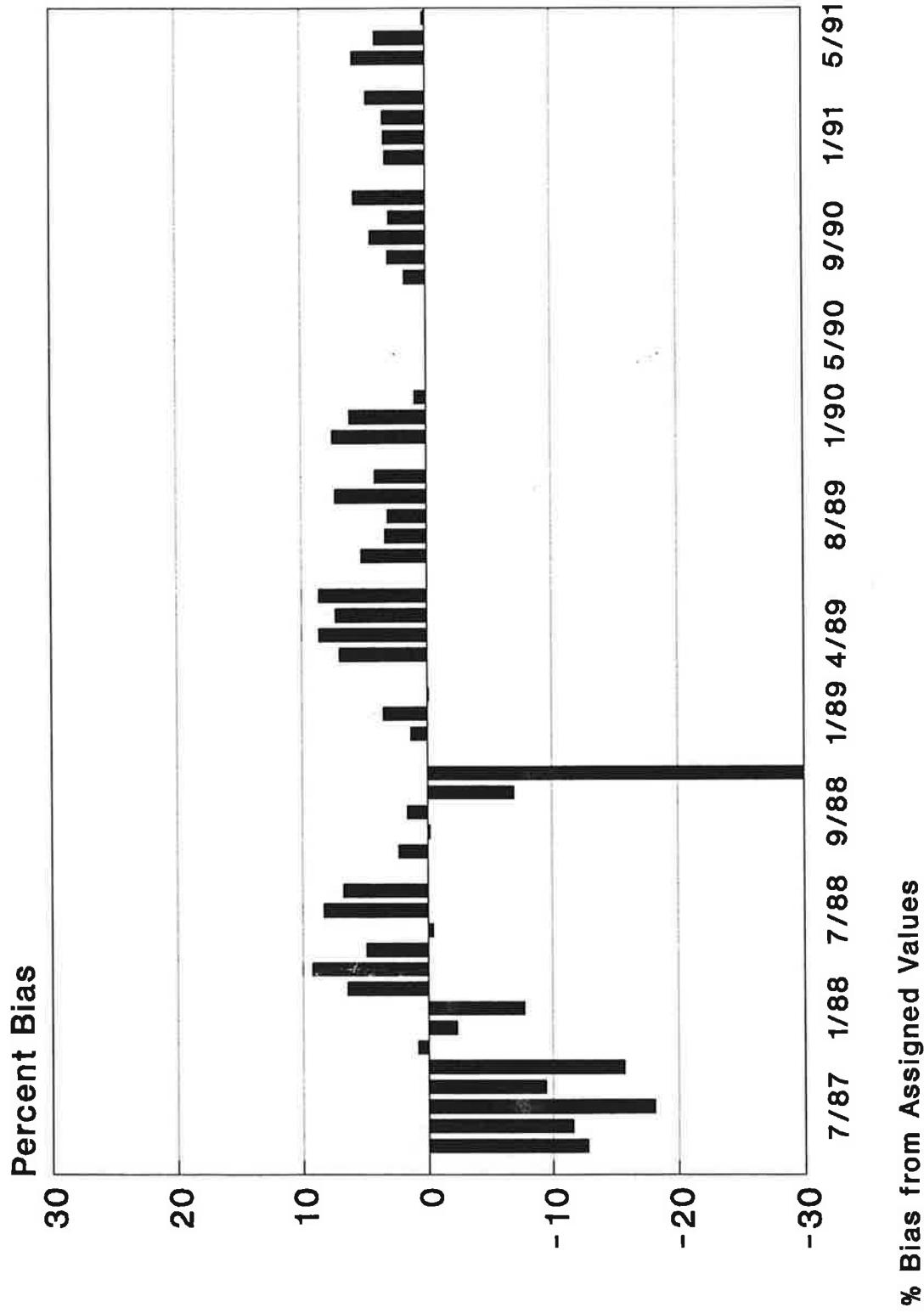
Appendix F. Representative “Individualized Report” for RR22

Each participant in RR22 received graphical summaries of their own measurement performance for total retinol, α -tocopherol, and/or total β -carotene. In RR22, one set of graphs was prepared:

- “Percent Bias” relative to the “Assigned Value” (the “Trimmed Average” of the Tables in Appendix E) for of the serum-based samples distributed from 7/1987 through 5/91.

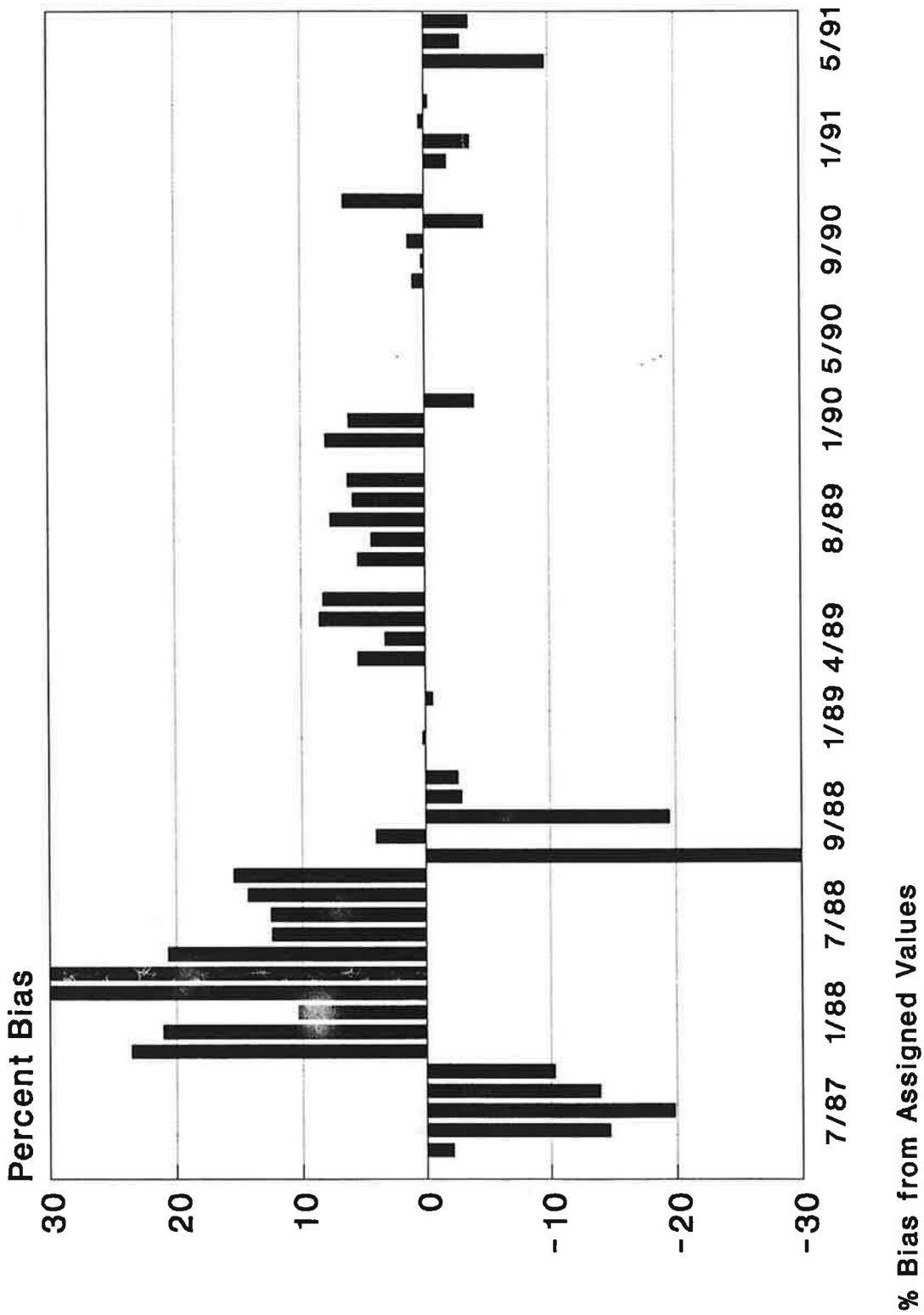
The following three pages constitute the individualized report for participant FSV-BA.

Laboratory FSV-BA
Retinol

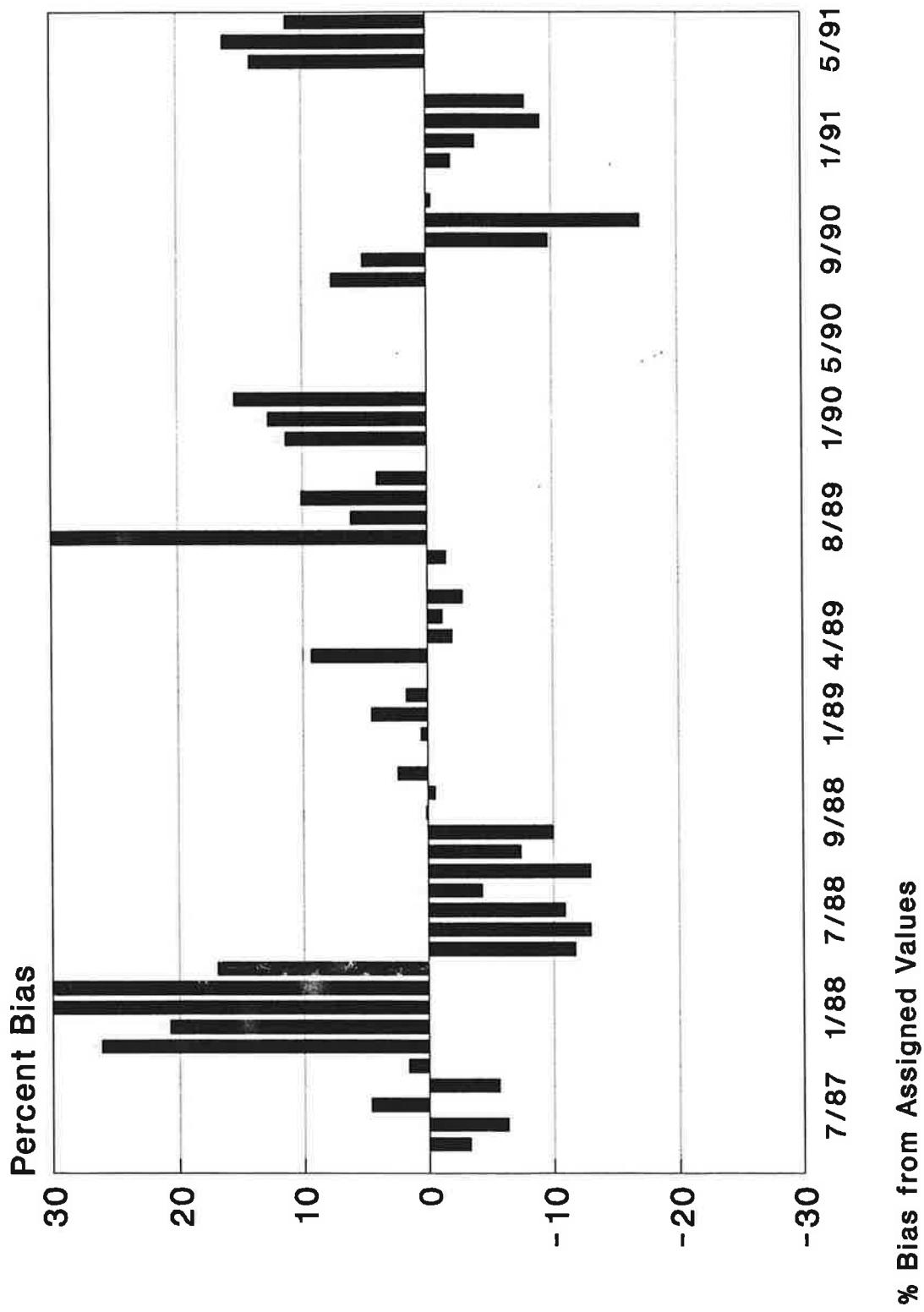


F2

Laboratory FSV-BA
Alpha-Tocopherol



Laboratory FSV-BA
Total Beta-Carotene



Appendix G. Updated “All-Lab Report” for RR22

The following five pages are an updated version of an “All-Lab” report for RR22. This report has three parts:

- pages 1 and 3 list results for all analytes reported at least twice, counting both participants and NIST analysts.
- page 4 provides a legend for pages 1 thru 3.
- page 5 summarizes each participant’s performance for retinol, α -tocopherol, and total β -carotene. These summaries are compatible with the percent bias evaluation advice given in the RR22 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 Average” used in the original and detailed in Appendix E. These original reference values were estimated from on-time results, 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 RR22. 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 results designated NISTa, NISTb, and NISTc in this updated All-Lab report are designated NIST-1, NIST-3, and NIST-2 in the Tables of Appendix E.

Round Robin XXII Laboratory Results

Lab	Total β-Cryptoxanthin			Total Lutein			Total Zeaxanthin			Total Lutein&Zeaxanthin		
	148	149	150	148	149	150	148	149	150	148	149	150
FSV-BA	0.039	0.095	0.063							0.112	0.224	0.179
FSV-BD												
FSV-BE												
FSV-BF												
FSV-BG												
FSV-BH	0.042	0.127	0.075	0.035	0.122	0.089	0.013	0.035	0.029	0.048	0.157	0.118
FSV-BI	0.032	0.093	0.066							0.051	0.145	0.126
FSV-BJ												
FSV-BK												
FSV-BL												
FSV-BM												
FSV-BN	0.022	0.063	0.047	0.021	0.060	0.043	0.016	0.035	0.021	0.037	0.095	0.064
FSV-BP	0.055	0.208	0.193									
FSV-BS												
FSV-BY												
FSV-BZ												
FSV-CA												
FSV-CB												
FSV-CH												
FSV-CJ	0.024	0.097	0.074							0.060	0.178	0.130
FSV-CK	0.057	0.103	0.088	0.073	0.201	0.185						
FSV-CM												
FSV-CO												
FSV-CP	0.026	0.063	0.041				0.044	0.108	0.087			
FSV-CQ												
FSV-CT				0.034	0.102	0.093	0.017	0.041	0.016	0.051	0.143	0.109
FSV-CU												
FSV-CV												
FSV-CX												
FSV-CY												
FSV-DC												
FSV-DE												
FSV-DG												
FSV-DH												
FSV-DL	0.031	0.089	0.061									
FSV-DM												
FSV-DW	0.021	0.061	0.037	0.045	0.110	0.083						
FSV-DY												
FSV-EA												
FSV-EB												
FSV-ED												
FSV-EF												
FSV-EI												
FSV-EJ												
FSV-EV												
FSV-EY												
FSV-FL												
FSV-FM												
FSV-FS												
FSV-FU												
n	10	10	10	5	5	5	4	4	4	6	6	6
Min	0.021	0.061	0.037	0.021	0.060	0.043	0.013	0.035	0.016	0.037	0.095	0.064
Mean	0.035	0.100	0.074	0.042	0.119	0.098	0.022	0.055	0.038	0.060	0.157	0.121
Max	0.057	0.208	0.193	0.073	0.201	0.185	0.044	0.108	0.087	0.112	0.224	0.179
SD	0.013	0.043	0.045	0.020	0.051	0.052	0.014	0.036	0.033	0.027	0.043	0.037
CV	23	21	23	27	26	28	33	33	38	24	19	21
NISTa												
NISTb												
NISTc	0.018	0.058	0.047	0.032	0.086	0.079	0.013	0.040	0.037	0.045	0.126	0.116
Median	0.032	0.094	0.065	0.035	0.110	0.089				0.051	0.151	0.122
eSD	0.013	0.030	0.021	0.014	0.018	0.009				0.009	0.026	0.016
eCV	40	32	33	41	16	10				17	17	13

Round Robin XXII Laboratory Results

Table Legend

Symbol	Interpretation
<i>na</i>	Not available (not reported by participant)
<i>nd</i>	Not detected (not reported or reported as '0', 'not determined', etc.)
<i>italics</i>	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): $100 \times SD / Mean$
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, $100 \times eSD / Median$

Round Robin XXII Laboratory Results

%Bias Summary

Lab	TR	aT	bC	Label	Definition
FSV-BA	3±2	-5±3	11±1	Lab	Participant code
FSV-BD	8±2	6±3		TR	Total Retinol
FSV-BE	3±2	-1±4		aT	a-Tocopherol
FSV-BF	9±3	0±3	19±19	bC	Total or trans-b-Carotene
FSV-BG	1±3	3±4	29±68	% Bias	(Mean ± SD) of individual serum biases
FSV-BH	-4±2	-1±2	25±2	Mean	Average of $(x_i - \text{Median}_i)/\text{Median}_i$
FSV-BI	-2±2	-2±4	-13±2	SD	Standard deviation of $(x_i - \text{Median}_i)/\text{Median}_i$
FSV-BJ	-1±3	3±5	8±7	x_i	Result for analyte in serum _i
FSV-BK	22±20	1±6		Median _i	Median of non-NIST results in serum _i
FSV-BL	15±8	6±6			
FSV-BM	1±1	-1±1			
FSV-BN	3±6	5±4	-6±4		
FSV-BP	74±85	41±23	-35±11		
FSV-BS	0±33		-22±15		
FSV-BY	-3±1	1±3	13±6		
FSV-BZ		-24±24			
FSV-CA	14±5	-3±4			
FSV-CB	-39±9	-36±11			
FSV-CH	14±2	8±5	-27±6		
FSV-CJ	-7±9	-6±18	-35±10		
FSV-CK	-13±4	-7±7	5±9		
FSV-CM		-3±1			
FSV-CO	-6±1	-7±4	9±5		
FSV-CP			-25±4		
FSV-CQ	-17±6	-4±6	-10±3		
FSV-CT	0±13		-18±8		
FSV-CU	48±33	-13±15	-7±14		
FSV-CV	2±3	14±9	31±57		
FSV-CX	-6±3	-2±2	-8±2		
FSV-CY	-3±5	1±1	13±15		
FSV-DC	5±2	-2±9	2±7		
FSV-DE		1±2			
FSV-DG	4±8	5±1	7±2		
FSV-DH	-10±7	-9±9	25±53		
FSV-DL	-5±4	-7±6	-20±6		
FSV-DM	30±6	-4±16	-15±13		
FSV-DW			-26±4		
FSV-DY	11±11		-6±1		
FSV-EA	-20±1	-27±3	-3±1		
FSV-EB	-7±4	6±7	-7±10		
FSV-ED	0±2	-1±4	11±9		
FSV-EF	2±2	2±9			
FSV-EI	39±14	-3±6	38±11		
FSV-EJ	-5±6	4±4	23±11		
FSV-EV	1±2	9±7			
FSV-EY	22±4	17±3			
FSV-FL	3±6	-2±2	14±12		
FSV-FM	-32±3	-4±5	-1±16		
FSV-FS	-1±5	2±5	42±16		
FSV-FU	-61±26	-53±24			
NISTa	-3±4	-3±1	10±18		
NISTb	1±3	-5±4	0±18		
NISTc	7±6	-11±6			

G6

Appendix H. Shipping Package Inserts for RR23

Two items were included in each package shipped to an RR23 participant:

- **Cover letter.** The original letter has been lost. It likely would have described the five sample materials (lyophilized sera 155, 156, 157, and 159; serum extract 158), given guidance on reconstituting the lyophilized samples, stated a date for return of results, to whom they should be sent, and who to contact with technical questions.
- **Datasheet.** Page H2 reproduces the form.

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

In addition to the information shipped with the samples, participants received two communications (likely via fax or airmail) prior to sample shipment:

- Workshop notification and instructions on the analysis of the serum extract. Page H3 reproduces this letter.
- Workshop reminder and request for return of results by September 13, 1991. Page H4 reproduces this letter

ROUND ROBIN XXIII RESULTS FROM LABORATORY # _____
 DATE OF ANALYSIS _____
 RESULTS IN µg/mL

SAMPLE #	ANALYTE	RESULT			
SERUM 155 VIAL # _____	RETINOL ALPHA-TOCOPHEROL BETA-CAROTENE (TOTAL)	_____			
SERUM 156 VIAL # _____	RETINOL ALPHA-TOCOPHEROL BETA-CAROTENE (TOTAL)	_____			
SERUM 157 VIAL # _____	RETINOL ALPHA-TOCOPHEROL	_____			
SERUM EXTRACT 158 VIAL # _____	RETINOL ALPHA-TOCOPHEROL BETA-CAROTENE (TOTAL)	_____			
SERUM 159 VIAL # _____	RETINOL ALPHA-TOCOPHEROL BETA-CAROTENE (TOTAL)	_____			
OPTIONAL ANALYTES : SUPPLY ONE RESULT IF AVAILABLE					
	SERUM #	155	156	157	159
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.
 SERUM EXTRACT 158 IS IN HEXANE.
 NIST FAX # 301-926-8671



NIST

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

July 31, 1991

Dear Colleague:

Our annual QA Workshop will be held on Saturday, October 19 here at NIST. Among other things, the agenda will include: a review of 1990 Round-Robin results; an update on NIST fat-soluble vitamin analysis automation activities; a discussion of QA needs for new analytes; discussion of long-term QA program structure. More detailed information concerning the program agenda will be forthcoming. We invite your suggestions for discussion topics. Depending on interest/need, we are willing to arrange a Friday hands-on laboratory session for new labs and those experiencing chronic measurement problems. Please indicate your interest on the enclosed form.

Samples for Round-Robin XXIII will be shipped on Tuesday, August 6. Four serum samples and an extract are provided. One mL of the extract corresponds to 1 mL of serum. Therefore, for those who do quantitation based on externals, simply provide the concentrations of retinol and α -tocopherol in the solution provided. Retinyl acetate (1.0 μ g/mL) and α -tocopherol acetate (40.0) and tocol (20.7) have been added for those who base their quantitation on internal standards. Process the hexane extract provided as you would any serum/plasma extract routinely analyzed in your lab. Report concentrations of retinol and α -tocopherol relative to the most appropriate of the internal standards that we have added.

Sincerely

ORIGINAL SIGNED BY
WILLIE E. MAY

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

Enclosure

cc: N. E. Craft



NIST

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

August 13, 1991

Dear Colleague:

Subject: Due date for Round Robin XXIII Results.

As you are aware from our last correspondence, the QA Workshop will be held on Saturday, October 19 here at NIST. In order for us to have ample time to compile the results from Round Robin XXIII for discussion at the workshop, we are requesting that you have your results in to us by September 13.

Sincerely,

A handwritten signature in black ink that reads "WILLIE E. MAY".

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

P.S. Results can be FAXed to 301/926-8671.

Appendix I. Final Report for RR23

The following 11 pages present the available components of the final report for RR23. The report as originally distributed would have had the following components:

- A “Summary of 1991 Activities”; no version of this component is now available.
- Tables that summarize results for retinol, α -tocopherol, and total β -carotene for RR21 and RR22. These tables reiterate the results provided in the RR21 and RR22 Final Reports and so are not included here.
- Tables listing the results and various summary values for retinol, α -tocopherol, total β -carotene in RR23. These tables are numbered as Table 1 thru Table 3 in the following pages.
- Tables listing the results and simple summary statistics for *trans*- β -carotene, α -carotene, 13-*cis*- β -carotene, retinyl palmitate, γ -tocopherol, lutein, zeaxanthin, lutein & zeaxanthin, β -cryptoxanthin, and lycopene. These tables are numbered as Table 4 to 15 in the following pages.
- Six graphical presentations related to interlaboratory precision for total retinol, α -tocopherol, and total β -carotene for all round robin studies from March 1986 thru September 1991. The first three of these figures display “Interlaboratory Precision vs Time”, estimated as a relative standard deviation, for all participants (with subjective removal of discordant results). The next three display the relative standard deviation for just the so-called “core labs” (laboratories that participated in round robin studies prior to RR22).

When the isomeric form of an analyte is not specified, it is likely that most participants reported “total” (the sum of all isomers.) Since resolution of γ -tocopherol and β -tocopherol is challenging, the results reported as γ -tocopherol can be confidently assumed to be γ/β -tocopherol.

Due to the complex formatting used in the tabular listings, the original laboratory codes have been deleted without replacement. However, Appendix J provides a complete listing of the RR23 results where the original codes have been altered to ensure confidentiality. Appendix J also provides more relevant summary statistics.

ROUND ROBIN XXIII

Table 11.

Zeaxanthin & Lutein

Lab #	Serum # 155	Serum # 156	Serum # 157	Serum # 159	Serum # 149
=====					
	0.100	0.139	0.083	0.136	0.145
	0.113	0.144	0.074	0.144	0.178
	0.166	0.209	0.126	0.200	0.224
	0.185	0.239	0.171	0.317	
	0.084	0.109	0.069	0.109	
	0.081	0.092	0.060	0.081	
=====					
AVG	0.122	0.155	0.097	0.164	0.182
SD	0.044	0.057	0.043	0.085	0.040
RSD	36.0	36.9	44.2	51.5	21.8

Table 12.

Beta-Cryptoxanthin

Lab #	Serum # 155	Serum # 156	Serum # 157	Serum # 159	Serum # 149
=====					
	0.061	0.069	0.052	0.099	0.093
	0.081	0.064	0.069	0.096	0.097
	0.066	0.063	0.057	0.083	0.095
	0.102	0.098	0.107	0.139	0.127
	0.051	0.054	0.067	0.082	0.103
	0.049	0.043	0.037	0.067	
	0.051	0.048	0.050	0.082	0.089
	0.297	0.283	0.320	0.319	0.208
	0.033	0.032	0.015	0.044	0.063
=====					
AVG	0.088	0.084	0.086	0.112	
SD	0.081	0.077	0.091	0.082	
RSD	92.1	91.9	106.1	72.6	

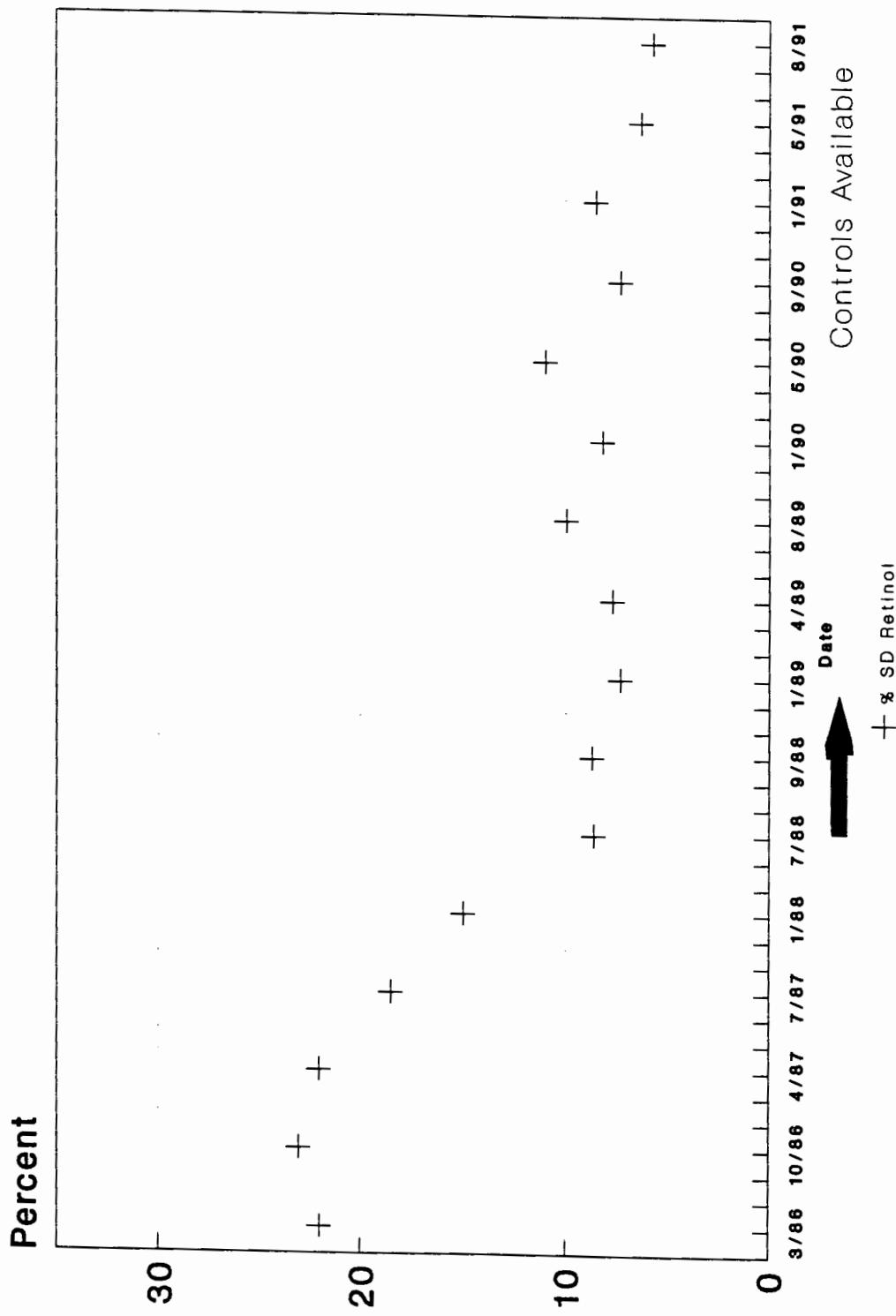
Table 13.

Lycopene

Lab #	Serum # 155	Serum # 156	Serum # 157	Serum # 159	Serum # 149
=====					
	0.583	0.423	0.827	0.352	0.660
	0.507	0.372	0.706	0.361	0.290
	0.339	0.419	0.844	0.312	
	0.602	0.416	0.773	0.426	0.360
	0.507	0.411	0.745	0.434	
	0.571	0.422	0.811	0.800	0.488
	0.674	0.512	1.039	0.497	0.431
	0.107	0.181	0.378	0.143	0.388
	0.776	0.585	1.097	0.573	
	0.780	0.722	1.520	0.727	0.420
	0.206	0.172	0.305	0.156	0.147
	0.779	0.653	0.941	0.590	0.655
	0.470	0.327	0.633	0.276	
	0.257	0.228	0.385	0.227	0.239
	0.455	0.303	0.743		0.576
	0.577	0.481	0.748	0.425	0.392
	0.506	0.322	0.652	0.302	0.366
=====					
AVG	0.512	0.409	0.773	0.413	0.400
SD	0.196	0.153	0.289	0.189	0.158
RSD	38.4	37.3	37.4	45.8	39.6

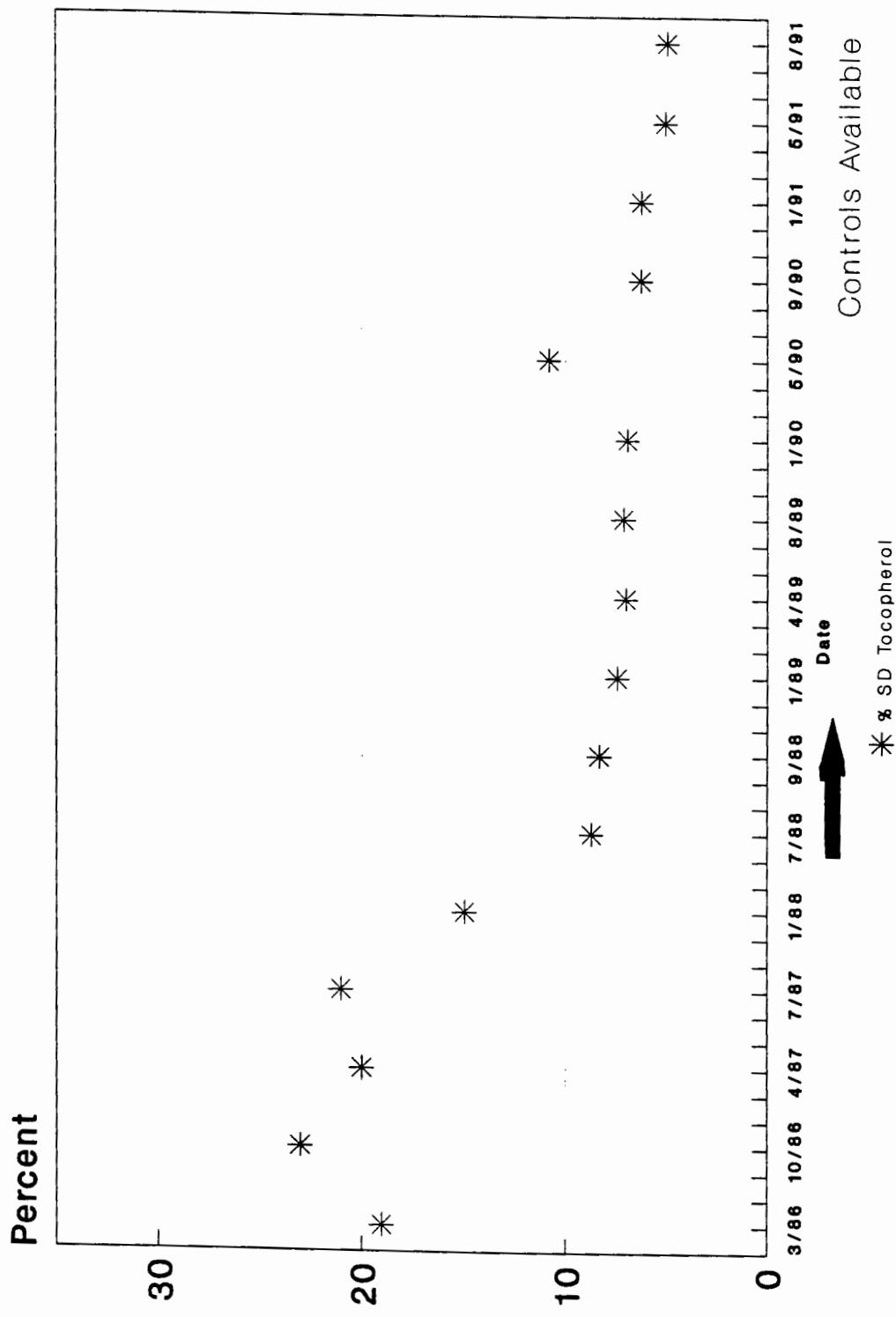
Interlaboratory Precision vs Time

Retinol

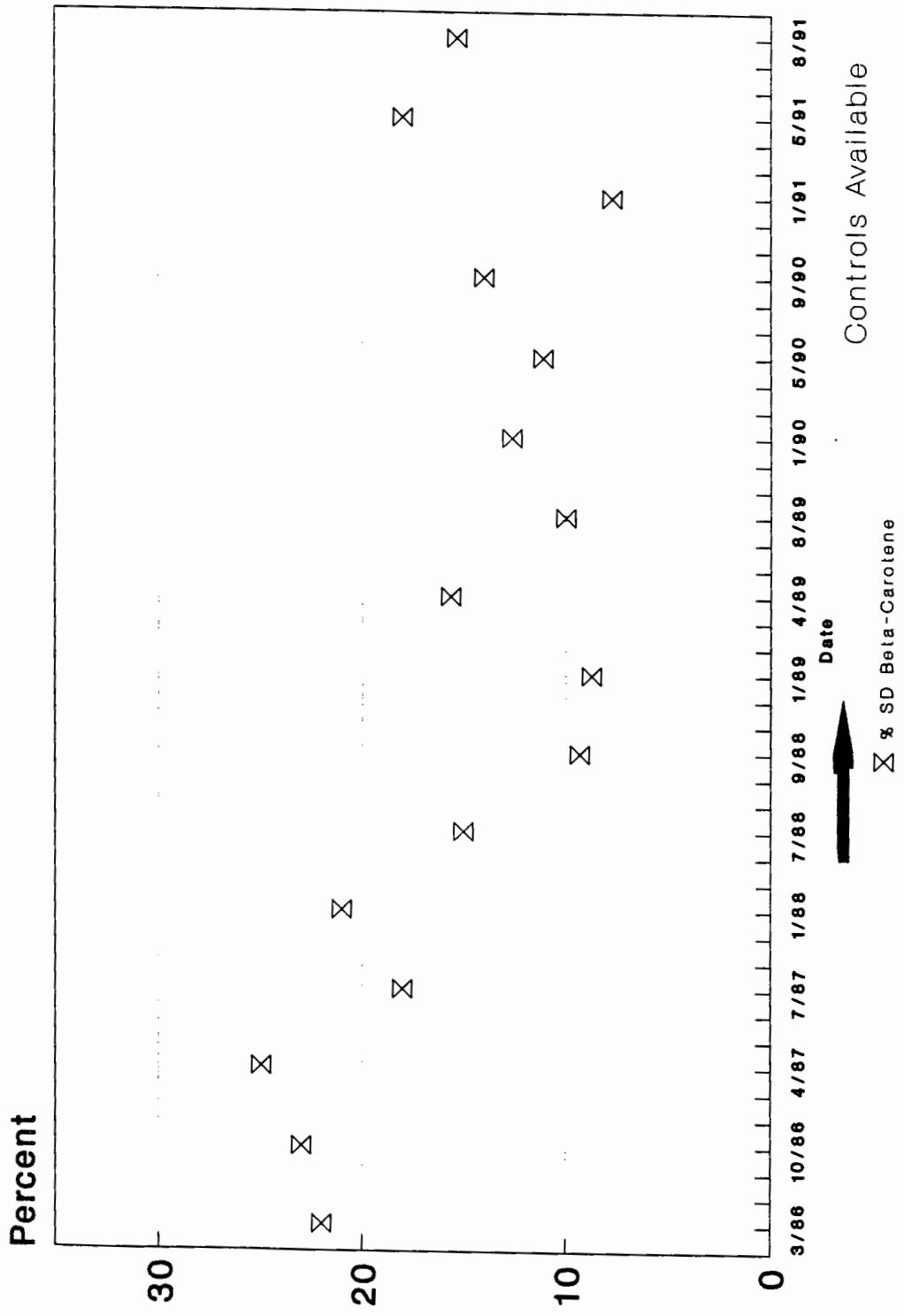


Interlaboratory Precision vs Time

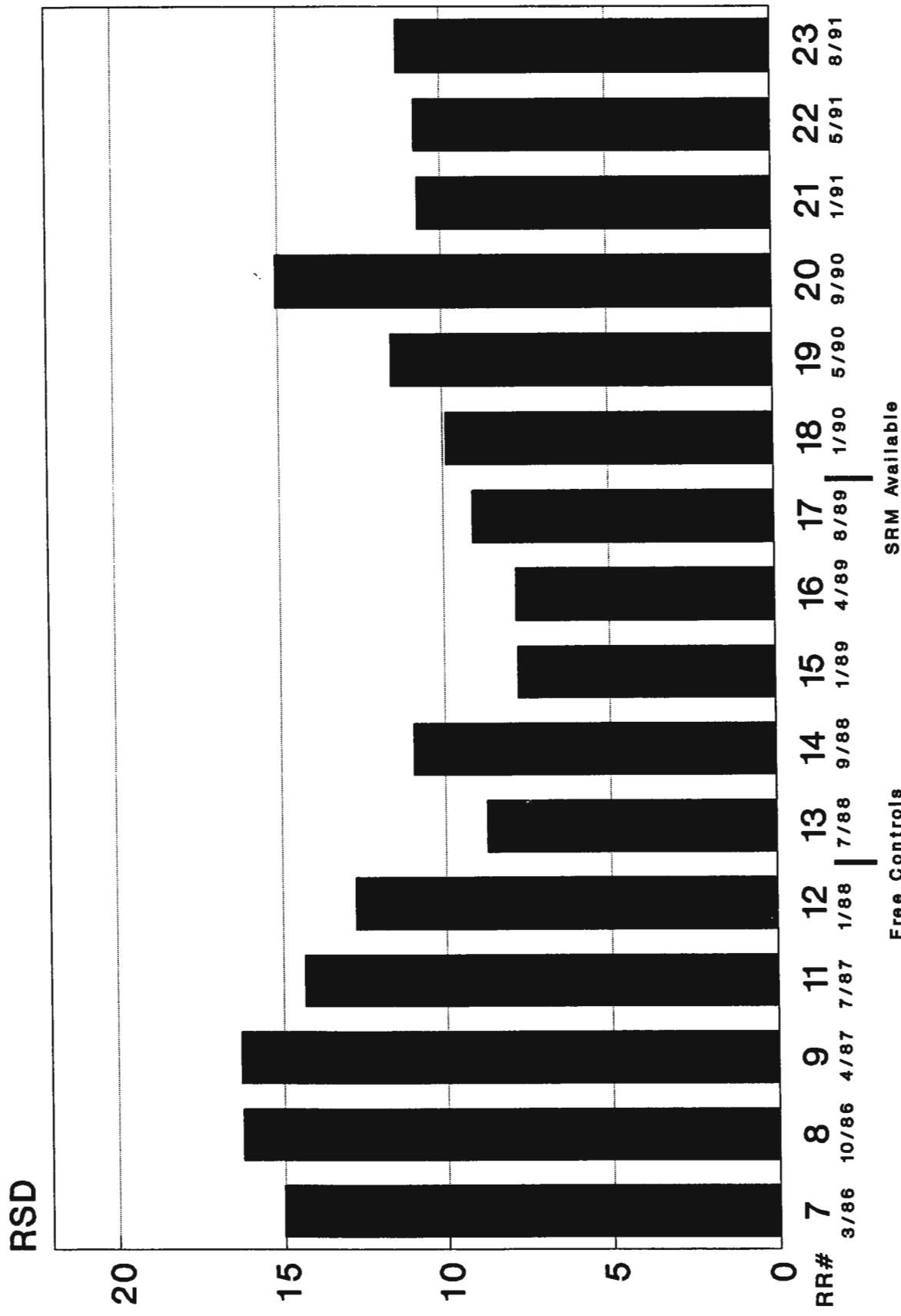
Alpha-Tocopherol



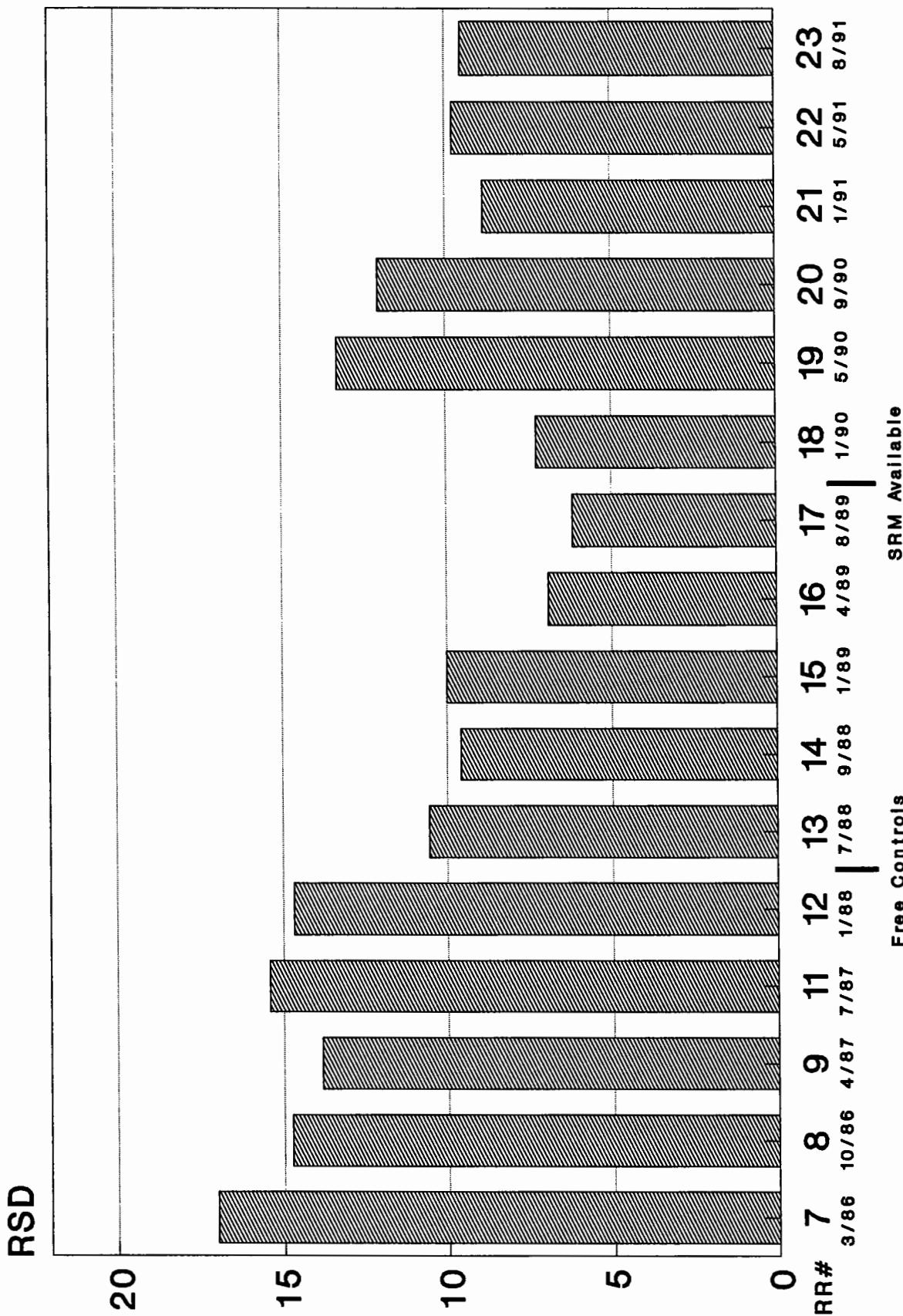
Interlaboratory Precision vs Time Beta-Carotene



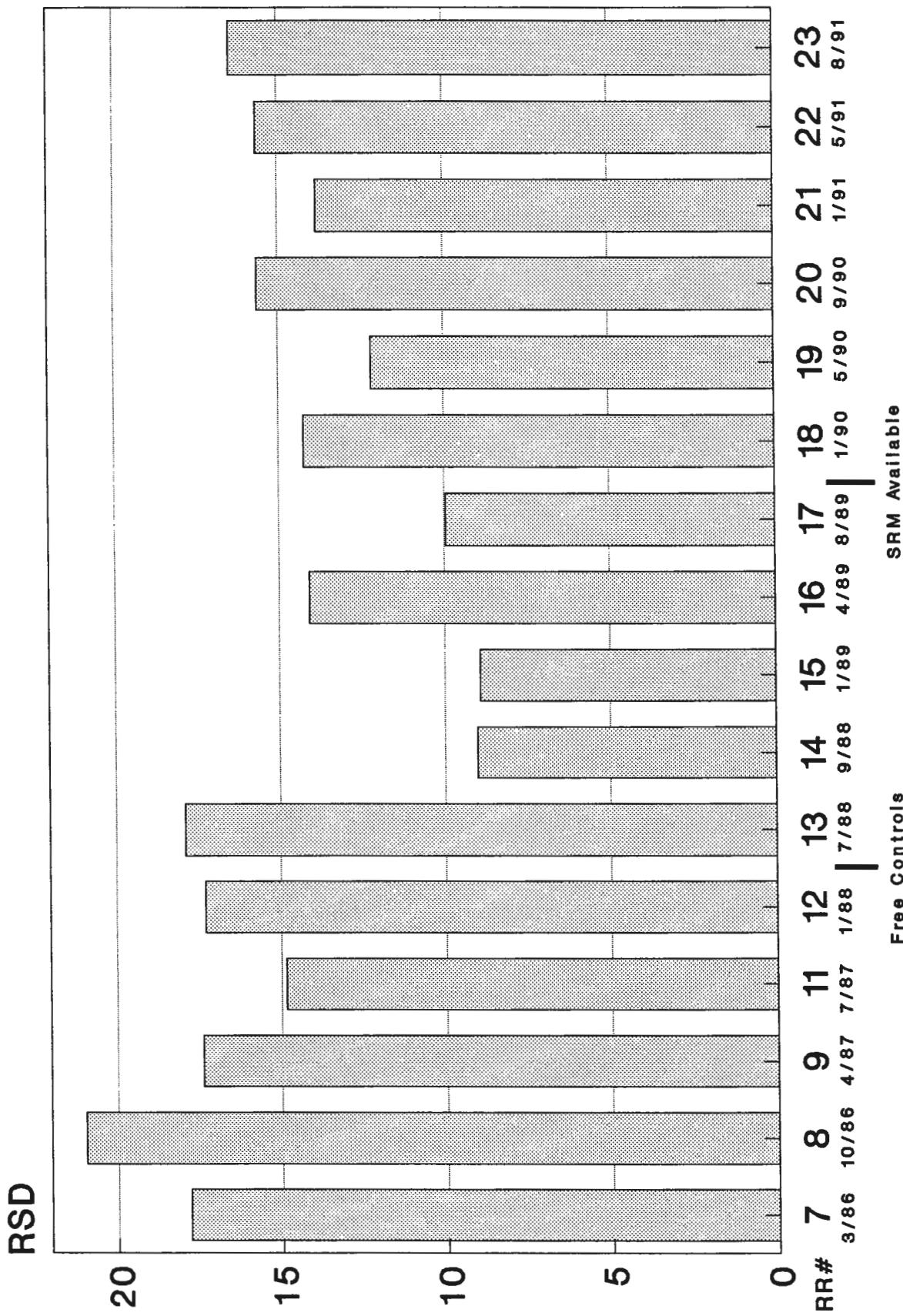
Average Relative Standard Deviation
Core Labs
Retinol



Average Relative Standard Deviation
Core Labs
Alpha-Tocopherol



Average Relative Standard Deviation
Core Labs
Total Beta-Carotene



Appendix J. Updated “All-Lab Report” for RR23

The following five pages are an updated version of an “All-Lab” report for RR23. 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 lists values for all analytes reported by only once. This page also provides a legend for pages 1 thru 4.
- page 6 summarizes each participant’s performance for total retinol, α -tocopherol, and total β -carotene. These summaries are compatible with the percent bias evaluation advice given in the RR23 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 RR23. The only attributed results are those reported by NIST. The NIST results are not used in the assessment of the consensus summary results of the study.

Round Robin XXIII Laboratory Results

Lab	Total β-Carotene					trans-β-Carotene					Total cis-β-Carotene				
	155	156	157	158	159	155	156	157	158	159	155	156	157	158	159
FSV-BA	0.396	0.818	0.092		0.902	0.360	0.780	0.086		0.826	0.036	0.380	0.006		0.073
FSV-BD															
FSV-BE															
FSV-BF	0.437	1.046	0.090		1.200										
FSV-BG	0.283	0.851	0.106		0.757										
FSV-BH	0.411	0.921	0.112		1.050	0.388	0.877	0.112		0.984	0.023	0.044	nd		0.066
FSV-BI	0.333	0.750	0.083		0.827										
FSV-BJ	0.362	0.933	0.098		0.908										
FSV-BK															
FSV-BL															
FSV-BM															
FSV-BN						0.359	0.575	0.069		0.610					
FSV-BP	0.482	1.049	0.214		0.962										
FSV-BS	0.074	0.382	0.020		0.246										
FSV-BX															
FSV-BY	0.442	0.980	0.093		0.891										
FSV-BZ															
FSV-CA															
FSV-CH	0.408	0.851	0.112		0.923										
FSV-CJ	0.293	0.593	0.065		0.689										
FSV-CK	0.080	0.455	0.044		0.403	0.080	0.396	0.039		0.353	nd	0.059	0.005		0.050
FSV-CM															
FSV-CO	0.389	0.853	0.109		0.935										
FSV-CP	0.303	0.697	0.067		0.602										
FSV-CT	0.353	0.766	0.089		0.718										
FSV-CU	0.420	0.823	0.124		0.982	0.388	0.755	0.112		0.899	0.032	0.068	0.012		0.083
FSV-CV	0.390	0.891	0.080		0.872	0.290	0.710	0.010		0.740					
FSV-CX															
FSV-CY	0.440	0.820	0.090		0.950										
FSV-DE															
FSV-DG	0.348	0.891	0.080		0.782										
FSV-DH	0.344	0.965	0.118		1.065										
FSV-DL	0.234	0.658	0.057		0.688										
FSV-DM	0.433	0.873	0.099		0.826										
FSV-DO	0.342	0.656	0.170		0.893										
FSV-DW						0.241	0.634	0.084	0.019	ns					
FSV-DY	0.379	0.852	0.094		0.930										
FSV-EA	0.258	0.542	0.054		0.660										
FSV-EB	0.376	0.829	0.061		0.865										
FSV-EF															
FSV-EI	0.308	0.881	0.069		0.904										
FSV-EJ	na	0.597	0.024		ns										
FSV-EV															
FSV-EY															
FSV-FL	0.423	0.842	0.113		0.950										
FSV-FM	0.239	0.397	0.052		0.593										
FSV-FR	0.418	0.893	0.091		0.998										
FSV-FS	0.381	1.055	0.092	nd	1.836										
FSV-FU															
FSV-GB	0.420	0.823	0.124		0.982										
n	32	33	33	0	32	7	7	7	1	6	3	4	3	0	4
Min	0.074	0.382	0.020		0.246	0.080	0.396	0.010		0.353	0.023	0.044	0.005		0.050
Mean	0.350	0.795	0.090		0.868	0.301	0.675	0.073	0.019	0.735	0.030	0.138	0.008		0.068
Max	0.482	1.055	0.214		1.836	0.388	0.877	0.112		0.984	0.036	0.380	0.012		0.083
SD	0.095	0.176	0.038		0.261	0.112	0.158	0.038		0.228	0.007	0.162	0.004		0.014
CV	20	17	18		14	29	18	34		23	18	43	31		17
NISTc	0.419	0.853	0.089		0.786										
Median	0.378	0.842	0.091		0.898	0.359	0.710	0.084		0.783					
eSD	0.064	0.117	0.031		0.125	0.043	0.113	0.042		0.214					
eCV	17	14	34		14	12	16	49		27					

Round Robin XXIII Laboratory Results

Lab	Total Lutein					Total Zeaxanthin					Total Lutein&Zeaxanthin					
	155	156	157	158	159	155	156	157	158	159	155	156	157	158	159	
FSV-BA											0.166	0.209	0.126		0.200	
FSV-BD																
FSV-BE																
FSV-BF																
FSV-BG																
FSV-BH	0.087	0.110	0.071		0.109	0.026	0.036	0.024		0.038	0.113	0.146	0.095		0.147	
FSV-BI											0.100	0.139	0.083		0.136	
FSV-BJ																
FSV-BK																
FSV-BL																
FSV-BM																
FSV-BN	0.050	0.063	0.038		0.055	0.023	0.020	0.012		0.020	0.081	0.092	0.060		0.081	
FSV-BP																
FSV-BS																
FSV-BX																
FSV-BY																
FSV-BZ																
FSV-CA																
FSV-CH																
FSV-CJ											0.113	0.144	0.074		0.144	
FSV-CK	0.223	0.248	0.183		0.270											
FSV-CM																
FSV-CO																
FSV-CP																
FSV-CT	0.091	0.116	0.063		0.114	0.048	0.064	0.027		0.061	0.084	0.109	0.069		0.109	
FSV-CU											0.139	0.180	0.090		0.175	
FSV-CV																
FSV-CX																
FSV-CY																
FSV-DE																
FSV-DG																
FSV-DH																
FSV-DL																
FSV-DM																
FSV-DO																
FSV-DW																
FSV-DY	0.138	0.179	0.126		0.257	0.047	0.060	0.045		0.060	0.185	0.239	0.171		0.317	
FSV-EA																
FSV-EB																
FSV-EF																
FSV-EI																
FSV-EJ																
FSV-EV																
FSV-EY																
FSV-FL																
FSV-FM																
FSV-FR																
FSV-FS																
FSV-FU																
FSV-GB																
n	5	5	5	5	0	5	4	4	4	0	4	8	8	8	0	8
Min	0.050	0.063	0.038		0.055	0.023	0.020	0.012		0.020	0.081	0.092	0.060		0.081	
Mean	0.118	0.143	0.096		0.161	0.036	0.045	0.027		0.045	0.123	0.157	0.096		0.164	
Max	0.223	0.248	0.183		0.270	0.048	0.064	0.045		0.061	0.185	0.239	0.171		0.317	
SD	0.067	0.072	0.058		0.096	0.013	0.021	0.014		0.020	0.038	0.049	0.036		0.072	
CV	30	29	32		36	28	32	30		32	20	21	21		23	
NISTc	0.074	0.089	0.069		0.079	0.033	0.045	0.034		0.034	0.107	0.134	0.103		0.113	
Median	0.091	0.116	0.071		0.114						0.113	0.145	0.087		0.146	
eSD	0.061	0.079	0.049		0.087						0.040	0.052	0.022		0.049	
eCV	67	68	69		77						36	36	26		34	

Round Robin XXIII Laboratory Results

Table Legend

Symbol	Interpretation
<i>na</i>	Not available (not reported by participant)
<i>nd</i>	Not detected (not reported or reported as '0', 'not determined', etc.)
<i>ns</i>	No sample (damaged in shipping or not provided by NIST)
<i>italics</i>	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): $100 * SD / Mean$
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, $100 * eSD / Median$

Round Robin XXIII Laboratory Results

%Bias Summary

Lab	TR	aT	bC	Label	Definition	
FSV-BA	4±2	1±1	1±3	Lab	Participant code	
FSV-BD	9±3	9±4		TR	Total Retinol	
FSV-BE	-2±3	-2±3		aT	a-Tocopherol	
FSV-BF	5±4	3±4	18±15	bC	Total or trans-b-Carotene	
FSV-BG	-1±3	-4±3	-6±18	% Bias	(Mean ± SD) of individual serum biases	
FSV-BH	5±6	-1±2	14±7	Mean	Average of $(x_i - \text{Median}_i)/\text{Median}_i$	
FSV-BI	-1±1	-6±2	-10±2	SD	Standard deviation of $(x_i - \text{Median}_i)/\text{Median}_i$	
FSV-BJ	0±1	2±1	4±7	x_i	Result for analyte in serum _i	
FSV-BK	19±9	3±4		Median _i	Median of non-NIST results in serum _i	
FSV-BL	12±4	-3±2				
FSV-BM	0±2	7±7				
FSV-BN	-11±4	2±4				
FSV-BP	6±6	56±10	48±58	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.		
FSV-BS	-28±21		-71±12			
FSV-BX	-2±3	2±3				
FSV-BY	4±8	5±5	9±9			
FSV-BZ		-10±6				
FSV-CA	7±3	2±2				
FSV-CH	11±1	10±4	9±10			
FSV-CJ	-24±2	-6±6	-26±4			
FSV-CK	29±10	17±6	-58±14			
FSV-CM		-7±6				
FSV-CO	-4±2	-3±1	7±8			
FSV-CP			-24±7			
FSV-CT	-5±7		-10±7			
FSV-CU	-10±5	-5±1	14±16			
FSV-CV	-1±3	-11±5	-2±8			
FSV-CX	-18±4	-6±4				
FSV-CY	-1±4	0±4	5±9			
FSV-DE		1±2				
FSV-DG	1±1	2±2	-7±9			
FSV-DH	8±17	15±18	13±16			
FSV-DL	-6±3	-16±4	-30±9			
FSV-DM	-8±1	6±20	5±10			
FSV-DO	2±6	-18±19	14±49			
FSV-DW			-23±14			
FSV-DY	6±5		2±2			
FSV-EA	-12±4	-19±15	-34±6			
FSV-EB	0±5	1±1	-10±16			
FSV-EF	4±6	1±2				
FSV-EI	21±6	-5±34	-9±14			
FSV-EJ	-27±8	9±2	-51±32			
FSV-EV	14±8	4±2				
FSV-EY	-1±1	9±2				
FSV-FL	-2±2	-4±3	10±10			
FSV-FM	-24±3	-12±18	-42±8			
FSV-FR	1±1	5±2	7±5			
FSV-FS	-20±18	0±6	33±49			
FSV-FU	56±14	-16±1				
FSV-GB	7±3	-2±2	14±16			
NISTc	4±3	0±2	-1±10			