NISTIR 7880-35

NIST Micronutrients Measurement Quality Assurance Program Winter, Spring, and Fall 1989 Comparability Studies

Results for Round Robins XV, XVI, and XVII Fat-Soluble Vitamins and Carotenoids in Human Serum

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National Institute of Standards and Technology
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August 2016

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 -soluble vitamins and carotenoids in human serum and plasma. This report describes the design of and results for the Winter, Spring and Fall 1989 MMQAP measurement comparability improvement studies: 1) Round Robin XV Fat-Soluble Vitamins and Carotenoids in Human Serum, 2) Round Robin XVI Fat-Soluble Vitamins and Carotenoids in Human Serum, and 3) Round Robin XVII Fat-Soluble Vitamins and Carotenoids in Human Serum. The materials for Round Robin XV were shipped to participants in January 1989; participants were requested to provide their measurement results by February 6, 1989. The materials for Round Robin XVI were shipped to participants in April 1989; participants were requested to provide their measurement results by May 15, 1989. The materials for Round Robin XVII were shipped to participants in August 1989; participants were requested to provide their measurement results by October 1, 1989.

Keywords

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

Table of Contents

Introduction

Beginning in 1984, the National Institute of Standards and Technology (NIST), formerly the National Bureau of Standards (NBS), has coordinated the Micronutrients Measurement Quality Assurance Program (MMQAP) for laboratories that measure fat-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 XV: Fat-Soluble Vitamins and Carotenoids in Human Serum

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin XV comparability study (hereafter referred to as RR15) received three 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 1989. The communication materials included in the sample shipment are described in Appendix A.

Participants were requested to report values for retinol, α-tocopherol, and total and *trans*-β-carotene. Not all participants reported values for the target analytes, and some participants reported values for non-target analytes.

The final report delivered to every participant in RR15 is reproduced as Appendix B. This report included:

- An individualized letter that discussed the participant community's results and a summary analysis of the individual particpnt's results.
- Tabular presentations of all results and several summary values.
- Graphical presentations of the community's interlaboratory precision over time.
- Graphical presentations of the individual participant's results.

Appendix C lists all of the measurement results reported for RR15 in a more accessible format.

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

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin XVI comparability study (hereafter referred to as RR16) received four lyophilized human serum test samples and one ethanolic solution of retinol, α-tocopherol, and *trans*-β-carotene for analysis. Unless multiple vials were previously requested, participants received one vial of each material. These sample materials were shipped on dry ice to participants in April 1989. The communication materials included in the sample shipment are described in Appendix D.

Participants were requested to report values for retinol, α-tocopherol, and total and *trans*-β-carotene. Not all participants reported values for the target analytes, and some participants reported values for non-target analytes.

The final report delivered to every participant in RR16 is reproduced as Appendix E. This report included:

- An individualized letter that discussed the participant community's results and a summary analysis of the individual participnt's results.
- Tabular presentations of all results and several summary values.
- Graphical presentations of the community's interlaboratory precision over time.
- Graphical presentations of the individual participant's results.

Appendix F lists all of the measurement results reported for RR16 in a more accessible format.

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

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin XVII comparability study (hereafter referred to as RR17) received five lyophilized human serum test samples and one ethanolic solution of retinol, α-tocopherol, and a complex mixture of β-carotene isomers 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 1989. The communication materials included in the sample shipment are described in Appendix G.

Participants were requested to report values for retinol, α-tocopherol, and total and *trans*-β-carotene. Not all participants reported values for the target analytes, and some participants reported values for non-target analytes.

The final report delivered to every participant in RR17 is reproduced as Appendix H. This report included:

- An individualized letter that discussed the participant community's results and a summary analysis of the individual participnt's results.
- Tabular presentations of all results and several summary values.
- Graphical presentations of the community's interlaboratory precision over time.
- Graphical presentations of the individual participant's results.

Appendix I lists all of the measurement results reported for RR17 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 RR15

Two items were included in each package shipped to an RR15 participant:

- **Cover letter**. The original letter has been lost. It would have described the three lyophilized sample materials (sera 100 to 102) distributed for the study, given guidance on reconstituting the serum samples and use of the calibration solutions, stated that results were due in February 6, 1989 and to whom they should be sent, and who to contact with technical questions.

- **Datasheets**. Page A2 reproduces the form.

These items were attached to the shipping box.

REPORT ON NIST/NCI SAMPLES FROM LABORATORY # _____

DATE OF ANALYSIS _____

RESULTS IN mg/L

SAMPLE #	ANALYTE	Result	
		Trans	Total
SERUM 100	RETINOL		
VIAL # _____	B-CAROTENE		
	A-TOCOPHEROL		
		Trans	Total
SERUM 101	RETINOL		
VIAL # _____	B-CAROTENE		
	A-TOCOPHEROL		
		Trans	Total
SERUM 102	RETINOL		
VIAL # _____	B-CAROTENE		
	A-TOCOPHEROL		

Reconstitute the Serum Sample with 1.00 mL water.

Please return results by 6 February 1989.

Appendix B. Final Report for RR15

The following twelve pages are the available components of the final report for RR15 as provided to all participants. The original report consisted of:

- An individualized cover letter and discussion. The pages reproduced here are for participant FSV-BA.
- Tables 1 to 3 list all of the participants' results and several summary values for retinol, α-tocopherol, and total β-carotene. These tables do not report any NIST results nor participant results for other analytes.
- Graphical presentations of interlaboratory precision over time for retinol, α-tocopherol, and total β-carotene.
- Graphical presentations of the indiviual participant's bias relative to an assigned value over time for retinol, α-tocopherol, and total β-carotene. The pages reproduced here are for participant FSV-BA.

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 RR15 results where the original codes have been altered to ensure confidentiality. Appendix C also provides more relevant summary statistics.

UNITED STATES DEPARTMENT OF COMMERCE
National Institute of Standards and Technology
[formerly National Bureau of Standards]
Gaithersburg, Maryland 20899

March 1, 1989

Personalized letters were sent to participants. -
This block contained their formal name and address. -

Dear (Personal name),

Enclosed is our statistical summary of the results from Round-Robin XV. As usual, results are presented in both tabular and graphical form. Tables 1-3 provide data submitted by each laboratory for retinol, α-tocopherol, and β-carotene, respectively. These tables also provide the laboratory grand average, standard deviation (SD), and relative standard deviation (RSD) for interlaboratory measurements of each analyte along with data representing each laboratory's bias on a sample-by-sample basis for each analyte. Two types of graphical data are provided. Figures A-C demonstrate the improvement in interlaboratory precision that has been accomplished over the past three years. The three additional figures are Control Charts which represent summaries of your lab's performance vs. the assigned value for each analyte over the past two years.

Over the past two years, individual laboratory performance on each sample has been judged against an assigned value derived from the laboratory grand average (33%) and data from two separate NIST measurement procedures (67%). At last year's workshop, we reviewed data that documented the improvement of interlaboratory measurement capabilities to the extent that value assignment for round-robin samples based solely on laboratory grand means is now possible. This is being implemented. We also agreed to tighten laboratory performance standards. In the past, deviation's of 0-15% from the assigned value was deemed to represent acceptable performance; a bias of >15-30% was deemed to represent marginal performance; bias of >30% represented unacceptable performance. Under our new performance standards, 0-5% bias from the assigned value will represent exceptional performance (AA); >5-10% bias from the assigned value will represent acceptable performance (A); >10-20% bias from the assigned value will represent marginal performance (M); and >20% bias will represent unacceptable performance (U). Keep in mind that these descriptors are meant only to inform you of your performance relative to the state of the practice for these analyses.

Your laboratory's performance in RRXV was:

Retinol	AA
α-tocopherol	AA
Total β-Carotene	AA

Interlaboratory measurement capabilities continue to improve both in terms of accuracy and precision. The level of interlaboratory measurement comparability is now approaching what we had hoped for when the program began. The data presented in Figures A-C show a threefold improvement in interlaboratory precision over the past three years. The most dramatic improvement came approximately one year ago, upon the development and distribution on Control Materials for use in validating and trouble shooting your methods. A dramatic improvement in the accuracy of individual laboratory measurements has accompanied the increase in interlaboratory precision. One year ago, in RRXII, only 9 of 31 labs (29%) reported values for retinol that were within 10% of the assigned value. Now 40% of the labs measure retinol to within 5% of the assigned value; 72% accurate to within 10%; and all to within 20%. In RRXII, 11 of 28 labs (39%) reported values for α-tocopherol that were accurate to within 10%. In RRXV, 50% of the labs report data accurate to within 5%; 73% accurate to within 10%; 95% accurate to within 20%. In RRXII, 4 of 20 labs (20%) reported data for total β-carotene accurate to within 10%. In RRXV, 46% reported data accurate to within 5%; 69% accurate to within 10%; and 92% accurate to within 20%.

Our experiences over the past year have clearly demonstrated the efficacy of the use of reference materials for improving accuracy and interlaboratory precision. The Control Materials that you have been using as reference materials for the past year were designed for a pilot effort to demonstrate the benefits that could be derived through proper use of reference materials. The supply of those samples has been depleted. However, measurements are nearing completion for the certification of three pools of serum for distribution through the NIST Office of Standard Reference Materials as SRM 968, "Fat Soluble Vitamins in Serum", at a cost of $150 per unit. Each unit will consist of two vials each of three levels. Preliminary analysis of these materials indicate that the certified concentrations (μg/mL), upon reconstitution of the freeze-dried samples with 1.00 mL water, will be approximately:

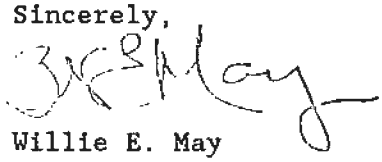
	RETINOL	α-TOCOPHEROL	TOTAL β-CAROTENE
Low Level	0.31	5.0	0.11
Medium Level	0.48	7.8	0.69
High Level	1.15	12.6	1.53

The concentrations of the three analytes will be certified with an uncertainty of approximately 5%. Information values will be provided for γ-tocopherol, all-trans β-carotene and additional carotenoids.

The SRM should be available by May 1. We strongly recommend that you incorporate its use into your normal QC and QA protocols. Data acquired through our round-robin studies demonstrate that use of this SRM will assist you in achieving accuracy, and thus ensure interlaboratory comparability of data.

Samples for RRXVI will be shipped during the first week of April. Further information regarding that exercise will be forthcoming within the next three weeks.

Sincerely,



Willie E. May
Chief
Organic Analytical Research Division
Center for Analytical Chemistry

Enclosure

Table 1.

Results Round Robin XV
Retinol

Lab#	Serum 100	Serum 101	Serum 102
	0.315	0.450	1.139
	0.291	0.472	1.046
	0.308	0.473	1.150
	0.325	0.500	1.250
	0.302	0.446	1.176
	0.334	0.518	1.201
	0.327	0.384	1.098
	0.345	0.435	1.066
	0.303	0.469	1.160
	0.283	0.441	1.400
	0.284	0.438	1.075
	0.323	0.427	1.152
	0.312	0.463	1.143
	0.313	0.477	1.137
	0.317	0.485	1.170
	0.334	0.555	1.236
	0.323	0.498	1.238
	0.305	0.438	1.073
	0.334	0.543	1.372
NC	0.360	0.520	1.180
	0.306	0.471	1.161
	0.289	0.456	1.141
NL	0.329	0.483	1.121
NL	0.260	0.420	1.030
NL	0.312	0.533	1.306
AVG	0.313	0.468	1.171
SD	0.017	0.040	0.091
RSD	5.6	8.5	7.7

Percent Bias from Grand Average
Retinol

Lab#	Serum 100	Serum 101	Serum 102
	0.7	-3.9	-2.7
	-7.2	0.7	-10.7
	-1.6	1.0	-1.8
	3.9	6.7	6.8
	-3.5	-4.8	0.5
	6.6	10.6	2.6
	4.5	-18.0	-6.2
	10.2	-7.1	-8.9
	-3.2	0.1	-0.9
	-9.6	-5.9	19.6
	-9.2	-6.5	-8.2
	3.2	-8.8	-1.6
	-0.4	-1.3	-2.4
	0.0	1.8	-2.9
	1.3	3.5	-0.1
	6.7	18.5	5.6
	3.2	6.3	5.8
	-2.6	-6.6	-8.3
	6.7	15.9	17.2
NC	15.0	11.0	0.8
	-2.2	0.5	-0.8
	-7.6	-2.7	-2.5
NL	5.1	3.1	-4.2
NL	-16.9	-10.3	-12.0
NL	-0.3	13.8	11.6

NC = No Controls, Values not included in the Grand Average.
NL = New Laboratories, Values not included in the Grand Average.

Table 2.

Results Round Robin XV
Alpha-Tocopherol

Lab#	Serum 100	Serum 101	Serum 102
	4.73	7.53	11.81
	4.69	7.50	12.41
	4.23	7.14	11.03
	5.10	7.95	13.00
	5.03	7.97	12.32
	4.50	6.75	11.18
	5.51	8.02	12.50
	4.93	7.89	12.63
	4.75	7.66	13.45
	4.29	6.81	9.74
	5.26	8.20	13.40
	5.15	7.80	12.90
	4.95	8.10	12.71
	4.94	7.82	12.29
	5.28	9.53	13.09
	5.10	8.03	12.94
	4.75	7.43	11.86
	5.29	8.29	13.52
NC	6.70	5.30	10.40
	5.19	8.09	12.17
NL	5.30	7.90	12.70
NL	4.78	8.01	10.42
AVG	4.93	7.82	12.37
SD	0.34	0.60	0.94
RSD	7.0	7.7	7.6

Percent Bias from Grand Average
Alpha-Tocopherol

Lab#	Serum 100	Serum 101	Serum 102
	-4.0	-3.6	-4.5
	-4.9	-4.0	0.4
	-14.2	-8.7	-10.8
	3.5	1.7	5.1
	2.0	2.0	-0.4
	-8.7	-13.6	-9.6
	11.8	2.6	1.1
	-0.1	0.9	2.2
	-3.6	-2.0	8.8
	-12.9	-12.9	-21.3
	6.7	4.9	8.4
	4.5	-0.2	4.3
	0.4	3.6	2.8
	0.2	0.0	-0.6
	7.1	21.9	5.9
	3.4	2.8	4.6
	-3.6	-5.0	-4.1
	7.3	6.0	9.3
NC	35.9	-32.2	-15.9
	5.3	3.5	-1.6
NL	7.5	1.1	2.7
NL	-3.0	2.5	-15.7

NC = No Controls, Values not included in the Grand Average.
NL = New Laboratories, Values not included in the Grand Average.

Table 3.

Results Round Robin XV
Total Beta-Carotene

Lab#	Serum 100	Serum 101	Serum 102
	0.112	0.687	1.505
	0.108	0.649	1.520
	0.111	0.703	1.476
	0.136	0.730	1.537
	0.147	0.698	1.684
	0.105	0.674	1.577
	0.110	0.674	1.518
	0.113	0.724	1.385
	0.115	0.706	1.550
	0.119	0.670	1.482
	0.115	0.663	1.491
	0.082	0.533	1.559
NL	0.098	0.612	1.427
AVG	0.114	0.676	1.524
SD	0.016	0.051	0.071
RSD	13.9	7.6	4.7

Percent Bias from Grand Average
Total Beta-Carotene

Lab#	Serum 100	Serum 101	Serum 102
	-2.1	1.7	-1.2
	-5.8	-4.0	-0.2
	-3.0	4.0	-3.1
	18.9	8.0	0.8
	28.5	3.3	10.5
	-8.0	-0.3	3.5
	-3.9	-0.2	-0.4
	-1.2	7.1	-9.1
	0.5	4.5	1.7
	4.2	-0.9	-2.7
	0.2	-2.0	-2.1
	-28.3	-21.1	2.3
NL	-14.3	-9.4	-6.3

NL = New Laboratories, Values not included in the Grand Average.

FIGURE A

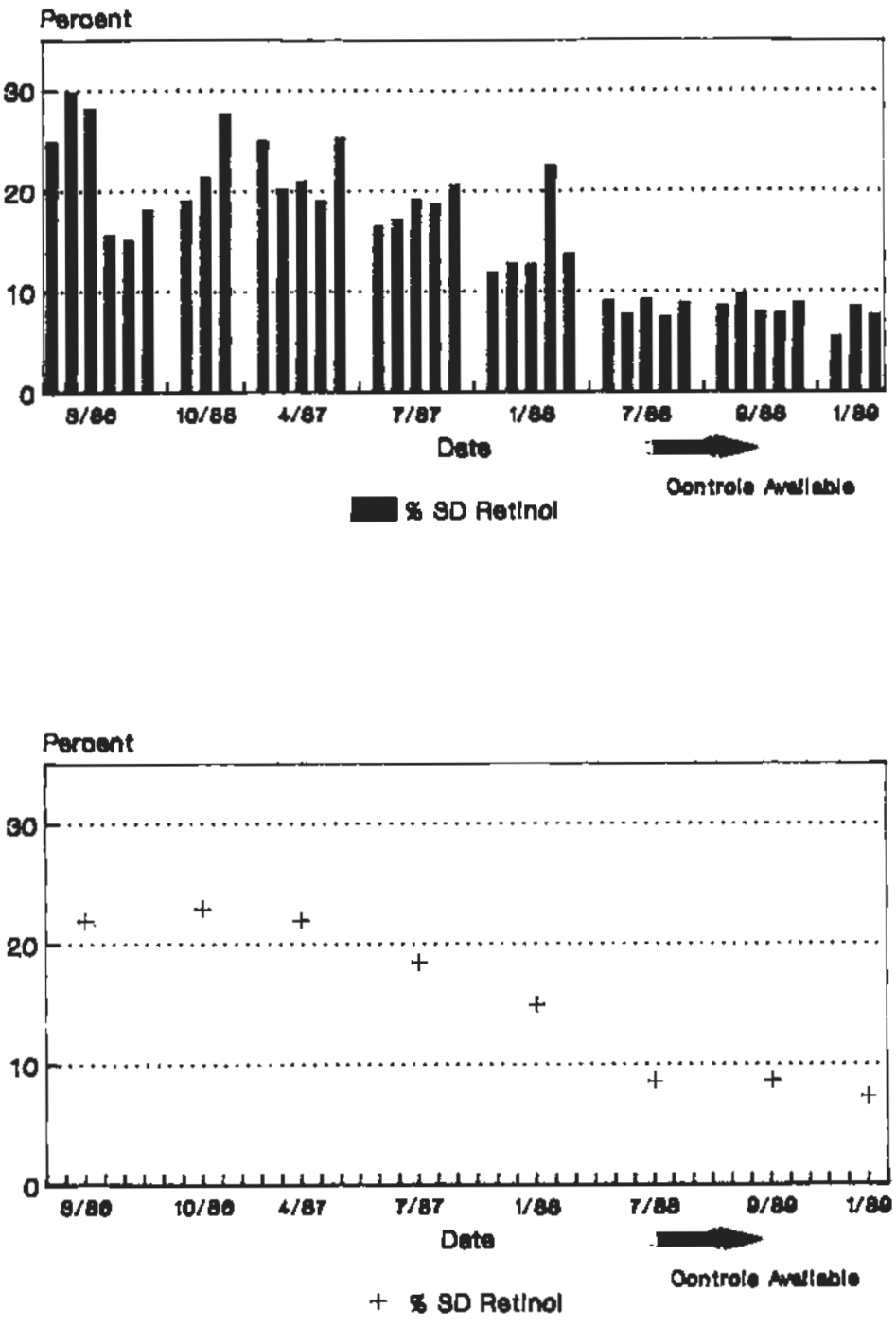


FIGURE B

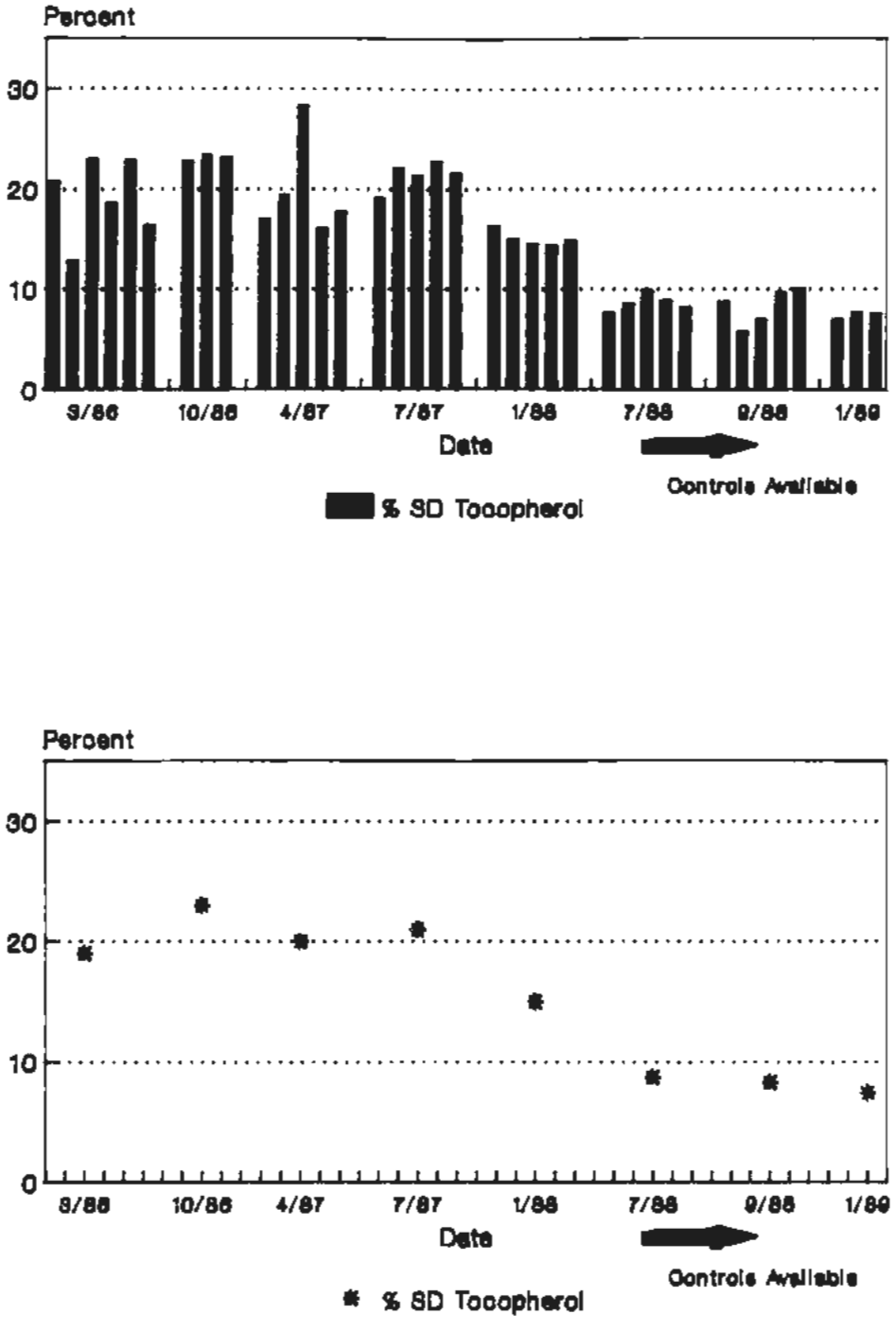
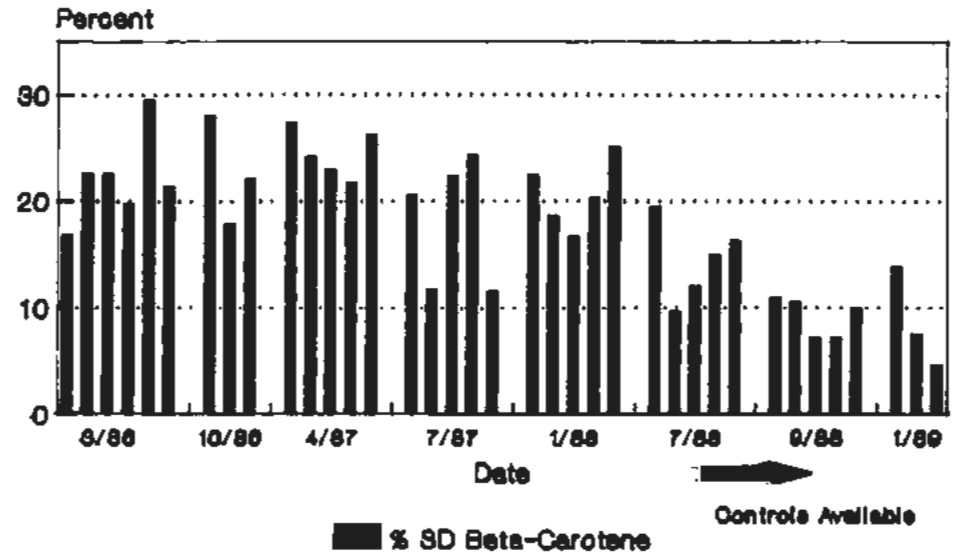
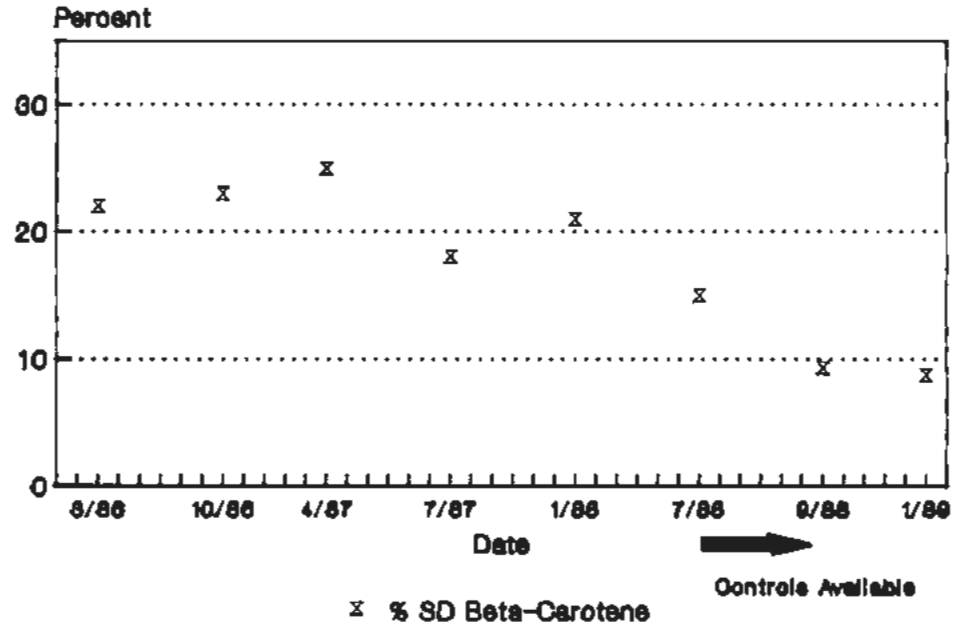
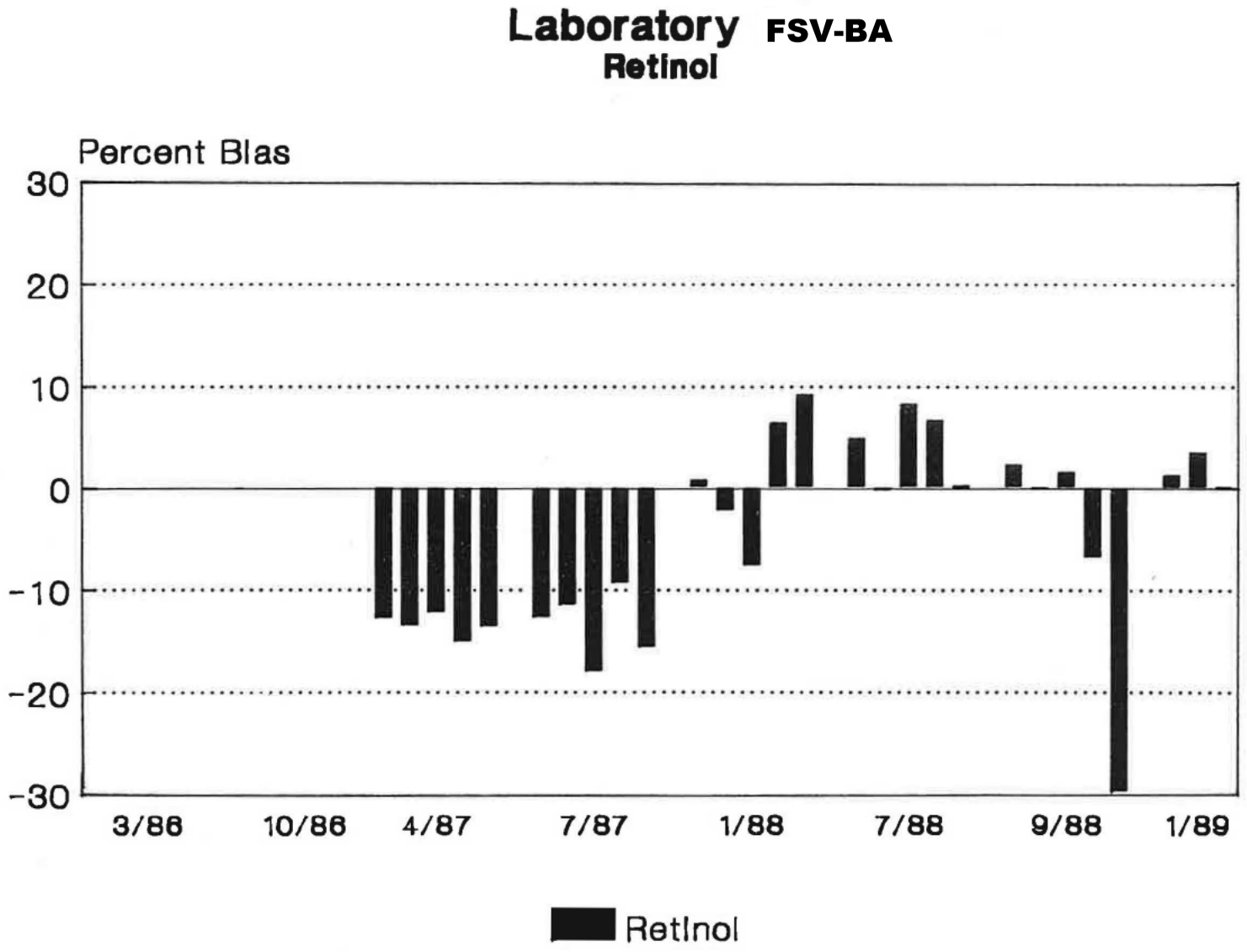
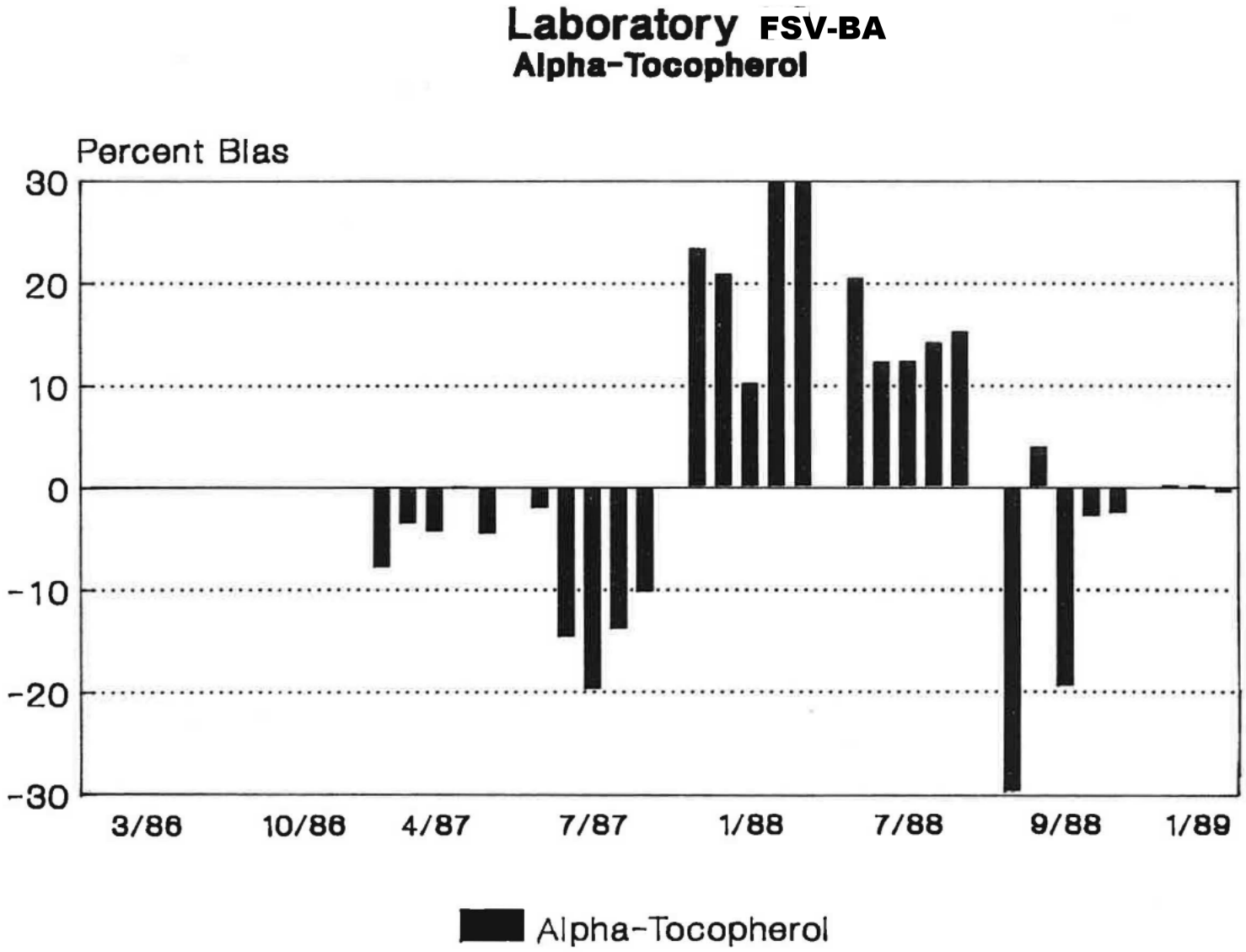


FIGURE C

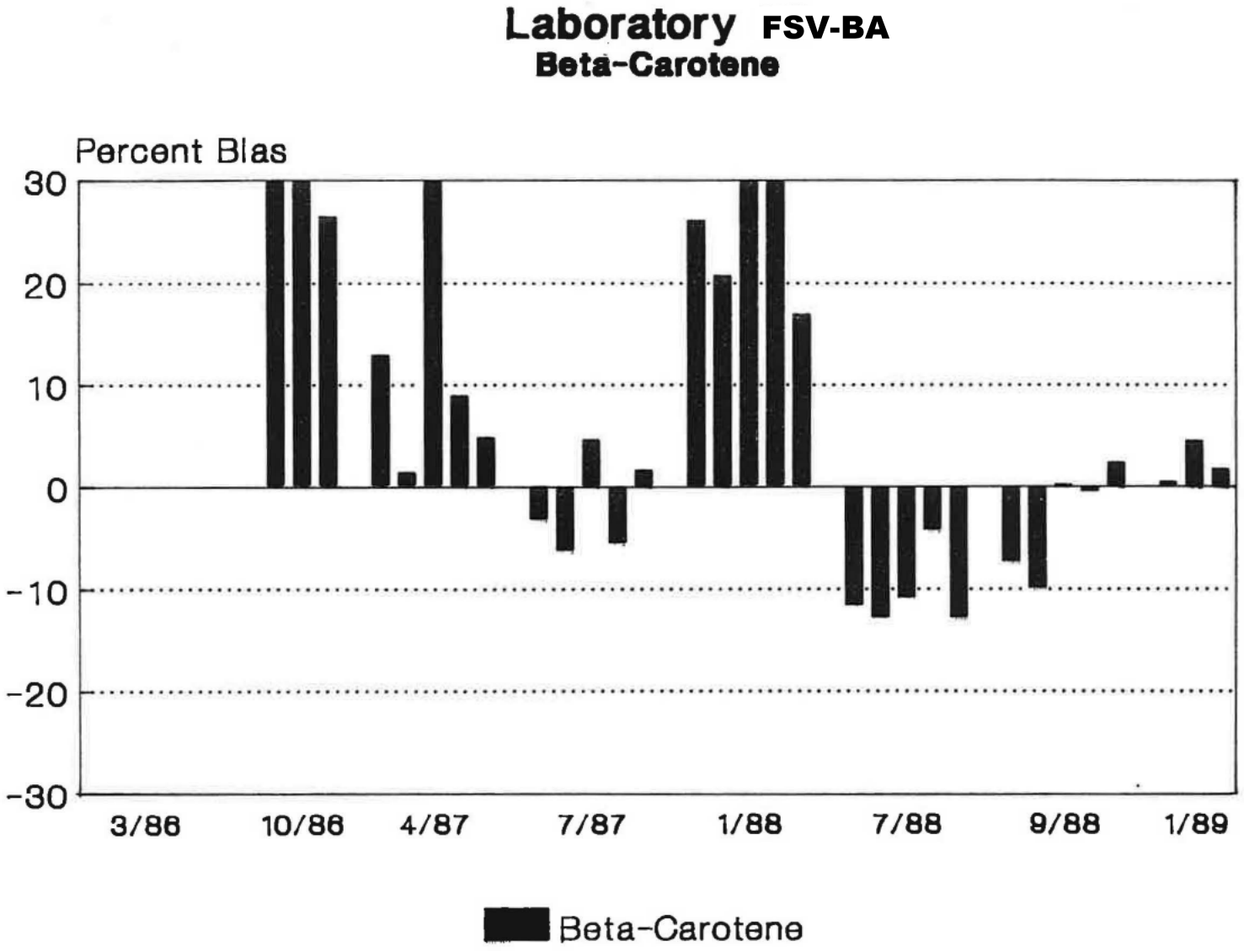








% Bias from Assigned Values



Appendix C. Updated "All-Lab Report" for RR15

The following three pages are a revised version of an "All-Lab" report for RR15. This report has three parts:

- Page 1 lists results for all analytes reported at least twice, counting both participants and NIST analysts. When the isomeric form of an analyte was 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, results reported as γ-tocopherol can be confidently assumed to be γ/β-tocopherol
- Page 2 lists results reported by only one participant and provides the legend for page 1.
- Page 3 summarizes each participants' performance for retinol, α-tocopherol, and total β-carotene. These summaries are compatible with the percent bias evaluation advice given in the original RR15 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 "Grand Average" used in the original and detailed in Appendix B.

To ensure confidentiality, the laboratory identifiers used in this "All-Lab Report" have been altered from those used in RR15. 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 XV Laboratory Results

	Total Retinol			α-Tocopherol			Total β-Carotene			trans-β-Carotene			Total cis-β-Carotene		
Lab	100	101	102	100	101	102	100	101	102	100	101	102	100	101	102
FSV-BA	0.317	0.485	1.170	4.94	7.82	12.29	0.115	0.706	1.550	0.111	0.664	1.480	*0.004*	*0.042*	*0.070*
FSV-BD	0.327	0.384	1.098	4.50	6.75	11.18									
FSV-BE	0.323	0.427	1.152	5.15	7.80	12.90									
FSV-BF	0.303	0.469	1.160	4.93	7.89	12.63	0.105	0.674	1.577						
FSV-BG	0.308	0.473	1.150	4.23	7.14	11.03	0.111	0.703	1.476						
FSV-BH	0.305	0.438	1.073	4.75	7.43	11.86	0.115	0.663	1.491						
FSV-BI	0.291	0.472	1.046	4.69	7.50	12.41	0.108	0.649	1.520						
FSV-BL	0.312	0.533	1.306	4.78	8.01	10.42									
FSV-BY	0.315	0.450	1.139	4.73	7.53	11.81	0.112	0.687	1.505						
FSV-BZ	0.360	0.520	1.180	6.70	5.30	10.40									
FSV-CA	0.302	0.446	1.176	5.10	7.95	13.00									
FSV-CJ	0.330	0.519	1.409	5.82	8.48	13.39	0.110	0.661	1.297						
FSV-CK	0.334	0.543	1.372	5.29	8.29	13.52	≥0.094	≥0.504	≥1.104	0.094	0.504	1.104			
FSV-CM	0.260	0.420	1.030	5.30	7.90	12.70									
FSV-CN	0.345	0.435	1.066	5.51	8.02	12.50	0.147	0.698	1.684						
FSV-CO	0.334	0.517	1.191	5.03	7.93	12.23	0.136	0.725	1.529						
FSV-CQ	0.283	0.441	1.400	4.75	7.66	13.45	≥0.073	≥0.639	≥1.670	0.073	0.639	1.670			
FSV-CY	0.290	0.450	1.160	4.91	7.53	12.60	≤0.1	0.620	1.420						
FSV-DC	0.313	0.477	1.137				0.113	0.724	1.385						
FSV-DE				5.26	8.20	13.40									
FSV-DG	0.312	0.463	1.143	4.95	8.10	12.71	0.110	0.674	1.518						
FSV-DN	0.307	0.483	1.290	4.73	7.60	12.25	0.103	0.620	1.330						
FSV-DO	0.325	0.500	1.250												
FSV-DT	0.284	0.438	1.075	4.29	6.81	9.74									
FSV-DY	0.329	0.483	1.121				0.098	0.612	1.427						
FSV-DZ	0.334	0.555	1.236	5.28	9.53	13.09									
FSV-ED	0.323	0.498	1.238	5.10	8.03	12.94	0.119	0.670	1.482						
FSV-EF	0.289	0.456	1.141	5.19	8.09	12.17									
FSV-FE	0.306	0.471	1.161				0.082	0.533	1.559						
FSV-FX				4.15	7.15	12.41									
n	28	28	28	26	26	26	15	16	16	3	3	3	1	1	1
Min	0.260	0.384	1.030	4.15	5.30	9.74	0.082	0.533	1.297						
Mean	0.313	0.473	1.181	5.00	7.71	12.27	0.112	0.664	1.484	0.093	0.602	1.418	0.004	0.042	0.070
Max	0.360	0.555	1.409	6.70	9.53	13.52	0.147	0.725	1.684						
SD	0.021	0.040	0.101	0.52	0.74	0.99	0.015	0.049	0.096						
CV	6.8	8.5	8.5	10.4	9.6	8.1	13.3	7.4	6.5						
NISTa	0.313	0.479	1.160	4.98	7.32	11.97	0.112	0.660	1.450	0.109	0.582	1.340	*0.003*	*0.078*	*0.110*
NISTd	0.315	0.469	1.175	4.77	7.26	11.82	0.160	0.655	1.348	0.119	0.513	1.115	*0.041*	*0.142*	*0.233*
Median	0.313	0.472	1.160	4.95	7.86	12.46	0.111	0.672	1.498						
eSD	0.020	0.041	0.075	0.341	0.427	0.764	0.006	0.043	0.084						
eCV	6.4	8.6	6.5	6.9	5.4	6.1	5.3	6.3	5.6						

C2

Round Robin XV Laboratory Results

Analytes Reported By One Laboratory

Values in µg/mL

Analyte	Code	100	101	102
γ/β-Tocopherol	FSV-DE	2.163	2.218	2.860

Table Legend

Symbol	Interpretation
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*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
≤x	Concentration at or below the limit of quantification, x
≥x	Concentration greater than or equal to x

Round Robin XV Laboratory Results

%Bias Summary

Lab	TR	aT	bC
FSV-BA	2±1	-1±1	4±1
FSV-BD	-6±12	-11±3	
FSV-BE	-2±7	2±3	
FSV-BF	-1±2	1±1	0±5
FSV-BG	-1±1	-12±3	1±3
FSV-BH	-6±3	-5±1	0±2
FSV-BI	-6±5	-3±3	-2±3
FSV-BL	8±7	-6±9	
FSV-BY	-2±3	-5±1	1±1
FSV-BZ	9±7	-5±36	
FSV-CA	-2±3	3±2	
FSV-CJ	12±8	11±6	-5±7
FSV-CK	13±6	7±1	-22±6
FSV-CM	-13±3	3±3	
FSV-CN	-2±11	5±6	16±15
FSV-CO	6±4	0±2	11±11
FSV-CQ	2±17	1±7	-9±23
FSV-CY	-4±4	-1±3	-6±2
FSV-DC	0±2		1±8
FSV-DE		6±2	
FSV-DG	-1±1	2±2	0±1
FSV-DN	4±7	-3±1	-9±2
FSV-DO	6±2		
FSV-DT	-8±1	-16±5	
FSV-DY	1±4		-8±3
FSV-DZ	10±6	11±9	
FSV-ED	5±2	3±1	2±5
FSV-EF	-4±3	2±4	
FSV-FE	-1±1		-14±16
FSV-FX		-8±8	
NISTa	1±1	-3±4	-1±2
NISTd	1±1	-5±2	11±29

Label	Definition
Lab	Participant code
TR	Total Retinol
aT	a-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 "Avg" 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-based reference value.

Appendix D. Shipping Package Inserts for RR16

The following two items were attached to each package shipped to an RR16 participant:

- Cover letter
- Datasheet

UNITED STATES DEPARTMENT OF COMMERCE
National Institute of Standards and Technology
(formerly National Bureau of Standards)
Gaithersburg, Maryland 20899

April 3, 1989

Dear Colleague,

Samples for RR-XVI are enclosed. The sample set is composed of four serum pools and a ready-to-inject ethanol solution. The serum samples are to be analyzed for retinol, α-tocopherol and total β-carotene as usual. The ethanol solution contains retinol, α-tocopherol and a complex mixture of β-carotene isomers. We request that you provide data for the concentration of retinol, α-tocopherol and all-trans β-carotene in the solution on the enclosed form. We also request that you provide us with a copy of the chromatogram from your analysis of the solution. Results are due by May 15.

Over the past year, we have observed that the use of a common pool of value assigned "Control Materials" to validate your in-house methods, has led to dramatic improvements in the accuracy of results submitted by individual laboratories and interlaboratory precision. The limited supply of those materials has been exhausted but is being replaced by Standard Reference Material 968. SRM 968 should be available for sale at $150.00 per unit by May 1, 1989. SRM 968 will contain two vials of lyophilized serum for each of three concentration levels of retinol, α-tocopherol, and total β-carotene. Information values will be provided for γ-tocopherol and all-trans β-carotene. SRM 968 is intended for use in validating the accuracy of methods used for the determination of these three fat-soluble vitamins in human serum and plasma samples. Employment as a benchmark for value assigning your in-house controls would represent a cost effective use of SRM 968.

The certified values for SRM 968 are given below:

ANALYTE	LOW LEVEL	MEDIUM LEVEL	HIGH LEVEL
Retinol	0.313 ± .010	0.477 ± .019	1.15 ± .05
α-tocopherol	4.94 ± .16	7.67 ± .29	12.4 ± .5
Total β-carotene	0.124 ± .017	0.677 ± .022	1.49 ± .06

The uncertainties listed represent two standard deviations of the certified values and include allowances for analytical imprecision, bottle-to-bottle variability, as well as imprecision associated with the reconstitution process.

Inquiries concerning this SRM should be placed through the NIST (formerly NBS) Office of Standard Reference Materials. They may be reached at 301/975-6776.

Sincerely,

Willie E. May, Ph.D.
Chief
Organic Analytical Research Division
Center for Analytical Chemistry

cc: R. Schaffer

REPORT ON NIST/NCI SAMPLES FROM LABORATORY # _____

DATE OF ANALYSIS _____

RESULTS IN mg/L

SAMPLE #	ANALYTE	Result	
SERUM 109	RETINOL		
		All Trans	Total
VIAL # _____	B-CAROTENE		
	A-TOCOPHEROL		
SERUM 110	RETINOL		
		All Trans	Total
VIAL # _____	B-CAROTENE		
	A-TOCOPHEROL		
SERUM 111	RETINOL		
		All Trans	Total
VIAL # _____	B-CAROTENE		
	A-TOCOPHEROL		
SERUM 112	RETINOL		
		All Trans	Total
VIAL # _____	B-CAROTENE		
	A-TOCOPHEROL		
SERUM 113	RETINOL		
		All Trans	
VIAL # _____	B-CAROTENE		
	A-TOCOPHEROL		

Reconstitute the Serum Sample 109 – 112 with 1.0 mL water.

Serum 113 is and Ethanolic solution of retinol, alpha-tocopherol and beta-carotene. It is to be directly injected for the determination of retinol, alpha-tocopherol and all-trans beta-carotene.

Appendix E. Final Report for RR16

The following nineteen pages are the the final report for RR16 as provided to all participants. This report consisted of:

- An individualized cover letter and discussion. The pages reproduced here are for participant FSV-BA.
- Tables 1 to 3B list all of the participants' results and several summary values for retinol, α-tocopherol, total β-carotene, and *trans*-β-carotene. These tables do not report any NIST results nor participant results for other analytes.
- Graphical presentations of interlaboratory precision over time for retinol, α-tocopherol, and total β-carotene.
- Graphical presentations of the indiviual participant's bias relative to an assigned value over time for retinol, α-tocopherol, and total β-carotene. The pages reproduced here are for participant FSV-BA.
- Graphical presentations of the indiviual participant's assays of "Control B" over time for retinol, α-tocopherol, and total β-carotene. The pages reproduced here are for participant FSV-BA.

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 RR16 results where the original codes have been altered to ensure confidentiality. Appendix C also provides more relevant summary statistics.

UNITED STATES DEPARTMENT OF COMMERCE
National Institute of Standards and Technology
[formerly National Bureau of Standards]
Gaithersburg, Maryland 20899

July 13, 1989

Personalized letters were sent to partiipants.
This block contained their formal name and address.

Dear (Personall name):

Enclosed is our statistical summary of the results of your analysis of the four serum samples provided to you in Round Robin XVI. The results from the analysis of the ethanolic solution (Serum #113) are not included. It became apparent as we received results, that our instructions for its analysis were not clear. We will provide more precise instructions and ask that it be reanalyzed in Round Robin XVII. As usual, results are presented in both tabular and graphical form. Tables 1-3 provide data submitted by each laboratory for retinol, α-tocopherol, and β-carotene, respectively. These tables also provide the laboratory grand average, standard deviation (SD), and relative standard deviation (RSD) for interlaboratory measurements of each analyte, along with data representing each laboratory's bias on a sample-by-sample basis for each analyte.

The tables also provide data documenting the stability of the Grand Averages from recent Round Robins. Each of the four serum samples used in Round Robin XVI has been analyzed in previous Round Robins. The grand average, date of the previous analyses, and difference between this and previous grand averages are provided at the bottom of each table. The agreement among the grand average values from this and previous Round Robins support data that has been acquired from NIST measurements regarding the stability of retinol, α-tocopherol, and β-carotene in freeze-dried serum stored at -80°C.

Three types of graphical data are provided.

- Figures A-C which demonstrate the improvement in interlaboratory precision for the measurement of retinol, α-tocopherol, and β-carotene, that has been accomplished in our program over the past three years.

- Control Charts for retinol, α-tocopherol, and β-carotene, respectively, which represent a summary of each lab's performance vs. the assigned value for each analyte over the past three years.

- A comparison of the values that each lab has reported for "Control B" the three times that it has been distributed as an unknown. Note that "Control B" was run as a blind duplicate in Round Robin XIV.

As mentioned in our last communication with you, the interlaboratory Grand Average has been determined to be an acceptable approximation of the true value for each sample, and will be used as the assigned value except in rare cases. NIST measurements will continue to be performed to verify that no significant bias exists. Under our new performance standards, 0-5% bias from the assigned value will represent exceptional performance (AA); >5-10% bias from the assigned value will represent acceptable performance (A); >10-20% bias from the assigned value will represent marginal performance (M); and >20% bias will represent unacceptable performance (U). Keep in mind that these descriptors are meant only to inform you of your performance relative to the state of the practice for these analysis.

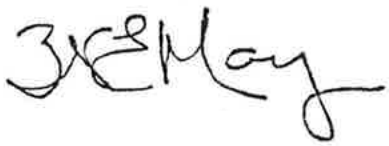
Your laboratory's performance in Round Robin XVI was

Retinol	A
α-tocopherol	AA
Total β-carotene	A
Trans β-carotene	A

Our experiences over the past year have clearly demonstrated the efficacy of the use of reference materials for improving accuracy and interlaboratory precision. The Control Materials provided to you a year ago were developed as a pilot effort to demonstrate the benefits that could be derived through proper use of reference materials. The supply of those samples has been depleted. These Control Materials have been replaced with Standard Reference Material 968, "Fat-Soluble Vitamins in Freeze-Dried Human Serum." A copy of the SRM certificate is enclosed for your information. This SRM can be ordered from the NIST Office of Standard Reference Materials (301/975-6776) at a cost of $185 per unit, including shipping.

Samples for Round Robin XVII will be shipped during the third week of August. Further information regarding that exercise will be forthcoming within the next three weeks along with a preliminary agenda for this years Workshop. The dates for the Workshop are October 31-November 1.

Sincerely,



Willie E. May
Chief
Organic Analytical Research Division
Center for Analytical Chemistry

Enclosures

Tabular Data for Interlaboratory Analyses
of Retinol, α-Tocopherol and β-Carotene

Table 1. Round Robin XVI Results

Retinol					Retinol % Bias from Grand Average			
	Serum	Serum	Serum	Serum	Serum	Serum	Serum	Serum
Lab #	109	110	111	112	109	110	111	112
	0.371	0.335	0.747	0.412	-5.3	-5.7	-2.7	-4.8
	0.381	0.344	0.781	0.434	-2.7	-3.2	1.7	0.3
	0.398	0.373	0.756	0.435	1.6	5.0	-1.5	0.5
	0.368	0.339	0.799	0.456	-6.0	-4.6	4.1	5.3
	0.397	0.380	0.730	0.444	1.4	7.0	-4.9	2.6
	0.399	0.355	0.765	0.437	1.9	-0.1	-0.4	1.0
	0.429	0.391	0.767	0.443	9.5	10.1	-0.1	2.3
	0.484	0.408	0.732	0.502	23.6	14.9	-4.7	16.0
	0.376	0.327	0.754	0.407	-4.0	-7.9	-1.8	-6.0
	0.511	1.105	0.475	0.576	30.5	211.1	-38.1	33.1
	0.387	0.355	0.747	0.431	-1.2	-0.1	-2.7	-0.4
	0.418	0.374	0.850	0.474	6.7	5.3	10.7	9.5
	0.390	0.355	0.755	0.410	-0.4	-0.1	-1.7	-5.3
	0.378	0.355	0.756	0.404	-3.5	-0.1	-1.5	-6.7
	0.418	0.397	0.999	0.513	6.7	11.8	30.1	18.5
	0.410	0.377	0.765	0.454	4.7	6.1	-0.4	4.9
	0.355	0.344	0.702	0.392	-9.4	-3.2	-8.6	-9.4
	0.435	0.371	0.799	0.465	11.1	4.4	4.1	7.4
	0.383	0.329	0.763	0.433	-2.2	-7.5	-0.7	-0.1
	0.855	0.837	1.723	1.015	118.3	135.6	124.4	134.5
	0.337	0.322	0.711	0.387	-14.0	-9.3	-7.4	-10.6
	0.370	0.320	0.760	0.410	-5.5	-9.9	-1.0	-5.3
	0.369	0.322	0.797	0.411	-5.8	-9.3	3.8	-5.0
	0.390	0.370	0.720	0.410	-0.4	4.2	-6.2	-5.3
	0.366	0.327	0.704	0.392	-6.6	-7.9	-8.3	-9.4
	0.374	0.315	0.713	0.392	-4.5	-11.3	-7.1	-9.4
	0.390	0.360	0.770	0.430	-0.4	1.4	0.3	-0.7
AVG	0.392	0.355	0.768	0.433				
SD	0.031	0.026	0.061	0.033				
RSD	7.9	7.3	7.9	7.7				

* = Value Removed
NL = New Laboratory Values not included in the Average.

Previous Value	0.396	0.361	0.750	0.431	0.421
% Bias	-1.1	-1.6	2.4	0.4	2.8
Previous Date Analyzed	7/88	1/88	7/88	7/88	1/88

Table 2. Round Robin XVI Results

Alpha-Tocopherol

Lab #	Serum 109	Serum 110	Serum 111	Serum 112
	7.18	6.18	4.92	5.74
	6.60	5.77	4.66	5.66
	7.47	6.26	5.76	6.43
	7.02	6.13	5.16	6.30
	7.10	5.90	4.80	6.19
	7.03	5.72	5.19	6.01
	7.69	6.30	5.07	7.20
	6.54	5.72	4.41	
	7.14	5.67	4.79	5.97
	7.07	5.46	5.85	6.14
	6.48	5.66	4.55	5.69
	7.09	6.11	5.04	6.16
	6.89	5.95	5.01	5.95
	7.28	6.21	5.05	5.98
	7.40	6.20	5.13	6.35
	7.02	8.39	4.26	6.61
	7.24	5.96	4.92	6.03
	7.05	5.95	5.05	6.10
	6.44	5.71	4.74	5.45
	6.45	5.59	4.81	5.86
	7.18	5.67	4.78	5.77
	7.85	5.86	4.99	5.98
	7.12	6.16	5.23	6.18
	4.93	4.25	3.54	5.13
	6.14	4.65	3.56	5.21
	7.14	5.93	5.09	6.11
AVG	7.06	6.02	4.96	6.08
SD	0.37	0.57	0.36	0.37
RSD	5.3	9.5	7.3	6.1

Alpha-Tocopherol % Bias from Grand Average

Serum 109	Serum 110	Serum 111	Serum 112
1.7	2.6	-0.9	-5.6
-6.5	-4.2	-6.1	-6.9
5.8	3.9	16.0	5.8
-0.5	1.8	4.0	3.6
0.6	-2.0	-3.3	1.8
-0.4	-5.0	4.6	-1.1
9.0	4.6	2.1	18.4
-7.3	-5.0	-11.2	
1.2	-5.9	-3.5	-1.8
0.2	-9.4	17.8	1.0
-8.2	-6.1	-8.3	-6.3
0.5	1.5	1.5	1.3
-2.4	-1.2	0.9	-2.1
3.2	3.1	1.6	-1.7
4.9	2.9	3.4	4.4
-0.6	39.2	-14.2	8.8
2.6	-1.0	-0.9	-0.8
-0.1	-1.2	1.7	0.3
-8.8	-5.2	-4.5	-10.4
-8.6	-7.2	-3.1	-3.6
1.7	-5.9	-3.7	-5.1
11.2	-2.7	0.5	-1.6
0.9	2.3	5.4	1.7
-30.1	-29.4	-28.7	-15.6
-13.0	-22.8	-28.3	-14.3
1.2	-1.6	2.4	0.4

* = Value Removed

NL = New Laboratory Values not included in the Average.

Previous Value	7.02	5.84	4.96	6.05	5.99
% Bias	0.5	3.1	0.1	0.5	1.5
Previous Date Analyzed	7/88	1/88	7/88	7/88	1/88

Table 3A. Round Robin XVI Results

	Total Beta-Carotene				Total Beta-Carotene % Bias from Grand Average			
	Serum	Serum	Serum	Serum	Serum	Serum	Serum	Serum
Lab #	109	110	111	112	109	110	111	112
	0.083	2.838	0.341	0.210	-0.2	11.7	-5.4	2.9
	0.066	2.157	0.299	0.165	-20.6	-15.1	-17.1	-19.1
	0.059	2.580	0.318	0.179	-29.1	1.5	-11.8	-12.3
	0.069	2.219	0.443	0.192	-17.0	-12.7	22.9	-5.9
	0.087	2.369	0.374	0.205	4.6	-6.8	3.7	0.5
	0.136	2.365	0.461	0.219	63.5	-6.9	27.9	7.4
	0.075	2.876	0.323	0.252	-9.8	13.2	-10.4	23.5
	0.108	2.390	0.374	0.261	29.9	-6.0	3.7	27.9
	0.080	2.718	0.359	0.200	-3.8	6.9	-0.4	-2.0
	0.090	3.050	0.455	0.222	8.2	20.0	26.2	8.8
	0.094	2.470	0.330	0.209	13.0	-2.8	-8.5	2.5
	0.087	2.277	0.304	0.199	4.6	-10.4	-15.7	-2.5
	0.077	2.675	0.365	0.205	-8.0	5.2	1.1	0.5
	0.060	2.380	0.320	0.180	-27.9	-6.4	-11.2	-11.8
	0.077	2.760	0.342	0.162	-7.4	8.6	-5.1	-20.6
	0.046	1.329	0.208	0.121	-44.3	-47.7	-42.3	-40.7
	0.130	4.200	0.610	0.380	56.3	65.3	69.2	86.3
AVG	0.083	2.542	0.361	0.204				
SD	0.020	0.266	0.053	0.028				
RSD	23.6	10.5	14.8	13.6				

* = Value Removed
NL = New Laboratory Values not included in the Average.

Previous Value	0.085	2.479	0.343	0.206	0.205
% Bias	-2.2	2.5	5.1	-1.0	-0.5
Previous Date Analyzed	7/88	1/88	7/88	7/88	1/88

Table 3B. Round Robin XVI Results

Trans Beta-Carotene					Trans Beta-Carotene % Bias from Grand Average			
	Serum	Serum	Serum	Serum	Serum	Serum	Serum	Serum
Lab #	109	110	111	112	109	110	111	112
	0.053	0.395	2.000	0.145	-37.9	-83.9	510.4	-27.1
	0.105	2.495	0.330	0.205	23.0	2.0	0.7	3.0
	0.076	2.330	0.310	0.196	-10.9	-4.7	-5.4	-1.5
	0.075	2.513	0.343	0.196	-12.1	2.7	4.7	-1.5
	0.078	2.194	0.293	0.164	-8.6	-10.3	-10.6	-17.6
AVG	0.085	2.446	0.328	0.199				
SD	0.017	0.101	0.017	0.005				
RSD	20.0	4.1	5.1	2.6				

* = Value Removed

NL = New Laboratory Values not included in the Average.

Previous Value	0.061	2.746	0.261	0.163	0.206
% Bias	39.9	-10.9	25.5	22.1	-3.4
Previous Date Analyzed	7/88	1/88	7/88	7/88	1/88

Interlaboratory Precision
vs
Time

FIGURE A

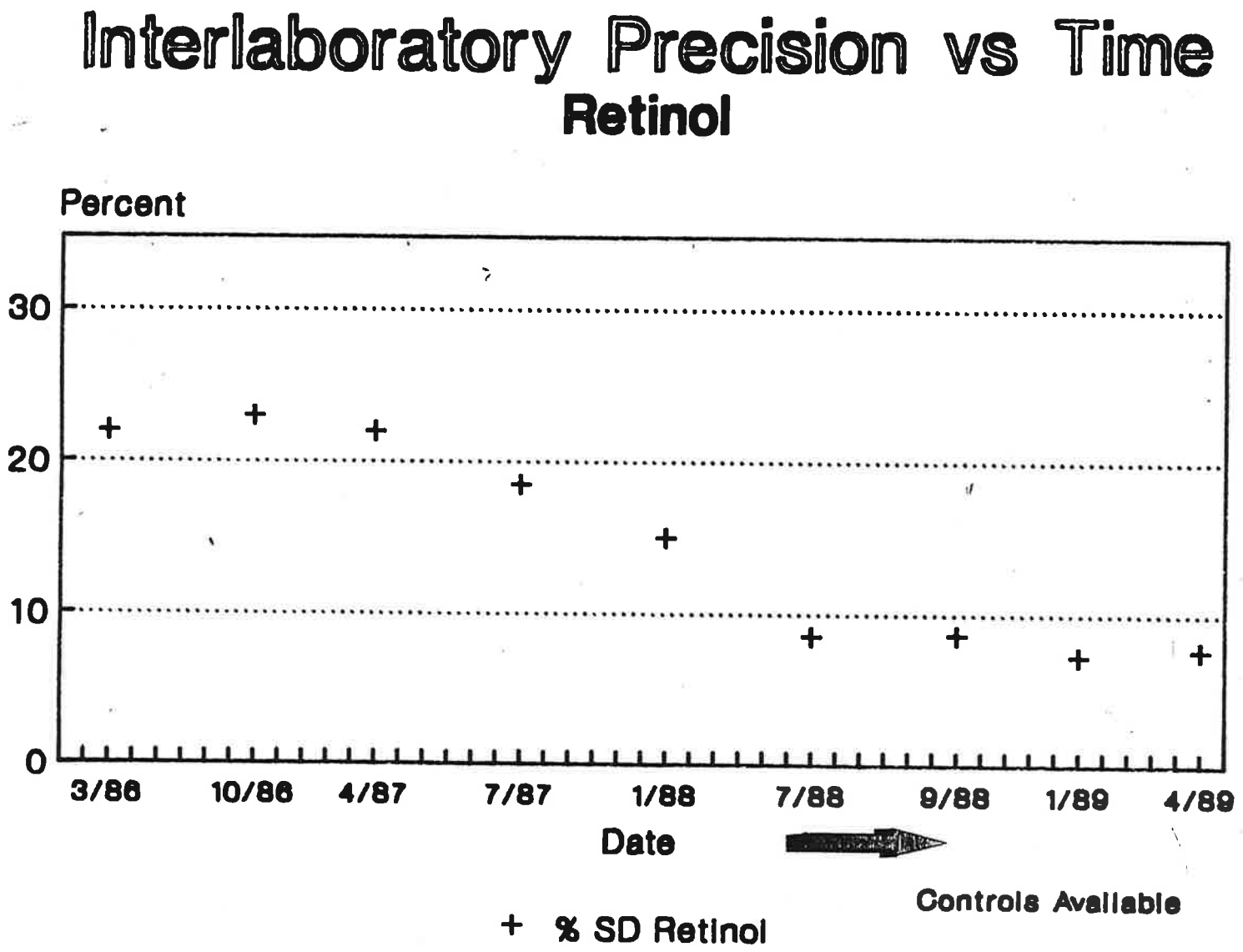


FIGURE B

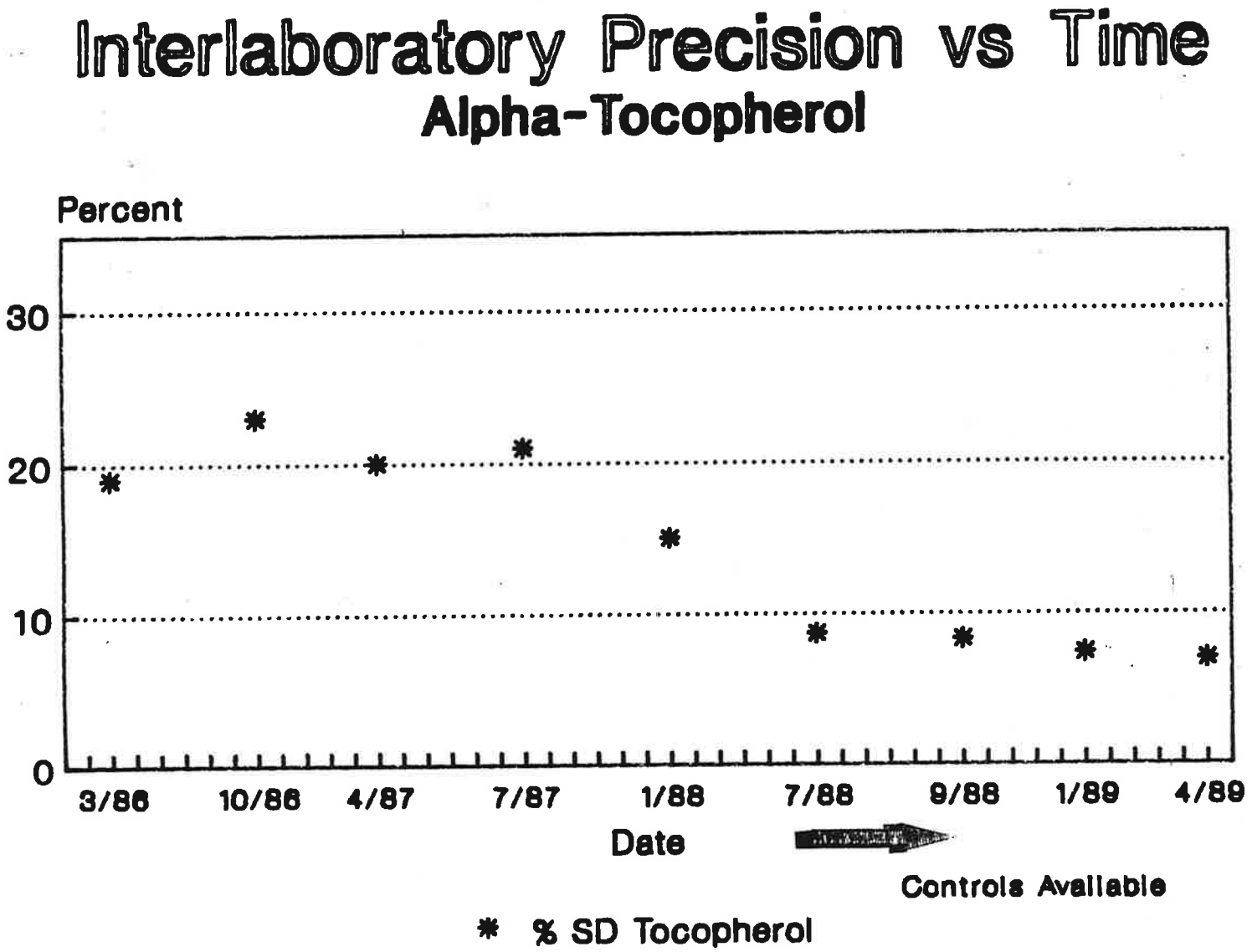
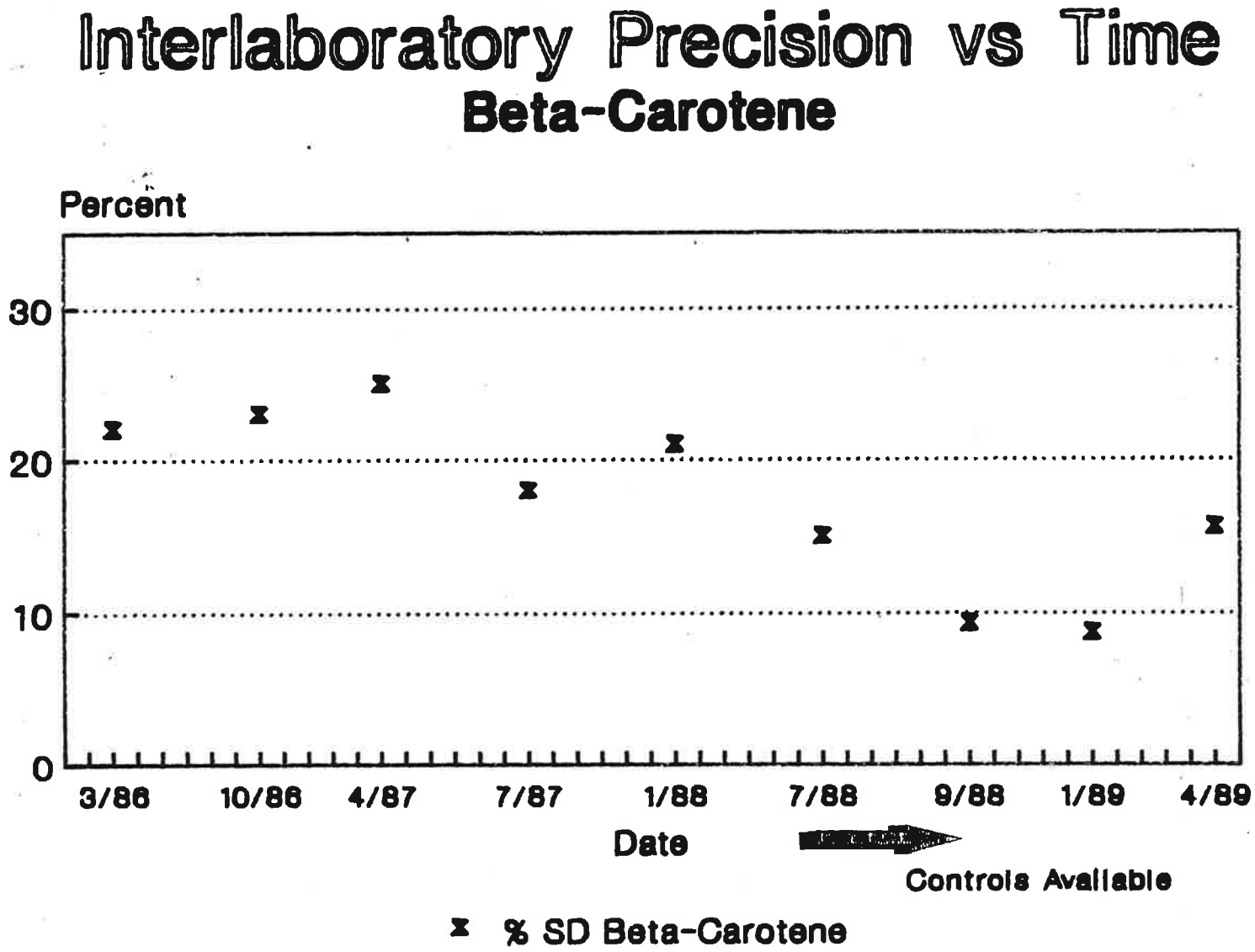
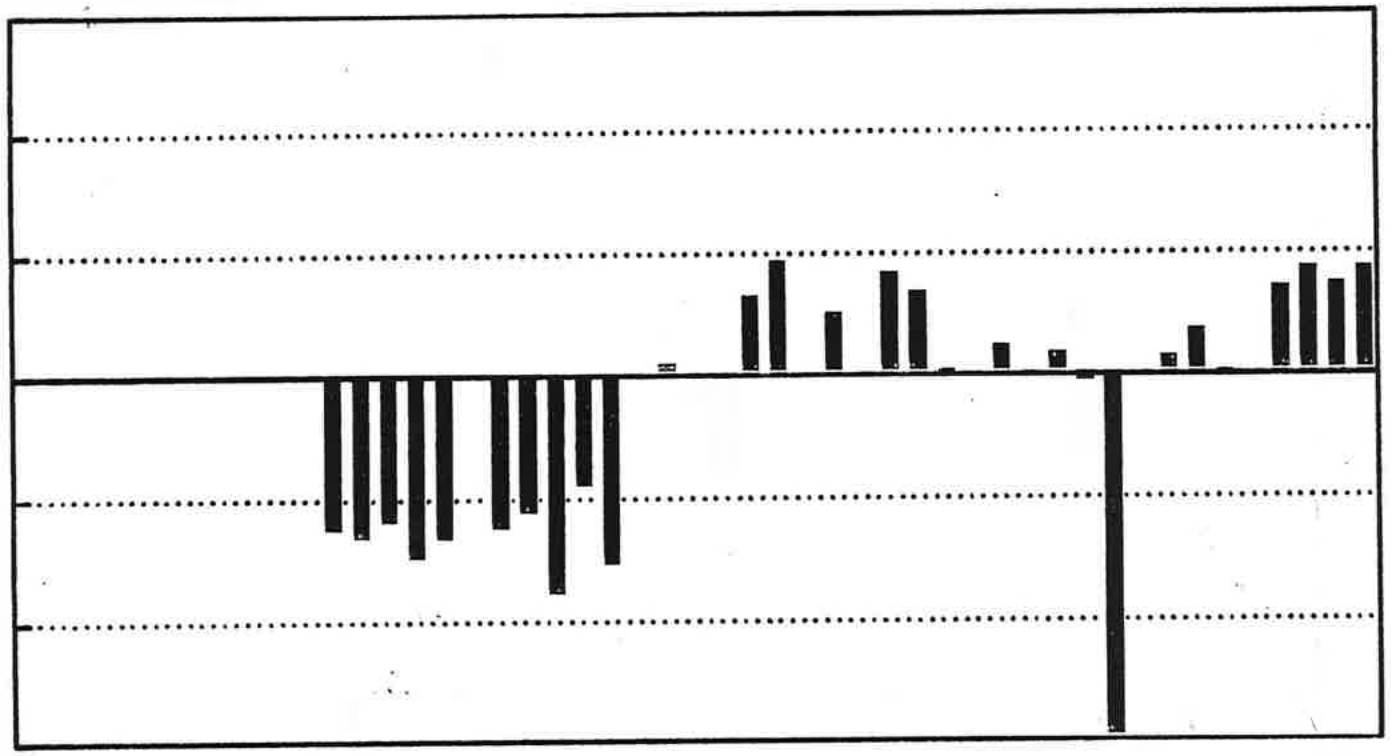


FIGURE C



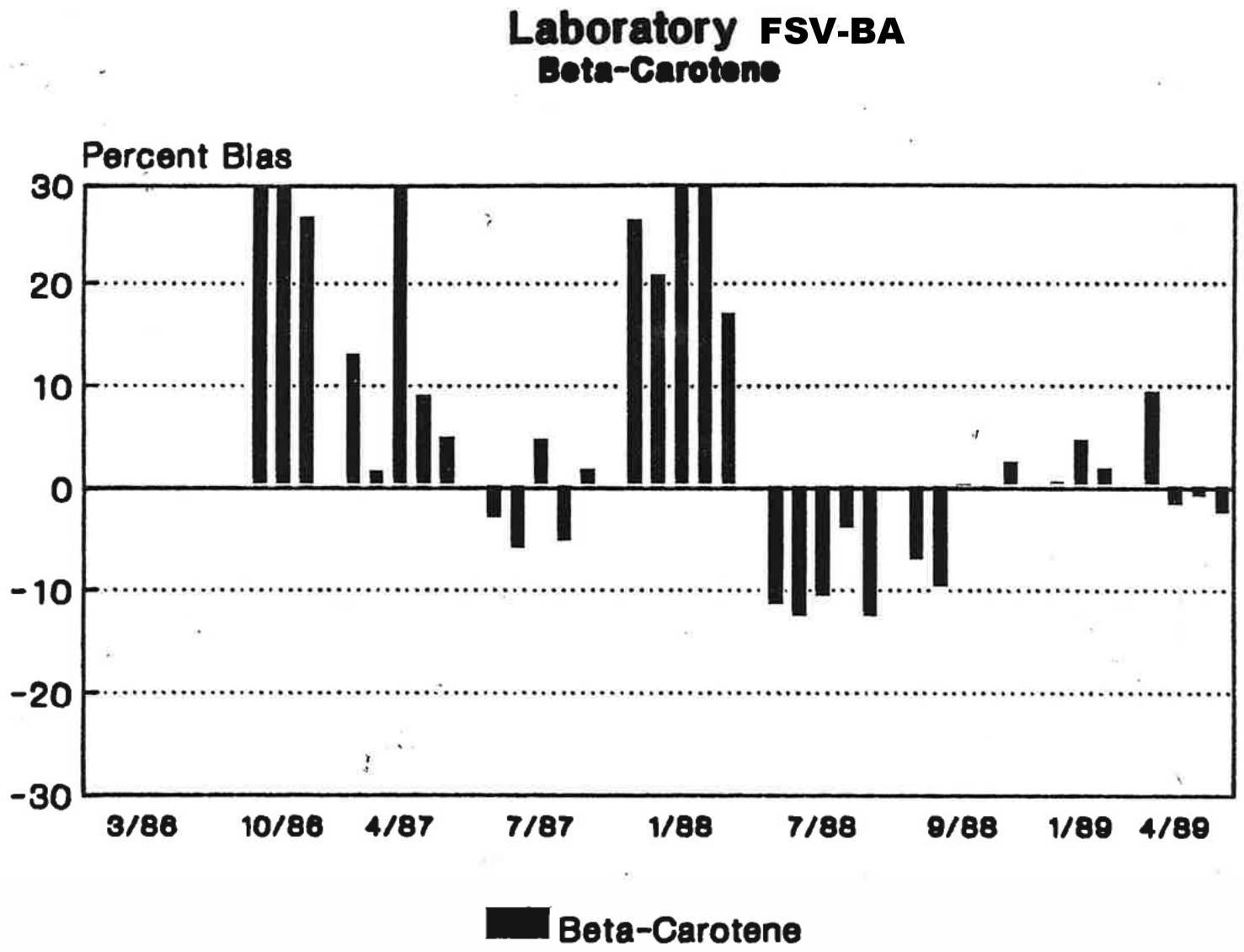
Individual Laboratory Accuracy
vs
Time

Laboratory FSV-BA
Retinol

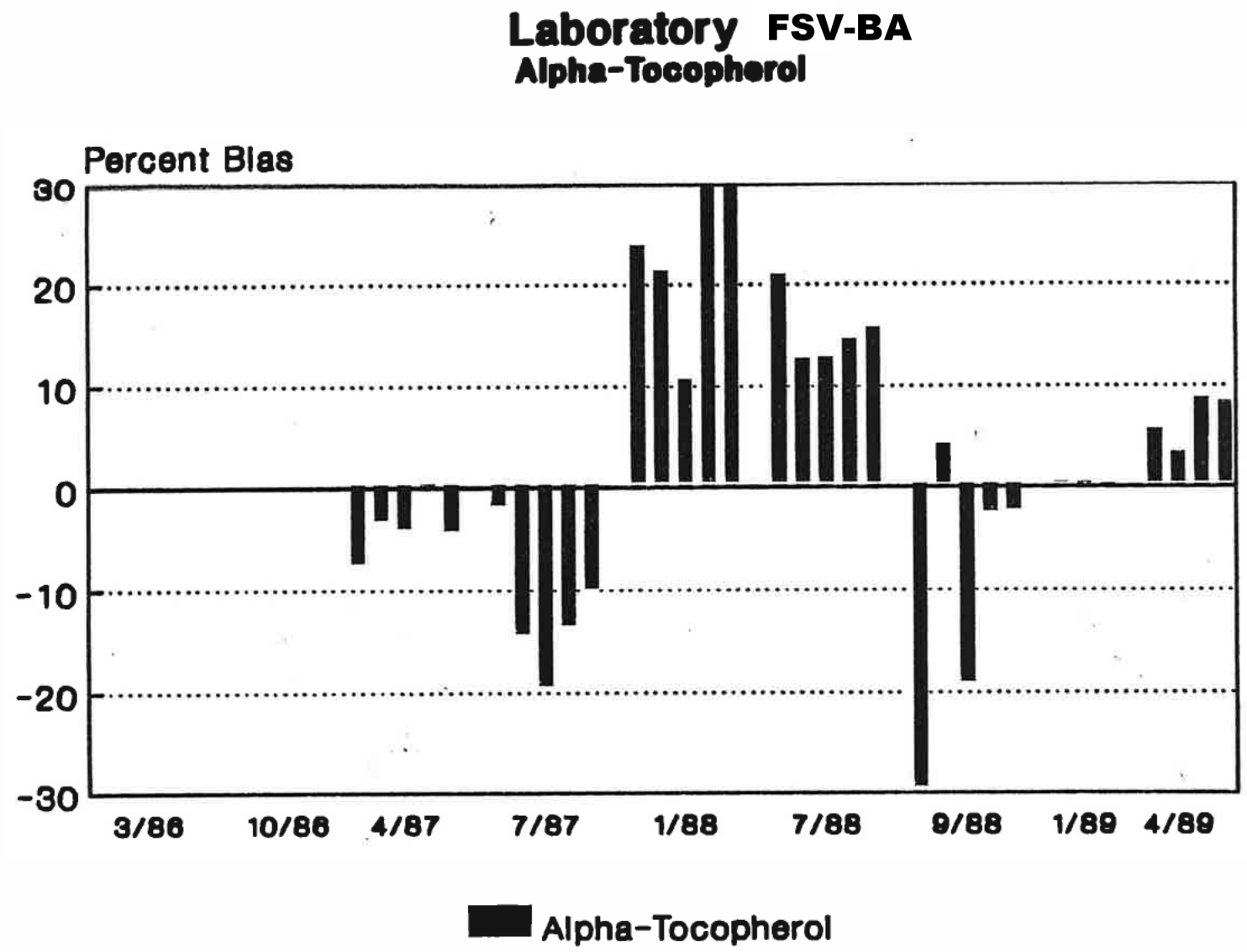


Retinol

% Bias from Assigned Values



% Bias from Assigned Values

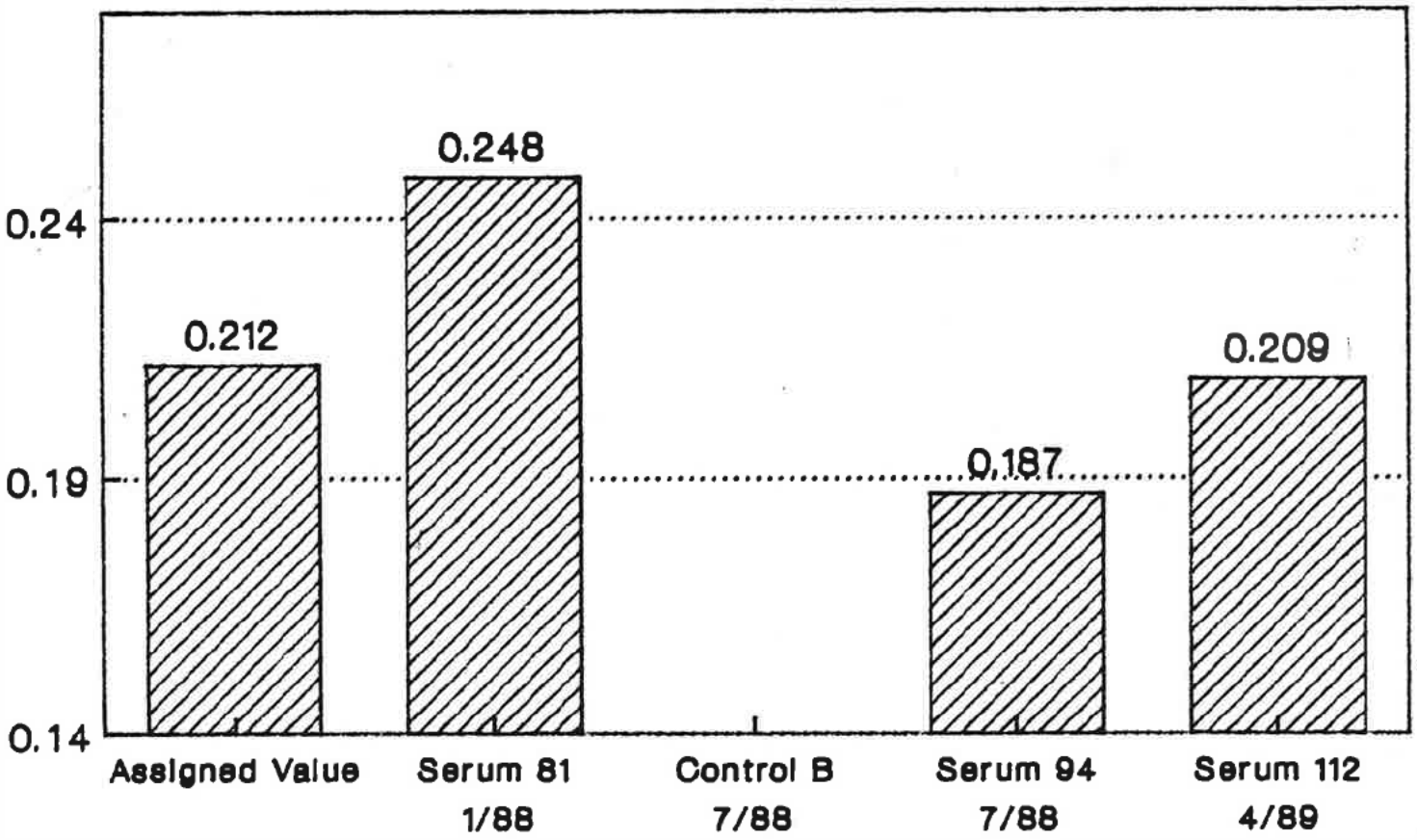


Blind Relicate Analyses of
“Control B”
Over Time

Laboratory **FSV-BA**

Control Material B - Blind Comparison

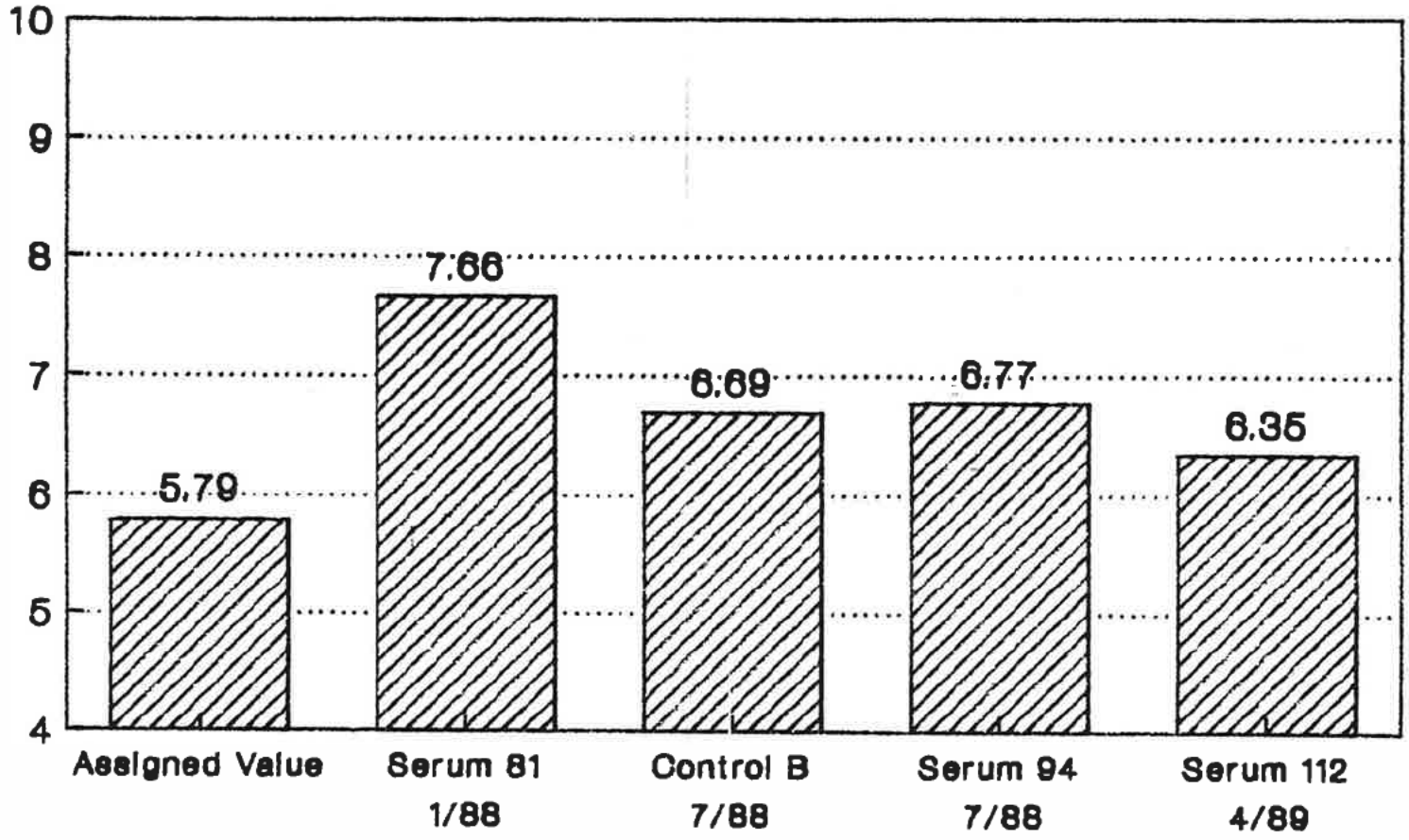
Beta-Carotene



Laboratory **FSV-BA**

Control Material B - Blind Comparison

Alpha-Tocopherol



Appendix F. Updated "All-Lab Report" for RR16

The following three pages are an updated version of an "All-Lab" report for RR16. This report has three parts:

- page 1 lists results for all analytes that were reported at least twice, counting both participants and NIST analysts.
- page 2 provides a legend for page 1.
- page 3 summarizes each participants' performance for retinol, α-tocopherol, and total β-carotene. These summaries are compatible with the percent bias evaluation advice given in the RR16 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 ("Avg") used in the original and detailed in Appendix E.

To ensure confidentiality, the laboratory identifiers used in this "All-Lab Report" have been altered from those used in RR16. 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 XVI Laboratory Results

	Total Retinol					α-Tocopherol					Total β-Carotene					trans-β-Carotene				
Code	109	110	111	112	113	109	110	111	112	113	109	110	111	112	113	109	110	111	112	113
FSV-BA	0.410	0.377	0.765	0.454	0.888	7.40	6.20	5.13	6.35	16.97	0.094	2.470	0.330	0.209	1.289	0.076	2.330	0.310	0.196	0.561
FSV-BD	0.429	0.391	0.767	0.443	0.721	7.69	6.30	5.07	7.20	14.39										
FSV-BE	0.418	0.374	0.850	0.474	1.090	6.89	5.95	5.01	5.95	18.36										
FSV-BF	0.376	0.327	0.754	0.407	1.023	7.14	5.67	4.79	5.97	17.34	0.075	2.876	0.323	0.252	1.328					
FSV-BG	0.398	0.373	0.756	0.435	0.985	7.47	6.26	5.76	6.43	18.19	0.059	2.580	0.318	0.179	1.275					
FSV-BH	0.383	0.329	0.763	0.433	0.958	7.05	5.95	5.05	6.10	17.00	0.077	2.675	0.365	0.205	≥0.729	0.075	2.513	0.343	0.196	0.729
FSV-BI	0.381	0.344	0.781	0.434	0.983	6.60	5.77	4.66	5.66	17.95	0.066	2.157	0.299	0.165	1.372					
FSV-BL	0.366	0.327	0.704	0.392		7.12	6.16	5.23	6.18											
FSV-BX											≥0.078	≥2.194	≥0.293	≥0.164	≥1.230	0.078	2.194	0.293	0.164	1.230
FSV-BY	0.371	0.335	0.747	0.412	0.987	7.18	6.18	4.92	5.74	18.07	0.083	2.838	0.341	0.210						
FSV-BZ	0.855	0.837	1.723	1.015	0.889	6.44	5.71	4.74	5.45	7.79										
FSV-CA	0.397	0.380	0.730	0.444	0.908	7.10	5.90	4.80	6.19	16.01										
FSV-CJ	0.368	0.339	0.799	0.456		7.02	6.13	5.16	6.30		0.069	2.219	0.443	0.192						
FSV-CM	0.390	0.370	0.720	0.410	0.430	7.85	5.86	4.99	5.98	4.91										
FSV-CN	0.484	0.408	0.732	0.502	0.416	6.54	5.72	4.41		9.08	0.136	2.365	0.461	0.219	1.220					
FSV-CO	0.399	0.355	0.765	0.437	1.014	7.03	5.72	5.19	6.01	19.33	0.087	2.369	0.374	0.205	1.331					
FSV-CQ	0.511	0.475	1.105	0.576	1.334	7.07	5.85	5.46	6.14	19.56	≥0.053	≥2.000	≥0.395	≥0.145	≥1.389	0.053	2.000	0.395	0.145	1.389
FSV-CS	0.437	0.356	0.652	0.483		3.35	5.42	3.36	2.84											
FSV-CY	0.370	0.320	0.760	0.410	0.950	7.18	5.67	4.78	5.77	19.26	0.060	2.380	0.320	0.180	≥1.110					1.110
FSV-DC	0.378	0.355	0.756	0.404	1.057						0.080	2.718	0.359	0.200	1.545					
FSV-DE						7.09	6.11	5.04	6.16	18.41										
FSV-DG	0.390	0.355	0.755	0.410	0.924	7.28	6.21	5.05	5.98	18.34	≥0.105	≥2.495	≥0.330	≥0.205	1.685	0.105	2.495	0.330	0.205	0.454
FSV-DH	0.374	0.315	0.713	0.392	0.930	7.14	5.93	5.09	6.11	21.41	0.046	1.329	0.208	0.121	≥1.260					1.260
FSV-DT	0.387	0.355	0.747	0.431	1.076	6.48	5.66	4.55	5.69	19.62	0.108	2.390	0.374	0.261	1.464					0.619
FSV-DY	0.369	0.322	0.797	0.411	0.719						0.077	2.760	0.342	0.162	≥1.096					1.096
FSV-DZ	0.355	0.344	0.702	0.392	0.919	7.02	8.39	4.26	6.61	15.08										
FSV-ED	0.435	0.371	0.799	0.465	0.999	7.24	5.96	4.92	6.03	17.68	0.087	2.277	0.304	0.199	1.178					
FSV-EO	0.418	0.397	0.999	0.513	0.892						0.090	3.050	0.455	0.222	≥0.306					0.306
FSV-FA	0.390	0.360	0.770	0.430	0.870						0.130	4.200	0.610	0.380	≥2.400					2.400
FSV-FE	0.337	0.322	0.711	0.387	0.859	6.45	5.59	4.81	5.86	19.10										
FSV-FX						5.54	4.45	3.55	5.17	21.50										
n	28	28	28	28	25	26	26	26	25	23	17	17	17	17	10	5	5	5	5	11
Min	0.337	0.315	0.652	0.387	0.416	3.35	4.45	3.36	2.84	4.91	0.046	1.329	0.208	0.121	1.178	0.053	2.000	0.293	0.145	0.306
Mean	0.413	0.375	0.808	0.459	0.913	6.86	5.95	4.84	5.91	16.75	0.084	2.568	0.366	0.209	1.369	0.077	2.306	0.334	0.181	1.014
Max	0.855	0.837	1.723	1.015	1.334	7.85	8.39	5.76	7.20	21.50	0.136	4.200	0.610	0.380	1.685	0.105	2.513	0.395	0.205	2.400
SD	0.094	0.096	0.200	0.117	0.190	0.851	0.618	0.510	0.750	4.164	0.024	0.570	0.088	0.055	0.155	0.018	0.215	0.039	0.026	0.587
CV	23	26	25	25	21	12	10	11	13	25	28	22	24	26	11	24	9	12	14	58
NISTd	0.390	0.334	0.727	0.381		6.75	5.59	4.37	5.37		*nq*	2.447	0.304	0.177		0.078	2.208	0.259	0.149	
Median	0.390	0.355	0.758	0.433	0.930	7.08	5.91	4.96	6.01	18.07	0.080	2.470	0.342	0.205	1.329	0.076	2.330	0.330	0.196	1.096
eSD	0.030	0.031	0.040	0.034	0.089	0.35	0.34	0.26	0.30	1.76	0.016	0.368	0.047	0.025	0.121	0.004	0.245	0.030	0.013	0.544
eCV	7.6	8.8	5.3	8.0	9.6	5.0	5.8	5.2	5.0	9.8	20.4	14.9	13.9	12.3	9.1	5.0	10.5	9.0	6.8	49.6

F2

Round Robin XVI Laboratory Results

Analytes Reported By One Laboratory

Values in µg/mL

Analyte	Code	109	110	111	112	113
γ/β-Tocopherol	FSV-DE	2.720	1.740	2.440	3.07	*nd*

Table Legend

Symbol	Interpretation
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*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
nd	Not detected (i.e., no detectable peak for analyte)
nq	Detected but not quantitatively determined
≥x	Concentration greater than or equal to x

Round Robin XVI Laboratory Results

%Bias Summary

Lab	TR	aT	bC
FSV-BA	4±2	5±1	4±9
FSV-BD	6±5	9±7	
FSV-BE	9±3	0±2	
FSV-BF	-5±3	-2±2	7±15
FSV-BG	2±2	9±5	-10±13
FSV-BH	-2±4	1±1	3±6
FSV-BI	-1±3	-5±2	-16±3
FSV-BL	-8±1	3±2	
FSV-BX			-12±7
FSV-BY	-4±2	0±4	5±7
FSV-BZ	129±8	-7±3	
FSV-CA	2±4	0±3	
FSV-CJ	0±6	3±3	0±20
FSV-CM	-2±5	3±6	
FSV-CN	13±12	-7±4	27±33
FSV-CO	1±1	0±3	4±7
FSV-CQ	36±7	3±5	-17±22
FSV-CS	2±12	-36±21	
FSV-CY	-5±4	-3±3	-7±4
FSV-DC	-3±3		3±6
FSV-DE		2±1	
FSV-DG	-1±3	2±2	7±16
FSV-DH	-8±3	1±1	-42±3
FSV-DT	-1±1	-7±2	17±17
FSV-DY	-4±6		-3±14
FSV-DZ	-7±3	9±24	
FSV-ED	7±3	1±1	-3±9
FSV-EO	17±11		19±11
FSV-FA	1±1		74±10
FSV-FE	-10±3	-5±3	
FSV-FX		-22±6	
NISTd	-6±5	-8±4	-9±7

Label	Definition
Lab	Participant code
TR	Total Retinol
aT	a-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 "Avg" 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-based reference value.

Appendix G. Shipping Package Inserts for RR17

The following two items were attached to each package shipped to an RR17 participant:

- **Cover letter**.
- **Datasheet**.

UNITED STATES DEPARTMENT OF COMMERCE
National Institute of Standards and Technology
[formerly National Bureau of Standards]
Gaithersburg, Maryland 20899

August 11, 1989

Dear Colleague:

Samples for RR-XVII are enclosed. The sample set is composed of four serum pools and an ethanol solution. The serum samples are to be analyzed for retinol, α-tocopherol and total β-carotene as usual. The ethanol solution contains retinol, α-tocopherol and a complex mixture of β-carotene isomers. We request that you provide data for the concentration of retinol, α-tocopherol and all-trans β-carotene in the solution by the procedure that you think most appropriate. We also request that you provide us with a copy of the chromatogram from your analysis of the solution; since the primary objective associated with the analysis of the solution is the resolution of β-carotene isomers. Results are due by October 1.

The 1989 NCI/NBS Micronutrient Analysis Workshop will be held on November 1 at the National Institute of Standards and Technology in the Metrology Building, Room A340. A copy of the preliminary agenda for the meeting is enclosed. Everyone involved in the QA Program is invited. Laboratories joining the program during the past year are strongly encouraged to attend. Please fill out the QA Workshop registration form and return it in the enclosed envelop indicating your intentions to attend or not to attend the meeting. Rooms have been reserved at the Gaithersburg Quality Inn (301/963-5900) at the rate of $51/night. You must make your reservation before October 17 and mention that you are attending the "Micronutrient Analysis Workshop" to get this special rate. A fee of $10 will be collected from each participant which will cover lunch and refreshments for the day.

Hands-on "Vitamins in Serum Measurement Workshops" are being held on October 31 and November 2 to assist laboratories that are experiencing problems with their analyses. Please contact Neal Craft (301/975-3111) to reserve space in one of these sessions.

I look forward to your participation in this year's Workshop.

Sincerely,

Willie E. May, Ph.D.
Chief
Organic Analytical Research Division
Center for Analytical Chemistry

cc: R. Schaffer
N. Craft

PLEASE PROVIDE: Telephone Number: _____; Telefax Number: _____
Telegram Number: _____

REPORT ON NIST/NCI SAMPLES FROM LABORATORY # _____

RR XVII DATE OF ANALYSIS _____

RESULTS IN µg/mL

SAMPLE #	ANALYTE	RESULT	
SERUM 115	RETINOL		
VIAL # ___		All Trans	Total
	B-CAROTENE		
	A-TOCOPHEROL		
SERUM 116	RETINOL		
VIAL # ___		All Trans	Total
	B-CAROTENE		
	A-TOCOPHEROL		
SERUM 117	RETINOL		
VIAL # ___		All Trans	Total
	B-CAROTENE		
	A-TOCOPHEROL		
SERUM 118	RETINOL		
VIAL # ___		All Trans	Total
	B-CAROTENE		
	A-TOCOPHEROL		
SERUM 119	RETINOL		
VIAL # ___		All Trans	Total
	B-CAROTENE		
	A-TOCOPHEROL		
SOLUTION 114	RETINOL		
EtOH solution		All Trans	
VIAL # ___	B-CAROTENE		
	A-TOCOPHEROL		

Add 1.0 mL of Distilled water to samples 115-119.

Appendix H. Final Report for RR17

The following fourteen pages are the the available components of the final report for RR17 as provided to all participants. The samples distributed in RR17 were the same as had been distributed in RR09. The original report consisted of:

- An individualized cover letter and discussion. The available letters are the proof copies of mail-merge letter forms sent to:
  - Experienced participants who sent in results for both RR09 and RR17.
  - Experienced participants who sent in results for RR17 but had not participanted in RR09.
  - New participants who sent in results for RR17.
  - Participants who did not send in results for RR17.
- Tables 1 to 4 list all of the participants' results and several summary values for retinol, α-tocopherol, total β-carotene, and *trans*-β-carotene. These tables do not report any NIST results nor participant results for other analytes.
- Graphical presentations of interlaboratory precision over time for retinol, α-tocopherol, and total β-carotene. Copies of these figures are no longer available, but would have been similar to those for RR16 in Appendix E.
- Graphical presentations of the indiviual participant's bias relative to an assigned value over time for retinol, α-tocopherol, and total β-carotene. Copies of these figures are no longer available, but would have been similar to those for RR16 in Appendix E.
- Graphical comparisons of the indiviual participant's assays of their RR09 and RR17 results for retinol, α-tocopherol, and total β-carotene. Copies of these figures are no longer available, but would have been similar in format to those for the "Control B" analyses in the RR16 report 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 RR17 results where the original codes have been altered to ensure confidentiality. Appendix C also provides more relevant summary statistics.

This letter was sent to experienced participants who sent in results for both RR09 and RR17.

January 2, 1990

^F1^
^F2^

Individualized letters were sent to study participants. The "^F1^" and "^F2^" were mail-merge commands for inserting a participant's name and address.

HAPPY NEW YEAR!!

A statistical summary of the results from your measurements of retinol, α-tocopherol and β-carotene in the four serum samples sent to you as RR-XVII is provided to you in both graphical and tabular form. Tables 1-4 (section A) provide a summary of the data submitted for retinol, α-tocopherol, total β-carotene, and all-trans β-carotene, respectively, in the four serum samples. These tables also provide assigned values (based on the laboratory grand averages), standard deviations (SD), relative standard deviations (RSD) for the interlaboratory measurement of each analyte, and data representing each laboratory's bias on a sample-by-sample basis for each analyte.

The tables also provide data documenting the "ruggedness" of the grand average values derived through our study group. Each of the serum samples used in RR-XVII had been analyzed in previous Round Robins. The current grand averages, grand averages from each previous round robin, and the differences between them are provided at the bottom left side of each table. The agreement among the grand averages from the most recent and past round robins along with data from NIST analyses of these and other sera similarly stored, document the stability of retinol, α-tocopherol, and β-carotene in freeze-dried sera stored at -80°C.

Three types of graphical data are provided:

- Figures A-C (in Section B) which demonstrate the improvement in interlaboratory precision for the measurement of retinol, α-tocopherol, and β-carotene, that has been achieved in our program over the past three years.

- Control Charts (in Section C) for retinol, α-tocopherol, and β-carotene, respectively, which represent a summary of your lab's performance vs. the assigned value for each analyte over the past three years.

- A comparison (in Section A) of the values that your lab has reported for a single sample for each of the three times that it has been distributed as an unknown.

As mentioned in the two most recent reports, interlaboratory Grand Averages have been determined to be acceptable approximations of the true values for each sample, and will be used as assigned values except in rare cases. NIST measurements will continue to be performed to verify that no significant bias exists. Under our new performance standards, 0-5% bias from the assigned value will represent exceptional performance (AA); >5-10% bias from the assigned value will represent acceptable performance (A); >10-20% bias from the assigned value will represent marginal performance (M); and >20% bias will represent unacceptable performance (U).

Your laboratory's performance in round-robin XVII was deemed to be:

RETINOL	_____
α-TOCOPHEROL	_____
TOTAL β-CAROTENE	_____
TRANS β-CAROTENE	_____

Our experiences over the past year have clearly demonstrated the efficacy of the use of reference materials for improving accuracy and interlaboratory precision. The Control Materials provided to the group in the spring of 1988 were developed as a pilot effort to demonstrate the benefits that could be derived through proper use of reference materials. The supply of those samples has been depleted. These Control Materials have been replaced with Standard Reference Material 968, "Fat-Soluble Vitamins in Freeze-Dried Human Serum." This SRM can be ordered from the NIST Office of Standard Reference Materials (301/975-6776) at a cost of $185 per unit, including shipping.

If your lab's performance is consistently marginal or unsatisfactory for any analyte, please feel free to contact me (301/975-3108) or Neal Craft (301/975-3111) for discussions regarding possible sources of bias in your methodology.

In addition to serum samples, you have been asked to analyze a solution containing retinol, α-tocopherol, and β-carotene isomers in the past two round robins. Our intentions were two fold. First, we wanted to determine the relative contributions of sample preparation (extraction, solvent exchange, etc.) and chromatographic analysis to the ~7-10 % interlaboratory variability that we now consistently observe for retinol, α-tocopherol and β-carotene. We felt the ready-to-analyze solution, would effectively avoid the variability associated with sample preparation, leaving only that associated with chromatographic analysis. The results provided in Table 5 are far worse than those provided for serum, indicating that the purpose and protocols for conducting our "simple" experiment were perhaps not clearly explained to you.

Secondly, we wanted labs to see the need to modify their chromatographic systems (stationary phase/mobile phase combinations) for resolution of individual β-carotenoid isomers. We asked you to provide data for both total- and all trans-β-carotene. The results for total β-carotene were only slightly worse than we normally see for serum. The limited data reported for trans-β-carotene were, as we expected, very variable.

ROUND ROBIN XVIII will consist of three serum samples and another ethanol solution of the retinol, α-tocopherol, and β-carotene. These samples **will be shipped to you on January 15, 1990.** Analyze the serum samples as you normally do. We also invite you to provide us with data on retinol palmitate, lycopene, and γ-tocopherol in these samples.

We wish to give the solution experiment one additional try with what we hope are more clear instructions. Please complete your serum analyses before you analyze the unknown solution. For analysis of the unknown solution, we ask that you calibrate your system with the set of solution standards provided. Quantitation should be accomplished by comparing the response from the unknown with that from the calibration solutions. No extractions, additions of internal standards, or solvent exchanges should be required for labs using reversed-phase systems. The assigned values for the target analytes in calibration solutions to be used for analysis of the unknown solution follows:

(μg/mL)

	RETINOL	α-TOCOPHEROL	TRANS β-CAROTENE
Solution 10	0.195	2.22	0.135
Solution 11	0.977	11.1	0.945
Solution 12	1.95	22.2	1.62

These solutions should be allowed to equilibrate at room temperature for at least 4 hours, but no more than 24 hours prior to use.

Results from the analysis of **RR-XVIII are due by March 5, 1990.**

Sincerely,

Willie E. May, Ph.D.
Chief
Organic Analytical Research Division
Center for Analytical Chemistry

cc: Robert Schaffer
Winfred Malone

This letter was sent to experienced participants who sent in results for RR17 but had not participanted in RR09.

January 2, 1990

^F1^
^F2^

Individualized letters were sent to study participants. The "^F1^" and "^F2^" were mail-merge commands for inserting a participant's name and address.

HAPPY NEW YEAR!!

A statistical summary of the results from your measurements of retinol, α-tocopherol and β-carotene in the four serum samples sent to you as RR-XVII is provided to you in both graphical and tabular form. Tables 1-4 (section A) provide a summary of the data submitted for retinol, α-tocopherol, total β-carotene, and all-trans β-carotene, respectively, in the four serum samples. These tables also provide assigned values (based on the laboratory grand averages), standard deviations (SD), relative standard deviations (RSD) for the interlaboratory measurement of each analyte, and data representing each laboratory's bias on a sample-by-sample basis for each analyte.

The tables also provide data documenting the "ruggedness" of the grand average values derived through our study group. Each of the serum samples used in RR-XVII had been analyzed in previous Round Robins. The current grand averages, grand averages from each previous round robin, and the differences between them are provided at the bottom left side of each table. The agreement among the grand averages from the most recent and past round robins along with data from NIST analyses of these and other sera similarly stored, document the stability of retinol, α-tocopherol, and β-carotene in freeze-dried sera stored at -80°C.

Two types of graphical data are provided:

- Figures A-C (in Section B) which demonstrate the improvement in interlaboratory precision for the measurement of retinol, α-tocopherol, and β-carotene, that has been achieved in our program over the past three years.

- Control Charts (in Section C) for retinol, α-tocopherol, and β-carotene, respectively, which represent a summary of your lab's performance vs. the assigned value for each analyte over the past three years.

As mentioned in the two most recent reports, interlaboratory Grand Averages have been determined to be acceptable approximations of the true values for each sample, and will be used as assigned values except in rare cases. NIST measurements will continue to be performed to verify that no significant bias exists. Under our new performance standards, 0-5% bias from the assigned value will represent exceptional performance (AA); >5-10% bias from the assigned value will represent acceptable performance (A); >10-20% bias from the assigned value will represent marginal performance (M); and >20% bias will represent unacceptable performance (U).

Your laboratory's performance in round-robin XVII was deemed to be:

	73I	73E
RETINOL	_____	_____
α-TOCOPHEROL	_____	_____
TOTAL β-CAROTENE	_____	_____
TRANS β-CAROTENE	_____	_____

Our experiences over the past year have clearly demonstrated the efficacy of the use of reference materials for improving accuracy and interlaboratory precision. The Control Materials provided to the group in the spring of 1988 were developed as a pilot effort to demonstrate the benefits that could be derived through proper use of reference materials. The supply of those samples has been depleted. These Control Materials have been replaced with Standard Reference Material 968, "Fat-Soluble Vitamins in Freeze-Dried Human Serum." This SRM can be ordered from the NIST Office of Standard Reference Materials (301/975-6776) at a cost of $185 per unit, including shipping.

If your lab's performance is consistently marginal or unsatisfactory for any analyte, please feel free to contact me (301/975-3108) or Neal Craft (301/975-3111) for discussions regarding possible sources of bias in your methodology.

In addition to serum samples, you have been asked to analyze a solution containing retinol, α-tocopherol, and β-carotene isomers in the past two round robins. Our intentions were two fold. First, we wanted to determine the relative contributions of sample preparation (extraction, solvent exchange, etc.) and chromatographic analysis to the ~7-10 % interlaboratory variability that we now consistently observe for retinol, α-tocopherol and β-carotene. We felt the ready-to-analyze solution, would effectively avoid the variability associated with sample preparation, leaving only that associated with chromatographic analysis. The results provided in Table 5 are far worse than those provided for serum, indicating that the purpose and protocols for conducting our "simple" experiment were perhaps not clearly explained to you.

Secondly, we wanted labs to see the need to modify their chromatographic systems (stationary phase/mobile phase combinations) for resolution of individual β-carotenoid isomers. We asked you to provide data for both total- and all trans-β-carotene. The results for total β-carotene were only slightly worse than we normally see for serum. The limited data reported for trans-β-carotene were, as we expected, very variable.

ROUND ROBIN XVIII will consist of three serum samples and another ethanol solution of the retinol, α-tocopherol, and β-carotene. These samples **will be shipped to you on January 15, 1990.** Analyze the serum samples as you normally do. We also invite you to provide us with data on retinol palmitate, lycopene, and γ-tocopherol in these samples.

We wish to give the solution experiment one additional try with what we hope are more clear instructions. Please complete your serum analyses before you analyze the unknown solution. For analysis of the unknown solution, we ask that you calibrate your system with the set of solution standards provided. Quantitation should be accomplished by comparing the response from the unknown with that from the calibration solutions. No extractions, additions of internal standards, or solvent exchanges should be required for labs using reversed-phase systems. The assigned values for the target analytes in calibration solutions to be used for analysis of the unknown solution follows:

(μg/mL)

	RETINOL	α-TOCOPHEROL	TRANS β-CAROTENE
Solution 10	0.195	2.22	0.135
Solution 11	0.977	11.1	0.945
Solution 12	1.95	22.2	1.62

These solutions should be allowed to equilibrate at room temperature for at least 4 hours, but no more than 24 hours prior to use.

Results from the analysis of **RR-XVIII are due by March 5, 1990.**

Sincerely,

Willie E. May, Ph.D.
Chief
Organic Analytical Research Division
Center for Analytical Chemistry

cc: Robert Schaffer
Winfred Malone

This letter was sent to new participants who sent in results for RR17.

January 2, 1990

^F1^
^F2^

Individualized letters were sent to study participants. The "^F1^" and "^F2^" were mail-merge commands for inserting a participant's name and address.

HAPPY NEW YEAR!!

A statistical summary of the results from your measurements of retinol, α-tocopherol and β-carotene in the four serum samples sent to you for analysis as part of the NIST/NCI QA program is provided to you in Tables I-III. These tables also provide the NIST assigned value for each sample, the grand average for measurements made by six new laboratories joining the studies, standard deviation (SD) and relative standard deviation (RSD) for the interlaboratory measurement of each analyte, and data representing each laboratory's bias, vs the NIST assigned value, on a sample-by-sample basis for each analyte. Tables IV-VII represent data provided by the regular participants in our program for the most recent round robin study. Figures A-C demonstrate the improvement in interlaboratory precision for the measurement of retinol, α-tocopherol, and β-carotene, that have been accomplished in our program over the past three years.

It has been demonstrated that the interlaboratory Grand Average for measurements made by regular participants in our program is an acceptable approximation of the true value for each sample. This value will be used as the assigned value except in rare cases. NIST measurements will be performed to verify that no significant bias exists. Under our current performance standards, 0-5% bias from the assigned value represents exceptional performance (AA); >5-10% bias from the assigned value represents acceptable performance (A); >10-20% bias from the assigned value represents marginal performance (M); and >20% bias represents unacceptable performance (U).

Your laboratory's performance on the four samples provided to you was deemed to be:

RETINOL _____

α-TOCOPHEROL _____

TOTAL β-CAROTENE _____

These descriptors are subjective, and are intended only to provide you with information concerning your performance relative to labs that have been active in our program for three or more years. We anticipate that your performance will follow the trend shown for regular participants shown in the Figures A-C.

Our experiences over the past year have clearly demonstrated the efficacy of the use of reference materials for improving accuracy and interlaboratory precision. Standard Reference Material 968, "Fat-Soluble Vitamins in Freeze-Dried Human Serum" can be ordered from the NIST Office of Standard Reference Materials (301/975-6776) at a cost of $185 per unit, including shipping. A copy of the Certificate of Analysis for this SRM is provided for your information.

If your lab's performance is marginal or unsatisfactory for any analyte, please feel free to contact me (301/975-3108) or Neal Craft (301/975-3111) for discussions regarding possible sources of bias in your methodology.

In addition to serum samples, we asked the regular participants in our studies to analyze an ethanol solution containing retinol, α-tocopherol, and β-carotene isomers in each of the last two round robins. Our intentions were two-fold. First, we wanted to determine the relative contributions of sample preparation (extraction, solvent exchange, etc.) and chromatographic analysis to the ~7-8 % interlaboratory variability that we now consistently observe for retinol and α-tocopherol, and 10% for β-carotene. We felt that by providing a ready-to-analyze solution, we could effectively remove variability associated with sample preparation, leaving only that associated with chromatographic analysis of a simulated solvent exchanged extract. The results obtained were far worse that those provided for serum, indicating that our "simple" experiment was perhaps not adequately explained.

Secondly, we wanted the labs to see the need for labs to modify their chromatographic systems (stationary phase/mobile phase combinations) for resolution of individual β-carotenoid isomers. We asked labs to provide data for both total- and all trans- β-carotene. The results for total β-carotene were only slightly worse than we normally see for serum. The results for trans-β-carotene were, as we expected, very variable.

ROUND ROBIN XVIII will consist of three serum samples and another ethanol solution of the retinol, α-tocopherol, and β-carotene. These samples **will be shipped to you on January 15, 1990.** Analyze the serum samples as you normally do. We also invite you to provide us with data on retinol palmitate, lycopene, and γ-tocopherol in these samples.

We wish to give the solution experiment one additional try with what we hope are more clear instructions. Please complete your serum analyses before you analyze the unknown solution. For analysis of the unknown solution, we ask that you calibrate your system with the set of solution standards provided. Quantitation should be accomplished by comparing the response from the unknown with that from the calibration solutions. No extractions, additions of internal standards, or solvent exchanges should be required for labs using reversed-phase systems. The assigned values for the target analytes in calibration solutions to be used for analysis of the unknown solution follows:

(μg/mL)

	RETINOL	α-TOCOPHEROL	TRANS β-CAROTENE
Solution 10	0.195	2.22	0.135
Solution 11	0.977	11.1	0.945
Solution 12	1.95	22.2	1.62

These solutions should be allowed to equilibrate at room temperature for at least 4 hours, but no more than 24 hours prior to use.

Results from the analysis of **RR-XVIII are due by March 5, 1990.**

Sincerely,

Willie E. May, Ph.D.
Chief
Organic Analytical Research Division
Center for Analytical Chemistry

cc: Robert Schaffer
Winfred Malone

This letter was sent to participants who did not send in results for RR17.

January 4, 1990

^F1^ Individualized letters were sent to study participants. The "^F1^" and "^F2^"
^F2^ were mail-merge commands for inserting a participant's name and address.

HAPPY NEW YEAR!
Samples for analysis of **ROUND ROBIN XVIII** will be shipped to you on January 16, 1990, and will include three serum samples and an ethanol solution of retinol, γ-tocopherol and β-carotene. Analyze the serum samples as you normally do. We also invite you to provide us with data on retinyl palmitate, lycopene, and γ-tocopherol in these samples.

Please complete your serum analyses before you analyze the unknown solution. For analysis of the unknown solution, we ask that you calibrate your system using the set of solution standards provided. Quantitation should be accomplished by comparing the response from the unknown with that from the calibration solutions. No extractions, additions of internal standards, or solvent exchanges should be required for labs using reversed-phase systems. The assigned values for the target analytes in calibration solutions to be used for analysis of the unknown solution follows:

(μg/mL)

	RETINOL	α-TOCOPHEROL	TRANS β-CAROTENE
Solution 10	0.195	2.22	0.135
Solution 11	0.977	11.1	0.945
Solution 12	1.95	22.2	1.62

These solutions should be allowed to equilibrate at room temperature for at least 4 hours, but no more than 24 hours prior to use.

Results from the analysis of **RR-XVIII samples are due by March 5, 1990.**

Sincerely,

Willie E. May, Ph.D.
Chief
Organic Analytical Research Division
Center for Analytical Chemistry

cc: Robert Schaffer
Winfred Malone

Table 1. Round Robin XVII Results

Retinol						Retinol % Bias from Grand Average				
Lab #	Serum # 115	Serum # 116	Serum # 117	Serum # 118	Serum # 119	Serum # 115	Serum # 116	Serum # 117	Serum # 118	Serum # 119
	0.352	0.964	0.646	0.492	0.809	-1.3	-2.5	-1.5	-5.0	-9.5
	0.360	0.999	0.677	0.501	0.917	1.0	1.1	3.2	-3.3	2.6
	0.374	1.111	0.675	0.573	1.053	4.9	12.4	2.9	10.6	17.8
	0.334	1.193	0.643	0.515	1.113	-6.3	20.7	-2.0	-0.6	24.5
	0.380	1.050	0.550	0.650		6.6	6.2	-16.2	25.4	
	0.327	0.949	0.623	0.486	0.850	-8.3	-4.0	-5.0	-6.2	-4.9
	0.343	0.919	0.618	0.488	0.855	-3.8	-7.0	-5.8	-5.8	-4.4
	0.376	1.029	0.710	0.514	0.868	5.5	4.1	8.2	-0.8	-2.9
	0.350	0.971	0.680	0.560	0.860	-1.8	-1.7	3.7	8.1	-3.8
	0.393	1.157	0.702	0.560	0.999	10.3	17.1	7.0	8.1	11.7
	0.296	0.919	0.663	0.491	0.848	-17.0	-7.0	1.1	-5.2	-5.1
	0.315	0.936	0.613	0.469	0.858	-11.6	-5.3	-6.5	-9.5	-4.0
	0.380	0.920	0.650	0.530	0.860	6.6	-6.9	-0.9	2.3	-3.8
	0.387	1.002	0.621	0.543	0.914	8.6	1.4	-5.3	4.8	2.2
	0.413	1.141	0.793	0.585	0.936	15.9	15.5	20.9	12.9	4.7
	0.325	0.940	0.682	0.473	0.742	-8.8	-4.9	4.0	-8.7	-17.0
	0.333	0.964	0.641	0.497	0.889	-6.6	-2.5	-2.3	-4.1	-0.6
	0.347	0.908	0.596	0.486	0.869	-2.7	-8.1	-9.1	-6.2	-2.8
	*0.845	*1.874	*1.379	*0.875	*1.745	137.1	89.6	110.2	68.9	95.2
	0.320	0.950	0.610	0.490	0.820	-10.2	-3.9	-7.0	-5.4	-8.3
	0.280	0.930	0.660	0.450	0.820	-21.4	-5.9	0.6	-13.2	-8.3
	0.430	0.950	0.710	0.520	0.890	20.6	-3.9	8.2	0.4	-0.4
	0.390	0.750	0.580	0.380	0.780	9.4	-24.1	-11.6	-26.7	-12.8
	0.350	1.100	0.660	0.640	1.080	-1.8	11.3	0.6	23.5	20.8
	0.400	0.966	0.740	0.543	0.932	12.2	-2.3	12.8	4.8	4.3
	0.340	0.997	0.671	0.497	0.911	-4.6	0.9	2.3	-4.1	1.9
	*0.740	1.070	0.665	0.490	0.840	107.6	8.3	1.4	-5.4	-6.0
	0.322	1.128	0.718	0.535	0.971	-9.7	14.1	9.5	3.2	8.6
	0.530	*1.920	0.830	0.650	0.910	48.7	94.3	26.5	25.4	1.8
	0.375	1.021	0.676	0.556	0.931	5.2	3.3	3.1	7.3	4.1
AVG	0.356	0.988	0.656	0.518	0.894					
SD	0.037	0.098	0.053	0.059	0.092					
RSD	10.4	9.9	8.1	11.3	10.3					

* Value Removed

NL = New Laboratory Values not included in the Average.

4/87 Value	0.359	1.027	0.648	0.528	0.930
% Bias	0.7	3.9	-1.2	1.9	4.0
7/87 Value		1.049		0.540	
% Bias		6.1		4.2	
9/88 Value	0.355		0.633		0.905
% Bias	-0.4		-3.5		1.2

Table 2. Round Robin XVII Results

Alpha-Tocopherol						Alpha Tocopherol % Bias from Grand Average				
Lab#	Serum # 115	Serum # 116	Serum # 117	Serum # 118	Serum # 119	Serum # 115	Serum # 116	Serum # 117	Serum # 118	Serum # 119
	11.65	7.18	5.09	9.88	8.84	-0.2	-5.8	-4.1	-3.8	-6.4
	11.43	7.37	5.07	9.92	9.06	-2.1	-3.3	-4.4	-3.4	-4.0
	12.84	8.45	4.70	12.60	*5.47	10.0	10.9	-11.4	22.7	-42.1
	12.06	8.21	5.54	10.71	9.64	3.3	7.7	4.4	4.3	2.1
	*4.80	*4.80	4.60	*4.60	*0.12	-58.9	-37.0	-13.3	-55.2	-98.7
	12.11	7.65	5.44	10.13	9.41	3.8	0.4	2.6	-1.3	-0.3
	11.92	7.56	5.38	10.21	9.54	2.1	-0.8	1.4	-0.5	1.1
	12.20	7.50	5.10	9.40	8.80	4.5	-1.6	-3.9	-8.4	-6.8
	11.10	7.73	5.23	9.72	8.73	-4.9	1.4	-1.4	-5.3	-7.5
	11.92	8.61	5.27	10.84	10.00	2.1	13.0	-0.6	5.6	5.9
	11.60	7.01	5.97	10.51	11.88	-0.6	-8.0	12.5	2.4	25.8
	12.53	7.95	5.56	10.72	9.82	7.4	4.3	4.8	4.4	4.0
	12.06	7.92	5.54	10.01	9.45	3.3	3.9	4.4	-2.5	0.1
	12.04	7.43	5.75	10.45	9.36	3.2	-2.5	8.4	1.8	-0.9
	10.04	8.11	5.61	9.75	9.28	-14.0	6.4	5.8	-5.0	-1.7
	12.52	7.65	5.61	10.52	9.70	7.3	0.4	5.8	2.5	2.7
	11.80	7.60	5.50	10.30	9.80	1.1	-0.3	3.7	0.3	3.8
	11.02	6.72	4.64	8.89	8.91	-5.6	-11.8	-12.5	-13.4	-5.6
	11.28	7.07	5.19	10.00	8.52	-3.3	-7.2	-2.2	-2.6	-9.8
	9.60	7.10	5.30	10.50	9.20	-17.7	-6.8	-0.1	2.3	-2.6
	12.40	8.20	5.60	11.00	9.90	6.3	7.6	5.6	7.1	4.9
	10.73	7.16	5.94	9.18	8.19	-8.0	-6.1	12.0	-10.6	-13.2
	13.50	8.32	5.84	11.30	9.92	15.7	9.2	10.1	10.1	5.1
	12.30	7.95	5.71	10.86	10.03	5.4	4.3	7.6	5.8	6.2
AVG	11.67	7.62	5.30	10.27	9.44					
SD	0.81	0.50	0.37	0.75	0.74					
RSD	6.9	6.6	6.9	7.3	7.8					

* Value Removed
NL = New Laboratory Values not included in the Average.

4/87 Value	13.00	8.53	5.83	11.34	10.62
% Bias	11.4	11.9	9.9	10.4	12.5
7/87 Value		8.21		11.09	
% Bias		7.7		8.0	
9/88 Value	11.74		5.39		9.26
% Bias	0.6		1.6		-1.9

Table 3. Round Robin XVII Results

Total Beta-Carotene

Lab#	Serum # 115	Serum # 116	Serum # 117	Serum # 118	Serum # 119
	0.169	1.089	0.067	0.473	1.649
	0.161	1.022	0.069	0.441	1.534
	0.175	1.154	0.069	0.509	1.682
	0.181	1.005	0.073	0.480	1.642
	0.193	1.079	0.090	0.491	1.526
	0.226	1.090	*0.158	0.569	1.920
	0.197	1.048	0.089	0.446	1.666
	0.200	1.080	0.090	0.520	1.600
	0.177	1.120	0.069	0.517	1.690
	0.220	1.261	0.100	0.540	2.084
	0.178	1.049	0.089	0.466	1.604
	0.175	1.082	0.073	0.487	1.723
	0.190	1.020	0.060	0.470	1.490
	*0.500	1.160	*0.440	*0.730	1.600
	*0.340	*0.760	*0.290	0.420	1.140
	0.194	1.030	0.089	0.559	1.660
	0.222	0.988	*0.199	*0.659	1.729
	0.185	1.461	0.083	0.543	1.745
AVG	0.188	1.085	0.078	0.493	1.678
SD	0.019	0.067	0.013	0.037	0.163
RSD	10.2	6.2	16.2	7.5	9.7

Total Beta-Carotene % Bias from Grand Average

Lab#	Serum # 115	Serum # 116	Serum # 117	Serum # 118	Serum # 119
	-10.0	0.4	-14.3	-4.1	-1.7
	-14.3	-5.8	-11.8	-10.5	-8.6
	-6.8	6.4	-11.8	3.2	0.3
	-3.6	-7.3	-6.6	-2.6	-2.1
	2.7	-0.5	15.1	-0.4	-9.0
	20.3	0.5	102.1	15.4	14.4
	4.9	-3.4	13.8	-9.5	-0.7
	6.5	-0.4	15.1	5.5	-4.6
	-5.8	3.3	-11.8	4.9	0.7
	17.1	16.3	27.9	9.5	24.2
	-5.2	-3.3	14.2	-5.5	-4.4
	-6.8	-0.2	-6.6	-1.2	2.7
	1.1	-6.0	-23.3	-4.7	-11.2
	166.2	7.0	462.7	48.1	-4.6
	81.0	-29.9	270.9	-14.8	-32.0
	3.3	-5.0	13.8	13.4	-1.1
	18.2	-8.9	154.5	33.7	3.1
	-1.5	34.7	6.1	10.1	4.0

* Value Removed
NL = New Laboratory Values not included in the Average.

4/87 Value	0.186	1.152	0.080	0.487	1.789
% Bias	-1.0	6.2	2.8	-1.2	6.6
7/87 Value		1.144		0.502	
% Bias		5.4		1.7	
9/88 Value	0.177		0.088		1.687
% Bias	-5.8		12.5		0.6

Table 4. Round Robin XVII Results

Trans Beta-Carotene

Lab#	Serum # 115	Serum # 116	Serum # 117	Serum # 118	Serum # 119
	*0.320	1.040	*0.970	0.250	
	0.179	0.971	0.050	0.497	1.620
	0.165	1.012	0.070	0.455	1.606
	0.102	0.773	0.057	0.448	1.357
	0.173	1.071	0.079	0.5	1.627
AVG	0.149	0.949	0.059	0.413	1.528
SD	0.041	0.121	0.010	0.110	0.148
RSD	27.6	12.7	17.2	26.8	9.7

Trans Beta-Carotene % Bias from Grand Average

Serum # 115	Serum # 116	Serum # 117	Serum # 118	Serum # 119
115.2	9.6	1544.1	-39.4	
20.4	2.3	-15.3	20.5	6.0
11.0	6.6	18.6	10.3	5.1
-31.4	-18.5	-3.4	8.6	-11.2
16.4	12.9	33.9	21.2	6.5

* Value Removed

NL = New Laboratory Values not included in the Average.

Appendix I. Updated "All-Lab Report" for RR17

The following three pages are an updated version of an "All-Lab" report for RR17. This report has three parts:

- Page 1 lists results for all analytes that were reported at least twice, counting both participants and NIST analysts.
- Page 2 lists values for all analytes reported by only once. This page also provides a legend for page 1.
- Page 3 summarizes each participants' performance for total retinol, α-tocopherol, and total β-carotene. These summaries are compatible with the percent bias evaluation advice given in the RR17 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 "AVG" used in the original and detailed in Appendix H.

To ensure confidentiality, the laboratory identifiers used in this "All-Lab Report" have been altered from those used in RR17. 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 XVII Laboratory Results

	Total Retinol						α-Tocopherol						Total β-Carotene						trans-β-Carotene					
Lab	115	116	117	118	119	114	115	116	117	118	119	114	115	116	117	118	119	114	115	116	117	118	119	114
FSV-BA	0.375	1.021	0.676	0.556	0.931	0.498	12.30	7.95	5.71	10.86	10.03	8.34	0.185	1.461	0.083	0.543	1.745	0.619	0.173	1.071	0.079	0.500	1.627	0.222
FSV-BD	0.376	1.029	0.710	0.514	0.868	0.521	12.20	7.50	5.10	9.40	8.80	8.40												
FSV-BE	0.315	0.936	0.613	0.469	0.858	0.686	12.06	7.92	5.54	10.01	9.45	13.03												
FSV-BF	0.393	1.157	0.702	0.560	0.999	0.674	11.92	8.61	5.27	10.84	10.00	9.30	0.197	1.048	0.089	0.446	1.666	0.744						
FSV-BG	0.374	1.111	0.675	0.573	1.053	0.479	12.84	8.45	4.70	12.60	5.47	8.80	0.175	1.154	0.069	0.509	1.682	0.589						
FSV-BH	0.347	0.908	0.596	0.486	0.869	0.533	11.80	7.60	5.50	10.30	9.80	9.80	0.175	1.082	0.073	0.487	1.723	≥0.409	0.165	1.012	0.070	0.455	1.606	0.409
FSV-BI	0.360	0.999	0.677	0.501	0.917	0.605	11.43	7.37	5.07	9.92	9.06	11.11	0.161	1.022	0.069	0.441	1.534	≥0.882						0.882
FSV-BL	0.400	0.966	0.740	0.543	0.932	0.598	10.73	7.16	5.94	9.18	8.19	9.57												
FSV-BY	0.352	0.964	0.646	0.492	0.809		11.65	7.18	5.09	9.88	8.84		0.169	1.089	0.067	0.473	1.649							
FSV-BZ	0.845	1.874	1.379	0.875	1.745	0.475	11.02	6.72	4.64	8.89	8.91	4.33												
FSV-CA	0.327	0.949	0.623	0.486	0.850	0.530	12.11	7.65	5.44	10.13	9.41	9.46												
FSV-CJ	0.334	1.193	0.643	0.515	1.113	0.472	12.06	8.21	5.54	10.71	9.64	11.10	0.181	1.005	0.073	0.480	1.642	0.799						
FSV-CM	0.350	1.100	0.660	0.640	1.080	0.420	12.40	8.20	5.60	11.00	9.90	10.80												
FSV-CN	0.350	0.971	0.680	0.560	0.860	0.569	11.10	7.73	5.23	9.72	8.73	3.44	0.226	1.090	0.158	0.569	1.920	1.800						
FSV-CO	0.343	0.919	0.618	0.488	0.855	0.269	11.92	7.56	5.38	10.21	9.54	4.39	0.193	1.079	0.090	0.491	1.526	0.258						
FSV-CQ	0.296	0.919	0.663	0.491	0.848	0.684	11.60	7.01	5.97	10.51	11.88	13.21	≥0.179	≥0.971	≥0.050	≥0.497	≥1.620	≥0.793	0.179	0.971	0.050	0.497	1.620	0.793
FSV-CS	0.530	1.920	0.830	0.650	0.910	1.000																		
FSV-CY	0.320	0.950	0.610	0.490	0.820	0.500	11.28	7.07	5.19	10.00	8.52	9.47	0.190	1.020	0.060	0.470	1.490							
FSV-DC	0.387	1.002	0.621	0.543	0.914	0.600							0.177	1.120	0.069	0.517	1.690							
FSV-DE							12.53	7.95	5.56	10.72	9.82	11.80												
FSV-DG	0.380	0.920	0.650	0.530	0.860	0.400	12.04	7.43	5.75	10.45	9.36	9.82	0.200	1.080	0.090	0.520	1.600	0.810						0.400
FSV-DH	0.340	0.997	0.671	0.497	0.911	0.472	13.50	8.32	5.84	11.30	9.92	9.33	0.194	1.030	0.089	0.559	1.660	≥0.675						0.675
FSV-DN	0.292	1.020	0.656	0.454	0.752	0.787	11.80	8.20	5.30		9.60	9.70	0.203	1.190	0.100	0.522	1.630	1.200						
FSV-DO	0.380	1.050	0.550	0.650		0.750	4.80	4.80	4.60	4.60		0.12	≥0.320	≥1.040	≥0.970	≥0.250		≥1.000	0.320	1.040	0.970	0.250		1.000
FSV-DT	0.323	0.905	0.609	0.480	0.799	0.579	13.06	8.51	5.91	11.15	10.25	10.56												
FSV-DY	0.430	0.950	0.710	0.520	0.890	0.500							0.500	1.160	0.440	0.730	1.600	0.870						
FSV-DYa	0.390	0.750	0.580	0.380	0.780								0.340	0.760	0.290	0.420	1.140							
FSV-DZ	0.325	0.940	0.682	0.473	0.742	0.512	10.04	8.11	5.61	9.75	9.28	8.45												
FSV-ED	0.333	0.964	0.641	0.497	0.889	0.639	12.52	7.65	5.61	10.52	9.70	12.23	0.178	1.049	0.089	0.466	1.604							
FSV-EF	0.280	0.930	0.660	0.450	0.820	0.520	9.60	7.10	5.30	10.50	9.20	8.60												
FSV-EO	0.413	1.141	0.793	0.585	0.936	0.093							0.220	1.261	0.100	0.540	2.084	0.144						
FSV-FA	0.322	1.128	0.718	0.535	0.971	0.510							0.222	0.988	0.199	0.659	1.729	1.111	0.102	0.773	0.057	0.448	1.357	1.111
FSV-FV	0.740	1.070	0.665	0.490	0.840	0.510																		
n	32	32	32	32	31	30	26	26	26	25	25	25	19	19	19	19	19	11	5	5	5	5	4	8
Min	0.280	0.750	0.550	0.380	0.742	0.093	4.80	4.80	4.60	4.60	5.47	0.12	0.161	0.760	0.060	0.420	1.140	0.144	0.102	0.773	0.050	0.250	1.357	0.222
Mean	0.385	1.052	0.686	0.530	0.917	0.546	11.55	7.61	5.40	10.13	9.33	9.01	0.215	1.089	0.121	0.518	1.648	0.813	0.188	0.973	0.245	0.430	1.553	0.686
Max	0.845	1.920	1.379	0.875	1.745	1.000	13.50	8.61	5.97	12.60	11.88	13.21	0.500	1.461	0.440	0.730	2.084	1.800	0.320	1.071	0.970	0.500	1.627	1.111
SD	0.118	0.240	0.139	0.086	0.177	0.159	1.62	0.77	0.38	1.38	1.08	3.05	0.079	0.135	0.096	0.075	0.184	0.454	0.080	0.118	0.405	0.103	0.131	0.317
CV	31	23	20	16	19	29	14	10	7	14	12	34	37	12	79	14	11	56	43	12	165	24	8	46
NISTa	0.363	1.060	0.659	0.495	0.976	0.538	11.98	7.51	5.30	10.09	9.22	8.37	0.153	0.972	0.074	0.395	1.360	0.837	0.141	0.941	0.071	0.337	1.325	0.289
Median	0.351	0.984	0.662	0.508	0.869	0.521	11.92	7.65	5.47	10.30	9.45	9.47	0.193	1.080	0.089	0.509	1.649	0.799	0.173	1.012	0.070	0.455		0.734
eSD	0.043	0.087	0.059	0.046	0.073	0.079	0.72	0.71	0.36	0.62	0.67	1.62	0.024	0.086	0.024	0.053	0.082	0.311	0.012	0.061	0.019	0.062		0.438
eCV	12.2	8.9	8.9	9.1	8.4	15.2	6.0	9.3	6.5	6.0	7.1	17.1	12.3	8.0	26.7	10.5	5.0	39.0	6.9	6.0	27.5	13.7		59.7

Round Robin XVII Laboratory Results

Table Legend

Symbol	Interpretation
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*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
≥x	Concentration greater than or equal to x

Round Robin XVII Laboratory Results

%Bias Summary

Lab	TR	aT	bC
FSV-BA	6±3	5±1	7±17
FSV-BD	4±3	-4±5	
FSV-BE	-6±3	1±2	
FSV-BF	12±4	4±6	-2±6
FSV-BG	11±7	-3±25	-5±12
FSV-BH	-5±4	1±2	-5±9
FSV-BI	2±2	-5±2	-13±7
FSV-BL	8±6	-6±9	
FSV-BY	-3±3	-5±2	-9±10
FSV-BZ	103±25	-11±4	
FSV-CA	-5±2	0±1	
FSV-CJ	9±15	3±3	-7±6
FSV-CM	12±13	5±2	
FSV-CN	2±5	-5±3	27±35
FSV-CO	-4±2	-1±1	-2±4
FSV-CQ	-6±6	5±13	-13±18
FSV-CS	41±34		
FSV-CY	-6±2	-6±3	-14±13
FSV-DC	4±6		-5±11
FSV-DE		4±1	
FSV-DG	1±6	1±3	1±2
FSV-DH	0±3	9±3	1±5
FSV-DN	-8±9	1±4	6±6
FSV-DO	7±18	-42±20	250±495
FSV-DT	-7±1	9±1	
FSV-DY	6±10		120±166
FSV-DYa	-12±15		45±111
FSV-DZ	-6±6	-3±8	
FSV-ED	-2±3	2±2	-4±4
FSV-EF	-9±8	-6±8	
FSV-EO	15±5		15±7
FSV-FA	6±9		33±53
FSV-FV	23±50		
NISTa	4±6	-2±1	-18±5

Label	Definition
Lab	Participant code
TR	Total Retinol
aT	a-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 "Avg" 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-based reference value.