NISTIR 7880-36

NIST Micronutrients Measurement Quality Assurance Program Winter, Spring, and Fall 1988 Comparability Studies

Results for Round Robins XII, XIII, and XIV Fat-Soluble Vitamins and Carotenoids in Human Serum

Neal E. Craft (Former Employee)
David L. Duewer
Margaret C. Kline
Willie E. May
Jeanice B. Thomas

This publication is available free of charge from
http://dx.doi.org/10.6028/NIST.IR.7880-36

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

NISTIR 7880-36

NIST Micronutrients Measurement Quality Assurance Program Winter, Spring, and Fall 1988 Comparability Studies

Results for Round Robins XII, XIII, and XIV Fat-Soluble Vitamins and Carotenoids in Human Serum

Neal E. Craft (Former Employee)
David L. Duewer
Jeanice B. Thomas
Chemical Sciences Division,
Materials Measurement Laboratory

Margaret C. Kline
Biomolecular Measurement Division,
Materials Measurement Laboratory

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

This publication is available free of charge from
http://dx.doi.org/10.6028/NIST.IR.7880-36

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

(This page intentionally blank)

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 1988 MMQAP measurement comparability improvement studies: 1) Round Robin XII Fat-Soluble Vitamins and Carotenoids in Human Serum, 2) Round Robin XIII Fat-Soluble Vitamins and Carotenoids in Human Serum, and 3) Round Robin XIV Fat-Soluble Vitamins and Carotenoids in Human Serum. The materials for Round Robin XII were shipped to participants in January 1988; participants were requested to provide their measurement results by March 2, 1988. The materials for Round Robin XIII were shipped to participants in May 1988; participants were requested to provide their measurement results by July 1, 1988. The materials for Round Robin XIV were shipped to participants in August 1988; participants were requested to provide their measurement results by October 1, 1988.

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 XII: Fat-Soluble Vitamins and Carotenoids in Human Serum

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin XII comparability study (hereafter referred to as RR12) received five 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 1988. 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 RR12 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 RR12 in a more accessible format.

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

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin XIII comparability study (hereafter referred to as RR13) received five 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 1988. 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 RR13 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 RR13 in a more accessible format.

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

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin XIV comparability study (hereafter referred to as RR14) received five lyophilized human sera. 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 1988. 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 RR14 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 RR14 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 RR12

Two items were attached to each package shipped to an RR12 participant:

- **Cover letter**. The original letter has been lost. It would have described the five lyophilized sample materials (sera 77 to 81) distributed for the study and stated that results were due in March 2, 1988.

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

REPORT ON NBS/NCI SAMPLES FROM LABORATORY # _____

DATE OF ANALYSIS _____

RESULTS IN mg/L

SAMPLE #	ANALYTE	Result	
SERUM 77			
	RETINOL		
VIAL # ____		All Trans	Total
	B-CAROTENE		
	A-TOCOPHEROL		
SERUM 78			
	RETINOL		
VIAL # ____		All Trans	Total
	B-CAROTENE		
	A-TOCOPHEROL		
SERUM 79			
	RETINOL		
VIAL # ____		All Trans	Total
	B-CAROTENE		
	A-TOCOPHEROL		
SERUM 80			
	RETINOL		
VIAL # ____		All Trans	Total
	B-CAROTENE		
	A-TOCOPHEROL		
SERUM 81			
	RETINOL		
VIAL # ____		All Trans	Total
	B-CAROTENE		
	A-TOCOPHEROL		

RECONSTITUTE THE SERUM SAMPLE WITH 1.0 mL WATER.

Appendix B. Final Report for RR12

The following sixteen pages are the final report for RR12 as provided to participants. The original report consisted of:

- An individualized cover letter and discussion. The pages reproduced here are for participant FSV-BA.
- Four tables that list the reported results and several summary values for retinol, α-tocopherol, total β-carotene, and *trans*-β-carotene. 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 RR12 results where the original codes have been altered to ensure confidentiality. Appendix C also provides more relevant summary statistics. (The labels "NBS A" and "NBS B" in these tables refer to NIST analysts NISTa and NISTd.)
- For all participants, graphical presentations of % bias relative to the assigned value of all participants for retinol, α-tocopherol, total β-carotene, and *trans*-β-carotene. The laboratory codes originally below the horizontal axis have been deleted without replacement.
- For all participants who had participated in Round Robin IX (May 1987), graphical calibration-style presentations of their results for Sera 62 to 66 relative to the assigned values for retinol, α-tocopherol, and total β-carotene. The pages reproduced here are for participant FSV-BA.

UNITED STATES DEPARTMENT OF COMMERCE
National Bureau of Standards
Gaithersburg, Maryland 20899

April 11, 1988

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 the fat-soluble vitamins component of RR-XII. Individual laboratory performance is judged against an assigned value derived from the laboratory grand average (33%) and two separate NBS measurement procedures (67%). Based on results from past round-robins and our assessment of the state of the art for fat-soluble vitamin measurement capabilities, 0-15% deviation from the assigned value is deemed acceptable performance; 16-30% represents marginal performance; >30% represents unacceptable performance.

Results from this round-robin, however, indicate that laboratory performance has begun to improve, and that the performance guidelines cited above might be a bit conservative and not truly reflect the measurement capabilities that exist in many laboratories. For example, of the thirty-one labs that measured retinol in RR-XII, twenty reported results that were classified as being acceptable (0.15% deviation from the assigned value), nine of the thirty-one laboratories reported values for all five samples that varied from the assigned values by ≤10%. The performance of five laboratories was deemed to be unacceptable.

For the twenty eight labs that measured alpha-tocopherol, fifteen reported results that were deemed acceptable, with eleven reporting results for all samples that varied from the assigned value by ≤10%. The performance of only one laboratory was classified as being unacceptable.

For the twenty labs that provided data for "total beta-carotene", ten reported results that were deemed acceptable. Four of the ten labs provided data that varied ≤10% for all five samples. For the five labs that reported data for all-trans beta carotene (the agent supposedly under investigation), one lab reported results that were deemed to be of unacceptable quality; the remaining four reported results that were classified as being marginal.

Your laboratory's overall performance in this round-robin was:

	ACCEPTABLE	MARGINAL	UNACCEPTABLE
Retinol	X		
α-Tocopherol		X	
All-trans β-carotene		X	
Total β-carotene			X

Problems with the measurement and interlaboratory comparison of measurements of serum beta-carotene continue. We feel that this problem is due mainly to the fact that different reversed-phase columns provide different selectivity for carotenoids. Most commercial C-18 olumns ("monomerically bound") provide separations that combine the all-trans and cis isomers of beta-carotene into one peak. Data obtained from such columns should be reported as "total beta carotene". "Polymerically bound" C-18 columns allow separation of the all-trans from the cis isomers, but the resolution of the cis isomers from other carotenoids is not consistent from manufacturer to manufacturer, or lot to lot for the same manufacturer. It is therefore possible to obtain a value for all trans beta-carotene, but obtaining values for total beta-carotene (by addition of responses for the all trans and the individual cis isomers) is complicated by the elution of other carotenoid compounds in the same region of the chromatogram. Research is in progress at NBS to develop test mixtures that will allow labs to classify their columns, and gain better knowledge of what they are measuring and reporting. Additional NBS investigations are being directed toward the development of a phase specifically tailored for carotenoid separations. We expect to begin providing the test mixtures by this summer and carotenoid columns by early next year.

As promised at last years workshop, we are now ready to distribute "Control Materials" with assigned concentration values for retinol, alpha-tocopherol, and total beta-carotene. These materials, and instructions for their use are being shipped to you under separate cover.

Sincerely,

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

Enclosure

cc R Schaffer
H Pierson
R Paule

RR XII Retinol Results, mg/L.

Lab #	Serum 79	Serum 78	Serum 81	Serum 80	Serum 77
	0.309	0.305	0.257	0.520	0.801
	0.360	0.400	0.460	1.000	1.100
	0.388	0.406	0.485	1.026	1.174
	0.336	0.362	0.419	0.973	1.030
	0.403	0.435	0.496	1.198	1.194
	*0.750	0.510	0.510	1.440	*1.650
	0.391	0.401	0.484	1.011	1.059
	0.359	0.374	0.424	0.958	0.999
	0.325	0.409	0.370	0.851	1.029
	0.270	0.280	0.358	0.800	0.930
	0.370	0.450	0.443	0.892	1.073
	0.373	0.404	0.490	0.990	1.101
	0.350	0.350	0.440	0.850	0.930
	0.340	0.380	0.380	0.820	1.040
	0.438	0.355	0.433	0.957	1.098
	0.367	0.417	0.437	1.026	1.068
	0.443	0.494	0.564	1.140	1.253
	0.380	0.420	0.460	1.030	1.150
	*0.540	*0.600	*0.710	1.500	1.200
	0.397	0.432	0.468	1.063	1.142
	0.380	0.370	0.405	0.910	1.130
	0.398	0.426	0.488	1.119	1.182
	0.353	0.392	0.438	1.030	1.090
	0.320	0.360	0.460	0.980	1.030
	0.456	0.430	0.522	0.958	0.663
	0.371	0.413	0.464	1.030	1.120
	0.331	0.362	0.425	0.854	0.993
	0.270	0.325	0.349	0.747	0.869
	0.350	0.370	0.420	0.880	0.960
	0.280	0.320	0.370	0.310	*0.280
	0.368	0.402	0.462	0.960	1.095
Average	0.361	0.392	0.439	0.962	1.047
SD**	0.046	0.050	0.061	0.218	0.125
% SD**	12.8	12.9	13.8	22.7	12.0
Fr Reject	2/31	1/31	1/31	0/31	2/31

* = Rejected Value
** = Standard deviation of a single measurement.

NBS A	0.333	0.352	0.413	0.881	1.010
NBS B	0.346	0.362	0.411	0.919	1.010

ASSIGNED VALUE = (NBS A + NBS B + Average)/3

ASSIGNED VALUE	0.347	0.369	0.421	0.921	1.022

RR XII Retinol % Bias from Assigned Value.

Lab #	Serum 79	Serum 78	Serum 81	Serum 80	Serum 77
	-10.9	-17.3	-39.0	-43.5	-21.6
	3.8	8.5	9.2	8.6	7.6
	11.9	10.1	15.2	11.4	14.9
	-3.1	-1.8	-0.5	5.7	0.8
	16.2	18.0	17.8	30.1	16.8
	116.3	38.4	21.1	56.4	61.4
	12.8	8.8	14.9	9.8	3.6
	3.5	1.5	0.7	4.1	-2.3
	-6.3	11.0	-12.1	-7.6	0.7
	-22.1	-24.0	-15.0	-13.1	-9.0
	6.7	22.1	5.2	-3.1	5.0
	7.6	9.6	16.4	7.5	7.7
	0.9	-5.0	4.5	-7.7	-9.0
	-1.9	3.1	-9.8	-10.9	1.7
	26.3	-3.7	2.8	3.9	7.4
	5.8	13.1	3.8	11.4	4.5
	27.8	34.0	33.9	23.8	22.6
	9.6	13.9	9.2	11.9	12.5
	55.7	62.8	68.6	62.9	17.4
	14.5	17.2	11.1	15.5	11.7
	9.6	0.4	-3.8	-1.2	10.5
	14.8	15.6	15.9	21.5	15.6
	1.8	6.3	4.0	11.9	6.6
	-7.7	-2.3	9.2	6.4	0.8
	31.5	16.7	24.0	4.1	-35.1
	7.0	12.0	10.2	11.9	9.6
	-4.5	-1.8	0.9	-7.2	-2.9
	-22.1	-11.8	-17.1	-18.9	-15.0
	0.9	0.4	-0.3	-4.4	-6.1
	-19.2	-13.2	-12.1	-66.3	-72.6
	6.1	9.1	9.7	4.3	7.1
NBS A	-4.0	-4.5	-1.9	-4.3	-1.2
NBS B	-0.2	-1.8	-2.4	-0.2	-1.2
ASSIGNED VALUE	0.347	0.369	0.421	0.921	1.022

% Bias= 100*(Lab value - Assigned Value)/Assigned Value

RR XII A-Tocopherol Results, mg/L.

Lab #	Serum 78	Serum 81	Serum 79	Serum 77	Serum 80
	5.250	5.700	5.490	8.750	9.730
	5.600	5.300	5.600	8.800	9.700
	6.590	6.630	6.560	10.000	11.090
	5.070	5.340	5.150	9.090	9.750
	5.090	6.120	5.940	9.570	10.670
	5.800	6.100	6.400	11.000	9.300
	5.200	5.900	5.400	9.500	10.400
	3.580	4.020	3.620	6.140	6.530
	5.080	5.000	5.180	7.750	8.540
	5.260	5.430	4.560	8.000	8.950
	5.800	6.300	6.100	9.600	10.700
	5.050	5.670	6.310	9.320	9.650
	4.700	5.440	5.470	7.400	9.230
	5.780	6.140	6.290	10.050	10.800
	5.550	6.050	5.750	9.025	10.330
	6.530	6.860	6.980	10.360	12.140
	6.900	7.600	7.400	12.100	12.900
	6.390	7.590	6.680	10.710	11.030
	5.230	5.415	5.755	9.585	10.595
	6.820	7.660	6.630	11.590	14.120
	6.503	6.601	7.014	6.170	10.533
	6.000	6.190	5.970	9.650	10.880
	5.350	5.750	5.650	8.850	9.950
	4.084	4.661	4.600	7.818	8.825
	4.600	5.000	4.900	7.700	10.200
	6.200	6.800	6.400	9.000	9.100
	6.420	7.020	6.550	11.070	11.280
	4.620	5.310	5.220	7.270	8.480
Average	5.537	5.986	5.842	9.138	10.193
SD**	0.828	0.894	0.849	1.494	1.457
% SD**	15.0	14.9	14.5	16.3	14.3
Fr Reject	0/28	0/28	0/28	0/28	0/28

* = Rejected Value
** = Standard deviation of a single measurement.

NBS A	5.292	5.408	5.568	9.208	10.143
NBS B	6.080	5.970	6.630	9.810	11.270

ASSIGNED VALUE = (NBS A + NBS B + Average)/3

ASSIGNED VALUE	5.636	5.788	6.013	9.385	10.535

RR XII A-Tocopherol % Bias from Assigned Value

Lab #	Serum 78	Serum 81	Serum 79	Serum 77	Serum 80
	-6.9	-1.5	-8.7	-6.8	-7.6
	-0.6	-8.4	-6.9	-6.2	-7.9
	16.9	14.5	9.1	6.5	5.3
	-10.0	-7.7	-14.4	-3.1	-7.5
	-9.7	5.7	-1.2	2.0	1.3
	2.9	5.4	6.4	17.2	-11.7
	-7.7	1.9	-10.2	1.2	-1.3
	-36.5	-30.5	-39.8	-34.6	-38.0
	-9.9	-13.6	-13.9	-17.4	-18.9
	-6.7	-6.2	-24.2	-14.8	-15.0
	2.9	8.8	1.4	2.3	1.6
	-10.4	-2.0	4.9	-0.7	-8.4
	-16.6	-6.0	-9.0	-21.2	-12.4
	2.5	6.1	4.6	7.1	2.5
	-1.5	4.5	-4.4	-3.8	-1.9
	15.9	18.5	16.1	10.4	15.2
	22.4	31.3	23.1	28.9	22.4
	13.4	31.1	11.1	14.1	4.7
	-7.2	-6.4	-4.3	2.1	0.6
	21.0	32.3	10.3	23.5	34.0
	15.4	14.0	16.6	-34.3	0.0
	6.4	6.9	-0.7	2.8	3.3
	-5.1	-0.7	-6.0	-5.7	-5.6
	-27.5	-19.5	-23.5	-16.7	-16.2
	-18.4	-13.6	-18.5	-18.0	-3.2
	10.0	17.5	6.4	-4.1	-13.6
	13.9	21.3	8.9	17.9	7.1
	-18.0	-8.3	-13.2	-22.5	-19.5
NBS A	-6.1	-6.6	-7.4	-1.9	-3.7
NBS B	7.9	3.1	10.3	4.5	7.0
ASSIGNED VALUE	5.636	5.788	6.013	9.385	10.535

% Bias= 100*(Lab value - Assigned Value)/Assigned Value.

RR XII Total B-Carotene Results, mg/L.

Lab #	Serum 78	Serum 81	Serum 80	Serum 77	Serum 79
	0.187	0.179	0.800	0.945	2.479
	0.160	0.160	0.740	0.840	2.200
	0.196	0.147	0.847	1.025	2.949
	0.202	0.173	0.735	0.857	2.316
	0.204	0.175	0.719	0.894	2.302
	0.206	0.216	0.845	1.052	2.611
	0.217	0.210	0.902	1.078	2.828
	0.231	0.341	1.128	0.977	2.040
	0.140	0.150	0.640	0.790	2.120
	0.234	0.257	0.782	0.940	2.550
	0.113	0.121	0.473	0.612	1.483
	0.210	0.230	0.980	1.100	2.830
	0.214	0.214	0.873	1.050	2.660
	0.204	0.204	0.868	1.066	2.459
	0.229	0.216	0.920	1.090	2.690
	0.245	0.248	1.134	1.295	3.341
	0.203	0.197	0.822	0.977	2.417
	0.220	0.230	0.947	1.118	2.825
	0.144	0.153	0.614	0.542	2.000
	0.271	0.278	1.135	1.593	*4.526
Average	0.201	0.205	0.845	0.992	2.479
SD**	0.038	0.052	0.173	0.224	0.416
% SD**	18.7	25.2	20.4	22.6	16.8
Fr Reject	0/20	0/20	0/20	0/20	1/20

* = Rejected Value
** = Standard deviation of a single measurement.

NBS A	0.233	0.238	0.850	1.080	2.620
NBS B	0.173	0.192	0.694	1.010	2.460

ASSIGNED VALUE = (NBS A + NBS B + Average)/3

ASSIGNED VALUE	0.202	0.212	0.796	1.027	2.520

RR XII Total B-Carotene % Bias from Assigned Value.

Lab #	Serum 78	Serum 81	Serum 80	Serum 77	Serum 79
	-7.7	-15.4	0.5	-8.0	-1.6
	-21.0	-24.4	-7.1	-18.2	-12.7
	-3.2	-30.5	6.4	-0.2	17.0
	-0.2	-18.3	-7.7	-16.6	-8.1
	0.7	-17.3	-9.7	-13.0	-8.6
	1.7	2.1	6.1	2.4	3.6
	7.2	-0.8	13.3	4.9	12.2
	14.1	61.1	41.6	-4.9	-19.0
	-30.9	-29.1	-19.6	-23.1	-15.9
	15.6	21.4	-1.8	-8.5	1.2
	-44.2	-42.8	-40.6	-40.4	-41.1
	3.7	8.7	23.1	7.1	12.3
	5.7	1.1	9.6	2.2	5.6
	0.7	-3.6	9.0	3.8	-2.4
	13.1	2.1	15.5	6.1	6.8
	20.7	16.9	42.3	26.1	32.6
	0.2	-6.9	3.2	-4.9	-4.1
	8.6	8.7	18.9	8.8	12.1
	-28.8	-27.9	-22.9	-47.2	-20.6
	33.8	31.4	42.5	55.1	79.6
NBS A	15.1	12.5	6.7	5.1	4.0
NBS B	-14.6	-9.3	-12.9	-1.7	-2.4
ASSIGNED VALUE	0.202	0.212	0.796	1.027	2.520

% Bias= 100*(Lab value - Assigned Value)/Assigned Value.

RR XII Trans-B-Carotene Results, mg/L.

Lab #	Serum 78	Serum 81	Serum 80	Serum 77	Serum 79
	0.257	0.307	1.328	1.294	4.034
	0.218	0.203	0.875	1.050	2.610
	0.227	0.227	1.066	1.217	3.147
	0.130	0.142	0.560	0.504	1.920
	0.150	0.150	0.550	0.780	2.020
Average	0.196	0.206	0.876	0.969	2.746
SD**	0.054	0.067	0.334	.3 6	0 873
% SD**	27.5	32.5	38.1	33.7	31.8
Fr Reject	0/5	0/5	0/5	0/5	0/5

* = Rejected Value
** = Standard deviation of a single measurement.

NBS A	0.177	0.208	0.732	0.969	2.342
NBS B	0.161	0.177	0.647	0.940	2.270

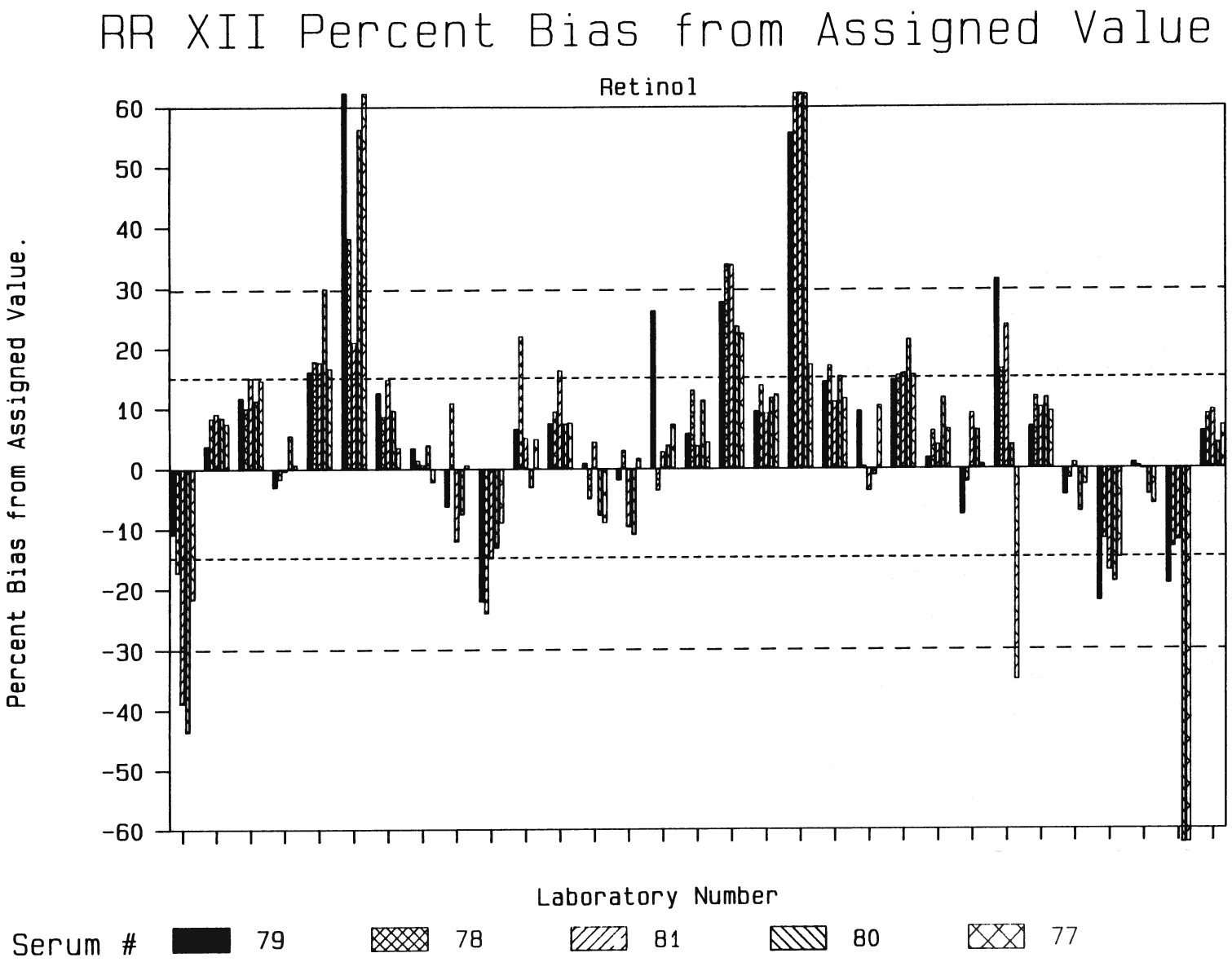
ASSIGNED VALUE = (NBS A + NBS B + Average)/3

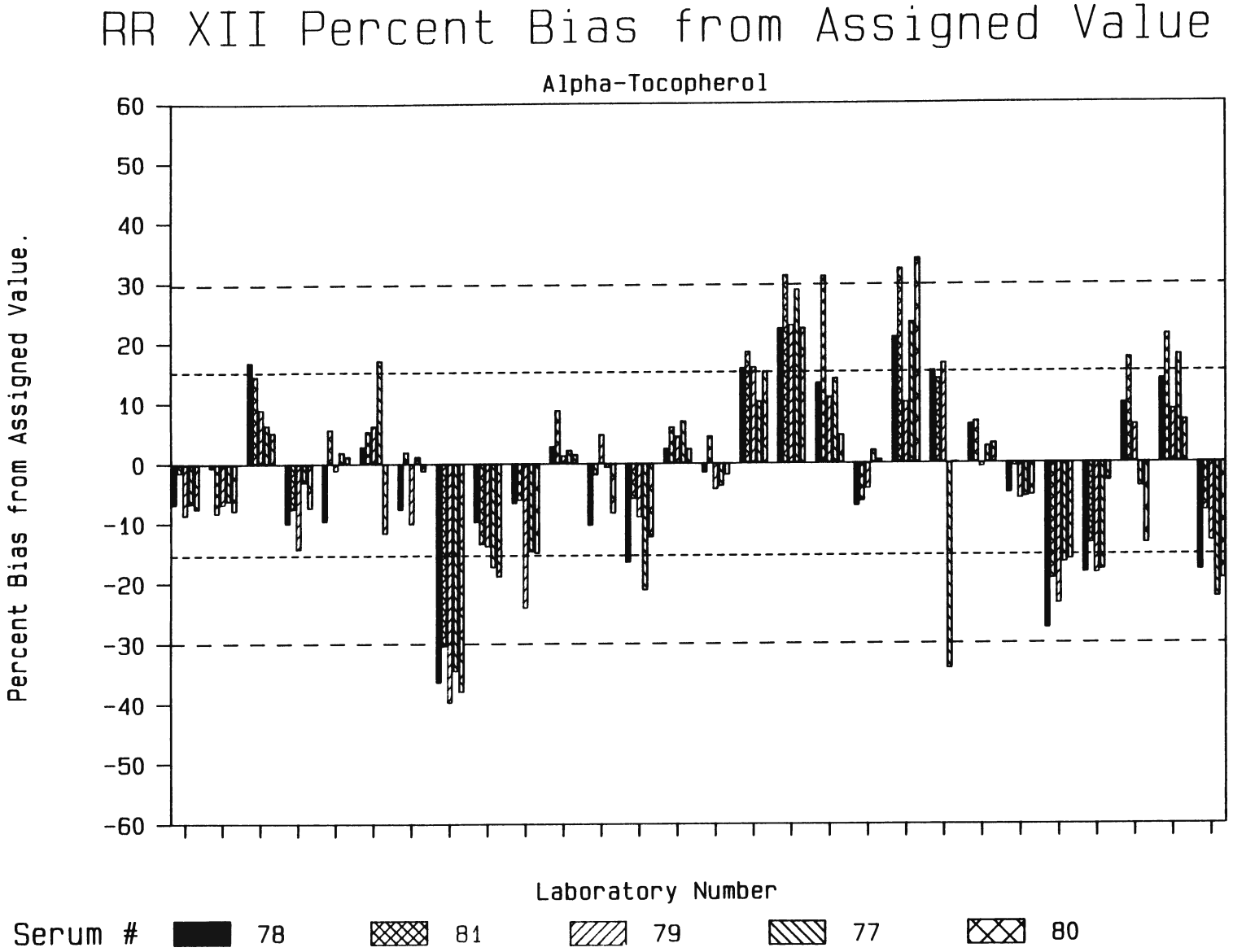
ASSIGNED VALUE	0.178	0.197	0.752	0.959	2.453

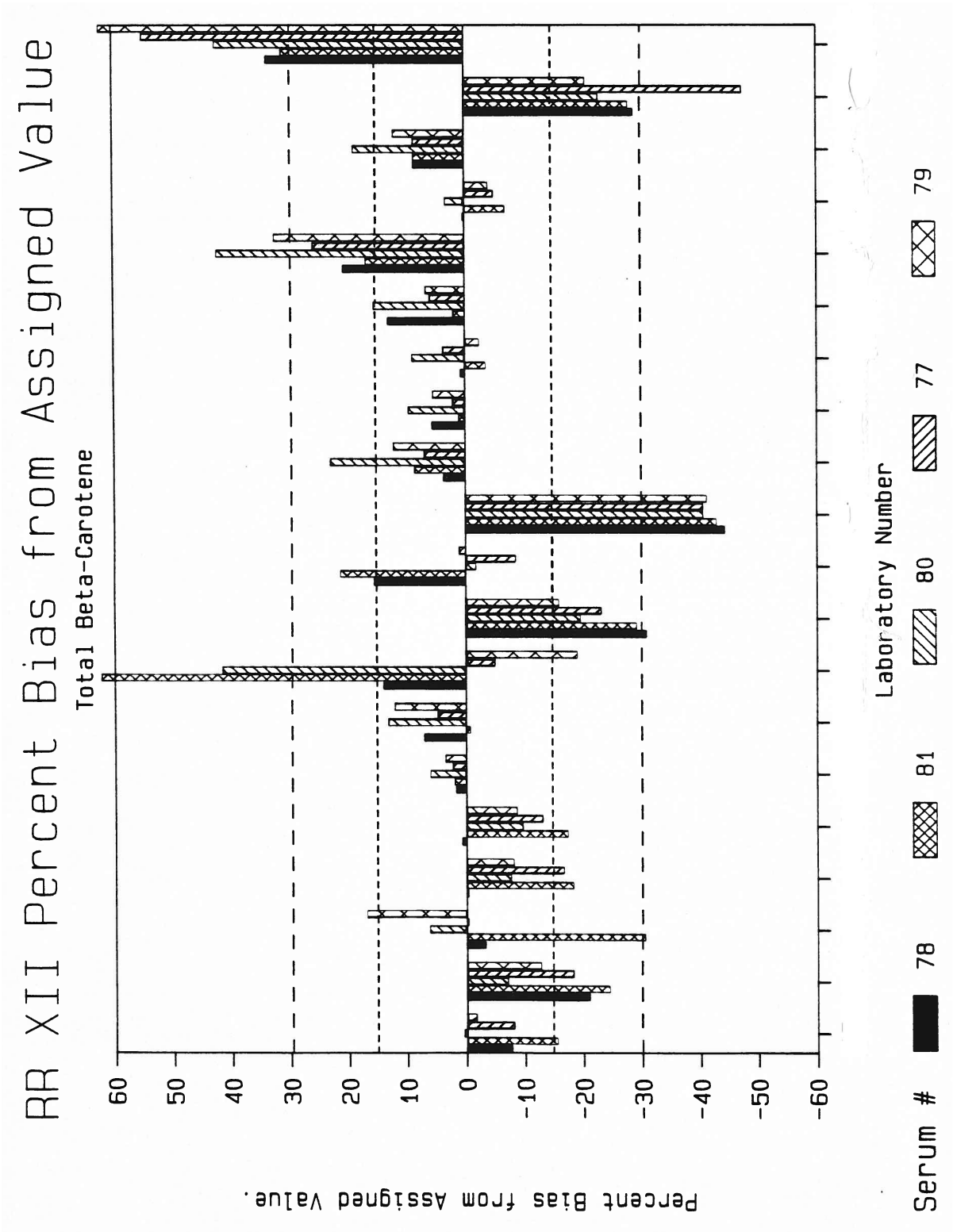
RR XII Trans-B-Carotene % Bias from Assigned Value.

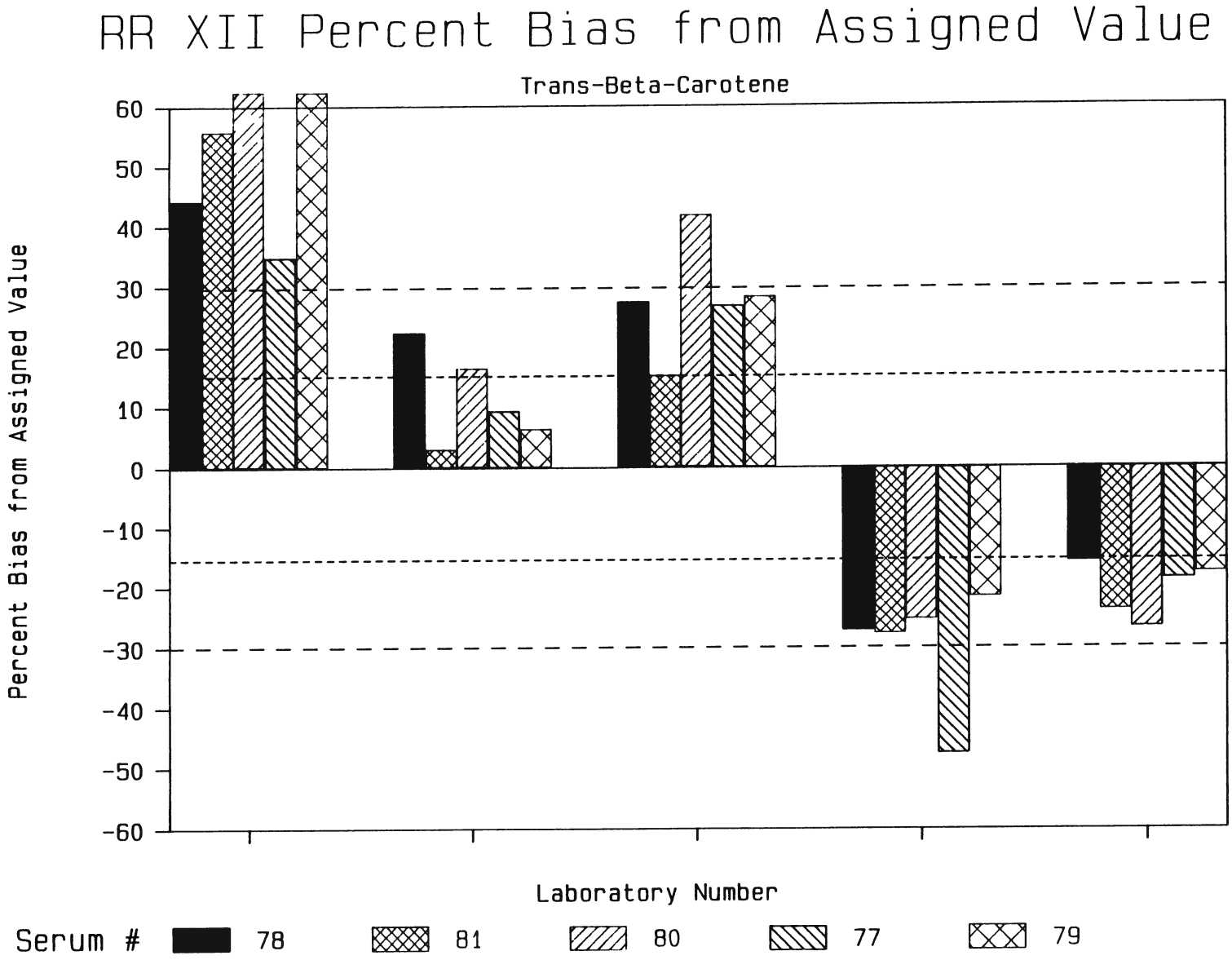
Lab #	Serum 78	Serum 81	Serum 80	Serum 77	Serum 79
	44.3	55.9	76.7	34.9	64.5
	22.4	3.1	16.4	9.5	6.4
	27.4	15.3	41.8	26.9	28.3
	-27.2	-27.8	-25.5	-47.5	-21.7
	-15.8	-23.8	-26.8	-18.7	-17.7
NBS A	-0.6	5.6	-2.6	1.0	-4.5
NBS B	-9.6	-10.1	-13.9	-2.0	-7.5
ASSIGNED VALUE	0.178	0.197	0.752	0.959	2.453

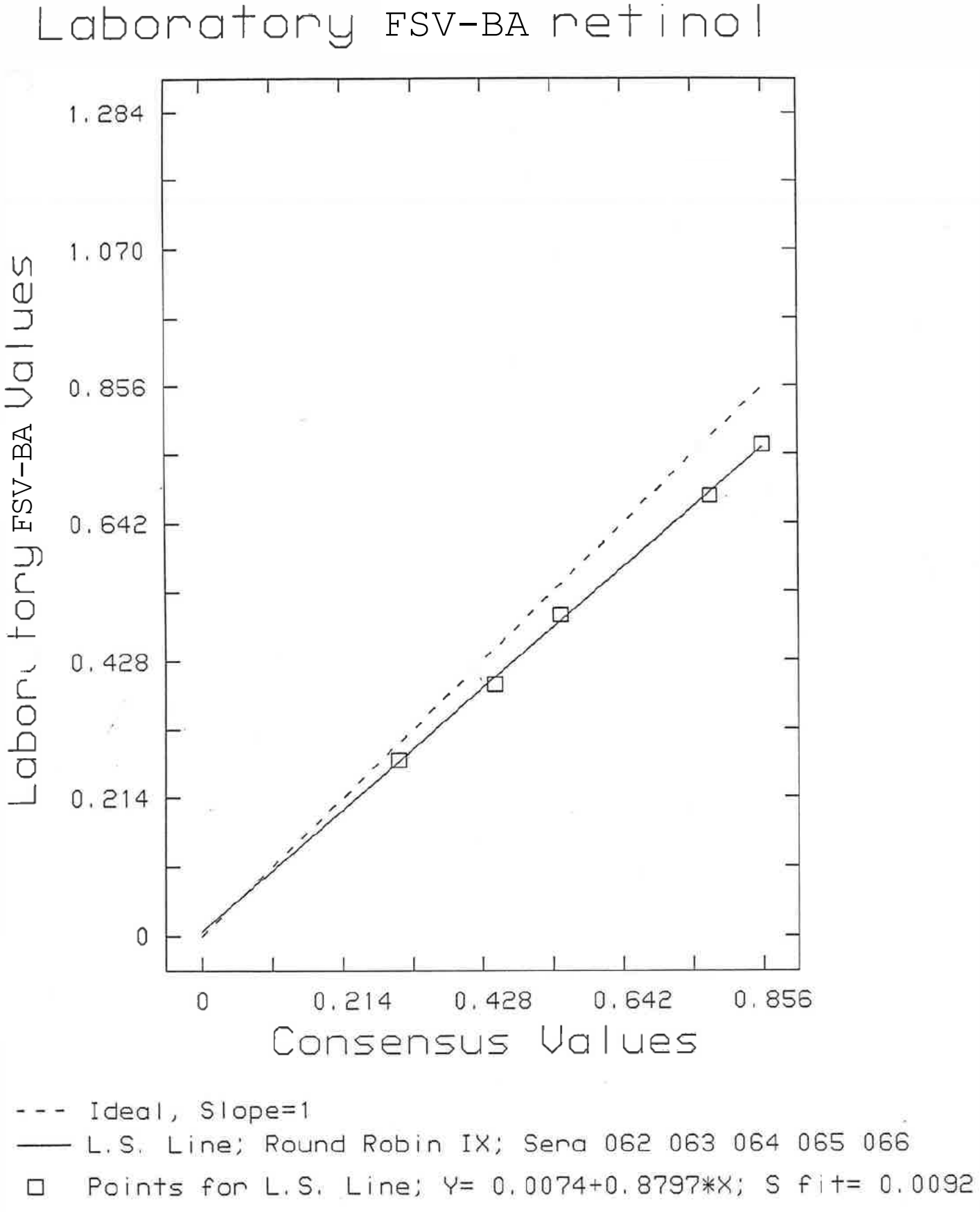
% Bias= 100*(Lab value - Assigned Value)/Assigned Value

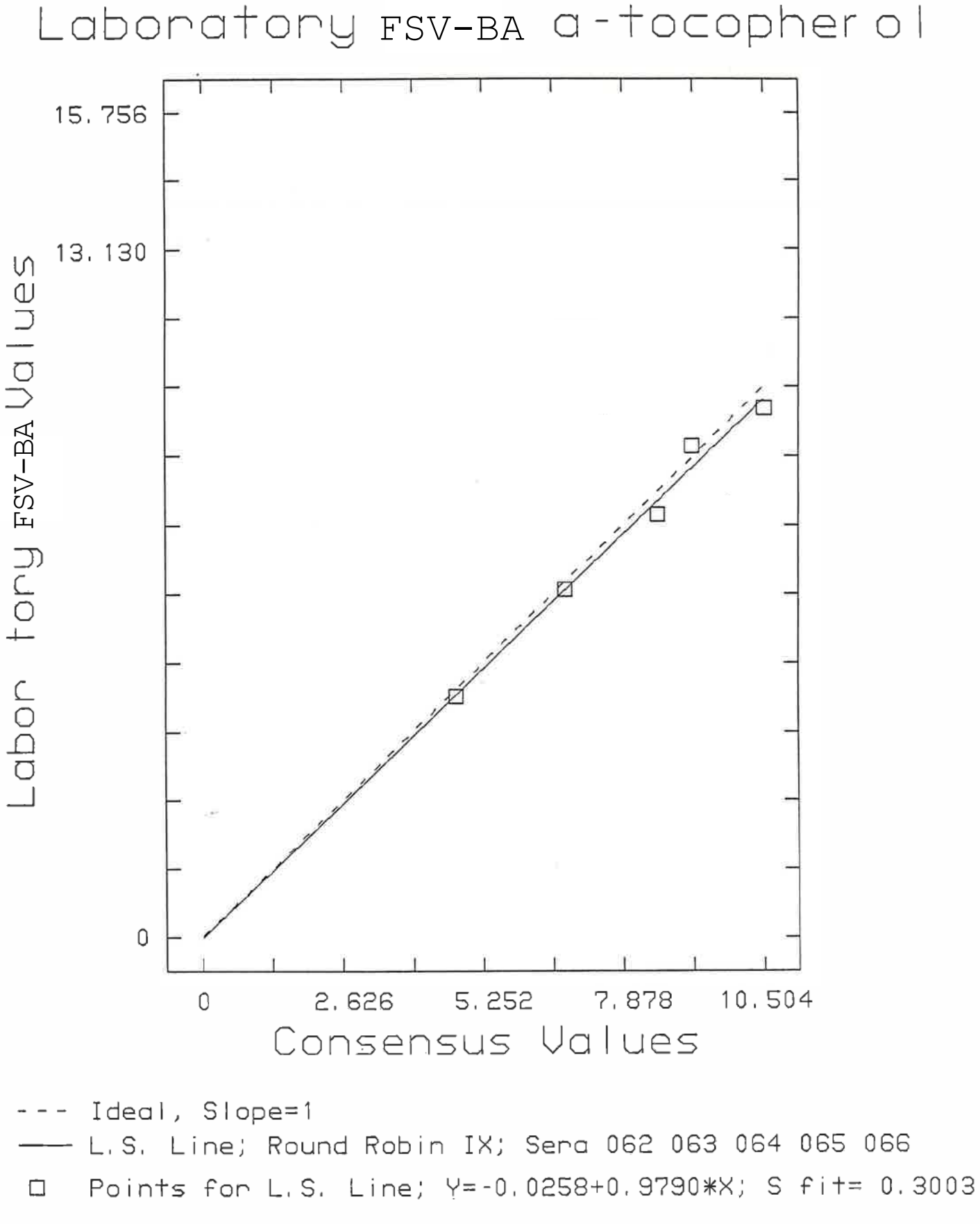


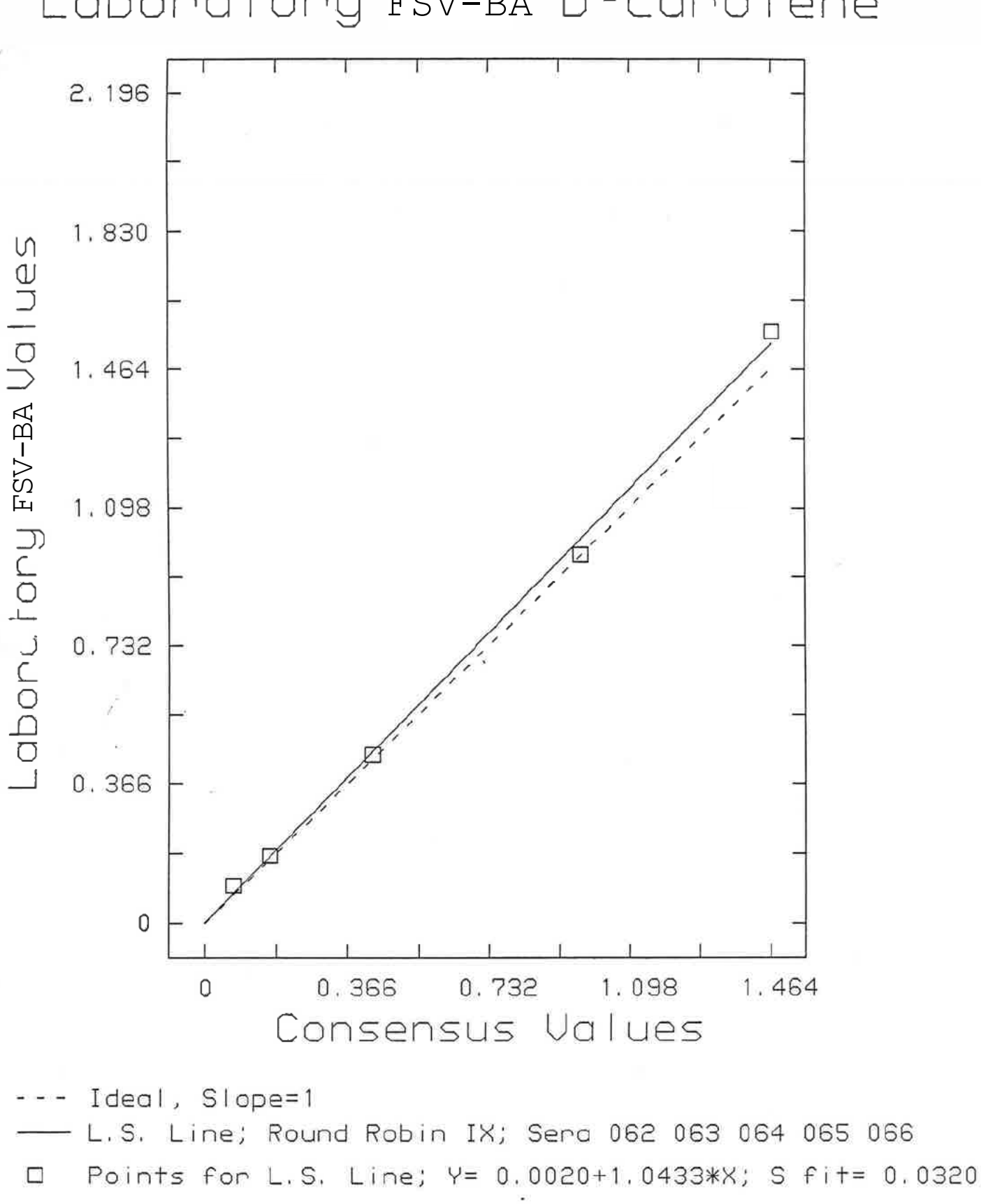












Appendix C. Updated "All-Lab Report" for RR12

The following three pages are a revised version of an "All-Lab" report for RR12. This report has three parts:

- Page 1 lists results for all analytes reported.
- Page 2 provides the legend for page 1.
- Page 3 summarizes each participants' performance for retinol, α-tocopherol, total β-carotene, and trans-β-carotene. These summaries are compatible with the percent bias evaluation advice given in the original RR12 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 "Assigned Value" 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 RR12. 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 XII Laboratory Results

C2

	Total Retinol					α-Tocopherol					Total β-Carotene					trans-β-Carotene					Total cis-β-Carotene				
Lab	77	78	79	80	81	77	78	79	80	81	77	78	79	80	81	77	78	79	80	81	77	78	79	80	81
FSV-BA	1.030	0.360	0.320	0.980	0.460	11.59	6.82	6.63	14.12	7.66	1.295	0.245	3.341	1.134	0.248	1.217	0.227	3.147	1.066	0.227	*0.078*	*0.018*	*0.194*	*0.068*	*0.021*
FSV-BD	1.029	0.409	0.325	0.851	0.370	11.00	5.80	6.40	9.30	6.10															
FSV-BE	1.253	0.494	0.443	1.140	0.564	9.03	5.55	5.75	10.33	6.05	0.612	0.113	1.483	0.473	0.121										
FSV-BF	1.040	0.380	0.340	0.820	0.380	9.60	5.80	6.10	10.70	6.30	≥1.294	≥0.257	≥4.034	≥1.328	≥0.307	1.294	0.257	4.034	1.328	0.307					
FSV-BG	1.174	0.406	0.388	1.026	0.485	8.80	5.60	5.60	9.70	5.30	1.025	0.196	2.949	0.847	0.147										
FSV-BH	0.993	0.362	0.331	0.854	0.425	8.85	5.35	5.65	9.95	5.75	1.118	0.220	2.825	0.947	0.230										
FSV-BI	1.100	0.400	0.360	1.000	0.460	8.75	5.25	5.49	9.73	5.70	0.840	0.160	2.200	0.740	0.160										
FSV-BX	0.960	0.370	0.350	0.880	0.420	7.70	4.60	4.90	10.20	5.00	≥0.780	≥0.150	≥2.020	≥0.550	≥0.150	0.780	0.150	2.020	0.550	0.150					
FSV-BY	0.801	0.305	0.309	0.520	0.257						0.945	0.187	2.479	0.800	0.179										
FSV-BZ	0.280	0.320	0.280	0.310	0.370	9.00	6.20	6.40	9.10	6.80															
FSV-CA	1.059	0.401	0.391	1.011	0.484	9.09	5.07	5.15	9.75	5.34															
FSV-CJ	1.194	0.435	0.403	1.198	0.496	10.00	6.59	6.56	11.09	6.63	0.894	0.204	2.302	0.719	0.175										
FSV-CK	0.869	0.325	0.270	0.747	0.349	7.82	4.08	4.60	8.83	4.66	0.542	0.144	2.000	0.614	0.153	0.504	0.130	1.920	0.560	0.142	*0.039*	*0.015*	*0.080*	*0.054*	*0.010*
FSV-CL	0.930	0.350	0.350	0.850	0.440	8.00	5.26	4.56	8.95	5.43	0.790	0.140	2.120	0.640	0.150										
FSV-CN	1.073	0.450	0.370	0.892	0.443	6.14	3.58	3.62	6.53	4.02	0.977	0.231	2.040	1.128	0.341										
FSV-CO	0.999	0.374	0.359	0.958	0.424	9.57	5.09	5.94	10.67	6.12	1.052	0.206	2.611	0.845	0.216										
FSV-DC	1.182	0.426	0.398	1.119	0.488						1.066	0.204	2.459	0.868	0.204										
FSV-DE						10.05	5.78	6.29	10.80	6.14															
FSV-DG	1.150	0.420	0.380	1.030	0.460	10.36	6.53	6.98	12.14	6.86	1.100	0.210	2.830	0.980	0.230										
FSV-DH	1.101	0.404	0.373	0.990	0.490	7.75	5.08	5.18	8.54	5.00															
FSV-DN	0.965	0.250	0.283	0.800	0.364	9.60	5.35	5.65	10.45	6.00	1.099	0.219	2.853	0.891	0.205										
FSV-DO	1.650	0.510	0.750	1.440	0.510																				
FSV-DT	1.098	0.355	0.438	0.957	0.433	9.32	5.05	6.31	9.65	5.67	0.940	0.234	2.550	0.782	0.257										
FSV-DZ	0.663	0.430	0.456	0.958	0.522	6.17	6.50	7.01	10.53	6.60															
FSV-ED	1.120	0.413	0.371	1.030	0.464	9.65	6.00	5.97	10.88	6.19	0.977	0.203	2.417	0.822	0.197										
FSV-EG	1.030	0.362	0.336	0.973	0.419						0.857	0.202	2.316	0.735	0.173										
FSV-EN	1.130	0.370	0.380	0.910	0.405	9.59	5.23	5.76	10.60	5.42															
FSV-EO	1.090	0.392	0.353	1.030	0.438						1.090	0.229	2.690	0.920	0.216	1.050	0.218	2.610	0.875	0.203	*0.040*	*0.011*	*0.080*	*0.045*	*0.013*
FSV-EP	1.068	0.417	0.367	1.026	0.437	7.40	4.70	5.47	9.23	5.44															
FSV-EU	1.200	0.600	0.540	1.500	0.710	12.10	6.90	7.40	12.90	7.60															
FSV-EW	1.142	0.432	0.397	1.063	0.468	10.71	6.39	6.68	11.03	7.59	1.050	0.214	2.660	0.873	0.214										
FSV-FE	1.095	0.402	0.368	0.960	0.462	7.27	4.62	5.22	8.48	5.31	1.593	0.271	4.526	1.135	0.278										
FSV-FI						11.07	6.42	6.55	11.28	7.02															
n	31	31	31	31	31	28	28	28	28	28	20	20	20	20	20	5	5	5	5	5	3	3	3	3	3
Min	0.280	0.250	0.270	0.310	0.257	6.14	3.58	3.62	6.53	4.02	0.542	0.113	1.483	0.473	0.121	0.504	0.130	1.920	0.550	0.142					
Mean	1.047	0.398	0.380	0.962	0.448	9.14	5.54	5.85	10.19	5.99	0.993	0.202	2.583	0.845	0.205	0.969	0.196	2.746	0.876	0.206	0.052	0.014	0.118	0.056	0.015
Max	1.650	0.600	0.750	1.500	0.710	12.10	6.90	7.40	14.12	7.66	1.593	0.271	4.526	1.135	0.341	1.294	0.257	4.034	1.328	0.307					
SD	0.215	0.064	0.088	0.218	0.077	1.495	0.826	0.845	1.457	0.893	0.225	0.038	0.612	0.172	0.052	0.326	0.054	0.873	0.334	0.067	0.022	0.003	0.066	0.012	0.005
CV	21	16	23	23	17	16	15	14	14	15	23	19	24	20	25	34	28	32	38	32	43	23	56	21	36
NISTa	1.010	0.362	0.346	0.919	0.411	9.81	6.08	6.63	11.27	5.97	1.010	0.173	2.460	0.694	0.192	0.940	0.161	2.270	0.647	0.177	*0.070*	*0.012*	*0.190*	*0.047*	*0.015*
NISTd	1.010	0.352	0.333	0.881	0.413	9.21	5.29	5.68	10.14	5.41	1.080	0.233	2.620	0.850	0.238	0.969	0.177	2.342	0.732	0.208	*0.111*	*0.056*	*0.278*	*0.118*	*0.030*
Median	1.073	0.401	0.368	0.973	0.443	9.21	5.45	5.85	10.27	6.03	1.001	0.205	2.515	0.846	0.205	1.050	0.218	2.610	0.875	0.203					
eSD	0.110	0.046	0.043	0.120	0.056	1.483	0.704	0.875	1.023	0.901	0.146	0.024	0.463	0.153	0.045	0.362	0.058	0.875	0.467	0.079					
eCV	10	11	12	12	13	16	13	15	10	15	15	12	18	18	22	34	27	34	53	39					

Round Robin XII 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 XII Laboratory Results

%Bias Summary

Lab	TR	aT	bC	t-bC
FSV-BA	-5±7	26±9	27±7	22±7
FSV-BD	-9±7	5±11		
FSV-BE	21±4	0±2	-42±2	
FSV-BF	-9±6	5±1	44±16	52±18
FSV-BG	6±3	-5±5	-3±16	
FSV-BH	-9±3	-3±1	11±2	
FSV-BI	1±2	-5±1	-17±5	
FSV-BX	-8±3	-13±7	-26±6	-37±6
FSV-BY	-31±13		-7±4	
FSV-BZ	-41±28	4±11		
FSV-CA	4±4	-7±4		
FSV-CJ	13±6	12±5	-10±6	
FSV-CK	-22±3	-20±5	-30±10	-36±10
FSV-CL	-9±6	-12±7	-24±6	
FSV-CN	1±7	-35±2	18±33	
FSV-CO	-4±2	1±4	3±3	
FSV-DC	10±3		1±3	
FSV-DE		6±3		
FSV-DG	5±2	17±3	11±5	
FSV-DH	3±4	-14±4		
FSV-DN	-21±10	0±3	7±5	
FSV-DO	50±34			
FSV-DT	1±11	-2±6	6±14	
FSV-DZ	2±24	4±22		
FSV-ED	4±2	5±3	-3±1	
FSV-EG	-6±4		-10±6	
FSV-EN	-3±7	-2±6		
FSV-EO	0±4		8±2	0±0
FSV-EP	1±3	-12±5		
FSV-EU	45±19	27±2		
FSV-EW	7±1	16±7	5±1	
FSV-FE	1±2	-15±4	48±21	
FSV-FI		15±4		
NISTa	-7±2	8±6	-8±8	-26±8
NISTd	-9±3	-3±4	9±7	-16±8

Label	Definition
Lab	Participant code
TR	Total Retinol
aT	a-Tocopherol
bC	Total b-Carotene
t-bC	Trans-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 "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-based reference value.

Appendix D. Shipping Package Inserts for RR13

Two items were attached to each package shipped to an RR12 participant:

- **Cover letter**. The original letter as attached to the packages has been lost, page D2 reproduces the proof copy of the mail-merge letter form.

- **Datasheet**. Page D3 reproduces the form.

May 19, 1988

^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.

Five proficiency testing samples for fat-soluble vitamin analyses (Round-Robin XIII) are being shipped to you on May 23 under a separate cover. These samples are freeze-dried plasma and should be reconstituted with 1.00 mL of water. You are to report only one value for each sample on the attached data entry form.

You were sent a set of Control Materials with assigned values for retinol, α-tocopherol, and β-carotene about one month ago. A copy of the letter that accompanied those samples and a Control Materials data form is enclosed. You were to analyze them immediately and make adjustments to your methods as needed. You are also to analyze a set of these materials concurrently with RR-XIII samples. All results from the analyses of Control Materials are to be provided to us along with RR-XIII data.

For both the RR-XIII samples and the Control Materials, we will accept data for both "trans β-carotene" and "total β-carotene" (trans and cis isomers). For laboratories that resolve trans β-carotene from its cis isomers, we ask that you report a value for trans β-carotene on the data form. As an option, you might report total β-carotene also by summing the responses for the cis and trans isomers. For laboratories that measure the cis and trans forms as a single peak, your data should be reported as "total β-carotene". Please contact me (301/975-3108) or Neal Craft (301/975-3111) if you have questions.

This year's Workshop will be held sometimes this fall; we have not settled on a date yet. Please indicate your willingness to participate and let us know of any period between October 1 and December 15 that is inconvenient for you on the bottom of the RR-XIII data form.

Sincerely,

ORIGINAL SIGNED BY
WILLIE E. MAY

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

cc: R. Schaffer
R. Paule

To be included in the statistical analysis,
PLEASE submit results by July 1, 1988. Thank you.

REPORT ON NBS/NCI SAMPLES FROM LABORATORY # _____

DATE OF ANALYSIS _____

RESULTS IN mg/L

SAMPLE #	ANALYTE	Result
SERUM 90	RETINOL	
		All Trans Total
VIAL # _____	B-CAROTENE	
	A-TOCOPHEROL	
SERUM 91	RETINOL	
		All Trans Total
VIAL # _____	B-CAROTENE	
	A-TOCOPHEROL	
SERUM 92	RETINOL	
		All Trans Total
VIAL # _____	B-CAROTENE	
	A-TOCOPHEROL	
SERUM 93	RETINOL	
		All Trans Total
VIAL # _____	B-CAROTENE	
	A-TOCOPHEROL	
SERUM 94	RETINOL	
		All Trans
VIAL # _____	B-CAROTENE	
	A-TOCOPHEROL	

Reconstitute the Serum Sample with 1.0 mL water.

YES, I PLAN TO ATTEND THE Q.A. WORKSHOP THIS FALL _____
NO, I CANNOT ATTEND THE Q.A. WORKSHOP THIS FALL _____
THE FOLLOWING FALL TIME PERIOD IS INCONVENIENT FOR ME:

Appendix E. Final Report for RR13

The following twenty pages are the final report for RR13 as provided to participants. This report consisted of:

- An individualized cover letter and discussion. The pages reproduced here are for participant FSV-BA.
- Four tables that list the erported results and several summary values for retinol, α-tocopherol, total β-carotene, and *trans*-β-carotene. Due to the complex formatting used in the tables, the originally listed laboratory codes have been deleted without replacement. However, Appendix F provides a complete listing of the RR13 results where the original codes have been altered to ensure confidentiality. Appendix F also provides more relevant summary statistics. (The labels "NBS A" and "NBS B" in these tables refer to NIST analysts NISTa and NISTd.)
- For all particpants, graphical presentations of their own measurements of the "Control B" material for retinol, α-tocopherol, and *trans*-β-carotene. The pages reproduced here are for participant FSV-BA.
- For all particpants, graphical presentations of their own measurements of the "Control D" material for retinol, α-tocopherol, total β-carotene, and *trans*-β-carotene. The pages reproduced here are for participant FSV-BA.
- For all participants, graphical presentations of their own over-time bias relative to an assigned value for retinol, α-tocopherol, total β-carotene, and total β-carotene. The pages reproduced here are for participant FSV-BA.

UNITED STATES DEPARTMENT OF COMMERCE
National Bureau of Standards
Gaithersburg, Maryland 20899

August 2, 1988

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 XIII. Data is reported in tabular form and, as before, individual laboratory performance is judged against an assigned value that is derived from the laboratory grand average (33%) and two separate NBS measurement procedures (67%). As before, we deem deviations of 0-15% from the assigned value to be acceptable performance, 16-30% deviations to represent marginal performance, and >30% deviations to be unacceptable. The summary also includes two types of graphical presentations. The first presents your results from the analysis of the same sample in both Round Robin XII and Round Robin XIII. For many of you, the results also reflect your replication error, since this same sample was run as both a control and an unknown in Round Robin XIII. The second series of graphs are historical presentations of your results vs. the assigned value for each analyte over the past two years.

The results from Round Robin XIII reflect a significant improvement in measurements for retinol and α-tocopherol, and noticeable improvement in β-carotene measurements. We feel that this improvement is directly linked to the use of "Value Assigned Control Materials". We feel that the performance criteria stated above (i.e. 0-15% = Acceptable, etc.) should be changed to reflect the new state-of-the-practice as measurement performance improves. This will be a topic for discussion at the Workshop. These criteria are being maintained, for the present however, as a basis for comparison with past performance.

For retinol measurements, the relative standard deviation was in the 12-15% range for the past three Round Robins. The relative standard deviation of results from Round Robin XIII dropped to the 8-9% range. As in Round Robin XII, approximately 67% of the participating labs reported results that were deemed to be acceptable. In Round Robin XII, the results from five laboratories were deemed to be unacceptable. In Round Robin XIII, three of these five laboratories reported results with biases ≤10%; one laboratory dropped out of the program; and one laboratory again reported results that were deemed to be unacceptable. The one laboratory that again reported unacceptable results did not run the Control Materials as requested.

For α-tocopherol measurements, the relative standard deviation was in the 12-15% range during the previous three Round Robins. The relative standard deviation of results for Round Robin XIII dropped to the 8-10% range. As in Round Robin XII, approximately 60% of the participating laboratories reported results that were deemed to be acceptable. None of the laboratories reported results that were deemed to be unacceptable.

Only four labs reported results for trans β-carotene in Round Robin XIII. One of the four reported that results were deemed to be unacceptable. Eighteen labs reported data for "Total β-carotene". Interlaboratory precision improved from the 20% (Round Robin XII) to the 15% range in Round Robin XIII. As in the previous Round Robin, approximately 50% of the labs reported acceptable results. In Round Robin XIII, the four labs that reported data deemed to be unacceptable did not run the Control Materials as requested. Measurement methods for determination of β-carotene(s) will be a major topic at the workshop this year.

Your laboratory's overall performance in this Round Robin was:

	ACCEPTABLE	MARGINAL	UNACCEPTABLE
Retinol	X		
α-Tocopherol		X	
All-trans β-Carotene	X		
Total β-Carotene	X		

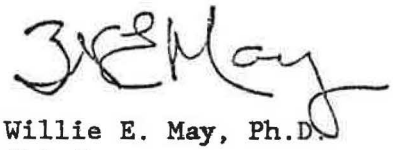
This year's workshop will be held November 2 and 3 at NBS and will address the measurement of fat-soluble vitamins only. The agenda for this workshop is being put together. It will include tours of the NBS analytical facilities as well as discussions of the following topics:

- FY88 Round Robin Results for retinol, α-tocopherol and β-carotene.
  - data presentation and discussion
  - use and value of control materials
  - development and use of SRM's
- Accuracy of Analytical Results vs. Extraction and Separations Methods Used for Retinol and α-tocopherol Analyses.
- Carotenoid Analysis: Problems with Extraction and Resolution of the all-trans and cis isomers of β-carotene.
- QA Program for the coming year (including discussions of accuracy and precision goals).

You will receive additional information regarding the Workshop within the next few weeks. Please come prepared to participate in the discussions. Feel free to bring along visual aids to support ideas or points that you wish to make. We strongly encourage the attendance of laboratory personnel as well as Principal Investigators at this year's workshop.

The next set of Round Robin samples will be shipped on August 22 or 23.

Sincerely,



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

cc: R. Schaffer
R. Paule
H. Pierson

Retinol Results for RRXIII

Lab #	Serum 90	Serum 91	Serum 92	Serum 93	Serum 94
	0.362	1.003	0.577	0.726	0.403
	0.396	0.990	0.574	0.732	0.415
	0.454	1.169	0.649	0.873	0.483
	0.388	1.044	0.568	0.748	0.443
	0.446	1.154	0.640	0.827	0.471
	0.375	0.975	0.562	0.787	0.450
	0.401	1.015	0.623	0.687	0.420
	0.412	1.054	0.590	0.777	0.454
	0.367	1.096	0.610	0.673	0.372
NC	0.430	1.004	0.580	0.734	0.436
	0.472	1.018	0.702	0.808	0.522
NC	0.372	1.037	0.544	0.718	0.437
	0.421	1.155	0.650	0.859	0.487
	0.364	0.963	0.553	0.771	0.408
NC	0.436	1.214	0.796	0.892	0.640
	0.400	1.090	0.600	0.780	0.430
	0.350	0.958	0.518	0.716	0.361
	0.385	1.058	0.575	0.770	0.428
	0.369	0.958	0.449	0.766	0.410
	0.402	1.010	0.625	0.761	0.416
L	0.346	0.927	0.564	0.744	0.375
	0.416	1.037	0.610	0.712	0.437
	0.382	1.020	0.584	0.750	0.424
	0.430	1.150	0.580	0.720	0.430
	0.380	0.880	0.530	0.650	0.420
	0.372	1.035	0.588	0.749	0.419
	0.450	0.920	0.560	0.700	0.470
	0.320	0.890	0.490	0.660	0.360
	0.388	0.914	0.517	0.823	0.452
	0.389	0.866	0.515	0.832	0.445
LAB AVG	0.396	1.027	0.584	0.750	0.431
SD	0.036	0.081	0.054	0.057	0.039
Rel SD	9.2	7.8	9.3	7.6	9.0
NBS AVG	0.377	1.004	0.574	0.695	0.406
Assigned Value	0.383 +/-0.032	1.017 +/-0.032	0.577 +/-0.023	0.713 +/-0.068	0.418 +/-0.025

NC = No Control Data. Results from labs with NC are not included in the Average.

L = Late results not included in the Average.

Assigned Value is derived from the mean of the Laboratory Grand Average and two NBS methods.
The Uncertainty is expressed as two standard deviations of the Assigned Value.

Retinol Results RRXIII

% Bias from the Assigned Value.

Lab #	Serum 90	Serum 91	Serum 92	Serum 93	Serum 94	Rating
	-5.5	-1.4	0.0	1.8	-3.5	AAA
	3.4	-2.6	-0.6	2.6	-0.6	AAA
	18.5	15.0	12.4	22.4	15.7	M
	1.3	2.7	-1.6	4.9	6.1	AA
	16.4	13.5	10.9	15.9	12.8	M
	-2.1	-4.1	-2.6	10.3	7.8	AA
	4.7	-0.2	7.9	-3.7	0.6	AA
	7.5	3.7	2.2	8.9	8.7	AA
	-4.2	7.8	5.7	-5.7	-10.9	A
NC	12.2	-1.3	0.5	2.9	4.4	A
	23.2	0.1	21.6	13.3	25.0	M
NC	-2.9	2.0	-5.8	0.7	4.6	AA
	9.9	13.6	12.6	20.4	16.6	M
	-5.0	-5.3	-4.2	8.1	-2.3	AA
NC	13.8	19.4	37.9	25.0	53.3	U
	4.4	7.2	3.9	9.3	3.0	AA
	-8.6	-5.8	-10.3	0.4	-13.6	A
	0.5	4.1	-0.4	7.9	2.5	AA
	-3.7	-5.8	-22.2	7.4	-1.8	M
	4.9	-0.7	8.3	6.7	-0.4	AA
L	-9.7	-8.8	-2.3	4.3	-10.2	AA
	8.6	2.0	5.7	-0.2	4.6	AA
	-0.3	0.3	1.1	5.1	1.5	AAA
	12.2	13.1	0.5	0.9	3.0	A
	-0.8	-13.5	-8.2	-8.9	0.6	A
	-2.9	1.8	1.9	5.0	0.3	AAA
	17.5	-9.5	-3.0	-1.9	12.6	M
	-16.5	-12.5	-15.1	-7.5	-13.8	M
	1.4	-10.1	-10.4	15.4	8.2	M
	1.6	-14.9	-10.8	16.6	6.7	M

Alpha-Tocopherol Results for RRXIII

Lab #	Serum 90	Serum 91	Serum 92	Serum 93	Serum 94
	6.85	9.42	5.91	4.60	5.91
	6.40	8.69	5.45	4.54	5.64
	7.37	8.73	5.59	4.75	5.62
	7.66	10.11	6.06	5.40	6.32
	6.94	9.49	6.08	4.88	6.13
	6.86	9.23	5.74	4.79	5.88
	6.13	8.39	5.75	4.79	6.62
NC	7.84	9.84	6.15	5.02	6.36
	7.74	10.28	7.14	5.56	6.78
NC	6.15	7.58	4.25	3.37	4.41
	6.50	8.84	5.51	4.72	5.35
	6.62	9.23	6.35	5.62	5.51
	7.11	9.30	5.88	4.80	5.86
NC	8.30	10.55	6.95	5.00	7.35
	7.00	10.76	6.91	4.60	5.89
	6.20	7.14	4.58	4.09	5.11
	8.47	10.57	6.67	5.40	6.77
L	6.41	9.39	6.35	5.99	5.85
	7.19	9.62	6.24	5.25	6.25
	6.95	9.25	5.65	4.90	5.80
	6.92	8.99	5.47	4.44	5.58
	6.90	10.40	6.80	5.90	6.50
	7.61	9.99	6.07	5.11	6.18
	7.39	9.51	6.51	5.42	6.68
	6.81	8.97	5.35	4.72	5.84
	6.93	9.30	5.99	4.82	6.85
	7.17	7.82	5.03	5.07	6.10
	6.35	7.82	4.71	4.93	6.06
LAB AVG	7.02	9.37	5.99	4.96	6.05
SD	0.54	0.81	0.59	0.44	0.50
Rel SD	7.7	8.6	9.9	8.9	8.2
NBS AVG	7.02	9.44	5.90	4.61	5.90
Assigned Value	7.02 +/-0.69	9.40 +/-0.55	5.93 +/-0.17	4.73 +/-0.39	5.87 +/-0.48

NC = No Control Data. Results from labs with NC are not included in the Average.

L = Late results not included in the Average.

Assigned Value is derived from the mean of the Laboratory Grand Average and two NBS methods. The Uncertainty is expressed as two standard deviations of the Assigned Value.

Alpha-Tocopherol Results RRXIII

% Bias from the Assigned Value.

Lab #	Serum 90	Serum 91	Serum 92	Serum 93	Serum 94	Rating
	-2.4	0.2	-0.3	-2.7	0.7	AAA
	-8.8	-7.6	-8.1	-4.0	-3.9	AA
	5.0	-7.1	-5.7	0.4	-4.2	AA
	9.1	7.5	2.2	14.2	7.7	A
	-1.1	1.0	2.6	3.2	4.5	AAA
	-2.3	-1.8	-3.2	1.3	0.2	AAA
	-12.7	-10.7	-3.0	1.3	12.8	A
NC	11.7	4.7	3.7	6.1	8.4	A
	10.3	9.4	20.4	17.6	15.5	M
NC	-12.4	-19.4	-28.4	-28.7	-24.8	M
	-7.4	-6.0	-7.0	-0.2	-8.8	AA
	-5.7	-1.8	7.1	18.8	-6.1	M
	1.3	-1.1	-0.8	1.5	-0.1	AAA
NC	18.2	12.2	17.2	5.7	25.2	M
	-0.3	14.5	16.6	-2.7	0.4	M
	-11.7	-24.0	-22.7	-13.5	-12.9	M
	20.6	12.4	12.5	14.3	15.4	M
L	-8.7	-0.1	7.1	26.7	-0.3	M
	2.4	2.3	5.3	11.0	6.5	A
	-1.0	-1.6	-4.7	3.6	-1.2	AAA
	-1.4	-4.4	-7.7	-6.1	-4.9	AA
	-1.7	10.6	14.7	24.8	10.8	M
	8.4	6.3	2.4	8.0	5.3	AA
	5.3	1.2	9.8	14.6	13.8	A
	-3.0	-4.6	-9.7	-0.2	-0.5	AA
	-1.3	-1.1	1.0	1.9	16.7	A
	2.1	-16.9	-15.1	7.2	3.9	M
	-9.5	-16.8	-20.5	4.1	3.2	M

Total Beta Carotene Results for RRXIII

Lab #	Serum 90	Serum 91	Serum 92	Serum 93	Serum 94
	0.116	0.910	0.977	0.359	0.213
	0.072	0.836	1.111	*0.288	0.176
	0.079	1.113	1.403	0.365	0.194
	0.071	0.962	1.193	0.274	0.167
	0.078	0.999	1.193	0.343	0.210
	0.089	1.034	1.252	0.346	0.205
NC	0.122	1.440	1.830	0.475	0.282
	0.075	1.004	1.345	0.457	0.262
NC	0.058	0.845	1.056	0.287	0.149
	0.089	0.950	1.190	0.337	0.190
	0.061	0.904	0.982	0.253	0.145
	0.080	1.120	1.410	0.360	0.200
	0.076	0.920	1.133	0.346	0.192
	0.076	0.915	1.153	0.320	0.187
	0.097	0.928	1.183	0.328	0.202
	0.103	1.148	1.453	0.440	0.228
	0.119	1.099	1.385	0.327	0.271
	0.170*	0.880	1.100	0.350	0.250
LAB AVG	0.085	0.983	1.216	0.343	0.206
SD	0.017	0.095	0.147	0.052	0.034
Rel SD	19.6	9.7	12.1	15.1	16.4
NBS AVG	0.087	1.119	1.333	0.330	0.224
Assigned Value	0.086 +/-0.030	1.050 +/-0.139	1.294 +/-0.187	0.334 +/-0.128	0.215 0.043

NC = No Control Data. Results from labs with NC are not included in the Average.

* = Value not included in the Average.

Assigned Value is derived from the mean of the Laboratory Grand Average and two NBS methods.
The Uncertainty is expressed as two standard deviations of the Assigned Value.

Total Beta Carotene Results RRXIII

% Bias from the Assigned Value.

Lab #	Serum 90	Serum 91	Serum 92	Serum 93	Serum 94	Rating
	34.7	-13.3	-24.5	7.3	-0.8	M
	-16.4	-20.4	-14.1	-13.9	-18.1	M
	-8.3	6.0	8.4	9.1	-9.7	AA
	-17.5	-8.4	-7.8	-18.1	-22.2	M
	-9.4	-4.9	-7.8	2.6	-2.2	AA
	3.4	-1.5	-3.2	3.5	-4.6	AAA
NC	41.7	37.1	41.4	42.0	31.3	U
	-12.9	-4.4	4.0	36.6	22.0	M
NC	-32.6	-19.5	-18.4	-14.2	-30.6	U
	3.4	-9.5	-8.0	0.8	-11.5	A
	-29.2	-13.9	-24.1	-24.4	-32.5	M
	-7.1	6.7	9.0	7.6	-6.9	AA
	-12.3	-12.4	-12.4	3.5	-10.6	A
	-11.7	-12.9	-10.9	-4.3	-12.9	A
	12.6	-11.6	-8.6	-1.9	-6.0	A
	19.0	9.3	12.3	31.6	5.9	M
	38.2	4.7	7.0	-2.2	26.2	M
	97.4	-16.2	-15.0	4.7	16.4	M

Trans-Beta Carotene Results for RRXIII

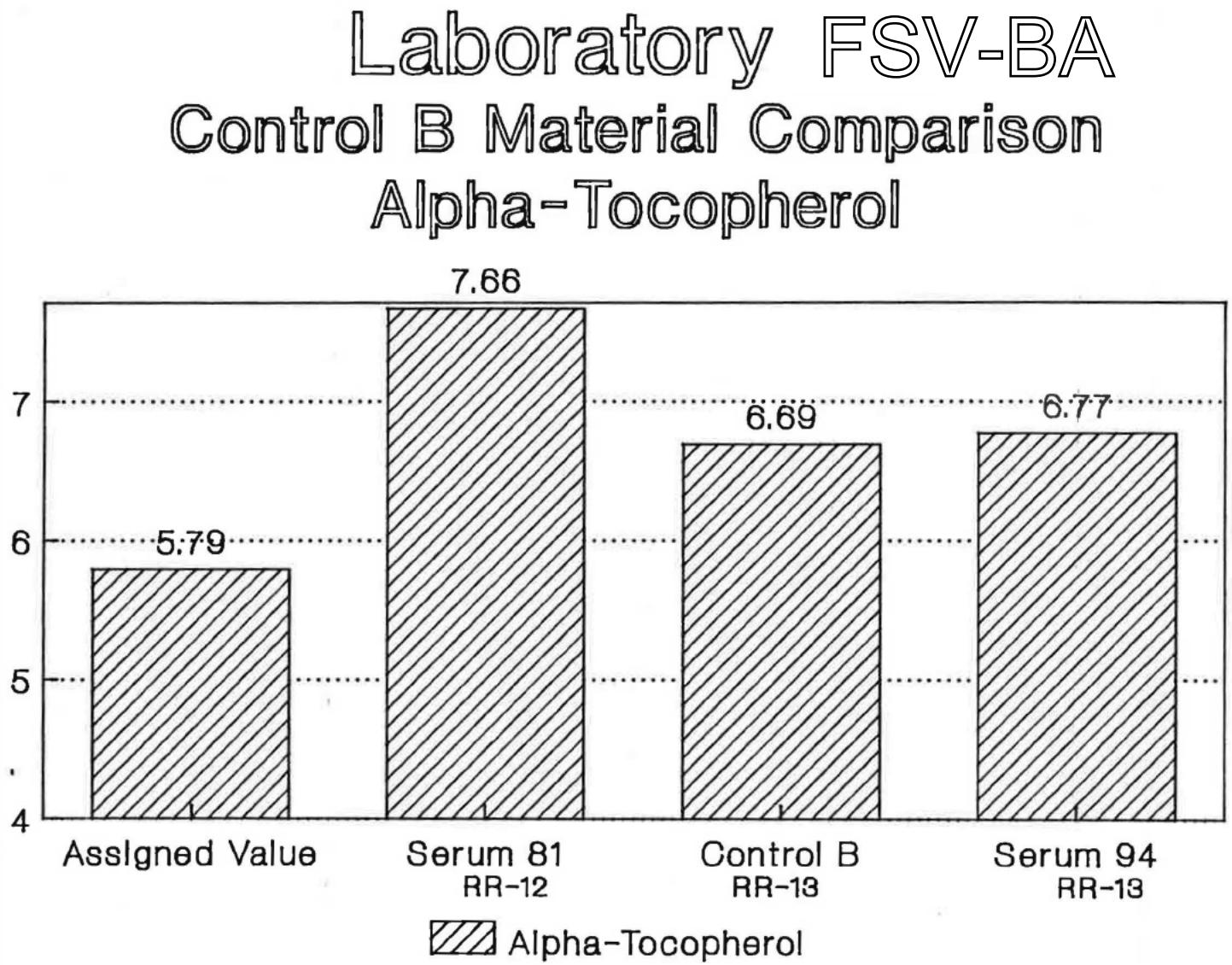
Lab #	Serum 90	Serum 91	Serum 92	Serum 93	Serum 94
	0.074	0.876	1.111	0.301	0.176
		0.830	0.980	0.250	0.170
	0.050	0.612	0.896	0.269	0.165
	0.058	0.645	0.965	0.225	0.141
LAB AVG	0.061	0.741	0.988	0.261	0.163
SD	0.012	0.132	0.090	0.032	0.016
Rel SD	20.2	17.8	9.1	12.3	9.5
NBS AVG	0.079	1.036	1.247	0.295	0.200
Assigned Value	0.073 +/-0.032	0.948 +/-0.201	1.161 +/-0.275	0.283 +/-0.104	0.192 +/-0.041

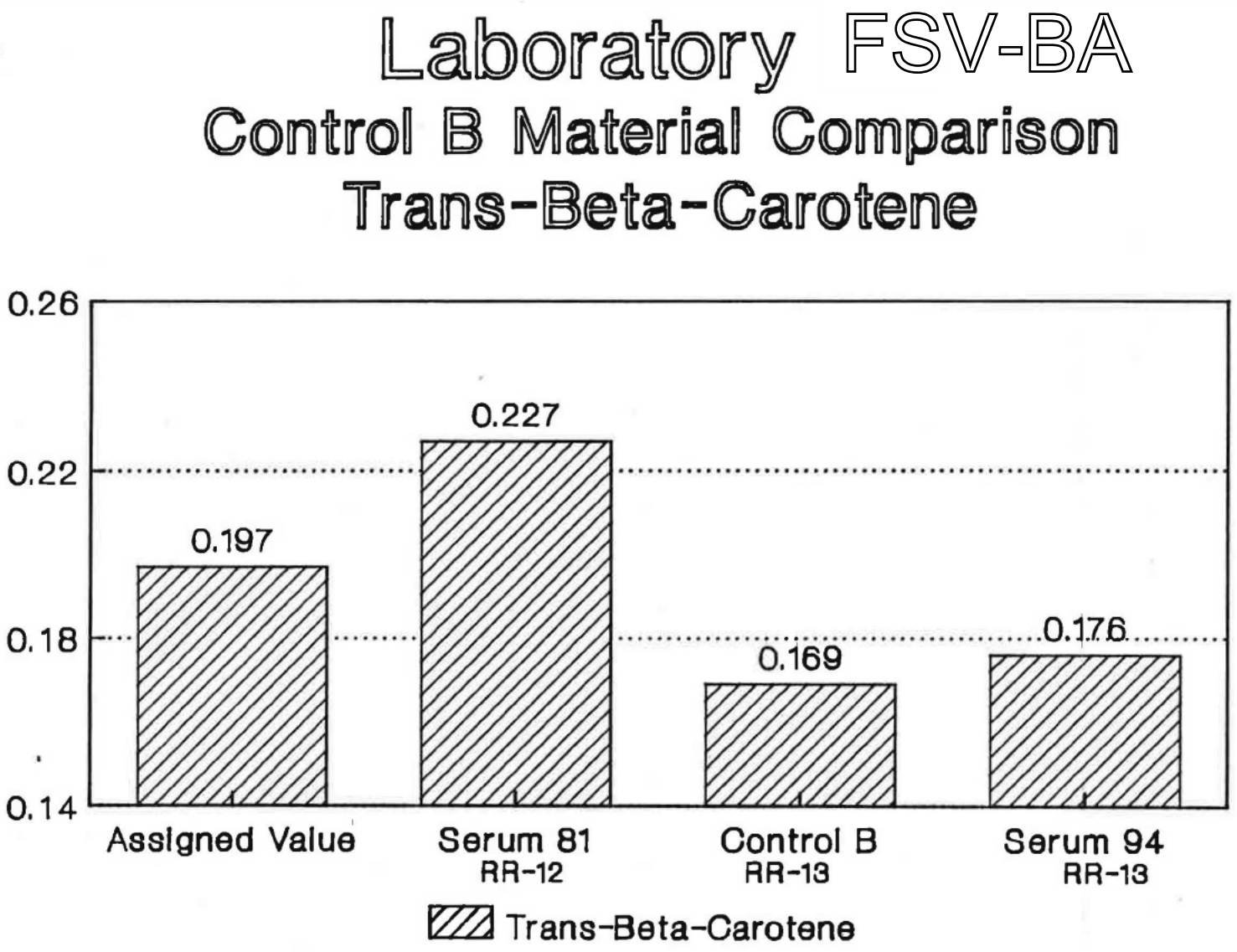
Assigned Value is derived from the mean of the Laboratory Grand Average and two NBS methods.
The Uncertainty is expressed as two standard deviations of the Assigned Value.

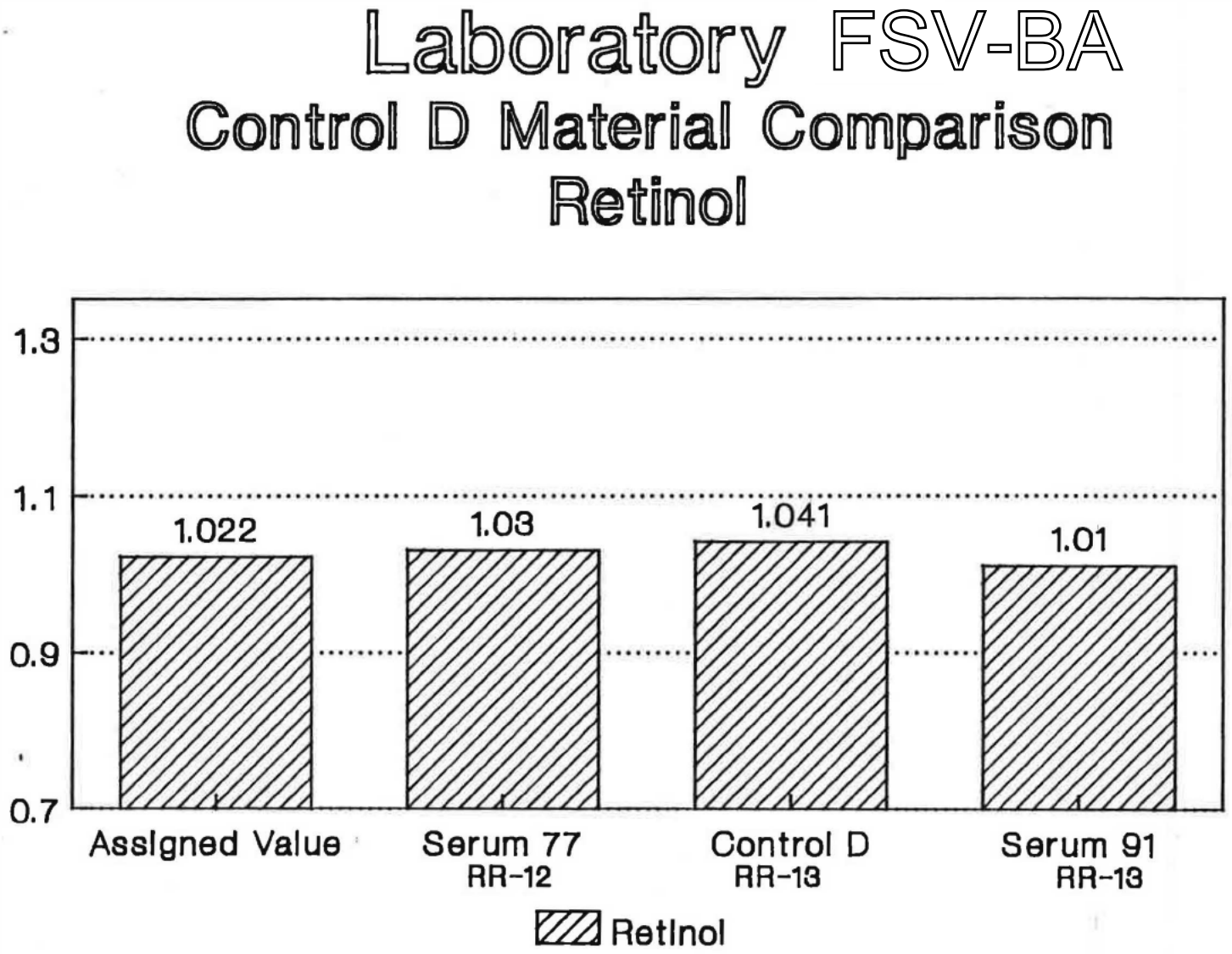
Trans-Beta Carotene Results RRXIII

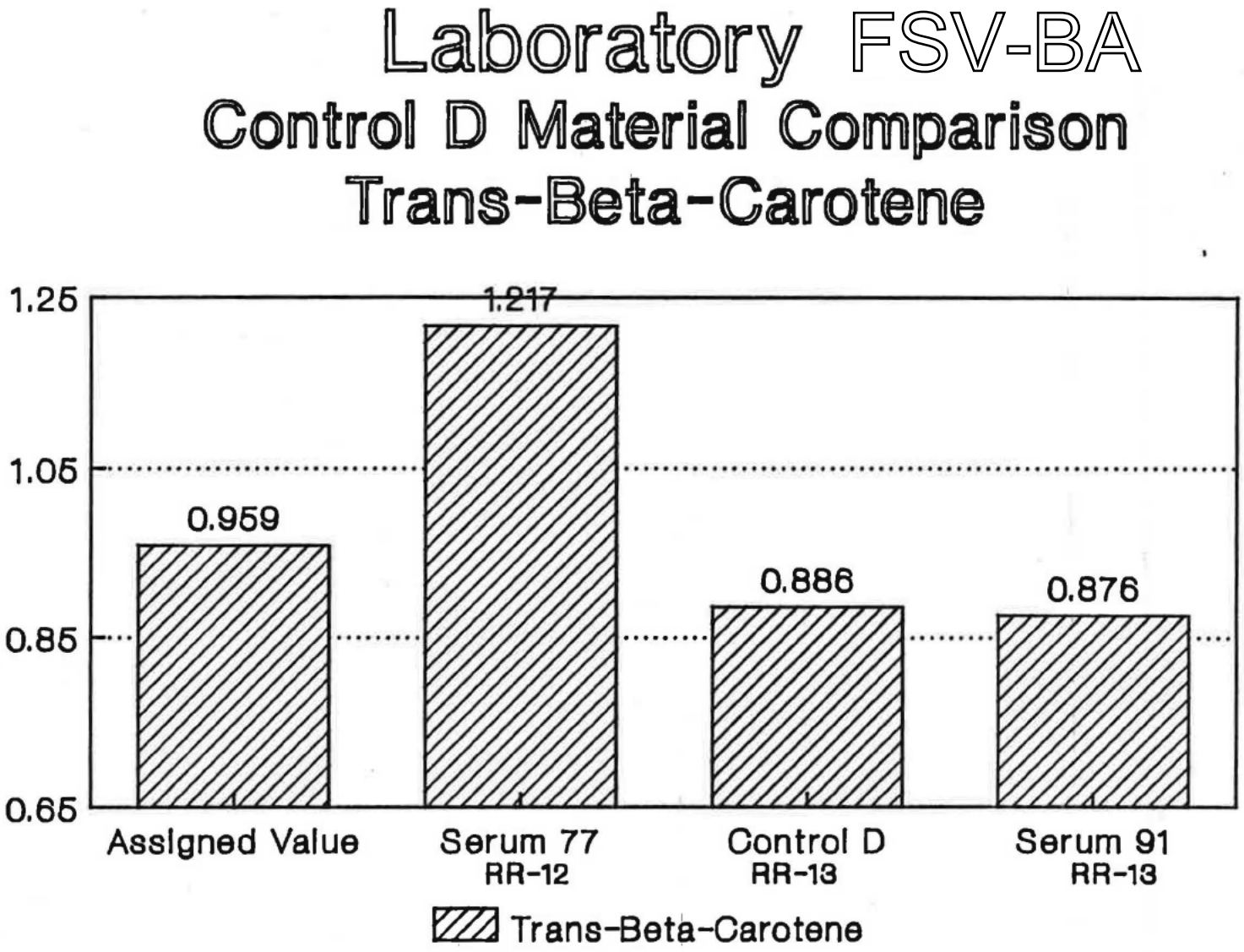
% Bias from the Assigned Value.

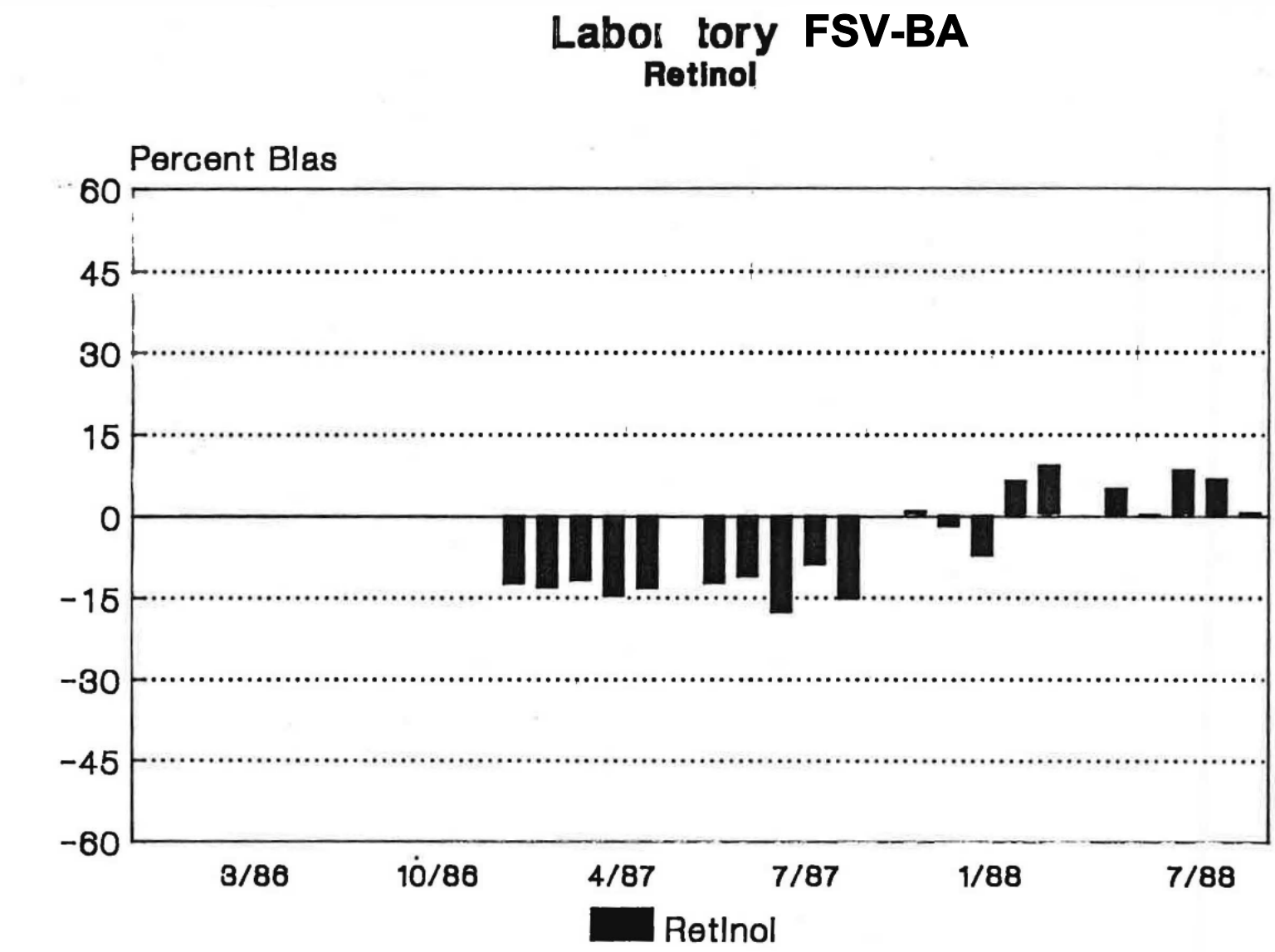
Lab #	Serum 90	Serum 91	Serum 92	Serum 93	Serum 94	Rating
	2.0	-7.6	-4.3	6.2	-8.4	AA
		-12.5	-15.6	-11.8	-11.5	A
	-31.2	-35.5	-22.8	-5.1	-13.9	U
	-19.9	-31.9	-16.9	-20.7	-26.8	M

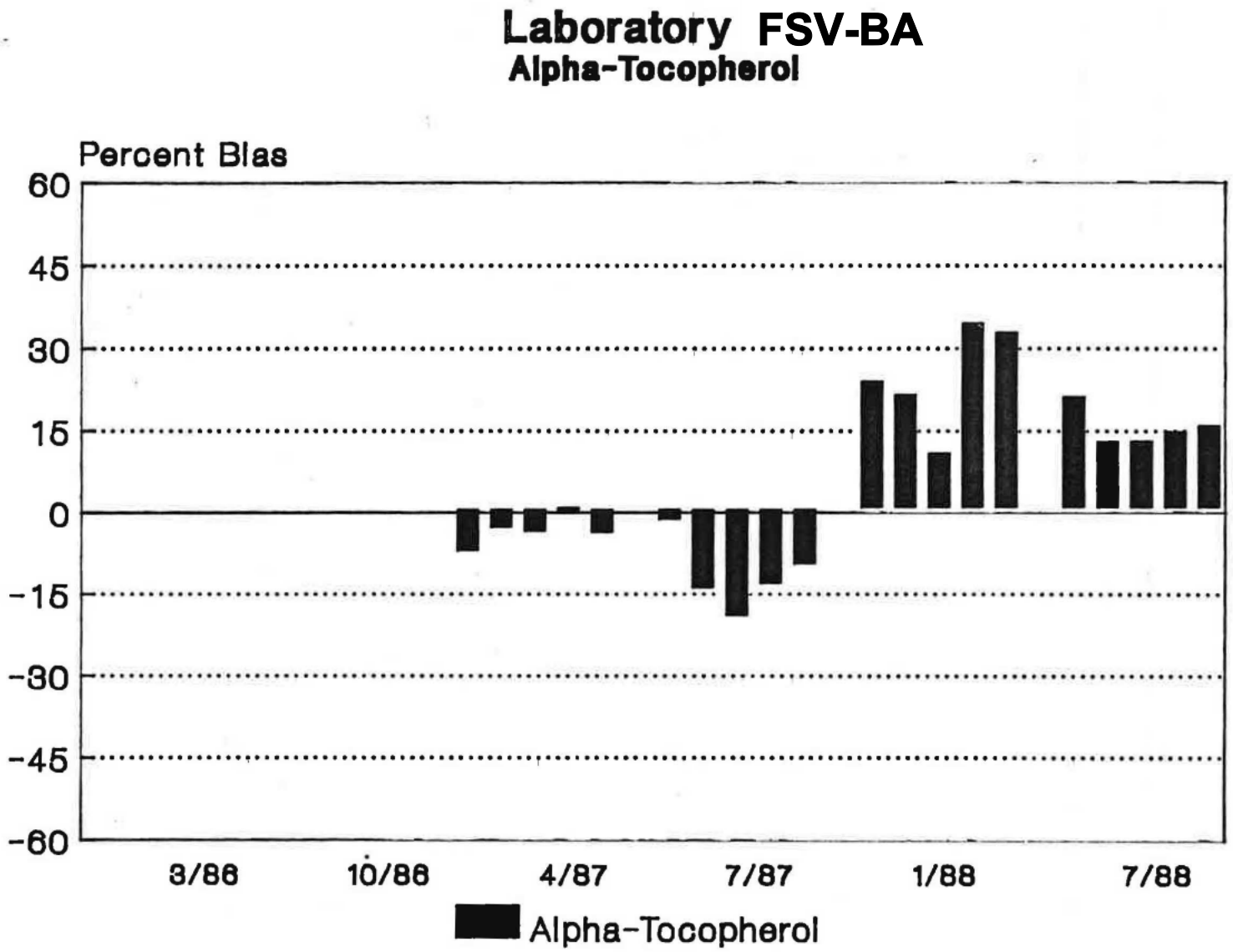




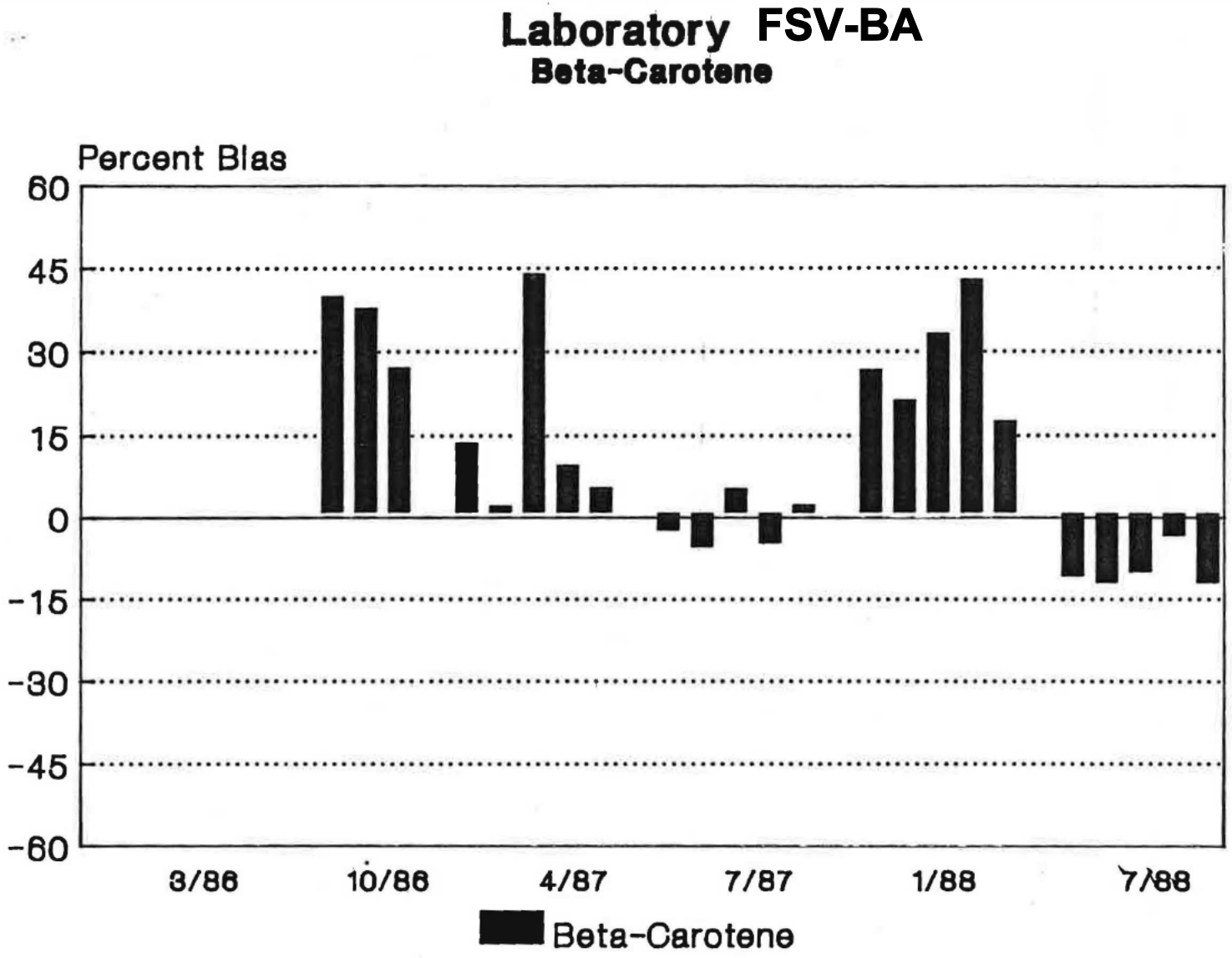








% Bias from Assigned Values



Appendix F. Updated "All-Lab Report" for RR13

The following three pages are an updated version of an "All-Lab" report for RR13. 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 RR13 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 "Assigned Value" 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 RR13. 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 XIII Laboratory Results

	Total Retinol					α-Tocopherol					Total β-Carotene					trans-β-Carotene					Total cis-β-Carotene				
Lab	90	91	92	93	94	90	91	92	93	94	90	91	92	93	94	90	91	92	93	94	90	91	92	93	94
FSV-BA	0.402	1.010	0.625	0.761	0.416	8.47	10.57	6.67	5.40	6.77	0.076	0.915	1.153	0.320	0.187	0.074	0.876	1.111	0.301	0.176	*0.002*	*0.039*	*0.042*	*0.019*	*0.011*
FSV-BD	0.367	1.096	0.610	0.673	0.372	6.13	8.39	5.75	4.79	6.62															
FSV-BE	0.436	1.214	0.796	0.892	0.640	8.30	10.55	6.95	5.00	7.35															
FSV-BF	0.421	1.155	0.650	0.859	0.487	6.50	8.84	5.51	4.72	5.35	0.089	0.950	1.190	0.337	0.190										
FSV-BG	0.454	1.169	0.649	0.873	0.483	7.37	8.73	5.59	4.75	5.62	0.079	1.113	1.403	0.365	0.194										
FSV-BH	0.382	1.005	0.582	0.752	0.428	7.00	9.30	5.60	5.00	5.80	0.105	1.140	1.450	0.470	0.230										
FSV-BI	0.396	0.990	0.574	0.732	0.415	6.40	8.69	5.45	4.54	5.64	0.072	0.836	1.111	0.288	0.176										
FSV-BX	0.430	1.150	0.580	0.720	0.430	6.92	8.99	5.47	4.44	5.58						*nq*	0.830	0.980	0.250	0.170					
FSV-BY	0.362	1.003	0.577	0.726	0.403	6.85	9.42	5.91	4.60	5.91	0.116	0.910	0.977	0.359	0.213										
FSV-BZ	0.380	0.880	0.530	0.650	0.420	6.90	10.40	6.80	5.90	6.50															
FSV-CA	0.401	1.015	0.623	0.687	0.420	6.94	9.49	6.08	4.88	6.13															
FSV-CJ	0.446	1.154	0.640	0.827	0.471	7.66	10.11	6.06	5.40	6.32	0.078	0.999	1.193	0.343	0.210										
FSV-CL	0.372	1.037	0.544	0.718	0.437	6.15	7.58	4.25	3.37	4.41	0.058	0.845	1.056	0.287	0.149										
FSV-CN	0.472	1.018	0.702	0.808	0.522	7.74	10.28	7.14	5.56	6.78	0.075	1.004	1.345	0.457	0.262										
FSV-CO	0.412	1.054	0.590	0.777	0.454	6.86	9.23	5.74	4.79	5.88	0.089	1.034	1.252	0.346	0.205										
FSV-CQ											0.061	0.904	0.982	0.253	0.145										
FSV-CY	0.450	0.920	0.560	0.700	0.470	6.81	8.97	5.35	4.72	5.84	0.170	0.880	1.100	0.350	0.250										
FSV-DC	0.385	1.058	0.575	0.770	0.428						0.061	0.904	0.982	0.253	0.145										
FSV-DE						7.11	9.30	5.88	4.80	5.86															
FSV-DG	0.400	1.090	0.600	0.780	0.430	7.00	10.76	6.91	4.60	5.89	0.080	1.120	1.410	0.360	0.200										
FSV-DN	0.430	1.004	0.580	0.734	0.436	7.84	9.84	6.15	5.02	6.36	0.122	1.440	1.830	0.475	0.282										
FSV-DO	0.375	0.975	0.562	0.787	0.450																				
FSV-DT	0.364	0.963	0.553	0.771	0.408	6.62	9.23	6.35	5.62	5.51															
FSV-DZ	0.346	0.927	0.564	0.744	0.375	6.41	9.39	6.35	5.99	5.85															
FSV-ED	0.416	1.037	0.610	0.712	0.437	7.19	9.62	6.24	5.25	6.25	0.097	0.928	1.183	0.328	0.202										
FSV-EF	0.320	0.890	0.490	0.660	0.360	6.93	9.30	5.99	4.82	6.85															
FSV-EG	0.388	1.044	0.568	0.748	0.443						0.071	0.962	1.193	0.274	0.167										
FSV-EN	0.350	0.958	0.518	0.716	0.361	6.20	7.14	4.58	4.09	5.11															
FSV-EO	0.369	0.958	0.449	0.766	0.410						0.076	0.920	1.133	0.346	0.192										
FSV-FE	0.372	1.035	0.588	0.749	0.419	7.39	9.51	6.51	5.42	6.68	0.119	1.099	1.385	0.327	0.271										
FSV-FI						7.61	9.99	6.07	5.11	6.18															
FSV-GH	0.388	0.914	0.517	0.823	0.452	7.17	7.82	5.03	5.67	6.10	0.050	0.612	0.896	0.269	0.165										
FSV-GI	0.389	0.866	0.515	0.832	0.445	6.35	7.82	4.71	4.93	6.06	0.058	0.645	0.965	0.225	0.141										
n	30	30	30	30	30	28	28	28	28	28	21	21	21	21	21	1	2	2	2	2	1	1	1	1	1
Min	0.320	0.866	0.449	0.650	0.360	6.13	7.14	4.25	3.37	4.41	0.050	0.612	0.896	0.225	0.141										
Mean	0.396	1.020	0.584	0.758	0.437	7.03	9.26	5.90	4.97	6.04	0.086	0.960	1.199	0.335	0.199	0.074	0.853	1.046	0.276	0.173	0.002	0.039	0.042	0.019	0.011
Max	0.472	1.214	0.796	0.892	0.640	8.47	10.76	7.14	5.99	7.35	0.170	1.440	1.830	0.475	0.282										
SD	0.035	0.090	0.065	0.061	0.053	0.61	0.92	0.72	0.55	0.60	0.028	0.174	0.217	0.069	0.042		0.033	0.093	0.036	0.004					
CV	9	9	11	8	12	9	10	12	11	10	33	18	18	20	21										
NISTa	0.392	1.008	0.589	0.668	0.403	7.33	9.73	6.05	4.75	6.15	0.068	1.048	1.263	0.252	0.197	0.062	1.013	1.200	0.234	0.178	*0.006*	*0.035*	*0.063*	*0.018*	*0.019*
Median	0.389	1.013	0.579	0.751	0.430	6.94	9.30	5.95	4.90	5.98	0.078	0.928	1.183	0.337	0.194										
eSD	0.033	0.077	0.047	0.050	0.030	0.64	0.82	0.62	0.45	0.52	0.025	0.113	0.240	0.042	0.040										
eCV	9	8	8	7	7	9	9	10	9	9	32	12	20	12	21										

F2

Round Robin XIII 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 XIII Laboratory Results

%Bias Summary

Lab	TR	aT	bC
FSV-BA	2±4	14±5	-3±1
FSV-BD	-3±10	-3±9	
FSV-BE	27±15	15±8	
FSV-BF	12±2	-7±3	3±6
FSV-BG	15±2	-3±5	10±9
FSV-BH	0±1	-1±3	28±9
FSV-BI	-1±2	-7±1	-10±3
FSV-BX	4±8	-6±4	
FSV-BY	-4±3	-2±3	9±25
FSV-BZ	-8±5	11±8	
FSV-CA	0±6	1±1	
FSV-CJ	12±2	7±4	4±4
FSV-CL	-2±4	-23±8	-17±7
FSV-CN	14±10	14±4	18±17
FSV-CO	4±2	-2±1	8±5
FSV-CQ			-18±9
FSV-CY	1±11	-4±3	28±52
FSV-DC	1±2		-18±9
FSV-DE		-1±2	
FSV-DG	4±3	5±10	10±9
FSV-DN	2±5	6±4	51±7
FSV-DO	0±4		
FSV-DT	-4±4	2±9	
FSV-DZ	-7±5	4±11	
FSV-ED	2±5	5±1	5±11
FSV-EF	-15±3	3±7	
FSV-EG	1±2		-7±10
FSV-EN	-9±5	-18±5	
FSV-EO	-7±9		-1±3
FSV-FE	-1±3	8±4	25±22
FSV-FI		5±3	
FSV-GH	-1±9	-2±14	-26±9
FSV-GI	-2±10	-9±10	-27±6
NISTa	-3±5	2±3	-3±16

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 "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-based reference value.

Appendix G. Shipping Package Inserts for RR14

The following two items were attached to each package shipped to an RR14 participant:

- **Cover letter**. The original letter as attached to the packages has been lost, page G2 reproduces the proof copy of the mail-merge letter form.

- **Datasheet**. Page G3 reproduces the form.

August 16, 1988

^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.

Five proficiency testing samples for fat-soluble vitamin analyses (Round Robin XIV) are being shipped to you on August 22 under a separate cover. These samples are freeze-dried plasma and should be reconstituted with 1.00 mL of water. You are to report only one value for each sample on the attached data entry form.

Data from Round-Robin XIII indicate that use of the value Assigned Control Materials provided to you in April, has resulted in a significant improvement in the quality of interlaboratory measurements. A copy of the letter that accompanied those samples and a Control Materials data form are enclosed. Analyze a set of the control materials between now and September 1, and concurrently with RR-XIV samples. Results from these analyses are to be provided to us along with RR-XIV data by October 1.

The 1988 NCI/NBS Micronutrient Analysis Workshop will be held on November 2 and 3 at the National Bureau of Standards in the Metrology Building, Room A340. Rooms have been set aside for this meeting at the Gaithersburg Quality Inn (301/963-5900) at the rate of $49/night. You must make your reservation before October 24 and mention you are attending the "Micronutrient Analysis Workshop" to get this special rate. A fee of $15 will be collected from each participant which will cover lunch and refreshments for both days.

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
R. Paule

REPORT ON NBS/NCI SAMPLES FROM LABORATORY # _____

DATE OF ANALYSIS _____

RESULTS IN mg/L

SAMPLE #	ANALYTE	RESULT	
SERUM 95	RETINOL		
VIAL # ___		All Trans	Total
	B-CAROTENE		
	A-TOCOPHEROL		
SERUM 96	RETINOL		
VIAL # ___		All Trans	Total
	B-CAROTENE		
	A-TOCOPHEROL		
SERUM 97	RETINOL		
VIAL # ___		All Trans	Total
	B-CAROTENE		
	A-TOCOPHEROL		
SERUM 98	RETINOL		
VIAL # ___		All Trans	Total
	B-CAROTENE		
	A-TOCOPHEROL		
SERUM 98	RETINOL		
VIAL # ___		All Trans	Total
	B-CAROTENE		
	A-TOCOPHEROL		

Reconstitute the Serum Sample with 1.0 mL Water.

Appendix H. Final Report for RR14

The following twelve pages are the the available components of the final report for RR14 as provided to participants. This report consisted of:

- An individualized cover letter and discussion. The original has been lost; however, page H2 displays a draft of the discussion of the history of the distributed samples.
  - Sera 96, 97, and 99 had been distributed in RR12 but with instructions to reconstitute with 1.2 mL water rather than the 1.0 mL specified for RR14.
  - Sera 95 and 98 are duplicates of a material originally distributed in RR13.
- Tables listing 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 every experienced participant's (volume-corrected) bias for all measurements of the four different sample materials, relative to an assigned value, 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 I provides a complete listing of the RR14 results where the original codes have been altered to ensure confidentiality. Appendix I also provides more relevant summary statistics.

NCI/NBS Round Robin XIV, Fat Soluble Vitamins.

Round Robin XIV consists of serum samples repeated from previous shipments, with one serum sample prepared in duplicate. Sera 66, 62, and 64 were numbered as sera 96, 97, and 99 respectively. The designated analyte values for these sera were assigned by reconstituting the samples with 1.2 mL of water. The instructions for this round robin are to reconstitute the samples with 1.0 mL water. The adjusted assigned values are listed in the table below.

The remaining serum samples in RR XIV consists of serum 92 renumbered in duplicate as sera 95 and 98. Serum 92 was originally prepared to be reconstituted with 1.0 mL. Thus, the resulting sera (95 and 98) are in smaller vials than 96, 97, and 99.

Table 1. RRXIV Analyte concentrations. (mg/L).

Analyte		Serum 95	Serum 96	Serum 97	Serum 98	Serum 99
Retinol	1.0 mL	0.577	0.952	0.376	0.577	0.683
	1.2 mL		0.793	0.313		0.569
Alpha-Tocopherol	1.0 mL	5.93	10.15	13.16	5.93	5.76
	1.2 mL		8.46	10.97		4.80
Total Beta-Carotene	1.0 mL	1.294	1.621	0.175	1.294	0.083
	1.2 mL		1.351	0.146		0.069
Trans-Beta-Carotene	1.0 mL	1.161	1.452	0.164	1.161	0.084
	1.2 mL		1.210*	0.137*		0.070*

* NBS Value used.

ROUND-ROBIN XIV

TABULAR DATA

Lab Results Round Robin XIV.

Retinol

Lab #		Serum 95	Serum 98	Serum 96	Serum 97	Serum 99
		0.546	0.533	0.909	0.366	0.658
		0.553	0.583	0.843	0.353	0.632
		0.616	0.652	1.021	0.386	0.728
		0.570	0.572	0.920	0.337	0.658
L	*	0.531	0.537	0.810	0.259	0.537
L	*		0.579	0.968	0.325	0.628
		0.625	0.725	1.000	0.375	0.775
		0.548	0.564	0.869	0.334	0.654
		0.573	0.572	0.902	0.363	0.658
		0.588	0.530	0.876	0.331	0.639
		0.675	0.578	0.835	0.337	0.577
		0.561	0.550	0.809	0.369	0.650
		0.554	0.551	0.896	0.335	0.638
		0.542	0.593	0.890	0.349	0.671
		0.729	0.670	1.112	0.388	0.739
		0.600	0.610	0.970	0.390	0.720
		0.595	0.584	0.915	0.339	0.684
		0.590	0.576	0.967	0.350	*0.35
		*0.846	*0.535	*0.891	*0.636	*0.632
		0.585	0.580	0.835	0.282	0.554
		0.490	0.440	0.870	0.300	0.560
		0.570	0.580	0.920	0.340	0.600
		0.567	0.590	0.874	0.364	0.645
		0.653	0.641	0.986	0.379	0.677
		0.643	0.630	1.013	0.383	0.710
AVG		0.584	0.582	0.912	0.349	0.655
Previous AVG		0.582	0.582	0.930	0.359	0.648
SD		0.051	0.057	0.073	0.028	0.059
Rel% SD		8.7	9.8	8.0	7.9	8.9
Assigned Value		0.577	0.577	0.952	0.376	0.683
%Bias		1.2	0.8	-4.2	-7.1	-4.1

* = Values not included in the Average.
L = Late results not included in the Average.
% Bias = The Average compared to the Assigned Value.

Percent Bias from Assigned Value.

Retinol

Lab #		Serum 95	Serum 98	Serum 96	Serum 97	Serum 99
		-5.4	-7.6	-4.5	-2.7	-3.7
		-4.2	1.0	-11.4	-6.1	-7.5
		6.8	13.0	7.2	2.7	6.6
		-1.2	-0.9	-3.4	-10.4	-3.7
L	*	-8.0	-6.9	-14.9	-31.1	-21.4
L	*		0.3	1.7	-13.6	-8.1
		8.3	25.6	5.0	-0.3	13.5
		-5.0	-2.3	-8.7	-11.2	-4.2
		-0.7	-0.9	-5.3	-3.5	-3.7
		1.9	-8.1	-8.0	-12.0	-6.4
		17.0	0.2	-12.3	-10.4	-15.5
		-2.8	-4.7	-15.0	-1.9	-4.8
		-4.0	-4.5	-5.9	-10.9	-6.6
		-6.1	2.8	-6.5	-7.2	-1.8
		26.3	16.1	16.8	3.2	8.2
		4.0	5.7	1.9	3.7	5.4
		3.1	1.2	-3.9	-9.8	0.1
		2.3	-0.2	1.6	-6.9	-48.8
		46.6	-7.3	-6.4	69.1	-7.5
		1.4	0.5	-12.3	-25.0	-18.9
		-15.1	-23.7	-8.6	-20.2	-18.0
		-1.2	0.5	-3.4	-9.6	-12.2
		-1.7	2.3	-8.2	-3.2	-5.6
		13.2	11.1	3.5	0.7	-1.0
		11.4	9.2	6.4	1.8	4.0
Assigned Value		0.577	0.577	0.952	0.376	0.683

Lab Results Round Robin XIV.

Alpha-Tocopherol

Lab #	Serum 95	Serum 98	Serum 96	Serum 97	Serum 99
	6.04	5.65	9.32	11.01	5.41
	5.89	5.79	9.49	12.02	5.50
	4.93	5.37	9.34	11.58	4.98
L *	5.76	5.95	8.16	11.97	5.27
L *		5.71	9.33	11.66	5.10
	5.99	6.07	10.17	12.63	5.94
	5.67	5.63	9.21	11.72	5.32
	7.00	5.80	9.30	13.40	5.90
	5.50	5.60	9.10	11.60	5.10
	5.57	6.16	8.74	11.34	5.47
	5.19	5.63	9.16	11.00	4.89
	4.90	4.95	7.91	10.10	4.67
	5.72	5.82	9.67	11.84	5.43
	5.95	5.95	9.13	8.48	4.16
	5.92	6.01	9.76	12.13	5.52
	*3.14	6.17	8.17	12.78	5.61
	*11.19	*10.99	*10.16	*12.69	*7.28
	6.02	6.11	9.77	11.60	5.72
	5.30	6.10	10.80	13.20	6.40
	5.36	5.32	9.22	11.81	4.58
	5.51	5.51	9.17	12.02	5.12
	5.99	5.95	9.78	12.11	5.16
	5.71	5.78	9.74	12.33	5.48
AVG	5.67	5.76	9.30	11.68	5.32
Previous AVG	5.99	5.99	10.62	13.00	5.83
SD	0.50	0.33	0.65	1.13	0.54
Rel% SD	8.8	5.8	7.0	9.7	10.1
Assigned Value	5.93	5.93	10.15	13.16	5.76
%Bias	-4.3	-2.9	-8.4	-11.2	-7.7

* = Values not included in the Average.
L = Late results not included in the Average.
% Bias = The Average compared to the Assigned Value.

Percent Bias from Assigned Value.

Alpha-Tocopherol

Lab #	Serum 95	Serum 98	Serum 96	Serum 97	Serum 99
	1.9	-4.7	-8.2	-16.3	-6.1
	-0.7	-2.4	-6.5	-8.7	-4.5
	-16.9	-9.4	-8.0	-12.0	-13.5
L *	-2.9	0.3	-19.6	-9.0	-8.5
L *		-3.7	-8.1	-11.4	-11.5
	1.0	2.4	0.2	-4.0	3.1
	-4.4	-5.1	-9.3	-10.9	-7.6
	18.0	-2.2	-8.4	1.8	2.4
	-7.3	-5.6	-10.3	-11.9	-11.5
	-6.1	3.9	-13.9	-13.8	-5.0
	-12.5	-5.1	-9.8	-16.4	-15.1
	-17.4	-16.5	-22.1	-23.3	-18.9
	-3.5	-1.9	-4.7	-10.0	-5.7
	0.3	0.3	-10.0	-35.6	-27.8
	-0.2	1.3	-3.8	-7.8	-4.2
	-47.0	4.0	-19.5	-2.9	-2.6
	88.7	85.3	0.1	-3.6	26.4
	1.5	3.0	-3.7	-11.9	-0.7
	-10.6	2.9	6.4	0.3	11.1
	-9.6	-10.3	-9.2	-10.3	-20.5
	-7.1	-7.1	-9.7	-8.7	-11.1
	1.1	0.4	-3.6	-8.0	-10.4
	-3.7	-2.5	-4.0	-6.3	-4.9
Assigned Value	5.93	5.93	10.15	13.16	5.76

Lab Results Round Robin XIV.

Total Beta-Carotene

Lab #		Serum 95	Serum 98	Serum 96	Serum 97	Serum 99
		1.508	1.370	1.792	0.174	0.077
		1.147	1.167	1.488	0.150	0.068
		1.322	1.295	1.827	0.179	0.083
		1.380	1.304	1.859	0.188	*0.15
L	*	1.194	1.157	1.549	0.178	0.084
L	*	1.235	1.213	1.593	0.192	0.092
		1.149	1.165	1.555	0.191	0.087
		1.550	1.650	1.720	0.180	0.083
		1.075	1.204	1.590	0.151	0.101
		1.377	1.403	1.864	0.174	0.082
		1.290	1.310	1.740	0.180	0.080
		1.169	1.159	1.573	0.182	0.080
		1.198	1.165	1.623	0.174	0.085
		1.274	1.247	1.698	0.184	0.095
		1.280	1.250	1.780	0.190	0.090
		1.273	1.217	1.640	0.160	0.072
		1.279	1.292	1.807	0.179	0.083
AVG		1.286	1.284	1.701	0.177	0.084
Previous AVG		1.216	1.216	1.789	0.186	0.080
SD		0.142	0.136	0.124	0.013	0.009
Rel% SD		11.0	10.6	7.3	7.3	10.1
Assigned Value		1.294	1.294	1.621	0.175	0.083
%Bias		-0.6	-0.8	4.9	1.0	1.5

* = Values not included in the Average.
L = Late results not included in the Average.
% Bias = The Average compared to the Assigned Value.

Percent Bias from Assigned Value.

Total Beta-Carotene

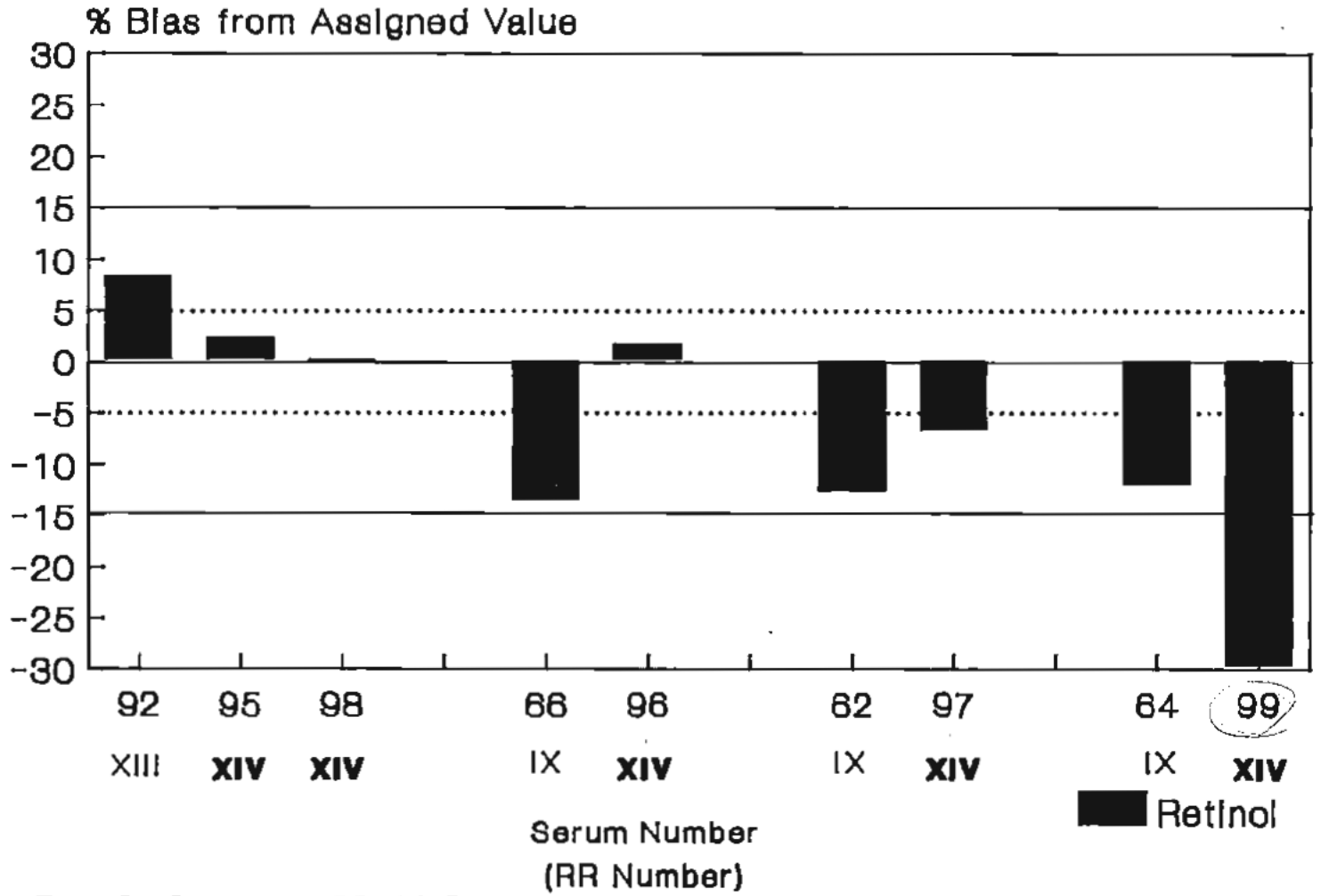
Lab #	Serum 95	Serum 98	Serum 96	Serum 97	Serum 99
	16.5	5.9	10.5	-0.6	-7.2
	-11.4	-9.8	-8.2	-14.3	-18.1
	2.2	0.1	12.7	2.3	0.0
	6.6	0.8	14.7	7.4	80.7
L *	-7.7	-10.6	-4.4	1.7	1.2
L *	-4.6	-6.3	-1.7	9.7	10.8
	-11.2	-10.0	-4.1	9.1	4.8
	19.8	27.5	6.1	2.9	0.0
	-16.9	-7.0	-1.9	-13.7	21.7
	6.4	8.4	15.0	-0.6	-1.2
	-0.3	1.2	7.3	2.9	-3.6
	-9.7	-10.4	-3.0	4.0	-3.6
	-7.4	-10.0	0.1	-0.6	2.4
	-1.5	-3.6	4.8	5.1	14.7
	-1.1	-3.4	9.8	8.6	8.4
	-1.6	-6.0	1.2	-8.9	-13.4
	-1.1	-0.2	11.5	2.3	-0.1
Assigned Value	1.294	1.294	1.621	0.175	0.083

ROUND-ROBIN XIV

GRAPHICAL DATA

Laboratory FSV-BA

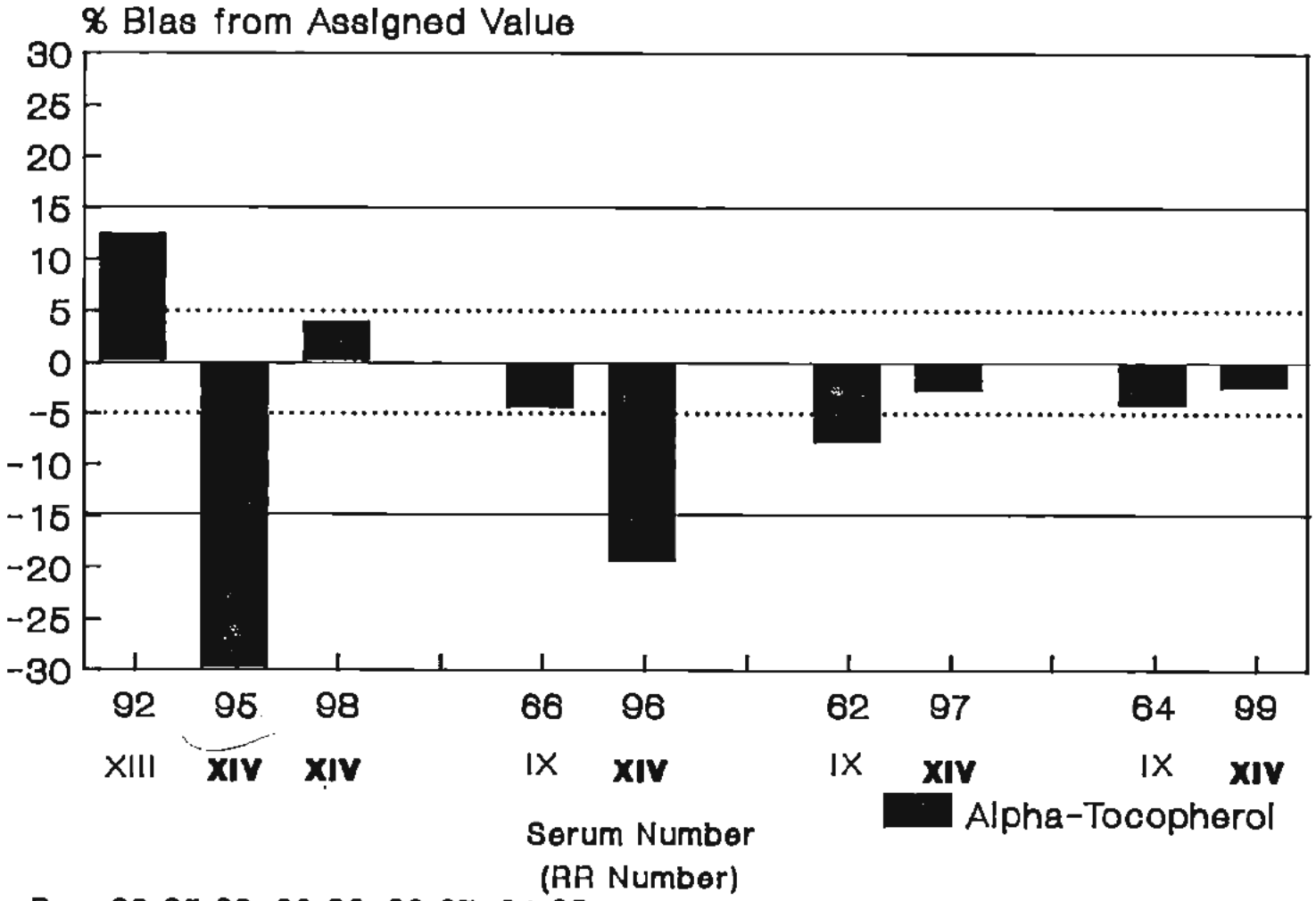
Retinol RR XIV Results



Sera Dup. 92 95 98; 66 96; 62 97; 64 99.

Laboratory FSV-BA

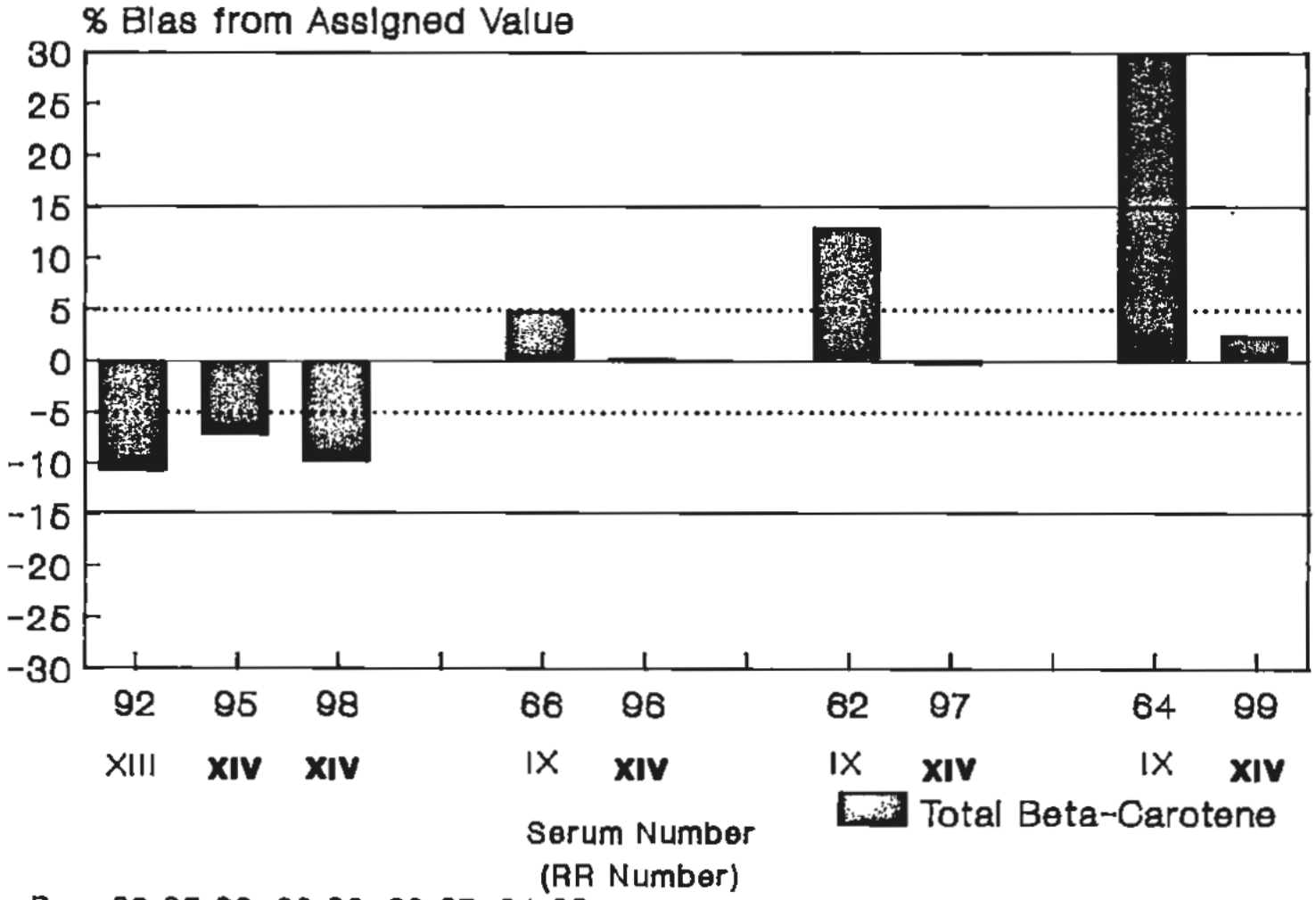
Alpha-Tocopherol RR XIV Results



Sera Dup. 92 95 98; 66 96; 62 97; 64 99.

Laboratory FSV-BA

Beta-Carotene RR XIV Results



Sera Dup. 92 95 98; 66 96; 62 97; 64 99.

Appendix I. Updated "All-Lab Report" for RR14

The following three pages are an updated version of an "All-Lab" report for RR14. 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 RR14 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 "Assigned Value" 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 RR14. 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 XIV Laboratory Results

	Total Retinol					α-Tocopherol					Total β-Carotene					trans-β-Carotene					Total cis-β-Carotene				
Lab	95	96	97	98	99	95	96	97	98	99	95	96	97	98	99	95	96	97	98	99	95	96	97	98	99
FSV-BA	0.590	0.967	0.350	0.576	0.350	3.14	8.17	12.78	6.17	5.61	1.198	1.623	0.174	1.165	0.085	1.120	1.500	0.157	1.086	0.076	*0.078*	*0.123*	*0.017*	*0.079*	*0.009*
FSV-BD	0.588	0.876	0.331	0.530	0.639	7.00	9.30	13.40	5.80	5.90															
FSV-BE	0.729	1.112	0.388	0.670	0.739	5.95	9.13	8.48	5.95	4.16															
FSV-BF	0.554	0.896	0.335	0.551	0.638	5.19	9.16	11.00	5.63	4.89	1.377	1.864	0.174	1.403	0.082										
FSV-BG	0.616	1.021	0.386	0.652	0.728	4.93	9.34	11.58	5.37	4.98	1.322	1.827	0.179	1.295	0.083										
FSV-BH	0.571	0.812	0.308	0.567	0.616	5.50	9.20	11.70	5.60	5.20	1.289	1.746	0.170	1.282	0.075	1.218	1.644	0.160	1.207	0.072	*0.071*	*0.102*	*0.010*	*0.075*	*0.003*
FSV-BI	0.553	0.843	0.353	0.583	0.632	5.89	9.49	12.02	5.79	5.50	1.147	1.488	0.150	1.167	0.068										
FSV-BY	0.546	0.909	0.366	0.533	0.658	6.04	9.32	11.01	5.65	5.41	1.508	1.792	0.174	1.370	0.077										
FSV-BZ	0.490	0.870	0.300	0.440	0.560	5.30	10.80	13.20	6.10	6.40															
FSV-CA	0.548	0.869	0.334	0.564	0.654	5.99	10.17	12.63	6.07	5.94															
FSV-CJ	*na*	0.968	0.325	0.579	0.628	*na*	9.33	11.66	5.71	5.10	1.235	1.593	0.192	1.213	0.092										
FSV-CJ	0.531	0.810	0.259	0.537	0.537	5.76	8.16	11.97	5.95	5.27	1.194	1.549	0.178	1.157	0.084										
FSV-CN	0.561	0.809	0.369	0.550	0.650	5.57	8.74	11.34	6.16	5.47	1.075	1.590	0.151	1.204	0.101										
FSV-CO	0.573	0.902	0.363	0.572	0.658	5.67	9.21	11.72	5.63	5.32	1.149	1.555	0.191	1.165	0.087										
FSV-CY	0.570	0.920	0.340	0.580	0.600	5.36	9.22	11.81	5.32	4.58	1.280	1.780	0.190	1.250	0.090										
FSV-DC	0.595	0.915	0.339	0.584	0.684						1.169	1.573	0.182	1.159	0.080										
FSV-DE						5.72	9.67	11.84	5.82	5.43															
FSV-DG	0.600	0.970	0.390	0.610	0.720	5.92	9.76	12.13	6.01	5.52	1.290	1.740	0.180	1.310	0.080										
FSV-DN	0.675	0.835	0.337	0.578	0.577	5.50	9.10	11.60	5.60	5.10	1.550	1.720	0.180	1.650	0.083										
FSV-DO	0.625	1.000	0.375	0.725	0.775																				
FSV-DT	0.542	0.890	0.349	0.593	0.671	4.90	7.91	10.10	4.95	4.67															
FSV-DZ	0.846	0.891	0.636	0.535	0.632	11.19	10.16	12.69	10.99	7.28															
FSV-ED	0.585	0.835	0.282	0.580	0.554	6.02	9.77	11.60	6.11	5.72	1.274	1.698	0.184	1.247	0.095										
FSV-EF	0.567	0.874	0.364	0.590	0.645	5.51	9.17	12.02	5.51	5.12															
FSV-EG	0.570	0.920	0.337	0.572	0.658						1.380	1.859	0.188	1.304	0.150										
FSV-GH	0.643	1.013	0.383	0.630	0.710	5.71	9.74	12.33	5.78	5.41	1.279	1.807	0.179	1.292	0.083										
FSV-GI	0.653	0.956	0.379	0.642	0.677	5.99	9.78	12.11	8.95	5.15	1.273	1.640	0.160	1.217	0.072										
n	25	26	26	26	26	23	24	24	24	24	18	18	18	18	18	2	2	2	2	2	2	2	2	2	2
Min	0.490	0.809	0.259	0.440	0.350	3.14	7.91	8.48	4.95	4.16	1.075	1.488	0.150	1.157	0.068										
Average	0.597	0.911	0.357	0.582	0.638	5.82	9.33	11.78	6.11	5.38	1.277	1.691	0.176	1.269	0.087	1.169	1.572	0.159	1.147	0.074	0.074	0.113	0.014	0.077	0.006
Max	0.846	1.112	0.636	0.725	0.775	11.19	10.80	13.40	10.99	7.28	1.550	1.864	0.192	1.650	0.150										
SD	0.072	0.074	0.066	0.054	0.082	1.36	0.65	1.00	1.26	0.62	0.122	0.118	0.012	0.120	0.018										
CV	12.1	8.1	18.4	9.2	12.8	23.3	7.0	8.5	20.6	11.5	9.5	7.0	7.0	9.5	20.3										
Median	0.573	0.899	0.350	0.579	0.648	5.71	9.31	11.83	5.80	5.36	1.277	1.709	0.179	1.249	0.083										
eSD	0.037	0.083	0.028	0.031	0.045	0.41	0.47	0.59	0.30	0.38	0.120	0.159	0.009	0.087	0.007										
eCV	6.5	9.3	8.1	5.4	6.9	7.3	5.0	5.0	5.2	7.0	9.4	9.3	5.0	6.9	8.9										

Round Robin XIV 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
na	Not reported

Round Robin XIV Laboratory Results

%Bias Summary

Lab	TR	aT	bC
FSV-BA	-1±5	-1±5	5±10
FSV-BD	-2±3	2±1	-13±5
FSV-BE	11±2	-6±5	3±3
FSV-BF	0±2		21±33
FSV-BG	-1±6	-2±2	1±8
FSV-BH	-13±8	-2±6	-5±5
FSV-BI	15±8		
FSV-BY	-3±2	7±3	
FSV-BZ	1±2	-1±1	-3±8
FSV-CA	-3±4	9±10	
FSV-CJ	-1±11	-3±1	11±15
FSV-CJ	-3±2	-6±3	5±7
FSV-CN	0±4	-14±1	
FSV-CO		1±2	
FSV-CY	18±7	-9±15	
FSV-DC	8±3	4±1	1±3
FSV-DE	2±3		-5±4
FSV-DG	-7±22	-8±22	-4±4
FSV-DN	24±39	48±43	
FSV-DO	-8±9	4±3	3±7
FSV-DT	-6±5	-2±1	-2±5
FSV-DZ	-14±7	9±10	
FSV-ED	-2±4	-6±6	4±4
FSV-EF	0±3	-3±3	
FSV-EG	-2±6	-1±5	-4±15
FSV-GH	11±2	2±2	2±3
FSV-GI	9±4	13±24	-6±6

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 "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-based reference value.