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Results for Round Robins I, II, III and V Fat-Soluble Vitamins and Carotenoids in Human Serum

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National Institute of Standards and Technology
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February 2019

U.S. Department of Commerce
Wilbur L. Ross, Jr., Secretary

National Institute of Standards and Technology
Walter Copan, NIST Director and Under Secretary of Commerce for Standards and Technology

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Abstract

From 1984 to 2017, the National Institute of Standards and Technology (NIST) and its precursor the National Bureau of Standards (NBS) coordinated what became 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 1984 and 1985 MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum measurement comparability improvement studies: Round Robins I, II, III, and V (selenium and zinc were the only analytes in Round Robin IV). The test samples were: calibration solutions in Round Robins I and II, fortified sera in Round Robin III, and natural plasma in Round Robin V. Participant results were received for: Round Robin I between November 11 and December 24, 1984; Round Robin II between January 14 and February 19, 1985; Round Robin III between March 2 and May 30, 1985; and Round Robin V between August 7 and December 12, 1985.

Keywords

Calibration Solutions, Human Serum
Retinol, α-Tocopherol, β-Carotene

Table of Contents

Introduction

From 1984 to 2017, the National Institute of Standards and Technology (NIST), formerly the National Bureau of Standards (NBS), coordinated what became the Micronutrients Measurement Quality Assurance Program (MMQAP) for laboratories that measure fat-soluble vitamins and carotenoids in human serum and plasma. The MMQAP provided 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, and *trans*- and total beta-carotene) and performance-evaluation standards were distributed by NIST to laboratories for analysis.

Participants used the methodology of their choice to determine analyte content in the control and study materials. Participants provided their data to NIST, where it was compiled and evaluated for trueness relative to the NIST value, within-laboratory precision, and concordance within the participant community. NIST provided the participants with a technical summary report concerning their performance for each exercise and suggestions for methods development and refinement. Participants who had concerns regarding their laboratory's performance were encouraged to consult with the MMQAP coordinators.

All MMQAP interlaboratory studies consisted of individual units of batch-prepared samples that were distributed to each participant. For historical reasons these studies are referred to as "Round Robins". The MMQAP and the nature of its studies are described in [1]. Results of MMQAP studies through 1993 are collected in NIST Special Publication 874 [2].

Round Robin I: Fat-Soluble Vitamins and Carotenoids in Calibration Solutions

Two ampoules of each of six solutions (solutions 5 to 10) were distributed to 22 participants in the fat-soluble vitamins and carotenoids component of the Round Robin I comparability study (hereafter referred to as RR01). The solutions 5, 6, and 7 were in an ethanol matrix; solutions 8, 9, 10 were in a hexane matrix. These solutions were shipped to participants in November 1984. Participants were requested to report two values for retinol, α-tocopherol, and β-carotene. The isomeric form of the β-carotene, total or *trans*, was not specified. Not all participants reported values for all target analytes.

Five participants in the ascorbic acid component of RR01 received three solutions (solutions 11, 12, and 13) prepared in 3:1 acetonitrile:water with dithiothreitol as an anti-oxidant. In the selenium and zinc component, 16 participants received three solutions (14, 15, and 16) in 0.1 % aqueous nitric acid. The results for these materials are not discussed in this document.

Our records for this study are incomplete. Appendix A presents 1) a fragmentary cover letter describing the test samples and calibration solutions and 2) an example of the data report form. Appendix B reproduces the data and its summary provided to all participants. Appendix C lists the fat-soluble vitamin and carotenoid measurement results reported for RR01 in a more accessible format.

Round Robin II: Fat-Soluble Vitamins and Carotenoids in Calibration Solutions

Two ampoules of each of four ethanolic solutions (solutions 5, 17, 18, and 19) were distributed to 21 participants in the fat-soluble vitamins and carotenoids component of the Round Robin II comparability study (hereafter referred to as RR02). The solutions 5 and 17 were value-assigned calibrants; solutions 18 and 19 were test samples. Participants were requested to report two values for retinol, α-tocopherol, and β-carotene in the solution 18 and 19 test samples, using two calibration approaches: 1) solutions 5 and 17 and 2) the participants own "in-house" calibrants. The isomeric form of the β-carotene, total or *trans*, was not specified. Not all participants reported values for all target analytes. These solutions were shipped to participants in January 1985.

Eight participants in the ascorbic acid component of RR02 received four solutions (solutions 11, 13, 20, and 13) prepared in 3:1 acetonitrile:water with dithiothreitol as an anti-oxidant. The solutions 11 and 13 were value-assigned calibrants; solutions 20 and 21 were test samples. Participants were requested to report two values for ascorbic acid in the solution 20 and 21 test samples, using two calibration approaches: 1) solutions 11 and 13 and 2) the participants own "in-house" calibrants. The results for these materials are not discussed in this document.

Our records for this study are incomplete. Appendix D presents 1) a fragmentary cover letter describing the test samples and calibration solutions and 2) an example of the data report form. Appendix E reproduces the data and its summary provided to all participants. Appendix F lists the fat-soluble vitamin and carotenoid measurement results reported for RR02 in a more accessible format.

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

Four human serum test samples (sera 6 to 9) were distributed to 20 participants in the fat-soluble vitamins and carotenoids component of Round Robin III (hereafter referred to as RR03). Participants were requested to report values for retinol, α-tocopherol, total and β-carotene, and other related analytes they wished to report. The isomeric form of the β-carotene, total or *trans*, was not specified. Not all participants reported values for all target analytes. These sample materials were shipped to participants in February 1985.

Five participants in the ascorbic acid component of RR03 received three human serum test samples (sera 10, 11, and 12). The results for these materials are not discussed in this document.

Our records for this study are incomplete. Appendix G presents an example of the data report form. Appendix H reproduces a letter sent to all participants that describes the tabular analysis for retinol, α-tocopherol, and β-carotene, the data, and statistical summary tables. Appendix I lists the measurement results reported for RR03 in a more accessible format.

Round Robin IV: Selenium and Zinc in Human Serum

The only analytes in MMQAP Round Robin IV were selenium and zinc. The 14 participants each received three human serum test samples (sera 16, 17, and 18). The results from this study are not discussed in this document.

Round Robin V: Fat-Soluble Vitamins and Carotenoids in Human Plasma

One ampoule of three liquid-frozen human plasma test samples (sera 19 to 21) and one ampoule of two three-component ethanolic calibrations solutions were distributed to 26 participants in the fat-soluble vitamins and carotenoids component of Round Robin V (hereafter referred to as RR05). While named as "Sera", the matrix for the three test samples was citrated plasma rather than heparin-treated serum. The sample materials were prepared from blood donated by three individuals who had consumed vitamins A and C supplements, vitamin C supplements, and carrots and β-carotene supplements, respectively. In addition to the native level, Serum 20 was spiked with ascorbic acid and ≈0.95 mg/L of dithiothreitol as stabilizer.

Participants were requested to separately report values for retinol, α-tocopherol, and/or β-carotene as calibrated with their own standards and as calibrated with the NBS-provided calibration solutions. The isomeric form of the β-carotene, total or *trans*, was not specified. Not all participants reported values for all target analytes. These sample materials were shipped to participants in August 1985.

Seven participants in the ascorbic acid component of RR05 received one ampoule of Serum 20 and one ampoule of two acetonitrile-water calibration solutions. The ascorbic acid results are not discussed in this document.

Appendix J reproduces the cover letter included in the sample shipment and the two data report forms. Appendix K reproduces the final report as provided to the RR05 participants. Appendix K also reproduces a discussion of a graphical analysis technique that was prepared as part of the Round Robin VI report. Appendix L lists the measurement results reported for RR05 in a more accessible format.

Round Robin VI: Selenium and Zinc in Human Serum

The only analytes in MMQAP Round Robin VI were selenium and zinc. The 13 participants each received six lyophilized human serum test samples (sera 22 to 27). Sera 25, 26, and 27 are the same materials distributed as sera 16, 17, and 18. The results from this study are not discussed in this document.

References

1 Duewer DL, Brown Thomas J, Kline MC, MacCrehan WA, Schaffer R, Sharpless KE, May WE, Crowell JA. NIST/NCI Micronutrients Measurement Quality Assurance Program: Measurement Repeatabilities and Reproducibilities for Fat-Soluble Vitamin-Related Compounds in Human Sera. Anal Chem 1997;69(7):1406-1413.

2 Thomas JB, Sharpless KE, eds. Methods for Analysis of Cancer Chemopreventive Agents in Human Serum. NIST Special Publication 874, NIST, 1995. https://nvlpubs.nist.gov/nistpubs/Legacy/SP/nistspecialpublication874.pdf

Appendix A. Shipping Package Inserts for RR01

Two items were attached to each package shipped to an RR01 participant:

- **Cover letter**. Unfortunately, the original letter has been lost. It would have described: the six gravimetrically prepared solutions (solutions 5 to 10), how they were to be analyzed, and how the results were to be reported. Based upon comments in the "brief commentary" provided to participants (see Appendix B), many participants did not fully understand what was expected of them.

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

Report on NBS/NCI Samples from Laboratory #____

Date of Analysis _____

Results in mg/L

Samples		Result 1	Result 2
Solution 5 #____	β-carotene		
	retinol		
	α-tocopherol		
Solution 6 #____	β-carotene		
	retinol		
	α-tocopherol		
Solution 7 #____	β-carotene		
	retinol		
	α-tocopherol		
Solution 8 #____	β-carotene		
	retinol		
	α-tocopherol		
Solution 9 #____	β-carotene		
	retinol		
	α-tocopherol		
Solution 10 #____	β-carotene		
	retinol		
	α-tocopherol		

Appendix B. Final Report for RR01

The following 14 pages present the following:

- Page B2: A “Call for Results” letter.
- Page B3: Cover letter.
- Page B4: “Comments on the Tables of Individual Laboratory Results”.
- Page B5: “Explanation of Column Heading on Statistical Summary”.
- Page B6: “Statistical Summary”.
- Pages B7 to B14: Individual results.

The ascorbic acid information provided in the original tables has been deleted. The format of the tables has been edited to improve readability.

UNITED STATES DEPARTMENT OF COMMERCE
National Bureau of Standards
Washington, D.C. 20234

December 24, 1984

Dear

Those of you who returned results on time for the analyses of Solutions 5 through 16 will be disappointed to see that this is not a report on the cumulative results on those samples. Only half of the results were here in the hoped-for 2 weeks after the samples were sent to you. The more tardy results were increasingly caught in the heavy crush of Christmas mail, some arriving in letters postmarked 10 days before being received. Now with the Christmas-to-New Years week ahead, a time many of us at NBS use for vacation, that report is delayed even more. Meanwhile, the results are being entered into the computer.

While the majority of labs reported two results per solution, as requested, others gave only single results: averages or single determinations per solution. As a consequence, some data will not be considered for statistical treatment, but that will not greatly effect the interlaboratory comparisons, as I believe you will see when the report is in your hands.

Please hereafter, conform to our requests and report back to us on time!

Now about what is ahead. Since the trace metal results look almost uniformly good, but not the vitamin results, we expect to complete this study of simple solutions with one more round of similar samples for the vitamins. We will send some solutions having assigned values and others as unknowns, and ask you to obtain values for the unknowns from use of your own standards and, separately, the "knowns" we send. This will help to differentiate whether interlab differences are due to problems with standards or methods. We expect to send these new samples early in January. About 2 weeks after that we intend to to send samples having a serum matrix. We will notify you when to expect each of these shipments.

We realize that the meeting that we thought to hold here on February 6 and 7, 1985, is coming too soon to be as beneficial as we would want, because of insufficient interlaboratory results. Therefore, the meeting dates have been put off until April 11 and 12, 1985.

With best wishes.

Sincerely,

Robert Schaffer
Supervisory Research Chemist
Organic Analytical Research Division

February 1, 1985

Dear

Dr. Schaffer is out ill, and I am writing to you on his behalf. Enclosed is a statistical summary and brief commentary on the data for Study I of the NBS/NCI micronutrient QA program. We apologize for the lengthy delay in returning the data summary to you and will strive in the future to have our statistical analysis completed and returned to you within three weeks of the final data submission date.

We have received approximately one-third of the results from Study II. Tardy results were one of the contributing factors to the delay in returning the statistical summaries from Study I to you. In the future, in order to be fair to those who submit data in a timely fashion, we will not include any data in our summary that is received later than three weeks after samples are distributed.

As Dr. Schaffer mentioned to you earlier, we have a program workshop tentatively scheduled for April 11-12, 1985. We consider this meeting as being very essential to the establishment of an effective program--one that is responsive to the needs of both the sponsor (NCI) and you, the participants. It is our understanding that NCI will allow grantee laboratories to support their travel to this meeting out of their grants. The preliminary agenda for the meeting is shown in Attachment A. We will gladly accept suggestions on additional topics for discussion. Please communicate your interest in attending this meeting to Dr. Schaffer in writing or to Margaret Hunt or Beverly Stevenson by telephone on 301/921-3778 before February 15.

We have prepared a pilot batch of fortified serum and are currently examining analyte stability. If all goes well, we will distribute our first serum samples on or about February 18. We look forward to continued and mutually beneficial collaboration with you in this program.

Sincerely,

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

Enclosures

Comments on Tables of Individual Laboratory Results for Each Analyte, by Solution Number

1. Only the results received by 21 December 1984 are included in these tables. This was an arbitrary cut-off date, but it was many days after we had indicated the deadline would be. As our working together becomes more routine, we expect this problem to diminish.

2. All the results received by that date are listed; that is, up to two results per ampoule. If a laboratory analyzed the samples by more than one method, the laboratory identification number is preceded by a letter, and the results were treated as if they were from another laboratory.

3. Some laboratories analyzed both of the ampoules for the same solution; other laboratories, just one. The results for only one ampoule were accepted for consideration by the statistical analysis. Under the column heading USED, the results accepted for consideration are listed as YES or OUT (outliers); the nonaccepted as NFI (not following instructions).

4. The results accepted for consideration, as defined in #3 above, were inspected for obvious outliers. If standard statistical tests for outliers had been employed, more of the results would have been classified as outliers (OUT under the heading USED). About 10-25 percent were considered outliers.

5. Some laboratories reported only one value instead of two. We asked for duplicate results in order to be able to estimate within-laboratory as well as between-laboratory precision. Under the USED column, single results are shown at NFI.

6. The column headings for the tables of individual laboratory results should need little explanation. Laboratory identification numbers run from to The last listing is NBS. The listed standard deviations are for the laboratory mean values. The grand averages are from the statistical summary, and the biases are calculated from the grand averages.

Explanation of Column Heading on Statistical Summary

GRAND AVG = grand average (consensus value from analysis of variance) = $\bar{\bar{x}}$ (mg/L)

S.E.AVG = one standard deviation (std. dev.) of $\bar{\bar{x}}$ = $S_{\bar{\bar{x}}}$

S WITHIN = within laboratory component of std. dev. = S_w (square root of within laboratory component of variance from the ANOVA; a pooled estimate of within laboratory imprecision)

S BETWEEN = between laboratory component of standard deviation = S_b

% CV AVG = percent coefficient of variation of the grand average = 100 $S_{\bar{\bar{x}}}/\bar{\bar{x}}$

% CV X = percent coefficient of variation of a single measurement made by a single laboratory = 100 $S_x/\bar{\bar{x}}$, where $S_x = \sqrt{S_w^2 + S_b^2}$ (NOTE: S_x is not equal to the std. dev. of all measurements)

FR REJECT = ratio of rejected to total laboratory results considered for statistical evaluation

STATISTICAL SUMMARY

ANALYTE=RETINOL

SOL	GRAND AVG	S.E. AVG	S WITHIN	S BETWEEN	% CV AVG	% CV X	FR REJECT
005	0.824	0.017	0.040	0.047	2.0	7.5	2/13
006	2.115	0.062	0.070	0.198	2.9	9.9	2/13
007	3.336	0.093	0.078	0.302	2.8	9.4	2/13
008	0.843	0.050	0.067	0.113	5.9	15.6	0/ 6
009	2.545	0.116	0.102	0.275	4.6	11.5	0/ 6
010	3.568	0.241	0.128	0.584	6.8	16.8	0/ 6

ANALYTE=A-TOCOPHEROL

SOL	GRAND AVG	S.E. AVG	S WITHIN	S BETWEEN	% CV AVG	% CV X	FR REJECT
006	3.323	0.152	0.124	0.421	4.6	13.2	1/ 9
005	9.261	0.484	0.292	1.353	5.2	14.9	1/ 9
007	23.856	1.098	0.433	3.090	4.6	13.1	1/ 9
008	7.242	0.325	0.511	0.630	4.5	11.2	1/ 6
009	10.032	0.428	0.322	0.931	4.3	9.8	1/ 6
010	23.371	1.825	2.164	3.782	7.8	18.6	1/ 6

ANALYTE=B-CAROTENE

SOL	GRAND AVG	S.E. AVG	S WITHIN	S BETWEEN	% CV AVG	% CV X	FR REJECT
005	1.405	0.167	0.097	0.496	11.9	36.0	0/ 9
006	5.083	0.308	0.246	0.909	6.1	18.5	0/ 9
007	5.906	0.538	0.243	1.606	9.1	27.5	0/ 9
008	1.376	0.286	0.060	0.493	20.8	36.1	0/ 3
009	6.165	1.083	0.599	1.828	17.6	31.2	0/ 3
010	10.212	2.121	0.486	3.657	20.8	36.1	0/ 3

RETINOL SOLUTION #005 GRAND AVG = 0.824

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	S DEV	% BIAS
	016	NFI	1.220	1.220	1.220	0.000	48.05
	078	NFI	1.200		1.200	0.000	45.62
	070	YES	0.820	0.860	0.840	0.020	1.94
	032	OUT	1.340	1.370	1.355	0.015	64.43
	077	NFI	1.300	1.370	1.335	0.035	62.01
	046	NFI	0.551	0.551	0.551	0.000	-33.13
	063	NFI	0.537	0.537	0.537	0.000	-34.83
	042	NFI	0.818	0.820	0.819	0.001	-0.61
	079	YES	0.825	0.827	0.826	0.001	0.24
	034	NFI	2.000	2.000	2.000	0.000	142.71
	058	OUT	4.000	4.000	4.000	0.000	385.41
	000	YES	0.750	0.890	0.820	0.070	-0.49
	000	YES	0.811	0.824	0.818	0.006	-0.79
	040	YES	0.870	0.820	0.845	0.025	2.54
	072	NFI	0.870	0.890	0.880	0.010	6.79
	004	YES	0.850	0.852	0.851	0.001	3.27
	099	NFI	0.850	0.850	0.850	0.000	3.15
	000	YES	0.791	0.832	0.812	0.021	-1.52
	047	YES	0.870	0.940	0.905	0.035	9.82
	076	NFI	0.890	0.950	0.920	0.030	11.64
	000	YES	0.788	0.796	0.792	0.004	-3.89
	057	YES	0.719	0.654	0.687	0.033	-16.69
	084	NFI	0.748	0.782	0.765	0.017	-7.17
	039	NFI	0.910	0.910	0.910	0.000	10.43
	062	NFI	0.890	0.896	0.893	0.003	8.37
	05	YES	0.860	0.880	0.870	0.010	5.58
	096	YES	0.850	0.860	0.855	0.005	3.76
NBS	0	NBS	0.900		0.900	0.000	9.22

RETINOL SOLUTION #008 GRAND AVG = 0.843

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	S DEV	% BIAS
	000	YES	0.930	0.880	0.905	0.025	7.39
	000	YES	0.766	0.767	0.767	0.001	-9.05
	018	NFI	0.520		0.520	0.000	-38.30
	076	NFI	0.730		0.730	0.000	-13.38
	041	YES	0.630	0.620	0.625	0.005	-25.84
	077	NFI	0.710	0.700	0.705	0.005	-16.35
	043	NFI	0.870	0.880	0.875	0.005	3.83
	084	YES	0.960	0.910	0.935	0.025	10.95
	062	YES	0.800	1.020	0.910	0.110	7.98
	031	YES	0.930	0.900	0.915	0.015	8.57
	050	NFI	0.920	0.890	0.905	0.015	7.39
NBS	0	NBS	0.780		0.780	0.000	-7.45

RETINOL SOLUTION #006 GRAND AVG = 2.115

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	S DEV	% BIAS
	016	NFI	2.820	2.820	2.820	0.000	33.31
	078	NFI	3.020	3.020	3.020	0.000	42.77
	065	YES	1.950	2.050	2.000	0.050	-5.45
	031	OUT	3.330	3.470	3.400	0.070	60.73
	078	NFI	3.420	3.500	3.460	0.040	63.57
	017	NFI	1.550	1.550	1.550	0.000	-26.73
	082	NFI	1.570	1.570	1.570	0.000	-25.78
	023	NFI	2.110	2.110	2.110	0.000	-0.25
	07	YES	2.370	2.370	2.370	0.000	12.04
	011	NFI	5.000	5.100	5.050	0.050	138.73
	067	OUT	5.700	5.800	5.750	0.050	171.82
	0	YES	2.030	2.230	2.130	0.100	0.69
		YES	2.020	2.060	2.040	0.020	-3.56
	028	YES	2.040	1.910	1.975	0.065	-6.64
	071	NFI	2.020	1.940	1.980	0.040	-6.40
	022	YES	2.080	2.090	2.085	0.005	-1.44
	086	NFI	2.080	2.080	2.080	0.000	-1.67
		YES	1.806	1.822	1.814	0.008	-14.25
	018	YES	2.250	2.440	2.345	0.095	10.86
	090	NFI	2.350	2.590	2.470	0.120	16.76
		YES	2.390	2.420	2.405	0.015	13.69
	046	NFI	1.860	1.770	1.815	0.045	-14.20
	062	YES	1.860	1.850	1.855	0.005	-12.31
	051	NFI	1.912	1.912	1.912	0.000	-9.61
	058	NFI	1.853	1.853	1.853	0.000	-12.40
	042	NFI	2.280	2.260	2.270	0.010	7.31
	04	YES	2.270	2.230	2.250	0.020	6.36
NBS	0	NBS	2.160		2.160	0.000	2.11

RETINOL SOLUTION #009 GRAND AVG = 2.545

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	S DEV	% BIAS
	000	YES	2.540	2.370	2.455	0.085	-3.54
	000	YES	2.160	2.410	2.285	0.125	-10.22
	071	NFI	1.230		1.230	0.000	-51.67
	009	YES	2.200	2.120	2.160	0.040	-15.13
	085	NFI	1.860	1.830	1.845	0.015	-27.50
	047	NFI	2.660	2.400	2.530	0.130	-0.59
	073	YES	2.770	2.790	2.780	0.010	9.23
	026	YES	2.720	2.860	2.790	0.070	9.63
	024	YES	2.760	2.840	2.800	0.040	10.02
	098	NFI	2.710	2.700	2.705	0.005	6.29
NBS	0	NBS	3.060		3.060	0.000	20.24

RETINOL SOLUTION #007 GRAND AVG = 3.336

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	S DEV	% BIAS
		NFI	4.850	4.850	4.850	0.000	45.39
		NFI	4.870	4.870	4.870	0.000	45.99
		YES	3.110	3.030	3.070	0.040	-7.97
		OUT	5.310	5.680	5.495	0.185	64.73
		NFI	5.340	5.640	5.490	0.150	64.58
		NFI	2.960	2.960	2.960	0.000	-11.27
		NFI	3.000	3.000	3.000	0.000	-10.07
		NFI	3.340	3.370	3.355	0.015	0.58
		YES	3.560	3.580	3.570	0.010	7.02
		OUT	9.000	10.000	9.500	0.500	184.79
		NFI	9.500	9.000	9.250	0.250	177.29
		YES	3.690	3.630	3.660	0.030	9.72
		YES	3.110	3.190	3.150	0.040	-5.57
		YES	3.360	3.360	3.360	0.000	0.72
		NFI	2.220	2.190	2.205	0.015	-33.90
		YES	3.340	3.340	3.340	0.000	0.13
		NFI	3.330	3.380	3.355	0.025	0.58
		YES	2.759	2.769	2.764	0.005	-17.14
		NFI	2.610	2.800	2.705	0.095	-18.91
		YES	3.330	3.630	3.480	0.150	4.32
		YES	3.730	3.630	3.680	0.050	10.32
		YES	2.920	3.050	2.985	0.065	-10.52
		NFI	2.688	2.688	2.688	0.000	-19.42
		NFI	2.787	2.787	2.787	0.000	-16.45
		YES	3.650	3.620	3.635	0.015	8.97
		NFI	3.520	3.720	3.620	0.100	8.52
NBS	0	NBS	3.750		3.750	0.000	12.42

RETINOL SOLUTION #010 GRAND AVG = 3.568

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	S DEV	% BIAS
	000	YES	3.740	3.630	3.685	0.055	3.29
	000	YES	3.530	3.500	3.515	0.015	-1.47
	002	NFI	1.430		1.430	0.000	-59.92
	097	NFI	1.640		1.640	0.000	-54.03
	018	YES	2.400	2.470	2.435	0.035	-31.74
	062	NFI	2.440	2.560	2.500	0.060	-29.92
	034	YES	3.840	3.620	3.730	0.110	4.56
	100	NFI	3.160	2.990	3.075	0.085	-13.81
	037	YES	3.760	4.120	3.940	0.180	10.44
	02	YES	4.090	4.110	4.100	0.010	14.93
	05	NFI	4.090	4.090	4.090	0.000	14.65
NBS	0	NBS	4.320		4.320	0.000	21.09

A-TOCOPHEROL SOLUTION #006 GRAND AVG = 9.261

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	S DEV	% BIAS
	000	OUT	0.910	0.850	0.880	0.030	-90.50
	000	YES	10.020	10.040	10.030	0.010	8.30
	043	NFI	10.000		10.000	0.000	7.98
	075	NFI	6.000		6.000	0.000	-35.21
	004	YES	9.700	9.700	9.700	0.000	4.74
	000	YES	10.500	9.700	10.100	0.400	9.06
	047	YES	10.730	10.570	10.650	0.080	15.00
	076	NFI	10.850	10.570	10.710	0.140	15.64
	000	YES	8.360	8.880	8.620	0.260	-6.92
	057	YES	6.540	6.040	6.290	0.250	-32.08
	084	NFI	7.230	7.480	7.355	0.125	-20.58
	039	NFI	9.005	9.005	9.005	0.000	-2.77
	062	NFI	8.898	8.898	8.898	0.000	-3.92
	055	YES	9.070	8.690	8.880	0.190	-4.12
	096	NFI	8.850	9.510	9.180	0.330	-0.88
	000	YES	9.910	9.730	9.820	0.090	6.03
NBS	0	NBS	10.000		10.000	0.000	7.98

A-TOCOPHEROL SOLUTION #008 GRAND AVG = 7.242

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	S DEV	% BIAS
	000	OUT	0.670	0.680	0.675	0.005	-90.68
	000	YES	7.780	7.710	7.745	0.035	6.95
	000	NFI	21.500	17.000	19.250	2.250	165.81
	043	NFI	6.170	6.210	6.190	0.020	-14.53
	084	YES	6.480	6.100	6.290	0.190	-13.15
	062	YES	7.400	8.680	8.040	0.640	11.02
	031	YES	7.150	6.270	6.710	0.440	-7.35
	050	NFI	7.180	7.260	7.220	0.040	-0.30
	000	YES	7.540	7.310	7.425	0.115	2.53
NBS	0	NBS	7.800		7.800	0.000	7.71

A-TOCOPHEROL SOLUTION #006 GRAND AVG = 3.323

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	S DEV	% BIAS
	000	OUT	0.270	0.290	0.280	0.010	-91.57
	000	YES	3.300	3.210	3.255	0.045	-2.03
	013	NFI	13.000		13.000	0.000	291.27
	081	NFI	10.000		10.000	0.000	200.98
	022	YES	3.400	3.300	3.350	0.050	0.83
	000	YES	3.600	3.500	3.550	0.050	6.85
	018	YES	3.850	4.040	3.945	0.095	18.74
	090	NFI	4.190	3.810	4.000	0.190	20.39
	000	YES	3.330	3.470	3.400	0.070	2.33
	046	NFI	2.310	2.270	2.290	0.020	-31.08
	062	YES	2.510	2.300	2.405	0.105	-27.61
	051	NFI	4.535	4.535	4.535	0.000	36.49
	058	NFI	4.459	4.459	4.459	0.000	34.21
	042	NFI	3.840	3.660	3.750	0.090	12.87
	047	YES	3.460	3.120	3.290	0.170	-0.98
	000	YES	3.360	3.410	3.385	0.025	1.88
NBS	0	NBS	3.400		3.400	0.000	2.33

A-TOCOPHEROL SOLUTION #009 GRAND AVG = 10.032

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	S DEV	% BIAS
	000	OUT	0.900	0.900	0.900	0.000	-91.03
	000	YES	10.070	10.120	10.095	0.025	0.63
	000	NFI	24.000	20.000	22.000	2.000	119.30
	047	NFI	6.920	7.250	7.085	0.165	-29.38
	073	YES	9.460	9.300	9.380	0.080	-6.50
	026	YES	11.000	11.920	11.460	0.460	14.23
	024	YES	9.160	8.760	8.960	0.200	-10.69
	098	NFI	9.320	9.330	9.325	0.005	-7.05
	000	YES	10.260	10.270	10.265	0.003	2.32
NBS	0	NBS	10.400		10.400	0.000	3.67

A-TOCOPHEROL SOLUTION #007 GRAND AVG = 23.856

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	S DEV	% BIAS
	000	OUT	2.420	2.370	2.395	0.025	-89.96
	000	YES	25.590	25.670	25.630	0.039	7.44
	003	NFI	62.000		62.000	0.000	159.89
	074	NFI	39.000		39.000	0.000	63.48
	017	YES	25.200	25.400	25.300	0.100	6.05
	000	YES	25.800	25.600	25.700	0.100	7.73
	024	NFI	26.120	27.680	26.900	0.780	12.76
	066	YES	26.470	26.140	26.305	0.165	10.26
	000	YES	20.200	21.300	20.750	0.550	-13.02
	052	YES	17.000	17.900	17.450	0.450	-26.85
	014	NFI	18.364	18.364	18.364	0.000	-23.02
	026	NFI	18.635	18.635	18.635	0.000	-21.89
	025	YES	24.700	25.580	25.140	0.440	5.38
	096	NFI	25.690	25.280	25.485	0.205	6.83
	000	YES	24.630	24.520	24.575	0.055	3.01
NBS	0	NBS	25.100		25.100	0.000	5.21

A-TOCOPHEROL SOLUTION #010 GRAND AVG = 23.371

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	S DEV	% BIAS
	000	OUT	2.140	2.150	2.145	0.005	-90.82
	000	YES	24.060	24.020	24.040	0.019	2.86
	002	NFI	39.500		39.500	0.000	69.01
	097	NFI	41.000		41.000	0.000	75.43
	034	YES	17.700	17.300	17.500	0.200	-25.12
	100	NFI	16.400	14.300	15.350	1.050	-34.32
	037	YES	32.380	25.600	28.990	3.390	24.04
	020	YES	23.160	23.030	23.095	0.065	-1.18
	061	NFI	23.420	23.230	23.325	0.095	-0.20
	000	YES	22.820	23.640	23.230	0.410	-0.60
NBS	0	NBS	23.800		23.800	0.000	1.84

B-CAROTENE SOLUTION #005 GRAND AVG = 1.405

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	S DEV	% BIAS
	070	YES	1.200	1.210	1.205	0.005	-14.23
	032	YES	1.920	1.940	1.930	0.010	37.37
	077	NFI	1.870	2.000	1.935	0.065	37.72
	046	NFI	0.680	0.680	0.680	0.000	-51.60
	063	NFI	0.815	0.815	0.815	0.000	-41.99
	042	YES	1.020	1.030	1.025	0.005	-27.05
	042	YES	2.400	2.360	2.380	0.020	69.40
	000	YES	1.030	1.050	1.040	0.010	-25.98
	040	NFI	2.010	2.010	2.010	0.000	43.06
	072	NFI	2.030	2.030	2.030	0.000	44.48
	004	YES	1.110	1.120	1.115	0.005	-20.64
	099	NFI	1.110	1.090	1.100	0.010	-21.71
	047	YES	1.090	1.180	1.135	0.045	-19.22
	076	NFI	1.140	1.270	1.205	0.065	-14.23
	000	YES	1.010	1.020	1.015	0.005	-27.76
	057	YES	2.000	1.600	1.800	0.200	28.11
	084	NFI	2.140	2.140	2.140	0.000	52.31
	039	NFI	2.682	2.682	2.682	0.000	90.89
	062	NFI	2.696	2.696	2.696	0.000	91.89
NBS	0	NBS	1.160		1.160	0.000	-17.44

B-CAROTENE SOLUTION #008 GRAND AVG = 1.376

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	S DEV	% BIAS
	000	YES	1.860	1.800	1.830	0.030	32.98
	000	YES	0.894	0.803	0.849	0.045	-38.34
	041	NFI	1.620	1.620	1.620	0.000	17.72
	077	NFI	1.620	1.620	1.620	0.000	17.72
	043	NFI	1.400	1.500	1.450	0.050	5.37
	084	YES	1.400	1.500	1.450	0.050	5.37
NBS	0	NBS	0.920		0.920	0.000	-33.15

B-CAROTENE SOLUTION #006 GRAND AVG = 5.083

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	S DEV	% BIAS
	065	YES	5.000	4.900	4.950	0.050	-2.62
	031	YES	5.670	5.910	5.790	0.120	13.90
	078	NFI	5.840	6.030	5.935	0.095	16.75
	017	NFI	5.510	5.510	5.510	0.000	8.39
	082	NFI	4.700	4.700	4.700	0.000	-7.54
	023	YES	3.600	3.600	3.600	0.000	-29.18
	000	YES	6.860	6.280	6.570	0.290	29.25
	000	YES	4.730	4.530	4.630	0.100	-8.92
	028	NFI	5.720	5.720	5.720	0.000	12.52
	071	NFI	5.740	5.740	5.740	0.000	12.92
	022	YES	5.650	5.650	5.650	0.000	11.15
	086	NFI	5.600	5.520	5.560	0.040	9.38
	018	YES	4.660	4.400	4.530	0.130	-10.89
	090	NFI	5.020	4.860	4.940	0.080	-2.82
	000	YES	4.430	4.130	4.280	0.150	-15.80
	046	NFI	4.300		4.300	0.000	-15.41
	062	YES	5.400	6.100	5.750	0.350	13.11
	051	NFI	8.176	8.176	8.176	0.000	60.84
	058	NFI	8.110	8.110	8.110	0.000	59.54
NBS	0	NBS	5.060		5.060	0.000	-0.46

B-CAROTENE SOLUTION #009 GRAND AVG = 6.165

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	S DEV	% BIAS
	000	YES	7.940	8.380	8.160	0.220	32.36
	000	YES	4.450	4.420	4.435	0.015	-28.06
	009	NFI	6.930	6.930	6.930	0.000	12.41
	085	NFI	6.600	6.600	6.600	0.000	7.06
	047	NFI	4.900		4.900	0.000	-20.52
	073	YES	5.200	6.600	5.900	0.700	-4.30
NBS	0	NBS	4.640		4.640	0.000	-24.74

B-CAROTENE SOLUTION #007 GRAND AVG = 5.906

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	S DEV	% BIAS
	013	YES	5.800	5.700	5.750	0.050	-2.63
	018	YES	7.130	7.690	7.410	0.280	25.48
	072	NFI	7.220	7.650	7.435	0.215	25.90
	064	NFI	5.660	5.660	5.660	0.000	-4.16
	093	NFI	6.010	6.010	6.010	0.000	1.77
	022	YES	3.920	4.040	3.980	0.060	-32.61
	000	YES	8.220	8.120	8.170	0.050	38.34
	000	YES	4.240	4.430	4.335	0.095	-26.59
	004	NFI	6.200	6.200	6.200	0.000	4.99
	050	NFI	6.120	6.120	6.120	0.000	3.63
	017	YES	7.300	7.200	7.250	0.050	22.77
	040	NFI	7.120	7.090	7.105	0.015	20.31
	024	NFI	3.010	3.310	3.160	0.150	-46.49
	066	YES	3.890	3.780	3.835	0.055	-35.06
	000	YES	6.860	6.980	6.920	0.060	17.18
	052	YES	5.100	5.900	5.500	0.400	-6.87
	014	NFI	7.568	7.568	7.568	0.000	28.15
	026	NFI	7.926	7.926	7.926	0.000	34.21
NBS	0	NBS	4.300		4.300	0.000	-27.19

B-CAROTENE SOLUTION #010 GRAND AVG = 10.212

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	S DEV	% BIAS
	000	YES	14.440	13.760	14.100	0.340	38.08
	000	YES	10.120	9.350	9.735	0.385	-4.67
	018	NFI	13.320	13.300	13.310	0.010	30.34
	062	NFI	12.600	12.600	12.600	0.000	23.39
	034	YES	6.500	7.100	6.800	0.300	-33.41
	100	NFI	9.000	8.300	8.650	0.350	-15.29
NBS	0	NBS	10.600		10.600	0.000	3.80

Appendix C. Updated "All-Lab Report" for RR01

The following three pages are the modernized "All-Lab" report for RR01. This report has three parts:

- Page B2 lists the participant mean results for all analytes reported.
- Page B3 provides the legend for page B2.
- Page B4 summarizes each participants' performance for retinol, α-tocopherol, and β-carotene, using the "Comparability Summary" calculations from the 1999 to 2017 Round Robins.

To ensure confidentiality, the laboratory identifiers used in this "All-Lab Report" have been altered from those used in RR01. 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 I Laboratory Results

	Total Retinol, µg/mL						α-Tocopherol, µg/mL						Total β-Carotene, µg/mL						Total α-Carotene, µg/mL					
Lab	5s	6s	7s	8s	9s	10s	5s	6s	7s	8s	9s	10s	5s	6s	7s	8s	9s	10s	5s	6s	7s	8s	9s	10s
FSV-BD	0.863	1.978	2.783	0.665	2.003	2.468							2.020	5.730	6.160	1.620	6.765	12.950						
FSV-BE							11.26	4.93	19.16	8.86	9.74	28.44												
FSV-BF	0.715	1.820	2.985	0.890	2.725	3.270	7.01	2.45	17.43	6.34	8.36	17.05	2.065	4.830	5.060	1.375	5.055	7.715						
FSV-BG	1.345	3.430	5.493										1.933	5.863	7.476									
FSV-BI	0.840	2.000	3.070										1.205	4.950	5.750									
FSV-BY	1.210	2.920	4.860																					
FSV-CA	0.820	2.130	3.660	0.905	2.455	3.685	8.80	2.80	23.95	6.75	9.00	21.45	2.380	8.170	8.170	1.830	8.160	14.100						
FSV-CJ	0.823	2.240	3.463										1.025	3.980	3.980									
FSV-CL	0.792	2.405	3.680				8.62	3.40	20.75				1.015	4.280	6.920				0.636		0.073			
FSV-CN	0.812	1.814	2.764				10.10	3.55	25.70															
FSV-CO	0.769	2.109	3.555	1.065	3.205	3.771	10.05	3.95	28.45	9.50	13.05	25.65												
FSV-DE							9.82	3.39	24.58	7.43	10.27	23.23												
FSV-DG	0.900	2.200	3.400	0.800	3.100	4.300	10.00	3.40	25.00	7.80	10.00	24.00	1.200	5.000	7.300	0.900	4.600	11.000						
FSV-DH	0.913	2.408	3.093				10.68	3.97	26.60				1.170	4.735	5.378									
FSV-DN	0.851	2.083	3.348				9.70	3.35	25.30				1.108	5.605	7.178				1.225	0.114	0.150			
FSV-DO	3.000	5.400	9.375																					
FSV-DT				0.910	2.790	3.940				8.04	11.46	28.99												
FSV-EG	0.544	1.560	2.980										0.748	5.105	5.835									
FSV-EP	0.863	2.260	3.628	0.910	2.753	4.095	9.03	3.52	25.31	6.97	9.14	23.21												
FSV-ER	0.818	2.040	3.150	0.767	2.285	3.515	10.03	3.26	25.63	7.75	10.10	24.04	1.040	4.335	4.335	0.849	4.435	9.735						
FSV-FO	0.900	1.883	2.738				8.95	4.50	18.50				2.689	8.143	7.747									
FSV-GA				0.625	1.230	1.535	8.00	11.50	50.50	19.25	22.00	40.25				1.490	5.610	10.400						
n	18	18	18	9	9	9	14	14	14	10	10	10	13	13	13	6	6	6	2	1	2	0	0	0
Min	0.544	1.560	2.738	0.625	1.230	1.535	7.01	2.45	17.43	6.34	8.36	17.05	0.748	3.980	3.980	0.849	4.435	7.715						
Median	0.845	2.119	3.374	0.890	2.725	3.685	9.76	3.46	25.15	7.77	10.05	24.02	1.200	5.000	6.160	1.433	5.333	10.700						
Max	3.000	5.400	9.375	1.065	3.205	4.300	11.26	11.50	50.50	19.25	22.00	40.25	2.689	8.170	8.170	1.830	8.160	14.100						
eSD	0.080	0.281	0.452	0.133	0.556	0.608	1.14	0.52	1.97	1.36	1.45	3.11	0.274	0.986	1.631	0.434	1.208	2.383						
eCV	9	13	13	15	20	16	12	15	8	17	14	13	23	20	26	30	23	22						
NIST	0.730	1.970	3.260	0.780	3.100	4.370	10.03	3.40	25.08	7.80	10.36	23.80	1.160	5.760	7.150	0.920	4.640	10.550						
NAV	0.845	2.119	3.374	0.890	2.725	3.685	9.76	3.46	25.15	7.77	10.05	24.02	1.200	5.000	6.160	1.433	5.333	10.700						
NAU	0.080	0.281	0.452	0.133	0.556	0.608	1.14	0.52	1.97	1.36	1.45	3.11	0.274	0.986	1.631	0.434	1.208	2.383						

C2

Round Robin I Laboratory Results

Legend

Term	Definition
n	Number of (non-NIST) quantitative values reported for this analyte
Min	Minimum (non-NIST) quantitative value reported
Median	Median (non-NIST) quantitative value reported
Max	Maximum (non-NIST) quantitative value reported
eSD	Estimated standard deviation, calculated from the median absolute deviation from the median of the non-NIST results
eCV	Coefficient of Variation for (non-NIST) results: 100*eSD/Median
NAV	NIST Assigned Value, our estimate of the "true" analyte concentration
NAU	NIST Assigned Uncertainty, our estimate of the total (sample heterogeniety and inter- and intra-laboratory) standard deviation

Round Robin I Laboratory Results

Comparability Summary

Lab	TR	aT	bC
FSV-BD	2		2
FSV-BE		3	
FSV-BF	1	3	2
FSV-BG	4		2
FSV-BI	1		1
FSV-BY	4		
FSV-CA	1	1	3
FSV-CJ	1		2
FSV-CL	1	2	1
FSV-CN	2	1	
FSV-CO	1	2	
FSV-DE		1	
FSV-DG	1	1	1
FSV-DH	1	1	1
FSV-DN	1	1	1
FSV-DO	4		
FSV-DT	1	2	
FSV-EG	3		2
FSV-EP	1	1	
FSV-ER	1	1	1
FSV-FO	2	3	4
FSV-GA	3	4	1
NIST	1	1	1
n	21	16	15

Label	Definition
Lab	laboratory number
TR	"Standard Score" for Retinol
aT	"Standard Score" for α-Tocopherol
bC	"Standard Score" for Total β-Carotene
n	number of (non-NIST) laboratories providing data for this analyte

"Standard Score"

Given that our knowledge of the shape, location, and width of the measurement distributions is approximate and that a limited number of labs are involved, we summarize comparability with the following four-level "Standard Score" (StS)…

StS	Definition
1	All StV within ±1 SD
2	All StV within ±2 SD
3	All StV within ±3 SD
4	At least one StV >3 SD

where:	
SD	Total measurement standard deviation (SD), including serum heterogeneity and inter-and intra-laboratory variability.
StV	Standardized Value, the distance in SD units your value is from the "true" concentration: StV = (your value - NAV) / NAU
NAV	NIST Assigned Value, our estimate of the "true" analyte concentration
NAU	NIST Assigned Uncertainty, our estimate of the SD

	TR	aT	bC	Expected
% 1	62	56	53	68.2 %
% 2	14	19	33	27.3 %
% 3	10	19	7	4.3 %
% 4	14	6	7	0.3 %

These are the observed and normal-population-expected proportions of each Standard Score (StS), based upon each laboratory's largest StV for the four sera.

Appendix D. Shipping Package Inserts for RR02

The following three items were attached to each package shipped to RR02 participants:

- Cover and corrigendum letters, describing the samples and calibration solutions used in the study.
- “In-House Standards” datasheet for reporting results based upon the participants’ own calibration solutions.
- “NBS Reference Solutions” for reporting results based upon the NIST-provided calibration solutions.

January 15, 1985

[Name and Address]

Dear [Formal name]:

The samples in this shipment are NBS/NCI Solutions 5, 11, 13, and 17-21. While two ampoules of each solution are enclosed, we want results for only one of each kind. Solutions 5, 11, and 13 are from sample lots you received previously. Now, however, concentrations have been assigned to Solutions 5, 11, 13, and 17; and we ask that you post results only for Solutions 18-21. The results for these unknowns are to be determined in two ways: first, by comparison to the "known" solutions we are providing; and second, by comparison to your "in-house" standards. A separate form is provided for each. We hope this comparison of results will allow us to evaluate the problems of standards purity. I next consider the fat-soluble vitamin samples, then the ascorbic acid.

Solutions 5 and 17-19 contain α- and β-carotene, retinol, and α- and γ-tocopherol dissolved in ethanol and toluene. The proportions of toluene are low and different in each solution. The NBS values for Solutions 5 and 17 (in mg/L) are as follows:

	Solution 5	Solution 17
β-carotene	0.90	2.30
retinol	1.16	2.04
α-tocopherol	10.0	19.3

(You will note that this study does not include analogous hexane solutions. That is because we found that all of the laboratories but one analyzed the ethanol solutions, that that one laboratory analyzed only the hexane solution, and that only a few of the other laboratories analyzed the hexane solutions also.)

In order to minimize differences in practices among the laboratories, we ask that Solutions 18 and 19 be analyzed in the following order: 18, 19, 18, 19. We recommend, but do not require, that Solutions 5 and 17 be analyzed both before and after Solutions 18 and 19. You might choose to do Solutions 5 and 17 just once, but this is your decision. We want from you only duplicate results for Solution 18 and the same for Solution 19. Please do not send averages or single results for Solution 18 or 19. Use the report form for Results from Use of NBS Reference Solutions.

We also want duplicate results for Solutions 18 and 19, made similarly, but based on use of your own "in-house" standards. Follow the same order as above when measuring Solutions 18 and 19, but precede and follow them with your own standards if you choose to do your standards that way.

The data for all of these results could be obtained from one set of analyses organized as follows: your standards, the two NBS reference solutions, Solutions 18 and 19, and the NBS reference solutions and your standards again if you choose to repeat them.

Ascorbic acid is in Solutions 11, 13, 20, and 21, with dithiothreitol as stabilizer. The solvent is acetonitrile-water (3:1 by volume). The ascorbic acid level is 4.04 mg/L in Solution 11 and 29.0 mg/L in Solution 13. As for the fat-soluble vitamin samples, we request two pairs of duplicate results for Solutions 20 and 21, the first pair to be obtained with Solutions 11 and 13 for calibration and the second pair with your "in-house" standards used for calibration. Please analyze Solutions 20 and 21 in the following order: 20, 21, 20, 21. Precede and follow these measurements with measurements of Solutions 11 and 13. Use the same pattern for Solutions 20 and 21 when analyzing them with your "in-house" standards. You are free to use the calibration materials as you see fit. Please refer also to the previous paragraph.

The values assigned to Solutions 5, 11, 13, and 17 are reasonably accurate, based on current work at NBS. We expect in the future to provide more definitive information on the purity of the analyte materials that can be used in the analyses for these analytes.

We expect to send you the statistical analysis on the initial round of results before we send the first of the serum-based samples, and the latter will be two or three weeks from now.

Please plan to have these results in the mail to us by January 30.

Sincerely,

Robert Schaffer, Ph.D.
Supervisory Research Chemist
Organic Analytical Research Division
Center for Analytical Chemistry

PS Only one ampoule of Solution 5 is enclosed. Sorry, there were not enough to send two. We do have a few extra if yours arrives broken.

January 22, 1985

[Name and Address]

Dear [Formal name]:

I am writing this letter to provide you with a written notice of the numerical errors in my letter of January 15, 1985, on the values for retinol and β-carotene in Solution 5. The correct values are 0.90 mg/L for retinol and 1.16 mg/L for β-carotene.

Sincerely,

Robert Schaffer, Ph.D.
Supervisory Research Chemist
Organic Analytical Research Division
Center for Analytical Chemistry

Report on NBS/NCI Samples from Laboratory #____

Results from Use of NBS Solutions

Data of Analysis _____

Results in mg/L

Samples		Result 1	Result 2
Solution 18 #____	β-carotene		
	retinol		
	α-tocopherol		
Solution 19 #____	β-carotene		
	retinol		
	α-tocopherol		
Solution 20 #____	ascorbic acid		
Solution 21 #____	ascorbic acid		

Report on NBS/NCI Samples from Laboratory #____

Results from Use of "In-House" Standards

Data of Analysis _____

Results in mg/L

Samples		Result 1	Result 2
Solution 18 #____	β-carotene		
	retinol		
	α-tocopherol		
Solution 19 #____	β-carotene		
	retinol		
	α-tocopherol		
Solution 20 #____	ascorbic acid		
Solution 21 #____	ascorbic acid		

Appendix E. Final Report for RR02

The following ten pages present the following:

- Page E2: Cover letter.
- Page E3: “Comments on the Tables of Laboratory Results”.
- Page E4: “Conclusions from the Statistical Summary” and “Explanation of Column Heading on Statistical Summary”.
- Page E5: “Statistical Summary”.
- Pages E6 to E8: “Standard Lab” results, obtained using the participants’ own “In-House Standards” calibration solutions.
- Pages E9 to E11: “Standard NBS” results, obtained using the “NBS Reference Solutions” calibration solutions.

The ascorbic acid information provided in the original tables has been deleted. The format of the tables has been edited to improve readability.

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

February 28, 1985

[Name and Address]

Deare[Formal Name]:

Your results and the statistical summary for Study II (Solutions 18-21) are enclosed. The summary of the statistical conclusions is given on the next two pages. Our comments on the tables of laboratory results and the individual laboratory results are on the following pages.

We hope to be sending you serum based materials next week. They should leave NBS on March 6 and arrive in your labs on March 7, which is a Thursday. Our one participating lab in Helsinki should receive these samples on March 8. Only if there is a change in this shipping schedule will you hear from us again about this.

Plans for the workshop meeting at NBS on April 11 and 12 are progressing, at least as far as participants from NCI supported labs are concerned. Possible funding for others does not seem good. Everyone, please note that we hope to have the first set of serum-based sample results available for discussion in the workshop program, and then to share with you. We will need everyone's cooperation to have those samples analyzed and all of the results returned to us in time for the meeting. Thanks.

Sincerely,

Robert Schaffer, Ph.D.
Supervisory Research Chemist
Organic Analytical Research Division
Center for Analytical Chemistry

Enclosure

Comments on the Tables of Laboratory Results

1. Spaces are provided on your report forms for noting the vial numbers of the samples analyzed. We keep records of all the vials numbers you receive. We would like your duplicate results to be duplicate analyses of a single vial, and it is the number on that vial that we want you to identify on the report form. (The second vial of each sample is provided to reduce your possible need for replacement samples because of breakage.)

2. Only a few results are classified as NFI--those from a laboratory that reported only single values, not duplicates, and those from two laboratories which use HPLC methods that do not resolve α- and β-carotene and therefore reported α plus β. For their information, the α-carotene in Solutions 18 and 19 were about 1.1 and 2.3 mg/L, respectively.

3. The outlier results were identified by inspection as obvious; that is, statistical tests for outliers were not used. This results in a minimum number of rejections. You will remember we acted similarly for removing outliers from our initial multilaboratory test results. The proportion of outliers was somewhat lower for the present results.

4. Three sets of results are identified as NBS results. NBS M01 results are simply gravimetric values for ascorbic acid, β-carotene, and α-tocopherol, and gravimetric values that are corrected for molar absorbance for retinol. NBS M02 and M03 are our measured HPLC values with UV and electrochemical detection, respectively, and are based on the NBS M01 solutions.

Conclusions from the Statistical Summary

1. In general, S_b is appreciably larger than S_w. It is the major source of variability.

2. As expected, the beneficial effect of using the NBS reference solutions for calibration is reflected in smaller S_b values (and indirectly in smaller % CV X values). A strong beneficial effect of using the NBS reference solutions is clearly observed for the higher concentration solution for α-tocopherol, β-carotene, and retinol, and for the low concentration solution for retinol.

3. The % CV X is generally 5-25 percent.

4. The % CV AVE is generally 2-10 percent.

5. The % CV is approximately constant with increasing concentration.

Explanation of Column Heading on Statistical Summary

SOL = solution number

GRAND AVG = grand average (consensus value from analysis of variance) = $\bar{x}$ (mg/L)

S.E.AVG = one standard deviation (std. dev.) of $\bar{x}$ = $S_{\bar{x}}$

S WITHIN = within laboratory component of std. dev. = S_w (square root of within laboratory component of variance from the ANOVA; a pooled estimate of within laboratory imprecision)

S BETWEEN = between laboratory component of standard deviation = S_b

% CV AVG = percent coefficient of variation of the grand average = $100\ S_{\bar{x}}/\bar{x}$

% CV X = percent coefficient of variation of a single measurement made by a single laboratory = $100\ S_x/\bar{x}$, where $S_x = \sqrt{S_w^2 + S_b^2}$ (NOTE: S_x is not equal to the std. dev. of all measurements)

FR REJECT = ratio of rejected to total laboratory results considered for statistical evaluation

Statistical Summary

STANDARD LAB ANALYTE=RETINOL

SOL	GRAND AVG	S.E. AVG	S WITHIN	S BETWEEN	% CV AVG	% CV X	FR REJECT
018	0.514	0.030	0.015	0.122	5.8	24.0	1/18
019	1.571	0.079	0.058	0.335	5.1	21.6	0/18

STANDARD LAB ANALYTE=α-TOCOPHEROL

SOL	GRAND AVG	S.E. AVG	S WITHIN	S BETWEEN	% CV AVG	% CV X	FR REJECT
018	6.290	0.227	0.160	0.777	3.6	12.6	0/12
019	12.600	0.449	0.214	1.549	3.6	12.4	0/12

STANDARD LAB ANALYTE=β-CAROTENE

SOL	GRAND AVG	S.E. AVG	S WITHIN	S BETWEEN	% CV AVG	% CV X	FR REJECT
018	0.412	0.036	0.013	0.113	8.7	27.7	2/12
019	2.624	0.283	0.076	0.979	10.8	37.4	0/12

STANDARD NBS ANALYTE=RETINOL

SOL	GRAND AVG	S.E. AVG	S WITHIN	S BETWEEN	% CV AVG	% CV X	FR REJECT
018	0.505	0.008	0.015	0.029	1.6	6.5	1/16
019	1.536	0.053	0.058	0.213	3.4	14.3	0/17

STANDARD NBS ANALYTE=α-TOCOPHEROL

SOL	GRAND AVG	S.E. AVG	S WITHIN	S BETWEEN	% CV AVG	% CV X	FR REJECT
018	6.186	0.222	0.166	0.760	3.6	12.6	0/12
019	12.474	0.311	0.229	1.065	2.5	8.7	0/12

STANDARD NBS ANALYTE=β-CAROTENE

SOL	GRAND AVG	S.E. AVG	S WITHIN	S BETWEEN	% CV AVG	% CV X	FR REJECT
018	0.435	0.034	0.015	0.112	7.8	26.0	2/13
019	2.924	0.216	0.051	0.746	7.4	25.6	0/12

STANDARD LAB ANALYTE=RETINOL SOLUTION #018 GRAND AVG = 0.514

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	012	YES	0.700	0.710	0.705	0.005	37.18
	015	YES	0.460	0.460	0.460	0.000	-10.50
	000	YES	0.830	0.830	0.830	0.000	61.50
	073	YES	0.500	0.500	0.500	0.000	-2.71
	065	YES	0.530	0.530	0.530	0.000	3.12
	032	OUT	1.150	0.950	1.050	0.100	104.30
	000	YES	0.500	0.560	0.530	0.030	3.12
	000	YES	0.400	0.410	0.405	0.005	-21.20
	038	YES	0.462	0.462	0.462	0.000	-10.11
	042	YES	0.570	0.520	0.545	0.025	6.04
	022	YES	0.540	0.530	0.535	0.005	4.10
	023	YES	0.461	0.483	0.472	0.011	-8.16
	000	YES	0.552	0.539	0.546	0.007	6.14
	027	YES	0.440	0.440	0.440	0.000	-14.39
	088	YES	0.242	0.253	0.248	0.005	-51.84
	021	YES	0.530	0.520	0.525	0.005	2.15
	033	YES	0.450	0.450	0.450	0.000	-12.44
	056	YES	0.560	0.550	0.555	0.005	7.99
NBS	M01	NBS	0.510		0.510	0.000	-0.77
NBS	M02	NBS	0.500		0.500	0.000	-2.71
NBS	M03	NBS	0.570		0.570	0.000	10.91

STANDARD LAB ANALYTE=RETINOL SOLUTION #019 GRAND AVG = 1.571

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	038	YES	2.090	2.100	2.095	0.005	33.32
	034	YES	1.350	1.340	1.345	0.005	-14.41
	000	YES	2.390	2.440	2.415	0.025	53.69
	038	YES	1.440	1.440	1.440	0.000	-8.36
	022	YES	1.470	1.460	1.465	0.005	-6.77
	021	YES	2.000	2.250	2.125	0.125	35.23
	000	YES	1.690	1.530	1.610	0.080	2.46
	000	YES	1.270	1.230	1.250	0.020	-20.45
	078	YES	1.348	1.361	1.354	0.007	-13.80
	024	YES	1.310	1.310	1.310	0.000	-16.63
	034	YES	1.600	1.550	1.575	0.025	0.23
	032	YES	1.230	1.090	1.160	0.070	-26.18
	000	YES	1.440	1.490	1.465	0.025	-6.77
	008	YES	1.290	1.290	1.290	0.000	-17.91
	051	YES	1.499	1.511	1.505	0.006	-4.22
	026	YES	1.820	1.760	1.790	0.030	13.91
	055	YES	1.420	1.440	1.430	0.010	-9.00
	054	YES	1.670	1.650	1.660	0.010	5.64
NBS	M01	NBS	1.530		1.530	0.000	-2.63
NBS	M02	NBS	1.500		1.500	0.000	-4.54
NBS	M03	NBS	1.580		1.580	0.000	0.55

STANDARD LAB ANALYTE=α-TOCOPHEROL SOLUTION #018 GRAND AVG = 6.290

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	000	YES	5.000	5.300	5.150	0.150	-18.13
	000	YES	8.300	8.000	8.150	0.150	29.56
	038	YES	6.610	6.520	6.565	0.045	4.37
	022	YES	6.300	6.000	6.150	0.150	-2.23
	023	YES	6.200	6.570	6.385	0.185	1.50
	000	YES	5.700	5.710	5.705	0.005	-9.31
	027	YES	5.800	5.800	5.800	0.000	-7.80
	088	YES	6.409	6.389	6.399	0.010	1.73
	021	YES	6.000	6.300	6.150	0.150	-2.23
	033	YES	5.500	5.500	5.500	0.000	-12.56
	000	YES	6.470	6.370	6.420	0.050	2.06
	056	YES	6.950	7.270	7.110	0.160	13.03
NBS	M01	NBS	6.400		6.400	0.000	1.74
NBS	M02	NBS	6.290		6.290	0.000	-0.01
NBS	M03	NBS	6.670		6.670	0.000	6.04

STANDARD LAB ANALYTE=α-TOCOPHEROL SOLUTION #019 GRAND AVG = 12.600

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	000	YES	12.200	12.200	12.200	0.000	-3.17
	000	YES	16.600	16.600	16.600	0.000	31.75
	023	YES	13.000	13.000	13.000	0.000	3.18
	034	YES	12.300	12.200	12.250	0.050	-2.78
	032	YES	12.850	12.230	12.540	0.310	-0.48
	000	YES	9.950	9.980	9.965	0.015	-20.91
	008	YES	11.800	11.700	11.750	0.050	-6.75
	051	YES	12.118	11.980	12.049	0.069	-4.37
	026	YES	12.000	12.400	12.200	0.200	-3.17
	055	YES	12.200	11.500	11.850	0.350	-5.95
	000	YES	13.110	12.990	13.050	0.060	3.57
	054	YES	13.780	13.710	13.745	0.035	9.09
NBS	M01	NBS	12.860		12.860	0.000	2.06
NBS	M02	NBS	12.500		12.500	0.000	-0.79
NBS	M03	NBS	13.280		13.280	0.000	5.40

STANDARD LAB			ANALYTE=β-CAROTENE		SOLUTION #018	GRAND AVG =	0.412
LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	012	YES	0.510	0.460	0.485	0.025	17.63
	015	YES	0.540	0.540	0.540	0.000	30.97
	000	NFI	1.840	1.820	1.830	0.010	343.85
	073	NFI	1.280	1.380	1.330	0.050	222.58
	065	YES	0.450	0.450	0.450	0.000	9.14
	000	OUT	1.470	1.630	1.550	0.080	275.94
	000	YES	0.510	0.530	0.520	0.010	26.12
	038	YES	0.429	0.445	0.437	0.008	5.99
	049	NFI	1.390		1.390	0.000	237.13
	022	YES	0.432	0.437	0.435	0.003	5.38
	023	YES	0.354	0.365	0.360	0.005	-12.81
	000	YES	0.375	0.385	0.380	0.005	-7.83
	027	YES	0.380	0.380	0.380	0.000	-7.83
	088	YES	0.135	0.139	0.137	0.002	-66.77
	056	OUT	2.060	2.100	2.080	0.020	404.49
NBS	M01	NBS	0.580		0.580	0.000	40.67
NBS	M02	NBS	0.470		0.470	0.000	13.99
NBS	M03	NBS	0.540		0.540	0.000	30.97

STANDARD LAB			ANALYTE=β-CAROTENE		SOLUTION #019	GRAND AVG =	2.624
LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	038	YES	2.670	2.620	2.645	0.025	0.81
	034	YES	2.160	2.130	2.145	0.015	-18.24
	000	NFI	5.163	5.196	5.180	0.017	97.41
	038	NFI	3.890	3.990	3.940	0.050	50.17
	022	YES	2.210	2.220	2.215	0.005	-15.58
	000	YES	4.120	4.420	4.270	0.150	62.75
	000	YES	2.810	2.700	2.755	0.055	5.01
	078	YES	2.210	2.340	2.275	0.065	-13.29
	087	NFI	3.920		3.920	0.000	49.41
	034	YES	2.040	2.060	2.050	0.010	-21.87
	032	YES	1.360	1.340	1.350	0.010	-48.55
	000	YES	1.980	1.930	1.955	0.025	-25.49
	008	YES	2.060	2.000	2.030	0.030	-22.63
	051	YES	3.055	3.103	3.079	0.024	17.35
	054	YES	4.760	4.670	4.715	0.045	79.71
NBS	M01	NBS	2.880		2.880	0.000	9.77
NBS	M02	NBS	2.770		2.770	0.000	5.58
NBS	M03	NBS	2.940		2.940	0.000	12.06

STANDARD NBS ANALYTE=RETINOL SOLUTION #018 GRAND AVG = 0.505

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	012	YES	0.510	0.510	0.510	0.000	1.04
	000	YES	0.510	0.510	0.510	0.000	1.04
	000	YES	0.510	0.510	0.510	0.000	1.04
	073	YES	0.520	0.530	0.525	0.005	4.02
	065	YES	0.500	0.510	0.505	0.005	0.05
	032	OUT	0.900	0.850	0.875	0.025	73.36
	000	YES	0.470	0.530	0.500	0.030	-0.94
	000	YES	0.500	0.520	0.510	0.010	1.04
	062	YES	0.516	0.512	0.514	0.002	1.84
	022	YES	0.510	0.500	0.505	0.005	0.05
	023	YES	0.397	0.416	0.407	0.009	-19.46
	000	YES	0.552	0.539	0.546	0.007	8.08
	027	YES	0.510	0.510	0.510	0.000	1.04
	021	YES	0.540	0.530	0.535	0.005	6.00
	033	YES	0.460	0.500	0.480	0.020	-4.90
	056	YES	0.510	0.500	0.505	0.005	0.05
NBS	M01	NBS	0.510		0.510	0.000	1.04
NBS	M02	NBS	0.500		0.500	0.000	-0.94
NBS	M03	NBS	0.570		0.570	0.000	12.93

STANDARD NBS ANALYTE=RETINOL SOLUTION #019 GRAND AVG = 1.536

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	038	YES	1.520	1.520	1.520	0.000	-1.05
	000	YES	1.510	1.510	1.510	0.000	-1.70
	000	YES	1.500	1.510	1.505	0.005	-2.02
	038	YES	1.510	1.510	1.510	0.000	-1.70
	022	YES	1.540	1.520	1.530	0.010	-0.40
	021	YES	1.500	1.750	1.625	0.125	5.79
	000	YES	1.610	1.450	1.530	0.080	-0.40
	000	YES	1.480	1.440	1.460	0.020	-4.95
	023	YES	1.507	1.508	1.507	0.001	-1.86
	084	YES	1.500	1.500	1.500	0.000	-2.35
	032	YES	1.060	0.938	0.999	0.061	-34.96
	000	YES	1.490	1.440	1.465	0.025	-4.63
	008	YES	1.500	1.490	1.495	0.005	-2.67
	051	YES	2.104	2.180	2.142	0.038	39.45
	026	YES	1.820	1.770	1.795	0.025	16.86
	055	YES	1.490	1.490	1.490	0.000	-3.00
	054	YES	1.540	1.520	1.530	0.010	-0.40
NBS	M01	NBS	1.530		1.530	0.000	-0.40
NBS	M02	NBS	1.500		1.500	0.000	-2.35
NBS	M03	NBS	1.580		1.580	0.000	2.86

STANDARD NBS ANALYTE=α-TOCOPHEROL SOLUTION #018 GRAND AVG = 6.186

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	000	YES	4.900	5.200	5.050	0.150	-18.36
	000	YES	7.700	7.500	7.600	0.100	22.86
	062	YES	6.570	6.460	6.515	0.055	5.32
	022	YES	6.200	6.000	6.100	0.100	-1.39
	023	YES	6.090	6.440	6.265	0.175	1.28
	000	YES	5.930	5.940	5.935	0.005	-4.05
	027	YES	6.100	6.200	6.150	0.050	-0.58
	088	YES	6.780	6.628	6.704	0.076	8.38
	021	YES	6.400	6.800	6.600	0.200	6.70
	033	YES	4.700	4.500	4.600	0.100	-25.64
	000	YES	6.480	6.370	6.425	0.055	3.87
	056	YES	6.120	6.450	6.285	0.165	1.60
NBS	M01	NBS	6.400		6.400	0.000	3.46
NBS	M02	NBS	6.290		6.290	0.000	1.69
NBS	M03	NBS	6.670		6.670	0.000	7.83

STANDARD NBS ANALYTE=α-TOCOPHEROL SOLUTION #019 GRAND AVG = 12.474

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	000	YES	11.800	11.800	11.800	0.000	-5.40
	000	YES	14.600	14.600	14.600	0.000	17.05
	023	YES	13.000	12.900	12.950	0.050	3.82
	084	YES	12.900	12.700	12.800	0.100	2.62
	032	YES	12.610	12.000	12.305	0.305	-1.35
	000	YES	10.500	10.600	10.550	0.050	-15.42
	008	YES	12.400	12.400	12.400	0.000	-0.59
	051	YES	12.382	12.456	12.419	0.037	-0.44
	026	YES	12.900	13.300	13.100	0.200	5.02
	055	YES	11.200	10.400	10.800	0.400	-13.42
	000	YES	13.110	13.000	13.055	0.055	4.66
	054	YES	12.940	12.870	12.905	0.035	3.46
NBS	M01	NBS	12.860		12.860	0.000	3.10
NBS	M02	NBS	12.500		12.500	0.000	0.21
NBS	M03	NBS	13.280		13.280	0.000	6.46

STANDARD NBS			ANALYTE=β-CAROTENE		SOLUTION #018	GRAND AVG =	0.435
	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	012	YES	0.490	0.450	0.470	0.020	7.97
	000	YES	0.440	0.440	0.440	0.000	1.08
	000	NFI	1.270	1.230	1.250	0.020	187.15
	073	NFI	1.020	1.100	1.060	0.040	143.50
	065	YES	0.470	0.470	0.470	0.000	7.97
	000	OUT	1.260	1.270	1.265	0.005	190.59
	000	YES	0.420	0.440	0.430	0.010	-1.22
	062	YES	0.567	0.542	0.555	0.013	27.38
	049	NFI	1.240		1.240	0.000	184.85
	022	YES	0.507	0.514	0.511	0.003	17.27
	023	YES	0.425	0.442	0.434	0.009	-0.42
	000	YES	0.401	0.412	0.407	0.005	-6.62
	027	YES	0.490	0.480	0.485	0.005	11.41
	088	YES	0.116	0.121	0.119	0.002	-72.78
	056	OUT	2.080	2.030	2.055	0.025	372.07
NBS	MO1	NBS	0.580		0.580	0.000	33.24
NBS	MO2	NBS	0.470		0.470	0.000	7.97
NBS	MO3	NBS	0.540		0.540	0.000	24.05

STANDARD NBS			ANALYTE=β-CAROTENE		SOLUTION #019	GRAND AVG =	2.924
LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	038	YES	2.600	2.550	2.575	0.025	-11.93
	000	YES	2.940	2.960	2.950	0.010	0.90
	000	NFI	3.510	3.630	3.570	0.060	22.10
	038	NFI	3.300	3.390	3.345	0.045	14.41
	022	YES	2.890	2.870	2.880	0.010	-1.50
	000	YES	3.550	3.460	3.505	0.045	19.88
	000	YES	2.770	2.660	2.715	0.055	-7.14
	023	YES	2.930	2.840	2.885	0.045	-1.33
	019	NFI	3.500		3.500	0.000	19.71
	084	YES	2.630	2.590	2.610	0.020	-10.73
	032	YES	1.640	1.620	1.630	0.010	-44.25
	000	YES	2.690	2.620	2.655	0.035	-9.19
	008	YES	2.630	2.550	2.590	0.040	-11.42
	051	YES	3.335	3.235	3.285	0.050	12.36
	054	YES	4.760	4.850	4.805	0.045	64.34
NBS	MO1	NBS	2.880		2.880	0.000	-1.50
NBS	MO2	NBS	2.770		2.770	0.000	-5.26
NBS	MO3	NBS	2.940		2.940	0.000	0.56

Appendix F. Updated "All-Lab Report" for RR02

The following three pages are the modernized "All-Lab" report for RR02. This report has three parts:

- Page F2 lists the participant mean results for all analytes reported.
- Page F3 provides the legend for page F2.
- Page F4 summarizes each participants' performance for retinol, α-tocopherol, and β-carotene, using the "Comparability Summary" calculations from the 1999 to 2017 Round Robins.

To ensure confidentiality, the laboratory identifiers used in this "All-Lab Report" have been altered from those used in RR02. 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 III Laboratory Results

	Total Retinol, µg/mL				α-Tocopherol, µg/mL				Total β-Carotene, µg/mL				Total Carotene, µg/mL			
Lab	6	7	8	9	6	7	8	9	6	7	8	9	6	7	8	9
FSV-BD	1.330	0.810	0.325	1.135					0.495	0.380	0.100	0.350				
FSV-BE	1.090	0.580	0.255	1.005	13.6	8.2	3.90	10.9	0.130	0.110	0.090	0.090				
FSV-BF	1.020	0.640	0.225	0.880	11.0	7.8	2.25	11.7	0.470	0.375	*nq*	0.725				
FSV-BG	1.900	1.190	0.380	1.695									1.05	0.44	*nq*	0.97
FSV-BI	1.300	0.853	0.335	1.145					0.008	0.005	*nq*	0.006				
FSV-BY	1.593	1.074	0.441	0.381					0.227	0.109	*nq*	0.052				
FSV-CA	1.305	0.860	0.330	1.200	9.0	5.3	2.20	6.7	0.890	0.600	0.280	0.640				
FSV-CJ	1.399	0.897	0.336	1.205					0.301	0.174	0.015	0.240				
FSV-CL	3.360	2.355	0.865	3.360	30.8	23.0	8.23	22.8	0.420	0.230	*nq*	0.240				
FSV-CN	1.379	0.961	0.518	1.170	4.2	7.6	0.81	2.9								
FSV-DE					12.0	8.3	3.21	9.1								
FSV-DG	2.800	2.100	0.630	2.700	22.1	15.4	5.37	16.8	0.660	0.365	0.035	0.640				
FSV-DH	1.325	0.887	0.336	1.265	8.5	6.6	2.73	5.4	*nq*	0.031	*nq*	0.041				
FSV-DN	1.150	0.700	0.275	0.920	8.5	5.7	2.05	5.5	0.051	0.034	*nq*	0.027				
FSV-DO	1.740	1.410	0.420	1.360												
FSV-DT	0.980	0.720	0.290	0.950	7.6	4.9	2.15	5.4								
FSV-EG	1.585	1.040	0.345	1.470									1.01	0.61	*nq*	0.57
FSV-EP	1.310	0.830	0.290	1.115	6.0	4.6	1.77	4.5								
FSV-ER	1.205	0.770	0.310	1.045	8.8	6.3	2.47	7.3	*nq*	0.095	*nq*	*nq*				
FSV-EU	2.285	1.425	0.175	2.000					0.132	0.081	*nd*	0.136				
n	19	19	19	19	12	12	12	12	11	13	5	12	2	2	0	2
Min	0.980	0.580	0.175	0.381	4.2	4.6	0.81	2.9	0.008	0.005	0.015	0.006	1.01	0.44		0.57
Median	1.330	0.887	0.335	1.170	8.9	7.1	2.36	7.0	0.301	0.110	0.090	0.188	1.03	0.53		0.77
Max	3.360	2.355	0.865	3.360	30.8	23.0	8.23	22.8	0.890	0.600	0.280	0.725	1.05	0.61		0.97
eSD	0.356	0.248	0.067	0.282	3.7	2.0	0.72	3.4	0.254	0.117	0.082	0.228				
eCV	27	28	20	24	42	28	30	49	84	106	91	121				
NIST	1.350	0.890	0.370	1.250	11.0	7.1	2.60	7.2	0.090	0.400	0.130	0.140				
NAV	1.330	0.887	0.335	1.170	8.9	7.1	2.36	7.0	0.301	0.110	0.090	0.188				
NAU	0.356	0.248	0.067	0.282	3.7	2.0	0.72	3.4	0.254	0.117	0.082	0.228				

Round Robin III Laboratory Results

Analytes Reported By One Laboratory

Analyte	Code	6	7	8	9
γ/β-Tocopherol, μg/mL	FSV-DE	2.585	1.635	0.795	1.850

Legend

Term	Definition
n	Number of (non-NIST) quantitative values reported for this analyte
Min	Minimum (non-NIST) quantitative value reported
Median	Median (non-NIST) quantitative value reported
Max	Maximum (non-NIST) quantitative value reported
eSD	Estimated standard deviation, calculated from the median absolute deviation from the median of the non-NIST results
eCV	Coefficient of Variation for (non-NIST) results: 100*eSD/Median
NAV	NIST Assigned Value, our estimate of the "true" analyte concentration
NAU	NIST Assigned Uncertainty, our estimate of the total (serum heterogeniety and inter- and intra-laboratory) standard deviation

Round Robin III Laboratory Results

Comparability Summary

Lab	TR	aT	bC
FSV-BD	1		2
FSV-BE	1	2	1
FSV-BF	2	1	2
FSV-BG	2		
FSV-BI	1		1
FSV-BY	2		1
FSV-CA	1	1	3
FSV-CJ	1		1
FSV-CL	4	4	1
FSV-CN	2	2	
FSV-DE		1	
FSV-DG	4	4	2
FSV-DH	1	1	1
FSV-DN	1	1	1
FSV-DO	2		
FSV-DT	1	1	
FSV-EG	1		
FSV-EP	1	1	
FSV-ER	1	1	
FSV-EU	3		1
NIST	1	1	2
n	20	13	13

Label	Definition
Lab	laboratory number
TR	"Standard Score" for Retinol
aT	"Standard Score" for α-Tocopherol
bC	"Standard Score" for Total β-Carotene
n	number of (non-NIST) laboratories providing data for this analyte

"Standard Score"

Given that our knowledge of the shape, location, and width of the measurement distributions is approximate and that a limited number of labs are involved, we summarize comparability with the following four-level "Standard Score" (StS)…

StS	Definition
1	All StV within ±1 SD
2	All StV within ±2 SD
3	All StV within ±3 SD
4	At least one StV >3 SD

where:	
SD	Total measurement standard deviation (SD), including serum heterogeneity and inter-and intra-laboratory variability.
StV	Standardized Value, the distance in SD units your value is from the "true" concentration: StV = (your value - NAV) / NAU
NAV	NIST Assigned Value, our estimate of the "true" analyte concentration
NAU	NIST Assigned Uncertainty, our estimate of the SD

	TR	aT	bC	Expected
% 1	60	69	62	68.2 %
% 2	25	15	31	27.3 %
% 3	5	0	8	4.3 %

These are the observed and normal-population-expected proportions of each Standard Score (StS), based upon each laboratory's largest StV for the four sera.

Appendix G. Shipping Package Inserts for RR03

Two items were attached to each package shipped to an RR03 participant:

- **Cover letter**. The original letter has been lost. It would have described the four liquid-frozen (sera 6, 7, 8, and 9) distributed to participants who were interested in the fat-soluble component of the study and the three dithiothreitol-stabilized samples (sera 10, 11, and 12) distributed to participants interested in the ascorbic acid component.

  Sera 6 and 7 had the same matrix as serum 8 but were gravimetrically spiked with retinol, α-tocopherol, and β-carotene. Serum 9 was prepared in a "stripped" serum matrix to have the same nominal retinol, α-tocopherol, and β-carotene levels as serum 6.

- **Datasheet**. Page G2 reproduces the report form. The same sheet was used to report both the fat-soluble analytes and/or the ascorbic acid.

Report on NBS/NCI Samples from Laboratory #____

Results in mg/L

Samples		Result 1	Result 2
Serum 6 Vial No. ____ Analysis Date 3/ /85	β-carotene		
	retinol		
	α-tocopherol		
Serum 7 Vial No. ____ Analysis Date 3/ /85	β-carotene		
	retinol		
	α-tocopherol		
Serum 8 Vial No. ____ Analysis Date 3/ /85	β-carotene		
	retinol		
	α-tocopherol		
Serum 9 Vial No. ____ Analysis Date 3/ /85	β-carotene		
	retinol		
	α-tocopherol		
Serum 10 Vial No. ____ Analysis Date 3/ /85	ascorbic acid		
Serum 11 Vial No. ____ Analysis Date 3/ /85	ascorbic acid		
Serum 12 Vial No. ____ Analysis Date 3/ /85	ascorbic acid		

Appendix H. Final Report for RR03

The following 10 pages present the following:

- Page H2: Cover letter.
- Page H3: “Statistical Conclusions” and “Comments on the Samples.”
- Page H4: “Statistical Summary” and “Explanation of Columns Headings.”
- Pages H5 to H10: “Standard Lab” results for retinol, α-tocopherol, and β-carotene. These results were obtained using the participants’ own calibration solutions.

April 29, 1985

[Name and address]

Dear [First Name]:

We enclosed our statistical conclusions with the tabular statistical summary and tables of lab results for Study III, the analyses of sera 6-9 and 13-15. The tabular materials were distributed at the workshop on April 11 and 12.

Our comments on the analytical samples which are given on the same page of the enclosure as the statistical conclusions were discussed at the workshop.

Natural sera are to be used for the next interlaboratory comparisons. When these samples will be available for distribution is not yet certain. We will keep you informed.

Sincerely,

Robert Schaffer, Ph.D.
Supervisory Research Chemist
Organic Analytical Research Division
Center for Analytical Chemistry

Enclosure

Statistical Conclusions for Round Robin III Serum Samples

1. For β-carotene, the relative imprecision of a single measurement (% CV X) is very large (~ 100%).

2. For the other analytes, excluding β-carotene, the relative imprecision of a single measurement (% CV X) is generally 5-40%. This is approximately the same as observed with the non-serum samples of Round Robins I and II.

3. The variability between different laboratories, s_b, is generally the largest source of variability.

- -

Comments on the Samples

Serum samples 6 and 7 were prepared by adding known quantities of β-carotene, retinol, and α-tocopherol to serum 8. α-carotene and γ-tocoperol were also added. Expected values for the analytes in 6 and 7 were calculated from the amounts weighed-in plus the grand average values the labs found for serum 8. Serum 9 was spiked like serum 6, but with a different serum as the matrix. This matrix for serum 9 contained very low levels of the analytes. In mg/L, the expected values were:

	Serum 6	Serum 7	Serum 8 (Grand Avg)	Serum 9
β-carotene	0.6	0.3	0.02	0.6
retinol	2.0	1.2	0.36	1.6
α-tocopherol	12.4	7.4	2.35	10

Relative to these expected values, the grand averages that the labs obtained for these analytes were low; this is particularly evident for β-carotene. The analysis for β-carotene done at NBS revealed extraction-resistant yellow pigment in the protein precipitates for sera 6, 7 and 9. These pigmentation levels paralleled the spiking levels. At this time we have no explanation to account for any of these differences.

The amounts of ascorbic acid added to sera 14 and 15 gave concentrations that were exceptionally high. Nevertheless, the dithiothreitol in the sera stabilized the ascorbic acid levels. These samples appear, however, to be suited only for HPLC analysis.

Statistical Summary

STANDARD LAB ANALYTE=RETINOL

SOL	GRAND AVG	S.E. AVG	S WITHIN	S BETWEEN	%CV AVG	%CV X	FR REJECT
006	1.351	0.063	0.054	0.249	4.7	18.9	2/18
007	0.889	0.053	0.047	0.209	6.0	24.1	2/18
008	0.355	0.024	0.022	0.099	6.8	28.5	1/18
009	1.121	0.072	0.054	0.285	6.4	25.9	2/18

STANDARD LAB ANALYTE=α-TOCOPHEROL

SOL	GRAND AVG	S.E. AVG	S WITHIN	S BETWEEN	%CV AVG	%CV X	FR REJECT
006	8.903	0.877	0.667	2.734	9.9	31.6	2/12
007	6.521	0.440	0.334	1.373	6.8	21.7	2/12
008	2.353	0.262	0.077	0.828	11.1	35.3	2/12
009	6.927	0.893	0.339	2.814	12.9	40.9	2/12

STANDARD LAB ANALYTE=β-CAROTENE

SOL	GRAND AVG	S.E. AVG	S WITHIN	S BETWEEN	%CV AVG	%CV X	FR REJECT
006	0.251	0.070	0.040	0.232	28.1	93.8	0/11
007	0.173	0.043	0.039	0.141	25.0	84.3	0/11
008	0.022	0.011	0.021	0.034	51.4	183.8	0/11
009	0.219	0.077	0.040	0.255	35.3	117.8	0/11

Explanation of Column Headings

SOL = solution number

GRAND AVG = grand average (consensus value from analysis of variance) = $\bar{x}$ (mg/L)

S.E.AVG = one standard deviation (std. dev.) of $\bar{x}$ = $S_{\bar{x}}$

S WITHIN = within laboratory component of std. dev. = S_w (square root of within laboratory component of variance from the ANOVA; a pooled estimate of within laboratory imprecision)

S BETWEEN = between laboratory component of standard deviation = S_b

%CV AVG = percent coefficient of variation of the grand average = 100 $S_{\bar{x}}/\bar{x}$

%CV X = percent coefficient of variation of a single measurement made by a single laboratory = 100 $S_x/\bar{x}$, where $S_x = \sqrt{S_w^2 + S_b^2}$ (NOTE: S_x is not equal to the std. dev. of all measurements)

FR REJECT = ratio of rejected to total laboratory results considered for statistical evaluation

STANDARD LAB ANALYTE=RETINOL SOLUTION #006 GRAND AVG = 1.351

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	097	YES	1.627	1.559	1.593	0.034	17.94
	078	YES	1.280	1.320	1.300	0.020	-3.75
	009	YES	1.890	1.910	1.900	0.010	40.67
	045	YES	1.560	1.610	1.585	0.025	17.35
	077	YES	1.427	1.371	1.399	0.028	3.58
	024	YES	1.800	1.680	1.740	0.060	28.83
	039	YES	1.260	1.350	1.305	0.045	-3.38
	019	YES	1.230	1.180	1.205	0.025	-10.78
	013	YES	1.360	1.300	1.330	0.030	-1.53
	004	YES	1.100	1.200	1.150	0.050	-14.86
	021	YES	1.360	1.397	1.379	0.018	2.06
	068	YES	1.420	1.230	1.325	0.095	-1.90
	085	OUT	3.280	3.440	3.360	0.080	148.77
	035	YES	0.990	1.050	1.020	0.030	-24.48
	081	YES	1.000	0.960	0.980	0.020	-27.44
	087	YES	1.310	1.310	1.310	0.000	-3.01
	000	YES	1.090	1.090	1.090	0.000	-19.30
	080	OUT	2.800	2.800	2.800	0.000	107.31
NBS	007	NBS	1.350		1.350	0.000	-0.05

STANDARD LAB ANALYTE=RETINOL SOLUTION #007 GRAND AVG = 0.889

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	018	YES	1.011	1.136	1.074	0.063	20.78
	070	YES	0.855	0.850	0.853	0.003	-4.08
	006	YES	1.170	1.210	1.190	0.020	33.89
	023	YES	1.010	1.070	1.040	0.030	17.01
	024	YES	0.918	0.876	0.897	0.021	0.92
	042	YES	1.320	1.500	1.410	0.090	58.64
	077	YES	0.880	0.840	0.860	0.020	-3.24
	089	YES	0.760	0.780	0.770	0.010	-13.36
	008	YES	0.830	0.790	0.810	0.020	-8.86
	035	YES	0.670	0.730	0.700	0.030	-21.24
	007	YES	0.968	0.954	0.961	0.007	8.13
	067	YES	0.874	0.899	0.887	0.013	-0.26
	050	OUT	2.500	2.210	2.355	0.145	164.97
	027	YES	0.640	0.640	0.640	0.000	-27.99
	087	YES	0.680	0.760	0.720	0.040	-18.99
	092	YES	0.820	0.840	0.830	0.010	-6.61
	000	YES	0.590	0.570	0.580	0.010	-34.74
	047	OUT	2.130	2.070	2.100	0.030	136.28
NBS	004	NBS	0.890		0.890	0.000	0.14

STANDARD LAB ANALYTE=RETINOL SOLUTION #008 GRAND AVG = 0.355

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	042	YES	0.452	0.429	0.441	0.011	23.95
	016	YES	0.320	0.350	0.335	0.015	-5.74
	038	YES	0.390	0.370	0.380	0.010	6.93
	005	YES	0.330	0.360	0.345	0.015	-2.92
	069	YES	0.342	0.331	0.336	0.006	-5.31
	022	YES	0.440	0.400	0.420	0.020	18.18
	052	YES	0.340	0.320	0.330	0.010	-7.14
	015	YES	0.320	0.300	0.310	0.010	-12.77
	018	YES	0.360	0.290	0.325	0.035	-8.55
	073	YES	0.270	0.280	0.275	0.005	-22.62
	031	YES	0.503	0.533	0.518	0.015	45.76
	077	YES	0.318	0.355	0.336	0.018	-5.31
	044	OUT	0.910	0.820	0.865	0.045	143.40
	001	YES	0.220	0.230	0.225	0.005	-36.69
	009	YES	0.320	0.260	0.290	0.030	-18.40
	013	YES	0.290	0.290	0.290	0.000	-18.40
	000	YES	0.250	0.260	0.255	0.005	-28.25
	017	YES	0.630	0.630	0.630	0.000	77.27
NBS	029	NBS	0.370		0.370	0.000	4.11

STANDARD LAB ANALYTE=RETINOL SOLUTION #009 GRAND AVG = 1.121

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	034	YES	0.358	0.404	0.381	0.023	-66.02
	023	YES	1.130	1.160	1.145	0.015	2.12
	022	YES	1.600	1.790	1.695	0.095	51.17
	024	YES	1.390	1.550	1.470	0.080	31.10
	068	YES	1.252	1.157	1.205	0.047	7.42
	054	YES	1.400	1.320	1.360	0.040	21.29
	048	YES	1.200	1.200	1.200	0.000	7.02
	093	YES	1.030	1.060	1.045	0.015	-6.80
	005	YES	1.150	1.120	1.135	0.015	1.23
	090	YES	0.870	0.970	0.920	0.050	-17.95
	020	YES	1.163	1.176	1.170	0.007	4.30
	011	YES	1.270	1.260	1.265	0.005	12.82
	086	OUT	3.470	3.250	3.360	0.110	199.67
	007	YES	0.880	0.880	0.880	0.000	-21.52
	098	YES	0.940	0.960	0.950	0.010	-15.27
	069	YES	1.110	1.120	1.115	0.005	-0.56
	000	YES	1.010	1.000	1.005	0.005	-10.37
	030	OUT	2.670	2.730	2.700	0.030	140.80
NBS	065	NBS	1.250		1.250	0.000	11.48

STANDARD LAB ANALYTE=α-TOCOPHEROL SOLUTION #006 GRAND AVG = 8.903

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	039	YES	9.700	8.200	8.950	0.750	0.52
	019	YES	8.570	8.930	8.750	0.180	-1.72
	004	YES	8.500	8.500	8.500	0.000	-4.53
	021	YES	4.179	4.130	4.155	0.025	-53.34
	068	YES	8.480	8.570	8.525	0.045	-4.25
	085	OUT	32.200	29.300	30.750	1.450	245.37
	035	YES	9.770	12.180	10.975	1.205	23.27
	081	YES	7.960	7.180	7.570	0.390	-14.98
	087	YES	6.060	5.930	5.995	0.065	-32.67
	012	YES	12.000	11.950	11.975	0.025	34.50
	000	YES	13.780	13.500	13.640	0.140	53.20
	080	OUT	22.150	22.000	22.075	0.075	147.94
NBS	007	NBS	11.000		11.000	0.000	23.55

STANDARD LAB ANALYTE=α-TOCOPHEROL SOLUTION #007 GRAND AVG = 6.521

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	077	YES	5.700	4.900	5.300	0.400	-18.73
	089	YES	6.480	6.160	6.320	0.160	-3.09
	035	YES	5.900	5.400	5.650	0.250	-13.36
	007	YES	7.508	7.600	7.554	0.046	15.83
	067	YES	6.360	6.750	6.555	0.195	0.52
	050	OUT	22.100	23.800	22.950	0.850	251.92
	027	YES	7.930	7.670	7.800	0.130	19.61
	087	YES	5.400	4.400	4.900	0.500	-24.86
	092	YES	4.630	4.560	4.595	0.035	-29.54
	043	YES	8.390	8.300	8.345	0.045	27.96
	000	YES	8.190	8.200	8.195	0.006	25.66
	047	OUT	15.410	15.410	15.410	0.000	136.30
NBS	004	NBS	7.120		7.120	0.000	9.18

STANDARD LAB ANALYTE=α-TOCOPHEROL SOLUTION #008 GRAND AVG = 2.353

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	052	YES	2.200	2.200	2.200	0.000	-6.50
	015	YES	2.460	2.480	2.470	0.010	4.97
	073	YES	2.100	2.000	2.050	0.050	-12.88
	031	YES	0.850	0.759	0.805	0.046	-65.81
	077	YES	2.810	2.650	2.730	0.080	16.02
	044	OUT	8.340	8.120	8.230	0.110	249.77
	001	YES	2.270	2.230	2.250	0.020	-4.38
	009	YES	2.180	2.120	2.150	0.030	-8.63
	013	YES	1.740	1.790	1.765	0.025	-24.99
	003	YES	3.160	3.260	3.210	0.050	36.42
	000	YES	3.780	4.020	3.900	0.120	65.75
	017	OUT	5.410	5.330	5.370	0.040	128.22
NBS	029	NBS	2.600		2.600	0.000	10.50

STANDARD LAB ANALYTE=α-TOCOPHEROL SOLUTION #009 GRAND AVG = 6.927

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	048	YES	6.300	7.100	6.700	0.400	-3.28
	093	YES	7.360	7.140	7.250	0.110	4.66
	090	YES	5.600	5.300	5.450	0.150	-21.33
	020	YES	3.010	2.849	2.930	0.080	-57.71
	011	YES	5.110	5.760	5.435	0.325	-21.54
	086	OUT	22.300	23.300	22.800	0.500	229.13
	007	YES	11.380	11.980	11.680	0.300	68.60
	098	YES	5.720	5.060	5.390	0.330	-22.19
	069	YES	4.340	4.600	4.470	0.130	-35.47
	044	YES	8.880	9.330	9.105	0.225	31.43
	000	YES	10.910	10.820	10.865	0.045	56.84
	030	OUT	17.190	16.450	16.820	0.370	142.80
NBS	065	NBS	7.170		7.170	0.000	3.50

STANDARD LAB			ANALYTE=β-CAROTENE		SOLUTION #006	GRAND AVG =	0.251
LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	089	YES	0.221	0.233	0.227	0.006	-9.58
	078	YES	0.008	0.008	0.008	0.000	-96.81
	009	NFI	1.060	1.030	1.045	0.015	316.26
	045	NFI	1.010	1.010	1.010	0.000	302.32
	029	YES	0.356	0.245	0.301	0.056	19.70
	039	NFI	0.890		0.890	0.000	254.52
	019	YES	0.000	0.000	0.000	0.000	-100.00
	013	YES	0.530	0.460	0.495	0.035	97.18
	004	YES	0.045	0.057	0.051	0.006	-79.68
	068	YES	0.000	0.000	0.000	0.000	-100.00
	085	YES	0.390	0.450	0.420	0.030	67.30
	035	YES	0.410	0.530	0.470	0.060	87.22
	000	YES	0.130	0.130	0.130	0.000	-48.22
	080	YES	0.670	0.650	0.660	0.010	162.90
NBS	007	NBS	0.090		0.090	0.000	-64.15

STANDARD LAB			ANALYTE=β-CAROTENE		SOLUTION #007	GRAND AVG =	0.173
LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	085	YES	0.105	0.112	0.109	0.004	-37.40
	070	YES	0.004	0.005	0.005	0.001	-97.40
	006	NFI	0.440	0.440	0.440	0.000	153.87
	023	NFI	0.670	0.550	0.610	0.060	251.95
	031	YES	0.175	0.172	0.174	0.002	0.10
	077	NFI	0.600		0.600	0.000	246.18
	089	YES	0.080	0.110	0.095	0.015	-45.19
	008	YES	0.400	0.360	0.380	0.020	119.25
	035	YES	0.030	0.038	0.034	0.004	-80.38
	067	YES	0.030	0.032	0.031	0.001	-82.11
	050	YES	0.220	0.240	0.230	0.010	32.70
	027	YES	0.460	0.290	0.375	0.085	116.37
	000	YES	0.120	0.100	0.110	0.010	-36.53
	047	YES	0.350	0.380	0.365	0.015	110.60
NBS	004	NBS	0.400		0.400	0.000	130.79

STANDARD LAB			ANALYTE=β-CAROTENE		SOLUTION #008	GRAND AVG =	0.022
LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	078	YES	0.006	0.000	0.003	0.003	-86.42
	016	YES	0.000	0.000	0.000	0.000	-100.00
	038	NFI	0.000	0.000	0.000	0.000	-100.00
	005	NFI	0.000	0.000	0.000	0.000	-100.00
	011	YES	0.015	0.015	0.015	0.000	-32.10
	052	NFI	0.280		0.280	0.000	1167.49
	015	YES	0.000	0.000	0.000	0.000	-100.00
	018	YES	0.150	0.050	0.100	0.050	352.67
	073	YES	0.000	0.000	0.000	0.000	-100.00
	077	YES	0.000	0.000	0.000	0.000	-100.00
	044	YES	0.000	0.000	0.000	0.000	-100.00
	001	YES	0.000	0.000	0.000	0.000	-100.00
	000	YES	0.090	0.090	0.090	0.000	307.41
	017	YES	0.040	0.030	0.035	0.005	58.44
NBS	029	NBS	0.130		0.130	0.000	488.48

STANDARD LAB ANALYTE=β-CAROTENE SOLUTION #009 GRAND AVG = 0.219

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
015	009	YES	0.051	0.053	0.052	0.001	-76.26
016	023	YES	0.006	0.006	0.006	0.000	-97.26
017	022	NFI	0.890	1.040	0.965	0.075	340.55
018	024	NFI	0.470	0.670	0.570	0.100	160.22
019	029	YES	0.292	0.187	0.240	0.052	9.34
021	048	NFI	0.640		0.640	0.000	192.18
023	093	YES	0.000	0.000	0.000	0.000	-100.00
026	005	YES	0.370	0.330	0.350	0.020	59.78
027	090	YES	0.032	0.021	0.027	0.006	-87.90
030	011	YES	0.036	0.045	0.041	0.004	-81.51
031	086	YES	0.240	0.240	0.240	0.000	9.57
032	007	YES	0.650	0.800	0.725	0.075	230.98
046	000	YES	0.090	0.090	0.090	0.000	-58.91
047	030	YES	0.640	0.640	0.640	0.000	192.18
NBS	065	NBS	0.140		0.140	0.000	-36.09

Appendix I. Updated "All-Lab Report" for RR03

The following three pages are the modernized "All-Lab" report for RR03. This report has three parts:

- Page I2 lists the participant mean results for all analytes reported.
  Note: two participants indicated that their method did not fully resolve α- carotene from β-carotene and they quantified the composite peak area as if it were just β-carotene. The reported "β-carotene" values for these participants were recorded as "Total carotene".
- Page I3 lists the unsolicited γ-tocopherol results reported by one participant. This page also provides the legend for page I2.
- Page I4 summarizes each participants' performance for retinol, α-tocopherol, and β-carotene, using the "Comparability Summary" calculations from the 1999 to 2017 Round Robins.

To ensure confidentiality, the laboratory identifiers used in this "All-Lab Report" have been altered from those used in RR03. 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 III Laboratory Results

	Total Retinol, µg/mL				α-Tocopherol, µg/mL				Total β-Carotene, µg/mL				Total Carotene, µg/mL			
Lab	6	7	8	9	6	7	8	9	6	7	8	9	6	7	8	9
FSV-BD	1.330	0.810	0.325	1.135					0.495	0.380	0.100	0.350				
FSV-BE	1.090	0.580	0.255	1.005	13.6	8.2	3.90	10.9	0.130	0.110	0.090	0.090				
FSV-BF	1.020	0.640	0.225	0.880	11.0	7.8	2.25	11.7	0.470	0.375	*nq*	0.725				
FSV-BG	1.900	1.190	0.380	1.695									1.05	0.44	*nq*	0.97
FSV-BI	1.300	0.853	0.335	1.145					0.008	0.005	*nq*	0.006				
FSV-BY	1.593	1.074	0.441	0.381					0.227	0.109	*nq*	0.052				
FSV-CA	1.305	0.860	0.330	1.200	9.0	5.3	2.20	6.7	0.890	0.600	0.280	0.640				
FSV-CJ	1.399	0.897	0.336	1.205					0.301	0.174	0.015	0.240				
FSV-CL	3.360	2.355	0.865	3.360	30.8	23.0	8.23	22.8	0.420	0.230	*nq*	0.240				
FSV-CN	1.379	0.961	0.518	1.170	4.2	7.6	0.81	2.9								
FSV-DE					12.0	8.3	3.21	9.1								
FSV-DG	2.800	2.100	0.630	2.700	22.1	15.4	5.37	16.8	0.660	0.365	0.035	0.640				
FSV-DH	1.325	0.887	0.336	1.265	8.5	6.6	2.73	5.4	*nq*	0.031	*nq*	0.041				
FSV-DN	1.150	0.700	0.275	0.920	8.5	5.7	2.05	5.5	0.051	0.034	*nq*	0.027				
FSV-DO	1.740	1.410	0.420	1.360												
FSV-DT	0.980	0.720	0.290	0.950	7.6	4.9	2.15	5.4								
FSV-EG	1.585	1.040	0.345	1.470									1.01	0.61	*nq*	0.57
FSV-EP	1.310	0.830	0.290	1.115	6.0	4.6	1.77	4.5								
FSV-ER	1.205	0.770	0.310	1.045	8.8	6.3	2.47	7.3	*nq*	0.095	*nq*	*nq*				
FSV-EU	2.285	1.425	0.175	2.000					0.132	0.081	*nd*	0.136				
n	19	19	19	19	12	12	12	12	11	13	5	12	2	2	0	2
Min	0.980	0.580	0.175	0.381	4.2	4.6	0.81	2.9	0.008	0.005	0.015	0.006	1.01	0.44		0.57
Median	1.330	0.887	0.335	1.170	8.9	7.1	2.36	7.0	0.301	0.110	0.090	0.188	1.03	0.53		0.77
Max	3.360	2.355	0.865	3.360	30.8	23.0	8.23	22.8	0.890	0.600	0.280	0.725	1.05	0.61		0.97
eSD	0.356	0.248	0.067	0.282	3.7	2.0	0.72	3.4	0.254	0.117	0.082	0.228				
eCV	27	28	20	24	42	28	30	49	84	106	91	121				
NIST	1.350	0.890	0.370	1.250	11.0	7.1	2.60	7.2	0.090	0.400	0.130	0.140				
NAV	1.330	0.887	0.335	1.170	8.9	7.1	2.36	7.0	0.301	0.110	0.090	0.188				
NAU	0.356	0.248	0.067	0.282	3.7	2.0	0.72	3.4	0.254	0.117	0.082	0.228				

Round Robin III Laboratory Results

Analytes Reported By One Laboratory

Analyte	Code	6	7	8	9
γ/β-Tocopherol, μg/mL	FSV-DE	2.585	1.635	0.795	1.850

Legend

Term	Definition
n	Number of (non-NIST) quantitative values reported for this analyte
Min	Minimum (non-NIST) quantitative value reported
Median	Median (non-NIST) quantitative value reported
Max	Maximum (non-NIST) quantitative value reported
eSD	Estimated standard deviation, calculated from the median absolute deviation from the median of the non-NIST results
eCV	Coefficient of Variation for (non-NIST) results: 100*eSD/Median
NAV	NIST Assigned Value, our estimate of the "true" analyte concentration
NAU	NIST Assigned Uncertainty, our estimate of the total (serum heterogeniety and inter- and intra-laboratory) standard deviation

Round Robin III Laboratory Results

Comparability Summary

Lab	TR	aT	bC
FSV-BD	1		2
FSV-BE	1	2	1
FSV-BF	2	1	2
FSV-BG	2		
FSV-BI	1		1
FSV-BY	2		1
FSV-CA	1	1	3
FSV-CJ	1		1
FSV-CL	4	4	1
FSV-CN	2	2	
FSV-DE		1	
FSV-DG	4	4	2
FSV-DH	1	1	1
FSV-DN	1	1	1
FSV-DO	2		
FSV-DT	1	1	
FSV-EG	1		
FSV-EP	1	1	
FSV-ER	1	1	
FSV-EU	3		1
NIST	1	1	2
n	20	13	13

Label	Definition
Lab	laboratory number
TR	"Standard Score" for Retinol
aT	"Standard Score" for α-Tocopherol
bC	"Standard Score" for Total β-Carotene
n	number of (non-NIST) laboratories providing data for this analyte

"Standard Score"

Given that our knowledge of the shape, location, and width of the measurement distributions is approximate and that a limited number of labs are involved, we summarize comparability with the following four-level "Standard Score" (StS)…

StS	Definition
1	All StV within ±1 SD
2	All StV within ±2 SD
3	All StV within ±3 SD
4	At least one StV >3 SD

where:	
SD	Total measurement standard deviation (SD), including serum heterogeneity and inter-and intra-laboratory variability.
StV	Standardized Value, the distance in SD units your value is from the "true" concentration: StV = (your value - NAV) / NAU
NAV	NIST Assigned Value, our estimate of the "true" analyte concentration
NAU	NIST Assigned Uncertainty, our estimate of the SD

	TR	aT	bC	Expected
% 1	60	69	62	68.2 %
% 2	25	15	31	27.3 %
% 3	5	0	8	4.3 %

These are the observed and normal-population-expected proportions of each Standard Score (StS), based upon each laboratory's largest StV for the four sera.

Appendix J. Shipping Package Inserts for RR03

The following three items were attached to each package shipped to RR03 participants:

- Cover letter, describing the samples and calibration solutions used in the study.
- “Lab standard” datasheet for reporting results based upon the participants’ own calibration solutions.
- “NBS calibration solution” for reporting results based upon the NBS-provided calibration solutions.

Report on NBS/NCI Samples from Laboratory #____

Results in mg/L

Based on lab standards

Samples		Result 1	Result 2
Serum No: 19 Vial No. _____ Analysis Date / /85	Retinol β-Carotene α-Tocopherol		
Serum No: 20 Vial No. _____ Analysis Date / /85	Retinol β-Carotene α-Tocopherol Ascorbic Acid		
Serum No: 21 Vial No. _____ Analysis Date / /85	Retinol β-Carotene α-Tocopherol		

Report on NBS/NCI Samples from Laboratory #____

Results in mg/L

Based on NBS calibration solutions

Samples		Result 1	Result 2
Serum No: 19 Vial No. _____ Analysis Date / /85	Retinol β-Carotene α-Tocopherol		
Serum No: 20 Vial No. _____ Analysis Date / /85	Retinol β-Carotene α-Tocopherol Ascorbic Acid		
Serum No: 21 Vial No. _____ Analysis Date / /85	Retinol β-Carotene α-Tocopherol		

Appendix K. Final Report for RR05

The following 17 pages present the following:

- Pages K2 to K3: Cover letter.
- Pagea K4 to K5: “Statistical Summary” and its legend.
- Pages K6 to K9: “Standard Lab” results obtained using the participants’ own calibration solutions.
- Pages K10 to K14: “Standard NBS” results, obtained using the NBS-provided calibration solutions.
- Page K15: “Distribution of Biases” table.
- Pages K16 to K18: Statistican’s report for RR06 that, in addition to the Se and Zn results of RR06, describes the individualized graphical analysis of the RR05 β-carotene results. None of the original graphs are extant.
- Page K19: A table listing least-square parameter values of intercept, slope, and “S fit” (more commonly known as the root-mean square error, RMSE, of the fit) for the RR05 β-carotene results. A modern re-creation of the graphical analysis as an approximate example of the original graphs (which would have been larger and would not have used color).

November 21, 1985

[Name and address]

Dear [First Name]:

This letter concerns Round Robin V (RR V).e Enclosed are:
1)e the RR V data,e

2)e the statistical summary and conclusions, from Dr. Paule, and,e

3)e a table showing the distribution of biases around the grand averages.

I am sorry it has taken so much time to report these results to you. The laboratories that turned in widely deviant results did hear from promptly. (Afterward analysts from one of those labs visited to discuss possible problems with their methods.) The rest of you have been very patient.

The three plasma samples (19, 20, 21) used for RR V were from blood donated by three individuals who had consumed vitamins A and C, vitamin C, and β-carotene and carrots, respectively. Although plasma samples 19 and 21 were not modified, plasma 20 was spiked (with ascorbic acid and dithiothreitol). In enclosure 2, Dr. Paule contrasts the results on these samples with the results from RR III. You may recall that the RR III samples were made from "stripped" sera and everyone seemed to agree that assessments of laboratory performance for these analytes should employ "natural" samples. The results obtained with the samples for RR V were much better than those for RR III.

The second statistical conclusion for RR V is a surprise, i.e., the "standard NBS" results were not more precise than the "standard lab" results. The use of the NBS standard solutions had helped in RR II, where the standards were similar to the unknowns.

We plan to continue to supply NBS solutions, but also to investigate the use of standards that are more closely matched to natural samples.

Enclosure 3 shows frequency distributions for the "standard lab" results. Note that the percent biases are grouped according to absolute differences from the grand averages. The distributions show that many of the results were quite good: more than half of the retinol and α-tocopherol results are within 10% of their averages. Somewhat less than a quarter of the β-carotene values are within 10% of their averages. The distributionse of the "standard NBS" results are similar and are therefore not provided.

We have samples for the next round, and are planning to send with them a questionaire on what we think are significant details of methodology. Answers will be correlated with results. We expect that all of us will benefit from what is learned.

March 17 and 18 have been booked for the next workshop which will again be held at NBS. Please mark your 1986 calendars!

Sincerely,

Robert Schaffer, Ph.D.
Supervisory Research Chemist
Organic Analytical Research Division
Center for Analytical Chemistry

Enclosures

RR V STATISTICAL SUMMARY

SER	GRAND AVG	S.E. AVG	S WITHIN	S BETWEEN	% CV AVG	% CV X	FR REJECT
			STANDARD LAB: ANALYTE=RETINOL				
21	0.421	0.021	0.014	0.092	5.1	22.2	1/20
20	0.469	0.024	0.023	0.104	5.2	22.7	1/20
19	0.507	0.028	0.024	0.121	5.5	24.3	0/19
			STANDARD LAB: ANALYTE=α-TOCOPHEROL				
21	5.916	0.339	0.231	1.258	5.7	21.6	1/15
20	6.935	0.270	0.282	0.953	3.9	14.3	2/15
19	28.287	1.195	0.434	4.299	4.2	15.3	1/14
			STANDARD LAB: ANALYTE=β-CAROTENE				
20	0.136	0.013	0.038	0.048	9.5	44.9	0/18
21	0.382	0.025	0.016	0.104	6.6	27.4	1/18
19	0.504	0.045	0.039	0.172	8.9	35.0	1/16
			STANDARD NBS: ANALYTE=RETINOL				
21	0.390	0.020	0.028	0.085	5.1	22.9	0/19
20	0.423	0.024	0.023	0.105	5.7	25.3	0/19
19	0.476	0.026	0.017	0.114	5.5	24.3	0/19
			STANDARD NBS: ANALYTE=α-TOCOPHEROL				
21	6.304	0.261	0.381	0.904	4.1	15.6	1/14
20	6.991	0.446	0.311	1.653	6.4	24.1	0/14
19	29.296	1.329	0.583	4.772	4.5	16.4	1/14
			STANDARD NBS: ANALYTE=β-CAROTENE				
20	0.114	0.013	0.009	0.048	11.4	43.1	3/17
21	0.405	0.025	0.058	0.090	6.1	26.4	1/17
19	0.480	0.035	0.031	0.132	7.2	28.2	2/17

STATISTICAL CONCLUSIONS FROM ROUND ROBIN V RESULTS

1) For α-tocopherol and β-carotene, the relative precisions [CV X] from the current Round Robin V test with serum samples are much improved: the CV Xs are 1/3 to 1/2 of those observed from the RR III test. For retinol, the relative precisions from the two round robins are about the same. For ascorbic acid, the analyte levels between the two round robins are very different and valid comparisons are not possible.

2) In the current RR the use of NBS ethanolic standards did not result in improved precisions over the lab standards. As usual, the variability between different laboratories, S_b, is the largest source of variability. Use of the NBS solution standards did little to reduce S_b.

Explanation of Column Heading on Statistical Summary

GRAND AVG = grand average (consensus value from analysis of variance) = $\bar{\bar{x}}$ (mg/L)

S.E.AVG = one standard deviation (std. dev.) of $\bar{\bar{x}} = S_{\bar{\bar{x}}}$

S WITHIN = within laboratory component of std. dev. = S_w (square root of within laboratory component of variance from the ANOVA; a pooled estimate of within laboratory imprecision)

S BETWEEN = between laboratory component of standard deviation = S_b

% CV AVG = percent coefficient of variation of the grand average = $100\ S_{\bar{\bar{x}}}/\bar{\bar{x}}$

% CV X = percent coefficient of variation of a single measurement made by a single laboratory = $100\ S_x/\bar{\bar{x}}$, where $S_x = \sqrt{S_w^2 + S_b^2}$ (NOTE: S_x is not equal to the std. dev. of all measurements)

FR REJECT = ratio of rejected to total laboratory results considered for statistical evaluation

RR V DATA (Data Begins on This Page)

STANDARD LAB		ANALYTE=RETINOL	SERUM #19	GRAND AVG =	0.507
LAB	VIAL USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
59	YES	0.395	0.419	0.407	0.012	-19.67
57	YES	0.520	0.530	0.525	0.005	3.62
25	YES	0.420	0.350	0.385	0.035	-24.01
27	YES	0.610	0.600	0.605	0.005	19.41
20	NFI	0.491		0.491	0.000	-3.09
38	YES	0.220	0.200	0.210	0.010	-58.55
	YES	0.470	0.490	0.480	0.010	-5.26
89	YES	0.520	0.520	0.520	0.000	2.63
36	YES	0.495	0.523	0.509	0.014	0.46
8	YES	0.500	0.460	0.480	0.020	-5.26
13	YES	0.470	0.480	0.475	0.005	-6.25
6	YES	0.540	0.540	0.540	0.000	6.58
4	YES	0.451	0.450	0.451	0.001	-11.08
86	YES	0.550	0.480	0.515	0.035	1.65
68	YES	0.460	0.470	0.465	0.005	-8.22
53	YES	0.470	0.430	0.450	0.020	-11.18
23	YES	0.530	0.500	0.515	0.015	1.65
78	YES	0.640	0.570	0.605	0.035	19.41
98	YES	0.810	0.820	0.815	0.005	60.86
65	YES	0.671	0.679	0.675	0.004	33.23
62	NFI	0.850	0.960	0.905	0.055	78.62

STANDARD LAB		ANALYTE=RETINOL	SERUM #20	GRAND AVG =	0.469
LAB	VIAL USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
54	YES	0.289	0.308	0.299	0.010	-36.41
40	YES	0.490	0.510	0.500	0.010	6.52
36	YES	0.350	0.340	0.345	0.005	-26.50
46	YES	0.710	0.670	0.690	0.020	47.00
76	NFI	0.456		0.456	0.000	-2.85
74	YES	0.480	0.420	0.450	0.030	-4.13
	YES	0.440	0.430	0.435	0.005	-7.33
27	YES	0.490	0.450	0.470	0.020	0.13
45	YES	0.434	0.446	0.440	0.006	-6.26
59	YES	0.450	0.440	0.445	0.005	-5.20
22	YES	0.450	0.480	0.465	0.015	-0.94
80	YES	0.480	0.510	0.495	0.015	5.45
66	YES	0.439	0.441	0.440	0.001	-6.26
13	YES	0.460	0.510	0.485	0.025	3.32
23	YES	0.400	0.390	0.395	0.005	-15.85
63	YES	0.440	0.370	0.405	0.035	-13.72
5	YES	0.460	0.450	0.455	0.005	-3.07
50	YES	0.360	0.410	0.385	0.025	-17.98
17	YES	0.740	0.730	0.735	0.005	56.58
	OUT	0.800	0.840	0.820	0.020	74.69
64	YES	0.584	0.586	0.585	0.001	24.63
43	NFI	0.470		0.470	0.000	0.13
47	NFI	0.430	0.470	0.450	0.020	-4.13

STANDARD LAB		ANALYTE=RETINOL	SERUM #21	GRAND AVG =		
LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	7	YES	0.459	0.461	0.460	0.001	9.33
	36	YES	0.440	0.440	0.440	0.000	4.57
	24	YES	0.340	0.330	0.335	0.005	-20.38
	12	YES	0.500	0.510	0.505	0.005	20.02
	77	NFI	0.420		0.420	0.000	-0.18
	14	YES	0.290	0.280	0.285	0.005	-32.27
		YES	0.370	0.380	0.375	0.005	-10.88
	60	YES	0.460	0.450	0.455	0.005	8.14
	41	YES	0.406	0.415	0.410	0.005	-2.44
	44	YES	0.420	0.390	0.405	0.015	-3.75
	71	YES	0.440	0.430	0.435	0.005	3.38
	16	YES	0.340	0.360	0.350	0.010	-16.82
	85	YES	0.410	0.412	0.411	0.001	-2.32
	3	YES	0.360	0.290	0.325	0.035	-22.76
	57	YES	0.370	0.370	0.370	0.000	-12.06
	91	YES	0.370	0.350	0.360	0.010	-14.44
	96	YES	0.430	0.420	0.425	0.005	1.01
	82	YES	0.410	0.390	0.400	0.010	-4.93
	18	YES	0.710	0.700	0.705	0.005	67.55
		OUT	0.940	0.760	0.850	0.090	102.01
	31	YES	0.541	0.545	0.543	0.002	29.05
	51	NFI	0.447		0.447	0.000	6.24
	9	NFI	0.400	0.440	0.420	0.020	-0.18

STANDARD LAB ANALYTE=α-TOCOPHEROL SERUM #19 GRAND AVG = 28.287

LAB	VIAL USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
20	NFI	32.520		32.520	0.000	14.96
	YES	28.200	28.800	28.500	0.300	0.75
89	YES	29.550	29.600	29.575	0.025	4.55
36	YES	30.000	30.000	30.000	0.000	6.06
13	YES	26.400	26.300	26.350	0.050	-6.85
6	YES	24.370	22.810	23.590	0.780	-16.60
4	YES	32.330	32.400	32.365	0.033	14.42
86	YES	29.400	30.300	29.850	0.450	5.53
68	YES	27.600	27.100	27.350	0.250	-3.31
53	YES	25.600	25.200	25.400	0.200	-10.21
23	YES	28.340	28.960	28.650	0.310	1.28
74	YES	30.000	29.400	29.700	0.300	5.00
78	YES	19.350	19.050	19.200	0.150	-32.12
98	OUT	56.240	55.720	55.980	0.261	97.90
65	YES	37.300	37.100	37.200	0.101	31.51

STANDARD LAB ANALYTE=α-TOCOPHEROL SERUM #20 GRAND AVG = 6.935

LAB	VIAL USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
76	NFI	8.260		8.260	0.000	19.10
	YES	7.400	7.100	7.250	0.150	4.54
27	YES	6.960	7.340	7.150	0.190	3.10
45	YES	6.860	6.830	6.845	0.015	-1.30
22	YES	6.600	6.600	6.600	0.000	-4.83
80	YES	5.880	5.820	5.850	0.030	-15.65
66	YES	7.780	7.413	7.597	0.184	9.54
13	YES	8.400	7.200	7.800	0.600	12.47
23	YES	6.500	6.300	6.400	0.100	-7.72
63	YES	6.980	7.000	6.990	0.010	0.79
5	YES	7.020	6.800	6.910	0.110	-0.36
30	YES	7.090	6.990	7.040	0.050	1.51
50	YES	4.620	5.000	4.810	0.190	-30.64
17	OUT	11.670	11.640	11.655	0.016	68.06
	OUT	2.450	2.650	2.550	0.100	-63.23
64	YES	8.860	8.970	8.915	0.055	28.55

STANDARD LAB ANALYTE=α-TOCOPHEROL SERUM #21 GRAND AVG = 5.916

LAB	VIAL USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
77	NFI	7.140		7.140	0.000	20.68
	YES	6.400	6.200	6.300	0.100	6.49
60	YES	7.010	6.390	6.700	0.310	13.25
41	YES	6.240	6.340	6.290	0.050	6.32
71	YES	6.000	5.800	5.900	0.100	-0.27
16	YES	5.110	4.800	4.955	0.155	-16.25
85	YES	7.363	7.092	7.228	0.135	22.16
3	YES	5.400	4.700	5.050	0.350	-14.64
57	YES	5.600	5.400	5.500	0.100	-7.04
91	YES	6.300	6.500	6.400	0.100	8.18
96	YES	6.410	6.210	6.310	0.100	6.66
88	YES	6.370	6.180	6.275	0.095	6.06
82	YES	4.820	4.520	4.670	0.150	-21.06
18	OUT	12.100	12.220	12.160	0.060	105.54
	YES	2.860	3.060	2.960	0.100	-49.97
31	YES	8.160	8.420	8.290	0.130	40.12

STANDARD LAB ANALYTE=β-CAROTENE SERUM #19 GRAND AVG = 0.504

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	59	YES	0.295	0.415	0.355	0.060	-29.51
	57	YES	0.370	0.385	0.378	0.008	-25.04
	25	YES	0.400	0.280	0.340	0.060	-32.49
	27	YES	0.590	0.610	0.600	0.010	19.13
	89	YES	0.490	0.480	0.485	0.005	-3.70
	36	YES	0.519	0.484	0.502	0.017	-0.42
	13	YES	0.410	0.410	0.410	0.000	-18.59
	6	YES	0.450	0.440	0.445	0.005	-11.64
	4	OUT	0.063	0.062	0.063	0.001	-87.59
	86	YES	0.600	0.600	0.600	0.000	19.13
	68	YES	0.179	0.180	0.180	0.001	-64.36
	78	YES	0.770	0.800	0.785	0.015	55.87
	98	YES	0.800	0.900	0.850	0.050	68.77
	65	YES	0.648	0.661	0.655	0.007	29.96
	93	NFI	0.825		0.825	0.000	63.81
	62	YES	0.480	0.520	0.500	0.020	-0.72
	2	YES	0.488	0.455	0.472	0.016	-6.38

STANDARD LAB ANALYTE=β-CAROTENE SERUM #20 GRAND AVG = 0.136

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	54	YES	0.026	0.025	0.026	0.001	-81.30
	40	YES	0.100	0.100	0.100	0.000	-26.65
	36	YES	0.100	0.100	0.100	0.000	-26.65
	46	YES	0.140	0.120	0.130	0.010	-4.65
	76	YES	0.139	0.136	0.138	0.002	0.86
	27	YES	0.160	0.170	0.165	0.005	21.03
	45	YES	0.130	0.118	0.124	0.006	-9.05
	22	YES	0.110	0.110	0.110	0.000	-19.32
	80	YES	0.130	0.120	0.125	0.005	-8.31
	66	YES	0.000	0.220	0.110	0.110	-19.32
	13	YES	0.170	0.180	0.175	0.005	28.36
	23	YES	0.080	0.040	0.060	0.020	-55.99
	50	YES	0.250	0.260	0.255	0.005	87.04
	17	YES	0.230	0.240	0.235	0.005	72.37
		YES	0.170	0.190	0.180	0.010	32.03
	64	YES	0.162	0.159	0.161	0.001	17.73
	43	NFI	0.243		0.243	0.000	78.24
	47	YES	0.130	0.150	0.140	0.010	2.69
	31	YES	0.124	0.119	0.122	0.003	-10.88

STANDARD LAB ANALYTE=β-CAROTENE SERUM #21 GRAND AVG = 0.382

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	7	YES	0.349	0.329	0.339	0.010	-11.23
	36	YES	0.330	0.320	0.325	0.005	-14.90
	24	YES	0.300	0.320	0.310	0.010	-18.82
	12	YES	0.530	0.490	0.510	0.020	33.55
	77	YES	0.466	0.461	0.464	0.003	21.37
	60	YES	0.450	0.430	0.440	0.010	15.22
	41	YES	0.425	0.425	0.425	0.000	11.29
	71	YES	0.400	0.400	0.400	0.000	4.74
	16	YES	0.400	0.400	0.400	0.000	4.74
	85	YES	0.101	0.095	0.098	0.003	-74.34
	3	YES	0.400	0.440	0.420	0.020	9.98
	57	YES	0.310	0.350	0.330	0.020	-13.59
	82	YES	0.230	0.240	0.235	0.005	-38.46
	18	OUT	0.760	0.770	0.765	0.005	100.32
		YES	0.400	0.420	0.410	0.010	7.36
	31	YES	0.534	0.534	0.534	0.000	39.83
	51	NFI	0.559		0.559	0.000	46.38
	9	YES	0.400	0.440	0.420	0.020	9.98
		YES	0.440	0.425	0.433	0.008	13.25

STANDARD NBS		ANALYTE=RETINOL	SERUM #19	GRAND AVG =		0.476
LAB	VIAL USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	59 YES	0.386	0.410	0.398	0.012	-16.45
	57 YES	0.510	0.520	0.515	0.005	8.12
	25 YES	0.310	0.270	0.290	0.020	-39.12
	27 YES	0.580	0.570	0.575	0.005	20.71
	38 YES	0.160	0.170	0.165	0.005	-65.36
	YES	0.440	0.460	0.450	0.010	-5.53
	89 YES	0.510	0.510	0.510	0.000	7.07
	93 YES	0.470	0.496	0.483	0.013	1.40
	8 YES	0.470	0.480	0.475	0.005	-0.28
	13 YES	0.440	0.440	0.440	0.000	-7.63
	6 YES	0.620	0.620	0.620	0.000	30.16
	4 YES	0.379	0.381	0.380	0.001	-20.23
	86 YES	0.530	0.460	0.495	0.035	3.92
	68 YES	0.555	0.551	0.553	0.002	16.09
	53 YES	0.460	0.410	0.435	0.025	-8.68
	23 YES	0.470	0.460	0.465	0.005	-2.38
	78 YES	0.570	0.570	0.570	0.000	19.66
	98 YES	0.600	0.610	0.605	0.005	27.01
	65 YES	0.623	0.630	0.627	0.003	31.52
	62 NFI	0.780	0.900	0.840	0.060	76.34
NBS	NBS	0.435		0.435	0.000	-8.68

STANDARD NBS		ANALYTE=RETINOL	SERUM #20	GRAND AVG =		0.423
LAB	VIAL USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	54 YES	0.282	0.301	0.292	0.010	-31.08
	40 YES	0.475	0.490	0.483	0.008	14.08
	36 YES	0.260	0.270	0.265	0.005	-37.34
	46 YES	0.650	0.630	0.640	0.010	51.32
	74 YES	0.320	0.350	0.335	0.015	-20.79
	YES	0.410	0.400	0.405	0.005	-4.24
	27 YES	0.480	0.490	0.485	0.005	14.67
	45 YES	0.412	0.423	0.417	0.006	-1.29
	59 YES	0.430	0.450	0.440	0.010	4.03
	22 YES	0.420	0.430	0.425	0.005	0.49
	80 YES	0.550	0.590	0.570	0.020	34.77
	66 YES	0.355	0.356	0.356	0.000	-15.95
	13 YES	0.440	0.490	0.465	0.025	9.94
	23 YES	0.220	0.210	0.215	0.005	-49.17
	63 YES	0.420	0.360	0.390	0.030	-7.79
	5 YES	0.390	0.400	0.395	0.005	-6.61
	50 YES	0.450	0.350	0.400	0.050	-5.43
	17 YES	0.510	0.520	0.515	0.005	21.76
	NFI	0.460	0.500	0.480	0.020	13.49
	64 YES	0.543	0.545	0.544	0.001	28.62
	43 NFI	0.420		0.420	0.000	-0.70
	47 NFI	0.390	0.430	0.410	0.020	-3.06
NBS	NBS	0.432		0.432	0.000	2.14

STANDARD NBS ANALYTE=RETINOL SERUM #21 GRAND AVG = 0.390

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	7	YES	0.448	0.451	0.450	0.002	15.35
	36	YES	0.430	0.430	0.430	0.000	10.35
	24	YES	0.230	0.200	0.215	0.015	-44.83
	12	YES	0.460	0.480	0.470	0.010	20.61
	14	YES	0.200	0.200	0.200	0.000	-48.68
		YES	0.350	0.360	0.355	0.005	-8.90
	60	YES	0.450	0.440	0.445	0.005	14.20
	41	YES	0.385	0.393	0.389	0.004	-0.18
	44	YES	0.390	0.410	0.400	0.010	2.65
	71	YES	0.390	0.400	0.395	0.005	1.36
	16	YES	0.390	0.410	0.400	0.010	2.65
	85	YES	0.351	0.356	0.354	0.003	-9.29
	3	YES	0.330	0.250	0.290	0.040	-25.58
	57	YES	0.445	0.567	0.506	0.061	29.85
	91	YES	0.360	0.350	0.355	0.005	-8.90
	96	YES	0.370	0.380	0.375	0.005	-3.77
	82	YES	0.400	0.330	0.365	0.035	-6.33
	18	YES	0.500	0.510	0.505	0.005	29.59
		NFI	0.440	0.480	0.460	0.020	18.04
	31	YES	0.504	0.508	0.506	0.002	29.85
	51	NFI	0.398		0.398	0.000	2.13
	9	NFI	0.370	0.410	0.390	0.020	0.08
NBS		NBS	0.375		0.375	0.000	-3.77

STANDARD NBS ANALYTE=α-TOCOPHEROL SERUM #19 GRAND AVG = 29.296

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
		YES	27.800	28.400	28.100	0.300	-4.08
	89	YES	30.290	30.350	30.320	0.029	3.50
	93	YES	31.400	31.400	31.400	0.000	7.18
	13	YES	28.900	29.400	29.150	0.250	-0.50
	6	YES	26.710	25.000	25.855	0.855	-11.74
	4	YES	25.520	25.520	25.520	0.000	-12.89
	86	YES	34.700	35.800	35.250	0.550	20.32
	68	YES	29.100	29.200	29.150	0.050	-0.50
	53	YES	26.400	25.900	26.150	0.250	-10.74
	23	YES	26.710	26.710	26.710	0.000	-8.83
	74	YES	30.200	29.700	29.950	0.250	2.23
	78	YES	21.250	23.130	22.190	0.940	-24.26
	98	OUT	48.130	48.420	48.275	0.144	64.78
	65	YES	41.200	41.000	41.100	0.100	40.29
NBS		NBS	33.770		33.770	0.000	15.27

STANDARD NBS ANALYTE=α-TOCOPHEROL SERUM #20 GRAND AVG = 6.991

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
		YES	7.300	7.000	7.150	0.150	2.27
	27	YES	6.770	7.180	6.975	0.205	-0.23
	45	YES	7.170	7.140	7.155	0.015	2.34
	22	YES	7.200	7.300	7.250	0.050	3.70
	80	YES	6.440	6.380	6.410	0.030	-8.31
	66	YES	6.262	6.051	6.157	0.106	-11.94
	13	YES	9.900	8.400	9.150	0.750	30.88
	23	YES	3.400	3.400	3.400	0.000	-51.37
	63	YES	6.980	7.000	6.990	0.010	-0.02
	5	YES	6.320	6.230	6.275	0.045	-10.24
	30	YES	7.160	7.040	7.100	0.060	1.56
	50	YES	4.940	4.830	4.885	0.055	-30.13
	17	YES	9.300	9.600	9.450	0.150	35.17
		NFI	0.730	2.860	1.795	1.065	-74.32
	64	YES	9.470	9.590	9.530	0.060	36.31
NBS		NBS	7.704		7.704	0.000	10.20

STANDARD NBS ANALYTE=α-TOCOPHEROL SERUM #21 GRAND AVG = 6.304

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
		YES	6.300	6.100	6.200	0.100	-1.64
	60	YES	6.840	6.190	6.515	0.325	3.36
	41	YES	6.520	6.630	6.575	0.055	4.31
	71	YES	6.500	6.600	6.550	0.050	3.91
	16	YES	5.600	5.260	5.430	0.170	-13.86
	85	YES	5.777	5.624	5.701	0.076	-9.57
	3	YES	6.300	5.400	5.850	0.450	-7.19
	57	YES	6.300	7.700	7.000	0.700	11.05
	91	YES	6.300	6.500	6.400	0.100	1.53
	96	YES	5.380	5.720	5.550	0.170	-11.95
	88	YES	6.420	6.240	6.330	0.090	0.42
	82	YES	5.170	4.850	5.010	0.160	-20.52
	18	OUT	10.380	10.300	10.340	0.040	64.04
		NFI	2.980	3.090	3.035	0.055	-51.85
	31	YES	8.690	8.980	8.835	0.145	40.16
NBS		NBS	7.618		7.618	0.000	20.85

STANDARD NBS ANALYTE=β-CAROTENE SERUM #19 GRAND AVG = 0.480

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	59	YES	0.307	0.432	0.370	0.063	-23.10
	57	YES	0.365	0.380	0.373	0.008	-22.47
	25	YES	0.390	0.350	0.370	0.020	-22.99
	27	YES	0.480	0.510	0.495	0.015	3.02
	89	YES	0.490	0.470	0.480	0.010	-0.10
	93	YES	0.513	0.478	0.496	0.017	3.13
	8	OUT	1.000	1.590	1.295	0.295	169.53
	13	YES	0.460	0.480	0.470	0.010	-2.18
	6	YES	0.440	0.420	0.430	0.010	-10.50
	4	YES	0.356	0.373	0.364	0.009	-24.14
	86	YES	0.530	0.540	0.535	0.005	11.35
	68	YES	0.272	0.305	0.289	0.016	-39.95
	78	YES	0.860	0.810	0.835	0.025	73.79
	98	OUT	1.530	1.540	1.535	0.005	219.48
	65	YES	0.587	0.600	0.594	0.007	23.53
	93	NFI	0.914		0.914	0.000	90.23
	62	YES	0.590	0.650	0.620	0.030	29.04
	2	YES	0.477	0.499	0.488	0.011	1.57
NBS		NBS	0.388		0.388	0.000	-19.25

STANDARD NBS ANALYTE=β-CAROTENE SERUM #20 GRAND AVG = 0.114

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	54	YES	0.028	0.026	0.027	0.001	-76.21
	40	YES	0.100	0.095	0.098	0.003	-14.10
	36	YES	0.110	0.130	0.120	0.010	5.73
	46	YES	0.120	0.140	0.130	0.010	14.54
	27	YES	0.140	0.150	0.145	0.005	27.75
	45	YES	0.128	0.117	0.123	0.006	7.93
	59	OUT	0.740	1.170	0.955	0.215	741.41
	22	YES	0.110	0.100	0.105	0.005	-7.49
	80	YES	0.130	0.120	0.125	0.005	10.13
	66	YES	0.052	0.036	0.044	0.008	-61.23
	13	YES	0.170	0.180	0.175	0.005	54.19
	23	YES	0.026	0.040	0.033	0.007	-70.93
	50	OUT	0.260	0.300	0.280	0.020	146.70
	17	OUT	0.440	0.420	0.430	0.010	278.85
		NFI	0.210	0.230	0.220	0.010	93.83
	64	YES	0.151	0.149	0.150	0.001	32.16
	43	NFI	0.198		0.198	0.000	74.45
	47	YES	0.170	0.190	0.180	0.010	58.59
	31	YES	0.133	0.137	0.135	0.002	18.94
NBS		NBS	0.122		0.122	0.000	7.49

STANDARD NBS ANALYTE=β-CAROTENE SERUM #21 GRAND AVG = 0.405

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	7	YES	0.364	0.343	0.354	0.011	-12.75
	36	YES	0.320	0.320	0.320	0.000	-21.02
	24	YES	0.300	0.300	0.300	0.000	-25.95
	12	YES	0.420	0.470	0.445	0.025	9.83
	60	YES	0.440	0.420	0.430	0.010	6.13
	41	YES	0.419	0.419	0.419	0.000	3.42
	44	YES	0.510	0.810	0.660	0.150	62.90
	71	YES	0.430	0.430	0.430	0.000	6.13
	16	YES	0.390	0.390	0.390	0.000	-3.74
	85	YES	0.334	0.363	0.348	0.015	-13.98
	3	YES	0.400	0.370	0.385	0.015	-4.97
	57	YES	0.260	0.350	0.305	0.045	-24.72
	82	YES	0.240	0.270	0.255	0.015	-37.06
	18	OUT	1.540	1.480	1.510	0.030	272.70
		NFI	0.300	0.320	0.310	0.010	-23.49
	31	YES	0.486	0.486	0.486	0.000	19.95
	51	NFI	0.587		0.587	0.000	44.88
	9	YES	0.500	0.550	0.525	0.025	29.58
		YES	0.432	0.429	0.431	0.002	6.26
NBS		NBS	0.335		0.335	0.000	-17.32

RR V DISTRIBUTION OF BIASES IN "LAB STANDARD" RESULTS

Analyte and Serum No.	0 to ±10%	±10 to 20%	±20 to 30%	±30 to 40%	±40 to 50%	>50%
RETINOL						
21	9	4	4			1
20	11	3	2			1
19	10	5	1			2
α-TOCOPHEROL						
21	7	3	2		2	
20	9	2	1	1		
19	8	2		2		
β-CAROTENE						
20	5	3	4	1		4
21	5	7	1	1		1
19	4	4	3	1		2

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

May 12, 1986

[Name and Address]

Dear [First Name]:

Statistical results from our NBS/NCI Round-Robins IV, V, and VI, have been reported to you in previous mailings. Graphical evaluations of the individual laboratory results from some of these same Round-Robins (RR) have now been made. Graphs are enclosed for Se and Zn results from RR IV and VI, and β-carotene results from RR V. The range of concentrations for other analytes and round-robins are too limited to permit useful graphical presentation.

The motivation for the current work is to present results for easy comprehension, and to allow comparisons to be readily made. In addition, the graphs allow one to easily judge the suitability of calibration procedures.

Let's now examine Graph 1 (see also, similar Graphs 2-8). Graph 1 shows the laboratory 23, RR IV and VI results for Se for various sera. The laboratory values are shown on the ordinate scale. The corresponding consensus values for the same sera, obtained from "all" of the laboratory results, are shown on the abscissa scale. Since the scales for the ordinate and abscissa are the same, a perfect agreement between an individual laboratory result and the consensus value will appear as a point on the (dashed) (45°) line.

Plots for Se (and Zn) have only been made for those laboratories that had all acceptable (YES category) data for sera 22-24 from RR VI. The three points for sera 22-24 are plotted as X's and the associated least squares regression line is shown. The equation for each laboratory's regression line is given in the legend along with the value of S_{fit} (also called the standard error of estimate, or $S_{Y.X}$).

Sera 25-27 results from RR VI are plotted on these graphs as O's and are actually the same samples as sera 16-18 from RR IV which are plotted as H's. The O and H values are for human sera that have had the T3 and T4 components removed. The generally good agreement of the consensus (abscissa) values for the O's and H's indicate that these sera were stable over the three month period between the round-robins. The individual laboratory (ordinate) O and H values show much more scatter than the consensus values.

An examination of graphs 1-8 shows reasonable linearities for the sera 16-18 (H) values vs. consensus values, and for the sera 25-27(O) values vs. consensus values. These linearities, as well as for the sera 22-24(X) values vs. consensus values, are encouraging. The general deviations of these "lines" from the dashed 45° line, however, indicates that further calibrations may help. The differences between the H and O values suggest that these additional

calibrations would generally need to be done more frequently than once very three months.

The differences between the plotted regression line for the normal human sera (the X values) from RR VI, and the T3 and T4 depleted sera results from the same round-robin (the O values) suggest that interferences in different sera need to be recognized in order to make adequate calibrations. Graph 3, as an example, shows that the regression line is inadequate for the calibration of the T3 and T4 depleted sera. The calibration corrected values would, in fact, be more in error than the original, uncorrected values.

The quantitative values of the Se data from the laboratories and the regression results are given in Table 1. The averages and consensus values used are also listed. The "average values" for Se (and for Zn of Table 2) require some explanation in that they are not necessarily the average of the values in the corresponding column. For example, the listed column values for Se serum 18 of RR IV have an average value of .026 mg/L rather than the printed value of .024 mg/L. The reason for this later value is given below.

> Table 1 lists only the laboratory results for Se that were plotted. In order for a laboratory's results for sera 16-18 to be plotted, a laboratory had to submit three acceptable sera 22-24 results upon which a regression line could be based. Acceptable, but unplotted sera 16-18 results could, however, still be used to help determine better average values for sera 16-18. Thus the printed averages include some numbers which are not listed in Table 1.

For Se (and for Zn) the printed averages are identical to the consensus values that were used in the plots.

Graphs 9-21 shows the individual laboratory results for Zn and Table 2 lists the corresponding numerical values. The Zn measurements tend to be in better control than the Se measurements. Several of the plots show excellent results. Some of the laboratories, however, have problems with large differences between the results for the same sera from RR IV and VI. Several laboratories also have large offsets from the consensus values. The graphs allow a clear comparison to be made between the different laboratories. Laboratories that are far from the norm may wish to examine their procedures.

Graphs 22-35 shows the individual laboratory results for sera 19-21 for β-carotene. Table 3 lists the corresponding numerical values. To date, little round-robin work with stable sera has been done for β-carotene. Because of this, only a few conclusions can be reached. We can note that the deviations from the ideal 45° line tend to be moderately large. A general linearity of the X values vs. the consensus values is also observed. Note that the consensus values for sera 19-21 were obtained by excluding the results from laboratories 32 and 46, even though we had not previously excluded these results from the statistical analyses. We previously were mainly concerned with evaluating the round-robin uncertainties, and were not greatly concerned with getting the best consensus values for the sera. In the present work, however, we need to be more selective since we want to get the best consensus values for evaluating

the linearity of the individual laboratory results. A comparison of Graphs 22-35 shows large differences between the individual laboratories regression lines and the ideal 45° line.

Additional information will be developed as we do more round-robins.

Sincerely,

Robert C. Paule
Statistician

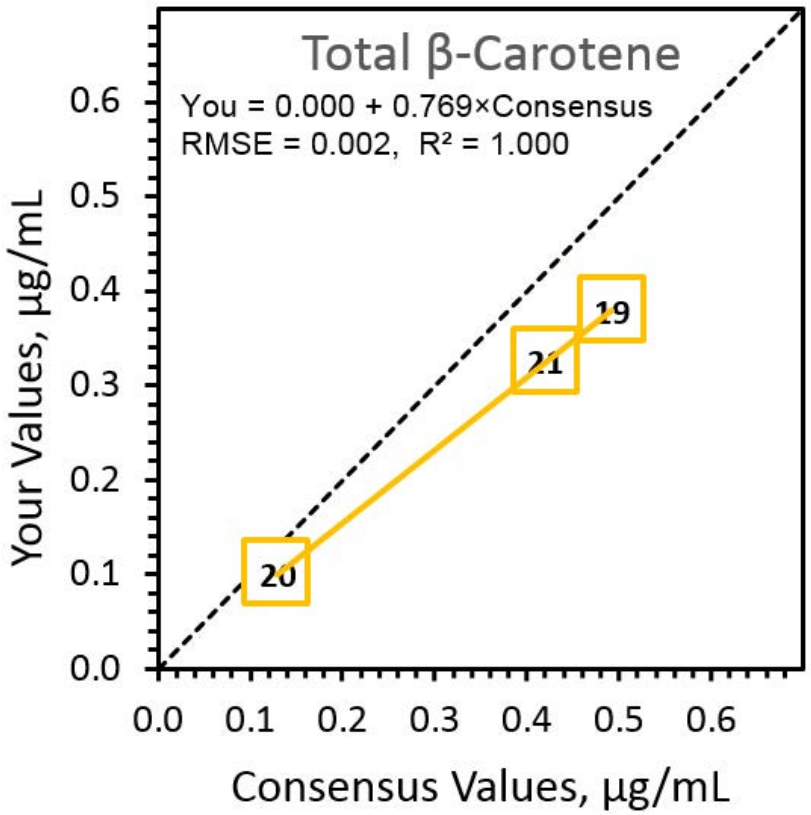
Fillmer C. Ruegg
Electronic Engineer

Table 3

Laboratories own standards used

Round Robin V: b-carotene

Lab	Intercept	Slope	S fit	Sera	020 Mean	021 Mean	019 Mean	
015	-0.0892	0.9725	0.0346		0.026	0.339	0.355	mg/L
016	0.0041	0.7784	0.0008		0.100	0.325	0.378	
017	0.0168	0.6892	0.0111		0.100	0.310	0.340	
018	-0.0323	1.3172	0.0022		0.130	0.510	0.600	
022	0.0542	0.9138	0.0110		0.165	0.440	0.485	
023	-0.0063	1.0536	0.0050		0.124	0.425	0.502	
027	0.0055	0.8902	0.0350		0.110	0.400	0.410	
028	0.0142	0.9138	0.0110		0.125	0.400	0.445	
031	0.0301	1.0885	0.0777		0.175	0.420	0.600	
032	0.0155	0.5154	0.1325		0.060	0.330	0.180	
046	0.0804	1.0185	0.3467		0.255	0.235	0.785	
050	-0.0096	1.3575	0.0222		0.161	0.534	0.655	
055	0.0159	0.9974	0.0102		0.140	0.420	0.500	
056	0.0001	1.0101	0.0199		0.122	0.433	0.472	
Average					0.128	0.394	0.479	
Consensus values used					0.123	0.413	0.479	



Appendix L. Updated "All-Lab Report" for RR05

The following three pages are the modernized "All-Lab" report for RR05. This report has three parts:

- Page L2 lists the participant mean results for all analytes reported.
- Page L3 provides the legend for page L2.
- Page L4 summarizes each participants' performance for retinol, α-tocopherol, and β-carotene, using the "Comparability Summary" calculations from the 1999 to 2017 Round Robins.

To ensure confidentiality, the laboratory identifiers used in this "All-Lab Report" have been altered from those used in RR05. 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 V Laboratory Results

	Total Retinol, µg/mL						α-Tocopherol, µg/mL						Total β-Carotene, µg/mL					
Lab	19	20	21	19a	20a	21a	19	20	21	19a	20a	21a	19	20	21	19a	20a	21a
FSV-BA													0.472	0.122	0.433	0.488	0.135	0.431
FSV-BD	0.480	0.445	0.405	0.475	0.440	0.400										1.300	0.955	0.660
FSV-BE	0.605	0.385	0.400	0.570	0.400	0.365	19.2	4.81	4.67	22.2	4.89	5.01	0.785	0.255	0.235	0.835	0.280	0.255
FSV-BF	0.465	0.395	0.370		0.215	0.506	27.4	6.40	5.50	29.2	3.40	7.00	0.180	0.060	0.330	0.289	0.033	0.305
FSV-BG	0.385	0.345	0.335	0.290	0.265	0.215							0.340	0.100	0.310	0.370	0.120	0.300
FSV-BI	0.525	0.500	0.440	0.515	0.483	0.430							0.378	0.100	0.325	0.373	0.098	0.320
FSV-BY	0.407	0.299	0.460	0.398	0.292	0.450							0.355	0.026	0.339	0.370	0.027	0.354
FSV-CA	0.480	0.435	0.375	0.450	0.405	0.355	28.5	7.25	6.30	28.1	7.15	6.20						
FSV-CJ	0.491	0.456	0.420				32.5	8.26	7.14				0.521	0.138	0.464			
FSV-CL	0.515	0.485	0.325	0.495	0.465	0.290	29.9	7.80	5.05	35.3	9.15	5.85	0.600	0.175	0.420	0.535	0.175	0.385
FSV-CN	0.540	0.495	0.350	0.620	0.570	0.400	23.6	5.85	4.96	25.9	6.41	5.43	0.445	0.125	0.400	0.430	0.125	0.390
FSV-CO	0.520	0.470	0.455	0.510	0.485	0.445	29.6	7.15	6.70	30.3	6.98	6.52	0.485	0.165	0.440	0.480	0.145	0.430
FSV-DC		0.470	0.447		0.420	0.398							0.825	0.243	0.559	0.914	0.198	0.587
FSV-DE							29.7	7.04	6.28	30.0	7.10	6.33						
FSV-DG	0.815	0.735	0.705	0.605	0.515	0.505	24.1	5.02	5.24	48.3	9.45	10.34	0.850	0.235	0.765	1.535	0.430	1.510
FSV-DH	0.451	0.440	0.411	0.380	0.356	0.354	32.4	7.60	7.23	25.5	6.16	5.70	0.063	0.110	0.098	0.364	0.044	0.348
FSV-DN	0.475	0.465	0.435	0.440	0.425	0.395	26.4	6.60	5.90	29.2	7.25	6.55	0.410	0.110	0.400	0.470	0.105	0.430
FSV-DO	0.210	0.450	0.285	0.165	0.335	0.200												
FSV-DT	0.450	0.405	0.360	0.553	0.390	0.355	25.4	6.99	6.40	26.2	6.99	6.40						
FSV-EG	0.605	0.690	0.505	0.575	0.640	0.470							0.600	0.130	0.510	0.495	0.130	0.445
FSV-EN	0.580	0.543	0.480				30.1	5.71	6.32									
FSV-EO	0.905	0.450	0.420	0.840	0.410	0.390							0.500	0.140	0.420	0.620	0.180	0.525
FSV-EP	0.515	0.455	0.425	0.465	0.395	0.375	28.7	6.91	6.31	26.7	6.28	5.55						
FSV-ER	0.509	0.440	0.410	0.483	0.417	0.389	30.0	6.85	6.29	31.4	7.16	6.58	0.502	0.124	0.425	0.496	0.123	0.419
FSV-EU		0.820	0.850		0.480	0.460		2.55	2.96		1.80	3.04		0.180	0.410		0.220	0.310
FSV-EW	0.675	0.585	0.540	0.627	0.544	0.506	37.2	8.92	8.29	41.1	9.53	8.84	0.655	0.161	0.534	0.594	0.150	0.486
n	22	24	24	19	22	22	16	17	17	14	15	15	18	19	19	18	19	19
Min	0.210	0.299	0.285	0.165	0.215	0.200	19.2	2.55	2.96	22.2	1.80	3.04	0.063	0.026	0.098	0.289	0.027	0.255
Median	0.512	0.456	0.420	0.495	0.419	0.397	29.1	6.91	6.29	29.2	6.99	6.33	0.493	0.130	0.420	0.492	0.135	0.419
Max	0.905	0.820	0.850	0.840	0.640	0.506	37.2	8.92	8.29	48.3	9.53	10.34	0.850	0.255	0.765	1.535	0.955	1.510
eSD	0.080	0.051	0.063	0.111	0.092	0.062	3.4	1.02	1.17	4.0	1.06	0.93	0.165	0.044	0.120	0.178	0.059	0.105
eCV	16	11	15	22	22	16	12	15	19	14	15	15	33	34	29	36	44	25
NIST	0.435	0.432	0.375				33.8	7.70	7.62				0.388	0.122	0.335			
NAV	0.512	0.456	0.420				29.1	6.91	6.29				0.493	0.130	0.420			
NAU	0.080	0.051	0.063				3.4	1.02	1.17				0.165	0.044	0.120			

L2

Round Robin V Laboratory Results

Legend

Term	Definition
n	Number of (non-NIST) quantitative values reported for this analyte
Min	Minimum (non-NIST) quantitative value reported
Median	Median (non-NIST) quantitative value reported
Max	Maximum (non-NIST) quantitative value reported
eSD	Estimated standard deviation, calculated from the median absolute deviation from the median of the non-NIST results
eCV	Coefficient of Variation for (non-NIST) results: 100*eSD/Median
NAV	NIST Assigned Value, our estimate of the "true" analyte concentration
NAU	NIST Assigned Uncertainty, our estimate of the total (serum heterogeniety and inter- and intra-laboratory) standard deviation

Round Robin V Laboratory Results

Comparability Summary

Lab	TR	aT	bC
FSV-BA			1
FSV-BD	1		
FSV-BE	2	3	3
FSV-BF	1	1	2
FSV-BG	2		1
FSV-BI	1		1
FSV-BY	3		2
FSV-CA	1	1	
FSV-CJ	1	2	1
FSV-CL	2	1	1
FSV-CN	1	2	1
FSV-CO	1	1	1
FSV-DC	1		3
FSV-DE		1	
FSV-DG	4	2	3
FSV-DH	1	1	3
FSV-DN	1	1	1
FSV-DO	3		
FSV-DT	1	1	
FSV-EG	4		1
FSV-EN	2	1	
FSV-EO	4		1
FSV-EP	1	1	
FSV-ER	1	1	1
FSV-EU	4	4	1
FSV-EW	3	3	1
NIST	1	2	1
n	25	18	20

Label	Definition
Lab	laboratory number
TR	"Standard Score" for Retinol
aT	"Standard Score" for α-Tocopherol
bC	"Standard Score" for Total β-Carotene
n	number of (non-NIST) laboratories providing data for this analyte

"Standard Score"

Given that our knowledge of the shape, location, and width of the measurement distributions is approximate and that a limited number of labs are involved, we summarize comparability with the following four-level "Standard Score" (StS)…

StS	Definition
1	All StV within ±1 SD
2	All StV within ±2 SD
3	All StV within ±3 SD
4	At least one StV >3 SD

where:

SD	Total measurement standard deviation (SD), including serum heterogeneity and inter-and intra-laboratory variability.
StV	Standardized Value, the distance in SD units your value is from the "true" concentration: StV = (your value - NAV) / NAU
NAV	NIST Assigned Value, our estimate of the "true" analyte concentration
NAU	NIST Assigned Uncertainty, our estimate of the SD

	TR	aT	bC	Expected
% 1	56	61	70	68.2 %
% 2	16	22	10	27.3 %
% 3	12	11	20	4.3 %
% 4	16	6	0	0.3 %

These are the observed and normal-population-expected proportions of each Standard Score (StS), based upon each laboratory's largest StV for the four sera.