NISTIR 7880-38

NIST Micronutrients Measurement Quality Assurance Program Spring and Fall 1986 Comparability Studies

Results for Round Robins VII and VIII Fat-Soluble Vitamins and Carotenoids in Human Serum

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

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

National Institute of Standards and Technology
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Abstract

From 1984 to 2017, the National Institute of Standards and Technology coordinated 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 Spring and Fall 1986 MMQAP measurement comparability improvement studies: 1) Round Robin VII Fat-Soluble Vitamins and Carotenoids in Human Serum and 2) Round Robin VIII Fat-Soluble Vitamins and Carotenoids in Human Serum. The first participant results for Round Robin VII were received March 5, 1986; the last results were received April 29, 1986. The first participant results for Round Robin VIII were received October 30, 1986; the last results were received February 6, 1987.

Keywords

Human Serum
Retinol, α-Tocopherol, β-Carotene

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Introduction

From 1984 to 2017, the National Institute of Standards and Technology (NIST), formerly the National Bureau of Standards (NBS), coordinated 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, *trans*- and total beta-carotene, and ascorbic acid) 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 program and the nature of its studies are described elsewhere. [1]

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

Six human serum test samples (sera 31 to 35 and 44) were distributed to 30 participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum component of Round Robin VII (hereafter referred to as RR07). Samples 31, 32, 34, and 44 were liquid frozen sera; sample 33 was a lyophilized version of sample 44; sample 35 was a lyophilized version of sample 34. Twenty-five participants received all six samples; five participants received just the {liquid, lyophilized} pairs: {44, 33} and {34, 35}. Participants received one vial of each test sample. These sera were shipped on dry ice to participants in March 1986.

Participants were requested to report 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.

Ten participants in the ascorbic acid component of RR07 received three freeze-dried human serum test samples (sera 37, 45, and 46) and 17 participants in the selenium and zinc component received six test samples (40, 41, 42, 43, 47, and 48). 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 measurement results reported for RR07 in a more accessible format.

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

Three lyophilized human serum test samples (sera 50 to 52) were distributed to 29 participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum component of Round Robin VIII (hereafter referred to as RR08). Unless multiple vials were previously requested, participants received one vial of each material. These sample materials were shipped on dry ice to participants in October 1986.

Participants were requested to report values for retinol, α-tocopherol, and total 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.

Eight participants in the ascorbic acid component of RR08 received four freeze-dried human serum test samples (sera 58 to 61) and 19 participants in the selenium and zinc component received five test samples (53 to 57). The results for these materials are not discussed in this document.

Our records for this study are incomplete. Appendix D presents an example of the data report form. Appendix E reproduces a letter sent to all participants that describes the tabular and graphical analysis for the study and standard solution preparation methods for retinol, α-tocopherol, and β-carotene. Appendix E also includes the data and statistical summary tables. Appendix F lists the measurement results reported for RR08 in a more accessible format.

Reference

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.

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Appendix A. Shipping Package Inserts for RR07

Two items were attached to each package shipped to an RR07 participant:

- **Cover letter**. The original letter has been lost. It would have described the four liquid-frozen (sera 31, 32, 34, and 44) and two lyophilized sample materials (sera 33 and 35) distributed for the study.

  Sample 33 was a lyophilized version of sample 44; sample 35 was a lyophilized version of sample 34. Twenty-five participants received all six samples; five participants received just the {liquid-frozen, lyophilized} pairs: {33, 44} and {35, 34}. The sample 32 material had been distributed as sample 21 in Round Robin V.

- **Datasheet**. Pages A2 and A3 reproduce the report form. The report form consisted of two pages to accommodate reporting results for the sera {33, 44} and {35, 34} {lyophilized, liquid-frozen} pairs.

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Report on NBS/NCI Samples from Laboratory #____

Results in mg/L

Samples		Result 1	Result 2
Serum No: 31 Vial No. ____ Analysis Date / /86	Retinol β-Carotene α-Tocopherol		
Serum No: 32 Vial No. ____ Analysis Date / /86	Retinol β-Carotene α-Tocopherol		

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Report on NBS/NCI Samples from Laboratory #____

Results in mg/L

Based on NBS Calibration Solutions

Samples		Result 1	Result 2
Serum No: 35 Vial No. ____ Analysis Date / /86	Retinol β-Carotene α-Tocopherol		
Serum No: 33 Vial No. ____ Analysis Date / /86	Retinol β-Carotene α-Tocopherol		
Serum No: 34 Vial No. ____ Analysis Date / /86	Retinol β-Carotene α-Tocopherol		
Serum No: 44 Vial No. ____ Analysis Date / /86	Retinol β-Carotene α-Tocopherol		

Note: Add 1.20 mL of water to reconstitute freeze-dried samples.

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Appendix B. Final Report for RR07

No copy of the original cover letter and discussion of results is now available. The following 11 pages list the following:

- The reported results for retinol, α-tocopherol, and β-carotene; the ascorbic acid, selenium, and zinc results included in the original report have been removed. Due to the complex formatting used in the tables, the original laboratory codes have been deleted without replacement. Appendix C provides a complete listing of the fat-soluble vitamin results where the original codes have been altered to ensure confidentiality. Appendix C also provides relevant summary statistics.
- A "Statistical Summary" table and its legend.

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RR VII Results

STANDARD LAB ANALYTE=RETINOL SERUM #31 GRAND AVG = 0.793

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	22	YES	0.761	0.767	0.764	0.003	-3.63
	25	YES	0.830	0.830	0.830	0.000	4.69
	55	YES	0.957	0.939	0.948	0.009	19.57
	57	YES	0.950	0.925	0.938	0.012	18.25
	42	YES	0.860	0.806	0.833	0.027	5.07
	69	OUT	0.150	0.120	0.135	0.015	-82.97
	76	YES	0.778	0.782	0.780	0.002	-1.62
	38	YES	0.763	0.790	0.777	0.014	-2.06
	67	YES	0.700	0.690	0.695	0.005	-12.34
	86	YES	0.860	0.820	0.840	0.020	5.95
	51	YES	0.690	0.670	0.680	0.010	-14.23
	90	YES	0.580	0.560	0.570	0.010	-28.10
	90	YES	0.730	0.710	0.720	0.010	-9.18
	??	YES	0.905	0.928	0.917	0.012	15.60
	45	NFI	0.630		0.630	0.000	-20.54
	53	YES	0.730	0.740	0.735	0.005	-7.29
	58	YES	0.680	0.670	0.675	0.005	-14.86
	83	YES	1.058	1.057	1.058	0.000	33.39
	119	YES	0.790	0.800	0.795	0.005	0.28
	62	YES	0.760	0.760	0.760	0.000	-4.14
	116	YES	0.861	0.801	0.831	0.030	4.82
	23	YES	0.770	0.820	0.795	0.025	0.28
	71	YES	0.711	0.709	0.710	0.001	-10.45
		NFI	0.697		0.697	0.000	-12.14
NBS		NBS	0.774		0.774	0.000	-2.37

STANDARD LAB ANALYTE=RETINOL SERUM #32 GRAND AVG = 0.417

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	10	YES	0.417	0.417	0.417	0.000	-0.07
	47	YES	0.430	0.430	0.430	0.000	3.05
	37	NFI	0.561	0.548	0.555	0.007	32.89
	63	YES	0.496	0.486	0.491	0.005	17.67
	20	YES	0.440	0.391	0.415	0.025	-0.43
	19	OUT	0.090	0.070	0.080	0.010	-80.83
	48	YES	0.404	0.400	0.402	0.002	-3.66
	87	YES	0.415	0.403	0.409	0.006	-1.98
	79	YES	0.340	0.350	0.345	0.005	-17.32
	75	YES	0.570	0.530	0.550	0.020	31.81
	92	YES	0.430	0.440	0.435	0.005	4.25
	90	YES	0.310	0.310	0.310	0.000	-25.71
	90	YES	0.390	0.410	0.400	0.010	-4.14
	??	YES	0.491	0.456	0.474	0.018	13.47
	34	NFI	0.330		0.330	0.000	-20.92
	78	YES	0.400	0.390	0.395	0.005	-5.34
	29	YES	0.380	0.370	0.375	0.005	-10.13
	06	YES	0.443	0.452	0.448	0.004	7.24
	73	YES	0.400	0.400	0.400	0.000	-4.14
	50	YES	0.460	0.440	0.450	0.010	7.84
	99	YES	0.519	0.456	0.488	0.031	16.83
	92	YES	0.300	0.301	0.301	0.001	-27.99
	71	YES	0.420	0.404	0.412	0.008	-1.26
		NFI	0.583		0.583	0.000	39.62
NBS		NBS	0.428		0.428	0.000	2.57

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STANDARD LAB		ANALYTE=RETINOL	SERUM #33	GRAND AVG =	0.341	
LAB	VIAL USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	36 YES	0.380	0.370	0.375	0.005	10.01
	19 YES	0.448	0.428	0.438	0.010	28.49
	60 YES	0.361	0.354	0.357	0.003	4.88
	54 NFI	0.395		0.395	0.000	15.88
	56 YES	0.300	0.320	0.310	0.010	-9.06
	63 YES	0.393	0.391	0.392	0.001	15.00
	06 YES	0.318	0.317	0.318	0.000	-6.86
	62 YES	0.350	0.344	0.347	0.003	1.80
	31 YES	0.330	0.350	0.340	0.010	-0.26
	45 YES	0.270	0.290	0.280	0.010	-17.86
	14 YES	0.330	0.320	0.325	0.005	-4.66
	24 YES	0.280	0.270	0.275	0.005	-19.32
	24 YES	0.360	0.350	0.355	0.005	4.15
	34 YES	0.385	0.416	0.401	0.015	17.49
	?? NFI	0.280		0.280	0.000	-17.86
	32 YES	0.220	0.170	0.195	0.025	-42.79
	12 YES	0.330	0.340	0.335	0.005	-1.72
	37 YES	0.330	0.320	0.325	0.005	-4.66
	44 OUT	0.851	0.809	0.830	0.021	143.49
	18 YES	0.320	0.320	0.320	0.000	-6.12
	18 YES	0.370	0.370	0.370	0.000	8.55
	25 YES	0.400	0.430	0.415	0.015	21.75
	07 YES	0.338	0.371	0.355	0.016	4.00
	50 YES	0.297	0.307	0.302	0.005	-11.40
	08 YES	0.348	0.345	0.347	0.001	1.65
	29 YES	0.391	0.338	0.364	0.027	6.93
	NFI	0.178		0.178	0.000	-47.72
NBS	NBS	0.359		0.359	0.000	5.32

STANDARD LAB		ANALYTE=RETINOL	SERUM #34	GRAND AVG =	0.346	
LAB	VIAL USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	12 YES	0.347	0.351	0.349	0.002	0.74
	22 YES	0.370	0.370	0.370	0.000	6.80
	40 YES	0.446	0.468	0.457	0.011	31.91
	24 YES	0.410	0.404	0.407	0.003	17.48
	13 YES	0.404	0.377	0.391	0.014	12.72
	57 YES	0.300	0.280	0.290	0.010	-16.29
	42 YES	0.400	0.405	0.403	0.002	16.18
	85 YES	0.364	0.356	0.360	0.004	3.91
	YES	0.365	0.353	0.359	0.006	3.63
	18 YES	0.340	0.330	0.335	0.005	-3.30
	07 YES	0.310	0.310	0.310	0.000	-10.52
	72 YES	0.320	0.310	0.315	0.005	-9.08
	55 YES	0.280	0.270	0.275	0.005	-20.62
	55 YES	0.350	0.350	0.350	0.000	1.03
	59 YES	0.394	0.435	0.415	0.021	19.65
	?? NFI	0.250		0.250	0.000	-27.84
	56 YES	0.140	0.130	0.135	0.005	-61.03
	36 YES	0.350	0.320	0.335	0.015	-3.30
	21 YES	0.280	0.280	0.280	0.000	-19.18
	04 OUT	0.779	0.822	0.800	0.022	131.06
	68 YES	0.320	0.330	0.325	0.005	-6.19
	68 YES	0.360	0.370	0.365	0.005	5.36
	74 YES	0.400	0.420	0.410	0.010	18.35
	71 YES	0.398	0.413	0.406	0.008	17.05
	75 YES	0.313	0.280	0.297	0.017	-14.42
	37 YES	0.356	0.353	0.355	0.002	2.33
	11 YES	0.341	0.399	0.370	0.029	6.80
	NFI	0.265		0.265	0.000	-23.42
NBS	NBS	0.354		0.354	0.000	2.18

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STANDARD LAB		ANALYTE=RETINOL	SERUM #35	GRAND AVG	0.337	
LAB	VIAL USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	19 YES	0.360	0.360	0.360	0.000	6.85
	58 YES	0.447	0.446	0.447	0.001	32.52
	48 YES	0.397	0.387	0.392	0.005	16.34
	47 YES	0.412	0.400	0.406	0.006	20.50
	64 YES	0.360	0.300	0.330	0.030	-2.06
	68 YES	0.392	0.392	0.392	0.000	16.34
	36 YES	0.321	0.327	0.324	0.003	-3.84
	50 YES	0.270	0.291	0.281	0.011	-16.75
	61 YES	0.320	0.330	0.325	0.005	-3.54
	70 YES	0.290	0.280	0.285	0.005	-15.41
	59 YES	0.300	0.300	0.300	0.000	-10.96
	54 YES	0.290	0.290	0.290	0.000	-13.93
	54 YES	0.340	0.340	0.340	0.000	0.91
	15 YES	0.358	0.377	0.368	0.009	9.07
	63 NFI	0.290		0.290	0.000	-13.93
	49 YES	0.150	0.160	0.155	0.005	-54.00
	60 YES	0.350	0.380	0.365	0.015	8.33
	14 YES	0.350	0.350	0.350	0.000	3.88
	02 OUT	0.765	0.777	0.771	0.006	128.83
	16 YES	0.320	0.340	0.330	0.010	-2.06
	16 YES	0.330	0.320	0.325	0.005	-3.54
	01 NFI	0.370		0.370	0.000	9.81
	20 YES	0.369	0.406	0.387	0.018	15.01
	31 YES	0.243	0.265	0.254	0.011	-24.61
	04 YES	0.350	0.344	0.347	0.003	2.99
	12 YES	0.370	0.425	0.398	0.027	17.98
	NFI	0.236		0.236	0.000	-30.11
NBS	NBS	0.363		0.363	0.000	7.74

STANDARD LAB		ANALYTE=RETINOL	SERUM #44	GRAND AVG =	0.352	
LAB	VIAL USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	56 YES	0.366	0.358	0.362	0.004	2.88
	25 YES	0.370	0.360	0.365	0.005	3.73
	26 YES	0.447	0.464	0.456	0.008	29.45
	38 YES	0.404	0.424	0.414	0.010	17.66
	07 NFI	0.393		0.393	0.000	11.69
	22 YES	0.300	0.300	0.300	0.000	-14.74
	19 YES	0.442	0.442	0.442	0.000	25.61
	44 YES	0.373	0.367	0.370	0.003	5.15
	YES	0.353	0.362	0.357	0.004	1.60
	36 YES	0.340	0.340	0.340	0.000	-3.37
	43 YES	0.310	0.320	0.315	0.005	-10.48
	24 YES	0.330	0.320	0.325	0.005	-7.64
	20 YES	0.280	0.280	0.280	0.000	-20.43
	20 YES	0.360	0.350	0.355	0.005	0.89
	37 YES	0.391	0.430	0.410	0.020	16.66
	?? NFI	0.280		0.280	0.000	-20.43
	28 YES	0.160	0.160	0.160	0.000	-54.53
	31 YES	0.330	0.350	0.340	0.010	-3.37
	04 YES	0.330	0.330	0.330	0.000	-6.22
	05 OUT	0.881	0.843	0.862	0.019	144.97
	10 YES	0.350	0.330	0.340	0.010	-3.37
	10 YES	0.380	0.380	0.380	0.000	7.99
	01 YES	0.410	0.400	0.405	0.005	15.10
	18 YES	0.370	0.432	0.401	0.031	13.96
	35 YES	0.286	0.311	0.299	0.013	-15.17
	23 YES	0.344	0.340	0.342	0.002	-2.81
	58 YES	0.370	0.344	0.357	0.013	1.46
	NFI	0.252		0.252	0.000	-28.44
NBS	NBS	0.345		0.345	0.000	-1.95

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STANDARD LAB ANALYTE=α-TOCOPHEROL SERUM #031 GRAND AVG = 11.296

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	25	YES	9.270	9.220	9.245	0.025	-18.16
	42	NFI	12.930		12.930	0.000	14.47
	76	YES	10.200	9.700	9.950	0.250	-11.92
	38	YES	11.930	12.320	12.125	0.195	7.34
	67	YES	12.700	12.400	12.550	0.150	11.10
	86	YES	13.600	13.900	13.750	0.150	21.73
	51	YES	10.300	10.100	10.200	0.100	-9.70
	??	YES	12.530	12.780	12.655	0.125	12.03
	45	NFI	11.000		11.000	0.000	-2.62
	53	YES	11.500	11.000	11.250	0.250	-0.41
	58	YES	8.020	9.170	8.595	0.575	-23.91
	94	NFI	11.700	11.800	11.750	0.050	4.02
	83	YES	12.000	12.100	12.050	0.050	6.68
	119	YES	12.410	12.470	12.440	0.030	10.13
	62	YES	6.800	6.600	6.700	0.100	-40.69
	116	YES	10.100	10.800	10.450	0.350	-7.49
	23	YES	13.620	13.780	13.700	0.080	21.28
		NFI	7.029		7.029	0.000	-37.77
	90	YES	12.850	12.500	12.675	0.175	12.21
	90	YES	12.450	12.350	12.400	0.050	9.77
NBS		NBS	11.900		11.900	0.000	5.35

STANDARD LAB ANALYTE=α-TOCOPHEROL SERUM #032 GRAND AVG = 6.120

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	47	YES	5.390	5.440	5.415	0.025	-11.52
	20	NFI	6.460		6.460	0.000	5.56
	48	YES	5.700	5.900	5.800	0.100	-5.23
	87	YES	6.530	6.290	6.410	0.120	4.74
	79	YES	7.400	8.700	8.050	0.650	31.54
	75	YES	7.700	7.800	7.750	0.050	26.63
	92	YES	5.700	5.400	5.550	0.150	-9.31
	??	YES	7.040	6.480	6.760	0.280	10.46
	34	NFI	6.200		6.200	0.000	1.31
	78	YES	5.600	5.400	5.500	0.100	-10.13
	29	YES	5.560	5.680	5.620	0.060	-8.17
	33	NFI	6.700	6.550	6.625	0.075	8.25
	06	YES	5.800	5.600	5.700	0.100	-6.86
	73	YES	6.620	6.600	6.610	0.010	8.01
	50	YES	5.800	4.700	5.250	0.550	-14.22
	99	YES	6.910	6.320	6.615	0.295	8.09
	92	YES	4.180	4.400	4.290	0.110	-29.90
		NFI	7.961		7.961	0.000	30.08
	90	YES	6.500	6.500	6.500	0.000	6.21
	90	YES	6.250	5.950	6.100	0.150	-0.33
NBS		NBS	6.556		6.556	0.000	7.12

STANDARD LAB ANALYTE=α-TOCOPHEROL SERUM #033 GRAND AVG = 6.510

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	36	YES	4.960	5.020	4.990	0.030	-23.35
	63	YES	6.500	6.700	6.600	0.100	1.39
	06	YES	6.720	6.780	6.750	0.030	3.69
	62	YES	7.080	7.140	7.110	0.030	9.22
	31	YES	6.290	6.510	6.400	0.110	-1.69
	45	YES	6.800	6.600	6.700	0.100	2.92

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STANDARD LAB ANALYTE=α-TOCOPHEROL SERUM #033 GRAND AVG = 6.510

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	14	YES	4.900	5.100	5.000	0.100	-23.19
	34	YES	7.790	8.320	8.055	0.265	23.74
	??	NFI	5.200		5.200	0.000	-20.12
	32	YES	4.013	4.127	4.070	0.057	-37.48
	12	YES	5.900	5.700	5.800	0.100	-10.90
	37	YES	5.710	5.580	5.645	0.065	-13.28
	04	NFI	6.790	6.970	6.880	0.090	5.69
	44	YES	9.500	9.100	9.300	0.200	42.86
	11	YES	6.310	6.300	6.305	0.005	-3.15
	25	YES	5.400	5.500	5.450	0.050	-16.28
	07	YES	6.150	6.530	6.340	0.190	-2.61
	50	YES	8.590	7.180	7.885	0.705	21.13
		NFI	2.040		2.040	0.000	-68.66
	24	YES	7.810	7.800	7.805	0.006	19.90
	24	YES	8.050	7.750	7.900	0.150	21.36
	18	YES	5.820	6.110	5.965	0.145	-8.37
	18	YES	5.860	6.390	6.125	0.265	-5.91
NBS		NBS	6.589		6.589	0.000	1.22

STANDARD LAB ANALYTE=α-TOCOPHEROL SERUM #034 GRAND AVG = 6.259

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	22	YES	5.200	5.020	5.110	0.090	-18.36
	42	YES	5.600	5.800	5.700	0.100	-8.93
	85	YES	6.690	6.570	6.630	0.060	5.93
		YES	7.250	7.000	7.125	0.125	13.84
	18	YES	5.670	5.780	5.725	0.055	-8.53
	07	YES	6.400	6.500	6.450	0.050	3.05
	72	YES	4.600	4.600	4.600	0.000	-26.50
	59	YES	8.000	8.630	8.315	0.315	32.85
	??	NFI	5.700		5.700	0.000	-8.93
	56	YES	4.185	4.089	4.137	0.048	-33.90
	36	YES	6.000	6.200	6.100	0.100	-2.54
	21	YES	5.270	5.300	5.285	0.015	-15.56
	51	NFI	6.860	6.990	6.925	0.065	10.64
	04	YES	8.200	8.600	8.400	0.200	34.21
	61	YES	6.000	6.250	6.125	0.125	-2.14
	74	YES	5.100	5.100	5.100	0.000	-18.52
	71	YES	5.910	6.350	6.130	0.220	-2.06
	75	YES	8.230	6.680	7.455	0.775	19.11
		NFI	6.932		6.932	0.000	10.76
	55	YES	7.780	7.490	7.635	0.145	21.99
	55	YES	6.650	6.800	6.725	0.075	7.45
	68	YES	5.880	5.910	5.895	0.015	-5.81
	68	YES	6.820	6.250	6.535	0.285	4.41
NBS		NBS	6.717		6.717	0.000	7.32

STANDARD LAB ANALYTE=α-TOCOPHEROL SERUM #035 GRAND AVG = 6.509

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	19	YES	5.130	5.010	5.070	0.060	-22.10
	68	YES	6.200	6.100	6.150	0.050	-5.51
	36	YES	6.670	6.800	6.735	0.065	3.48
	50	YES	5.600	6.070	5.835	0.235	-10.35
	61	YES	6.320	6.980	6.650	0.330	2.17

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STANDARD LAB ANALYTE=α-TOCOPHEROL SERUM #035 GRAND AVG = 6.509

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	70	YES	6.500	6.500	6.500	0.000	-0.13
	59	YES	4.800	4.800	4.800	0.000	-26.25
	15	YES	7.370	7.990	7.680	0.310	18.00
	63	NFI	5.900		5.900	0.000	-9.35
	49	YES	4.130	4.098	4.114	0.016	-36.79
	60	YES	6.500	6.600	6.550	0.050	0.64
	14	YES	5.610	5.720	5.665	0.055	-12.96
	69	NFI	6.860	6.950	6.905	0.045	6.09
	02	YES	8.200	8.700	8.450	0.250	29.83
	13	YES	6.280	6.260	6.270	0.010	-3.67
	01	NFI	5.600		5.600	0.000	-13.96
	20	YES	6.150	6.520	6.335	0.185	-2.67
	31	YES	7.940	8.170	8.055	0.115	23.76
		NFI	1.304		1.304	0.000	-79.97
	54	YES	8.580	8.090	8.335	0.245	28.06
	54	YES	7.450	7.550	7.500	0.050	15.23
	16	YES	6.470	6.190	6.330	0.140	-2.74
	16	YES	6.640	6.640	6.640	0.000	2.02
NBS		NBS	6.708		6.708	0.000	3.06

STANDARD LAB ANALYTE=α-TOCOPHEROL SERUM #044 GRAND AVG = 6.335

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	25	YES	4.990	5.030	5.010	0.020	-20.91
	19	YES	6.000	5.700	5.850	0.150	-7.65
	44	YES	6.940	6.640	6.790	0.150	7.18
		YES	6.790	7.000	6.895	0.105	8.84
	36	YES	6.050	6.480	6.265	0.215	-1.10
	43	YES	6.200	6.200	6.200	0.000	-2.13
	24	YES	4.500	4.700	4.600	0.100	-27.39
	37	YES	7.780	8.310	8.045	0.265	26.99
	??	NFI	6.000		6.000	0.000	-5.29
	28	YES	4.011	4.096	4.054	0.043	-36.01
	31	YES	6.500	6.300	6.400	0.100	1.03
	04	YES	5.940	5.500	5.720	0.220	-9.71
	08	NFI	6.900	6.990	6.945	0.045	9.63
	05	YES	8.800	7.500	8.150	0.650	28.65
	11	YES	6.760	6.670	6.715	0.045	6.00
	01	YES	5.100	4.900	5.000	0.100	-21.07
	18	YES	5.990	6.150	6.070	0.080	-4.18
	35	YES	8.420	7.450	7.935	0.485	25.26
		NFI	3.236		3.236	0.000	-48.92
	20	YES	7.930	7.740	7.835	0.095	23.68
	20	YES	7.500	6.650	7.075	0.425	11.68
	10	YES	5.340	5.520	5.430	0.090	-14.28
	10	YES	6.520	6.800	6.660	0.140	5.13
NBS		NBS	6.666		6.666	0.000	5.23

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STANDARD LAB ANALYTE=β-CAROTENE SERUM #31 GRAND AVG = 0.136

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	22	YES	0.125	0.125	0.125	0.000	-8.23
	25	YES	0.100	0.100	0.100	0.000	-26.59
	55	YES	0.176	0.206	0.191	0.015	40.22
	57	YES	0.150	0.134	0.142	0.008	4.25
	42	YES	0.136	0.140	0.138	0.002	1.31
	38	YES	0.138	0.152	0.145	0.007	6.45
	67	NFI	0.920	0.930	0.925	0.005	579.08
	86	YES	0.130	0.150	0.140	0.010	2.78
	51	YES	0.100	0.110	0.105	0.005	-22.92
	90	YES	0.150	0.150	0.150	0.000	10.12
	??	YES	0.126	0.136	0.131	0.005	-3.83
	45	NFI	0.110		0.110	0.000	-19.24
	83	OUT	0.260	0.270	0.265	0.005	94.55
	119	YES	0.130	0.120	0.125	0.005	-8.23
	62	YES	0.140	0.130	0.135	0.005	-0.89
	116	YES	0.141	0.145	0.143	0.002	4.98
	71	YES	0.134	0.140	0.137	0.003	0.58
NBS		NBS	0.115		0.115	0.000	-15.57

STANDARD LAB ANALYTE=β-CAROTENE SERUM #32 GRAND AVG = 0.434

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	10	YES	0.469	0.518	0.494	0.024	13.79
	47	YES	0.320	0.320	0.320	0.000	-26.22
	37	NFI	0.175	0.177	0.176	0.001	-59.42
	63	YES	0.462	0.455	0.459	0.004	5.72
	20	YES	0.430	0.418	0.424	0.006	-2.24
	87	YES	0.506	0.477	0.492	0.015	13.33
	79	NFI	1.040	0.920	0.980	0.060	125.96
	75	YES	0.470	0.480	0.475	0.005	9.52
	92	OUT	0.040	0.040	0.040	0.000	-90.78
	90	YES	0.370	0.380	0.375	0.005	-13.54
	??	YES	0.335	0.356	0.346	0.011	-20.34
	34	NFI	0.410		0.410	0.000	-5.47
	06	OUT	0.920	1.030	0.975	0.055	124.81
	73	YES	0.450	0.450	0.450	0.000	3.76
	50	YES	0.320	0.380	0.350	0.030	-19.30
	99	YES	0.516	0.501	0.509	0.008	17.24
	71	YES	0.513	0.513	0.513	0.000	18.28
NBS		NBS	0.399		0.399	0.000	-8.00

STANDARD LAB ANALYTE=β-CAROTENE SERUM #33 GRAND AVG = 0.091

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	46	YES	0.097	0.097	0.097	0.000	6.18
	36	YES	0.050	0.050	0.050	0.000	-45.27
	19	YES	0.110	0.123	0.117	0.007	27.53
	60	YES	0.088	0.067	0.078	0.011	-15.16
	54	NFI	0.103		0.103	0.000	12.75
	06	YES	0.065	0.065	0.065	0.000	-28.85
	62	YES	0.084	0.085	0.085	0.001	-7.50
	31	NFI	0.860	0.940	0.900	0.040	885.19
	45	YES	0.080	0.080	0.080	0.000	-12.43
	14	YES	0.090	0.090	0.090	0.000	-1.48
	24	YES	0.080	0.070	0.075	0.005	-17.90

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STANDARD LAB			ANALYTE=β-CAROTENE		SERUM #33 GRAND AVG =		0.091
LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	34	YES	0.099	0.094	0.097	0.003	5.63
	??	NFI	0.617		0.617	0.000	575.40
	11	YES	0.120	0.083	0.102	0.018	11.11
	18	YES	0.100	0.100	0.100	0.000	9.47
	18	YES	0.120	0.110	0.115	0.005	25.89
	25	YES	0.130	0.140	0.135	0.005	47.78
	07	YES	0.105	0.105	0.105	0.000	14.94
	08	YES	0.076	0.081	0.079	0.002	-14.07
	29	YES	0.090	0.082	0.086	0.004	-5.86
NBS		NBS	0.066		0.066	0.000	-27.75

STANDARD LAB			ANALYTE=β-CAROTENE		SERUM #34 GRAND AVG =		0.199
LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	39	YES	0.192	0.194	0.193	0.001	-2.87
	22	YES	0.110	0.110	0.110	0.000	-44.64
	40	YES	0.254	0.252	0.253	0.001	27.32
	24	YES	0.194	0.200	0.197	0.003	-0.86
	27	YES	0.235	0.241	0.238	0.003	19.78
	85	YES	0.147	0.172	0.160	0.013	-19.73
		YES	0.203	0.204	0.204	0.000	2.41
	18	NFI	0.970	0.990	0.980	0.010	393.19
	07	YES	0.190	0.170	0.180	0.010	-9.41
	72	YES	0.160	0.160	0.160	0.000	-19.48
	55	YES	0.250	0.270	0.260	0.010	30.85
	59	YES	0.205	0.197	0.201	0.004	1.15
	??	NFI	0.214		0.214	0.000	7.70
	61	OUT	0.570	0.570	0.570	0.000	186.86
	68	YES	0.170	0.170	0.170	0.000	-14.45
	68	YES	0.170	0.190	0.180	0.010	-9.41
	74	YES	0.200	0.200	0.200	0.000	0.65
	71	YES	0.217	0.177	0.197	0.020	-0.86
	37	YES	0.222	0.221	0.222	0.001	11.47
	11	YES	0.228	0.281	0.255	0.026	28.08
NBS		NBS	0.160		0.160	0.000	-19.48

STANDARD LAB			ANALYTE=β-CAROTENE		SERUM #35 GRAND AVG =		0.216
LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	52	YES	0.187	0.196	0.191	0.005	-11.25
	19	YES	0.110	0.110	0.110	0.000	-49.02
	58	YES	0.286	0.314	0.300	0.014	39.04
	48	YES	0.318	0.298	0.308	0.010	42.75
	40	YES	0.246	0.246	0.246	0.000	14.01
	36	YES	0.170	0.160	0.165	0.005	-23.53
	50	YES	0.184	0.198	0.191	0.007	-11.48
	61	NFI	0.990	0.990	0.990	0.000	358.83
	70	YES	0.180	0.190	0.185	0.005	-14.26
	59	YES	0.150	0.150	0.150	0.000	-30.48
	54	YES	0.280	0.290	0.285	0.005	32.09
	15	YES	0.185	0.169	0.177	0.008	-17.97
	63	NFI	0.171		0.171	0.000	-20.75
	13	YES	0.200	0.172	0.186	0.014	-13.79
	16	YES	0.200	0.200	0.200	0.000	-7.31
	16	YES	0.200	0.190	0.195	0.005	-9.62
	01	NFI	0.230		0.230	0.000	6.60
	20	YES	0.349	0.181	0.265	0.084	22.82
	04	YES	0.270	0.266	0.268	0.002	24.21
	12	YES	0.232	0.259	0.246	0.013	13.78
NBS		NBS	0.160		0.160	0.000	-25.85

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STANDARD LAB		ANALYTE=β-CAROTENE	SERUM #44 GRAND AVG =	0.088		
LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	9	YES	0.091	0.091	0.091	0.000	3.17
	25	YES	0.050	0.050	0.050	0.000	-43.31
	26	YES	0.116	0.113	0.115	0.002	29.82
	38	YES	0.071	0.081	0.076	0.005	-13.83
	07	NFI	0.089		0.089	0.000	0.91
	44	YES	0.072	0.067	0.070	0.002	-21.20
		YES	0.077	0.085	0.081	0.004	-8.16
	36	NFI	0.860	0.920	0.890	0.030	909.07
	43	YES	0.080	0.070	0.075	0.005	-14.97
	24	YES	0.100	0.090	0.095	0.005	7.71
	20	OUT	0.210	0.190	0.200	0.010	126.76
	37	YES	0.095	0.089	0.092	0.003	4.31
	??	NFI	0.070		0.070	0.000	-20.63
	11	OUT	0.260	0.300	0.280	0.020	217.46
	10	YES	0.080	0.080	0.080	0.000	-9.30
	10	YES	0.080	0.100	0.090	0.010	2.04
	01	YES	0.100	0.120	0.110	0.010	24.72
	18	YES	0.138	0.106	0.122	0.016	38.32
	23	YES	0.091	0.093	0.092	0.001	4.31
	58	YES	0.086	0.084	0.085	0.001	-3.63
NBS		NBS	0.062		0.062	0.000	-29.71

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RR VII Statistical Summary

STANDARD LAB ANALYTE=RETINOL

SER	GRAND AVG	S.E. AVG	S WITHIN	S BETWEEN	% CV AVG	% CV X	FR REJECT
031	0.793	0.024	0.019	0.109	3.0	13.9	1/22
032	0.417	0.013	0.017	0.058	3.2	14.5	1/21
033	0.341	0.011	0.015	0.050	3.1	15.4	1/24
034	0.346	0.013	0.014	0.063	3.7	18.7	1/26
035	0.337	0.013	0.016	0.060	3.8	18.4	1/24
044	0.352	0.012	0.014	0.059	3.5	17.3	1/25

STANDARD LAB ANALYTE=α-TOCOPHEROL

SER	GRAND AVG	S.E. AVG	S WITHIN	S BETWEEN	% CV AVG	% CV X	FR REJECT
031	11.296	0.488	0.304	1.942	4.3	17.4	0/16
032	6.120	0.236	0.353	0.910	3.9	15.9	0/16
033	6.510	0.279	0.287	1.231	4.3	19.4	0/20
034	6.259	0.257	0.311	1.126	4.1	18.7	0/20
035	6.509	0.265	0.222	1.146	4.1	17.9	0/19
044	6.335	0.260	0.337	1.137	4.1	18.7	0/20

STANDARD LAB ANALYTE=β-CAROTENE

SER	GRAND AVG	S.E. AVG	S WITHIN	S BETWEEN	% CV AVG	% CV X	FR REJECT
031	0.136	0.006	0.009	0.020	4.2	16.3	1/15
032	0.434	0.020	0.018	0.068	4.6	16.2	2/14
033	0.091	0.005	0.008	0.020	5.5	23.4	0/17
034	0.199	0.009	0.014	0.038	4.8	20.2	1/18
035	0.216	0.014	0.031	0.052	6.3	27.9	0/17
044	0.088	0.005	0.009	0.017	5.3	21.8	2/17

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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

Appendix C. Updated "All-Lab Report" for RR07

The following three pages are the modernized "All-Lab" report for RR07. This report has three parts:

- Page 1 lists the participant mean results for all analytes reported.
- Page 2 provides the legend for page 1.
- Page 3 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 RR07. 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.

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Round Robin VII Laboratory Results

	Retinol, µg/mL						α-Tocopherol, µg/mL						Total β-Carotene, µg/mL					
Lab	31	32	33	34	35	44	31	32	33	34	35	44	31	32	33	34	35	44
FSV-BD	0.695	0.345	0.340	0.335	0.325	0.340	12.55	8.05	6.40	5.73	6.65	6.27	0.925	0.980	0.900	0.980	0.990	0.890
FSV-BE	1.058	0.448					12.05	5.70	6.31	6.13	6.27	6.72	0.265	0.975	0.102	0.540	0.186	0.280
FSV-BF	0.630	0.330	0.280	0.250	0.290	0.280	11.00	6.20	5.20	5.70	5.90	6.00	0.110	0.410	0.617	0.214	0.171	0.070
FSV-BG	0.948	0.555	0.438	0.457	0.447	0.456							0.191	0.176	0.117	0.253	0.300	0.115
FSV-BI	0.830	0.430	0.375	0.370	0.360	0.365	9.25	5.42	4.99	5.11	5.07	5.01	0.100	0.320	0.050	0.110	0.110	0.050
FSV-BY	0.764	0.417		0.349		0.362							0.125	0.494	0.097	0.193	0.192	0.091
FSV-CA	0.780	0.402	0.392	0.403	0.392	0.442	9.95	5.80	6.60	5.70	6.15	5.85						
FSV-CJ	0.833	0.416	0.395	0.391	0.406	0.393	12.93	6.46					0.138	0.424	0.103	0.238	0.246	0.089
FSV-CL	0.917	0.474	0.401	0.415	0.368	0.411	12.66	6.76	8.06	8.32	7.68	8.05	0.131	0.346	0.097	0.201	0.177	0.092
FSV-CN	0.680	0.435	0.325	0.315	0.300	0.325	10.20	5.55	5.00	4.60	4.80	4.60	0.105	0.040	0.090	0.160	0.150	0.095
FSV-CO			0.318	0.360	0.324	0.370			6.75	6.63	6.74	6.79			0.065	0.160	0.165	0.070
FSV-DC	0.710	0.412	0.347	0.355	0.347	0.342							0.137	0.513	0.079	0.222	0.268	0.092
FSV-DE							11.75	6.63	6.88	6.93	6.91	6.95						
FSV-DG	0.795	0.400	0.320	0.325	0.330	0.340	12.44	6.61	5.97	5.90	6.33	5.43	0.125	0.450	0.100	0.170	0.200	0.080
FSV-DGa			0.370	0.365	0.325	0.380			6.13	6.54	6.64	6.66			0.115	0.180	0.195	0.090
FSV-DH	0.570	0.310	0.275	0.280	0.290	0.280	12.68	6.50	7.81	7.64	8.34	7.84	0.150	0.375	0.075	0.260	0.285	0.200
FSV-DHa	0.720	0.400	0.355	0.350	0.340	0.355	12.40	6.10	7.90	6.73	7.50	7.08						
FSV-DN	0.840	0.550	0.280	0.310	0.285	0.315	13.75	7.75	6.70	6.45	6.50	6.20	0.140	0.475	0.080	0.180	0.185	0.075
FSV-DO	0.135	0.080	0.310	0.290	0.330	0.300												
FSV-DT	0.735	0.395	0.335	0.335	0.365	0.340	11.25	5.50	5.80	6.10	6.55	6.40						
FSV-DZ	0.697	0.583	0.178	0.265	0.236	0.252	7.03	7.96	2.04	6.93	1.30	3.24						
FSV-EG	0.938	0.491	0.358	0.407	0.392	0.414							0.142	0.459	0.078	0.197	0.308	0.076
FSV-EN	0.795	0.301	0.302	0.297	0.254	0.299	13.70	4.29	7.89	7.46	8.06	7.94						
FSV-EO			0.365	0.370	0.398	0.357									0.086	0.255	0.246	0.085
FSV-EP	0.675	0.375	0.325	0.280	0.350	0.330	8.60	5.62	5.65	5.29	5.67	5.72						
FSV-ER	0.777	0.409	0.347	0.359	0.281	0.358	12.13	6.41	7.11	7.13	5.84	6.90	0.145	0.492	0.085	0.204	0.191	0.081
FSV-EU	0.760	0.450	0.415	0.410	0.370	0.405	6.70	5.25	5.45	5.10	5.60	5.00	0.135	0.350	0.135	0.200	0.230	0.110
FSV-EW	0.831	0.488	0.355	0.406	0.388	0.401	10.45	6.62	6.34	6.13	6.34	6.07	0.143	0.509	0.105	0.197	0.265	0.122
FSV-FO			0.195	0.135	0.155	0.160			4.07	4.14	4.11	4.05						
FSV-GC			0.830	0.801	0.771	0.862			9.30	8.40	8.45	8.15						
n	24	24	27	28	27	28	20	20	23	23	23	23	17	17	20	20	20	20
Min	0.135	0.080	0.178	0.135	0.155	0.160	6.70	4.29	2.04	4.14	1.30	3.24	0.100	0.040	0.050	0.110	0.110	0.050
Median	0.770	0.414	0.347	0.352	0.340	0.356	11.90	6.31	6.34	6.13	6.34	6.27	0.138	0.450	0.097	0.201	0.198	0.091
Max	1.058	0.583	0.830	0.801	0.771	0.862	13.75	8.05	9.30	8.40	8.45	8.15	0.925	0.980	0.900	0.980	0.990	0.890
eSD	0.098	0.056	0.043	0.069	0.070	0.058	1.43	0.82	1.14	1.18	0.85	1.01	0.018	0.093	0.026	0.039	0.059	0.022
eCV	13	13	12	19	21	16	12	13	18	19	13	16	13	21	27	19	30	25
NISTa	0.776	0.417	0.396	0.357	0.404	0.394	11.75	6.12	6.50	6.66	6.63	6.82	0.128	0.418	0.090	0.182	0.183	0.089
NAV	0.773	0.415	0.371	0.355	0.372	0.375	11.83	6.21	6.42	6.39	6.48	6.54	0.133	0.434	0.094	0.191	0.190	0.090
NAU	0.098	0.056	0.043	0.069	0.070	0.058	1.43	0.82	1.14	1.18	0.85	1.01	0.018	0.093	0.026	0.039	0.059	0.022

C2

Round Robin VII 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 heterogeneity and inter- and intra-laboratory) standard deviation

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ıarability Summary

Lab	TR	aT	bC
FSV-BD	1	1	4
FSV-BE	3	1	4
FSV-BF	2	1	4
FSV-BG	2		3
FSV-BI	1	2	2
FSV-BY	1		1
FSV-CA	1	1	
FSV-CJ	1	1	1
FSV-CL	2	2	1
FSV-CN	1	2	3
FSV-CO	1	1	1
FSV-DC	1		1
FSV-DE		1	
FSV-DG	1	1	1
FSV-DGa	1	1	1
FSV-DH	2	2	3
FSV-DHa	1	1	
FSV-DN	2	1	1
FSV-DO	4		
FSV-DT	1	1	
FSV-DZ	3	4	
FSV-EG	2		1
FSV-EN	2	2	
FSV-EO	1		1
FSV-EP	1	2	
FSV-ER	1	1	1
FSV-EU	1	2	1
FSV-EW	1	1	1
FSV-FO	4	3	
FSV-GC	4	3	
NISTa	1	1	1
n	30	25	21

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	60	67	68.2 %
% 2	23	28	5	27.3 %
% 3	7	8	14	4.3 %
% 4	10	4	14	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.

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Appendix D. Shipping Package Inserts for RR08

Two items were attached to each package shipped to an RR08 participant:

- **Cover letter**. The original letter as attached to the packages has been lost. It would have described the three lyophilized sample materials (sera 50, 51, and 52) distributed for the study.

- **Datasheet**. Page D2 reproduces the form.

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Report on NBS/NCI Samples from Laboratory #____

Results in mg/L

Samples		Result 1	Result 2
Serum No: 50 Vial No. ____ Analysis Date / /86	Retinol β-Carotene α-Tocopherol		
Serum No: 51 Vial No. ____ Analysis Date / /86	Retinol β-Carotene α-Tocopherol		
Serum No: 52 Vial No. ____ Analysis Date / /86	Retinol β-Carotene α-Tocopherol		

Note: Add 1.20 mL of water to reconstitute Freeze-dried samples.

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Appendix E. Final Report for RR08

The following 16 pages are the available fragments of the report for RR08 provided to participants. These fragments are:

- An individualized letter.
- The reported results for retinol, α-tocopherol, and β-carotene; the ascorbic acid, selenium, and zinc results included in the original report have been removed. Due to the complex formatting used in the tables, the original laboratory codes have been deleted without replacement. Appendix F provides a complete listing of the RR08 results where the original codes have been altered to ensure confidentiality. Appendix F also provides relevant summary statistics.
- The statistical summary table and its legend.
- Descriptions of standard solution preparation for retinol, β-carotene, and α-tocopherol.
- A description of graphical analysis, plotting each participant's results against consensus values, followed by tables listing least-square parameter values of intercept, slope, and "S fit" (more commonly known as the root-mean square error (RMSE) of the fit.) None of the original graphs are extant. A modern re-creation is included below each table as an approximate example of the original graphs (which would have been larger and would not have used color).

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

February 24, 1987

Individualized
name and address

Dear First name:

This letter accompanies:

(1) the combination of information we usually send when a round robin exercise is ended and the lab data have been assembled and analyzed (now it's RR VIII);

(2) copies of Reports of Analysis describing the methods of analysis utilized for the NBS values; and

(3) graphs that Paule and Ruegg have prepared from the RR VIII results and, separately, from the previous results of RRs V-VII:

(a) the RR VIII graphs are for every lab that reported on the analytes which you measured in RR VIII, and;
(b) the RR V-VII graphs are only for measurements you reported.

NOTE: A graph was drawn only if a lab reported values for all the samples having the analyte and all the values were accepted, i.e., none was an outlier or an NFI (not following instructions). Note also that the "perfect agreement" lines on the graphs involve the consensus values, not the NBS values now being listed for each sample. Except for beta-carotene, the consensus (or grand averages) and the NBS averages are at present in reasonable agreement. We hope, with further investigation, to uncover the major cause of the rather consistent differences for beta-carotene. (The consensus is higher than NBS.) We would expect the NBS values to be the benchmark values in the future, given that adequate documentation can be assembled to demonstrate that position.

As usual for the statistical analyses, as much of the reported data as possible were retained. (Less than 5% of RR VIII data were rejected as outliers.) For RR VIII we again see that the between-lab variability is greater than the within-lab variability. The % CV AVG values are generally 2 to 10%, and the % CVx values are generally 10 to 25%. The Statistical Summary lists NBS average values for the sample, but the NBS values are not incorporated in the statistics.

Sincerely,

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

Enclosures

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RESULTS FROM ROUND ROBIN VIII IN NCI/NBS MICRONUTRIENT MEASUREMENT PROGRAM

ANALYTE = RETINOL SERUM #50 GRAND AVG = 0.383 NBS AVG = 0.375

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	28	YES	0.338	0.330	0.334	0.004	-12.73
	41	YES	0.390	0.390	0.390	0.000	1.91
	93	YES	0.418	0.421	0.420	0.002	9.62
	40	YES	0.323	0.313	0.318	0.005	-16.91
	29	YES	0.416	0.399	0.408	0.009	6.48
	96	YES	0.090	0.110	0.100	0.010	-73.87
	100	YES	0.368	0.358	0.363	0.005	-5.15
	25	YES	0.362	0.359	0.361	0.001	-5.80
	117	YES	0.383	0.409	0.396	0.013	3.48
	20	YES	0.407	0.383	0.395	0.012	3.21
	114	YES	0.324	0.301	0.313	0.011	-18.34
	36	YES	0.420	0.390	0.405	0.015	5.83
	113	YES	0.380	0.400	0.390	0.010	1.91
	102	YES	0.381	0.395	0.388	0.007	1.38
	76	YES	0.470	0.480	0.475	0.005	24.12
	92	YES	0.360	0.330	0.345	0.015	-9.85
	57	YES	0.420	0.420	0.420	0.000	9.75
		YES	0.390	0.450	0.420	0.030	9.75
	112	YES	0.380	0.380	0.380	0.000	-0.71
	46	YES	0.770	0.740	0.755	0.015	97.28
	3	YES	0.444	0.470	0.457	0.013	19.41
	78	YES	0.266	0.256	0.261	0.005	-31.80
	50	YES	0.330	0.370	0.350	0.020	-8.54
	63	OUT	1.214	0.889	1.052	0.163	174.76
	74	YES	0.358	0.340	0.349	0.009	-8.81
	97	YES	0.391	0.362	0.377	0.014	-1.62
NBS	A*	NBS			0.386		0.86
NBS	B*	NBS			0.386		0.86
NBS	C*	NBS			0.374		-2.27
NBS	D*	NBS			0.353		-7.76

ANALYTE = RETINOL SERUM #51 GRAND AVG = 0.431 NBS AVG = 0.442

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	102	YES	0.449	0.402	0.426	0.024	-1.39
	93	YES	0.440	0.440	0.440	0.000	1.97
	85	YES	0.465	0.466	0.466	0.001	7.88
	66	YES	0.360	0.364	0.362	0.002	-16.10
	142	YES	0.436	0.441	0.439	0.003	1.63
	99	YES	0.250	0.250	0.250	0.000	-42.06
	64	YES	0.409	0.405	0.407	0.002	-5.67
	38	YES	0.404	0.402	0.403	0.001	-6.60
	63	YES	0.427	0.423	0.425	0.002	-1.50
	77	YES	0.413	0.418	0.415	0.003	-3.70
	61	YES	0.356	0.320	0.338	0.018	-21.66
	78	YES	0.420	0.460	0.440	0.020	1.97
	75	YES	0.430	0.440	0.435	0.005	0.82
	15	YES	0.439	0.421	0.430	0.009	-0.34
	21	YES	0.550	0.570	0.560	0.010	29.79

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ANALYTE = RETINOL SERUM #51 GRAND AVG = 0.431 NBS AVG = 0.442

LAB	VIAL	USED	RESULT1	RESULT2			
	16	YES	0.380	0.360	0.370	0.010	-14.25
	86	YES	0.460	0.450	0.455	0.005	5.45
		YES	0.460	0.480	0.470	0.010	8.93
	80	YES	0.440	0.430	0.435	0.005	0.82
	9	YES	0.690	0.720	0.705	0.015	63.39
	39	YES	0.503	0.536	0.520	0.016	20.40
	3	YES	0.252	0.247	0.250	0.002	-42.18
	4	YES	0.510	0.600	0.555	0.045	28.63
	13	OUT	1.088	1.030	1.059	0.029	145.43
	89	YES	0.377	0.379	0.378	0.001	-12.39
	79	YES	0.423	0.407	0.415	0.008	-3.82
NBS	A*	NBS			0.461		6.84
NBS	B*	NBS			0.427		-1.04
NBS	C*	NBS			0.446		3.37
NBS	D*	NBS			0.435		0.82

ANALYTE = RETINOL SERUM #52 GRAND AVG = 0.921 NBS AVG = 0.937

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	96	YES	0.835	0.820	0.828	0.008	-10.15
	67	YES	0.920	0.930	0.925	0.005	0.44
	71	YES	0.944	0.983	0.964	0.019	4.62
	82	YES	0.798	0.839	0.819	0.021	-11.13
	7	YES	1.014	1.024	1.019	0.005	10.64
	94	YES	0.400	0.370	0.385	0.015	-58.20
	72	YES	0.820	0.835	0.828	0.008	-10.15
	2	YES	0.849	0.855	0.852	0.003	-7.49
	64	YES	0.872	0.888	0.880	0.008	-4.45
	49	YES	0.873	0.846	0.860	0.014	-6.68
	73	NFI	0.459		0.459	0.000	-50.16
	9	YES	0.920	0.880	0.900	0.020	-2.28
	3	YES	0.990	0.980	0.985	0.005	6.95
	99	YES	0.873	0.804	0.839	0.034	-8.96
	112	YES	1.180	1.180	1.180	0.000	28.12
	32	YES	0.770	0.780	0.775	0.005	-15.85
	70	YES	1.010	1.040	1.025	0.015	11.29
		YES	1.090	0.990	1.040	0.050	12.92
	81	YES	0.960	0.950	0.955	0.005	3.69
	18	YES	1.640	1.680	1.660	0.020	80.24
	12	YES	1.140	1.180	1.160	0.020	25.95
	28	YES	0.390	0.436	0.413	0.023	-55.16
	38	YES	1.100	1.100	1.100	0.000	19.44
	90	OUT	1.704	2.146	1.925	0.221	109.02
	5	YES	0.817	0.787	0.802	0.015	-12.92
	75	YES	0.935	0.890	0.913	0.022	-0.92
NBS	A*	NBS			0.994		7.93
NBS	B*	NBS			0.909		-1.30
NBS	C*	NBS			0.918		-0.32
NBS	D*	NBS			0.926		0.55

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ANALYTE = α-TOCOPHEROL SERUM #50 GRAND AVG = 9.256 NBS AVG = 8.708

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	41	YES	7.590	7.470	7.530	0.060	-18.64
	29	YES	9.400	9.110	9.255	0.145	-0.01
	100	YES	7.980	8.010	7.995	0.015	-13.62
	25	YES	8.430	8.350	8.390	0.040	-9.35
	117	YES	9.630	9.570	9.600	0.030	3.72
	20	YES	7.000	8.100	7.550	0.550	-18.43
	114	YES	8.200	7.700	7.950	0.250	-14.11
	36	YES	8.600	8.000	8.300	0.300	-10.33
	113	YES	8.800	10.000	9.400	0.600	1.56
	102	YES	9.820	9.100	9.460	0.360	2.21
	76	YES	14.000	15.000	14.500	0.500	56.66
	92	YES	16.280	16.110	16.195	0.085	74.97
	57	YES	7.430	7.460	7.445	0.015	-19.56
	119	YES	9.190	9.160	9.175	0.015	-0.87
		YES	7.850	8.010	7.930	0.080	-14.32
	112	YES	8.460	8.260	8.360	0.100	-9.68
	46	YES	14.200	13.700	13.950	0.250	50.72
	3	YES	9.370	9.650	9.510	0.140	2.75
	78	YES	6.620	7.140	6.880	0.260	-25.67
	63	YES	6.963	8.665	7.814	0.851	-15.58
	74	YES	7.750	7.423	7.587	0.163	-18.03
	97	YES	9.000	8.700	8.850	0.150	-4.38
NBS	A*	NBS			8.840		-4.49
NBS	B*	NBS			8.470		-8.49
NBS	C*	NBS			9.020		-2.55
NBS	D*	NBS			8.500		-8.16

ANALYTE = α-TOCOPHEROL SERUM #51 GRAND AVG = 7.293 NBS AVG = 7.158

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	93	YES	6.230	6.010	6.120	0.110	-16.09
	142	YES	7.460	7.360	7.410	0.050	1.60
	64	YES	6.460	6.320	6.390	0.070	-12.39
	38	YES	6.860	6.810	6.835	0.025	-6.28
	63	YES	7.920	7.910	7.915	0.005	8.52
	61	YES	7.300	7.200	7.250	0.050	-0.59
	78	YES	6.900	7.100	7.000	0.100	-4.02
	75	YES	8.900	7.410	8.155	0.745	11.81
	15	YES	7.840	7.870	7.855	0.015	7.70
	21	YES	11.500	12.700	12.100	0.600	65.91
	16	YES	6.650	7.160	6.905	0.255	-5.32
	86	YES	6.540	6.480	6.510	0.030	-10.74
	65	YES	7.640	7.660	7.650	0.010	4.89
		YES	6.320	6.560	6.440	0.120	-11.70
	80	YES	7.140	7.000	7.070	0.070	-3.06
	9	YES	9.300	9.100	9.200	0.100	26.14
	39	YES	8.180	8.280	8.230	0.050	12.84
	3	YES	3.390	3.940	3.665	0.275	-49.75
	13	OUT	2.609	8.025	5.317	2.708	-27.10

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ANALYTE = α-TOCOPHEROL SERUM #51 GRAND AVG = 7.293 NBS AVG = 7.158

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	89	YES	6.172	6.160	6.166	0.006	-15.46
	79	YES	7.200	6.800	7.000	0.200	-4.02
NBS	A*	NBS			7.050		-3.34
NBS	B*	NBS			6.740		-7.59
NBS	C*	NBS			7.560		3.66
NBS	D*	NBS			7.280		-0.18

ANALYTE = α-TOCOPHEROL SERUM #52 GRAND AVG = 10.594 NBS AVG = 10.668

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	67	YES	8.980	9.030	9.005	0.025	-15.00
	7	YES	11.050	10.840	10.945	0.105	3.32
	72	YES	9.560	9.600	9.580	0.020	-9.57
	2	YES	10.260	10.280	10.270	0.009	-3.06
	64	YES	11.560	12.040	11.800	0.240	11.39
	49	YES	6.200	7.100	6.650	0.450	-37.23
	49	YES	9.200	8.400	8.800	0.400	-16.93
	73	NFI	9.200		9.200	0.000	-13.16
	9	YES	10.700	9.800	10.250	0.450	-3.25
	3	YES	12.590	12.200	12.395	0.195	17.00
	99	YES	11.280	10.820	11.050	0.230	4.31
	112	YES	17.200	18.100	17.650	0.450	66.61
	32	YES	9.530	9.530	9.530	0.000	-10.04
	70	YES	9.330	9.670	9.500	0.170	-10.32
	80	YES	11.480	11.230	11.355	0.125	7.19
		YES	9.790	10.300	10.045	0.255	-5.18
	81	YES	10.480	10.260	10.370	0.110	-2.11
	18	YES	15.400	16.100	15.750	0.350	48.67
	12	YES	11.600	12.000	11.800	0.200	11.39
	28	YES	8.310	7.420	7.865	0.445	-25.76
	90	YES	8.937	8.291	8.614	0.323	-18.69
	5	YES	9.352	9.126	9.239	0.113	-12.79
	75	YES	10.800	10.400	10.600	0.200	0.06
NBS	A*	NBS			10.880		2.70
NBS	B*	NBS			10.080		-4.85
NBS	C*	NBS			10.890		2.80
NBS	D*	NBS			10.820		2.14

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ANALYTE = β-CAROTENE SERUM #50 GRAND AVG = 0.094 NBS AVG = 0.066

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	130	YES	0.082	0.099	0.091	0.009	-4.09
	41	YES	0.070	0.070	0.070	0.000	-25.82
	93	YES	0.089	0.085	0.087	0.002	-7.80
	40	YES	0.030	0.030	0.030	0.000	-68.21
	87	YES	0.081	0.081	0.081	0.000	-14.16
	25	YES	0.091	0.090	0.091	0.001	-4.09
	117	YES	0.072	0.076	0.074	0.002	-21.58
	114	YES	0.039	0.044	0.041	0.003	-56.02
	36	YES	0.160	0.200	0.180	0.020	90.76
	102	YES	0.120	0.114	0.117	0.003	23.99
	76	YES	0.144	0.138	0.141	0.003	49.43
	112	YES	0.070	0.070	0.070	0.000	-25.82
	46	YES	0.190	0.100	0.145	0.045	53.66
	3	YES	0.114	0.114	0.114	0.000	20.81
	50	YES	0.080	0.070	0.075	0.005	-20.52
	72	YES	0.108	0.098	0.103	0.005	9.16
	74	YES	0.066	0.062	0.064	0.002	-32.18
	97	YES	0.130	0.120	0.125	0.005	32.47
NBS	A*	NBS			0.068		-27.94
NBS	B*	NBS			0.071		-24.76
NBS	C*	NBS			0.063		-33.24
NBS	D*	NBS			0.063		-33.24

ANALYTE = β-CAROTENE SERUM #51 GRAND AVG = 1.171 NBS AVG = 1.037

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	136	YES	0.878	0.959	0.919	0.041	-21.57
	93	YES	0.980	0.940	0.960	0.020	-18.03
	85	YES	1.369	1.358	1.364	0.006	16.42
	66	YES	0.566	0.706	0.636	0.070	-45.70
	20	YES	1.062	1.048	1.055	0.007	-9.92
	38	YES	1.159	1.146	1.153	0.006	-1.59
	63	YES	1.114	1.127	1.121	0.007	-4.33
	61	YES	0.874	0.725	0.800	0.075	-31.73
	78	YES	2.080	2.140	2.110	0.030	80.16
	15	YES	0.916	0.946	0.931	0.015	-20.51
	21	YES	2.227	2.040	2.134	0.093	82.17
	80	YES	1.080	1.020	1.050	0.030	-10.35
	9	YES	0.910	0.980	0.945	0.035	-19.31
	39	YES	1.150	1.190	1.170	0.020	-0.10
	4	YES	1.090	0.990	1.040	0.050	-11.20
	32	YES	1.466	1.463	1.465	0.002	25.05
	89	YES	0.907	0.946	0.927	0.019	-20.89
	79	YES	1.330	1.280	1.305	0.025	11.43
NBS	A*	NBS			0.998		-14.79
NBS	B*	NBS			1.057		-9.75
NBS	C*	NBS			1.050		-10.35
NBS	D*	NBS			1.041		-11.11

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ANALYTE = β-CAROTENE SERUM #52 GRAND AVG = 0.657 NBS AVG = 0.558

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	175	YES	0.531	0.506	0.518	0.013	-21.04
	67	YES	0.520	0.520	0.520	0.000	-20.81
	71	YES	0.664	0.714	0.689	0.025	4.92
	82	YES	0.335	0.465	0.400	0.065	-39.09
	92	YES	0.622	0.590	0.606	0.016	-7.72
	2	YES	0.641	0.645	0.643	0.002	-2.08
	64	YES	0.604	0.628	0.616	0.012	-6.19
	73	NFI	0.385		0.385	0.000	-41.37
	9	YES	1.160	1.060	1.110	0.050	69.03
	99	YES	0.567	0.583	0.575	0.008	-12.44
	112	YES	1.043	0.995	1.019	0.024	55.18
	81	YES	0.630	0.610	0.620	0.010	-5.59
	18	YES	0.550	0.530	0.540	0.010	-17.77
	12	YES	0.662	0.701	0.682	0.019	3.78
	38	YES	0.620	0.660	0.640	0.020	-2.54
	117	YES	0.731	0.764	0.748	0.017	13.83
	5	YES	0.518	0.508	0.513	0.005	-21.88
	75	YES	0.740	0.710	0.725	0.015	10.40
NBS	A*	NBS			0.571		-13.05
NBS	B*	NBS			0.615		-6.35
NBS	C*	NBS			0.521		-20.66
NBS	D*	NBS			0.523		-20.36

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Statistical Summary for ROUND ROBIN VIII

ANALYTE = RETINOL

SER	NBS	GRAND AVG	S.E. AVG	S WITHIN	S BETWEEN	% CV AVG	% CV X	FR REJECT
50	0.375	0.383	0.021	0.016	0.105	5.5	27.8	1/26
51	0.442	0.431	0.019	0.019	0.092	4.3	21.7	1/26
52	0.937	0.921	0.050	0.026	0.243	5.4	26.6	1/25

ANALYTE = α-TOCOPHEROL

SER	NBS	GRAND AVG	S.E. AVG	S WITHIN	S BETWEEN	% CV AVG	% CV X	FR REJECT
51	7.18	7.293	0.355	0.341	1.567	4.9	22.0	1/21
50	8.71	9.256	0.521	0.444	2.422	5.6	26.6	0/22
52	10.67	10.594	0.514	0.377	2.394	4.8	22.9	0/22

ANALYTE = β-CAROTENE

SER	NBS	GRAND AVG	S.E. AVG	S WITHIN	S BETWEEN	% CV AVG	% CV X	FR REJECT
50	0.066	0.094	0.009	0.017	0.036	9.4	41.9	0/18
52	0.558	0.657	0.043	0.034	0.175	6.5	27.2	0/17
51	1.04	1.171	0.094	0.056	0.396	8.0	34.2	0/18

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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

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PREPARATION OF STANDARD SOLUTIONS FOR β-CAROTENE, ALL TRANS-RETINOL, AND α-TOCOPHEROL

trans-Retinol and β-carotene samples are not pure. Therefore, standard solutions cannot be prepared directly by weight; instead solution concentrations are calculated from absorbance measurements. Errors can arise from this practice if impurities in the sample absorb at the wavelengths used for these measurements. Also it is necessary to have accurate values for the extinction coefficients, $E^{1\%}_{1cm}$, used for calculating the concentrations.

This communication provides procedures that should enable different laboratories to make standard solutions of trans-retinol and β-carotene that are reasonably accurate and consistent. It also provides our recommendations on standard solutions for α-tocopherol.

RETINOL

We have found some commercially supplied crystalline synthetic retinol, even though packaged under an inert gas in amber vials, to be as much as 50% impure. By using high performance liquid chromatography (HPLC) on silica, with hexane containing 20% (v/v) diethyl ether as the mobile phase, solutions of retinol of good purity can be obtained.

The purification and preparation of a standard solution are carried out as follows: A 25 mg quantity of crystalline retinol from a freshly opened vial is dissolved in 500 μL of hexane. A turbid solution is usually obtained. After standing in the dark at room temperature for an hour to allow insoluble matter to settle, 20 μL of the solution is injected onto an analytical scale (4.6mm ID x 250mm long) silica HPLC column, and 1.2-1.3 mL of effluent from the center of the main peak is collected. (Repetitive injections can be made.) For spectrophotometry, 1.0 mL of the collected fraction is transferred to a 100 mL volumetric flask, and the flask is filled to the mark with the desired solvent (i.e., acetonitrile, etc.). An equal volume of the mobile phase, similarly diluted, is used as the blank for the determination. The retinol concentration is usually between 0.1-0.2 mg/100 mL.

We have found solutions made this way to be stable for several days when stored in the dark at room temperatures. Their absorbances change less than 1%, even without an anti-oxidant. If an anti-oxidant such as BHT does not interfere with the analysis, its addition to these solutions may be prudent.

We have used the above method for preparing solutions for comparative measurement, basing the comparison on the widely accepted value of 1850 dL g^{-1} cm^{-1} for retinol in absolute ethanol at 325 nm. The literature provides differing values for the extinction coefficients of retinol. We recommend the use of the extinction coefficients given in the table below:

Solvent	$E^{1\%}_{1cm}$ dL g^{-1} cm^{-1}
Acetonitrile	1832
Isopropanol	1841
Ethyl Acetate	1854
Ethanol	1850
Hexane*	1791

*Since it is known that the presence of polar solvents in hexane causes an increase in the retinol extinction coefficient, we measured the effect of the ether by making multiple additions to the solvent. Extrapolation of the results suggests that the extinction coefficient in a hexane solution containing no ether may be as low as 1750 dL g^{-1} cm^{-1}, but probably not as low as 1700.

β-CAROTENE

For a similar comparative study with β-carotene, we used "Type IV" β-carotene from SIGMA, which we found to be low in α-carotene content. Although such material is supplied in sealed vials, its purity can become quite low. We have found that β-carotene having nearly optimum specifications can be recovered by dissolving the impure material in toluene and precipitating with methanol. It is advisable to do this under subdued light.

The purification is carried out as follows. The contents of a 5 mg vial of the material are taken up in about 500 μL of toluene. Undissolved particles can be removed by centrifuging. The β-carotene is precipitated by adding about 8 mL of methanol and is isolated by centrifuging and removing the supernatant liquid. The β-carotene is washed twice by suspension in methanol and centrifuging. It then dried under vacuum at room temperature for 2 hours.

The purified β-carotene is readily soluble in hexane. However, its rate of dissolution in absolute ethanol is very slow. We therefore recommend that ethanol solutions of β-carotene be made by dissolving the purified material in toluene, i.e., dissolve the purified material in about 1 mL of toluene, transfer about 100 μL of the solution to a volumetric flask, and dilute to 100 mL with absolute ethanol. The solution will contain about 0.2 mg β-carotene/100 mL. β-Carotene is readily soluble in chloroform, but is not stable in that solvent. For example, the absorbance of chloroform solutions declines 10-100 times faster than ethanol solutions. We recommend that chloroform not be used as a solvent for standard solutions of β-carotene.

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On examining information in the literature on the extinction coefficient of β-carotene, we think the best available value is: $E^{1\%}_{1cm}$ 2592 dL g^{-1} cm^{-1} in hexane at 450 nm. We use this value for the extinction coefficient in hexane and, by comparative measurements, have found the extinction coefficient in absolute ethanol solution, made as described above, to be 2560 dL g^{-1} cm^{-1} at 452 nm. We recommend that all laboratories in the NBS/NCI QA program use these values.

α-TOCOPHEROL

We have found samples of α-tocopherol obtained from Eastman Kodak and the US Pharmacopoeial Convention to be reasonably pure. We recommend the use of these materials for standard solutions. It is a good idea to make a spectrophotometric check of the concentration of solutions made by weight. We recommend the use of 75.8 dL g^{-1} cm^{-1} (292 nm, in ethanol) as the value for the extinction coefficient.

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GRAPHICAL MATERIAL

Graphical presentations of individual laboratory results have been made as part of our evaluation of the NCI Round Robin (RR) data. The graphs are intended to allow you to see how your laboratory is doing and to make easy comparisons with the results from the other laboratories. Graphs have been made only for laboratories that measured all samples for a given analyte and RR. It is hoped that the graphs will help individual laboratories to discover any shifts, drifts, or biases that may be associated with their methods, and to take appropriate corrective actions. .

Each graph is for one laboratory and analyte, and consists of the laboratory's values plotted against a set of "consensus" values. The graph's ordinate and abscissa scales are made to be the same so that perfect agreement between the laboratory and the consensus values is represented by the dotted 45 degree line. The solid line of the graph represents the least-squares regression line associated with the individual laboratory's data from one RR. The equation for the line is given in the legend, and the points associated with the line are plotted as squares. The consensus values are taken from the data shown in the attached tables. The consensus values are probably slightly better than the overall averages from the RR since we have used a selected subset of the total population of measurements. In addition, for the vitamins we have excluded several of the laboratories' extreme data that are listed in the enclosed tables.

The graphs for all laboratories that fully participated in RR VIII are enclosed. The samples were all new. The vitamin samples were all freeze dried, and the Se and Zn samples were liquid sera.

The graphs that use your laboratory's RR VII data for the l.s. line are also enclosed and may contain points associated with RR VI or V. These latter points sometimes show noticeable changes in the individual laboratory's measurements with time. In looking at your RR VII vitamin data in the graphs and in the enclosed tables you may wish to note that samples 33, 35, and 45 are freeze dried versions of the respective liquid samples 44, 34, and 46. For the vitamins, RR VII liquid sample 32 is the same as RR V liquid sample 21.

For Se and Zn, all samples have been liquid sera. The Se and Zn RR VII samples 47 and 48 are the same as RR VI samples 22 and 23, respectively. The Se and Zn RR VI samples 25-27 were treated to have the T3 and T4 components removed.

Laboratories that show relatively large deviations from the 45 degree line or that show relatively large shifts between round robins should examine their procedures and instruments. Several of the laboratories definitely need improvement. We hope that the graphs serve as a useful diagnostic tool.

Robert C. Paule
Statistician

Fillmer C. Ruegg
Electronic Engineer

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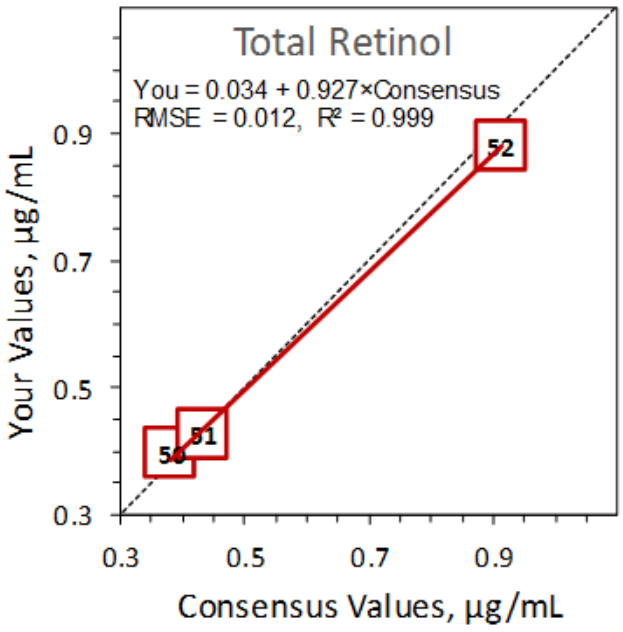
Round Robin VIII: retinol

Lab	Intercept	Slope	S fit	Sera 050 Mean	051 Mean	052 Mean
	0.0153	0.8933	0.0334	0.334	0.425	0.827
	0.0044	1.0097	0.0002	0.390	0.440	0.925
	0.0234	1.0307	0.0038	0.420	0.466	0.963
	-0.0450	0.9469	0.0022	0.318	0.362	0.818
	-0.0546	1.1759	0.0196	0.408	0.438	1.019
	-0.0049	0.4346	0.0911	0.100	0.250	0.385
	0.0287	0.8761	0.0003	0.363	0.407	0.827
	0.0034	0.9304	0.0027	0.360	0.403	0.852
	0.0339	0.9270	0.0122	0.396	0.425	0.880
	0.0415	0.8958	0.0171	0.395	0.416	0.859
	0.0390	0.9436	0.0085	0.405	0.440	0.900
	-0.0475	1.1318	0.0081	0.390	0.435	0.985
	0.0632	0.8503	0.0002	0.388	0.430	0.838
	-0.0176	1.3145	0.0139	0.475	0.560	1.180
	0.0227	0.8242	0.0114	0.345	0.370	0.775
	-0.0336	1.1598	0.0162	0.420	0.455	1.025
	-0.0334	1.1767	0.0061	0.420	0.470	1.040
	-0.0334	1.0841	0.0007	0.380	0.435	0.955
	-0.0070	1.8206	0.0998	0.755	0.705	1.660
	-0.0523	1.3294	0.0026	0.457	0.519	1.160
	0.1305	0.3084	0.0191	0.261	0.250	0.413
	-0.0807	1.3024	0.0995	0.350	0.555	1.100
	0.0116	0.8661	0.0100	0.349	0.378	0.802
	-0.0193	1.0213	0.0088	0.376	0.415	0.912
Average				0.386	0.435	0.921
Consensus values used				0.382	0.432	0.912



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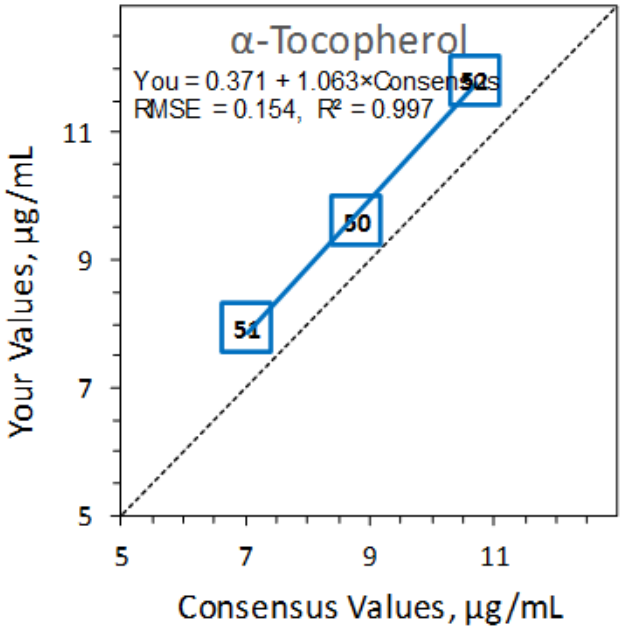
Round Robin VIII: a-tocopherol

Lab	Intercept	Slope	S fit	Sera 051 Mean	050 Mean	052 Mean
	0.5798	0.7883	0.0150	6.120	7.530	9.005
	0.6674	0.9652	0.1142	7.410	9.255	10.945
	0.2821	0.8713	0.0541	6.390	7.995	9.580
	0.1904	0.9394	0.0831	6.835	8.390	10.270
	0.3711	1.0629	0.1542	7.915	9.600	11.800
	0.6464	0.8899	0.2185	7.000	8.300	10.250
	-0.3100	1.1639	0.6532	8.155	9.400	12.395
	1.7365	0.8727	0.0522	7.855	9.460	11.050
	1.3202	1.5185	0.2261	12.100	14.500	17.650
	4.9885	0.6658	6.5501	6.905	16.195	9.530
	0.5628	0.8204	0.4141	6.510	7.445	9.500
	0.4237	1.0142	0.2140	7.650	9.175	11.355
	-0.5889	0.9868	0.2031	6.440	7.930	10.045
	0.6069	0.9038	0.2463	7.070	8.360	10.370
	-2.7738	1.7798	1.2985	9.200	13.950	15.750
	1.1931	0.9785	0.3608	8.230	9.510	11.800
	-3.9469	1.1401	0.9707	3.665	6.880	7.865
	0.2331	0.8402	0.0504	6.166	7.586	9.239
	0.1221	0.9831	0.0927	7.000	8.850	10.600
Average				7.296	9.490	11.000
Consensus values used				7.036	8.801	10.695



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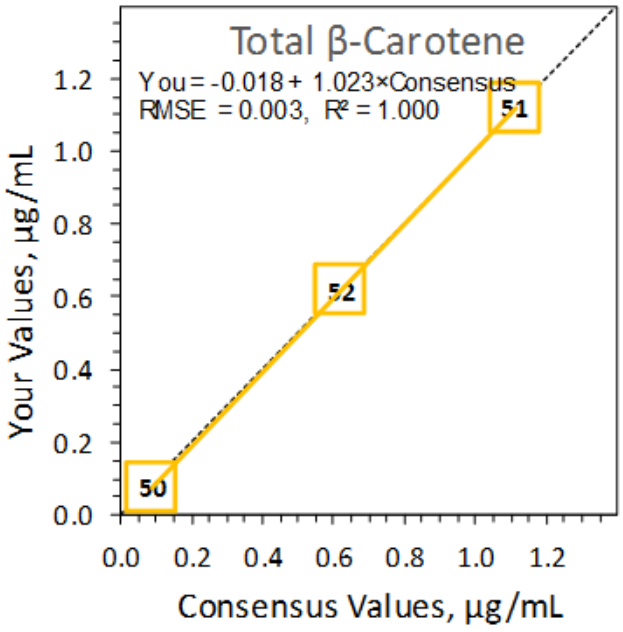
Round Robin VIII: b-carotene

Lab	Intercept	Slope	S fit	Sera	050 Mean	052 Mean	051 Mean
	0.0170	0.8095	0.0031		0.090	0.518	0.918
	-0.0122	0.8698	0.0116		0.070	0.520	0.960
	-0.0446	1.2464	0.0521		0.087	0.689	1.363
	-0.0059	0.5941	0.0440		0.030	0.400	0.636
	0.0013	0.9529	0.0139		0.081	0.606	1.055
	-0.0027	1.0384	0.0011		0.090	0.643	1.152
	-0.0186	1.0232	0.0031		0.074	0.616	1.120
	-0.0128	1.8850	0.0625		0.180	1.110	2.110
	0.0565	0.7969	0.0273		0.117	0.575	0.931
	-0.0840	1.9437	0.1316		0.141	1.019	2.133
	-0.0033	0.9593	0.0317		0.070	0.620	1.050
	0.0681	0.7816	0.0182		0.145	0.540	0.945
	0.0271	1.0330	0.0137		0.114	0.681	1.170
	0.0102	0.9453	0.0504		0.075	0.640	1.040
	-0.0366	1.3294	0.0535		0.103	0.747	1.464
	-0.0116	0.8433	0.0007		0.064	0.513	0.926
	0.0171	1.1534	0.0126		0.125	0.725	1.305
Average					0.097	0.657	1.193
Consensus values used					0.089	0.623	1.112



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Appendix F. Updated "All-Lab Report" for RR08

The following three pages are an updated version of an "All-Lab" report for RR08. This report has three parts:

- Page 1 lists results for all analytes.
- Page 2 provides a legend for page 1.
- Page 3 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 RR08. 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.

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Round Robin VIII Laboratory Results

	Retinol, µg/mL			α-Tocopherol, µg/mL			Total β-Carotene, µg/mL		
Lab	50	51	52	50	51	52	50	51	52
FSV-BA							0.103	1.475	0.748
FSV-BD	0.395	0.416	0.860	7.55	6.65	8.80			
FSV-BE	0.420	0.470	1.040	7.93	6.44	10.05			
FSV-BF	0.475	0.560	1.180	14.50	12.10	17.65	0.141	2.134	1.019
FSV-BG	0.420	0.466	0.964				0.087	1.364	0.689
FSV-BH	0.377	0.415	0.913	8.85	7.00	10.60	0.125	1.305	0.725
FSV-BI	0.390	0.440	0.925	7.53	6.12	9.01	0.070	0.960	0.520
FSV-BY	0.334	0.426	0.828				0.091	0.919	0.519
FSV-CA	0.363	0.407	0.828	8.00	6.39	9.58			
FSV-CJ	0.408	0.439	1.019	9.26	7.41	10.95	0.081	1.055	0.606
FSV-CK	0.395	0.484	0.954	9.39	8.12	12.51	0.068	0.937	0.502
FSV-CL	0.388	0.430	0.839	9.46	7.86	11.05	0.117	0.931	0.575
FSV-CN	0.405	0.440	0.900	8.30	7.00	10.25	0.180	2.110	1.110
FSV-CO	0.361	0.403	0.852	8.39	6.84	10.27	0.091	1.153	0.643
FSV-DC	0.350	0.555	1.100				0.075	1.040	0.640
FSV-DE				9.18	7.65	11.36			
FSV-DG	0.380	0.435	0.955	8.36	7.07	10.37	0.070	1.050	0.620
FSV-DH	0.390	0.435	0.985	9.40	8.16	12.40			
FSV-DN	0.313	0.338	0.459	7.95	7.25	9.20	0.042	0.800	0.385
FSV-DO	0.100	0.250	0.385						
FSV-DT	0.345	0.370	0.775	9.60	7.72	11.30			
FSV-DZ	1.052	1.059	1.925	7.81	5.32	8.61	0.103	1.465	0.748
FSV-ED	0.349	0.378	0.802	7.59	6.17	9.24	0.064	0.927	0.513
FSV-EG	0.318	0.363	0.819				0.030	0.636	0.400
FSV-EN	0.261	0.250	0.413	6.88	3.67	7.87			
FSV-EP	0.420	0.455	1.025	7.45	6.51	9.50			
FSV-ER	0.396	0.425	0.880	9.60	7.92	11.80	0.074	1.121	0.616
FSV-EU	0.330	0.315	0.705	5.60	4.30	6.05	0.145	0.945	0.540
FSV-EW	0.457	0.520	1.160	9.51	8.23	11.80	0.114	1.170	0.682
n	27	27	27	23	23	23	20	20	20
Min	0.100	0.250	0.385	5.60	3.67	6.05	0.030	0.636	0.385
Median	0.388	0.430	0.900	8.36	7.00	10.27	0.089	1.053	0.618
Max	1.052	1.059	1.925	14.50	12.10	17.65	0.180	2.134	1.110
eSD	0.047	0.053	0.126	1.23	1.07	1.59	0.029	0.183	0.146
eCV	12	12	14	15	15	15	33	17	24
NISTa	0.374	0.446	0.918	9.02	7.56	10.89	0.063	1.050	0.521
NISTb	0.386	0.461	0.994	8.84	7.05	10.88	0.068	0.998	0.571
NISTc	0.386	0.427	0.909	8.47	6.74	10.08	0.071	1.057	0.615
NAV	0.373	0.431	0.911	8.51	6.94	10.27	0.068	1.010	0.557
NAU	0.047	0.053	0.126	1.23	1.07	1.59	0.029	0.183	0.146

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Round Robin VIII 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 heterogeneity and inter- and intra-laboratory) standard deviation

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Round Robin VIII Laboratory Results

Comparability Summary

Lab	TR	aT	bC
FSV-BA			2
FSV-BD	1	1	
FSV-BE	1	1	
FSV-BF	3	4	4
FSV-BG	1		2
FSV-BH	1	1	2
FSV-BI	1	1	1
FSV-BY	1		1
FSV-CA	1	1	
FSV-CJ	1	1	1
FSV-CK	1	2	1
FSV-CL	1	1	1
FSV-CN	1	1	4
FSV-CO	1	1	1
FSV-DC	2		1
FSV-DE		1	
FSV-DG	1	1	1
FSV-DH	1	2	
FSV-DN	3	1	2
FSV-DO	4		
FSV-DT	2	1	
FSV-DZ	4	2	2
FSV-ED	1	1	1
FSV-EG	2		2
FSV-EN	4	3	
FSV-EP	1	1	
FSV-ER	1	1	1
FSV-EU	2	3	2
FSV-EW	2	2	1
NISTa	1	1	1
NISTb	1	1	1
NISTc	1	1	1
n	30	26	23

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	63	69	57	68.2 %
% 2	17	15	30	27.3 %
% 3	7	8	0	4.3 %
% 4	10	4	9	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.

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