

NISTIR 7880-34

NIST Micronutrients Measurement Quality Assurance Program Winter, Spring, and Fall 1990 Comparability Studies

**Results for Round Robins XVIII, XIX, and XX
Fat-Soluble Vitamins and Carotenoids in Human Serum**

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Chemical Sciences Division, Materials Measurement Laboratory

Margaret C. Kline

Biomolecular Measurement Division, Materials Measurement Laboratory

Katherine E. Sharpless

Office of Special Programs

Wille E. May

Under Secretary of Commerce for Standards and Technology and Director

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June 2015



U.S. Department of Commerce
Penny Pritzker, Secretary

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

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Abstract

The National Institute of Standards and Technology coordinates the Micronutrients Measurement Quality Assurance Program (MMQAP) for laboratories that measure fat- and water-soluble vitamins and carotenoids in human serum and plasma. This report describes the design of and results for the Winter, Spring and Fall 1990 MMQAP measurement comparability improvement studies: 1) Round Robin XVIII Fat-Soluble Vitamins and Carotenoids in Human Serum, 2) Round Robin XIX Fat-Soluble Vitamins and Carotenoids in Human Serum, and 3) Round Robin XX Fat-Soluble Vitamins and Carotenoids in Human Serum. The materials for Round Robin XVIII were shipped to participants in January 1990; participants were requested to provide their measurement results by March 5, 1990. The materials for Round Robin XIX were shipped to participants in May 1990; participants were requested to provide their measurement results by July 6, 1990. The materials for Round Robin XX were shipped to participants in August 1990; participants were requested to provide their measurement results by October 22, 1990.

Keywords

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

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Introduction

Beginning in 1988, the National Institute of Standards and Technology (NIST) has coordinated the Micronutrients Measurement Quality Assurance Program (MMQAP) for laboratories that measure fat- and water-soluble vitamins and carotenoids in human serum and plasma. The MMQAP provides participants with measurement comparability assessment through use of interlaboratory studies, Standard Reference Materials (SRMs) and control materials, and methods development and validation. Serum-based samples with assigned values for the target analytes (retinol, alpha-tocopherol, gamma/beta-tocopherol, *trans*- and total beta-carotene, and ascorbic acid) and performance-evaluation standards are distributed by NIST to laboratories for analysis.

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

All MMQAP interlaboratory studies consist of individual units of batch-prepared samples that are distributed to each participant. For historical reasons these studies are referred to as "Round Robins". The MMQAP program and the nature of its studies are described elsewhere. [1]

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

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin XVIII comparability study (hereafter referred to as RR18) received three lyophilized human serum test samples for analysis. Unless multiple vials were previously requested, participants received one vial of each serum. These sera were shipped on dry ice to participants in January 1990. The communication materials included in the sample shipment are described in Appendix A.

Participants are requested to report values for all fat-soluble vitamin-related analytes that are of interest to their organizations. Not all participants report values for the target analytes, and some participants report values for non-target analytes.

The final report delivered to every participant in RR18 is reproduced as Appendix B. This report included:

- Our analysis of the participants' results.
- Tabular presentations of all participant results.
- Graphical presentations of interlaboratory precision over time. These Figures are no longer available.

Each participant also received an "Individualized Report" that graphed their results for selected analytes. These figures are no longer available.

Appendix C lists all of the measurement results reported for RR18 in a more accessible format.

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

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin XIX comparability study (hereafter referred to as RR19) received three lyophilized human serum test samples for analysis. Unless multiple vials were previously requested, participants received one vial of each material. These sample materials were shipped on dry ice to participants in May 1990. The communication materials included in the sample shipment are described in Appendix D.

Participants are requested to report values for all fat-soluble vitamin-related analytes that are of interest to their organizations. Not all participants report values for the target analytes, and some participants report values for non-target analytes.

The final report delivered to every participant in RR19 is reproduced as Appendix E. This report included:

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

Each participant also received an "Individualized Report" that graphed their results for selected analytes. These figures are no longer available.

Appendix F lists all of the measurement results reported for RR19 in a more accessible format.

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

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin XX comparability study (hereafter referred to as RR20) received two liquid-frozen and three lyophilized human sera. Unless multiple vials were previously requested, participants received one vial of each material. These sample materials were shipped on dry ice to participants in August 1990. The communication materials included in the sample shipment are described in Appendix G.

Participants are requested to report values for all fat-soluble vitamin-related analytes that are of interest to their organizations. Not all participants report values for the target analytes, and some participants report values for non-target analytes.

It is probable that a preliminary report for RR20 was mailed to all participants shortly before the NIST/NCI Micronutrients Analysis Workshop held on November 15, 1990. However, no version of either the letter or the preliminary report is available. Likewise, only the tabular parts of the Final Report for RR20 are available; these tables are reproduced as Appendix H.

It is probable that each participant also received an "Individualized Report" that graphically analyzes their results for selected analytes. These figures are no longer available.

Appendix I lists all of the measurement results reported for RR20 in a more accessible format.

References

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

Appendix A. Shipping Package Inserts for RR18

Two items were included in each package shipped to an RR18 participant:

- **Cover letter.** The original letter has been lost. It would have described the three lyophilized sample materials (sera 120 to 122), ethanolic solution (sample 123), and three ethanolic calibration solutions distributed for the study, given guidance on reconstituting the serum samples and use of the calibration solutions, stated that results were due March 5, 1990 and to whom they should be sent, and who to contact with technical questions.

While there is no record of the cover letter, page A2 reproduces a letter sent at the same time as the Final Report for Round Robin XVII (RR17) that describes the RR18 materials. This notification letter was sent to laboratories that had not reported results for RR17; similar descriptions were included in the RR17 Final Reports provided to laboratories that had participated in RR17. These letters were sent about two weeks before the packages were to be shipped.

- **Datasheets.** Pages A3 and A4 reproduce the form. The first page of the form is for reporting the primary-focus analytes: retinol, α -tocopherol, and total and *trans*- β -carotene. The second page is for reporting three “optional” analytes: retinyl palmitate, γ -tocopherol, and lycopene. In the absence of other information, the lycopene results are assumed to report total lycopene.

These items were attached to the shipping box.

January 4, 1990

^F1^

^F2^

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

HAPPY NEW YEAR!

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

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

	(μg/mL)		
	<u>RETINOL</u>	<u>α-TOCOPHEROL</u>	<u>TRANS β-CAROTENE</u>
Solution 10	0.195	2.22	0.135
Solution 11	0.977	11.1	0.945
Solution 12	1.95	22.2	1.62

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

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

Sincerely,

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

cc: Robert Schaffer
Winfred Malone

REPORT ON NIST/NCI SAMPLES FROM LABORATORY # _____			
DATE OF ANALYSIS _____			
RESULTS IN $\mu\text{g/mL}$			
SAMPLE #	ANALYTE		
SERUM 120	RETINOL		
		Trans	Total
VIAL # ____	B-CAROTENE		
	A-TOCOPHEROL		
SERUM 121	RETINOL		
		Trans	Total
VIAL # ____	B-CAROTENE		
	A-TOCOPHEROL		
SERUM 122	RETINOL		
		Trans	Total
VIAL # ____	B-CAROTENE		
	A-TOCOPHEROL		
SERUM 123 (EtOH Sol)	RETINOL		
		Trans	Total
VIAL # ____	B-CAROTENE		
	A-TOCOPHEROL		

Add 1.0 mL of Distilled water to the Serum Samples.

REPORT ON NIST/NCI SAMPLES FROM LABORATORY # _____
 OPTIONAL ANALYTES
 DATE OF ANALYSIS _____

RESULTS IN µg/mL

SAMPLE #	ANALYTE
SERUM 120	RETINYL PALMITATE
VIAL # ____	LYCOPENE
	GAMMA-TOCOPHEROL
SERUM 121	RETINYL PALMITATE
VIAL # ____	LYCOPENE
	GAMMA-TOCOPHEROL
SERUM 122	RETINYL PALMITATE
VIAL # ____	LYCOPENE
	GAMMA-TOCOPHEROL

Appendix B. Final Report for RR18

The following six pages are the available components of the final report for RR18 as provided to all participants. The original report consisted of:

- A cover letter and discussion.
- Tables 1 to 3 that list results and various summary values for retinol, α -tocopherol, and total β -carotene.
- Four unnumbered tables that list results and simple summary statistics for retinyl palmitate, lycopene, γ -tocopherol, and *trans*- β -carotene. Note: the cover letter states that the first three of these analytes constitute “Table 5”; the *trans*- β -carotene values were doubtless intended to be “Table 4.”
- Graphical presentations of interlaboratory precision over time for retinol, α -tocopherol, and total β -carotene.

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

The graphical presentations for RR18 are no longer available; however, they would have been very similar to the graphs in the Final Report for RR19 in Appendix E.

Due to the complex formatting used in the Tables, the originally listed laboratory codes have been deleted without replacement. However, Appendix C provides a complete listing of the RR18 results where the original codes have been altered to ensure confidentiality. Appendix C also provides more relevant summary statistics.

May 18, 1990

Dear Colleague:

Please pardon our delay in providing you with the results from Round-Robin XVIII. Tables 1-4 provide the usual compilation of individual laboratory results and a summary of the data for the determination of serum/plasma levels of retinol, α -tocopherol, total β -carotene, and trans β -carotene, respectively. Figures 1-3 provide an overall picture of the interlaboratory measurement capability (with respect to precision) for retinol, α -tocopherol, and β -carotene over the past four years. Table 5 provides data compilations and summaries for serum/plasma determinations of three new analytes: retinyl palmitate, γ -tocopherol, and lycopene. As one might expect, interlaboratory variability is much greater (2-3 times) for the three new analytes than for our three core analytes.

As usual, the right side of Tables 1-3 provides data for use in evaluating your lab's performance in the study. The bias values given are based on the interlaboratory grand mean values for each analyte. The NIST value, based on measurements at NIST using one method only, is provided also for your information but not considered in computing the grand means. For retinol and α -tocopherol, the relatively close agreement between the NIST and the grand mean values allows us to continue the convention of 0-5% bias representing EXCEPTIONAL performance, 6-10% ACCEPTABLE performance, 11-20% MARGINAL performance, and >20% UNACCEPTABLE with respect to the current state-of-the-practice for measurement of these analytes in serum/plasma.

Data reported for β -carotene is much less precise, and the biases between the grand mean and the NIST values are relatively large, +19, -41, and -19% for serum samples 120, 121, and 122 respectively. The respective biases for retinol were 6, 7, and 7% and for α -tocopherol +3, +7, and -4%. The reduced precision and apparent bias associated with the serum β -carotene data make individual laboratory performance difficult to evaluate. We hope that these data are anomalous and that β -carotene results from RR-XIX will be more in line with those from recent studies. If the imprecision continues and bias between the grand means and NIST values remains, round-robin studies and NIST laboratory studies will be designed to address the problem.

For the third time, results from the solution experiment perplex us. For all three analytes, the data from the analysis of the solution are more scattered than those for the three serum samples. For β -carotene this is understandable. First, after conversations with many of you concerning the non-linearity of the response from the three calibration solutions the we supplied to you, we observed that β -carotene does not completely redissolve in calibration solutions 11 and 12 by simply allowing them to remain at room temperature for 4 hours as you were instructed to do; instead 12 hours at room temperature or ultrasonic agitation for 10 minutes is necessary. Second, we observed β -carotene degradation products in several vials of the unknown solution that you were asked to analyze. From our analyses of 12 randomly selected vials, we obtained eight distinctly different values for β -carotene, but identical values for retinol and α -tocopherol. We found that the integrity and solubility of retinol and α -tocopherol in both the calibration solutions and the unknown solutions to be good. In summary, scatter in the solution data for β -carotene is understandable due both to problems associated with dissolution of β -carotene in the calibration solutions provided

to you and what appears to be degradation of β -carotene in the unknown solution. Reasons for the scatter observed in the solution data for retinol and α -tocopherol are not apparent.

As you probably recall, the solution experiments were designed to serve two purposes: (1) to determine the relative contributions of solvent extraction/solvent exchange and chromatographic analysis to the overall 6-10% CV for retinol and α -tocopherol analyses and (2) to evaluate the need for you to modify your chromatographic systems in order to obtain values for individual β -carotenoid isomer determinations. Since the precision obtained from the determination of retinol and α -tocopherol in ethanolic solutions in this and previous round-robins has curiously been worse than for serum analyses, it is certainly not possible to isolate the relative contributions of sample preparation and chromatographic analyses to the total variance. Therefore, until a better experiment can be designed to isolate these relative contributions, CV's of roughly 6-7% appear to be the limit for interlaboratory serum determinations of retinol and α -tocopherol. The integrity of both the calibration solutions and unknown sample preclude any substantive evaluation of β -carotene data from sample 123. Problems associated with total- and β -carotene isomer determination will continue to be a major focus in these studies.

Results are due back to NIST July 6.

Sincerely,

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

Table 1. Round Robin XVIII Results

Retinol Lab #	Serum # 120	Serum # 121	Serum # 122	Solution 123	Retinol % Serum # 120	Bias from Serum # 121	Grand Average Serum # 122	Solution 123
	0.240	0.493	0.897	0.525	-7.2	1.7	5.7	2.3
	0.240	0.470	0.812	0.568	-7.2	-3.0	-4.3	10.7
	0.270	0.508	0.954	0.561	4.4	4.8	12.4	9.4
	0.216	0.503	0.904	0.514	-16.5	3.8	6.5	0.2
	0.223	0.420	0.778	0.570	-13.8	-13.3	-8.3	11.1
	0.255	0.467	0.780		-1.6	-3.7	-8.1	
	0.270	0.520	0.849	0.488	4.4	7.3	0.1	-4.9
	0.314	0.477	0.819		21.4	-1.6	-3.5	
	0.260	0.476	0.818	0.444	0.5	-1.8	-3.6	-13.5
	0.252	0.462	0.852	0.517	-2.6	4.7	0.4	0.8
	0.259	0.421	0.689	0.497	0.1	-4.1	-18.8	-3.1
	0.270	0.499	0.753	0.544	4.4	3.0	-11.3	6.0
	0.270	0.500	0.840	0.390	4.4	3.2	-1.0	-24.0
	0.282	0.524	0.896	0.593	9.5	8.1	-5.6	-15.6
	0.278	0.514	0.856	0.445	7.1	6.1	0.9	-13.3
	0.339	0.699	0.866	0.562	31.1	44.2	2.1	9.6
	*0.258	*0.510	0.852	0.440	-0.3	5.8	0.7	-14.9
	*0.327	*0.823	*1.092	*0.574	-15.0	69.2	28.2	-11.2
	0.220	0.450	0.770	0.560	8.6	-7.7	0.7	-25.9
	0.281	0.498	0.854	0.380	-11.1	17.6	118.0	-17.0
	0.230	0.570	1.850	0.600	-1.1	-13.6	-9.6	-8.6
	0.230	0.437	0.767	0.474	2.4	-19.8	-5.2	-1.6
	0.265	0.437	0.804	0.557	-4.1	1.9	-2.2	-0.2
	0.248	0.494	0.867	0.505	6.1	3.4	-3.9	20.9
	0.243	0.501	0.815	0.512	-0.5	1.1	-0.1	1.4
	0.260	0.490	0.848	0.620	12.1	7.3	21.4	-9.7
	0.290	0.520	1.030	0.520	-5.7	4.0	3.6	3.3
	0.244	0.504	1.030	0.463				
	0.269	0.473	0.879	0.530				
	0.275	0.521	0.905	0.501				
NIST								
Avg	0.259	0.485	0.848	0.513				
SD	0.023	0.030	0.080	0.062				
RSD	9.0	6.2	9.5	12.1				

* = Value not included in statistical analysis.

L = Late, Results not included in Statistical Analysis.

NIST value not included in Statistical Analysis.

Table 3. Round Robin XVIII Results

Table 3. Round Robin XVIII Results														
Total Beta-Carotene														
Lab #	Serum # 120	Serum # 121	Serum # 122	Solution # 123	Total Beta-Carotene & Bias from Grand Average					Solution 123				
	0.386	0.990	1.950	1.363	Serum # 120	Serum # 121	Serum # 122							
	0.357	0.874	1.539	1.102	-5.5	-4.6	-1.3		28.0					
	0.417	1.049	2.145	1.213	-12.0	-15.8	-22.1		3.5					
	0.392	1.071	2.100	0.630	-4.1	1.1	8.6		13.9					
	0.457	1.126	2.155	0.925	11.7	3.2	6.3		-40.9					
	0.357	1.042	1.332	0.566	-12.6	0.4	9.0		-13.2					
	0.470	1.092	2.446	1.367	15.0	5.2	-32.8		-46.9					
	0.292	0.847	1.651	0.930	-28.5	-18.4	-16.4		-28.3					
	0.440	1.130	1.690	1.629	7.6	-8.9	-14.5		-12.7					
	0.345	0.948	1.942	0.420	-15.3	-8.7	15.4		-39.1					
	0.455	1.170	2.280	*0.420	11.0	12.7	-8.8		-60.6					
	0.384	*0.437	*1.802	*1.390	15.4	17.9	-16.3		30.5					
	*0.472	1.020	*1.644	1.200	-4.6	-17.8	-2.6		12.7					
	0.390	1.430	1.930		-10.4	37.8	36.8		-38.0					
	0.407	0.831	2.700	0.660	10.4	-19.9	-1.8		47.4					
	0.457	1.010	1.940	1.570	11.8	-2.7	8.3		13.6					
	0.450	1.120	2.060	1.210	10.1	7.9	4.3		-47.6					
	0.480	1.069	2.485	0.558	17.5	3.0	25.8		-49.9					
	0.394	1.050	1.760	0.534	-3.6	1.2	-10.9							
	0.447	0.664	1.420	0.499	9.4	-36.0	-28.1							
	0.487	0.626	1.600											
NIST'														
Avg	0.409	1.038	1.976	1.065										
SD	0.053	0.090	0.319	0.364										
RSD	13.1	8.6	16.2	34.2										

* = Value not included in statistical analysis.

L = Late, Results not included in Statistical Analysis.

NIST value not included in Statistical Analysis.

Retinyl Palmitate

Lab #	Serum # 120	Serum # 121	Serum # 122
NIST	ND	ND	ND
	ND	ND	ND
	ND	ND	ND
	0.051	0.136	0.268
	0.025	0.030	0.037
	0.002	0.002	0.005
	ND	ND	ND

TSIN

B7

Lycopene

Lab #	Serum # 120	Serum # 121	Serum # 122
	0.383	0.495	0.166
	0.210	0.270	0.110
	1.861	2.352	0.689
	0.239	0.305	0.114
	0.060	0.077	0.037
	0.186	0.241	0.088
	0.267	0.106	0.054
	0.180	0.200	0.050
	0.456	0.505	0.176
	0.260	0.340	0.110
Avg	0.273	0.308	0.108
SSD	0.098	0.138	0.046
RSD	36	45	42

* = Value not used in statistical analysis.

Gamma Tocopherol

Lab #	Serum # 120	Serum # 121	Serum # 122
	2.12	0.95	0.69
	2.20	0.93	0.82
		1.12	* 3.57
	2.32	1.13	0.85
	3.47	1.53	* 9.50
	2.24	0.92	0.72
	2.56	1.62	0.99
	* 2.34	* 0.47	* 0.24
	1.67	0.77	0.62
	2.59	1.55	2.02
	2.52	1.13	1.06
NIST	2.24	0.89	0.70

NIST

Avg	2.410	1.164	0.846
SD	0.488	0.300	0.164
RSD	20	26	19

* = Value not used in statistical analysis.

NIST Value not used in statistical analysis.

Trans Beta-Carotene				
Lab #	Serum # 120	Serum # 121	Serum # 122	Solution # 123
NIST	0.413	1.090	2.140	0.274
	0.361	1.005	1.696	0.227
	0.463	0.407	1.536	0.410
				0.231
	0.409	0.526	1.380	0.199
Avg	0.412	0.834	1.791	0.286
	0.051	0.372	0.313	0.086
	12.4	44.6	17.5	30.0

Appendix C. Updated “All-Lab Report” for RR18

The following four pages are a revised version of an “All-Lab” report for RR18. This report has three parts:

- pages 1 and 2 list results for all analytes reported at least twice, counting both participants and NIST analysts.
- page 3 provides a legend for pages 1 and 2.
- page 4 summarizes each participants’ performance for retinol, α -tocopherol, and total β -carotene. These summaries are compatible with the percent bias evaluation advice given in the RR18 Report. However, the current bias summaries are estimated relative to the median of all reported values for each analyte in each serum rather than to the “Trimmed Average” used in the original and detailed in Appendix B. These original reference values were estimated from on-time results, with subjective exclusion of results deemed non-representative.

To ensure confidentiality, the laboratory identifiers used in this “All-Lab Report” have been altered from those used in RR18. The only attributed results are those reported by NIST. The NIST results are not used in the assessment of the consensus summary results of the study.

Note: The results designated NISTa in this updated All-Lab report are designated as NIST in the Tables of Appendix B.

Round Robin XVIII Laboratory Results

Lab	Total Retinol				Retinyl Palmitate				α -Tocopherol				γ/β -Tocopherol			
	120	121	122	123	120	121	122	123	120	121	122	123	120	121	122	123
FSV-BA	0.278	0.514	0.856	0.445	0.025	0.030	0.037		5.22	7.09	9.24	8.1				
FSV-BD	0.270	0.520	0.849	0.488					4.90	7.60	10.10	8.5				
FSV-BE	0.270	0.499	0.753	0.544					5.00	6.50	9.20	9.0				
FSV-BF	0.260	0.476	0.818	0.444					4.72	6.36	9.53	8.1	2.3	1.13	0.85	
FSV-BG	0.270	0.508	0.954						4.69	6.58	10.65	9.7				
FSV-BH	0.258	0.510	0.852	0.440	<0.02	<0.02	<0.02		4.51	6.63	9.31	8.0	2.2	0.95	0.72	
FSV-BI	0.240	0.470	0.812	0.546	nd	nd	nd		4.48	6.27	8.66	9.9	2.2	0.93	0.82	
FSV-BJ	0.243	0.501	0.815	0.512					4.75	7.00	10.85	10.4	2.5	1.13	1.06	
FSV-BL	0.267	0.482	0.798	0.505					4.96	6.38	8.71	9.1				
FSV-BM	0.248	0.494	0.867	0.505					4.90	7.10	10.50	10.1				
FSV-BY	0.240	0.493	0.897	0.525	nd	nd	nd		4.71	6.53	9.99	8.0	2.1	0.95	0.69	
FSV-BZ									5.50	6.69	9.42	7.4				
FSV-CA	0.223	0.420	0.778	0.570					4.56	6.07	8.78	11.3				
FSV-CJ	0.216	0.503	0.904	0.514					5.09	7.45	10.11	8.7				
FSV-CK	0.327	0.823	1.092	0.574					5.10	10.49	11.97	11.4	2.6	1.62	0.99	
FSV-CM				0.480					4.71	6.68	9.74	9.8				
FSV-CN	0.314	0.477	0.819						5.23	6.72	8.76					
FSV-CO	0.255	0.467	0.780		nd	nd	nd		4.75	6.57	9.14					
FSV-CQ	0.252	0.462	0.852	0.517	0.051	0.136	0.268		na	7.00	9.50	9.3	3.5	1.53	9.50	
FSV-CV	0.260	0.490	0.848	0.620					4.90	6.45	12.03	10.9				
FSV-CY	0.220	0.450	0.770	0.560					5.17	6.82	9.04	10.4	1.3	0.47	0.24	
FSV-DC	0.282	0.544	0.896	0.593												
FSV-DE									4.88	6.68	9.34	9.5	2.2	0.92	0.72	nd
FSV-DG	0.270	0.500	0.840	0.390					4.39	7.05	9.08	10.0				
FSV-DH	0.230	0.419	0.767	0.474	0.058	0.108	0.226		4.02	5.12	7.69	8.4	2.0	0.73	0.74	
FSV-DL	0.279	0.493	0.825	0.530												
FSV-DT	0.259	0.421	0.689	0.497					4.98	6.23	9.10	8.5				
FSV-DY	0.230	0.570	1.850	0.600												
FSV-DZ	0.339	0.699	0.866	0.562					4.42		6.81	9.7				
FSV-EB	0.244	0.504	1.020	0.463					4.24	7.14	10.30	8.3				
FSV-EF	0.281	0.498	0.854	0.380					4.63	6.60	9.20	8.4	1.7	0.77	0.62	
FSV-EV	0.290	0.520	1.030	0.520					4.85	6.00	9.70	8.7				
FSV-FA	0.265	0.437	0.804	0.557	0.002	0.002	0.005		4.72	5.86	8.64	7.4	2.6	1.55	1.02	
n	30	30	30	28	4	4	4	0	29	29	30	28	12	12	12	0
Min	0.216	0.419	0.689	0.380	0.002	0.002	0.005		4.02	5.12	6.81	7.39	1.34	0.47	0.24	
Mean	0.263	0.505	0.885	0.513	0.034	0.069	0.134		4.79	6.75	9.50	9.18	2.26	1.06	1.50	
Max	0.339	0.823	1.850	0.620	0.058	0.136	0.268		5.50	10.49	12.03	11.40	3.47	1.62	9.50	
SD	0.029	0.079	0.201	0.059	0.026	0.063	0.132		0.32	0.87	1.07	1.11	0.53	0.35	2.53	
CV	11	16	23	12	75	92	99		7	13	11	12	23	34	169	
NISTa	0.275	0.521	0.905		nd	nd	nd		4.99	7.16	9.21		2.2	0.89	0.70	
NISTc	0.244	0.433	0.726						4.47	6.18	8.27		2.5	1.43	1.25	
Median	0.260	0.496	0.849	0.516					4.75	6.63	9.33	9.05	2.22	0.95	0.78	
eSD	0.026	0.029	0.068	0.062					0.31	0.53	0.82	1.22	0.39	0.27	0.18	
eCV	10	6	8	12					7	8	9	13	18	28	24	

Round Robin XVIII Laboratory Results

Lab	Total β -Carotene				trans- β -Carotene				Total cis- β -Carotene				Total Lycopene			
	120	121	122	123	120	121	122	123	120	121	122	123	120	121	122	123
FSV-BA	0.455	1.17	2.28	0.65	0.413	1.090	2.140	0.274	0.042	0.080	0.140	0.375	0.19	0.24	0.088	
FSV-BD																
FSV-BE																
FSV-BF	0.470	1.09	2.45	0.57									0.24	0.31	0.114	
FSV-BG	0.417	1.05	2.15	1.10												
FSV-BH	0.384	1.06	1.80	0.42	0.361	1.005	1.696	0.227	0.023	0.051	0.106	0.193	0.39	0.55	0.154	
FSV-BI	0.357	0.87	1.54	1.14									0.21	0.27	0.110	
FSV-BJ	0.450	1.12	2.06	1.57									0.26	0.34	0.110	
FSV-BL																
FSV-BM																
FSV-BY	0.386	0.99	1.95										0.38	0.50	0.166	
FSV-BZ																
FSV-CA																
FSV-CJ	0.392	1.07	2.10	1.21												
FSV-CK	0.472	0.44	1.64	1.39	0.463	0.407	1.536	0.410	0.009	0.029	0.109	0.980	0.27	0.11	0.054	
FSV-CM																
FSV-CN	0.357	1.04	1.33	0.93												
FSV-CO	0.457	1.13	2.15	0.63												
FSV-CQ	0.292	0.85	1.65										0.06	0.08	0.037	
FSV-CV	0.480	1.07	2.49	1.21												
FSV-CY	0.390	1.02	1.93	1.20									0.18	0.20	0.050	
FSV-DC	0.345	0.95	1.95	1.63												
FSV-DE																
FSV-DG	0.440	1.13	1.69	0.93												
FSV-DH	0.407	0.83	1.94	0.66									0.37	0.40	0.177	
FSV-DL	0.451	1.50	1.49													
FSV-DT																
FSV-DY	0.365	1.43	2.70													
FSV-DZ																
FSV-EB	0.394	1.05	1.76	0.56												
FSV-EF																
FSV-EV																
FSV-FA	0.457	1.01	2.15					0.231					0.46	0.51	0.176	
n	21	21	21	16	3	3	3	4	3	3	3	3	11	11	11	0
Min	0.292	0.437	1.332	0.420	0.361	0.407	1.536	0.227	0.009	0.029	0.106	0.193	0.060	0.077	0.037	
Mean	0.410	1.041	1.962	0.987	0.412	0.834	1.791	0.286	0.025	0.053	0.118	0.516	0.273	0.317	0.112	
Max	0.480	1.495	2.700	1.629	0.463	1.090	2.140	0.410	0.042	0.080	0.140	0.980	0.456	0.548	0.177	
SD	0.050	0.211	0.348	0.377	0.051	0.372	0.313	0.086	0.017	0.025	0.019	0.412	0.117	0.159	0.051	
CV	12	20	18	38	12	45	17	30	69	48	16	80	43	50	46	
NISTa	0.487	0.63	1.60		0.409	0.526	1.383		0.078	0.100	0.213					
NISTc	0.448	0.98	1.71		0.369	0.853	1.530		0.080	0.124	0.175		0.21	0.22	0.127	
Median	0.407	1.050	1.950	1.016									0.260	0.305	0.110	
eSD	0.065	0.104	0.303	0.536									0.119	0.156	0.083	
eCV	16	10	16	53									46	51	75	

Round Robin XVIII Laboratory Results

Table Legend

Symbol	Interpretation
<i>na</i>	Not available (not reported by participant)
<i>nd</i>	Not detected (not reported or reported as '0', 'not determined', etc.)
<i>italics</i>	Value calculated from reported results
n	Number of non-NIST laboratories reporting quantitative results
Min	Minimum non-NIST reported value.
Mean	Average over all non-NIST reported values.
Max	Maximum non-NIST reported value.
SD	Standard deviation over all non-NIST values.
CV	Coefficient of Variation (% relative standard deviation): $100 \cdot \text{SD} / \text{Mean}$
Median	Median over all non-NIST reported values
eSD	Robust estimate of SD based on the adjusted median absolute difference from the median (MADe)
eCV	Robust estimate of CV, $100 \cdot \text{eSD} / \text{Median}$

Round Robin XVIII Laboratory Results

Lab	TR	aT	bC
FSV-BA	4±3	5±6	13±3
FSV-BD	3±3	9±6	
FSV-BE	-2±8	1±4	
FSV-BF	-3±2	-1±3	15±11
FSV-BG	6±5	4±9	4±5
FSV-BH	1±2	-2±3	-4±4
FSV-BI	-6±2	-6±1	-17±4
FSV-BJ	-3±4	7±8	8±3
FSV-BL	-2±4	-2±6	
FSV-BM	-1±3	8±5	
FSV-BY	-1±7	2±5	-4±3
FSV-BZ		6±9	
FSV-CA	-13±4	-6±2	
FSV-CJ	-3±12	9±3	2±6
FSV-CK	40±22	31±26	-19±37
FSV-CM		1±3	
FSV-CN	4±14	2±8	-15±16
FSV-CO	-5±3	-1±1	10±3
FSV-CQ	-3±4	4±3	-21±7
FSV-CV	0±1	10±17	16±13
FSV-CY	-11±4	3±6	-3±2
FSV-DC	8±2		-8±8
FSV-DE		1±1	
FSV-DG	1±2	-1±7	1±12
FSV-DH	-12±3	-19±4	-7±12
FSV-DL	1±5		10±33
FSV-DT	-11±10	-1±6	
FSV-DY	40±68		21±28
FSV-DZ	24±20	-17±14	
FSV-EB	5±14	2±12	-4±5
FSV-EF	3±4	-1±1	
FSV-EV	13±8	-1±7	
FSV-FA	-5±7	-7±6	6±9
NISTa	6±1	4±5	-13±30
NISTc	-11±4	-8±3	-3±12

Label	Definition
Lab	Participant code
TR	Total Retinol
aT	a-Tocopherol
bC	Total b-Carotene
% Bias	(Mean ± SD) of individual serum biases
Mean	Average of $(x_i - \text{Median}_i) / \text{Median}_i$
SD	Standard deviation of $(x_i - \text{Median}_i) / \text{Median}_i$
x_i	Result for analyte in serum _i
Median _i	Median of non-NIST results in serum _i

The original analysis listed % Bias for each result for each serum calculated relative to the "Trimmed Core Lab Average" of that analyte in the serum. The summary values reported here are the (arithmetic mean ± standard deviation) of each laboratory's reported results for the analyte estimated relative to each serum's median value.

The results for the calibration solution 123 do not necessarily reflect a participant's measurement performance for serum samples. Therefore, these summary values are calculated using only results for the four serum samples: 120, 121, and 122.

Appendix D. Shipping Package Inserts for RR19

Two items were included in each package shipped to an RR19 participant:

- **Cover letter.** The original letter has been lost. It would have described the four lyophilized sample materials (sera 130 to 133) distributed for the study, given guidance on reconstituting these samples, stated that results were due July 6, 1990 and to whom they should be sent, and who to contact with technical questions.
- **Datasheets.** The following two pages reproduce the form. The first page of the form is for reporting the primary-focus analytes: retinol, α -tocopherol, and total and *trans*- β -carotene. The second page is for reporting three “optional” analytes: retinyl palmitate, γ -tocopherol, and lycopene. In the absence of other information, the lycopene results are assumed to report total lycopene.

These items were attached to the shipping box.

REPORT ON NIST/NCI SAMPLES FROM LABORATORY # _____			
DATE OF ANALYSIS _____			
RESULTS IN $\mu\text{g/mL}$			
SAMPLE #	ANALYTE		
SERUM 130	RETINOL		
VIAL # ____		Trans	Total
	B-CAROTENE		
	A-TOCOPHEROL		
SERUM 131	RETINOL		
VIAL # ____		Trans	Total
	B-CAROTENE		
	A-TOCOPHEROL		
SERUM 132	RETINOL		
VIAL # ____		Trans	Total
	B-CAROTENE		
	A-TOCOPHEROL		
SERUM 133	RETINOL		
VIAL # ____		Trans	Total
	B-CAROTENE		
	A-TOCOPHEROL		

Add 1.0 mL of Distilled water to the Serum Samples.

REPORT ON NIST/NCI SAMPLES FROM LABORATORY # _____
 OPTIONAL ANALYTES
 DATE OF ANALYSIS _____

RESULTS IN µg/mL

SAMPLE #	ANALYTE
SERUM 130	RETINYL PALMITATE
VIAL # ____	LYCOPENE
	GAMMA-TOCOPHEROL
SERUM 131	RETINYL PALMITATE
VIAL # ____	LYCOPENE
	GAMMA-TOCOPHEROL
SERUM 132	RETINYL PALMITATE
VIAL # ____	LYCOPENE
	GAMMA-TOCOPHEROL
SERUM 133	RETINYL PALMITATE
VIAL # ____	LYCOPENE
	GAMMA-TOCOPHEROL

Appendix E. Final Report for RR19

The following eight pages are the final report for RR19 as provided to all participants:

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

It is likely that most participants reported “total lycopene” (the sum of all isomers.) Since resolution of γ -tocopherol and β -tocopherol is challenging, the results reported as γ -tocopherol can be confidently assumed to be γ/β -tocopherol.

Due to the complex formatting used in the Tables, the originally listed laboratory codes have been deleted without replacement. However, Appendix F provides a complete listing of the RR19 results where the original codes have been altered to ensure confidentiality. Appendix F also provides more relevant summary statistics.

August 23, 1990



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

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

Dear (Personal name),

By now you should have received a letter from Bob Schaffer regarding this years QA Workshop. I hope to see you here on November 15th. Discussions regarding our analysis of data from Round-Robin XIX follows.

Tables 1-3 each provide the usual compilation of both individual laboratory results and a summary of the interlaboratory data from the determination of serum/plasma levels of retinol, α -tocopherol, total β -carotene, respectively. Table 4 provides data compilations and summaries for serum/plasma determinations of trans β -carotene and three new analytes: retinyl palmitate, γ -tocopherol, and lycopene. Upon inspection of the data reported for these analytes, you will readily notice that, as in Round-Robin XVIII, interlaboratory variability is much greater for the three new compounds than for our three core analytes for which QA/QC procedure and practices have been put in place. Variations in interlaboratory precision for the determination of serum/plasma levels of retinol, α -tocopherol, and β -carotene respectively, over the past four years are shown in Figures 1-3. You are also provided with your lab's "Blind Control Charts" for each of the three core analytes.

As usual, the right side of Tables 1-3 provides data for use in evaluating your lab's performance in the study. The bias values given are based on the interlaboratory grand means for each analyte. NIST values are also provided for your information, but are not used in computing the grand means against which laboratory performance is being evaluated. A copy of a report describing NIST Analyses of Round Robin XIX samples is provided for your information. For all three analytes, the relatively close agreement between the NIST and grand mean values allows us to continue the convention of 0-5% bias from the grand mean representing EXCEPTIONAL performance, 6-10% ACCEPTABLE performance, 11-20% MARGINAL performance, and >20% UNACCEPTABLE with respect to the current state-of-the-practice for measurement of these analytes in serum/plasma.

Round-Robin XX samples will be shipped to you on September 10th. Results are required by October 22nd.

Sincerely,

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

Enclosures

Table 1.

Round Robin XIX
Retinol Results

Percent Bias from the Average.

Lab #	Serum # 130	Serum # 131	Serum # 132	Serum # 133	Serum #Serum 130 131	Serum #Serum 132 133	Serum #Serum 130 132	Serum #Serum 131 133
	0.188	0.425	0.244	0.745	-16.6	-17.4	-16.2	0.9
	0.230	0.488	0.273	0.729	2.0	-5.2	-6.3	-1.2
	0.243	0.569	0.292	0.847	7.8	10.6	-6.3	-14.8
	0.196	0.359	0.216	0.522	-13.1	-30.2	-25.8	-29.3
	0.192	0.478	0.309	0.754	-14.9	-7.1	-6.1	-2.2
	0.222	0.523	0.290	0.731	-1.8	1.6	-0.6	-1.0
	0.231	0.514	0.282	0.756	2.4	-0.1	-3.2	2.4
	0.420	0.990	0.540	1.350	86.2	92.4	85.4	82.9
		0.484	0.315	0.657		-5.9	8.2	-11.0
	0.264	0.455	0.299	0.801	17.1	-11.6	2.7	8.5
	0.236	0.482	0.292	0.748	4.6	-6.3	0.3	1.3
	0.247	0.571	0.301	0.800	9.5	11.0	3.4	8.4
	0.208	0.504	0.274	0.724	-7.8	-2.0	-5.9	-1.9
	0.177	0.402	0.234	0.601	-21.5	-21.9	-19.6	-18.6
	0.248	0.686	0.311	0.695	-10.0	3.4	-6.8	-5.2
	0.222	0.527	0.285	0.757	-1.6	2.4	-3.5	-1.1
	0.211	0.499	0.281	0.730	-6.4	-3.0	-2.5	-1.7
	0.230	0.510	0.290	0.780	-2.0	-0.9	-0.4	5.7
	0.250	0.550	0.330	0.840	10.9	-6.1	-3.3	13.8
	0.424	0.442	0.248	0.614	88.0	-14.3	-14.8	-16.8
	0.235	0.451	0.270	0.695	-9.5	-12.3	-7.3	-5.6
	0.257	0.578	0.332	0.875	4.2	-6.8	14.0	18.7
	0.279	0.462	0.318	0.674	14.0	-12.3	9.2	-8.7
	0.238	0.452	0.303	0.668	23.7	-8.2	4.0	17.5
	0.252	0.529	0.303	0.668	11.7	-12.8	9.2	-9.3
	0.205	0.526	0.291	0.745	-9.3	2.7	3.0	0.3
	0.218	0.518	0.288	0.712	-3.3	0.7	-0.1	1.3
	0.215	0.506	0.276	0.750	-4.6	-1.7	-5.2	-1.6
	0.223	0.556	0.321	0.719	-1.1	8.1	10.2	-2.0
	0.233	0.526	0.292	0.775	3.3	2.5	24.6	25.0
	0.238	0.661	0.363	0.926	5.5	28.0	0.3	5.0
	0.235	0.530	0.292	0.775	4.2	3.0	0.4	3.6
	0.255	0.610	0.290	0.765	13.1	18.6	-0.4	3.6
	0.188	0.229	0.263	0.575	-16.6	-5.5	-9.7	-22.1
	0.212	0.524	0.307	0.820				
NIST								
Avg	0.226	0.515	0.291	0.738				
SD	0.023	0.067	0.028	0.081				
RSD	10.1	13.1	9.8	11.0				

* = Value not included in statistical analysis.

L = Late, Results not included in statistical analysis.

NIST value not included in statistical analysis.

Table 2.

Round Robin XIX
Alpha-Tocopherol Results

Percent Bias from the Average

Lab #	Serum # 130	Serum # 131	Serum # 132	Serum # 133	Serum #Serum 130 131	Serum #Serum 132 133	Serum #Serum 130 131	Serum #Serum 132 133
	3.73	6.55	4.43	11.48	-0.4	-1.4	-0.4	-3.4
	3.82	6.57	4.55	11.91	2.0	-1.1	2.0	0.2
	4.17	7.12	4.99	12.69	11.4	7.1	11.4	6.8
	13.04	7.41	7.21	7.31	248.3	11.4	248.3	-38.5
	3.84	6.89	5.02	13.09	2.6	3.7	2.6	10.2
	3.66	6.73	4.55	11.71	-2.4	1.3	-2.4	-1.5
	3.65	6.43	4.66	11.90	-2.5	1.2	-2.5	0.2
	1.60	2.80	1.90	4.60	-57.3	-57.3	-57.3	-61.3
	3.55	6.73	4.38	12.14	-5.2	-5.3	-5.2	2.2
	3.57	6.73	4.63	12.70	-4.7	0.1	-4.7	6.9
	3.75	6.53	4.52	11.93	0.2	-0.4	0.2	0.4
	3.84	6.62	4.67	11.12	2.6	-5.5	2.6	-6.4
	4.01	6.28	4.37	12.45	7.1	-2.9	7.1	4.8
	3.91	6.46	4.37	9.61	4.4	-31.9	-14.4	-19.1
	3.79	4.52	3.96	11.81	1.2	3.8	1.2	-0.6
	3.88	6.90	4.63	11.41	3.6	2.6	3.6	-3.9
	6.26	6.82	4.54	11.65	67.1	15.0	67.1	-2.0
	3.83	7.65	4.84	11.95	2.3	-2.0	2.3	0.6
	3.20	6.51	4.65	11.60	-14.5	-5.3	-14.5	-2.4
	3.40	6.30	4.20	13.60	22.1	8.6	22.1	14.5
	4.57	7.88	5.39	14.31	-13.0	-22.6	-13.0	20.4
	3.26	5.47	4.33	12.96	-31.2	-22.1	-31.2	-9.1
	2.81	5.17	2.06	9.12	-24.9	-27.1	-24.9	-18.6
	3.80	4.84	3.52	9.40	1.5	9.8	1.5	-23.2
	3.83	7.30	4.90	12.40	2.3	5.6	2.3	4.4
	3.74	6.63	4.75	12.21	-0.1	-0.3	-0.1	2.8
	3.58	6.85	4.46	12.12	-4.4	3.1	-4.4	2.0
	4.47	6.37	4.85	11.23	19.4	-4.2	19.4	-5.5
	3.80	7.40	4.80	10.10	1.5	11.3	1.5	-15.0
	4.81	6.18	4.21	12.80	0.2	-7.0	0.2	-7.7
	4.81	7.70	5.84	11.08	28.3	15.9	28.3	-6.3
	1.59	0.18	1.12	13.70	-57.6	-97.4	-57.6	15.3
	3.32	6.75	4.43	2.95				-75.2
				11.46				
NIST								
	3.74	6.65	4.62	11.88				
Avg	0.46	0.76	0.43	1.21				
SD	12.3	11.4	9.3	10.2				
RSD								

* = Value not included in statistical analysis.

L = Late, Results not included in statistical analysis.

NIST value not included in statistical analysis.

Table 3.

Round Robin XIX
Total Beta-Carotene Results

Percent Bias from the Average

Lab #	Serum # 130	Serum # 131	Serum # 132	Serum # 133	Serum #Serum #Serum # 130 131 132 133
	0.993	0.181	1.153	0.526	1.9
	0.864	0.176	0.994	0.439	-11.3
	0.950	0.182	1.083	0.484	-2.5
	1.134	0.244	1.219	0.695	16.4
	0.958	0.205	1.135	0.534	-1.7
	1.800	0.410	2.360	1.100	84.7
	1.015	0.195	1.092	0.494	4.2
	1.210	0.000	0.621	0.415	24.2
	1.047	0.212	1.183	0.582	7.4
	0.964	0.198	1.096	0.532	-1.1
	0.947	0.194	1.241	0.522	-2.8
	0.823	0.190	1.034	0.487	-15.5
	0.962	0.178	1.112	0.463	-1.3
	0.910	0.160	1.140	0.560	-6.6
	0.290	0.053	0.354	0.182	-70.2
	0.874	0.185	1.053	0.554	-10.3
	0.801	0.157	0.926	0.535	-17.8
	0.974	0.223	1.156	0.521	0.0
	1.025	0.180	1.226	0.535	5.2
	1.163	0.186	1.111	0.590	19.3
	0.902	0.159	1.110	0.524	-7.4
	1.415	0.450	1.465	0.935	45.2
	0.974	0.849	1.370	0.496	0.0
	0.984	0.208	1.110	0.502	0.0
NIST					
Avg	0.974	0.189	1.145	0.524	
SD	0.106	0.022	0.121	0.059	
RSD	10.9	11.7	10.6	11.3	

* = Value not included in staistical analysis.

L = Late, Results not included in statistical analysis.

NIST value not included in statistical analysis.

Table 4.

RR XIX Results

Trans-Beta Carotene

Lab #	Serum # 130	Serum # 131	Serum # 132	Serum # 133
	0.916	0.168	1.058	0.441
	0.770	0.167	0.944	0.492
NIST	0.864	0.184	1.020	0.432
Avg	0.843	0.168	1.001	0.467

Retinyl Palmitate

Lab #	Serum # 130	Serum # 131	Serum # 132	Serum # 133
	0.008	ND	ND	ND
	ND	ND	ND	ND
	ND	ND	ND	ND
	ND	ND	ND	ND
	0.056	0.020	0.069	0.051
	0.025	0.026	0.068	0.046
	0.037	ND	ND	0.058
	0.010	0.012	0.014	0.016
	0.108			
Avg	0.041	0.019	0.050	0.043

Lycopene

Lab #	Serum # 130	Serum # 131	Serum # 132	Serum # 133
	0.135	0.312	0.254	0.297
	0.084	0.182	0.149	0.172
	0.170	0.480	0.380	0.460
	0.097	0.218	0.179	0.192
	0.091	0.204	0.171	0.196
	0.112	0.241	0.204	0.216
	0.130	0.210	0.210	0.240
	0.117	0.266	0.219	0.263
	0.080	0.213	0.165	0.192
	0.068	0.134	0.112	0.156
	0.750	0.200	0.173	0.203
	0.489	0.263	0.723	0.452
Avg	0.194	0.244	0.245	0.253

Gamma-Tocopherol

Lab #	Serum # 130	Serum # 131	Serum # 132	Serum # 133
	0.696	2.601	1.522	2.234
	0.830	2.810	1.770	2.530
	0.730	2.750	1.730	2.590
	0.220	0.710	0.420	0.600
	0.770	2.810	1.620	2.350
	0.700	2.540	1.500	2.210
	0.850	2.780	1.640	2.540
	0.731	2.555	1.547	2.220
	0.570	2.050	1.410	
	0.750	3.000	1.800	3.000
	1.290	5.130	3.150	4.570
	0.532	2.139	1.292	2.052
	0.648	2.305	1.450	2.130
	0.860	3.127	1.923	2.735
	0.750	2.590	1.780	2.050
NIST	0.867	3.030	1.510	2.530
Avg	0.728	2.660	1.637	2.254

Interlaboratory Precision vs Time

Retinol

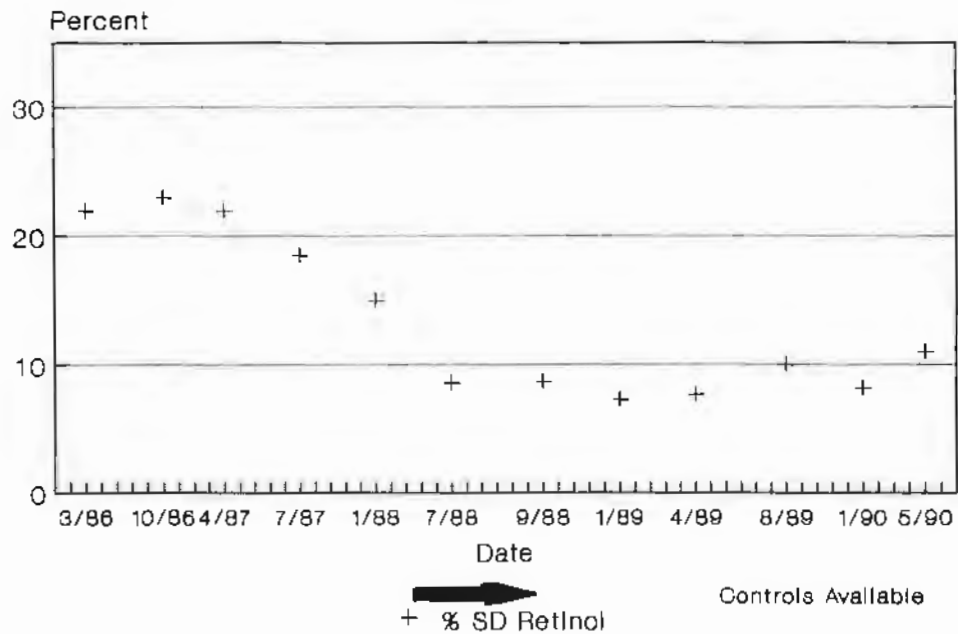
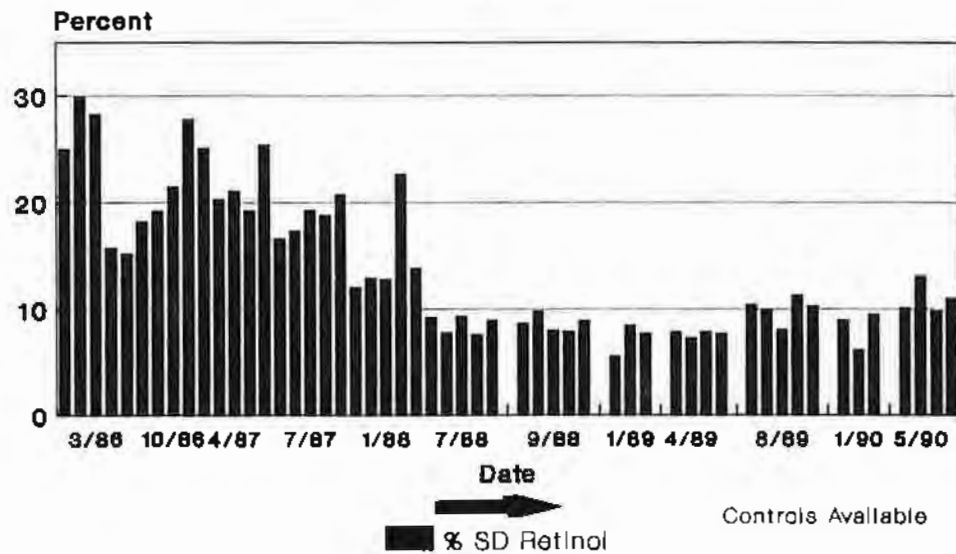


FIGURE 1

Interlaboratory Precision vs Time

Alpha-Tocopherol

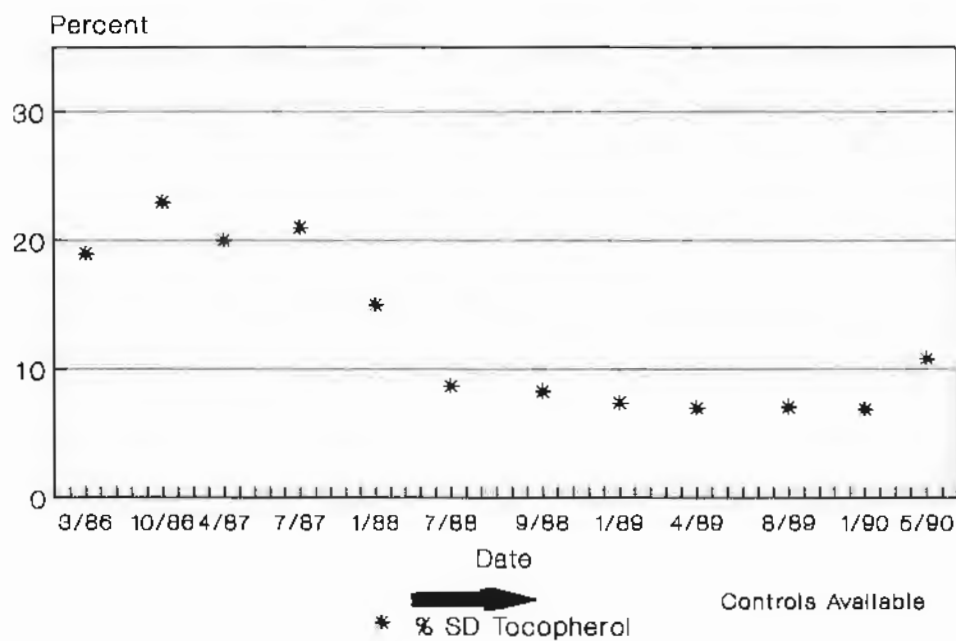
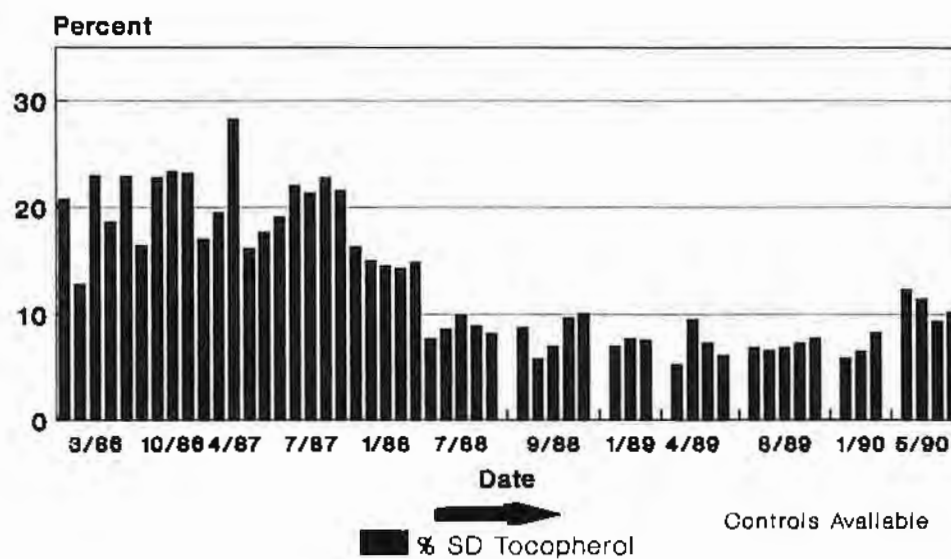
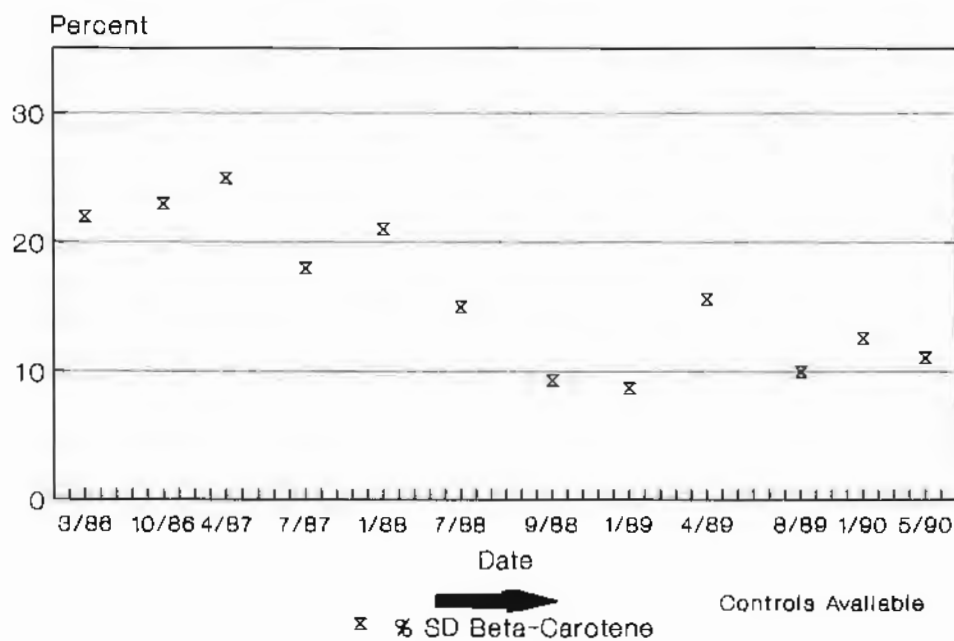
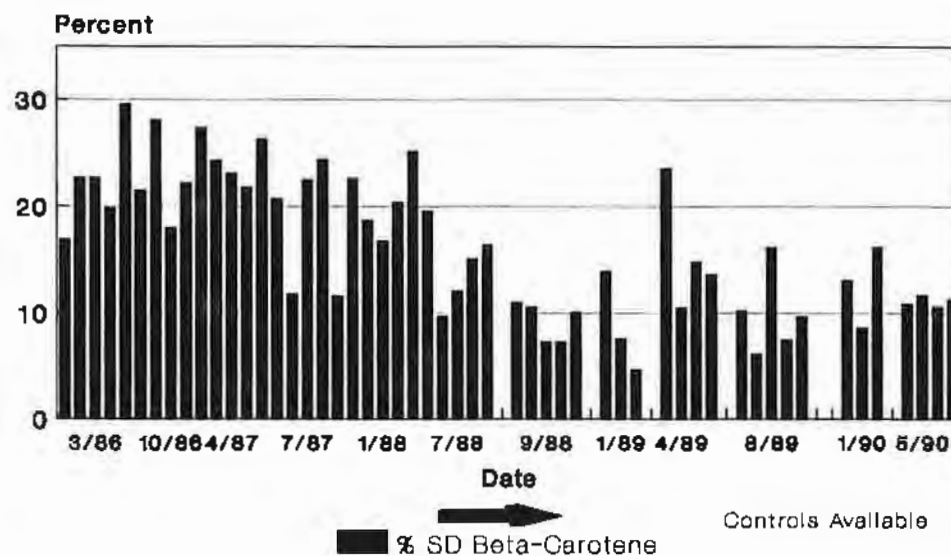


FIGURE 2

Interlaboratory Precision vs Time

Beta-Carotene



Appendix F. Updated “All-Lab Report” for RR19

The following four pages are an updated version of an “All-Lab” report for RR19. This report has three parts:

- pages 1 and 2 list results for all analytes reported at least twice, counting both participants and NIST analysts.
- page 3 provides a legend for pages 1 and 2.
- page 4 summarizes each participants’ performance for retinol, α -tocopherol, and total β -carotene. These summaries are compatible with the percent bias evaluation advice given in the RR19 Report. However, the current bias summaries are estimated relative to the median of all reported values for each analyte in each serum rather than to the trimmed average (“Avg”) used in the original and detailed in Appendix E. The original reference values were estimated from on-time results, with subjective exclusion of results deemed non-representative.

To ensure confidentiality, the laboratory identifiers used in this “All-Lab Report” have been altered from those used in RR19. The only attributed results are those reported by NIST. The NIST results are not used in the assessment of the consensus summary results of the study.

Note: The results designated NISTa in this updated All-Lab report are designated NIST in the Tables of Appendix E.

Round Robin XIX Laboratory Results

Lab	Total Retinol				Retinyl Palmitate				α -Tocopherol				γ/β -Tocopherol			
	130	131	132	133	130	131	132	133	130	131	132	133	130	131	132	133
FSV-BA	0.234	0.536	0.297	0.786					3.76	6.48	4.53	11.09				
FSV-BD	0.231	0.514	0.282	0.756					3.65	6.43	4.66	11.90	0.73	2.75	1.73	2.59
FSV-BE	0.236	0.482	0.292	0.748					3.84	6.28	4.67	11.12	0.85	2.78	1.64	2.54
FSV-BH	0.211	0.499	0.281	0.730	nd	nd	nd	nd	3.88	6.82	4.54	11.41	0.73	2.56	1.55	2.22
FSV-BI	0.230	0.488	0.273	0.729	nd	nd	nd	nd	3.82	6.57	4.55	11.91	0.83	2.81	1.77	2.53
FSV-BJ	0.218	0.518	0.288	0.712					3.83	7.02	4.75	12.21	0.86	3.13	1.92	2.74
FSV-BK	0.215	0.506	0.276	0.750					3.74	6.63	4.46	12.12				
FSV-BL	0.204	0.451	0.270	0.695					4.57	7.88	5.39	14.31	1.29	5.13	3.15	4.57
FSV-BM	0.205	0.526	0.291	0.748					3.80	7.30	4.90	12.40				
FSV-BY	0.188	0.425	0.244	0.745	0.008	nd	nd	nd	3.73	6.55	4.43	11.48	0.70	2.60	1.52	2.23
FSV-BZ									6.26	7.65	4.84	11.65				
FSV-CA	0.192	0.478	0.309	0.754					3.84	6.89	5.02	13.09				
FSV-CH	0.255	0.610	0.290	0.765					4.81	7.70	5.84	13.70				
FSV-CJ	0.243	0.569	0.292	0.847					4.17	7.12	4.99	12.69				
FSV-CM									3.40	7.20	4.60	13.60				
FSV-CN	na	0.484	0.315	0.657					3.55	6.73	4.38	12.14	0.77	2.81	1.62	2.35
FSV-CO	0.222	0.523	0.290	0.731					3.66	6.73	4.55	11.71				
FSV-CQ	0.264	0.455	0.299	0.801					3.57	6.53	4.63	12.70				
FSV-CS	0.238	0.529	0.303	0.745					2.81	4.84	3.52	9.12				
FSV-CV	0.233	0.526	0.292	0.775					4.47	6.37	4.85	10.10	0.75	2.59	1.78	2.05
FSV-CY	0.230	0.510	0.290	0.780					3.83	6.51	4.65	11.95	0.57	2.05	1.41	1.88
FSV-DC	0.208	0.504	0.274	0.724	nd	nd	nd	nd								
FSV-DE									3.75	6.62	4.52	11.93	0.70	2.54	1.50	2.21
FSV-DG	0.247	0.571	0.301	0.800					4.01	6.46	4.37	12.45				
FSV-DH	0.235	0.578	0.332	0.875	0.056	0.020	0.069	0.051	3.26	6.47	4.33	12.96	0.53	2.14	1.29	2.05
FSV-DL	0.260	0.489	0.288	0.739												
FSV-DN	0.420	0.990	0.540	1.350					1.60	2.80	1.90	4.60	0.22	0.71	0.42	0.60
FSV-DO	0.196	0.359	0.216	0.522					13.04	7.41	7.21	7.31				
FSV-DY	0.424	0.442	0.248	0.614												
FSV-DZ	0.248	0.686	0.311	0.695					3.91	4.52	3.96	9.61				
FSV-EB	0.235	0.530	0.292	0.775					3.75	6.18	4.21	11.08				
FSV-ED	0.222	0.527	0.285	0.757					3.79	6.90	4.63	11.81				
FSV-EF	0.250	0.550	0.330	0.840					3.20	6.30	4.20	11.60	0.75	3.00	1.80	3.00
FSV-EJ	0.188	0.229	0.263	0.575					1.92	2.05	1.12	2.95				
FSV-EO	0.177	0.402	0.234	0.601												
FSV-EV	0.238	0.661	0.363	0.926					3.80	7.40	4.80	12.80				
FSV-EY	0.223	0.556	0.321	0.719					3.58	6.85	4.89	11.23				
FSV-FA	0.252	0.452	0.303	0.668	<0.01	0.012	0.014	0.016	2.58	5.17	2.06	9.67	0.65	2.31	1.45	2.13
FSV-FQ	0.257	0.482	0.318	0.674	0.025	0.026	0.068	0.046								
FSV-FV	0.279	0.560	0.318	0.867	0.037	nq	nq	0.058								
n	36	37	37	37	4	3	3	4	34	34	34	34	15	15	15	15
Min	0.177	0.229	0.216	0.522	0.008	0.012	0.014	0.016	1.60	2.05	1.12	2.95	0.22	0.71	0.42	0.60
Mean	0.239	0.519	0.298	0.756	0.032	0.019	0.050	0.043	3.98	6.39	4.44	11.25	0.73	2.66	1.64	2.38
Max	0.424	0.990	0.540	1.350	0.056	0.026	0.069	0.058	13.04	7.88	7.21	14.31	1.29	5.13	3.15	4.57
SD	0.051	0.112	0.049	0.129	0.020	0.007	0.031	0.019	1.78	1.24	1.05	2.34	0.22	0.89	0.55	0.81
CV	21	22	17	17	64	36	63	43	45	19	24	21	30	33	33	34
NISTa	0.212	0.524	0.307	0.820					3.32	6.75	4.43	11.46	0.87	3.03	1.51	2.53
Median	0.234	0.514	0.292	0.748					3.78	6.60	4.58	11.86	0.73	2.60	1.62	2.23
eSD	0.026	0.053	0.024	0.053					0.24	0.44	0.35	1.11	0.12	0.31	0.24	0.44
eCV	11	10	8	7					6	7	8	9	17	12	15	20

Round Robin XIX Laboratory Results

Lab	Total β -Carotene				trans- β -Carotene				Total cis- β -Carotene				Total Lycopene			
	130	131	132	133	130	131	132	133	130	131	132	133	130	131	132	133
FSV-BA	0.909	0.201	1.086	0.522												
FSV-BD																
FSV-BE																
FSV-BH	0.962	0.178	1.112	0.463	0.916	0.168	1.058	0.441	0.046	0.010	0.054	0.022	0.112	0.241	0.204	0.216
FSV-BI	0.864	0.176	0.994	0.439									0.084	0.182	0.149	0.172
FSV-BJ	1.025	0.180	1.226	0.535									0.068	0.134	0.112	0.156
FSV-BK																
FSV-BL																
FSV-BM																
FSV-BY	0.993	0.181	1.153	0.526									0.135	0.312	0.254	0.297
FSV-BZ																
FSV-CA																
FSV-CH	1.415	0.450	1.465	0.935												
FSV-CJ	0.950	0.182	1.083	0.484												
FSV-CM																
FSV-CN	1.015	0.195	1.092	0.494									0.097	0.218	0.179	0.192
FSV-CO	0.958	0.205	1.135	0.534												
FSV-CQ	1.210	nd	0.621	0.415												
FSV-CS																
FSV-CV	1.163	0.186	1.111	0.590									0.108	0.200	0.173	0.203
FSV-CY	0.910	0.160	1.140	0.560									0.130	0.210	0.210	0.240
FSV-DC	0.964	0.198	1.096	0.532									0.091	0.204	0.171	0.196
FSV-DE																
FSV-DG	1.047	0.212	1.183	0.582	0.770	0.167	0.944	0.492	0.104	0.018	0.109	0.062	0.117	0.266	0.219	0.263
FSV-DH	0.874	0.185	1.053	0.554												
FSV-DL	1.067	0.166	1.138	0.527												
FSV-DN	1.800	0.410	2.360	1.100									0.170	0.480	0.380	0.460
FSV-DO	1.134	0.244	1.219	0.695												
FSV-DY	0.290	0.053	0.354	0.182												
FSV-DZ																
FSV-EB	0.902	0.159	1.110	0.524												
FSV-ED	0.823	0.190	1.034	0.487												
FSV-EF																
FSV-EJ	1.586	0.176	1.370	0.496									0.489	0.263	0.723	0.452
FSV-EO	0.947	0.194	1.241	0.522												
FSV-EV																
FSV-EY																
FSV-FA	0.974	0.223	1.156	0.521									0.080	0.213	0.165	0.192
FSV-FQ	0.801	0.157	0.926	0.535												
FSV-FV																
n	25	24	25	25	2	2	2	2	2	2	2	2	12	12	12	12
Min	0.290	0.053	0.354	0.182	0.770	0.167	0.944	0.441	0.046	0.010	0.054	0.022	0.068	0.134	0.112	0.156
Mean	1.023	0.203	1.138	0.550	0.843	0.168	1.001	0.467	0.075	0.014	0.082	0.042	0.140	0.244	0.245	0.253
Max	1.800	0.450	2.360	1.100	0.916	0.168	1.058	0.492	0.104	0.018	0.109	0.062	0.489	0.480	0.723	0.460
SD	0.280	0.078	0.333	0.167									0.113	0.087	0.165	0.102
CV	27	39	29	30									81	36	67	40
NISTa	0.984	0.208	1.110	0.502	0.864	0.184	1.020	0.432	0.120	0.024	0.090	0.070				
Median	0.964	0.186	1.112	0.526									0.110	0.216	0.192	0.210
eSD	0.123	0.021	0.087	0.047									0.033	0.044	0.040	0.050
eCV	13	11	8	9									30	20	21	24

Round Robin XIX Laboratory Results

Table Legend

Symbol	Interpretation
<i>na</i>	Not available (not reported by participant)
<i>nd</i>	Not detected (not reported or reported as '0', 'not determined', etc.)
<x	Value less than x
<i>italics</i>	Value calculated from reported results
n	Number of non-NIST laboratories reporting quantitative results
Min	Minimum non-NIST reported value.
Mean	Average over all non-NIST reported values.
Max	Maximum non-NIST reported value.
SD	Standard deviation over all non-NIST values.
CV	Coefficient of Variation (% relative standard deviation): $100 \cdot \text{SD} / \text{Mean}$
Median	Median over all non-NIST reported values
eSD	Robust estimate of SD based on the adjusted median absolute difference from the median (MADe)
eCV	Robust estimate of CV, $100 \cdot \text{eSD} / \text{Median}$

Round Robin XIX Laboratory Results

%Bias Summary

Lab	TR	aT	bC
FSV-BA	3±2	-2±3	0±6
FSV-BD	-1±2	-1±2	
FSV-BE	-1±3	-2±4	
FSV-BH	-5±3	0±3	-4±6
FSV-BI	-4±2	0±1	-11±5
FSV-BJ	-3±3	4±2	4±6
FSV-BK	-4±4	0±2	
FSV-BL	-10±3	20±1	
FSV-BM	-3±7	6±4	
FSV-BY	-13±9	-2±1	1±3
FSV-BZ		21±30	
FSV-CA	-5±10	7±4	
FSV-CH	7±9	22±7	75±49
FSV-CJ	7±6	9±1	-3±3
FSV-CM		4±11	
FSV-CN	-3±10	-1±4	1±6
FSV-CO	-2±3	-1±2	3±5
FSV-CQ	3±10	0±5	-13±35
FSV-CS	2±2	-25±2	
FSV-CV	1±2	2±14	8±10
FSV-CY	0±3	1±1	-3±9
FSV-DC	-6±4		2±4
FSV-DE		0±1	
FSV-DG	7±3	1±5	10±3
FSV-DH	11±7	-3±10	-2±6
FSV-DL	1±7		1±9
FSV-DN	84±6	-59±2	107±15
FSV-DO	-26±7	69±124	23±11
FSV-DY	9±49		-69±3
FSV-DZ	10±17	-15±15	
FSV-EB	2±2	-5±3	-5±7
FSV-ED	-1±3	1±2	-7±7
FSV-EF	10±3	-8±6	
FSV-EJ	-27±20	-67±12	19±33
FSV-EO	-21±2		3±6
FSV-EV	20±12	6±5	
FSV-EY	2±8	0±6	
FSV-FA	-3±10	-32±16	6±10
FSV-FQ	1±10		-12±9
FSV-FV	13±5		
NISTa	2±8	-4±6	2±7

Label	Definition
Lab	Participant code
TR	Total Retinol
aT	a-Tocopherol
bC	Total b-Carotene
% Bias	(Mean ± SD) of individual serum biases
Mean	Average of $(x_i - \text{Median}_i) / \text{Median}_i$
SD	Standard deviation of $(x_i - \text{Median}_i) / \text{Median}_i$
x_i	Result for analyte in serum _i
Median _i	Median of non-NIST results in serum _i

The original analysis listed % Bias for each result for each serum calculated relative to the "Trimmed Core Lab Average" of that analyte in the serum. The summary values reported here are the (arithmetic mean ± standard deviation) of each laboratory's reported results for the analyte estimated relative to each serum's median value.

Appendix G. Shipping Package Inserts for RR20

Two items were included in each package shipped to an RR20 participant:

- **Cover letter.** The original letter has been lost. It likely would have described the five sample materials (liquid frozen sera 138 and 139; lyophilized sera 140, 141, and 142), given guidance on reconstituting the lyophilized samples, stated that results were required by October 22, 1990, to whom they should be sent, and who to contact with technical questions. It is likely that this letter also provided details on the 1990 QA Workshop, held at NIST on November 15, 1990.
- **Datasheet.** The following two pages reproduce the form.

The cover letter and datasheets were attached to the shipping box.

REPORT ON NIST/NCI SAMPLES FROM LABORATORY # _____			
RR XX		DATE OF ANALYSIS _____	
RESULTS IN $\mu\text{g/mL}$			
SAMPLE #	ANALYTE		
SERUM 138	RETINOL		
VIAL # ____		Trans	Total
	B-CAROTENE		
	A-TOCOPHEROL		
SERUM 139	RETINOL		
VIAL # ____		Trans	Total
	B-CAROTENE		
	A-TOCOPHEROL		
SERUM 140	RETINOL		
VIAL # ____		Trans	Total
	B-CAROTENE		
	A-TOCOPHEROL		
SERUM 141	RETINOL		
VIAL # ____		Trans	Total
	B-CAROTENE		
	A-TOCOPHEROL		
SERUM 142	RETINOL		
VIAL # ____		Trans	Total
	B-CAROTENE		
	A-TOCOPHEROL		

Add 1.0 mL of Distilled water to samples 140, 141, and 142.

REPORT ON NIST/NCI SAMPLES FROM LABORATORY # _____
 OPTIONAL ANALYTES
 DATE OF ANALYSIS _____

RESULTS IN µg/mL

SAMPLE #	ANALYTE
SERUM 138	RETINYL PALMITATE
VIAL # ____	LYCOPENE
	GAMMA-TOCOPHEROL
SERUM 139	RETINYL PALMITATE
VIAL # ____	LYCOPENE
	GAMMA-TOCOPHEROL
SERUM 140	RETINYL PALMITATE
VIAL # ____	LYCOPENE
	GAMMA-TOCOPHEROL
SERUM 141	RETINYL PALMITATE
VIAL # ____	LYCOPENE
	GAMMA-TOCOPHEROL
SERUM 142	RETINYL PALMITATE
VIAL # ____	LYCOPENE
	GAMMA-TOCOPHEROL

Appendix H. Final Report for RR20

There is no available record of the complete final report for RR20. The following four pages present the results and summary statistics for the samples as interpreted at the time:

- Tables 1 thru 3 list retinol, α -tocopherol, total β -carotene;
- Tables 4 to 7 list *trans*- β -carotene, retinyl palmitate, lycopene, and γ -tocopherol.

It is likely that most participants reported “total lycopene” (the sum of all isomers.) Since resolution of γ -tocopherol and β -tocopherol is challenging, the results reported as γ -tocopherol can be confidently assumed to be γ/β -tocopherol.

Due to the complex formatting used in the tabular listings, the original laboratory codes have been deleted without replacement. However, Appendix I provides a complete listing of the RR20 results where the original codes have been altered to ensure confidentiality. Appendix I also provides more relevant summary statistics.

Table 1.

Round Robin XX

Retinol Results

Percent Bias from the Average.

Lab #	Serum # 138	Serum # 139	Serum # 140	Serum # 141	Serum # 142	Serum # 138	Serum # 139	Serum # 140	Serum # 141	Serum # 142
	0.611	0.715	0.738	*0.438	0.209	10.5	14.0	1.3	-47.6	-5.4
	0.532	0.594	0.714	0.795	0.216	-3.4	-5.0	-2.0	-5.0	-2.1
	*0.627	0.740	*0.884	0.953	*0.257	13.4	18.0	21.3	13.0	16.1
	*0.350	*0.420	*0.470	*0.550	0.200	-36.7	-33.0	-35.5	-34.3	-4.6
	0.500	0.599	0.700	0.861	0.231	-9.5	-4.5	-4.0	0.2	1.4
	0.588	0.632	0.739	0.833	0.232	-0.8	-12.3	1.4	-12.4	4.8
	0.586	0.550	0.727	0.766	0.180	6.4	-28.2	-0.8	-11.5	-10.3
	0.360	*0.450	0.570	0.740	0.244	-34.9	21.0	-0.3	-8.4	3.8
	*0.619	*0.759	0.727	0.812	0.215	12.0	-0.8	-0.3	-2.9	-8.7
	*0.762	0.570	*0.593	0.729	0.202	-14.2	-9.1	-4.6	-12.7	-6.6
	0.474	0.631	0.671	0.789	0.236	-0.7	0.5	-2.9	-5.8	-0.7
	0.549	0.580	0.750	0.861	0.220	-7.7	-3.0	4.4	2.9	5.7
	0.510	0.646	0.761	0.860	0.234	1.7	3.8	-6.8	-12.0	-3.9
	0.562	0.651	0.679	0.736	0.204	-0.5	7	-3.0	-5.2	-7.8
	0.550	0.688	0.707	0.880	0.195	-0.1	9.4	7.0	-7.4	-3.9
	0.552	0.538	*0.613	0.758	*0.186	-10.5	-30.8	-15.9	-27.6	-15.2
	*0.384	*0.434	*0.570	*0.606	*0.150	-30.5	-30.1	-21.8	-29.6	-32.2
	0.508	0.589	0.724	0.781	0.225	-8.1	-6.3	-0.7	-6.6	1.3
	0.560	0.581	0.680	*0.589	0.230	2.0	-7.5	-6.1	-8.5	-1.7
	0.553	0.650	0.737	0.908	0.229	1.0	3.7	1.2	6.9	5.5
	0.510	0.610	0.825	0.894	0.229	-7.8	31.7	23.6	23.2	2.9
	*0.723	*0.827	*0.900	*1.031	*0.272	30.5	10.9	3.1	19.3	1.7
	0.551	0.657	0.738	0.997	0.225	-0.3	4.8	-10.1	-13.5	-8.3
	0.525	0.659	0.655	0.724	*0.203	13.1	5.1	13.5	20.0	3.9
	0.625	0.659	*0.877	0.988	0.269	-5.8	6.0	2.4	0.1	3.5
	0.583	0.669	*0.827	0.896	0.230	-2.5	-0.5	1.3	7.1	3.3
	0.537	0.624	0.746	0.838	0.229	-2.8	-0.5	2.4	0.2	3.5
NIST										
NIST										
AVG	0.553	0.627	0.729	0.837	0.221					
SD	0.044	0.053	0.031	0.081	0.013					
RSD	8.0	8.5	4.3	9.6	5.9					
PREV AVG		0.738	0.848	0.226						

* = Value greater than or less than 2 SD from the Mean.
 L = Late results not included in the statistical analysis

Table 2.

Round Robin XX

Alpha-Tocopherol Results

Percent Bias from the Average.

Lab #	Serum # 138	Serum # 139	Serum # 140	Serum # 141	Serum # 142	Serum # 138	Serum # 139	Serum # 140	Serum # 141	Serum # 142
	10.45	10.38	11.73	*6.24	3.66	-0.1	7.5	1.5	-34.1	-1.4
	10.04	9.61	11.25	8.89	3.65	-4.0	-2.9	-2.8	-6.1	-1.6
	11.63	10.61	12.57	10.40	4.09	11.2	-9.9	-8.9	-9.9	10.3
	*6.05	8.76	*6.36	*5.76	*4.89	-42.1	-9.2	-45.0	-39.1	31.8
	9.44	9.18	11.48	9.56	3.60	-9.7	-4.9	-0.7	1.0	-3.9
	10.45	9.83	11.61	8.90	3.58	-0.1	14.0	-0.4	-7.0	-3.5
	10.30	8.30	11.90	10.40	3.60	-1.5	-0.5	-5.1	-6.0	-2.9
	10.30	9.50	11.20	9.77	3.78	-1.0	-0.5	-3.4	9.2	-2.9
	9.41	8.90	11.60	9.10	3.39	-15.0	-1.7	-0.2	3.3	-1.6
	12.02	9.80	11.33	9.25	3.46	-2.0	1.0	-2.0	-2.3	-6.7
	10.22	9.65	11.39	9.18	3.70	-2.3	0.4	-2.4	-3.0	-0.2
	10.23	9.88	11.62	9.92	3.40	-4.2	2.2	-1.0	-4.8	10.3
	9.70	9.45	11.13	8.89	4.09	-7.2	-2.0	-3.7	-6.1	4.6
	9.55	9.67	11.71	9.01	3.95	-0.9	2.2	-1.3	-4.1	6.5
	10.37	9.86	11.53	9.29	3.51	-0.9	-10.1	-13.5	-1.8	-2.6
	8.70	8.60	*10.00	10.30	*3.58	-16.8	2.0	-0.8	-4.5	0.0
	10.20	9.80	11.90	9.09	3.60	-2.4	1.5	-3.0	-3.5	-24.0
	11.48	*11.24	*13.27	9.80	*4.60	9.8	16.4	14.2	6.2	4.7
	11.38	8.49	10.70	10.05	*3.94	-12.0	-1.4	-9.1	-13.9	-5.1
	9.20	9.60	10.50	*8.15	3.52	-2.4	-0.5	-4.7	0.4	1.1
	12.03	10.95	12.16	9.50	3.90	-12.0	13.4	5.2	2.3	5.8
	9.83	9.14	11.02	*11.39	3.68	-2.4	-0.5	-3.4	4.4	4.8
	11.56	10.75	*13.02	10.68	3.77	-6.0	11.3	12.7	8.7	7.2
	10.10	9.83	11.90	*10.95	*4.20	-3.4	1.9	3.0	15.7	13.0
	11.50	10.60	12.60	9.60	3.70	10.4	9.8	9.9	1.4	2.3
	11.92	10.23	12.76	10.11	3.76	4.8	6.0	6.3	8.2	1.0
	*13.57	*11.41	*13.09	*11.48	*5.09	29.8	18.4	13.8	21.6	37.1
NIST	10.64	10.06	11.35	8.85	3.30	1.8	4.2	-1.1	-6.5	-11.0
NIST	10.10	9.45	11.40	8.85	3.45	-3.4	-2.1	-1.4	-6.5	-7.0
Avg	10.46	9.65	11.56	9.46	3.71					
SD	0.88	0.69	0.50	0.56	0.20					
RSD	8.4	7.2	4.4	5.9	5.3					
PREV AVG			11.88	9.62	3.74					

* = Value greater than or less than 2 SD from the Mean.
 L = Late results not included in the Statistical analysis

Table 3.

Round Robin XX

		Total Beta-Carotene Results					Percent Bias from the Average.				
Lab #	Serum #	Serum #	Serum #	Serum #	Serum #	Serum #	Serum #	Serum #	Serum #	Serum #	Serum #
	138	139	140	141	142	138	139	140	141	142	
	1.903	0.506	0.525	1.136	0.930	17.6	14.6	0.5	-40.3	7.6	
	1.495	0.416	0.482	1.532	0.885	-7.6	-5.6	-7.8	-19.5	2.4	
	1.924	0.483	0.579	2.533	*1.084	18.9	9.6	10.8	-33.0	25.4	
	*2.320	0.430	0.720	1.170	0.880	43.4	-2.6	37.9	-38.6	1.8	
	1.702	0.470	0.564	2.163	0.911	5.2	6.9	7.2	-13.5	-0.5	
	1.490	0.410	0.490	1.400	0.860	-7.4	-6.0	-6.2	-26.6	3.5	
	1.673	0.498	0.648	2.278	0.922	3.4	13.6	2.0	-19.4	7.7	
	*3.540	0.372	0.477	*3.530	*1.550	118.0	-15.4	24.7	85.4	-6.3	
	1.586	0.451	0.477	1.898	0.819	-2.0	2.8	-8.7	-0.3	2.3	
	1.420	0.380	0.530	1.750	0.910	-12.2	1.0	1.4	-8.1	5.2	
	1.770	0.445	0.503	2.246	0.883	9.4	5.1	-3.7	17.9	2.2	
	1.740	0.463	0.472	1.580	0.861	7.6	5.1	-9.4	-11.0	4.5	
	1.681	0.463	0.505	2.118	0.938	3.9	5.1	-3.4	11.5	0.6	
			0.510	2.370	0.890			-2.4	24.5	3.0	
	1.254	0.371	0.489	1.950	0.781	-22.5	8.5	-6.4	-23.4	-9.1	
	1.670	0.390	0.402	1.649	0.682	-3.2	-11.6	-1.4	-13.9	1.2	
	*0.829	0.350	0.390	*0.935	*0.595	-48.8	-20.6	-23.1	-50.7	-21.3	
	*2.099	0.551	0.599	2.393	*1.092	29.7	25.1	-14.6	25.7	29.5	
	1.460	0.489	0.625	2.140	0.929	-9.7	11.0	19.6	12.4	7.5	
	1.607	0.401	0.508	1.703	0.840	-30.1	-9.0	-3.7	-10.6	5.8	
	1.130	0.387	0.503	1.730	0.745	-14.7	12.5	-17.0	-12.4	-13.8	
	1.380	0.443	0.434	1.668	0.952	21.8	59.8	-1.6	-17.0	1.8	
	1.970	0.704	0.514	1.580	0.952	14.4	19.8	12.3	-0.2	7.7	
	1.850	0.528	0.587	1.900	0.932						
NIST											
NIST											
AVG	1.618	0.441	0.523	1.904	0.864						
SD	0.189	0.054	0.081	0.408	0.075						
RSD	11.7	12.2	15.6	21.5	8.7						
PREV AVG			0.524	1.976	0.974						

* = Value greater than or less than 2 SD from the Mean.
 L = Late results not included in the statistical analysis

Table 4.

Round Robin XX Trans Beta-Carotene Results

Lab #	Serum #138	Serum #139	Serum #140	Serum #141	Serum #142
	1.660	0.420	0.429	1.460	0.793
	1.610	0.437	0.480	1.987	0.878
	1.200	0.345	0.448	1.822	0.722
NIST	1.840	0.533	0.444	1.350	0.845
NIST	1.690	0.441	0.466	1.930	0.817
	1.600	0.435	0.453	1.710	0.811
AVG	0.239	0.067	0.020	0.287	0.059
SD	15.0	15.4	4.4	16.8	7.3
RSD					
PREV AVG			0.467	1.791	0.843

Table 5.

Round Robin XX Retinyl Palmitate

Lab #	Serum #138	Serum #139	Serum #140	Serum #141	Serum #142
	0.259	0.079	ND	ND	ND
	0.214	0.052	ND	ND	ND
	0.212	0.064	ND	ND	ND
	0.084	0.081	0.036	0.021	0.011
	0.352	0.109	ND	ND	ND
NIST	0.220	0.072	0.016		
	0.224	0.076	0.026	0.021	0.011
AVG	0.087	0.019			
SD	38.7	25.3			
RSD					
PREV AVG			0.043		0.041

Table 6.

Round Robin XX Lycopene

Lab #	Serum #138	Serum #139	Serum #140	Serum #141	Serum #142
	0.482	0.522	0.250	0.451	0.129
	0.251	0.259	0.165	0.080	0.076
	0.435	0.533	0.274	0.166	0.135
	0.268	0.260	0.158	0.099	0.072
	0.369	0.391	0.219	0.137	0.101
	0.371	0.418	0.220	0.180	0.140
	0.270	0.330	0.275	0.169	0.113
	1.209	1.090	0.810	0.098	0.080
	0.555	0.575	0.374	0.423	0.237
NIST	0.379	0.443	0.215	0.250	0.190
			0.099	0.099	0.071
	0.459	0.482	0.289	0.196	0.122
AVG	0.281	0.240	0.182	0.129	0.053
SD	61.2	49.8	63.1	66.0	43.2
RSD					
PREV AVG			0.253	0.108	0.140

Table 7.

Round Robin XX Gamma Tocopherol

Lab #	Serum #138	Serum #139	Serum #140	Serum #141	Serum #142
	1.15	2.01	2.15	0.77	0.67
	1.46	2.25	2.43	0.72	0.78
	1.49	2.17	1.98	0.76	0.91
	1.27	2.16	2.19	0.68	0.72
	1.33	2.20	2.21	0.68	0.71
			2.61	0.66	0.85
	0.98	1.77	1.79	0.43	0.42
	1.40	2.07	2.21	0.85	0.87
	1.93	3.17	2.88	0.97	0.88
	1.12	1.88	2.00	0.70	0.59
	1.47	2.26	2.58	0.71	0.75
NIST	1.74	2.75	2.76	0.87	0.87
	1.394	2.244	2.316	0.733	0.751
AVG	0.275	0.396	0.336	0.133	0.143
SD	19.7	17.6	14.5	18.1	19.0
RSD					
PREV AVG			2.254	0.846	0.728

Appendix I. Updated “All-Lab Report” for RR20

The following four pages are an updated version of an “All-Lab” report for RR20. This report has three parts:

- pages 1 and 2 list results for all analytes reported by at least twice, counting both participants and NIST analysts.
- page 3 lists values for all analytes reported by only once. This page also provides a legend for pages 1 and 2.
- page 4 summarizes each participants’ performance for total retinol, α -tocopherol, and total β -carotene. These summaries are compatible with the percent bias evaluation advice given in the RR20 Report. However, the current bias summaries are estimated relative to the median of all reported values for each analyte in each serum rather than to the “Trimmed Core Lab Average” used in the original and detailed in Appendix J. These original reference values were estimated from on-time results of the more experienced participants, with subjective exclusion of results deemed non-representative.

In the original data sheets for RR20, one participant commented that the two “Serum 141” vials they received yielded quite discordant results. Only the most concordant of their two sets of results were included in the original Final Report (see Appendix H). When reviewing the data for this report, it was noted that the excluded Serum 141 results from this participant as well as those reported by two other participants matched the results expected for Serum 121 distributed in RR18; the intended Serum 141 material was distributed as Serum 122 in RR18. Thus it is quite plausible that several vials of Serum 121 were incorrectly relabeled and distributed in RR20. For this reason, the updated All-Lab Report lists the three sets of discordant Serum 141 results under the heading “Serum 141a”. The median values listed for Serum 141a are those for Serum 121 in RR18.

To ensure confidentiality, the laboratory identifiers used in this “All-Lab Report” have been altered from those used in RR20. The only attributed results are those reported by NIST. The NIST results are not used in the assessment of the consensus summary results of the study.

Note: The results designated NISTa and NISTc in this updated All-Lab report are both designated “NIST” in the Tables of Appendix H.

Round Robin XX Laboratory Results

Lab	Total Retinol					Retinyl Palmitate					α-Tocopherol					γ/β-Tocopherol									
	138	139	140	141	141a	142	138	139	140	141	141a	142	138	139	140	141	141a	142	138	139	140	141	141a	142	
FSV-BA	0.562	0.646	0.761	0.86		0.234							10.6	9.67	11.71	9.0		3.95							
FSV-BD	0.588	0.550	0.739	0.73		0.232							10.3	8.30	10.90	8.9		3.60							
FSV-BE	0.549	0.631	0.671	0.79		0.236							10.0	9.88	11.62	9.9		4.09							
FSV-BF	0.619	0.759	0.727	0.77		0.244							9.4	9.50	11.60	9.8		3.78							
FSV-BH	0.550	0.651	0.679	0.74		0.204							10.4	9.86	11.53	9.3		3.61						0.91	
FSV-BI	0.532	0.594	0.714	0.80		0.216							10.0	9.38	11.25	8.9		3.65						0.71	
FSV-BJ	0.564	0.581	0.680	0.59		0.230							12.0	10.95	12.16	11.4		3.91						0.78	
FSV-BK	0.560	0.630	0.737	0.91		0.218							9.8	9.14	11.17	9.7		3.68							
FSV-BM	0.508	0.589	0.724	0.78		0.225							10.2	9.60	12.10	9.5		3.90							
FSV-BY	0.611	0.715	0.738		0.438	0.209							10.5	10.38	11.73		6.24	3.66							
FSV-BZ													8.7	8.60	10.00	10.3		5.50						0.67	
FSV-CA	0.500	0.599	0.700	0.86	0.485	0.231							9.4	9.18	11.48	9.6	6.50	3.60							
FSV-CH	0.625	0.659	0.877	0.99		0.269							13.6	11.41	13.09	11.5		5.09							
FSV-CJ	0.627	0.740	0.884	0.95		0.257							11.6	10.61	12.57	10.4		4.09							
FSV-CM													10.2	9.80	11.90	9.8		4.60							
FSV-CO	0.549	0.632	0.760	0.84		0.224							10.4	9.83	11.61	8.8		3.58							
FSV-CQ	0.762	0.622	0.760	0.81		0.215							12.0	8.90	11.30	9.1		3.39							
FSV-CV	0.510	0.610	0.755	0.86		0.229							10.1	9.83	11.90	11.0		4.20						0.59	
FSV-CY	ns	ns	0.780	0.88		0.230							ns	ns	11.46	9.1		3.68						0.85	
FSV-DC																									
FSV-DE													10.2	9.65	11.39	9.2		3.70						0.72	
FSV-DG	0.510	0.580	0.750	0.86		0.220							9.7	9.45	11.13	8.9		3.87							
FSV-DH	0.552	0.688	0.707	0.77		0.195							11.5	11.24	13.27	10.1		3.36						0.42	
FSV-DL	0.551	0.657	0.655	0.72		0.203																			
FSV-DN	0.360	0.450	0.570	0.74		0.180							10.3	9.60	11.20	10.4		3.60							
FSV-DO	0.350	0.420	0.470		0.550	0.200							6.1	8.76	6.36		5.76	4.89							
FSV-DT	0.474	0.570	0.593	0.73		0.202							10.2	9.80	11.33	9.3		3.46							
FSV-EB	0.572	0.694	0.738	1.00		0.225							10.9	10.23	10.76	10.1		3.76							
FSV-EF	0.490	0.540	0.720	0.83		0.230							9.9	9.30	14.20	12.8		4.70						1.00	
FSV-EV	0.723	0.827	0.900	1.03		0.272							11.5	10.60	12.60	9.6		3.70							
FSV-EY	0.553	0.650	0.825	0.89		0.229							11.6	10.75	13.02	10.3		3.77							
FSV-FA	0.384	0.434	0.570	0.61		0.150							9.2	8.49	10.50	8.2		3.52						0.87	
FSV-FQ	0.493	0.538	0.613	0.76		0.186							11.4	9.79	11.70	8.3		1.94							
n	28	28	29	27	3	29							30	30	31	29	3	31		10	10	11	10	1	11
Min	0.350	0.420	0.470	0.589	0.438	0.150							6.05	8.30	6.36	8.15	5.76	1.94		0.98	1.77	1.79	0.43		0.421
Mean	0.544	0.616	0.717	0.818	0.491	0.220							10.39	9.75	11.57	9.75	6.17	3.87		1.34	2.16	2.26	0.76		0.763
Max	0.762	0.827	0.900	1.031	0.550	0.272							13.57	11.41	14.20	12.80	6.50	5.50		1.93	3.17	2.88	1.20		1.000
SD	0.090	0.092	0.095	0.106	0.056	0.025							1.29	0.78	1.29	1.00	0.38	0.63		0.26	0.39	0.31	0.21		0.165
CV	17	15	13	13	11	12							12	8	11	10	6	16		19	18	14	27		22
NISTa	0.583	0.669	0.827	0.90		0.230							10.6	10.06	11.35	8.9		3.30		1.69	2.60	2.94	0.81		0.86
NISTc	0.537	0.624	0.746	0.84		0.229							10.1	9.45	11.40	8.9		3.45		1.74	2.75	2.76	0.87		0.87
Median	0.551	0.626	0.727	0.812	0.485	0.225							10.27	9.73	11.60	9.60	6.24	3.70		1.31	2.12	2.21	0.71		0.78
eSD	0.062	0.067	0.050	0.101		0.015							0.74	0.69	0.59	0.87		0.27		0.23	0.18	0.31	0.07		0.15
eCV	11	11	7	12		7							7	7	5	9		7		17	8	14	10		19

Round Robin XX Laboratory Results

Lab	Total β -Carotene						trans- β -Carotene						Total cis- β -Carotene						Total Lycopene																	
	138	139	140	141	141a	142	138	139	140	141	141a	142	138	139	140	141	141a	142	138	139	140	141	141a	142												
FSV-BA	1.74	0.46	0.472	1.58		0.86	1.660	0.420	0.429	1.460		0.793	0.080	0.043	0.043	0.120		0.068																		
FSV-BD																																				
FSV-BE	1.67	0.50	0.490	2.28		0.92																														
FSV-BF	1.68	0.46	0.505	2.12		0.94																														
FSV-BH	1.50	0.42	0.482	1.53		0.89																														
FSV-BI	2.10	0.55	0.599	2.39		1.09																														
FSV-BJ																																				
FSV-BK																																				
FSV-BM																																				
FSV-BY	1.90	0.51	0.525		1.14	0.93																														
FSV-BZ																																				
FSV-CA																																				
FSV-CH	1.38	0.44	0.434	1.67		0.75																														
FSV-CJ	1.92	0.48	0.579	2.53		1.08																														
FSV-CM																																				
FSV-CO	1.70	0.47	0.564	2.16		0.91																														
FSV-CQ	3.54	0.37	0.648	3.53		1.55																														
FSV-CV	1.46	0.49	0.625	2.14		1.12																														
FSV-CY	ns	ns	0.510	2.37		0.89																														
FSV-DC	1.77	0.45	0.503	2.25		0.88																														
FSV-DE																																				
FSV-DG	1.42	0.38	0.530	1.75		0.91																														
FSV-DH	1.25	0.37	0.489	1.95		0.78																														
FSV-DL	1.13	0.39	0.503	1.73		0.84																														
FSV-DN	1.49	0.41	0.490	1.40		0.86																														
FSV-DO	2.32	0.43	0.720		1.17	0.88																														
FSV-DT	1.59	0.45	0.477	1.90		0.82																														
FSV-EB	1.61	0.40	0.508	1.70		0.93																														
FSV-EF																																				
FSV-EV																																				
FSV-EY																																				
FSV-FA	0.83	0.35	0.390	0.94		0.60																														
FSV-FQ	1.67	0.39	0.402	1.65		0.68																														
n	21	21	22	20	2	22	3	3	3	3	3	0	3	3	3	3	3	3	3	3	3	3	3	3												
Min	0.829	0.350	0.390	0.935	1.136	0.595	1.200	0.345	0.429	1.460		0.722	0.054	0.026	0.025	0.120		0.059	0.251	0.259	0.158	0.080		0.251	0.259	0.158	0.080		0.072	0.468	0.486	0.297	0.178	0.127	0.072	
Mean	1.699	0.437	0.520	1.978	1.153	0.914	1.490	0.401	0.452	1.756		0.798	0.068	0.032	0.036	0.126		0.062	0.468	0.486	0.297	0.178		0.468	0.486	0.297	0.178		0.127	1.209	1.090	0.810	0.423	0.237	0.237	
Max	3.540	0.551	0.720	3.530	1.170	1.550	1.660	0.437	0.480	1.987		0.878	0.080	0.043	0.043	0.131		0.068	1.209	1.090	0.810	0.423		1.209	1.090	0.810	0.423		0.237	0.297	0.254	0.191	0.106	0.053	0.053	
SD	0.532	0.053	0.078	0.536		0.187	0.252	0.049	0.026	0.270		0.078	0.013	0.010	0.010	0.006		0.005	0.297	0.254	0.191	0.106		0.297	0.254	0.191	0.106		0.053	0.053	0.053	0.053	0.053	0.053		
CV	31	12	15	27		20	17	12	6	15		10	19	31	27	5		8	63	52	64	59		63	52	64	59		41	63	52	64	59	41	63	
NISTa	1.97	0.70	0.514	1.58		0.95	1.840	0.533	0.444	1.350		0.845	0.130	0.171	0.070	0.230		0.107	0.38	0.44	0.215	0.099		0.38	0.44	0.215	0.099		0.071	0.371	0.418	0.235	0.166	0.121	0.121	
NISTc	1.85	0.53	0.587	1.90		0.93	1.690	0.441	0.466	1.930		0.817	0.160	0.087	0.121	nd		0.115	0.153	0.170	0.059	0.099		0.153	0.170	0.059	0.099		0.045	0.371	0.418	0.235	0.166	0.121	0.121	
Median	1.670	0.443	0.504	1.924	1.153	0.888																														
eSD	0.311	0.062	0.039	0.443		0.067																														
eCV	19	14	8	23		8																														

Round Robin XX Laboratory Results

Analytes Reported By One Laboratory

Values in µg/mL

Analyte	Code	138	139	140	141	141a	142
Total α-Carotene	FSV-BJ	0.083	0.041	0.032	0.043		0.036

Table Legend

Symbol	Interpretation
<i>na</i>	Not available (not reported by participant)
<i>nd</i>	Not detected (not reported or reported as '0', 'not determined', etc.)
<i>ns</i>	No sample (sample either damaged or not sent)
<i>italics</i>	Value calculated from reported results
<i>n</i>	Number of non-NIST laboratories reporting quantitative results
Min	Minimum non-NIST reported value.
Mean	Average over all non-NIST reported values.
Max	Maximum non-NIST reported value.
SD	Standard deviation over all non-NIST values.
CV	Coefficient of Variation (% relative standard deviation): 100*SD/Mean
Median	Median over all non-NIST reported values
eSD	Robust estimate of SD based on the adjusted median absolute difference from the median (MADe)
eCV	Robust estimate of CV, 100*eSD/Median

Round Robin XX Laboratory Results

%Bias Summary

Lab	TR	aT	bC
FSV-BA	4±1	1±5	-4±9
FSV-BD	-2±8	-6±6	
FSV-BE	-1±5	3±5	
FSV-BF	7±11	-1±4	6±9
FSV-BH	-4±6	-1±2	4±4
FSV-BI	-3±1	-4±2	-8±8
FSV-BJ	-7±12	12±6	23±3
FSV-BK	2±6	-3±3	
FSV-BM	-4±3	1±3	
FSV-BY	2±11	1±5	9±5
FSV-BZ		3±27	
FSV-CA	-2±5	-3±3	
FSV-CH	16±7	24±10	-12±7
FSV-CJ	17±3	10±2	19±9
FSV-CM		6±10	
FSV-CO	2±2	-2±4	7±5
FSV-CQ	8±18	-2±11	57±50
FSV-CV	0±5	6±7	12±16
FSV-CY	6±3	-2±3	8±13
FSV-DC			4±7
FSV-DE		-1±2	
FSV-DG	-2±6	-3±5	-6±9
FSV-DH	-2±8	7±10	-11±10
FSV-DL	-5±7		-12±12
FSV-DN	-23±10	0±5	-10±10
FSV-DO	-21±21	-15±31	18±22
FSV-DT	-12±4	-2±3	-4±4
FSV-EB	8±9	2±6	-4±7
FSV-EF	-4±8	15±18	
FSV-EV	27±5	6±6	
FSV-EY	6±6	9±4	
FSV-FA	-28±5	-11±4	-36±15
FSV-FQ	-13±4	-10±23	-14±9
NISTa	8±4	-3±7	14±28
NISTc	1±2	-4±3	10±8

Label	Definition
Lab	Participant code
TR	Total Retinol
aT	a-Tocopherol
bC	Total b-Carotene
% Bias	(Mean ± SD) of individual serum biases
Mean	Average of $(x_i - \text{Median}_i) / \text{Median}_i$
SD	Standard deviation of $(x_i - \text{Median}_i) / \text{Median}_i$
x_i	Result for analyte in serum _i
Median _i	Median of non-NIST results in serum _i

The original analysis listed % Bias for each result for each serum calculated relative to the trimmed "Avg" of that analyte in the serum. The summary values reported here are the (arithmetic mean ± standard deviation) of each laboratory's reported results for the analyte estimated relative to each serum's median-based reference value.

For all sera except 141a, the reference value is the median of results in this study. For serum 141a, the reference value is the median of of results for serum 121 in Round Robin XVIII.