NIST Micronutrients Measurement Quality Assurance Program Spring and Fall 1987 Comparability Studies

Results for Round Robins IX and XI
Fat-Soluble Vitamins and Carotenoids in Human Serum

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

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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 1987 MMQAP measurement comparability improvement studies: 1) Round Robin IX Fat-Soluble Vitamins and Carotenoids in Human Serum and 2) Round Robin XI Fat-Soluble Vitamins and Carotenoids in Human Serum. The first participant results for Round Robin IX were received April 22, 1987; the last results were received June 23, 1987. The first participant results for Round Robin XI were received July 8, 1987; the last results were received September 4, 1987. The analytes in Round Robin X were selenium and zinc and are not discussed in this report.

Keywords

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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 IX: Fat-Soluble Vitamins and Carotenoids in Human Serum

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin IX comparability study (hereafter referred to as RR09) received five lyophilized human serum test samples (sera 62 to 66) for analysis and three ethanolic calibration solutions. Unless multiple vials were previously requested, participants received one vial of each test sample. These sera were shipped on dry ice to participants in April 1987. The communication materials included in the sample shipment are described in Appendix A.

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.

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 RR09 in a more accessible format.

Round Robin X: Selenium and Zinc in Human Serum

The only analytes in MMQAP Round Robin X were selenium and zinc; the results from this study are not included in this report. The eleven participants each received five lyophilized human serum test samples (sera 67 to 71).

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

Five lyophilized human serum test samples (sera 72 to 76) were distributed in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin XI comparability study (hereafter referred to as RR11). Twenty participants received all five sera; four received sera 73, 75, and 76; and five received sera 72 and 74. Most laboratories receiving partial sets were relatively new to the MMQAP. Unless multiple vials were previously requested, participants received one vial of each material. These sample materials were shipped on dry ice to participants in July 1987. The communication materials included in the sample shipment are described in Appendix D.

Participants were requested to report values for retinol, α-tocopherol, and total and β-carotene. The isomeric form of the β-carotene, total or *trans*, was not specified. Not all participants reported values for all target analytes.

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 1) describes revisions to the statistical summary data tables, 2) the design for Round Robin XII, and 3) a proposed way to clarify β-carotene reporting. Appendix E also includes the (revised) data and statistical summary tables. Appendix F lists the measurement results reported for RR11 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.

Appendix A. Shipping Package Inserts for RR09

Two items were attached to each package shipped to an RR09 participant:

- **Cover letter**. The original letter has been lost. It would have described the three ethanolic calibration solutions (solutions 7 to 9) and five lyophilized sample materials (sera 62 to 66) distributed for the study. Page A2 reproduces what appears to be a draft message about the use of the calibration solutions.

- **Datasheet**. Page A3 reproduces the report form.

Dear _____:

This shipment contains serum amples for RR IX and calibration solutions for beta-carotene, retinol and alpha-tocopherol measurements only. We are not concerning ourselves with the other analytes in this round robin. We want to learn whether interlaboratory measurement comparability would improve for these analytes if all the laboratories in the NBS/NCI program use identical calibration solutions in their analyses.

The calibration solutions were made from chromatographically purified (but not entirely purified) solutions of retinol and beta-carotene. The solutions were mixed, and then the alpha-tocopherol was added. The less concentrated solutions are dilutions of the most concentrated one. These solutions are to be employed specifically for determining the response factors you use for the RR IX-sample analyses. The concentrations assigned to these calibration solutions are correct enough for the purpose of this round robin. They could also serve as a rough check on the calibration solutions you prepare yourself. (However, we prefer that you not consider them to be reference calibration materials.)

The serum samples are similar to those you analyzed for RR VIII. They require the addition of 1.2 mL of water for reconstitution.

After the results from RR VIII were sent to everyone, some labs were sent additional RR VIII samples to measure, for them to use to find out why some of their results were 50% or more from the consensus. They were asked to telephone or write to let us know their new results. If yours is one of the few labs that has not yet responded, please do so before you run the samples for RR IX.

Results in mg/L

Based on NBS Calibration Solutions

Samples	Result 1	Result 2

Serum No: 62   Retinol
 	
                β-Carotene
 	
Analysis Date   α-Tocopherol
 	
   / /87		

Serum No: 63   Retinol
 	
                β-Carotene
 	
Analysis Date   α-Tocopherol
 	
   / /87		

Serum No: 64   Retinol
 	
                β-Carotene
 	
Analysis Date   α-Tocopherol
 	
   / /87		

Serum No: 65   Retinol
 	
                β-Carotene
 	
Analysis Date   α-Tocopherol
 	
   / /87		

Serum No: 66   Retinol
 	
                β-Carotene
 	
Analysis Date   α-Tocopherol
 	
   / /87		

Note: Add 1.20 mL of water to reconstitute

   freeze-dried samples.

Appendix B. Final Report for RR09

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

- The reported results for retinol, α-tocopherol, and β-carotene. 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 RR09 results where the original codes have been altered to ensure confidentiality. Appendix C also provides more relevant summary statistics.

- The statistical summary table and its legend.

```
STANDARD NBS     ANALYTE=α-TOCOPHEROL   SERUM #062 GRAND AVG = 10.830; NBS=11.10
LAB   VIAL USED   RESULT1   RESULT2    MEAN     SD MEAN    % BIAS
      205  YES   10.400    10.000    10.200    0.200     -5.82
      195  YES   12.800    12.500    12.650    0.150     16.80
      235  YES   10.870    11.030    10.950    0.080      1.11
      217  YES    9.040     8.590     8.815    0.225    -18.61
      218  YES    9.880    10.000     9.940    0.060     -8.22
      228  YES   11.900    12.300    12.100    0.200     11.73
      150  YES   10.300    10.800    10.550    0.250     -2.59
      258  YES    9.690     9.170     9.430    0.260    -12.93
      133  YES    9.780    10.250    10.015    0.235     -7.53
      222  YES    9.900     9.600     9.750    0.150     -9.97
      214  YES   10.160    10.590    10.375    0.215     -4.20
      206  YES   14.377    13.944    14.161    0.217     30.75
      219  YES   10.270    10.400    10.335    0.065     -4.57
      131  YES    9.380     9.560     9.470    0.090    -12.56
      148  YES   10.400    10.600    10.500    0.100     -3.05
      229  YES    9.990    10.390    10.190    0.200     -5.91
      140  YES   10.149    10.049    10.099    0.050     -6.75
           YES   12.046    13.609    12.828    0.781     18.44
      116  YES   11.200    11.200    11.200    0.000      3.42
      123  YES   14.000    13.200    13.600    0.400     25.58
      246  YES   10.100    10.450    10.275    0.175     -5.13
NBS        NBS   11.100              11.100    0.000      2.49

STANDARD NBS     ANALYTE=α-TOCOPHEROL   SERUM #063 GRAND AVG =  7.107; NBS=6.67
LAB   VIAL USED   RESULT1   RESULT2    MEAN     SD MEAN    % BIAS
      215  YES    6.700     6.400     6.550    0.150     -7.83
      23   YES    8.300     8.500     8.400    0.100     18.20
      241  YES    6.870     6.950     6.910    0.040     -2.77
      235  YES    5.910     5.690     5.800    0.110    -18.39
      245  YES    6.670     6.930     6.800    0.130     -4.32
      242  YES    8.900     7.300     8.100    0.800     13.98
      138  YES    6.600     6.800     6.700    0.100     -5.72
      253  YES    6.320     5.730     6.025    0.295    -15.22
      134  YES    6.400     6.280     6.340    0.060    -10.79
      244  YES    6.200     6.200     6.200    0.000    -12.76
      199  YES    6.480     6.340     6.410    0.070     -9.80
      218  YES    9.433     9.805     9.619    0.186     35.35
      201  YES    6.790     6.960     6.875    0.085     -3.26
      109  YES    6.080     6.210     6.145    0.065    -13.53
      131  YES    7.100     6.800     6.950    0.150     -2.21
      210  YES    6.500     6.790     6.645    0.145     -6.50
      231  YES    6.594     6.686     6.640    0.046     -6.57
           YES    7.913     7.901     7.907    0.006     11.26
      118  YES    6.900     7.000     6.950    0.050     -2.21
      146  YES    9.800    10.800    10.300    0.500     44.93
      207  YES    7.200     6.750     6.975    0.225     -1.85
NBS        NBS    6.670               6.670    0.000     -6.15

STANDARD NBS     ANALYTE=α-TOCOPHEROL   SERUM #064 GRAND AVG =  4.858; NBS=4.75
LAB   VIAL USED   RESULT1   RESULT2    MEAN     SD MEAN    % BIAS
      199  YES    4.900     4.600     4.750    0.150     -2.23
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STANDARD NBS        ANALYTE=α-TOCOPHEROL   SERUM #064 GRAND AVG =  4.858; NBS=4.75
LAB   VIAL USED    RESULT1   RESULT2    MEAN     SD MEAN     % BIAS
      215  YES     5.300     6.000      5.650    0.350       16.30
      237  YES     5.000     4.900      4.950    0.050        1.89
      218  YES     4.140     4.070      4.105    0.035      -15.51
      220  YES     4.460     4.710      4.585    0.125       -5.63
      238  YES     6.000     7.600      6.800    0.800       39.97
      141  YES     4.400     4.700      4.550    0.150       -6.35
      228  YES     4.820     3.970      4.395    0.425       -9.54
      148  YES     4.520     4.270      4.395    0.125       -9.54
      211  YES     4.400     4.300      4.350    0.050      -10.46
      207  YES     4.440     4.540      4.490    0.050       -7.58
      229  YES     7.069     7.305      7.187    0.118       47.93
      204  YES     4.660     4.770      4.715    0.055       -2.95
      219  YES     3.560     3.690      3.625    0.065      -25.39
      124  YES     4.900     4.800      4.850    0.050       -0.17
      194  YES     4.300     4.140      4.220    0.080      -13.14
      139  YES     4.499     4.690      4.595    0.096       -5.43
           YES     5.579     5.581      5.580    0.001       14.85
      119  YES     4.400     4.400      4.400    0.000       -9.43
      158  OUT     9.000     9.500      9.250    0.250       90.39
      235  YES     5.150     4.800      4.975    0.175        2.40
NBS        NBS     4.750                4.750    0.000       -2.23

STANDARD NBS        ANALYTE=α-TOCOPHEROL   SERUM #065 GRAND AVG =  9.448; NBS=9.30
LAB   VIAL USED    RESULT1   RESULT2    MEAN     SD MEAN     % BIAS
      13   YES     9.000     8.900      8.950    0.050       -5.27
      244  YES     9.900     9.800      9.850    0.050        4.25
      04   YES     9.870     9.960      9.915    0.045        4.94
      246  YES     7.970     7.900      7.935    0.035      -16.02
      253  YES     9.150     9.190      9.170    0.020       -2.94
      248  YES     9.600    10.200      9.900    0.300        4.78
      188  YES     8.900     9.200      9.050    0.150       -4.21
      257  YES     8.380     8.150      8.265    0.115      -12.52
      178  YES     8.980     8.500      8.740    0.240       -7.50
      012  YES     8.700     8.400      8.550    0.150       -9.51
      217  YES    10.140     9.000      9.570    0.570        1.29
      006  YES    12.556    12.660     12.608    0.052       33.44
      249  YES     9.120     9.190      9.155    0.035       -3.10
      172  YES     8.270     7.820      8.045    0.225      -14.85
      065  YES    10.100    10.000     10.050    0.050        6.37
      236  YES     8.410     8.260      8.335    0.075      -11.78
      166  YES     8.993     9.734      9.363    0.371       -0.90
           YES    10.395    10.376     10.386    0.010        9.92
      195  YES     9.000     8.800      8.900    0.100       -5.80
      131  YES    11.500    12.200     11.850    0.350       25.42
      232  YES    10.050     9.600      9.825    0.225        3.99
NBS        NBS     9.300                9.300    0.000       -1.57

STANDARD NBS        ANALYTE=α-TOCOPHEROL   SERUM #066 GRAND AVG =  8.849; NBS=8.07
LAB   VIAL USED    RESULT1   RESULT2    MEAN     SD MEAN     % BIAS
      240  YES     8.400     8.300      8.350    0.050       -5.63
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STANDARD NBS        ANALYTE=α-TOCOPHEROL   SERUM #066 GRAND AVG =   8.849; NBS=8.07
LAB   VIAL USED     RESULT1   RESULT2     MEAN     SD MEAN      % BIAS
      245  YES      10.600     9.400     10.000     0.600      13.01
      233  YES       8.970     9.090      9.030     0.060       2.05
      242  YES       6.640     7.180      6.910     0.270     -21.91
      239  YES       8.550     7.700      8.125     0.425      -8.18
      249  YES      10.200     9.300      9.750     0.450      10.19
      178  YES       8.000     8.400      8.200     0.200      -7.33
      230  YES       8.480     7.340      7.910     0.570     -10.61
      136  YES       8.090     8.270      8.180     0.090      -7.56
      225  YES       7.900     8.100      8.000     0.100      -9.59
      218  YES       8.980     8.600      8.790     0.190      -0.66
      234  YES      12.251    12.166     12.209     0.043      37.97
      215  YES       8.480     8.470      8.475     0.005      -4.22
      142  YES       8.230     7.640      7.935     0.295     -10.32
      150  YES       9.100     9.400      9.250     0.150       4.54
      205  YES       8.410     7.510      7.960     0.450     -10.04
      134  YES       7.977     8.169      8.073     0.096      -8.77
           YES       9.732    10.116      9.924     0.192      12.15
      138  YES       7.900     8.000      7.950     0.050     -10.16
      171  YES      12.000    11.200     11.600     0.400      31.09
      228  YES       9.150     9.250      9.200     0.050       3.97
NBS        NBS       8.070                8.070     0.000      -8.80


STANDARD NBS        ANALYTE=β-CAROTENE   SERUM #062 GRAND AVG =   0.155; NBS=0.137
LAB   VIAL USED     RESULT1   RESULT2     MEAN     SD MEAN      % BIAS
      201  YES       0.146     0.143      0.145     0.002      -6.92
      205  YES       0.140     0.140      0.140     0.000      -9.81
      195  YES       0.213     0.196      0.205     0.008      31.74
      115  YES       0.142     0.148      0.145     0.003      -6.59
      217  YES       0.149     0.145      0.147     0.002      -5.31
      218  YES       0.230     0.250      0.240     0.010      54.60
      150  YES       0.140     0.150      0.145     0.005      -6.59
      133  YES       0.170     0.170      0.170     0.000       9.51
      222  YES       0.155     0.159      0.157     0.002       1.14
      131  YES       0.140     0.140      0.140     0.000      -9.81
      148  YES       0.080     0.060      0.070     0.010     -54.91
      229  YES       0.145     0.145      0.145     0.000      -6.59
      245  YES       0.240     0.230      0.235     0.005      51.38
      207  YES       0.146     0.149      0.147     0.002      -4.98
      209  YES       0.173     0.177      0.175     0.002      12.73
           YES       0.138     0.149      0.144     0.005      -7.56
      116  YES       0.090     0.090      0.090     0.000     -42.02
NBS        NBS       0.137                0.137     0.000     -11.75


STANDARD NBS        ANALYTE=β-CAROTENE   SERUM #063 GRAND AVG =   0.960; NBS=0.877
LAB   VIAL USED     RESULT1   RESULT2     MEAN     SD MEAN      % BIAS
      222  YES       0.986     0.904      0.945     0.041      -1.56
      215  YES       0.840     0.820      0.830     0.010     -13.54
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STANDARD NBS     ANALYTE=β-CAROTENE  SERUM #063 GRAND AVG =  0.960; NBS=0.877
LAB   VIAL USED  RESULT1  RESULT2   MEAN    SD MEAN    % BIAS
      223  YES    1.286    1.241    1.264    0.023      31.62
      130  YES    1.292    1.131    1.212    0.081      26.20
      224  YES    0.883    0.874    0.879    0.005      -8.49
      235  YES    0.886    0.859    0.873    0.014      -9.11
      245  YES    0.830    0.780    0.805    0.025     -16.14
      138  YES    0.890    0.910    0.900    0.010      -6.25
      134  YES    1.040    1.070    1.055    0.015       9.90
      244  YES    1.093    1.061    1.077    0.016      12.19
      109  YES    0.970    0.980    0.975    0.005       1.57
      131  OUT    0.320    0.350    0.335    0.015     -65.10
      210  YES    0.832    0.870    0.851    0.019     -11.35
      229  YES    1.380    1.180    1.280    0.100      33.34
      257  YES    0.899    1.002    0.951    0.051      -0.99
      212  YES    0.969    0.978    0.974    0.004       1.41
           YES    0.914    0.919    0.917    0.003      -4.53
      118  YES    0.540    0.530    0.535    0.005     -44.27
NBS        NBS    0.877             0.877    0.000      -8.64

STANDARD NBS     ANALYTE=β-CAROTENE  SERUM #064 GRAND AVG =  0.067; NBS=0.070
LAB   VIAL USED  RESULT1  RESULT2   MEAN    SD MEAN    % BIAS
      201  YES    0.066    0.062    0.064    0.002      -4.58
      199  YES    0.070    0.070    0.070    0.000       4.37
      215  YES    0.089    0.082    0.086    0.004      27.47
      122  YES    0.044    0.050    0.047    0.003     -29.93
      138  YES    0.064    0.058    0.061    0.003      -9.05
      218  YES    0.076    0.073    0.075    0.001      11.07
      220  YES    0.080    0.080    0.080    0.000      19.27
      141  YES    0.062    0.057    0.060    0.002     -11.29
      148  YES    0.060    0.070    0.065    0.005      -3.09
      211  YES    0.071    0.074    0.073    0.002       8.09
      219  YES    0.050    0.060    0.055    0.005     -18.00
      124  YES    0.060    0.050    0.055    0.005     -18.00
      194  YES    0.051    0.062    0.057    0.006     -15.76
      195  YES    0.100    0.090    0.095    0.005      41.64
      209  YES    0.063    0.060    0.061    0.001      -8.61
      197  YES    0.095    0.097    0.096    0.001      43.13
           YES    0.070    0.069    0.070    0.001       3.62
      119  YES    0.040    0.040    0.040    0.000     -40.36
NBS        NBS    0.070             0.070    0.000       4.37

STANDARD NBS     ANALYTE=β-CAROTENE  SERUM #065 GRAND AVG =  0.406; NBS=0.376
LAB   VIAL USED  RESULT1  RESULT2   MEAN    SD MEAN    % BIAS
      254  YES    0.472    0.436    0.454    0.018      11.76
      13   YES    0.390    0.390    0.390    0.000      -3.99
      244  YES    0.496    0.523    0.510    0.013      25.42
      171  YES    0.452    0.472    0.462    0.010      13.73
      186  YES    0.411    0.416    0.414    0.002       1.79
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STANDARD NBS ANALYTE=β-CAROTENE SERUM #065 GRAND AVG = 0.406; NBS=0.376

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	246	YES	0.399	0.396	0.398	0.002	-2.15
	253	YES	0.390	0.380	0.385	0.005	-5.22
	188	YES	0.370	0.390	0.380	0.010	-6.46
	178	YES	0.450	0.460	0.455	0.005	12.01
	012	YES	0.458	0.444	0.451	0.007	11.02
	172	YES	0.420	0.420	0.420	0.000	3.39
	065	YES	0.180	0.190	0.185	0.005	-54.46
	236	YES	0.371	0.328	0.350	0.022	-13.96
	256	YES	0.520	0.590	0.555	0.035	36.62
	009	YES	0.426	0.458	0.442	0.016	8.81
	255	YES	0.437	0.447	0.442	0.005	8.81
		YES	0.389	0.393	0.391	0.002	-3.75
	195	YES	0.230	0.230	0.230	0.000	-43.38
NBS		NBS	0.376		0.376	0.000	-7.44

STANDARD NBS ANALYTE=β-CAROTENE SERUM #066 GRAND AVG = 1.491; NBS=1.210

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	206	YES	1.708	1.564	1.636	0.072	9.73
	240	YES	1.250	1.240	1.245	0.005	-16.49
	245	YES	1.897	1.915	1.906	0.009	27.84
	162	YES	1.844	1.824	1.834	0.010	23.02
	180	YES	1.372	1.347	1.360	0.012	-8.81
	242	YES	1.204	1.288	1.246	0.042	-16.42
	239	YES	1.160	1.160	1.160	0.000	-22.19
	178	YES	1.330	1.400	1.365	0.035	-8.44
	136	YES	1.520	1.500	1.510	0.010	1.28
	225	YES	1.436	1.555	1.496	0.060	0.31
	142	YES	1.630	1.480	1.555	0.075	4.30
	150	OUT	0.470	0.510	0.490	0.020	-67.13
	205	YES	1.260	1.210	1.235	0.025	-17.16
	209	YES	1.820	2.040	1.930	0.110	29.45
	254	YES	1.405	1.414	1.410	0.005	-5.46
	221	YES	1.554	1.571	1.563	0.008	4.80
		YES	1.408	1.402	1.405	0.003	-5.76
	138	OUT	0.550	0.530	0.540	0.010	-63.78
NBS		NBS	1.210		1.210	0.000	-18.84

STANDARD NBS ANALYTE=RETINOL SERUM #062 GRAND AVG = 0.299; NBS=0.327

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	211	YES	0.261	0.240	0.251	0.011	-16.14
	205	YES	0.330	0.330	0.330	0.000	10.47
	195	YES	0.370	0.371	0.371	0.001	24.03
		YES	0.279	0.264	0.272	0.008	-9.11
	230	OUT	0.060	0.070	0.065	0.005	-78.24
	235	YES	0.329	0.331	0.330	0.001	10.47
	217	YES	0.280	0.269	0.275	0.005	-8.11
	218	YES	0.375	0.372	0.374	0.002	25.03

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STANDARD NBS      ANALYTE=RETINOL   SERUM #062 GRAND AVG =  0.299; NBS=0.327
LAB   VIAL USED   RESULT1  RESULT2   MEAN    SD MEAN    % BIAS
      228  YES    0.275    0.250    0.263    0.013    -12.13
      150  YES    0.250    0.280    0.265    0.015    -11.29
      258  YES    0.260    0.260    0.260    0.000    -12.96
      133  YES    0.250    0.250    0.250    0.000    -16.31
      222  YES    0.260    0.260    0.260    0.000    -12.96
      214  YES    0.225    0.227    0.226    0.001    -24.35
      206  YES    0.374    0.376    0.375    0.001     25.53
      131  YES    0.310    0.310    0.310    0.000      3.77
      148  YES    0.340    0.360    0.350    0.010     17.16
      229  YES    0.282    0.337    0.310    0.027      3.61
      106  OUT    0.500    0.490    0.495    0.005     65.70
      207  YES    0.321    0.330    0.326    0.005      8.96
      140  YES    0.272    0.273    0.273    0.000     -8.78
           YES    0.376    0.375    0.376    0.001     25.70
      116  YES    0.250    0.250    0.250    0.000    -16.31
      123  YES    0.290    0.270    0.280    0.010     -6.27
NBS        NBS    0.327             0.327    0.000      9.46

STANDARD NBS      ANALYTE=RETINOL   SERUM #063 GRAND AVG =  0.856; NBS=0.910
LAB   VIAL USED   RESULT1  RESULT2   MEAN    SD MEAN    % BIAS
      234  YES    0.667    0.680    0.674    0.007    -21.32
      215  YES    0.940    0.950    0.945    0.005     10.39
      223  YES    1.081    1.035    1.058    0.023     23.59
      130  YES    0.842    0.841    0.842    0.001     -1.70
      256  OUT    0.420    0.400    0.410    0.010    -52.10
      241  YES    0.948    0.951    0.950    0.001     10.92
      235  YES    0.795    0.770    0.783    0.012     -8.59
      245  YES    0.863    0.899    0.881    0.018      2.92
      242  YES    0.870    0.905    0.888    0.017      3.68
      138  YES    0.790    0.830    0.810    0.020     -5.38
      253  YES    0.800    0.800    0.800    0.000     -6.54
      134  YES    0.700    0.700    0.700    0.000    -18.23
      244  YES    0.760    0.780    0.770    0.010    -10.05
      199  YES    0.861    0.888    0.875    0.014      2.16
      218  YES    0.947    0.904    0.926    0.022      8.12
      109  YES    0.960    0.970    0.965    0.005     12.73
      131  YES    0.980    1.000    0.990    0.010     15.65
      210  YES    0.865    0.908    0.887    0.022      3.56
      150  OUT    1.400    1.410    1.405    0.005     64.13
      257  YES    0.872    0.906    0.889    0.017      3.85
      231  YES    0.763    0.763    0.763    0.000    -10.87
           YES    0.996    0.985    0.991    0.006     15.71
      118  YES    0.720    0.730    0.725    0.005    -15.31
      146  YES    0.730    0.720    0.725    0.005    -15.31
NBS        NBS    0.910             0.910    0.000      6.31

STANDARD NBS      ANALYTE=RETINOL   SERUM #064 GRAND AVG =  0.540; NBS=0.597
LAB   VIAL USED   RESULT1  RESULT2   MEAN    SD MEAN    % BIAS
      212  YES    0.547    0.561    0.554    0.007      2.54
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STANDARD NBS        ANALYTE=RETINOL   SERUM #064 GRAND AVG =  0.540; NBS=0.597
LAB   VIAL USED    RESULT1   RESULT2    MEAN     SD MEAN     % BIAS
      199  YES     0.620     0.620      0.620    0.000       14.75
      215  YES     0.679     0.656      0.668    0.012       23.55
      122  YES     0.544     0.559      0.552    0.007        2.08
      205  YES     0.390     0.400      0.395    0.005      -26.89
      237  YES     0.558     0.563      0.561    0.003        3.74
      218  YES     0.510     0.489      0.500    0.010       -7.55
      220  YES     0.609     0.642      0.626    0.016       15.77
      238  YES     0.440     0.485      0.463    0.023      -14.40
      141  YES     0.510     0.540      0.525    0.015       -2.83
      228  YES     0.490     0.500      0.495    0.005       -8.38
      148  YES     0.490     0.480      0.485    0.005      -10.23
      211  YES     0.460     0.510      0.485    0.025      -10.23
      207  YES     0.567     0.569      0.568    0.001        5.13
      229  YES     0.597     0.593      0.595    0.002       10.13
      219  YES     0.560     0.590      0.575    0.015        6.43
      124  YES     0.690     0.670      0.680    0.010       25.86
      194  YES     0.579     0.592      0.586    0.007        8.37
      133  OUT     0.930     0.930      0.930    0.000       72.13
      209  YES     0.590     0.550      0.570    0.020        5.50
      139  YES     0.498     0.500      0.499    0.001       -7.64
           YES     0.639     0.687      0.663    0.024       22.71
      119  YES     0.440     0.450      0.445    0.005      -17.64
      158  YES     0.260     0.380      0.320    0.060      -40.77
NBS        NBS     0.597                0.597    0.000       10.50

STANDARD NBS        ANALYTE=RETINOL   SERUM #065 GRAND AVG =  0.440; NBS=0.479
LAB   VIAL USED    RESULT1   RESULT2    MEAN     SD MEAN     % BIAS
      251  YES     0.438     0.404      0.421    0.017       -4.38
      13   YES     0.490     0.490      0.490    0.000       11.29
      244  YES     0.499     0.529      0.514    0.015       16.74
      171  YES     0.448     0.394      0.421    0.027       -4.38
      226  YES     0.310     0.300      0.305    0.005      -30.73
      04   YES     0.493     0.524      0.508    0.016       15.49
      246  YES     0.416     0.407      0.412    0.005       -6.54
      253  YES     0.490     0.510      0.500    0.010       13.56
      248  YES     0.395     0.395      0.395    0.000      -10.29
      188  YES     0.410     0.430      0.420    0.010       -4.61
      257  YES     0.410     0.410      0.410    0.000       -6.88
      178  YES     0.420     0.410      0.415    0.005       -5.75
      012  YES     0.390     0.390      0.390    0.000      -11.42
      217  YES     0.419     0.392      0.406    0.014       -7.90
      006  YES     0.500     0.503      0.502    0.002       13.90
      172  YES     0.480     0.460      0.470    0.010        6.74
      065  YES     0.550     0.540      0.545    0.005       23.78
      236  YES     0.473     0.474      0.474    0.000        7.54
      132  OUT     0.750     0.750      0.750    0.000       70.34
      009  YES     0.492     0.485      0.489    0.004       10.95
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STANDARD NBS      ANALYTE=RETINOL   SERUM #065 GRAND AVG =  0.440; NBS=0.479
LAB   VIAL USED   RESULT1  RESULT2   MEAN    SD MEAN     % BIAS
      166  YES     0.397    0.383    0.390    0.007     -11.42
           YES     0.519    0.515    0.517    0.002      17.42
      195  YES     0.360    0.360    0.360    0.000     -18.24
      131  YES     0.400    0.350    0.375    0.025     -14.83
NBS        NBS     0.479             0.479    0.000       8.79


STANDARD NBS      ANALYTE=RETINOL   SERUM #066 GRAND AVG =  0.775; NBS=0.811
LAB   VIAL USED   RESULT1  RESULT2   MEAN    SD MEAN     % BIAS
      247  YES     0.661    0.698    0.680    0.019     -12.36
      240  YES     0.850    0.850    0.850    0.000       9.63
      245  YES     0.893    0.897    0.895    0.002      15.43
      162  YES     0.770    0.752    0.761    0.009      -1.85
      210  OUT     0.240    0.250    0.245    0.005     -68.40
      233  YES     0.827    0.847    0.837    0.010       7.95
      242  YES     0.639    0.696    0.668    0.028     -13.91
      239  YES     0.770    0.776    0.773    0.003      -0.30
      249  YES     0.645    0.640    0.643    0.003     -17.13
      178  YES     0.700    0.740    0.720    0.020      -7.14
      230  YES     0.800    0.710    0.755    0.045      -2.62
      136  YES     0.640    0.660    0.650    0.010     -16.17
      225  YES     0.710    0.670    0.690    0.020     -11.01
      218  YES     0.854    0.876    0.865    0.011      11.56
      234  YES     0.830    0.812    0.821    0.009       5.89
      142  YES     0.910    0.880    0.895    0.015      15.43
      150  YES     0.950    0.990    0.970    0.020      25.11
      205  YES     0.805    0.750    0.778    0.028       0.28
      143  OUT     1.260    1.260    1.260    0.000      62.51
      254  YES     0.823    0.775    0.799    0.024       3.05
      134  YES     0.695    0.673    0.684    0.011     -11.78
           YES     0.962    1.029    0.996    0.033      28.40
      138  YES     0.650    0.650    0.650    0.000     -16.17
      171  YES     0.660    0.700    0.680    0.020     -12.30
NBS        NBS     0.811             0.811    0.000       4.60
```

Summary of RR IX Results
"Fat Soluble Vitamins in Serum"

STANDARD NBS   ANALYTE=α-TOCOPHEROL

SER NBS	GRAND AVG	S.E. AVG	S WITHIN	S BETWEEN	% CV AVG	% CV X	FR REJECT
064 4.75	4.858	0.193	0.331	0.831	4.0	18.4	1/21
063 6.67	7.107	0.252	0.339	1.130	3.5	16.6	0/21
066 8.07	8.849	0.279	0.410	1.244	3.2	14.8	0/21
065 9.30	9.448	0.253	0.296	1.139	2.7	12.5	0/21
062 11.1	10.830	0.311	0.356	1.403	2.9	13.4	0/21

STANDARD NBS   ANALYTE=β-CAROTENE

SER NBS	GRAND AVG	S.E. AVG	S WITHIN	S BETWEEN	% CV AVG	% CV X	FR REJECT
064 0.070	0.067	0.004	0.004	0.015	5.3	23.1	0/18
062 0.137	0.155	0.010	0.007	0.043	6.7	27.7	0/17
065 0.376	0.406	0.021	0.018	0.087	5.1	21.8	0/18
063 0.877	0.960	0.044	0.052	0.179	4.6	19.4	1/18
066 1.210	1.491	0.060	0.062	0.235	4.0	16.3	2/18

STANDARD NBS   ANALYTE=RETINOL

SER NBS	GRAND AVG	S.E. AVG	S WITHIN	S BETWEEN	% CV AVG	% CV X	FR REJECT
062 0.327	0.299	0.010	0.012	0.047	3.4	16.3	2/24
065 0.479	0.440	0.013	0.015	0.060	2.9	14.1	1/24
064 0.597	0.540	0.018	0.025	0.086	3.4	16.6	1/24
066 0.811	0.775	0.022	0.027	0.104	2.9	13.8	2/24
063 0.910	0.856	0.022	0.018	0.105	2.6	12.4	2/24

Explanation of Column Heading on <u>Statistical Summary</u>

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

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

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

S BETWEEN = between laboratory component of standard deviation = S_b

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

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

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

Appendix C. Updated "All-Lab Report" for RR09

The following three pages are modernized "All-Lab" report for RR09. This report has three parts:

- Page 1 lists results for all analytes reported.
- Page 2 provides the legend for page 1.
- Page 3 summarizes each participants' performance for retinol, α-tocopherol, and β-carotene, using the "Comparability Summary" calculations used 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 RR09. The only attributed results are those reported by NIST. The NIST results are not used in the assessment of the consensus summary results of the study.

Round Robin IX Laboratory Results

Lab	Total Retinol, µg/mL 62	63	64	65	66	α-Tocopherol, µg/mL 62	63	64	65	66	Total β-Carotene, µg/mL 62	63	64	65	66
FSV-BA	0.273	0.763	0.499	0.390	0.684	10.10	6.64	4.59	9.36	8.07	0.175	0.974	0.096	0.442	1.563
FSV-BD	0.263	0.888	0.463	0.395	0.643	12.10	8.10	6.80	9.90	9.75					
FSV-BF	0.260	0.770	0.485	0.390	0.690	9.75	6.20	4.35	8.55	8.00	0.157	1.077	0.073	0.451	1.496
FSV-BG	0.371	1.058	0.668	0.514	0.895	12.65	8.40	5.65	9.85	10.00	0.205	1.264	0.086	0.510	1.906
FSV-BI	0.330	0.945	0.620	0.490	0.850	10.20	6.55	4.75	8.95	8.35	0.140	0.830	0.070	0.390	1.245
FSV-BX	0.250	0.725	0.445	0.360	0.650	11.20	6.95	4.40	8.90	7.95	0.090	0.535	0.040	0.230	0.540
FSV-BY	0.251	0.674	0.554	0.421	0.680						0.145	0.945	0.064	0.454	1.636
FSV-BZ	0.280	0.725	0.320	0.375	0.680	13.60	10.30	9.25	11.85	11.60					
FSV-CA	0.330	0.950	0.561	0.509	0.837	10.95	6.91	4.95	9.92	9.03					
FSV-CJ	0.290	0.956	0.642	0.438	1.251	11.30	7.43	5.20	10.08	9.32	0.145	0.879	0.061	0.414	1.360
FSV-CK	0.376	0.991	0.663	0.517	0.996	12.83	7.91	5.58	10.39	9.92	0.144	0.917	0.070	0.391	1.405
FSV-CL	0.250	0.700	0.485	0.415	0.650	10.02	6.34	4.40	8.74	8.18	0.170	1.055	0.065	0.455	1.510
FSV-CO	0.275	0.783	0.500	0.412	0.668	8.82	5.80	4.11	7.94	6.91	0.147	0.873	0.075	0.398	1.246
FSV-DC	0.495	1.405	0.930	0.750	1.260						0.235	1.280	0.095	0.555	1.930
FSV-DE						10.34	6.88	4.72	9.16	8.48					
FSV-DG	0.310	0.965	0.575	0.470	0.895	9.47	6.15	3.63	8.05	7.94	0.140	0.975	0.055	0.420	1.555
FSV-DH	0.260	0.800	0.495	0.410	0.755	9.43	6.03	4.40	8.27	7.91					
FSV-DN	0.265	0.810	0.525	0.420	0.720	10.55	6.70	4.55	9.05	8.20	0.145	0.900	0.060	0.380	1.365
FSV-DO	0.065	0.410	0.395	0.305	0.245										
FSV-DT	0.226	0.875	0.568	0.406	0.865	10.38	6.41	4.49	9.57	8.79					
FSV-DZ	0.391	1.200	0.728	0.590	0.935	13.75	8.51	5.41	11.35	9.75					
FSV-ED	0.319	0.872	0.590	0.458	0.812	9.80	5.63	4.07	8.47	7.73	0.133	0.721	0.059	0.358	1.222
FSV-EG	0.272	0.842	0.550	0.421	0.765						0.120	1.212	0.047	0.462	1.834
FSV-EO	0.326	0.889	0.570	0.489	0.799						0.148	0.951	0.061	0.442	1.410
FSV-EP	0.375	0.926	0.595	0.502	0.821	14.16	9.62	7.19	12.61	12.21					
FSV-ER	0.374	0.881	0.626	0.500	0.773	9.94	6.80	4.59	9.17	8.13	0.240	0.805	0.080	0.385	1.160
FSV-EU	0.350	0.990	0.680	0.545	0.970	10.50	6.95	4.85	10.05	9.25	0.070	0.335	0.055	0.185	0.490
FSV-EW	0.310	0.887	0.586	0.474	0.778	10.19	6.65	4.22	8.34	7.96	0.145	0.851	0.057	0.350	1.235
FSV-FI						10.28	6.98	4.98	9.83	9.20					
n	27	27	27	27	27	24	24	24	24	24	19	19	19	19	19
Min	0.065	0.410	0.320	0.305	0.245	8.82	5.63	3.63	7.94	6.91	0.070	0.335	0.040	0.185	0.490
Median	0.290	0.881	0.568	0.438	0.778	10.36	6.84	4.65	9.27	8.41	0.145	0.917	0.064	0.414	1.405
Max	0.495	1.405	0.930	0.750	1.260	14.16	10.30	9.25	12.61	12.21	0.240	1.280	0.096	0.555	1.930
eSD	0.059	0.125	0.102	0.071	0.145	0.89	0.81	0.46	1.01	0.83	0.018	0.129	0.013	0.056	0.237
eCV	20	14	18	16	19	9	12	10	11	10	12	14	20	13	17
NISTa	0.332	0.900	0.558	0.501	0.781	10.01	6.37	4.29	8.46	7.53	0.129	0.793	0.070	0.389	1.104
NISTb	0.349	0.923	0.613	0.499	0.832	12.26	7.11	5.15	10.07	8.45	0.154	1.000	0.074	0.412	1.410
NNIST	4	4	4	4	4	4	4	4	4	4	4	4	4	4	4
Mean$_{NIST}$	0.340	0.912	0.585	0.500	0.806	11.14	6.74	4.72	9.27	7.99	0.141	0.898	0.072	0.389	1.258
NAV	0.315	0.896	0.577	0.469	0.792	10.75	6.79	4.69	9.27	8.20	0.143	0.907	0.068	0.401	1.331
NAU	0.069	0.126	0.103	0.084	0.146	1.69	0.81	0.61	1.15	0.88	0.023	0.158	0.014	0.091	0.259

C2

Round Robin IX Laboratory Results

Analytes Reported By One Laboratory
Values in µg/mL

Analyte	Code	62	63	64	65	66
Total cis-β-Carotene	NISTa	0.013	0.094	0.002	0.035	0.110
trans-β-Carotene	NISTa	0.141	0.906	0.072	0.377	1.300

Table Legend [a]

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	Adjusted median absolute deviation from the median of the non-NIST results
eCV	Coefficient of Variation for (non-NIST) results: 100*SD/Median
N_{NIST}	Number of results reported by NIST (NBS) analysts
$Mean_{NIST}$	Mean of NIST results
NAV	NIST Assigned Value: (Median + NIST)/2
NAU	NIST Assigned Uncertainty: $\sqrt{(S^2 + S_{btw}^2)}$
	S is the maximum of (0.05*NAV, eSD, SD_{LT}) and S_{btw} is the standard deviation between Median and $Mean_{NIST}$. The expected long-term SD, SD_{LT}, is defined in: Duewer et al., Anal Chem 1997;69(7):1406-1413.
a	What is now "NIST" was the National Bureau of Standards (NBS) when this study was conducted.

C3

Round Robin IX Laboratory Results

Comparability Summary

Lab	TR	aT	bC
FSV-BA	1	1	2
FSV-BD	1	2	
FSV-BF	1	1	1
FSV-BG	2	2	2
FSV-BI	1	1	1
FSV-BX	1	1	3
FSV-BY	1		1
FSV-BZ	2	4	
FSV-CA	1	1	
FSV-CJ	2	1	1
FSV-CK	2	2	1
FSV-CL	1	1	1
FSV-CO	1	2	1
FSV-DC	4		3
FSV-DE		1	
FSV-DG	1	2	1
FSV-DH	1	1	
FSV-DN	1	1	1
FSV-DO	3		
FSV-DT	1	1	
FSV-DZ	2	2	
FSV-ED	1	1	1
FSV-EG	1		2
FSV-EO	1		1
FSV-EP	1	4	
FSV-ER	1	1	3
FSV-EU	2	1	3
FSV-EW	1	1	1
FSV-FI		1	
NISTa	1	1	1
NISTb	1	1	1
n	29	26	21

	TR	aT	bC
% 1	72	69	67
% 2	21	23	14
% 3	3	0	19
% 4	3	8	0

Label	Definition
Lab	Participant code
TR	Total Retinol
aT	α-Tocopherol
bC	β-Carotene
n	Number of values
n	number of participants providing quantitative data
% 1	Percent of CS = 1 (within 1 SD of medians)
% 2	Percent of CS = 2 (within 2 SD of medians)
% 3	Percent of CS = 3 (within 3 SD of medians)
% 4	Percent of CS = 4 (3 or more SD from medians)

"Comparability Score"

The Comparability Score (CS) summarizes your measurement performance for a given analyte relative to the consensus medians in this study. CS is the average distance (in units of standard deviation) of your measurement performance characteristics from the consensus performance. CS is calculated when the number of quantitative values you reported, N_{you}, is at least two and at least six participants reported quantitative values for the analyte.

We define CS as follows:

$$CS = \text{MINIMUM}\left(4, \text{INTEGER}\left(1+\sqrt{C^2 + AP^2}\right)\right)$$

$$C = \text{Concordance} = \frac{\sum_{i=1}^{N_{you}} \frac{You_i - Median_i}{NAU_i}}{N_{you}}$$

$$AP = \text{Apparent Precision} = \sqrt{\frac{\sum_{i=1}^{N_{you}}\left(\frac{You_i - Median_i}{NAU_i}\right)^2}{N_{you} - 1}}$$

NAU = NIST Assigned Uncertainty

For further details, please see
Duewer DL, Kline MC, Sharpless KE, Brown Thomas J, Gary KT. Micronutrients Measurement Quality Assurance Program: Helping participants use interlaboratory comparison exercise results to improve their long-term measurement performance. Anal Chem 1999;71(9):1870-8.

C4

Appendix D. Shipping Package Inserts for RR11

Two items were attached to each package shipped to an RR11 participant:

- **Cover letter**. The original letter as attached to the packages has been lost.
- **Datasheet**. Page D2 reproduces the form.

DATE OF ANALYSIS _____

RESULTS IN mg/L

```
========================================================================
            SAMPLES                    RESULT 1        RESULT 2
========================================================================
Serum 72    RETINOL
            ----------------------------------------------------
VIAL #____   B-CAROTENE
            ----------------------------------------------------
            A-TOCOPHEROL
========================================================================
Serum 73    RETINOL
            ----------------------------------------------------
VIAL #____   B-CAROTENE
            ----------------------------------------------------
            A-TOCOPHEROL
========================================================================
Serum 74    RETINOL
            ----------------------------------------------------
VIAL #____   B-CAROTENE
            ----------------------------------------------------
            A-TOCOPHEROL
========================================================================
Serum 75    RETINOL
            ----------------------------------------------------
VIAL #____   B-CAROTENE
            ----------------------------------------------------
            A-TOCOPHEROL
========================================================================
Serum 76    RETINOL
            ----------------------------------------------------
VIAL #____   B-CAROTENE
            ----------------------------------------------------
            A-TOCOPHEROL
========================================================================
```

Appendix E. Final Report for RR11

The following 14 pages are the available fragments of the report for RR11 provided to participants. These fragments are:

- An individualized letter sent two months after the original letter that informed the participant of revisions to the summary table for β-carotene, identified a mechanism for chromatographically distinguishing total and *trans*-β-carotene, and discussed plans for the next study, Round Robin XII.

- The reported results for retinol, α-tocopherol, and β-carotene. 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 RR11 results where the original codes have been altered to ensure confidentiality. Appendix F also provides more relevant summary statistics.

- The statistical summary table and its legend.

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

December 29, 1987

Individualized
name and address

Dear First Name:

Enclosed is a revision of the statistical summary for RR-XI mailed to you in November. The table for beta-carotene had errors that were introduced while rearranging the individual sample results in the order of increasing concentration. The tables for retinol and alpha-tocopherol were o.k.

Round-Robin XII will consist of proficiency testing samples for the fat-soluble vitamins, ascorbic acid, and the metals (zinc and selenium). The samples should be shipped on January 11, 1988.
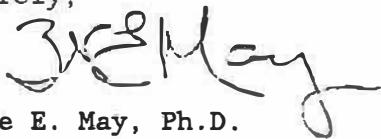
The samples for fat-soluble vitamin analyses are freeze-dried plasma. Data entry forms are provided and please note that these samples are to be reconstituted with 1.00 mL of water and that you are to report only one value for each sample. Results must arrive at NBS by February 29, 1988 to be included in our statistical summaries.

In the past we have discussed the dilemma that we face in evaluating beta-carotene results. NBS and a few other labs feel that they have been reporting beta-carotene data based on the concentration of the all-trans isomer only. The majority of labs measure a single peak that contains both the all-trans and the cis-isomers of beta-carotene. In order to facilitate the interlaboratory comparison of data, we request that all laboratories report a value for total beta-carotene and that labs also report concentrations for all trans beta-carotene where possible. Laboratories that use "polymerically-bound" ODS columns should report data for both all-trans and total beta-carotene. Total beta-carotene should be determined by summing the all-trans and cis-isomers. A chromatogram representative of the analysis of an NBS sample on a "polymerically-bound" column is provided in figure 1. Laboratories that use "monomerically-bound" ODS columns (the vast majority) should, as they have in the past, report only total beta-carotene concentrations. Figure 2 is a chromatogram representative of the analysis of an NBS sample on a "monomerically-bound" column. Please feel free to contact me (301/975-3108) or Neal Craft (301/975-3111) if you have questions.

The samples for ascorbic acid analyses and those for analysis of zinc and selenium are similar to those sent previously. The metal samples will be shipped as frozen liquids. The ascorbic acid samples are freeze-dried and should be reconstituted with 1.00 mL of water. The reporting format and data forms are as before, i.e. results for each sample should be reported in duplicate. Results for the samples must arrive at NBS by March 15, 1988 to be included in our statistical summaries.

Dr. Robert Schaffer will retire within a few weeks. He will, however, continue to serve as Clinical Coordinator for this program. You should continue to communicate and interact with him, as in the past, through his NBS address and telephone number. However, official correspondence from NBS to you will come from me.

Sincerely,

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

Enclosures

cc: R. Schaffer
      M. Kline

Fig 1

POLYMERICALLY-BOUND ODS COLUMN

Serum Extract

ABSORBANCE UNITS (10^{-3})

Trans (β-carotene)

α-carotene

cis-isomers of β-carotene

Lycopene

TIME (MIN)

Fig 2

MONOMERICALLY-BOUND ODS COLUMN

Serum Extract

β-carotene

Lycopene

α-carotene

ABSORBANCE UNITS (10^{-3})

10

5

0

TIME (MIN)

0   10   20   30   40   50   60   70   80
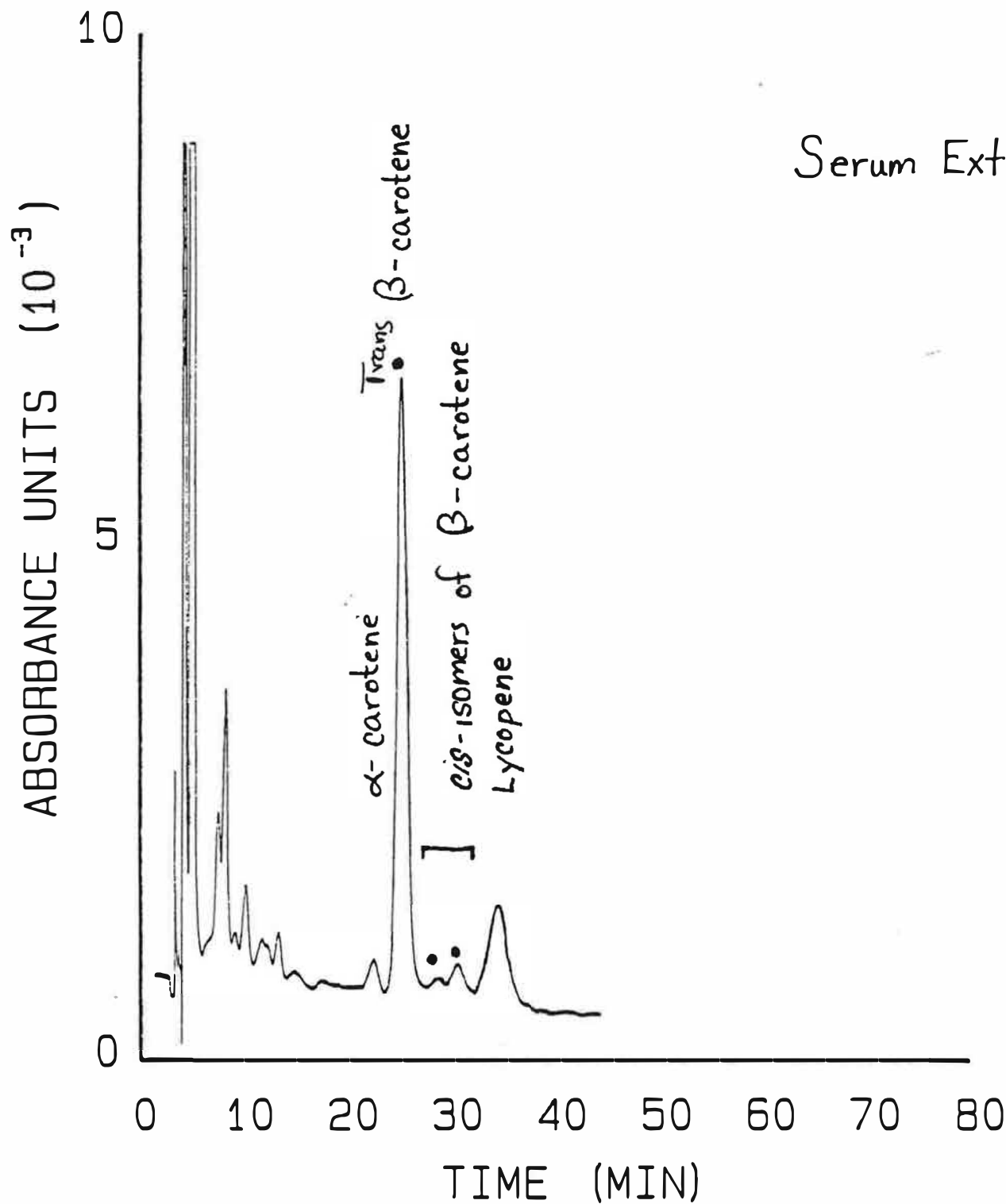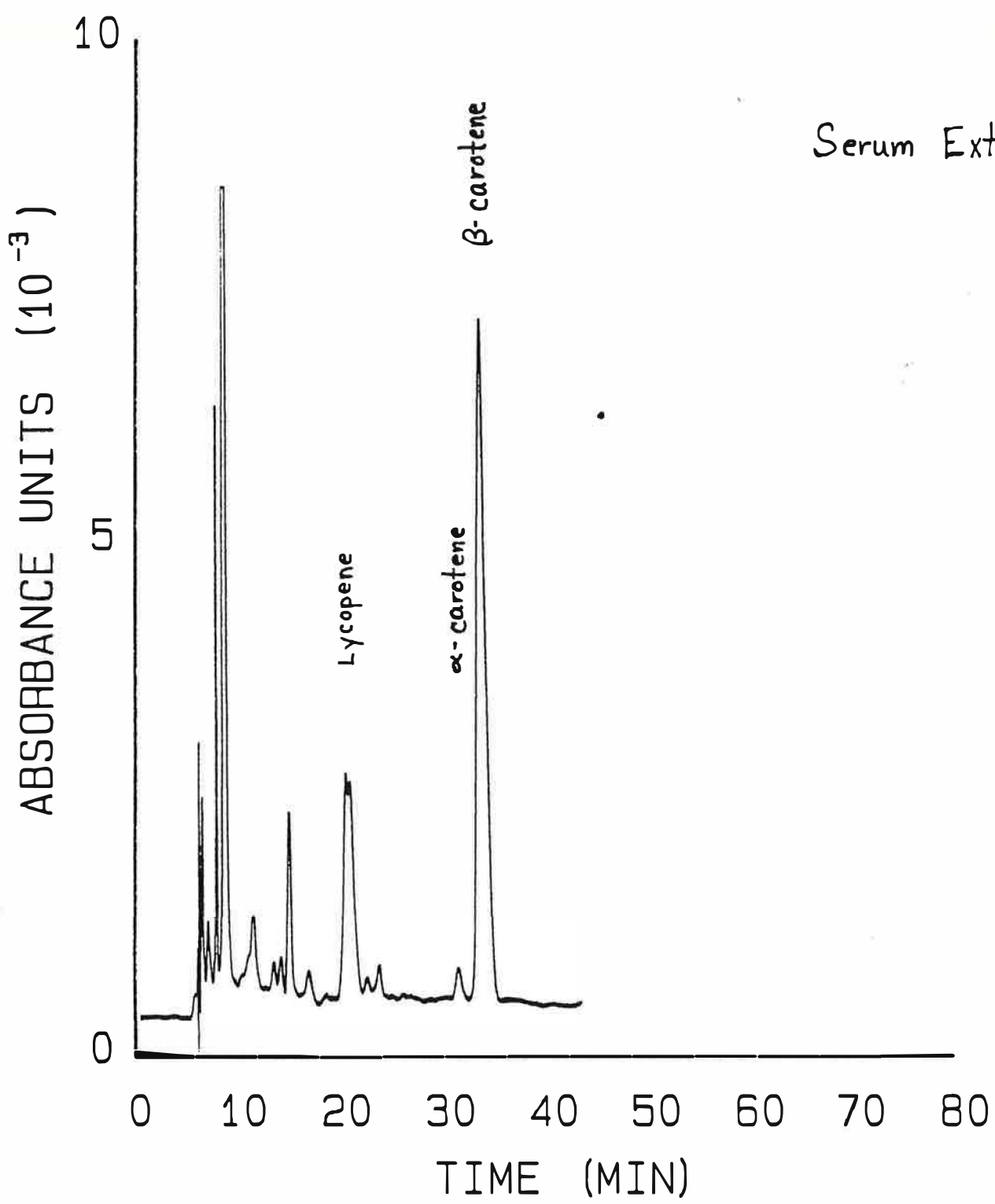
```
STANDARD LAB      ANALYTE=α-TOCOPHEROL   SERUM #72 GRAND AVG =   6.840; NBS = 6.76
LAB   VIAL USED    RESULT1    RESULT2    MEAN      SD MEAN     % BIAS
      48   YES     6.500      6.400      6.450     0.050       -5.70
      16   YES     9.000      8.800      8.900     0.100       30.12
      34   YES     6.920      6.780      6.850     0.070        0.15
      46   YES     6.640      6.630      6.635     0.005       -3.00
      02   YES     6.200      5.530      5.865     0.335      -14.25
      26   YES     6.700      6.600      6.650     0.050       -2.78
      14   OUT    11.400     11.800     11.600     0.200       69.59
      28   YES     6.690      6.490      6.590     0.100       -3.65
      06   YES     7.100      7.400      7.250     0.150        5.99
      21   YES     6.860      6.500      6.680     0.180       -2.34
      33   YES     6.250      6.360      6.305     0.055       -7.82
      23   YES     6.290      6.240      6.265     0.025       -8.41
      13   YES     6.050      6.570      6.310     0.260       -7.75
      12   OUT     8.550      4.710      6.630     1.920       -3.07
      11   YES     6.500      5.800      6.150     0.350      -10.09
      04   NFI     5.476                 5.476     0.000      -19.94
      27   YES     7.400      7.300      7.350     0.050        7.46
      15   YES     8.800      7.900      8.350     0.450       22.08




STANDARD LAB      ANALYTE=α-TOCOPHEROL   SERUM #73 GRAND AVG = 11.241; NBS = 10.87
LAB   VIAL USED    RESULT1    RESULT2    MEAN      SD MEAN     % BIAS
      08   YES    10.200     10.200     10.200     0.000       -9.26
      29   YES    14.300     15.900     15.100     0.800       34.33
      26   YES    11.640     11.780     11.710     0.070        4.17
      18   YES    10.460     10.790     10.625     0.165       -5.48
      08   YES     9.230      9.610      9.420     0.190      -16.20
      28   YES    11.700     11.100     11.400     0.300        1.41
      35   OUT    18.100     19.200     18.650     0.550       65.91
      20   YES    12.000     11.200     11.600     0.400        3.19
      27   YES    12.990     11.870     12.430     0.560       10.57
      24   YES    13.000     12.500     12.750     0.250       13.42
      22   YES    12.500     13.400     12.950     0.450       15.20
      04   YES    10.800     10.780     10.790     0.010       -4.01
      32   YES    10.390     10.390     10.390     0.000       -7.57
      06   YES    10.500     10.900     10.700     0.200       -4.82
      41   YES     9.420     10.130      9.775     0.355      -13.04
      49   OUT     6.000      6.330      6.165     0.165      -45.16
      10   YES    11.380     10.240     10.810     0.570       -3.84
      23   NFI     9.193                 9.193     0.000      -18.22
      14   YES    10.156      9.848     10.002     0.154      -11.02
      27   YES    10.400     10.500     10.450     0.050       -7.04




STANDARD LAB      ANALYTE=α-TOCOPHEROL   SERUM #74 GRAND AVG =   9.239; NBS = 9.36
LAB   VIAL USED    RESULT1    RESULT2    MEAN      SD MEAN     % BIAS
      46   YES     8.200      8.400      8.300     0.100      -10.16
```

E6

STANDARD LAB   ANALYTE=α-TOCOPHEROL   SERUM #74 GRAND AVG =   9.239; NBS = 9.36

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	21	YES	11.000	12.300	11.650	0.650	26.10
	29	YES	9.330	9.290	9.310	0.020	0.77
	35	YES	8.870	8.930	8.900	0.030	-3.67
	42	YES	7.640	7.590	7.615	0.025	-17.57
	31	YES	10.100	10.400	10.250	0.150	10.95
	15	OUT	14.500	15.600	15.050	0.550	62.90
	10	YES	9.430	9.880	9.655	0.225	4.51
	43	YES	10.500	11.200	10.850	0.350	17.44
	34	YES	6.900	8.300	7.600	0.700	-17.74
	40	YES	8.710	9.120	8.915	0.205	-3.50
	48	YES	8.430	8.360	8.395	0.035	-9.13
	19	YES	7.450	8.050	7.750	0.300	-16.11
	18	OUT	5.470	5.600	5.535	0.065	-40.09
	32	YES	8.430	8.350	8.390	0.040	-9.19
	45	NFI	7.483		7.483	0.000	-19.00
	20	YES	10.100	10.000	10.050	0.050	8.78
	26	YES	9.900	12.000	10.950	1.050	18.52

STANDARD LAB   ANALYTE=α-TOCOPHEROL   SERUM #75 GRAND AVG =   8.869; NBS = 8.98

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	04	YES	8.600	8.500	8.550	0.050	-3.60
	01	YES	11.900	12.900	12.400	0.500	39.81
	10	YES	9.060	9.610	9.335	0.275	5.25
	03	YES	8.810	8.490	8.650	0.160	-2.47
	14	YES	7.700	7.510	7.605	0.095	-14.25
	11	YES	8.600	9.400	9.000	0.400	1.47
	18	OUT	14.800	14.700	14.750	0.050	66.30
	23	YES	8.800	8.600	8.700	0.100	-1.91
	22	YES	12.810	12.070	12.440	0.370	40.26
	17	YES	10.200	10.500	10.350	0.150	16.70
	09	YES	8.620	9.280	8.950	0.330	0.91
	06	YES	8.550	8.320	8.435	0.115	-4.90
	24	YES	8.340	8.480	8.410	0.070	-5.18
	02	YES	8.000	7.900	7.950	0.050	-10.36
	25	YES	7.590	8.190	7.890	0.300	-11.04
	39	YES	6.060	5.600	5.830	0.230	-34.27
	04	YES	9.390	8.670	9.030	0.360	1.81
	07	NFI	7.651		7.651	0.000	-13.74
	08	YES	7.618	7.625	7.622	0.003	-14.07
	13	YES	8.500	8.500	8.500	0.000	-4.16

STANDARD LAB   ANALYTE=α-TOCOPHEROL   SERUM #76 GRAND AVG =   7.073; NBS = 7.25

LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	11	YES	7.000	6.800	6.900	0.100	-2.45

E7

```
STANDARD LAB        ANALYTE=α-TOCOPHEROL   SERUM #76 GRAND AVG =  7.073; NBS = 7.25
LAB   VIAL USED     RESULT1  RESULT2    MEAN     SD MEAN     % BIAS
      42   YES      8.700    9.500      9.100    0.400       28.65
      30   YES      7.550    7.330      7.440    0.110        5.18
      35   YES      7.260    7.070      7.165    0.095        1.30
      32   YES      6.480    6.190      6.335    0.145      -10.44
      28   YES      7.200    5.900      6.550    0.650       -7.40
      41   OUT     11.800   12.500     12.150    0.350       71.77
      45   YES      7.600    7.100      7.350    0.250        3.91
      27   YES      9.030    9.480      9.255    0.225       30.84
      49   YES      8.200    8.700      8.450    0.250       19.46
      43   YES      6.200    6.630      6.415    0.215       -9.31
      36   YES      7.330    7.270      7.300    0.030        3.20
      23   YES      6.810    6.760      6.785    0.025       -4.08
      38   YES      6.800    6.900      6.850    0.050       -3.16
      25   YES      6.260    6.790      6.525    0.265       -7.75
      12   YES      4.400    4.910      4.655    0.255      -34.19
      44   YES      7.000    6.630      6.815    0.185       -3.65
      37   NFI      6.427               6.427    0.000       -9.14
      39   YES      6.641    6.518      6.580    0.061       -6.98
      34   YES      6.900    6.800      6.850    0.050       -3.16


STANDARD LAB        ANALYTE=β-CAROTENE   SERUM #72 GRAND AVG =  0.953; NBS = 0.864
LAB   VIAL USED     RESULT1  RESULT2    MEAN     SD MEAN     % BIAS
015   10   NFI      0.897               0.897    0.000       -5.84
      48   YES      0.870    0.850      0.860    0.010       -9.72
      16   YES      1.078    1.004      1.041    0.037        9.28
      17   YES      0.756    0.752      0.754    0.002      -20.85
      38   YES      1.032    1.006      1.019    0.013        6.97
      46   YES      1.021    1.025      1.023    0.002        7.39
      02   YES      0.842    0.815      0.829    0.014      -13.03
      14   OUT      2.440    2.320      2.380    0.060      149.84
      06   YES      0.819    1.156      0.988    0.169        3.66
      13   YES      0.960    1.020      0.990    0.030        3.92
      11   YES      0.894    0.860      0.877    0.017       -7.94
      22   YES      0.977    0.977      0.977    0.000        2.56
      43   YES      1.104    1.196      1.150    0.046       20.72
      04   YES      0.917    0.932      0.925    0.008       -2.95
      27   OUT      1.600    1.600      1.600    0.000       67.96


STANDARD LAB        ANALYTE=β-CAROTENE   SERUM #73 GRAND AVG =  0.629; NBS = 0.554
LAB   VIAL USED     RESULT1  RESULT2    MEAN     SD MEAN     % BIAS
      09   NFI      0.570               0.570    0.000       -9.35
      08   YES      0.560    0.540      0.550    0.010      -12.53
```

E8

```
STANDARD LAB        ANALYTE=β-CAROTENE   SERUM #73 GRAND AVG =  0.629; NBS = 0.554
LAB   VIAL USED     RESULT1   RESULT2    MEAN      SD MEAN     % BIAS
      29   YES      0.678     0.675      0.677     0.001       7.59
      11   YES      0.499     0.474      0.487     0.013     -22.63
      19   YES      0.649     0.629      0.639     0.010       1.62
      18   YES      0.693     0.695      0.694     0.001      10.37
      08   YES      0.536     0.543      0.540     0.003     -14.20
      35   OUT      1.660     1.560      1.610     0.050     156.05
      20   YES      0.660     0.680      0.670     0.010       6.55
      24   YES      0.580     0.709      0.645     0.064       2.50
      41   YES      0.610     0.640      0.625     0.015      -0.60
      10   YES      0.686     0.596      0.641     0.045       1.94
      36   YES      0.723     0.737      0.730     0.007      16.10
      23   YES      0.632     0.573      0.603     0.030      -4.18
      14   YES      0.566     0.543      0.555     0.011     -11.81
      27   YES      0.750     0.750      0.750     0.000      19.28


STANDARD LAB        ANALYTE=β-CAROTENE   SERUM #74 GRAND AVG =  0.418; NBS = 0.359
LAB   VIAL USED     RESULT1   RESULT2    MEAN      SD MEAN     % BIAS
      22   NFI      0.382                0.382     0.000      -8.69
      46   YES      0.360     0.350      0.355     0.005     -15.15
      21   YES      0.465     0.460      0.463     0.002      10.55
      37   YES      0.318     0.312      0.315     0.003     -24.71
      16   YES      0.444     0.439      0.442     0.002       5.53
      35   YES      0.466     0.468      0.467     0.001      11.62
      42   YES      0.359     0.351      0.355     0.004     -15.15
      15   OUT      1.150     0.960      1.055     0.095     152.17
      43   YES      0.434     0.518      0.476     0.042      13.77
      19   YES      0.330     0.350      0.340     0.010     -18.73
      32   YES      0.428     0.400      0.414     0.014      -1.05
      25   YES      0.508     0.526      0.517     0.009      23.57
      27   YES      0.423     0.470      0.447     0.024       6.72
      45   YES      0.419     0.443      0.431     0.012       3.02
      20   OUT      0.700     0.700      0.700     0.000      67.31


STANDARD LAB        ANALYTE=β-CAROTENE   SERUM #75 GRAND AVG =  0.083; NBS = 0.066
LAB   VIAL USED     RESULT1   RESULT2    MEAN      SD MEAN     % BIAS
      27   NFI      0.078                0.078     0.000      -5.54
      04   YES      0.080     0.070      0.075     0.005      -9.17
      01   YES      0.084     0.087      0.086     0.001       3.55
      12   YES      0.042     0.038      0.040     0.002     -51.56
      19   YES      0.090     0.087      0.089     0.002       7.18
      03   YES      0.096     0.093      0.095     0.002      14.45
      14   YES      0.049     0.059      0.054     0.005     -34.60
```

E9

```
STANDARD LAB        ANALYTE=β-CAROTENE   SERUM #75 GRAND AVG =  0.083; NBS = 0.066
LAB   VIAL USED     RESULT1   RESULT2     MEAN     SD MEAN      % BIAS
      18   OUT      0.190     0.200       0.195    0.005       136.16
      23   YES      0.100     0.090       0.095    0.005        15.05
      17   YES      0.076     0.079       0.078    0.002        -6.14
      25   YES      0.080     0.080       0.080    0.000        -3.11
      04   YES      0.095     0.076       0.086    0.009         3.55
      16   YES      0.114     0.112       0.113    0.001        36.85
      07   YES      0.082     0.085       0.084    0.002         1.12
      08   YES      0.060     0.068       0.064    0.004       -22.49
      13   YES      0.120     0.120       0.120    0.000        45.33




STANDARD LAB        ANALYTE=β-CAROTENE   SERUM #76 GRAND AVG =  1.192; NBS = 1.01
LAB   VIAL USED     RESULT1   RESULT2     MEAN     SD MEAN      % BIAS
      33   NFI      1.084                 1.084    0.000        -9.09
      11   YES      1.020     1.020       1.020    0.000       -14.46
      42   YES      1.265     1.353       1.309    0.044         9.78
      31   YES      1.161     1.125       1.143    0.018        -4.14
      47   YES      1.079     1.066       1.073    0.007       -10.05
      35   YES      1.239     1.221       1.230    0.009         3.15
      32   YES      1.010     1.010       1.010    0.000       -15.30
      41   OUT      3.000     3.120       3.060    0.060       156.63
      45   YES      1.420     1.400       1.410    0.010        18.25
      49   YES      1.332     1.487       1.410    0.077        18.21
      25   YES      1.110     1.180       1.145    0.035        -3.97
      44   YES      1.160     1.040       1.100    0.060        -7.75
      29   YES      1.330     1.332       1.331    0.001        11.62
      37   YES      1.191     1.208       1.200    0.008         0.60
      39   YES      1.035     1.013       1.024    0.011       -14.12
      34   YES      1.310     1.270       1.290    0.020         8.19




STANDARD LAB        ANALYTE=RETINOL    SERUM #72 GRAND AVG =  0.874; NBS = 0.916
LAB   VIAL USED     RESULT1   RESULT2     MEAN     SD MEAN      % BIAS
      10   NFI      0.713                 0.713    0.000       -18.39
      48   YES      0.920     0.900       0.910    0.010         4.16
      16   YES      0.912     0.882       0.897    0.015         2.67
      17   YES      1.107     1.110       1.109    0.002        26.88
      38   YES      1.035     1.042       1.038    0.004        18.86
      46   YES      0.811     0.847       0.829    0.018        -5.11
      02   YES      0.863     0.773       0.818    0.045        -6.37
      26   YES      0.873     0.919       0.896    0.023         2.55
      14   YES      0.600     0.620       0.610    0.010       -30.18
      28   YES      0.920     0.850       0.885    0.035         1.30
      06   YES      0.880     1.050       0.965    0.085        10.45
```

E10

```
STANDARD LAB        ANALYTE=RETINOL   SERUM #72 GRAND AVG =  0.874; NBS = 0.916
LAB  VIAL USED    RESULT1  RESULT2    MEAN     SD MEAN    % BIAS
     21   YES     0.877    0.867      0.872    0.005      -0.19
     33   YES     0.930    0.970      0.950    0.020       8.73
     13   YES     0.840    0.800      0.820    0.020      -6.14
     12   YES     0.930    0.980      0.955    0.025       9.31
     11   YES     0.802    0.710      0.756    0.046     -13.47
     22   YES     0.944    0.942      0.943    0.001       7.93
     43   YES     0.998    1.086      1.042    0.044      19.27
     04   NFI     0.785               0.785    0.000     -10.15
     27   YES     0.630    0.650      0.640    0.010     -26.75
     15   YES     0.670    0.660      0.665    0.005     -23.89


STANDARD LAB        ANALYTE=RETINOL   SERUM #73 GRAND AVG =  0.956; NBS  0.967
LAB  VIAL USED    RESULT1  RESULT2    MEAN     SD MEAN    % BIAS
     09   NFI     0.840               0.840    0.000     -12.17
     08   YES     1.010    1.020      1.015    0.005       6.12
     29   YES     0.962    0.990      0.976    0.014       2.05
     11   YES     1.260    1.198      1.229    0.031      28.50
     19   YES     1.132    1.119      1.126    0.007      17.68
     18   YES     0.904    0.909      0.907    0.003      -5.22
     08   YES     0.824    0.831      0.828    0.003     -13.48
     28   YES     0.972    0.963      0.968    0.005       1.16
     35   YES     0.610    0.640      0.625    0.015     -34.65
     20   YES     1.040    1.040      1.040    0.000       8.74
     27   YES     0.990    0.940      0.965    0.025       0.90
     24   YES     0.970    1.080      1.025    0.055       7.17
     22   YES     0.897    0.841      0.869    0.028      -9.14
     04   YES     0.960    0.940      0.950    0.010      -0.67
     06   YES     0.934    0.922      0.928    0.006      -2.97
     41   YES     0.920    0.870      0.895    0.025      -6.42
     49   YES     0.990    0.980      0.985    0.005       2.99
     10   YES     0.957    0.883      0.920    0.037      -3.81
     36   YES     0.994    0.992      0.993    0.001       3.82
     23   NFI     0.840               0.840    0.000     -12.17
     14   YES     1.000    0.955      0.978    0.022       2.20
     27   YES     0.906    0.912      0.909    0.003      -4.96


STANDARD LAB        ANALYTE=RETINOL   SERUM #74 GRAND AVG =  0.450; NBS  0.469
LAB  VIAL USED    RESULT1  RESULT2    MEAN     SD MEAN    % BIAS
     22   NFI     0.410               0.410    0.000      -8.88
     46   YES     0.450    0.440      0.445    0.005      -1.10
     21   YES     0.494    0.486      0.490    0.004       8.90
     37   YES     0.609    0.614      0.612    0.003      35.90
```

STANDARD LAB			ANALYTE=RETINOL		SERUM #74	GRAND AVG =	0.450; NBS = 0.469
LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	16	YES	0.516	0.517	0.517	0.001	14.79
	35	YES	0.442	0.466	0.454	0.012	0.90
	42	YES	0.419	0.410	0.415	0.005	-7.88
	31	YES	0.432	0.455	0.444	0.011	-1.43
	15	YES	0.270	0.310	0.290	0.020	-35.55
	10	YES	0.450	0.430	0.440	0.010	-2.21
	43	YES	0.490	0.550	0.520	0.030	15.57
	34	YES	0.500	0.444	0.472	0.028	4.90
	40	YES	0.430	0.480	0.455	0.025	1.12
	19	YES	0.380	0.360	0.370	0.010	-17.77
	18	YES	0.560	0.480	0.520	0.040	15.57
	32	YES	0.445	0.410	0.428	0.017	-4.99
	25	YES	0.427	0.427	0.427	0.000	-5.10
	27	YES	0.448	0.517	0.483	0.035	7.23
	45	NFI	0.382		0.382	0.000	-15.10
	20	YES	0.330	0.330	0.330	0.000	-26.66
	26	YES	0.580	0.300	0.440	0.140	-2.21

STANDARD LAB			ANALYTE=RETINOL		SERUM #75	GRAND AVG =	0.395; NBS = 0.393
LAB	VIAL	USED	RESULT1	RESULT2	MEAN	SD MEAN	% BIAS
	27	NFI	0.361		0.361	0.000	-8.55
	04	YES	0.400	0.420	0.410	0.010	3.86
	01	YES	0.406	0.398	0.402	0.004	1.84
	12	YES	0.510	0.507	0.508	0.002	28.82
	19	YES	0.444	0.453	0.449	0.005	13.62
	03	YES	0.365	0.383	0.374	0.009	-5.26
	14	YES	0.362	0.367	0.364	0.003	-7.66
	11	YES	0.354	0.359	0.357	0.003	-9.69
	18	YES	0.240	0.240	0.240	0.000	-39.20
	23	YES	0.420	0.440	0.430	0.010	8.93
	22	YES	0.390	0.380	0.385	0.005	-2.47
	17	YES	0.430	0.450	0.440	0.010	11.46
	09	YES	0.438	0.418	0.428	0.010	8.42
	06	YES	0.400	0.420	0.410	0.010	3.86
	02	YES	0.400	0.409	0.405	0.004	2.47
	25	YES	0.360	0.350	0.355	0.005	-10.07
	39	YES	0.420	0.390	0.405	0.015	2.60
	04	YES	0.375	0.347	0.361	0.014	-8.55
	16	YES	0.407	0.411	0.409	0.002	3.61
	07	NFI	0.350		0.350	0.000	-11.34
	08	YES	0.390	0.393	0.392	0.002	-0.82
	13	YES	0.372	0.372	0.372	0.000	-5.76

E12

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STANDARD LAB     ANALYTE=RETINOL   SERUM #76 GRAND AVG =  0.446; NBS = 0.437
LAB  VIAL USED    RESULT1  RESULT2    MEAN     SD MEAN    % BIAS
     33   NFI     0.405               0.405    0.000     -9.18
     11   YES     0.480    0.470      0.475    0.005      6.52
     42   YES     0.437    0.473      0.455    0.018      2.04
     31   YES     0.648    0.656      0.652    0.004     46.21
     47   YES     0.506    0.505      0.506    0.000     13.36
     35   YES     0.424    0.433      0.429    0.005     -3.91
     32   YES     0.422    0.403      0.413    0.009     -7.50
     28   YES     0.410    0.356      0.383    0.027    -14.11
     41   YES     0.260    0.270      0.265    0.005    -40.57
     45   YES     0.480    0.470      0.475    0.005      6.52
     27   YES     0.460    0.440      0.450    0.010      0.91
     49   YES     0.500    0.520      0.510    0.010     14.37
     43   YES     0.482    0.483      0.483    0.001      8.20
     36   YES     0.410    0.420      0.415    0.005     -6.94
     38   YES     0.414    0.411      0.413    0.002     -7.50
     25   YES     0.420    0.400      0.410    0.010     -8.06
     12   YES     0.470    0.470      0.470    0.000      5.40
     44   YES     0.410    0.372      0.391    0.019    -12.32
     29   YES     0.458    0.458      0.458    0.000      2.71
     37   NFI     0.371               0.371    0.000    -16.80
     39   YES     0.460    0.451      0.456    0.005      2.15
     34   YES     0.414    0.411      0.413    0.002     -7.50
```

E13

Round-Robin XI

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STANDARD LAB      ANALYTE=α-TOCOPHEROL
SER NBS      GRAND AVG S.E. AVG  S WITHIN  S BETWEEN  % CV AVG  % CV X  FR REJECT
72  6.76      6.840    0.214     0.282     0.804      3.1       12.4    2/17
76  7.25      7.073    0.249     0.339     1.028      3.5       15.3    1/19
75  8.98      8.869    0.373     0.349     1.563      4.2       18.1    1/19
74  9.36      9.239    0.333     0.561     1.227      3.6      .14.6    2/17
73 10.87     11.241    0.345     0.492     1.379      3.1       13.0    2/19


STANDARD LAB      ANALYTE=β-CAROTENE
SER NBS      GRAND AVG S.E. AVG  S WITHIN  S BETWEEN  % CV AVG  % CV X  FR REJECT
75  0.066     0.083    0.006     0.005     0.021      6.8       25.9    1/15
76  0.359     1.192    0.038     0.045     0.137      3.2       12.1    1/15
74  0.554     0.418    0.018     0.022     0.061      4.4       15.5    2/14
73  0.864     0.629    0.020     0.034     0.072      3.2       12.6    1/15
72  1.01      0.953    0.031     0.075     0.095      3.3       12.7    2/14


STANDARD LAB      ANALYTE=RETINOL
SER NBS      GRAND AVG S.E. AVG  S WITHIN  S BETWEEN  % CV AVG  % CV X  FR REJECT
75  0.393     0.395    0.012     0.011     0.051      2.9       13.3    0/20
76  0.437     0.446    0.016     0.014     0.072      3.6       16.4    0/20
74  0.469     0.450    0.016     0.052     0.061      3.7       17.9    0/19
72  0.916     0.874    0.031     0.043     0.132      3.5       15.8    0/19
73  0.967     0.956    0.026     0.029     0.116      2.8       12.6    0/20
```

Explanation of Column Heading on <u>Statistical Summary</u>

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

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

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

S BETWEEN = between laboratory component of standard deviation = S_b

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

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

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

Appendix F. Updated "All-Lab Report" for RR11

The following three pages are an updated version of an "All-Lab" report for RR11. 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 used 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 RR11. The only attributed results are those reported by NIST. The NIST results are not used in the assessment of the consensus summary results of the study.

Round Robin XI Laboratory Results

Lab	Total Retinol, µg/mL					α-Tocopherol, µg/mL					β-Carotene, µg/mL				
	72	73	74	75	76	72	73	74	75	76	72	73	74	75	76
FSV-BA	0.785	0.840	0.382	0.350	0.371	5.48	9.19	7.48	7.65	6.43	0.925	0.603	0.431	0.084	1.200
FSV-BD	0.896	0.968	0.444	0.357	0.383	6.65	11.40	10.25	9.00	6.55					
FSV-BE		0.928		0.405	0.413		10.70		7.95	6.85					
FSV-BF	0.965	1.025	0.520	0.440	0.510	7.25	12.75	10.85	10.35	8.45	0.988	0.645	0.476	0.078	1.410
FSV-BG	0.897	0.976	0.490	0.402	0.455	8.90	15.10	11.65	12.40	9.10	1.041	0.677	0.463	0.086	1.309
FSV-BH		0.909		0.372	0.413		10.45		8.50	6.85		0.750		0.120	1.290
FSV-BI	0.910	1.015	0.445	0.410	0.475	6.45	10.20	8.30	8.55	6.90	0.860	0.550	0.355	0.075	1.020
FSV-BX	0.640		0.330			7.35		10.05			1.600		0.700		
FSV-BY	0.713	0.840	0.410	0.361	0.405						0.897	0.570	0.382	0.078	1.084
FSV-BZ	0.665		0.440			8.35		10.95							
FSV-CJ	1.039	1.126	0.517	0.449	0.506	6.85	11.71	9.31	9.34	7.44	1.019	0.639	0.442	0.089	1.073
FSV-CK	0.787		0.426			6.00		8.64			0.863		0.377		
FSV-CN		1.040		0.430	0.475		11.60		8.70	7.35		0.670		0.095	1.410
FSV-CO	0.829	0.907	0.454	0.374	0.429	6.64	10.63	8.90	8.65	7.17	1.023	0.694	0.467	0.095	1.230
FSV-DC	0.943	0.993	0.427	0.409	0.458						0.977	0.730	0.517	0.113	1.331
FSV-DE						6.27	10.39	8.40	8.41	6.79					
FSV-DG	0.820	0.895	0.370	0.355	0.410	6.31	9.78	7.75	7.89	6.53	0.990	0.625	0.340	0.080	1.140
FSV-DH	0.885	0.965	0.440	0.385	0.450	6.59	12.43	9.66	12.44	9.26					
FSV-DN	0.610	0.625	0.290	0.240	0.265	11.60	18.65	15.05	14.75	12.15	2.380	1.610	1.055	0.195	3.060
FSV-DO	1.175	1.515	0.710	0.655	0.735										
FSV-DT	0.872	0.869	0.472	0.428	0.483	6.68	12.95	7.60	8.95	6.42					
FSV-ED		0.978		0.392	0.456		10.00		7.62	6.58		0.555		0.064	1.024
FSV-EG	1.109	1.229	0.612	0.508	0.652						0.754	0.487	0.315	0.040	1.143
FSV-EO	1.042		0.483								1.150		0.447		
FSV-EP	0.950	0.950	0.455	0.410	0.415	6.31	10.79	8.92	8.44	7.30					
FSV-ER	0.818	0.828	0.415	0.364	0.413	5.87	9.42	7.62	7.61	6.34	0.829	0.540	0.355	0.054	1.010
FSV-EU	0.955	0.985	0.520	0.405	0.470	6.63	6.17	5.54	5.83	4.66					
FSV-EW	0.756	0.920	0.428	0.361	0.391	6.15	10.81	8.39	9.03	6.82	0.877	0.641	0.414	0.086	1.100
FSV-FI						6.70		9.39							
n	23	22	23	22	22	20	20	20	20	20	16	16	16	16	16
Min	0.610	0.625	0.290	0.240	0.265	5.48	6.17	5.54	5.83	4.66	0.754	0.487	0.315	0.040	1.010
Median	0.885	0.958	0.444	0.397	0.439	6.63	10.75	8.91	8.60	6.85	0.982	0.640	0.436	0.085	1.171
Max	1.175	1.515	0.710	0.655	0.735	11.60	18.65	15.05	14.75	12.15	2.380	1.610	1.055	0.195	3.060
eSD	0.119	0.087	0.050	0.044	0.056	0.52	1.35	1.70	1.01	0.64	0.141	0.092	0.084	0.014	0.190
eCV	13	9	11	11	13	8	13	19	12	9	14	14	19	17	16
N_{RR}	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1
N_{past}	27	27	27	27	27	24	23	24	23	23	19	20	19	20	20
$Median_{past}$	0.881	0.900	0.438	0.388	0.430	6.84	10.27	9.27	8.36	7.00	0.917	0.618	0.414	0.089	1.053
SD_{past}	0.125	0.133	0.071	0.044	0.052	0.81	1.56	1.01	1.26	1.02	0.129	0.132	0.056	0.033	0.285
NIST	0.970	0.880	0.430	0.380	0.390	6.30	10.39	8.26	8.70	6.87	0.830	0.570	0.360	0.080	1.020
NAV	0.928	0.919	0.437	0.389	0.415	6.47	10.57	8.58	8.65	6.86	0.906	0.605	0.398	0.082	1.096
NAU	0.133	0.103	0.051	0.046	0.066	0.61	1.37	1.77	1.01	0.64	0.178	0.104	0.100	0.015	0.218

F2

Round Robin XI Laboratory Results

Table Legend [a]

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
N_{RR}	Number of prior RR studies that distributed this serum
N_{past}	Maximum number of (non-NIST) quantitative values reported for this analyte in prior studies
$Median_{past}$	Mean of the median values from prior studies
SD_{past}	Pooled eSD of prior studies
NAV	NIST Assigned Value: (Median + NIST)/2
NAU	NIST Assigned Uncertainty: $\sqrt{S^2 + S_{btw}^2}$
	S is the maximum of (0.05*NAV, eSD, SD_{LT}) and S_{btw} is the standard deviation between Median and $Mean_{NIST}$. The expected long-term SD, SD_{LT}, is defined in: Duewer et al., Anal Chem 1997;69(7):1406-1413.

[a] What is now "NIST" was the National Bureau of Standards (NBS) when this study was conducted.

F3

Round Robin XI Laboratory Results

Comparability Summary

Lab	TR	aT	bC
FSV-BA	2	2	1
FSV-BD	1	1	
FSV-BE	1	1	
FSV-BF	2	2	1
FSV-BG	1	4	1
FSV-BH	1	1	2
FSV-BI	1	1	1
FSV-BX	3	1	4
FSV-BY	1		1
FSV-BZ	2	3	
FSV-CJ	2	1	1
FSV-CK	1	1	1
FSV-CN	1	1	1
FSV-CO	1	1	1
FSV-DC	1		2
FSV-DE		1	
FSV-DG	1	1	1
FSV-DH	1	3	
FSV-DN	3	4	4
FSV-DO	4		
FSV-DT	1	1	
FSV-ED	1	1	2
FSV-EG	3		2
FSV-EO	2		1
FSV-EP	1	1	
FSV-ER	1	1	2
FSV-EU	1	3	
FSV-EW	1	1	1
FSV-FI		1	
NIST	1	1	1
n	28	25	20

	TR	aT	bC
% 1	68	72	65
% 2	18	8	25
% 3	11	12	0
% 4	4	8	10

Label	Definition
Lab	Participant code
TR	Total Retinol
aT	α-Tocopherol
bC	β-Carotene
n	Number of values
n	number of participants providing quantitative data
% 1	Percent of CS = 1 (within 1 SD of medians)
% 2	Percent of CS = 2 (within 2 SD of medians)
% 3	Percent of CS = 3 (within 3 SD of medians)
% 4	Percent of CS = 4 (3 or more SD from medians)

"Comparability Score"

The Comparability Score (CS) summarizes your measurement performance for a given analyte relative to the consensus medians in this study. CS is the average distance (in units of standard deviation) of your measurement performance characteristics from the consensus performance. CS is calculated when the number of quantitative values you reported, N_{you}, is at least two and at least six participants reported quantitative values for the analyte.

We define CS as follows:

$$CS = \text{MINIMUM}\left(4, \text{INTEGER}\left(1 + \sqrt{C^2 + AP^2}\right)\right)$$

$$C = \text{Concordance} = \frac{\sum_{i=1}^{N_{you}} \dfrac{You_i - Median_i}{NAU_i}}{N_{you}}$$

$$AP = \text{Apparent Precision} = \sqrt{\frac{\sum_{i=1}^{N_{you}} \left(\dfrac{You_i - Median_i}{NAU_i}\right)^2}{N_{you} - 1}}$$

NAU = NIST Assigned Uncertainty

For further details, please see
Duewer DL, Kline MC, Sharpless KE, Brown Thomas J, Gary KT. Micronutrients Measurement Quality Assurance Program: Helping participants use interlaboratory comparison exercise results to improve their long-term measurement performance. Anal Chem 1999;71(9):1870-8.

F4