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NIST Micronutrients Measurement Quality Assurance Program Winter 2011 Comparability Studies

Results for Round Robin LXIX
Fat-Soluble Vitamins and Carotenoids in Human Serum
and Round Robin 34 Ascorbic Acid in Human Serum

David L. Duewer
Jeanice B. Thomas

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April, 2013

U.S. Department of Commerce
Rebecca Blank, Acting Secretary

National Institute of Standards and Technology
Patrick D. Gallagher, 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 2011 MMQAP measurement comparability improvement studies: 1) Round Robin LXIX Fat-Soluble Vitamins and Carotenoids in Human Serum and 2) Round Robin 34 Total Ascorbic Acid in Human Serum. The materials for both studies were shipped to participants in November 2010; participants were requested to provide their measurement results by March 7, 2011.

Keywords

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

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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 total 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. [1,2]

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

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin LXIX comparability study (hereafter referred to as RR69) received five liquid-frozen 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 November 2010. The communication materials included in the sample shipment are provided 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 many participants report values for non-target analytes.

The final report delivered to every participant in RR69 consists of three documents:

- A cover letter for the current study, a brief description of the other two documents, and a discussion of our analysis of the overall results that may be of broad interest. This cover letter is reproduced as Appendix B.

- The "All-Lab Report" that lists all of the reported measurement results, a number of consensus statistics for analytes reported by more than one participant, and the mean median and pooled SD from any prior distributions of the serum. This report also provides a numerical "score card" for each participant's measurement comparability for the more commonly reported analytes. This report is reproduced as Appendix C.

- An “Individualized Report” that graphically analyzes each participant’s results for all analytes reported by at least five participants. This report also provides a graphical summary of their measurement comparability. The graphical tools used in this report are described in detail elsewhere [3]. An example of “Individualized Report” is reproduced as Appendix D.

Round Robin 34: Vitamin C in Human Serum

Participants in the MMQAP Vitamin C in Human Serum Round Robin 34 comparability study (hereafter referred to as RR34) received four frozen serum test samples, one frozen control serum, and a solid ascorbic acid control material 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 November 2010. The communication materials included in the sample shipment are provided in Appendix E.

The test and control serum materials were prepared by adding equal volumes of 10 % metaphosphoric acid (MPA) to human serum that had been spiked with ascorbic acid. While these samples contain some dehydroascorbic acid, its content is variable. Therefore, the participants report only total ascorbic acid (TAA, ascorbic acid plus dehydroascorbic acid). Participants are also encouraged to prepare calibration solutions from the supplied solid control to enable calibrating their serum measurements to the same reference standard.

The final report delivered to every participant in RR34 consists of three documents:

- A cover letter for the current study, a brief description of the other two documents, and a discussion of our analysis of overall results that may be of broad interest. This cover letter is reproduced as Appendix F.

- The “All-Lab Report” that summarizes all of the reported measurement results and provides several consensus statistics. This report is reproduced as Appendix G.

- An “Individualized Report” that graphically analyzes each participant’s results for TAA, including a graphical summary of their measurement comparability. The graphical tools used in this report are described in detail elsewhere [3]. An example “Individualized Report” is reproduced as Appendix H.

References

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

2 Margolis SA, Duewer DL. Measurement Of Ascorbic Acid in Human Plasma and Serum: Stability, Intralaboratory Repeatability, and Interlaboratory Reproducibility. Clin Chem 1996;42(8):1257-1262.

3 Duewer DL, Kline MC, Sharpless KE, Brown Thomas J, Gary KT, Sowell AL. Micronutrients Measurement Quality Assurance Program: Helping Participants Use Interlaboratory Comparison Exercise Results to Improve Their Long-Term Measurement Performance. Anal Chem 1999;71(9):1870-1878.

Appendix A. Shipping Package Inserts for RR69

The following three items were included in each package shipped to an RR69 participant:

- Cover letter
- Datasheet
- Packing List and Shipment Receipt Confirmation Form

The cover letter and datasheet were enclosed in a sealed waterproof bag along with the samples themselves. The packing list was placed at the top of the shipping box, between the cardboard covering and the foam insulation.

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

November 26, 2010

Dear Colleague:

Enclosed are samples for the first fat-soluble vitamins and carotenoids in serum study (Round Robin LXIX) for the 2011 NIST Micronutrients Measurement Quality Assurance Program. The set of samples (Sera 372- 376) consists of one vial each of five liquid-frozen serum samples for analysis along with a form for reporting your results. These samples should be stored in the dark at or below –20 °C upon receipt. When reporting your results, please submit one value for each analyte for a given serum sample. If a value obtained is below your limit of quantification, please indicate this result on the form by using NQ *(Not Quantified)*. Results are due to NIST by **March 7, 2011**. Results received more than two weeks after the due date may not be included in the summary report for this round robin study. The feedback report concerning the study will be distributed in April 2011.

Samples should be allowed to stand at room temperature under subdued light until thawed. We recommend that sample mixing be facilitated with 3 to 5 min agitation in an ultrasonic bath or at least 15 min at room temperature with intermittent swirling. (CAUTION: Vigorous shaking will cause foaming and possibly interfere with accurate measurement. The rubber stopper contains phthalate esters that may leach into the sample upon intermittent contact of the liquid sample with the stopper. These esters absorb strongly in the UV region and elute near retinol in most LC systems creating analytical problems.) **Water should not be added to the liquid-frozen samples.**

For consistency, we request that laboratories use the following absorptivities (dL/g • cm): retinol, 1843 at 325 nm (ethanol); retinyl palmitate, 975 at 325 nm (ethanol); α-tocopherol, 75.8 at 292 nm (ethanol); γ-tocopherol, 91.4 at 298 nm (ethanol); α-carotene, 2800 at 444 nm (hexane); β-carotene, 2560 at 450 nm (ethanol), 2592 at 452 nm (hexane); and lycopene, 3450 at 472 nm (hexane).

Please report your results for Round Robin LXIX by e-mail to david.duewer@nist.gov or fax to 301-977-0685. If you have questions or comments regarding this study, please call me at (301) 975-3120 or e-mail me at jbthomas@nist.gov.

Sincerely,

Jeanice Brown Thomas
Program Coordinator/Research Chemist
Analytical Chemistry Division
Material Measurement Laboratory

Cc: Lane C. Sander

NIST

Participant #: _____ Date: _____

Round Robin LXIX: Human Sera

NIST Micronutrients Measurement Quality Assurance Program

Analyte	372	373	374	375	376	Units*
total retinol						
trans-retinol						
retinyl palmitate						
retinyl stearate						
α-tocopherol						
γ/β-tocopherol						
δ-tocopherol						
total β-carotene						
trans-β-carotene						
total cis-β-carotene						
total α-carotene						
total lycopene						
trans-lycopene						
total β-cryptoxanthin						
total α-cryptoxanthin						
total lutein						
total zeaxanthin						
total lutein&zeaxanthin						
total coenzyme Q10						
ubiquinol (QH_2)						
ubiquinone (Qox)						
phylloquinone (K_1)						
25-hydroxyvitamin D						

Other measurands?						

* we prefer **µg/mL**

Were the liquid-frozen samples frozen when received? Yes | No

Comments:

Mail: M^2QAP
NIST, Stop 6392
Gaithersburg, MD 20899-6392

Please return results by
7-March-2011

Fax: 301-977-0685
Email: David.Duewer@NIST.gov

Participant #: _____ Date: _____

Round Robin LXIX: Human Sera

NIST Micronutrients Measurement Quality Assurance Program

Packing List and Shipment Receipt Confirmation Form

This box contains: one vial each of the following five FSV M^2QAP sera

Serum	Form	Reconstitute?	Vial/Cap
#372	Liquid frozen	No	10 mL amber, silver cap
#373	Liquid frozen	No	2 mL amber, green cap
#374	Liquid frozen	No	2 mL amber, blue cap
#375	Liquid frozen	No	2 mL amber, red cap
#376	Liquid frozen	No	2 mL amber, green cap

Please 1) Open the pack immediately
2) Check that it contains all of the above samples
3) Check if the vials are intact
4) Store the sera at -20 ˚C or below until analysis
5) Complete the following information
6) Fax the completed form to us at 301-977-0685
(or email requested information to david.duewer@nist.gov)

1) Date this shipment arrived: _____

2) Are all five sera vials intact? Yes | No
If "No", which one(s) were damaged?

3) Was there any dry-ice left in cooler? Yes | No

4) Did the liquid frozen samples arrive frozen? Yes | No

5) At what temperature are you storing the serum samples? _____ ˚C

6) When do you anticipate analyzing these samples? _____

Your prompt return of this information is appreciated.

The M^2QAP Gang

Mail: M^2QAP
NIST, Stop 6392
Gaithersburg, MD 20899-6392

Fax: 301-977-0685
Email: David.Duewer@NIST.gov

Appendix B. Final Report for RR69

The following two pages are the final report as provided to all participants:

- Cover letter.
- An information sheet that:
  - describes the contents of the “All-Lab” report,
  - describes the content of the “Individualized” report,
  - describes the nature of the test samples and details their previous distributions, if any, and
  - summarizes aspects of the study that we believe may be of interest to the participants.

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

April 7, 2011

Dear Colleague:

Enclosed is the summary report of the results for round robin LIX (RR69) of the 2011 NIST Micronutrients Measurement Quality Assurance Program (M^2QAP) for the fat-soluble vitamins and carotenoids in human serum. Included in this report are: 1) a summary of data and measurement comparability scores for all laboratories, 2) a detailed graphical analysis of your results; and 3) a graphical summary of your measurement comparability.

Your overall measurement comparability is summarized in the "Score Card" summary, page 6 of the All Lab Report. Combined results rated 1 to 3 are within 1 to 3 standard deviations of the assigned value, respectively; those rated 4 are >3 standard deviations from the assigned value. Similar information is presented graphically in the "target plots" that are the last page of your Individualized Report. If you have concerns regarding your laboratory's performance, please contact us for consultation.

As you may be aware, the MMQAP workshop is scheduled to be held in conjunction with the Experimental Biology conference on Wednesday, April 13 at the Washington Convention Center in Washington, DC. There is no registration fee for the workshop. However, we ask that you submit a registration form if you plan to attend. If you have not received a form, please contact me; a form will be sent to you as soon as possible.

Samples for the second 2011 QA interlaboratory exercise will be shipped around the middle of May. If you have any questions regarding this report, please contact Dave Duewer at david.duewer@nist.gov or me at jbthomas@nist.gov, tel: 301/975-3120, or fax: 301/977-0685.

Sincerely,

Jeanice Brown Thomas
Research Chemist
Analytical Chemistry Division
Chemical Science and Technology Laboratory

David L. Duewer, Ph.D.
Research Chemometrician
Analytical Chemistry Division
Chemical Science and Technology Laboratory

Cc: L.C. Sander

NIST

The NIST M^2QAP Round Robin LXIX (RR69) report consists of:

Page	"All Lab" Report
1-5	A listing of all results and statistics for all analytes.
6	A legend for the list of results and statistics.
7	The text Comparability Summary ("Score Card") of measurement performance.

Page	"Individualized" Report
1	Your values, the number of labs reporting values, and our assigned values.
2 to n	"Four Plot" summaries of your current and past measurement performance, one page for each analyte you report that is also reported by at least 8 other participants.
n+1	The graphical Comparability Summary (target plot) of measurement performance.

Samples. Five samples were distributed in RR69.

Serum	Description	Prior Distributions
372	Fresh-frozen, native, multi-donor serum prepared in Fall, 2007. This was SRM 968d.	#341 & #344:RR63-3/08, #351:RR64-9/08, #361:RR66-9/09
373	Fresh-frozen, native, multi-donor, prepared in 2009. This is Level III of SRM 968e.	#359:RR66-9/09
374	Fresh-frozen, native, multi-donor, prepared in 2009. This is Level II of SRM 968e.	#358:RR66-9/09
375	Fresh-frozen, native, multi-donor, prepared in 2009. This is Level I of SRM 968e.	#357:RR66-9/09
376	Fresh-frozen, native, multi-donor, prepared in 2008.	#356:RR65-3/09, #360:RR66-9/09

Results

1) Sera Stability. There was no significant change in the median level or measurement variability of any measurand in any of the materials, including the very low-level material that used to be SRM 968d.

2) SRM 968e. Sera #373 to #375 are the components of SRM 968e. All three of these materials were prepared by blending commercially available materials without spiking. The materials were designed to represent relatively low, middling, and relatively high levels of retinol, α-tocopherol and β-carotene. SRM 968e is now available for purchase.

3) Environmental Stability. Due to severe weather, RR69 shipping was more than usually chaotic. Several sets of RR69 materials arrived thoroughly defrosted. None of the affected labs measured the thawed and unthawed materials together under repeatability conditions. However, there were no significant (relative to interlaboratory imprecision) differences between results for any of the thawed RR69 materials and the measurements each of these labs made for the same materials in RR66. Any changes in these materials that may have occurred due to the shipping misadventures were thus well within interlaboratory imprecision.

Appendix C. "All-Lab Report" for RR69

The following six pages are the "All-Lab Report" as provided to all participants, with two exceptions:

- the participant identifiers (Lab) have been altered.
- the order in which the participant results are listed has been altered.

The data summary in the "All-Lab Report" has been altered to ensure confidentiality of identification codes assigned to laboratories.

Round Robin LXIX Laboratory Results

	Total Retinol, µg/mL					trans-Retinol, µg/mL					Retinyl Palmitate, µg/mL					α-Tocopherol, µg/mL					γ/β-Tocopherol, µg/mL				
Lab	372	373	374	375	376	372	373	374	375	376	372	373	374	375	376	372	373	374	375	376	372	373	374	375	376
FSV-BA	0.365	0.715	0.537	0.375	0.775						0.030	0.173	0.070	0.018	0.047	5.67	18.15	9.94	6.61	10.10	1.52	2.47	1.50	1.93	2.55
FSV-BB	0.378	0.708	0.554	0.393	0.795						0.008	0.075	0.022	0.009	0.038	5.89	18.23	10.26	6.65	10.45	1.56	2.25	1.40	1.80	2.41
FSV-BC	0.354	0.718	0.561	0.352	0.778																				
FSV-BD	0.366	0.692	0.606	0.371	0.719											5.80	20.10	11.40	6.40	9.90					
FSV-BE	0.418	0.799	0.598	0.422	0.868											4.20	19.21	10.18	6.54	10.41	1.62	2.54	1.52	2.00	2.58
FSV-BF	0.350	0.600	0.490	0.330	0.690											5.90	18.60	10.50	6.80	10.60					
FSV-BG	0.346	0.659	0.506	0.360	0.766						0.018	0.155	0.041	0.010	0.041	5.64	18.72	10.07	6.75	10.84	1.51	2.30	1.42	1.93	2.47
FSV-BH	0.242	0.573	0.459	0.304	0.646											5.54	16.93	9.84	6.41	9.87	1.25	1.91	1.25	1.60	2.09
FSV-BJ	0.335	0.643	0.507	0.352	0.741						*nq*	0.083	*nq*	*nq*	*nq*	6.21	20.27	11.46	7.14	11.20	1.52	2.43	1.51	1.93	2.50
FSV-BK	0.438	0.677	0.537	0.385	0.805											6.72	20.08	11.24	7.38	11.49					
FSV-BL	0.340	0.690	0.540	0.370	0.770											6.03	18.52	10.77	6.89	10.34					
FSV-BM	0.350	0.719	0.511	0.379	0.739											6.60	20.40	11.40	7.40	10.90					
FSV-BN	0.324	0.616	0.481	0.318	0.614											4.61	17.44	9.20	5.53	8.26					
FSV-BO	0.289	0.539	0.412	0.298	0.607											5.25	16.75	9.04	5.60	9.22	1.17	1.83	1.10	1.46	1.93
FSV-BP	0.350	0.650	0.520	0.350	0.730											6.13	15.74	10.00	6.77	9.50					
FSV-BQ	0.360	0.810	1.040	0.410	1.070											8.00	24.00	23.00	8.00	16.00					
FSV-BR	0.335	0.660	0.490	0.350	0.730											5.65	14.70	9.10	6.54	9.28					
FSV-BS	*≥0.291*	*≥0.661*	*≥0.476*	*≥0.321*	*≥0.745*	0.291	0.661	0.476	0.321	0.745															
FSV-BT	0.385	0.517	0.409	0.306	0.607											5.90	19.43	11.07	7.17	11.53	1.39	2.49	1.51	1.84	2.52
FSV-BU	0.284	0.694	0.483	0.341	0.669											5.95	20.12	12.23	7.36	11.68	1.39	2.21	1.23	1.54	2.18
FSV-BV	0.367	0.708	0.572	0.408	0.802											5.54	18.48	10.89	7.00	10.54	1.31	2.12	1.43	1.88	2.37
FSV-BW	0.332	0.623	0.472	0.357	0.707						0.002	0.123	0.027	0.010	0.060	5.49	20.20	10.27	7.04	11.31	1.37	2.32	1.37	1.83	2.57
FSV-CC	0.293	0.657	0.477	0.348	0.701	0.281	0.645	0.472	0.347	0.691						2.02	19.68	9.99	6.76	10.49					
FSV-CD	0.359	0.560	0.437	0.319	0.638						0.011	0.125	0.022	*nq*	0.047	8.52	20.86	11.96	8.05	12.28	1.93	2.45	1.55	1.94	2.59
FSV-CE	0.350	0.660	0.520	0.390	0.750											5.73	17.07	10.37	6.84	10.63					
FSV-CG	0.374	0.727	0.556	0.407	0.821											5.66	21.20	11.03	7.12	11.20	1.65	2.78	1.68	2.12	2.81
FSV-CI	0.407	0.758	0.585	0.405	0.850						0.015	0.103	0.042	0.015	0.050	5.77	18.63	10.56	6.79	10.70	1.38	2.17	1.37	1.73	2.28
FSV-CW	0.343	0.512	0.432	0.346	0.682						0.005	0.088	0.028	0.009	0.049	5.89	18.94	11.00	7.07	11.10	1.39	2.22	1.42	1.82	2.41
FSV-CZ	0.324	0.604	0.469	0.339	0.692											5.24	19.13	9.45	6.10	10.69	1.35	2.11	1.27	1.69	2.30
FSV-DD	0.380	0.670	0.550	0.500	0.720																				
FSV-DQ																5.95	28.06	12.68	6.62	12.87	1.51	2.36	1.62	2.21	2.62
FSV-DV	0.357	0.734	0.570	0.401	0.830											4.70	18.90	10.20	6.60	10.10					
FSV-EE																									
FSV-EZ	0.336	0.647	0.503	0.361	0.754						nq	0.073	nq	nq	0.034	5.81	18.93	10.62	7.02	11.19	1.47	2.40	1.51	1.96	2.66
FSV-FK	*≥0.321*	*≥0.610*	*≥0.474*	*≥0.336*	*≥0.690*	0.321	0.610	0.474	0.336	0.690						6.00	17.40	10.00	6.40	10.00					
N	31	31	31	31	31	3	3	3	3	3	7	9	7	6	8	31	31	31	31	31	18	18	18	18	18
Min	0.242	0.512	0.409	0.298	0.607	0.281	0.610	0.472	0.321	0.690	0.002	0.073	0.022	0.009	0.034	2.02	14.70	9.04	5.53	8.26	1.17	1.83	1.10	1.46	1.93
Median	0.350	0.660	0.511	0.360	0.739	0.291	0.645	0.474	0.336	0.691	0.011	0.103	0.028	0.010	0.047	5.80	18.93	10.50	6.79	10.63	1.43	2.31	1.43	1.86	2.49
Max	0.438	0.810	1.040	0.500	1.070	0.321	0.661	0.476	0.347	0.745	0.030	0.173	0.070	0.018	0.060	8.52	28.06	23.00	8.05	16.00	1.93	2.78	1.68	2.21	2.81
SD	0.024	0.071	0.058	0.037	0.073						0.008	0.033	0.009	0.001	0.007	0.341	1.742	0.783	0.371	0.841	0.125	0.208	0.130	0.128	0.165
CV	7	11	11	10	10						76	32	33	11	14	6	9	7	5	8	9	9	9	7	7
N_{past}	31	31	31	31	30	7	5	5	5	5	8	10	7	9	9	32	32	32	32	32	21	19	19	19	19
$Median_{past}$	0.341	0.655	0.504	0.353	0.740	0.318	0.549	0.471	0.330	0.692	0.010	0.090	0.021	0.008	0.045	5.744	18.634	10.300	6.822	10.564	1.403	2.125	1.373	1.751	2.383
SD_{past}	0.030	0.049	0.039	0.033	0.061	0.053	0.102	0.064	0.032	0.097	0.007	0.027	0.007	0.004	0.009	0.853	1.221	0.856	0.540	0.797	0.142	0.265	0.153	0.136	0.266
NAV	0.350	0.660	0.509	0.359	0.735	0.350	0.660	0.509	0.359	0.735	0.010	0.104	0.028	0.010	0.047	5.785	18.913	10.530	6.780	10.615	1.394	2.297	1.431	1.844	2.474
NAU	0.029	0.072	0.057	0.035	0.072	0.029	0.072	0.057	0.035	0.072	0.010	0.031	0.012	0.010	0.016	0.518	1.742	0.818	0.572	0.864	0.177	0.233	0.160	0.196	0.247

C2

Round Robin LXIX Laboratory Results

	δ-Tocopherol, µg/mL					Total β-Carotene, µg/mL					trans-β-Carotene, µg/mL					Total cis-β-Carotene, µg/mL					Total α-Carotene, µg/mL				
Lab	372	373	374	375	376	372	373	374	375	376	372	373	374	375	376	372	373	374	375	376	372	373	374	375	376
FSV-BA	0.086	0.199	0.062	0.083	0.255	0.078	0.388	0.226	0.086	0.260	0.075	0.372	0.216	0.082	0.248	0.003	0.017	0.010	0.003	0.011	0.007	0.015	0.031	0.005	0.048
FSV-BB	0.089	0.217	0.067	0.143	0.275	0.078	0.356	0.234	0.093	0.282	0.075	0.342	0.223	0.088	0.266	0.004	0.015	0.010	0.005	0.016	0.008	0.012	0.029	0.008	0.047
FSV-BC																									
FSV-BD																									
FSV-BE						0.078	0.430	0.241	0.089	0.247															
FSV-BF						0.071	0.373	0.220	0.072	0.304															
FSV-BG						0.078	0.463	0.244	0.100	0.279											0.009	0.016	0.032	0.010	0.045
FSV-BH						0.060	0.354	0.226	0.074	0.261	0.060	0.331	0.207	0.074	0.237	*nq*	0.023	0.019	*nq*	0.024	*nq*	*nq*	*nq*	*nq*	0.033
FSV-BJ						0.083	0.427	0.243	0.084	0.280											*nq*	*nq*	*nq*	*nq*	0.033
FSV-BK																									
FSV-BL																									
FSV-BM																									
FSV-BN						0.056	0.383	0.233	0.074	0.226											0.011	0.029	0.043	0.009	0.054
FSV-BO						0.101	0.443	0.254	0.098	0.274											0.008	0.009	0.026	0.007	0.041
FSV-BP						0.102	0.400	0.271	0.095	0.273											0.017	0.020	0.034	0.045	0.030
FSV-BQ																									
FSV-BR																									
FSV-BS						*≥0.129*	*≥0.710*	*≥0.451*	*≥0.159*	*≥0.425*	0.129	0.710	0.451	0.159	0.425						0.031	0.052	0.075	0.032	0.131
FSV-BT	0.119	0.278	0.129	0.143	0.333	0.076	0.322	0.205	0.079	0.219	0.075	0.331	0.208	0.081	0.228	0.006	0.018	0.013	0.004	0.010	0.011	0.019	0.025	0.008	0.030
FSV-BU						0.092	0.420	0.255	0.091	0.280											0.009	0.009	0.032	0.010	0.056
FSV-BV						0.069	0.402	0.269	0.104	0.299											0.010	0.018	0.045	0.010	0.068
FSV-BW						0.063	0.497	0.268	0.100	0.321											0.002		0.013	0.001	0.026
FSV-CC																									
FSV-CD						0.194	0.446	0.326	0.200	0.370											0.049	0.050	0.059	0.046	0.069
FSV-CE						0.090	0.410	0.230	0.090	0.320															
FSV-CG						0.069	0.431	0.251	0.093	0.285	0.065	0.411	0.238	0.088	0.269	nq	0.020	0.012	0.005	0.015	0.010	0.019	0.040	0.010	0.064
FSV-CI						0.065	0.453	0.259	0.091	0.282											0.014	0.052	0.050	0.012	0.057
FSV-CW	0.061	0.225	0.043	0.095	0.275	0.079	0.319	0.208	0.088	0.250											0.006	0.007	0.021	0.003	0.038
FSV-CZ						0.102	0.346	0.246	0.102	0.299															
FSV-DD																									
FSV-DQ						0.148	0.781	0.435	0.120	0.571											0.016	0.012	0.053	0.006	0.110
FSV-DV																									
FSV-EE																									
FSV-EZ											0.078	0.315	0.196	0.086	0.261										
FSV-FK																									
N	4	4	4	4	4	21	21	21	21	21	7	7	7	7	7	3	5	5	4	5	16	15	16	16	18
Min	0.061	0.199	0.043	0.083	0.255	0.056	0.319	0.205	0.072	0.219	0.060	0.315	0.196	0.074	0.228	0.003	0.015	0.010	0.003	0.010	0.002	0.007	0.013	0.001	0.026
Median	0.088	0.221	0.065	0.119	0.275	0.078	0.410	0.244	0.091	0.280	0.075	0.342	0.216	0.086	0.261	0.004	0.018	0.012	0.005	0.015	0.010	0.018	0.033	0.010	0.048
Max	0.119	0.278	0.129	0.143	0.333	0.194	0.781	0.435	0.200	0.571	0.129	0.710	0.451	0.159	0.425	0.006	0.023	0.019	0.005	0.024	0.049	0.052	0.075	0.046	0.131
SD	0.021	0.019	0.018	0.036	0.015	0.018	0.053	0.022	0.010	0.028	0.004	0.040	0.013	0.006	0.019		0.003	0.003	0.000	0.006	0.004	0.009	0.013	0.004	0.021
CV	24	9	28	30	5	23	13	9	11	10	6	12	6	7	7		16	25	10	40	37	49	41	39	45
N_{past}	5	5	4	4	5	22	22	22	22	21	9	8	8	8	8	5	7	7	4	6	16	17	18	16	18
$Median_{past}$	0.084	0.202	0.072	0.091	0.261	0.076	0.388	0.241	0.090	0.274	0.073	0.337	0.210	0.083	0.247	0.005	0.016	0.012	0.004	0.014	0.009	0.016	0.030	0.008	0.046
SD_{past}	0.022	0.034	0.022	0.023	0.026	0.015	0.081	0.036	0.015	0.034	0.010	0.059	0.024	0.007	0.012	0.003	0.001	0.001	0.001	0.002	0.003	0.006	0.004	0.004	0.007
NAV	0.088	0.221	0.065	0.119	0.275	0.078	0.415	0.245	0.092	0.280	0.075	0.342	0.216	0.086	0.261	0.075	0.342	0.216	0.086	0.261	0.010	0.018	0.032	0.009	0.047
NAU						0.019	0.059	0.036	0.016	0.041	0.011	0.040	0.024	0.012	0.028	0.011	0.040	0.024	0.012	0.028	0.004	0.009	0.016	0.004	0.021

C3

Round Robin LXIX Laboratory Results

Lab	Total Lycopene, µg/mL					trans-Lycopene, µg/mL					Total β-Cryptoxanthin, µg/mL					Total α-Cryptoxanthin, µg/mL					Total Lutein, µg/mL				
	372	373	374	375	376	372	373	374	375	376	372	373	374	375	376	372	373	374	375	376	372	373	374	375	376
FSV-BA	0.258	0.818	0.526	0.200	0.323	0.130	0.405	0.292	0.114	0.183	0.051	0.045	0.069	0.057	0.069	0.022	0.027	0.035	0.022	0.024					
FSV-BB	0.275	0.809	0.576	0.223	0.360	0.115	0.354	0.270	0.104	0.172	0.043	0.037	0.053	0.049	0.059	0.016	0.013	0.020	0.017	0.017	0.059	0.131	0.090	0.070	0.065
FSV-BC																									
FSV-BD																									
FSV-BE																									
FSV-BF																									
FSV-BG	0.272	0.993	0.549	0.211	0.347	0.138	0.500	0.321	0.131	0.199	0.039	0.023	0.045	0.048	0.059										
FSV-BH	0.276	0.927	0.615	0.220	0.367						0.054	0.045	0.074	0.059	0.076						0.043	0.097	0.075	0.059	0.051
FSV-BJ	0.290	0.954	0.627	0.209	0.366						*nq*	*nq*	*nq*	*nq*	0.052						0.061	0.120	0.104	0.089	0.080
FSV-BK																									
FSV-BL																									
FSV-BM																									
FSV-BN	0.212	1.041	0.639	0.194	0.307						0.037	0.032	0.051	0.045	0.056										
FSV-BO	0.268	0.614	0.462	0.182	0.276						0.029	0.017	0.036	0.040	0.046						0.069	0.125	0.109	0.101	0.073
FSV-BP	0.246	0.980	0.595	0.201	0.514						0.033	0.028	0.045	0.039	0.043										
FSV-BQ																									
FSV-BR																									
FSV-BS	0.369	1.546	1.007	0.306	0.394	0.121	0.493	0.369	0.127	0.139	0.058	0.050	0.083	0.100	0.104						0.045	0.127	0.072	0.073	0.077
FSV-BT	0.263	0.779	0.519	0.203	0.311	0.102	0.317	0.261	0.102	0.156	0.035	0.030	0.041	0.038	0.047	0.020	0.031	0.027	0.019	0.022	0.074	0.079	0.067	0.068	0.060
FSV-BU	0.347	1.096	0.695	0.261	0.399						0.045	0.028	0.048	0.054	0.066										
FSV-BV	0.244	0.964	0.693	0.262	0.412						0.045	0.035	0.071	0.071	0.082										
FSV-BW	0.240	1.470	0.710	0.260	0.460						0.074	0.044	0.064	0.062	0.089										
FSV-CC																									
FSV-CD	0.345	1.132	0.740	0.287	0.476						0.077	0.082	0.096	0.089	0.102	0.047	0.073	0.064	0.048	0.051					
FSV-CE																									
FSV-CG	0.263	1.093	0.673	0.254	0.417	0.121	0.489	0.347	0.131	0.228	0.057	0.071	0.085	0.074	0.092										
FSV-CI																					0.045	0.120	0.075	0.076	0.048
FSV-CW						0.124	0.353	0.281	0.119	0.187	0.041	0.026	0.045	0.046	0.053										
FSV-CZ																									
FSV-DD																									
FSV-DQ	0.369	0.996	0.600	0.204	0.465						0.041	0.039	0.062	0.056	0.077						0.074	0.228	0.143	0.111	0.110
FSV-DV																									
FSV-EE																									
FSV-EZ																									
FSV-FK																									
N	16	16	16	16	16	7	7	7	7	7	16	16	16	16	17	4	4	4	4	4	8	8	8	8	8
Min	0.212	0.614	0.462	0.182	0.276	0.102	0.317	0.261	0.102	0.139	0.029	0.017	0.036	0.038	0.043	0.016	0.013	0.020	0.017	0.017	0.043	0.079	0.067	0.059	0.048
Median	0.270	0.987	0.621	0.216	0.381	0.121	0.405	0.292	0.119	0.183	0.044	0.036	0.058	0.055	0.066	0.021	0.029	0.031	0.021	0.023	0.060	0.122	0.083	0.075	0.069
Max	0.369	1.546	1.007	0.306	0.514	0.138	0.500	0.369	0.131	0.228	0.077	0.082	0.096	0.100	0.104	0.047	0.073	0.064	0.048	0.051	0.074	0.228	0.143	0.111	0.110
SD	0.033	0.160	0.107	0.027	0.070	0.009	0.125	0.043	0.017	0.024	0.012	0.013	0.019	0.014	0.021	0.004	0.013	0.011	0.004	0.005	0.020	0.010	0.019	0.016	0.015
CV	12	16	17	13	18	7	31	15	15	13	27	35	33	25	31	21	46	36	18	22	34	8	23	21	22
N_{past}	17	17	17	17	16	8	8	8	8	8	18	18	18	18	17	8	4	4	4	8	11	9	9	9	9
$Median_{past}$	0.268	0.966	0.598	0.241	0.376	0.116	0.367	0.285	0.117	0.180	0.038	0.030	0.049	0.047	0.059	0.015	0.015	0.021	0.016	0.020	0.056	0.117	0.088	0.073	0.060
SD_{past}	0.059	0.224	0.092	0.013	0.046	0.018	0.064	0.013	0.017	0.021	0.006	0.007	0.006	0.008	0.009	0.002	0.002	0.004	0.004	0.009	0.013	0.031	0.021	0.013	0.010
NAV	0.268	0.993	0.627	0.220	0.367	0.121	0.405	0.292	0.119	0.183	0.044	0.036	0.058	0.055	0.066	0.021	0.029	0.031	0.021	0.023	0.060	0.122	0.083	0.075	0.069
NAU	0.062	0.181	0.124	0.053	0.080	0.021	0.125	0.053	0.021	0.032	0.012	0.013	0.019	0.014	0.021						0.020	0.023	0.019	0.016	0.015

C4

Round Robin LXIX Laboratory Results

	Total Zeaxanthin, µg/mL					Total Lutein&Zeaxanthin, µg/mL					Coenzyme Q10, µg/mL					Phylloquinone (K1), ng/mL					25-hydroxyvitamin D, µg/mL				
Lab	372	373	374	375	376	372	373	374	375	376	372	373	374	375	376	372	373	374	375	376	372	373	374	375	376
FSV-BA						0.083	0.168	0.142	0.108	0.099															
FSV-BB	0.038	0.058	0.046	0.037	0.037	0.097	0.189	0.137	0.107	0.102															
FSV-BC																									
FSV-BD																									
FSV-BE											0.749	1.400	0.930	0.842	0.839	0.323	3.047	0.650	0.568	1.361					
FSV-BF																									
FSV-BG						0.076	0.170	0.124	0.093	0.093															
FSV-BH	0.024	0.025	0.030	0.041	0.033	0.067	0.122	0.105	0.100	0.084															
FSV-BJ											0.717	1.221	0.920	0.849	0.766										
FSV-BK																									
FSV-BL																									
FSV-BM																									
FSV-BN						0.050	0.126	0.099	0.080	0.070											0.027	0.041	0.035	0.020	0.026
FSV-BO	0.006	0.019	0.016	0.011	0.013	*0.076*	*0.144*	*0.125*	*0.112*	*0.086*	0.786	1.796	0.984	0.898	1.001										
FSV-BP						0.082	0.138	0.140	0.118	0.104															
FSV-BQ																									
FSV-BR																									
FSV-BS	0.018	0.019	0.023	0.032	0.040	0.063	0.146	0.095	0.105	0.117															
FSV-BT	0.034	0.034	0.032	0.032	0.030	0.108	0.114	0.099	0.100	0.090	0.613	1.268	0.817	0.713	0.741										
FSV-BU						0.074	0.154	0.118	0.104	0.097	!1.61	1.600	1.140	1.050	1.190						0.013	0.018	0.013	0.009	0.016
FSV-BV						0.070	0.137	0.123	0.116	0.093															
FSV-BW						0.141	0.195	0.162	0.166	0.150	0.298	0.972	0.575	0.436	0.517										
FSV-CC																									
FSV-CD						0.380	0.567	0.465	0.454	0.385															
FSV-CE																									
FSV-CG						0.104	0.231	0.177	0.165	0.145															
FSV-CI	0.014	0.017	0.018	0.021	0.017	*0.059*	*0.137*	*0.093*	*0.097*	*0.065*	0.685	1.410	0.957	0.859	0.813	0.304	3.131	0.599	0.490	1.343					
FSV-CW						0.070	0.100	0.088	0.087	0.071															
FSV-CZ											0.747	1.784	1.086	0.937	0.965										
FSV-DD																									
FSV-DQ	0.023	0.032	0.030	0.033	0.033	*0.097*	*0.260*	*0.173*	*0.144*	*0.143*															
FSV-DV																									
FSV-EE											0.773	1.482	0.993	0.890	0.870										
FSV-EZ											0.773	1.482	0.993	0.890	0.870										
FSV-FK																									
N	7	7	7	7	7	17	17	17	17	17	9	10	10	10	10	2	2	2	2	2	2	2	2	2	2
Min	0.006	0.017	0.016	0.011	0.013	0.050	0.100	0.088	0.080	0.065	0.298	0.972	0.575	0.436	0.517	0.304	3.047	0.599	0.490	1.343	0.013	0.018	0.013	0.009	0.016
Median	0.023	0.025	0.030	0.032	0.033	0.076	0.146	0.124	0.107	0.097	0.747	1.446	0.971	0.875	0.854	0.314	3.089	0.625	0.529	1.352	0.020	0.029	0.024	0.014	0.021
Max	0.038	0.058	0.046	0.041	0.040	0.380	0.567	0.465	0.454	0.385	0.786	1.796	1.140	1.050	1.190	0.323	3.131	0.650	0.568	1.361	0.027	0.041	0.035	0.020	0.026
SD	0.013	0.010	0.010	0.007	0.006	0.019	0.036	0.037	0.015	0.019	0.044	0.246	0.068	0.043	0.148										
CV	58	42	35	21	18	25	24	30	14	20	6	17	7	5	17										
N_{past}	10	8	8	8	8	19	18	18	18	17	8	5	5	5	6	0	0	0	0	0	0	0	0	0	0
$Median_{past}$	0.024	0.032	0.030	0.036	0.029	0.085	0.142	0.130	0.110	0.097	0.629	1.440	0.990	0.887	0.922										
SD_{past}	0.009	0.017	0.017	0.013	0.015	0.018	0.026	0.025	0.009	0.020	0.128	0.169	0.067	0.037	0.150										
NAV	0.023	0.025	0.030	0.032	0.033	0.076	0.146	0.124	0.107	0.097	0.747	1.446	0.971	0.875	0.854										
NAU	0.013	0.010	0.010	0.009	0.009	0.019	0.036	0.037	0.022	0.020	0.075	0.246	0.097	0.088	0.148										

C5

Round Robin LXIX Laboratory Results

Analytes Reported By One Laboratory

Analyte	Code	372	373	374	375	376
Ubiquinol	FSV-BW	*nd*	0.424	0.376	0.282	0.167
Ubiquinone	FSV-BW	0.298	0.548	0.200	0.154	0.350
Phytofluene	FSV-BS	0.221	1.090	0.585	0.138	0.331
Phytoene	FSV-BS	*nd*	0.100	0.030	*nd*	*nd*

Term	Legend
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
SD	Adjusted median absolute deviation from the median of the non-NIST results
CV	Coefficient of Variation for (non-NIST) results: 100*SD/Median
N_{past}	Mean of N(s) from past RR(s)
$Median_{past}$	Mean of Median(s) from past RR(s)
SD_{past}	Pooled SD from past RR(s)
NIST	Mean of NIST results
NAV	NIST Assigned Value = (Median + NIST)/2 for analytes reported by NIST = Median for analytes reported by ≥ 5 labs but not NIST
NAU	NIST Assigned Uncertainty: $\sqrt{(S^2 + S_{btw}^2)}$ S is the maximum of (0.05*NAV, SD, SD_{past}, eSD) and S_{btw} is the standard deviation between Median and NIST. The expected long-term SD, eSD, is defined in: Duewer et al., Anal Chem 1997;69(7):1406-1413.
nq	Detected but not quantitatively determined
≥x	Concentration greater than or equal to x
!	Discrepant value: heterogeneous serum, damaged sample, malfunction, etc.
italics	Not explicitly reported but calculated by NIST from reported values

Round Robin LXIX Laboratory Results

Comparability Summary

Lab	TR	aT	g/bT	bC	tbC	aC	TLy	TbX	TLu	TZ	L&Z
FSV-BA	1	1	1	1	1	1	1	1			1
FSV-BB	1	1	1	1	1	1	1	1	1	2	1
FSV-BC	1										
FSV-BD	1	1									
FSV-BE	2	2	1	1							
FSV-BF	1	1		1							
FSV-BG	1	1	1	1		1	1	1			1
FSV-BH	3	1	2	1	1		1	1	1	1	1
FSV-BJ	1	1	1	1			1		1		
FSV-BK	2	2									
FSV-BL	1	1									
FSV-BM	1	2									
FSV-BN	2	3		1		1	1	1			2
FSV-BO	2	2	2	1		1	2	2	2	2	1
FSV-BP	1	2		1		4	1	1			1
FSV-BQ	4	4									
FSV-BR	1	2									
FSV-BS	2			4	4	4	3	2	1	1	1
FSV-BT	2	1	1	2	1	1	1	1	2	1	2
FSV-BU	2	2	2	1		1	1	1			1
FSV-BV	2	1	1	1		1	1	1			1
FSV-BW	1	1	1	1		2	2	2			3
FSV-CC	2	4									
FSV-CD	2	3	2	4		4	2	3			4
FSV-CE	1	1		1							
FSV-CG	2	1	2	1	2	1	1	2			3
FSV-CI	2	1	1	1		2			1	2	1
FSV-CW	2	1	1	1		1		1			2
FSV-CZ	1	1	1	1							
FSV-DD	3										
FSV-DQ		4	2	4		2	2	1	4	1	3
FSV-DV	2	2									
FSV-EZ	1	1	1		1						
FSV-FK	1	1									
n	33	31	18	22	7	16	16	16	8	7	17

	TR	aT	g/bT	bC	tbC	aC	TLy	TbX	TLu	TZ	L&Z
% 1	52	58	67	77	71	56	75	69	63	57	59
% 2	39	26	33	9	14	25	19	25	25	43	18
% 3	6	6	0	0	0	0	6	6	0	0	18
% 4	3	10	0	14	14	19	0	0	13	0	6

Label	Definition
Lab	Participant code
TR	Total Retinol
aT	α-Tocopherol
g/bT	γ/β-Tocopherol
bC	Total β-Carotene
tbC	trans-β-Carotene
aC	Total α-Carotene
TLy	Total Lycopene
TbX	Total β-Cryptoxanthin
TLu	Total Lutein
TZ	Total Zeaxanthin
L&Z	Total Lutein & Zeaxanthin

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.

Appendix D. Representative "Individualized Report" for RR69

Each participant in RR69 received an "Individualized Report" reflecting their reported results. Each report included a detailed analysis for analytes that were assayed by at least five participants. The following analytes met this criterion in RR69:

- Total Retinol
- Retinyl Palmitate
- α-Tocopherol
- γ/β-Tocopherol
- Total β-Carotene
- *trans*-β-Carotene
- Total *cis*-β-Carotene
- Total α-Carotene
- Total Lycopene
- *trans*-Lycopene
- Total β-Cryptoxanthin
- Total Lutein
- Total Zeaxanthin
- Total Lutein & Zeaxanthin
- Coenzyme Q10

The following fourteen pages are the "Individualized Report" for the analytes evaluated by participant FSV-BA.

Individualized Round Robin LXIX Report: FSV-MA

Summary

	Serum 372			Serum 373			Serum 374			Serum 375			Serum 376		
Analyte	You	NAV	n	You	NAV	n	You	NAV	n	You	NAV	n	You	NAV	n
Total Retinol	0.365	0.350	32	0.715	0.660	32	0.537	0.509	32	0.375	0.359	32	0.775	0.735	32
Retinyl Palmitate	0.03	0.01	8	0.2	0.1	10	0.1	0.0	8	0.02	0.01	7	0.05	0.05	9
α-Tocopherol	5.67	5.79	32	18.15	18.91	32	9.94	10.53	32	6.61	6.78	32	10.10	10.62	32
γ/β-Tocopherol	1.517	1.394	19	2.469	2.297	19	1.501	1.431	19	1.933	1.844	19	2.554	2.474	19
δ-Tocopherol	0.086	0.088	4	0.199	0.221	4	0.062	0.065	4	0.083	0.119	4	0.255	0.275	4
Total β-Carotene	0.078	0.078	22	0.388	0.415	22	0.226	0.245	22	0.086	0.092	22	0.260	0.280	22
trans-β-Carotene	0.075	0.075	7	0.372	0.342	7	0.216	0.216	7	0.082	0.086	7	0.248	0.261	7
Total cis-β-Carotene	0.003	0.075	3	0.017	0.342	5	0.010	0.216	5	0.003	0.086	4	0.011	0.261	5
Total α-Carotene	0.007	0.010	17	0.015	0.018	15	0.031	0.032	17	0.005	0.009	17	0.048	0.047	19
Total Lycopene	0.258	0.268	17	0.818	0.993	17	0.526	0.627	17	0.200	0.220	17	0.323	0.367	17
trans-Lycopene	0.130	0.121	7	0.405	0.405	7	0.292	0.292	7	0.114	0.119	7	0.183	0.183	7
Total β-Cryptoxanthin	0.051	0.044	16	0.045	0.036	16	0.069	0.058	16	0.057	0.055	16	0.069	0.066	17
Total α-Cryptoxanthin	0.022	0.021	4	0.027	0.029	4	0.035	0.031	4	0.022	0.021	4	0.024	0.023	4
Total Lutein&Zeaxanthin	0.083	0.076	17	0.168	0.146	17	0.142	0.124	17	0.108	0.107	17	0.099	0.097	17

You : Your reported values for the listed analytes (micrograms/milliliter)
NAV : NIST Assigned Values, here equal to this RR's median
n : Number of non-NIST laboratories reporting quantitative values for this analyte in this serum

D2

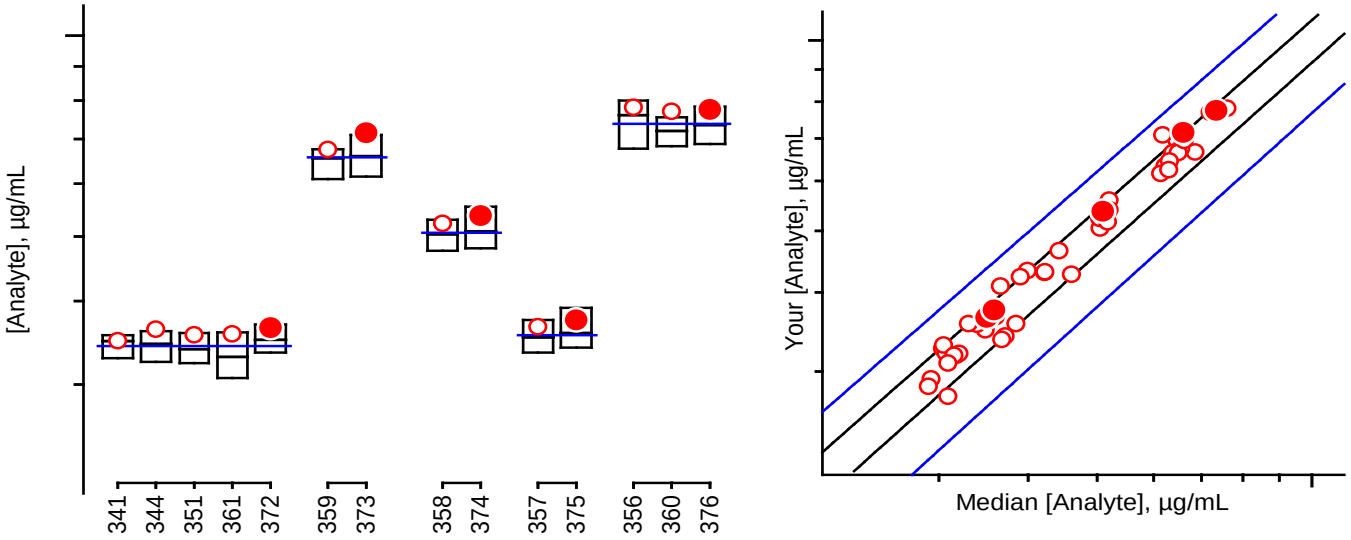
Please check our records against your records. Send corrections and/or updates to...

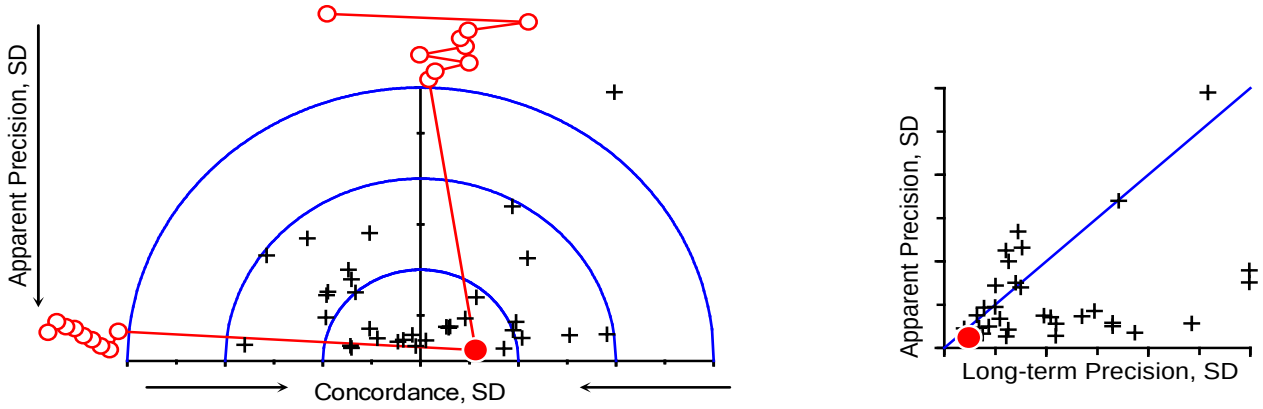
Micronutrients Measurement Quality Assurance Program
National Institute of Standards and Technology
100 Bureau Drive Stop 8392
Gaithersburg, MD 20899-8392 USA

Tel: (301) 975-3935
Fax: (301) 977-0685
Email: david.duewer@nist.gov

Individualized RR LXIX Report: FSV-BA

Total Retinol, µg/mL





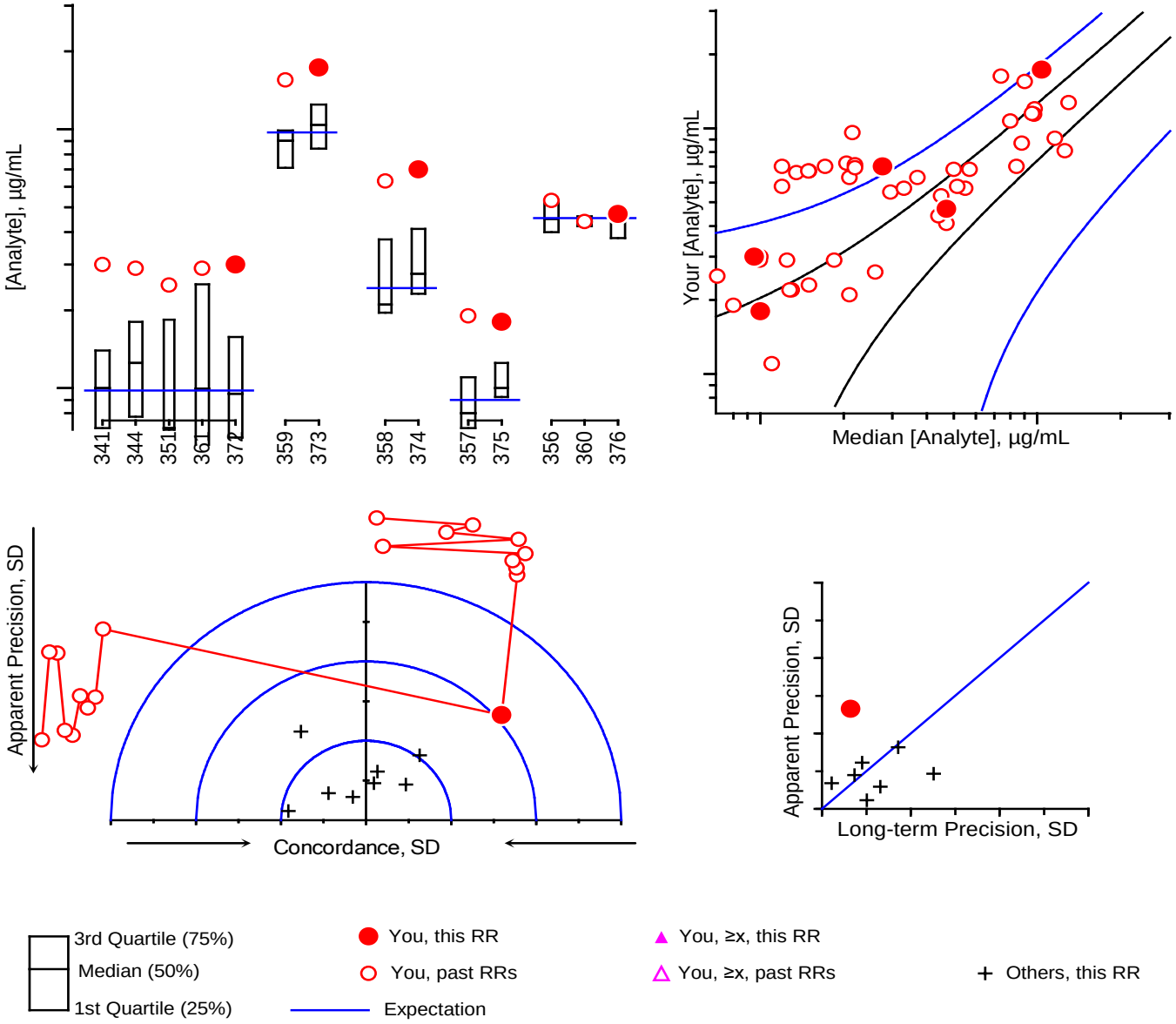
3rd Quartile (75%)
Median (50%)
1st Quartile (25%)
You, this RR
You, past RRs
Expectation
You, ≥x, this RR
You, ≥x, past RRs
Others, this RR

For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

Serum	Comments	History
#372	Fresh-frozen, native, multi-donor: SRM968d	63:#341, 63:#344, 64:#351, 66:#361
#373	Fresh-frozen, native, multi-donor: SRM968e III	66:#359
#374	Fresh-frozen, native, multi-donor: SRM 968e II	66:#358
#375	Fresh-frozen, native, multi-donor: SRM 968e I	66:#357
#376	Fresh-frozen, native, multi-donor: prepared in 2008	64:#356, 66:#360

Individualized RR LXIX Report: FSV-BA

Retinyl Palmitate, µg/mL



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Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

Serum	Comments	History
#372	Fresh-frozen, native, multi-donor: SRM968d	63:#341, 63:#344, 64:#351, 66:#361
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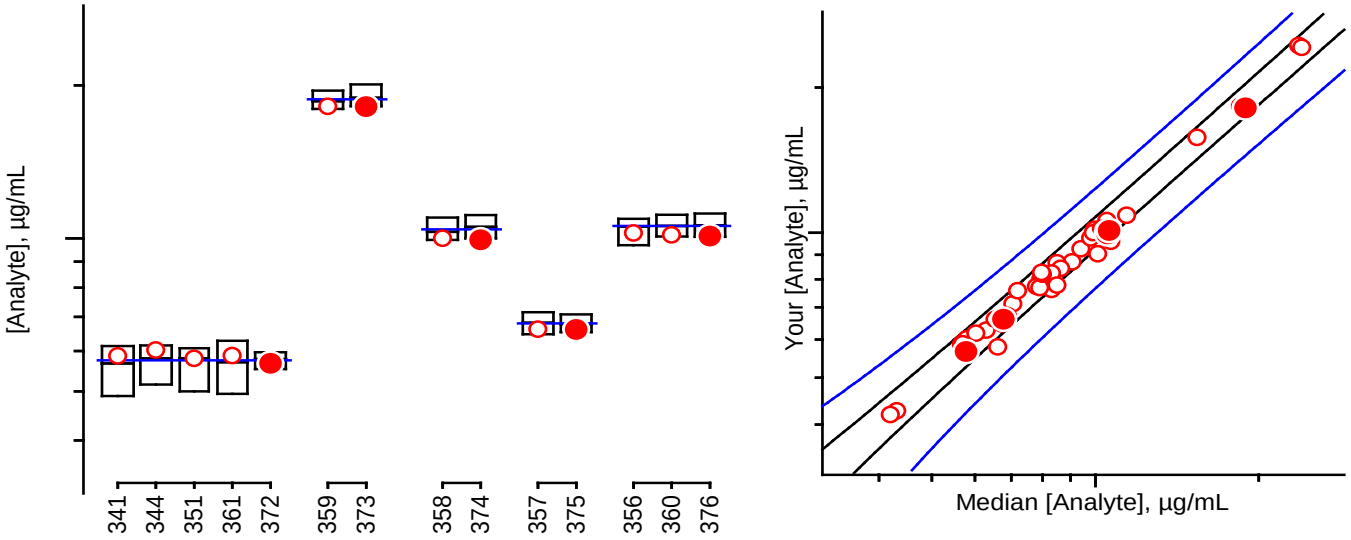
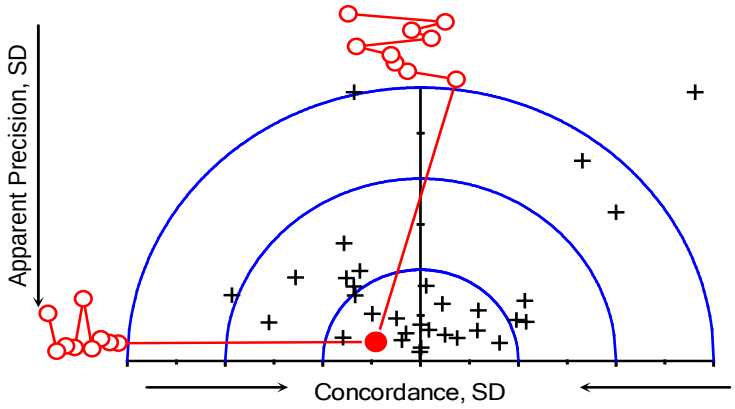
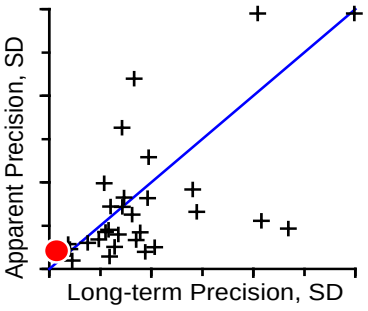
#374	Fresh-frozen, native, multi-donor: SRM 968e II	66:#358
#375	Fresh-frozen, native, multi-donor: SRM 968e I	66:#357
#376	Fresh-frozen, native, multi-donor: prepared in 2008	64:#356, 66:#360

Individualized RR LXIX Report: FSV-BA

α-Tocopherol, µg/mL







3rd Quartile (75%)
Median (50%)
1st Quartile (25%)
You, this RR
You, past RRs
Expectation
You, ≥x, this RR
You, ≥x, past RRs
Others, this RR

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Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

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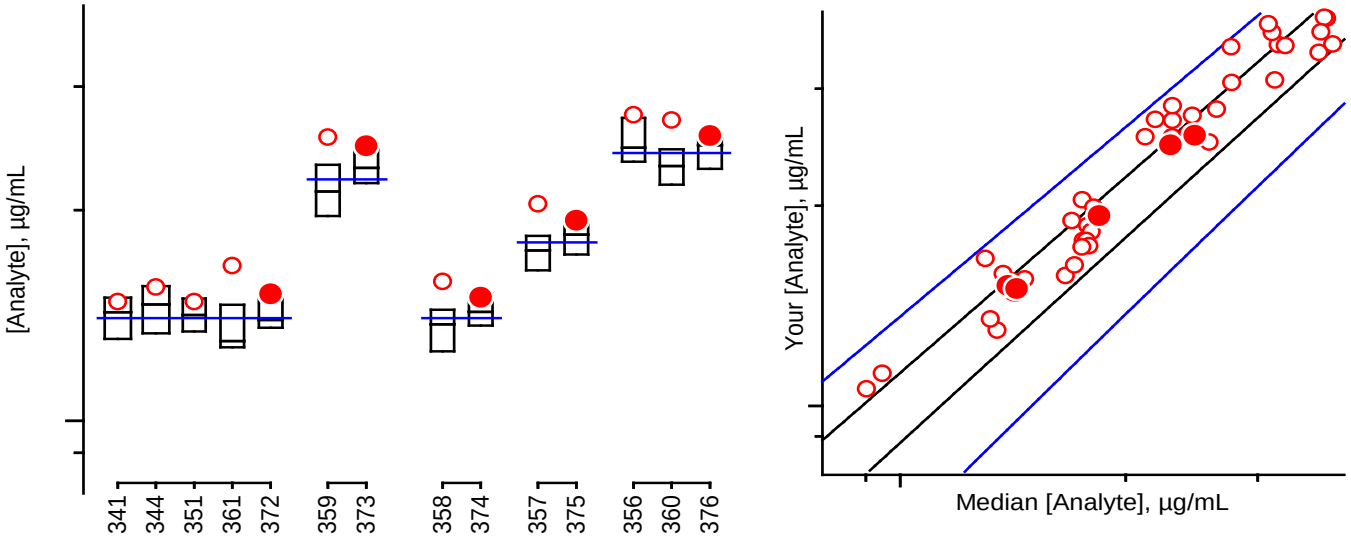
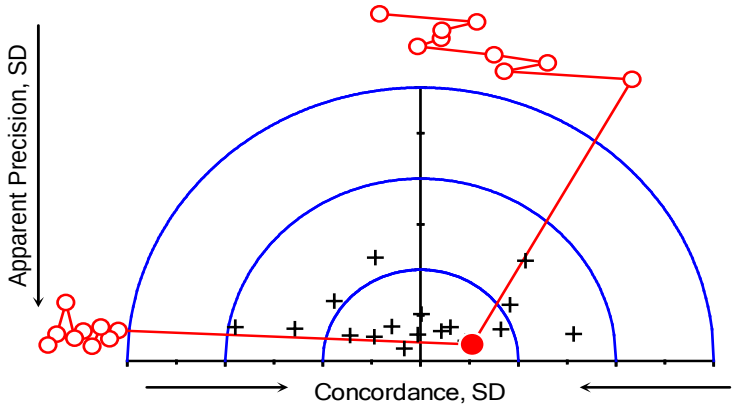
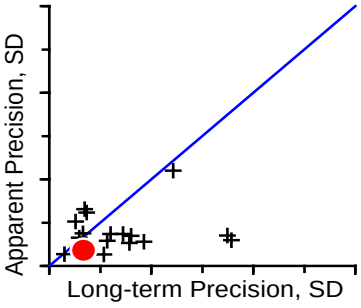
#374	Fresh-frozen, native, multi-donor: SRM 968e II	66:#358
#375	Fresh-frozen, native, multi-donor: SRM 968e I	66:#357
#376	Fresh-frozen, native, multi-donor: prepared in 2008	64:#356, 66:#360

Individualized RR LXIX Report: FSV-BA

γ/β-Tocopherol, µg/mL







3rd Quartile (75%)
Median (50%)
1st Quartile (25%)
You, this RR
You, past RRs
Expectation
You, ≥x, this RR
You, ≥x, past RRs
Others, this RR

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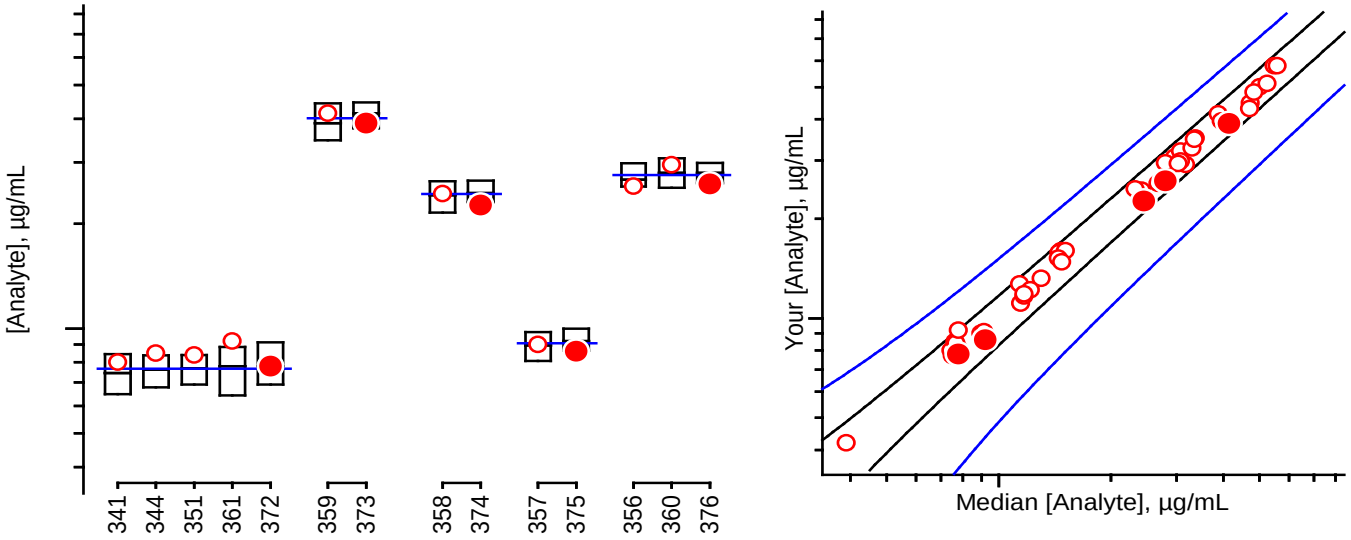
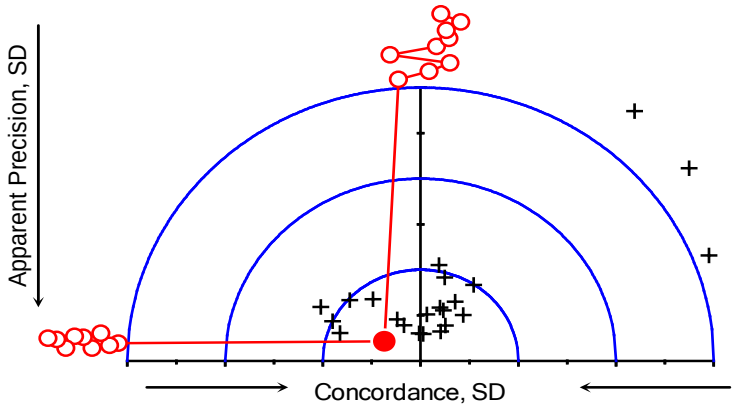
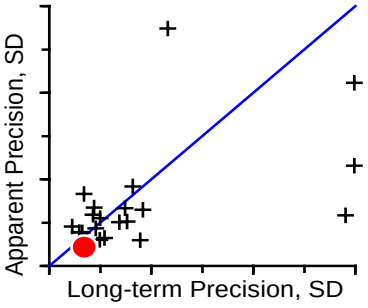
#375	Fresh-frozen, native, multi-donor: SRM 968e I	66:#357
#376	Fresh-frozen, native, multi-donor: prepared in 2008	64:#356, 66:#360

Individualized RR LXIX Report: FSV-BA

Total β-Carotene, µg/mL







3rd Quartile (75%)
Median (50%)
1st Quartile (25%)
You, this RR
You, past RRs
Expectation
You, ≥x, this RR
You, ≥x, past RRs
Others, this RR

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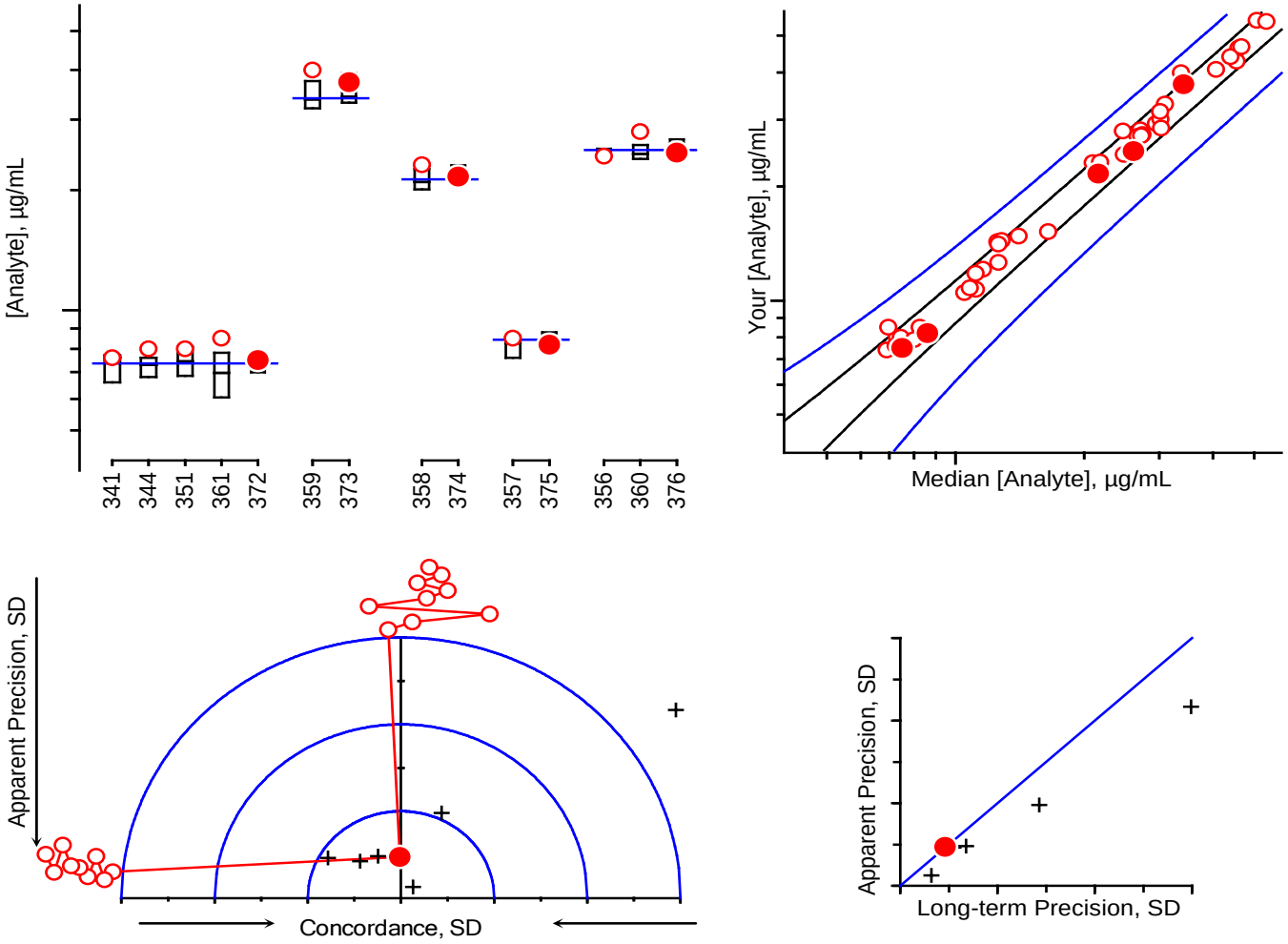
#375	Fresh-frozen, native, multi-donor: SRM 968e I	66:#357
#376	Fresh-frozen, native, multi-donor: prepared in 2008	64:#356, 66:#360

Individualized RR LXIX Report: FSV-BA

trans-β-Carotene, µg/mL



3rd Quartile (75%)
Median (50%)
1st Quartile (25%)
You, this RR
You, past RRs
Expectation
You, ≥x, this RR
You, ≥x, past RRs
Others, this RR

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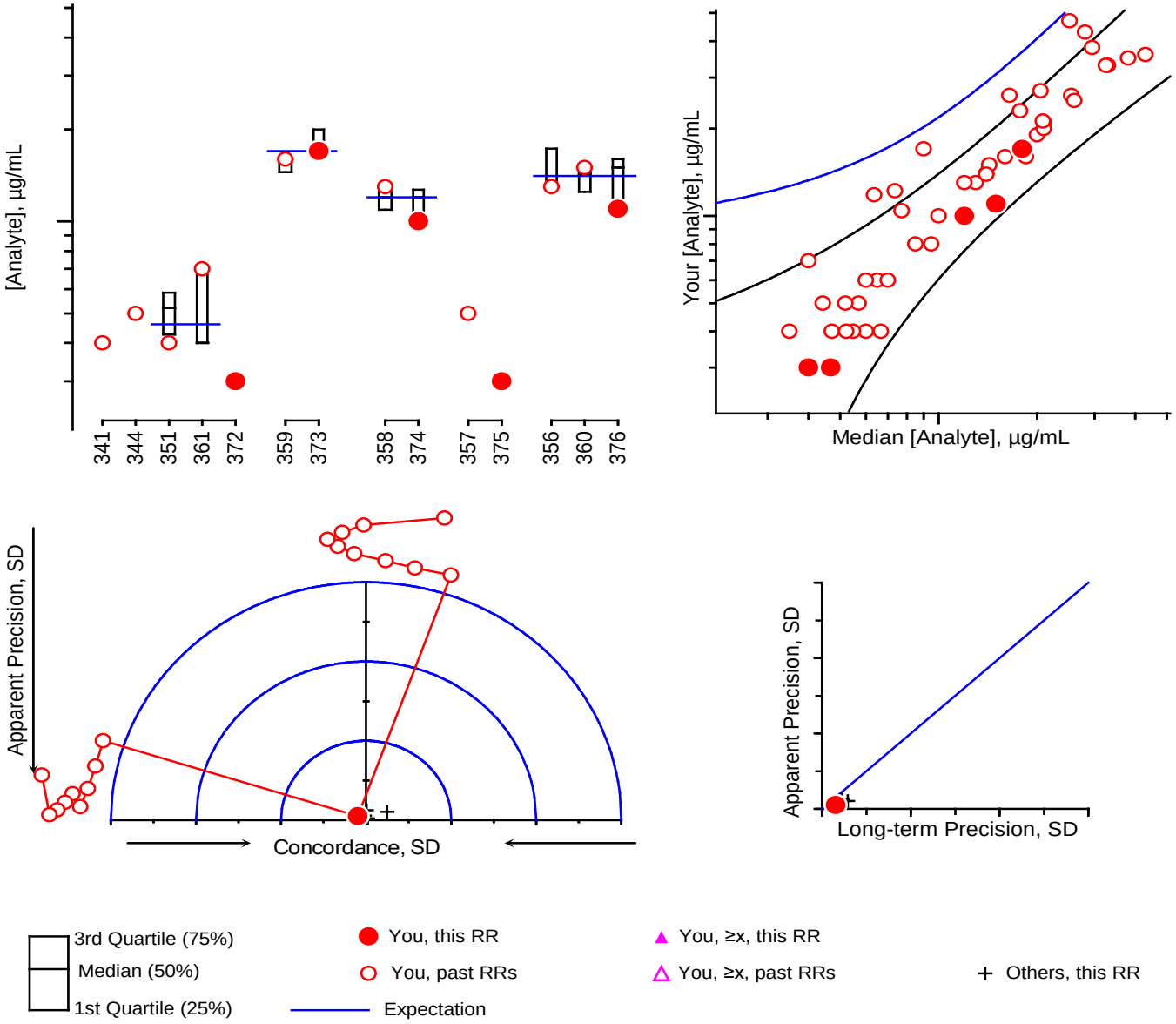
#376	Fresh-frozen, native, multi-donor: prepared in 2008	64:#356, 66:#360

Individualized RR LXIX Report: FSV-BA

Total cis-β-Carotene, µg/mL



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Serum	Comments	History
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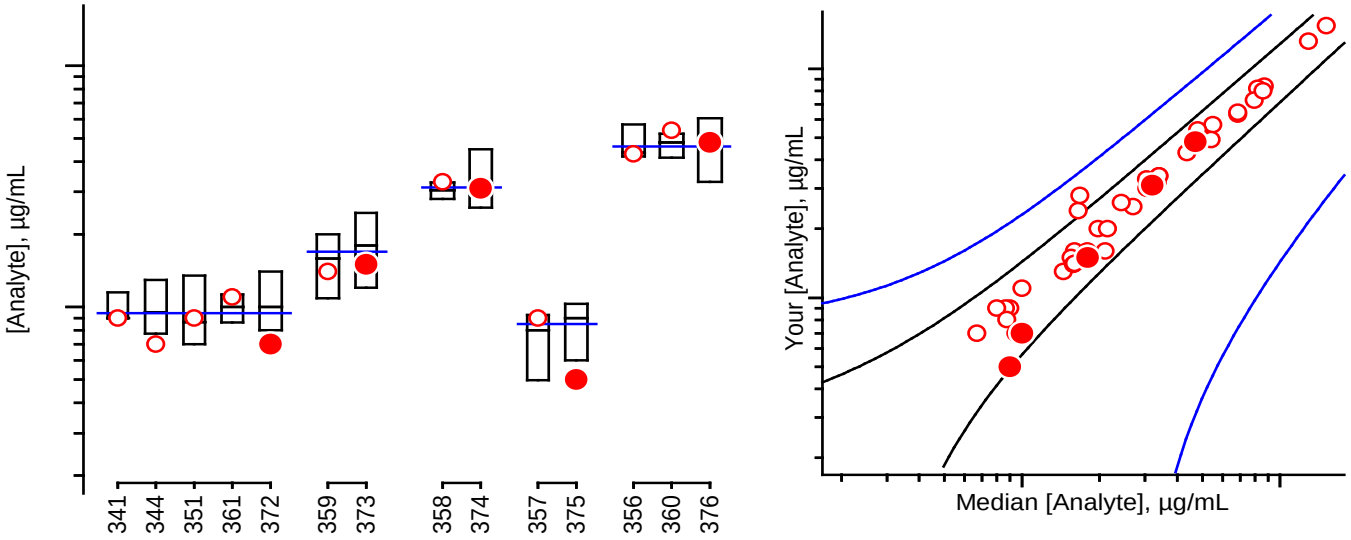
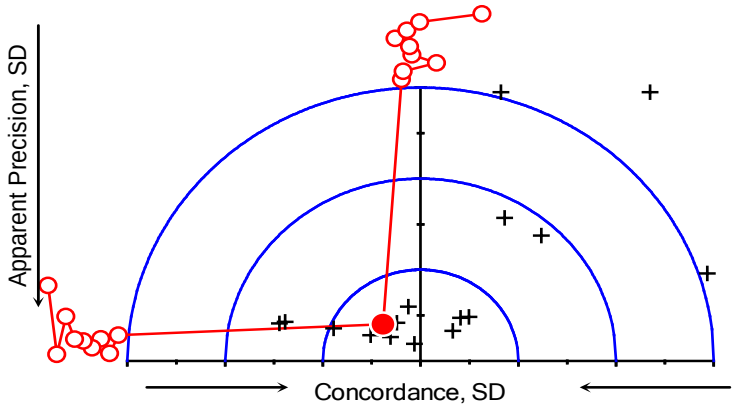
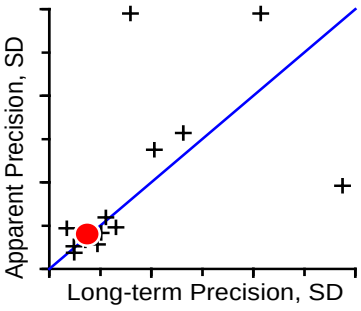
#376	Fresh-frozen, native, multi-donor: prepared in 2008	64:#356, 66:#360

Individualized RR LXIX Report: FSV-BA

Total α-Carotene, µg/mL







3rd Quartile (75%)
Median (50%)
1st Quartile (25%)
You, this RR
You, past RRs
Expectation
You, ≥x, this RR
You, ≥x, past RRs
Others, this RR

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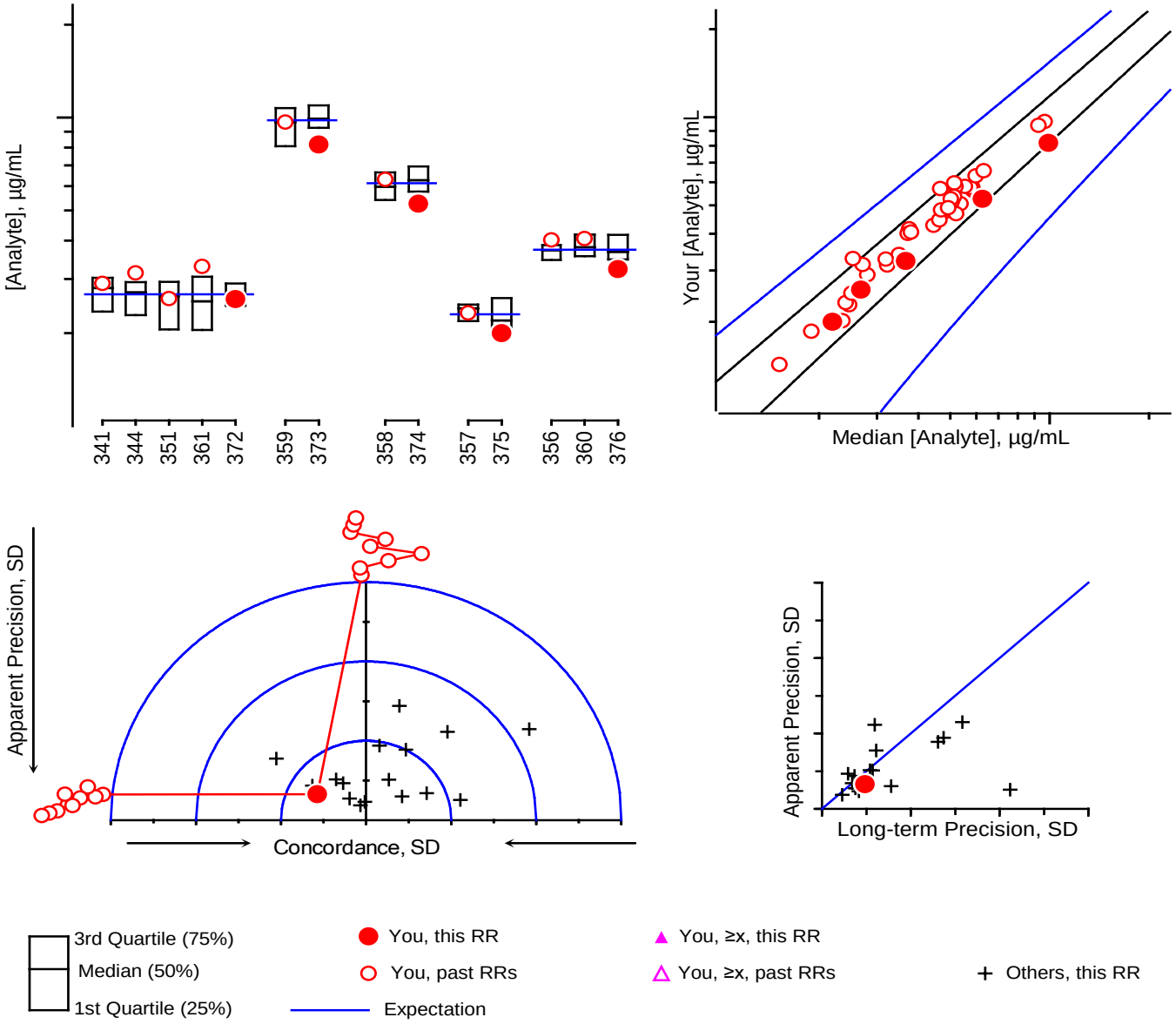
#376	Fresh-frozen, native, multi-donor: prepared in 2008	64:#356, 66:#360

Individualized RR LXIX Report: FSV-BA

Total Lycopene, µg/mL



For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

Serum	Comments	History
#372	Fresh-frozen, native, multi-donor: SRM968d	63:#341, 63:#344, 64:#351, 66:#361
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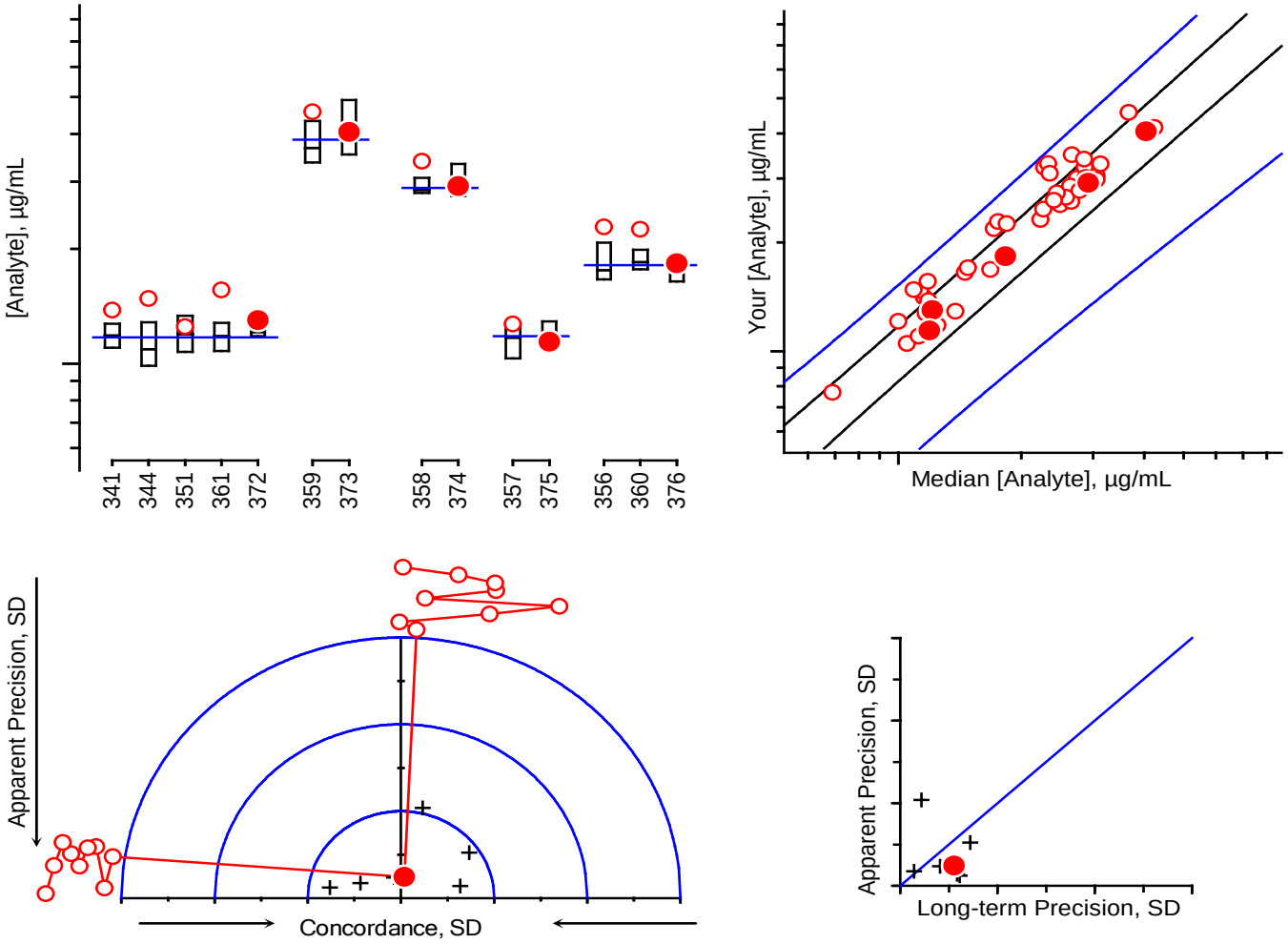
#375	Fresh-frozen, native, multi-donor: SRM 968e I	66:#357
#376	Fresh-frozen, native, multi-donor: prepared in 2008	64:#356, 66:#360

Individualized RR LXIX Report: FSV-BA

trans-Lycopene, µg/mL



3rd Quartile (75%)
Median (50%)
1st Quartile (25%)
You, this RR
You, past RRs
Expectation
You, ≥x, this RR
You, ≥x, past RRs
Others, this RR

For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

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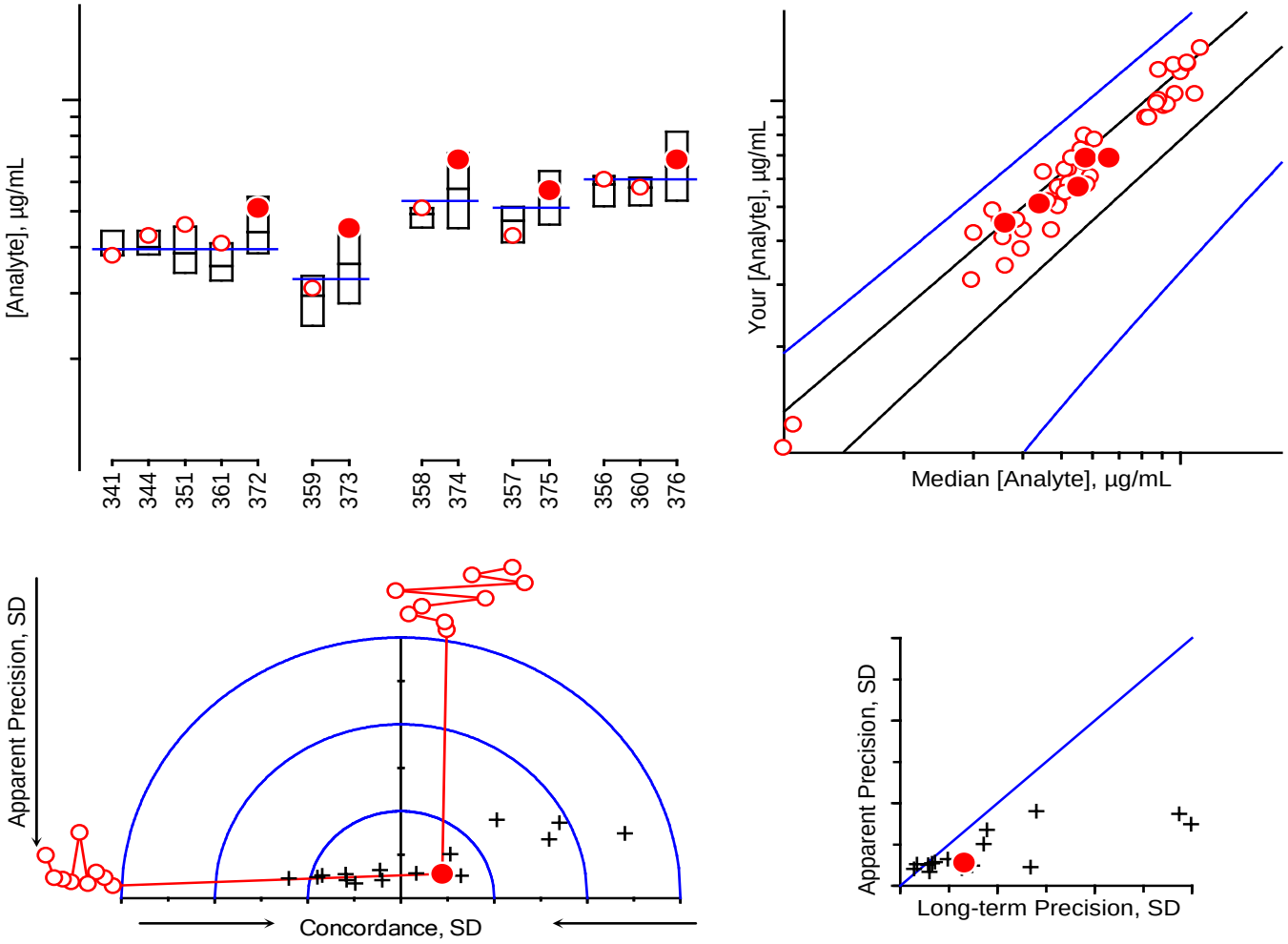
#375	Fresh-frozen, native, multi-donor: SRM 968e I	66:#357
#376	Fresh-frozen, native, multi-donor: prepared in 2008	64:#356, 66:#360

Individualized RR LXIX Report: FSV-BA

Total β-Cryptoxanthin, µg/mL



3rd Quartile (75%)
Median (50%)
1st Quartile (25%)
You, this RR
You, past RRs
Expectation
You, ≥x, this RR
You, ≥x, past RRs
Others, this RR

For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

Serum	Comments	History
#372	Fresh-frozen, native, multi-donor: SRM968d	63:#341, 63:#344, 64:#351, 66:#361
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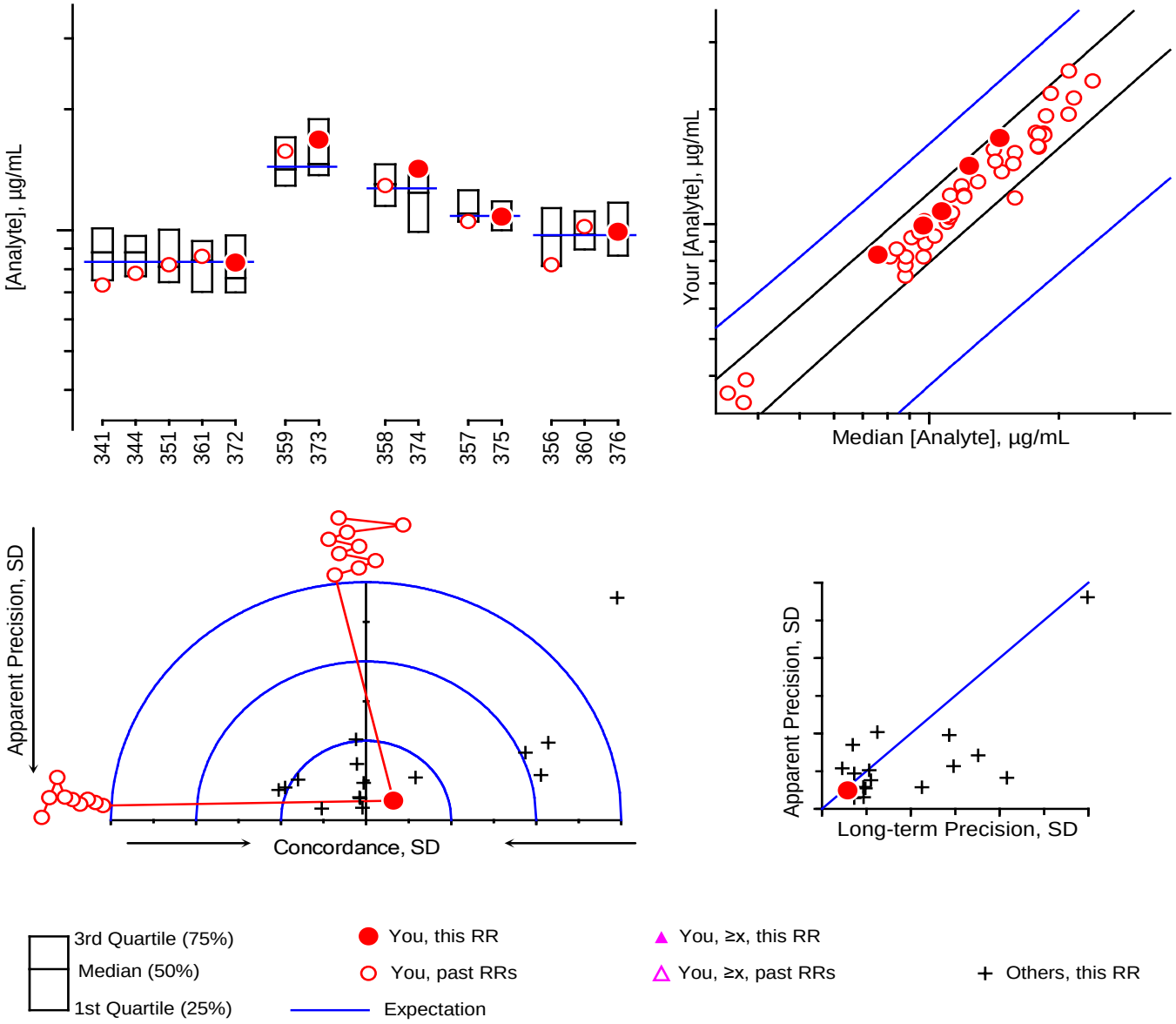
#375	Fresh-frozen, native, multi-donor: SRM 968e I	66:#357
#376	Fresh-frozen, native, multi-donor: prepared in 2008	64:#356, 66:#360

Individualized RR LXIX Report: FSV-BA

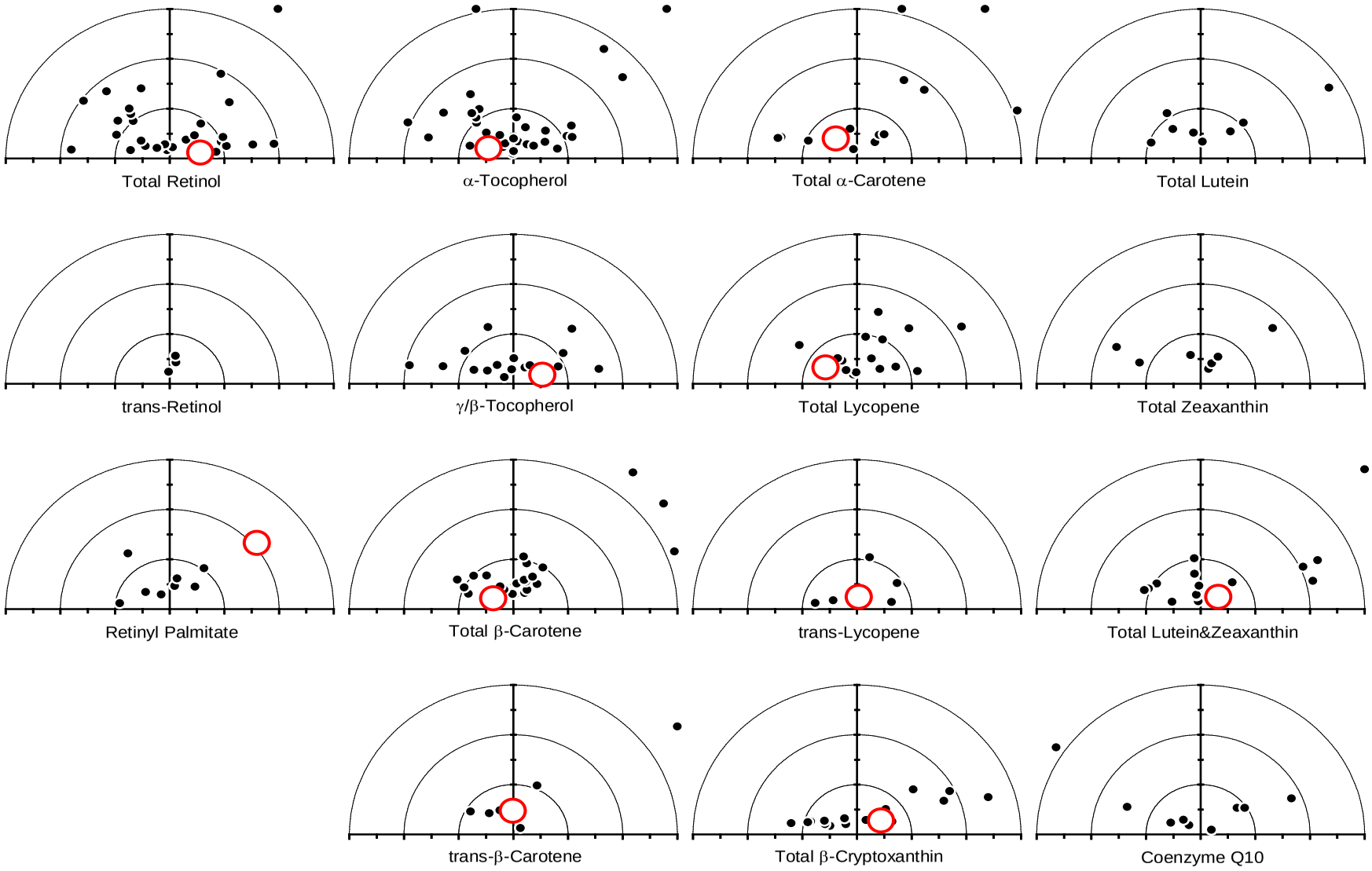
Total Lutein&Zeaxanthin, µg/mL



For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

Serum	Comments	History
#372	Fresh-frozen, native, multi-donor: SRM968d	63:#341, 63:#344, 64:#351, 66:#361
#373	Fresh-frozen, native, multi-donor: SRM968e III	66:#359
#374	Fresh-frozen, native, multi-donor: SRM 968e II	66:#358
#375	Fresh-frozen, native, multi-donor: SRM 968e I	66:#357
#376	Fresh-frozen, native, multi-donor: prepared in 2008	64:#356, 66:#360

Individualized Round Robin LXIX Report: FSV-BA
Graphical Comparability Summary



D15

Appendix E. Shipping Package Inserts for RR34

The following five items were included in each package shipped to an RR34 participant:

- Cover letter
- Protocol for Preparation and Analysis of the Ascorbic Acid Solid Control Material
- Preparation and Validation of Ascorbic Acid Solid Control Material Datasheet
- Analysis of Control Materials and Test Samples Datasheet
- Packing List and Shipment Receipt Confirmation Form

The cover letter, preparation protocol, and the two datasheets were enclosed in a sealed waterproof bag along with the samples themselves. The packing list was placed at the top of the shipping box, between the cardboard covering and the foam insulation.

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

November 22, 2010

Dear Colleague:

The samples within this package constitute Vitamin C Round Robin 34 (RR34) of the 2011Micronutrients Measurement Quality Assurance Program. RR34 consists of four vials of frozen serum *test samples* (#341, #342, #343, and #344), one vial of frozen *control serum* (CS #2), and one vial of ascorbic acid *solid control material* (Control). Please follow the attached protocols when you prepare and analyze these samples. If you cannot prepare the *solid control* solutions gravimetrically, please prepare equivalent solutions volumetrically and report the exact volumes used. (Routine 0.5 g gravimetric measurements are generally 10-fold more accurate than routine 0.5 mL volumetric measurements.)

Please use the control serum to validate the performance of your measurement system before you analyze the *test samples*. The target value for *CS* #2 is 28.05 µmol/L of sample; the ≈95% confidence interval for the target value is 27.56 to 28.54 µmol/L of sample.

Please be aware that sample contact with any oxidant-contaminated surface (vials, glassware, etc.) may degrade your measurement system's performance (SA Margolis and E Park, "Stability of Ascorbic Acid in Solutions in Autosampler Vials", *Clinical Chemistry* 2001, *47*(8), 1463-1464). You should suspect such degradation if you observe unusually large variation in replicate analyses.

Please report your results (using the attached form) for RR34 by e-mail to david.duewer@nist.gov or fax to 301-977-0685 by **March 7, 2011**. If you have questions or comments regarding this study, please call me at (301) 975-3120 or e-mail me at jbthomas@nist.gov.

Sincerely,

Jeanice Brown Thomas
Program Coordinator/Research Chemist
Analytical Chemistry Division
Chemical Science and Technology Laboratory

Enclosures: Protocols, Preparation and Analysis of Control Materials and Analysis of Test Samples
RR34 Report Form for Ascorbic Acid Solid Control Material Preparation
RR34 Report Form for Control Material and Test Sample Analyses

NIST

Micronutrient Measurement Quality Assurance Program for
Vitamin C

Please Read Through Completely BEFORE Analyzing Samples

Protocol for Preparation and Analysis of the Ascorbic Acid Solid Control Material

The *ascorbic acid solid control material* (in the amber vial) should be prepared and used in the following manner:

1) Prepare at least 500 mL of 5% mass fraction metaphosphoric acid (MPA) in distilled water. This solution will be referred to as the "Diluent" below.

2) Weigh 0.20 to 0.22 g of the ascorbic acid solid control material to 0.0001 g (if possible), dissolve it in the Diluent in a 100 mL volumetric flask, and dilute with the Diluent to the 100 mL mark. Weigh the amount of Diluent added to 0.1 g. Record the weights. The resulting material will be referred to as the "Stock Solution" below.

3) Prepare three dilute solutions of the Stock Solution as follows:

   Dilute Solution 1: Weigh 0.500 mL of the Stock Solution to 0.0001 g into a 100 mL volumetric flask; dilute with Diluent to the 100 mL mark. Record the weight.

   Dilute Solution 2: Weigh 0.250 mL of the Stock Solution to 0.0001 g into a 100 mL volumetric flask; dilute with Diluent to the 100 mL mark. Record the weight.

   Dilute Solution 3: Weigh 0.125 mL of the Stock Solution to 0.0001 g into a 100 mL volumetric flask; dilute with Diluent to the 100 mL mark. Record the weight.

4) Calculate and record the total ascorbic acid concentrations, [TAA], in these Dilute Solutions. If you follow the above gravimetric preparation directions, the [TAA] in μmol/L is calculated:

$$[\mathrm{TAA}]_{\mathrm{DS}} = \frac{(\text{g Stock Solution in Dilute Solution}) \cdot (\text{g AA in Stock Solution}) \cdot (56785\ \mu\text{mol/g} \cdot \text{L})}{(\text{g AA in Stock Solution}) + (\text{g Diluent in Stock Solution})}$$

   For example, if you prepared the Stock Solution with 0.2000 g of solid ascorbic acid and 103.0 g of Diluent, then 0.5 mL of the Stock Solution should weigh (0.2+103)/200 = 0.52 g and $[\mathrm{TAA}]_{\mathrm{DS1}}$ = (0.52 g)(0.2 g)·(56785 μmol/g·L)/(0.2 + 103 g) = 57.2 μmol/L. Likewise, 0.25 mL of the Stock Solution should weigh 0.26 g and $[\mathrm{TAA}]_{\mathrm{DS2}}$ = 29.4 μmol/L and 0.125 mL should weigh 0.13 g and $[\mathrm{TAA}]_{\mathrm{DS3}}$ = 14.2 μmol/L.

5) Measure the ultraviolet absorbance spectrum of Dilute Solution 1 against the Diluent as the blank using paired 1 cm path length cuvettes. Record the absorbance at 242, 243, 244, and 245 nm. Record the maximum absorbance (A_{max}) within this region. Record the wavelength (λ_{max}) at which this maximum occurs.

   The extinction coefficient ($E^{1\%}$) of ascorbic acid at λ_{max} (using a cell with a 1 cm path length) of Dilute Solution #1 can be calculated:

$$E^{1\%}\left(\frac{dL}{g \cdot cm}\right) = \frac{(A_{max}) \cdot ((\text{g AA in Stock Solution}) + (\text{g Diluent in Stock Solution}))}{(\text{g Stock Solution in Dilute Solution 1}) \cdot (\text{g AA in Stock Solution})}$$

If your spectrophotometer is properly calibrated, λ_{max} should be between 243 and 244 nm and $E^{1\%}$ should be 550 ± 30 dL/g·cm. If they are not, you should recalibrate the wavelength and/or absorbance axes of your spectrophotometer and repeat the measurements.

6) Measure and record the concentration of total ascorbic acid in all three dilute solutions and in the 5% MPA Diluent in duplicate using *exactly* the same method that you will use for the serum control materials and test samples, including any enzymatic treatment. We recommend that you analyze these solutions in the following order: Diluent, Dilute Solution 1, Dilute Solution 2, Dilute Solution 3, Dilute Solution 3, Dilute Solution 2, Dilute Solution 1, Diluent.

   a) Compare the values of the duplicate measurements. *Are you satisfied that your measurement precision is adequate?*

   b) Compare the measured with the calculated [TAA] values. This is most conveniently done by plotting the measured values on the y-axis of a scatterplot against the calculated values on the x-axis. The line through the four {calculated, measured} data pairs should go through the origin with a slope of 1.0. *Are you satisfied with the agreement between the measured and calculated values?*

   Do **not** analyze the serum control materials or test samples until you are satisfied that your system is performing properly!

7) Once you have confirmed that your system is properly calibrated, analyze the serum control CS #2 (see protocol below). The target values for this materials is 28.1 ±1.0 µmol/L of sample.

   If your measured values are not close to this value, please review your sample preparation procedure and whether you followed *exactly* the same measurement protocol the solutions prepared from the solid control material as you used for these serum controls. If the protocols differ, please repeat from Step 6 using the proper protocol. If the proper protocol was used, your measurement system may not be suitable for MPA-preserved samples; please contact us at 301-975-3120 or jbthomas@NIST.gov.

   Do **not** analyze the test samples until you are satisfied that your system is performing properly and is suitable for the analysis of MPA-preserved serum!

Protocol for Analysis of the Serum Control Materials and Test Samples

The *serum control material* and *test samples* are in sealed ampoules. They were prepared by adding equal volumes of 10% MPA to spiked human serum. We have checked the samples for stability and homogeneity. Only the total ascorbic acid is stable. While these samples contain some dehydroascorbic acid, its content is variable. Therefore, only total ascorbic acid should be reported. The *serum control material* and *test samples* should be defrosted by warming at 20 °C for not more than 10 min otherwise some irreversible degradation may occur.

Each *serum test sample* contains between 0.0 and 80.0 µmol of total ascorbic acid/L of solution. The total ascorbic acid in each ampoule should be measured in duplicate. Please report your results in µmol/(L of the sample solution) rather than µmol/(L of serum NIST used to prepare the sample).

Participant #: _____ Date: _____

Vitamin C Round Robin 34

NIST Micronutrient Measurement Quality Assurance Program

Preparation and Validation of Ascorbic Acid Solid Control Material

STOCK SOLUTION

Mass of ascorbic acid in the Stock Solution ..... _____ g

Mass of 5% MPA Diluent added to the 100 mL volumetric flask..... _____ g

DILUTE SOLUTION 1

Mass of added stock solution (0.5 mL)..... _____ g

Mass of 5% MPA Diluent added to the 100 mL volumetric flask..... _____ g

Absorbance of Dilute Solution 1 at 242 nm..... _____ AU

Absorbance of Dilute Solution 1 at 243 nm..... _____ AU

Absorbance of Dilute Solution 1 at 244 nm..... _____ AU

Absorbance of Dilute Solution 1 at 245 nm..... _____ AU

Absorbance of Dilute Solution absorbance maximum ..... _____ AU

Wavelength of maximum absorbance..... _____ nm

Calculated $E^{1\%}$ ..... _____ dL/g·cm

Calculated $[TAA]_{DS1}$ ..... _____ µmol/L

DILUTE SOLUTION 2

Mass of added stock solution (0.25 mL)..... _____ g

Mass of 5% MPA Diluent added to the 100 mL volumetric flask..... _____ g

Calculated $[TAA]_{DS2}$ ..... _____ µmol/L

DILUTE SOLUTION 3

Mass of added stock solution (0.125 mL)..... _____ g

Mass of 5% MPA Diluent added to the 100 mL volumetric flask..... _____ g

Calculated $[TAA]_{DS3}$ ..... _____ µmol/L

Please return by **March 7, 2011**

MMQAP
100 Bureau Drive, Stop 6392
Gaithersburg, MD 20899-6392

Fax: 301-977-0685
Email: david.duewer@nist.gov

Participant #: _____ Date: _____

Vitamin C Round Robin 34

NIST Micronutrient Measurement Quality Assurance Program

Analysis of Control Materials and Test Samples

Sample	Replicate 1	Replicate 2	Units
Dilute Solution 1			µmol/L of Dilute Solution
Dilute Solution 2			µmol/L of Dilute Solution
Dilute Solution 3			µmol/L of Dilute Solution
5% MPA Diluent			µmol/L of Diluent
CS #2			µmol/L of Sample *Target:* 27.2 ±3.3 µmol/L
Serum Test Sample #341			µmol/L of Sample
Serum Test Sample #342			µmol/L of Sample
Serum Test Sample #343			µmol/L of Sample
Serum Test Sample #344			µmol/L of Sample

Were samples frozen upon receipt? Yes | No

Analysis method: HPLC-EC | HPLC-Fluor DAB | HPLC-OPD | HPLC-UV | AO-OPD | Other
If "Other", please describe:

COMMENTS:

Please return by **March 7, 2011**

MMQAP
100 Bureau Drive, Stop 6392
Gaithersburg, MD 20899-6392

Fax: 301-977-0685
Email: david.duewer@nist.gov

Participant #: _____ Date: _____

Vitamin C Round Robin 34

NIST Micronutrients Measurement Quality Assurance Program

Packing List and Shipment Receipt Confirmation Form

This box contains one vial each of the following **six** VitC M^2QAP samples:

Label	Form
VitC #341	Liquid frozen (1:1 serum:10% MPA)
VitC #342	Liquid frozen (1:1 serum:10% MPA)
VitC #343	Liquid frozen (1:1 serum:10% MPA)
VitC #344	Liquid frozen (1:1 serum:10% MPA)
CS #2	Liquid frozen (1:1 serum:10% MPA)
Control	Solid AA

Please
1) Open the pack immediately
2) Check that it contains one vial each of the above samples
3) Check if the samples arrived frozen
4) Store the samples at -20 ˚C or below until analysis
5) Complete the following information
6) Fax the completed form to us at 301-977-0685
(or email requested information to david.duewer@nist.gov)

1) Date this shipment arrived: _____

2) Are all of the vials intact? Yes | No
If "No", which one(s) were damaged?

3) Was there any dry-ice left in cooler? Yes | No

4) Did the samples arrive frozen? Yes | No

5) At what temperature are you storing the samples? _____ ˚C

6) When do you anticipate analyzing these samples? _____

Your prompt return of this information is appreciated.

The M^2QAP Gang

Mail: M^2QAP
NIST, Stop 6392
Gaithersburg, MD 20899-6392

Fax: 301-977-0685
Email: David.Duewer@NIST.gov

Appendix F. Final Report for RR34

The following two pages are the final report as provided to all participants:

- Cover letter.
- An information sheet that:
  - describes the contents of the “All-Lab” report,
  - describes the content of the “Individualized” report,
  - describes the nature of the test samples and details their previous distributions, if any, and
  - summarizes aspects of the study that we believe may be of interest to the participants.

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

April 7, 2011

Dear Colleague:

Enclosed is the summary report for Round Robin 34 (RR34) for the measurement of total ascorbic acid (TAA, ascorbic acid plus dehydroascorbic acid) in human serum. Included in this report are a summary of data for all laboratories and an individualized summary of your laboratory's measurement performance. The robust median is used to estimate the consensus value for all samples, the "median absolute deviation from the median" (MADe) is used to estimate the expected standard deviation, and the coefficient of variation (CV) is defined as 100×MADe/median.

RR34 consisted of four *test samples* (#341, #342, #343, and #344), one *serum control material* (CS#2), and one *solid control material* for preparation of TAA control solutions. Details regarding the samples can be found in the enclosed report.

If you have concerns regarding your laboratory's performance, we suggest that you obtain and analyze a unit of Standard Reference Material (SRM) 970, Vitamin C in Frozen Human Serum. SRM 970 can be purchased from the NIST SRM Program at phone: 301-975-6776; fax: 301-948-3730. If your measured values do not agree with the certified values, we suggest that you contact us for consultation.

As you may be aware, the MMQAP workshop is scheduled to be held in conjunction with the Experimental Biology conference on Wednesday, April 13 at the Washington Convention Center in Washington, DC. There is no registration fee for the workshop. We hope to see you there.

We hope to ship samples for the second vitamin C round robin (RR35) of the 2011 Micronutrients Measurement Quality Assurance Program (M^2QAP) around the middle of May. If you have questions or concerns regarding this report, please contact David Duewer at 301-975-3935; e-mail: david.duewer@nist.gov or me at 301-975-3120; e-mail: jbthomas@nist.gov; or fax: 301-977-0685.

Sincerely,

Jeanice Brown Thomas
Research Chemist
Analytical Chemistry Division
Chemical Science and Technology Laboratory
Laboratory

David L. Duewer
Research Chemometrician
Analytical Chemistry Division
Chemical Science and Technology

Enclosures

Cc: L. C. Sander

The NIST M^2QAP Vitamin C Round Robin 34 (RR34) report consists of

Page	"Individualized" Report
1	Summarizes your reported values for the nominal 55 mmol/L solution you prepared from the ascorbic acid solid control sample, the serum control sample, and the four serum test samples.
2	Graphical summary of your RR34 sample measurements.

Page	"All Lab" Report
1	A tabulation of results and summary statistics for Total Ascorbic Acid [TAA] in the RR34 samples and control/calibration solutions.

Serum-based Samples. One serum control and four unknowns were distributed in RR34.

CS#2 SRM 970 level 2, ampouled in mid-1998.

S34:1 SRM 970 level 1, ampouled in mid-1998, previously distributed many times.

S34:2 Serum 333, ampouled in late 2009, previously distributed as sample S33:3 (RR33, Fall 09

S34:3 Serum 324, ampouled in late 2009, previously distributed as sample S32:4 (RR32, Spring 09

S34:4 New material, ampouled in late 2009

Results.

1) Most participants who prepared the four 5% MPA control/calibration solutions (the three "Dilute Solutions" and the "Diluent") did so correctly. The criteria used to evaluate this success are: the density of the 5% MPA (≈1.03 gm/mL), the observed wavelength maximum of "Dilute Solution #1"(≈244 nm), the observed absorbance at that maximum (≈0.58 OD), the calculated $E^{1\%}$ #1"(≈560 dL/g·cm).

2) The Measured = a+b*Gravimetric calibration parameters for the control/calibration solutions (columns 10 to 13 of the All Lab Report) indicate that the measurement systems for all participants are linear (R^2 close to 1 and RMS close to 0.0) and well calibrated (intercepts range from -0.4 to 0.6 and slopes range from 0.94 to 1.14).

3) The Measured = p+q*Median regression parameters for samples S31:1 to S31:4 (columns 23 to 26 of the All Lab Report) confirm the linearity of most measurement systems (R^2 close to 1 and RMS close to 0.0).

4) There is no evidence of sample degradation.

Appendix G. "All-Lab Report" for RR34

The following single page is the "All-Lab Report" as provided to all participants, with two exceptions:

- the participant identifiers (Lab) have been altered.
- the order in which the participant results are listed has been altered.

The data summary in the "All-Lab Report" has been altered to ensure confidentiality of identification codes assigned to laboratories.

Micronutrients Measurement Quality Assurance Program for Total Ascorbic Acid
"Round Robin" 34 - March 2011

		Control / Calibration Samples											MPA	Dilute Solution 1			Samples								
		Grav, µmol/L			Measured, µmol/L				Measured = a + b*Grav				Density	Spectrophotometry			Measured, µmol/L					Measured = p+q*Median			
Lab	Date	Dil:1	Dil:2	Dil:3	Dil:1	Dil:2	Dil:3	MPA	Inter	Slope	R^2	RMS	g/mL	λ_{max}	A_{max}	$E^{1\%}$	CS#2	S34:1	S34:2	S34:3	S34:4	Inter	Slope	R^2	RMS
VC-MA	28/02/11	58.8	30.0	14.9	67.1	35.5	17.7	0.0	0.56	1.14	1.000	0.8	1.037	244.	0.5750	555.2	27.9	8.4	16.0	48.0	58.3	-0.48	1.03	0.998	1.3
VC-MB	14/01/11	57.7	28.6	14.4	57.0	28.4	13.2	0.0	-0.39	0.99	1.000	0.7	1.030	243.5	0.5590	549.8	27.4	7.7	13.9	45.7	57.0	-1.79	1.02	0.999	0.7
VC-MC	24/03/11	59.7	30.1	14.8	57.3	29.4	14.5	0.0	0.23	0.96	1.000	0.3	1.031	243.	0.5772	549.1	27.6	10.1	17.9	50.1	62.0	0.74	1.07	0.999	0.7
VC-ME	01/03/11	58.0	29.0	14.1	61.1	30.6	15.2	0.0	0.11	1.05	1.000	0.2	1.033	243.	0.5844	572.4	28.0	9.4	16.6	45.2	54.6	1.51	0.93	0.998	1.2
VC-MG	20/01/11	58.4	30.8	16.2	61.1	32.7	18.0	0.0	0.54	1.04	1.000	0.6	1.029	243.9	0.5850	568.4	28.3	9.0	15.6	48.4	59.5	-0.61	1.05	0.999	0.9
VC-MH	25/01/11	60.6	30.3	15.3	61.3	30.3	15.3	0.0	-0.16	1.01	1.000	0.2	1.029	244.1	0.5907	553.0	27.8	8.9	16.3	47.9	57.8	0.09	1.02	0.998	1.4
VC-MI	24/01/11	59.0	30.4	14.3	59.6	30.6	14.6	0.0	0.01	1.01	1.000	0.1	1.030				28.0	9.1	12.8	45.6	58.9	-1.97	1.05	0.997	1.6
VC-MJ	23/02/11	59.3	29.7	14.8	57.1	29.1	15.0	0.1	0.48	0.96	1.000	0.4	0.999	254a	0.365a	349.2a	34.5	10.5	22.3	53.3	63.9	3.01	1.07	0.992	2.7
VC-MN	02/02/11	59.5	29.9	14.9	59.2	29.4	14.6	0.0	-0.17	1.00	1.000	0.2	1.026	243.6	0.5881	561.4	26.1	10.2	14.9	40.8	48.4	2.75	0.80	0.997	1.2
VC-MP	28/01/11	60.2	30.2	14.7	56.7	28.9	14.0	0.0	0.18	0.94	1.000	0.3	1.032	242.	0.5598	527.7	28.5	9.4	14.8	41.7	58.0	-0.36	0.98	0.993	2.3
VC-NF	24/02/11	56.8	28.5	14.1	56.8	28.6	14.2	0.0	0.04	1.00	1.000	0.0	1.032	243.	0.5330	533.1	28.5	9.3	15.7	43.5	53.6	1.21	0.91	0.999	0.6
VC-NM	03/03/11	54.8	27.6	13.7	54.9	27.2	13.9	0.0	-0.05	1.00	1.000	0.3	1.034	242.	0.5440	563.8	26.9	7.5	13.2	41.4	52.6	-1.22	0.93	1.000	0.2
	N	12	12	12	12	12	12	12					N	12	10	10	10	12	12	12	12	12			
	Average	58.6	29.6	14.7	59.1	30.0	15.0	0.0					Average	1.028	243.2	0.5696	553.4	28.3	9.1	15.8	46.0	57.0			
	SD	1.6	1.0	0.7	3.3	2.2	1.5	0.0					SD	0.010	0.8	0.0198	14.4	2.1	0.9	2.5	3.8	4.2			
	Min	54.8	27.61	13.7	54.9	27.15	13.2	0.0					Min	0.999	242.0	0.5330	527.7	26.1	7.5	12.8	40.8	48.4			
	%25	57.9	28.89	14.3	57.0	28.83	14.1	0.0					%25	1.029	243.0	0.5592	549.3	27.5	8.7	14.6	43.1	54.4			
	Median	58.9	29.95	14.7	58.2	29.40	14.6	0.0					Median	1.031	243.3	0.5761	554.1	28.0	9.2	15.7	45.6	57.9			
	%75	59.5	30.27	14.9	61.1	30.56	15.2	0.0					%75	1.032	243.8	0.5849	563.2	28.3	9.6	16.3	48.1	59.1			
	Max	60.6	30.76	16.2	67.1	35.48	18.0	0.1					Max	1.037	244.1	0.5907	572.4	34.5	10.5	22.3	53.3	63.9			
	MADe	1.3	0.6	0.6	2.2	1.4	0.8	0.0					eSD	0.0	0.7	0.0197	12.5	0.6	0.8	1.4	3.8	3.7			
	CV	2	2	4	4	5	6						CV	0.26	0.30	3.4	2.3	2	9	9	8	6			

a) 5% Trichloroacetic acid solution

G2

Appendix H. Representative "Individualized Report" for RR34

Each participant in RR34 received an "Individualized Report" reflecting their reported results. The following two pages are the "Individualized Report" for participant "VC-MA".

Vitamin C "Round Robin" 34 Report: Participant VC-MA

Date	RR	Method	MPA Density g/mL	Dilute Solution 1 Spectrophotometry λ_{max}	A_{max}	$E^{1\%}$	Control/Calibration Solutions Y_{meas} = Inter + Slope* X_{grav} Inter	Slope	R^2	SEE
08/11/08	29	HPLC-EC	1.037	243.0	0.567	553.2	0.3	1.03	1.000	0.64
03/03/09	30	HPLC-EC	1.037	242.0	0.569	555.6	0.2	1.03	1.000	0.40
09/10/09	31	HPLC-EC	1.036	244.0	0.566	546.1	-0.1	1.02	1.000	0.20
02/24/10	32	HPLC-EC	1.035	242.0	0.566	545.1	0.3	1.03	1.000	0.46
09/27/10	33	HPLC-EC	1.037	244.0	0.560	540.5	0.4	1.08	1.000	0.43
02/28/11	34	HPLC-EC	1.039	244.0	0.575	555.2	0.6	1.14	1.000	0.78
		Mean	1.037	243.2	0.57	549.3			Pooled SEE	0.52
		SD	0.001	1.0	0.00	6.2				
		CV	0.14	0.40	0.9	1.1				

Date	RR	Sample	[TAA] mmol/Lsample Rep_1	Rep_2	F_{adj}	Mean	SD_{dup}	N	Mean	SD_{repeat}	SD_{reprod}
09/23/98	11	S11:1:A	15.5	13.9	0.5	7.4	0.6	13	8.2	0.3	0.5
04/02/99	12	S12:1:A	14.5	15.8	0.5	7.6	0.5				
09/17/01	13	S13:1	8.4	8.5	1.0	8.5	0.1				
09/27/01	14	S14:3	8.0	7.7	1.0	7.8	0.2				
09/18/01	15	S15:1	8.9	8.7	1.0	8.8	0.1				
11/18/02	16	S16:1	8.8	8.8	1.0	8.8	0.0				
11/13/03	19	S19:4	7.8	8.6	1.0	8.2	0.5				
02/23/04	20	S20:3	8.3	8.1	1.0	8.2	0.1				
10/17/05	23	S23:4	8.6	8.8	1.0	8.7	0.1				
08/28/06	25	S25:1	8.7	8.5	1.0	8.6	0.2				
08/11/08	29	S29:2	8.3	8.4	1.0	8.3	0.1				
09/10/09	31	S31:3	7.3	8.1	1.0	7.7	0.5				
02/28/11	34	S34:1	**8.5**	**8.3**	1.0	8.4	0.1				
09/27/10	33	S33:3	16.6	16.8	1.0	16.7	0.1	2	16.4	0.3	0.6
02/28/11	34	S34:2	**16.3**	**15.7**	1.0	16.0	0.4				
02/24/10	32	S32:4	49.0	48.6	1.0	48.8	0.3	2	48.4	0.2	0.6
02/28/11	34	S34:3	**47.9**	**48.1**	1.0	48.0	0.1				
02/28/11	34	S34:4	**58.1**	**58.4**	1.0	58.3	0.2	1	58.3	0.2	

Please check our records against your records. Send corrections and/or updates to...

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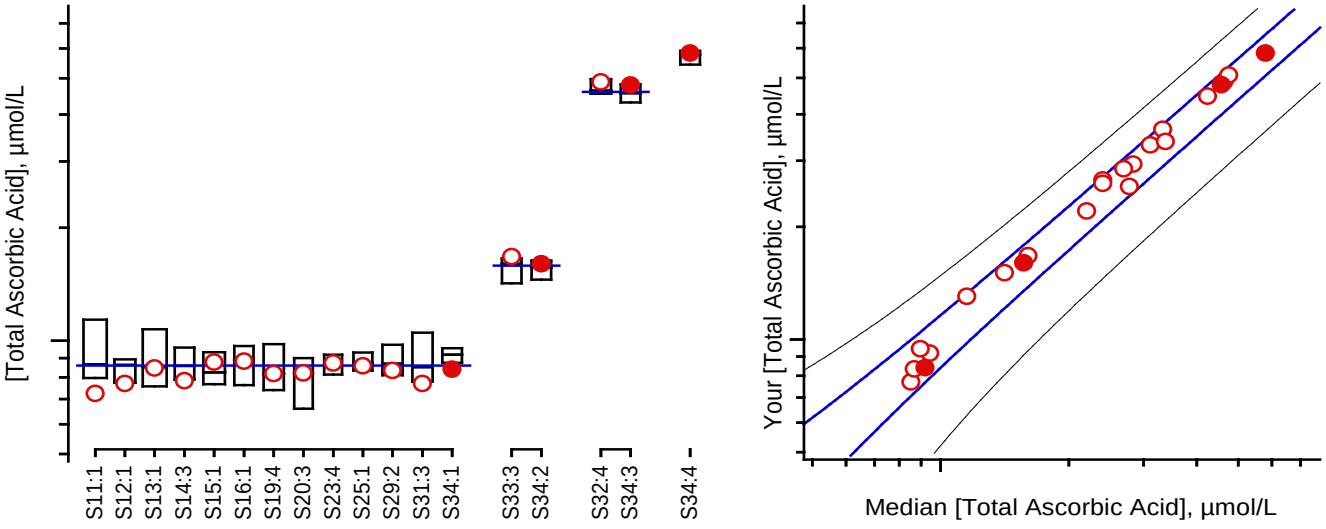
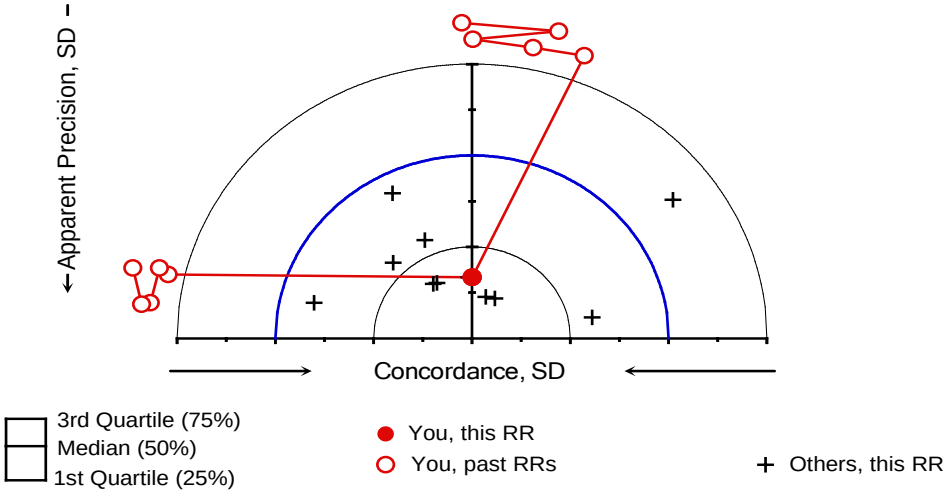
Vitamin C "Round Robin" 34 Report: Participant VC-MA

Total Ascorbic Acid, µmol/mL





For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

Sample	Comments
S33:1	VitC #341, previously distributed in RRs 11, 12, 13, 14, 15, 16, 19, 20, 23, 25, 29, 31
S33:2	VitC #342, previously distributed in RR 33
S33:3	VitC #343, previously distributed in RR 32
S33:4	VitC #344, new material