NISTIR 7880-2

NIST Micronutrients Measurement Quality Assurance Program Summer 2011 Comparability Studies

Results for Round Robin LXX
Fat-Soluble Vitamins and Carotenoids in Human Serum
and Round Robin 35 Ascorbic Acid in Human Serum

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

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April, 2013

U.S. Department of Commerce
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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 Summer 2011 MMQAP measurement comparability improvement studies: 1) Round Robin LXX Fat-Soluble Vitamins and Carotenoids in Human Serum and 2) Round Robin 35 Total Ascorbic Acid in Human Serum. The materials for both studies were shipped to participants in May 2011; participants were requested to provide their measurement results by September 19, 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 LXX: Fat-Soluble Vitamins and Carotenoids in Human Serum

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin LXX comparability study (hereafter referred to as RR70) received one lyophilized and four 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 May 2011. 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 RR70 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 "Individualized Report" is reproduced as Appendix D.

Round Robin 35: Vitamin C in Human Serum

Participants in the MMQAP Vitamin C in Human Serum Round Robin 35 comparability study (hereafter referred to as RR35) 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 May 2011. 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 RR35 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 RR70

The following three items were included in each package shipped to an RR70 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-

May 23, 2011

Dear Colleague:

Enclosed are samples for the second fat-soluble vitamins and carotenoids in serum study (Round Robin LXX) for the 2011 NIST Micronutrients Measurement Quality Assurance Program. The set of samples (Sera 377- 381) consists of one vial each of four liquid-frozen serum samples and one lyophilized sample 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 **September 19, 2011.** Results received more than <u>two weeks</u> 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 October 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 LXX 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 B Thomas

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

NIST

Participant #: ________ Date: ____________

Round Robin LXX: Human Sera

NIST Micronutrients Measurement Quality Assurance Program

Analyte	377	378	379	380	381	Units*
total retinol						
trans-retinol						
retinyl palmitate						
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						
Phytoene						
Phytofluene						

* we prefer **μg/mL**

Were the liquid-frozen samples (378 to 381) frozen when received? Yes | No

Comments:

Mail: M^2QAP
NIST, Stop 8392
Gaithersburg, MD 20899-8392

Please return results by
19-Sep-2011

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

Participant #: ________ Date: ____________

Fat-Soluble Vitamins Round Robin LXX

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^{2}QAP sera

Serum	Form	Reconstitute?	Vial/Cap
#377	Lyophilized	Yes	Amber/Red
#378	Liquid frozen	No	Amber/Blue
#379	Liquid frozen	No	Amber/Green
#380	Liquid frozen	No	Amber/Green
#381	Liquid frozen	No	Amber/Red

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 (#378 to #381) 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^{2}QAP Gang

Mail: M^{2}QAP
NIST, Stop 8392
Gaithersburg, MD 20899-8392

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

Appendix B. Final Report for RR70

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-

November 21, 2011

Dear Colleague:

Enclosed is the summary report of the results for round robin LXX (RR70) 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.

Samples for the first fat-soluble vitamins and carotenoids in serum interlaboratory exercise (RR71) of the 2012 M^2QAP will be shipped **starting January 9, 2011**. Please contact us immediately if this schedule is problematic for your laboratory.

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 B Thomas

Jeanice Brown Thomas, M.B.A.
Research Chemist
Analytical Chemistry Division
Material Measurement Laboratory

David Duewer

David L. Duewer, Ph.D.
Research Chemometrician
Analytical Chemistry Division
Material Measurement Laboratory

Enclosures

Cc: L.C. Sander

NIST

The NIST M^2QAP Round Robin LXX (RR70) report consists of:

Page	"All Lab" Report
1-4	A listing of all results and statistics for analytes reported by more than one participant.
5	A listing of the analytes reported by only one participant and a legend for the list of results and statistics.
6	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 RR70.

Serum	Description	Prior Distributions
377	Lyophilized, native serum prepared in 1999. Serum #380 is the liquid-frozen partner of this sample.	#270:RR49-3/01, #276:RR50-9/01, #367:RR68-9/10
378	Fresh-frozen, native, multi-donor serum prepared in 2005. This serum was prepared as a blend of the #381 pool and a second pool with generally higher analyte levels.	#315:RR58-9/05, #335:RR62-9/07
379	Fresh-frozen, native, multi-donor, prepared in 2009. This is Level III of SRM 968e.	#359:RR66-9/09, #363:RR67-3/10, #373:RR69-3/11
380	Liquid-frozen, native serum prepared in 1999. Serum #377 is the lyophilized partner of this sample.	#267:RR49-9/00, #274:RR50-9/01, #368:RR68-9/10
381	Fresh-frozen, native, multi-donor serum prepared in 2005. This serum was expected to have low levels of most analytes.	#316:RR58-9/05, #336:RR62-9/07

Results

1) Sera Stability. There was no significant change in the median level or measurement variability of any measurand in any of the materials.

2) SRM 968e. Sera #379 is the "high" retinol, α-tocopherol and β-carotene component of SRM 968e. It was prepared without spiking.

Appendix C. "All-Lab Report" for RR70

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 LXX Laboratory Results

	Total Retinol, µg/mL					trans-Retinol, µg/mL					Retinyl Palmitate, µg/mL					α-Tocopherol, µg/mL					γ/β-Tocopherol, µg/mL				
Lab	377	378	379	380	381	377	378	379	380	381	377	378	379	380	381	377	378	379	380	381	377	378	379	380	381
FSV-BA	0.692	0.532	0.713	0.698	0.430						0.018	0.053	0.181	0.021	0.012	5.93	9.28	18.1	6.17	4.13	2.37	1.86	2.44	2.44	1.81
FSV-BB	0.641	0.486	0.683	0.688	0.397						0.023	0.039	0.064	0.023	0.008	5.45	8.57	16.8	5.84	3.71	2.08	1.64	2.16	2.19	1.62
FSV-BC	0.652	0.480	0.676	0.669	0.400																				
FSV-BD	0.635	0.468	0.649	0.681	0.378											6.30	9.00	18.9	6.60	5.00					
FSV-BE	0.635	0.530	0.694	0.706	0.393											4.62	8.05	16.6	5.18	3.36	1.87	1.64	2.17	2.11	1.54
FSV-BF	0.600	0.480	0.670	0.640	0.350											5.40	8.30	16.5	5.60	3.90					
FSV-BG	0.729	0.501	0.674	0.721	0.395						0.017	0.041	0.115	0.017	0.012	6.95	9.54	19.2	6.87	4.27	2.67	1.80	2.32	2.62	1.74
FSV-BH	0.642	0.466	0.646	0.689	0.373											6.24	9.93	19.9	6.63	4.42	2.27	1.72	2.26	2.34	1.76
FSV-BJ	0.613	0.449	0.608	0.630	0.351											6.16	10.07	19.6	6.47	4.01	2.17	1.71	2.15	2.23	1.56
FSV-BK	0.648	0.499	0.656	0.681	0.403											6.00	9.41	18.8	6.50	4.23					
FSV-BL	0.660	0.520	0.690	0.660	0.370											6.46	10.34	19.8	6.46	4.31					
FSV-BM	0.660	0.500	0.640	0.660	0.396											6.20	9.80	18.7	6.90	5.50					
FSV-BN	0.649	0.417	0.626	0.598	0.333											5.67	7.31	16.8	5.28	3.43					
FSV-BO	0.520	0.420	0.540	0.580	0.330											5.10	8.40	15.7	5.50	3.70	1.90	1.60	1.90	2.10	1.50
FSV-BP	0.618	0.451	0.631	0.652	0.371											6.36	10.34	18.8	6.63	4.75					
FSV-BQ	0.620	0.400	0.580	0.530	0.310											5.00	9.00	17.0	5.00	4.00					
FSV-BR	0.630	0.490	0.650	0.650	0.340											6.50	8.90	19.0	6.20	4.30					
FSV-BS	≥0.605	≥0.474	≥0.719	≥0.648	≥0.387	0.605	0.474	0.719	0.648	0.387															
FSV-BT	0.559	0.461	0.596	0.616	0.356											5.67	9.14	18.3	6.28	4.11	2.00	1.62	2.26	2.13	1.51
FSV-BU	0.607	0.488	0.581	0.606	0.355											6.05	9.96	19.0	6.20	4.19	2.14	1.80	2.15	2.24	1.64
FSV-BV	0.636	0.518	0.657	0.701	0.390											6.65	10.75	19.9	6.99	4.38	2.36	1.92	2.37	2.54	1.78
FSV-BW	0.595	0.444	0.589	0.626	0.354						0.017	0.032	0.062	0.016	0.011	5.42	8.27	16.6	5.71	3.59	2.12	1.58	2.04	2.29	1.60
FSV-CC	0.647	0.488	0.668	0.664	0.377	0.639	0.483	0.663	0.654	0.371						6.26	9.83	20.0	6.39	4.30					
FSV-CD	0.577	0.459	0.584	0.677	0.339						0.021	0.074	0.107	0.020	0.019	6.93	10.64	19.0	7.50	5.23	3.50	1.75	2.12	2.29	1.64
FSV-CE	0.770	0.480	0.660	0.700	0.430											5.10	10.00	20.4	6.65	5.52					
FSV-CG	0.672	0.501	0.673	0.673	0.393											5.33	9.48	17.8	6.19	4.18	2.13	1.86	2.31	2.41	1.79
FSV-CI	0.704	0.541	0.741	0.747	0.405						0.012	0.050	0.108	0.014	0.001	5.71	8.93	18.2	6.18	4.02	2.36	1.69	2.39	2.42	1.68
FSV-CW	0.866	0.499	0.573	0.705	0.363						0.016	0.053	0.094	0.019	0.004	4.42	8.64	17.3	5.54	4.00	1.80	1.60	2.09	2.10	1.58
FSV-CZ	0.641	0.542	0.637	0.657	0.395											6.35	9.29	18.6	6.08	4.12	2.06	1.63	2.03	2.10	1.59
FSV-DD	0.620	0.460	0.640	0.600	0.360																				
FSV-DQ																5.70	10.36	22.5	7.02	4.47	2.47	2.07	3.15	2.89	2.03
FSV-DV	0.677	0.512	0.683	0.686	0.405											5.80	8.40	16.2	5.80	3.90					
FSV-EE																									
FSV-FK	0.597	0.452	0.609	0.609	0.369											6.00	9.10	17.5	6.40	4.30					
N	31	31	31	31	31	2	2	2	2	2	7	7	7	7	7	30	30	30	30	30	17	17	17	17	17
Min	0.520	0.400	0.540	0.530	0.310	0.605	0.474	0.663	0.648	0.371	0.012	0.032	0.062	0.014	0.001	4.42	7.31	15.7	5.00	3.36	1.80	1.58	1.90	2.10	1.50
Median	0.641	0.486	0.649	0.664	0.373	0.622	0.479	0.691	0.651	0.379	0.017	0.050	0.107	0.019	0.011	5.97	9.29	18.7	6.24	4.19	2.14	1.71	2.17	2.29	1.64
Max	0.866	0.542	0.741	0.747	0.430	0.639	0.483	0.719	0.654	0.387	0.023	0.074	0.181	0.023	0.019	6.95	10.75	22.5	7.50	5.52	3.50	2.07	3.15	2.89	2.03
SD	0.034	0.038	0.040	0.050	0.033						0.002	0.013	0.019	0.004	0.004	0.58	0.98	1.7	0.59	0.28	0.32	0.13	0.19	0.23	0.14
CV	5	8	6	8	9						11	26	18	19	36	10	11	9	9	7	15	8	9	10	9
N_{past}	36	33	32	39	34	11	6	7	11	6	10	11	10	10	8	40	35	33	42	35	22	22	19	24	22
$Median_{past}$	0.635	0.498	0.655	0.666	0.390	0.617	0.490	0.618	0.657	0.385	0.023	0.050	0.089	0.026	0.012	5.97	9.43	18.7	6.39	4.19	2.23	1.80	2.24	2.34	1.74
SD_{past}	0.052	0.045	0.060	0.060	0.029	0.040	0.031	0.087	0.048	0.037	0.010	0.009	0.029	0.012	0.004	0.49	0.65	1.4	0.63	0.31	0.17	0.13	0.21	0.21	0.10
NAV	0.641	0.486	0.649	0.664	0.373						0.017	0.050	0.107	0.019	0.011	5.97	9.29	18.7	6.24	4.19	2.14	1.71	2.17	2.29	1.64
NAU	0.051	0.039	0.051	0.053	0.033						0.011	0.016	0.028	0.011	0.010	0.58	0.98	1.7	0.59	0.44	0.32	0.18	0.22	0.23	0.18

Round Robin LXX 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	377	378	379	380	381	377	378	379	380	381	377	378	379	380	381	377	378	379	380	381	377	378	379	380	381
FSV-BA	0.101	0.107	0.208	0.079	0.105	0.501	0.291	0.419	0.518	0.038	0.473	0.271	0.398	0.496	0.036	0.028	0.020	0.021	0.021	0.003	0.020	0.056	0.015	0.019	0.004
FSV-BB	0.079	0.108	0.191	0.079	0.098	0.454	0.329	0.340	0.482	0.040	0.409	0.256	0.317	0.444	0.035	0.029	0.018	0.015	0.021	0.001	0.016	0.055	0.009	0.017	0.004
FSV-BC																									
FSV-BD																									
FSV-BE						0.462	0.272	0.405	0.484	0.036															
FSV-BF						0.440	0.240	0.380	0.480	0.030															
FSV-BG						0.507	0.262	0.460	0.587	0.041											0.016	0.045	0.015	0.017	0.007
FSV-BH						0.453	0.258	0.389	0.483	0.031	0.444	0.250	0.380	0.473	0.031	0.009	0.008	0.008	0.010	*nq*	0.012	0.042	nq	0.013	*nq*
FSV-BJ						0.481	0.248	0.392	0.526	0.030											*nq*	0.078	*nq*	*nq*	*nq*
FSV-BK																									
FSV-BL																									
FSV-BM																									
FSV-BN						0.429	0.199	0.343	0.395	0.021											0.030	0.047	0.019	0.025	*nd*
FSV-BO						0.397	0.296	0.368	0.473	0.051											0.016	0.056	0.006	0.016	0.005
FSV-BP						0.503	0.242	0.377	0.528	0.074											0.024	0.036	0.019	0.026	0.041
FSV-BQ																									
FSV-BR																									
FSV-BS						≥0.408	≥0.212	≥0.376	≥0.398	≥0.025	0.408	0.212	0.376	0.398	0.025						0.006	0.023	*nd*	0.005	*nd*
FSV-BT	0.025	0.021	0.029	0.021	0.019	0.502	0.282	0.390	0.510	0.050	0.445	0.246	0.337	0.455	0.042	0.021	0.014	0.023	0.019	0.003	0.015	0.038	0.020	0.016	0.007
FSV-BU						0.569	0.286	0.345	0.556	*nq*											0.026	0.059	0.025	0.024	*nq*
FSV-BV						0.492	0.279	0.400	0.564	0.039											0.016	0.057	0.010	0.018	0.006
FSV-BW						0.290	0.232	0.325	0.473	0.034											0.005	0.026	0.002	0.009	0.002
FSV-CC																									
FSV-CD						0.748	0.474	0.443	0.808	0.083											0.036	0.097	0.030	0.049	0.024
FSV-CE						0.350	0.280	0.450	0.390	0.090															
FSV-CG	0.129	0.179	0.280	0.129	0.134	0.389	0.297	0.384	0.484	0.042	0.373	0.280	0.368	0.467	0.039	0.017	0.017	0.016	0.017	*nq*	0.021	0.075	0.024	0.027	0.008
FSV-CI						0.464	0.238	0.435	0.502	0.029											0.027	0.049	0.042	0.028	0.006
FSV-CW	0.063	0.090	0.196	0.086	0.078	0.392	0.257	0.333	0.460	0.036											0.016	0.061	0.012	0.021	0.006
FSV-CZ						0.421	0.266	0.347	0.433	0.069															
FSV-DD																									
FSV-DQ						0.551	0.182	0.249	0.468	0.045											0.020	0.039	0.012	0.020	0.012
FSV-DV																									
FSV-EE																									
FSV-FK																									
N	5	5	5	5	5	21	21	21	21	20	6	6	6	6	6	5	5	5	5	3	17	18	15	17	13
Min	0.025	0.021	0.029	0.021	0.019	0.290	0.182	0.249	0.390	0.021	0.373	0.212	0.317	0.398	0.025	0.009	0.008	0.008	0.010	0.001	0.005	0.023	0.002	0.005	0.002
Median	0.079	0.107	0.196	0.079	0.098	0.462	0.266	0.384	0.484	0.040	0.427	0.253	0.372	0.461	0.035	0.021	0.017	0.016	0.019	0.003	0.016	0.052	0.015	0.019	0.006
Max	0.129	0.179	0.280	0.129	0.134	0.748	0.474	0.460	0.808	0.090	0.473	0.280	0.398	0.496	0.042	0.029	0.020	0.023	0.021	0.003	0.036	0.097	0.042	0.049	0.041
SD	0.032	0.026	0.018	0.012	0.029	0.061	0.036	0.055	0.039	0.014	0.027	0.018	0.025	0.022	0.006	0.010	0.004	0.007	0.003		0.006	0.014	0.008	0.007	0.003
CV	41	24	9	15	30	13	13	14	8	35	6	7	7	5	18	47	26	45	16		39	27	50	36	43
N_{past}	6	6	5	6	6	28	22	22	28	21	11	12	8	12	12	8	7	6	9	6	23	22	16	24	17
$Median_{past}$	0.108	0.114	0.215	0.119	0.100	0.492	0.286	0.399	0.521	0.040	0.439	0.272	0.340	0.475	0.038	0.026	0.019	0.018	0.026	0.003	0.020	0.056	0.017	0.022	0.008
SD_{past}	0.042	0.013	0.034	0.117	0.012	0.064	0.029	0.072	0.074	0.008	0.040	0.023	0.069	0.042	0.006	0.006	0.004	0.004	0.008	0.002	0.005	0.011	0.008	0.005	0.003
NAV	0.079	0.107	0.196	0.079	0.098	0.462	0.266	0.384	0.484	0.040	0.427	0.253	0.372	0.461	0.035	0.021	0.017	0.016	0.019	0.003	0.016	0.052	0.015	0.019	0.006
NAU	0.032	0.026	0.036	0.021	0.029	0.065	0.039	0.055	0.067	0.014	0.045	0.028	0.040	0.049	0.008	0.010	0.006	0.007	0.007		0.006	0.016	0.008	0.007	0.003

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Round Robin LXX Laboratory Results

Lab	Total Lycopene, µg/mL 377	378	379	380	381	trans-Lycopene, µg/mL 377	378	379	380	381
FSV-BA	0.221	0.225	0.95	0.231	0.136	0.136	0.128	0.446	0.147	0.072
FSV-BB	0.199	0.209	0.73	0.202	0.113	0.100	0.097	0.314	0.111	0.052
FSV-BC										
FSV-BD										
FSV-BE										
FSV-BF										
FSV-BG	0.205	0.211	0.96	0.229	0.144	0.128	0.116	0.500	0.160	0.083
FSV-BH	0.229	0.248	1.03	0.241	0.144					
FSV-BJ	0.143	0.168	0.81	0.156	0.076					
FSV-BK										
FSV-BL										
FSV-BM										
FSV-BN	0.224	0.199	0.91	0.206	0.113					
FSV-BO	0.180	0.188	0.61	0.185	0.104					
FSV-BP	0.254	0.233	1.00	0.278	0.253					
FSV-BQ										
FSV-BR										
FSV-BS	0.219	0.216	0.90	0.214	0.158	0.150	0.143	0.604	0.149	0.099
FSV-BT	0.209	0.216	0.83	0.212	0.151	0.142	0.135	0.388	0.148	0.082
FSV-BU	0.221	0.231	0.75	0.256	*nq*					
FSV-BV	0.218	0.245	0.98	0.258	0.150					
FSV-BW	0.100	0.200	0.86	0.220	0.100					
FSV-CC										
FSV-CD	0.332	0.369	1.05	0.349	0.214					
FSV-CE										
FSV-CG	0.195	0.272	0.97	0.244	0.161	0.124	0.154	0.478	0.151	0.088
FSV-CI										
FSV-CW						0.108	0.117	0.354	0.126	0.067
FSV-CZ										
FSV-DD										
FSV-DQ	0.350	0.188	0.68	0.302	0.217					
FSV-DV										
FSV-EE										
FSV-FK										
N	16	16	16	16	15	7	7	7	7	7
Min	0.100	0.168	0.61	0.156	0.076	0.100	0.097	0.314	0.111	0.052
Median	0.219	0.216	0.91	0.230	0.144	0.128	0.128	0.446	0.148	0.082
Max	0.350	0.369	1.05	0.349	0.253	0.150	0.154	0.604	0.160	0.099
SD	0.025	0.025	0.12	0.037	0.045	0.021	0.018	0.086	0.004	0.015
CV	11	12	14	16	32	17	14	19	3	19
N_{past}	23	20	17	24	20	10	9	8	10	9
$Median_{past}$	0.226	0.243	0.96	0.234	0.151	0.123	0.119	0.399	0.134	0.069
SD_{past}	0.034	0.037	0.19	0.036	0.029	0.024	0.019	0.092	0.021	0.012
NAV	0.219	0.216	0.91	0.230	0.144	0.128	0.128	0.446	0.148	0.082
NAU	0.053	0.052	0.17	0.055	0.045	0.022	0.022	0.086	0.026	0.015

Lab	Total β-Cryptoxanthin, µg/mL 377	378	379	380	381	Total α-Cryptoxanthin, µg/mL 377	378	379	380	381	Total Lutein, µg/mL 377	378	379	380	381
FSV-BA	0.065	0.056	0.054	0.065	0.043	0.039	0.022	0.033	0.037	0.016					
FSV-BB	0.041	0.039	0.032	0.045	0.029	0.019	0.010	0.013	0.021	0.007	0.099	0.068	0.145	0.102	0.0221
FSV-BC															
FSV-BD															
FSV-BE															
FSV-BF															
FSV-BG	0.055	0.044	0.024	0.053	0.036										
FSV-BH	0.050	0.053	0.037	0.054	0.039						0.083	0.063	0.123	0.085	0.0167
FSV-BJ	0.029	0.033	0.017	0.028	0.017						0.072	0.057	0.111	0.069	*nq*
FSV-BK															
FSV-BL															
FSV-BM															
FSV-BN	0.054	0.042	0.057	0.051	0.028										
FSV-BO	0.035	0.036	0.015	0.038	0.026						0.095	0.076	0.132	0.112	0.0270
FSV-BP	0.046	0.048	0.050	0.054	0.049										
FSV-BQ															
FSV-BR															
FSV-BS	0.033	0.021	0.060	0.029	0.022						0.062	0.035	0.078	0.066	0.0110
FSV-BT	0.037	0.034	0.031	0.042	0.026	0.018	0.014	0.028	0.020	0.020					
FSV-BU	0.044	0.042	0.027	0.045	*nq*										
FSV-BV	0.034	0.034	0.020	0.036	0.025										
FSV-BW															
FSV-CC															
FSV-CD	0.062	0.064	0.054	0.067	0.045	0.029	0.013	0.040	0.031	*nq*					
FSV-CE															
FSV-CG	0.065	0.070	0.063	0.067	0.049										
FSV-CI											0.080	0.054	0.102	0.082	0.0220
FSV-CW	0.040	0.040	0.026	0.049	0.029										
FSV-CZ															
FSV-DD															
FSV-DQ	0.067	0.060	0.044	0.070	0.049						0.086	0.079	0.162	0.095	0.0240
FSV-DV															
FSV-EE															
FSV-FK															
N	16	16	16	16	15	4	4	4	4	3	7	7	7	7	6
Min	0.029	0.021	0.015	0.028	0.017	0.018	0.010	0.013	0.020	0.007	0.062	0.035	0.078	0.066	0.0110
Median	0.045	0.042	0.035	0.050	0.029	0.024	0.014	0.031	0.026	0.016	0.083	0.063	0.123	0.085	0.0221
Max	0.067	0.070	0.063	0.070	0.049	0.039	0.022	0.040	0.037	0.020	0.099	0.079	0.162	0.112	0.0270
SD	0.015	0.012	0.022	0.015	0.010	0.008	0.003	0.009	0.008		0.017	0.014	0.030	0.023	0.0051
CV	33	28	64	29	35	34	23	29	31		20	22	25	28	23
N_{past}	22	21	17	24	21	5	5	4	6	9	13	14	9	13	13
$Median_{past}$	0.048	0.045	0.032	0.051	0.035	0.023	0.020	0.021	0.027	0.009	0.086	0.063	0.121	0.089	0.0242
SD_{past}	0.009	0.007	0.011	0.008	0.004	0.005	0.010	0.008	0.003	0.002	0.021	0.011	0.021	0.013	0.0057
NAV	0.045	0.042	0.035	0.050	0.029	0.024	0.014	0.031	0.026	0.016	0.083	0.063	0.123	0.085	0.0221
NAU	0.015	0.012	0.022	0.015	0.010						0.017	0.014	0.030	0.023	0.0052

Round Robin LXX 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	377	378	379	380	381	377	378	379	380	381	377	378	379	380	381	377	378	379	380	381	377	378	379	380	381
FSV-BA						0.149	0.112	0.182	0.146	0.0540															
FSV-BB	0.042	0.0265	0.064	0.044	0.0186	0.141	0.094	0.209	0.146	0.0407															
FSV-BC																									
FSV-BD																									
FSV-BE											0.840	0.66	1.39	0.87	0.650	2.841	1.241	3.610	6.081	0.242					
FSV-BF																									
FSV-BG						0.133	0.088	0.157	0.125	0.0375															
FSV-BH	0.054	0.0198	0.032	0.051	0.0142	0.138	0.083	0.154	0.136	0.0309											0.010	0.012	0.022	0.011	0.007
FSV-BJ											0.780	0.77	1.50	0.90	0.540										
FSV-BK																									
FSV-BL																									
FSV-BM																									
FSV-BN						0.099	0.052	0.116	0.091	0.0140											0.015	0.016	0.037	0.016	0.012
FSV-BO	0.026	0.0110	0.020	0.028	0.0050	*0.121*	*0.087*	*0.152*	*0.140*	*0.0320*	0.930	1.08	1.54	1.00	0.950										
FSV-BP						0.113	0.076	0.161	0.126	0.0370															
FSV-BQ																									
FSV-BR																									
FSV-BS	0.006	0.0010	0.006	0.006	0.0020	0.068	0.036	0.084	0.072	0.0130															
FSV-BT						0.100	0.077	0.126	0.113	0.0370	0.754	0.63	1.11	0.87	0.680										
FSV-BU						0.111	0.075	0.164	0.099	*nq*	0.910	0.64	1.27	0.92	0.700						0.011	0.015	0.023	0.010	0.006
FSV-BV						0.112	0.074	0.119	0.116	0.0320															
FSV-BW											0.609	0.59	1.31	0.81	0.496										
FSV-CC																									
FSV-CD						0.222	0.176	0.273	0.246	0.1060															
FSV-CE																									
FSV-CG						0.153	0.114	0.196	0.169	0.0530															
FSV-CI	0.018	0.0090	0.013	0.020	0.0060	*0.098*	*0.063*	*0.115*	*0.102*	*0.0280*	0.761	0.57	1.14	0.67	0.618	0.978	0.438	1.212	1.042	0.102					
FSV-CW						0.206	0.110	0.195	0.175	0.0463	0.775	0.68	1.41	0.68	0.626										
FSV-CZ											0.967	0.93	1.86	1.15	0.881										
FSV-DD																									
FSV-DQ	0.026	0.0110	0.020	0.028	0.0085	*0.112*	*0.090*	*0.182*	*0.123*	*0.0325*															
FSV-DV																									
FSV-EE											0.882	0.74	1.52	0.95	0.704										
FSV-FK																									
N	6	6	6	6	6	16	16	16	16	15	10	10	10	10	10	2	2	2	2	2	3	3	3	3	3
Min	0.006	0.0010	0.006	0.006	0.0020	0.068	0.036	0.084	0.072	0.0130	0.609	0.57	1.11	0.67	0.496	0.978	0.438	1.212	1.042	0.102	0.010	0.012	0.022	0.010	0.006
Median	0.026	0.0110	0.020	0.028	0.0073	0.117	0.085	0.159	0.125	0.0370	0.810	0.67	1.40	0.89	0.665	1.910	0.840	2.411	3.562	0.172	0.011	0.015	0.023	0.011	0.007
Max	0.054	0.0265	0.064	0.051	0.0186	0.222	0.176	0.273	0.246	0.1060	0.967	1.08	1.86	1.15	0.950	2.841	1.241	3.610	6.081	0.242	0.015	0.016	0.037	0.016	0.012
SD	0.018	0.0080	0.014	0.018	0.0056	0.027	0.016	0.051	0.031	0.0090	0.095	0.11	0.19	0.11	0.064										
CV	69	73	69	64	77	23	18	32	24	24	12	16	13	12	10										
N_{past}	11	11	8	12	12	21	21	18	23	20	7	6	8	10	6						7		8	7	
$Median_{past}$	0.036	0.0220	0.029	0.038	0.0160	0.124	0.086	0.143	0.128	0.0383	0.815	0.62	1.38	0.76	0.561						0.010		0.018	0.008	
SD_{past}	0.009	0.0046	0.013	0.011	0.0038	0.020	0.010	0.031	0.022	0.0106	0.171	0.10	0.20	0.18	0.102						0.004		0.009	0.005	
NAV	0.026	0.0110	0.020	0.028	0.0073	0.117	0.085	0.159	0.125	0.0370	0.810	0.67	1.40	0.89	0.665						0.011	0.015	0.023	0.011	0.007
NAU	0.018	0.0080	0.014	0.018	0.0056	0.027	0.018	0.051	0.031	0.0090	0.095	0.11	0.19	0.11	0.067										

Round Robin LXX Laboratory Results

Analytes Reported By One Laboratory

Analyte	Code	377	378	379	380	381
Phytofluene, µg/mL	FSV-BS	0.049	0.082	0.351	0.052	0.040
Phytoene, µg/mL	FSV-BS	*nd*	*nd*	0.124	*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.
nd	Not detected (i.e., no detectable peak for analyte)
nq	Detected but not quantitatively determined
?	Non-quantitative value: extrapolated beyond upper limit of calibration curve
italics	Not explicitly reported but calculated by NIST from reported values

Round Robin LXX Laboratory Results
Comparability Summary

Lab	TR	aT	g/bT	bC	tbC	aC	TLy	TbX	TLu	TZ	L&Z
FSV-BA	2	1	1	1	1	1	1	2			2
FSV-BB	1	1	1	1	1	1	1	1	1	3	1
FSV-BC	1										
FSV-BD	1	1									
FSV-BE	1	2	1	1							
FSV-BF	1	2		1							
FSV-BG	2	1	2	2		1	1	1			1
FSV-BH	1	1	1	1	1	1	1	1	1	2	1
FSV-BJ	1	1	1	1			2	2	1		
FSV-BK	1	1									
FSV-BL	1	1									
FSV-BM	1	2									
FSV-BN	2	2		2		2	1	1			2
FSV-BO	2	2	1	1		1	2	1	1	1	1
FSV-BP	1	1		2		4	2	2			1
FSV-BQ	2	2									
FSV-BR	1	1									
FSV-BS	1			2	2	2	1	2	2	2	3
FSV-BT	2	1	1	1	1	1	1	1			1
FSV-BU	1	1	1	2		2	1	1			1
FSV-BV	1	2	1	1		1	1	1			1
FSV-BW	1	2	1	2		2	2				
FSV-CC	1	1									
FSV-CD	2	2	3	4		4	3	2			4
FSV-CE	2	2		3							
FSV-CG	1	1	1	1	1	2	1	2			2
FSV-CI	2	1	1	1		2			1	1	1
FSV-CW	3	2	1	1		1		1			2
FSV-CZ	1	1	1	2							
FSV-DD	1										
FSV-DQ		2	3	2		1	2	2	1	1	1
FSV-DV	1	1									
FSV-FK	1	1									
n	32	30	17	22	6	17	16	16	7	6	16

	TR	aT	g/bT	bC	tbC	aC	TLy	TbX	TLu	TZ	L&Z
% 1	69	60	82	55	83	53	63	56	86	50	63
% 2	28	40	6	36	17	35	31	44	14	33	25
% 3	3	0	12	5	0	0	6	0	0	17	6
% 4	0	0	0	5	0	12	0	0	0	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:

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

$$\text{C} = \text{Concordance} = \frac{\sum_{i=1}^{N_{you}} \frac{\text{You}_i - \text{Median}_i}{\text{NAU}_i}}{N_{you}}$$

$$\text{AP} = \text{Apparent Precision} = \sqrt{\frac{\sum_{i=1}^{N_{you}} \left(\frac{\text{You}_i - \text{Median}_i}{\text{NAU}_i}\right)^2}{N_{you}-1}}$$

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 RR70

Each participant in RR70 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 RR70:

- Total Retinol
- Retinyl Palmitate
- 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 Lutein
- Total Zeaxanthin
- Total Lutein & Zeaxanthin
- Coenzyme Q10

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

Individualized Round Robin LXX Report: FSV-BA

Summary

Analyte	Serum 377 You	Serum 377 NAV	Serum 377 n	Serum 378 You	Serum 378 NAV	Serum 378 n	Serum 379 You	Serum 379 NAV	Serum 379 n	Serum 380 You	Serum 380 NAV	Serum 380 n	Serum 381 You	Serum 381 NAV	Serum 381 n
Total Retinol	0.641	0.641	31	0.486	0.486	31	0.683	0.649	31	0.688	0.669	31	0.397	0.373	31
Retinyl Palmitate	0.02	0.02	7	0.0	0.1	7	0.1	0.1	7	0.02	0.02	7	0.01	0.01	7
α-Tocopherol	5.45	5.97	30	8.57	9.29	30	16.79	18.65	30	5.84	6.24	30	3.71	4.19	30
γ/β-Tocopherol	2.081	2.144	17	1.644	1.709	17	2.156	2.170	17	2.186	2.285	17	1.624	1.642	17
δ-Tocopherol	0.079	0.079	5	0.108	0.107	5	0.191	0.196	5	0.079	0.079	5	0.098	0.098	5
Total β-Carotene	0.454	0.462	21	0.329	0.266	21	0.340	0.384	21	0.482	0.484	21	0.040	0.040	20
trans-β-Carotene	0.409	0.427	6	0.256	0.253	6	0.317	0.372	6	0.444	0.461	6	0.035	0.035	6
Total cis-β-Carotene	0.029	0.021	5	0.018	0.017	5	0.015	0.016	5	0.021	0.019	5	0.001	0.003	3
Total α-Carotene	0.016	0.016	17	0.055	0.052	18	0.009	0.015	15	0.017	0.019	17	0.004	0.006	13
Total Lycopene	0.199	0.219	16	0.209	0.216	16	0.733	0.907	16	0.202	0.230	16	0.113	0.144	15
trans-Lycopene	0.100	0.128	7	0.097	0.128	7	0.314	0.446	7	0.111	0.148	7	0.052	0.082	7
Total β-Cryptoxanthin	0.041	0.045	16	0.039	0.042	16	0.032	0.035	16	0.045	0.050	16	0.029	0.029	15
Total α-Cryptoxanthin	0.019	0.024	4	0.010	0.014	4	0.013	0.031	4	0.021	0.026	4	0.007	0.016	3
Total Lutein	0.099	0.083	7	0.068	0.063	7	0.145	0.123	7	0.102	0.085	7	0.022	0.022	6
Total Zeaxanthin	0.042	0.026	6	0.026	0.011	6	0.064	0.020	6	0.044	0.028	6	0.019	0.007	6
Total Lutein&Zeaxanthin	0.141	0.117	16	0.094	0.085	16	0.209	0.159	16	0.146	0.125	16	0.041	0.037	15

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

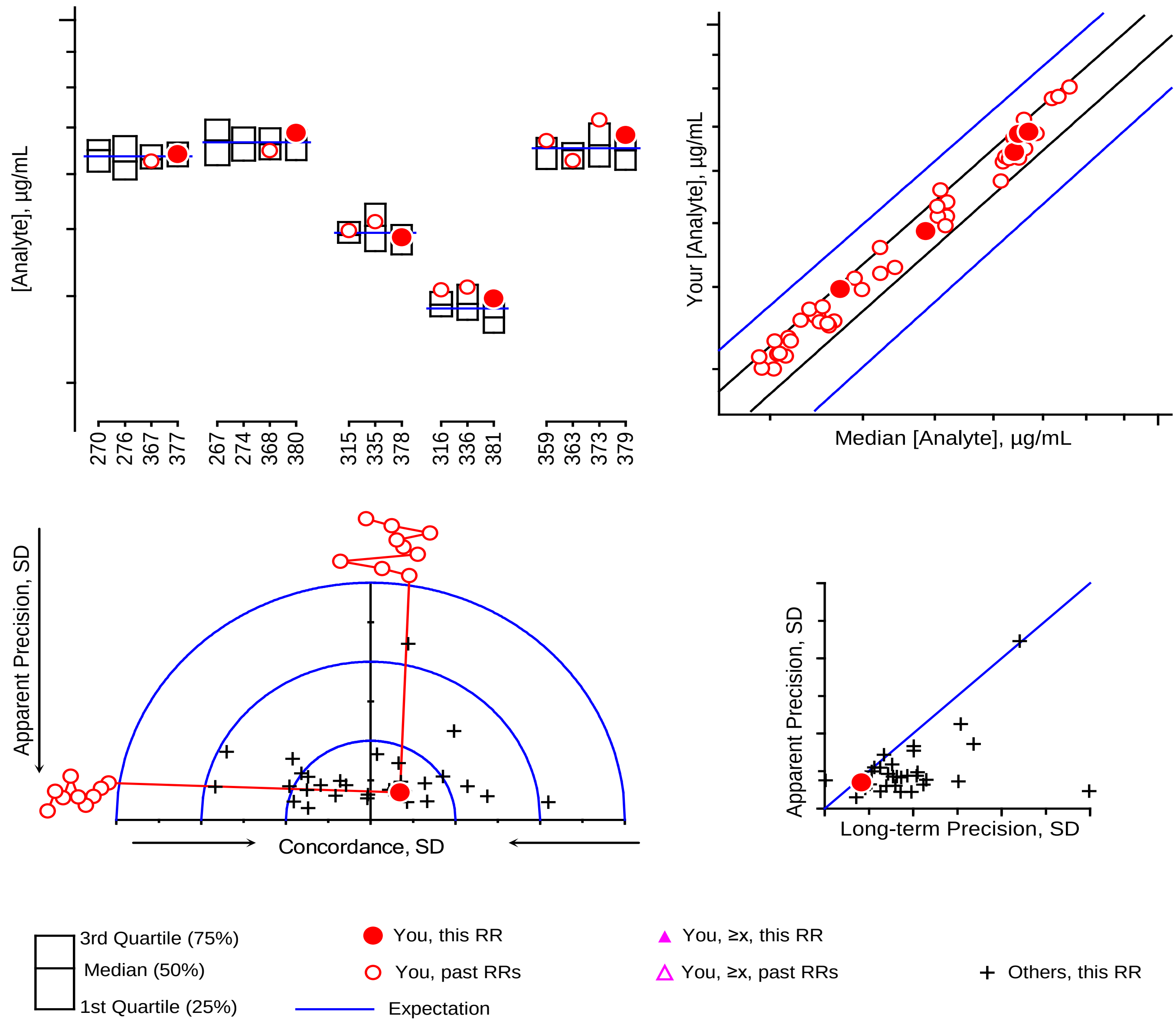
Please check our records against your records. Send corrections and/or updates to...

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 LXX Report: FSV-BA

Total Retinol, µg/mL

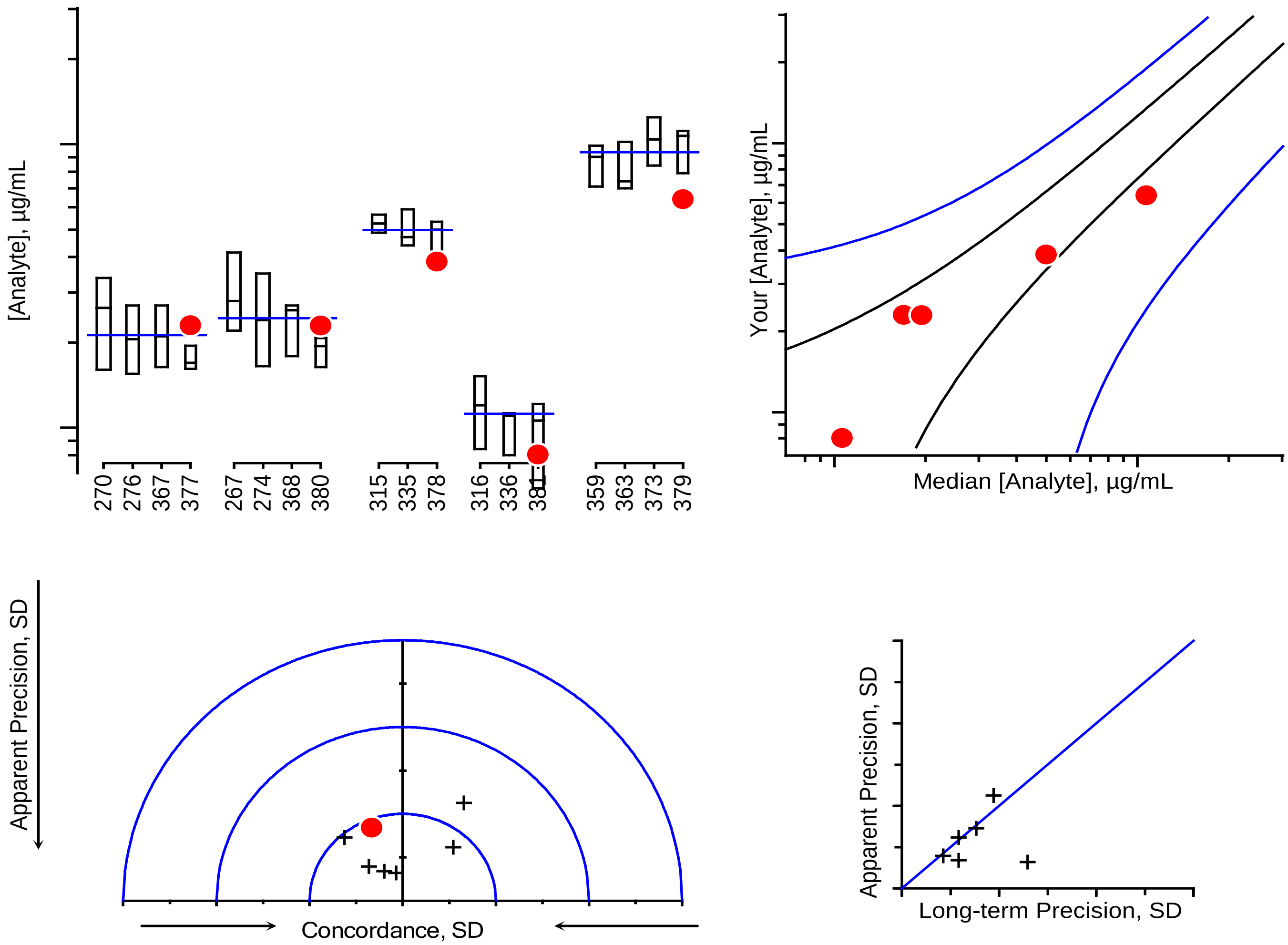


For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

Serum	Comments	History
#377	Lyophilized, same native pool as #380	49#270, 50#276, 68#367
#378	Fresh-frozen, native, multi-donor	58#315, 62#335
#379	Fresh-frozen, native, multi-donor: SRM 968e III	66#359, 67#363, 69#373
#380	Fresh-frozen, same native pool as #377	48#267, 50#274, 68#368
#381	Fresh-frozen, native, multi-donor	58#316, 62#336

Individualized RR LXX Report: FSV-BA

Retinyl Palmitate, 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

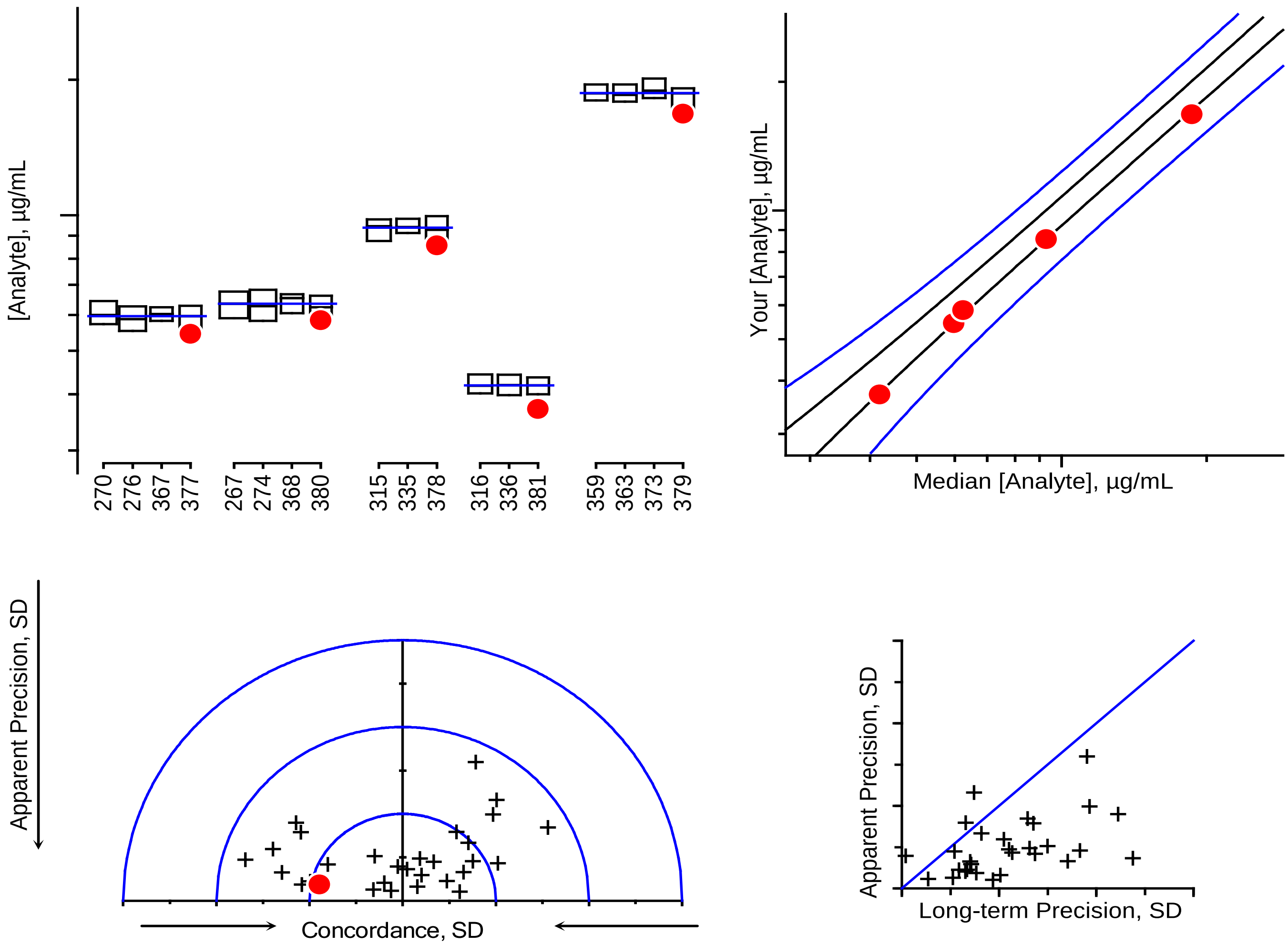
Others, this RR

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
#377	Lyophilized, same native pool as #380	49#270, 50#276, 68#367
#378	Fresh-frozen, native, multi-donor	58#315, 62#335
#379	Fresh-frozen, native, multi-donor: SRM 968e III	66#359, 67#363, 69#373
#380	Fresh-frozen, same native pool as #377	48#267, 50#274, 68#368
#381	Fresh-frozen, native, multi-donor	58#316, 62#336

Individualized RR LXX 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

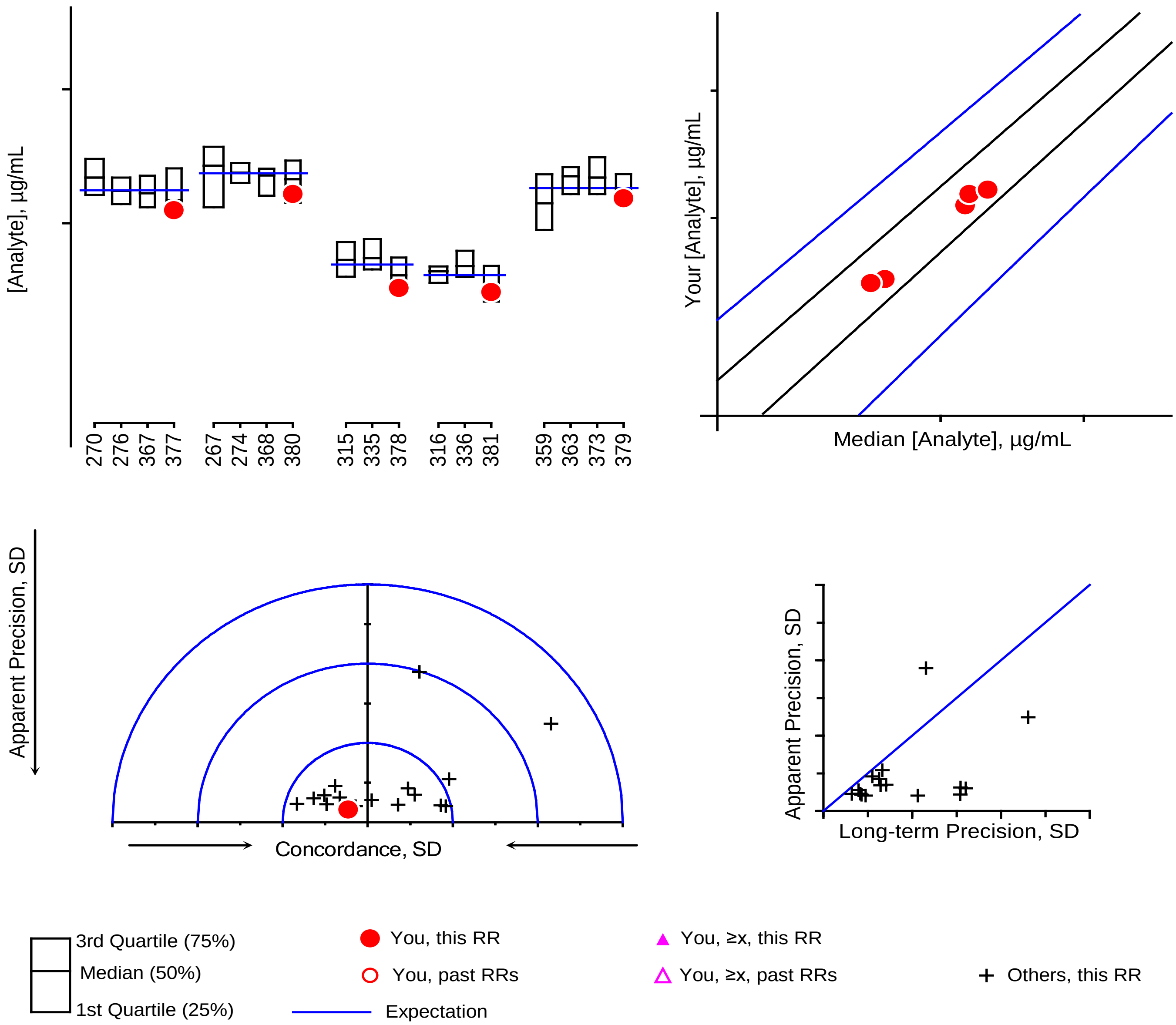
Others, this RR

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
#377	Lyophilized, same native pool as #380	49#270, 50#276, 68#367
#378	Fresh-frozen, native, multi-donor	58#315, 62#335
#379	Fresh-frozen, native, multi-donor: SRM 968e III	66#359, 67#363, 69#373
#380	Fresh-frozen, same native pool as #377	48#267, 50#274, 68#368
#381	Fresh-frozen, native, multi-donor	58#316, 62#336

Individualized RR LXX Report: FSV-BA

γ/β-Tocopherol, µg/mL

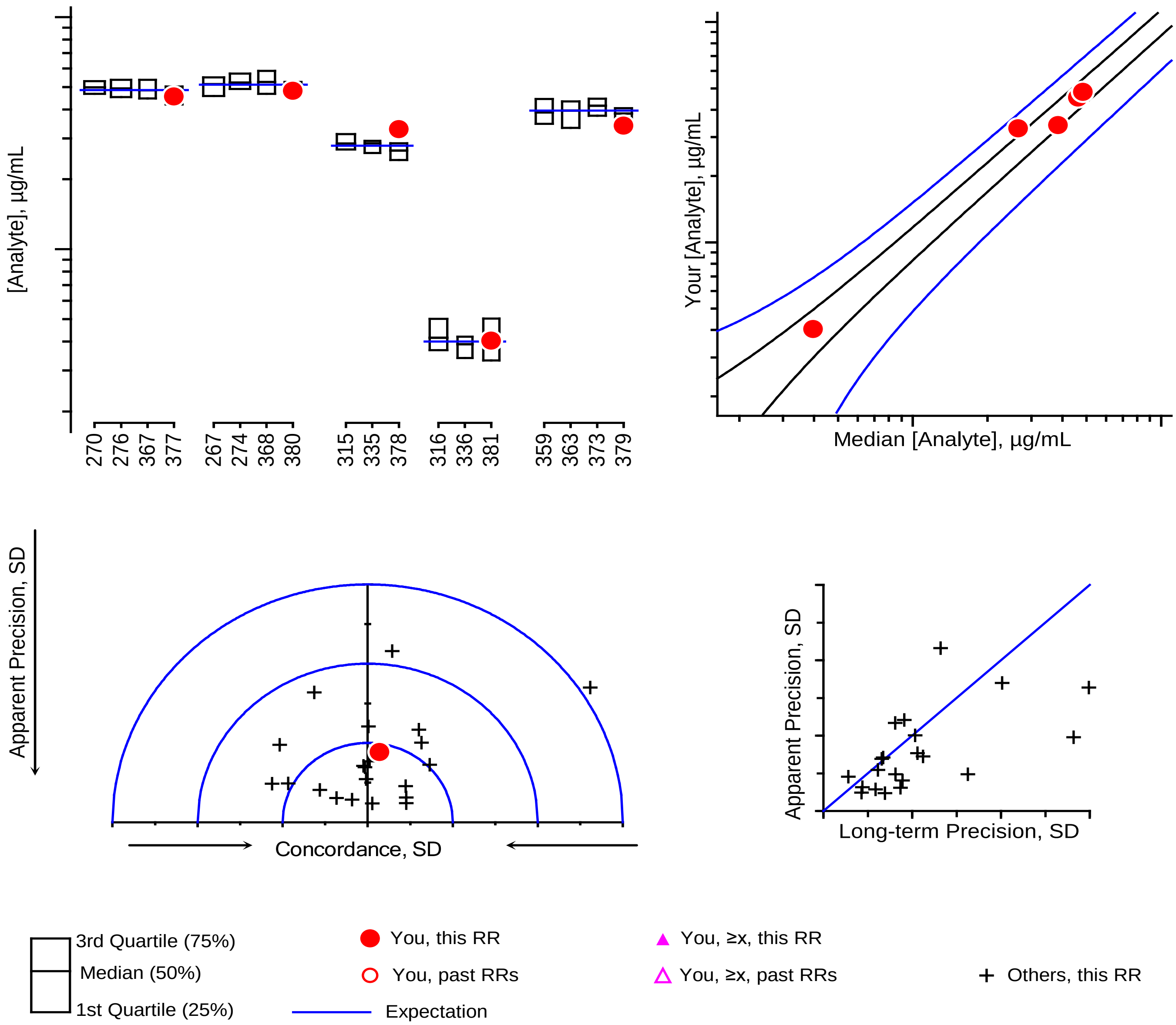


For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

Serum	Comments	History
#377	Lyophilized, same native pool as #380	49#270, 50#276, 68#367
#378	Fresh-frozen, native, multi-donor	58#315, 62#335
#379	Fresh-frozen, native, multi-donor: SRM 968e III	66#359, 67#363, 69#373
#380	Fresh-frozen, same native pool as #377	48#267, 50#274, 68#368
#381	Fresh-frozen, native, multi-donor	58#316, 62#336

Individualized RR LXX Report: FSV-BA

Total β-Carotene, µg/mL

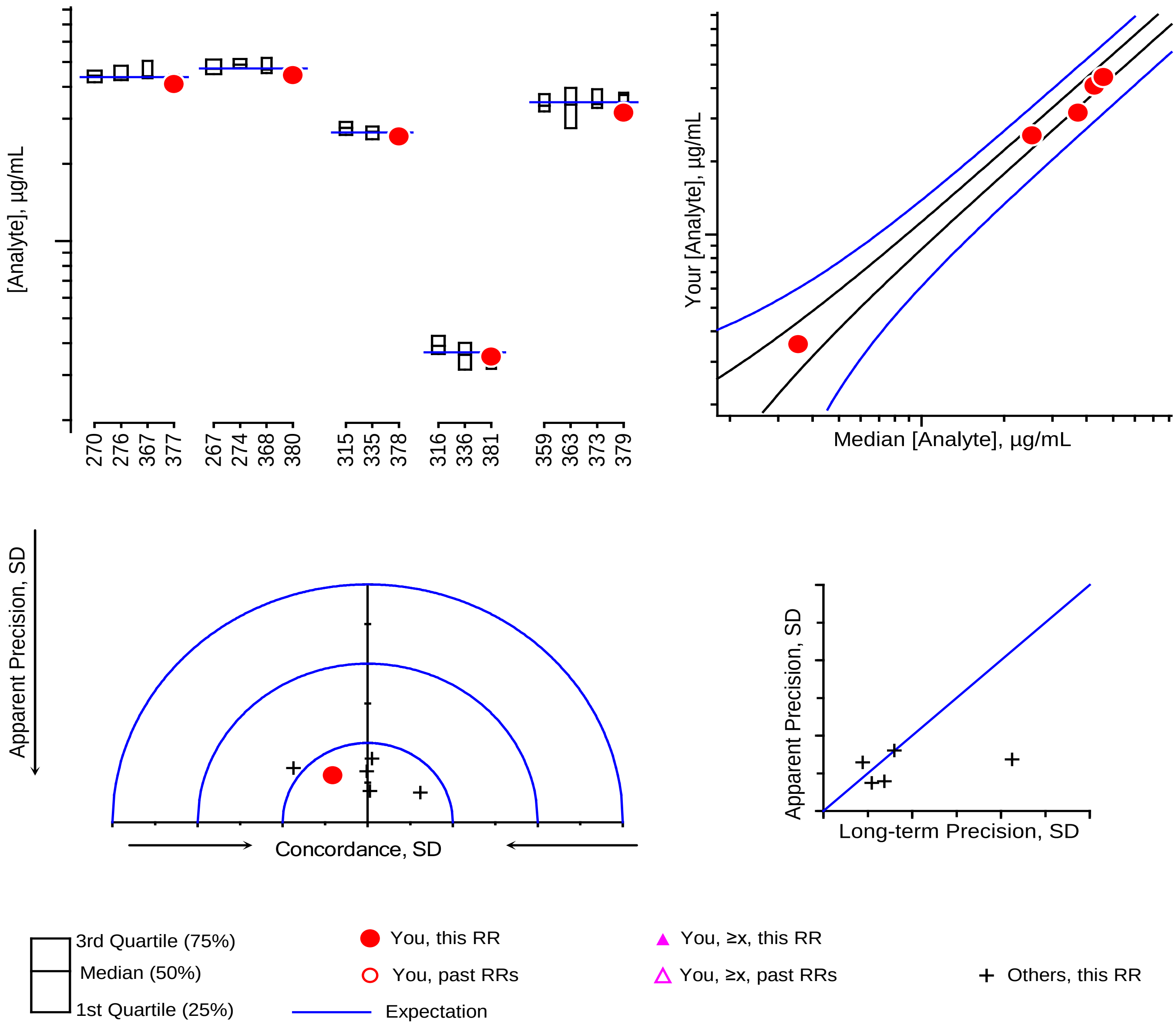


For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

Serum	Comments	History
#377	Lyophilized, same native pool as #380	49#270, 50#276, 68#367
#378	Fresh-frozen, native, multi-donor	58#315, 62#335
#379	Fresh-frozen, native, multi-donor: SRM 968e III	66#359, 67#363, 69#373
#380	Fresh-frozen, same native pool as #377	48#267, 50#274, 68#368
#381	Fresh-frozen, native, multi-donor	58#316, 62#336

Individualized RR LXX Report: FSV-BA

trans-β-Carotene, µg/mL

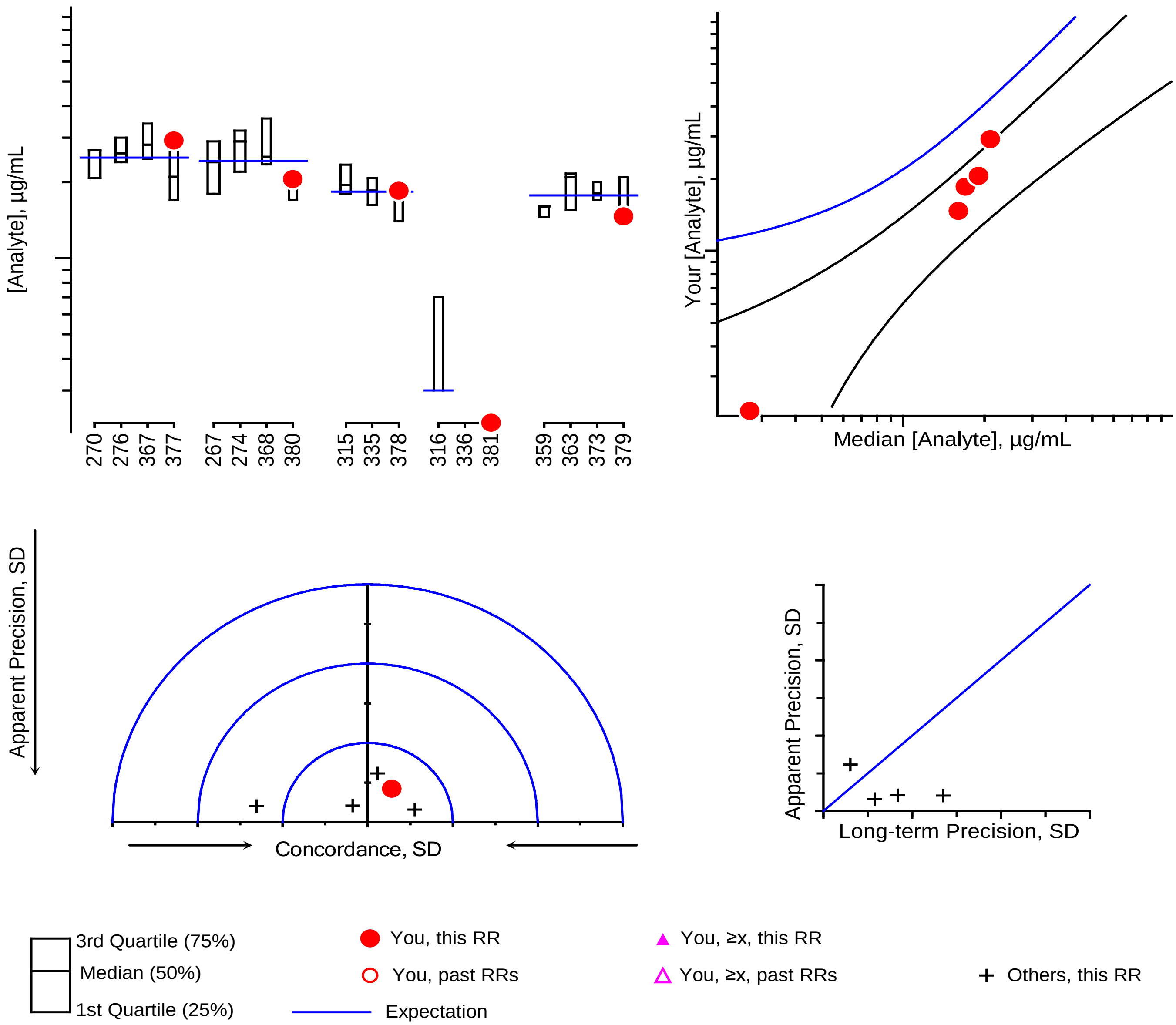


For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

Serum	Comments	History
#377	Lyophilized, same native pool as #380	49#270, 50#276, 68#367
#378	Fresh-frozen, native, multi-donor	58#315, 62#335
#379	Fresh-frozen, native, multi-donor: SRM 968e III	66#359, 67#363, 69#373
#380	Fresh-frozen, same native pool as #377	48#267, 50#274, 68#368
#381	Fresh-frozen, native, multi-donor	58#316, 62#336

Individualized RR LXX Report: FSV-BA

Total cis-β-Carotene, µg/mL

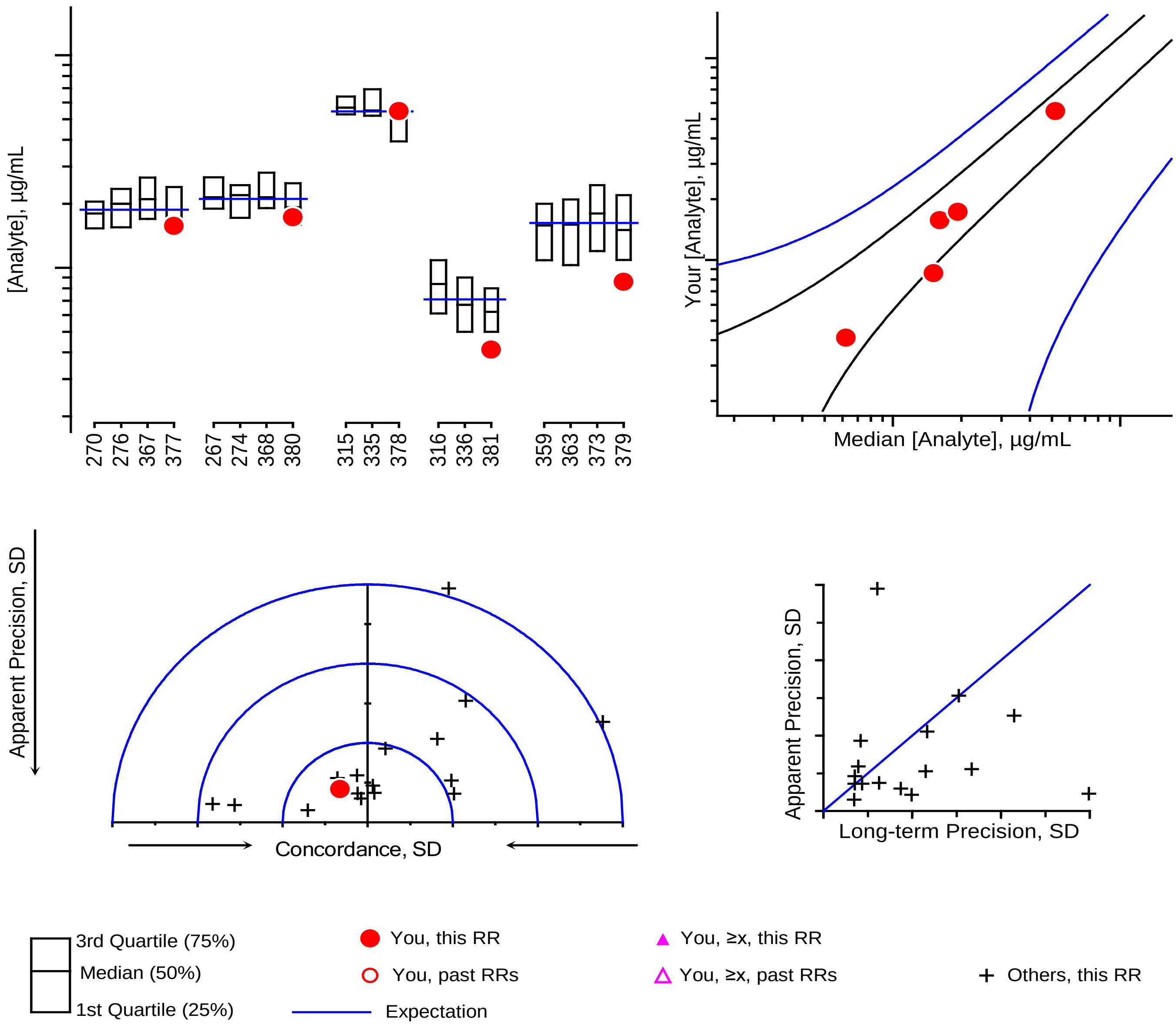


For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

Serum	Comments	History
#377	Lyophilized, same native pool as #380	49#270, 50#276, 68#367
#378	Fresh-frozen, native, multi-donor	58#315, 62#335
#379	Fresh-frozen, native, multi-donor: SRM 968e III	66#359, 67#363, 69#373
#380	Fresh-frozen, same native pool as #377	48#267, 50#274, 68#368
#381	Fresh-frozen, native, multi-donor	58#316, 62#336

Individualized RR LXX Report: FSV-BA

Total α-Carotene, µg/mL

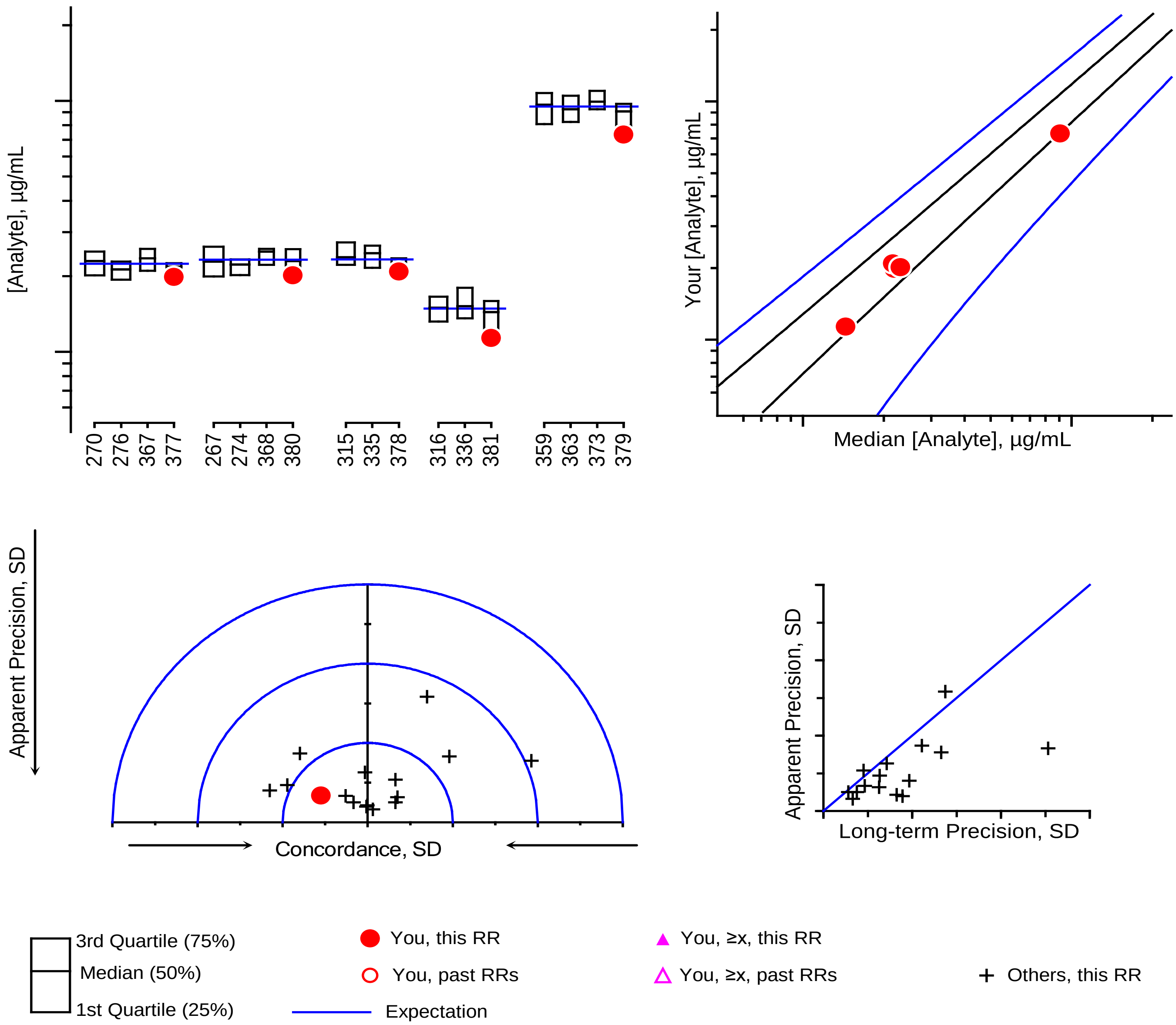


For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

Serum	Comments	History
#377	Lyophilized, same native pool as #380	49#270, 50#276, 68#367
#378	Fresh-frozen, native, multi-donor	58#315, 62#335
#379	Fresh-frozen, native, multi-donor: SRM 968e III	66#359, 67#363, 69#373
#380	Fresh-frozen, same native pool as #377	48#267, 50#274, 68#368
#381	Fresh-frozen, native, multi-donor	58#316, 62#336

Individualized RR LXX 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
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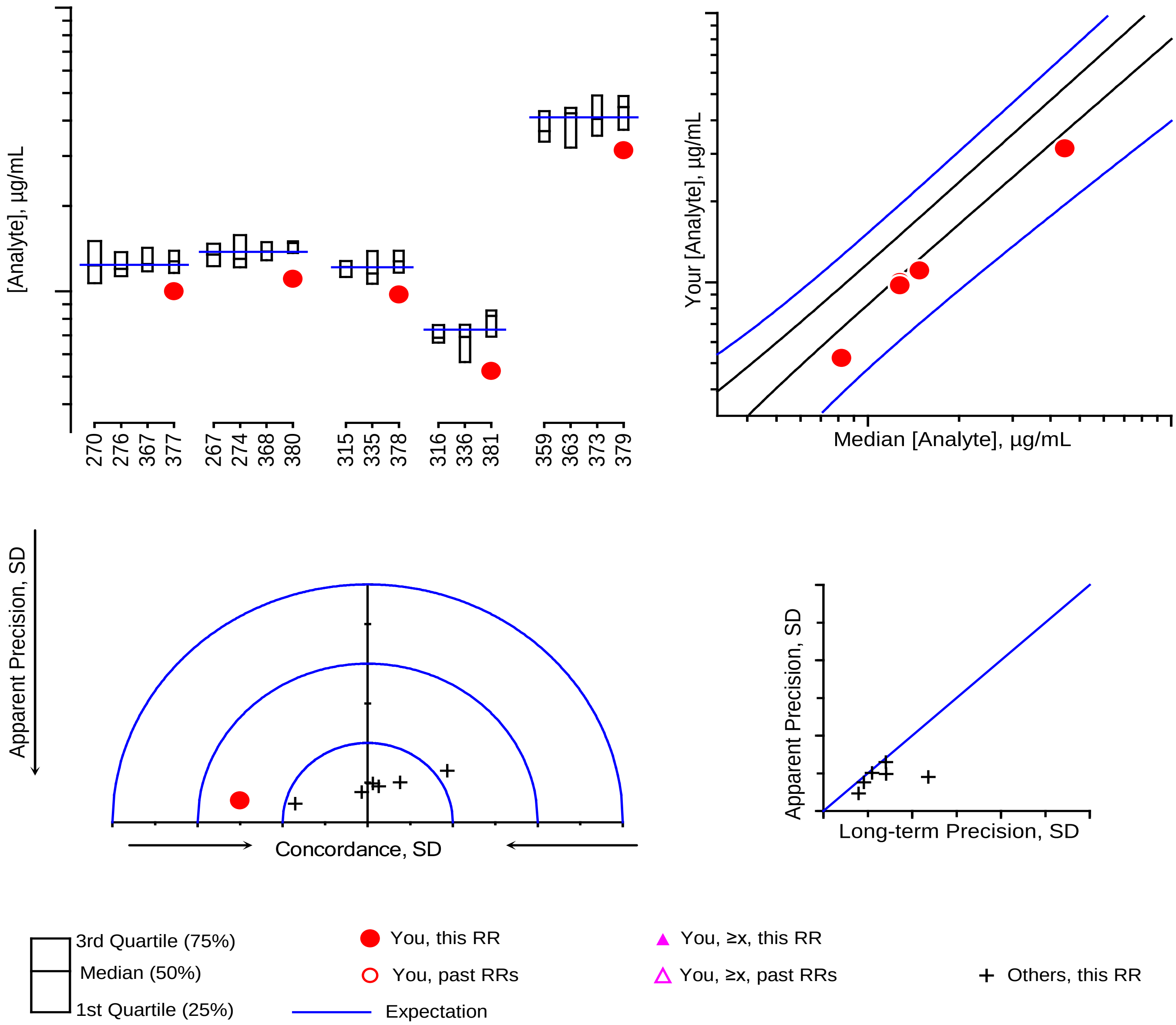
#379	Fresh-frozen, native, multi-donor: SRM 968e III	66#359, 67#363, 69#373
#380	Fresh-frozen, same native pool as #377	48#267, 50#274, 68#368
#381	Fresh-frozen, native, multi-donor	58#316, 62#336

Individualized RR LXX Report: FSV-BA

trans-Lycopene, µg/mL



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Serum	Comments	History
#377	Lyophilized, same native pool as #380	49#270, 50#276, 68#367
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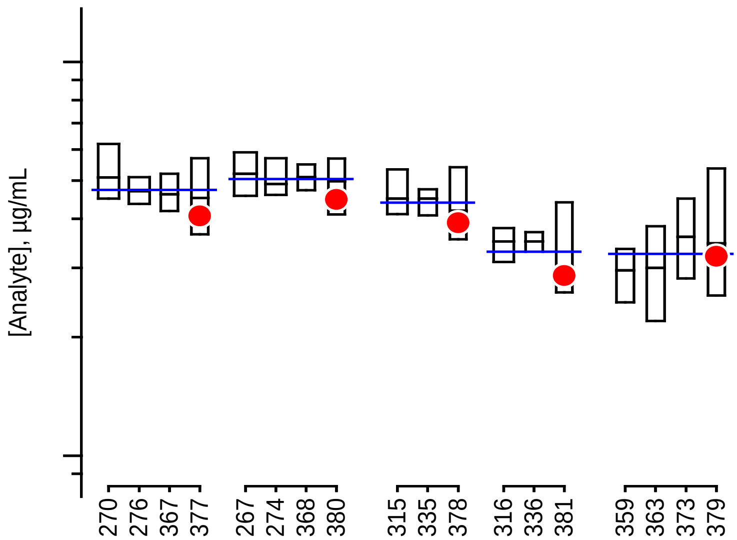
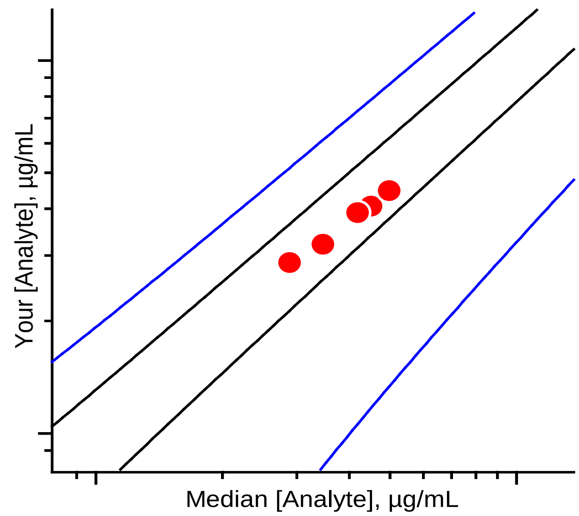
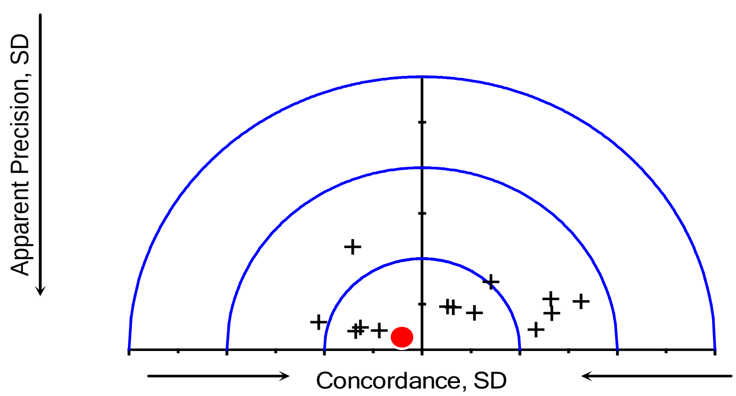
#379	Fresh-frozen, native, multi-donor: SRM 968e III	66#359, 67#363, 69#373
#380	Fresh-frozen, same native pool as #377	48#267, 50#274, 68#368
#381	Fresh-frozen, native, multi-donor	58#316, 62#336

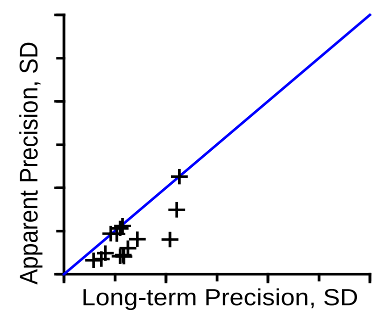
Individualized RR LXX Report: FSV-BA

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
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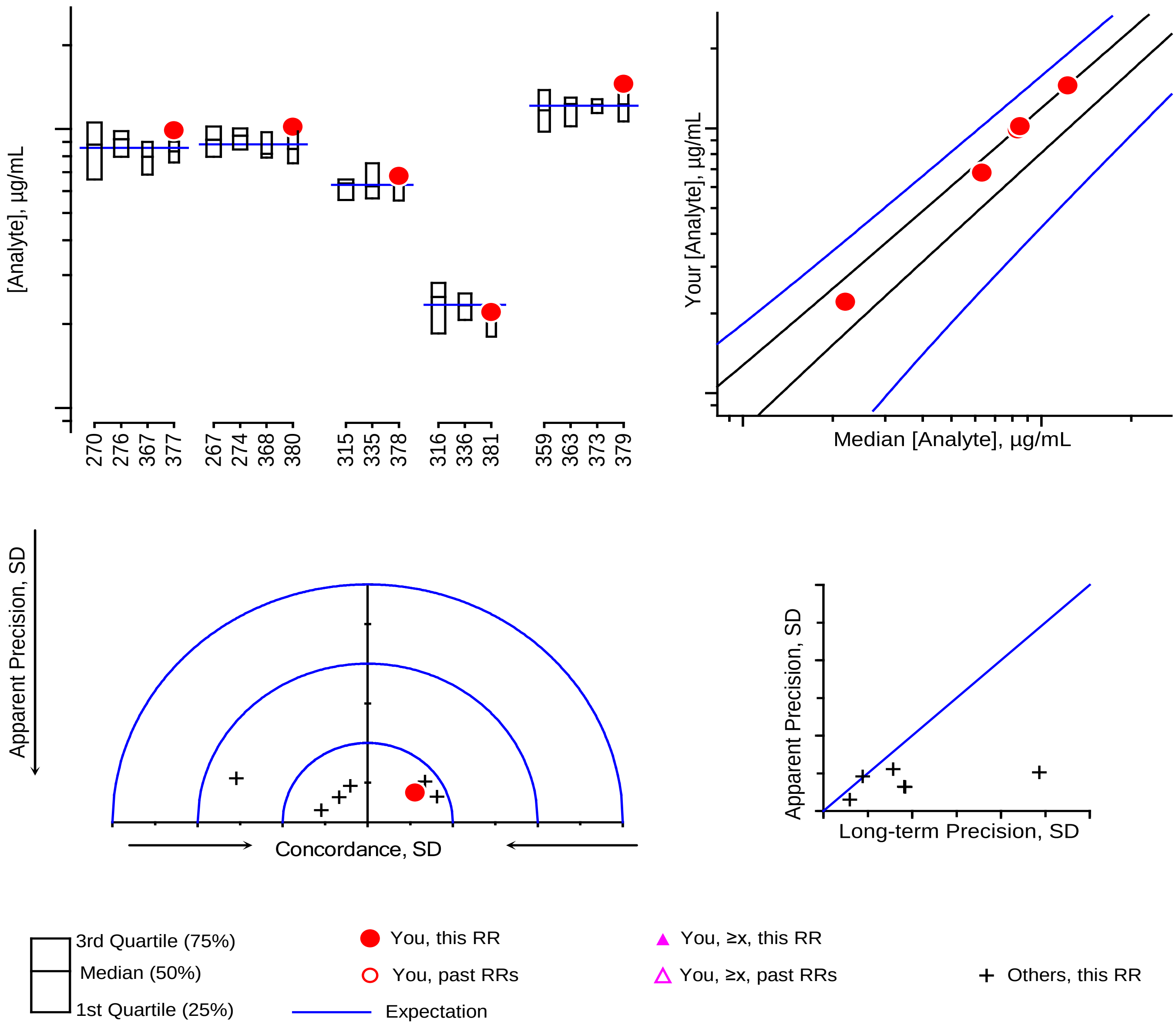
#379	Fresh-frozen, native, multi-donor: SRM 968e III	66#359, 67#363, 69#373
#380	Fresh-frozen, same native pool as #377	48#267, 50#274, 68#368
#381	Fresh-frozen, native, multi-donor	58#316, 62#336

Individualized RR LXX Report: FSV-BA

Total Lutein, µg/mL



For details of the construction and interpretation of these plots, see:
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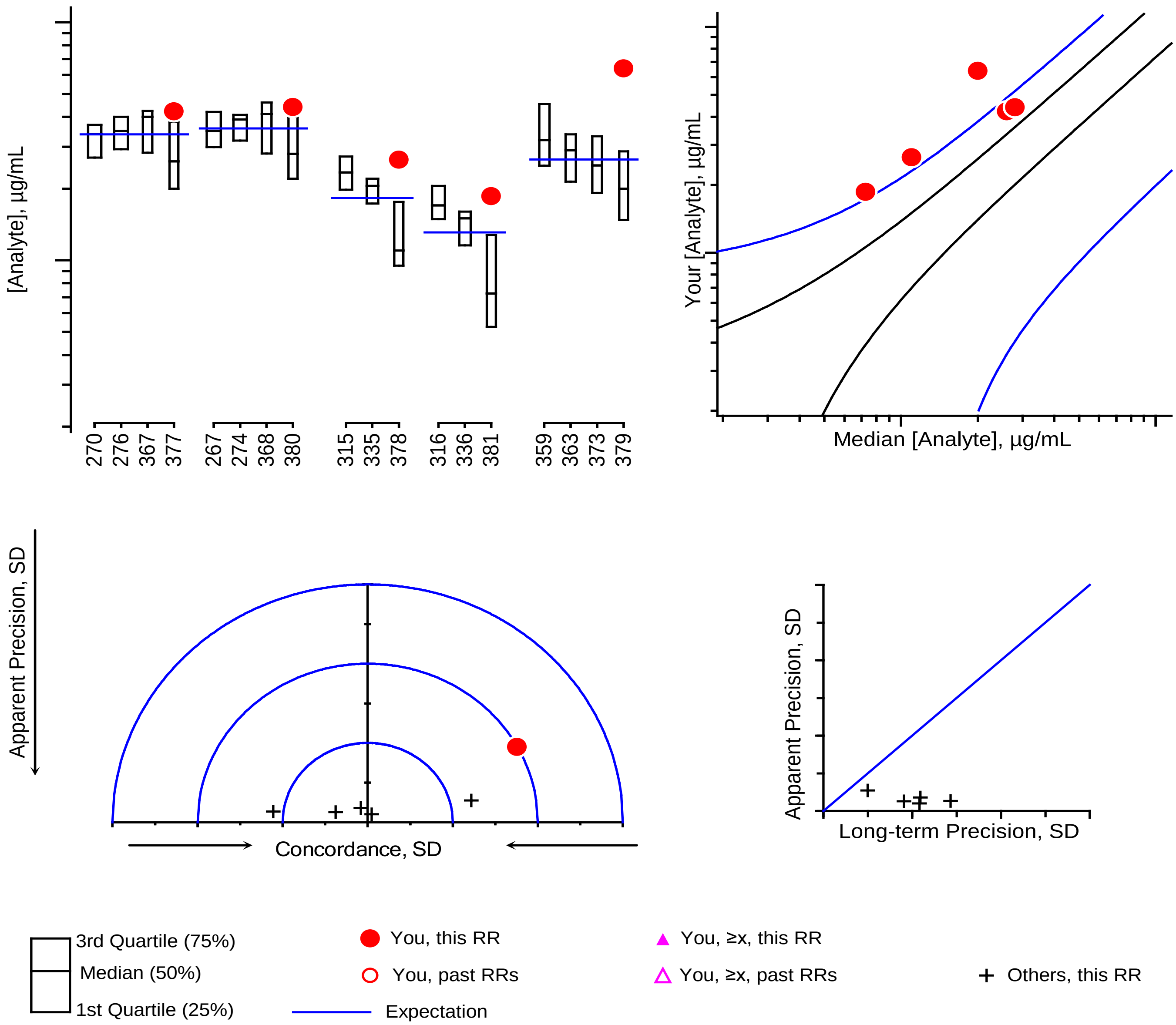
#377	Lyophilized, same native pool as #380	49#270, 50#276, 68#367
#378	Fresh-frozen, native, multi-donor	58#315, 62#335
#379	Fresh-frozen, native, multi-donor: SRM 968e III	66#359, 67#363, 69#373
#380	Fresh-frozen, same native pool as #377	48#267, 50#274, 68#368
#381	Fresh-frozen, native, multi-donor	58#316, 62#336

Individualized RR LXX Report: FSV-BA



For details of the construction and interpretation of these plots, see:
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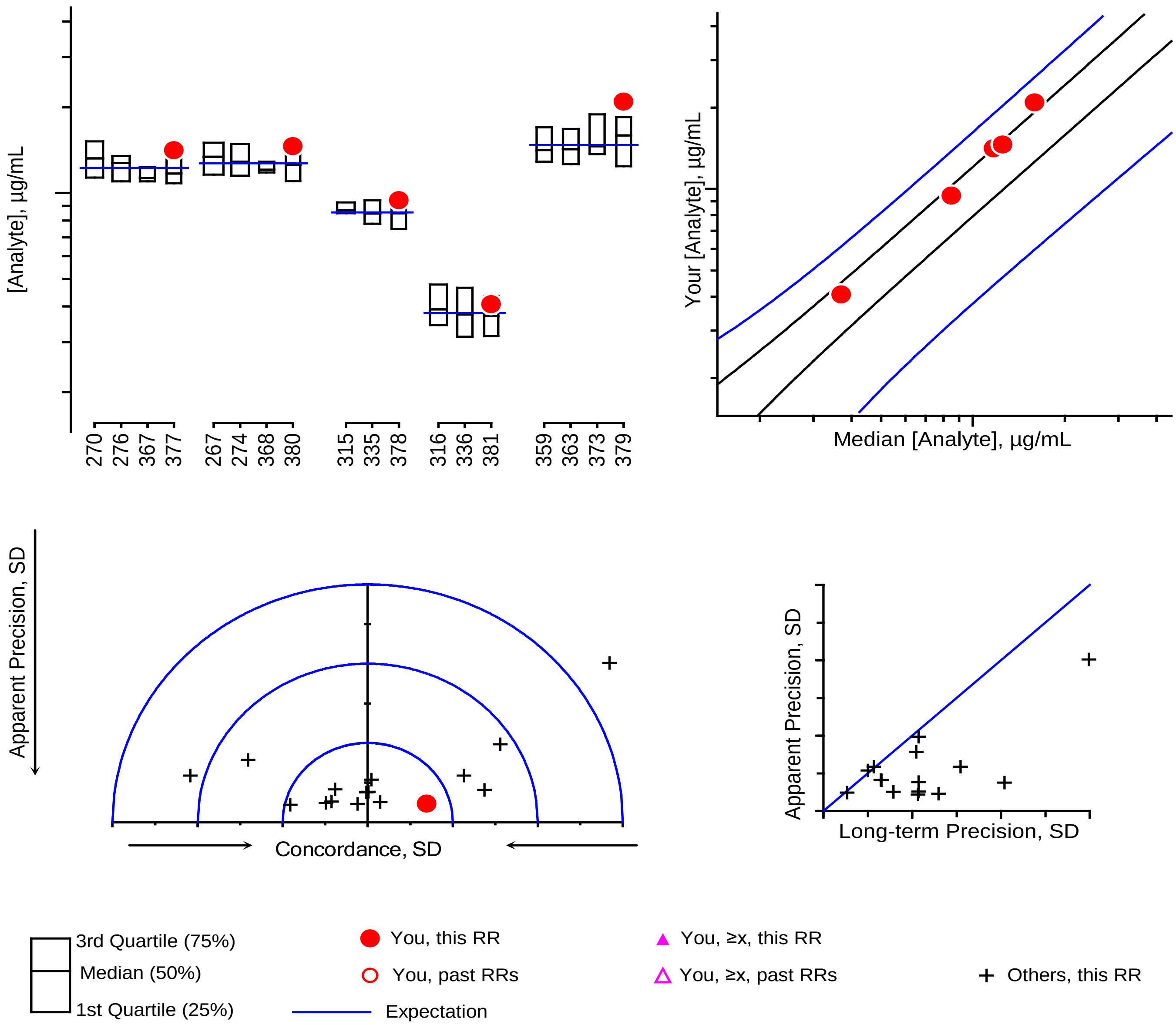
Serum	Comments	History
#377	Lyophilized, same native pool as #380	49#270, 50#276, 68#367
#378	Fresh-frozen, native, multi-donor	58#315, 62#335
#379	Fresh-frozen, native, multi-donor: SRM 968e III	66#359, 67#363, 69#373
#380	Fresh-frozen, same native pool as #377	48#267, 50#274, 68#368
#381	Fresh-frozen, native, multi-donor	58#316, 62#336

Individualized RR LXX Report: FSV-BA

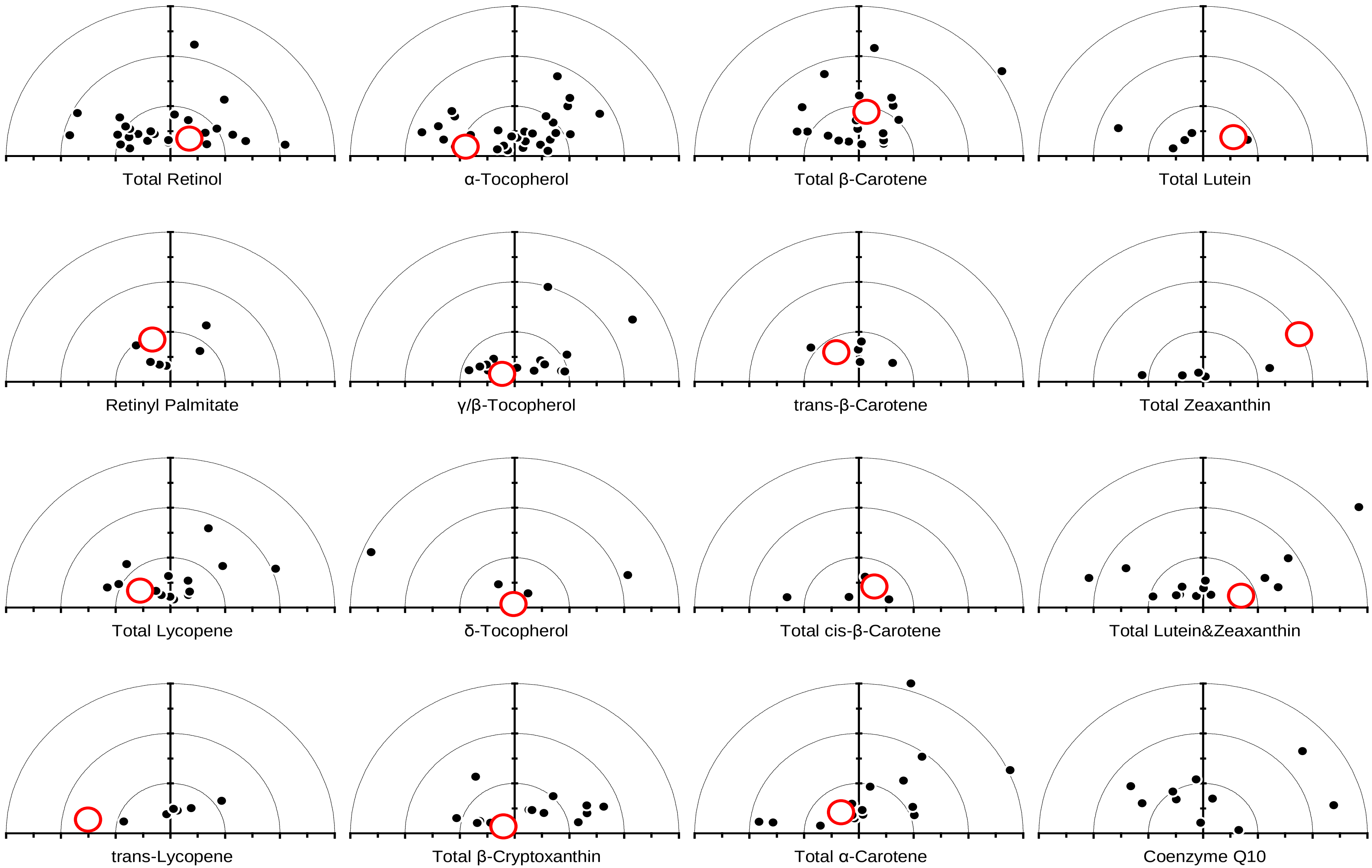
Total Lutein&Zeaxanthin, µg/mL



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
#377	Lyophilized, same native pool as #380	49#270, 50#276, 68#367
#378	Fresh-frozen, native, multi-donor	58#315, 62#335
#379	Fresh-frozen, native, multi-donor: SRM 968e III	66#359, 67#363, 69#373
#380	Fresh-frozen, same native pool as #377	48#267, 50#274, 68#368
#381	Fresh-frozen, native, multi-donor	58#316, 62#336

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



D17

Appendix E. Shipping Package Inserts for RR35

The following five items were included in each package shipped to an RR35 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-

May 23, 2011

Dear Colleague:

The samples within this package constitute Vitamin C Round Robin 35 (RR35) of the 2011Micronutrients Measurement Quality Assurance Program. RR35 consists of four vials of frozen serum *test samples* (#351, #352 #353, and #354), one vial of frozen *control serum* (CS #1), 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* #1 is 8.41 µmol/L of sample; the ≈95% confidence interval for the target value is 7.75 to 9.07 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 RR35 by e-mail to david.duewer@nist.gov or fax to 301-977-0685 by **September 19, 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 B Thomas

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
RR35 Report Form for Ascorbic Acid Solid Control Material Preparation
RR35 Report Form for Control Material and Test Sample Analyses

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:

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

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 35

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 **September 19, 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 35

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 #1			µmol/L of Sample *Target:* 8.41 µmol/L (7.75 µmol/L to 9.07 µmol/L; 95 % confidence interval)
Serum Test Sample #351			µmol/L of Sample
Serum Test Sample #352			µmol/L of Sample
Serum Test Sample #353			µmol/L of Sample
Serum Test Sample #354			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 **September 19, 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 35

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^{2}QAP samples:

Label	Form
VitC #351	Liquid frozen (1:1 serum:10% MPA)
VitC #352	Liquid frozen (1:1 serum:10% MPA)
VitC #353	Liquid frozen (1:1 serum:10% MPA)
VitC #354	Liquid frozen (1:1 serum:10% MPA)
CS #1	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^{2}QAP Gang

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

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

Appendix F. Final Report for RR35

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-

November 21, 2011

Dear Colleague:

Enclosed is the summary report of the results for Round Robin 35 (RR35) 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.

RR35 consisted of four *test samples* (S351, S352, S353, and S354), one *serum control material* (CS#1), 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.

Samples for the first vitamin C round robin (RR36) of the 2012 M^2QAP will be shipped **starting January 9, 2012.** Please contact us immediately if this schedule is problematic for your laboratory.

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 301-975-3320; e-mail: jbthomas@nist.gov; or fax: 301-977-0685.

Sincerely,

Jeanice Brown Thomas, M.B.A.
Research Chemist
Analytical Chemistry Division
Material Measurement Laboratory

David L. Duewer, Ph.D.
Research Chemometrician
Analytical Chemistry Division
Material Measurement Laboratory

Enclosures

Cc: L. C. Sander

NIST

The NIST M^2QAP Vitamin C Round Robin 35 (RR35) 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 RR35 sample measurements.

Page	"All Lab" Report
1	A tabulation of results and summary statistics for Total Ascorbic Acid [TAA] in the RR35 samples and control/calibration solutions.

Serum-based Samples. One serum control and four unknowns were distributed in RR35.

CS#1 SRM 970 level 1, ampouled in mid-1998.

S35:1 Ampouled in late 2009, previously distributed as sample S33:3 (RR33, Fall 09) and S34:2 (RR34, Spring 10)

S35:2 Ampouled in late 2009, previously distributed as sample S32:2 (RR32, Spring 09) and S34:2 (RR34, Spring 10)

S35:3 Ampouled in late 2009, previously distributed as sample S32:3 (RR32, Spring 09)

S35:4 Ampouled in late 2009, previously distributed as sample S32:4 (RR32, Spring 09) and S34:3 (RR34, Spring 10)

Results.

1) All 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 S35:1 to S35:4 (columns 23 to 26 of the All Lab Report) confirm the linearity of all 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 RR35

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" 35 - October 2011

		Control / Calibration Samples											MPA	Dilute Solution 1		
		Grav, µmol/L			Measured, µmol/L				Measured = a + b*Grav				Density	Spectrophotometry		
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}	$E^{1\%}$
VC-MA	24/08/11	58.9	28.9	14.4	60.8	29.5	15.1	0.0	0.00	1.03	1.000	0.2	1.036	242.	0.5680	547.7
VC-MB	13/06/11	57.9	29.2	14.6	60.1	30.2	15.2	0.0	0.02	1.04	1.000	0.1	1.029	244.	0.5650	553.9
VC-MC	18/08/11	60.1	30.3	15.0	61.9	31.3	15.4	0.0	-0.01	1.03	1.000	0.0	1.030	243.	0.5894	556.9
VC-ME	05/07/11	57.7	28.7	14.3	58.4	28.4	15.2	0.0	0.16	1.01	1.000	0.7	1.030	243.3	0.5681	558.8
VC-MG	29/08/11	60.4	30.8	15.7	63.5	31.9	15.9	0.0	-0.32	1.05	1.000	0.4	1.028	243.8	0.5900	555.0
VC-MH	26/07/11	60.6	30.7	15.1	59.4	30.1	14.9	0.0	0.03	0.98	1.000	0.1	1.031	243.7	0.5970	559.2
VC-MI	29/07/11	54.8	27.3	13.6	55.2	27.1	13.6	0.0	-0.11	1.01	1.000	0.2	1.034			
VC-MJ	13/09/11	57.5	27.6	15.0	56.4	27.3	14.0	0.3	-0.01	0.98	1.000	0.5	1.024	255a	0.35a	345.4a
VC-MN	02/08/11	56.4	28.4	14.5	58.5	29.4	14.7	0.0	-0.13	1.04	1.000	0.2	1.034	243.9	0.5347	538.5
VC-NM	08/08/11	55.0	28.5	15.2	57.4	29.0	16.2	0.0	-0.01	1.04	1.000	0.5	1.031	243.	0.5430	560.1
	N	10	10	10	10	10	10	10				N	10	8	8	8
	Average	57.9	29.0	14.7	59.2	29.4	15.0	0.0				Average	1.031	243.3	0.5694	553.8
	SD	2.1	1.2	0.6	2.5	1.6	0.8	0.1				SD	0.003	0.7	0.0224	7.3
	Min	54.8	27.29	13.6	55.2	27.07	13.6	0.0				Min	1.024	242.0	0.5347	538.5
	%25	56.7	28.41	14.4	57.6	28.57	14.8	0.0				%25	1.029	243.0	0.5595	552.3
	Median	57.8	28.80	14.8	58.9	29.43	15.1	0.0				Median	1.031	243.5	0.5681	556.0
	%75	59.8	30.03	15.1	60.6	30.17	15.3	0.0				%75	1.033	243.8	0.5896	558.9
	Max	60.6	30.80	15.7	63.5	31.90	16.2	0.3				Max	1.036	244.0	0.5970	560.1
	MADe	2.8	1.2	0.5	2.5	1.3	0.5	0.0				eSD	0.002	0.7	0.0321	4.5
	CV	5	4	3	4	4	3					CV	0.23	0.27	5.7	0.8

	Samples													
		Measured, µmol/L				Measured = p+q*Median					Calibrated, µmol/L			
Lab	CS#1	S35:1	S35:2	S35:3	S35:4	Inter	Slope	R^2	RMS	CS#1	S35:1	S35:2	S35:3	S35:4
VC-MA	9.0	16.6	24.6	33.4	50.0	-0.02	1.07	0.999	0.5	8.8	16.1	23.8	32.4	48.5
VC-MB	8.5	15.2	23.7	31.7	47.5	-0.33	1.03	1.000	0.1	8.2	14.7	22.9	30.6	45.8
VC-MC	8.6	16.9	26.2	33.6	51.4	0.24	1.10	0.999	0.5	8.4	16.4	25.4	32.6	49.9
VC-ME	8.4	15.0	22.2	28.2	41.9	1.96	0.86	1.000	0.3	8.2	14.7	21.9	27.8	41.5
VC-MG	8.1	14.4	23.2	31.1	48.3	-2.11	1.08	1.000	0.3	8.0	14.0	22.4	29.8	46.1
VC-MH	8.4	15.8	23.9	30.9	45.5	1.64	0.94	1.000	0.1	8.5	16.1	24.4	31.5	46.4
VC-MI	*b*	14.1	21.8	28.9	43.5	-0.15	0.94	1.000	0.1	*b*	14.1	21.8	28.8	43.4
VC-MJ	11.6	20.8	28.2	35.6	50.2	6.49	0.94	1.000	0.3	11.8	21.3	28.8	36.4	51.2
VC-MN	8.7	14.1	21.6	27.7	40.3	1.82	0.83	1.000	0.3	8.5	13.7	20.9	26.8	38.9
VC-NM	7.7	13.1	20.5	30.1	41.5	-0.33	0.92	0.988	1.6	7.4	12.6	19.7	29.0	39.9
N	9	10	10	10	10					9	10	10	10	10
Average	8.8	15.6	23.6	31.1	46.0					8.6	15.4	23.2	30.6	45.2
SD	1.1	2.2	2.3	2.5	4.0					1.3	2.4	2.6	2.8	4.2
Min	7.7	13.1	20.5	27.7	40.3					7.4	12.6	19.7	26.8	38.9
%25	8.4	14.2	21.9	29.2	42.3					8.2	14.0	21.8	28.8	42.0
Median	8.5	15.1	23.5	31.0	46.5					8.4	14.7	22.6	30.2	45.9
%75	8.7	16.4	24.4	33.0	49.6					8.5	16.1	24.2	32.2	48.0
Max	11.6	20.8	28.2	35.6	51.4					11.8	21.3	28.8	36.4	51.2
eSD	0.3	1.4	2.2	3.4	5.3					0.3	1.8	2.2	2.7	4.8
CV	3	10	9	11	11					3	12	10	9	10

a) 5% Trichloroacetic acid solution
b) By our err, provided with CS#2 rather than CS#1

Appendix H. Representative “Individualized Report” for RR35

Each participant in RR35 received an “Individualized Report” reflecting their reported results. The following two pages are the “Individualized Report” for participant “VC-MA”.

Vitamin C "Round Robin" 35 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
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
08/24/11	35	HPLC-EC	1.039	242.0	0.568	547.7	0.0	1.03	1.000	0.24
		Mean	1.037	243.0	0.57	548.4			Pooled SEE	0.46
		SD	0.002	1.1	0.00	5.9				
		CV	0.18	0.45	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}
09/27/10	33	S33:3	16.6	16.8	1.0	16.7	0.1	3	16.4	0.5	0.5
02/28/11	34	S34:2	16.3	15.7	1.0	16.0	0.4				
08/24/11	35	S35:1	**16.1**	**17.1**	1.0	16.6	0.7				
02/24/10	32	S32:2	26.2	26.0	1.0	26.1	0.1	3	25.8	0.2	1.1
09/27/10	33	S33:4	26.9	26.4	1.0	26.7	0.4				
08/24/11	35	S35:2	**24.7**	**24.5**	1.0	24.6	0.2				
02/24/10	32	S32:3	33.4	32.8	1.0	33.1	0.5	2	33.2	0.3	0.3
08/24/11	35	S35:3	**33.4**	**33.4**	1.0	33.4	0.1				
02/24/10	32	S32:4	49.0	48.6	1.0	48.8	0.3	3	48.9	0.2	1.0
02/28/11	34	S34:3	47.9	48.1	1.0	48.0	0.1				
08/24/11	35	S35:4	**50.2**	**49.8**	1.0	50.0	0.3				

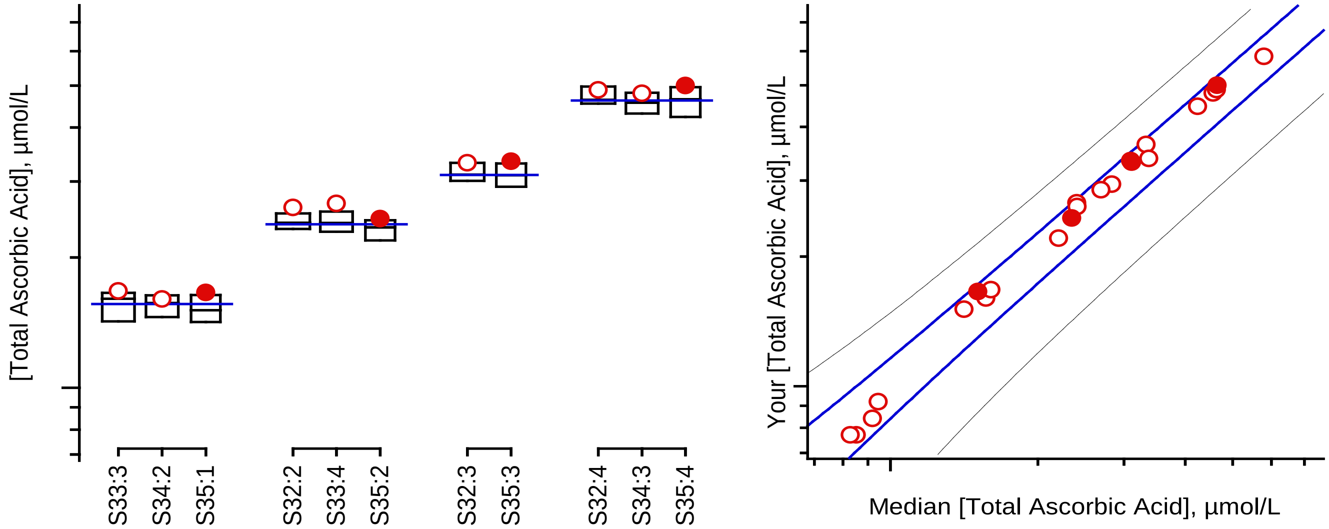
Please check our records against your records. Send corrections and/or updates to...

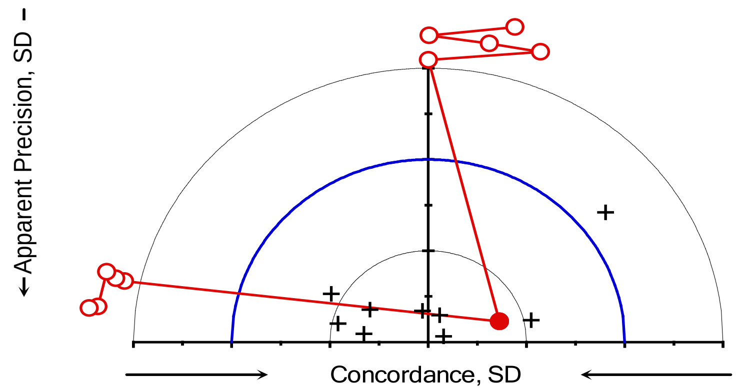
Micronutrients Measurement Quality Assurance Program
National Institute of Standards and Technology
100 Bureau Drive Stop 6392
Gaithersburg, MD 20899-6392 USA

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

Vitamin C "Round Robin" 35 Report: Participant VC-MA

Total Ascorbic Acid, µmol/mL





3rd Quartile (75%)
Median (50%)
1st Quartile (25%)

You, this RR
You, 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.

Sample	Comments
S35:1	VitC #351, previously distributed in RRs 33 and 34
S35:2	VitC #352, previously distributed in RR 32 and 33
S35:3	VitC #353, previously distributed in RR 32
S35:4	VitC #354, previously distributed in RR32 and 34