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NIST Micronutrients Measurement Quality Assurance Program Summer 2007 Comparability Studies

Results for Round Robin LXII
Fat-Soluble Vitamins and Carotenoids in Human Serum
and Round Robin 27 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 Summer 2007 MMQAP measurement comparability improvement studies: 1) Round Robin LXII Fat-Soluble Vitamins and Carotenoids in Human Serum and 2) Round Robin 27 Total Ascorbic Acid in Human Serum. The materials for both studies were shipped to participants in May 2007; participants were requested to provide their measurement results by September 28, 2007.

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 LXII: Fat-Soluble Vitamins and Carotenoids in Human Serum

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin LXII comparability study (hereafter referred to as RR62) 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 2007. 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 RR62 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 27: Vitamin C in Human Serum

Participants in the MMQAP Vitamin C in Human Serum Round Robin 27 comparability study (hereafter referred to as RR27) 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 2007. 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 RR27 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 RR62

The following three items were included in each package shipped to an RR62 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 29, 2007

Dear Colleague:

Enclosed are the samples (Sera 334-338) for the second fat-soluble vitamins and carotenoids in serum round robin study (Round Robin LXII) for the fiscal year (FY) 07 NIST Micronutrients Measurement Quality Assurance Program. You will find one vial of each of four liquid-frozen serum samples and one lyophilized sample for analysis along with a form for reporting your results. When reporting your results, please submit one value for each analyte for a given serum sample. If a value is obtained below your limit of quantification, please indicate this result on the form by using NQ *(Not Quantified)*. Results are due to NIST by **September 28, 2007**. Results received more than two weeks after the due date will not be included in the summary report for this round robin study.

Lyophilized samples should be reconstituted with 1.0 mL of HPLC-grade water or equivalent. We recommend that dissolution be facilitated with 3 to 5 min agitation in an ultrasonic bath or at least 30 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.) Pipette a known volume of serum from the vial for analysis. The final volume of the reconstituted sample is greater than 1.0 mL. **Water should not be added to the liquid-frozen samples (Sera 335-338).**

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); lycopene, 3450 at 472 nm (hexane). At the request of the participants who attended the QA workshop on May 2, also attached is a suggested protocol for preparing calibration solutions.

Please mail or fax your results to:

Micronutrients Measurement Quality Assurance Program
NIST
100 Bureau Drive Stop 8392
Gaithersburg, MD 20899-8392
Fax: (301) 977-0685

If you have questions or comments regarding this study, please call me at (301) 975-3120; e-mail me at jbthomas@nist.gov; or mail/fax queries to the above address.

Sincerely,

Jeanice B Thomas

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

Enclosures

NIST

Participant #: _____ Date: _____

Round Robin LXII: Human Sera

NIST Micronutrients Measurement Quality Assurance Program

Analyte	334	335	336	337	338	Units*
total retinol						
trans-retinol						
didehydroretinol						
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						

Other measurands?						

* we prefer **μg/mL**

Were the liquid-frozen samples (335 to 338) frozen when received? Yes | No

Comments:

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

Please return results **before**
28-Sep-2007

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

Participant #: _____ Date: _____

Fat-Soluble Vitamins Round Robin LXII

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?
#334	Lyophilized	Yes (1 ml H_2O)
#335	Liquid frozen	No
#336	Liquid frozen	No
#337	Liquid frozen	No
#338	Liquid frozen	No

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 8392
Gaithersburg, MD 20899-8392

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

Appendix B. Final Report for RR62

The following three 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-

December 7, 2007

Dear Colleague:

Enclosed is the summary report of the results for round robin LXII (RR62) of the 2007 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.

Data for evaluating laboratory performance in RR62 are provided in text "Score Card" summary, page 6 of the All Lab Report. Laboratory comparability is summarized as follows: 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 in the graphical "target plot" summary, last page of your Individualized Report.

We are sorry that SRM 968c, Fat-Soluble Vitamins, Carotenoids, and Cholesterol in Human Serum is no longer available. If you have concerns regarding your laboratory's performance, we suggest that you analyze an in-house control material. If you do not currently have such a material, please contact us.

Samples for the first 2008 QA interlaboratory exercise were shipped during the week of November 5, 2007. Please contact us immediately if you have not received your scheduled shipment. 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,

Janice B Thomas

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

Cc: Lane C. Sander
David L. Duewer

NIST

The NIST M^2QAP Round Robin LXI (RR62) report consists of:

Page	"All Lab" Report
1-4	A listing of all results and statistics for all analytes.
5	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.

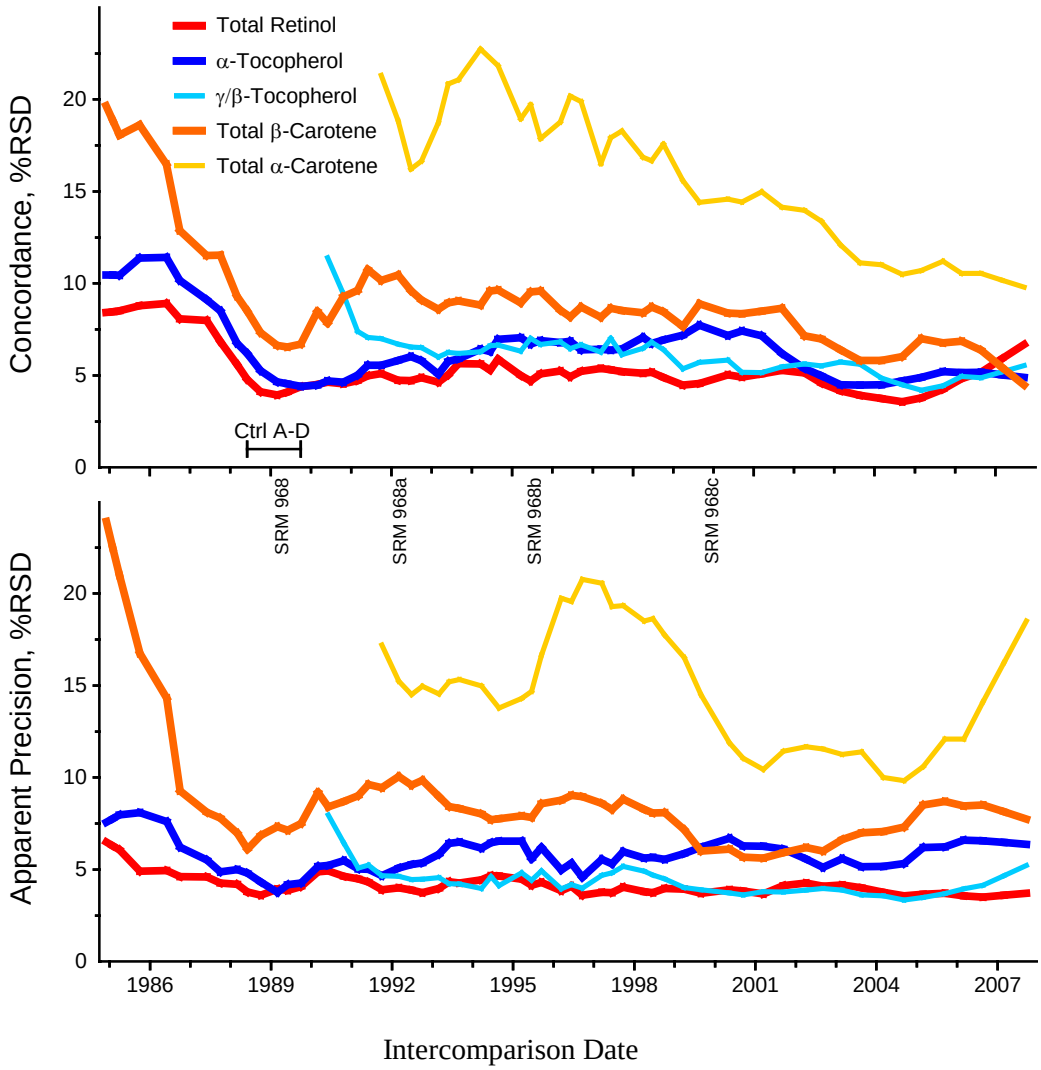
Samples. The five sera below were distributed in RR62.

Serum	Description	Prior Distributions
334	Lyophilized; augmented with retinyl palmitate, γ- and δ-tocopherol, trans-α- and β-carotene, trans-lycopene, and zeaxanthin; prepared in Fall 1997.	#241:RR42-3/98
335	Fresh-frozen, native, multi-donor, prepared in Spring 2005.	#315:RR58-9/05
336	Fresh-frozen, native, multi-donor, prepared in Spring 2005.	#316:RR58-9/05
337	Fresh-frozen, native, single-donor serum prepared in Spring 2006.	#329:RR61-3/07
338	Fresh-frozen, native, single-donor serum prepared in Spring 2006.	#326:RR60-9/06, #331:RR61-3/07

Results

There was no significant change in the median level or variability of any measurand in any of the sera, with the possible exception of zeaxanthin in Serum #334. A number of carotenoids in this material were augmented, using phospholipid slurries.

The following figure displays the "Concordance" (the agreement among participants) and the "Apparent Precision" (the average within-participant agreement among samples) for a number of analytes over the past 23 years. (These performance characteristics are more fully described in Duewer, Kline, Sharpless, and Thomas, NIST Micronutrients Measurement Quality Assurance Program: Characterizing the Measurement Community's Performance over Time, Analytical Chemistry 2000;72:4163-4170.) While concordance for most analytes has improved or remained stable over the past several years (total retinol may, unfortunately, be an exception), it appears that apparent (im)precision for α-carotene is increasing. We hope to present you with a number of new samples having higher levels of α-carotene and other "minor" carotenoids in the coming year.


Total Retinol
α-Tocopherol
γ/β-Tocopherol
Total β-Carotene
Total α-Carotene
Concordance, %RSD
Ctrl A-D
SRM 968
SRM 968a
SRM 968b
SRM 968c
Apparent Precision, %RSD
0
5
10
15
20
1986
1989
1992
1995
1998
2001
2004
2007
Intercomparison Date

Appendix C. "All-Lab Report" for RR62

The following seven 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. The only attributed results are those reported by NIST. The NIST results are not used in the assessment of the consensus summary results of the study.

Round Robin LXII Laboratory Results

(μg/mL)

	Total Retinol					trans-Retinol					Retinyl Palmitate					α-Tocopherol					γ/β-Tocopherol				
Lab	334	335	336	337	338	334	335	336	337	338	334	335	336	337	338	334	335	336	337	338	334	335	336	337	338
FSV-BA	0.465	0.505	0.423	0.540	0.321						0.029	0.041	0.011	0.068	0.067	6.27	9.26	4.19	24.22	8.44	3.50	1.83	1.77	1.12	3.85
FSV-BB	0.519	0.583	0.438	0.615	0.348						0.018	0.047	0.007	0.058	0.013	6.61	9.54	4.19	23.81	8.90	4.01	1.81	1.76	1.02	3.75
FSV-BC	0.428	0.512	0.412	0.538	0.316																				
FSV-BD	0.444	0.466	0.376	0.510	0.319											6.10	8.80	4.50	22.00	9.00					
FSV-BE	0.431	0.487	0.406	0.521	0.305											6.29	9.38	4.10	23.52	8.36	4.01	1.77	1.82	0.87	3.88
FSV-BF	0.430	0.490	0.400	0.510	0.310											6.42	9.41	4.42	24.70	8.70	3.46	1.73	1.84	0.94	3.45
FSV-BG	0.469	0.543	0.411	0.548	0.305						0.038	0.076	0.016	0.055	0.021	6.88	10.38	4.27	25.15	8.64	4.01	1.91	1.73	0.90	3.69
FSV-BH	0.382	0.411	0.336	0.443	0.255											5.79	8.33	3.76	21.43	7.24	3.60	1.63	1.65	0.80	3.38
FSV-BI	0.376	0.485	0.380	0.489	0.291											5.90	9.79	4.35	24.86	8.90	3.89	2.14	2.04	1.14	4.32
FSV-BJ	0.403	0.467	0.368	0.498	0.288						*nq*	0.045	nq	0.045	*nq*	6.50	9.98	4.54	27.69	8.73	3.91	1.79	1.84	0.92	3.77
FSV-BK	0.510	0.530	0.390	0.550	0.290											5.97	9.27	4.12	21.95	8.71					
FSV-BL	0.430	0.430	0.370	0.460	0.260											6.90	8.60	4.30	22.00	8.20					
FSV-BM	0.401	0.459	0.336	0.493	0.310											5.40	8.80	4.60	21.70	8.40					
FSV-BN	0.442	0.454	0.370	0.483	0.262						0.018	0.043	0.011	0.043	0.010	6.06	8.64	4.05	22.09	7.06	3.48	1.55	1.62	0.82	3.15
FSV-BO	0.372	0.424	0.347	0.435	0.275											6.20	9.40	4.30	24.00	8.30	3.40	1.80	1.60	1.20	3.40
FSV-BP	0.467	0.540	0.440	0.542	0.320											7.21	9.82	4.04	21.96	8.53					
FSV-BQ	0.541	0.565	0.408	0.566	0.338											6.92	10.60	4.31	28.10	8.79					
FSV-BR	*≥0.450*	*≥0.470*	*≥0.300*	*≥0.510*	*≥0.280*	0.450	0.470	0.300	0.510	0.280						6.30	9.80	3.60	24.50	9.20					
FSV-BS	*≥0.391*	*≥0.443*	*≥0.329*	*≥0.461*	*≥0.289*	0.391	0.443	0.329	0.461	0.289						7.22	10.56	4.36	26.51	8.72	4.19	2.05	1.90	1.32	4.03
FSV-BT	0.396	0.512	0.378	0.587	0.289											5.73	10.02	4.66	24.00	9.74	3.32	1.79	1.72	0.95	3.92
FSV-BU	0.441	0.463	0.367	0.481	0.280											6.25	9.40	4.28	26.24	8.96	3.52	1.80	1.71	0.90	3.38
FSV-BV	0.344	0.408	0.336	0.419	0.179											5.35	7.64	3.49	19.90	8.06	3.03	1.39	1.38	0.77	3.38
FSV-BW	0.400	0.500	0.380	0.510	0.310						0.012	0.072	0.012	0.050	0.012	6.35	9.45	3.97	23.80	8.56	5.30	2.46	2.40	1.22	5.15
FSV-BX	*≥0.413*	*≥0.482*	*≥0.367*	*≥0.493*	*≥0.286*	0.413	0.482	0.367	0.493	0.286						6.06	9.35	3.90	23.71	8.22	3.37	1.80	1.45	1.08	3.58
FSV-CC	0.460	0.520	0.370	0.520	0.310	0.440	0.500	0.370	0.500	0.310						6.95	10.37	4.72	24.69	9.31					
FSV-CE	0.500	0.620	0.470	0.650	0.370											5.72	9.55	3.61	25.11	8.71					
FSV-CF	0.531	0.570	0.436	0.595	0.349											6.70	9.50	4.80	24.80	9.40					
FSV-CG	0.596	0.544	0.411	0.571	0.333											6.41	9.44	4.09	25.00	8.46	4.06	1.94	1.81	1.02	3.90
FSV-CI	0.453	0.506	0.400	0.513	0.307						0.019	0.044	0.008	0.045	0.017	6.31	9.24	3.95	23.66	8.26	3.78	1.74	1.71	0.79	3.61
FSV-CS	0.583	0.581	0.469	0.549	0.348											6.40	9.12	3.98	22.70	7.87	4.07	1.90	1.79	1.05	3.60
FSV-CW	0.463	0.622	0.484	0.618	0.303						0.023	0.059	0.011	0.067	0.017	8.57	13.66	5.32	37.18	11.36	5.04	2.37	2.34	1.18	4.92
FSV-DD	*≥0.420*	*≥0.520*	*≥0.410*	*≥0.550*	*≥0.340*	0.42	0.52	0.41	0.55	0.34															
FSV-DF	0.459	0.544	0.419	0.462	0.331																				
FSV-DI	0.414	0.494	0.384	0.532	0.305						0.014	0.050	0.008	0.039	0.009	6.25	9.10	4.08	23.60	8.40	3.75	1.74	1.70	0.88	3.67
FSV-DV	*≥0.790*	*≥0.640*	*≥0.491*	*≥0.636*	*≥0.370*	0.790	0.640	0.491	0.636	0.370						7.30	10.30	4.50	25.60	8.80					
FSV-EE	0.369	0.456	0.316	0.415	0.282											5.50	8.50	3.90	20.40	7.60					
N	31	31	31	31	31	6	6	6	6	6	8	9	8	9	8	33	33	33	33	33	21	21	21	21	21
Min	0.344	0.408	0.316	0.415	0.179	0.391	0.443	0.300	0.461	0.280	0.012	0.041	0.007	0.039	0.009	5.35	7.64	3.49	19.90	7.06	3.03	1.39	1.38	0.77	3.15
Median	0.442	0.505	0.390	0.520	0.307	0.430	0.491	0.368	0.505	0.300	0.019	0.047	0.011	0.050	0.015	6.30	9.41	4.19	24.00	8.64	3.78	1.80	1.76	0.95	3.69
Max	0.596	0.622	0.484	0.650	0.370	0.790	0.640	0.491	0.636	0.370	0.038	0.076	0.016	0.068	0.067	8.57	13.66	5.32	37.18	11.36	5.30	2.46	2.40	1.32	5.15
SD	0.049	0.059	0.034	0.047	0.024						0.006	0.011	0.002	0.010	0.005	0.48	0.52	0.32	2.16	0.44	0.39	0.12	0.10	0.18	0.33
CV	11	12	9	9	8						30	24	22	19	32	8	6	8	9	5	10	7	6	19	9
NIST	0.437	0.491	0.399	0.517	0.307											6.49	9.46	4.16	23.72	8.51	3.73	1.79	1.78	0.92	3.68
N_{past}	47	35	36	32	32	0	6	6	5	5	7	12	8	10	9	46	36	36	33	35	21	23	23	21	23
$Median_{past}$	0.437	0.490	0.389	0.520	0.306		0.488	0.402	0.510	0.308	0.054	0.053	0.012	0.057	0.025	6.42	9.45	4.18	23.69	8.41	3.65	1.79	1.73	0.90	3.67
SD_{past}	0.042	0.025	0.023	0.042	0.026						0.015	0.006	0.005	0.026	0.023	0.53	0.76	0.30	1.56	0.55	0.53	0.14	0.09	0.15	0.32
NAV	0.441	0.500	0.384	0.513	0.307	0.430	0.491	0.368	0.505	0.300	0.018	0.045	0.010	0.045	0.013	6.29	9.40	4.19	23.81	8.56	3.75	1.80	1.73	0.94	3.67
NAU	0.050	0.060	0.032	0.049	0.026	0.035	0.040	0.030	0.041	0.025	0.011	0.015	0.010	0.015	0.011	0.54	0.74	0.44	2.12	0.68	0.41	0.19	0.19	0.16	0.36

C2

Round Robin LXII Laboratory Results

(μg/mL)

	δ-Tocopherol					Total β-Carotene					trans-β-Carotene					Total cis-β-Carotene				
Lab	334	335	336	337	338	334	335	336	337	338	334	335	336	337	338	334	335	336	337	338
FSV-BA	0.119	0.114	0.103	0.161	0.093	0.160	0.287	0.042	0.580	0.152	0.152	0.271	0.038	0.545	0.144	0.008	0.016	0.004	0.035	0.008
FSV-BB	0.166	0.098	0.096	0.120	0.071	0.152	0.262	0.036	0.528	0.118	0.145	0.245	0.033	0.495	0.113	0.008	0.017	0.003	0.033	0.005
FSV-BC																				
FSV-BD																				
FSV-BE						0.135	0.271	0.032	0.506	0.155										
FSV-BF																				
FSV-BG						0.158	0.233	0.043	0.593	0.148										
FSV-BH						0.150	0.259	0.033	0.583	0.129	0.150	0.259	0.033	0.550	0.129	*nq*	*nq*	*nq*	0.033	*nq*
FSV-BI						0.129	0.241	0.029	0.474	0.126										
FSV-BJ						0.104	0.257	0.026	0.566	0.144										
FSV-BK																				
FSV-BL																				
FSV-BM																				
FSV-BN						0.158	0.237	0.034	0.496	0.116	0.147	0.223	0.030	0.456	0.111	0.010	0.013	0.003	0.038	0.004
FSV-BO						0.202	0.292	0.045	0.537	0.173	0.166	0.238	0.045	0.450	0.128	0.037	0.054	*nd*	0.086	0.045
FSV-BP						0.188	0.293	*nd*	0.562	0.150										
FSV-BQ																				
FSV-BR																				
FSV-BS						*≥0.169*	*≥0.286*	*≥0.030*	*≥0.542*	*≥0.147*	0.169	0.286	0.030	0.542	0.147					
FSV-BT						0.189	0.295	0.045	0.519	0.137	0.176	0.274	0.041	0.480	0.127	0.012	0.021	0.004	0.039	0.010
FSV-BU						0.148	0.285	0.038	0.555	0.130										
FSV-BV						0.139	0.265	0.039	0.555	0.170										
FSV-BW						0.145	0.320	0.038	0.590	0.160										
FSV-BX						*≥0.165*	*≥0.254*	*≥0.039*	*≥0.529*	*≥0.135*	0.165	0.254	0.039	0.529	0.135					
FSV-CC																				
FSV-CE						0.140	0.290	0.040	0.620	0.160										
FSV-CF																				
FSV-CG	0.113	0.129	*nd*	*nd*	*nd*	0.164	0.287	0.039	0.574	0.145	0.155	0.267	0.036	0.534	0.136	0.009	0.020	*nd*	0.040	0.009
FSV-CI						*≥0.185*	*≥0.303*	*≥0.050*	*≥0.558*	*≥0.108*	0.185	0.303	0.050	0.558	0.108					
FSV-CS						0.222	0.347	0.050	0.604	0.139										
FSV-CW	0.177	0.128	0.122	0.037	0.095	*≥0.176*	*≥0.356*	*≥0.005*	*≥0.721*	*≥0.103*	0.176	0.356	0.005	0.721	0.103					
FSV-DD																				
FSV-DF																				
FSV-DI	0.140	0.105	0.096	0.042	0.072	0.142	0.294	0.041	0.505	0.133										
FSV-DV																				
FSV-EE																				
N	5	5	4	3	4	18	18	17	18	18	11	11	11	11	11	6	6	4	7	6
Min	0.113	0.098	0.096	0.037	0.071	0.104	0.233	0.026	0.474	0.116	0.145	0.223	0.005	0.450	0.103	0.008	0.013	0.003	0.033	0.004
Median	0.140	0.114	0.100	0.081	0.083	0.151	0.286	0.039	0.559	0.145	0.165	0.267	0.036	0.534	0.128	0.010	0.019	0.004	0.038	0.009
Max	0.177	0.129	0.122	0.161	0.095	0.222	0.347	0.050	0.620	0.173	0.185	0.356	0.050	0.721	0.147	0.037	0.054	0.004	0.086	0.045
SD	0.035	0.017	0.009	0.066	0.016	0.017	0.024	0.006	0.045	0.017	0.016	0.022	0.006	0.045	0.018	0.002	0.003	0.001	0.004	0.003
CV	25	15	9	82	20	11	9	15	8	12	10	8	18	8	14	25	18	21	11	35
NIST						0.166	0.280	0.045	0.553	0.148										
N_{past}	0	7	7	6	6	30	25	24	19	23	9	12	12	11	10	4	8	7	7	6
$Median_{past}$		0.115	0.100	0.058	0.088	0.171	0.285	0.042	0.548	0.146	0.174	0.277	0.039	0.507	0.133	0.008	0.020	0.003	0.033	0.010
SD_{past}		0.006	0.014	0.094	0.092	0.033	0.033	0.010	0.046	0.019	0.013	0.024	0.005	0.034	0.014		0.004	0.003	0.008	0.005
NAV	0.119	0.105	0.096	0.120	0.072	0.150	0.285	0.039	0.555	0.144	0.160	0.263	0.037	0.532	0.129	0.010	0.019	0.004	0.038	0.009
NAU	0.026	0.024				0.024	0.042	0.010	0.077	0.023	0.018	0.029	0.008	0.056	0.015	0.004	0.006		0.014	0.004

C3

Round Robin LXII Laboratory Results

(μg/mL)

	Total α-Carotene					Total Lycopene					trans-Lycopene					Total β-Cryptoxanthin					Total a-Cryptoxanthin				
Lab	334	335	336	337	338	334	335	336	337	338	334	335	336	337	338	334	335	336	337	338	334	335	336	337	338
FSV-BA	0.231	0.057	0.007	0.308	0.015	0.484	0.229	0.143	0.255	0.576	0.329	0.141	0.077	0.147	0.349	0.078	0.063	0.046	0.127	0.129	0.036	0.033	0.018	0.050	0.053
FSV-BB	0.236	0.050	0.004	0.297	0.011	0.479	0.216	0.136	0.249	0.485	0.261	0.114	0.070	0.109	0.248	0.060	0.045	0.045	0.106	0.096	0.020	0.032	0.020	0.056	0.052
FSV-BC																									
FSV-BD																									
FSV-BE																									
FSV-BF																									
FSV-BG	0.211	0.042	0.012	0.296	0.016	0.494	0.199	0.118	0.268	0.520	0.286	0.096	0.053	0.117	0.257	0.072	0.045	0.033	0.098	0.103					
FSV-BH	0.401	0.054	*nq*	0.326	*nq*	0.564	0.247	0.143	0.299	0.587						0.061	0.044	0.035	0.100	0.109					
FSV-BI	0.306	0.055	0.005	0.289	0.013	0.365	0.198	0.113	0.215	0.478						0.052	0.046	0.033	0.099	0.111					
FSV-BJ	0.275	0.060	nq	0.359	0.012	0.572	0.290	0.186	0.341	0.693						0.047	0.037	0.031	0.086	0.084					
FSV-BK																									
FSV-BL																									
FSV-BM																									
FSV-BN	0.350	0.048	0.006	0.294	0.012	0.477	0.213	0.129	0.262	0.501	0.315	0.117	0.068	0.121	0.266	0.050	0.037	0.026	0.089	0.094	0.012	0.002	*nd*	0.006	0.024
FSV-BO	0.384	0.053	0.006	0.292	0.014	0.592	0.255	0.184	0.263	0.615						0.055	0.043	0.035	0.090	0.105					
FSV-BP	0.280	0.053	*nd*	0.282	*nd*	0.405	0.598	0.521	0.336	0.586						0.070	0.040	*nd*	0.079	0.048					
FSV-BQ																									
FSV-BR																									
FSV-BS	0.431	0.071	0.007	0.355	0.036	0.401	0.191	0.111	0.211	0.462						0.044	0.036	0.026	0.084	0.095					
FSV-BT	0.300	0.055	0.008	0.245	0.021	0.451	0.255	0.161	0.260	0.535	0.311	0.141	0.081	0.095	0.289	0.059	0.048	0.036	0.094	0.103	0.023	0.017	0.010	0.024	0.035
FSV-BU	0.367	0.069	0.020	0.296	0.026	0.554	0.237	0.152	0.250	0.554						0.064	0.045	0.039	0.104	0.116					
FSV-BV	0.396	0.054	0.005	0.344	0.018	0.533	0.233	0.150	0.292	0.699						0.040	0.032	0.025	0.083	0.097					
FSV-BW	0.200	0.037	0.006	0.170	0.024	0.640	0.270	0.161	0.265	0.700						0.050	0.045	0.036	0.094	0.109					
FSV-BX	0.327	0.048	0.000	0.275	0.010						0.258	0.101	0.057	0.088	0.243	0.070	0.044	0.035	0.099	0.113					
FSV-CC																									
FSV-CE																									
FSV-CF																									
FSV-CG	0.447	0.071	*nd*	0.386	0.019	0.563	0.262	0.153	0.306	0.604	0.358	0.138	0.076	0.125	0.322	0.094	0.068	0.049	0.140	0.153					
FSV-CI	0.237	0.075	0.022	0.289	0.036																				
FSV-CS	0.375	0.070	0.007	0.343	0.010	0.675	0.343	0.199	0.346	0.680						0.068	0.051	0.037	0.086	0.093					
FSV-CW	0.472	0.070	0.004	0.391	0.006						0.319	0.108	0.048	0.086	0.267	0.068	0.050	0.035	0.123	0.119					
FSV-DD																									
FSV-DF																									
FSV-DI	0.270	0.060	0.015	0.245	0.045	0.505	0.265	0.180	0.300	0.510															
FSV-DV																									
FSV-EE																									
N	20	20	16	20	18	17	17	17	17	17	8	8	8	8	8	18	18	17	18	18	4	4	3	4	4
Min	0.200	0.037	0.000	0.170	0.006	0.365	0.191	0.111	0.211	0.462	0.258	0.096	0.048	0.086	0.243	0.040	0.032	0.025	0.079	0.048	0.012	0.002	0.010	0.006	0.024
Median	0.316	0.055	0.007	0.296	0.016	0.505	0.247	0.152	0.265	0.576	0.313	0.116	0.069	0.113	0.267	0.061	0.045	0.035	0.096	0.104	0.022	0.025	0.018	0.037	0.044
Max	0.472	0.075	0.022	0.391	0.045	0.675	0.598	0.521	0.346	0.700	0.358	0.141	0.081	0.147	0.349	0.094	0.068	0.049	0.140	0.153	0.036	0.033	0.020	0.056	0.053
SD	0.093	0.013	0.003	0.042	0.008	0.065	0.036	0.033	0.033	0.078	0.031	0.024	0.015	0.021	0.032	0.014	0.005	0.003	0.012	0.013	0.006	0.014		0.024	0.015
CV	29	23	44	14	54	13	15	21	13	13	10	21	21	19	12	23	11	8	13	12	28	58		64	34
NIST	0.281	0.054	*nq*	0.282	0.019											0.063	0.049	0.036	0.092	0.117					
N_{past}	25	23	18	19	19	26	23	23	17	20	9	9	9	7	7	25	24	24	17	20	4	6	6	4	4
$Median_{past}$	0.308	0.057	0.008	0.316	0.016	0.474	0.238	0.150	0.269	0.585	0.417	0.122	0.069	0.100	0.298	0.060	0.045	0.035	0.097	0.108	0.033	0.015	0.009	0.021	0.037
SD_{past}	0.094	0.008	0.004	0.044	0.005	0.085	0.038	0.025	0.024	0.063	0.080	0.012	0.007	0.016	0.049	0.014	0.009	0.005	0.017	0.027		0.001	0.002	0.002	0.004
NAV	0.311	0.055	0.007	0.295	0.016	0.505	0.247	0.152	0.265	0.576	0.299	0.116	0.069	0.113	0.262	0.060	0.045	0.035	0.094	0.103	0.022	0.025	0.018	0.037	0.044
NAU	0.086	0.017	0.004	0.075	0.008	0.104	0.058	0.039	0.062	0.116	0.054	0.029	0.015	0.021	0.047	0.014	0.011	0.009	0.021	0.023					

C4

Round Robin LXII Laboratory Results

(μg/mL)

	Total Lutein					Total Zeaxanthin					Total Lutein&Zeaxanthin					Coenzyme Q10				
Lab	334	335	336	337	338	334	335	336	337	338	334	335	336	337	338	334	335	336	337	338
FSV-BA											0.117	0.085	0.039	0.174	0.214					
FSV-BB	0.050	0.069	0.018	0.172	0.161	0.094	0.034	0.019	0.042	0.109	0.144	0.103	0.037	0.214	0.270					
FSV-BC																				
FSV-BD																				
FSV-BE																				
FSV-BF																				
FSV-BG	0.072	0.055	0.030	0.153	0.155	0.035	0.017	0.016	0.029	0.056	0.088	0.061	0.034	0.165	0.178					
FSV-BH	0.037	0.046	0.013	0.115	0.095	0.101	0.016	0.012	0.023	0.072	0.138	0.062	0.025	0.138	0.167					
FSV-BI	0.043	0.054	0.021	0.122	0.122	0.123	0.025	0.016	0.036	0.072	*0.166*	*0.079*	*0.037*	*0.158*	*0.194*					
FSV-BJ	0.063	0.057	0.025	0.135	0.130											0.56	0.62	0.52	0.84	1.04
FSV-BK																				
FSV-BL																				
FSV-BM																				
FSV-BN	0.161	0.048	0.004	0.138	0.128	0.017	0.021	0.011	0.031	0.059	0.183	0.075	0.020	0.177	0.200					
FSV-BO	0.044	0.060	0.022	0.147	0.145	0.119	0.018	0.011	0.019	0.062	0.163	0.078	0.033	0.166	0.207					
FSV-BP											0.170	0.068	0.066	0.102	0.089					
FSV-BQ																				
FSV-BR																				
FSV-BS											0.117	0.078	0.007	0.200	0.278					
FSV-BT											0.116	0.096	0.049	0.179	0.199					
FSV-BU											0.138	0.085	0.039	0.189	0.231					
FSV-BV											0.133	0.080	0.034	0.177	0.252					
FSV-BW											0.160	0.086	0.025	0.170	0.220	0.31	0.44	0.46	0.48	1.29
FSV-BX	0.061	0.080	0.024	0.180	0.187	0.110	0.010	0.007	0.022	0.066	*0.171*	*0.090*	*0.031*	*0.202*	*0.252*					
FSV-CC																				
FSV-CE																				
FSV-CF																				
FSV-CG											0.199	0.125	0.058	0.256	0.301					
FSV-CI	0.076	0.089	0.038	0.182	0.189	0.093	0.022	0.022	0.028	0.054	*0.169*	*0.111*	*0.060*	*0.210*	*0.243*	0.60	0.64	0.62	0.74	1.20
FSV-CS	0.053	0.065	0.024	0.025	0.058	0.116	0.020	0.015	0.128	0.132	*0.169*	*0.085*	*0.039*	*0.153*	*0.190*					
FSV-CW	0.063	0.096	0.023	0.222	0.206	0.094	0.022	0.016	0.027	0.057	0.157	0.118	0.049	0.249	0.263	0.14	0.47	0.40	0.41	1.03
FSV-DD																				
FSV-DF																				
FSV-DI	0.170	0.074	0.028	0.141	0.165											0.64	0.67	0.63	0.77	1.02
FSV-DV																				
FSV-EE																0.51	0.61	0.65	0.75	1.22
N	12	12	12	12	12	10	10	10	10	10	18	18	18	18	18	6	6	6	6	6
Min	0.037	0.046	0.004	0.025	0.058	0.017	0.010	0.007	0.019	0.054	0.088	0.061	0.007	0.102	0.089	0.14	0.44	0.40	0.41	1.02
Median	0.062	0.062	0.023	0.144	0.150	0.098	0.021	0.016	0.029	0.064	0.159	0.085	0.037	0.177	0.217	0.54	0.61	0.57	0.74	1.12
Max	0.170	0.096	0.038	0.222	0.206	0.123	0.034	0.022	0.128	0.132	0.199	0.125	0.066	0.256	0.301	0.64	0.67	0.65	0.84	1.29
SD	0.018	0.015	0.004	0.031	0.033	0.016	0.003	0.003	0.008	0.011	0.026	0.012	0.011	0.027	0.042	0.17	0.10	0.11	0.16	0.13
CV	29	25	17	22	22	16	17	21	28	17	16	14	30	15	19	32	16	20	22	12
NIST	0.061	0.061	0.027	0.151	0.144	0.094	0.017	0.012	0.022	0.061	*0.155*	*0.078*	*0.039*	*0.173*	*0.205*					
N_{past}	13	16	14	13	14	11	12	13	11	11	24	23	22	17	20	0	6	6	7	6
$Median_{past}$	0.047	0.064	0.025	0.157	0.146	0.112	0.023	0.017	0.026	0.062	0.158	0.087	0.039	0.187	0.211		0.62	0.55	0.70	1.14
SD_{past}	0.014	0.008	0.007	0.016	0.036	0.031	0.006	0.004	0.004	0.018	0.039	0.006	0.010	0.020	0.055		0.10	0.09	0.05	0.09
NAV	0.057	0.062	0.023	0.144	0.141	0.098	0.019	0.014	0.029	0.064	0.152	0.082	0.036	0.176	0.210	0.54	0.61	0.57	0.74	1.12
NAU	0.022	0.013	0.005	0.026	0.026	0.033	0.006	0.005	0.009	0.017	0.036	0.017	0.008	0.037	0.044	0.17	0.10	0.11	0.16	0.13

C5

Round Robin LXII Laboratory Results

(µg/mL)

	Ubiquinol					Ubiquinone					Phylloquinone (K1) x1000					25-hydroxyvitamin D x1000				
Lab	334	335	336	337	338	334	335	336	337	338	334	335	336	337	338	334	335	336	337	338
FSV-BA																				
FSV-BB																0.020	0.011	*nq*	0.018	0.011
FSV-BC																				
FSV-BD																				
FSV-BE											0.259	0.853	0.181	1.698	0.645					
FSV-BF																				
FSV-BG																				
FSV-BH																				
FSV-BI																				
FSV-BJ																				
FSV-BK																				
FSV-BL																				
FSV-BM																				
FSV-BN																0.005	0.016	0.006	0.023	0.009
FSV-BO																				
FSV-BP																				
FSV-BQ																				
FSV-BR																				
FSV-BS																				
FSV-BT																				
FSV-BU																				
FSV-BV																				
FSV-BW	0.06	0.25	0.27	0.19	1.12	0.25	0.20	0.19	0.29	0.17										
FSV-BX																				
FSV-CC																				
FSV-CE																				
FSV-CF																				
FSV-CG																				
FSV-CI											0.164	0.667	0.130	1.272	0.523					
FSV-CS																				
FSV-CW																				
FSV-DD																				
FSV-DF																				
FSV-DI	0.13	0.64	0.93	0.05	1.62	0.48	0.24	0.05	0.65	0.16	0.270	0.600	0.230	0.940	0.520					
FSV-DV																				
FSV-EE																				
N	2	2	2	2	2	2	2	2	2	2	3	3	3	3	3	2	2	1	2	2
Min	0.059	0.247	0.271	0.045	1.120	0.249	0.197	0.050	0.290	0.160	0.16	0.60	0.13	0.94	0.52	0.005	0.011		0.018	0.009
Median	0.096	0.444	0.601	0.118	1.370	0.362	0.218	0.119	0.470	0.164	0.26	0.67	0.18	1.27	0.52	0.013	0.014	0.006	0.020	0.010
Max	0.133	0.640	0.930	0.191	1.620	0.475	0.239	0.187	0.650	0.168	0.27	0.85	0.23	1.70	0.65	0.020	0.016		0.023	0.011
SD																				
CV																				
NIST																				
N_{past}	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	4	7
$Median_{past}$																			0.015	0.008
SD_{past}																			0.001	0.001
NAV											0.259	0.667	0.181	1.272	0.523					
NAU																				

C6

Round Robin LXII Laboratory Results

Term	Legend
N	Number of quantitative values reported for this analyte
Min	Minimum quantitative value reported
Median	Median quantitative value reported
Max	Maximum quantitative value reported
SD	Standard deviation for results: (Median Absolute Difference from the median)/0.674
CV	Coefficient of Variation for 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)
NAV	NIST Assigned Value: Median, when sufficient data are available
NAU	NIST Assigned Uncertainty: Max(0.05*NAV, SD, eSD). 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
≤x	Concentration at or below the limit of quantification, x
italics	Not explictly reported but calculated by NIST from reported values

Comparability Summary

Lab	TR	aT	g/bT	bC	tbC	aC	TLy	TbX	TLu	TZ	L&Z
0???											
FSV-BA	1	1	1	1	1	1	1	2			1
FSV-BB	2	1	1	1	1	1	1	1	1	2	1
FSV-BC	1										
FSV-BD	1	1									
FSV-BE	1	1	1	1							
FSV-BF	1	1	1								
FSV-BG	1	1	1	1		2	1	1	1	1	2
FSV-BH	2	2	1	1	1	1	1	1	2	1	2
FSV-BI	1	1	2	1		1	1	1	1	1	1
FSV-BJ	1	1	1	2		1	1	1	1		
FSV-BK	1	1									
FSV-BL	2	1									
FSV-BM	1	2									
FSV-BN	1	2	2	1	2	1	1	1	3	2	2
FSV-BO	2	1	2	2	2	1	1	1	1	1	1
FSV-BP	1	2		1		1	4	2			3
FSV-BQ	2	2									
FSV-BR	2	1									
FSV-BS	2	2	2	1	1	2	1	1			3
FSV-BT	1	2	1	1	1	1	1	1			2
FSV-BU	1	1	1	1		2	1	1			1
FSV-BV	3	2	2	1		1	1	2			1
FSV-BW	1	1	4	1		2	1	1			1
FSV-BX	1	1	2	1	1	1		1	2	2	1
FSV-CC	1	2									
FSV-CE	3	1		1							
FSV-CF	2	1									
FSV-CG	2	1	1	1	1	2	1	3			3
FSV-CI	1	1	1	2	2	3			2	1	2
FSV-CS	2	1	1	2		1	2	1	3	4	1
FSV-CW	2	4	3	3	4	2		1	3	1	2
FSV-DD	1										
FSV-DF	1										
FSV-DI	1	1	1	1		3	1		3		
FSV-DV	4	2									
FSV-EE	2	2									
NIST	1	1	1	1		1		1	1	1	1
n	37	34	22	23	11	21	17	19	13	11	19

	TR	aT	g/bT	bC	tbC	aC	TLy	TbX	TLu	TZ	L&Z
% 1	59	65	64	78	64	62	88	79	46	64	53
% 2	32	32	27	17	27	29	6	16	23	27	32
% 3	5	0	5	4	0	10	0	5	31	0	16
% 4	3	3	5	0	9	0	6	0	0	9	0

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) of summarizes your measurement performance for a given measurand, relative to the consensus medians. CS is the average distance, in standard deviation units, that your measurement performance characteristics are from the consensus performance. CS is calculated when the number of quantitative values you reported for a measurand, N_{you}, is at least two and the measurand has been reported by 10 or more participants.

$$CS = MIN(4, INT(1 + \sqrt{C^2 + AP^2}))$$

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

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

NAU = NIST Assigned Uncertainty, our estimate of the overall measurement standard deviation for each sample. The estimate includes serum heterogeneity, analytical repeatability, and among-participant reproducibility variance components.

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 RR62

Each participant in RR62 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 RR62:

- Total Retinol
- *trans*-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 13 pages are the "Individualized Report" for the analytes evaluated by participant FSV-BA.

Individualized Round Robin LXII Report: FSV-BA

Summary

	Serum 334			Serum 335			Serum 336			Serum 337			Serum 338		
Analyte	You	NAV	n	You	NAV	n	You	NAV	n	You	NAV	n	You	NAV	n
Total Retinol	0.465	0.441	31	0.505	0.500	31	0.423	0.384	31	0.540	0.513	31	0.321	0.307	31
Retinyl Palmitate	0.03	0.02	8	0.0	0.0	9	0.0	0.0	8	0.07	0.05	9	0.07	0.01	8
α-Tocopherol	6.27	6.29	33	9.26	9.40	33	4.19	4.19	33	24.22	23.81	33	8.44	8.56	33
γ/β-Tocopherol	3.501	3.750	21	1.829	1.795	21	1.774	1.730	21	1.119	0.940	21	3.848	3.670	21
δ-Tocopherol	0.119	0.119	5	0.114	0.105	5	0.103	0.096	4	0.161	0.120	4	0.093	0.072	4
Total β-Carotene	0.160	0.150	18	0.287	0.285	18	0.042	0.039	17	0.580	0.555	18	0.152	0.144	18
trans-β-Carotene	0.152	0.160	11	0.271	0.263	11	0.038	0.037	11	0.545	0.532	11	0.144	0.129	11
Total cis-β-Carotene	0.008	0.010	6	0.016	0.019	6	0.004	0.004	4	0.035	0.038	7	0.008	0.009	6
Total α-Carotene	0.231	0.311	20	0.057	0.055	20	0.007	0.007	16	0.308	0.295	20	0.015	0.016	18
Total Lycopene	0.484	0.505	17	0.229	0.247	17	0.143	0.152	17	0.255	0.265	17	0.576	0.576	17
trans-Lycopene	0.329	0.299	8	0.141	0.116	8	0.077	0.069	8	0.147	0.113	8	0.349	0.262	8
Total β-Cryptoxanthin	0.078	0.060	18	0.063	0.045	18	0.046	0.035	17	0.127	0.094	18	0.129	0.103	18
Total α-Cryptoxanthin	0.036	0.022	4	0.033	0.025	4	0.018	0.018	3	0.050	0.037	4	0.053	0.044	4
Total Lutein&Zeaxanthin	0.117	0.152	18	0.085	0.082	18	0.039	0.036	18	0.174	0.176	18	0.214	0.210	18

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

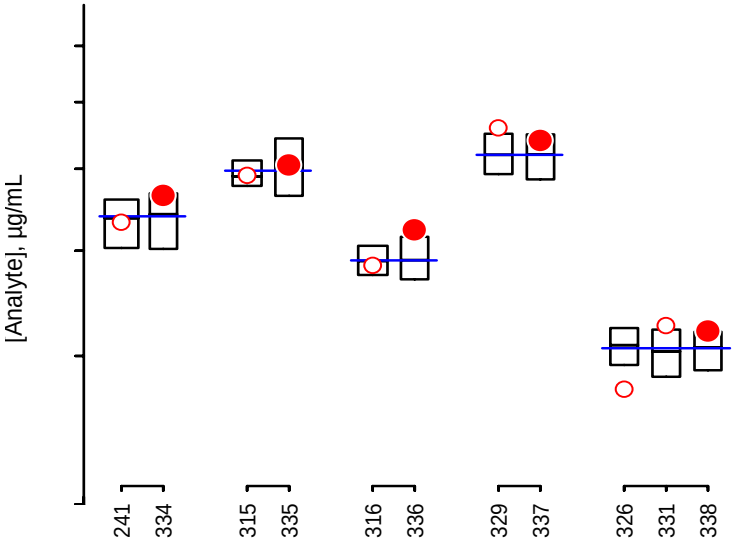
Please check our records against your records. Send corrections and/or updates to...

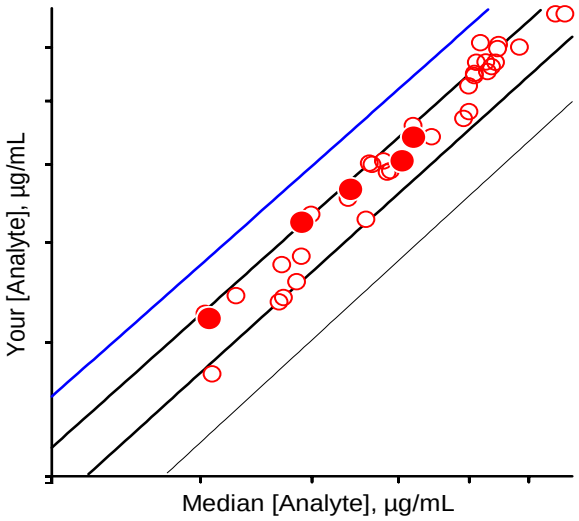
Micronutrients Measurement Quality Assurance Program
National Institute of Standards and Technology
100 Bureau Drive Stop 8392
Gaithersburg, MD 20899-8392 USA

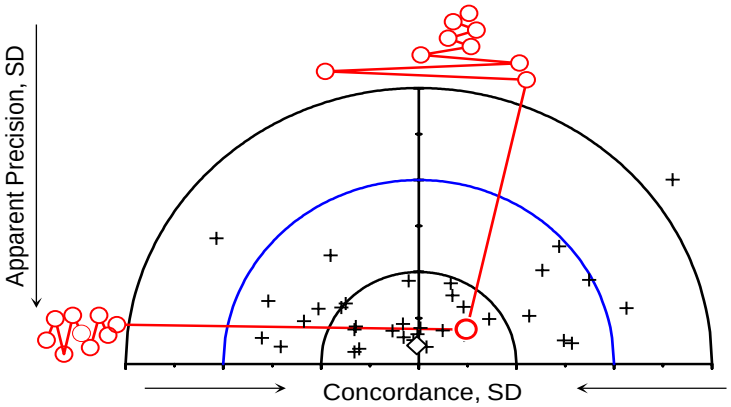
Tel: (301) 975-3935
Fax: (301) 977-0685
Email: david.duewer@nist.gov

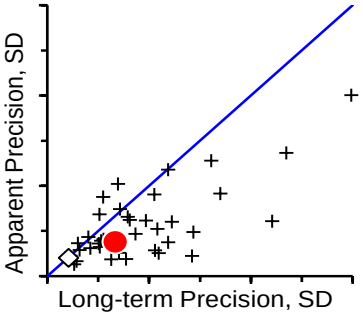
Individualized RR LXII Report: FSV-BA

Total Retinol, µg/mL









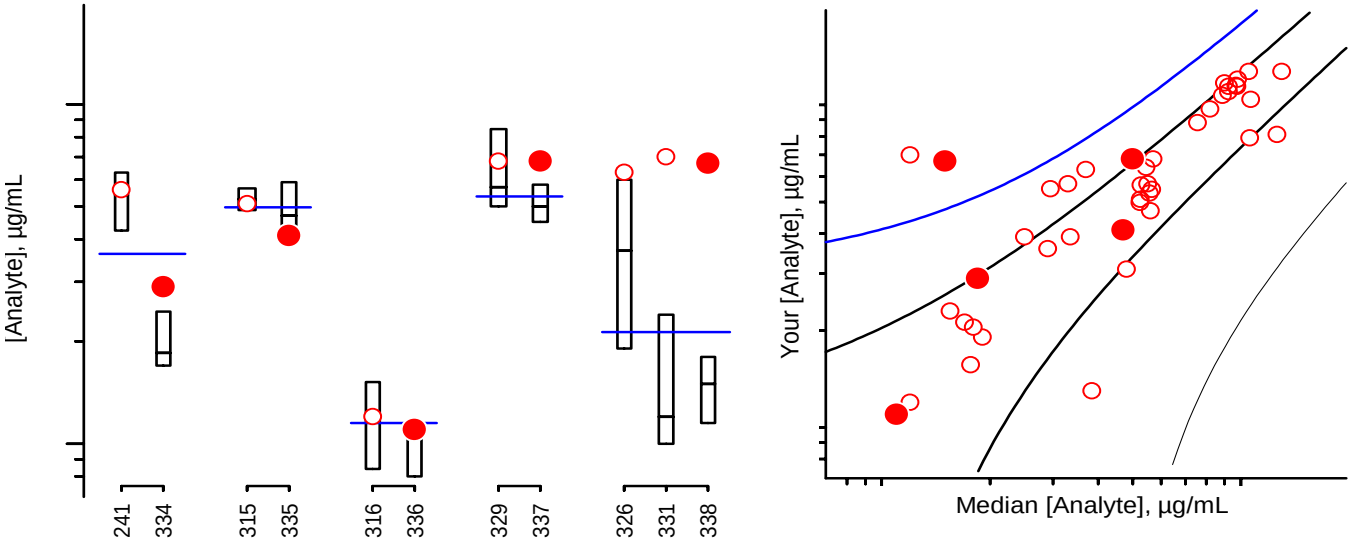
3rd Quartile (75%)
Median (50%)
1st Quartile (25%)
You, this RR
You, past RRs
Expectation
You, ≥x, this RR
You, ≥x, past RRs
Others, this RR

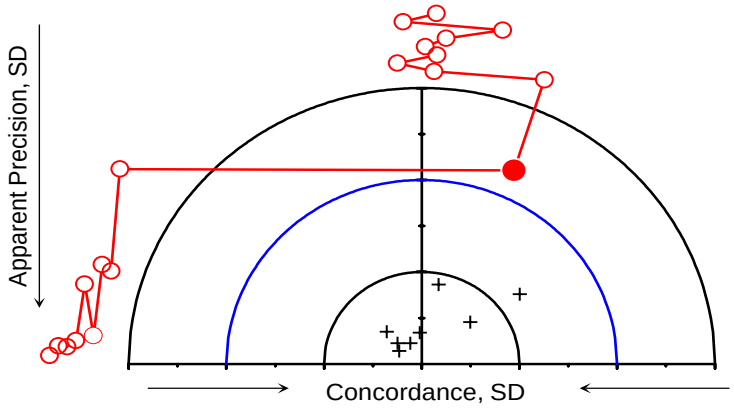
For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

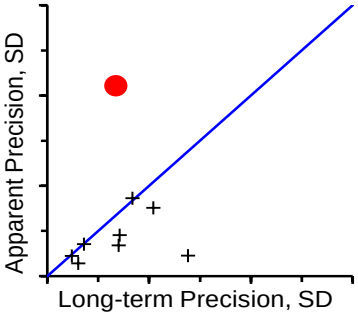
Serum	History	Comments
#334	Lyophilized, 42:#241	Augmented (RP,gT,dT,bC,aC,Z,Ly)
#335	Fresh frozen, 58:#315	Native, multi-source
#336	Fresh frozen, 58:#316	Native, multi-source
#337	Fresh frozen, 61:#329	Native, single-source
#338	Fresh frozen: 60:#326, 61:#331	Native, single-source

Individualized RR LXII 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

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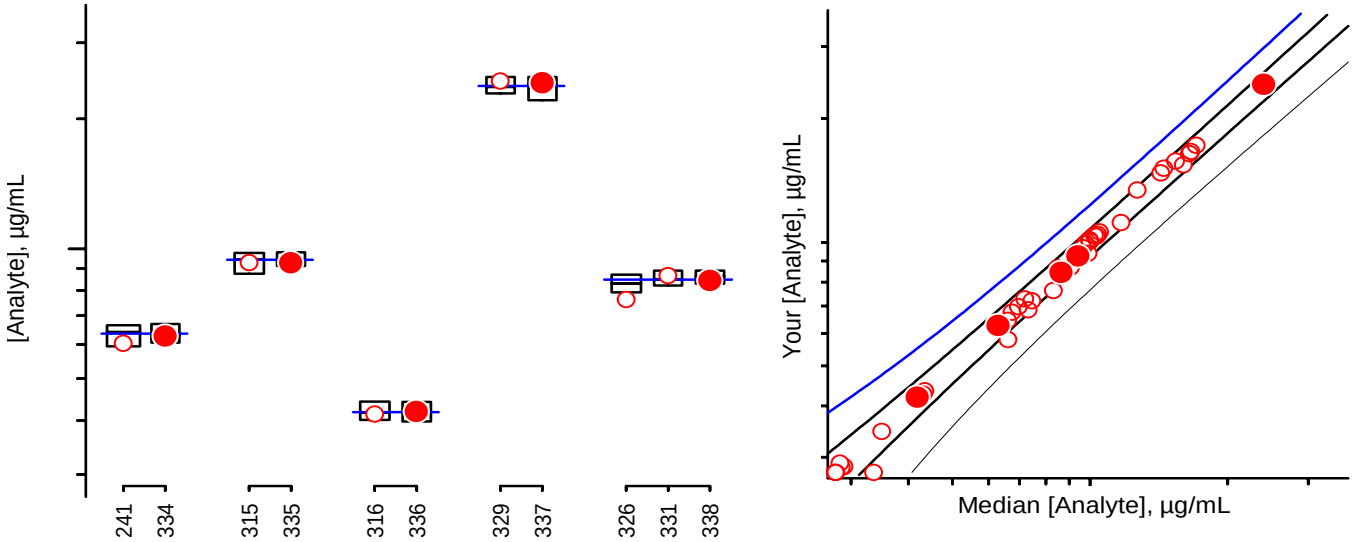
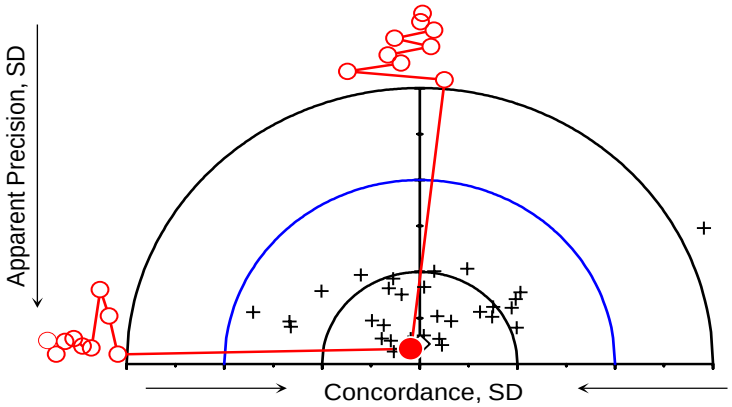
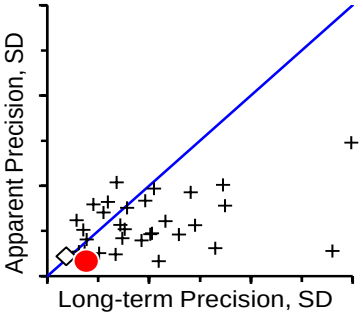
#338	Fresh frozen: 60:#326, 61:#331	Native, single-source

Individualized RR LXII 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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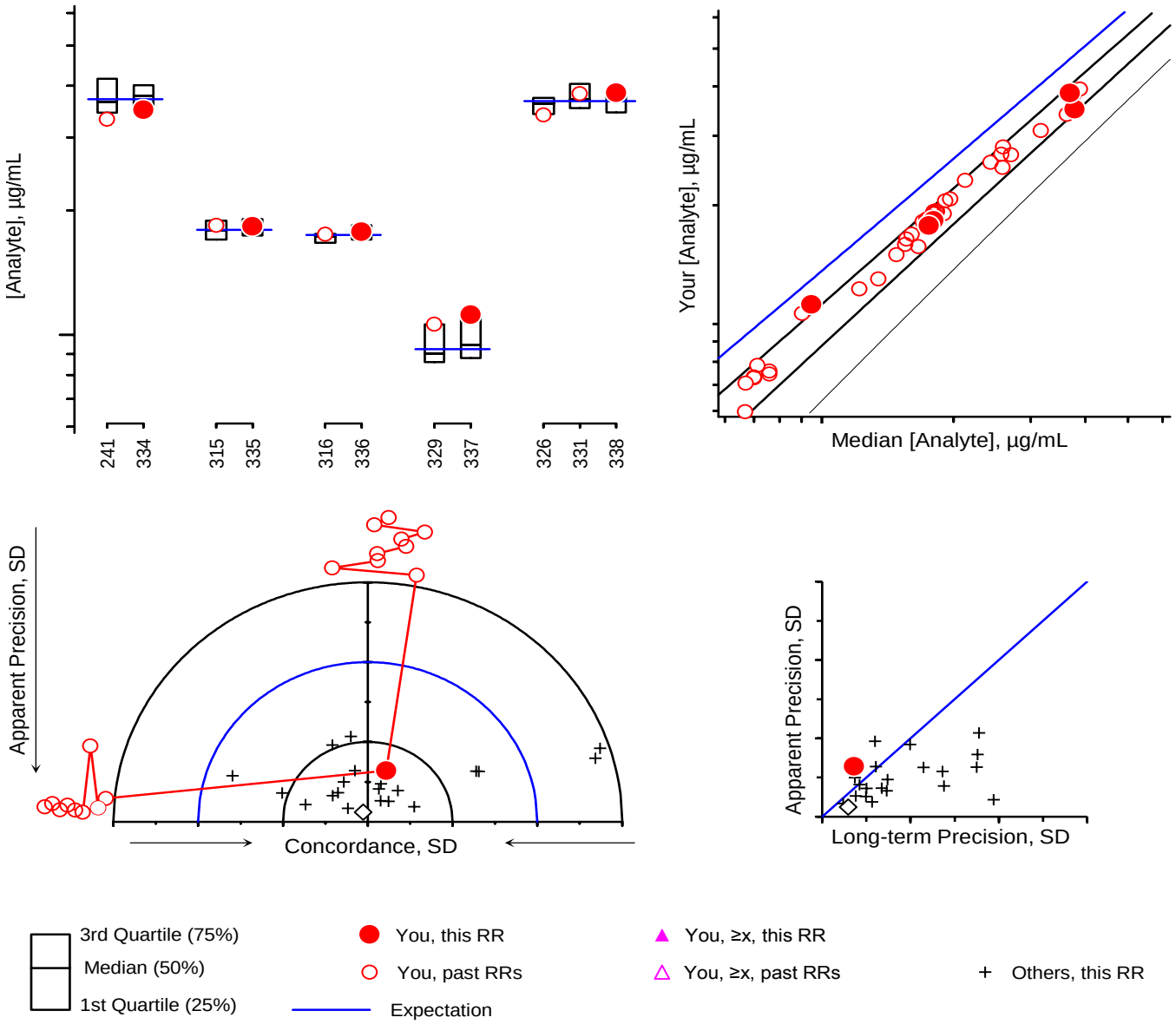
#337	Fresh frozen, 61:#329	Native, single-source
#338	Fresh frozen: 60:#326, 61:#331	Native, single-source

Individualized RR LXII Report: FSV-BA

γ/β-Tocopherol, µg/mL

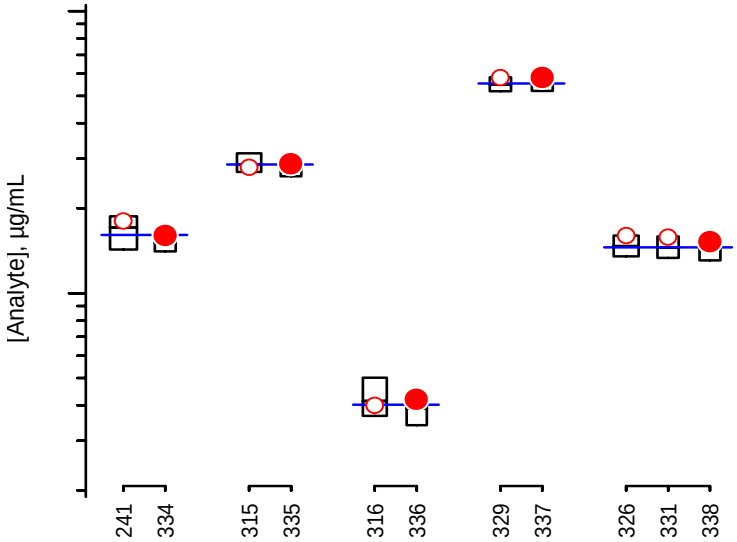


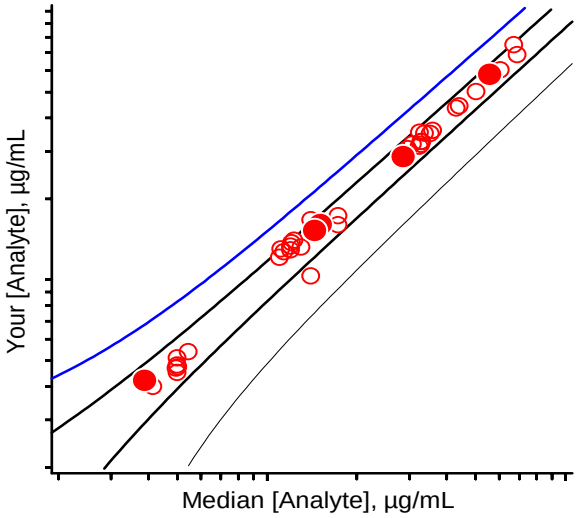
For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

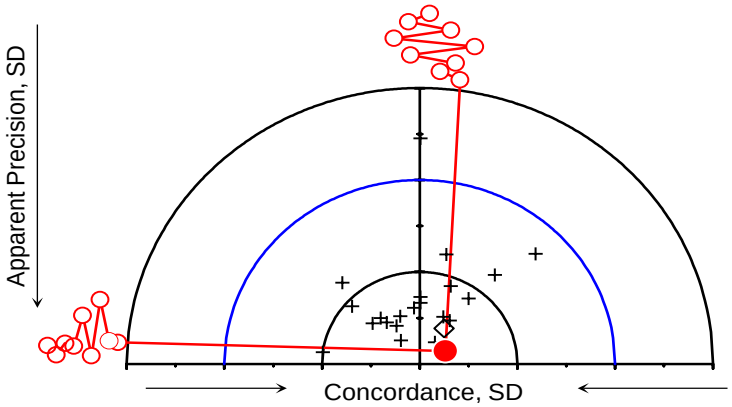
Serum	History	Comments
#334	Lyophilized, 42:#241	Augmented (RP,gT,dT,bC,aC,Z,Ly)
#335	Fresh frozen, 58:#315	Native, multi-source
#336	Fresh frozen, 58:#316	Native, multi-source
#337	Fresh frozen, 61:#329	Native, single-source
#338	Fresh frozen: 60:#326, 61:#331	Native, single-source

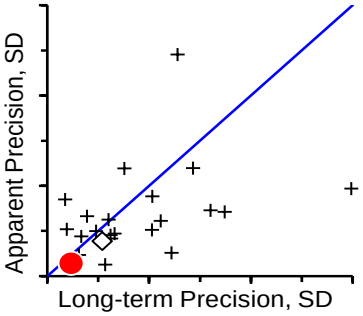
Individualized RR LXII Report: FSV-BA

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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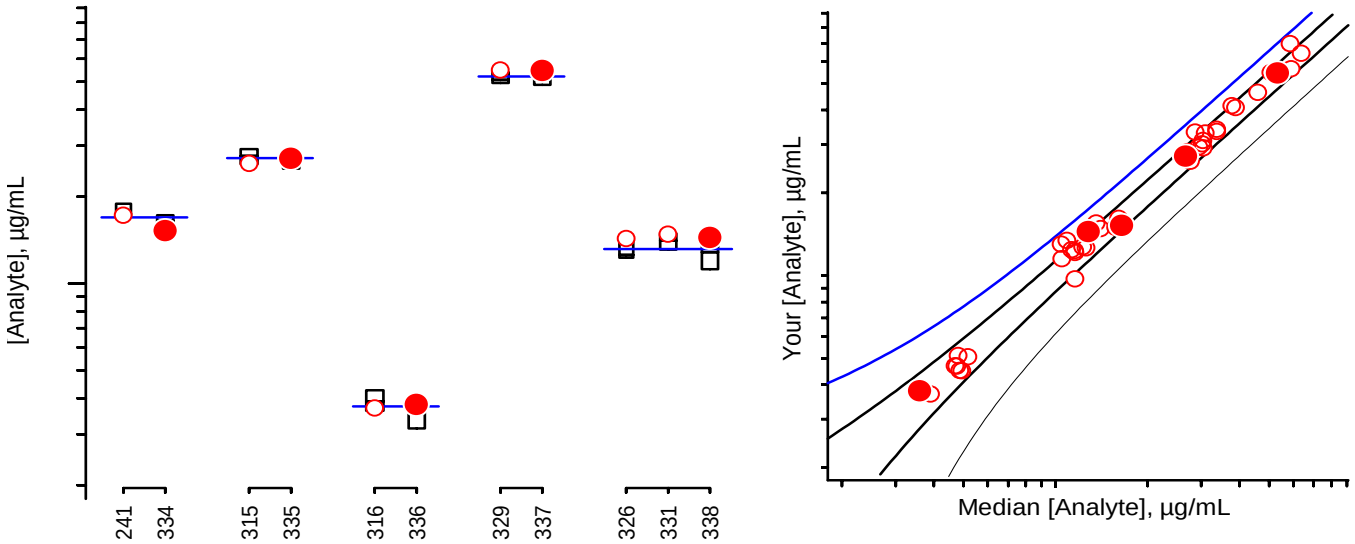
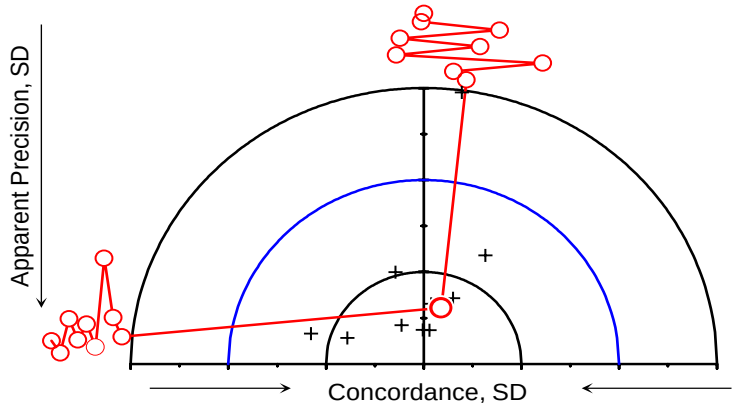
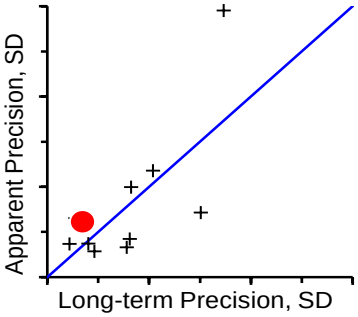
#337	Fresh frozen, 61:#329	Native, single-source
#338	Fresh frozen: 60:#326, 61:#331	Native, single-source

Individualized RR LXII 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
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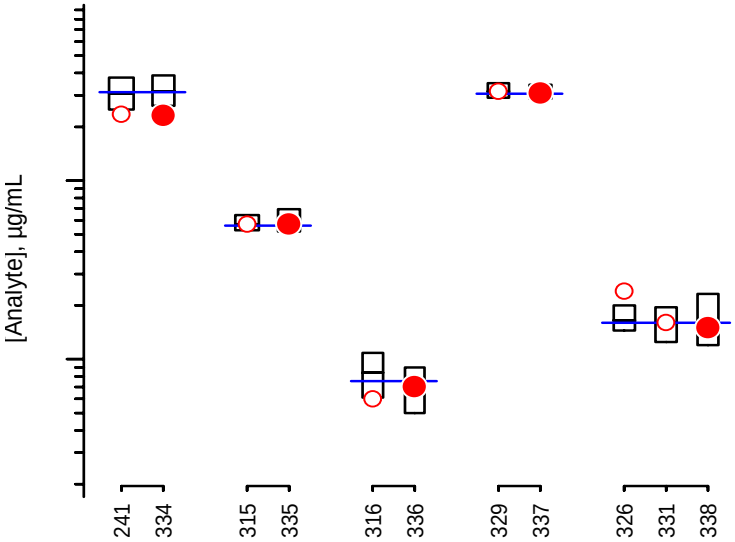
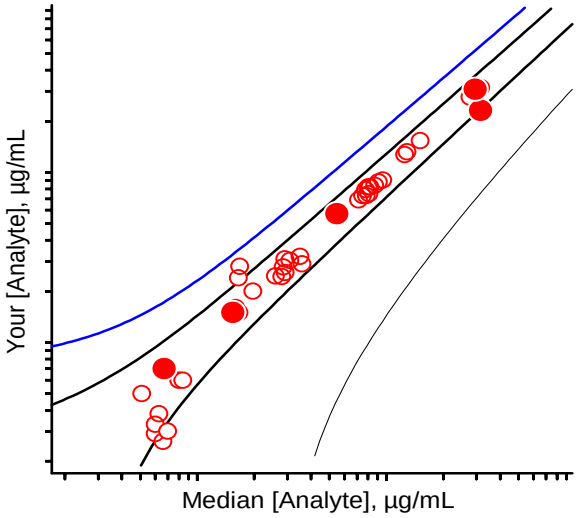
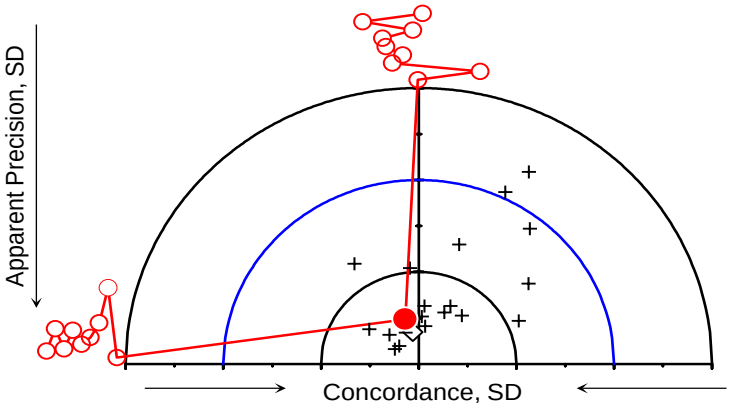
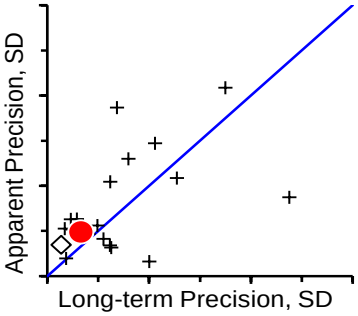
Individualized RR LXII Report: FSV-BA

Total α-Carotene, µg/mL









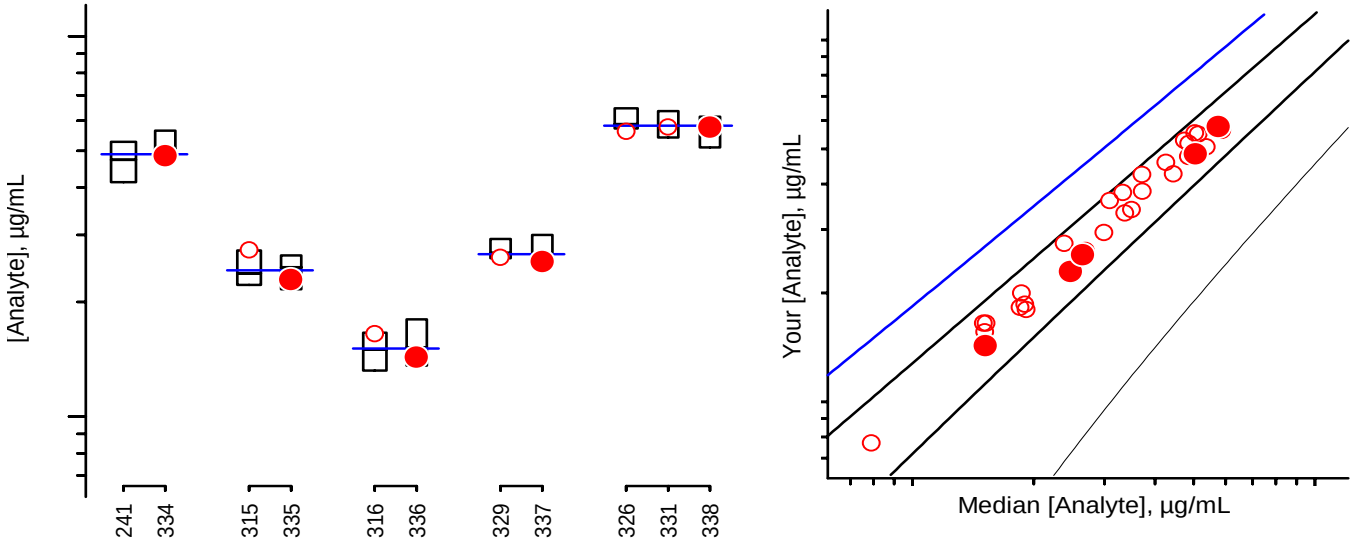
3rd Quartile (75%)
Median (50%)
1st Quartile (25%)
You, this RR
You, past RRs
Expectation
You, ≥x, this RR
You, ≥x, past RRs
Others, this RR

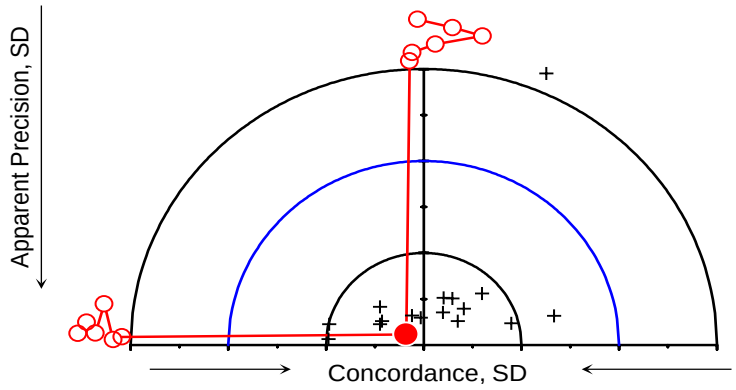
For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

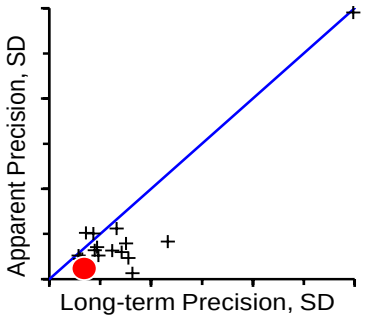
Serum	History	Comments
#334	Lyophilized, 42:#241	Augmented (RP,gT,dT,bC,aC,Z,Ly)
#335	Fresh frozen, 58:#315	Native, multi-source
#336	Fresh frozen, 58:#316	Native, multi-source
#337	Fresh frozen, 61:#329	Native, single-source
#338	Fresh frozen: 60:#326, 61:#331	Native, single-source

Individualized RR LXII Report: FSV-BA

Total Lycopene, µg/mL







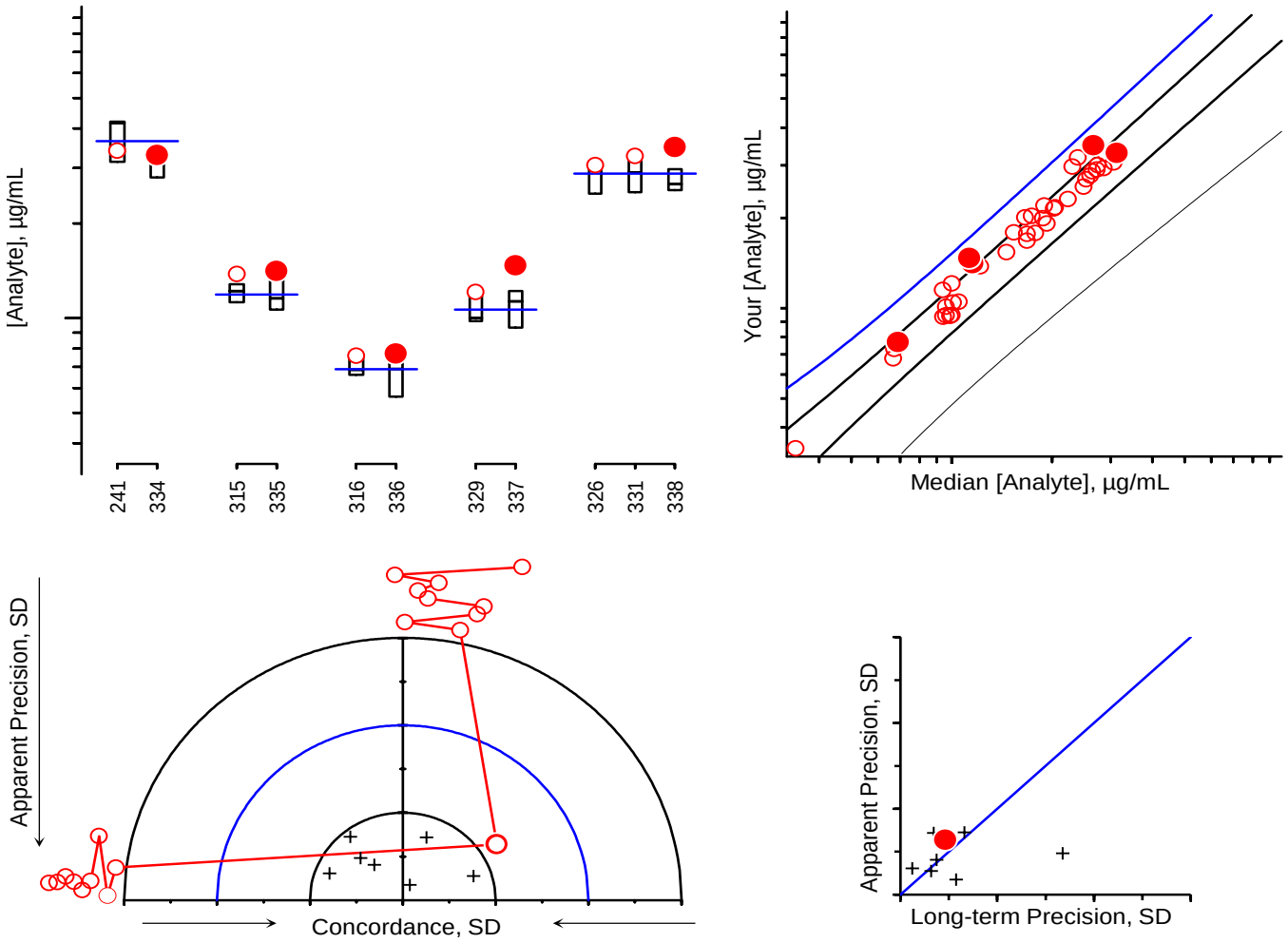
3rd Quartile (75%)
Median (50%)
1st Quartile (25%)
You, this RR
You, past RRs
Expectation
You, ≥x, this RR
You, ≥x, past RRs
Others, this RR

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.

Serum	History	Comments
#334	Lyophilized, 42:#241	Augmented (RP,gT,dT,bC,aC,Z,Ly)
#335	Fresh frozen, 58:#315	Native, multi-source
#336	Fresh frozen, 58:#316	Native, multi-source
#337	Fresh frozen, 61:#329	Native, single-source
#338	Fresh frozen: 60:#326, 61:#331	Native, single-source

Individualized RR LXII Report: FSV-BA

trans-Lycopene, µg/mL



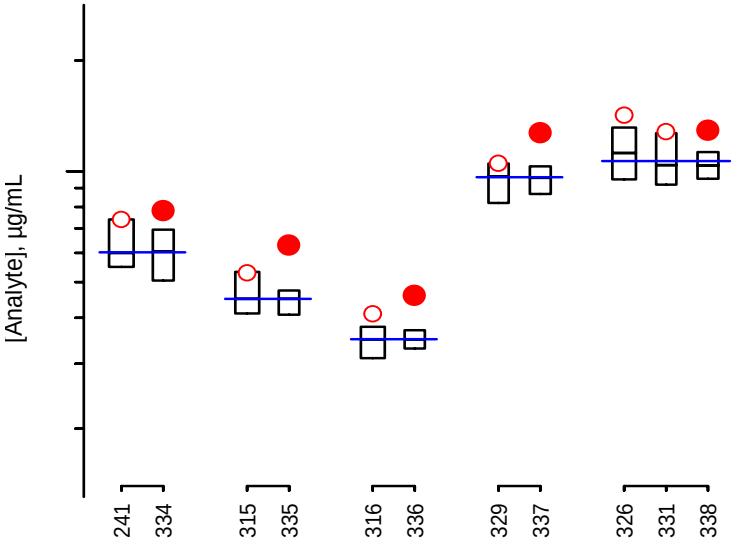
3rd Quartile (75%)
Median (50%)
1st Quartile (25%)
You, this RR
You, past RRs
Expectation
You, ≥x, this RR
You, ≥x, past RRs
Others, this RR

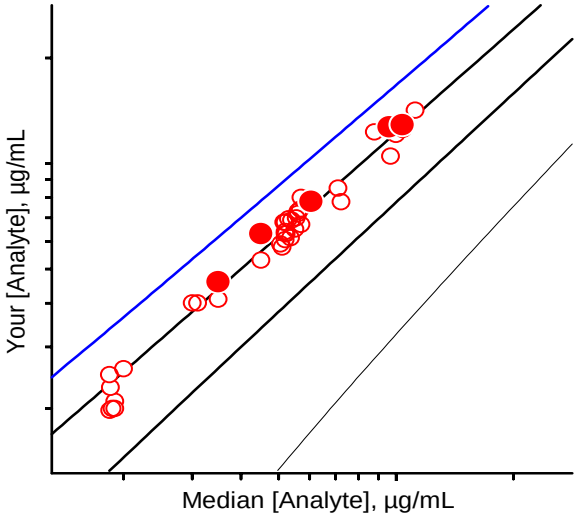
For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

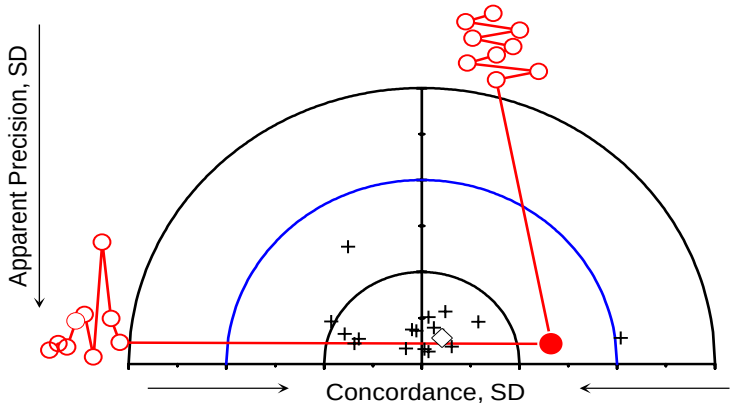
Serum	History	Comments
#334	Lyophilized, 42:#241	Augmented (RP,gT,dT,bC,aC,Z,Ly)
#335	Fresh frozen, 58:#315	Native, multi-source
#336	Fresh frozen, 58:#316	Native, multi-source
#337	Fresh frozen, 61:#329	Native, single-source
#338	Fresh frozen: 60:#326, 61:#331	Native, single-source

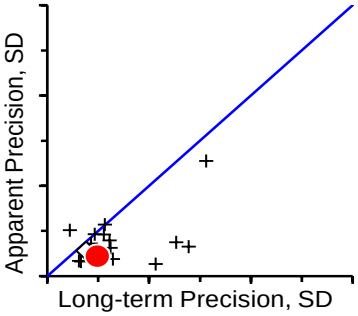
Individualized RR LXII Report: FSV-BA

Total β-Cryptoxanthin, µg/mL









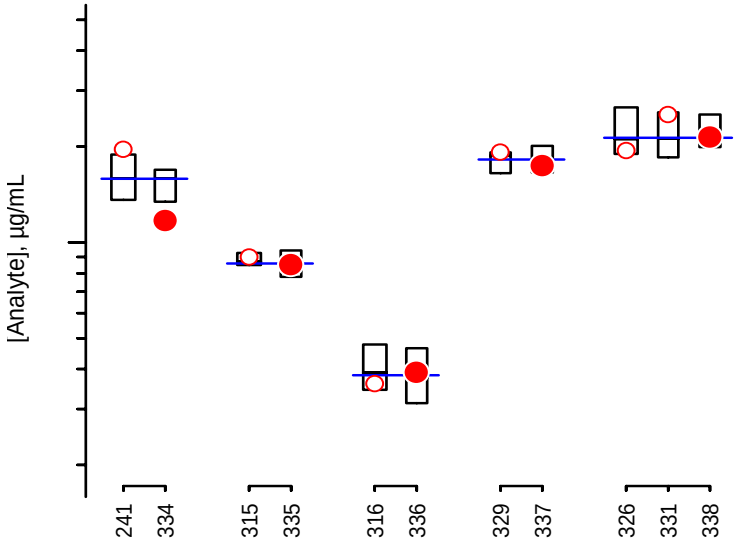
3rd Quartile (75%)
Median (50%)
1st Quartile (25%)
You, this RR
You, past RRs
Expectation
You, ≥x, this RR
You, ≥x, past RRs
Others, this RR

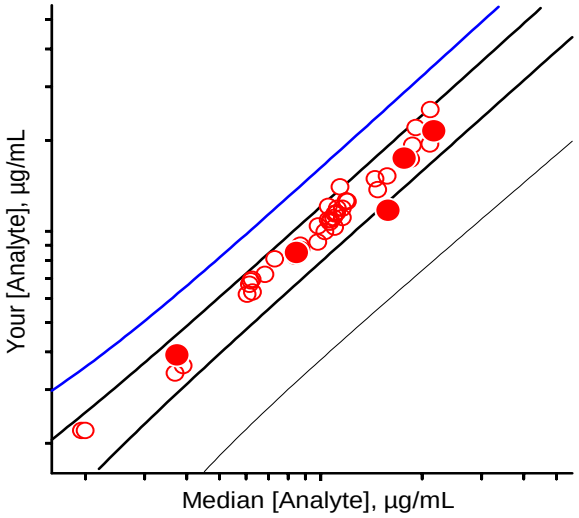
For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

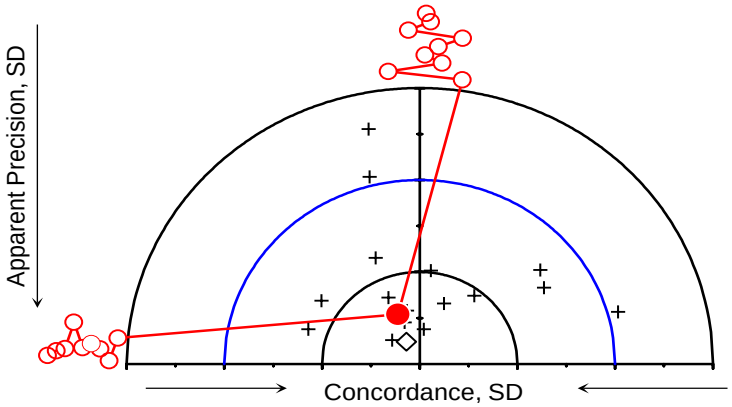
Serum	History	Comments
#334	Lyophilized, 42:#241	Augmented (RP,gT,dT,bC,aC,Z,Ly)
#335	Fresh frozen, 58:#315	Native, multi-source
#336	Fresh frozen, 58:#316	Native, multi-source
#337	Fresh frozen, 61:#329	Native, single-source
#338	Fresh frozen: 60:#326, 61:#331	Native, single-source

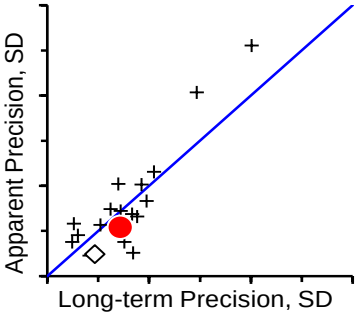
Individualized RR LXII Report: FSV-BA

Total Lutein&Zeaxanthin, µg/mL







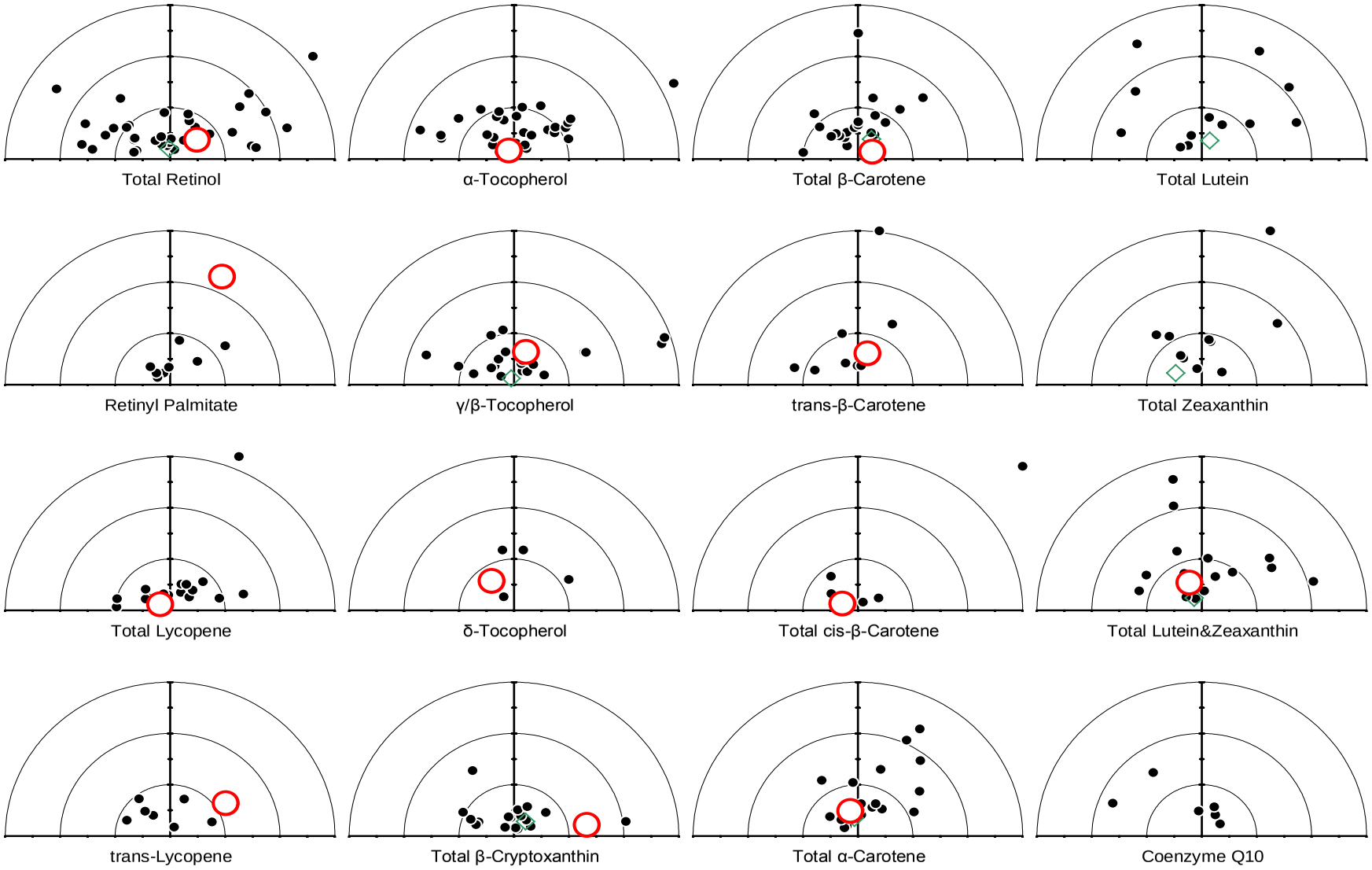


3rd Quartile (75%)
Median (50%)
1st Quartile (25%)
You, this RR
You, past RRs
Expectation
You, ≥x, this RR
You, ≥x, past RRs
Others, this RR

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	History	Comments
#334	Lyophilized, 42:#241	Augmented (RP,gT,dT,bC,aC,Z,Ly)
#335	Fresh frozen, 58:#315	Native, multi-source
#336	Fresh frozen, 58:#316	Native, multi-source
#337	Fresh frozen, 61:#329	Native, single-source
#338	Fresh frozen: 60:#326, 61:#331	Native, single-source

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



D14

Appendix E. Shipping Package Inserts for RR27

The following five items were included in each package shipped to an RR27 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 29, 2007

Dear Colleague:

The samples within this package constitute Vitamin C Round Robin 27 (RR27) of the 2007 Micronutrients Measurement Quality Assurance Program.

RR27 consists of four vials of frozen serum *test samples* (#36, #56, #115, and #118), 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 and ≈95% confidence interval for target value and ≈95% confidence interval for *CS #2* is 28.1 ±1.0 μmol/L of sample.

The report for RR26 was mailed the week of May 4, 2007. If you find your results for RR26 unsatisfactory, we recommend that you obtain **Standard Reference Material (SRM) 970 Ascorbic Acid in Serum** to validate your methodology and value assign in-house control materials. This SRM may be purchased from the Standard Materials Reference Program at NIST (Tel: 301-975-6776, Fax: 301-948-3730, or e-mail: srminfo@nist.gov).

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.

If you have any questions or concerns about the Vitamin C Micronutrients Measurement Quality Assurance Program please contact Jeanice Brown Thomas at tel: 301-975-3120, fax: 301-977-0685, or e-mail: jbthomas@nist.gov.

We ask that you return your results for these RR27 samples by **September 28, 2007**. We would appreciate receiving your results as soon as they become available. Please use the attached form. Your results will be kept confidential.

Sincerely,

Jeanice B Thomas

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

Enclosures: Protocols, Preparation and Analysis of Control Materials and Analysis of Test Samples
RR27 Report Form for Ascorbic Acid Solid Control Material Preparation
RR27 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 $[\mathrm{TAA}]_{DS1}$ = (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 $[\mathrm{TAA}]_{DS2}$ = 28.4 μmol/L and 0.125 mL should weigh 0.13 g and $[\mathrm{TAA}]_{DS3}$ = 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 calibrate 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: 301-975-3120 or Jeanice.BrownThomas@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 27

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 *before* **September 28, 2007**

MMQAP
100 Bureau Drive, Stop 8392
Gaithersburg, MD 20899-8392

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

Participant #: _____ Date: _____

Vitamin C Round Robin 27

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:* 28.1 ±1.0 µmol/L
Serum Test Sample #36	_____	_____	µmol/L of Sample
Serum Test Sample #46	_____	_____	µmol/L of Sample
Serum Test Sample #56	_____	_____	µmol/L of Sample
Serum Test Sample #114	_____	_____	µmol/L of Sample
Serum Test Sample #118	_____	_____	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 *before* **September 28, 2007**

MMQAP
100 Bureau Drive, Stop 8392
Gaithersburg, MD 20899-8392

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

Participant #: _____ Date: _____

Vitamin C Round Robin 27

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 #36	Liquid frozen (1:1 serum:10% MPA)
VitC #56	Liquid frozen (1:1 serum:10% MPA)
VitC #114	Liquid frozen (1:1 serum:10% MPA)
VitC #118	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 8392
Gaithersburg, MD 20899-8392

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

Appendix F. Final Report for RR27

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-

February 15, 2013

Dear Colleague:

Enclosed is the summary report of the results for Round Robin 27 (RR27) 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.

RR 27 consisted of four *test samples* (#36, #56, #114, and #118), one *serum control material*, 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.

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 B Thomas

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

Enclosures

Cc: L. C. Sander
D.L. Duewer

NIST

The NIST M^2QAP Vitamin C Round Robin 27 (RR27) 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 RR27 sample measurements.

Page	"All Lab" Report
1	A tabulation of results and summary statistics for Total Ascorbic Acid [TAA] in the RR27 samples and control/calibration solutions.

Serum-based Samples. One serum control and four unknowns were distributed in RR27.

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

S27:1 Serum 36, ampouled in late 2001, previously distributed as sample S17:2 (RR17, Fall 02), S18:1 (RR18, Spring 03), S20:1 (RR20, Spring 04), S22:2 (RR22, Spring 05), and S23:2 (RR23, Fall 05).

S27:2 Serum 56, ampouled in late 2001, previously distributed as sample S16:3 (RR16, Spring 02), S17:3 (RR17, Fall 02),S20:2 (RR20, Spring 04), S21:4 (RR21, Fall 04), and S23:3 (RR23, Fall 05).

S27:3 Serum 114, ampouled in 1995, previously distributed as sample 188a in (RR9, Summer 96).

S27:4 Serum 118, ampouled in 1995, previously distributed as sample 688a in (RR8, Fall 95) and (RR9, Summer 96).

Results.

1) All participants who prepared the four 5% MPA control/calibration solutions 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) Judging from the calibration parameters calculated for the control/calibration solutions (intercepts close to 0.0, slopes close to 1.0, R^2 close to 1, and RMS close to 0.0), the measurement systems for most participants are linear and fairly well calibrated. However, several measurement systems perform somewhat differently for the control solutions than with the serum samples.

3) Sample S27:2 (Serum 56): One participant with concordant results for the other serum materials reported low results for this high-level material. While other participants reported systematically low results for all materials, there were no other anomalous results for this material. Evaluation of the participant's prior results for S27:2 material suggests that variable results with high-level materials may be characteristic of the particular measurement system. While a seal-defect in the ampoule analyzed is possible, we do not believe that this six-year old material *per se* has degraded.

4) Sample S27:4 (Serum 118): The median result for this material is unchanged from 1995 values. However, four participants reported discordantly high values for this low-level material. Since "degradation" should not *increase* [TAA], we do not believe that the apparent increased variability results from storage – particularly since several of these participants reported systematically higher than expected results. However, it is plausible that some ampoules have become miss-identified over the years. If we distribute this material again, it will be accompanied by the low-level CS#1.

Appendix G. "All-Lab Report" for RR27

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" 27 - September 2007

		Control / Calibration Samples											MPA	Dilute Solution 1			Samples									
		Gravimetric, µmol/L			Measured, µmol/L				Calibration Parameters				Density	Spectrophotometry			Measured, µmol/L					Calibrated to Gravimetric, µmol/L				
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		S27:1	S27:2	S27:3	S27:4	CS#2	S27:1	S27:2	S27:3	S27:4
VC-MA	05/10/07	57.2	28.5	14.0	56.8	28.1	13.9	0.0	-0.05	0.99	1.000	0.1	1.032	242.	0.5610	557.2	27.7		22.6	48.1	28.5	13.8	28.0	22.9	48.5	28.8	13.9
VC-MB	09/07/07	57.6	28.6	14.2	58.6	29.7	14.7	0.0	0.23	1.02	1.000	0.3	1.029	244.	0.5650	557.3	27.1		18.1	45.9	27.4	13.2	26.4	17.5	44.9	26.7	12.7
VC-MC	16/07/07	56.0	28.1	13.9	59.0	30.3	15.2	0.0	0.39	1.05	1.000	0.5	1.030	243.	0.5486	556.0	28.4		22.6	47.9	28.4	13.4	26.6	21.2	45.2	26.6	12.4
VC-ME	18/07/07	56.9	29.2	14.3	59.2	29.1	14.4	0.0	-0.45	1.04	0.999	0.8	1.037	243.	0.5610	559.7	27.6		22.1	33.4	27.5	14.5	26.9	21.6	32.6	26.9	14.4
VC-MG	13/08/07	61.1	31.0	16.5	62.3	30.5	15.8	0.0	-0.62	1.02	0.999	0.7	1.029	243.5	0.6170	573.8	29.0		26.1	51.4	35.1	18.3	29.0	26.1	50.9	35.0	18.5
VC-MH	28/08/07	62.4	31.1	15.5	62.5	31.1	15.4	0.0	-0.08	1.00	1.000	0.1	1.031	244.1	0.6220	566.4	27.4		21.1	44.7	28.4	13.9	27.4	21.1	44.6	28.3	13.9
VC-MI	06/08/07	56.5	28.3	14.0	59.5	29.2	14.6	0.0	-0.21	1.05	1.000	0.4	1.027				27.5		20.3	45.3	26.7	13.2	26.3	19.4	43.2	25.5	12.7
VC-MJ	25/09/07	48.2	24.6	12.3	51.3	27.8	14.1	10.1	6.84	0.89	0.974	3.7	1.029	*254*[a]	*0.303*[a]	*356.8*[a]	29.7		26.0	51.2	36.4	18.2	25.7	21.4	49.8	33.2	12.8
VC-MK	13/08/07	57.5	28.7	14.5	60.8	27.8	15.9	4.8	2.61	0.98	0.990	3.0	1.030	244.	0.5682	560.7	35.9		26.5	51.1	42.4	20.1	34.0	24.4	49.4	40.6	17.8
VC-MN	28/09/07	61.1	30.6	15.0	63.4	30.9	14.3	0.0	-0.70	1.04	0.999	0.8	1.031	244.	0.6051	562.3	29.3		25.8	48.9	31.9	21.3	28.8	25.4	47.5	31.3	21.1
VC-MU	19/09/07	57.2	28.8	13.8	54.5	25.9	12.8	0.0	-0.46	0.95	0.999	0.9	1.030	245.	0.5426	538.4	23.0		14.8	39.8	19.3	9.1	24.6	16.0	42.3	20.8	10.0
	N	11	11	11	11	11	11	11					N	11	9	9	9	11		11	11	11	11	11	11	11	11
	Average	57.4	28.9	14.4	58.9	29.1	14.6	1.4					Average	1.030	243.6	0.5767	559.1	28.4		22.3	46.1	30.2	15.4	27.6	21.5	45.4	29.4	14.6
	SD	3.7	1.8	1.1	3.6	1.6	0.9	3.2					SD	0.003	0.9	0.0299	9.5	3.1		3.7	5.5	6.1	3.6	2.5	3.1	5.1	5.3	3.2
	Min	48.2	24.58	12.3	51.3	25.85	12.8	0.0					Min	1.027	242.0	0.5426	538.4	23.0		14.8	33.4	19.3	9.1	24.6	16.0	32.6	20.8	10.0
	%25	56.7	28.41	13.9	57.7	27.95	14.2	0.0					%25	1.029	243.0	0.5610	557.2	27.4		20.7	45.0	27.5	13.3	26.3	20.3	43.9	26.6	12.7
	Median	57.2	28.74	14.2	59.2	29.15	14.6	0.0					Median	1.030	244.0	0.5650	559.7	27.7		22.6	47.9	28.4	13.9	26.9	21.4	45.2	28.3	13.9
	%75	59.3	29.93	14.8	61.5	30.43	15.3	0.0					%75	1.031	244.0	0.6051	562.3	29.2		25.9	50.0	33.5	18.3	28.4	23.6	49.0	32.2	16.1
	Max	62.4	31.08	16.5	63.4	31.05	15.9	10.1					Max	1.037	245.0	0.6220	573.8	35.9		26.5	51.4	42.4	21.3	34.0	26.1	50.9	40.6	21.1
	MADe	1.1	0.8	0.5	3.6	2.0	0.9	0.0					eSD	0.0	0.7	0.0243	3.9	0.9		4.7	4.7	2.5	1.1	1.6	2.9	4.3	4.2	1.8
	CV	2	3	3	6	7	6						CV	0.13	0.30	4.3	0.7	3		21	10	9	8	6	14	9	15	13

a) 5% Trichloroacetic acid solution

G2

Appendix H. Representative "Individualized Report" for RR27

Each participant in RR27 received an "Individualized Report" reflecting their reported results. The following two pages are the "Individualized Report" for participant "VC-MA".

Vitamin C "Round Robin" 27 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
03/08/05	22	HPLC-EC	1.034	243.0	0.559	562.9	0.2	1.06	1.000	0.24
10/17/05	23	HPLC-EC	1.030	244.0	0.562	567.9	-0.6	1.09	0.998	1.47
03/09/06	24	HPLC-EC	1.031	244.0	0.568	586.7	0.2	1.13	1.000	0.41
08/28/06	25	HPLC-EC	1.039	242.0	0.555	557.4	0.8	0.95	0.999	0.92
03/20/07	26	HPLC-EC	1.033	244.0	0.573	554.3	0.3	1.00	1.000	0.31
10/05/07	27	HPLC-EC	1.032	242.0	0.561	557.2	-0.1	0.99	1.000	0.14
		Mean	1.033	243.2	0.56	564.4				0.58
		SD	0.003	1.0	0.01	12.0				0.51
		CV	0.31	0.40	1.2	2.1				

[TAA] mmol/Lsample

Date	RR	Sample	Rep_1	Rep_2	F_{adj}	Mean	SD_{dup}	N	Mean	SD_{repeat}	SD_{reprod}
03/08/05	22	CS#2	29.0	29.0	1.0	29.0	0.0	5	28.7	0.5	1.0
10/17/05	23	CS#2	29.4	30.5	1.0	30.0	0.8				
03/09/06	24	CS#2	29.2	29.1	1.0	29.2	0.1				
08/28/06	25	CS#2	27.2	28.1	1.0	27.6	0.6				
10/05/07	27	CS#2	**28.1**	**27.4**	1.0	27.7	0.5				
12/12/02	17	S17:2	23.3	23.4	1.0	23.4	0.1	6	23.6	0.5	1.0
03/20/03	18	S18:1	22.7	23.7	1.0	23.2	0.7				
02/23/04	20	S20:1	25.1	24.1	1.0	24.6	0.7				
03/08/05	22	S22:2	22.7	22.7	1.0	22.7	0.0				
10/17/05	23	S23:2	25.5	24.4	1.0	24.9	0.8				
10/05/07	27	S27:1	**22.9**	**22.4**	1.0	22.6	0.4				
11/18/02	16	S16:3	49.9	44.9	1.0	47.4	3.5	6	48.6	1.5	1.4
12/12/02	17	S17:3	49.7	49.1	1.0	49.4	0.4				
02/23/04	20	S20:2	50.6	50.0	1.0	50.3	0.4				
09/13/04	21	S21:4	47.1	47.0	1.0	47.0	0.0				
10/17/05	23	S23:3	49.8	48.8	1.0	49.3	0.7				
10/05/07	27	S27:2	**48.6**	**47.6**	1.0	48.1	0.8				
06/19/96	09	S09:2	51.7	51.1	0.5	25.7	0.2	2	27.1	0.2	2.0
10/05/07	27	S27:3	**28.5**	**28.6**	1.0	28.5	0.1				
ND	08							2	13.5	1.3	1.0
06/19/96	09	S09:1	29.1	23.8	0.5	13.2	1.9				
10/05/07	27	S27:4	**13.9**	**13.7**	1.0	13.8	0.2				

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

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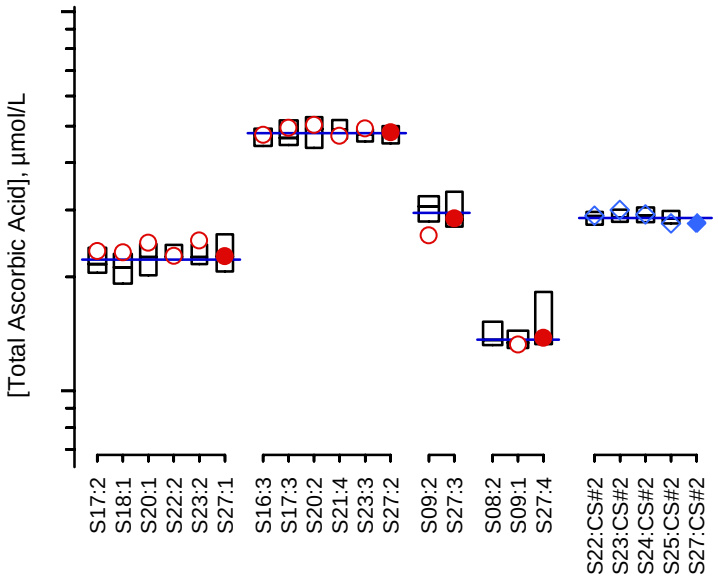
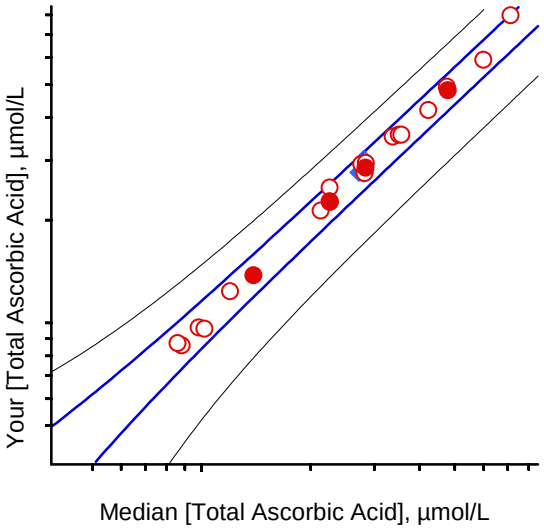
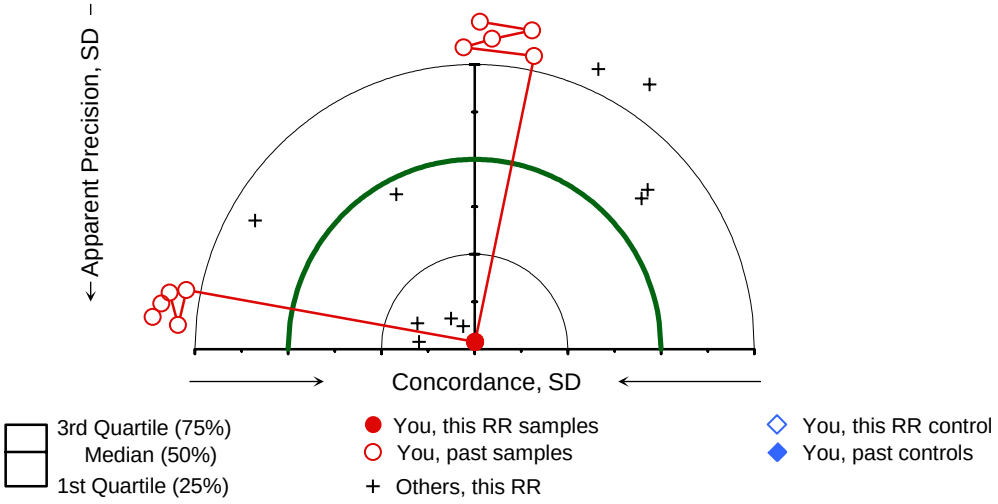
Vitamin C "Round Robin" 27 Report: Participant VC-MA

Total Ascorbic Acid







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
S27:1	VitC #36, previously distributed in RRs 17, 18, 20, 22, and 27
S27:2	VitC #56, previously distributed in RRs 16, 17, 20, 21, and 23
S27:3	VitC #114, previous distributed in RR 9
S27:4	VitC #118, previous distributed in RR 8 and 9