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NIST Micronutrients Measurement Quality Assurance Program Summer 2008 Comparability Studies

Results for Round Robin LXIV
Fat-Soluble Vitamins and Carotenoids in Human Serum
and Round Robin 29 Ascorbic Acid in Human Serum

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

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Materials Measurement Laboratory

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 2008 MMQAP measurement comparability improvement studies: 1) Round Robin LXIV Fat-Soluble Vitamins and Carotenoids in Human Serum and 2) Round Robin 29 Total Ascorbic Acid in Human Serum. The materials for both studies were shipped to participants in May 2008; participants were requested to provide their measurement results by September 12, 2008.

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 LXIV: Fat-Soluble Vitamins and Carotenoids in Human Serum

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin LXIV comparability study (hereafter referred to as RR64) received two lyophilized and three 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 2008. 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 RR64 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 29: Vitamin C in Human Serum

Participants in the MMQAP Vitamin C in Human Serum Round Robin 29 comparability study (hereafter referred to as RR29) 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 2008. 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 RR29 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 RR64

The following three items were included in each package shipped to an RR64 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 12, 2008

Dear Colleague:

Enclosed are the samples (Sera 347-351) for the second fat-soluble vitamins and carotenoids in serum round robin study (Round Robin LXIV) for the fiscal year (FY) 08 NIST Micronutrients Measurement Quality Assurance Program. You will find one vial of each of three liquid-frozen serum samples and two lyophilized samples 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 12, 2008**. Results received more than two weeks after the due date will not be included in the summary report for this round robin study. For your convenience, we have also included one vial of **Serum 198** (which was distributed in four past round robin studies) that can be used as a control and to help validate your method. We have included the averaged assigned values for the measurands in this serum as well.

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.**

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).

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

Reference Values for NIST Serum 198, μg/mL

Analyte	Value[a]	U_{95}[b]	P_{95}[c]
Total Retinol	0.73	0.02	0.11
Retinyl Palmitate	0.25	0.04	0.14
α-Tocopherol	14.1	0.3	2.0
β/γ-Tocopherol	2.57	0.06	0.31
Total β-Carotene	0.68	0.01	0.15
trans- β-Carotene	0.634	0.006	0.093
Total *cis*- β-Carotene	0.055	0.011	0.036
Total α-Carotene	0.039	0.007	0.020
Total Lycopene	0.155	0.030	0.079
trans-Lycopene	0.070	0.008	0.031
Total β-Cryptoxanthin	0.019	0.004	0.012
Total Lutein	0.052	0.003	0.019
Total Zeaxanthin	0.020	0.003	0.011
Total Lutein&Zeaxanthin	0.073	0.006	0.030

a Expected value; the average of the interlaboratory median results and mean of NIST results in M^2QAP RR31, RR35, RR43, and RR57

b Approximate 95% uncertainty interval; the interval Value ± U_{95} is believed with approximately 95% confidence to contain the true value of the analyte

c Approximate 95% prediction interval; the interval Value ± P_{95} contains approximately 95% of valid measurements obtained with measurement systems similar to those in use in M^2QAP RR31, RR35, RR43, and RR57

Participant #: _____ Date: _____

Round Robin LXIV: Human Sera

NIST Micronutrients Measurement Quality Assurance Program

Analyte	347	348	349	350	351	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 (349 to 351) frozen when received? Yes | No

Comments:

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

Please return results **before** 12-Sep-2008

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

Participant #: _____ Date: _____

Fat-Soluble Vitamins Round Robin LXIV

NIST Micronutrients Measurement Quality Assurance Program

Packing List and Shipment Receipt Confirmation Form

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

Serum	Form	Reconstitute?	Vial/Cap
#347	Lyophilized	Yes (1 ml H_2O)	5 mL clear, silver
#348	Lyophilized	Yes (1 ml H_2O)	2 mL amber, blue
#349	Liquid frozen	No	2 mL amber, silver
#350	Liquid frozen	No	2 mL amber, silver
#351	Liquid frozen	No	10 mL amber, silver

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 RR64

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 20, 2008

Dear Colleague:

Enclosed is the summary report of the results for round robin LXIV (RR64) of the 2008 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.

We have received your inquiries regarding the availability of the renewal material SRM 968d, Fat-Soluble Vitamins, Carotenoids, and Cholesterol in Human Serum. The material has been value-assigned. Our SRM office is currently finalizing the packaging of the SRM prior to sale. Orders can be placed directly through our on-line SRM order request system at: https://srmors.nist.gov/index.cfm. You can also call the SRM office directly at (301) 975-2200 for more details regarding the SRM's availability.

Samples for the first 2009 QA interlaboratory exercise will be shipped during the week of December 8, 2009. If you have any questions regarding this report, please contact Dave Duewer at david.duewer@nist.gov or me at jbthomas@nist.gov, tel: 301/975-3120, or fax: 301/977-0685.

Sincerely,

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

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

NIST

The NIST M^2QAP Round Robin LXIV (RR64) 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. Five samples were distributed in RR64.

Serum	Description	Prior Distributions
347	Lyophilized, augmented, multi-donor serum prepared in 1994. This material is a 1:1 blend of stripped serum and a serum pool augmented with retinol, retinyl palmitate, and α-tocopherol.	#195:RR31-6/94, #214:RR35-9/95; #244:RR43-6/98, #328:RR60-9/06
348	Lyophilized, native, single-donor, commercially obtained serum prepared in 2002. The same material was used to prepare #349.	#290:RR53-2/03, #300:RR55-3/04, #312:RR57-3/05, #322:RR59-3/06, #333:RR61-3/07
349	Fresh-frozen, native, single-donor, commercially obtained serum prepared in 2002. The same material was used to prepare #348.	#292:RR53-2/03, #301:RR55-3/04, #313:RR57-3/05, #323:RR59-3/06, #332:RR61-3/07
350	Fresh-frozen, native, multi-donor *plasma* commercially prepared in 2006.	#340:RR63-3/08
351	Fresh-frozen, native, multi-donor serum prepared in Fall, 2007 (SRM 968d)	#341 & #344:RR63-3/08

Results

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

2) Candidate SRM 1950, Metabolites in Human Plasma (#350). As in RR63, no analysis problems were reported for this material. We anticipate this material being available for purchase mid-2009.

3) SRM 968d Fat-Soluble Vitamins and Carotenoids in Human Serum (#351). As in RR63, no analysis problems were reported for this material. It will be available for purchase in the near future.

Appendix C. "All-Lab Report" for RR64

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. 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 LXIV Laboratory Results

	Total Retinol, µg/mL					trans-Retinol, µg/mL					Retinyl Palmitate, µg/mL					α-Tocopherol, µg/mL					γ/β-Tocopherol, µg/mL					δ-Tocopherol, µg/mL				
Lab	347	348	349	350	351	347	348	349	350	351	347	348	349	350	351	347	348	349	350	351	347	348	349	350	351	347	348	349	350	351
FSV-BA	0.409	0.633	0.665	0.431	0.356						0.091	0.070	0.087	0.023	0.025	7.11	9.04	9.59	7.78	5.80	1.35	1.63	1.74	1.73	1.48	0.081	0.046	0.053	0.075	0.080
FSV-BB	0.429	0.680	0.679	0.438	0.396						0.079	0.066	0.067	0.012	0.007	6.94	10.19	10.30	8.02	5.99	1.29	1.67	1.71	1.65	1.38	0.069	0.054	0.053	0.068	0.073
FSV-BC	0.355	0.630	0.673	0.419	0.370																									
FSV-BD	0.407	0.589	0.604	0.413	0.334											7.90	10.30	10.80	9.30	6.10										
FSV-BE	0.448	0.696	0.746	0.464	!0.294											6.99	9.37	9.80	7.46	!0.47	1.33	1.72	1.86	1.75	!0.55					
FSV-BF	0.350	0.590	0.600	0.410	0.330											7.00	10.00	10.20	8.50	6.20										
FSV-BG	0.383	0.607	0.644	0.428	0.358						0.101	0.094	0.104	0.014	*nd*	7.30	10.13	10.68	8.54	6.49	1.36	1.82	1.87	1.87	1.59					
FSV-BH	0.305	0.558	0.620	0.381	0.312											7.73	10.96	11.58	9.04	6.60	1.32	1.73	1.86	1.76	1.44					
FSV-BI	0.385	0.583	0.616	0.397	0.323						0.116	0.102	0.102	*nd*	*nd*	6.52	10.03	10.53	8.16	5.80	1.40	1.80	1.91	1.86	1.52					
FSV-BJ	0.368	0.545	0.606	0.385	0.314						0.116	0.071	0.082	*nq*	*nq*	7.58	9.94	11.04	8.60	6.06	1.31	1.56	1.75	1.74	1.43					
FSV-BK	0.472	0.702	0.738	0.526	0.470											7.35	10.45	11.25	9.18	6.45										
FSV-BL	0.340	0.660	0.690	0.430	0.340											6.46	9.48	9.91	7.75	5.60										
FSV-BM	0.350	0.620	0.650	0.420	0.350											6.80	10.60	10.80	9.00	5.90										
FSV-BN	0.364	0.548	0.554	0.372	0.314						0.122	0.084	0.088	0.026	0.018	6.09	8.50	8.61	6.99	4.90	1.22	1.59	1.63	1.62	1.36					
FSV-BO	0.344	0.739	0.633	0.464	0.438											6.68	10.21	10.98	8.61	6.42	1.22	1.71	1.75	1.74	1.44					
FSV-BP	0.444	0.633	0.648	0.447	0.358											7.89	9.21	10.30	9.44	5.42										
FSV-BQ	0.382	0.634	0.656	0.425	0.323											6.99	8.74	9.00	6.89	4.70										
FSV-BR	*≥0.420*	*≥0.690*	*≥0.630*	*≥0.430*	*≥0.340*	0.420	0.690	0.630	0.430	0.340						6.26	8.35	7.70	6.22	3.00										
FSV-BS	*≥0.392*	*≥0.424*	*≥0.353*	*≥0.592*	*≥0.694*	0.392	0.424	0.353	0.592	0.694																				
FSV-BT	0.409	0.661	0.568	0.440	0.438											7.27	10.18	10.99	8.70	4.93	1.31	1.72	1.82	1.79	1.19					
FSV-BU	0.342	0.557	0.606	0.442	0.324											5.06	11.09	10.60	7.19	4.19	1.42	1.61	1.75	1.93	1.33					
FSV-BV	0.305	0.642	0.648	0.394	0.330											7.05	10.62	10.14	7.45	4.88	1.19	1.71	1.67	1.55	1.22					
FSV-BW	0.400	0.620	0.690	0.430	0.350						0.143	0.122	0.131	0.017	0.007	7.68	10.09	11.17	8.88	6.08	1.21	1.53	1.66	1.61	1.32					
FSV-CD	0.320	0.550	0.570	0.370	0.290											7.35	10.45	10.84	8.66	5.94	1.21	1.69	1.75	1.73	1.37					
FSV-CE	0.380	0.660	0.700	0.450	0.340											7.80	5.50	12.10	9.70	6.00										
FSV-CF	0.447	0.685	0.724	0.483	0.394											7.40	10.10	10.70	8.90	5.80										
FSV-CG	0.369	0.625	0.608	0.401	0.336											6.61	9.51	9.54	7.14	*nd*	1.28	1.78	1.79	1.78	0.81	*nd*	*nd*	*nd*	*nd*	0.142
FSV-CI	0.288	0.676	0.701	0.458	0.379						0.152	0.093	0.109	0.015	<0.02	8.07	10.24	11.09	8.45	5.88	1.61	1.86	1.99	1.95	1.61					
FSV-CW	0.350	0.593	0.605	0.404	0.339						0.076	0.069	0.070	0.013	0.005	6.97	10.29	10.90	8.62	6.21	1.37	1.79	1.91	1.85	1.50	0.075	0.041	0.048	0.082	0.086
FSV-CZ	0.322	0.552	0.565	0.363	0.314											6.39	10.92	8.62	7.92	3.48	1.77	2.20	2.20	2.45	1.67					
FSV-DD	0.360	0.530	0.580	0.360	0.300																									
FSV-DQ																7.72	8.43	11.02	8.22	5.42	1.34	1.57	1.84	1.71	1.41					
FSV-DV	*≥0.386*	*≥0.603*	*≥0.641*	*≥0.407*	*≥0.334*	0.386	0.603	0.641	0.407	0.334						5.40	8.90	9.60	7.30	5.00										
FSV-EE	0.335	0.562	0.569	0.366	0.291											7.10	10.40	10.70	8.50	5.40										
N	30	30	30	30	29	3	3	3	3	3	9	9	9	7	5	31	31	31	31	29	19	19	19	19	18	3	3	3	3	4
Min	0.288	0.530	0.554	0.360	0.290	0.386	0.424	0.353	0.407	0.334	0.076	0.066	0.067	0.012	0.005	5.06	5.50	7.70	6.22	3.00	1.19	1.530	1.63	1.55	0.81	0.069	0.041	0.048	0.068	0.073
Median	0.366	0.623	0.638	0.423	0.339	0.392	0.603	0.630	0.430	0.340	0.116	0.084	0.088	0.015	0.007	7.05	10.10	10.68	8.50	5.80	1.32	1.710	1.79	1.75	1.42	0.075	0.046	0.053	0.075	0.083
Max	0.472	0.739	0.746	0.526	0.470	0.420	0.690	0.641	0.592	0.694	0.152	0.122	0.131	0.026	0.025	8.07	11.09	12.10	9.70	6.60	1.77	2.196	2.20	2.45	1.67	0.081	0.054	0.053	0.082	0.142
SD	0.047	0.069	0.054	0.035	0.026						0.023	0.018	0.016	0.005	0.009	0.63	0.79	0.84	0.88	0.80	0.09	0.125	0.09	0.10	0.11					0.016
CV	13	11	9	8	8						20	21	18	32	121	9	8	8	10	14	7	7	5	6	8					19
N_{past}	41	33	33	30	30	5	7	7	7	7	11	11	11	9	9	42	35	36	36	34	21	22	22	24	24	10	6	6	5	6
$Median_{past}$	0.381	0.608	0.642	0.423	0.348	0.429	0.611	0.644	0.405	0.332	0.114	0.094	0.099	0.013	0.011	7.04	9.89	10.42	7.95	5.74	1.33	1.75	1.83	1.77	1.45	0.074	0.066	0.065	0.075	0.084
SD_{past}	0.042	0.043	0.045	0.023	0.021	0.060	0.037	0.048	0.038	0.043	0.025	0.019	0.020	0.004	0.007	0.70	0.70	0.65	0.82	0.84	0.12	0.14	0.16	0.12	0.15	0.007	0.030	0.037	0.009	0.028
$Mean_{NIST}$	0.335	0.602	0.647	0.408	0.337											6.61	10.11	10.93	8.00	6.09	1.3	1.8	1.8	1.8	1.5					
S_{rep}	0.015	0.030	0.009	0.005	0.005											0.12	0.19	0.20	0.02	0.22	0.0	0.0	0.0	0.0	0.0					
S_{het}	0.030	0.027	0.001	0.006	0.003											0.12	0.12	0.02	0.02	0.10	0.0	0.0	0.0	0.0	0.0					
S_{NIST}	0.033	0.040	0.009	0.008	0.006											0.17	0.22	0.20	0.03	0.24	0.0	0.0	0.0	0.0	0.0					
NAV	0.350	0.612	0.643	0.415	0.338	0.392	0.603	0.630	0.430	0.340	0.116	0.084	0.088	0.015	0.007	6.83	10.11	10.81	8.25	5.94	1.33	1.75	1.82	1.79	1.45	0.075	0.046	0.053	0.075	0.083
NAU	0.052	0.070	0.055	0.036	0.028						0.029	0.023	0.024	0.011	0.010	0.70	0.79	0.86	0.95	0.83	0.15	0.19	0.20	0.20	0.17					

C2

Round Robin LXIV Laboratory Results

	Total β-Carotene, µg/mL					trans-β-Carotene, µg/mL					Total cis-β-Carotene, µg/mL					Total α-Carotene, µg/mL					Total Lycopene, µg/mL					trans-Lycopene, µg/mL				
Lab	347	348	349	350	351	347	348	349	350	351	347	348	349	350	351	347	348	349	350	351	347	348	349	350	351	347	348	349	350	351
FSV-BA	0.348	0.111	0.122	0.081	0.084	0.315	0.107	0.118	0.076	0.080	0.033	0.004	0.004	0.005	0.004	0.016	0.073	0.080	0.026	0.009	0.079	0.446	0.517	0.328	0.259	0.034	0.259	0.302	0.170	0.125
FSV-BB	0.309	0.107	0.110	0.073	0.075	0.287	0.103	0.105	0.070	0.071	0.022	0.004	0.004	0.004	0.004	0.015	0.065	0.066	0.022	0.008	0.071	0.474	0.492	0.336	0.280	0.027	0.229	0.237	0.138	0.108
FSV-BC																														
FSV-BD																														
FSV-BE	0.175	0.084	0.079	0.068	!0.0387																									
FSV-BF	0.367	0.099	0.099	0.064	0.075																									
FSV-BG	0.354	0.146	0.153	0.087	0.093											0.017	0.085	0.091	0.024	0.010	0.076	0.591	0.621	0.361	0.325	0.035	0.335	0.356	0.184	0.153
FSV-BH	0.371	0.118	0.121	0.075	0.078	0.346	0.118	0.121	0.075	0.078	0.025	*nq*	*nq*	*nq*	*nq*	*nq*	0.086	0.090	*nq*	*nq*	0.075	0.520	0.537	0.347	0.289					
FSV-BI	0.325	0.117	0.116	0.074	0.070											0.014	0.072	0.078	0.025	0.007	0.066	0.396	0.416	0.257	0.202					
FSV-BJ	0.314	0.107	0.110	0.061	0.069											0.029	0.080	0.092	0.020	0.015	0.057	0.446	0.579	0.286	0.269					
FSV-BK																														
FSV-BL																														
FSV-BM																														
FSV-BN	0.320	0.108	0.108	0.072	0.077	0.279	0.100	0.100	0.066	0.069	0.038	0.006	0.004	0.003	0.005	-	0.072	0.078	0.024	0.008	0.069	0.428	0.440	0.288	0.248	0.023	0.234	0.241	0.140	0.114
FSV-BO	0.373	0.136	0.127	0.083	0.089	0.289	0.116	0.108	0.077	0.084	0.084	0.020	0.019	0.005	0.005	0.018	0.079	0.089	0.026	0.005	0.070	0.504	0.588	0.400	0.392					
FSV-BP	0.064	0.070	0.082	0.090	0.040											*nd*	0.031	0.038	0.014	0.013	0.177	0.155	0.152	0.080	0.040					
FSV-BQ																														
FSV-BR																														
FSV-BS	*≥0.314*	*≥0.123*	*≥0.115*	*≥0.057*	*≥0.067*	0.314	0.123	0.115	0.057	0.067						0.023	0.097	0.104	0.034	0.022	0.048	0.350	0.336	0.191	0.198					
FSV-BT	0.382	0.128	0.127	0.094	0.094	0.355	0.123	0.122	0.089	0.088	0.033	0.007	0.007	0.007	0.007	0.022	0.111	0.110	0.044	0.021	0.086	0.453	0.451	0.338	0.293	0.039	0.320	0.327	0.210	0.150
FSV-BU	0.341	0.126	0.135	0.083	0.083											0.018	0.092	0.097	0.027	0.011	0.071	0.573	0.570	0.367	0.296					
FSV-BV	0.330	0.123	0.124	0.067	0.067											0.018	0.090	0.090	0.024	0.007	0.070	0.564	0.572	0.310	0.247					
FSV-BW	0.400	0.130	0.145	0.083	0.082											0.009	0.059	0.065	0.019	0.004	0.090	0.616	0.695	0.400	0.310					
FSV-CD	0.422	0.197	0.201	0.168	0.182											0.019	0.061	0.071	0.022	<0.005						0.039	0.200	0.239	0.119	0.105
FSV-CE	0.280	0.031	0.140	0.110	0.070																									
FSV-CF																														
FSV-CG	0.301	0.112	0.113	0.066	0.054	0.274	0.103	0.106	0.061	0.048	0.028	0.009	0.007	0.006	0.006	0.021	0.091	0.091	0.028	0.007	0.074	0.490	0.496	0.289	0.191	0.033	0.288	0.297	0.149	0.090
FSV-CI	0.294	0.106	0.114	0.057	0.050											0.016	0.055	0.059	0.018	<0.009										
FSV-CW	0.340	0.132	0.135	0.084	0.090											0.020	0.085	0.086	0.026	0.007						0.025	0.272	0.279	0.147	0.128
FSV-CZ																														
FSV-DD																														
FSV-DQ	0.378	0.052	0.088	0.061	0.053											0.013	0.051	0.062	0.011	0.021	0.160	0.250	0.391	0.267	0.207					
FSV-DV	0.309	0.136	0.135	0.089	0.078																									
FSV-EE																														
N	22	22	22	22	21	8	8	8	8	8	7	6	6	6	6	16	19	19	18	16	16	16	16	16	16	8	8	8	8	8
Min	0.064	0.031	0.079	0.057	0.040	0.274	0.100	0.100	0.057	0.048	0.022	0.004	0.004	0.003	0.004	0.009	0.031	0.038	0.011	0.004	0.048	0.155	0.152	0.080	0.040	0.023	0.200	0.237	0.119	0.090
Median	0.335	0.115	0.122	0.078	0.077	0.302	0.112	0.111	0.073	0.075	0.033	0.007	0.005	0.005	0.005	0.018	0.079	0.086	0.024	0.009	0.073	0.464	0.507	0.319	0.264	0.034	0.266	0.288	0.148	0.120
Max	0.422	0.197	0.201	0.168	0.182	0.355	0.123	0.122	0.089	0.088	0.084	0.020	0.019	0.007	0.007	0.029	0.111	0.110	0.044	0.022	0.177	0.616	0.695	0.400	0.392	0.039	0.335	0.356	0.210	0.153
SD	0.045	0.017	0.018	0.014	0.011	0.028	0.012	0.010	0.009	0.009	0.007	0.003	0.002	0.001	0.001	0.003	0.019	0.017	0.004	0.005	0.008	0.082	0.103	0.051	0.065	0.007	0.047	0.050	0.025	0.020
CV	14	15	15	18	14	9	11	9	12	12	21	45	38	23	22	19	23	19	17	56	11	18	20	16	25	21	18	17	17	16
N_{past}	31	24	24	22	22	10	11	11	9	9	7	6	6	5	4	24	21	21	17	15	24	21	21	18	18	8	8	8	8	8
$Median_{past}$	0.341	0.114	0.122	0.075	0.075	0.320	0.110	0.118	0.069	0.075	0.027	0.006	0.007	0.006	0.005	0.019	0.075	0.080	0.027	0.009	0.071	0.483	0.512	0.323	0.276	0.038	0.250	0.264	0.146	0.114
SD_{past}	0.043	0.016	0.017	0.012	0.013	0.028	0.011	0.011	0.007	0.007	0.011	0.003	0.002	0.002	0.002	0.006	0.012	0.011	0.005	0.003	0.021	0.054	0.064	0.036	0.049	0.008	0.045	0.048	0.019	0.018
$Mean_{NIST}$	0.334	0.115	0.129	0.079	0.081											0.015	0.078	0.079	0.032	0.010	nq	0.438	0.432	0.301	0.273					
S_{rep}	0.008	0.005	0.004	0.002	0.003											0.002	0.004	0.003	0.003	0.001		0.012	0.004	0.026	0.003					
S_{het}	0.020	0.000	0.006	0.000	0.001											0.001	0.003	0.001	0.001	0.002		0.005	0.012	0.002	0.002					
S_{NIST}	0.022	0.005	0.008	0.002	0.003											0.002	0.005	0.003	0.003	0.002		0.013	0.013	0.026	0.003					
NAV	0.334	0.115	0.125	0.079	0.079	0.302	0.112	0.111	0.073	0.075	0.033	0.007	0.005	0.005	0.005	0.017	0.079	0.083	0.028	0.009	0.073	0.451	0.469	0.310	0.268	0.034	0.266	0.288	0.148	0.120
NAU	0.048	0.019	0.021	0.014	0.014	0.032	0.014	0.014	0.011	0.011	0.012	0.003	0.003	0.003	0.003	0.007	0.023	0.025	0.010	0.005	0.022	0.099	0.117	0.073	0.065	0.008	0.048	0.052	0.026	0.021

C3

Round Robin LXIV Laboratory Results

	Total β-Cryptoxanthin, µg/mL					Total α-Cryptoxanthin, µg/mL					Total Lutein, µg/mL					Total Zeaxanthin, µg/mL					Total Lutein&Zeaxanthin, µg/mL				
Lab	347	348	349	350	351	347	348	349	350	351	347	348	349	350	351	347	348	349	350	351	347	348	349	350	351
FSV-BA	0.009	0.064	0.069	0.049	0.046	0.003	0.029	0.032	0.022	0.019											0.036	0.101	0.104	0.092	0.082
FSV-BB	0.010	0.052	0.053	0.036	0.040	0.003	0.022	0.023	0.014	0.015	0.037	0.099	0.101	0.085	0.070	0.014	0.048	0.050	0.037	0.037	0.051	0.147	0.151	0.122	0.107
FSV-BC																									
FSV-BD																									
FSV-BE																									
FSV-BF																									
FSV-BG	0.009	0.063	0.069	0.040	0.050						0.025	0.084	0.088	0.058	0.045	0.008	0.032	0.027	0.018	0.009	0.030	0.106	0.108	0.073	0.054
FSV-BH	*nq*	0.059	0.062	0.041	0.046						0.013	0.046	0.036	0.045	0.045	0.006	0.019	0.015	0.015	0.020	0.019	0.065	0.051	0.060	0.065
FSV-BI	0.008	0.051	0.053	0.033	0.036						0.024	0.071	0.074	0.061	0.049	0.010	0.031	0.032	0.021	0.025	*0.034*	*0.102*	*0.106*	*0.082*	*0.074*
FSV-BJ	*nq*	0.037	0.045	0.026	0.032						0.023	0.071	0.079	0.062	0.054										
FSV-BK																									
FSV-BL																									
FSV-BM																									
FSV-BN	0.027	0.039	0.040	0.025	0.031	0.002	0.013	0.013	0.004	0.007	0.033	0.077	0.079	0.064	0.059	0.009	0.027	0.030	0.017	0.020	0.039	0.104	0.107	0.079	0.077
FSV-BO	0.010	0.048	0.051	0.034	0.044						0.021	0.072	0.073	0.066	0.057	0.006	0.026	0.027	0.022	0.023	0.027	0.098	0.101	0.088	0.080
FSV-BP	0.008	0.018	0.015	0.025	0.018																0.054	0.116	0.123	0.182	0.132
FSV-BQ																									
FSV-BR																									
FSV-BS	0.016	0.075	0.080	0.041	0.056																0.026	0.114	0.133	0.084	0.070
FSV-BT	0.010	0.049	0.050	0.036	0.040						0.029	0.086	0.075	0.072	0.098	0.012	0.045	0.037	0.029	0.040	0.041	0.134	0.115	0.103	0.140
FSV-BU	0.011	0.048	0.047	0.032	0.034																0.030	0.106	0.106	0.084	0.078
FSV-BV	0.007	0.055	0.055	0.033	0.034																0.037	0.125	0.128	0.102	0.091
FSV-BW	*nq*	0.052	0.057	0.031	0.038																0.033	0.103	0.108	0.090	0.075
FSV-CD	0.006	0.045	0.046	0.032	0.039																0.059	0.196	0.211	0.168	0.144
FSV-CE																									
FSV-CF																									
FSV-CG	0.015	0.077	0.077	0.049	0.047																0.044	0.138	0.142	0.109	0.095
FSV-CI											0.021	0.055	0.054	0.047	0.030	<0.006	0.014	0.018	0.012	0.010	*≤0.021*	*0.069*	*0.072*	*0.059*	*0.040*
FSV-CW	0.005	0.051	0.053	0.031	0.036						0.026	0.079	0.080	0.069	0.058	0.007	0.040	0.043	0.029	0.037	0.033	0.119	0.123	0.098	0.095
FSV-CZ																									
FSV-DD																									
FSV-DQ	0.016	0.035	0.056	0.034	0.033						0.010	0.098	0.101	0.091	0.077	0.019	0.027	0.027	0.024	0.025	*0.029*	*0.125*	*0.128*	*0.115*	*0.102*
FSV-DV																									
FSV-EE																									
N	15	18	18	18	18	3	3	3	3	3	11	11	11	11	11	9	10	10	10	10	17	18	18	18	18
Min	0.005	0.018	0.015	0.025	0.018	0.002	0.013	0.013	0.004	0.007	0.010	0.046	0.036	0.045	0.030	0.006	0.014	0.015	0.012	0.009	0.019	0.065	0.051	0.059	0.040
Median	0.010	0.051	0.053	0.034	0.039	0.003	0.022	0.023	0.014	0.015	0.024	0.077	0.079	0.064	0.057	0.009	0.029	0.029	0.021	0.024	0.034	0.110	0.112	0.091	0.081
Max	0.027	0.077	0.080	0.049	0.056	0.003	0.029	0.032	0.022	0.019	0.037	0.099	0.101	0.091	0.098	0.019	0.048	0.050	0.037	0.040	0.059	0.196	0.211	0.182	0.144
SD	0.004	0.009	0.009	0.006	0.008						0.005	0.010	0.008	0.008	0.013	0.003	0.009	0.007	0.008	0.010	0.008	0.017	0.016	0.019	0.019
CV	38	18	18	18	22						20	13	10	13	23	37	29	23	36	43	25	15	15	20	24
N_{past}	17	21	21	18	18	6	5	6	5	5	12	14	14	12	12	9	12	12	10	10	19	22	22	19	19
$Median_{past}$	0.012	0.052	0.055	0.036	0.040	0.006	0.024	0.026	0.014	0.015	0.028	0.079	0.081	0.065	0.059	0.011	0.030	0.031	0.021	0.024	0.039	0.109	0.114	0.098	0.088
SD_{past}	0.004	0.008	0.010	0.009	0.005	0.001	0.006	0.006	0.001	0.002	0.006	0.015	0.014	0.014	0.012	0.005	0.007	0.008	0.005	0.006	0.008	0.018	0.016	0.019	0.017
$Mean_{NIST}$	*nq*	0.049	0.049	0.034	0.033						0.028	0.075	0.076	0.076	0.051	0.013	0.030	0.031	0.021	0.023	*0.041*	*0.104*	*0.107*	*0.097*	*0.074*
S_{rep}		0.000	0.002	0.005	0.001						0.001	0.002	0.003	0.003	0.002	0.002	0.003	0.003	0.005	0.002	0.004	0.001	0.005	0.002	0.001
S_{het}		0.001	0.001	0.004	0.001						0.001	0.001	0.004	0.003	0.002	0.001	0.004	0.003	0.001	0.001	0.000	0.006	0.006	0.004	0.002
S_{NIST}		0.001	0.003	0.006	0.002						0.002	0.003	0.005	0.004	0.003	0.003	0.005	0.004	0.005	0.002	0.004	0.006	0.008	0.004	0.002
NAV	0.010	0.050	0.051	0.034	0.036	0.003	0.022	0.023	0.014	0.015	0.026	0.076	0.077	0.070	0.054	0.011	0.030	0.030	0.021	0.024	0.037	0.107	0.109	0.094	0.077
NAU	0.004	0.012	0.013	0.009	0.010						0.006	0.016	0.016	0.016	0.014	0.005	0.009	0.008	0.008	0.010	0.010	0.023	0.023	0.019	0.020

C4

Round Robin LXIV Laboratory Results

	Coenzyme Q10, µg/mL					Phylloquinone (K1), ng/mL				
Lab	347	348	349	350	351	347	348	349	350	351
FSV-BA										
FSV-BB										
FSV-BC										
FSV-BD										
FSV-BE	0.306	0.764	0.791	0.850	0.786	0.534	0.492	0.466	0.526	0.280
FSV-BF										
FSV-BG										
FSV-BH										
FSV-BI										
FSV-BJ	0.276	0.697	0.763	0.540	0.573					
FSV-BK										
FSV-BL										
FSV-BM										
FSV-BN										
FSV-BO										
FSV-BP										
FSV-BQ										
FSV-BR										
FSV-BS										
FSV-BT	0.127	0.223	0.252	0.349	0.354					
FSV-BU										
FSV-BV										
FSV-BW	0.356	0.783	0.787	0.633	0.602					
FSV-CD										
FSV-CE										
FSV-CF										
FSV-CG										
FSV-CI	0.310	0.760	0.800	0.850	0.750	0.625	0.421	0.386	0.424	0.206
FSV-CW	0.109	0.310	0.279	0.247	0.207					
FSV-CZ	0.323	0.848	0.696	0.743	0.626					
FSV-DD										
FSV-DQ										
FSV-DV										
FSV-EE	0.285	0.634	0.653	0.700	0.496					
N	8	8	8	8	8	2	2	2	2	2
Min	0.109	0.223	0.252	0.247	0.207	0.53	0.42	0.39	0.42	0.21
Median	0.296	0.729	0.730	0.667	0.588	0.58	0.46	0.43	0.48	0.24
Max	0.356	0.848	0.800	0.850	0.786	0.63	0.49	0.47	0.53	0.28
SD	0.055	0.160	0.170	0.206	0.146					
CV	19	22	23	31	25					
N_{past}	6	5	5	9	9					
$Median_{past}$	0.350	0.701	0.751	0.683	0.633					
SD_{past}	0.018	0.096	0.104	0.166	0.122					
$Mean_{NIST}$										
S_{rep}										
S_{het}										
S_{NIST}										
NAV	0.296	0.729	0.730	0.667	0.588					
NAU	0.055	0.160	0.170	0.206	0.146					

C5

Round Robin LXIV Laboratory Results

Analytes Reported By One Laboratory

Analyte	Code	347	348	349	350	351
Ubiquinol	FSV-BW	0.277	0.620	0.725	0.568	0.135
Ubiquinone	FSV-BW	*nq*	0.163	0.062	0.065	0.467

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	Standard deviation for (non-NIST) results: 0.741*(3rd Quartile - 1st Quartile)
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)
$Mean_{NIST}$	Mean of NIST results
S_{rep}	NIST's within-vial pooled standard deviation
S_{het}	NIST's among-vial pooled standard deviation
S_{NIST}	Combined standard deviation for NIST analyses: $(S_{rep}^2 + S_{het}^2)^{0.5}$
NAV	NIST Assigned Value = (Median + $Mean_{NIST}$)/2 for analytes reported by NIST analyst(s) = Median for analytes reported by ≥ 5 labs but not NIST
NAU	NIST Assigned Uncertainty: $(S^2 + S_{btw}^2)^{0.5}$ S is the maximum of (0.05*NAV, SD, S_{NIST}, eSD) and S_{btw} is the standard deviation between Median and $Mean_{NIST}$. The expected long-term SD, eSD, is defined in: Duewer et al. Anal Chem 1997;69(7):1406-1413.
-	Not analyzed
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
≥x	Concentration greater than or equal to x
!	Discrepant value: heterogeneous serum, damaged sample, malfunction, etc.
italics	Not explicitly reported but calculated by NIST from reported values

Comparability Summary

Lab	TR	aT	g/bT	bC	tbC	aC	TLy	TbX	TLu	TZ	L&Z
FSV-BA	1	1	1	1	1	1	1	2			1
FSV-BB	2	1	1	1	1	1	1	1	2	2	2
FSV-BC	1										
FSV-BD	1	1									
FSV-BE	2	1	1	3							
FSV-BF	1	1		1							
FSV-BG	1	1	1	2		1	1	1	1	1	1
FSV-BH	1	1	1	1	1	1	1	1	2	2	2
FSV-BI	1	1	1	1		1	1	1	1	1	1
FSV-BJ	1	1	1	1		2	1	1	1		
FSV-BK	3	1									
FSV-BL	1	1									
FSV-BM	1	1									
FSV-BN	2	2	1	1	1	1	1	3	1	1	1
FSV-BO	2	1	1	1	1	1	2	1	1	1	1
FSV-BP	1	2		4		2	4	3			3
FSV-BQ	1	2									
FSV-BR	1	3									
FSV-BS	4			1	1	2	2	2			1
FSV-BT	2	1	1	1	2	2	1	1	2	2	2
FSV-BU	1	2	1	1		1	1	1			1
FSV-BV	1	1	1	1		1	1	1			1
FSV-BW	1	1	1	1		1	2	1			1
FSV-CD	2	1	1	4		1		1			4
FSV-CE	1	4		3							
FSV-CF	2	1									
FSV-CG	1	2	2	1	2	1	1	2			2
FSV-CI	2	1	2	2		1			2	2	2
FSV-CW	1	1	1	1		1		1	1	2	1
FSV-CZ	2	2	3								
FSV-DD	2										
FSV-DQ		2	1	3		2	3	2	2	2	1
FSV-DV	1	2		1							
FSV-EE	2	1									
NIST	1	1	1	1		1	1	1	1	1	1
n	34	32	20	24	8	20	17	19	12	11	19

	TR	aT	g/bT	bC	tbC	aC	TLy	TbX	TLu	TZ	L&Z
% 1	62	69	85	71	75	75	71	68	58	45	63
% 2	32	25	10	8	25	25	18	21	42	55	26
% 3	3	3	5	13	0	0	6	11	0	0	5
% 4	3	3	0	8	0	0	6	0	0	0	5

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 RR64

Each participant in RR64 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 RR64:

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

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

Individualized Round Robin LXIV Report: FSV-BA

Summary

	Serum 347			Serum 348			Serum 349			Serum 350			Serum 351		
Analyte	You	NAV	n	You	NAV	n	You	NAV	n	You	NAV	n	You	NAV	n
Total Retinol	0.409	0.350	30	0.633	0.612	30	0.665	0.643	30	0.431	0.415	30	0.356	0.338	29
Retinyl Palmitate	0.09	0.12	9	0.1	0.1	9	0.1	0.1	9	0.02	0.02	7	0.03	0.01	5
α-Tocopherol	7.11	6.83	31	9.04	10.11	31	9.59	10.81	31	7.78	8.25	31	5.80	5.94	29
γ/β-Tocopherol	1.351	1.333	19	1.629	1.753	19	1.742	1.816	19	1.733	1.792	19	1.480	1.448	18
δ-Tocopherol	0.081	0.075	3	0.046	0.046	3	0.053	0.053	3	0.075	0.075	3	0.080	0.083	4
Total β-Carotene	0.348	0.334	22	0.111	0.115	22	0.122	0.125	22	0.081	0.079	22	0.084	0.079	21
trans-β-Carotene	0.315	0.302	8	0.107	0.112	8	0.118	0.111	8	0.076	0.073	8	0.080	0.075	8
Total cis-β-Carotene	0.033	0.033	7	0.004	0.007	6	0.004	0.005	6	0.005	0.005	6	0.004	0.005	6
Total α-Carotene	0.016	0.017	16	0.073	0.079	19	0.080	0.083	19	0.026	0.028	18	0.009	0.009	16
Total Lycopene	0.079	0.073	16	0.446	0.451	16	0.517	0.469	16	0.328	0.310	16	0.259	0.268	16
trans-Lycopene	0.034	0.034	8	0.259	0.266	8	0.302	0.288	8	0.170	0.148	8	0.125	0.120	8
Total β-Cryptoxanthin	0.009	0.010	15	0.064	0.050	18	0.069	0.051	18	0.049	0.034	18	0.046	0.036	18
Total α-Cryptoxanthin	0.003	0.003	3	0.029	0.022	3	0.032	0.023	3	0.022	0.014	3	0.019	0.015	3
Total Lutein&Zeaxanthin	0.036	0.037	17	0.101	0.107	18	0.104	0.109	18	0.092	0.094	18	0.082	0.077	18

D2

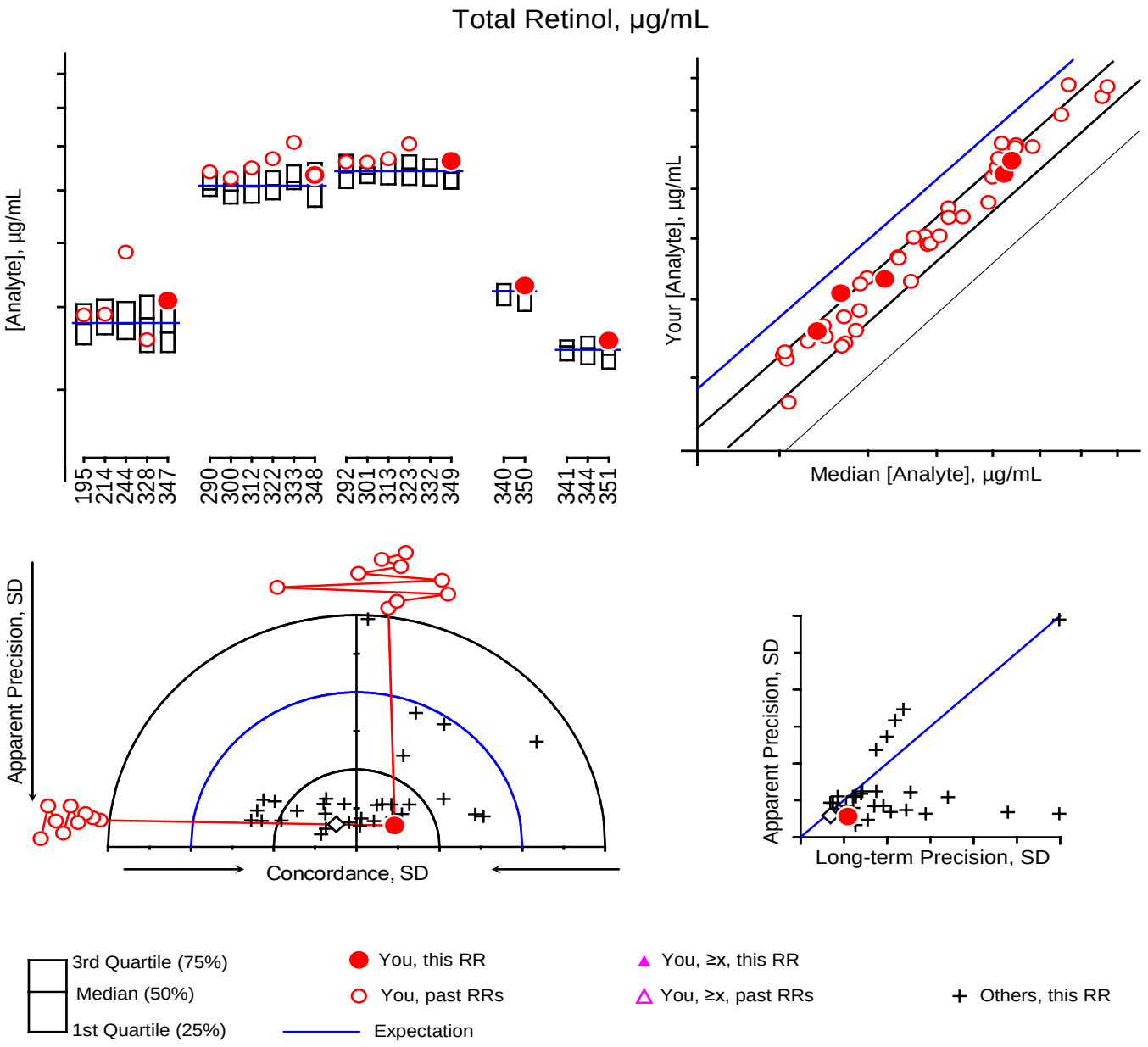
You : Your reported values for the listed analytes (micrograms/milliliter)
NAV : NIST Assigned Values, here equal to this RR's median
n : Number of non-NIST laboratories reporting quantitative values for this analyte in this serum

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 LXIV Report: FSV-BA



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
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#348	53:#290, 55:#300,57:#312, 59:#322:RR59,61:#333	Lyophilized, native, single-donor
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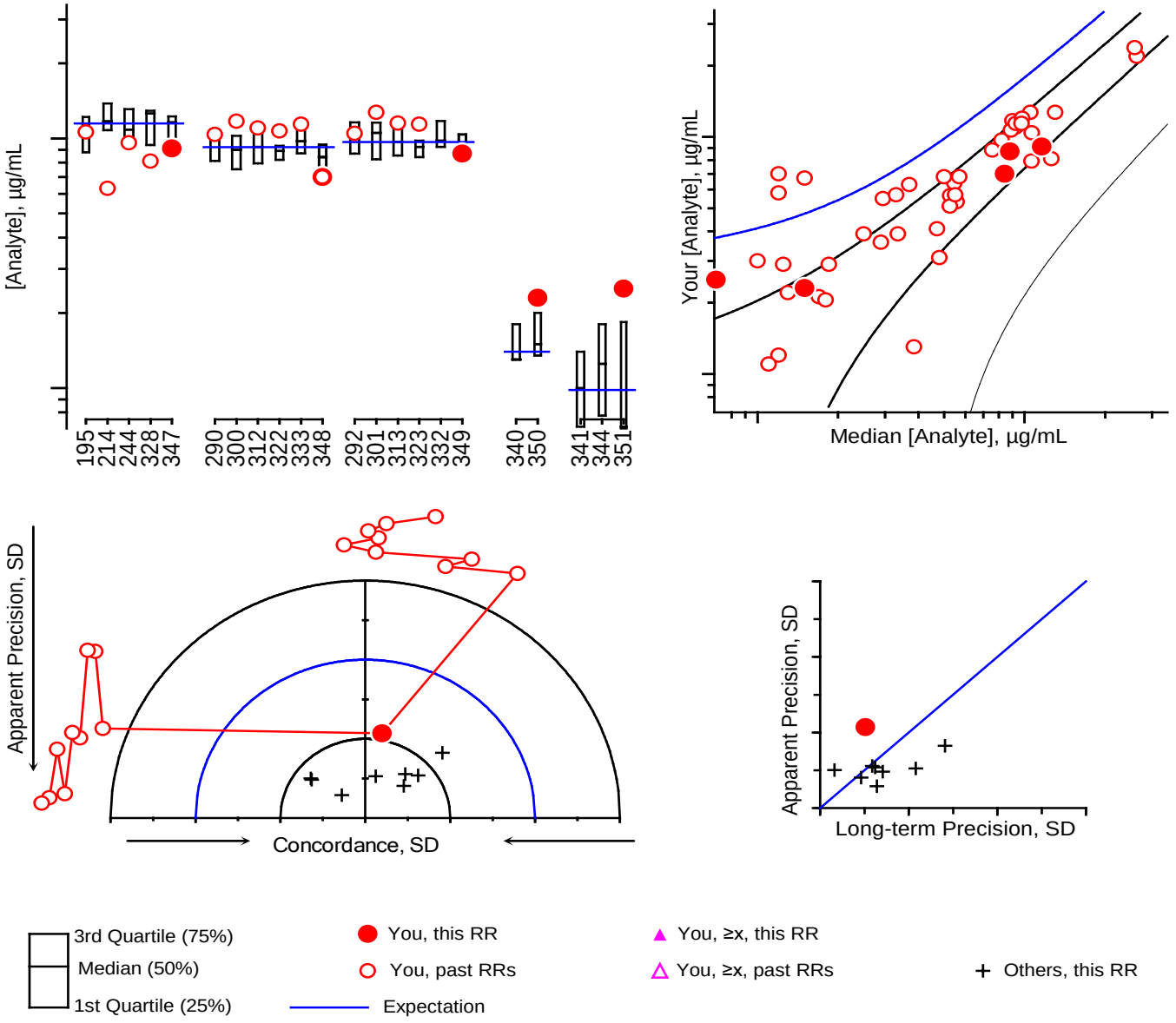
#351	63:#341, 63:#344	Fresh-frozen, native, multi-donor

Individualized RR LXIV Report: FSV-BA

Retinyl Palmitate, µg/mL



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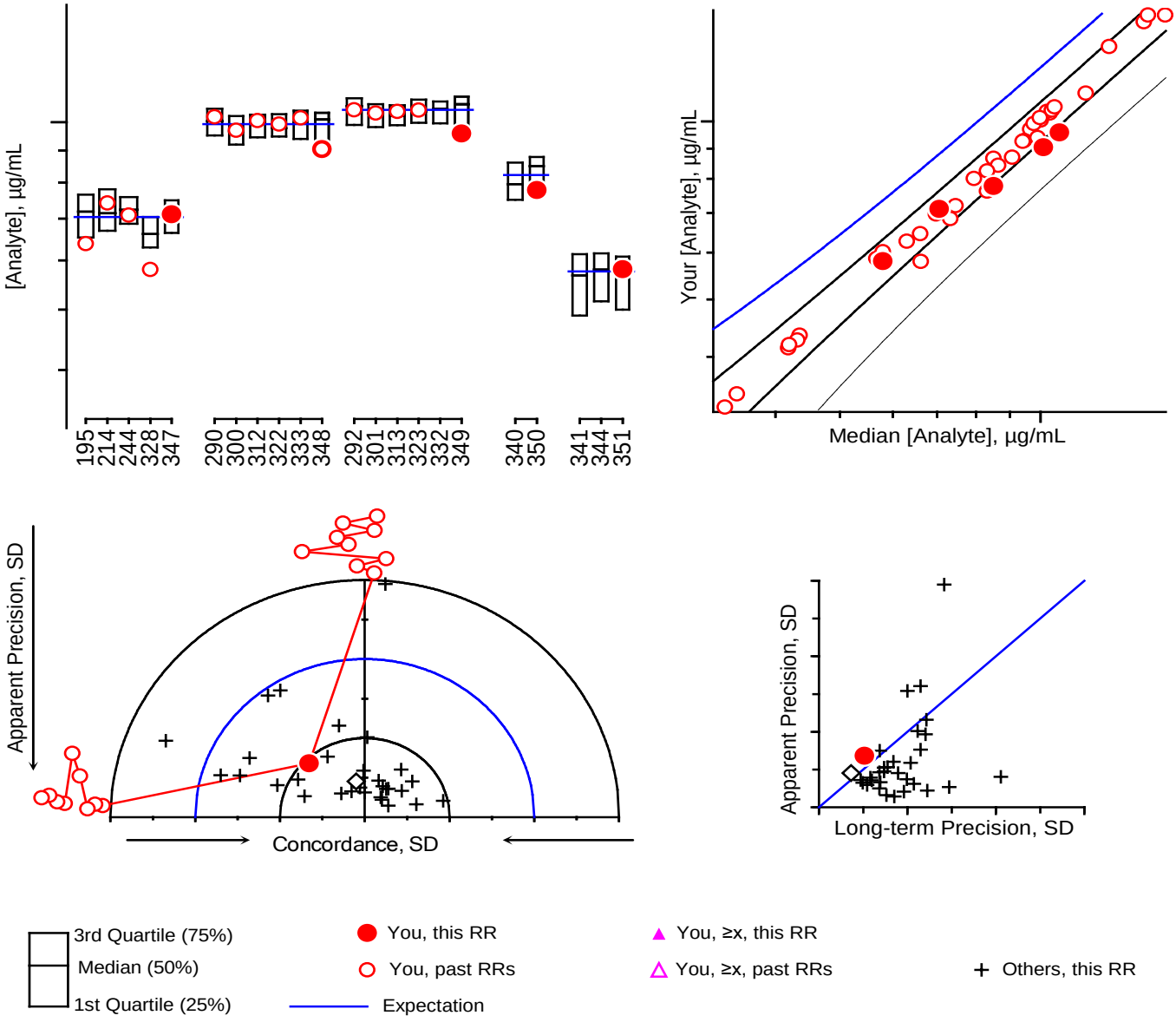
#351	63:#341, 63:#344	Fresh-frozen, native, multi-donor

Individualized RR LXIV Report: FSV-BA

α-Tocopherol, µg/mL



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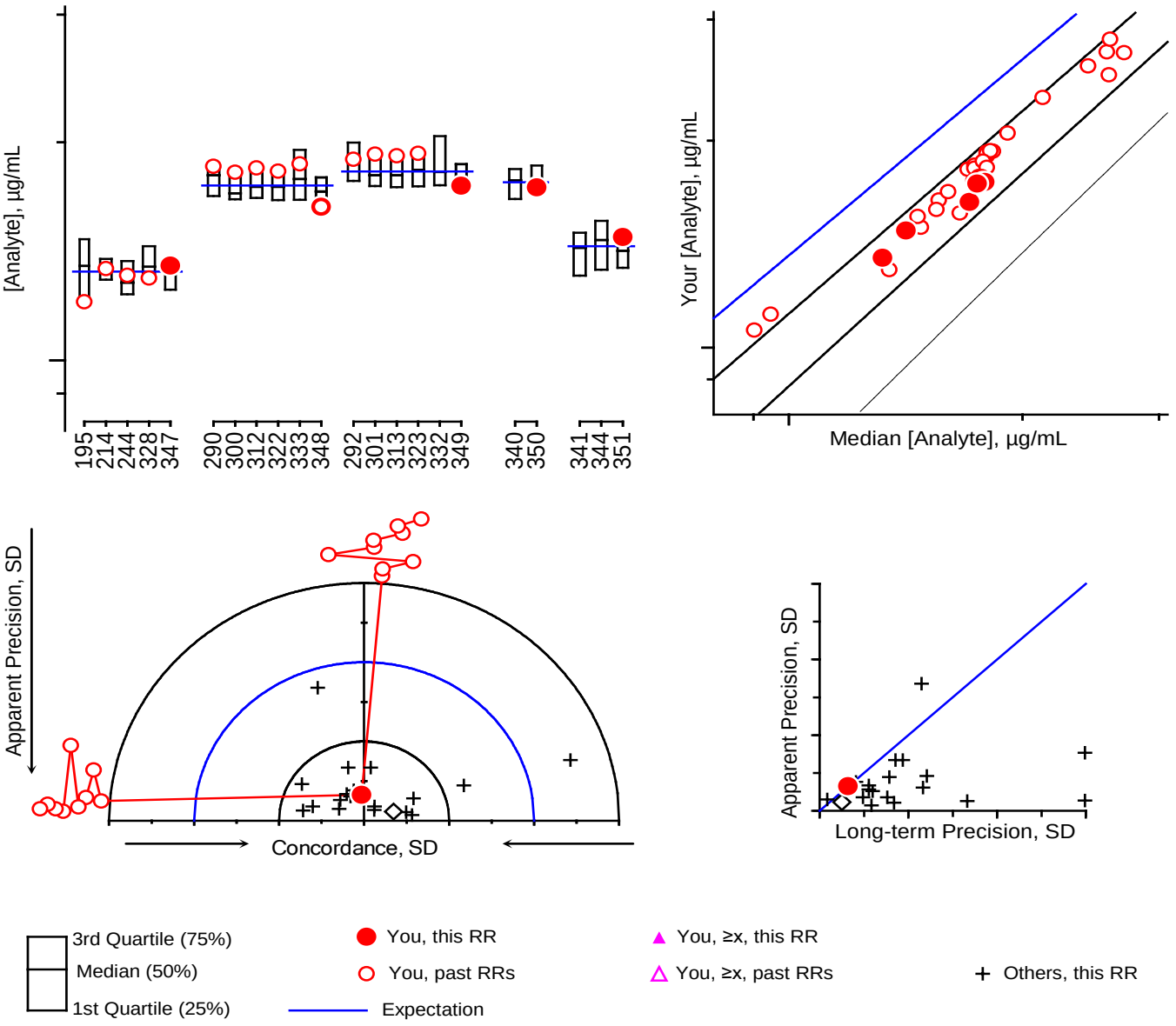
#351	63:#341, 63:#344	Fresh-frozen, native, multi-donor

Individualized RR LXIV Report: FSV-BA

γ/β-Tocopherol, µg/mL



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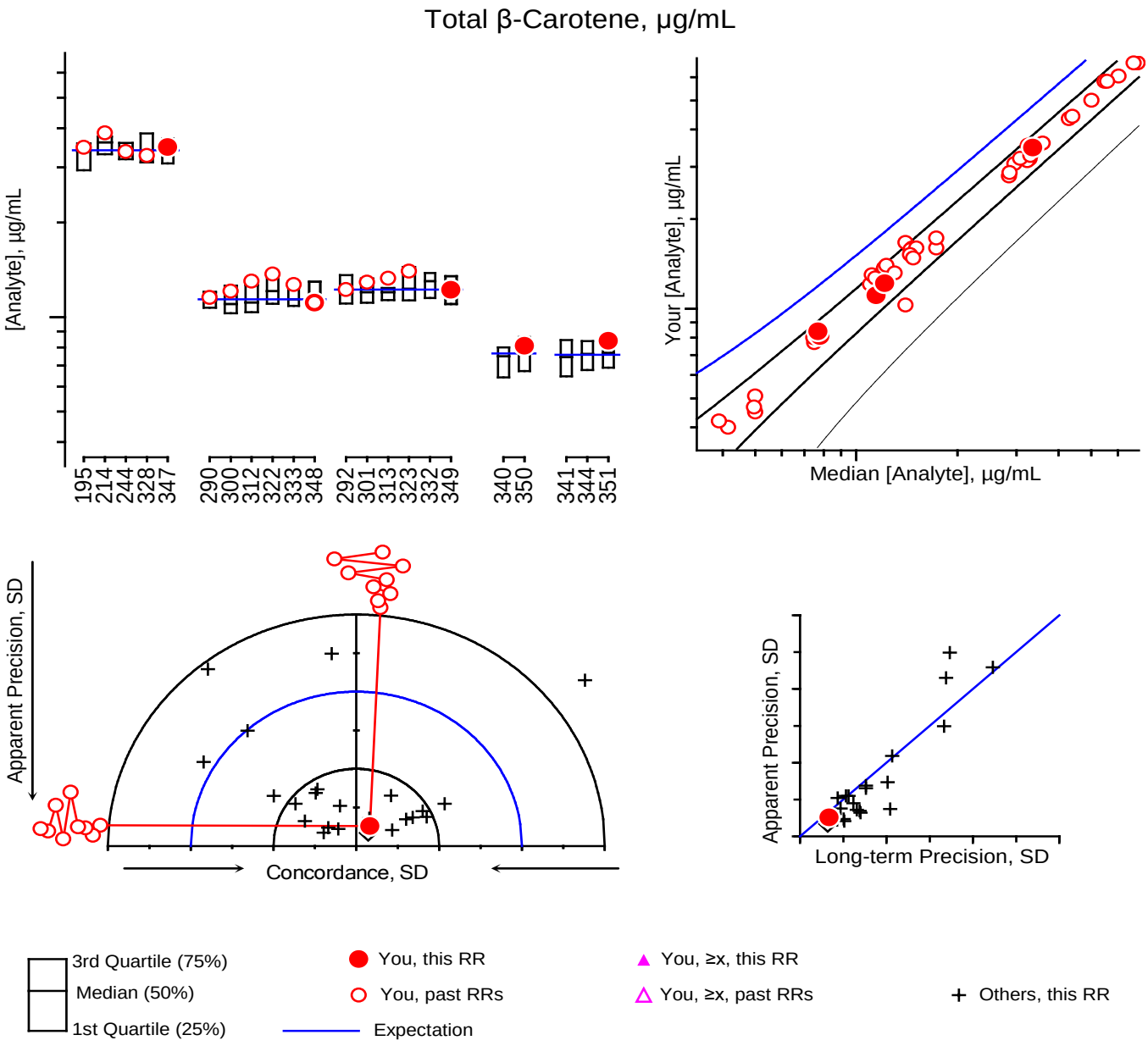
Individualized RR LXIV Report: FSV-BA



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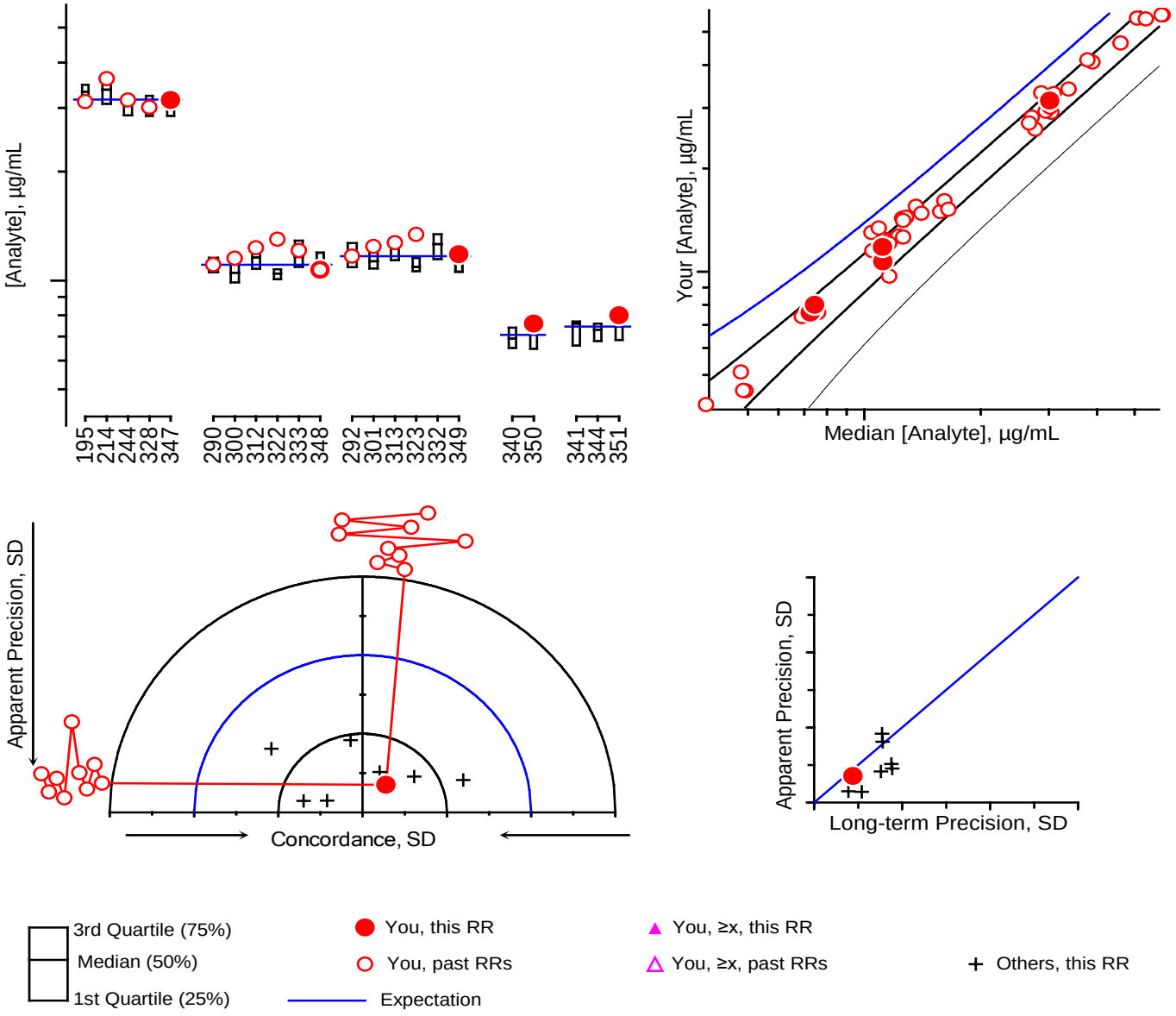
#351	63:#341, 63:#344	Fresh-frozen, native, multi-donor

Individualized RR LXIV Report: FSV-BA

trans-β-Carotene, µg/mL



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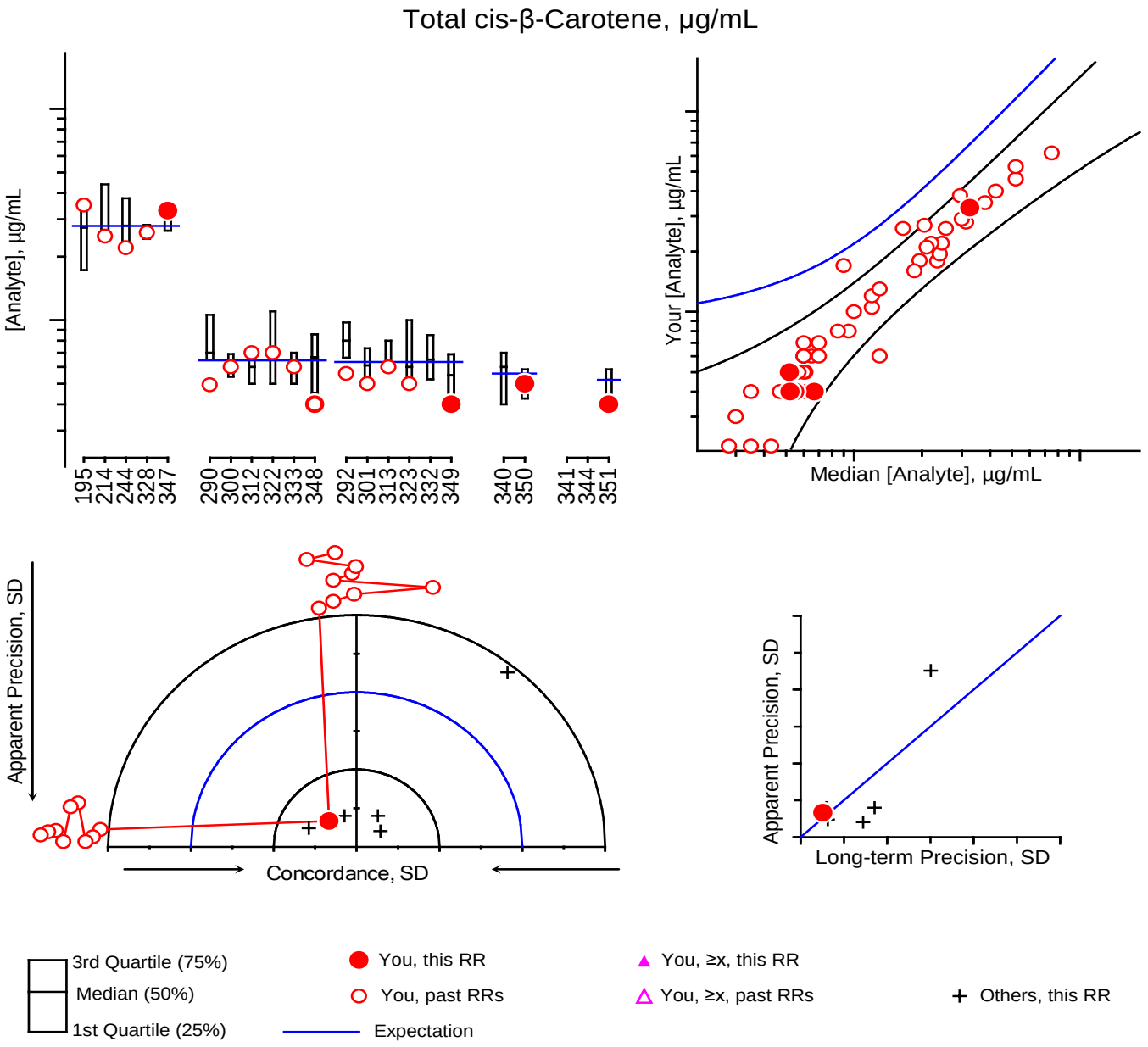
Individualized RR LXIV Report: FSV-BA



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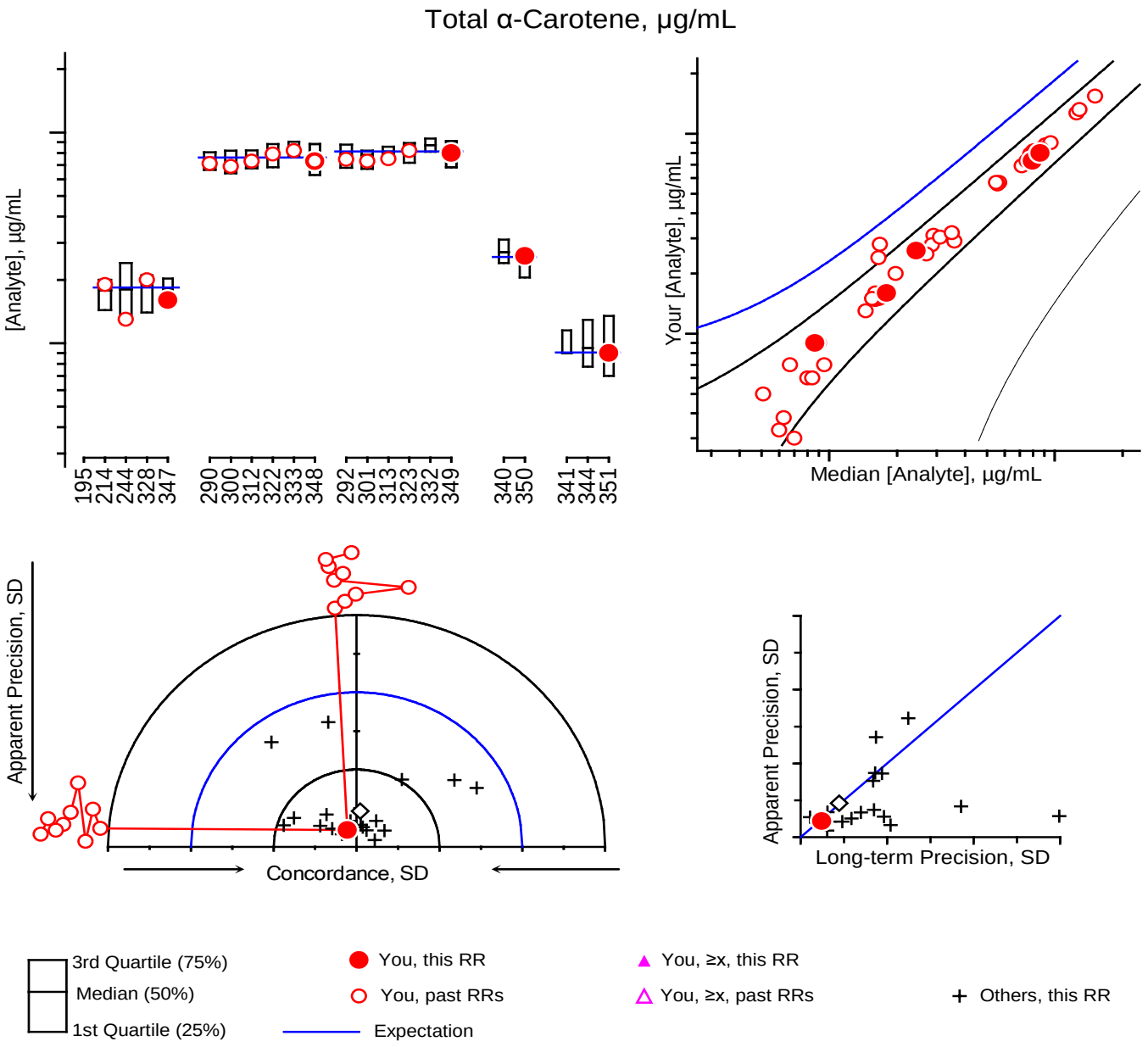
Individualized RR LXIV Report: FSV-BA



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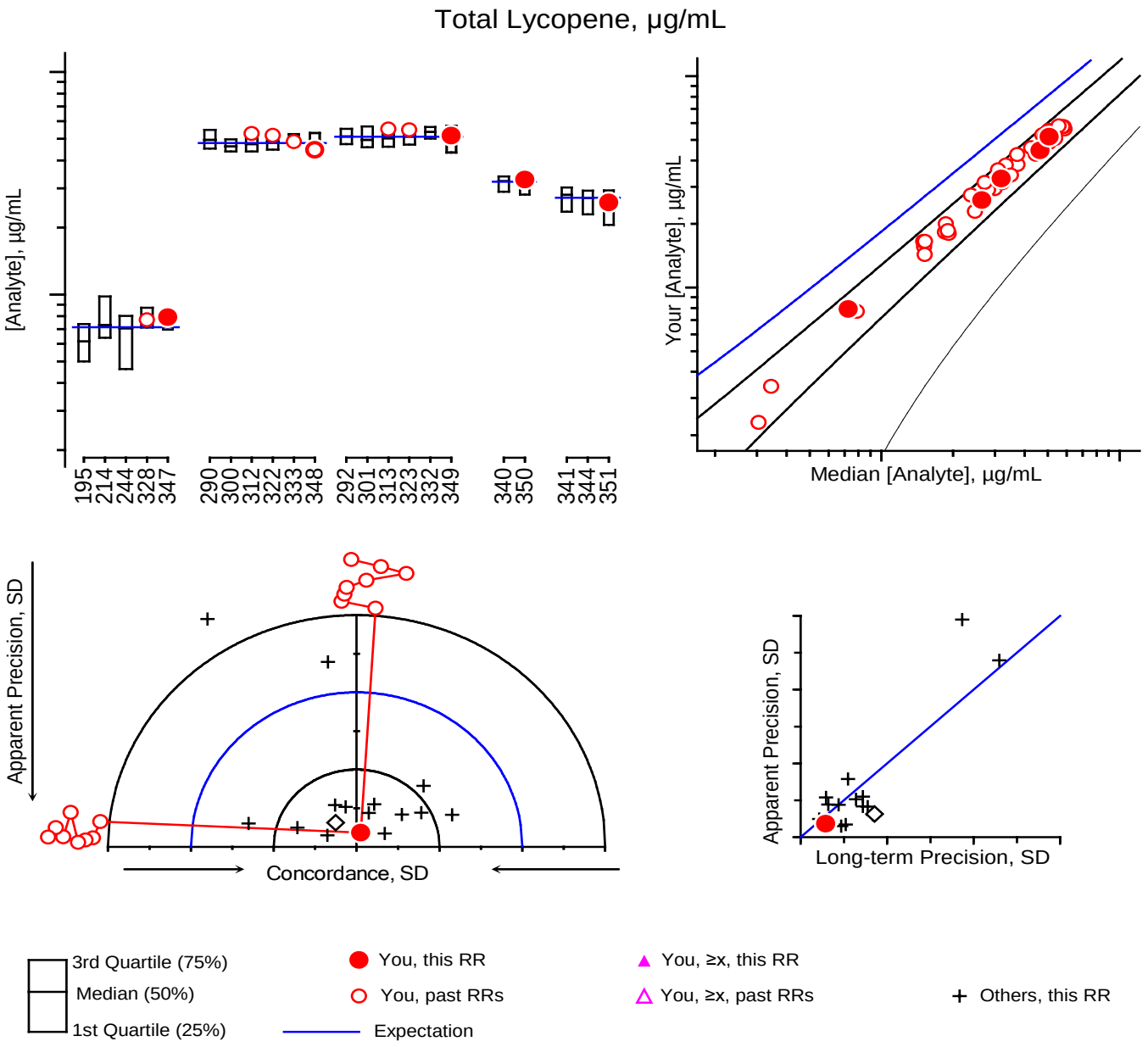

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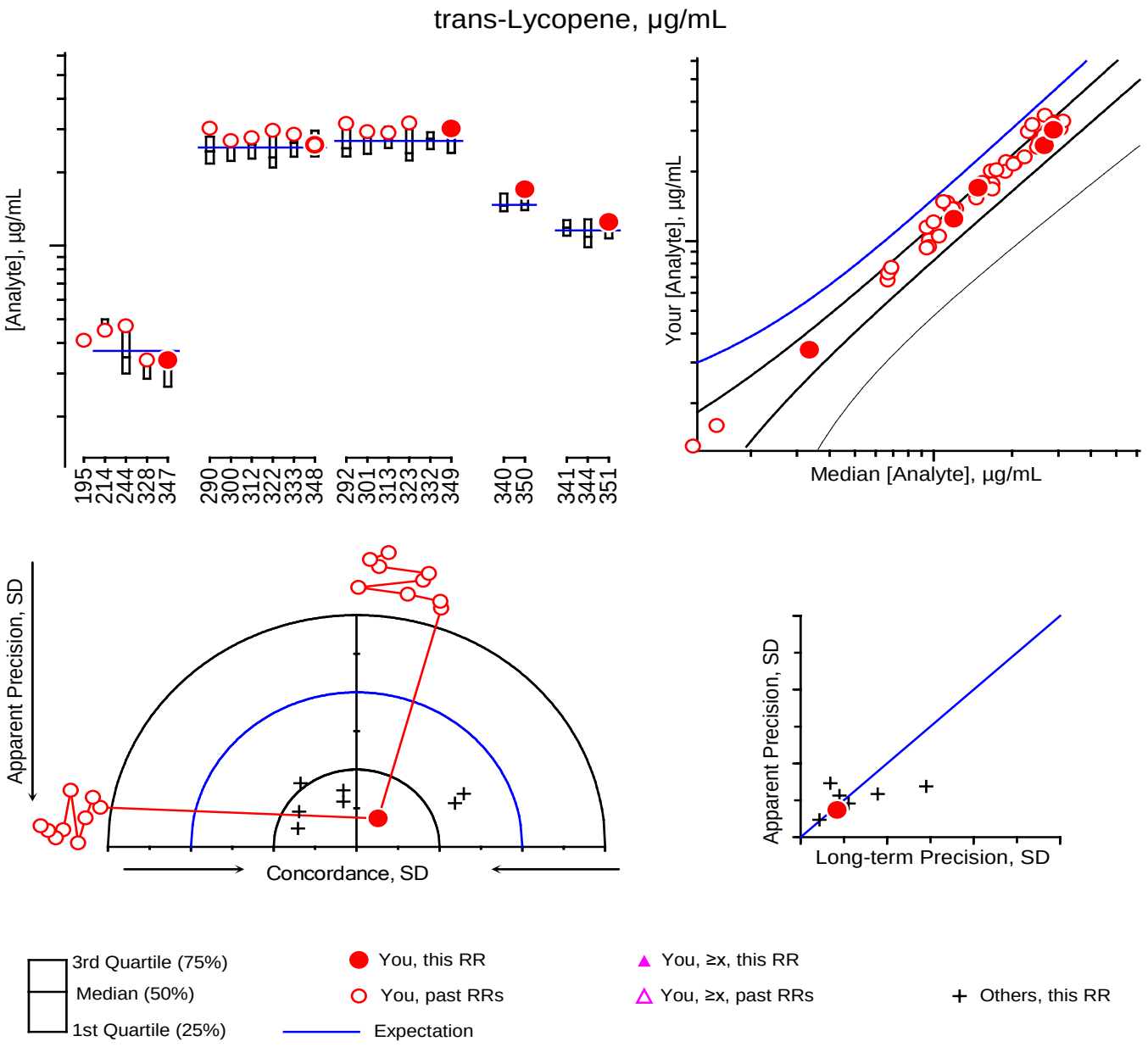

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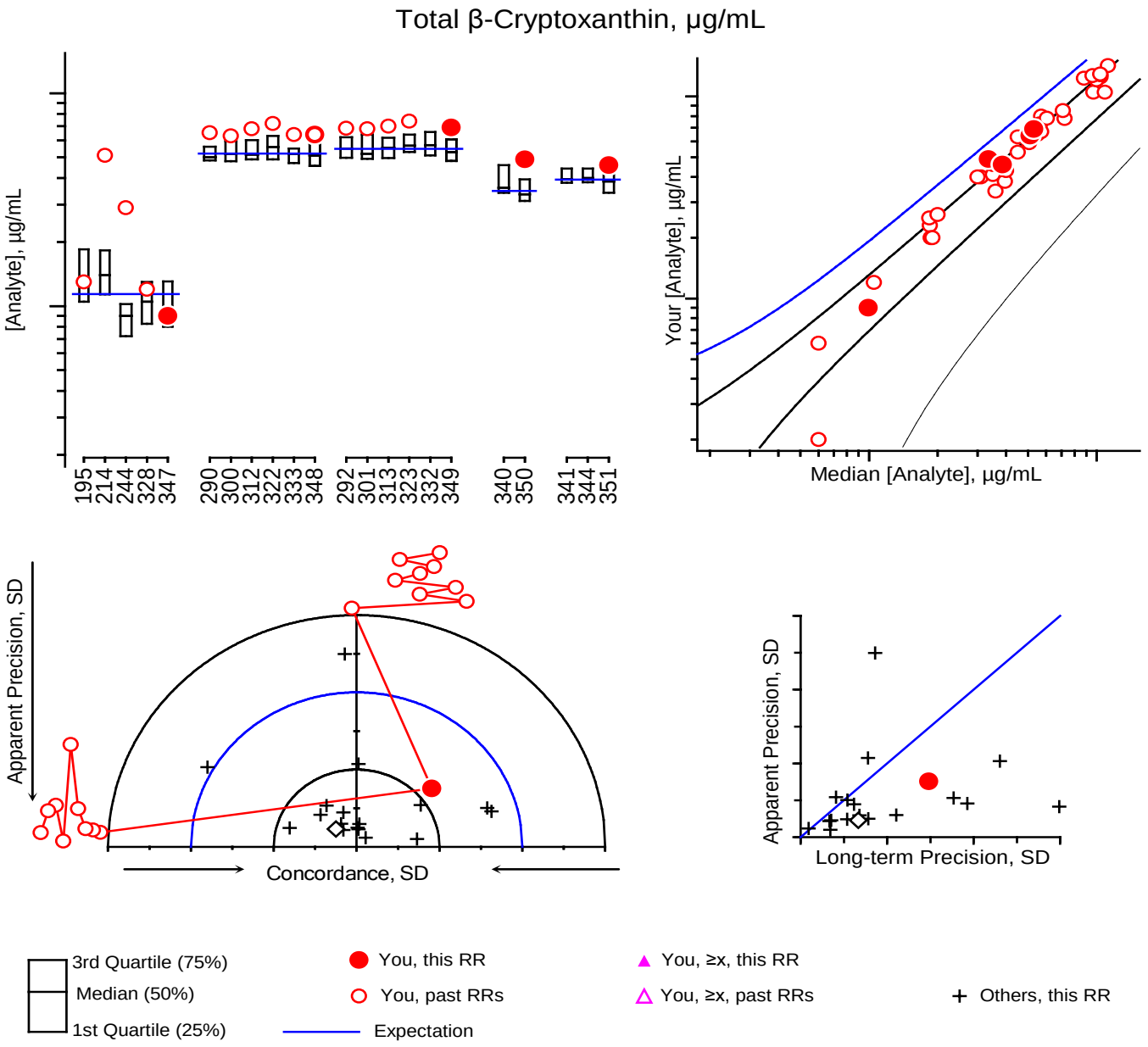

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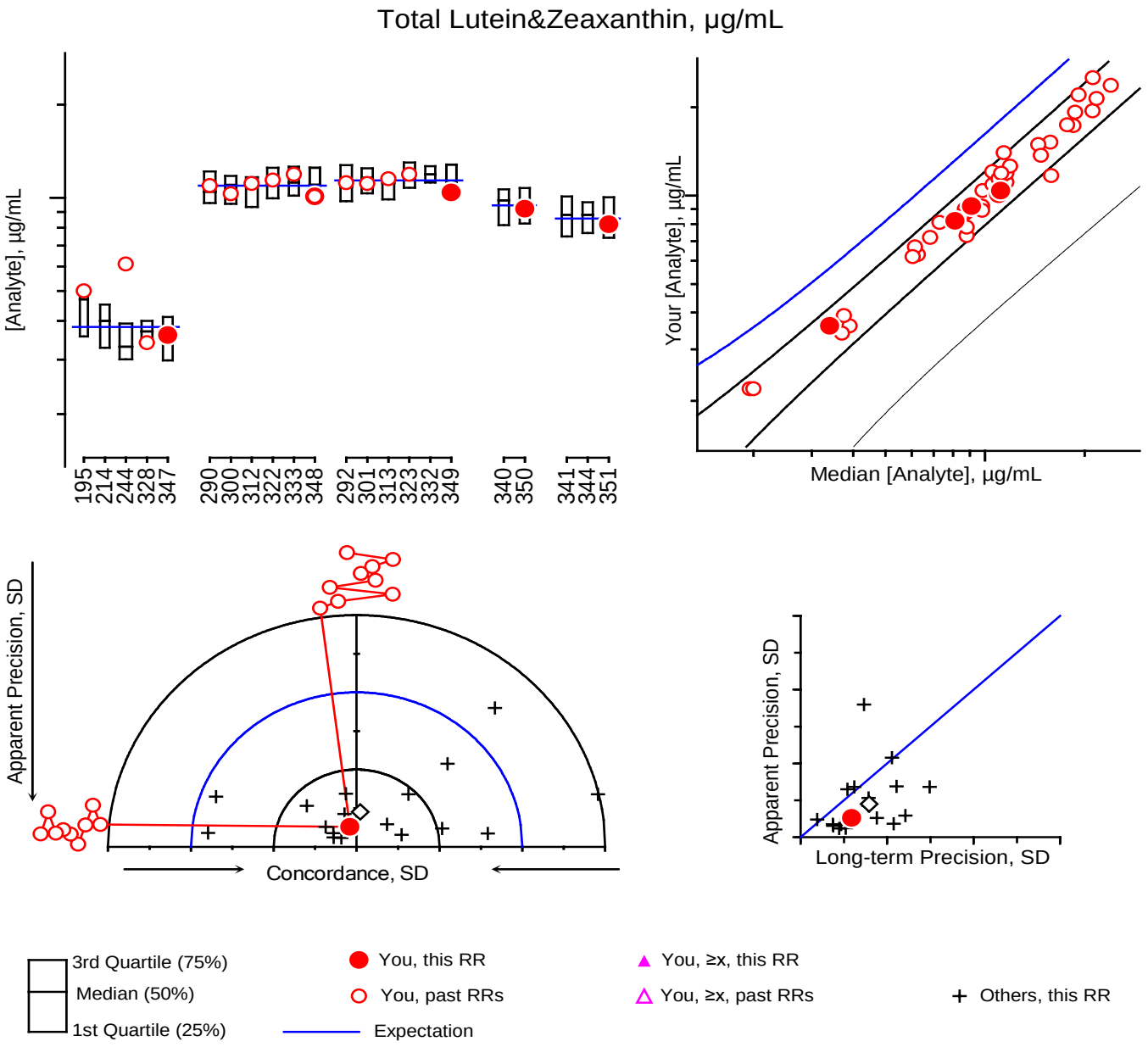
Individualized RR LXIV Report: FSV-BA



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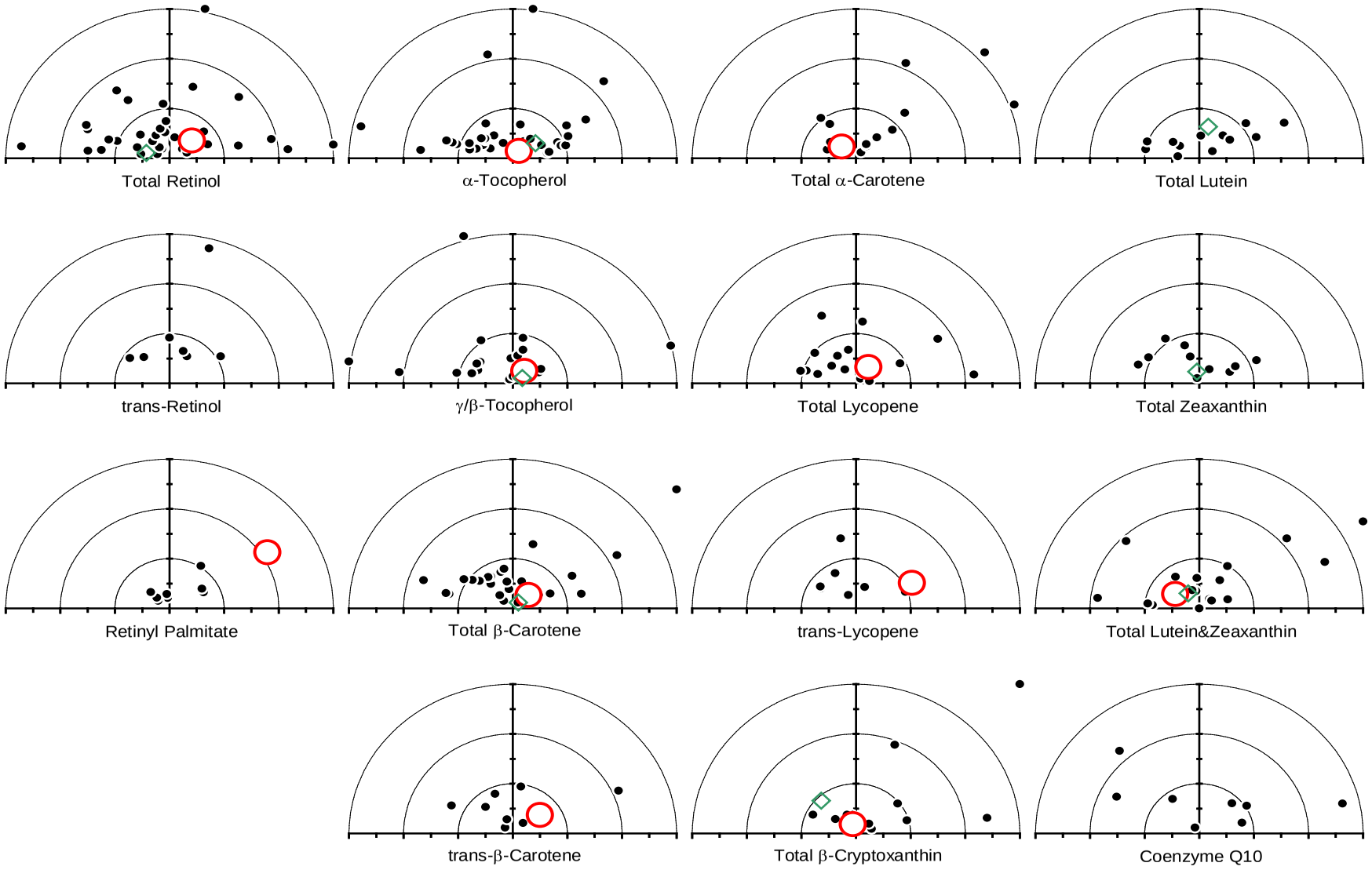
#347	31:#195, 35:#214, 43:#244, 60:#328	Lyophilized, multi-donor, aug(TR, RP,aT)
#348	53:#290, 55:#300,57:#312, 59:#322:RR59,61:#333	Lyophilized, native, single-donor
#349	53:#292, 55:#301,57:#313, 59:#323:RR59,61:#332	Fresh-frozen, native, single-donor
#350	63:#340	Plasma, fresh-frozen, native, multi-donor
#351	63:#341, 63:#344	Fresh-frozen, native, multi-donor

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



D15

Appendix E. Shipping Package Inserts for RR29

The following five items were included in each package shipped to an RR29 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 12, 2008

Dear Colleague:

The samples within this package constitute Vitamin C Round Robin 29 (RR29) of the 2008 Micronutrients Measurement Quality Assurance Program.

RR29 consists of four vials of frozen serum *test samples* (#15, #73, #90, and #111), 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 RR28 was mailed May 2, 2008. If you find your results for RR28 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 Ė 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 RR29 samples *before* **Sep 12, 2008**. 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 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
RR29 Report Form for Ascorbic Acid Solid Control Material Preparation
RR29 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:

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

   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 $[TAA]_{DS1}$ = (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 $[TAA]_{DS2}$ = 29.4 μmol/L and 0.125 mL should weigh 0.13 g and $[TAA]_{DS3}$ = 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 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 29

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* **12-Sep-2008**

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 29

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 #15			µmol/L of Sample
Serum Test Sample #73			µmol/L of Sample
Serum Test Sample #90			µmol/L of Sample
Serum Test Sample #111			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* **12-Sep-2008**

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 29

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 #15	Liquid frozen (1:1 serum:10% MPA)
VitC #73	Liquid frozen (1:1 serum:10% MPA)
VitC #90	Liquid frozen (1:1 serum:10% MPA)
VitC #111	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 RR29

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 25, 2008

Dear Colleague:

Enclosed is the summary report of the results for Round Robin 29 (RR29) 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 29 consisted of four *test samples* (#15, #73, #90, and #111), one *serum control material* (CS#2), and one *solid control material* for preparation of TAA control solutions. Details regarding the samples can be found in the enclosed report.

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

Samples for the first vitamin C round robin (RR30) of the 2009 M^2QAP will be shipped during the week of December 8, 2009. If you have questions or concerns regarding this report, please contact David Duewer at 301-975-3935; e-mail: david.duewer@nist.gov or me at 301-975-3120; e-mail: jbthomas@nist.gov; or fax: 301-977-0685.

Sincerely,

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

Enclosures

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

NIST

The NIST M^2QAP Vitamin C Round Robin 29 (RR29) 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 RR29 sample measurements.

Page	"All Lab" Report
1	A tabulation of results and summary statistics for Total Ascorbic Acid [TAA] in the RR29 samples and control/calibration solutions.

Serum-based Samples. One serum control and four unknowns were distributed in RR29.

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

S29:1 A "blank" stripped serum, ampouled in late 2001, previously distributed as sample S19:1 (RR19, 9/03), S21:1 (RR21, 9/04), S23:1 (RR23, 9/05), and S26:1 (RR26, 3/07).

S29:1 SRM 970 level 1, ampouled in mid-1998.

S29:2 SRM 970 level 2, ampouled in mid-1998.

S29:4 Serum 111, ampouled in 1989. It was used in some early experiments but was first distributed in the current MMQAP as S25:3 (RR25, 9/06).

Results.

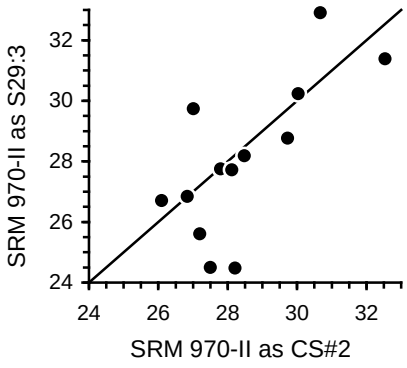
1) Nearly all participants who prepared the four 5% MPA control/calibration solutions (the three "Dilute Solutions" and the "Diluent") did so correctly. The criteria used to evaluate this success are: the density of the 5% MPA (≈1.03 gm/mL), the observed wavelength maximum of "Dilute Solution #1"(≈244 nm), the observed absorbance at that maximum (≈0.58 OD), the calculated $E^{1\%}$ #1"(≈560 dL/g·cm). On the evidence of MPA density, one participant prepared the solutions in 0% MPA.

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 reasonably well calibrated (intercepts range from -1.2 to 1.5 and slopes range from 0.88 to 1.04).

3) The Measured = p+q*Median regression parameters for samples S29:1 to S29:4 (columns 23 to 26 of the All Lab Report) mostly confirm the linearity of the measurement systems (R^2 close to 1 and RMS close to 0.0). As in RR28, there appears to be no correlation between regression intercepts and slopes and those for gravimetric calibration, again implying the existence of serum matrix effects.

4) There is no evidence of sample degradation in either of the SRM 970 materials.

5) There is little evidence for any "attraction to the known-value" in the analysis of the control material. However, comparison of the SRM 970-II material analyzed as CS#2 and as S29:3 (see Youden plot to the right) provides direct evidence that most of the observed among-participant differences in reported value result from systematic biases among the measurement systems.



Appendix G. "All-Lab Report" for RR29

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" 29 - September 2008

		Control / Calibration Samples											MPA	Dilute Solution 1			Samples									
		Grav, μmol/L			Measured, μmol/L				Measured = a + b*Grav				Density	Spectrophotometry			Measured, μmol/L						Measured = p + q*Median			
Lab	Date	Dil:1	Dil:2	Dil:3	Dil:1	Dil:2	Dil:3	MPA	Inter	Slope	R^2	RMS	g/mL	λ_{max}	A_{max}	$E^{1\%}$	CS#2	S29:1	S29:1	S29:2	S29:3	S29:4	Inter	Slope	R^2	RMS
VC-MA	11/08/08	58.2	28.9	14.8	59.9	30.8	15.5	0.0	0.34	1.03	1.000	0.6	1.036	243.	0.5670	553.2	27.2	0.0	0.0	8.3	25.6	13.0	0.74	0.92	0.988	1.4
VC-MB	20/06/08	59.1	29.4	14.6	60.7	30.5	14.2	0.0	-0.27	1.03	1.000	0.6	1.029	244.	0.5640	542.1	26.8	0.0	0.0	8.0	26.8	11.1	-0.15	0.97	1.000	0.2
VC-MC	20/06/08	56.2	28.6	14.3	58.2	28.8	13.4	0.0	-0.73	1.04	0.999	0.8	1.029	243.	0.5460	551.8	29.7	0.0	0.0	8.6	28.8	10.3	-0.57	1.04	0.996	1.0
VC-ME	19/09/08	57.3	28.8	14.2	57.6	31.2	14.6	0.0	0.58	1.01	0.998	1.3	1.031	243.	0.5955	590.3	30.0	4.2	4.2	9.8	30.2	12.3	2.60	0.96	0.983	1.8
VC-MG	21/08/08	61.1	31.7	17.0	62.2	31.2	14.8	0.0	-1.16	1.03	0.998	1.4	1.029	243.8	0.5980	555.3	28.1	0.0	0.0	6.9	27.7	10.0	-1.02	1.02	0.994	1.1
VC-MH	19/06/08	61.3	30.4	15.3	62.4	30.7	15.4	0.0	-0.13	1.02	1.000	0.2	1.032	244.1	0.6130	567.9	27.8	0.3	0.3	8.1	27.8	12.2	0.16	1.00	0.998	0.6
VC-MI	10/09/08	57.1	28.4	13.9	53.1	27.9	14.5	0.0	0.91	0.92	0.999	1.0	1.032				28.5	0.0	0.0	8.7	28.2	10.2	-0.45	1.02	0.996	0.9
VC-MJ	22/08/08	59.0	28.4	14.8	58.1	28.7	15.0	0.0	0.37	0.98	1.000	0.5	1.027	254[a]	0.356[a]	342.3[a]	32.5	4.9	4.9	11.7	31.4	17.5	4.78	0.97	0.987	1.6
VC-MK	12/06/08	60.4	30.6	14.9	54.7	28.5	15.4	1.0	1.53	0.88	0.999	0.7	0.999	244.	0.5865	551.0	27.0	5.7	5.7	13.9	29.7	12.8	5.14	0.87	0.976	1.9
VC-MN	08/09/08	60.4	29.8	14.9	60.9	31.0	15.3	0.8	0.77	1.00	1.000	0.4	1.038	243.7	0.5600	526.4	30.7	7.2	7.2	16.4	32.9	16.9	7.36	0.92	0.993	1.1
VC-MP	10/09/08																28.2	0.0	0.0	9.0	24.5	11.5	0.83	0.87	0.994	0.9
VC-MS	25/06/08																27.5	0.0	0.0	9.0	24.5	8.0	-0.11	0.87	0.980	1.8
VC-MU	27/08/08																26.1	0.6	0.6	7.4	26.7	6.2	-1.26	0.96	0.953	3.0
	N	10	10	10	10	10	10	10					N	10	8	8	8	13	13	13	13	13	13			
	Average	59.0	29.5	14.9	58.8	29.9	14.8	0.2					Average	1.028	243.6	0.5788	554.8	28.5	1.8	1.8	9.7	28.1	11.7			
	SD	1.8	1.1	0.8	3.1	1.3	0.7	0.4					SD	0.011	0.5	0.0229	18.6	1.8	2.7	2.7	2.7	2.5	3.1			
	Min	56.2	28.39	13.9	53.1	27.87	13.4	0.0					Min	0.999	243.0	0.5460	526.4	26.1	0.0	0.0	6.9	24.5	6.2			
	%25	57.5	28.69	14.4	57.8	28.74	14.6	0.0					%25	1.029	243.0	0.5630	548.8	27.2	0.0	0.0	8.1	26.7	10.2			
	Median	59.1	29.12	14.8	59.1	30.55	14.9	0.0					Median	1.030	243.8	0.5768	552.5	28.1	0.0	0.0	8.7	27.8	11.5			
	%75	60.4	30.24	14.9	60.9	30.92	15.3	0.0					%75	1.032	244.0	0.5961	558.5	29.7	4.2	4.2	9.8	29.7	12.8			
	Max	61.3	31.71	17.0	62.4	31.25	15.5	1.0					Max	1.038	244.1	0.6130	590.3	32.5	7.2	7.2	16.4	32.9	17.5			
	MADe	2.3	1.0	0.4	2.6	1.0	0.6	0.0					eSD	0.0	0.4	0.0263	9.8	1.6	0.0	0.0	1.0	2.9	2.0			
	CV	4	3	3	4	3	4						CV	0.25	0.17	4.6	1.8	6			12	11	17			

G2

Appendix H. Representative "Individualized Report" for RR29

Each participant in RR29 received an "Individualized Report" reflecting their reported results. The following two pages are the "Individualized Report" for participant "VC-MA".

Vitamin C "Round Robin" 29 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
03/09/06	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
03/04/08	28	HPLC-EC	1.035	243.0	0.572	562.2	0.7	1.03	0.999	0.99
08/11/08	29	HPLC-EC	1.037	243.0	0.567	553.2	0.3	1.03	1.000	0.64
		Mean	1.035	243.0	0.57	561.8				0.57
		SD	0.003	0.9	0.01	12.6				0.34
		CV	0.28	0.37	1.2	2.2				

[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	6	28.4	0.5	1.1
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				
08/11/08	29	CS#2	**27.2**	**27.2**	1.0	27.2	0.0				
11/18/02	16	S16:2	0.0	0.1	1.0	0.0	0.0	4	0.0	0.0	0.0
11/13/03	19	S19:1	na	na	1.0						
09/13/04	21	S21:1	na	na	1.0						
10/17/05	23	S23:1	0.0	0.0	1.0	0.0	0.0				
03/20/07	26	S26:1	0.0	0.0	1.0	0.0	0.0				
08/11/08	29	S29:1	**0.0**	**0.0**	1.0	0.0	0.0				
11/18/02	16	S16:1	8.8	8.8	1.0	8.8	0.0	6	8.5	0.2	0.3
11/13/03	19	S19:4	7.8	8.6	1.0	8.2	0.5				
02/23/04	20	S20:3	8.3	8.1	1.0	8.2	0.1				
10/17/05	23	S23:4	8.6	8.8	1.0	8.7	0.1				
08/28/06	25	S25:1	8.7	8.5	1.0	8.6	0.2				
08/11/08	29	S29:2	**8.3**	**8.4**	1.0	8.3	0.1				
03/20/03	18	S18:3	28.8	29.2	1.0	29.0	0.3	5	27.4	0.3	1.8
02/23/04	20	S20:4	25.9	25.2	1.0	25.5	0.5				
03/08/05	22	S22:4	29.4	29.4	1.0	29.4	0.0				
08/28/06	25	S25:2	27.6	27.4	1.0	27.5	0.1				
08/11/08	29	S29:3	**25.6**	**25.7**	1.0	25.6	0.1				
08/28/06	25	S25:3	12.3	12.4	1.0	12.4	0.1	2	12.7	0.2	0.5
08/11/08	29	S29:4	**12.8**	**13.3**	1.0	13.0	0.3				

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

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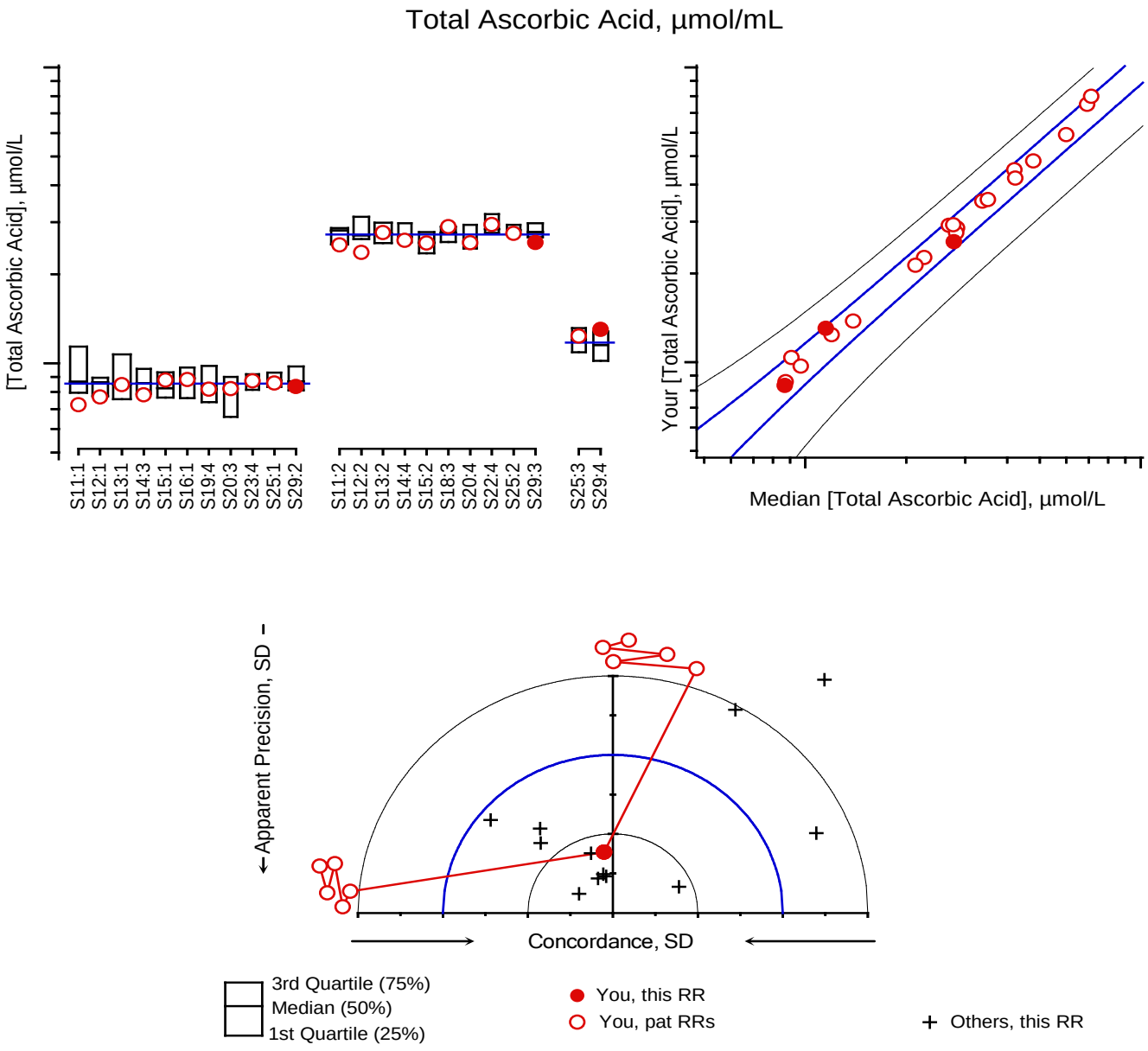
Vitamin C "Round Robin" 29 Report: Participant VC-MA



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
S29:1	VitC #15 (not displayed), serum blank previously distributed in RRs 16, 19, 21, 23, and 26
S29:2	VitC #73, previously distributed in RRs 11, 12, 13, 14, 15, 16, 19, 20, 23, and 25
S29:3	VitC #90, previously distributed in RRs 11, 12, 13, 14, 15, 18, 20, 22, and 25
S29:4	VitC #111, previously distributed in RR 25