NISTIR 7880-6

NIST Micronutrients Measurement Quality Assurance Program Summer 2009 Comparability Studies

Results for Round Robin LXVI Fat-Soluble Vitamins and Carotenoids in Human Serum and Round Robin 31 Ascorbic Acid in Human Serum

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http://dx.doi.org/10.6028/NIST.IR.7880-6

National Institute of Standards and Technology
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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 2009 MMQAP measurement comparability improvement studies: 1) Round Robin LXVI Fat-Soluble Vitamins and Carotenoids in Human Serum and 2) Round Robin 31 Total Ascorbic Acid in Human Serum. The materials for both studies were shipped to participants in June 2009; participants were requested to provide their measurement results by September 28, 2009.

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 LXVI: Fat-Soluble Vitamins and Carotenoids in Human Serum

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin LXVI comparability study (hereafter referred to as RR66) received five liquid-frozen human serum test samples for analysis. Unless multiple vials were previously requested, participants received one vial of each serum. These sera were shipped on dry ice to participants in June 2009. 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 RR66 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 31: Vitamin C in Human Serum

Participants in the MMQAP Vitamin C in Human Serum Round Robin 31 comparability study (hereafter referred to as RR31) 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 June 2009. 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 RR31 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 RR66

The following three items were included in each package shipped to an RR66 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-

June 1, 2009

Dear Colleague:

Enclosed are samples for the second fat-soluble vitamins and carotenoids in serum study (Round Robin LXVI) for the 2009 NIST Micronutrients Measurement Quality Assurance Program. The set of samples (Sera 357 - 361) consists of one vial of each of five liquid-frozen serum samples for analysis along with a form for reporting your results. These samples should be stored in the dark at or below –20 °C upon receipt. When reporting your results, please submit one value for each analyte for a given serum sample. If a value obtained is below your limit of quantification, please indicate this result on the form by using NQ *(Not Quantified)*. Results are due to NIST by **September 28, 2009**. Results received more than two weeks after the due date may not be included in the summary report for this round robin study. The feedback report concerning the study will be distributed in October 2009.

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

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

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

Sincerely,

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

Enclosures

Participant #: _____ Date: _____

Round Robin LXVI: Human Sera

NIST Micronutrients Measurement Quality Assurance Program

Analyte	357	358	359	360	361	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 (357 to 361) frozen when received? Yes | No

Comments:

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

Please return results by
28-Sep-2009

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

Participant #: _____ Date: _____

Fat-Soluble Vitamins Round Robin LXVI

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
#357	Liquid frozen	No	2 mL amber, red cap
#358	Liquid frozen	No	2 mL amber, blue cap
#359	Liquid frozen	No	2 mL amber, green cap
#360	Liquid frozen	No	2 mL amber, green cap
#361	Liquid frozen	No	10 mL amber, silver cap

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

1) Date this shipment arrived: _____

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

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

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

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

6) When do you anticipate analyzing these samples? _____

Your prompt return of this information is appreciated.

The M^2QAP Gang

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

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

Appendix B. Final Report for RR66

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-

October 30, 2009

Dear Colleague:

Enclosed is the summary report of the results for round robin LXVI (RR66) of the 2009 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 are in the process of finalizing the value-assignment for SRM 968e, Fat-Soluble Vitamins, Carotenoids, and Cholesterol in Human Serum. This SRM will consist of three different levels for the core analytes (retinol, α-tocopherol, and β-carotene). We will notify you when the SRM becomes available for purchase (estimated Spring 2010).

Samples for the first 2010 QA interlaboratory exercise will be shipped **during the week of December 7, 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 B. Thomas, M.B.A
Research Chemist
Analytical Chemistry Division
Chemical Science and Technology Laboratory

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

Cc: L.C. Sander

NIST

The NIST M^2QAP Round Robin LXVI (RR66) 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 RR66.

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

Results

1) Sera Stability. There was no significant change in the median level or measurement variability of any measurand in either of the two previously distributed materials. However, measurement variability for some analytes appears to have increased in the SRM 968d material. We will closely monitor the stability of this material; we hope to replace it with in the very near future.

2) Candidate SRM 968e. Sera #357 to #359 are the components of candidate SRM 968e. All three of these materials were prepared by blending commercially available materials without spiking. The materials were designed to represent relatively low, middling, and relatively high levels of retinol, α-Tocopherol and β-carotene. We anticipate this material being available for purchase early-2010.

3) Environmental Stability. The initial set of samples for Lab 110 arrived thawed. After a replacement set was successfully delivered, both sets (listed as 110 and 110.1) were analyzed under reproducibility conditions. The is appreciable difference in the reported values for any analyte in any of the materials.

Appendix C. "All-Lab Report" for RR66

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 LXVI Laboratory Results

	Total Retinol, µg/mL					trans-Retinol, µg/mL					Retinyl Palmitate, µg/mL					α-Tocopherol, µg/mL					γ/β-Tocopherol, µg/mL				
Lab	357	358	359	360	361	357	358	359	360	361	357	358	359	360	361	357	358	359	360	361	357	358	359	360	361
110.2???	0.328	0.433	0.522	0.608	0.275						0.002	0.020	0.089	0.042	0.002	7.09	10.03	17.00	9.68	4.91	1.18	0.98	1.58	1.59	0.83
110.3???	0.293	0.463	0.522	0.623	0.281						0.006	0.021	0.093	0.043	0.000	6.28	10.66	17.54	10.00	5.07	1.13	1.05	1.53	1.66	0.82
119.1???	*≥0.274*	*≥0.380*	*≥0.480*	*≥0.571*	*≥0.289*	0.274	0.380	0.480	0.571	0.289						6.80	10.51	18.74	11.11	6.14	1.83	1.48	0.24	2.39	1.43
FSV-BA	0.366	0.523	0.675	0.770	0.357						0.019	0.063	0.155	0.044	0.029	6.62	9.99	18.18	10.16	5.88	2.04	1.58	2.54	2.69	1.67
FSV-BB	0.357	0.522	0.655	0.733	0.361						0.007	0.021	0.070	0.036	0.007	6.49	9.94	17.88	10.14	5.81	1.82	1.43	2.27	2.41	1.47
FSV-BC	0.373	0.530	0.670	0.772	0.356																				
FSV-BD	0.387	0.531	0.732	0.851	0.375											7.10	10.70	20.50	11.90	6.50					
FSV-BE	0.343	0.504	0.675	0.713	0.287											5.66	9.22	18.15	9.34	2.41	1.77	1.44	2.49	2.41	1.30
FSV-BF	0.390	0.560	0.710	0.840	0.370											7.10	10.50	18.80	10.90	5.00					
FSV-BG	0.350	0.497	0.645	0.748	0.336						0.016	0.049	0.219	0.045	0.024	6.43	9.95	18.40	10.41	5.86	1.84	1.44	2.34	2.50	1.46
FSV-BH	0.347	0.511	0.619	0.720	0.315											6.98	10.64	18.71	10.45	5.67	2.29	1.75	2.70	2.91	1.66
FSV-BJ	0.331	0.473	0.626	0.718	0.321						*nq*	*nq*	0.092	*nq*	*nq*	6.08	9.99	18.34	10.24	5.50	1.79	1.43	2.28	2.48	1.50
FSV-BK	0.364	0.494	0.659	0.722	0.450											6.86	10.46	19.26	10.83	6.37					
FSV-BL	0.320	0.430	0.540	0.600	0.290											7.32	11.20	19.38	11.20	6.46					
FSV-BM	0.370	0.480	0.690	0.730	0.370											7.10	10.20	19.70	10.90	6.20					
FSV-BN	0.316	0.428	0.550	0.638	0.291						0.003	0.014	0.074	0.032	0.003	6.10	8.80	15.53	9.27	4.51	1.37	1.01	1.62	1.83	1.08
FSV-BNa	0.309	0.453	0.552	0.630	0.284						0.005	0.017	0.081	0.033	*nd*	5.96	9.23	15.44	9.01	4.48	1.36	1.08	1.64	1.78	1.06
FSV-BO	0.381	0.549	0.668	0.925	0.371											6.60	10.20	18.10	10.70	5.70	1.60	1.30	2.00	2.10	1.30
FSV-BP	0.351	0.497	0.654	0.752	0.314											6.52	9.71	17.53	8.70	5.24					
FSV-BQ	0.440	0.660	0.840	0.960	0.430											8.40	12.20	22.20	13.50	7.70					
FSV-BR	0.360	0.530	0.720	0.720	0.340											6.70	11.00	19.40	10.75	4.05					
FSV-BS	*≥0.655*	*≥0.554*	*≥0.455*	*≥0.421*	*≥1.075*	0.655	0.554	0.455	0.421	1.075															
FSV-BT	*≥0.330*	*≥0.428*	*≥0.549*	*≥0.704*	*≥0.375*	0.330	0.428	0.549	0.704	0.375						6.48	10.02	17.76	10.10	5.38	1.70	1.37	2.30	2.23	1.23
FSV-BU	0.377	0.547	0.677	0.706	0.291											6.40	9.54	18.56	8.99	4.46	1.67	1.17	2.00	2.26	1.26
FSV-BV	0.400	0.559	0.627	0.719	0.330											7.730	10.990	16.820	10.760	5.870	1.89	1.39	1.95	2.37	1.39
FSV-BW	0.350	0.495	0.655	0.732	0.327						0.008	0.026	0.101	0.053	0.005	6.14	9.59	18.00	10.06	4.91	1.69	1.27	2.08	2.31	1.25
FSV-CC	0.393	0.520	0.688	0.758	0.303	0.352	0.487	0.638	0.693	0.243						7.73	11.54	20.42	11.73	*nq*					
FSV-CD	0.320	0.450	0.580	0.670	0.300						0.011	<0.01	0.057	0.051	<0.01	7.54	11.48	20.58	11.57	6.74	1.61	1.20	1.97	2.11	1.30
FSV-CE	0.353	0.451	0.554	0.601	0.310											8.52	12.96	23.96	12.94	7.12					
FSV-CF	0.361	0.506	0.664	0.645	0.332											7.60	11.20	19.50	10.40	6.80					
FSV-CG	0.308	0.459	0.497	0.688	0.337											5.47	8.76	13.35	8.88	4.47	1.50	1.24	1.83	2.13	1.29
FSV-CI	0.344	0.507	0.643	0.715	0.340						0.008	*nd*	0.091	0.043	0.013	6.84	10.87	19.42	10.74	5.52	1.72	1.34	2.18	2.26	1.31
FSV-CW	0.493	0.624	0.784	0.993	0.700						0.008	0.019	0.061	0.046	0.029	8.15	12.01	21.03	12.09	6.11	2.01	1.55	2.50	2.62	1.50
FSV-CZ	0.311	0.436	0.554	0.605	0.296											6.50	9.89	18.03	10.42	4.98	1.75	1.30	2.13	2.22	1.29
FSV-DD	0.330	0.480	0.650	0.690	0.310																				
FSV-DV	*≥0.330*	*≥0.471*	*≥0.603*	*≥0.675*	*≥0.256*	0.330	0.471	0.603	0.675	0.256						6.10	9.20	15.60	8.90	3.00					
FSV-EE	0.339	0.479	0.601	0.678	0.312											6.90	10.40	19.60	12.90	7.00					
N	33	33	33	33	33	5	5	5	5	5	11	9	12	11	9	34	34	34	34	33	21	21	21	21	21
Min	0.293	0.428	0.497	0.600	0.275	0.274	0.380	0.455	0.421	0.243	0.002	0.014	0.057	0.032	0.000	5.47	8.76	13.35	8.70	2.41	1.13	0.98	0.24	1.59	0.82
Median	0.351	0.497	0.654	0.719	0.327	0.330	0.471	0.549	0.675	0.289	0.008	0.021	0.090	0.043	0.007	6.75	10.30	18.48	10.44	5.67	1.72	1.34	2.08	2.26	1.30
Max	0.493	0.660	0.840	0.993	0.700	0.655	0.554	0.638	0.704	1.075	0.019	0.063	0.219	0.053	0.029	8.52	12.96	23.96	13.50	7.70	2.29	1.75	2.70	2.91	1.67
SD	0.033	0.049	0.052	0.061	0.044	0.032	0.064	0.102	0.043	0.069	0.005	0.005	0.020	0.004	0.009	0.52	0.86	1.39	0.85	1.04	0.180	0.158	0.375	0.225	0.188
CV	9	10	8	8	14	10	14	19	6	24	58	26	23	10	125	8	8	8	8	18	10	12	18	10	14
Npast	0	0	0	28	30	0	0	0	5	9	0	0	0	9	7	0	0	0	31	32	0	0	0	18	22
Medianpast				0.760	0.344				0.708	0.332				0.045	0.010				10.552	5.759				2.455	1.437
SDpast				0.066	0.025				0.130	0.043				0.012	0.006				0.812	0.764				0.285	0.144
NIST	0.346	0.490	0.672	0.702	0.313											7.01	10.25	19.56	10.98	5.94	2.01	1.50	2.33	2.35	1.40
NAV	0.349	0.495	0.662	0.710	0.321	0.330	0.471	0.549	0.675	0.289	0.008	0.021	0.090	0.043	0.007	6.904	10.326	19.057	10.716	5.811	1.865	1.419	2.204	2.307	1.351
NAU	0.034	0.045	0.056	0.067	0.046	0.032	0.064	0.102	0.053	0.069	0.010	0.011	0.024	0.015	0.010	0.608	0.815	1.612	0.895	1.115	0.275	0.197	0.414	0.239	0.202

C2

Round Robin LXVI Laboratory Results

	δ-Tocopherol, µg/mL					Total β-Carotene, µg/mL					trans-β-Carotene, µg/mL					Total cis-β-Carotene, µg/mL					Total α-Carotene, µg/mL				
Lab	357	358	359	360	361	357	358	359	360	361	357	358	359	360	361	357	358	359	360	361	357	358	359	360	361
110.2???						0.094	0.265	0.369	0.299	0.071											0.005	0.007	0.006	0.007	0.004
110.3???						0.092	0.277	0.373	0.301	0.065											0.006	0.008	0.006	0.007	0.004
119.1???						0.090	0.255	0.389	0.303	0.083	0.090	0.254	0.386	0.301	0.082	*nd*	*0.001*	*0.003*	*0.002*	*0.001*	0.008	0.028	0.020	0.038	0.010
FSV-BA	0.097	0.067	0.202	0.267	0.073	0.090	0.244	0.415	0.295	0.092	0.085	0.231	0.399	0.280	0.085	0.005	0.013	0.016	0.015	0.007	0.009	0.033	0.014	0.054	0.011
FSV-BB	0.084	0.076	0.205	0.255	0.085	0.086	0.225	0.346	0.265	0.079	0.082	0.213	0.330	0.249	0.074	0.004	0.012	0.016	0.016	0.004	0.005	0.028	0.012	0.043	0.009
FSV-BC																									
FSV-BD																									
FSV-BE						0.078	0.234	0.441	0.273	0.065															
FSV-BF						0.069	0.208	0.350	0.252	0.053															
FSV-BG						0.094	0.264	0.462	0.311	0.082											0.009	0.032	0.020	0.048	0.009
FSV-BH						0.079	0.236	0.387	0.241	0.059	0.079	0.224	0.371	0.241	0.059	*nq*	0.012	0.016	*nq*	*nq*	*nq*	*nq*	*nq*	0.034	*nq*
FSV-BJ						0.088	0.269	0.502	0.310	0.092											*nq*	0.030	*nq*	0.049	*nq*
FSV-BK																									
FSV-BL																									
FSV-BM																									
FSV-BN						0.088	0.213	0.364	0.258	0.074	0.083	0.203	0.344	0.244	0.065	*0.005*	*0.010*	*0.020*	*0.015*	*0.008*	0.012	0.031	0.016	0.053	0.011
FSV-BNa						0.092	0.235	0.366	0.257	0.070	0.085	0.219	0.348	0.242	0.063	*0.007*	*0.016*	*0.018*	*0.015*	*0.007*	0.014	0.037	0.017	0.053	0.014
FSV-BO						0.089	0.219	0.307	0.271	0.084											0.003	0.022	0.005	0.045	0.008
FSV-BP						0.063	0.261	0.407	0.287	0.056											*nd*	0.028	0.018	0.043	0.006
FSV-BQ																									
FSV-BR																									
FSV-BS						*≥0.029*	*≥0.145*	*≥0.330*	*≥0.234*	*≥0.012*	0.029	0.145	0.330	0.234	0.012						0.006	0.031	0.024	0.060	0.012
FSV-BT	0.066	0.046	0.102	0.106	0.033	0.091	0.214	0.299	0.257	0.079	0.089	0.206	0.291	0.252	0.077	0.004	0.013	0.015	0.012	0.004	0.008	0.028	0.017	0.039	0.010
FSV-BU						0.102	0.289	0.472	0.310	0.100											0.006	0.031	0.006	0.044	0.017
FSV-BV						0.111	0.292	0.444	0.352	0.090											0.010	0.043	0.018	0.070	0.010
FSV-BW						0.099	0.266	0.493	0.314	0.064											0.003	0.017	0.004	0.040	*nd*
FSV-CC																									
FSV-CD						0.182	0.245	0.321	0.250	0.167											0.029	0.037	0.027	0.048	0.025
FSV-CE						0.168	0.344	0.585	0.486	0.153															
FSV-CF																									
FSV-CG	*nq*	*nq*	0.179	0.238	0.127	0.071	0.205	0.283	0.226	0.066	0.067	0.192	0.269	0.212	0.061	*nq*	0.013	0.014	0.014	*nq*	0.009	0.035	0.013	0.052	0.011
FSV-CI						0.075	0.206	0.402	0.229	0.055											0.013	0.044	0.043	0.052	0.013
FSV-CW	0.172	0.100	0.262	0.312	0.096	0.100	0.239	0.386	0.303	0.051											0.005	0.027	0.011	0.049	0.002
FSV-CZ						0.089	0.199	0.277	0.229	0.077															
FSV-DD																									
FSV-DV																									
FSV-EE																									
N	4	4	5	5	5	24	24	24	24	24	9	9	9	9	9	5	8	8	7	6	18	20	19	21	18
Min	0.066	0.046	0.102	0.106	0.033	0.063	0.199	0.277	0.226	0.051	0.029	0.145	0.269	0.212	0.012	0.004	0.001	0.003	0.002	0.001	0.003	0.007	0.004	0.007	0.002
Median	0.091	0.072	0.202	0.255	0.085	0.090	0.241	0.386	0.280	0.075	0.083	0.213	0.344	0.244	0.065	0.005	0.013	0.016	0.015	0.006	0.008	0.030	0.016	0.048	0.010
Max	0.172	0.100	0.262	0.312	0.127	0.182	0.344	0.585	0.486	0.167	0.090	0.254	0.399	0.301	0.085	0.007	0.016	0.020	0.016	0.008	0.029	0.044	0.043	0.070	0.025
SD	0.023	0.022	0.034	0.025	0.018	0.010	0.036	0.070	0.038	0.016	0.006	0.016	0.039	0.012	0.013	0.001	0.001	0.002	0.001	0.003	0.004	0.004	0.006	0.007	0.003
CV	25	31	17	10	21	11	15	18	14	21	7	8	11	5	19	26	6	12	6	46	51	14	39	15	33
Npast	0	0	0	4	5	0	0	0	20	22	0	0	0	7	9	0	0	0	5	5	0	0	0	17	15
Medianpast				0.267	0.084				0.268	0.076				0.248	0.075				0.013	0.005				0.044	0.009
SDpast				0.027	0.023				0.020	0.013				0.010	0.008				0.003	0.002				0.007	0.003
NIST	nq	nq	nq	0.271	nq	0.115	0.248	0.440	0.266	0.074	0.094	0.196	0.380	nq	nq	*0.021*	*0.052*	*0.060*	nd	nd	*nq*	0.034	*nq*	0.041	0.007
NAV	0.091	0.072	0.202	0.263	0.085	0.103	0.243	0.413	0.269	0.075	0.088	0.204	0.362	0.244	0.065	0.013	0.032	0.038	0.015	0.006	0.008	0.032	0.015	0.044	0.008
NAU			0.036	0.044	0.022	0.024	0.039	0.090	0.045	0.018	0.014	0.027	0.047	0.027	0.013	0.012	0.033	0.034	0.005	0.003	0.004	0.010	0.008	0.016	0.005

C3

Round Robin LXVI Laboratory Results

	Total Lycopene, µg/mL					trans-Lycopene, µg/mL					Total β-Cryptoxanthin, µg/mL					Total α-Cryptoxanthin, µg/mL					Total Lutein, µg/mL				
Lab	357	358	359	360	361	357	358	359	360	361	357	358	359	360	361	357	358	359	360	361	357	358	359	360	361
110.2???	0.219	0.573	0.815	0.312	0.192						0.058	0.080	0.026	0.058	0.033										
110.3???	0.204	0.594	0.833	0.338	0.163						0.048	0.053	0.027	0.058	0.031										
119.1???	0.243	0.691	1.004	0.437	0.304	0.101	0.283	0.424	0.177	0.120	0.032	0.033	0.023	0.038	0.028						0.060	0.059	0.067	0.053	0.046
FSV-BA	0.233	0.631	0.966	0.405	0.329	0.127	0.339	0.457	0.225	0.156	0.043	0.051	0.031	0.058	0.041	0.015	0.017	0.013	0.017	0.016					
FSV-BB	0.248	0.640	0.936	0.398	0.308	0.101	0.255	0.337	0.162	0.110	0.047	0.050	0.034	0.056	0.040	0.017	0.020	0.014	0.017	0.015	0.073	0.102	0.140	0.069	0.060
FSV-BC																									
FSV-BD																									
FSV-BE																									
FSV-BF																									
FSV-BG	0.242	0.665	1.038	0.418	0.285	0.141	0.383	0.533	0.248	0.151	0.050	0.051	0.032	0.060	0.041										
FSV-BH	0.236	0.642	0.965	0.375	0.254						0.059	0.071	0.047	0.074	0.045						0.071	0.088	0.114	0.055	0.044
FSV-BJ	0.234	0.594	1.215	0.387	0.270						0.036	0.045	0.034	0.056	0.034						0.064	0.087	0.126	0.071	0.048
FSV-BK																									
FSV-BL																									
FSV-BM																									
FSV-BN	0.255	0.536	0.808	0.355	0.205	0.113	0.281	0.374	0.175	0.101	0.047	0.046	0.024	0.055	0.033						0.073	0.075	0.098	0.058	0.044
FSV-BNa	0.210	0.558	0.818	0.328	0.166	0.115	0.294	0.383	0.173	0.085	0.047	0.048	0.025	0.054	0.030						0.062	0.078	0.096	0.059	0.039
FSV-BO	0.195	0.471	0.632	0.314	0.253						0.091	0.050	0.028	0.062	0.042						0.122	0.127	0.143	0.106	0.094
FSV-BP	0.268	0.933	1.522	0.546	0.182						0.044	0.064	0.043	0.068	0.036										
FSV-BQ																									
FSV-BR																									
FSV-BS	0.169	0.490	1.055	0.366	0.127						0.015	0.018	0.012	0.032	0.029										
FSV-BT	0.241	0.575	0.775	0.379	0.305	0.121	0.279	0.304	0.189	0.120	0.042	0.041	0.028	0.051	0.032	0.020	0.027	0.031	0.023	0.016	0.064	0.059	0.071	0.059	0.056
FSV-BU	0.250	0.598	1.073	0.380	0.308						0.047	0.050	0.029	0.049	0.035										
FSV-BV	0.308	0.803	1.113	0.515	0.342						0.049	0.048	0.023	0.061	0.037										
FSV-BW	0.241	0.682	1.140	0.432	0.208						0.041	0.036	0.017	0.075	0.029										
FSV-CC																									
FSV-CD	0.148	0.219	0.303	0.178	0.148						0.041	0.047	0.030	0.045	0.035	0.012	0.022	0.016	<0.01	<0.01					
FSV-CE																									
FSV-CF																									
FSV-CG	0.192	0.541	0.696	0.326	0.245	0.104	0.296	0.337	0.180	0.116	0.056	0.068	0.048	0.072	0.052										
FSV-CI																					0.078	0.092	0.117	0.065	0.049
FSV-CW						0.127	0.287	0.361	0.190	0.089	0.052	0.049	0.031	0.060	0.048						0.108	0.114	0.138	0.086	0.096
FSV-CZ																									
FSV-DD																									
FSV-DV																									
FSV-EE																									
N	19	19	19	19	19	9	9	9	9	9	20	20	20	20	20	4	4	4	3	3	10	10	10	10	10
Min	0.148	0.219	0.303	0.178	0.127	0.101	0.255	0.304	0.162	0.085	0.015	0.018	0.012	0.032	0.028	0.012	0.017	0.013	0.017	0.015	0.060	0.059	0.067	0.053	0.039
Median	0.236	0.594	0.965	0.379	0.253	0.115	0.287	0.374	0.180	0.116	0.047	0.049	0.029	0.058	0.035	0.016	0.021	0.015	0.017	0.016	0.072	0.088	0.115	0.062	0.048
Max	0.308	0.933	1.522	0.546	0.342	0.141	0.383	0.533	0.248	0.156	0.091	0.080	0.048	0.075	0.052	0.020	0.027	0.031	0.023	0.016	0.122	0.127	0.143	0.106	0.096
SD	0.025	0.079	0.219	0.061	0.081	0.017	0.012	0.055	0.013	0.022	0.007	0.005	0.006	0.006	0.008	0.004	0.004	0.002			0.012	0.020	0.031	0.010	0.009
CV	11	13	23	16	32	15	4	15	7	19	16	10	21	11	22	23	18	15			16	23	27	17	19
Npast	0	0	0	15	17	0	0	0	7	8	0	0	0	16	18	0	0	0	5	7	0	0	0	8	12
Medianpast				0.372	0.272				0.176	0.116				0.059	0.039				0.020	0.015				0.055	0.058
SDpast				0.033	0.052				0.027	0.018				0.010	0.006				0.009	0.002				0.010	0.014
NIST	0.175	0.389	0.733	nq	0.262	0.157	0.319	0.603	0.187	nd	0.042	0.038	0.017	0.056	0.026						0.067	0.102	0.132	0.056	0.047
NAV	0.205	0.492	0.842	0.377	0.258	0.136	0.303	0.488	0.184	0.116	0.044	0.043	0.023	0.057	0.030	0.016	0.021	0.015	0.017	0.016	0.070	0.095	0.123	0.059	0.048
NAU	0.070	0.187	0.251	0.082	0.079	0.036	0.057	0.176	0.032	0.022	0.012	0.014	0.011	0.014	0.011						0.015	0.023	0.033	0.013	0.010

C4

Round Robin LXVI Laboratory Results

	Total Zeaxanthin, µg/mL					Total Lutein&Zeaxanthin, µg/mL					Coenzyme Q10, µg/mL					Phylloquinone (K1), ng/mL					25-hydroxyvitamin D, µg/mL				
Lab	357	358	359	360	361	357	358	359	360	361	357	358	359	360	361	357	358	359	360	361	357	358	359	360	361
110.2???						0.154	0.148	0.159	0.113	0.083															
110.3???						0.127	0.156	0.164	0.114	0.076															
119.1???	0.035	0.032	0.034	0.031	0.023	0.119	0.112	0.124	0.104	0.085															
FSV-BA						0.105	0.129	0.157	0.102	0.086											0.007	0.013	0.020	0.015	0.013
FSV-BB	0.037	0.046	0.059	0.039	0.031	0.110	0.148	0.199	0.108	0.091															
FSV-BC																									
FSV-BD																									
FSV-BE											0.862	0.945	1.440	0.806	0.656	0.454	0.538	2.971	1.174	0.267					
FSV-BF																									
FSV-BG						0.105	0.131	0.174	0.097	0.079															
FSV-BH	0.039	0.028	0.026	0.025	0.020	0.110	0.117	0.141	0.080	0.044											*nq*	0.017	0.022	0.013	0.012
FSV-BJ																									
FSV-BK																									
FSV-BL																									
FSV-BM																									
FSV-BN	0.052	0.045	0.044	0.039	0.031	0.112	0.109	0.132	0.086	0.065															
FSV-BNa	0.041	0.045	0.042	0.040	0.029	0.093	0.111	0.128	0.087	0.058															
FSV-BO	0.006	0.009	0.013	0.007	0.004	0.128	0.136	0.156	0.113	0.098															
FSV-BP						0.068	0.139	0.105	0.081	0.060															
FSV-BQ																									
FSV-BR																									
FSV-BS						0.133	0.162	0.216	0.164	0.147															
FSV-BT	0.029	0.027	0.030	0.025	0.022	0.104	0.097	0.133	0.095	0.089	0.979	1.023	1.199	1.124	0.654										
FSV-BU						0.116	0.134	0.128	0.095	0.074															
FSV-BV						0.104	0.107	0.118	0.088	0.069															
FSV-BW						0.105	0.122	0.142	0.094	0.076															
FSV-CC																									
FSV-CD						0.185	0.211	0.236	0.157	0.122															
FSV-CE																									
FSV-CF																									
FSV-CG						0.108	0.122	0.120	0.098	0.095															
FSV-CI	0.025	0.022	0.022	0.020	0.019	*0.103*	*0.114*	*0.139*	*0.085*	*0.067*						0.360	0.440	2.669	1.117	0.163					
FSV-CW	0.075	0.053	0.048	0.052	0.058	0.183	0.167	0.187	0.138	0.153	0.953	1.141	1.605	0.957	0.663										
FSV-CZ											0.871	0.990	1.554	0.943	0.701										
FSV-DD																									
FSV-DV																									
FSV-EE											0.887	0.663	1.336	0.872	0.682										
N	9	9	9	9	9	20	20	20	20	20	5	5	5	5	5	2	2	2	2	2	1	2	2	2	2
Min	0.006	0.009	0.013	0.007	0.004	0.068	0.097	0.105	0.080	0.044	0.862	0.663	1.199	0.806	0.654	0.360	0.440	2.669	1.117	0.163		0.013	0.020	0.013	0.012
Median	0.037	0.032	0.034	0.031	0.023	0.110	0.130	0.142	0.098	0.081	0.887	0.990	1.440	0.943	0.663	0.407	0.489	2.820	1.146	0.215	0.007	0.015	0.021	0.014	0.013
Max	0.075	0.053	0.059	0.052	0.058	0.185	0.211	0.236	0.164	0.153	0.979	1.141	1.605	1.124	0.701	0.454	0.538	2.971	1.174	0.267		0.017	0.022	0.015	0.013
SD	0.012	0.019	0.015	0.012	0.009	0.010	0.027	0.026	0.016	0.019	0.037	0.067	0.169	0.106	0.013										
CV	32	59	45	38	38	9	21	18	16	24	4	7	12	11	2										
Npast	0	0	0	7	10	0	0	0	16	19	0	0	0	7	9	0	0	0	0	0	0	0	0	0	0
Medianpast				0.030	0.024				0.097	0.086				0.900	0.618										
SDpast				0.015	0.009				0.024	0.018				0.184	0.147										
NIST	0.027	0.030	0.025	0.028	0.023	*0.095*	*0.132*	*0.157*	*0.084*	*0.070*															
NAV	0.032	0.031	0.030	0.030	0.023	0.102	0.130	0.149	0.091	0.077	0.887	0.990	1.440	0.943	0.663										
NAU	0.014	0.019	0.017	0.012	0.009	0.025	0.027	0.031	0.023	0.020	0.089	0.099	0.169	0.106	0.066										

C5

Round Robin LXVI Laboratory Results

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: $\sqrt{(S_{rep}^2 + S_{het}^2)}$
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: $\sqrt{(S^2 + S_{btw}^2)}$ 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.
nd	Not detected (i.e., no detectable peak for analyte)
nq	Detected but not quantitatively determined
≥x	Concentration greater than or equal to x
italics	Not explicitly reported but calculated by NIST from reported values

Round Robin LXVI Laboratory Results

Comparability Summary

Lab	TR	aT	g/bT	bC	tbC	aC	TLy	TbX	TLu	TZ	L&Z
110.2???	2	1	3	1		2	1	2			1
110.3???	2	1	3	1		2	1	1			1
119.1???	3	1	3	1	2	1	1	2	2	1	1
FSV-BA	1	1	2	1	2	1	1	1			1
FSV-BB	1	1	1	1	1	1	1	1	1	1	1
FSV-BC	1										
FSV-BD	2	2									
FSV-BE	1	2	1	1							
FSV-BF	2	1		1							
FSV-BG	1	1	1	1		1	1	1			1
FSV-BH	1	1	3	1	1		1	2	1	1	1
FSV-BJ	1	1	1	1		1	1	1	1		
FSV-BK	2	1									
FSV-BL	2	1									
FSV-BM	1	1									
FSV-BN	2	2	2	1	1	1	1	1	1	1	1
FSV-BNa	2	2	2	1	1	1	1	1	1	1	1
FSV-BO	2	1	1	1		1	1	2	4	2	1
FSV-BP	1	2		1		1	2	1			2
FSV-BQ	4	3									
FSV-BR	1	1									
FSV-BS	4			3	3	1	1	2			3
FSV-BT	2	1	1	1	1	1	1	1	1	1	1
FSV-BU	1	2	1	2		2	1	1			1
FSV-BV	2	2	1	2		1	2	1			1
FSV-BW	1	1	1	1		2	1	1			1
FSV-CC	1	2									
FSV-CD	1	2	1	4		3	3	1			3
FSV-CE	2	3		4							
FSV-CF	1	1									
FSV-CG	2	3	1	1	2	1	1	2			1
FSV-CI	1	1	1	1		2			1	1	1
FSV-CW	4	2	2	1		1		1	3	3	3
FSV-CZ	2	1	1	1							
FSV-DD	1										
FSV-DV	1	2									
FSV-EE	1	2									
NIST	1	1	1	1	1	1	1	1	1	1	1
n	38	35	22	26	10	21	20	21	11	10	21

	TR	aT	g/bT	bC	tbC	aC	TLy	TbX	TLu	TZ	L&Z
% 1	53	57	64	81	60	71	85	71	73	80	81
% 2	37	34	18	8	30	24	10	29	9	10	5
% 3	3	9	18	4	10	5	5	0	9	10	14
% 4	8	0	0	8	0	0	0	0	9	0	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.

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

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

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

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 RR66

Each participant in RR66 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 RR66:

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

Individualized Round Robin LXVI Report: FSV-BA

Summary

	Serum 357			Serum 358			Serum 359			Serum 360			Serum 361		
Analyte	You	NAV	n	You	NAV	n	You	NAV	n	You	NAV	n	You	NAV	n
Total Retinol	0.366	0.349	33	0.523	0.495	33	0.675	0.662	33	0.770	0.710	33	0.357	0.321	33
Retinyl Palmitate	0.02	0.01	11	0.1	0.0	9	0.2	0.1	12	0.04	0.04	11	0.03	0.01	9
α-Tocopherol	6.62	6.90	34	9.99	10.33	34	18.18	19.06	34	10.16	10.72	34	5.88	5.81	33
γ/β-Tocopherol	2.041	1.865	21	1.581	1.419	21	2.540	2.204	21	2.687	2.307	21	1.666	1.351	21
δ-Tocopherol	0.097	0.091	4	0.067	0.072	4	0.202	0.202	5	0.267	0.263	5	0.073	0.085	5
Total β-Carotene	0.090	0.103	24	0.244	0.243	24	0.415	0.413	24	0.295	0.269	24	0.092	0.075	24
trans-β-Carotene	0.085	0.088	9	0.231	0.204	9	0.399	0.362	9	0.280	0.244	9	0.085	0.065	9
Total cis-β-Carotene	0.005	0.013	5	0.013	0.032	8	0.016	0.038	8	0.015	0.015	7	0.007	0.006	6
Total α-Carotene	0.009	0.008	18	0.033	0.032	20	0.014	0.015	19	0.054	0.044	21	0.011	0.008	18
Total Lycopene	0.233	0.205	19	0.631	0.492	19	0.966	0.842	19	0.405	0.377	19	0.329	0.258	19
trans-Lycopene	0.127	0.136	9	0.339	0.303	9	0.457	0.488	9	0.225	0.184	9	0.156	0.116	9
Total β-Cryptoxanthin	0.043	0.044	20	0.051	0.043	20	0.031	0.023	20	0.058	0.057	20	0.041	0.030	20
Total α-Cryptoxanthin	0.015	0.016	4	0.017	0.021	4	0.013	0.015	4	0.017	0.017	3	0.016	0.016	3
Total Lutein&Zeaxanthin	0.105	0.102	20	0.129	0.130	20	0.157	0.149	20	0.102	0.091	20	0.086	0.077	20
25-hydroxyvitamin D	0.007		1	0.013		2	0.020		2	0.015		2	0.013		2

D2

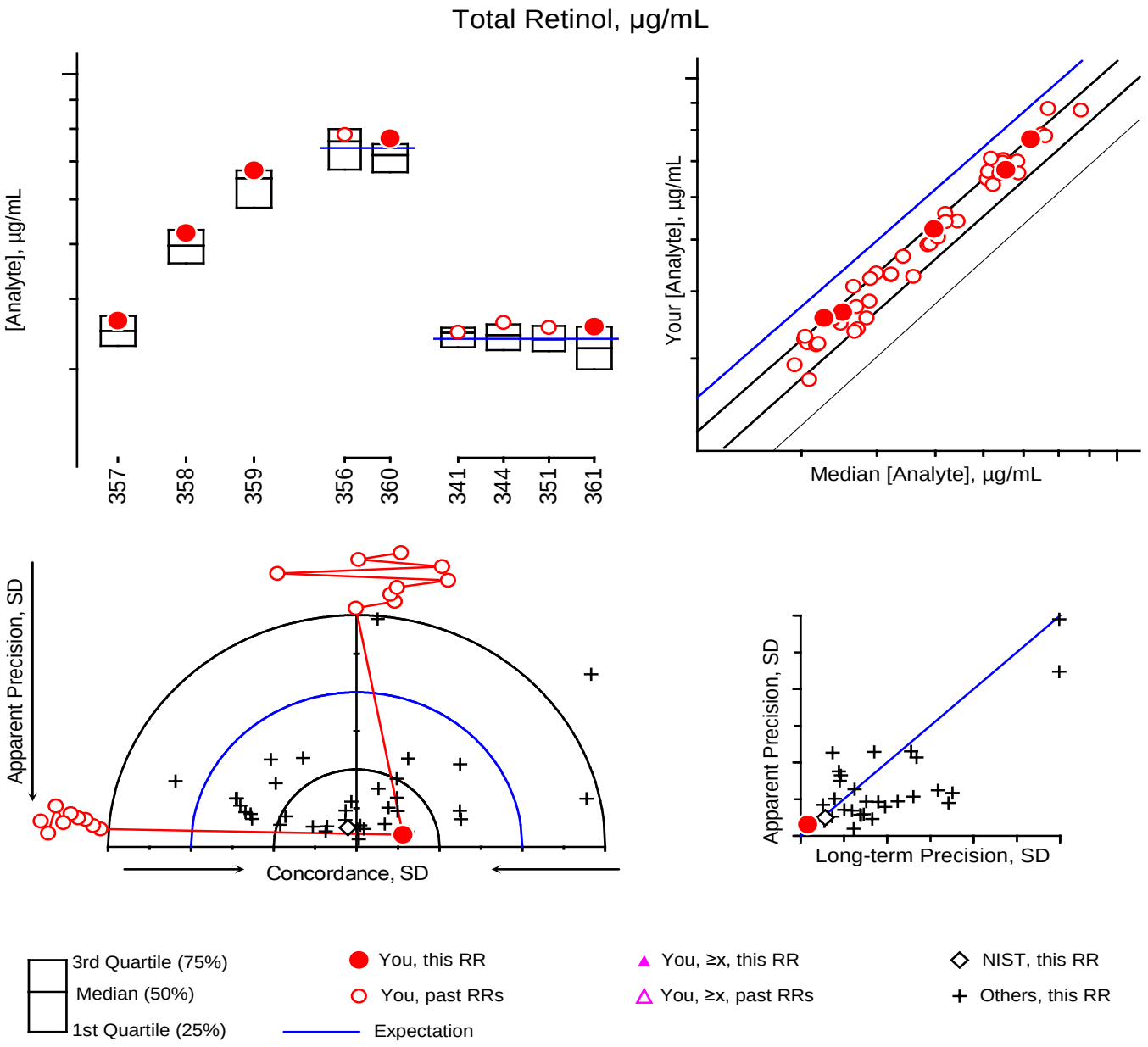
You : Your reported values for the listed analytes (micrograms/milliliter)
NAV : NIST Assigned Values, here equal to this RR's median
n : Number of non-NIST laboratories reporting quantitative values for this analyte in this serum

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 LXVI Report: FSV-BA

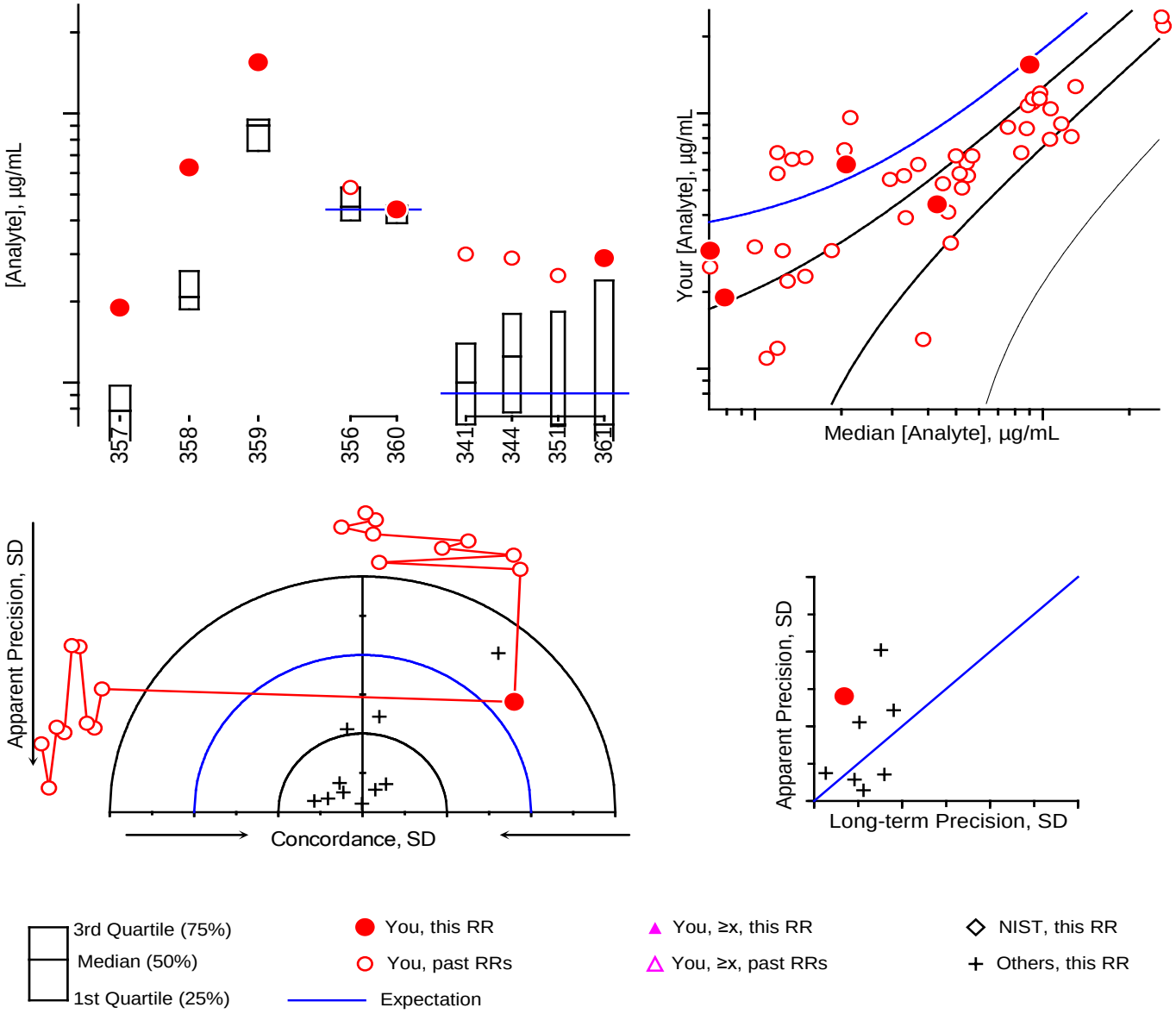


For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

Serum	History	Comments
#357	New	Fresh-frozen, native, multi-donor
#358	New	Fresh-frozen, native, multi-donor
#359	New	Fresh-frozen, native, multi-donor
#360	65:#356	Fresh-frozen, native, multi-donor
#351	63:#341, 63:#344, 64:#351	Fresh-frozen, native, multi-donor

Individualized RR LXVI Report: FSV-BA

Retinyl Palmitate, µg/mL



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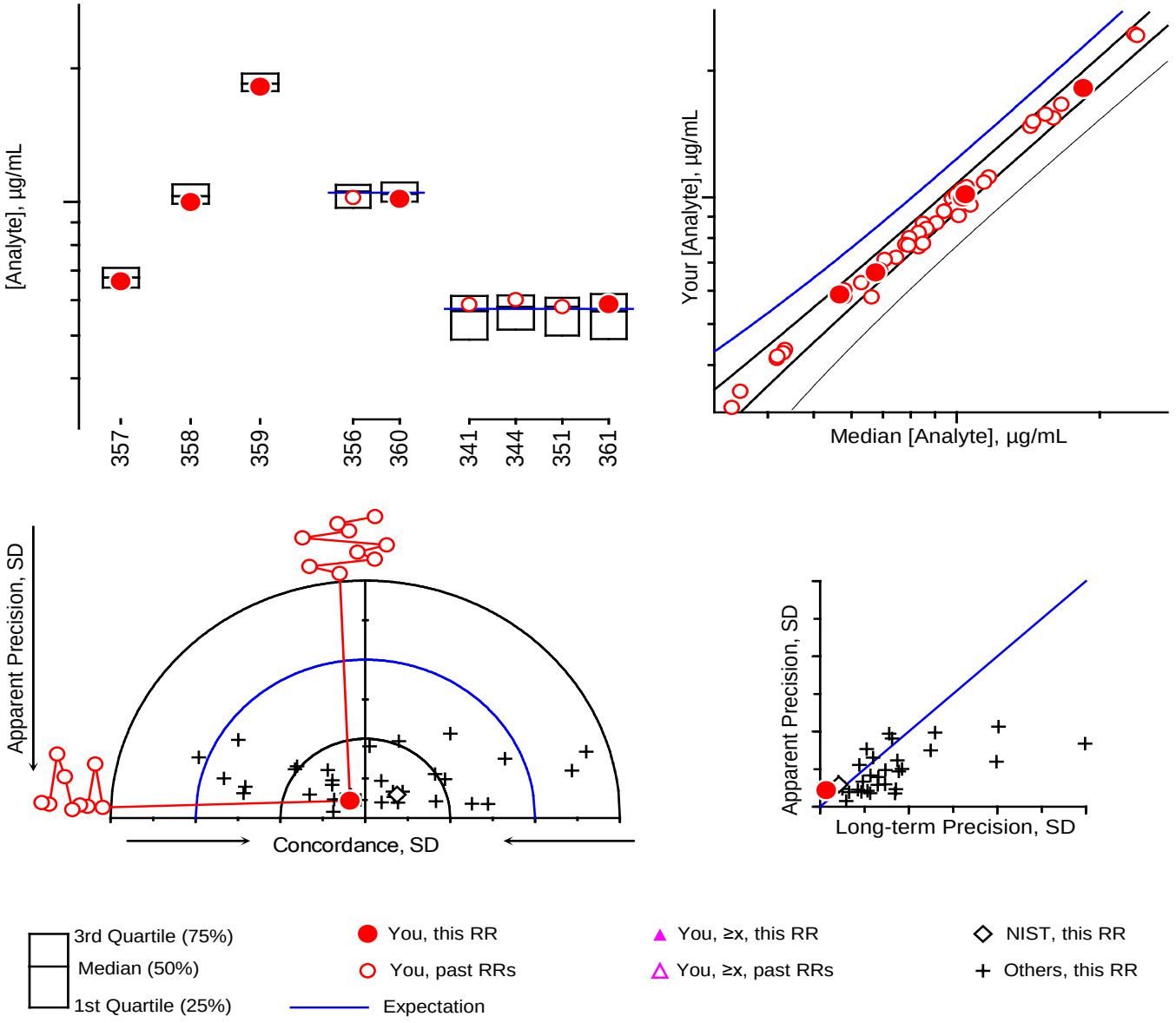
#351	63:#341, 63:#344, 64:#351	Fresh-frozen, native, multi-donor

Individualized RR LXVI Report: FSV-BA

α-Tocopherol, µg/mL



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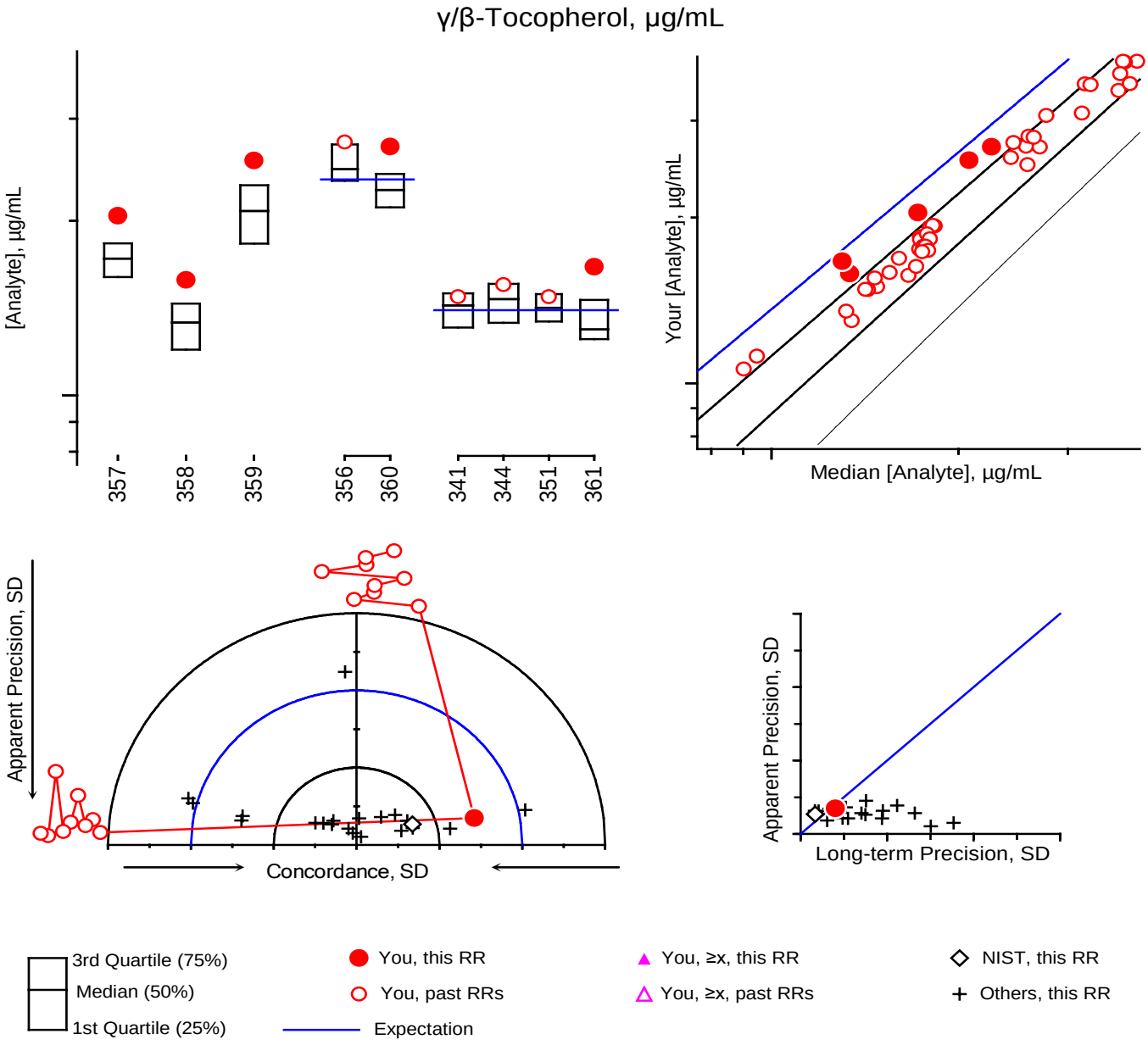
Individualized RR LXVI Report: FSV-BA



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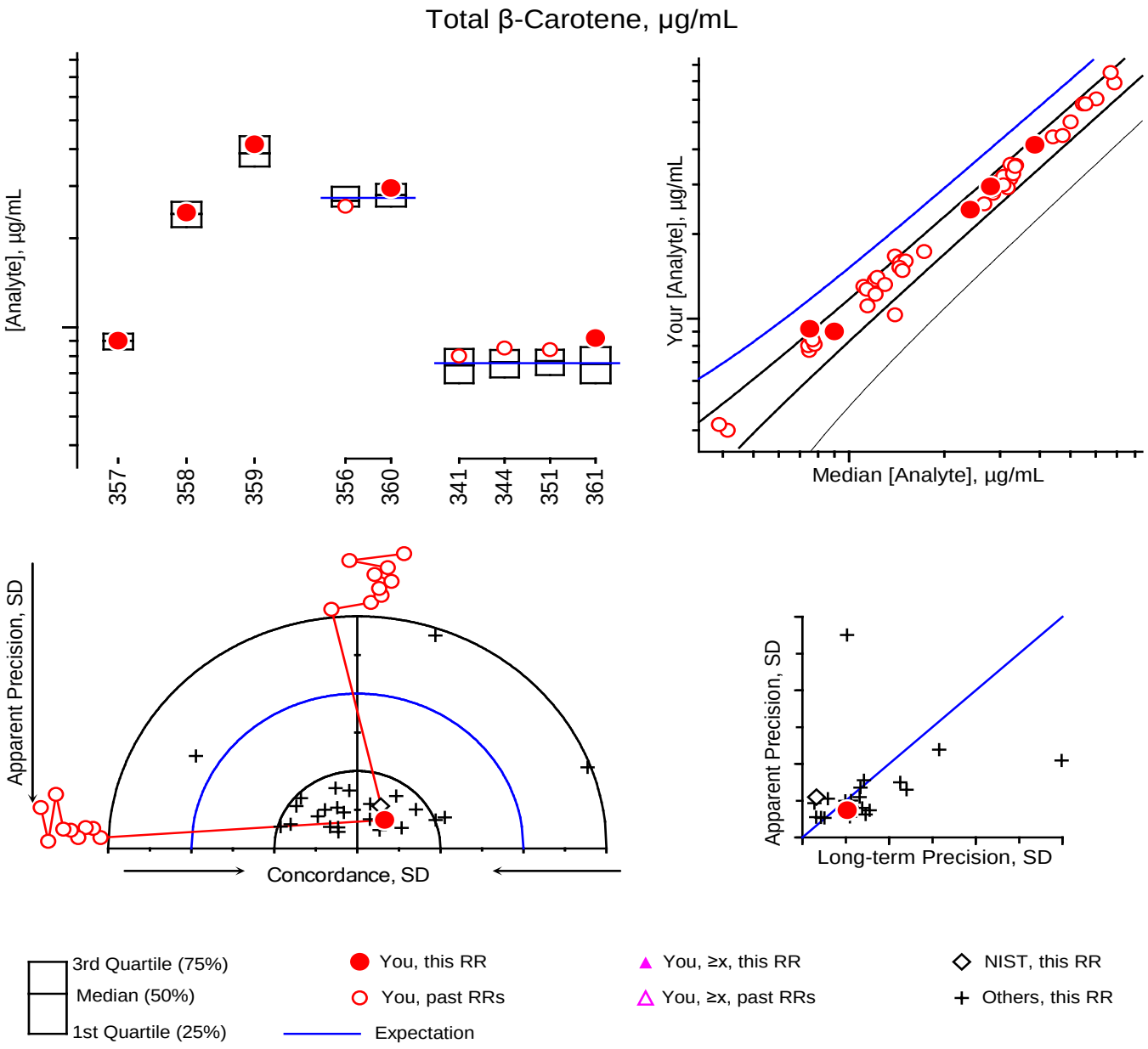
Individualized RR LXVI Report: FSV-BA



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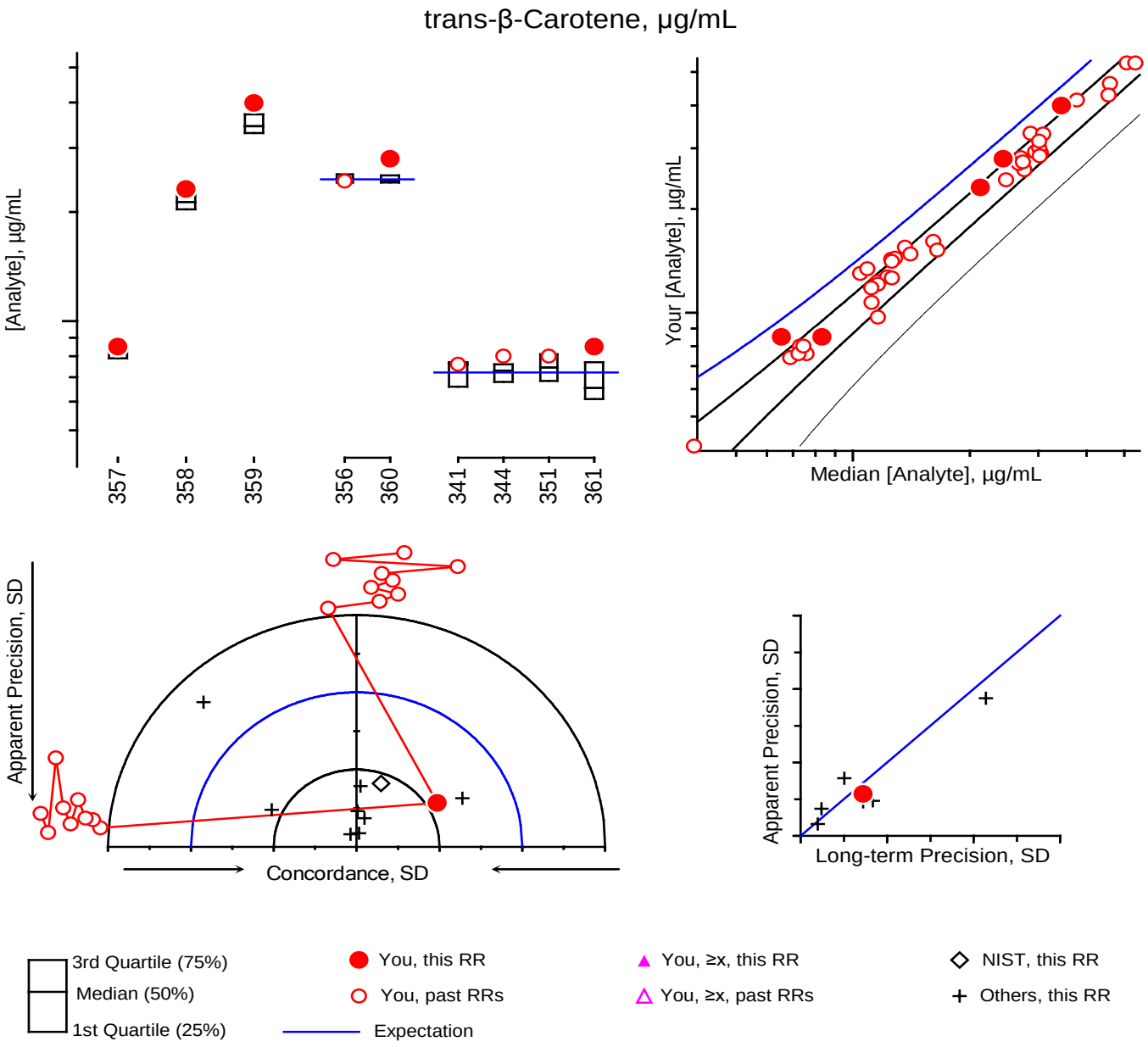
Individualized RR LXVI Report: FSV-BA



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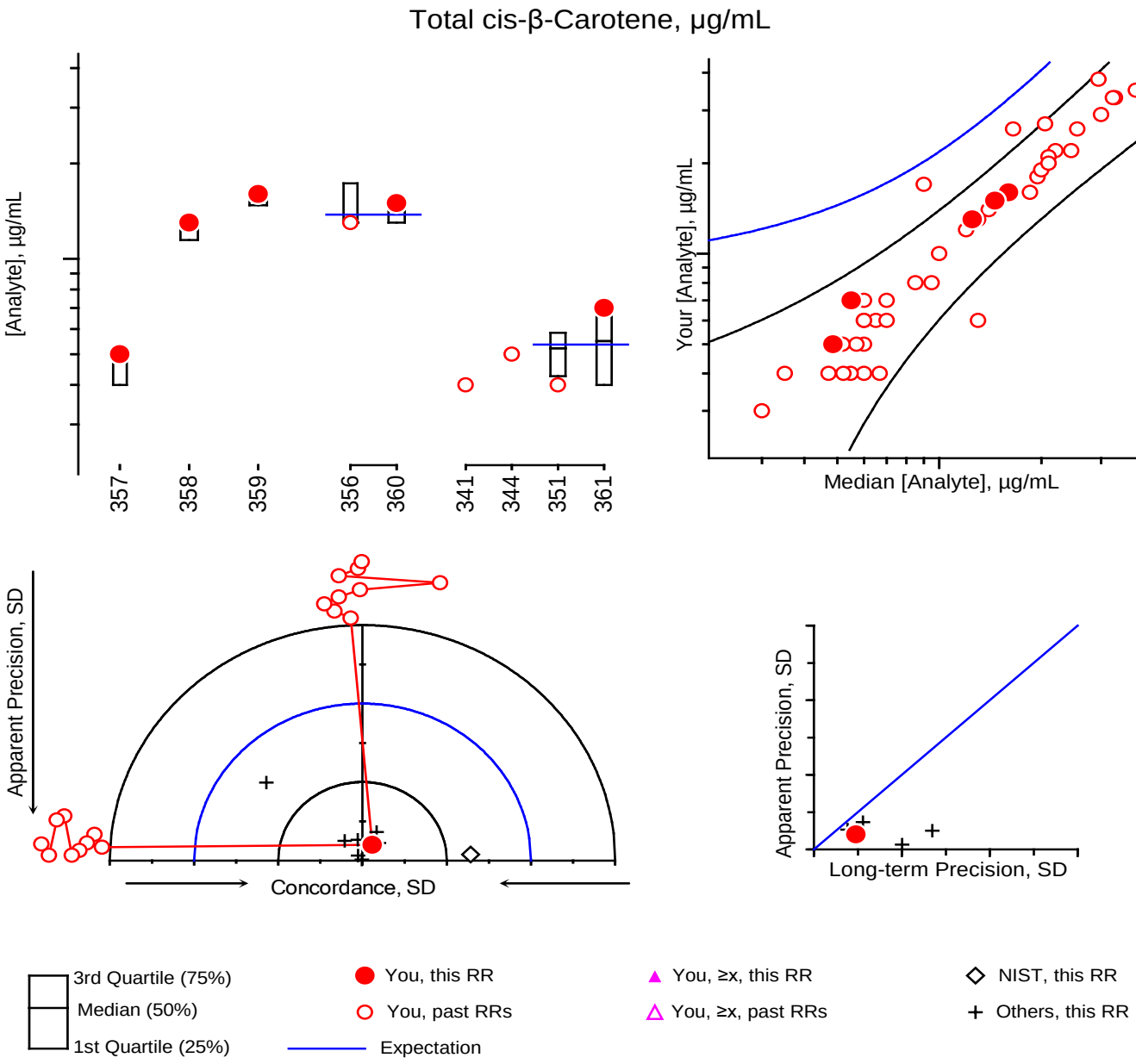
Individualized RR LXVI Report: FSV-BA



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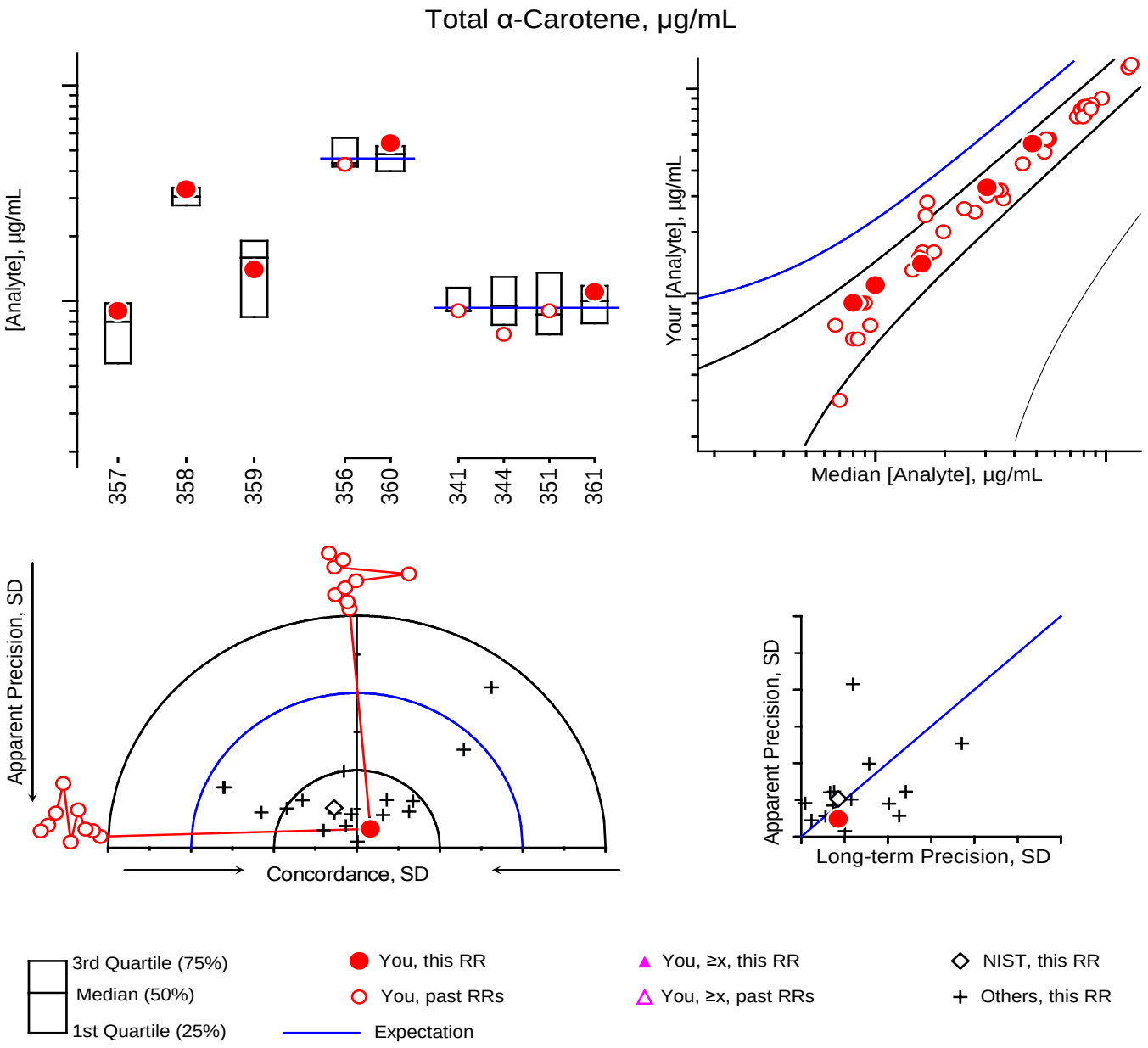
Individualized RR LXVI Report: FSV-BA



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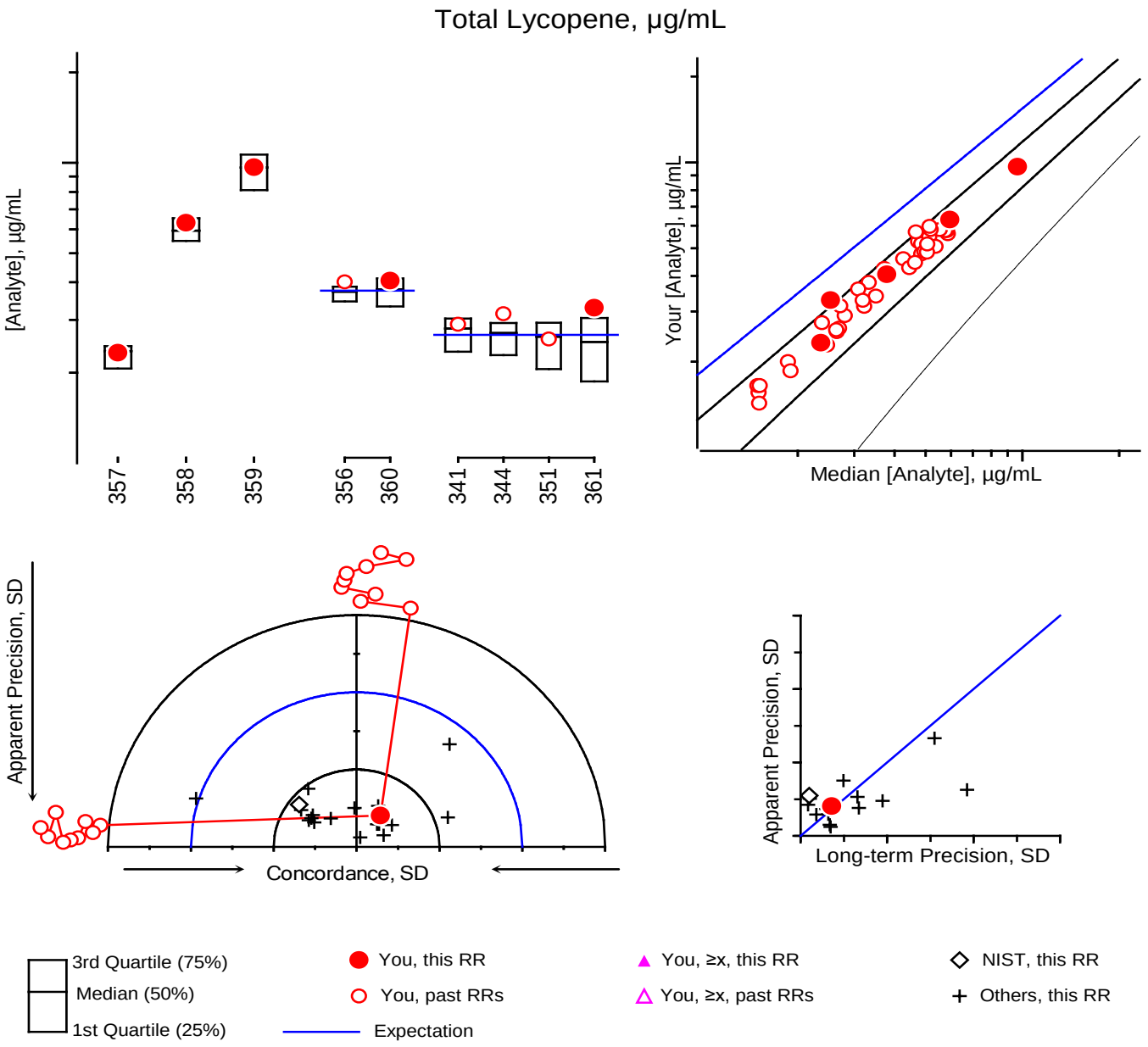
Individualized RR LXVI Report: FSV-BA



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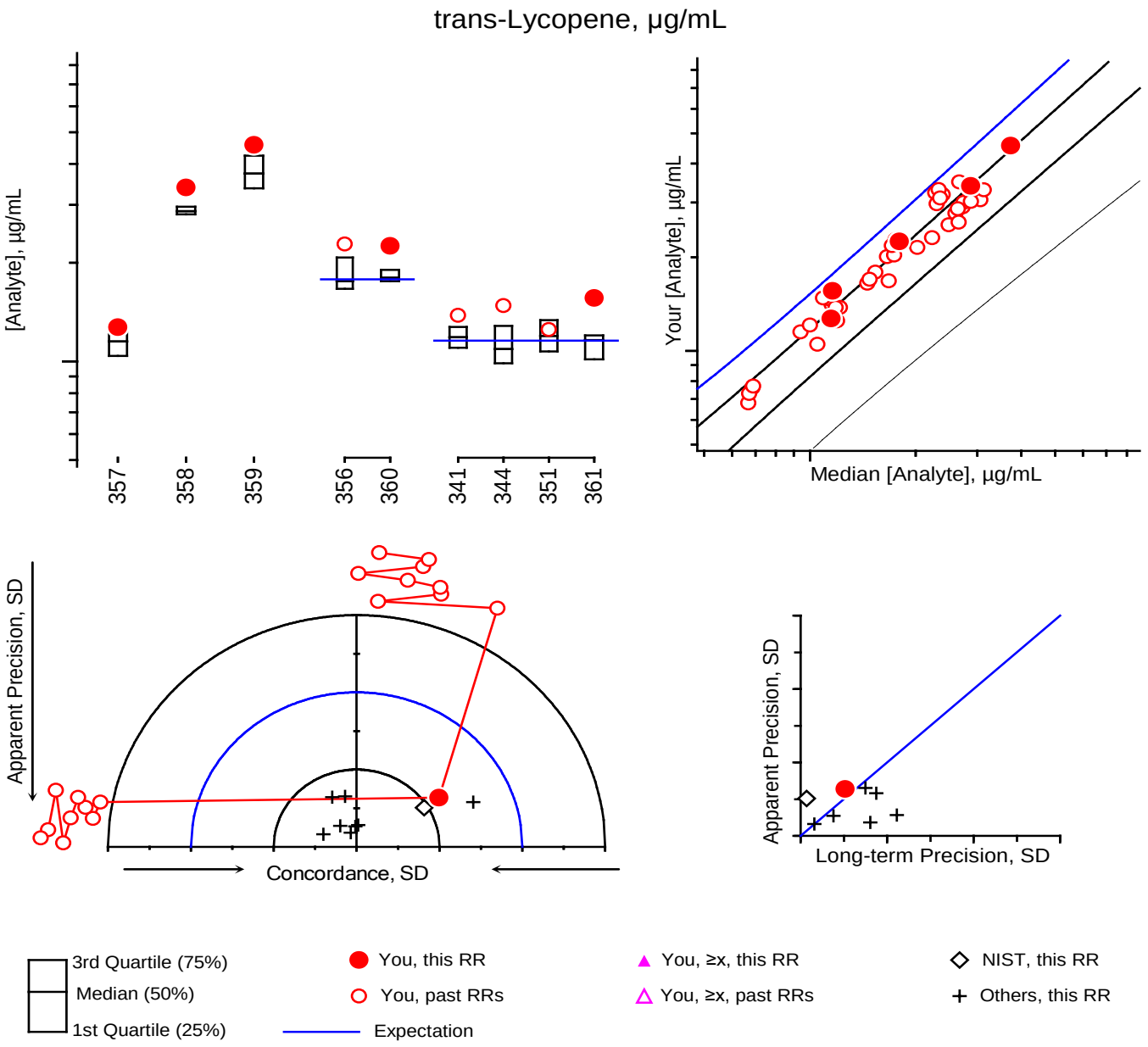
Individualized RR LXVI Report: FSV-BA



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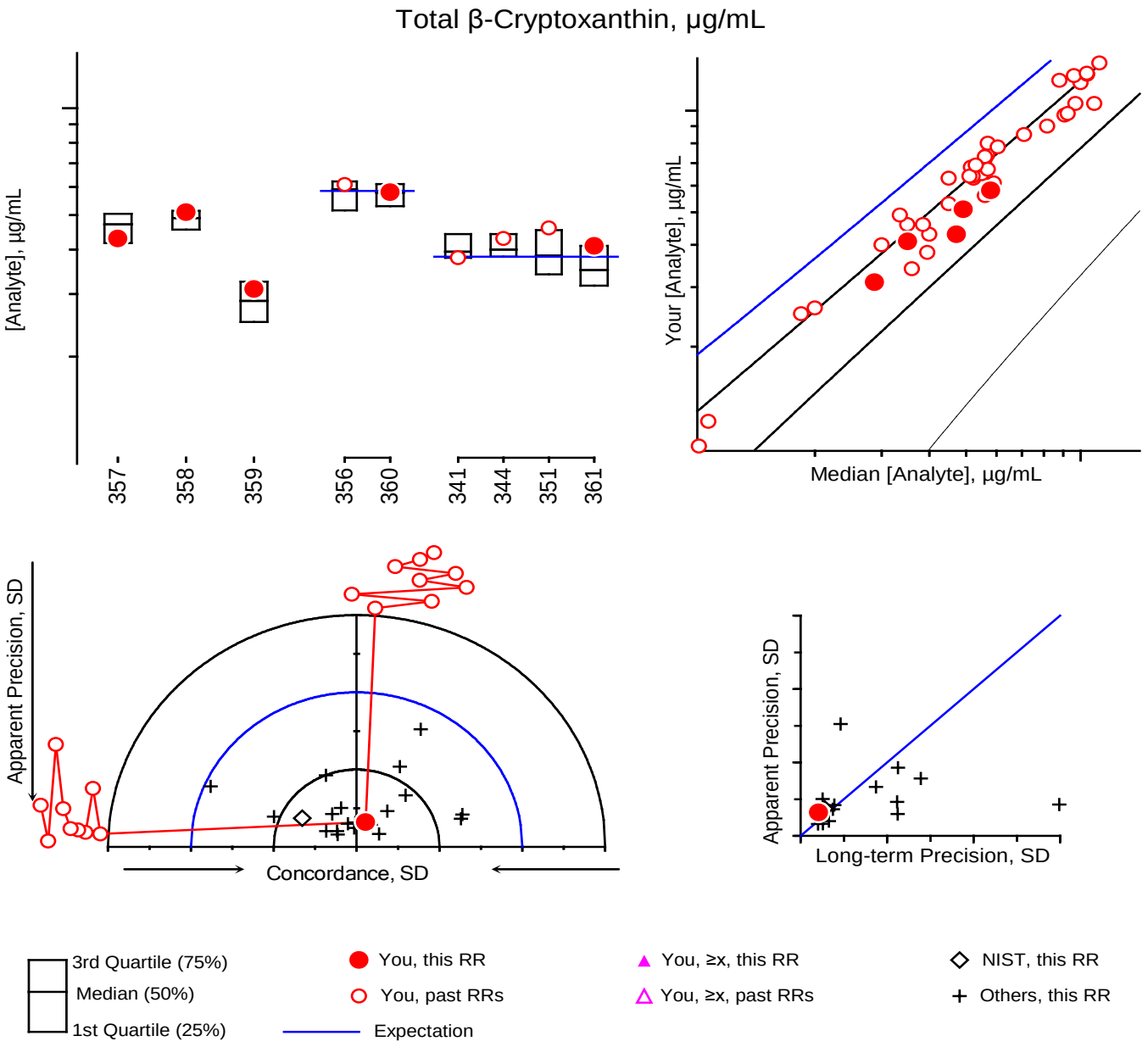
Individualized RR LXVI Report: FSV-BA



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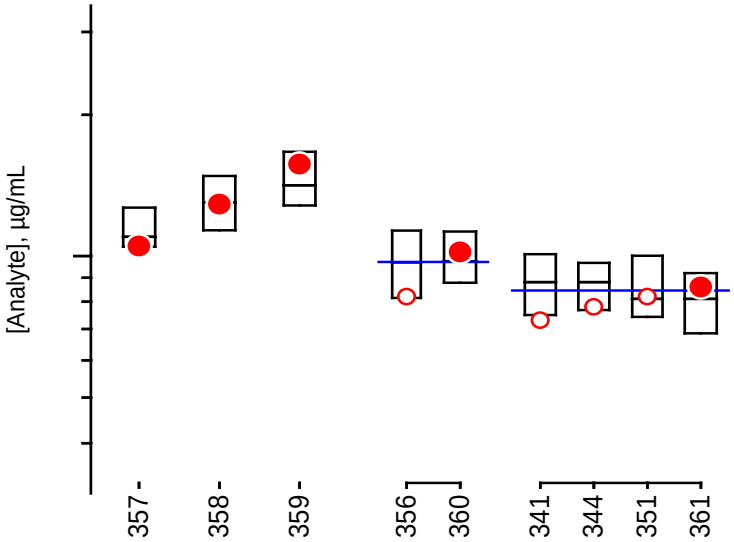
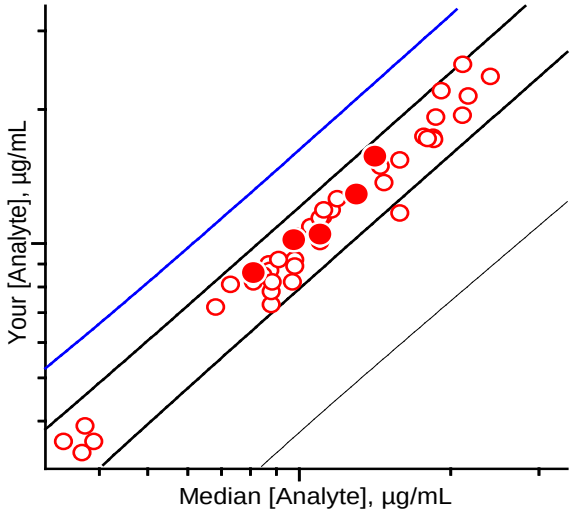
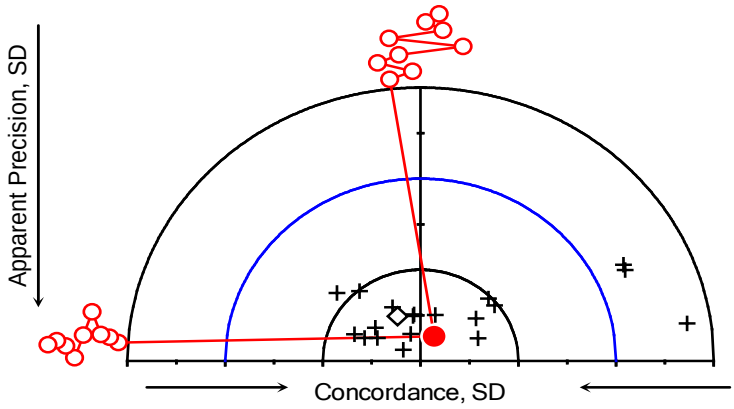
#351	63:#341, 63:#344, 64:#351	Fresh-frozen, native, multi-donor

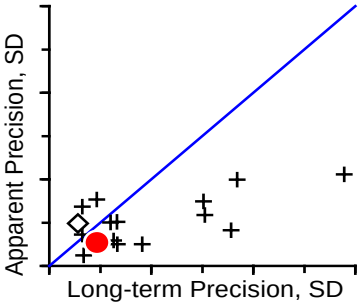
Individualized RR LXVI Report: FSV-BA

Total Lutein&Zeaxanthin, µg/mL









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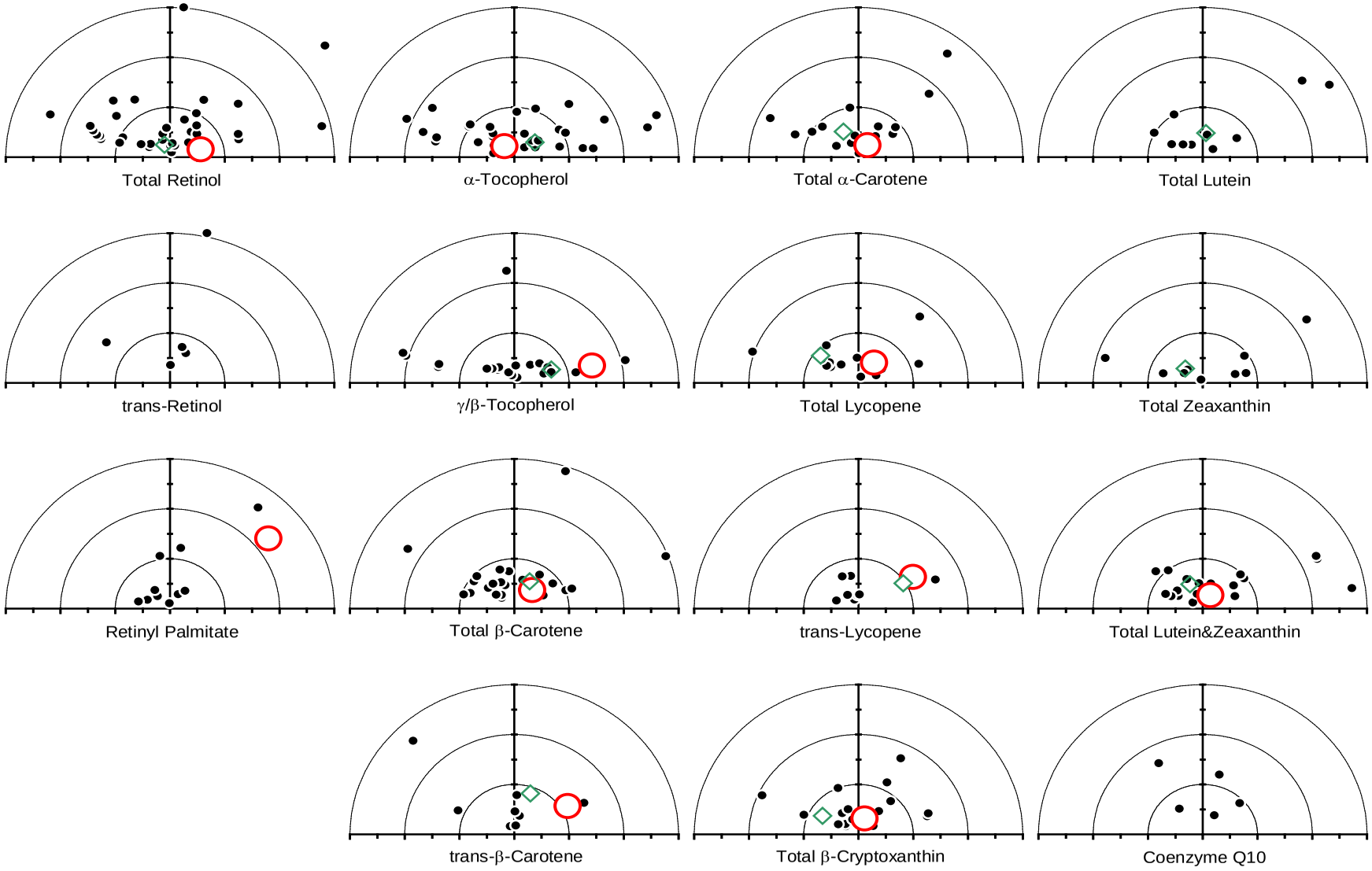
Individualized Round Robin LXVI Report: FSV-BA

Graphical Comparability Summary



D15

Appendix E. Shipping Package Inserts for RR31

The following five items were included in each package shipped to an RR31 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-

June 1, 2009

Dear Colleague:

The samples within this package constitute Vitamin C Round Robin 31 (RR31) of the 2009 Micronutrients Measurement Quality Assurance Program. RR31 consists of four vials of frozen serum *test samples* (#37, #47, #74, and #114), 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 RR30 was e-mailed May 15, 2009. If you find your results for RR30 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 phone: 301-975-3120, fax: 301-977-0685, or e-mail: jbthomas@nist.gov.

We ask that you return your results for these RR31 samples by **September 28, 2009**. 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
RR31 Report Form for Ascorbic Acid Solid Control Material Preparation
RR31 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 #1 (see protocol below). The target values for this materials is 8.4 ±0.7 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 31

NIST Micronutrient Measurement Quality Assurance Program

Preparation and Validation of Ascorbic Acid Solid Control Material

STOCK SOLUTION

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

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

DILUTE SOLUTION 1

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

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

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

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

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

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

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

Wavelength of maximum absorbance..... _____ nm

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

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

DILUTE SOLUTION 2

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

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

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

DILUTE SOLUTION 3

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

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

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

Please return by **September 28, 2009**

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 31

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 #37			µmol/L of Sample
Serum Test Sample #47			µmol/L of Sample
Serum Test Sample #74			µmol/L of Sample
Serum Test Sample #114			µmol/L of Sample

Were samples frozen upon receipt? Yes | No

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

COMMENTS:

Please return by **September 28, 2009**

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 31

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 #37	Liquid frozen (1:1 serum:10% MPA)
VitC #47	Liquid frozen (1:1 serum:10% MPA)
VitC #74	Liquid frozen (1:1 serum:10% MPA)
VitC #114	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 RR31

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-

October 30, 2009

Dear Colleague:

Enclosed is the summary report of the results for Round Robin 31 (RR31) 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.

RR31 consisted of four *test samples* (#37, #47, #74, and #114), 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 (RR32) of the 2010 NIST Micronutrients Measurement Quality Assurance Program will be shipped **during the week of December 7, 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 B. Thomas, M.B.A.
Research Chemist
Analytical Chemistry Division
Chemical Science and Technology Laboratory

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

Enclosures

Cc: L. C. Sander

NIST

The NIST M^2QAP Vitamin C Round Robin 31 (RR31) 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 RR31 sample measurements.

Page	"All Lab" Report
1	A tabulation of results and summary statistics for Total Ascorbic Acid [TAA] in the RR31 samples and control/calibration solutions.

Serum-based Samples. One serum control and four unknowns were distributed in RR31.

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

S31:1 Serum 37, 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), S23:2 (RR23, Fall 05), S27:1 (RR27, Fall 07).

S31:2 Serum 47, ampouled in late 2001, previously distributed as sample S18:2 (RR18, Spring 03), S19:3 (RR19, Fall 03), S21:3 (RR21, Fall 04), S22:3 (RR22, Spring 05) and S24:2 (RR24, Spring 06)., S26:2 (RR26, Spring 07)

S31:3 SRM 970 level 1, ampouled in mid-1998

S31:4 Serum 114, ampouled in 1995, previously distributed as sample 188a in (RR9, Summer 96) and 27:3 (RR27, Fall 07)

Results.

1) Most participants who prepared the four 5% MPA control/calibration solutions (the three "Dilute Solutions" and the "Diluent") did so correctly. The criteria used to evaluate this success are: the density of the 5% MPA (≈1.03 gm/mL), the observed wavelength maximum of "Dilute Solution #1"(≈244 nm), the observed absorbance at that maximum (≈0.58 OD), the calculated $E^{1\%}$ #1"(≈560 dL/g·cm). On the evidence of MPA density, one participant prepared the solutions in 2.5% MPA; this may have contributed to systematically low results for the unknowns. On the evidence of the maximum absorbance and calculated $E^{1\%}$ #1, one participant had spectrophotometer issues; this had no impact on the [TAA] measurements.

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 -1.0 to 0.9 and slopes range from 0.96 to 1.14).

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

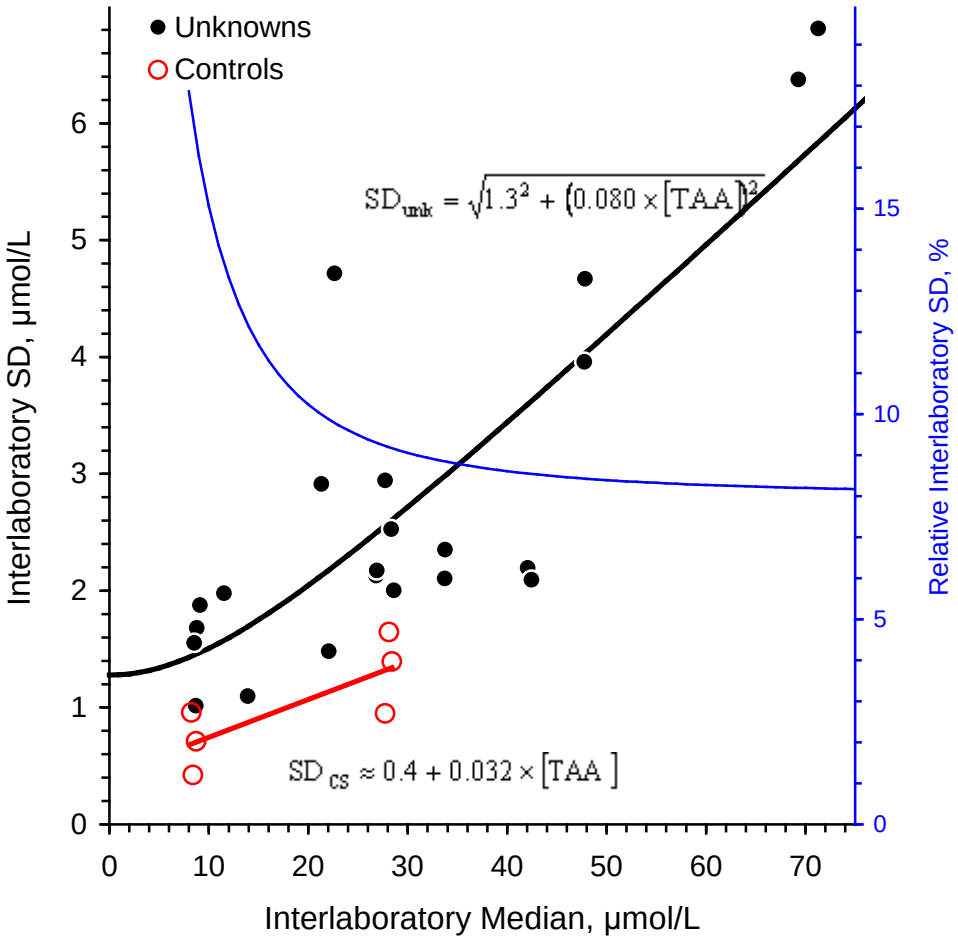
4) There is no evidence of sample degradation in any of the materials.

5) The Figure below displays the interlaboratory MADe (a robust estimate of the standard deviation) as a function of the interlaboratory median for both the "Unkowns" and the "Controls". The interlab

reproducibility precision, SD_{unk}, for the unknowns is reasonably well described by the function

$$SD_{unk} = \sqrt{1.3^2 + (0.080 \times [TAA])^2} .$$

That is, SD_{unk} is 1) at least 1.3 µmol/L regardless of the [TAA] and 2) at high [TAA] levels the *relative* SD_{unk}, RSD_{unk}, is about 8%. The SD_{unk} is displayed as a thick black line, RSD_{unk} is displayed as a blue line.



While there are too few data for confident assessment, the reproducibility precision for the two materials that have been provided as Controls, SD_{CS}, is consistently smaller than for the Unknowns. It appears, therefore, that there is "room for improvement" in the interlaboratory comparability of [TAA] measurements.

Appendix G. "All-Lab Report" for RR31

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" 31 - Fall 2009

		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	S31:1	S31:2	S31:3	S31:4	Inter	Slope	R^2	RMS
VC-MA	10/09/09	58.8	29.6	14.8	60.2	30.0	14.9	0.0	-0.14	1.02	1.000	0.2	1.036	244.	0.5660	546.1	32.0	22.1	33.8	7.7	29.4	-1.09	1.05	0.999	0.5
VC-MB	05/08/09	58.2	28.5	14.6	60.7	30.2	15.5	0.0	0.23	1.04	1.000	0.3	1.030	243.	0.5800	565.7	28.4	23.2	35.4	8.5	29.9	-0.46	1.06	1.000	0.2
VC-MC	02/10/09	59.1	29.6	15.0	56.9	28.6	14.2	0.0	-0.06	0.96	1.000	0.1	1.016	243.	0.5663	544.0	27.5	16.7	28.5	7.6	23.2	-0.14	0.82	0.987	1.3
VC-ME	23/07/09	56.7	28.4	14.2	57.8	28.6	14.7	0.0	0.03	1.02	1.000	0.3	1.031	243.	0.5714	572.0	31.8	25.7	38.8	9.7	34.9	-0.17	1.18	0.995	1.1
VC-MG	28/08/09	62.8	31.5	16.4	64.1	31.8	16.1	0.0	-0.32	1.02	1.000	0.4	1.028	243.6	0.6100	551.3	28.0	22.1	33.7	8.2	28.6	-0.31	1.01	1.000	0.1
VC-MH	13/10/09	61.2	30.5	14.9	61.0	30.5	15.1	0.0	0.08	1.00	1.000	0.1	1.027	244.1	0.6038	560.3	28.2	21.4	32.7	8.3	28.1	0.04	0.97	1.000	0.2
VC-MI	23/09/09	55.4	28.3	13.9	56.7	27.5	14.8	0.0	-0.06	1.02	0.999	1.0	1.030				28.4	21.5	35.3	10.5	28.7	1.55	0.97	0.988	1.4
VC-MJ	25/09/09	59.2	30.5	15.2	67.2	33.4	15.3	0.0	-1.01	1.14	0.999	1.1	1.023	254a	0.368a	352.7a	30.1	26.0	37.8	11.1	*b*	2.23	1.06	1.000	0.4
VC-MK	16/09/09	57.9	29.3	14.5	57.7	29.3	15.0	1.0	0.85	0.98	1.000	0.2	1.029	245.	0.1761	172.6	30.0	24.5	36.0	10.5	28.0	2.29	0.97	0.983	1.7
VC-MN	24/09/09	63.0	31.6	15.8	62.5	30.8	14.3	0.0	-0.64	1.00	0.999	0.7	1.029	243.7	0.6178	556.7	28.7	22.1	32.7	11.3	27.0	4.00	0.83	0.995	0.8
VC-MP	04/08/09																	21.2	32.5	8.5	31.5	0.00	1.01	0.972	2.3
VC-MU	11/09/09																25.9	18.2	31.3	7.4	24.7	-1.17	0.93	0.989	1.3
VC-NE	02/11/09	60.3	29.6	14.2	60.3	30.1	14.3	0.0	0.14	1.00	1.000	0.3	1.032	243.5	0.5950	560.1	27.9	21.5	32.1	7.8	25.7	-0.11	0.94	0.995	0.9
N		10	10	10	10	10	10	10					N	10	8	8	11	12	12	12	11				
Average		59.2	29.8	14.9	60.5	30.1	15.0	0.1					Average	1.028	243.7	0.5364	508.6	29.0	22.1	34.0	9.1	28.5			
SD		2.5	1.2	0.7	3.4	1.7	0.6	0.3					SD	0.005	0.7	0.1470	136.1	1.8	2.7	2.8	1.4	3.1			
Min		55.4	28.29	13.9	56.7	27.50	14.2	0.0					Min	1.016	243.0	0.1761	172.6	25.9	16.7	28.5	7.4	23.2			
%25		58.0	28.69	14.5	57.7	28.77	14.7	0.0					%25	1.027	243.0	0.5662	545.6	28.1	21.4	32.6	8.1	27.5			
Median		59.0	29.64	14.9	60.4	30.09	15.0	0.0					Median	1.029	243.7	0.5757	554.0	28.4	22.1	33.7	8.5	28.6			
%75		60.7	30.53	15.1	62.1	30.69	15.3	0.0					%75	1.030	244.0	0.6054	561.7	30.0	23.6	35.5	10.5	29.7			
Max		63.0	31.58	16.4	67.2	33.36	16.1	1.0					Max	1.036	245.0	0.6178	572.0	32.0	26.0	38.8	11.3	34.9			
MADe		2.4	1.5	0.5	4.0	1.7	0.5	0.0					eSD	0.0	0.8	0.0280	13.2	1.4	1.5	2.3	1.6	2.0			
CV		4	5	3	7	6	3						CV	0.22	0.33	4.9	2.4	5	7	7	18	7			

G2

a) 5% Trichloroacetic acid solution

b) Mislabeled sample

Appendix H. Representative "Individualized Report" for RR31

Each participant in RR31 received an "Individualized Report" reflecting their reported results. The following two pages are the "Individualized Report" for participant "VC-MA".

Vitamin C "Round Robin" 31 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
03/20/07	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
03/03/09	30	HPLC-EC	1.037	242.0	0.569	555.6	0.2	1.03	1.000	0.40
09/10/09	31	HPLC-EC	1.036	244.0	0.566	546.1	-0.1	1.02	1.000	0.20
		Mean	1.035	243.0	0.57	554.8			Pooled SEE	0.53
		SD	0.002	0.9	0.00	5.3				
		CV	0.18	0.37	0.8	0.9				

[TAA] mmol/Lsample

Date	RR	Sample	Rep_1	Rep_2	F_{adj}	Mean	SD_{dup}	N	Mean	SD_{repeat}	SD_{reprod}
10/17/05	23	CS#2	29.4	30.5	1.0	30.0	0.8	6	28.9	0.5	1.8
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				
09/10/09	31	CS#2	**31.8**	**32.2**	1.0	32.0	0.3				
03/20/03	18	S18:2	35.1	36.0	1.0	35.6	0.6	7	34.9	0.3	1.1
11/13/03	19	S19:3	35.9	35.8	1.0	35.9	0.1				
09/13/04	21	S21:3	33.2	32.9	1.0	33.0	0.2				
03/08/05	22	S22:3	35.7	35.6	1.0	35.6	0.1				
03/09/06	24	S24:2	35.8	35.5	1.0	35.6	0.2				
03/20/07	26	S26:2	35.0	35.4	1.0	35.2	0.3				
09/10/09	31	S31:2	**33.9**	**33.7**	1.0	33.8	0.2				
09/23/98	11	S11:1:A	15.5	13.9	0.5	7.4	0.6	12	8.2	0.3	0.5
04/02/99	12	S12:1:A	14.5	15.8	0.5	7.6	0.5				
09/17/01	13	S13:1	8.4	8.5	1.0	8.5	0.1				
09/27/01	14	S14:3	8.0	7.7	1.0	7.8	0.2				
09/18/01	15	S15:1	8.9	8.7	1.0	8.8	0.1				
11/18/02	16	S16:1	8.8	8.8	1.0	8.8	0.0				
11/13/03	19	S19:4	7.8	8.6	1.0	8.2	0.5				
02/23/04	20	S20:3	8.3	8.1	1.0	8.2	0.1				
10/17/05	23	S23:4	8.6	8.8	1.0	8.7	0.1				
08/28/06	25	S25:1	8.7	8.5	1.0	8.6	0.2				
08/11/08	29	S29:2	8.3	8.4	1.0	8.3	0.1				
09/10/09	31	S31:3	**7.3**	**8.1**	1.0	7.7	0.5				
06/19/96	09	S09:2	51.7	51.1	0.5	25.7	0.2	3	27.9	0.4	2.0
10/05/07	27	S27:3	28.5	28.6	1.0	28.5	0.1				
09/10/09	31	S31:4	**29.0**	**29.9**	1.0	29.4	0.7				

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

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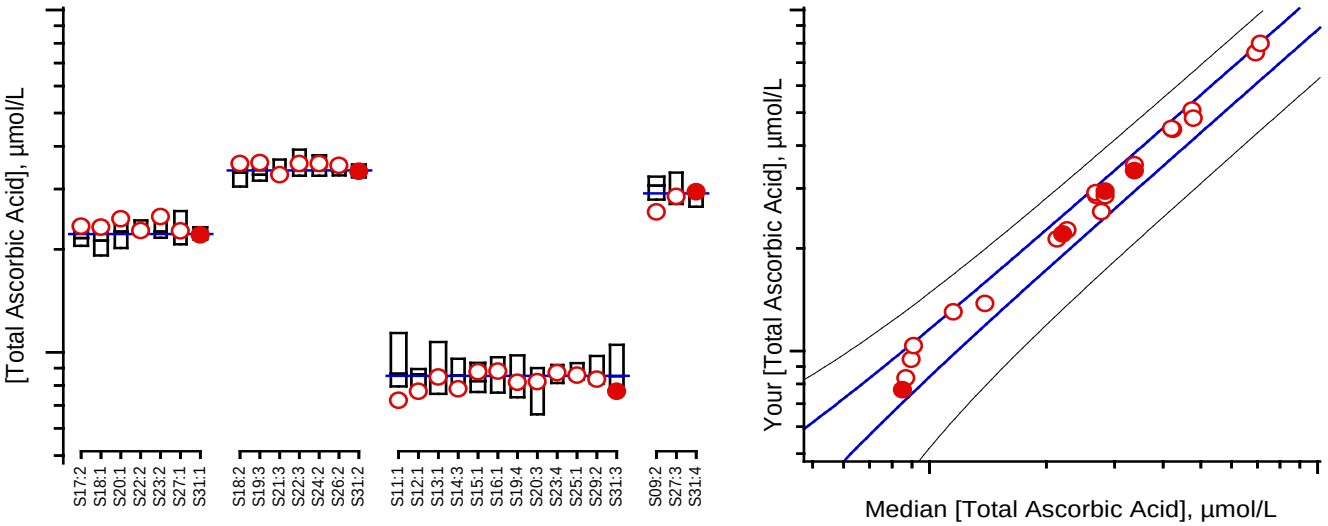
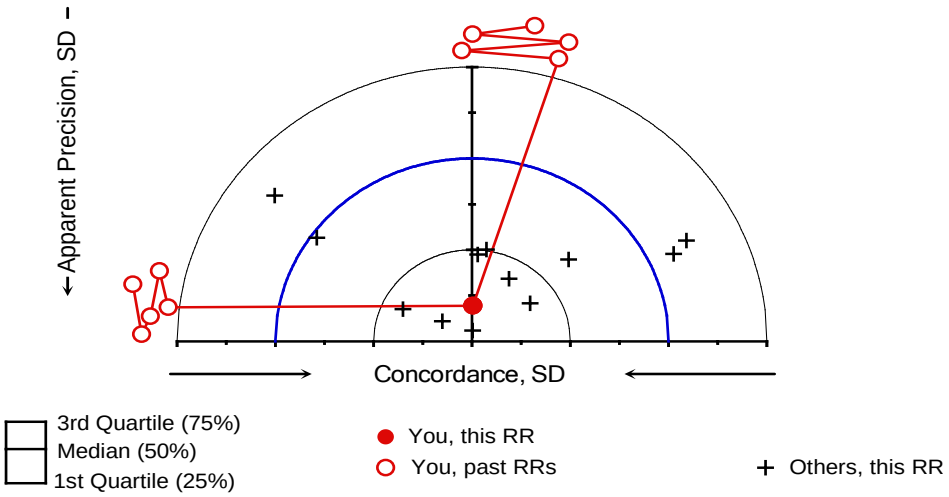
Vitamin C "Round Robin" 31 Report: Participant VC-MA

Total Ascorbic Acid, µmol/mL





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

Sample	Comments
S31:1	VitC #37 previously distributed in RRs 17, 18, 20, 22, 23, 27
S31:2	VitC #47, previously distributed in RRs 18, 19, 21, 22, 24, 26
S31:3	VitC #74, previously distributed in RRs 11, 12, 13, 14, 15, 16, 19, 20, 23, 25, 29
S31:4	VitC #114, previously distributed in RRs 9, 27