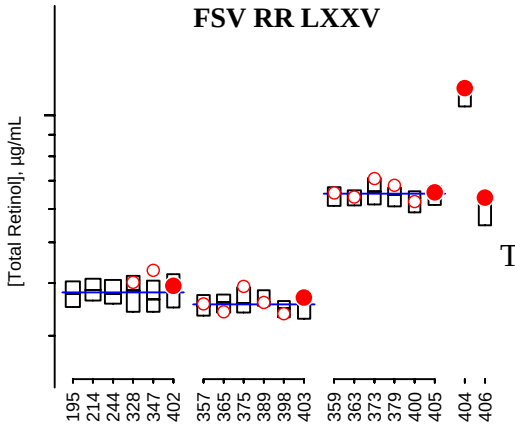
NISTIR 7880-43

NIST Micronutrients Measurement Quality Assurance Program Winter 2014 Comparability Studies

Results for Round Robin LXXV
Fat-Soluble Vitamins and Carotenoids in Human Serum
and Round Robin 40 Ascorbic Acid in Human Serum



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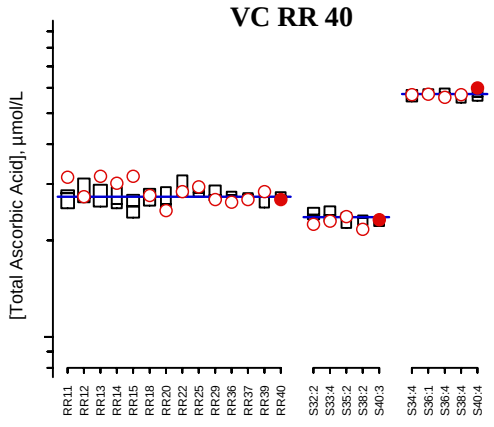

National Institute of Standards and Technology
U.S. Department of Commerce

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Results for Round Robin LXXV Fat-Soluble Vitamins and Carotenoids in Human Serum and Round Robin 40 Ascorbic Acid in Human Serum

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Materials Measurement Laboratory

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June 2015

U.S. Department of Commerce
Penny Pritzker, Secretary

National Institute of Standards and Technology
Willie E. May, Under Secretary of Commerce for Standards and Technology and Director

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Abstract

The National Institute of Standards and Technology coordinates the Micronutrients Measurement Quality Assurance Program (MMQAP) for laboratories that measure fat- and water-soluble vitamins and carotenoids in human serum and plasma. This report describes the design of and results for the Winter 2014 MMQAP measurement comparability improvement studies: 1) Round Robin LXXV Fat-Soluble Vitamins and Carotenoids in Human Serum and 2) Round Robin 40 Total Ascorbic Acid in Human Serum. The materials for both studies were shipped to participants in January 2014. To help reduce overhead costs and avoid raising participation fees, the materials for Round Robin LXXVI Fat-Soluble Vitamins and Carotenoids in Human Serum and Round Robin 41 Total Ascorbic Acid in Human Serum were also shipped in the same shipping container. Participants in Round Robins LXXV and 41 were requested to provide measurement results by April 18, 2014.

Keywords

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

Table of Contents

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 LXXV: Fat-Soluble Vitamins and Carotenoids in Human Serum

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin LXXV comparability study (hereafter referred to as RR75) received one lyophilized and four liquid-frozen human serum test samples for analysis. Unless multiple vials were previously requested, participants received one vial of each serum. These sera were shipped on dry ice to participants in January 2014 in the same shipping package as the Round Robin LXXVI (RR76) samples but in a separately labeled plastic bag. Participants were requested to provide measurement results by April 18, 2014. 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 RR75 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 All-Lab 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 the Individualized Report are described in detail elsewhere [3]. An example Individualized Report is reproduced as Appendix D.

Round Robin 40: Vitamin C in Human Serum

Participants in the MMQAP Vitamin C in Human Serum Round Robin 40 comparability study (hereafter referred to as RR40) received four frozen serum test samples and two frozen control sera. Unless multiple vials were previously requested, participants received one vial of each material. These materials were shipped on dry ice to participants in January 2014 in the same shipping package as the Round Robin 41 (RR41) samples but in a separately labeled plastic bag. Participants were requested to provide measurement results by April 18, 2014. 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).

The final report delivered to every participant in RR40 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 All-Lab 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 the Individualized 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 RR75

The following three items were included in each package shipped to an RR75 participant:

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

The RR75 samples were backed in a sealed waterproof bag and labeled:

NIST MMQAP-FSV: **RR LXXV**
Micronutrients Measurement Fat-Soluble Vitamin
Quality Assurance Program

Winter 2014 Samples

Results due on or before:
April 18, 2014

A cover letter describing both RR75 and RR76, datasheets for both studies, and packing lists for both studies were enclosed in a sealed waterproof bag 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-

February 10, 2014

Dear Colleague:

Enclosed are samples for the fat-soluble vitamins and carotenoids in serum studies for the 2014 NIST Micronutrients Measurement Quality Assurance Program. Sample details are provided below.

Comparability study/Round Robin (RR)	Sample description	Results due
RR75	Sera 402 - 406	April 18, 2014
RR76	Sera 407 – 411	September 15, 2014

The package for each study consists of one vial of lyophilized serum and one vial each of four 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 each serum sample. If a value obtained is below your limit of quantification, please indicate this result on the form as "*nq*" (*Not Quantified*) or "<*x*" where *x* is your established limit of quantification. Results are due to NIST for each study as indicated above. Results received more than two weeks after the due date may not be included in the summary report for the study. The feedback report concerning each study will be distributed in May and October 2014, respectively. Please contact us immediately if this schedule is problematic for your laboratory.

Samples should be allowed to stand at room temperature under subdued light until thawed. Add 1.00 mL reagent-grade water to the lyophilized serum. 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 by e-mail to david.duewer@nist.gov or fax to 301-977-0685. If you have questions or comments regarding the studies, please contact us at 301-975-3120 (Jeanice); jbthomas@nist.gov or 301-975-3935 (Dave); david.duewer@nist.gov.

Sincerely,

Jeanice B. Thomas, M.B.A.
Research Chemist
Chemical Sciences Division
Material Measurement Laboratory

David L. Duewer, Ph.D.
Research Chemometrician
Chemical Sciences Division
Material Measurement Laboratory

Enclosures

NIST

Participant #: _____ Date: _____

Round Robin LXXV: Human Sera

NIST Micronutrients Measurement Quality Assurance Program

Analyte	402	403	404	405	406	Units*
total retinol						
trans-retinol						
retinyl palmitate						
α-tocopherol						
γ/β-tocopherol						
δ-tocopherol						
total β-carotene						
trans-β-carotene						
total cis-β-carotene						
total α-carotene						
total lycopene						
trans-lycopene						
total β-cryptoxanthin						
total α-cryptoxanthin						
total lutein						
total zeaxanthin						
total lutein&zeaxanthin						
total coenzyme Q10						
ubiquinol (QH_2)						
ubiquinone (Qox)						
phylloquinone (K_1)						
25-hydroxyvitamin D						
Phytoene						
Phytofluene						

* we prefer **µg/mL**

Were the samples frozen when received? Yes | No

Comments:

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

Please return results by
18-April-2014

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

Participant #: _____ Date: _____

Fat-Soluble Vitamins Round Robin LXXV

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
#402	Lyophilized	Yes	5 mL clear / silver
#403	Liquid frozen	No	3 mL amber / red
#404	Liquid frozen	No	2 mL clear / green
#405	Liquid frozen	No	2 mL amber / green
#406	Liquid frozen	No	3 mL amber / green

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) Email (david.duewer@nist.gov) or fax (301-977-0685) us the following information:

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 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 RR75

The following four pages are the final report for RR75 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.

May 13, 2014

UNITED STATES DEPARTMENT OF COMMERCE
National Institute of Standards and Technology
Gaithersburg, Maryland 20899-8390

Dear Colleague:

Enclosed is the summary report of the results for "Round Robin" LXXV (RR75) of the 2014 NIST Micronutrients Measurement Quality Assurance Program (MMQAP) 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. RR75 (Sera 402 - 406) consisted of one vial of lyophilized serum and one vial each of four liquid-frozen serum samples. Details regarding the samples can be found in the enclosed report.

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 pleased that MMQAP participant Professor Garry Duthie of the Rowett Institute of Nutrition and Health, University of Aberdeen, Scotland is providing the (**free**) on-line recipe book Stovies Reloaded: Traditional Scottish Recipes Made Healthier at http://www.abdn.ac.uk/rowett/policy-industry/stovies.php. Prof. Duthie states that *"Stovies Reloaded" is our light-hearted take on traditional Scottish recipes and how they can be adapted in the light of increased nutritional knowledge. … We hope that this book may play a small part in encouraging people to rediscover cooking skills, take an interest in traditional Scottish fare and eat a more healthy diet*." This website also provides links to a wealth of other information developed at the Rowett Institute relating to nutrition, food safety, and human health.

The results for fat-soluble vitamins and carotenoids in serum RRLXXVI and vitamin C in serum RR41 are due on or before **September 15, 2014.** We will send you a reminder in August. 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, M.B.A.
Research Chemist
Chemical Sciences Division
Material Measurement Laboratory

David L. Duewer, Ph.D.
Research Chemometrician
Chemical Sciences Division
Material Measurement Laboratory

Enclosures

Cc: L.C. Sander

The NIST MMQAP Round Robin LXXV (RR75) report consists of:

Page	All-Lab Report
1-4	A listing of all results and statistics for analytes reported by more than one participant.
5	The 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 eight other participants.
n+1	The graphical Comparability Summary (target plot) of measurement performance.

Samples. Five samples were distributed to each participant in RR75.

Serum	Description	Prior Distributions
402	Lyophilized, augmented, multi-donor, 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; #347:RR64-9/08
403	Fresh-frozen, native, multi-donor, prepared in 2009. This is Level I of SRM 968e.	#357:RR66-9/09, #365:RR67-3/10, #376:RR69-3/11, #389:RR72-9/12
404	Fresh-frozen, augmented, single donor, prepared in 2013. This material was prepared from the same (reasonably) normal serum as #406 but was augmented with *trans*-retinol, retinyl palmitate, and β-tocopherol,	First MMQAP distribution
405	Fresh-frozen, native, multi-donor, prepared in 2009. This is Level III of SRM 968e.	#359:RR66-9/09, #363:RR67-3/10, #373:RR69-3/11, #379:RR70-9/11
406	Fresh-frozen, augmented, single donor, prepared in 2013. This material was prepared from the same (reasonably) normal serum as #404 but was augmented with *cis*-retinol and γ-tocopherol.	First MMQAP distribution

Results

1) Stability: There have been no significant changes in either the level nor the variability in any of the analytes in the 20-year old lyophilized Serum 402 nor in the five-year old fresh-frozen SRM 968e materials, Sera 403 and 405.

2) γ/β-Tocopherol: A year or two ago, we were asked why we designate as "γ/β-tocopherol" what most literature reports as "γ-tocopherol." While in our experience reliably separating γ from β requires specialized methods, it had been some years since we last investigated tocopherol separations and agreed that it was quite possible that some of you may have developed methods that resolved these two isomers. The Sera {404,406} pair was produced in part to test this. The native γ/β concentration

in the serum used to prepare the pair was about 1.1 µg/mL. Serum 404 was spiked with β-tocopherol and Serum 406 was spiked with γ-tocopherol, both to have high-normal levels of about 4 µg/mL.

Figure 1 compares the reported values for γ/β-tocopherol in Serum 404 to those in Serum 406, with each solid circle representing the values reported by one participant. The solid red box represents the native γ/β content of the original serum; the red line represents the expected ratio of the two spikes. Scatter along the red line represents calibration issues that affect both samples proportionally. Scatter perpendicular to the red line combines measurement imprecision with potential bias due to the nature of the spikes. The relative absence of perpendicular scatter strongly suggests that all participants measure the same "γ/β-tocopherol" measurand regardless of whether "γ/β" is mostly γ or mostly β. Further, there were no reported comments on the chromatographic shape of the γ/β-tocopherol peak in either serum. We conclude that all participants are indeed measuring "γ/β-tocopherol."

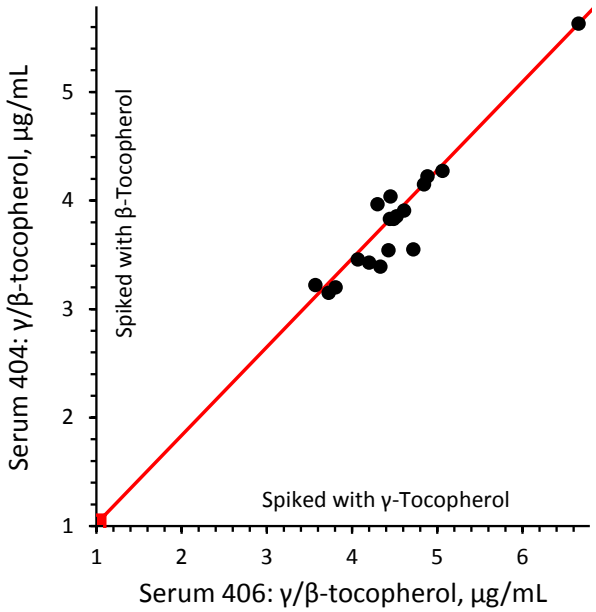

Figure 1: Comparison of Reported Values of γ/β-Tocopherol in Sera 404 and 406

3) <u>*cis*-/*trans*-Retinol</u>: The Sera {404,406} pair was also designed to evaluate measurement systems that report "total retinol" or "*trans*-retinol." The total retinol concentration in the serum used to prepare the pair was about 0.42 µg/mL. Serum 404 was spiked with all-*trans*-retinol to have a very high level of about 1.1 µg/mL and Serum 406 was spiked with a smaller quantity of 13-*cis*-retinol to have a fairly normal total retinol level of about 0.6 µg/mL.

Figure 2 compares the reported values for total retinol in Serum 406 to those in Serum 404. As above, the solid red box represents the native total content of the original serum, the red line represents the expected ratio of the two spikes, scatter along the red line represents calibration issues that affect both samples proportionally, and scatter perpendicular to the red line combines measurement imprecision with potential bias due to the nature of the spikes. In contrast to "γ/β-tocopherol", several participants reported total retinol values for Serum 406 that apparently exclude the contribution from the 13-*cis* spike.

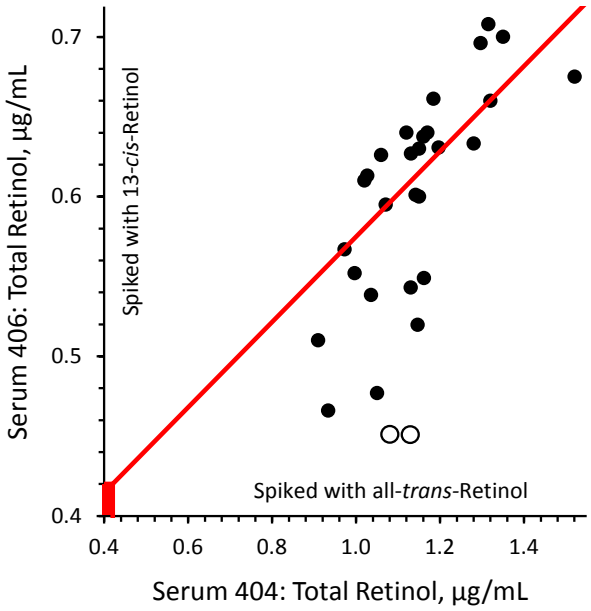

Figure 2: Comparison of Reported values of Total Retinol in Sera 404 and 406

Indeed, the open circles in Figure 2 represent results reported by participants who resolved the two isomers. Figure 3 presents the relevant sections of their chromatograms for Sera 404 and 406. The peak for 13-*cis*-retinol elutes somewhat earlier than does all-*trans*-retinol in both methods.

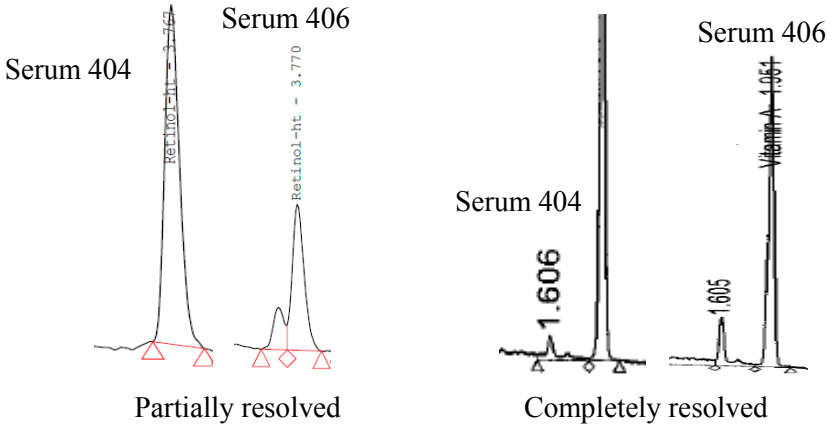

Figure 3: Chromatograms of Retinol in Sera 404 and Serum 406

Given the evidence of a somewhat variable definition of "total retinol" among the participants, we strongly encourage all of you to examine the retinol sections of your chromatograms for Sera 404 and 406. If you are resolving the 13-*cis* but not including it in the total integration, you may want to either report *trans*-retinol or modify your method. If you are not resolving the 13-*cis* peak but report *trans*-retinol, you may want to report total retinol or modify your method.

Appendix C. "All-Lab Report" for RR75

The following six pages are the "All-Lab Report" for RR75 as provided to all participants, with two exceptions:

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

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

Round Robin LXXV Laboratory Results

	Total Retinol, µg/mL					trans-Retinol, µg/mL					Retinyl Palmitate, µg/mL					α-Tocopherol, µg/mL					γ/β-Tocopherol, µg/mL				
Lab	402	403	404	405	406	402	403	404	405	406	402	403	404	405	406	402	403	404	405	406	402	403	404	405	406
FSV-BB	0.394	0.370	1.16	0.657	0.637						0.080	0.007	0.43	0.087	0.026	7.24	7.03	8.99	18.2	8.91	1.37	1.86	3.83	2.27	4.45
FSV-BC	0.354	0.318	1.08	0.630	0.451																				
FSV-BD	0.421	0.414	1.52	0.762	0.675											8.20	7.20	8.90	20.6	8.70					
FSV-BE	0.303	0.285	0.93	0.520	0.466											6.44	5.93	7.68	17.6	7.73	1.15	1.54	3.20	2.05	3.81
FSV-BF	0.390	0.360	1.17	0.680	0.640											6.50	6.30	7.50	17.4	7.50					
FSV-BFa	0.420	0.400	1.32	0.730	0.660											7.40	7.30	9.10	20.5	9.20					
FSV-BG	0.420	0.357	1.20	0.651	0.631						0.088	0.016	0.59	0.107	0.022	6.66	6.35	8.57	19.4	8.54	1.36	1.86	4.04	2.23	4.45
FSV-BH	0.282	0.321	1.05	0.589	0.477											6.31	5.81	7.75	17.0	7.67	1.22	1.65	3.43	2.17	4.20
FSV-BJ	0.376	0.325	1.28	0.662	0.633											7.51	6.51	8.29	19.6	8.49	1.44	1.87	4.15	2.37	4.85
FSV-BK	0.369	0.343	1.07	0.619	0.595											6.65	6.47	8.03	18.7	8.15					
FSV-BL	0.400	0.370	1.15	0.660	0.600											6.50	6.50	7.80	16.8	7.80					
FSV-BM	0.340	0.260	1.02	0.580	0.610											7.30	5.10	8.40	17.2	8.30					
FSV-BN	0.426	0.365	1.25	0.687	0.639											6.55	5.79	7.11	17.0	6.98					
FSV-BO	0.360	0.370	1.15	0.680	0.630											7.19	6.76	8.10	19.2	8.40	1.03	1.57	3.22	1.99	3.57
FSV-BR	*≥0.300*	*≥0.370*	*≥1.120*	*≥0.650*	*≥0.640*	0.300	0.370	1.120	0.650	0.640						7.60	6.50	7.90	18.8	7.30					
FSV-BS	0.422	0.346	1.18	0.661	0.661																				
FSV-BT	0.428	0.361	0.97	0.608	0.567											6.90	6.80	7.81	18.8	8.11	1.29	1.74	3.15	2.56	3.73
FSV-BU	0.207	0.340	1.15	0.650	0.520											6.99	6.66	8.41	19.2	8.70	1.54	1.75	3.91	2.35	4.61
FSV-BV	0.298	0.360	1.13	0.581	0.543											6.95	6.86	8.35	16.9	8.79	1.23	1.84	3.55	2.10	4.72
FSV-BW	0.510	0.590	1.35	0.820	0.700						0.228	0.011	1.00	0.197	0.048	6.20	6.25	7.30	17.6	7.17	1.74	2.53	5.63	3.12	6.66
FSV-CD	0.350	0.316	1.00	0.660	0.552						*nq*	*nq*	0.51	*nq*	*nq*	6.82	6.45	7.91	18.3	8.19	1.14	1.64	3.46	2.08	4.07
FSV-CE	0.357	0.329	1.04	0.637	0.538											5.53	7.95	8.43	21.7	8.91					
FSV-CG	0.431	0.399	1.13	0.670	0.627											7.08	6.83	8.27	18.8	8.36	1.42	2.00	3.85	2.50	4.52
FSV-CI	0.418	0.402	1.30	0.726	0.696						0.119	0.017	0.54	0.110	0.027	8.00	7.28	8.90	20.2	9.00	1.47	1.97	4.22	2.47	4.89
FSV-CO																6.16	6.25	7.68	18.5	8.05	1.34	1.92	3.97	2.56	4.82
FSV-CZ	0.395	0.431	1.32	0.696	0.708											6.92	7.04	8.89	17.3	8.83	1.36	1.71	3.39	2.64	4.34
FSV-DD	0.320	0.300	0.91	0.530	0.510																				
FSV-DV	0.360	0.358	1.14	0.673	0.601											7.60	7.20	8.60	18.0	9.40					
FSV-EE																									
FSV-EZ	0.391	0.346	1.16	0.672	0.549						0.104	<0.02	0.55	0.073	<0.02	6.74	6.53	8.22	19.3	7.97	1.38	1.79	3.97	2.28	4.30
FSV-FK	0.401	0.335	1.06	0.611	0.626											7.30	6.30	8.20	17.6	8.00					
FSV-FZ	*≥0.386*	*≥0.346*	*≥1.129*	*≥0.637*	*≥0.451*	0.386	0.346	1.129	0.637	0.451	0.063	0.012	0.25	0.081	0.017	7.48	6.63	8.58	18.9	8.11	1.41	1.75	3.54	2.24	4.43
FSV-GD	0.341	0.344	1.03	0.613	0.613											6.79	6.61	8.11	18.6	8.18	1.27	1.82	3.83	2.31	4.49
N	29	29	29	29	29	2	2	2	2	2	6	5	7	6	5	29	29	29	29	29	18	18	18	18	18
Min	0.207	0.260	0.91	0.520	0.451	0.300	0.346	1.120	0.637	0.451	0.063	0.007	0.25	0.073	0.017	5.53	5.10	7.11	16.8	6.98	1.03	1.54	3.15	1.99	3.57
Median	0.390	0.357	1.15	0.660	0.613	0.343	0.358	1.125	0.644	0.546	0.096	0.012	0.54	0.097	0.026	6.92	6.53	8.22	18.6	8.19	1.36	1.80	3.83	2.29	4.45
Max	0.510	0.590	1.52	0.820	0.708	0.386	0.370	1.129	0.650	0.640	0.228	0.017	1.00	0.197	0.048	8.20	7.95	9.10	21.7	9.40	1.74	2.53	5.63	3.12	6.66
eSD	0.048	0.033	0.14	0.045	0.070						0.029	0.006	0.08	0.021	0.006	0.56	0.41	0.53	1.5	0.68	0.13	0.12	0.45	0.27	0.38
eCV	12	9	13	7	11						30	46	15	22	23	8	6	6	8	8	9	7	12	12	9
N_{past}	39	30	0	31	0	8	8	0	8	0	10	7	0	9	0	40	30	0	31	0	20	17	0	18	0
$Median_{past}$	0.378	0.356		0.650		0.429	0.347		0.618		0.115	0.011		0.094		7.04	6.79		18.7		1.33	1.79		2.22	
SD_{past}	0.040	0.028		0.053		0.055	0.035		0.087		0.031	0.004		0.025		0.71	0.54		1.4		0.12	0.17		0.24	
NAV	0.390	0.357	1.15	0.660	0.613						0.096	0.012	0.54	0.097	0.026	6.92	6.53	8.22	18.6	8.19	1.36	1.80	3.83	2.29	4.45
NAU	0.048	0.033	0.14	0.052	0.070						0.029	0.011	0.12	0.026	0.012	0.58	0.56	0.66	1.5	0.68	0.15	0.19	0.45	0.27	0.39

C2

Round Robin LXXV Laboratory Results

Lab	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				
	402	403	404	405	406	402	403	404	405	406	402	403	404	405	406	402	403	404	405	406	402	403	404	405	406
FSV-BB	0.078	0.094	0.129	0.202	0.136	0.314	0.082	0.415	0.371	0.399	0.286	0.079	0.378	0.338	0.367	0.028	0.003	0.037	0.033	0.033	0.014	0.006	0.043	0.008	0.029
FSV-BC																									
FSV-BD																									
FSV-BE						0.276	0.080	0.348	0.395	0.345															
FSV-BF						0.481	0.083	0.318	0.399	0.382															
FSV-BFa																									
FSV-BG						0.276	0.086	0.366	0.405	0.344											0.017	0.010	0.036	0.014	0.032
FSV-BH						0.230	0.066	0.317	0.376	0.304											*nq*	*nd*	0.028	*nq*	0.026
FSV-BJ						0.390	0.086	0.464	0.574	0.522											*nq*	*nq*	0.026	*nq*	*nq*
FSV-BK																									
FSV-BL																									
FSV-BM																									
FSV-BN						0.326	0.089	0.379	0.406	0.371											0.041	0.007	0.055	0.027	0.053
FSV-BO						0.339	0.082	0.363	0.336	0.374											0.018	0.005	0.042	0.009	0.041
FSV-BR																									
FSV-BS						0.308	0.103	0.396	0.478	0.350															
FSV-BT	0.078	0.108	0.121	0.187	0.126	0.354	0.106	0.381	0.431	0.412	0.322	0.097	0.355	0.413	0.386	0.032	0.009	0.026	0.018	0.025	0.014	0.008	0.037	0.017	0.037
FSV-BU						0.352	0.103	0.393	0.393	0.400											0.017	0.007	0.046	0.012	0.049
FSV-BV						0.351	0.101	0.449	0.428	0.443											0.022	0.014	0.046	0.015	0.045
FSV-BW						0.440	0.108	0.532	0.590	0.518											0.010	0.015	0.047	0.069	0.043
FSV-CD						0.315	0.094	0.395	0.420	0.420											*nq*	*nq*	*nq*	*nq*	*nq*
FSV-CE						0.229	0.201	0.748	0.928	0.838															
FSV-CG	0.284	0.418	0.444	0.588	0.445	0.298	0.085	0.362	0.391	0.370	0.274	0.080	0.340	0.371	0.348	0.023	0.005	0.022	0.020	0.022	0.020	0.008	0.046	0.018	0.047
FSV-CI						0.260	0.075	0.348	0.404	0.357											0.021	0.016	0.043	0.038	0.045
FSV-CO						0.235	0.095	0.380	0.398	0.374											0.010	0.007	0.038	0.011	0.041
FSV-CZ						0.235	0.104	0.272	0.305	0.288															
FSV-DD																									
FSV-DV																									
FSV-EE																									
FSV-EZ						*≥0.398*	*≥0.070*	*≥0.396*	*≥0.300*	*≥0.375*	0.398	0.070	0.396	0.300	0.375										
FSV-FK						*≥0.411*	*≥0.090*	*≥0.415*	*≥0.393*	*≥0.404*	0.411	0.090	0.415	0.393	0.404										
FSV-FZ						0.337	0.095	0.416	0.401	0.404															
FSV-GD						0.307	0.111	0.369	0.410	0.369	0.270	0.094	0.326	0.362	0.326	0.037	0.017	0.043	0.048	0.043	0.014	0.009	0.045	0.013	0.043
N	3	3	3	3	3	21	21	21	21	21	6	6	6	6	6	4	4	4	4	4	12	12	14	12	13
Min	0.078	0.094	0.121	0.187	0.126	0.229	0.066	0.272	0.305	0.288	0.270	0.070	0.326	0.300	0.326	0.023	0.003	0.022	0.018	0.022	0.010	0.005	0.026	0.008	0.026
Median	0.078	0.108	0.129	0.202	0.136	0.314	0.094	0.380	0.404	0.374	0.304	0.085	0.367	0.367	0.371	0.030	0.007	0.031	0.027	0.029	0.017	0.008	0.043	0.015	0.043
Max	0.284	0.418	0.444	0.588	0.445	0.481	0.201	0.748	0.928	0.838	0.411	0.097	0.415	0.413	0.404	0.037	0.017	0.043	0.048	0.043	0.041	0.016	0.055	0.069	0.053
eSD						0.057	0.015	0.047	0.024	0.043	0.048	0.011	0.042	0.041	0.028	0.007	0.005	0.011	0.011	0.008	0.005	0.002	0.005	0.005	0.006
eCV						18	16	12	6	11	16	13	11	11	8	22	68	35	43	27	28	29	13	35	14
N_{past}	13	7	0	6	0	29	20	0	21	0	9	6	0	7	0	7	6	0	7	0	22	14	0	15	0
$Median_{past}$	0.074	0.110		0.210		0.340	0.090		0.396		0.316	0.084		0.351		0.028	0.005		0.018		0.018	0.008		0.016	
SD_{past}	0.008	0.025		0.030		0.042	0.013		0.064		0.030	0.006		0.058		0.010	0.003		0.005		0.005	0.003		0.008	
NAV	0.078	0.108	0.129	0.202	0.136	0.314	0.094	0.380	0.404	0.374	0.304	0.085	0.367	0.367	0.371	0.030	0.007	0.031	0.027	0.029	0.017	0.008	0.043	0.015	0.043
NAU						0.057	0.016	0.054	0.057	0.053	0.048	0.012	0.042	0.041	0.039						0.006	0.004	0.013	0.006	0.014

C3

Round Robin LXXV 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	402	403	404	405	406	402	403	404	405	406	402	403	404	405	406	402	403	404	405	406	402	403	404	405	406
FSV-BB	0.063	0.210	0.262	0.81	0.256	0.026	0.099	0.118	0.322	0.114	0.010	0.046	0.063	0.036	0.060	0.004	0.016	0.026	0.014	0.027	0.033	0.090	0.080	0.175	0.071
FSV-BC																									
FSV-BD																									
FSV-BE																									
FSV-BF																									
FSV-BFa																									
FSV-BG	0.062	0.224	0.283	1.02	0.257	0.028	0.117	0.141	0.463	0.131	0.012	0.050	0.068	0.038	0.065										
FSV-BH	0.057	0.211	0.269	1.05	0.259						*nd*	0.053	0.066	0.027	0.065						0.017	0.067	0.059	0.102	0.057
FSV-BJ	0.075	0.226	0.289	1.31	0.303						*nq*	*nq*	0.057	*nq*	0.061						*nq*	0.095	0.088	0.149	0.096
FSV-BK																									
FSV-BL																									
FSV-BM																									
FSV-BN	0.054	0.201	0.269	1.01	0.249						0.014	0.075	0.095	0.045	0.092										
FSV-BO	0.065	0.187	0.227	0.68	0.234						0.005	0.055	0.069	0.028	0.072						0.024	0.106	0.090	0.131	0.093
FSV-BR																									
FSV-BS	0.126	0.227	0.302	1.23	0.228	0.081	0.140	0.175	0.681	0.142	0.021	0.046	0.055	0.030	0.055						0.031	0.098	0.084	0.123	0.081
FSV-BT	0.058	0.147	0.167	0.58	0.169	0.041	0.129	0.143	0.506	0.147	0.010	0.039	0.048	0.039	0.049	0.004	0.013	0.018	0.021	0.018	0.031	0.085	0.063	0.114	0.070
FSV-BU	0.051	0.203	0.263	0.99	0.303						0.010	0.046	0.057	0.031	0.063										
FSV-BV	0.064	0.236	0.294	0.94	0.289						0.006	0.049	0.064	0.026	0.064										
FSV-BW	0.060	0.241	0.243	1.17	0.275																				
FSV-CD	0.063	0.222	0.312	1.06	0.321						*nq*	0.053	0.065	*nq*	0.063	*nq*	*nq*	0.028	*nq*	0.030					
FSV-CE																									
FSV-CG	0.067	0.224	0.274	0.98	0.281	0.029	0.120	0.141	0.440	0.144	0.014	0.068	0.085	0.060	0.087										
FSV-CI																					0.018	0.062	0.061	0.102	0.060
FSV-CO	0.047	0.209	0.245	0.87	0.239						0.009	0.047	0.061	0.028	0.061										
FSV-CZ																									
FSV-DD																									
FSV-DV																									
FSV-EE																									
FSV-EZ						0.059	0.166	0.229	0.504	0.221											0.021	0.086	0.064	0.123	0.074
FSV-FK																									
FSV-FZ																									
FSV-GD	0.068	0.202	0.275	0.85	0.251																				
N	15	15	15	15	15	6	6	6	6	6	10	12	13	11	13	2	2	3	2	3	7	8	8	8	8
Min	0.047	0.147	0.167	0.58	0.169	0.026	0.099	0.118	0.322	0.114	0.005	0.039	0.048	0.026	0.049	0.004	0.013	0.018	0.014	0.018	0.017	0.062	0.059	0.102	0.057
Median	0.063	0.211	0.269	0.99	0.257	0.035	0.125	0.142	0.483	0.143	0.010	0.050	0.064	0.031	0.063	0.004	0.014	0.026	0.018	0.027	0.024	0.088	0.072	0.123	0.073
Max	0.126	0.241	0.312	1.31	0.321	0.081	0.166	0.229	0.681	0.221	0.021	0.075	0.095	0.060	0.092	0.004	0.016	0.028	0.021	0.030	0.033	0.106	0.090	0.175	0.096
eSD	0.007	0.019	0.030	0.18	0.034	0.012	0.017	0.019	0.049	0.012	0.004	0.005	0.007	0.007	0.003						0.010	0.013	0.017	0.022	0.015
eCV	12	9	11	18	13	34	13	13	10	8	44	11	12	24	5						43	15	24	18	21
N_{past}	22	15	0	16	0	8	7	0	7	0	17	14	0	16	0	7	6	0	5	0	12	7	0	8	0
$Median_{past}$	0.071	0.220		0.93		0.037	0.115		0.408		0.011	0.051		0.032		0.006	0.018		0.023		0.027	0.077		0.124	
SD_{past}	0.019	0.027		0.17		0.008	0.018		0.095		0.005	0.011		0.014		0.002	0.003		0.008		0.006	0.016		0.027	
NAV	0.063	0.211	0.269	0.99	0.257	0.035	0.125	0.142	0.483	0.143	0.010	0.050	0.064	0.031	0.063			0.026		0.027	0.024	0.088	0.072	0.123	0.073
NAU	0.019	0.051	0.062	0.18	0.060	0.012	0.022	0.025	0.092	0.025	0.004	0.012	0.015	0.008	0.015						0.010	0.017	0.017	0.023	0.015

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Round Robin LXXV Laboratory Results

	Total Zeaxanthin, µg/mL					Total Lutein&Zeaxanthin, µg/mL					Coenzyme Q10, µg/mL					Phylloquinone (K1), ng/mL				
Lab	402	403	404	405	406	402	403	404	405	406	402	403	404	405	406	402	403	404	405	406
FSV-BB	0.010	0.038	0.037	0.053	0.048	0.043	0.128	0.117	0.228	0.120										
FSV-BC																				
FSV-BD																				
FSV-BE											0.360	1.02	1.04	1.97	1.060	1.509	0.955	1.862	5.463	1.818
FSV-BF																				
FSV-BFa																				
FSV-BG						0.038	0.111	0.110	0.172	0.111										
FSV-BH	0.007	0.048	0.038	0.029	0.036	0.024	0.115	0.097	0.131	0.093										
FSV-BJ											0.388	1.09	0.66	1.40	0.809					
FSV-BK																				
FSV-BL																				
FSV-BM																				
FSV-BN						0.013	0.091	0.091	0.113	0.075										
FSV-BO	0.009	0.019	0.012	0.020	0.011	*0.033*	*0.125*	*0.102*	*0.151*	*0.104*	0.320	0.84	0.90	1.54	0.840					
FSV-BR																				
FSV-BS	0.006	0.032	0.029	0.022	0.029	0.037	0.130	0.113	0.145	0.110										
FSV-BT	0.009	0.034	0.028	0.027	0.032	0.040	0.119	0.091	0.141	0.102	0.252	0.69	0.73	1.53	0.585					
FSV-BU						0.025	0.105	0.102	0.157	0.090										
FSV-BV						0.030	0.108	0.100	0.121	0.097										
FSV-BW											0.250	0.71	0.74	1.15	0.705					
FSV-CD						*nq*	0.125	0.108	0.160	0.116										
FSV-CE																				
FSV-CG						0.035	0.110	0.100	0.144	0.100										
FSV-CI	0.006	0.022	0.019	0.014	0.025	*0.024*	*0.084*	*0.080*	*0.116*	*0.085*	0.270	0.85	0.88	1.03	0.850	0.399	0.232	0.343	1.345	0.479
FSV-CO						0.044	0.112	0.110	0.143	0.112										
FSV-CZ											0.640	0.74	1.26	1.94	1.007					
FSV-DD																				
FSV-DV																				
FSV-EE											0.298	0.93	0.97	1.44	0.874					
FSV-EZ											0.323	0.68	0.78	1.18	0.749					
FSV-FK																0.400	0.260	0.410	1.540	0.440
FSV-FZ																				
FSV-GD											0.300	0.83	0.76	1.27	0.800					
N	6	6	6	6	6	12	13	13	13	13	10	10	10	10	10	3	3	3	3	3
Min	0.006	0.019	0.012	0.014	0.011	0.013	0.084	0.080	0.113	0.075	0.250	0.68	0.66	1.03	0.585	0.399	0.232	0.343	1.345	0.440
Median	0.008	0.033	0.029	0.024	0.030	0.034	0.112	0.102	0.144	0.102	0.310	0.84	0.83	1.42	0.825	0.400	0.260	0.410	1.540	0.479
Max	0.010	0.048	0.038	0.053	0.048	0.044	0.130	0.117	0.228	0.120	0.640	1.09	1.26	1.97	1.060	1.509	0.955	1.862	5.463	1.818
eSD	0.002	0.012	0.013	0.007	0.008	0.011	0.011	0.012	0.019	0.013	0.067	0.16	0.14	0.29	0.093					
eCV	28	37	47	27	27	32	9	12	13	13	22	20	17	20	11					
N_{past}	9	6	0	7	0	19	15	0	16	0	7	8	0	9	0	0	0	0	0	0
$Median_{past}$	0.010	0.034		0.027		0.038	0.111		0.148		0.323	0.86		1.36						
SD_{past}	0.004	0.012		0.013		0.008	0.020		0.036		0.027	0.10		0.29						
NAV	0.008	0.033	0.029	0.024	0.030	0.034	0.112	0.102	0.144	0.102	0.310	0.84	0.83	1.42	0.825	0.400	0.260	0.410	1.540	0.479
NAU	0.003	0.012	0.013	0.007	0.009	0.011	0.023	0.021	0.030	0.021	0.067	0.16	0.14	0.29	0.093					

C5

Round Robin LXXV Laboratory Results

Analytes Reported By One Laboratory

Values in µg/mL

Analyte	Code	402	403	404	405	406
25-hydroxyvitamin D	FSV-BH	0.014	0.007	0.021	0.022	0.020
Phytoene	FSV-BS	*nd*	*nd*	*nd*	*nd*	*nd*
Phytofluene	FSV-BS	0.024	0.058	0.056	0.265	0.058

Table Legend

Term	Definition
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
eSD	Adjusted median absolute deviation from the median of the non-NIST results
eCV	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)
NAV	NIST Assigned Value: Median for analytes reported by ≥ 5 labs
NAU	NIST Assigned Uncertainty: maximum of (0.05*NAV, eSD, SDpast, eSD_{calc})
	eSD_{calc}, 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 LXXV Laboratory Results

Comparability Summary

Lab	TR	aT	g/bT	bC	tbC	aC	TLy	TbX	TLu	TZ	L&Z
FSV-BB	1	1	1	1	1	1	1	1	2		1
FSV-BC	2										1
FSV-BD	2	2									
FSV-BE	3	1	2	1							
FSV-BF	1	1		2							
FSV-BFa	2	2									
FSV-BG	1	1	1	1		1	1	1		3	2
FSV-BH	2	2	1	2		2	1	1	1		
FSV-BJ	1	1	1	3			1	1	2	1	1
FSV-BK	1	1									
FSV-BL	1	1									
FSV-BM	2	2									
FSV-BN	1	2		1		3	1	2			2
FSV-BO	1	1	2	1		1	1	1	1	1	1
FSV-BR	1	1								2	1
FSV-BS	1			1			2	2	1	1	1
FSV-BT	1	1	2	1	1	1	2	1	1		
FSV-BU	2	1	1	1		1	1	1			1
FSV-BV	2	1	1	1		1	1	1			
FSV-BW	4	2	4	3		4	1				1
FSV-CD	1	1	2	1			1	1			1
FSV-CE	1	3		4							1
FSV-CG	1	1	1	1	1	1	1	3			
FSV-CI	2	2	1	1		3			1	1	1
FSV-CO		1	1	1		1	1	1			
FSV-CZ	2	1	1	2							
FSV-DD	2										
FSV-DV	1	2									
FSV-EZ	1	1	1	2	2				1		
FSV-FK	1	1		1	2						
FSV-FZ	2	1	1	1							
FSV-GD	1	1	1	1	1	1	1				
n	31	29	18	23	6	13	15	13	8	6	13

	TR	aT	g/bT	bC	tbC	aC	TLy	TbX	TLu	TZ	L&Z
% 1	58	69	72	70	67	69	87	77	75	67	85
% 2	35	28	22	17	33	8	13	15	25	17	15
% 3	3	3	0	9	0	15	0	8	0	17	0
% 4	3	0	6	4	0	8	0	0	0	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) summarizes your measurement performance for a given analyte relative to the consensus medians in this study. CS is the average distance (in units of standard deviation) of your measurement performance characteristics from the consensus performance. CS is calculated when the number of quantitative values you reported, N_{you}, is at least two and at least six participants reported quantitative values for the analyte.

We define CS as follows:

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

$$C = \text{Concordance} = \frac{\sum_{i=1}^{N_{you}} \frac{You_i - Median_i}{NAU_i}}{N_{you}}$$

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

NAU = NIST Assigned Uncertainty

For further details, please see

Duewer DL, Kline MC, Sharpless KE, Brown Thomas J, Gary KT. Micronutrients Measurement Quality Assurance Program: Helping participants use interlaboratory comparison exercise results to improve their long-term measurement performance. Anal Chem 1999;71(9):1870-8.

Appendix D. Representative Individualized Report for RR75

Each participant in RR75 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:

- Total Retinol
- Retinyl Palmitate
- α-Tocopherol
- γ/β-Tocopherol
- Total 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-BB.

Individualized Round Robin LXXV Report: FSV-BB

Summary

	Serum 402			Serum 403			Serum 404			Serum 405			Serum 406		
Analyte	You	NAV	n	You	NAV	n	You	NAV	n	You	NAV	n	You	NAV	n
Total Retinol	0.394	0.390	29	0.370	0.357	29	1.160	1.147	29	0.657	0.660	29	0.637	0.613	29
Retinyl Palmitate	0.08	0.10	6	0.0	0.0	5	0.4	0.5	7	0.09	0.10	6	0.03	0.03	5
α-Tocopherol	7.24	6.92	29	7.03	6.53	29	8.99	8.22	29	18.22	18.64	29	8.91	8.19	29
γ/β-Tocopherol	1.369	1.359	18	1.865	1.803	18	3.829	3.829	18	2.273	2.293	18	4.445	4.449	18
δ-Tocopherol	0.078	0.078	3	0.094	0.108	3	0.129	0.129	3	0.202	0.202	3	0.136	0.136	3
Total β-Carotene	0.314	0.314	21	0.082	0.094	21	0.415	0.380	21	0.371	0.404	21	0.399	0.374	21
trans-β-Carotene	0.286	0.304	6	0.079	0.085	6	0.378	0.367	6	0.338	0.367	6	0.367	0.371	6
Total cis-β-Carotene	0.028	0.030	4	0.003	0.007	4	0.037	0.031	4	0.033	0.027	4	0.033	0.029	4
Total α-Carotene	0.014	0.017	12	0.006	0.008	12	0.043	0.043	14	0.008	0.015	12	0.029	0.043	13
Total Lycopene	0.063	0.063	15	0.210	0.211	15	0.262	0.269	15	0.807	0.990	15	0.256	0.257	15
trans-Lycopene	0.026	0.035	6	0.099	0.125	6	0.118	0.142	6	0.322	0.483	6	0.114	0.143	6
Total β-Cryptoxanthin	0.010	0.010	10	0.046	0.050	12	0.063	0.064	13	0.036	0.031	11	0.060	0.063	13
Total α-Cryptoxanthin	0.004		2	0.016		2	0.026	0.026	3	0.014		2	0.027	0.027	3
Total Lutein	0.033	0.024	7	0.090	0.088	8	0.080	0.072	8	0.175	0.123	8	0.071	0.073	8
Total Zeaxanthin	0.010	0.008	6	0.038	0.033	6	0.037	0.029	6	0.053	0.024	6	0.048	0.030	6
Total Lutein&Zeaxanthin	0.043	0.034	12	0.128	0.112	13	0.117	0.102	13	0.228	0.144	13	0.120	0.102	13

D2

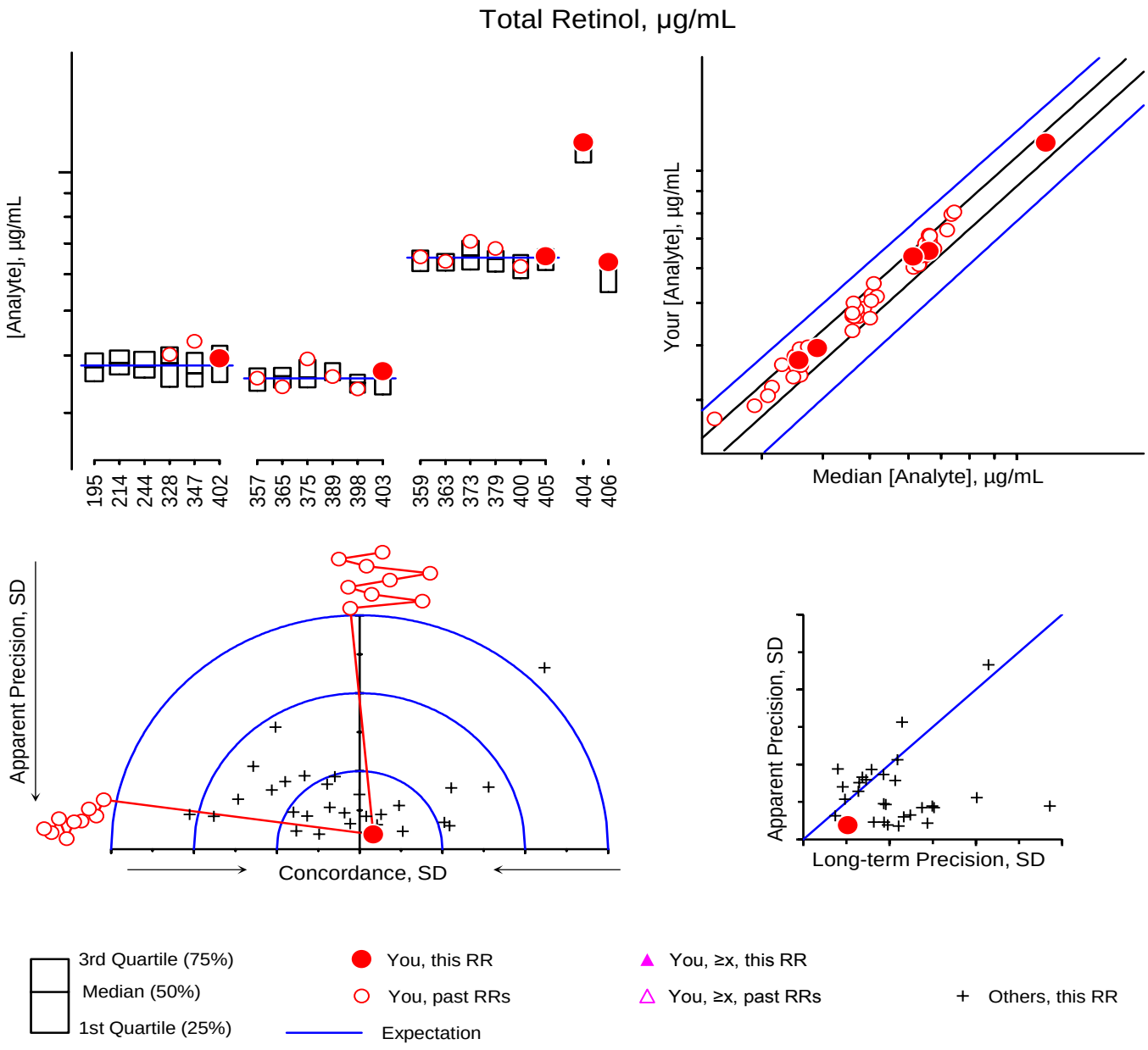
You : Your reported values for the listed analytes (micrograms/milliliter)
NAV : NIST Assigned Values, here equal to this RR's median
n : Number of non-NIST laboratories reporting quantitative values for this analyte in this serum

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 LXXV Report: FSV-BB

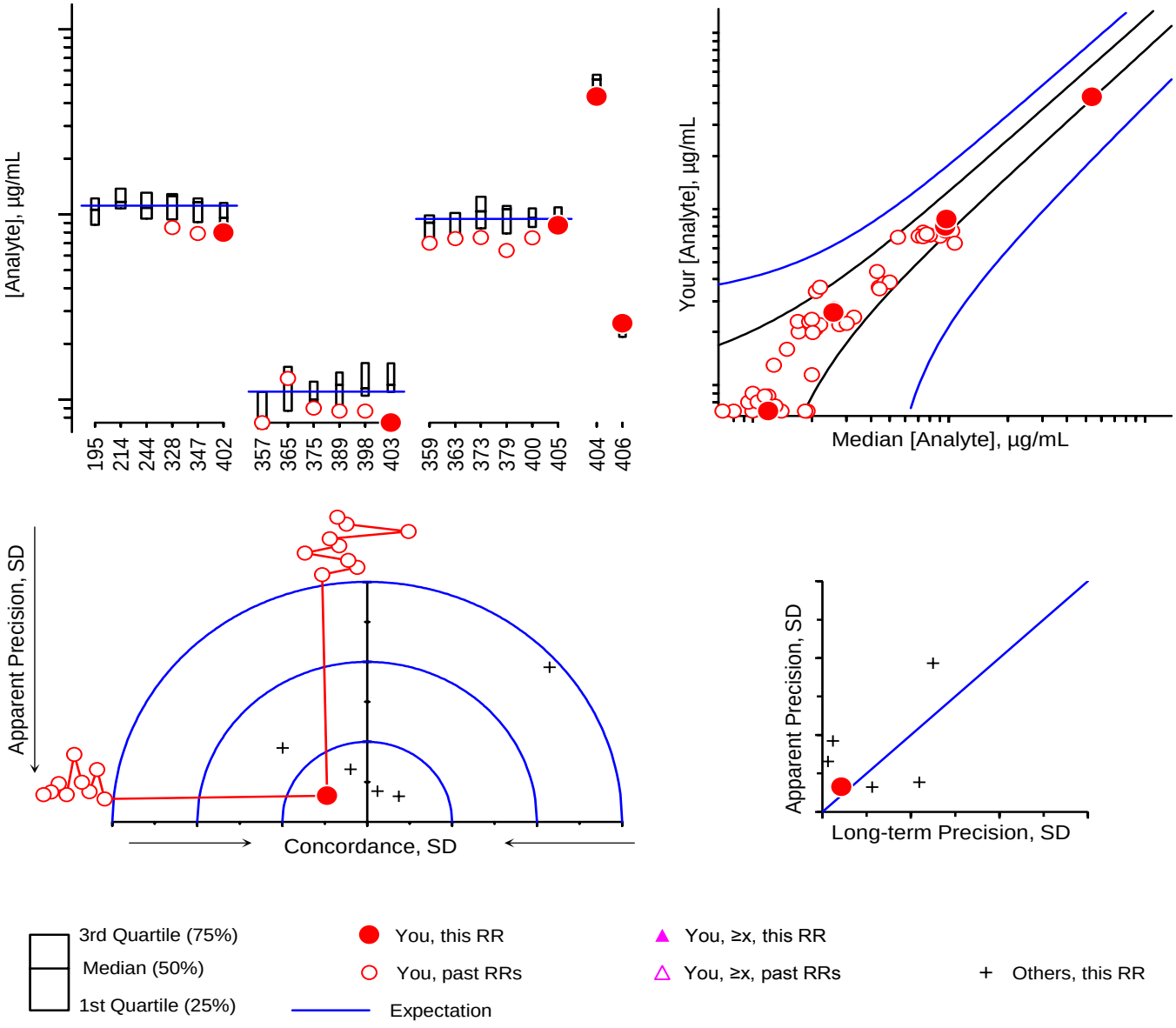


For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

Serum	Comments	History
#402	Lyophilized, augmented, multi-donor	31#195, 35#214, 43#244, 60#328, 64#347
#403	Fresh-frozen, native, multi-donor: SRM 968e I	66#357, 67#365, 69#375, 72#389, 74#398
#404	Fresh-frozen, augmented {tR,RP,βT}, single-donor	Initial distribution
#405	Fresh-frozen, native, multi-donor: SRM 968e III	66#359, 67#363, 69#373, 70#379, 74#400
#406	Fresh-frozen, augmented {cR,γT}, single-donor	Initial distribution

Individualized RR LXXV Report: FSV-BB

Retinyl Palmitate, µg/mL



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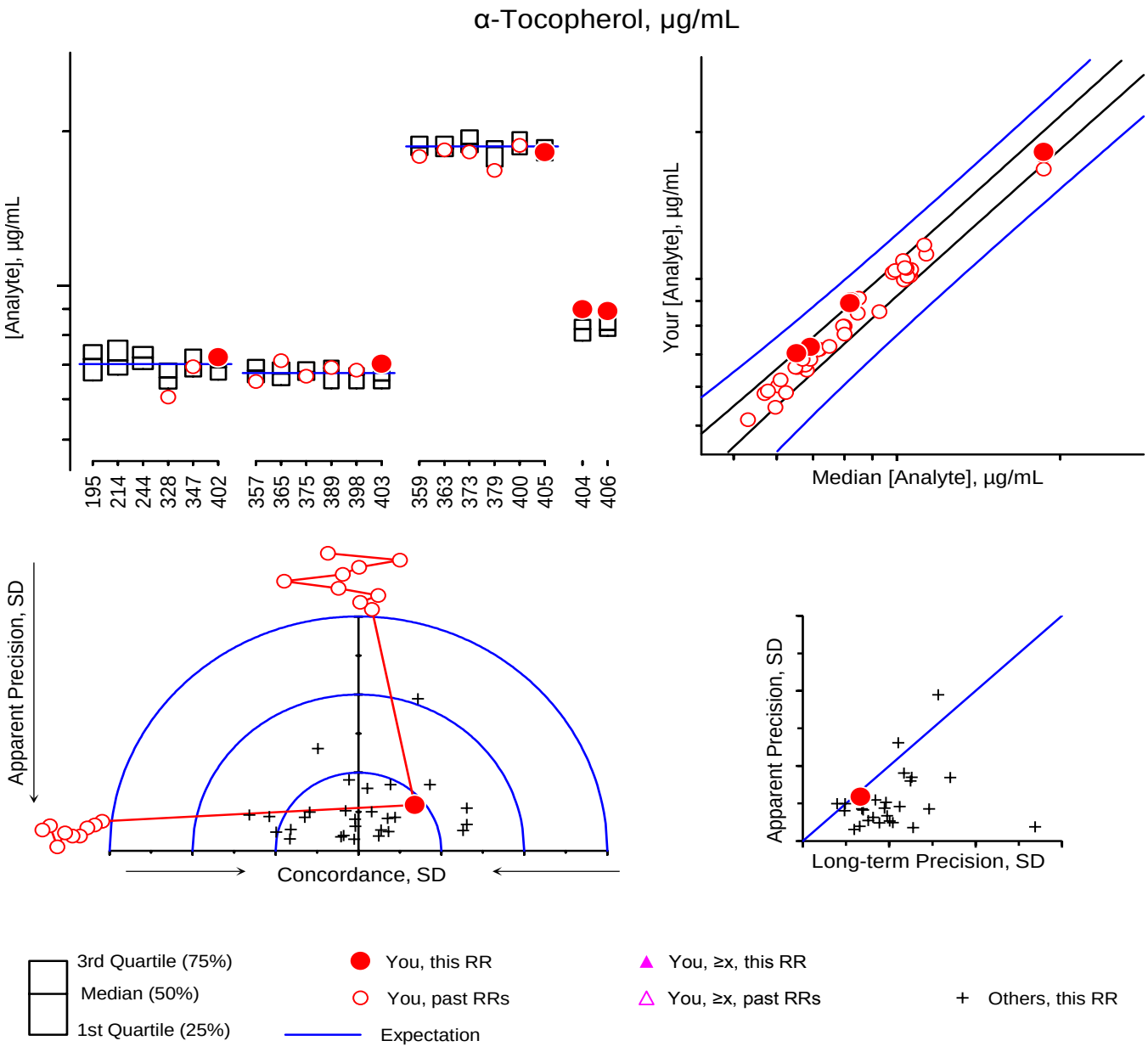
Individualized RR LXXV Report: FSV-BB



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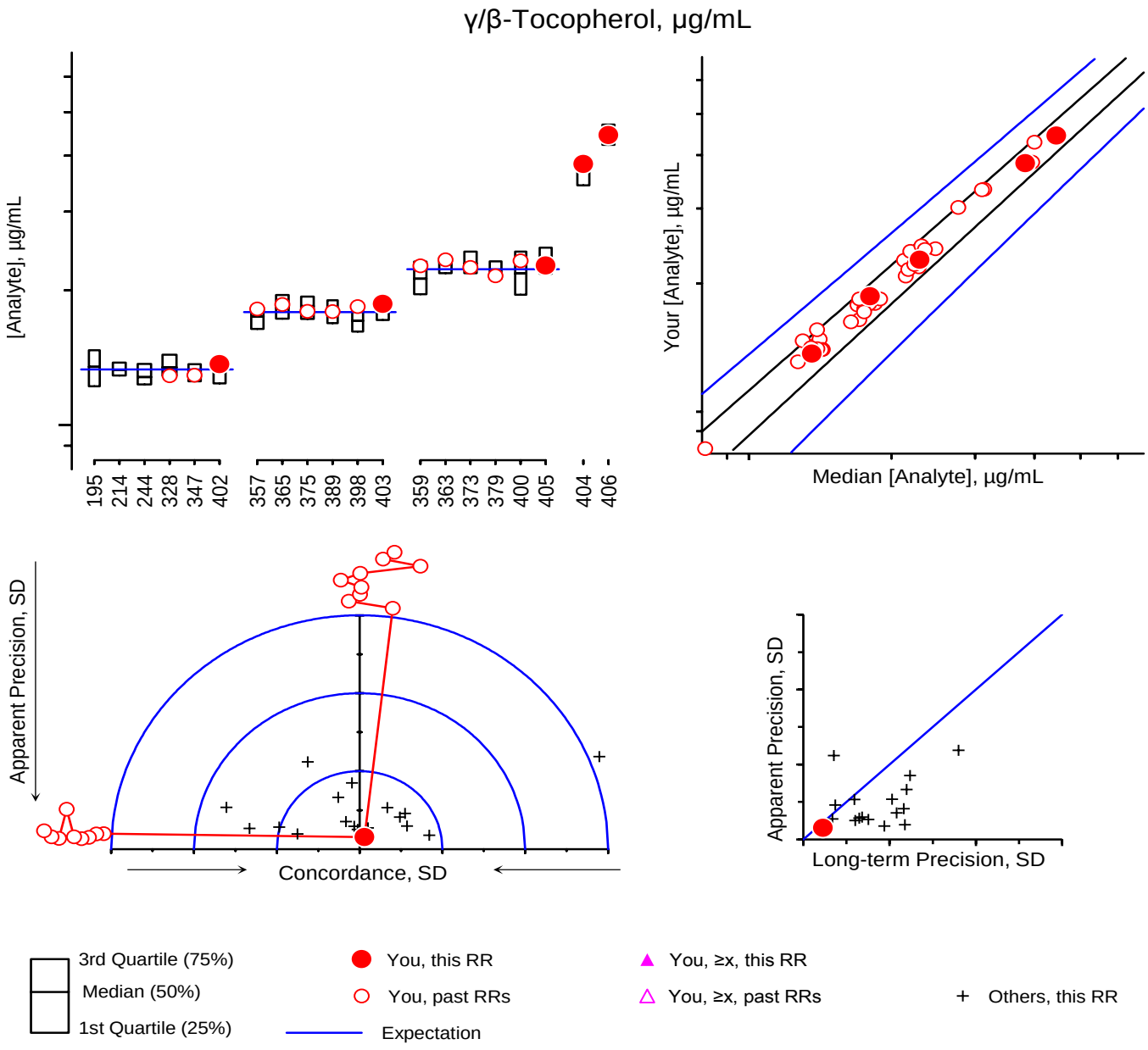
Individualized RR LXXV Report: FSV-BB



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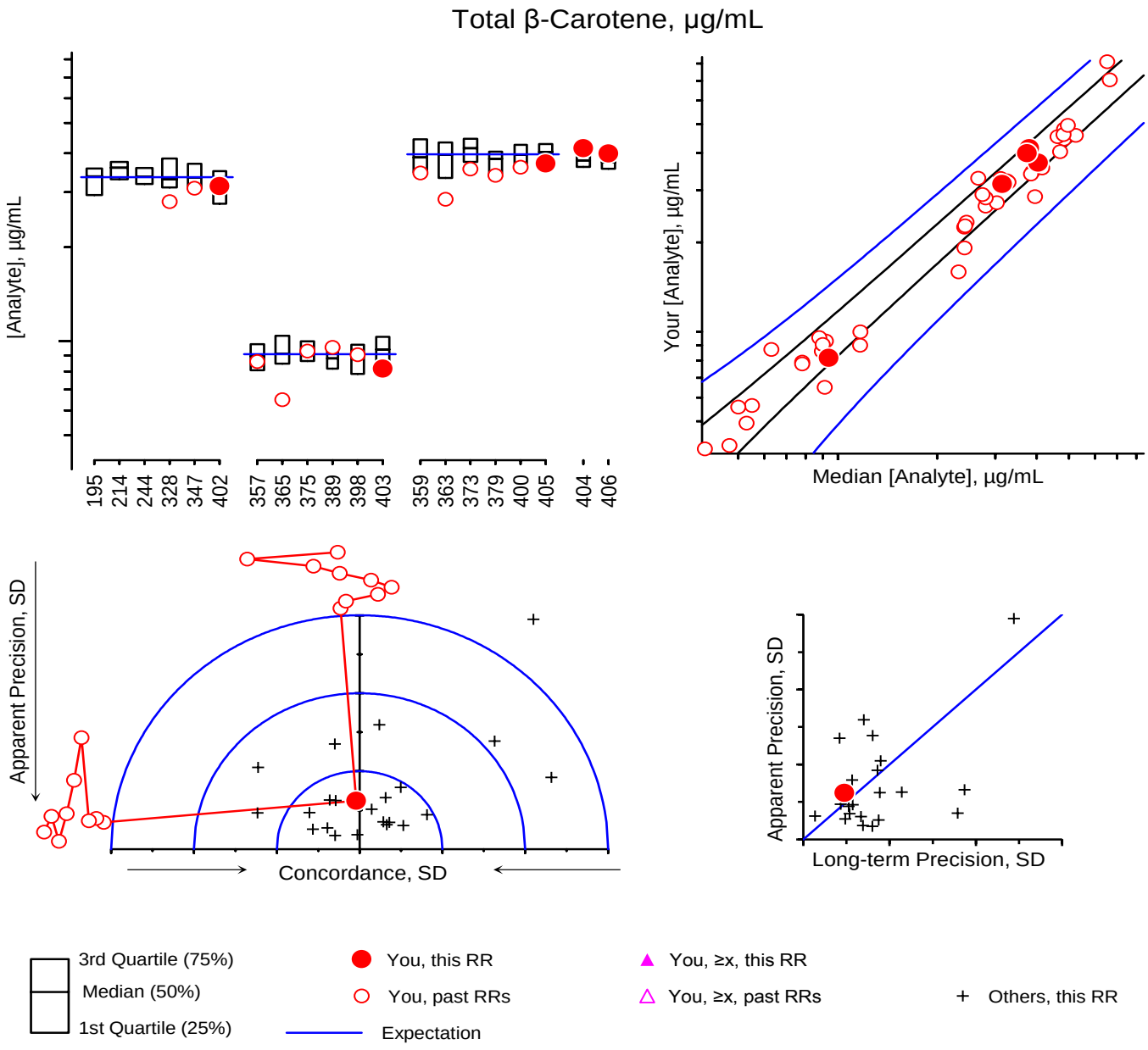
Individualized RR LXXV Report: FSV-BB



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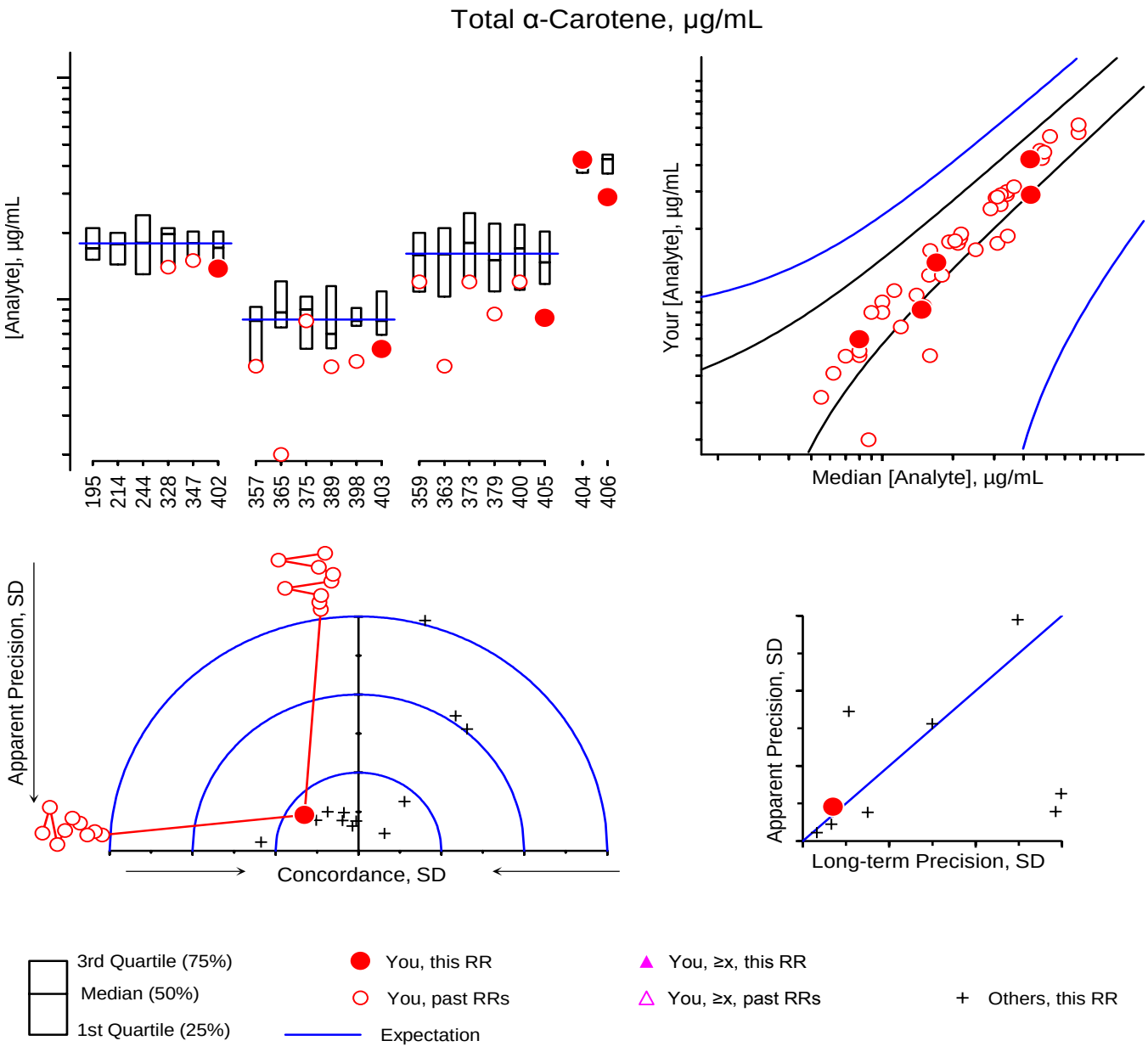
Individualized RR LXXV Report: FSV-BB



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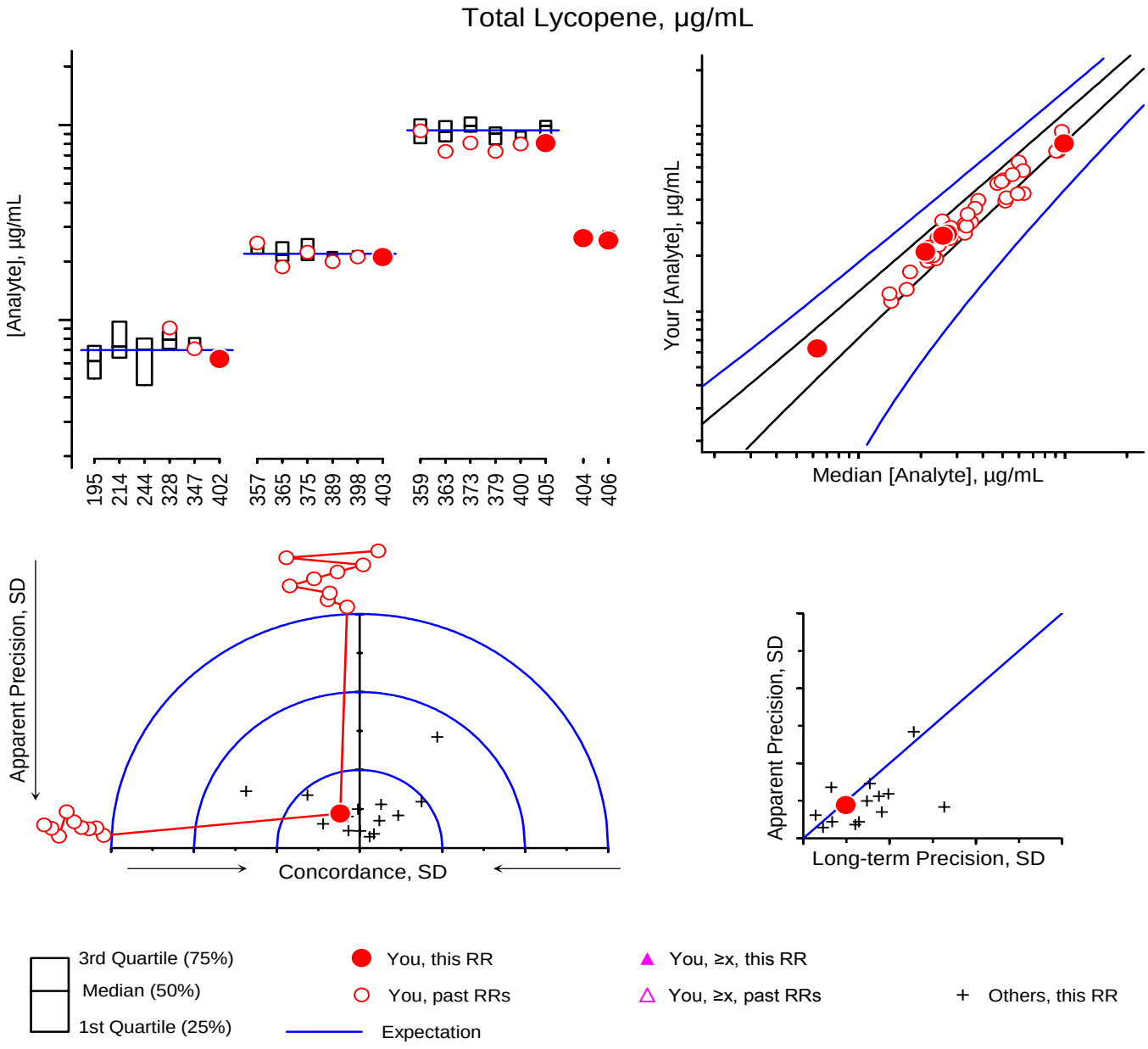
Individualized RR LXXV Report: FSV-BB



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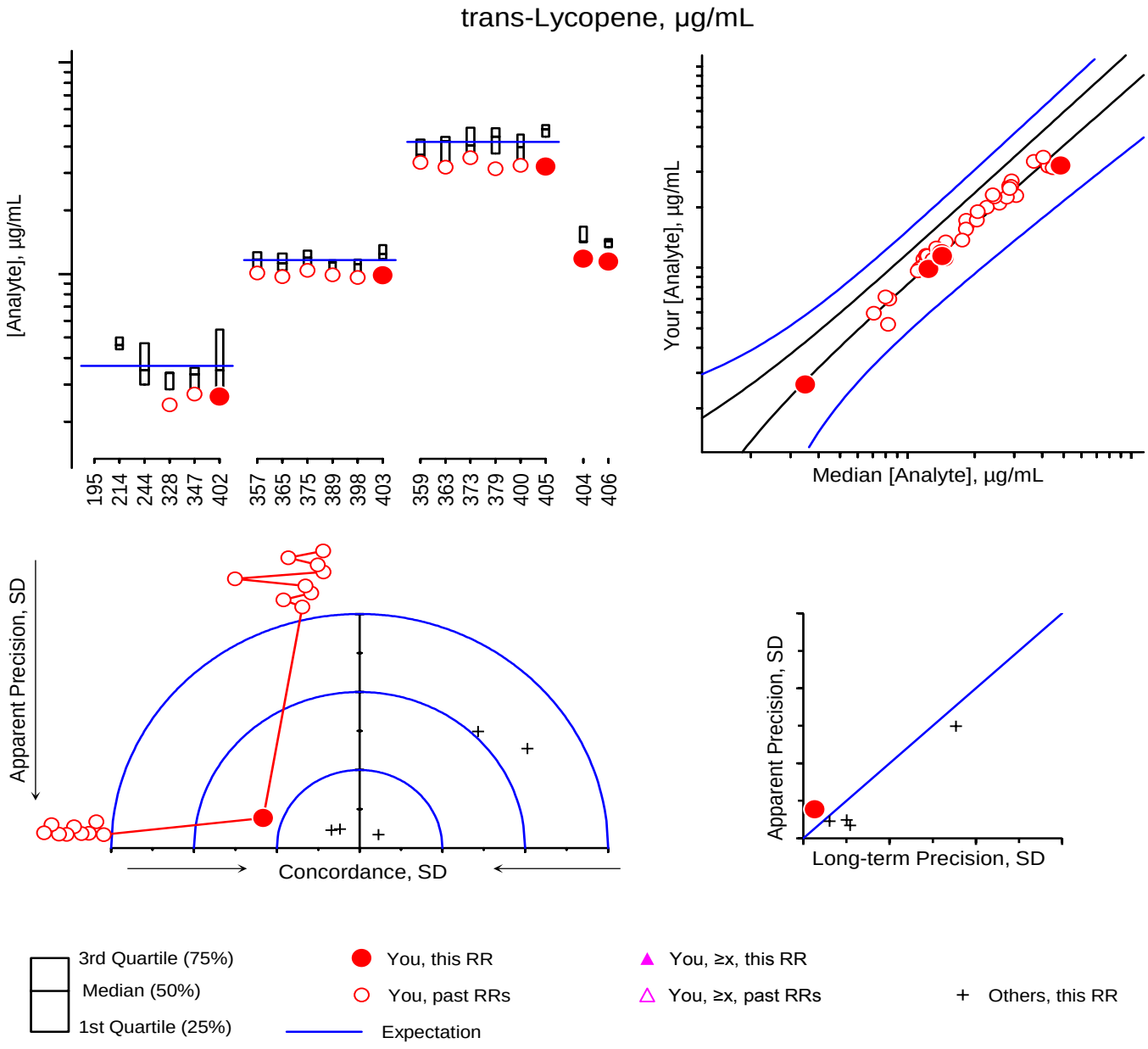
Individualized RR LXXV Report: FSV-BB



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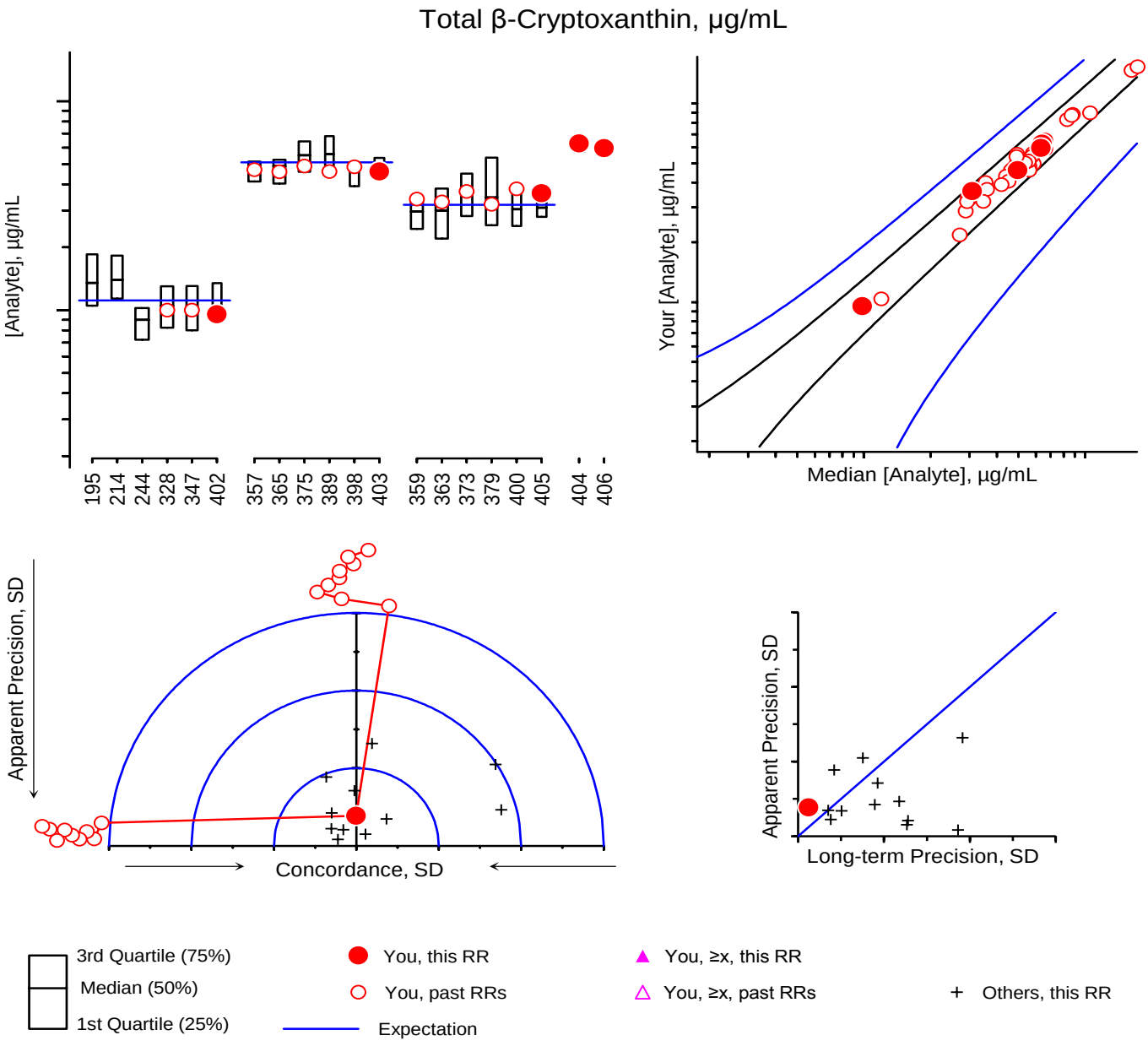
Individualized RR LXXV Report: FSV-BB



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Serum	Comments	History
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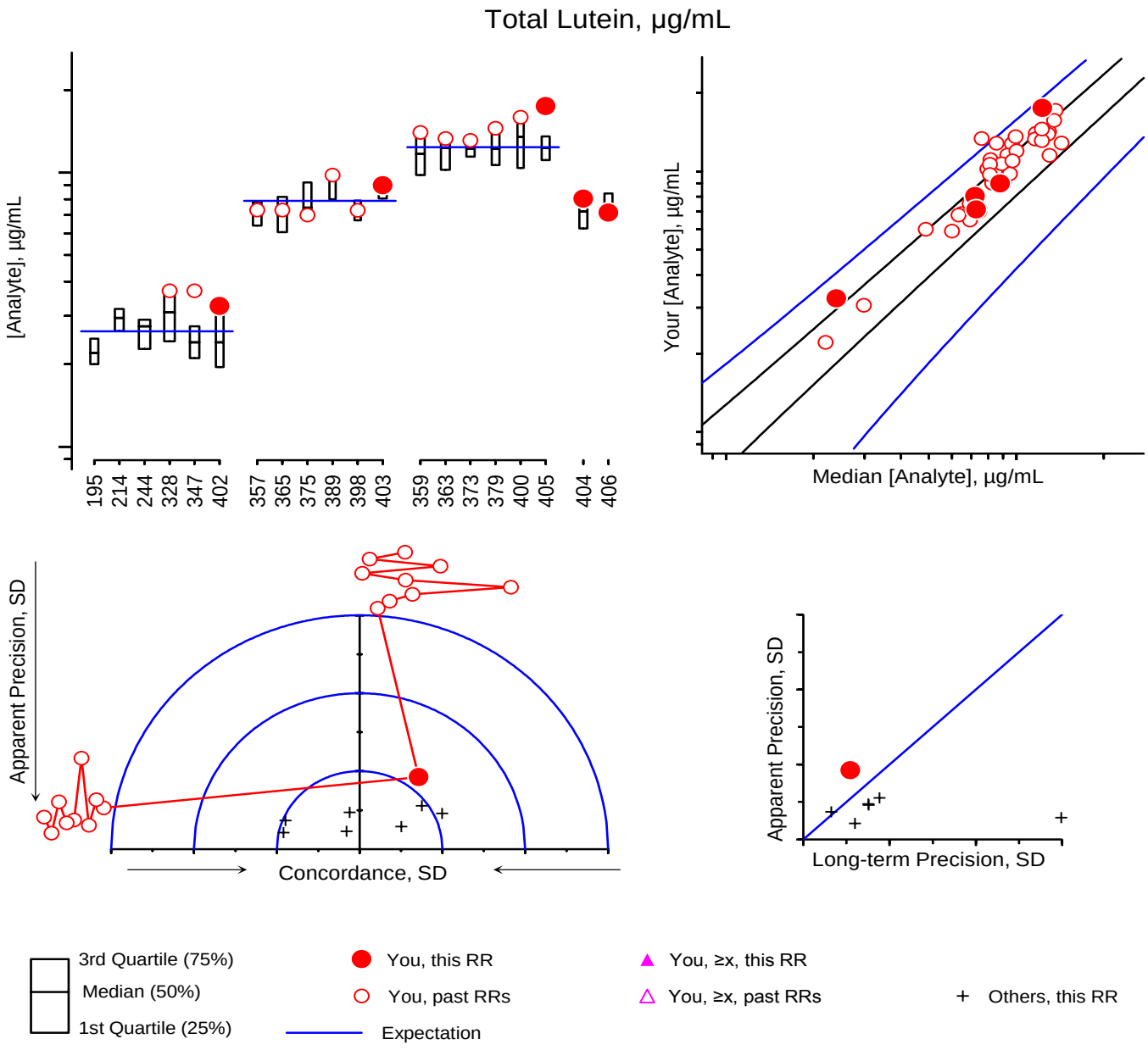
Individualized RR LXXV Report: FSV-BB



For details of the construction and interpretation of these plots, see:
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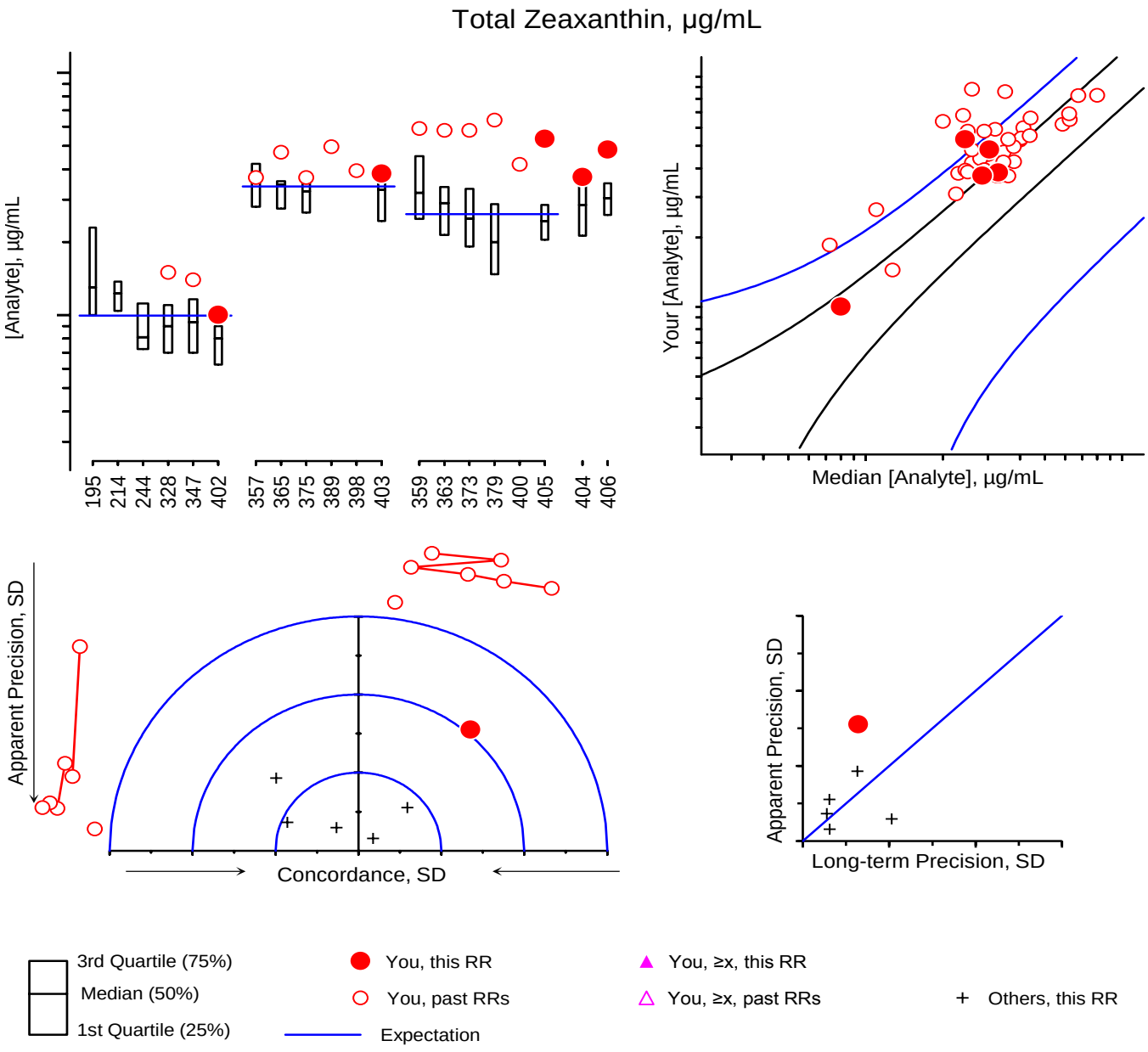
Individualized RR LXXV Report: FSV-BB



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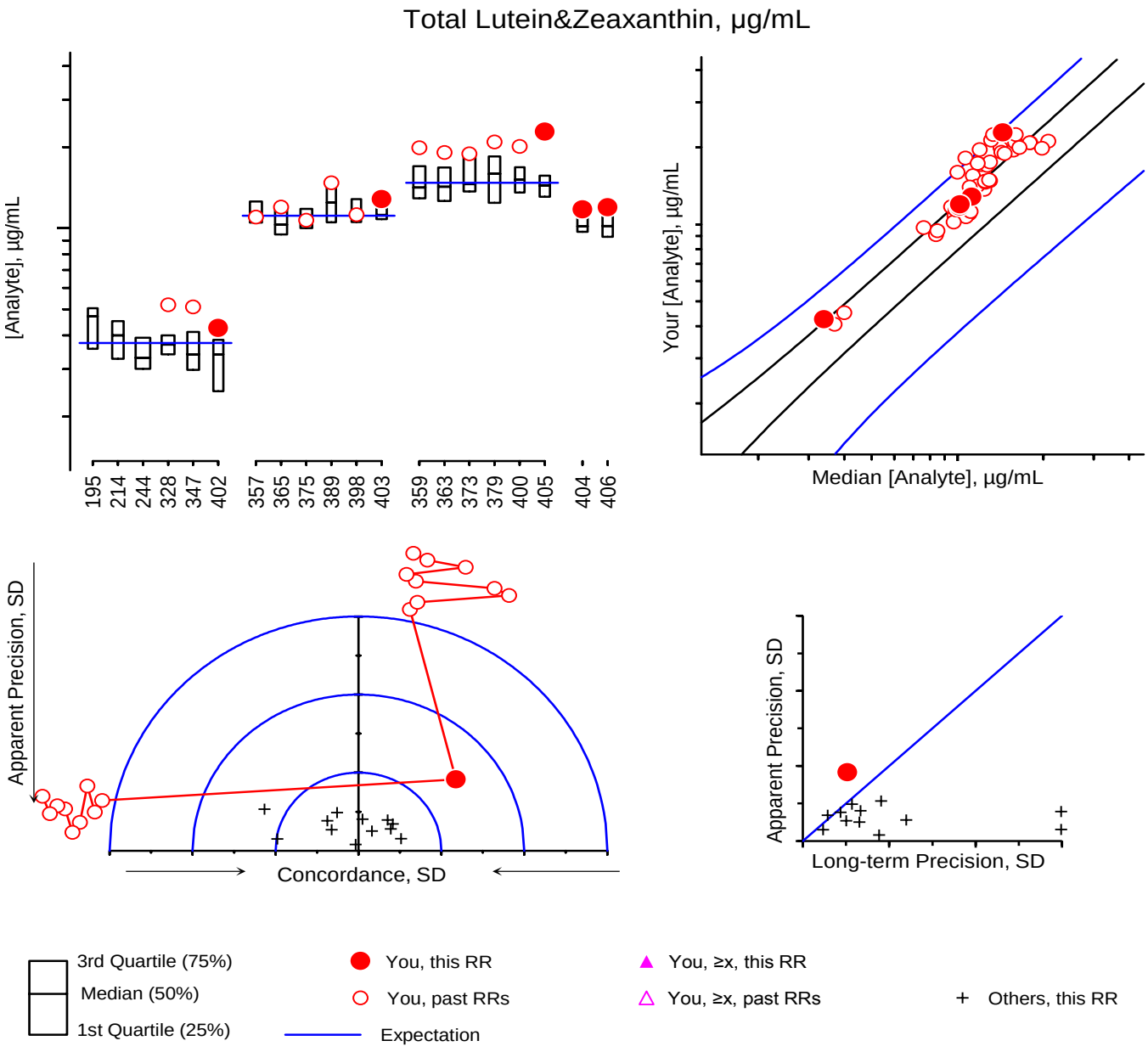
#404	Fresh-frozen, augmented {tR,RP,βT}, single-donor	Initial distribution
#405	Fresh-frozen, native, multi-donor: SRM 968e III	66#359, 67#363, 69#373, 70#379, 74#400
#406	Fresh-frozen, augmented {cR,γT}, single-donor	Initial distribution

Individualized RR LXXV Report: FSV-BB



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Serum	Comments	History
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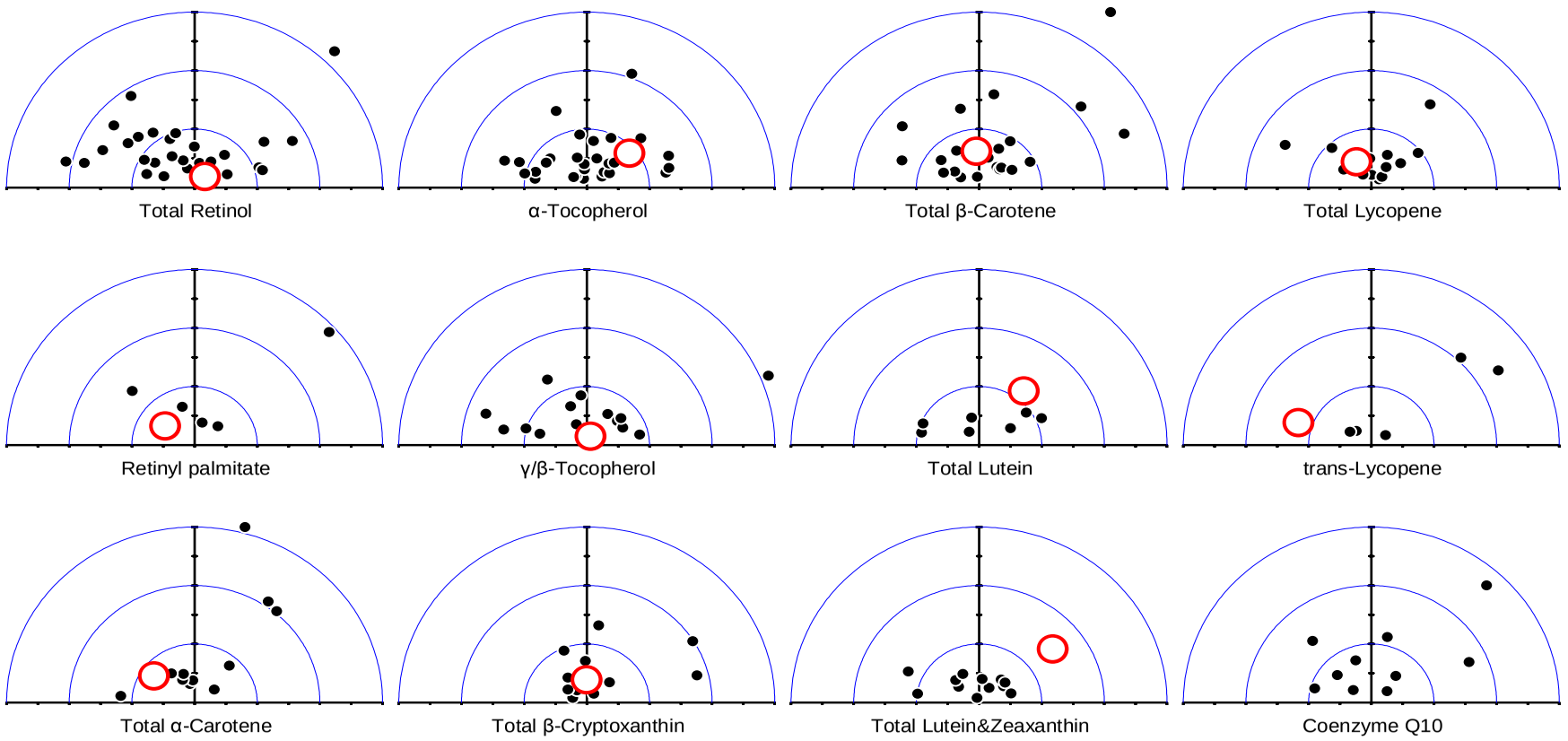
#404	Fresh-frozen, augmented {tR,RP,βT}, single-donor	Initial distribution
#405	Fresh-frozen, native, multi-donor: SRM 968e III	66#359, 67#363, 69#373, 70#379, 74#400
#406	Fresh-frozen, augmented {cR,γT}, single-donor	Initial distribution

Individualized Round Robin LXXV Report: FSV-BB
Graphical Comparability Summary

D15



Appendix E. Shipping Package Inserts for RR40

The following three items were included in each package shipped to an RR40 participant:

- Cover letter
- Analysis of Control Materials and Test Samples Datasheet
- Packing List and Shipment Receipt Confirmation Form

The RR41 samples were packed in a sealed waterproof bag and labeled:

NIST MMQAP-VC: **RR 40**
Micronutrients Measurement Vitamin C
Quality Assurance Program

Winter 2014 Controls & Samples

Results due on or before:
April 18, 2014

Cover letters describing the RR40 and RR41 studies, datasheets for both studies, and packing lists for both studies were enclosed in a sealed waterproof bag 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-

February 10, 2014

Dear Colleague:

The samples within this package constitute Vitamin C Round Robin 40 (RR40) of the 2014 Micronutrients Measurement Quality Assurance Program. RR40 consists of one vial each of four frozen serum *test samples* (#401, #402 #403, and #404) and one vial each of two frozen *control sera* (CS#3 and CS#4). These materials 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 total ascorbic acid is stable. While these samples contain some dehydroascorbic acid, its content is variable. Therefore, only total ascorbic acid should be analyzed and reported.

Please use the *control sera* to validate the performance of your measurement system before you analyze the *test samples*. The target value for CS#3 is (15.5 ± 1.6; 13.9 to 17.1) µmol/L and the target for CS#4 is (46.1 ± 4.6; 41.5 to 50.7) µmol/L. We expect your results for both of these controls to be within this ± 10% target range. If your results are significantly outside this range, your analysis system may not be suited to the analysis of MPA-preserved samples. In this case, please do **not** proceed to the analysis of the *test samples* but contact us at jbthomas@nist.gov or 301-975-3120.

The *test samples* and *control sera* should be defrosted by warming at 20 °C for not more than 10 min otherwise some irreversible degradation may occur. Please be aware that sample contact with any oxidant-contaminated surface (vials, glassware, etc.) may degrade your measurement system's performance (SA Margolis and E Park, "Stability of Ascorbic Acid in Solutions in Autosampler Vials", *Clinical Chemistry* 2001, *47*(8), 1463-1464). You should suspect such degradation if you observe unusually large variation in replicate analyses.

Please measure the total ascorbic acid in each ampoule in duplicate, reporting in units of µmol/(L sample solution) rather than µmol/(L serum used to prepare the sample). Please email (david.duewer@nist.gov) or fax (301-977-0685) your results to us as soon as possible but no later than **April 18, 2014**.

Please report your results by e-mail to david.duewer@nist.gov or fax to 301-977-0685. If you have questions or comments regarding the studies, please contact us at 301-975-3120 (Jeanice); jbthomas@nist.gov or 301-975-3935 (Dave); david.duewer@nist.gov.

Sincerely,

Jeanice B. Thomas, M.B.A.
Research Chemist
Chemical Sciences Division
Material Measurement Laboratory

David L. Duewer, Ph.D.
Research Chemometrician
Chemical Sciences Division
Material Measurement Laboratory

Enclosure: RR40 Report Form for Control and Test Sample Analyses

NIST

Participant #: _____ Date: _____

Vitamin C Round Robin 40

NIST Micronutrient Measurement Quality Assurance Program

Analysis of Control Materials and Test Samples

Sample	Replicate 1	Replicate 2	Units
Control serum CS#3			µmol/L of Sample *Target: (15.5 ±1.6) µmol/L*
Control serum CS#4			µmol/L of Sample *Target: (46.1 ±4.6) µmol/L*
Test sample #401			µmol/L of Sample
Test sample #402			µmol/L of Sample
Test sample #403			µmol/L of Sample
Test sample #404			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:

Nature of samples you typically analyze: native | MPA-preserved | DTT-preserved | Other
If "Other", please describe:

COMMENTS:

Please return by **April 18, 2014**

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 40

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 #401	Liquid frozen (1:1 serum:10% MPA)
VitC #402	Liquid frozen (1:1 serum:10% MPA)
VitC #403	Liquid frozen (1:1 serum:10% MPA)
VitC #404	Liquid frozen (1:1 serum:10% MPA)
CS#3	Liquid frozen (1:1 serum:10% MPA)
CS#4	Liquid frozen (1:1 serum:10% MPA)

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) Email (david.duewer@nist.gov) or fax (301-977-0685) us the following information:

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

Your prompt return of this information is appreciated.

The M^2QAP Gang

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

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

Appendix F. Final Report for RR40

The following six pages are the final report for RR40 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.

May 13, 2014

UNITED STATES DEPARTMENT OF COMMERCE
National Institute of Standards and Technology
Gaithersburg, Maryland 20899-8390

Dear Colleague:

Enclosed is the summary report of the results for Round Robin 40 (RR40) 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 robust median absolute deviation from the median is used as an estimate of the consensus interlaboratory standard deviation (eSD). We estimate the coefficient of interlaboratory variation (eCV) as 100×eSD/median.

RR40 consisted of four test samples (#401, #402 #403, and #404) and one vial each of two frozen control serum control samples (CS #3 and CS #4). 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 www.nist.gov/srm; 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.

We are pleased to announce that MMQAP participant Professor Garry Duthie of the Rowett Institute of Nutrition and Health, University of Aberdeen, Scotland is providing the (**free**) on-line recipe book Stovies Reloaded: Traditional Scottish Recipes Made Healthier at http://www.abdn.ac.uk/rowett/policy-industry/stovies.php. Prof. Duthie states that *"Stovies Reloaded" is our light-hearted take on traditional Scottish recipes and how they can be adapted in the light of increased nutritional knowledge. … We hope that this book may play a small part in encouraging people to rediscover cooking skills, take an interest in traditional Scottish fare and eat a more healthy diet.*" This website also provides links to a wealth of other information developed at the Rowett Institute relating to nutrition, food safety, and human health.

The results for vitamin C in serum RR41 and fat-soluble vitamins and carotenoids in serum RR41 are due on or before **September 15, 2014.** We will send you a reminder in August. 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, M.B.A.
Research Chemist
Chemical Sciences Division
Material Measurement Laboratory

David L. Duewer, Ph.D.
Research Chemometrician
Chemical Sciences Division
Material Measurement Laboratory

Enclosures

cc: L. C. Sander

The NIST MMQAP Vitamin C Round Robin 40 (RR40) report consists of:

Page	"Individualized" Report
1	Summary of your reported values for the two serum control and four serum test samples.
2	Graphical summary of measurements made on the RR40 test samples.
3	Your RR40 measurements as a function of their expected values.

Page	"All-Lab" Report
1	A tabulation of results and summary statistics for total ascorbic acid [TAA] in the RR40 control and test samples. Results and summary statistics are also presented for the test samples calibrated to the results for the control samples. The consensus [TAA] content and inter-participant standard deviations are estimated using robust estimators.

Serum-Based Samples. Two serum controls and four test samples were distributed in RR40.

CS#3 a (15.4 ±0.4) μmol/L material ampouled in 2009

CS#4 a (46.2 ±1.2) μmol/L material ampouled in 2009

S40:1 SRM 970 level 2, ampouled in mid-1998, previously distributed as a test sample in RRs 11 to 15, 18, 20 22, 25, 29, 36, 37, and 39

S40:2 For most participants, this was a blank material of nearly zero [TAA] content, ampouled in 2001, and previously distributed in RRs 16, 19, 21, 23, 26, 29, and 38. Unfortunately, due to a labelling error four participants received augmented materials. The results for these miss-labeled samples agree well with the historical values for materials that were stored in the same container as the blank (these values are displayed on page 3 of the affected participants' Individualized Reports.) However, since our primary purpose in distributing blank samples is to assess current detection/quantification limits, the results for all of the miss-labeled samples are recorded in the All-Lab Report as "ne" (short for NIST error.)

S40:3 Ampouled in late 2009, previously distributed in RRs 32, 33, 35, and 38

S40:4 Ampouled in late 2009, previously distributed in RRs 34, 36, and 38

Results.

1) The reported [TAA] contents of the two control sera, CS#3 and CS#4, are unchanged from the values estimated for these materials when they were distributed as test samples.

2) All participants who received the intended blank material, S40:2, reported either suitably small values or quantification limits. We interpret [TAA] = 0 as "not detected" (and *vice versa*).

3) There is no evidence for any significant change in the [TAA] level or interlaboratory variability for samples S40:1, S40:3, and S40:4.

4) The primary focus of these VitC QAP studies has been and remains improving interlaboratory comparability of [TAA] measurements. In addition to providing you with objective assessment of your results against the community's consensus and helping to certify SRM 970, we have used these studies to explore other ways to further improve comparability. Chief among these has been the use of various control materials to linearly recalibrate your measurement systems:

$$[\mathrm{TAA}]_{\mathrm{ControlReported}} = a + b \times [\mathrm{TAA}]_{\mathrm{ControlReference}}$$

where the $[TAA]_{ControlReported}$ are the reported values for two or more control materials, $[TAA]_{ControlReference}$ are established reference values for these materials, and a and b are the intercept and slope of the linear calibration function. The (re)calibrated values for test samples are then given by:

$$[TAA]_{TestCalibrated} = ([TAA]_{TestReported} - a) / b \, .$$

Beginning in 1996, we distributed a solid ascorbic acid control material and asked you to gravimetrically prepare solutions of known [TAA] in 5% MPA. In addition to enabling you to independently verify the performance of your measurement systems before measuring the test samples, the information you provided for these solutions enabled us to determine if the use of a common simple-matrix calibrant could improve comparability.

Figure 1 plots a robust estimate of interlaboratory standard deviation, eSD, as a function of the consensus median [TAA] value for the 45 samples distributed as test samples from RR26 (2007) through RR36 (2012). The red circles represent the {eSD, Median} values for the results as reported. The green triangles represent the {eSD, Median} values for those results after recalibration to the gravimetrically prepared control solutions. The solid lines represent "best fits" to the non-linear function:

$$\text{eSD} = \sqrt{\alpha^2 + (\beta \times \text{Median})^2}$$

where α is the expected constant variability and β is the expected proportional variability. This or similar functional relationships are routinely observed in interlaboratory studies whenever the analyte content in some samples approaches the quantification limits of a sizable minority of the measurement systems.

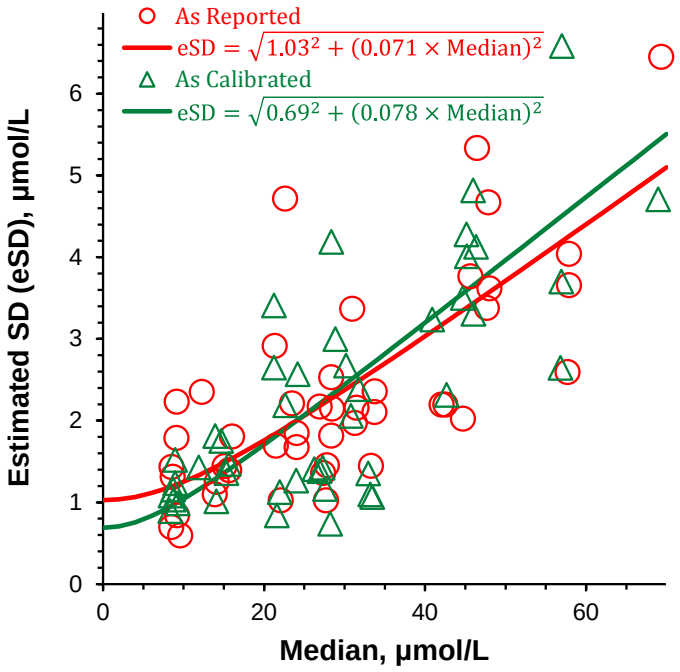


Figure 1: Estimated Standard Deviation as a Function of Median Value
Before and After Recalibration to Gravimetrically Prepared [TAA] in 5 % MPA Solutions

There is no evidence that recalibration to the 5 % MPA solutions improves comparability. (This is the primary reason why we stopped distributing the solid AA control with RR36.)

Beginning in RR20 (2004), we periodically distributed the two SRM 970 materials in the same RR, either explicitly as controls and/or as test samples. This allowed recalibrating results for the test samples with two models: 1) proportional, using regression to estimate a slope, *b*, when *a* is zero and 2) linear, solving for the *a* and *b* of the line between the two points. Figure 2 displays the summary statistics for the 38 available test samples using both models. The panel to the left displays proportionally recalibrated results as solid green triangles; the panel to the right displays the linearly recalibrated values as open green triangles.

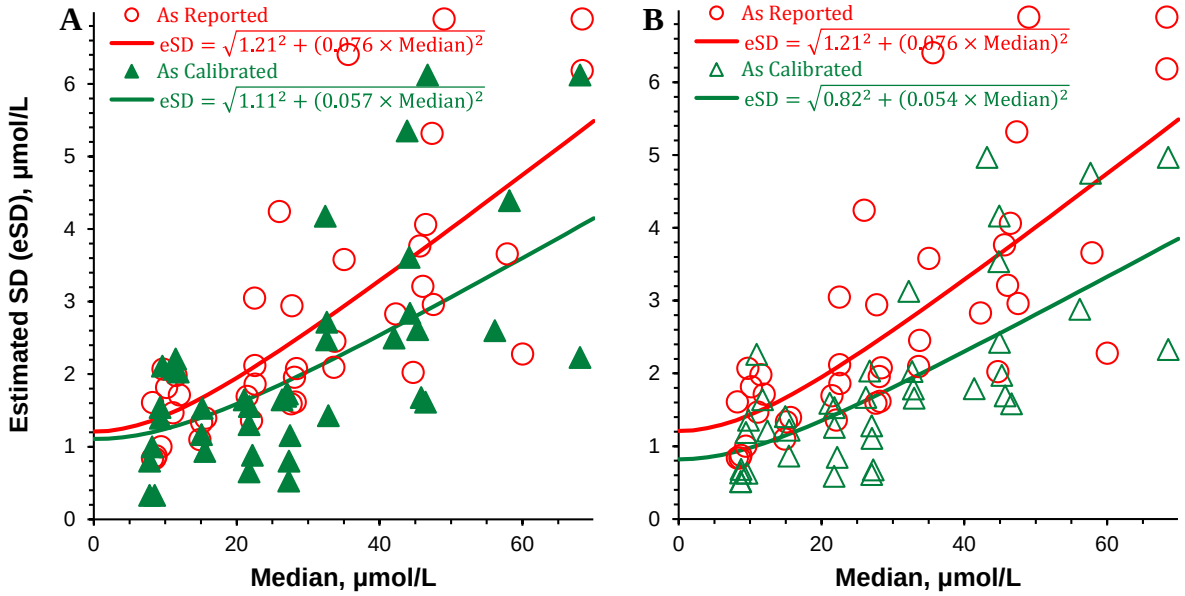


Figure 2: Estimated Standard Deviation as a Function of Median Value
before and after Recalibration with SRM 970 Levels I and II
Panel A: Proportional Model (*a* = 0). Panel B: Linear Model

Proportional recalibration to the SRM 970 materials marginally improves comparability for the very high [TAA] content samples, from a limiting relative standard deviation, %RSD = 100 × eSD / Median, of about 7.6 % to about 5.7 %. However, it does little for the low [TAA] content materials. Linear recalibration is somewhat superior, reducing the % RSD to about 5.4 % and reducing the content-independent standard deviation from about 1.2 µmol/L to about 0.8 µmol/L.

While perhaps academically interesting, the improved comparability with either model hardly seems worth the effort.

However, the 8.5 µmol/L long-term consensus [TAA] content of SRM 970 Level I is not much greater than the quantification limit reported by several participants and the 27.4 µmol/L of SRM 970 Level II is less than 50% of the maximum [TAA] content of the samples thus far distributed. To investigate whether increasing the [TAA] contents of the control samples used in the recalibration from the relatively low values of the SRM 970 materials, beginning with RR34 (2011) we periodically distributed (either explicitly as controls and/or as test samples) two new materials, now designated as CS#3 with [TAA] = 15.2 µmol/L and as CS#4 with [TAA] = 45.9 µmol/L. Figure 3 displays the summary statistics for the 20 samples that can be recalibrated with these materials, using both the proportional and linear models.

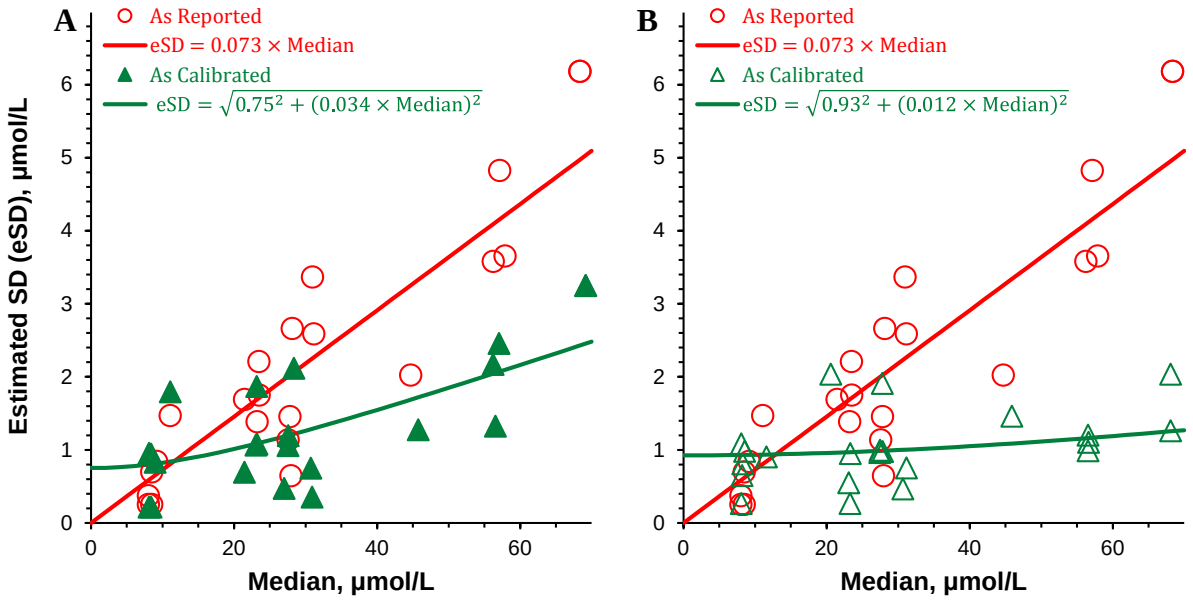


Figure 3: Estimated Standard Deviation as a Function of Median Value
before and after Recalibration with CS#3 and CS#4
Panel A: Proportional Model ($a = 0$). Panel B: Linear Model

The data are as yet too limited for drawing strong conclusions, but the linear recalibration model considerably improves comparability for the mid to high [TAA] content samples, essentially reducing the expected variability to an almost constant 1 µmol/L independent of [TAA] content.

Our current hypothesis is that interlaboratory comparability is limited by small differences in how measurement processes respond to components of the sample matrix other than TAA itself. At least for the samples distributed in the VitC QAP, these biases are greatly reduced by using matrix-matched calibrants having appropriately designed [TAA] content. Whether this would hold true for real samples is an intriguing, but yet unanswerable question.

Appendix G. "All-Lab Report" for RR40

The following two pages are the "All-Lab Report" for RR40 as provided to all participants, with the following exceptions:

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

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" 40 - Winter 2014

		Samples						Calibrated Results: [TAA]' = (TAA-a)/b					
		Total Ascorbic Acid ([TAA]), µmol/L						Parameters		[TAA]', µmol/L			
Lab	Date	CS#3	CS#4	S40:1	S40:2	S40:3	S40:4	*b*	*a*	S40:1'	S40:2'	S40:3'	S40:4'
VC-MB	24/02/14	13.9	47.7	26.8	ne	23.2	59.9	1.10	-2.8	26.9	-	23.6	56.9
VC-MC	18/04/14	15.5	45.0	27.6	ne	23.3	56.2	0.96	0.9	27.8	-	23.3	57.5
VC-MG	07/03/14	15.4	48.5	28.5	1.2	23.7	58.9	1.08	-1.0	27.3	2.0	22.9	55.6
VC-MH	03/03/14	14.5	43.2	23.8	ne	21.7	51.4	0.94	0.2	25.1	-	22.9	54.6
VC-MI	18/04/14	15.6	45.4	28.1	<1.4	24.6	56.4	0.97	0.8	28.1	<0.6	24.4	57.2
VC-MJ	27/03/14	22.0	52.8	33.0	2.3	31.9	69.3	1.01	6.7	26.2	-4.4	25.1	62.3
VC-MN	01/04/14	15.1	44.6	27.5	0.0	22.5	54.8	0.96	0.5	28.1	-0.5	22.9	56.5
VC-NF	10/04/14	16.0	44.7	28.4	0.0	23.5	54.6	0.94	1.7	28.4	-1.8	23.3	56.4
VC-NM	17/04/14	14.1	43.8	24.2	<5.7	20.3	52.1	0.97	-0.7	25.7	<6.6	21.6	54.5
VC-NX	15/04/14	14.5	47.1	27.6	ne	22.1	58.5	1.06	-1.7	27.5	-	22.4	56.6
	N	10	10	10	4	10	10			10	4	10	10
	Average	15.6	46.3	27.5	0.9	23.7	57.2			27.1	-1.2	23.2	56.8
	SD	2.3	2.9	2.5	1.1	3.1	5.1			1.1	2.7	1.0	2.2
	Min	13.9	43.2	23.8	0.0	20.3	51.4			25.1	-4.4	21.6	54.5
	%25	14.5	44.6	27.0		22.2	54.6			26.4		22.9	55.8
	Median	15.3	45.2	27.6	0.6	23.2	56.3			27.4	-1.1	23.1	56.5
	%75	15.6	47.6	28.3		23.7	58.8			28.0		23.5	57.2
	Max	22.0	52.8	33.0	2.3	31.9	69.3			28.4	2.0	25.1	62.3
	eSD	1.1	2.5	1.1		1.4	3.6			1.0		0.6	1.2
	eCV	7	5	4		6	6			4		2	2

G2

Micronutrients Measurement Quality Assurance Program for Total Ascorbic Acid
"Round Robin" 40 - Winter 2014

Legend

Term	Definition
Lab	Participant code
Date	Date results received at NIST
b	Slope and Intercept of the line connecting the expected values
a	for control samples CS#3 and CS#4 with the results reported by each participant
N	Number of quantitative values reported for this analyte
Average	Mean of the reported quantitative values
SD	Standard deviation of the reported quantitative values
Min	Minimum quantitative value reported
%25	First quartile of the reported quantitative values
Median	Median (second quartile) of the reported quantitative values
%75	Third quartile of the reported quantitative values
Max	Maximum quantitative value reported
eSD	Robust standard deviation, estimated using the adjusted median absolute deviation from the median (MADe)
eCV	Robust Coefficient of Variation, estimated as 100*eSD/Median
<x	Concentration less than x
ne	NIST error (participant was shipped the wrong sample)

G3

Appendix H. Representative "Individualized Report" for RR40

Each participant in RR40 received an "Individualized Report" reflecting their reported results. The following three pages are the "Individualized Report" for participant "VC-MB".

Vitamin C "Round Robin" 40 Report: Participant VC-MB

[TAA] mmol/Lsample

Date	RR	Sample	Rep_1	Rep_2	F_{adj}	Mean	SD_{dup}	N	Mean	SD_{repeat}	SD_{reprod}
07/23/10	33	S33:3	13.4	13.4	1.0	13.4	0.0	7	14.1	1.3	1.1
01/14/11	34	S34:2	13.9	13.9	1.0	13.9	0.0				
06/13/11	35	S35:1	15.0	15.5	1.0	15.2	0.4				
08/06/12	37	CS#3	10.3	15.0	1.0	12.6	3.3				
02/27/13	38	CS#3	13.9	13.9	1.0	13.9	0.0				
08/01/13	39	CS#3	15.5	15.5	1.0	15.5	0.0				
02/24/14	40	CS#3	**13.9**	**13.9**	1.0	13.9	0.0				
01/13/10	32	S32:4	45.4	45.9	1.0	45.7	0.4	7	46.6	0.9	1.1
01/14/11	34	S34:3	45.4	45.9	1.0	45.7	0.4				
06/13/11	35	S35:4	47.5	47.5	1.0	47.5	0.0				
08/06/12	37	CS#4	44.9	48.0	1.0	46.5	2.2				
02/27/13	38	CS#4	45.9	44.9	1.0	45.4	0.7				
08/01/13	39	CS#4	48.0	47.5	1.0	47.7	0.4				
02/24/14	40	CS#4	**47.5**	**48.0**	1.0	47.7	0.4				
09/25/98	11	S11:2	63.0	62.0	0.5	31.3	0.4	14	28.4	1.3	2.2
02/26/99	12	S12:2	55.0	55.0	0.5	27.5	0.0				
03/03/00	13	S13:2	63.0	64.0	0.5	31.8	0.4				
03/26/01	14	S14:4	60.9	59.9	0.5	30.2	0.4				
09/05/01	15	S15:2	63.0	64.0	0.5	31.8	0.4				
12/19/02	18	S18:3	55.8	54.7	0.5	27.6	0.4				
03/01/04	20	S20:4	21.7	27.9	1.0	24.8	4.4				
03/02/05	22	S22:4	28.4	28.4	1.0	28.4	0.0				
05/24/06	25	S25:2	29.4	29.4	1.0	29.4	0.0				
06/20/08	29	S29:3	26.8	26.8	1.0	26.8	0.0				
03/07/12	36	S36:3	25.8	26.8	1.0	26.3	0.7				
08/06/12	37	S37:3	25.8	27.9	1.0	26.8	1.5				
08/01/13	39	S39:2	28.4	28.4	1.0	28.4	0.0				
02/24/14	40	S40:1	**26.8**	**26.8**	1.0	26.8	0.0				
02/08/02	16	S16:2	3.1	2.0	0.5	1.3	0.4	5	0.9	0.4	1.4
05/01/03	19	S19:1	5.2	7.2	0.5	3.1	0.7				
07/14/04	21	S21:1	<5.2	<5.2	1.0						
05/25/05	23	S23:1	0.0	0.0	1.0	0.0	0.0				
11/26/06	26	S26:1	0.0	0.0	1.0	0.0	0.0				
06/20/08	29	S29:1	0.0	0.0	1.0	0.0	0.0				
02/27/13	38	S38:1	<5.2	<5.2	1.0						
02/24/14	40	S40:2	**ne**	**ne**	1.0			ne = NIST error			
01/13/10	32	S32:2	22.2	22.7	1.0	22.5	0.4	5	22.8	0.5	0.8
07/23/10	33	S33:4	23.2	22.7	1.0	23.0	0.4				
06/13/11	35	S35:2	23.7	23.7	1.0	23.7	0.0				
02/27/13	38	S38:2	21.2	22.2	1.0	21.7	0.7				
02/24/14	40	S40:3	**23.7**	**22.7**	1.0	23.2	0.7				
01/14/11	34	S34:4	56.8	57.3	1.0	57.0	0.4	5	57.5	0.6	1.5
03/07/12	36	S36:1	57.8	56.8	1.0	57.3	0.7				
03/07/12	36	S36:4	56.8	55.2	1.0	56.0	1.1				
02/27/13	38	S38:4	57.3	56.8	1.0	57.0	0.4				
02/24/14	40	S40:4	**59.9**	**59.9**	1.0	59.9	0.0				

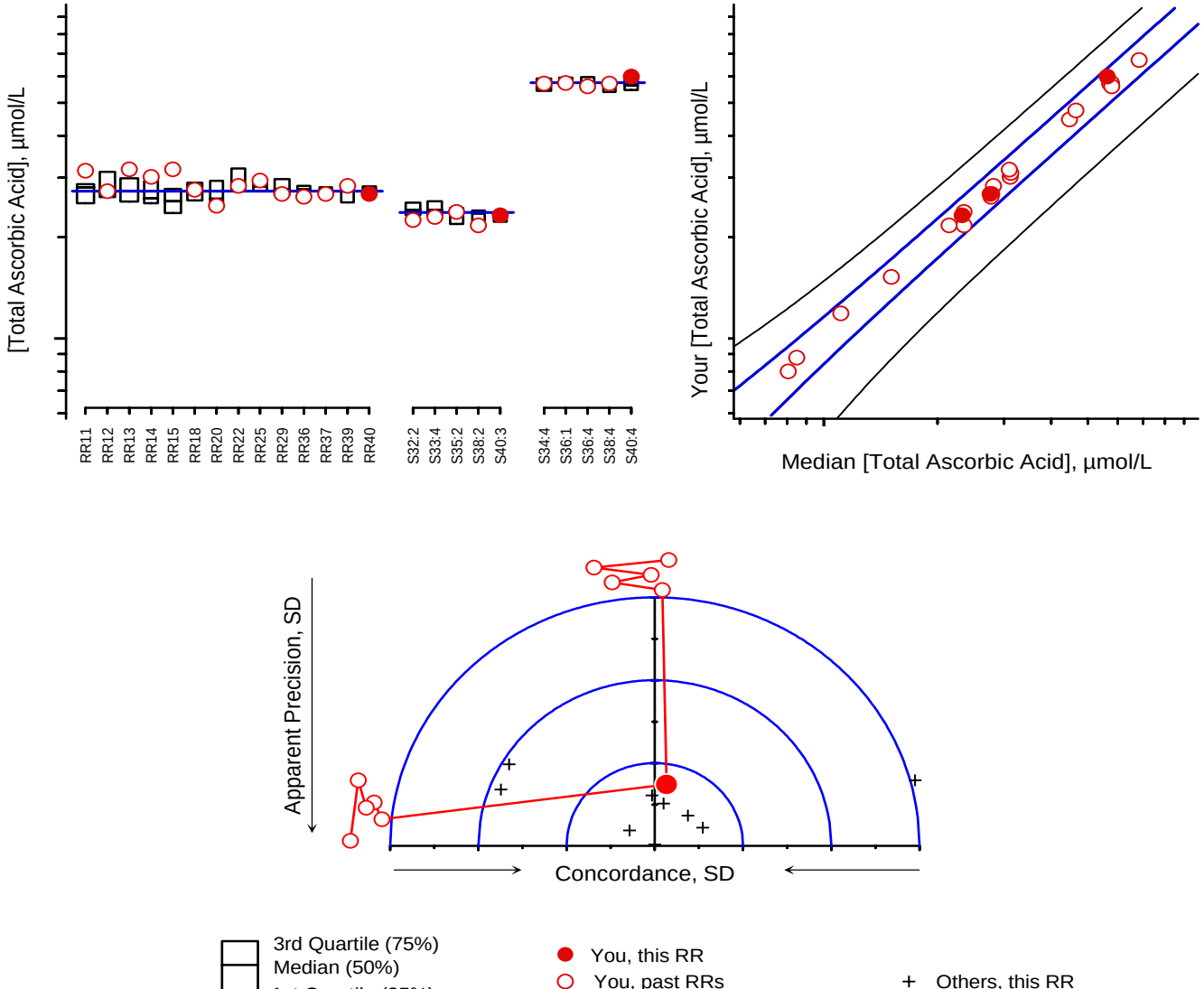
Please check our records against your records. Send corrections and/or updates to...

Micronutrients Measurement Quality Assurance Program
National Institute of Standards and Technology
Fax: (301) 977-0685
Email: david.duewer@nist.gov

H2

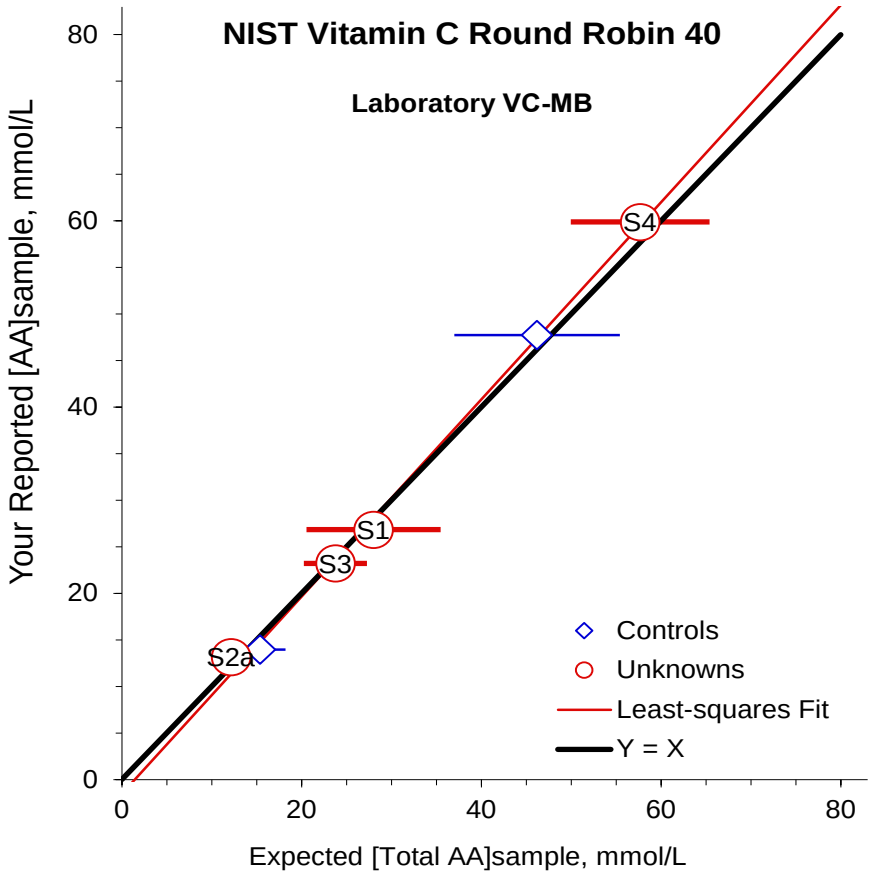
Vitamin C "Round Robin" 40 Report: Participant VC-MB

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
CS#3	Distributed as an unknown in RRs 33,34,35; as a control in RRs 37,38,39
CS#4	Distributed as an unknown in RRs 32,34,35; as a control in RRs 37,38,39
S39:1	SRM970 Lv II; distributed as an unknown in RRs 11,12,13,14,15,18,20,22,25,29,36,37,39
S39:2	Zero-level control serum, distributed in RRs 16,19,21,23,26,29,38
S39:3	Distributed in RRs 32,33,35,38
S39:4	Distributed in RRs 34,36(dups),38



Params	Values	Units
Intercept	-1.5 ± 1.1	µmol/L
Slope	1.059 ± 0.031	(Ratio)
N	6	
R^2	0.9966	
RMSE	1.2	µmol/L

Data error-bars are 2×SD

The "Expected" values are the most recent historical data available for these materials.