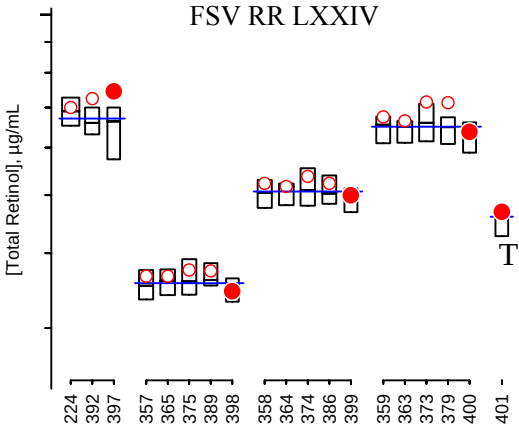
NISTIR 7880-42

NIST Micronutrients Measurement Quality Assurance Program Summer 2013 Comparability Studies

Results for Round Robin LXXIV Fat-Soluble Vitamins and Carotenoids in Human Serum and Round Robin 39 Ascorbic Acid in Human Serum



David L. Duewer
Jeanice B. Thomas

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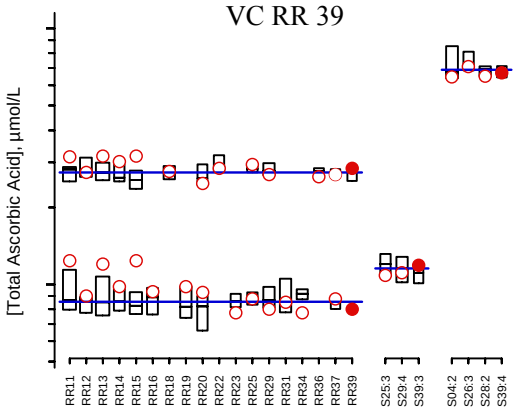

National Institute of Standards and Technology
U.S. Department of Commerce

NISTIR 7880-42

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Results for Round Robin LXXIV Fat-Soluble Vitamins and Carotenoids in Human Serum and Round Robin 39 Ascorbic Acid in Human Serum

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Materials Measurement Laboratory

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November 2014

U.S. Department of Commerce
Penny Pritzker, Secretary

National Institute of Standards and Technology
Willie E. May, Acting Under Secretary of Commerce for Standards and Technology and Acting 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 2013 MMQAP measurement comparability improvement studies: 1) Round Robin LXXIV Fat-Soluble Vitamins and Carotenoids in Human Serum and 2) Round Robin 39 Total Ascorbic Acid in Human Serum. The materials for both studies were shipped to participants in June 2013; participants were requested to provide their measurement results by September 30, 2013.

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 LXXIV: Fat-Soluble Vitamins and Carotenoids in Human Serum

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin LXXIV comparability study (hereafter referred to as RR74) 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 June 2013. 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 RR74 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 39: Vitamin C in Human Serum

Participants in the MMQAP Vitamin C in Human Serum Round Robin 39 comparability study (hereafter referred to as RR39) 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 sample materials were shipped on dry ice to participants in June 2013. 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 RR39 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 RR74

The following three items were included in each package shipped to an RR74 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, 2013

Dear Colleague:

Enclosed are samples for the second fat-soluble vitamins and carotenoids in serum study (Round Robin LXXIV) for the 2013 NIST Micronutrients Measurement Quality Assurance Program. The set of samples (Sera 397- 401) 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 by **September 30, 2013**. 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 2013.

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 LXXIV 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
Program Coordinator/Research Chemist
Analytical Chemistry Division
Material Measurement Laboratory

Enclosure

Participant #: _____ Date: _____

Round Robin LXXIV: Human Sera

NIST Micronutrients Measurement Quality Assurance Program

Analyte	397	398	399	400	401	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						
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
30-Sep-2013

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

Participant #: _____ Date: _____

Fat-Soluble Vitamins Round Robin LXXIV

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
#397	Lyophilized	Yes	5 mL clear / silver
#398	Liquid frozen	No	TBD
#399	Liquid frozen	No	TBD
#400	Liquid frozen	No	TBD
#401	Liquid frozen	No	TBD

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 RR74

The following six pages are the final report for RR74 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 20899-8390

December 3, 2013

Dear Colleague:

Enclosed is the summary report of the results for "Round Robin" LXXIV (RR74) of the 2013 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. RR74 (Sera 397 - 401) 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.

Samples for the 2014 MMQAP will be shipped **starting January 27, 2014**. We will ship the samples for both exercises for each study at the same time. Results are due in April for the first study; written feedback will be provided to laboratories in May. We will send you a notification around June about the reporting deadline for the second study. Please contact us immediately if this schedule is problematic for your laboratory.

We have completed on-line documentation of data and reports for the MMQAP studies from 1996 to 2012. For your convenience, enclosed are the links to these documents. Data summaries have been altered to ensure confidentiality of identification codes assigned to laboratories. We anticipate that the results for Round Robins 73 and 74 Fat-Soluble Vitamins and Carotenoids in Human Serum and Round Robins 38 and 39 Ascorbic Acid in Human Serum will be av**a**ilable online by spring 2014.

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

MMQAP Comparability Studies (1996 to 2012)

D.L. Duewer, M.C. Kline, K.E. Sharpless, J.M. Brown Thomas, S.A. Margolis, NIST Micronutrients Measurement Quality Assurance Program Winter, Spring, and Fall 1996 Comparability Studies: Results for Round Robin XXXVI, XXXVII, and XXXVIII Fat-Soluble Vitamins and Carotenoids in Human Serum and Round Robin 9 Ascorbic Acid in Human Serum, NIST Interagency/Internal Report (NISTIR) 7880-28

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D.L. Duewer, J.M. Brown Thomas, NIST Micronutrients Measurement Quality Assurance Program Winter 2004 Comparability Studies: Results for Round Robin LV Fat-Soluble Vitamins and Carotenoids in Human Serum and Round Robin 20 Ascorbic Acid in Human Serum, NIST Interagency/Internal Report (NISTIR) 7880-17

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D.L. Duewer, J.M. Brown Thomas, NIST Micronutrients Measurement Quality Assurance Program Summer 2012 Comparability Studies, NIST Interagency/Internal Report (NISTIR) 7880-1

The NIST MMQAP Round Robin LXXIV (RR74) 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 RR74.

Serum	Description	Prior Distributions
397	Lyophilized, multi-donor stripped serum augmented with retinol, retinyl palmitate, α- and γ-tocopherol, α- and β-carotene, lycopene, β-cryptoxanthin, lutein, and zeaxanthin prepared in 1995.	#224:RR38-9/96, #392:RR73-3/13
398	Fresh-frozen, native, multi-donor, prepared in 2009. This is Level I of SRM 968e.	#357:RR66-9/09, #365:RR67-3/10, #375:RR69-3/11, #389:RR72-9/12
399	Fresh-frozen, native, multi-donor, prepared in 2009. This is Level II of SRM 968e.	#358:RR66-9/09, #364:RR67-3/10, #374:RR69-3/11, #386:RR71-3/12
400	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
401	Liquid-frozen, multi-donor heparin-treated plasma material augmented with glycyrrhetinic acid prepared in 1989.	First MMQAP distribution

Results

1) Serum 397. This highly augmented experimental material was distributed in RR74 to confirm that the levels of retinyl palmitate, β-carotene, and total and trans-lycopene are less than they were in 1995 but that the levels of the other analytes are largely unchanged.

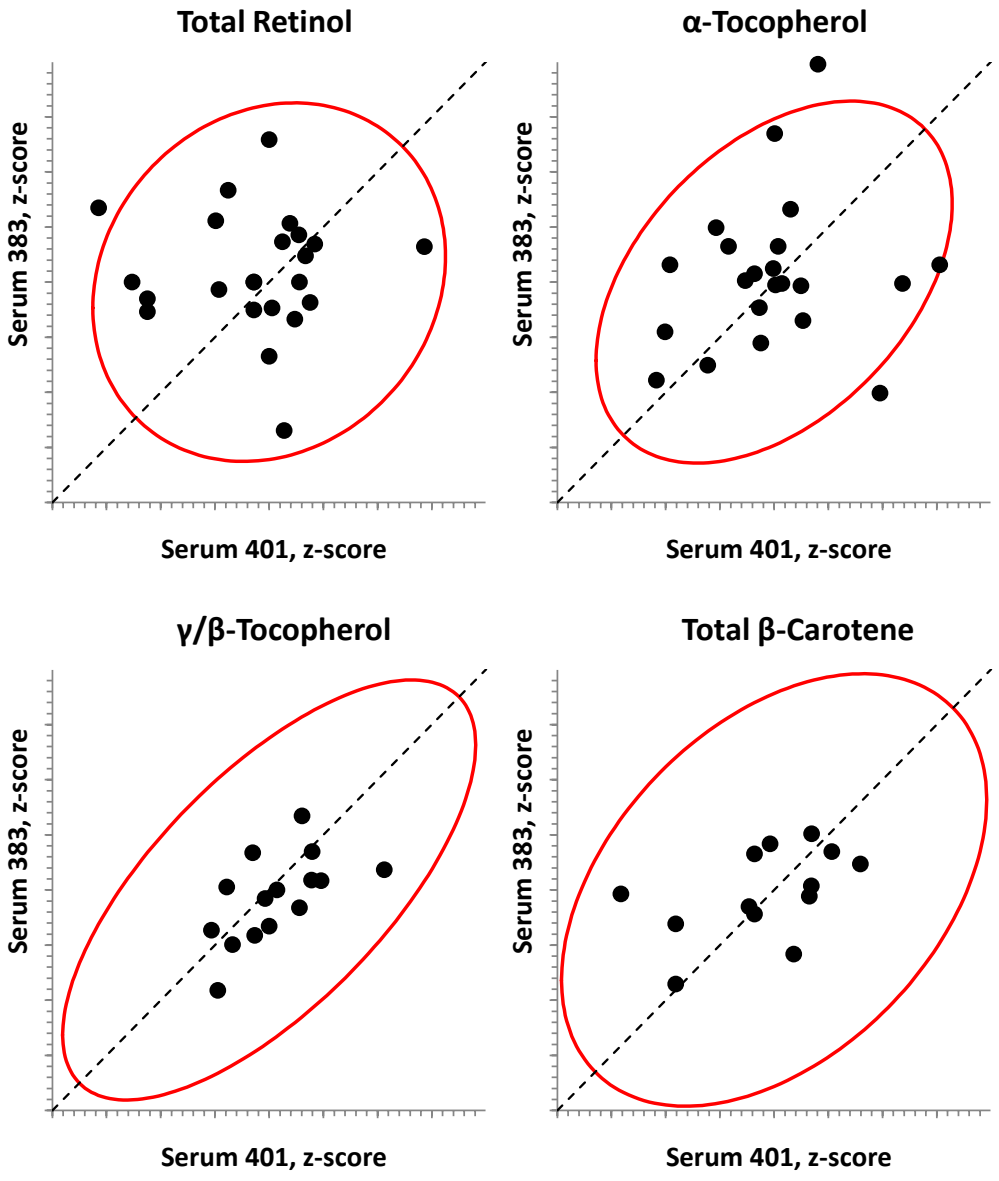
2) Sera 388 to 400. There have been no significant changes in either the level nor variability in any of the analytes in these SRM 968e materials.

3) Serum 401. This is the second distribution of a material prepared as part of an investigation into the chemopreventative potential of glycyrrhetinic acid. This relatively polar compound's absorbance maxima are at 248 nm and should not interfere with the analysis of the usual MMQAP analytes. The following "Youden Plots" provide a way to check for systematic biases in the analysis of these samples, plotting normalized values for serum 393 (Y-axis), a material with a different glycyrrhetinic

acid concentration distributed in RR73, against those for serum 401 (X-axis) for each participant that reported both values. Each solid black circle represents the results reported by one participant.

The z-score normalization was accomplished using the median as a robust estimator of location and the "MADe" (median absolute deviation from the median, adjusted to have the same coverage as a standard deviation) as a robust estimate of dispersion: z-score = (value – Median)/MADe. The solid red ellipse is a 95 % joint probability ellipse for the paired data given the number of pairs and a robust estimate of the "typical" between sera correlation for these two materials. Assuming that the results are normally distributed, about 95% of "typical" results should lie within the ellipse.

The dashed line marks where the paired results are expected to cluster when there is significant systematic bias in a participant's method. Since none of the paired results that lie outside the 95 % ellipse are close to this line, there is little evidence for such a bias in the analysis of total retinol, α- and γ/β-tocopherol, and total β-carotene. There are too few paired results to test for systematic bias in any of the other analytes.



Appendix C. "All-Lab Report" for RR74

The following six pages are the "All-Lab Report" for RR74 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 LXXIV Laboratory Results

C2

	Total Retinol, µg/mL					Retinyl Palmitate, µg/mL					α-Tocopherol, µg/mL					γ/β-Tocopherol, µg/mL					δ-Tocopherol, µg/mL				
Lab	397	398	399	400	401	397	398	399	400	401	397	398	399	400	401	397	398	399	400	401	397	398	399	400	401
FSV-BA	0.744	0.345	0.499	0.637	0.469	0.082	0.024	0.075	0.156	0.039	9.65	7.03	10.91	19.9	5.38	4.96	2.06	1.65	2.71	1.53	0.85	0.089	0.078	0.241	0.094
FSV-BB	0.711	0.338	0.461	0.625	0.474	0.072	0.009	0.022	0.075	0.011	9.12	6.83	10.55	18.8	5.14	4.29	1.84	1.41	2.33	1.31	0.78	0.070	0.050	0.201	0.066
FSV-BC	0.661	0.349	0.499	0.637	0.433																				
FSV-BD	0.660	0.357	0.510	0.626	0.462						9.50	8.00	10.60	18.2	6.40										
FSV-BE	0.677	0.348	0.513	0.642	0.477						7.92	6.20	10.34	19.6	4.53	3.33	1.51	1.22	1.95	1.09					
FSV-BF	0.480	0.320	0.460	0.530	0.370						8.50	6.90	10.50	20.2	5.00										
FSV-BG	0.709	0.363	0.523	0.654	0.509	0.047	0.010	0.032	0.105	0.011	8.20	6.49	10.30	18.3	5.10	4.02	1.93	1.46	2.27	1.48					
FSV-BJ	0.736	0.339	0.492	0.645	0.490						7.72	6.30	10.38	21.0	5.36	4.10	1.83	1.40	2.43	1.42					
FSV-BK	0.636	0.313	0.443	0.582	-						7.86	6.23	9.70	18.0	-										
FSV-BL	0.690	0.370	0.520	0.690	0.460						9.00	6.90	11.20	18.9	5.60										
FSV-BM	0.500	0.330	0.480	0.620	0.450						9.00	7.30	11.10	21.0	5.50										
FSV-BN	0.567	0.301	0.434	0.541	0.427						7.57	6.21	9.74	17.6	4.83										
FSV-BO	0.590	0.324	0.464	0.547	0.450						8.49	6.89	11.11	20.5	5.43	3.33	1.59	1.29	2.13	1.19					
FSV-BR	0.610	0.350	0.510	0.650	0.450						10.40	7.00	10.30	18.0	6.70										
FSV-BT	0.703	0.340	0.495	0.608	0.460						8.86	6.88	9.86	18.4	5.21	3.46	1.65	1.52	2.50	1.48	0.26	0.093	0.111	0.150	0.102
FSV-BU	0.533	0.353	0.504	0.636	0.348						9.82	6.68	10.37	19.0	5.72	4.60	1.74	1.35	2.18	1.43					
FSV-BV	0.568	0.344	0.453	0.548	0.371						8.42	6.32	8.95	14.9	4.49	4.44	1.76	1.30	1.96	1.20					
FSV-BW	0.770	0.370	0.520	0.690	0.480	0.085	0.017	0.023	0.085	0.018	7.24	5.95	9.46	17.0	4.42	3.98	1.71	1.24	2.09	1.25					
FSV-CE	0.560	0.300	0.440	0.540	0.380						10.15	7.98	11.02	21.3	6.22										
FSV-CG	0.640	0.363	0.508	0.582	0.425						5.20	5.24	7.56	11.0	3.61	2.70	1.66	1.29	1.51	1.06	0.34	*nq*	*nq*	*nq*	*nq*
FSV-CI	0.547	0.358	0.513	0.664	0.380	0.104	0.011	0.028	0.109	0.022	8.84	6.70	9.89	18.3	5.58	3.08	1.30	1.00	1.64	1.02					
FSV-CO											7.86	6.70	9.86	19.2	5.26	4.45	2.06	1.58	2.59	1.48					
FSV-CZ	0.666	0.383	0.570	0.706	0.487						8.90	7.11	10.87	20.0	5.37	3.45	1.58	1.22	1.88	0.99					
FSV-DD	0.740	0.370	0.530	0.680	0.470																				
FSV-DV	0.674	0.386	0.539	0.743	0.562						7.70	5.80	9.40	18.5	5.40										
FSV-EE																									
FSV-FK	0.691	0.372	0.501	0.651	0.484						8.20	6.40	10.10	18.0	4.90										
FSV-FZ	0.679	0.328	0.490	0.663	0.467	0.051	0.012	0.041	0.087	0.024	8.67	6.58	10.58	19.7	5.25	4.00	1.68	1.44	2.45	1.27					
N	26	26	26	26	25	6	6	6	6	6	25	25	25	25	24	15	15	15	15	15	4	3	3	3	3
Min	0.480	0.300	0.434	0.530	0.348	0.047	0.009	0.022	0.075	0.011	5.20	5.24	7.56	11.0	3.61	2.70	1.30	1.00	1.51	0.99	0.26	0.070	0.050	0.150	0.066
Median	0.664	0.348	0.500	0.637	0.460	0.077	0.012	0.030	0.096	0.020	8.50	6.70	10.34	18.8	5.31	4.00	1.71	1.35	2.18	1.27	0.56	0.089	0.078	0.201	0.094
Max	0.770	0.386	0.570	0.743	0.562	0.104	0.024	0.075	0.156	0.039	10.40	8.00	11.20	21.3	6.70	4.96	2.06	1.65	2.71	1.53	0.85	0.093	0.111	0.241	0.102
eSD	0.075	0.024	0.029	0.042	0.036	0.025	0.003	0.011	0.018	0.009	0.92	0.49	0.72	1.2	0.42	0.81	0.18	0.16	0.37	0.27	0.38				
eCV	11	7	6	7	8	33	26	37	19	46	11	7	7	7	8	20	10	12	17	21	67				
Npast	39	31	32	32	0	12	7	8	10	0	38	31	32	32	0	21	18	18	19	0	5	6	5	5	0
Medianpast	0.675	0.358	0.508	0.653		0.158	0.011	0.026	0.094		8.37	6.81	10.30	18.7		3.91	1.81	1.41	2.22		0.70	0.110	0.073	0.210	
SDpast	0.056	0.029	0.044	0.055		0.092	0.005	0.009	0.027		0.58	0.56	0.78	1.5		0.42	0.16	0.12	0.20		0.10	0.025	0.024	0.030	
NAV	0.664	0.348	0.500	0.637	0.460	0.077	0.012	0.030	0.096	0.020	8.50	6.70	10.34	18.8	5.31	4.00	1.71	1.35	2.18	1.27	0.56	0.089	0.078	0.201	0.094
NAU	0.075	0.029	0.040	0.051	0.037	0.025	0.011	0.013	0.025	0.011	0.92	0.57	0.80	1.5	0.49	0.81	0.18	0.16	0.37	0.27					

Round Robin LXXIV Laboratory Results

	Total β-Carotene, µg/mL					trans-β-Carotene, µg/mL					Total cis-β-Carotene, µg/mL					Total α-Carotene, µg/mL				
Lab	397	398	399	400	401	397	398	399	400	401	397	398	399	400	401	397	398	399	400	401
FSV-BA	0.303	0.099	0.245	0.425	0.060	0.287	0.089	0.234	0.403	0.051	0.016	0.010	0.011	0.022	0.009	0.38	0.008	0.028	0.017	0.012
FSV-BB	0.328	0.091	0.227	0.361	0.049	0.317	0.086	0.219	0.347	0.046	0.011	0.005	0.008	0.014	0.004	0.40	0.005	0.028	0.012	0.010
FSV-BC																				
FSV-BD																				
FSV-BE	0.253	0.081	0.257	0.427	0.042															
FSV-BF	0.219	0.078	0.262	0.379	0.053															
FSV-BG	0.398	0.100	0.259	0.450	0.054											0.37	0.009	0.033	0.019	0.011
FSV-BJ	0.348	0.062	0.261	0.504	0.067											0.49	*nq*	*nq*	*nq*	*nq*
FSV-BK																				
FSV-BL																				
FSV-BM																				
FSV-BN	0.348	0.076	0.234	0.393	0.040											0.53	0.011	0.054	0.043	0.011
FSV-BO	0.475	0.090	0.215	0.314	0.050											0.41	0.005	0.027	0.010	0.010
FSV-BR																				
FSV-BT	0.311	0.099	0.246	0.396	0.057	0.298	0.093	0.231	0.376	0.053	0.013	0.006	0.015	0.020	0.004	0.34	0.008	0.031	0.017	0.014
FSV-BU	0.365	0.087	0.240	0.395	0.064											0.43	0.008	0.033	0.025	0.013
FSV-BV	0.368	0.093	0.235	0.384	0.050											0.53	0.009	0.031	0.012	0.012
FSV-BW	0.214	0.089	0.243	0.464	0.040											0.64	0.009	0.023	0.010	0.006
FSV-CE	0.230	0.120	0.250	0.460	0.080															
FSV-CG	0.265	0.062	0.162	0.231	0.033	0.263	0.062	0.160	0.231	0.033	*0.002*	*nd*	*0.002*	*nd*	*nd*	0.46	*nq*	*nq*	*nq*	*nq*
FSV-CI	0.308	0.079	0.231	0.412	0.055											0.48	0.011	0.053	0.045	0.021
FSV-CO	0.421	0.094	0.251	0.404	0.057											0.46	0.008	0.027	0.010	0.010
FSV-CZ	0.217	0.060	0.120	0.175	0.053															
FSV-DD																				
FSV-DV																				
FSV-EE																				
FSV-FK	0.228	0.097	0.196	0.334	0.046															
FSV-FZ	0.367	0.098	0.247	0.412	0.052															
N	19	19	19	19	19	4	4	4	4	4	4	3	4	3	3	13	11	11	11	11
Min	0.214	0.060	0.120	0.175	0.033	0.263	0.062	0.160	0.231	0.033	0.002	0.005	0.002	0.014	0.004	0.34	0.005	0.023	0.010	0.006
Median	0.311	0.090	0.243	0.396	0.053	0.293	0.087	0.225	0.362	0.048	0.012	0.006	0.010	0.020	0.004	0.46	0.008	0.031	0.017	0.011
Max	0.475	0.120	0.262	0.504	0.080	0.317	0.093	0.234	0.403	0.053	0.016	0.010	0.015	0.022	0.009	0.64	0.011	0.054	0.045	0.021
eSD	0.085	0.013	0.018	0.046	0.006	0.022	0.005	0.011	0.042	0.005	0.004		0.005			0.09	0.001	0.005	0.010	0.002
eCV	27	15	7	12	12	8	6	5	12	11	31		51			20	19	15	60	17
Npast	27	21	22	22	0	9	7	7	8	0	7	5	6	6	0	23	15	17	16	0
Medianpast	0.475	0.090	0.240	0.396		0.450	0.083	0.216	0.348		0.019	0.005	0.013	0.018		0.41	0.008	0.032	0.016	
SDpast	0.089	0.013	0.032	0.068		0.075	0.006	0.026	0.061		0.014	0.003	0.004	0.005		0.08	0.004	0.012	0.008	
NAV	0.311	0.090	0.243	0.396	0.053	0.293	0.087	0.225	0.362	0.048	0.012	0.006	0.010	0.020	0.004	0.46	0.008	0.031	0.017	0.011
NAU	0.085	0.016	0.036	0.056	0.011											0.11	0.004	0.010	0.010	0.005

C3

Round Robin LXXIV Laboratory Results

	Total Lycopene, µg/mL					trans-Lycopene, µg/mL					Total β-Cryptoxanthin, µg/mL					Total α-Cryptoxanthin, µg/mL				
Lab	397	398	399	400	401	397	398	399	400	401	397	398	399	400	401	397	398	399	400	401
FSV-BA	0.307	0.209	0.53	0.85	0.246	0.206	0.111	0.286	0.40	0.120	0.067	0.062	0.068	0.049	0.046	0.051	0.024	0.031	0.026	0.026
FSV-BB	0.335	0.210	0.55	0.80	0.228	0.190	0.096	0.248	0.33	0.097	0.056	0.049	0.054	0.038	0.032	0.043	0.018	0.022	0.015	0.016
FSV-BC																				
FSV-BD																				
FSV-BE																				
FSV-BF																				
FSV-BG	0.358	0.224	0.59	1.00	0.253	0.251	0.126	0.319	0.48	0.130	0.057	0.052	0.056	0.040	0.035					
FSV-BJ	0.341	0.211	0.59	1.47	0.308						*nq*	*nq*	0.034	*nq*	*nq*					
FSV-BK																				
FSV-BL																				
FSV-BM																				
FSV-BN	0.440	0.262	0.75	1.14	0.309						0.045	0.050	0.046	0.025	0.029					
FSV-BO	0.300	0.206	0.48	0.64	0.234						0.035	0.038	0.034	0.014	0.020					
FSV-BR																				
FSV-BT	0.201	0.135	0.34	0.53	0.162	0.177	0.117	0.299	0.46	0.137	0.038	0.031	0.031	0.026	0.020	0.024	0.013	0.021	0.019	0.016
FSV-BU	0.369	0.244	0.65	0.90	0.285						0.054	0.051	0.058	0.032	0.023					
FSV-BV	0.427	0.229	0.59	0.89	0.253						0.038	0.035	0.036	0.017	0.019					
FSV-BW	0.273	0.207	0.54	0.84	0.214															
FSV-CE																				
FSV-CG	0.288	0.188	0.46	0.62	0.201	0.221	0.095	0.240	0.28	0.099	0.041	0.043	0.049	0.033	0.030					
FSV-CI																				
FSV-CO	0.374	0.201	0.57	0.86	0.243						0.057	0.049	0.050	0.029	0.030					
FSV-CZ																				
FSV-DD																				
FSV-DV																				
FSV-EE																				
FSV-FK																				
FSV-FZ																				
N	12	12	12	12	12	5	5	5	5	5	10	10	11	10	10	3	3	3	3	3
Min	0.201	0.135	0.34	0.53	0.162	0.177	0.095	0.240	0.28	0.097	0.035	0.031	0.031	0.014	0.019	0.024	0.013	0.021	0.015	0.016
Median	0.338	0.210	0.56	0.86	0.245	0.206	0.111	0.286	0.40	0.120	0.049	0.049	0.049	0.030	0.029	0.043	0.018	0.022	0.019	0.016
Max	0.440	0.262	0.75	1.47	0.309	0.251	0.126	0.319	0.48	0.137	0.067	0.062	0.068	0.049	0.046	0.051	0.024	0.031	0.026	0.026
eSD	0.055	0.017	0.05	0.15	0.035	0.024	0.022	0.049	0.11	0.025	0.012	0.007	0.013	0.010	0.009					
eCV	16	8	9	17	14	12	20	17	27	21	24	14	27	32	29					
Npast	23	16	16	17	0	8	7	8	8	0	17	15	16	17	0	4	5	4	4	0
Medianpast	0.352	0.223	0.61	0.95		0.246	0.115	0.293	0.41		0.058	0.052	0.054	0.033		0.042	0.018	0.026	0.023	
SDpast	0.074	0.029	0.09	0.17		0.058	0.016	0.048	0.09		0.017	0.012	0.017	0.015		0.005	0.003	0.011	0.008	
NAV	0.338	0.210	0.56	0.86	0.245	0.206	0.111	0.286	0.40	0.120	0.049	0.049	0.049	0.030	0.029	0.043	0.018	0.022	0.019	0.016
NAU	0.075	0.051	0.11	0.16	0.058	0.037	0.022	0.052	0.11	0.025	0.012	0.012	0.013	0.010	0.009					

C4

Round Robin LXXIV Laboratory Results

	Total Lutein, µg/mL					Total Zeaxanthin, µg/mL					Total Lutein&Zeaxanthin, µg/mL					Coenzyme Q10, µg/mL					Phylloquinone (K1), ng/mL				
Lab	397	398	399	400	401	397	398	399	400	401	397	398	399	400	401	397	398	399	400	401	397	398	399	400	401
FSV-BA											0.182	0.099	0.122	0.151	0.098										
FSV-BB	0.115	0.073	0.110	0.159	0.075	0.083	0.039	0.039	0.042	0.039	0.198	0.112	0.149	0.201	0.114										
FSV-BC																									
FSV-BD																									
FSV-BE																1.18	1.04	1.14	1.63	0.97	1.063	0.629	0.820	4.273	1.022
FSV-BF																									
FSV-BG											0.196	0.102	0.136	0.171	0.105										
FSV-BJ	0.138	0.067	0.097	0.152	0.069											1.19	0.87	0.82	0.93	0.99					
FSV-BK																									
FSV-BL																									
FSV-BM																									
FSV-BN											0.228	0.111	0.108	0.133	0.084										
FSV-BO	0.195	0.105	0.115	0.135	0.092	0.018	0.018	0.024	0.031	0.023	*0.213*	*0.123*	*0.139*	*0.166*	*0.115*	0.86	0.83	0.97	1.17	0.86					
FSV-BR																									
FSV-BT	0.128	0.079	0.082	0.103	0.068	0.069	0.032	0.025	0.024	0.027	0.197	0.111	0.107	0.127	0.095	1.02	0.78	1.03	1.83	0.88					
FSV-BU											0.193	0.134	0.129	0.181	0.098										
FSV-BV											0.261	0.134	0.139	0.156	0.117										
FSV-BW																0.27	0.56	0.72	0.79	0.28					
FSV-CE																									
FSV-CG											0.212	0.143	0.137	0.142	0.114										
FSV-CI	0.130	0.064	0.080	0.104	0.063	0.066	0.026	0.015	0.015	0.018	*0.196*	*0.090*	*0.095*	*0.119*	*0.081*	0.70	0.64	0.70	1.04	0.62	0.550	0.251	0.406	1.519	0.473
FSV-CO											0.240	0.109	0.122	0.138	0.094										
FSV-CZ																1.32	1.02	1.42	2.02	1.11					
FSV-DD																									
FSV-DV																									
FSV-EE																0.98	0.87	1.02	1.35	0.83					
FSV-FK																									
FSV-FZ																									
N	5	5	5	5	5	4	4	4	4	4	11	11	11	11	11	8	8	8	8	8	2	2	2	2	2
Min	0.115	0.064	0.080	0.103	0.063	0.018	0.018	0.015	0.015	0.018	0.182	0.090	0.095	0.119	0.081	0.27	0.56	0.70	0.79	0.28	0.550	0.251	0.406	1.519	0.473
Median	0.130	0.073	0.097	0.135	0.069	0.068	0.029	0.025	0.028	0.025	0.198	0.111	0.129	0.151	0.098	1.00	0.85	0.99	1.26	0.87	0.807	0.440	0.613	2.896	0.748
Max	0.195	0.105	0.115	0.159	0.092	0.083	0.039	0.039	0.042	0.039	0.261	0.143	0.149	0.201	0.117	1.32	1.04	1.42	2.02	1.11	1.063	0.629	0.820	4.273	1.022
eSD	0.012	0.009	0.022	0.036	0.009	0.012	0.010	0.007	0.012	0.007	0.021	0.017	0.015	0.027	0.021	0.27	0.18	0.24	0.52	0.16					
eCV	9	13	23	27	13	18	34	30	43	27	11	15	11	18	22	27	21	24	41	19					
Npast	11	8	8	9	0	9	7	9	7	0	18	16	17	17	0	8	8	9	9	0	0	0	0	0	0
Medianpast	0.146	0.078	0.090	0.121		0.081	0.035	0.030	0.027		0.215	0.111	0.127	0.147		1.03	0.87	0.96	1.38						
SDpast	0.026	0.017	0.020	0.024		0.017	0.013	0.013	0.013		0.033	0.021	0.035	0.037		0.23	0.07	0.07	0.20						
NAV	0.130	0.073	0.097	0.135	0.069	0.068	0.029	0.025	0.028	0.025	0.198	0.111	0.129	0.151	0.098	1.00	0.85	0.99	1.26	0.87					
NAU	0.024	0.015	0.022	0.036	0.014						0.041	0.023	0.027	0.032	0.021	0.27	0.18	0.24	0.52	0.16					

C5

Round Robin LXXIV Laboratory Results

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
SD	Adjusted median absolute deviation from the median of the non-NIST results
CV	Coefficient of Variation for (non-NIST) results: 100*SD/Median
N_{past}	Mean of N(s) from past RR(s)
$Median_{past}$	Mean of Median(s) from past RR(s)
SD_{past}	Pooled SD from past RR(s)
NIST	Mean of NIST results
NAV	NIST Assigned Value = (Median + NIST)/2 for analytes reported by NIST = Median for analytes reported by ≥ 5 labs but not NIST
NAU	NIST Assigned Uncertainty: $\sqrt{(S^2 + S_{btw}^2)}$ S is the maximum of (0.05*NAV, SD, SD_{past}, eSD) and S_{btw} is the standard deviation between Median and NIST. The expected long-term SD, eSD, is defined in: Duewer et al., Anal Chem 1997;69(7):1406-1413.
-	Not analyzed
nd	Not detected (i.e., no detectable peak for analyte)
nq	Detected but not quantitatively determined
italics	Not explicitly reported but calculated by NIST from reported values

Comparability Summary

Lab	TR	aT	g/bT	bC	aC	TLy	TbX	TLu	L&Z
FSV-BA	1	1	2	1	1	1	2		1
FSV-BB	1	1	1	1	1	1	1	1	1
FSV-BC	1								
FSV-BD	1	2							
FSV-BE	1	1	1	1					
FSV-BF	2	1		1					
FSV-BG	1	1	1	1	1	1	1		1
FSV-BJ	1	1	1	2		2		1	
FSV-BK	2	1							
FSV-BL	1	1							
FSV-BM	2	1							
FSV-BN	2	1		1	2	2	1		1
FSV-BO	2	1	1	2	1	1	2	2	1
FSV-BR	1	2							
FSV-BT	1	1	1	1	1	2	2	1	1
FSV-BU	2	1	1	1	1	1	1		1
FSV-BV	2	2	1	1	1	1	2		1
FSV-BW	1	2	1	2	2	1			
FSV-CE	2	2		2					
FSV-CG	1	4	2	3		1	1		1
FSV-CI	2	1	2	1	2			1	1
FSV-CO		1	2	1	1	1	1		1
FSV-CZ	2	1	1	3					
FSV-DD	1								
FSV-DV	2	2							
FSV-FK	1	1		1					
FSV-FZ	1	1	1	1					
n	26	25	15	19	11	12	10	5	11

	TR	aT	g/bT	bC	aC	TLy	TbX	TLu	L&Z
% 1	58	72	73	68	73	75	60	80	100
% 2	42	24	27	21	27	25	40	20	0
% 3	0	0	0	11	0	0	0	0	0
% 4	0	4	0	0	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
aC	Total α-Carotene
TLy	Total Lycopene
TbX	Total β-Cryptoxanthin
TLu	Total Lutein
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 RR74

Each participant in RR74 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 Lutein & Zeaxanthin
- Coenzyme Q10

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

Individualized Round Robin LXXIV Report: FSV-BA

Summary

	Serum 397			Serum 398			Serum 399			Serum 400			Serum 401		
Analyte	You	NAV	n	You	NAV	n	You	NAV	n	You	NAV	n	You	NAV	n
Total Retinol	0.744	0.664	26	0.345	0.348	26	0.499	0.500	26	0.637	0.637	26	0.469	0.460	25
Retinyl Palmitate	0.08	0.08	6	0.0	0.0	6	0.1	0.0	6	0.16	0.10	6	0.04	0.02	6
α-Tocopherol	9.65	8.50	25	7.03	6.70	25	10.91	10.34	25	19.92	18.77	25	5.38	5.37	24
γ/β-Tocopherol	4.963	4.000	15	2.063	1.710	15	1.648	1.353	15	2.714	2.182	15	1.527	1.270	15
δ-Tocopherol	0.848	0.559	4	0.089	0.089	3	0.078	0.078	3	0.241	0.201	3	0.094	0.094	3
Total β-Carotene	0.303	0.311	19	0.099	0.090	19	0.245	0.243	19	0.425	0.396	19	0.060	0.053	19
trans-β-Carotene	0.287	0.293	4	0.089	0.087	4	0.234	0.225	4	0.403	0.362	4	0.051	0.048	4
Total cis-β-Carotene	0.016	0.012	4	0.010	0.006	3	0.011	0.010	4	0.022	0.020	3	0.009	0.004	3
Total α-Carotene	0.382	0.459	13	0.008	0.008	11	0.028	0.031	11	0.017	0.017	11	0.012	0.012	11
Total Lycopene	0.307	0.338	12	0.209	0.210	12	0.532	0.557	12	0.849	0.856	12	0.246	0.245	12
trans-Lycopene	0.206	0.206	5	0.111	0.111	5	0.286	0.286	5	0.399	0.399	5	0.120	0.120	5
Total β-Cryptoxanthin	0.067	0.049	10	0.062	0.049	10	0.068	0.049	11	0.049	0.030	10	0.046	0.029	10
Total α-Cryptoxanthin	0.051	0.043	3	0.024	0.018	3	0.031	0.022	3	0.026	0.019	3	0.026	0.016	3
Total Lutein&Zeaxanthin	0.182	0.198	11	0.099	0.111	11	0.122	0.129	11	0.151	0.151	11	0.098	0.098	11

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

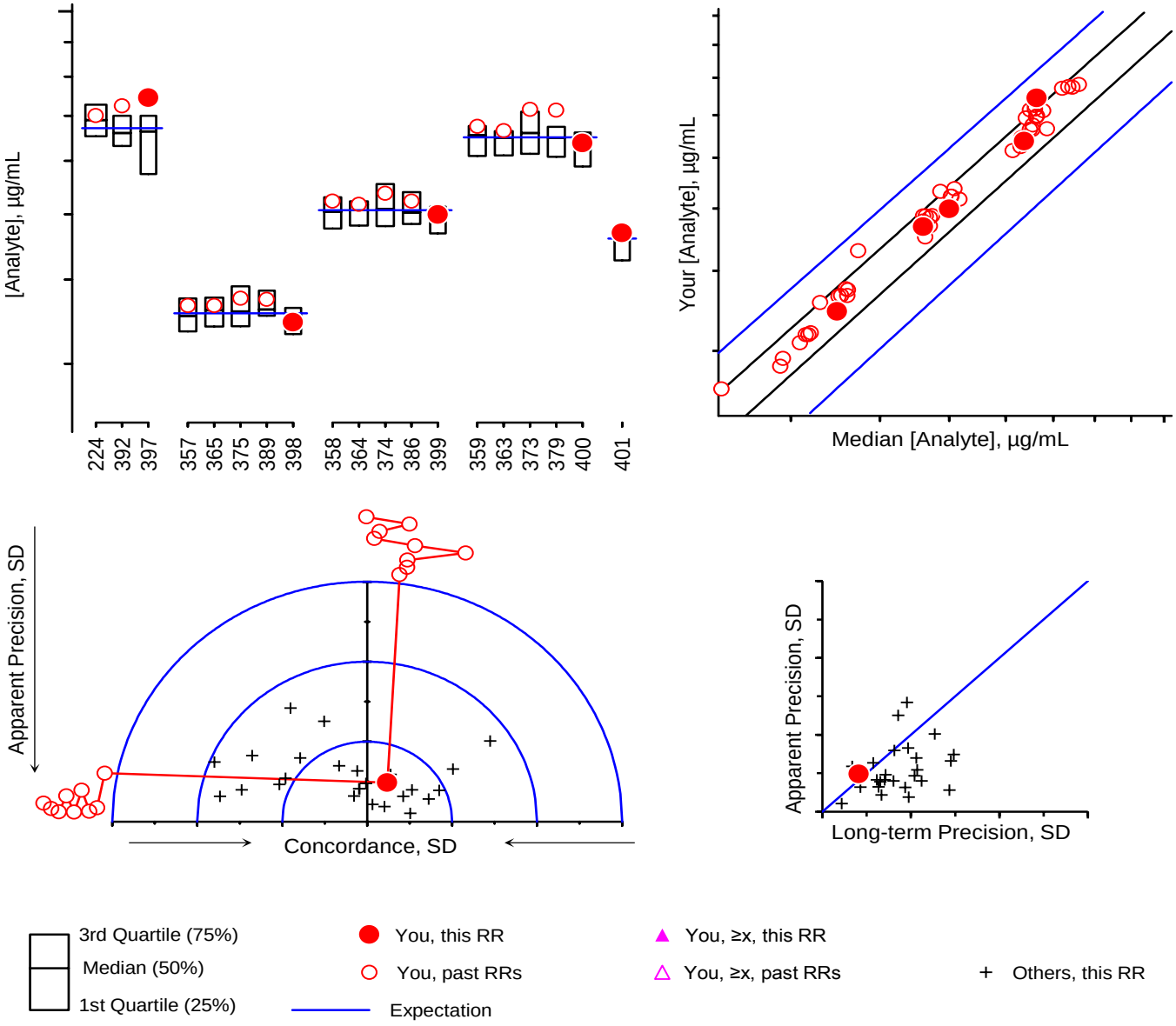
Please check our records against your records. Send corrections and/or updates to...

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 LXXIV Report: FSV-BA

Total Retinol, µg/mL

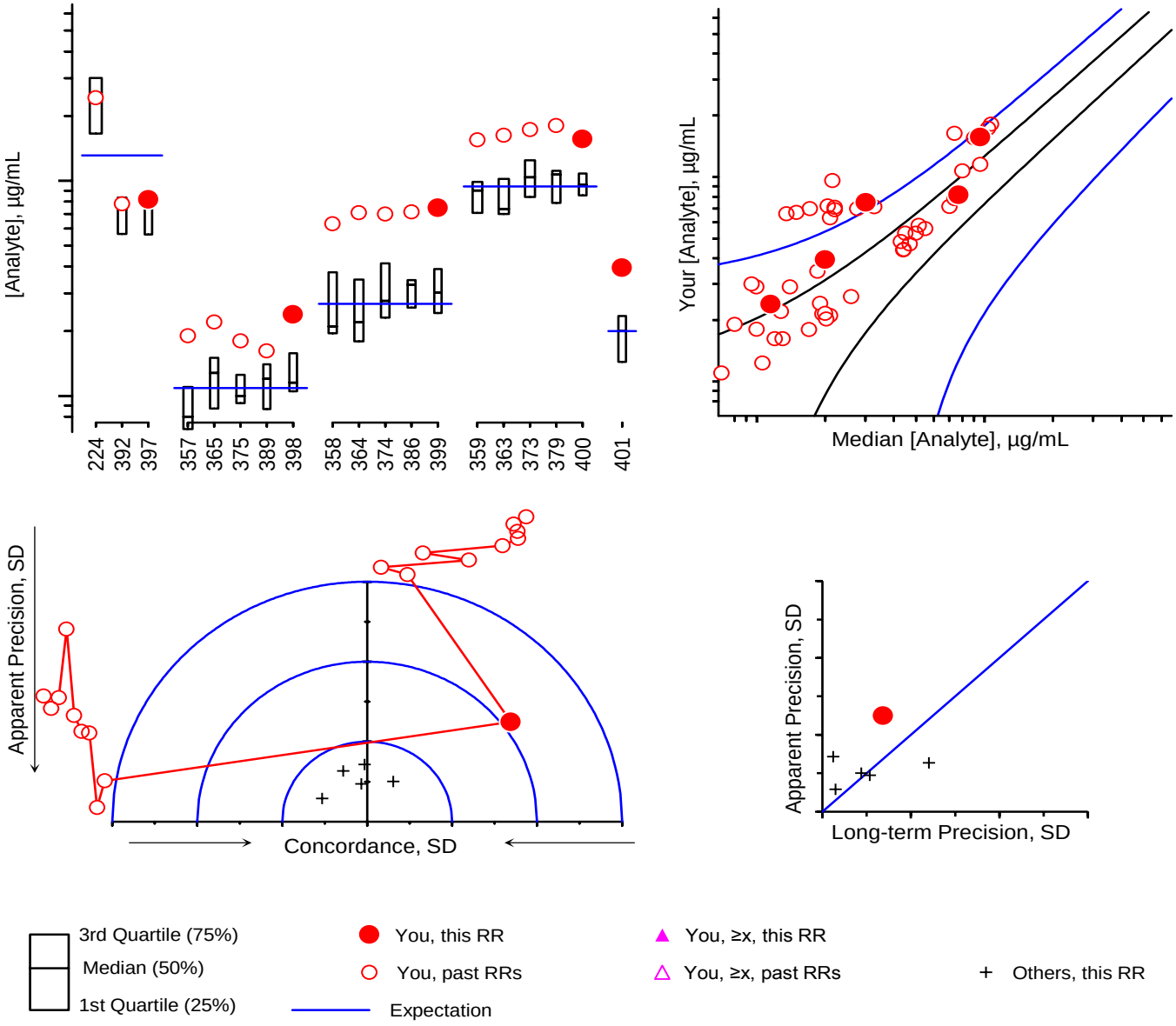


For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

Serum	Comments	History
#397	Lyophilized, augmented, multi-donor	38#224, 73#392
#398	Fresh-frozen, native, multi-donor: SRM 968e I	66#357, 67#365, 69#375, 72#389
#399	Fresh-frozen, native, multi-donor: SRM 968e II	66#358, 67#364, 69#374, 71#386
#400	Fresh-frozen, native, multi-donor: SRM 968e III	66#359, 67#363, 69#373, 70#379
#401	Fresh-frozen, native, multi-donor	Initial distribution

Individualized RR LXXIV Report: FSV-BA

Retinyl Palmitate, µg/mL



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

Serum	Comments	History
#397	Lyophilized, augmented, multi-donor	38#224, 73#392
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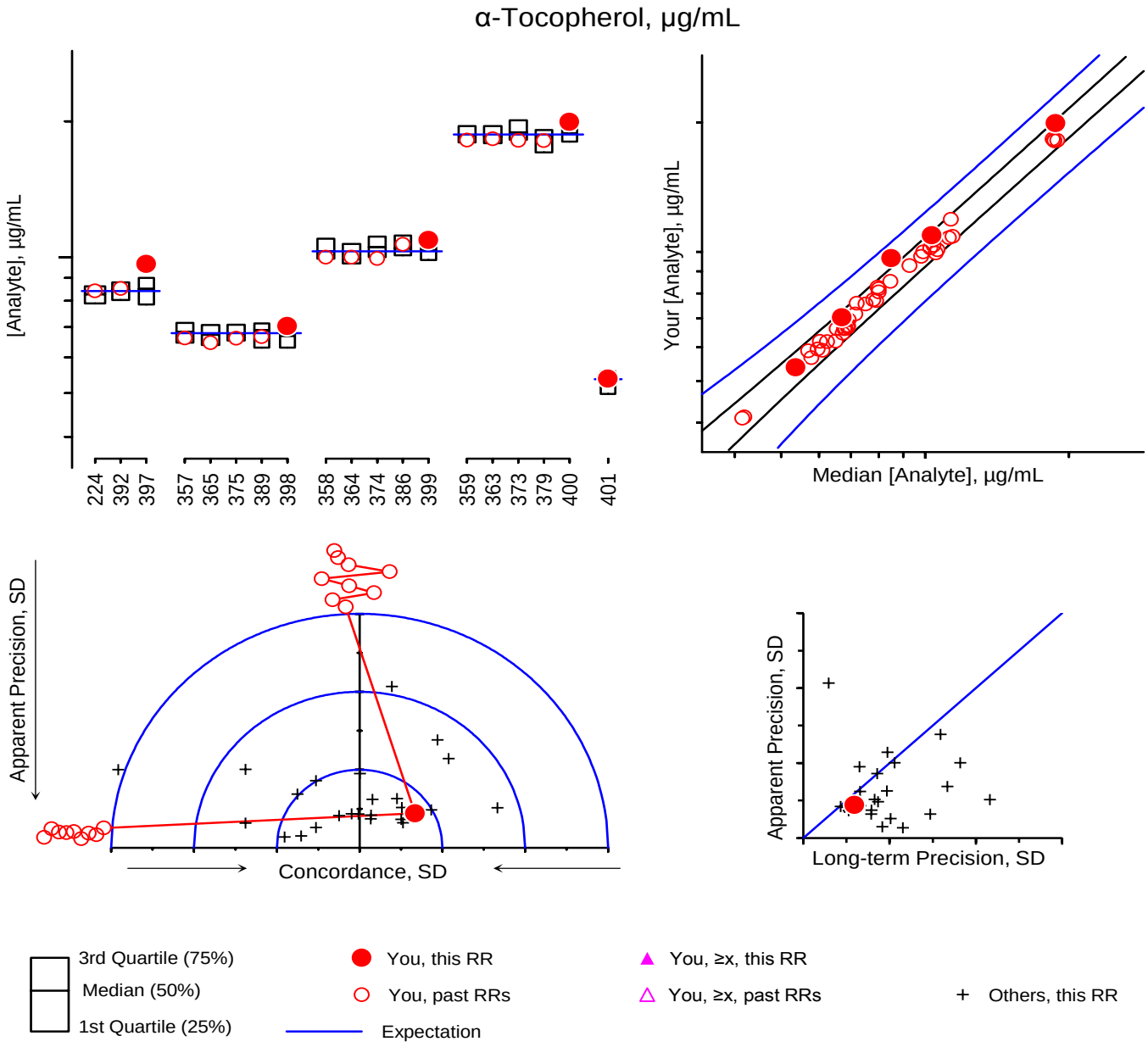
#401	Fresh-frozen, native, multi-donor	Initial distribution

Individualized RR LXXIV 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.

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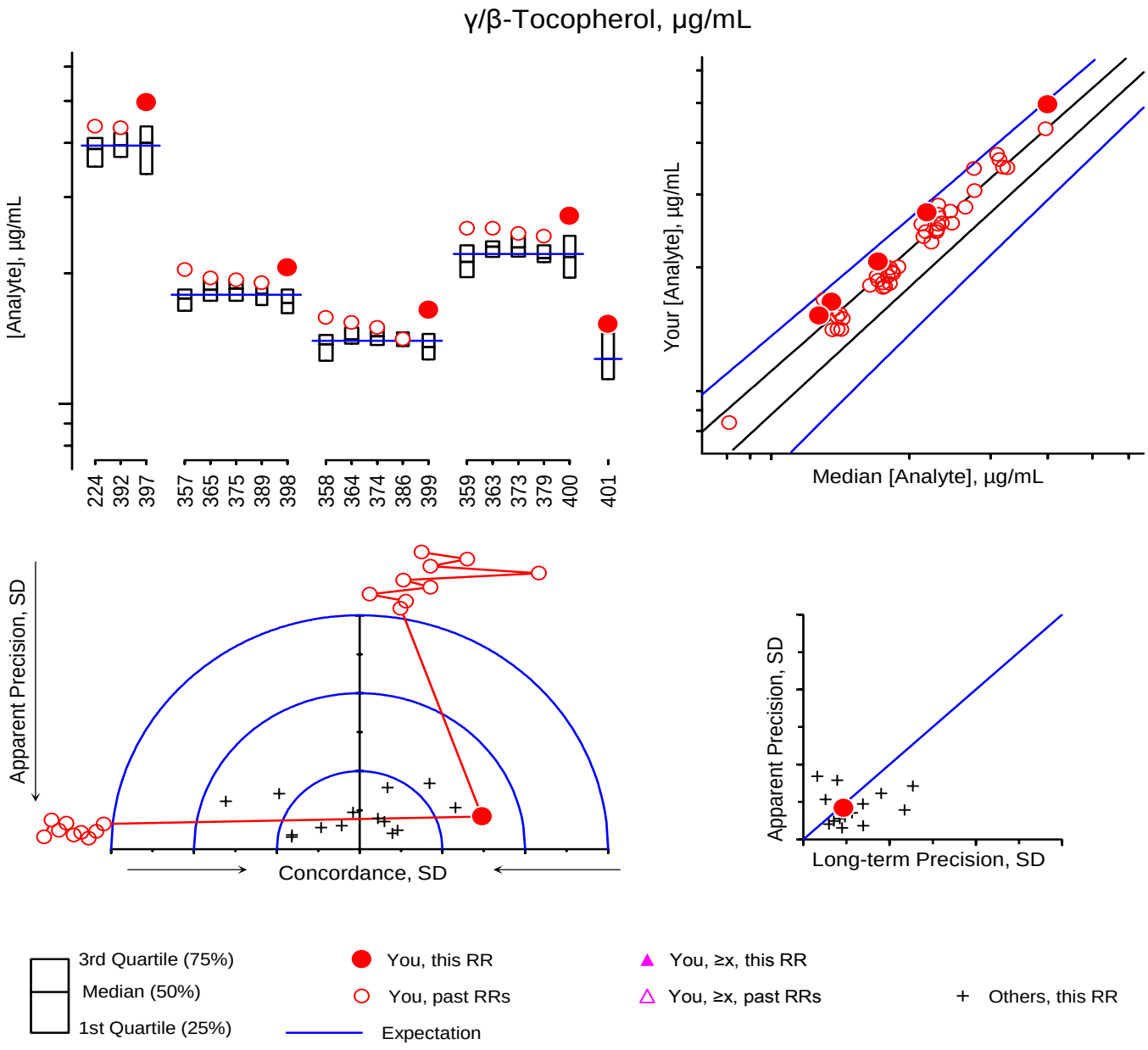
#401	Fresh-frozen, native, multi-donor	Initial distribution

Individualized RR LXXIV 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.

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#397	Lyophilized, augmented, multi-donor	38#224, 73#392
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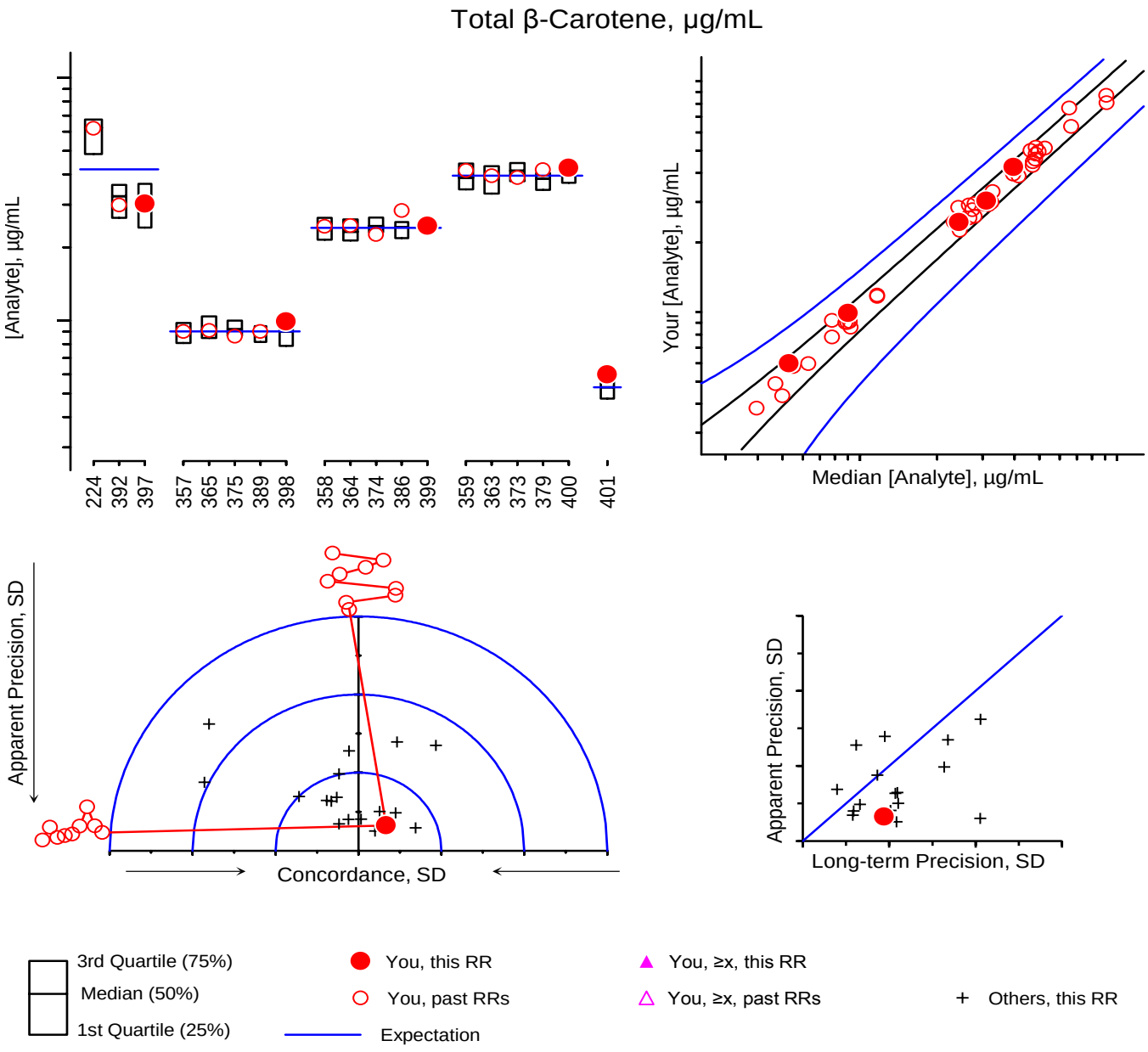
Individualized RR LXXIV Report: FSV-BA



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	Comments	History
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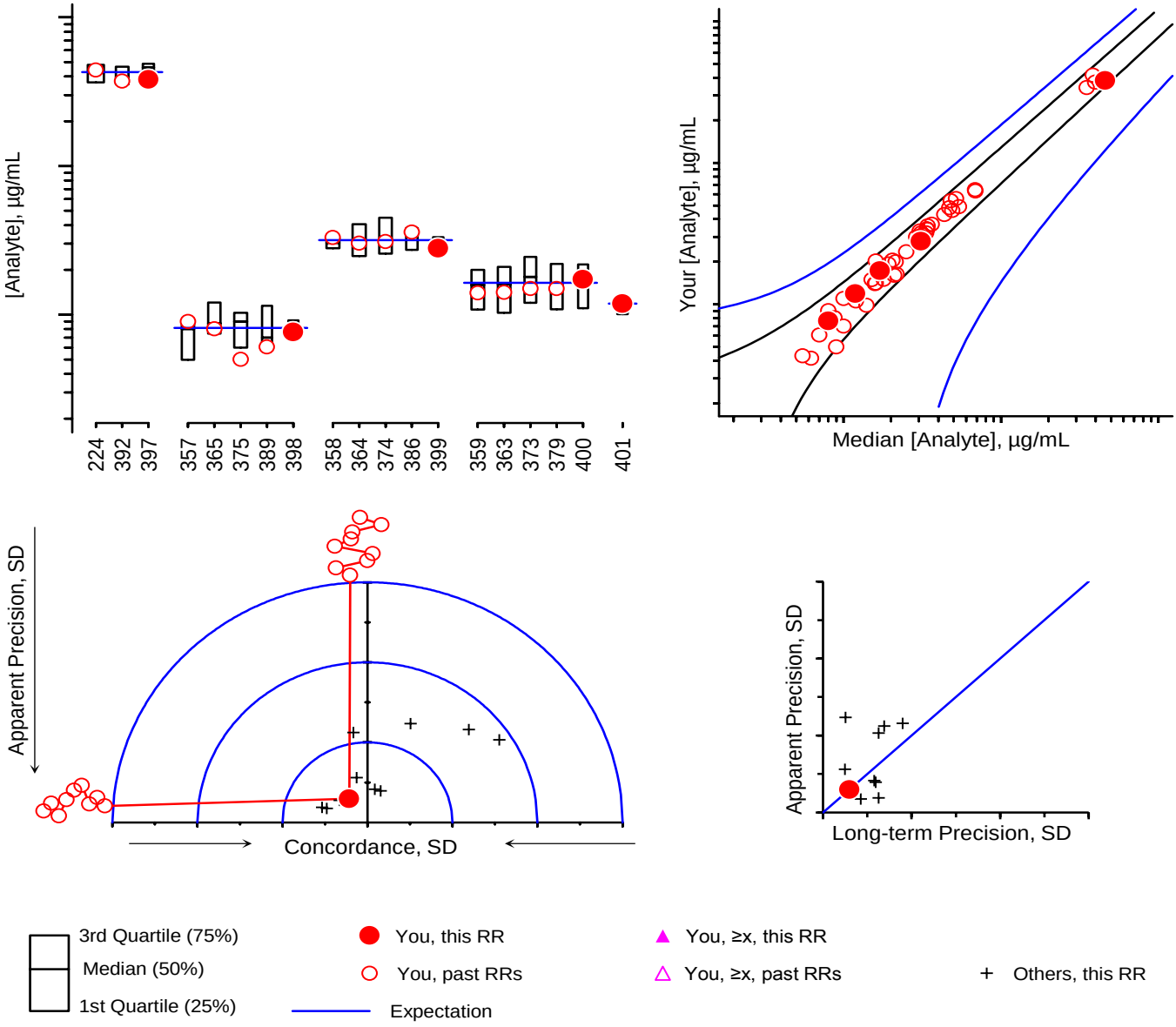
#401	Fresh-frozen, native, multi-donor	Initial distribution

Individualized RR LXXIV Report: FSV-BA

Total α-Carotene, µg/mL



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

Serum	Comments	History
#397	Lyophilized, augmented, multi-donor	38#224, 73#392
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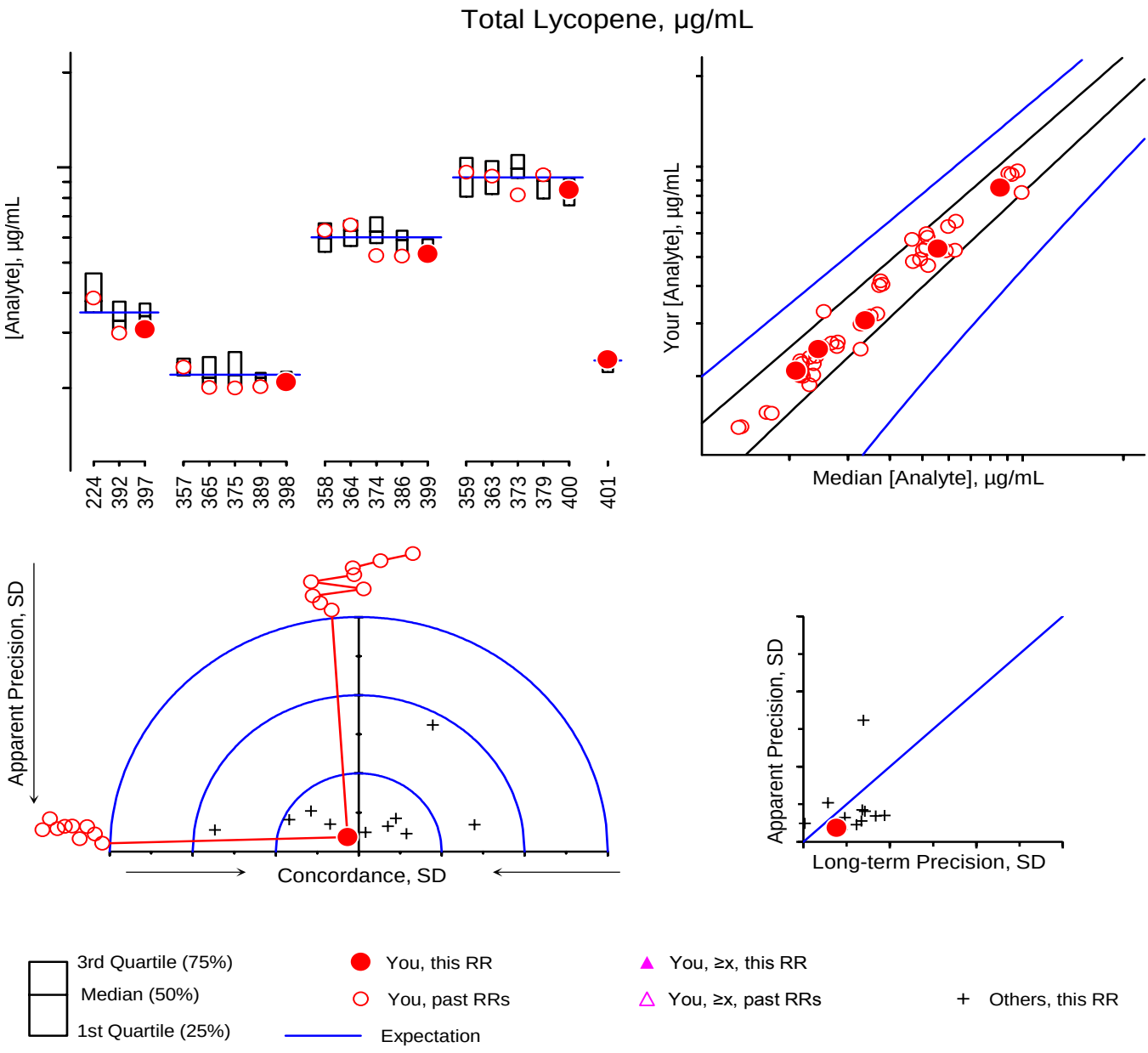
#401	Fresh-frozen, native, multi-donor	Initial distribution

Individualized RR LXXIV Report: FSV-BA



For details of the construction and interpretation of these plots, see:
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Serum	Comments	History
#397	Lyophilized, augmented, multi-donor	38#224, 73#392
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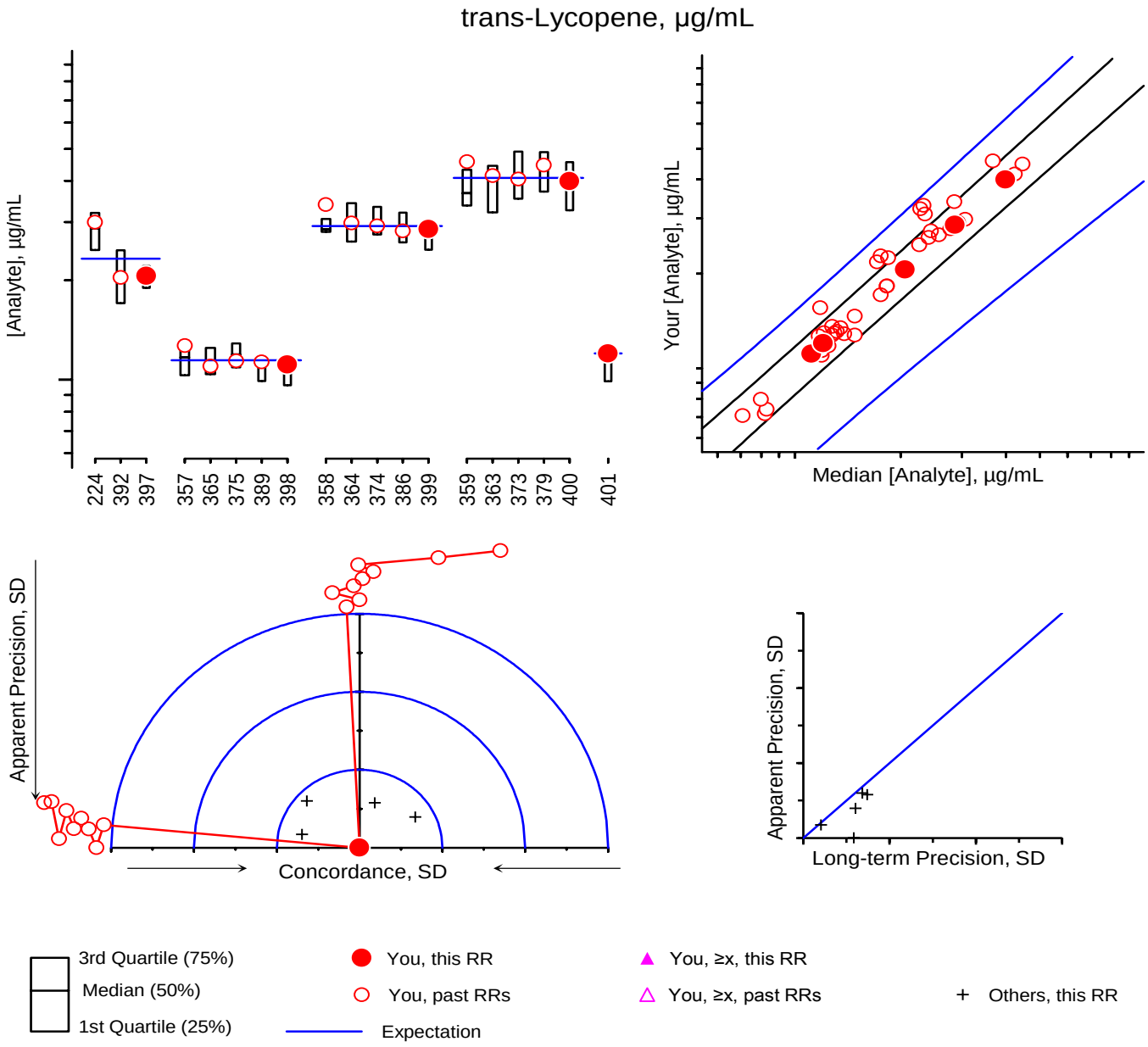
#401	Fresh-frozen, native, multi-donor	Initial distribution

Individualized RR LXXIV 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.

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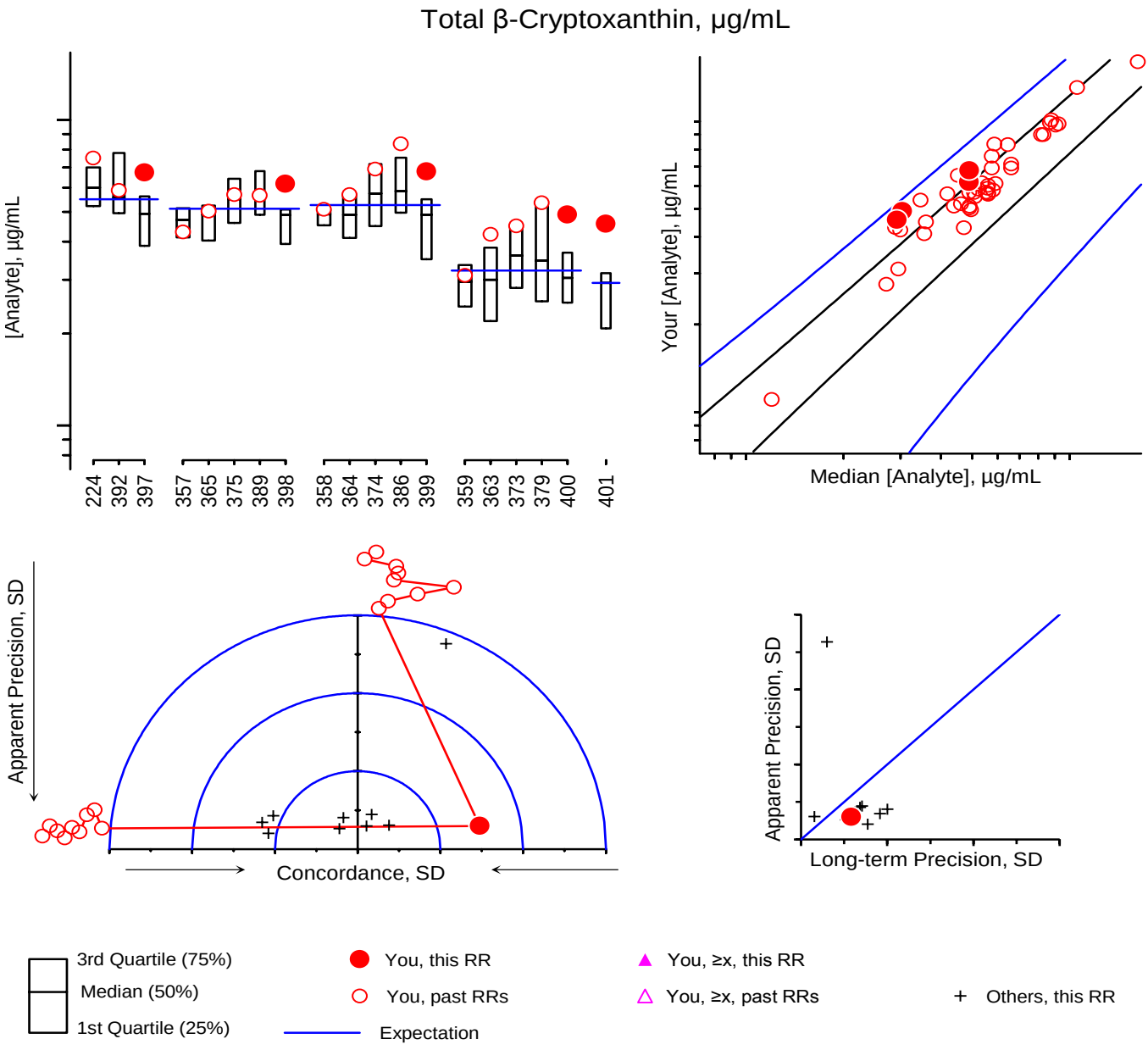
#401	Fresh-frozen, native, multi-donor	Initial distribution

Individualized RR LXXIV Report: FSV-BA



For details of the construction and interpretation of these plots, see:
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Serum	Comments	History
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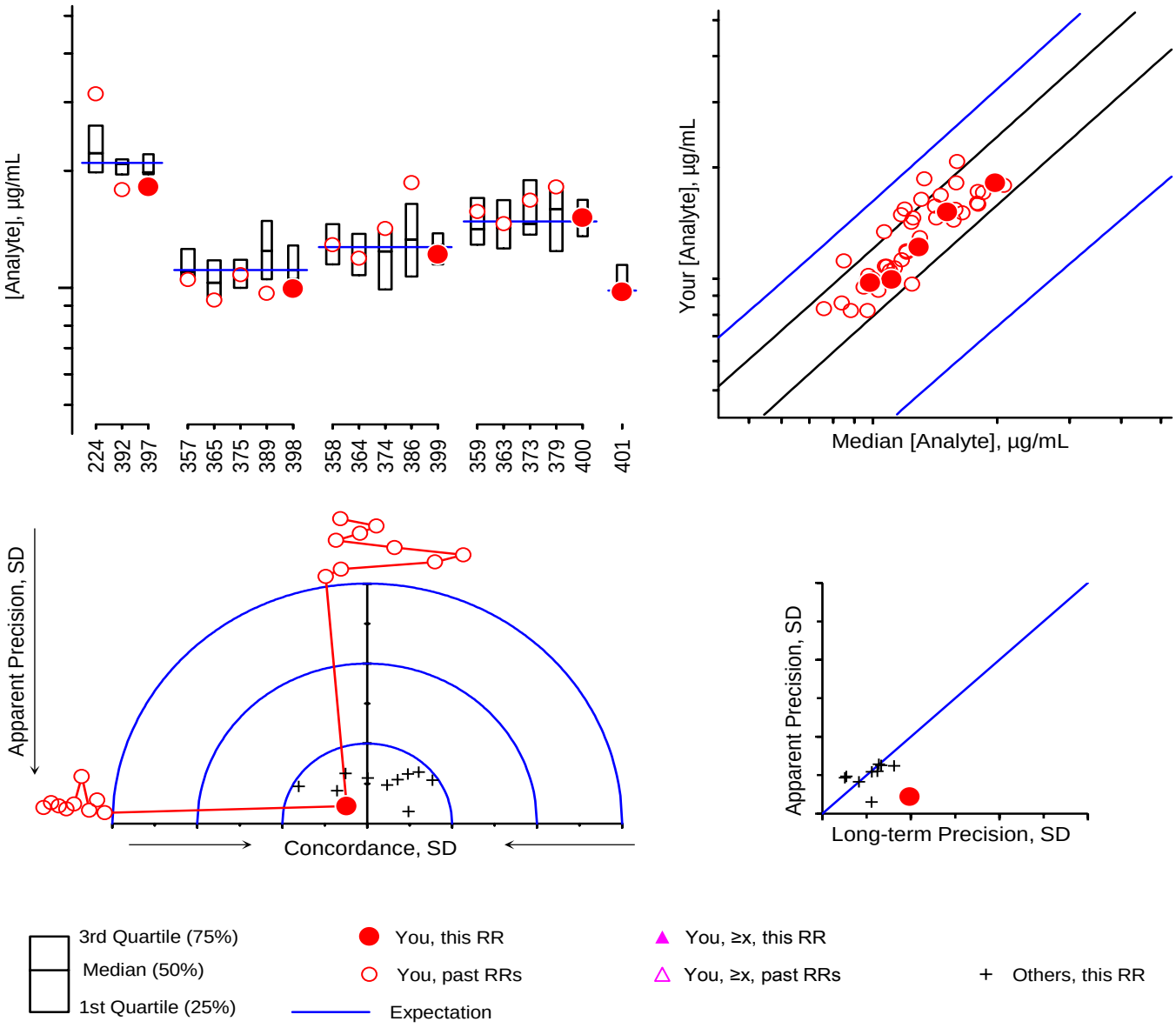
#401	Fresh-frozen, native, multi-donor	Initial distribution

Individualized RR LXXIV Report: FSV-BA

Total Lutein&Zeaxanthin, µg/mL



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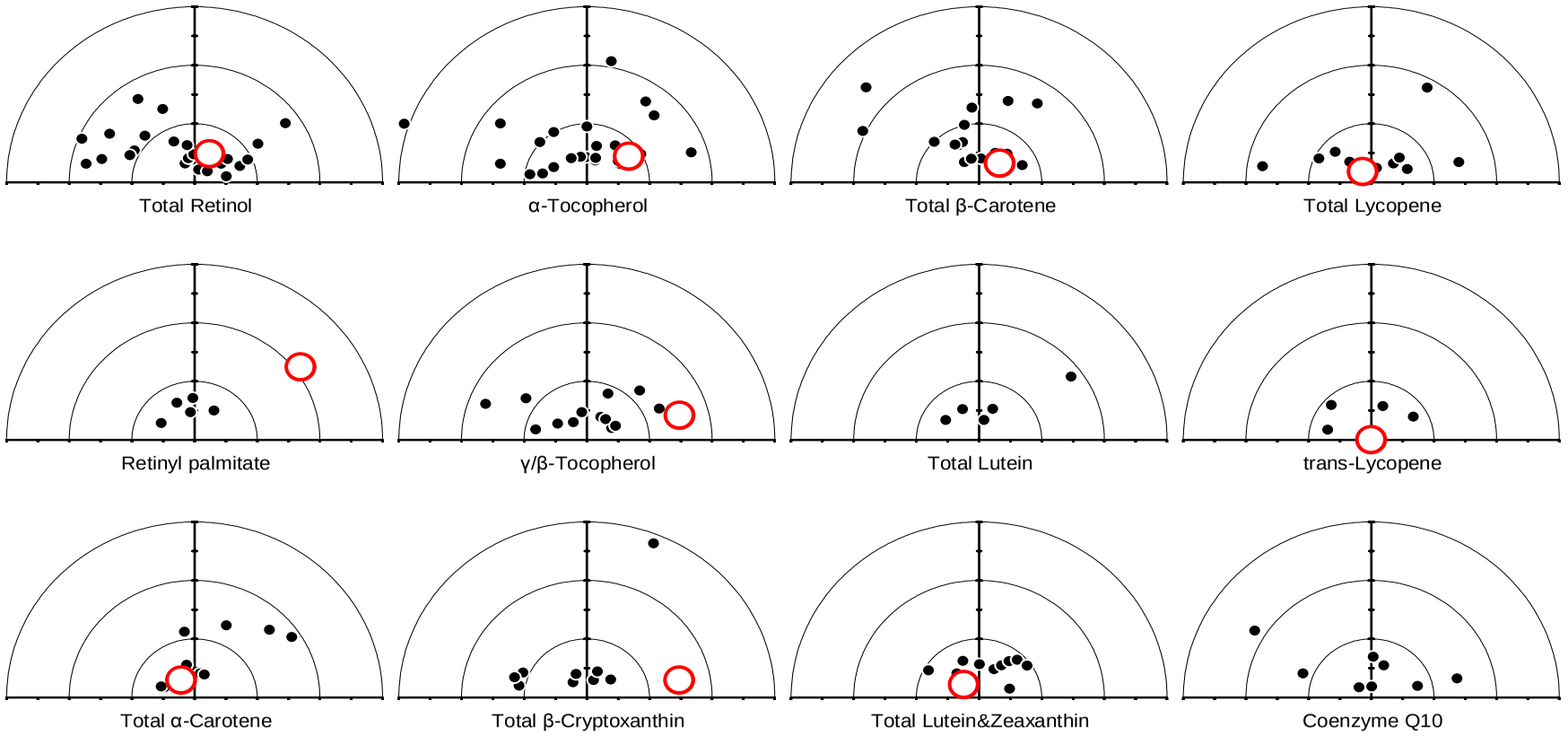
#401	Fresh-frozen, native, multi-donor	Initial distribution

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

D13



Appendix E. Shipping Package Inserts for RR39

The following three items were included in each package shipped to an RR39 participant:

- Cover letter
- Analysis of Control Materials and Test Samples 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, 2013

Dear Colleague:

The samples within this package constitute Vitamin C Round Robin 39 (RR39) of the 2013 Micronutrients Measurement Quality Assurance Program. RR39 consists of one vial each of four frozen serum *test samples* (#391, #392 #393, and #394) 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 301-975-3120 or jbthomas@nist.gov.

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 **September 30, 2013**.

If you have questions or comments regarding this study, please e-mail me at jbthomas@nist.gov or call me at (301) 975-3120.

Sincerely,

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

Enclosure: RR39 Report Form for Control Material and Test Sample Analyses

NIST

Participant #: _____ Date: _____

Vitamin C Round Robin 39

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 #391			µmol/L of Sample
Test sample #392			µmol/L of Sample
Test sample #393			µmol/L of Sample
Test sample #394			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 **September 30 2013**

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 39

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 #391	Liquid frozen (1:1 serum:10% MPA)
VitC #392	Liquid frozen (1:1 serum:10% MPA)
VitC #393	Liquid frozen (1:1 serum:10% MPA)
VitC #394	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

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 6392
Gaithersburg, MD 20899-6392

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

Appendix F. Final Report for RR39

The following six pages are the final report for RR39 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 20899-8390

December 3, 2013

Dear Colleague:

Enclosed is the summary report of the results for Round Robin 39 (RR39) 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 "adjusted median absolute deviation from the median" (MADe) is used to estimate the expected standard deviation, and we estimate the coefficient of variation (CV) as 100×MADe/median.

RR39 consisted of four test samples (#391, #392 #393, and #394) and one vial each of two frozen control serum control samples (CS #3 and CS #4), and one vial of solid control material (Control) 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 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.

Samples for the 2014 Micronutrients Measurements Quality Assurance Program (MMQAP) will be shipped **starting January 27, 2014**. We will ship the samples for both exercises for each study at the same time. Results are due in April for the first study; written feedback will be provided to laboratories in May. We will send you a notification around June about the reporting deadline for the second study. Please contact us immediately if this schedule is problematic for your laboratory.

We have completed on-line documentation of data and reports for the MMQAP studies from 1996 to 2012. For your convenience, enclosed are the links to these documents. Data summaries have been altered to ensure confidentiality of identification codes assigned to laboratories. We anticipate that the results for Round Robins 73 and 74 Fat-Soluble Vitamins and Carotenoids in Human Serum and Round Robins 38 and 39 Ascorbic Acid in Human Serum will be available online by Spring 2014.

If you have questions or concerns regarding this report, please contact David Duewer at david.duewer@nist.gov or me at jbthomas@nist.gov, 301-975-3120, 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

MMQAP Comparability Studies (1996 to 2012)

D.L. Duewer, M.C. Kline, K.E. Sharpless, J.M. Brown Thomas, S.A. Margolis, NIST Micronutrients Measurement Quality Assurance Program Winter, Spring, and Fall 1996 Comparability Studies: Results for Round Robin XXXVI, XXXVII, and XXXVIII Fat-Soluble Vitamins and Carotenoids in Human Serum and Round Robin 9 Ascorbic Acid in Human Serum, NIST Interagency/Internal Report (NISTIR) 7880-28

D.L. Duewer, M.C. Kline, K.E. Sharpless, J.M. Brown Thomas, S.A. Margolis, NIST Micronutrients Measurement Quality Assurance Program Winter, Spring, and Fall 1997 Comparability Studies: Results for Round Robin XXXIX, XL, and XLI Fat-Soluble Vitamins and Carotenoids in Human Serum and Round Robin 10 Ascorbic Acid in Human Serum, NIST Interagency/Internal Report (NISTIR) 7880-27

D.L. Duewer, M.C. Kline, K.E. Sharpless, J.M. Brown Thomas, S.A. Margolis, NIST Micronutrients Measurement Quality Assurance Program Winter, Spring, and Fall 1998 Comparability Studies: Results for Round Robin XLII, XLIII, and XLIV Fat-Soluble Vitamins and Carotenoids in Human Serum and Round Robin 11 Ascorbic Acid in Human Serum, NIST Interagency/Internal Report (NISTIR) 7880-26

David Lee Duewer, Sam A. Margolis, Katherine E Sharpless, Jeanice M Brown Thomas, NIST Micronutrients Measurement Quality Assurance Program Winter and Summer 1999 Comparability Studies: Results for Round Robin XLV and XLVI Fat-Soluble Vitamins and Carotenoids in Human Serum and Round Robin 12 Ascorbic Acid in Human Serum, NIST Interagency/Internal Report (NISTIR) 7880-25

D.L. Duewer, M.C. Kline, K.E. Sharpless, J.M. Brown Thomas, S.A. Margolis, NIST Micronutrients Measurement Quality Assurance Program Winter and Summer 2000 Comparability Studies: Results for Round Robins XLVII and XLVIII Fat-Soluble Vitamins and Carotenoids in Human Serum and Round Robin 13 Ascorbic Acid in Human Serum, NIST Interagency/Internal Report (NISTIR) 7880-24

D.L. Duewer, K.E. Sharpless, J.M. Brown Thomas, S.A. Margolis, NIST Micronutrients Measurement Quality Assurance Program Winter 2001 Comparability Studies: Results for Round Robin XLIX Fat-Soluble Vitamins and Carotenoids in Human Serum and Round Robin 14 Ascorbic Acid in Human Serum, NIST Interagency/Internal Report (NISTIR) 7880-23

D.L. Duewer, K.E. Sharpless, J.M. Brown Thomas, S.A. Margolis, NIST Micronutrients Measurement Quality Assurance Program Summer 2001 Comparability Studies: Results for Round Robin L Fat-Soluble Vitamins and Carotenoids in Human Serum and Round Robin 15 Ascorbic Acid in Human Serum, NIST Interagency/Internal Report (NISTIR) 7880-22

D.L. Duewer, K.E. Sharpless, J.M. Brown Thomas, S.A. Margolis, NIST Micronutrients Measurement Quality Assurance Program Winter 2002 Comparability Studies: Results for Round Robin LI Fat-Soluble Vitamins and Carotenoids in Human Serum and Round Robin 16 Ascorbic Acid in Human Serum, NIST Interagency/Internal Report (NISTIR) 7880-21

D.L. Duewer, M.C. Kline, K.E. Sharpless, J.M. Brown Thomas, S.A. Margolis, NIST Micronutrients Measurement Quality Assurance Program Summer 2002 Comparability Studies: Results for Round Robin LII Fat-Soluble Vitamins and Carotenoids in Human Serum and Round Robin 17 Ascorbic Acid in Human Serum, NIST Interagency/Internal Report (NISTIR) 7880-20

D.L. Duewer, K.E. Sharpless, J.M. Brown Thomas, NIST Micronutrients Measurement Quality Assurance Program Winter 2003 Comparability Studies: Results for Round Robin LIII Fat-Soluble Vitamins and Carotenoids in Human Serum and Round Robin 18 Ascorbic Acid in Human Serum, NIST Interagency/Internal Report (NISTIR) 7880-19

D.L. Duewer, K.E. Sharpless, J.M. Brown Thomas, NIST Micronutrients Measurement Quality Assurance Program Summer 2003 Comparability Studies: Results for Round Robin LIV Fat-Soluble Vitamins and Carotenoids in Human Serum and Round Robin 19 Ascorbic Acid in Human Serum, NIST Interagency/Internal Report (NISTIR) 7880-18

D.L. Duewer, J.M. Brown Thomas, NIST Micronutrients Measurement Quality Assurance Program Winter 2004 Comparability Studies: Results for Round Robin LV Fat-Soluble Vitamins and Carotenoids in Human Serum and Round Robin 20 Ascorbic Acid in Human Serum, NIST Interagency/Internal Report (NISTIR) 7880-17

D.L. Duewer, J.M. Brown Thomas, NIST Micronutrients Measurement Quality Assurance Program Summer 2004 Comparability Studies: Results for Round Robin LVI Fat-Soluble Vitamins and Carotenoids in Human Serum and Round Robin 21 Ascorbic Acid in Human Serum, NIST Interagency/Internal Report (NISTIR) 7880-16

D.L. Duewer, J.M. Brown Thomas, NIST Micronutrients Measurement Quality Assurance Program Winter 2005 Comparability Studies: Results for Round Robin LVII Fat-Soluble Vitamins and Carotenoids in Human Serum and Round Robin 22 Ascorbic Acid in Human Serum, NIST Interagency/Internal Report (NISTIR) 7880-15

D.L. Duewer, J.M. Brown Thomas, NIST Micronutrients Measurement Quality Assurance Program Summer 2005 Comparability Studies: Results for Round Robin LVIII Fat-Soluble Vitamins and Carotenoids in Human Serum and Round Robin 23 Ascorbic Acid in Human Serum, NIST Interagency/Internal Report (NISTIR) 7880-14

D.L. Duewer, J.M. Brown Thomas, NIST Micronutrients Measurement Quality Assurance Program Winter 2006 Comparability Studies: Results for Round Robin LIX Fat-Soluble Vitamins and Carotenoids in Human Serum and Round Robin 24 Ascorbic Acid in Human Serum, NIST Interagency/Internal Report (NISTIR) 7880-13

D.L. Duewer, J.M. Brown Thomas, NIST Micronutrients Measurement Quality Assurance Program Summer 2006 Comparability Studies: Results for Round Robin LX Fat-Soluble Vitamins and Carotenoids in Human Serum and Round Robin 25 Ascorbic Acid in Human Serum, NIST Interagency/Internal Report (NISTIR) 7880-12

D.L. Duewer, J.M. Brown Thomas, NIST Micronutrients Measurement Quality Assurance Program Winter 2007 Comparability Studies: Results for Round Robin LXI Fat-Soluble Vitamins and Carotenoids in Human Serum and Round Robin 26 Ascorbic Acid in Human Serum, NIST Interagency/Internal Report (NISTIR) 7880-11

D.L. Duewer, J.M. Brown Thomas, NIST Micronutrients Measurement Quality Assurance Program Summer 2007 Comparability Studies: Results for Round Robin LXII Fat-Soluble Vitamins and Carotenoids in Human Serum and Round Robin 27 Ascorbic Acid in Human Serum, NIST Interagency/Internal Report (NISTIR) 7880-10

D.L. Duewer, J.M. Brown Thomas, NIST Micronutrients Measurement Quality Assurance Program Winter 2008 Comparability Studies: Results for Round Robin LXIII Fat-Soluble Vitamins and Carotenoids in Human Serum and Round Robin 28 Ascorbic Acid in Human Serum, NIST Interagency/Internal Report (NISTIR) 7880-9

D.L. Duewer, J.M. Brown Thomas, NIST Micronutrients Measurement Quality Assurance Program Summer 2008 Comparability Studies: Results for Round Robin LXIV Fat-Soluble Vitamins and Carotenoids in Human Serum and Round Robin 29 Ascorbic Acid in Human Serum, NIST Interagency/Internal Report (NISTIR) 7880-8

D.L. Duewer, J.M. Brown Thomas, NIST Micronutrients Measurement Quality Assurance Program Winter 2009 Comparability Studies: Results for Round Robin LXV Fat-Soluble Vitamins and Carotenoids in Human Serum and Round Robin 30 Ascorbic Acid in Human Serum, NIST Interagency/Internal Report (NISTIR) 7880-7

D.L. Duewer, J.M. Brown Thomas, 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, NIST Interagency/Internal Report (NISTIR) 7880-6

D.L. Duewer, J.M. Brown Thomas, NIST Micronutrients Measurement Quality Assurance Program Winter 2010 Comparability Studies: Results for Round Robin LXVII Fat-Soluble Vitamins and Carotenoids in Human Serum and Round Robin 32 Ascorbic Acid in Human Serum, NIST Interagency/Internal Report (NISTIR) 7880-5

D.L. Duewer, J.M. Brown Thomas, NIST Micronutrients Measurement Quality Assurance Program Summer 2010 Comparability Studies: Results for Round Robin LXVIII Fat-Soluble Vitamins and Carotenoids in Human Serum and Round Robin 33 Ascorbic Acid in Human Serum, NIST Interagency/Internal Report (NISTIR) 7880-4

D.L. Duewer, J.M. Brown Thomas, NIST Micronutrients Measurement Quality Assurance Program Winter 2011 Comparability Studies: Results for Round Robin LXIX Fat-Soluble Vitamins and Carotenoids in Human Serum and Round Robin 34 Ascorbic Acid in Human Serum, NIST Interagency/Internal Report (NISTIR) 7880-3

D.L. Duewer, J.M. Brown Thomas, NIST Micronutrients Measurement Quality Assurance Program Summer 2011 Comparability Studies Results for Round Robin LXX Fat-Soluble Vitamins and Carotenoids in Human Serum and Round Robin 35 Ascorbic Acid in Human Serum, NIST Interagency/Internal Report (NISTIR) 7880-2

D.L. Duewer, J.M. Brown Thomas, NIST Micronutrients Measurement Quality Assurance Program Winter 2012 Comparability Studies Results for Round Robin LXXI Fat-Soluble Vitamins and Carotenoids in Human Serum and Round Robin 36 Ascorbic Acid in Human Serum, NIST Interagency/Internal Report (NISTIR) 7880

D.L. Duewer, J.M. Brown Thomas, NIST Micronutrients Measurement Quality Assurance Program Summer 2012 Comparability Studies, NIST Interagency/Internal Report (NISTIR) 7880-1

The NIST MMQAP Vitamin C Round Robin 39 (RR39) 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 your RR39 measurements.

Page	"All-Lab" Report
1	A tabulation of results and summary statistics for total ascorbic acid [TAA] in the RR39 control and test samples. Results and summary statistics are also presented for the test samples calibrated to the results for the control samples

Serum-Based Samples. Two serum controls and four test samples were distributed in RR39.

- 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
- S39:1 an unaugmented material (i.e., the [TAA] content is zero or nearly so), ampouled in 2001, previously distributed in RRs 16, 19, 21, 23, 26, and 29
- S39:2 Ampouled in late 2009, previously distributed in RRs 32, 33, and 35
- S39:3 Ampouled in late 2009, previously distributed in RRs 32, 35, and 36
- S39:4 Ampouled in late 2009, previously distributed in RRs 34 and 36

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 unknowns. The expected [TAA] content and inter-participant standard deviations are estimated with the robust median and adjusted median absolute deviation (eSD) statistics.

2) There is no evidence for any significant change in the [TAA] level for any of the four test materials.

3) The results for two control sera with well-separated [TAA] levels enable calibration of the reported results for the unknowns to the function:

$$[\mathrm{TAA}]_{\mathrm{reportedCS}} = a + b^{*}[\mathrm{TAA}]_{\mathrm{referenceCS}}$$

where $[\mathrm{TAA}]_{\mathrm{reportedCS}}$ are the reported values for the two sera and $[\mathrm{TAA}]_{\mathrm{referenceCS}}$ are the established reference values for these materials. The calibrated values for unknown samples are then given by:

$$[\mathrm{TAA}]_{\mathrm{calibrated}} = ([\mathrm{TAA}]_{\mathrm{reported}} - a) / b \,.$$

Figure 1 displays the eSD as a function of the Median values for the test samples distributed in RR37, RR38, and RR39 for both the "as reported" and "after calibration" results. Calibration to the control materials improves the robust inter-participant estimated coefficient of variation

$$\mathrm{eCV} = 100^{*}\mathrm{eSD/Median}$$

from about 9% to about 6%.

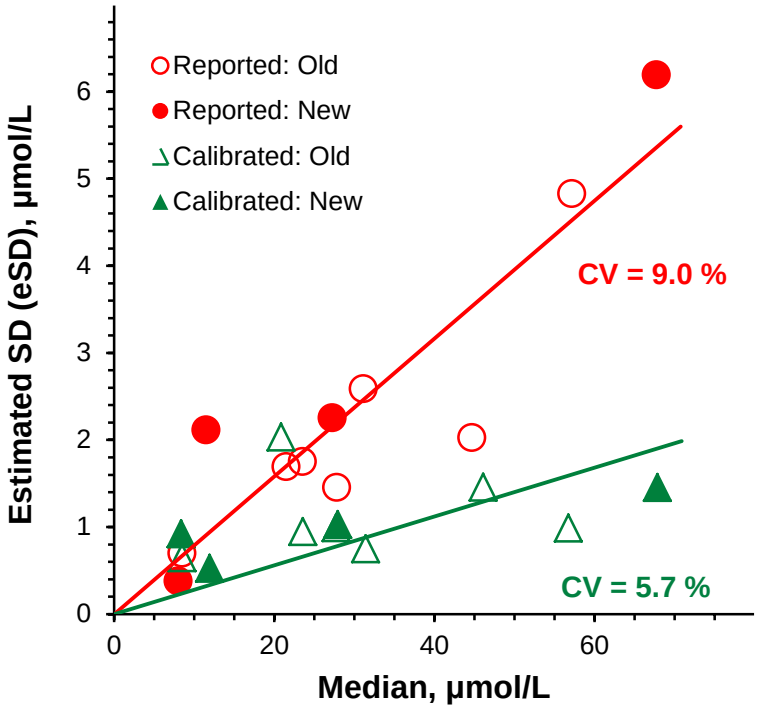


Figure 1: Estimated Standard Deviation as a Function of Median Value

Each open symbol represents the summary statistics for a test sample distributed in RR37 or RR38; each solid symbol represents the statistics for a test sample in RR39.

Appendix G. "All-Lab Report" for RR39

The following two pages are the "All-Lab Report" for RR39 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" 39 - Summer 2013

		Samples						Calibrated Results: [TAA]' = (TAA-a)/b					
		Total Ascorbic Acid ([TAA]), µmol/L						Parameters		[TAA]', µmol/L			
Lab	Date	CS#3	CS#4	S39:1	S39:2	S39:3	S39:4	*b*	*a*	S39:1'	S39:2'	S39:3'	S39:4'
VC-MB	01/08/13	15.5	47.7	8.0	28.4	11.9	67.1	1.05	-0.8	8.4	27.7	12.1	64.5
VC-MC	17/06/13	15.6	46.7	7.8	26.4	11.0	68.4	1.02	-0.2	7.9	26.1	11.0	67.4
VC-MG	01/08/13	14.8	46.1	8.3	28.4	11.1	69.6	1.02	-1.1	9.1	28.8	11.9	69.1
VC-MH	10/07/13	14.9	43.9	8.1	25.4	12.0	64.2	0.95	0.1	8.3	26.6	12.5	67.5
VC-MI	23/09/13	10.8	43.3	8.3	28.2	6.0	72.6	1.06	-5.7	13.2	31.9	11.0	73.7
VC-MJ	31/07/13	21.0	51.4	12.3	33.5	17.3	82.7	0.99	5.6	6.7	28.1	11.8	77.5
VC-MN	30/09/13	14.0	41.0	7.7	24.5	14.7	59.6	0.88	0.3	8.4	27.4	16.4	67.1
VC-NM	16/09/13	12.3	39.5	7.3	23.6	9.2	59.1	0.89	-1.5	9.9	28.2	12.0	68.2
VC-NX	03/12/13	15.0	47.2	8.6	28.7	10.1	71.1	1.05	-1.4	9.4	28.6	10.9	68.9
	N	9	9	9	9	9	9			9	9	9	9
	Average	14.9	45.2	8.5	27.4	11.5	68.2			9.0	28.1	12.2	69.3
	SD	2.8	3.7	1.5	3.0	3.2	7.2			1.8	1.7	1.7	3.9
	Min	10.8	39.5	7.3	23.6	6.0	59.1			6.7	26.1	10.9	64.5
	%25	14.0	43.3	7.8	25.4	10.1	64.2			8.3	27.4	11.0	67.4
	Median	14.9	46.1	8.1	28.2	11.1	68.4			8.4	28.1	11.9	68.2
	%75	15.5	47.2	8.3	28.4	12.0	71.1			9.4	28.6	12.1	69.1
	Max	21.0	51.4	12.3	33.5	17.3	82.7			13.2	31.9	16.4	77.5
	eSD	1.1	3.2	0.4	2.7	1.5	6.2			1.1	1.0	0.9	1.3
	eCV	7	7	5	9	13	9			13	3	8	2

G2

Micronutrients Measurement Quality Assurance Program for Total Ascorbic Acid
"Round Robin" 39 - Summer 2013

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

G3

Appendix H. Representative "Individualized Report" for RR39

Each participant in RR39 received an "Individualized Report" reflecting their reported results. The following two pages are the "Individualized Report" for participant "VC-MB".

Vitamin C "Round Robin" 39 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}
09/25/98	11	S11:1	25.0	25.0	0.5	12.5	0.0	15	9.2	1.7	2.1
02/26/99	12	S12:1	18.0	18.0	0.5	9.0	0.0				
03/03/00	13	S13:1	24.0	24.0	0.5	12.0	0.0				
03/26/01	14	S14:3	19.6	19.6	0.5	9.8	0.0				
09/05/01	15	S15:1	25.8	23.7	0.5	12.4	0.7				
02/08/02	16	S16:1	18.7	18.7	0.5	9.4	0.0				
05/01/03	19	S19:4	19.6	19.6	0.5	9.8	0.0				
03/01/04	20	S20:3	7.8	10.9	1.0	9.3	2.2				
05/25/05	23	S23:4	7.7	7.7	1.0	7.7	0.0				
05/24/06	25	S25:1	8.8	8.8	1.0	8.8	0.0				
06/20/08	29	S29:2	8.3	7.7	1.0	8.0	0.4				
08/05/09	31	S31:3	8.8	8.3	1.0	8.5	0.4				
01/14/11	34	S34:1	7.7	7.7	1.0	7.7	0.0				
08/06/12	37	S37:1	8.8	0.0	1.0	4.4	6.2				
08/01/13	39	S39:1	**8.3**	**7.7**	1.0	8.0	0.4				
09/25/98	11	S11:2	63.0	62.0	0.5	31.3	0.4	13	28.5	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				
05/24/06	25	S25:3	10.8	10.8	1.0	10.8	0.0	3	11.3	0.2	0.5
06/20/08	29	S29:4	10.8	11.4	1.0	11.1	0.4				
08/01/13	39	S39:3	**11.9**	**11.9**	1.0	11.9	0.0				
08/24/93	04	S04:2	2.3	2.3	28.4	64.7	0.8	4	67.0	2.6	3.2
11/26/06	26	S26:3	70.7	71.2	1.0	71.0	0.4				
11/15/07	28	S28:2	61.4	68.7	1.0	65.0	5.1				
08/01/13	39	S39:4	**67.1**	**67.1**	1.0	67.1	0.0				

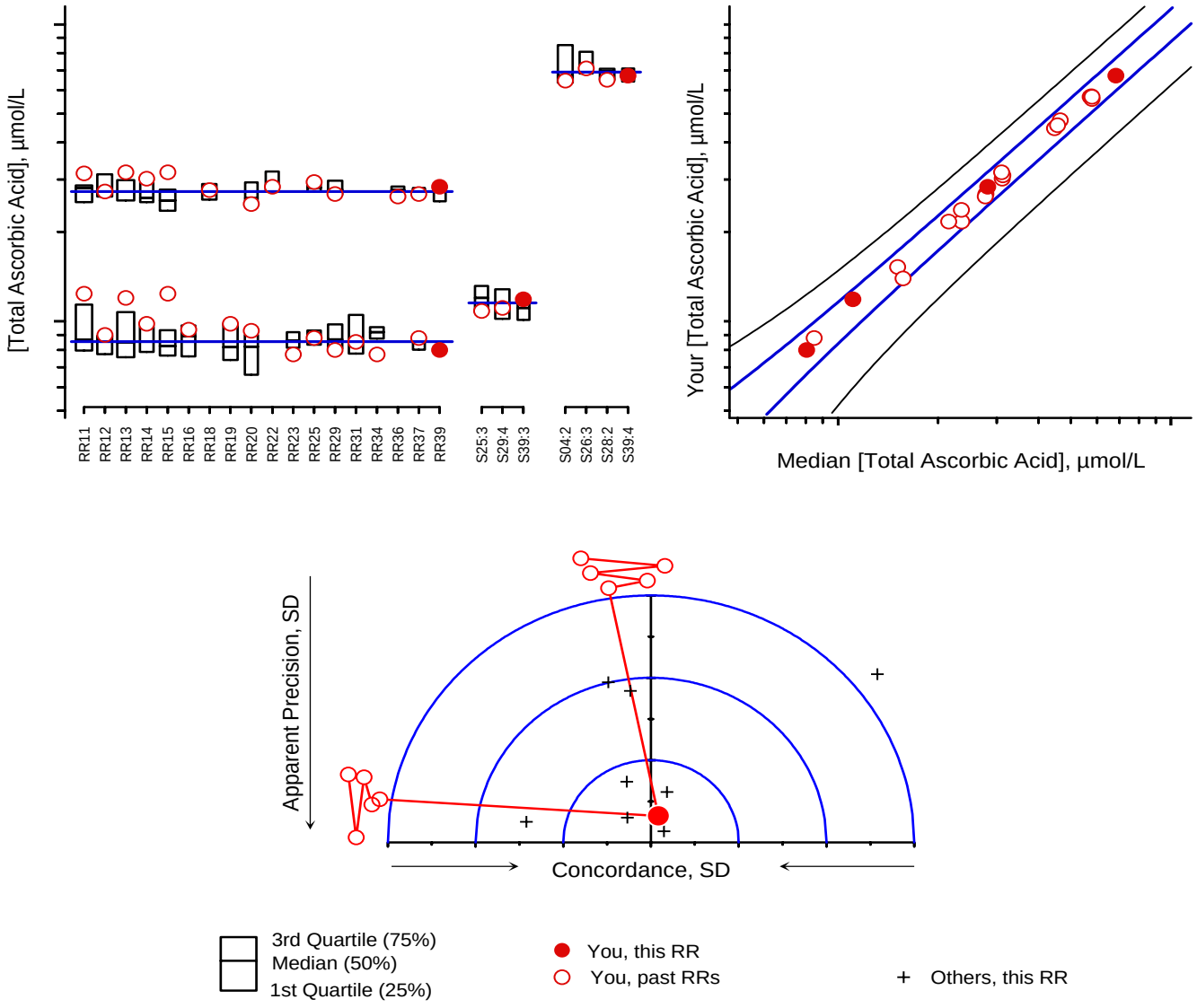
Please check our records against your records. Send corrections and/or updates to...

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" 39 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
S39:1	SRM970 Lv I - prepared 1998; distributed in RRs 11,12,13,14,15,16,19,20,23,25,29,31,34,37
S39:2	SRM970 Lv II - prepared 1998; distributed in RRs 11,12,13,14,15,18,20,22,25,29,36,37
S39:3	VitC #111 - prepared in 1989; distributed in RRs 25,29
S39:4	VitC #112 - prepared in 1993; distributed in RRs 4,26,28