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NIST Micronutrients Measurement Quality Assurance Program Summer 2012 Comparability Studies

Results for Round Robin LXXII Fat-Soluble Vitamins and Carotenoids in Human Serum and Round Robin 37 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

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March, 2013

U.S. Department of Commerce
Rebecca Blank, Acting Secretary

National Institute of Standards and Technology
Patrick D. Gallagher, Under Secretary of Commerce for Standards and Technology and Director

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Abstract

The National Institute of Standards and Technology coordinates the Micronutrients Measurement Quality Assurance Program (MMQAP) for laboratories that measure fat- and water-soluble vitamins and carotenoids in human serum and plasma. This report describes the design of and results for the Summer 2012 MMQAP measurement comparability improvement studies: 1) Round Robin LXXII Fat-Soluble Vitamins and Carotenoids in Human Serum and 2) Round Robin 37 Total Ascorbic Acid in Human Serum. The materials for both studies were shipped to participants in June 2012; participants were requested to provide their measurement results by September 28, 2012.

Keywords

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

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Introduction

Beginning in 1988, the National Institute of Standards and Technology (NIST) has coordinated the Micronutrients Measurement Quality Assurance Program (MMQAP) for laboratories that measure fat- and water-soluble vitamins and carotenoids in human serum and plasma. The MMQAP provides participants with measurement comparability assessment through use of interlaboratory studies, Standard Reference Materials (SRMs) and control materials, and methods development and validation. Serum-based samples with assigned values for the target analytes (retinol, alpha-tocopherol, gamma/beta-tocopherol, *trans*- and total beta-carotene, and total ascorbic acid) and performance-evaluation standards are distributed by NIST to laboratories for analysis.

Participants use the methodology of their choice to determine analyte content in the control and study materials. Participants provide their data to NIST, where it is compiled and evaluated for trueness relative to the NIST value, within-laboratory precision, and concordance within the participant community. NIST provides the participants with a technical summary report concerning their performance for each exercise and suggestions for methods development and refinement. Participants who have concerns regarding their laboratory's performance are encouraged to consult with the MMQAP coordinators.

All MMQAP interlaboratory studies consist of individual units of batch-prepared samples that are distributed to each participant. For historical reasons these studies are referred to as "Round Robins". The MMQAP program and the nature of its studies are described elsewhere. [1,2]

Round Robin LXXII: Fat-Soluble Vitamins and Carotenoids in Human Serum

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin LXXII comparability study (hereafter referred to as RR72) 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 2012. 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 RR72 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 37: Vitamin C in Human Serum

Participants in the MMQAP Vitamin C in Human Serum Round Robin 37 comparability study (hereafter referred to as RR37) received four frozen serum test samples, one frozen control serum, and a solid ascorbic acid control material for analysis. Unless multiple vials were previously requested, participants received one vial of each material. These sample materials were shipped on dry ice to participants in June 2012. The communication materials included in the sample shipment are provided in Appendix E.

The test and control serum materials were prepared by adding equal volumes of 10 % metaphosphoric acid (MPA) to human serum that had been spiked with ascorbic acid. While these samples contain some dehydroascorbic acid, its content is variable. Therefore, the participants report only total ascorbic acid (TAA, ascorbic acid plus dehydroascorbic acid). Participants are also encouraged to prepare calibration solutions from the supplied solid control to enable calibrating their serum measurements to the same reference standard.

The final report delivered to every participant in RR37 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 RR72

The following three items were included in each package shipped to an RR72 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 25, 2012

Dear Colleague:

Enclosed are samples for the second fat-soluble vitamins and carotenoids in serum study Round Robin LXXII (RR72) for the 2012 NIST Micronutrients Measurement Quality Assurance Program. The set of samples (Sera 387- 391) 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 a given serum sample. If a value obtained is below your limit of quantification, please indicate this result on the form by using NQ *(Not Quantified)*. Results are due to NIST by **September 28, 2012**. Results received more than two weeks after the due date may not be included in the summary report for RR72. The feedback report concerning the study will be distributed in November 2012.

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 RR72 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

Participant #: _____ Date: _____

Round Robin LXXII: Human Sera

NIST Micronutrients Measurement Quality Assurance Program

Analyte	387	388	389	390	391	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
28-Sep-2012

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

Participant #: _____ Date: _____

Fat-Soluble Vitamins Round Robin LXXII

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
#387	Lyophilized	Yes	2 mL amber, red cap
#388	Liquid frozen	No	2 mL amber, green cap
#389	Liquid frozen	No	2 mL amber, red cap
#390	Liquid frozen	No	2 mL amber, gold cap
#391	Liquid frozen	No	2 mL amber, green cap

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

1) Date this shipment arrived: _____

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

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

4) Did the 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 RR72

The following two pages are the final report as provided to all participants:

- Cover letter.
- An information sheet that:
  - describes the contents of the “All-Lab” report,
  - describes the content of the “Individualized” report,
  - describes the nature of the test samples and details their previous distributions, if any, and
  - summarizes aspects of the study that we believe may be of interest to the participants.

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

November 30, 2012

Dear Colleague:

Enclosed is the summary report of the results for round robin LXXII (RR72) of the 2012 NIST Micronutrients Measurement Quality Assurance Program (M^2QAP) for the fat-soluble vitamins and carotenoids in human serum. Included in this report are: 1) a summary of data and measurement comparability scores for all laboratories, 2) a detailed graphical analysis of your results; and 3) a graphical summary of your measurement comparability. RR72 (Sera 387 - 391) 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 first set of fat-soluble vitamins and carotenoids in serum interlaboratory exercise (RR73) of the 2013 M^2QAP will be shipped **starting January 22, 2013**. Please contact us immediately if this schedule is problematic for your laboratory.

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 LXXII (RR72) 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	A listing of the analytes reported by only one participant and a legend for the list of results and statistics.
6	The text Comparability Summary ("Score Card") of measurement performance.

Page	Individualized Report
1	Your values, the number of labs reporting values, and our assigned values.
2 to n	"Four Plot" summaries of your current and past measurement performance, one page for each analyte you report that is also reported by at least eight other participants.
n+1	The graphical Comparability Summary (target plot) of measurement performance.

Samples. Five samples were distributed in RR72.

Serum	Description	Prior Distributions
387	Lyophilized, native serum prepared in 1999. Serum #380 is the liquid-frozen partner of this sample.	#270:RR49-3/01, #276:RR50-9/01, #367:RR68-9/10, #377:RR70-9/11
388	Liquid-frozen, native serum prepared in 1999. Serum #377 is the lyophilized partner of this sample.	#267:RR49-9/00, #274:RR50-9/01, #368:RR68-9/10, #380:RR70-9/11
389	Liquid-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
390	Liquid-frozen, native, single-donor serum prepared in 2011. This material was designed to have low contents of α- and β-carotene and lycopene but a high content of β-cryptoxanthin..	#383:RR71
391	Liquid-frozen, native, multi-donor, prepared in 2008	#356:RR65-3/09, #360:RR66-9/09

Results

1) <u>SRM 968e Stability.</u> There was no significant change in the median level or measurement variability of any measurand in the SRM 968e Level I material.

2) <u>Lyophilized vs Liquid-frozen Stability</u>. After 13 years, there is no change in the median level or the measurement variability in any analyte in the lyophilized #387 material or the #388 liquid-frozen material prepared from the same serum pool. The analyte levels in the lyophilized material are slightly lower than in the liquid-frozen material due to reconstituting *with* 1.0 ml water rather than reconstituting *to* a total volume of 1.0 ml.

Appendix C. All-Lab Report for RR72

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

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

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

Round Robin LXXII Laboratory Results

	Total Retinol, µg/mL					trans-Retinol, µg/mL					Retinyl Palmitate, µg/mL					α-Tocopherol, µg/mL					γ/β-Tocopherol, µg/mL				
Lab	387	388	389	390	391	387	388	389	390	391	387	388	389	390	391	387	388	389	390	391	387	388	389	390	391
FSV-BA	0.664	0.711	0.374	0.487	0.774						0.022	0.020	0.016	0.016	0.044	5.89	6.19	6.66	6.72	10.35	2.30	2.44	1.90	1.83	2.56
FSV-BB	0.651	0.664	0.360	0.465	0.806						0.024	0.020	0.009	0.008	0.035	6.20	6.58	6.92	7.34	10.50	2.21	2.30	1.79	1.72	2.40
FSV-BC	0.621	0.672	0.401	0.484	0.752																				
FSV-BD	0.668	0.687	0.366	0.461	0.746											5.60	6.70	7.10	6.50	10.00					
FSV-BE	0.550	0.580	0.310	0.400	0.630											5.60	5.90	6.30	6.40	10.00	2.10	2.20	1.70	1.60	2.30
FSV-BF	0.600	0.760	0.400	0.480	0.790											5.70	6.70	7.00	6.90	10.80					
FSV-BG	0.658	0.700	0.360	0.467	0.768						0.017	0.022	0.014	0.015	0.044	5.85	6.20	6.35	6.45	10.42	2.32	2.45	1.92	1.78	2.50
FSV-BJ	0.650	0.681	0.373	0.454	0.744											5.68	6.01	6.64	6.49	10.06	2.21	2.29	1.85	1.72	2.30
FSV-BK	0.660	0.636	0.362	0.439	0.755											6.10	6.30	6.42	6.59	10.28					
FSV-BL	0.570	0.630	0.340	0.430	0.720											6.00	6.50	6.90	6.90	10.20					
FSV-BM	0.620	0.620	0.360	0.440	0.720											6.70	6.90	7.00	7.40	10.60					
FSV-BN	0.660	0.690	0.370	0.490	0.650											5.80	6.02	6.07	6.50	9.67					
FSV-BO	0.530	0.610	0.300	0.370	0.660											5.47	6.16	6.12	6.16	9.91	2.04	2.34	1.67	1.56	2.35
FSV-BQ	0.700	0.740	0.380	0.510	0.790											7.00	8.00	8.00	8.00	11.00					
FSV-BR	0.660	0.640	0.350	0.430	*na*											6.40	6.70	7.13	6.70	9.90					
FSV-BS	≥0.534	≥0.542	≥0.281	≥0.374	≥0.629	0.534	0.542	0.281	0.374	0.629															
FSV-BU	0.725	0.754	0.391	0.475	0.806											6.93	7.29	7.71	8.49	12.59	2.23	2.27	1.77	1.85	2.44
FSV-BV	0.732	0.731	0.406	0.518	0.722											6.67	6.00	6.16	6.96	9.11	2.30	2.19	1.66	1.75	2.14
FSV-BW	0.630	0.650	0.340	0.450	0.740						0.020	0.019	0.005	0.013	0.053	6.20	6.60	7.20	7.25	10.53	3.94	2.08	1.69	1.55	1.76
FSV-CC	0.638	0.678	0.358	0.448	0.743	0.632	0.676	0.358	0.444	0.737						6.22	6.60	6.98	6.85	10.96					
FSV-CE	0.531	0.670	0.361	0.464	0.676											7.30	5.58	5.77	7.34	11.30					
FSV-CG	0.691	0.699	0.395	0.498	0.790											6.08	6.47	6.90	6.96	10.89	2.60	2.78	2.16	1.96	2.89
FSV-CI	0.654	0.672	0.353	0.433	0.746						0.020	0.022	0.012	0.013	0.048	5.61	5.81	6.29	6.66	9.92	2.05	2.11	1.69	1.67	2.25
FSV-CZ	0.665	0.720	0.390	0.485	0.742											6.38	6.69	7.13	7.28	11.49	2.05	2.17	1.90	1.78	2.18
FSV-DD	0.660	0.690	0.360	0.470	0.700																				
FSV-DV	0.668	0.783	0.392	0.542	0.826											5.10	6.30	5.50	7.20	8.60					
FSV-EE																									
FSV-FK	0.607	0.626	0.335	0.441	0.706											7.30	7.50	8.00	8.10	12.10					
FSV-FZ	0.644	0.657	0.354	0.450	0.746											6.73	6.87	7.57	7.86	11.53	2.68	2.76	2.19	2.04	2.91
N	27	27	27	27	26	2	2	2	2	2	5	5	5	5	5	25	25	25	25	25	13	13	13	13	13
Min	0.530	0.580	0.300	0.370	0.630	0.534	0.542	0.281	0.374	0.629	0.017	0.019	0.005	0.008	0.035	5.10	5.58	5.50	6.16	8.60	2.04	2.08	1.66	1.55	1.76
Median	0.654	0.678	0.361	0.464	0.745	0.583	0.609	0.319	0.409	0.683	0.020	0.020	0.012	0.013	0.044	6.10	6.50	6.90	6.90	10.42	2.23	2.29	1.79	1.75	2.35
Max	0.732	0.783	0.406	0.542	0.826	0.632	0.676	0.358	0.444	0.737	0.024	0.022	0.016	0.016	0.053	7.30	8.00	8.00	8.49	12.59	3.94	2.78	2.19	2.04	2.91
SD	0.024	0.049	0.019	0.034	0.040						0.002	0.002	0.005	0.003	0.006	0.62	0.46	0.71	0.59	0.73	0.20	0.17	0.16	0.12	0.22
CV	4	7	5	7	5						11	9	41	23	13	10	7	10	9	7	9	8	9	7	10
N_{past}	35	37	32	31	30	12	12	7	0	5	10	10	8	5	9	38	39	33	29	32	21	22	19	16	19
$Median_{past}$	0.636	0.665	0.357	0.460	0.740	0.617	0.657	0.347		0.692	0.021	0.024	0.010	0.019	0.045	5.97	6.35	6.78	7.15	10.56	2.21	2.33	1.81	1.75	2.38
SD_{past}	0.048	0.058	0.032	0.022	0.061	0.040	0.048	0.035		0.097	0.009	0.011	0.005	0.012	0.009	0.51	0.62	0.49	0.67	0.80	0.21	0.22	0.16	0.23	0.27
NAV	0.654	0.678	0.361	0.464	0.745						0.020	0.020	0.012	0.013	0.044	6.10	6.50	6.90	6.90	10.42	2.23	2.29	1.79	1.75	2.35
NAU	0.052	0.054	0.030	0.037	0.059						0.011	0.011	0.011	0.011	0.015	0.62	0.56	0.71	0.59	0.81	0.23	0.23	0.19	0.19	0.24

C2

Round Robin LXXII Laboratory Results

	δ-Tocopherol, µg/mL					Total β-Carotene, µg/mL					trans-β-Carotene, µg/mL					Total cis-β-Carotene, µg/mL					Total α-Carotene, µg/mL				
Lab	387	388	389	390	391	387	388	389	390	391	387	388	389	390	391	387	388	389	390	391	387	388	389	390	391
FSV-BA	0.101	0.110	0.094	0.075	0.271	0.459	0.496	0.090	0.043	0.278	0.440	0.475	0.083	0.042	0.262	0.019	0.022	0.007	0.001	0.016	0.016	0.020	0.006	0.010	0.046
FSV-BB	0.109	0.117	0.100	0.059	0.265	0.462	0.496	0.096	0.056	0.290	0.424	0.455	0.080	0.041	0.230	0.019	0.019	0.006	0.003	0.015	0.019	0.018	0.005	0.010	0.046
FSV-BC																									
FSV-BD																									
FSV-BE						0.620	0.580	0.110	0.050	0.340															
FSV-BF						0.534	0.593	0.075	0.037	0.272															
FSV-BG						0.514	0.541	0.089	0.042	0.277											0.017	0.019	0.010	0.012	0.053
FSV-BJ						0.457	0.495	0.094	0.054	0.254											*nq*	*nq*	*nq*	*nq*	0.036
FSV-BK																									
FSV-BL																									
FSV-BM																									
FSV-BN						0.514	0.536	0.086	0.047	0.259											0.039	0.037	0.016	0.019	0.065
FSV-BO						0.473	0.546	0.114	0.066	0.311											0.016	0.017	0.006	0.010	0.042
FSV-BQ																									
FSV-BR																									
FSV-BS						≥0.607	≥0.435	≥0.106	≥0.029	≥0.111	0.607	0.435	0.106	0.029	0.111						0.071	0.061	0.045	0.039	0.052
FSV-BU						0.406	0.403	0.081	0.040	0.228											0.022	0.016	0.007	0.017	0.041
FSV-BV						0.482	0.485	0.088	0.052	0.274											0.023	0.022	0.006	0.014	0.062
FSV-BW						0.505	0.509	0.082	0.039	0.228											0.015	0.016	0.007	0.011	0.039
FSV-CC																									
FSV-CE						0.808	0.465	0.078	0.063	0.336															
FSV-CG	0.188	0.189	0.164	0.255	0.179	0.442	0.469	0.087	0.043	0.269	0.416	0.444	0.082	0.040	0.252	0.026	0.025	*nq*	*nq*	0.017	0.025	0.023	0.009	0.015	0.063
FSV-CI						0.564	0.559	0.099	0.050	0.321											0.027	0.026	0.013	0.020	0.064
FSV-CZ						0.382	0.407	0.072	0.068	0.220															
FSV-DD																									
FSV-DV																									
FSV-EE																									
FSV-FK																									
FSV-FZ																									
N	3	3	3	3	3	15	15	15	15	15	4	4	4	4	4	3	3	2	2	3	11	11	11	11	12
Min	0.101	0.110	0.094	0.059	0.179	0.382	0.403	0.072	0.037	0.220	0.416	0.435	0.080	0.029	0.111	0.019	0.019	0.006	0.001	0.015	0.015	0.016	0.005	0.010	0.036
Median	0.109	0.117	0.100	0.075	0.265	0.482	0.496	0.088	0.050	0.274	0.432	0.450	0.083	0.041	0.241	0.019	0.022	0.006	0.002	0.016	0.022	0.020	0.007	0.014	0.049
Max	0.188	0.189	0.164	0.255	0.271	0.808	0.593	0.114	0.068	0.340	0.607	0.475	0.106	0.042	0.262	0.026	0.025	0.007	0.003	0.017	0.071	0.061	0.045	0.039	0.065
SD						0.047	0.059	0.010	0.010	0.030	0.018	0.015	0.003	0.001	0.023						0.008	0.005	0.003	0.006	0.013
CV						10	12	11	21	11	4	3	3	4	10						36	25	42	42	27
N_{past}	6	6	5	4	5	26	27	22	18	21	10	11	8	5	8	8	8	5	0	6	21	23	16	14	18
$Median_{past}$	0.101	0.109	0.110	0.063	0.261	0.484	0.512	0.091	0.047	0.274	0.436	0.471	0.083	0.044	0.247	0.025	0.024	0.005		0.014	0.019	0.021	0.009	0.012	0.046
SD_{past}	0.040	0.101	0.025	0.043	0.026	0.063	0.067	0.014	0.009	0.034	0.037	0.038	0.006	0.004	0.012	0.007	0.007	0.003		0.002	0.006	0.006	0.004	0.004	0.007
NAV	0.109	0.117	0.100	0.075	0.265	0.482	0.496	0.088	0.050	0.274	0.432	0.450	0.083	0.041	0.241	0.019	0.022			0.016	0.022	0.020	0.007	0.014	0.049
NAU						0.067	0.069	0.016	0.011	0.040											0.008	0.007	0.004	0.006	0.015

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Round Robin LXXII Laboratory Results

	Total Lycopene, µg/mL					trans-Lycopene, µg/mL					Total β-Cryptoxanthin, µg/mL					Total α-Cryptoxanthin, µg/mL				
Lab	387	388	389	390	391	387	388	389	390	391	387	388	389	390	391	387	388	389	390	391
FSV-BA	0.188	0.205	0.202	0.151	0.318	0.123	0.135	0.113	0.080	0.183	0.050	0.060	0.057	0.182	0.076	0.023	0.033	0.022	0.027	0.029
FSV-BB	0.199	0.218	0.199	0.164	0.303	0.114	0.125	0.099	0.072	0.156	0.045	0.048	0.046	0.153	0.054	0.022	0.023	0.016	0.019	0.016
FSV-BC																				
FSV-BD																				
FSV-BE																				
FSV-BF																				
FSV-BG	0.237	0.245	0.222	0.190	0.367	0.138	0.152	0.116	0.086	0.194	0.053	0.056	0.056	0.186	0.068					
FSV-BJ	0.238	0.270	0.225	0.200	0.415						*nq*	*nq*	*nq*	0.107	0.046					
FSV-BK																				
FSV-BL																				
FSV-BM																				
FSV-BN	0.254	0.267	0.230	0.195	0.385						0.049	0.052	0.052	0.164	0.055					
FSV-BO	0.171	0.175	0.174	0.140	0.283						0.048	0.051	0.049	0.164	0.060					
FSV-BQ																				
FSV-BR																				
FSV-BS	0.252	0.186	0.221	0.077	0.140	0.082	0.065	0.088	0.043	0.061	0.080	0.064	0.073	0.082	0.048					
FSV-BU	0.205	0.214	0.194	0.177	0.349						0.060	0.064	0.071	0.189	0.070					
FSV-BV	0.222	0.214	0.216	0.192	0.351						0.044	0.044	0.044	0.188	0.055					
FSV-BW	0.274	0.257	0.216	0.177	0.413															
FSV-CC																				
FSV-CE																				
FSV-CG	0.230	0.240	0.236	0.185	0.390	0.134	0.144	0.121	0.086	0.205	0.063	0.067	0.068	0.201	0.085					
FSV-CI																				
FSV-CZ																				
FSV-DD																				
FSV-DV																				
FSV-EE																				
FSV-FK																				
FSV-FZ																				
N	11	11	11	11	11	5	5	5	5	5	9	9	9	10	10	2	2	2	2	2
Min	0.171	0.175	0.174	0.077	0.140	0.082	0.065	0.088	0.043	0.061	0.044	0.044	0.044	0.082	0.046	0.022	0.023	0.016	0.019	0.016
Median	0.230	0.218	0.216	0.177	0.351	0.123	0.135	0.113	0.080	0.183	0.050	0.056	0.056	0.173	0.058	0.023	0.028	0.019	0.023	0.023
Max	0.274	0.270	0.236	0.200	0.415	0.138	0.152	0.121	0.086	0.205	0.080	0.067	0.073	0.201	0.085	0.023	0.033	0.022	0.027	0.029
SD	0.036	0.040	0.021	0.022	0.058	0.016	0.014	0.011	0.009	0.033	0.006	0.012	0.015	0.023	0.015					
CV	15	18	10	12	16	13	10	10	11	18	12	21	26	13	26					
N_{past}	21	22	17	13	16	10	10	8	6	8	21	22	17	14	17	5	5	4	4	8
$Median_{past}$	0.224	0.233	0.226	0.171	0.376	0.124	0.138	0.116	0.083	0.180	0.047	0.050	0.050	0.163	0.059	0.023	0.027	0.018	0.025	0.020
SD_{past}	0.032	0.037	0.032	0.036	0.046	0.023	0.018	0.018	0.019	0.021	0.011	0.010	0.011	0.048	0.009	0.006	0.005	0.003	0.013	0.009
NAV	0.230	0.218	0.216	0.177	0.351	0.123	0.135	0.113	0.080	0.183	0.050	0.056	0.056	0.173	0.058					
NAU	0.055	0.053	0.052	0.045	0.078	0.021	0.024	0.020	0.014	0.033	0.012	0.013	0.015	0.036	0.015					

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Round Robin LXXII Laboratory Results

	Total Lutein, µg/mL					Total Zeaxanthin, µg/mL					Total Lutein&Zeaxanthin, µg/mL					Coenzyme Q10, µg/mL					Phylloquinone (K1), ng/mL				
Lab	387	388	389	390	391	387	388	389	390	391	387	388	389	390	391	387	388	389	390	391	387	388	389	390	391
FSV-BA											0.121	0.121	0.097	0.113	0.100										
FSV-BB	0.115	0.120	0.098	0.107	0.106	0.054	0.055	0.050	0.066	0.053	0.170	0.175	0.147	0.173	0.160										
FSV-BC																									
FSV-BD																									
FSV-BE																0.930	0.91	0.900	1.16	0.86	2.986	3.365	0.631	0.262	2.021
FSV-BF																									
FSV-BG											0.123	0.131	0.104	0.110	0.100										
FSV-BJ	0.078	0.086	0.080	0.089	0.081											0.760	0.86	0.910	1.04	0.95					
FSV-BK																									
FSV-BL																									
FSV-BM																									
FSV-BN											0.130	0.128	0.159	0.185	0.091										
FSV-BO	0.093	0.100	0.095	0.105	0.084	0.027	0.029	0.015	0.015	0.019	*0.120*	*0.129*	*0.110*	*0.120*	*0.103*	0.553	0.47	0.768	0.94	0.58					
FSV-BQ																									
FSV-BR																									
FSV-BS	0.126	0.110	0.153	0.058	0.051	0.065	0.060	0.053	0.057	0.053	0.191	0.170	0.206	0.115	0.104										
FSV-BU											0.132	0.146	0.139	0.113	0.099	0.720	0.67	0.840	0.92	0.91					
FSV-BV											0.124	0.127	0.109	0.146	0.076										
FSV-BW																0.780	0.97	0.910	1.37	0.81					
FSV-CC																									
FSV-CE																									
FSV-CG											0.146	0.156	0.149	0.172	0.136										
FSV-CI	0.069	0.066	0.066	0.073	0.054	0.026	0.032	0.026	0.031	0.018	*0.095*	*0.098*	*0.092*	*0.104*	*0.072*	0.800	0.83	0.840	1.09	0.81	1.433	1.429	0.263	0.091	0.776
FSV-CZ																1.207	1.28	0.881	1.14	1.08					
FSV-DD																									
FSV-DV																									
FSV-EE																0.823	0.68	1.436	0.88	0.67					
FSV-FK																									
FSV-FZ																									
N	5	5	5	5	5	4	4	4	4	4	10	10	10	10	10	8	8	8	8	8	2	2	2	2	2
Min	0.069	0.066	0.066	0.058	0.051	0.026	0.029	0.015	0.015	0.018	0.095	0.098	0.092	0.104	0.072	0.553	0.47	0.768	0.88	0.58	1.433	1.429	0.263	0.091	0.776
Median	0.093	0.100	0.095	0.089	0.081	0.041	0.044	0.038	0.044	0.036	0.127	0.130	0.125	0.118	0.100	0.790	0.85	0.891	1.07	0.84	2.210	2.397	0.447	0.177	1.399
Max	0.126	0.120	0.153	0.107	0.106	0.065	0.060	0.053	0.066	0.053	0.191	0.175	0.206	0.185	0.160	1.207	1.28	1.436	1.37	1.08	2.986	3.365	0.631	0.262	2.021
SD	0.033	0.021	0.022	0.024	0.038	0.021	0.020	0.020	0.026	0.025	0.010	0.018	0.035	0.016	0.010	0.076	0.22	0.052	0.16	0.14					
CV	36	21	23	27	46	51	45	53	59	71	8	14	28	13	10	10	26	6	15	17					
N_{past}	11	12	9	4	9	10	11	8	0	8	20	21	18	14	17	8	10	8	9	6	0	0	0	0	0
$Median_{past}$	0.086	0.088	0.072	0.097	0.060	0.034	0.036	0.034		0.029	0.122	0.127	0.107	0.131	0.097	0.814	0.80	0.857	1.08	0.92					
SD_{past}	0.020	0.016	0.015	0.028	0.010	0.012	0.013	0.009		0.015	0.022	0.024	0.013	0.041	0.020	0.156	0.16	0.073	0.06	0.15					
NAV	0.093	0.100	0.095	0.089	0.081	0.041	0.044	0.038	0.044	0.036	0.127	0.130	0.125	0.118	0.100	0.790	0.85	0.891	1.07	0.84					
NAU	0.033	0.021	0.022	0.024	0.038						0.026	0.027	0.035	0.024	0.021	0.079	0.22	0.089	0.16	0.14					

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Round Robin LXXII Laboratory Results

Analytes Reported By One Laboratory

Analyte	Lab	387	388	389	390	391
25-hydroxyvitamin D	FSV-BN	0.0021	0.0040	0.0054	0.0063	0.0047
Phytoene	FSV-BS	*nd*	*nd*	*nd*	0.089	0.081
Phytofluene	FSV-BS	0.123	0.119	0.132	0.110	0.116

Term	Legend
N	Number of (non-NIST) quantitative values reported for this analyte
Min	Minimum (non-NIST) quantitative value reported
Median	Median (non-NIST) quantitative value reported
Max	Maximum (non-NIST) quantitative value reported
SD	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)
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.
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 LXXII Laboratory Results

Comparability Summary

Lab	TR	aT	g/bT	bC	aC	TLy	TbX	TLu	L&Z
FSV-BA	1	1	1	1	1	1	1		1
FSV-BB	1	1	1	1	1	1	1	1	2
FSV-BC	1								
FSV-BD	1	1							
FSV-BE	2	1	1	2					
FSV-BF	2	1		2					
FSV-BG	1	1	1	1	1	1	1		1
FSV-BJ	1	1	1	1		1	2	1	
FSV-BK	1	1							
FSV-BL	2	1							
FSV-BM	1	1							
FSV-BN	1	1		1	2	1	1		2
FSV-BO	3	1	1	2	1	1	1	1	1
FSV-BQ	1	2							
FSV-BR	1	1							
FSV-BS	3			3	4	2	2	2	2
FSV-BU	2	3	1	2	1	1	1		1
FSV-BV	2	2	1	1	1	1	1		1
FSV-BW	1	1	4	1	1	1			
FSV-CC	1	1							
FSV-CE	2	2		3					
FSV-CG	1	1	2	1	1	1	2		2
FSV-CI	1	1	1	1	2			2	2
FSV-CZ	1	1	1	2					
FSV-DD	1								
FSV-DV	2	2							
FSV-FK	1	2							
FSV-FZ	1	2	3						
n	28	25	13	16	11	11	10	5	10

	TR	aT	g/bT	bC	aC	TLy	TbX	TLu	L&Z
% 1	68	72	77	56	73	91	70	60	50
% 2	25	24	8	31	18	9	30	40	50
% 3	7	4	8	13	0	0	0	0	0
% 4	0	0	8	0	9	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 RR72

Each participant in RR72 received an "Individualized Report" reflecting their reported results. Each report included a detailed analysis for analytes that were assayed by at least five participants. The following analytes met this criterion in RR72:

- 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 LXXII Report: FSV-BA

Summary

	Serum 387			Serum 388			Serum 389			Serum 390			Serum 391		
Analyte	You	NAV	n	You	NAV	n	You	NAV	n	You	NAV	n	You	NAV	n
Total Retinol	0.664	0.654	27	0.711	0.678	27	0.374	0.361	27	0.487	0.464	27	0.774	0.745	26
Retinyl Palmitate	0.02	0.02	5	0.0	0.0	5	0.0	0.0	5	0.02	0.01	5	0.04	0.04	5
α-Tocopherol	5.89	6.10	25	6.19	6.50	25	6.66	6.90	25	6.72	6.90	25	10.35	10.42	25
γ/β-Tocopherol	2.300	2.235	13	2.438	2.290	13	1.901	1.794	13	1.831	1.750	13	2.561	2.350	13
δ-Tocopherol	0.101	0.109	3	0.110	0.117	3	0.094	0.100	3	0.075	0.075	3	0.271	0.265	3
Total β-Carotene	0.459	0.482	15	0.496	0.496	15	0.090	0.088	15	0.043	0.050	15	0.278	0.274	15
trans-β-Carotene	0.440	0.432	4	0.475	0.450	4	0.083	0.083	4	0.042	0.041	4	0.262	0.241	4
Total cis-β-Carotene	0.019	0.019	3	0.022	0.022	3	0.007		2	0.001		2	0.016	0.016	3
Total α-Carotene	0.016	0.022	11	0.020	0.020	11	0.006	0.007	11	0.010	0.014	11	0.046	0.049	12
Total Lycopene	0.188	0.230	11	0.205	0.218	11	0.202	0.216	11	0.151	0.177	11	0.318	0.351	11
trans-Lycopene	0.123	0.123	5	0.135	0.135	5	0.113	0.113	5	0.080	0.080	5	0.183	0.183	5
Total β-Cryptoxanthin	0.050	0.050	9	0.060	0.056	9	0.057	0.056	9	0.182	0.173	10	0.076	0.058	10
Total α-Cryptoxanthin	0.023		2	0.033		2	0.022		2	0.027		2	0.029		2
Total Lutein&Zeaxanthin	0.121	0.127	10	0.121	0.130	10	0.097	0.125	10	0.113	0.118	10	0.100	0.100	10

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

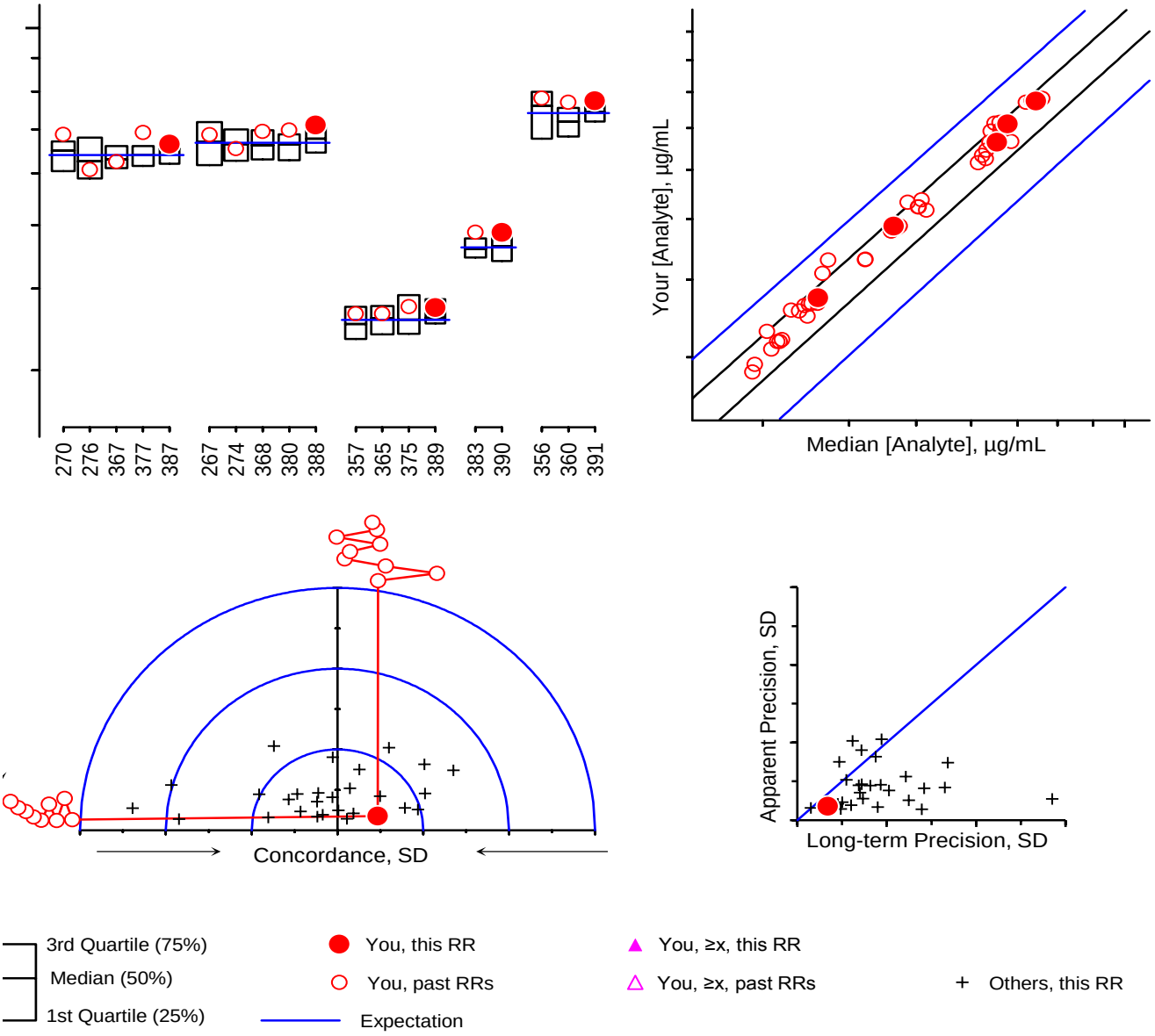
Please check our records against your records. Send corrections and/or updates to...

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 LXXII Report: FSV-BA

Total Retinol, µg/mL

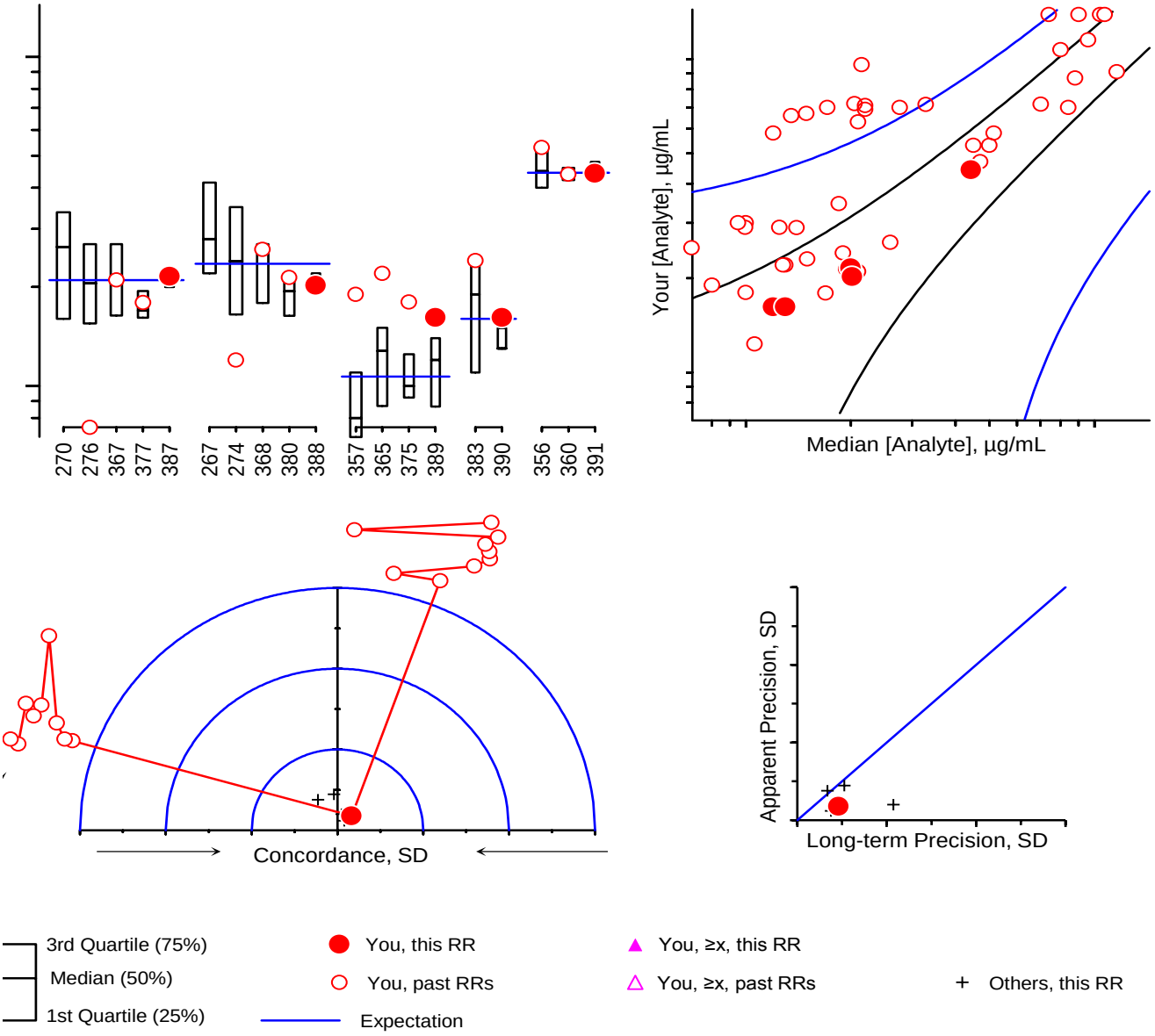


For details of the construction and interpretation of these plots, see:
Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

erum	Comments	History
387	Lyophilized, same native pool as #388	49#270, 50#276, 68#367, 70#377
388	Fresh-frozen, same native pool as #387	49#267, 50#274, 68#368, 70#380
389	Fresh-frozen, native, multi-donor: SRM 968e I	66#357, 67#365,69#375
390	Fresh-frozen, native, single-donor	71#383
391	Fresh-frozen, native, multi-donor	65#356, 66#360

Individualized RR LXXII Report: FSV-BA

Retinyl Palmitate, µg/mL



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

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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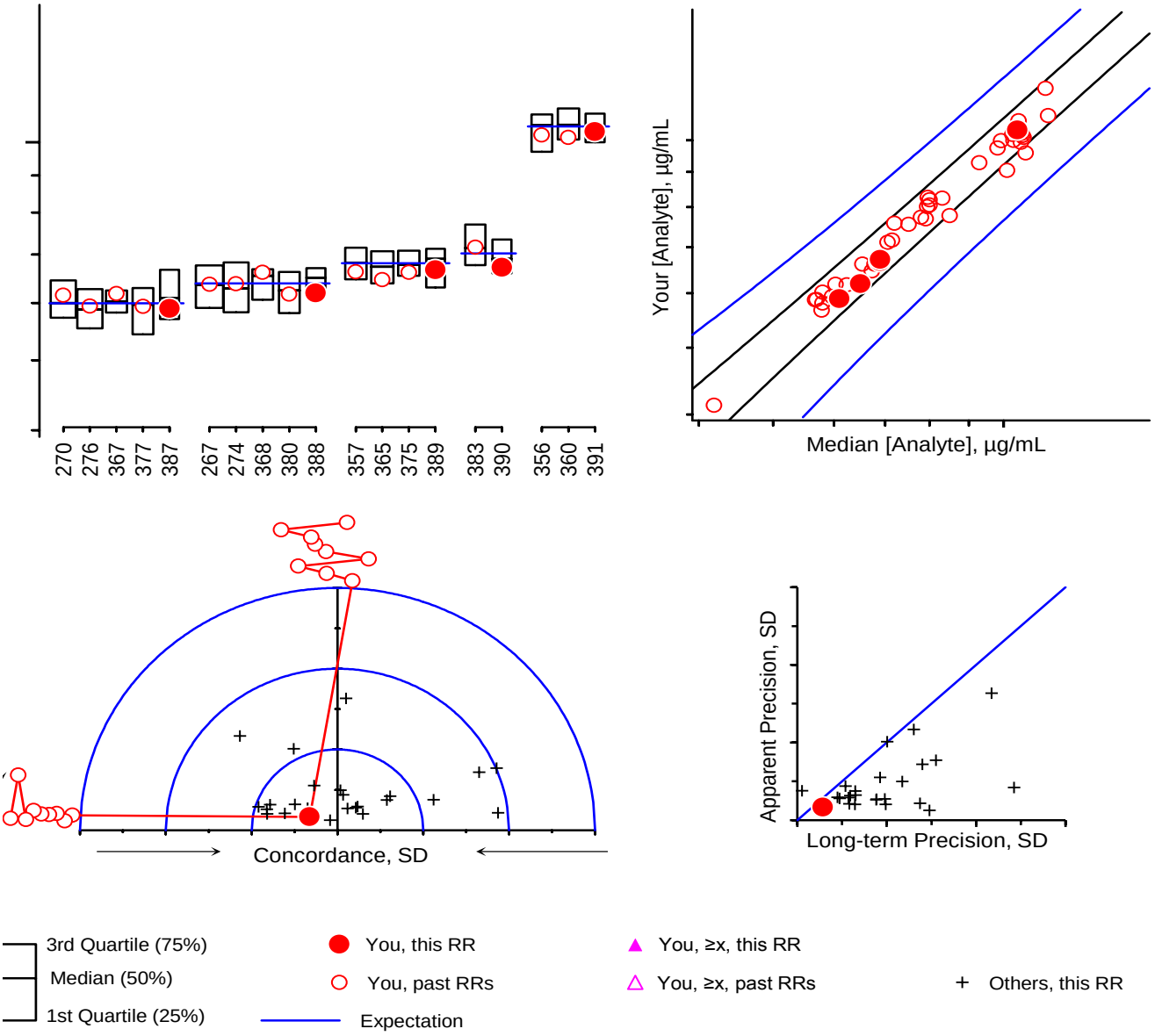
391	Fresh-frozen, native, multi-donor	65#356, 66#360

Individualized RR LXXII Report: FSV-BA

α-Tocopherol, µg/mL



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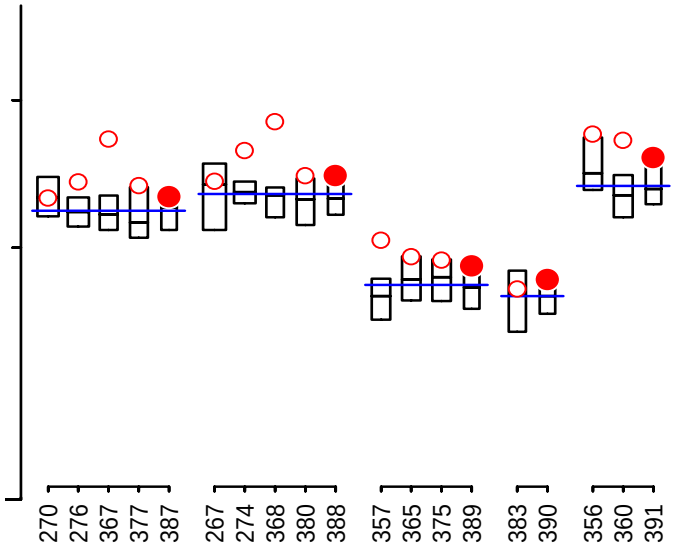
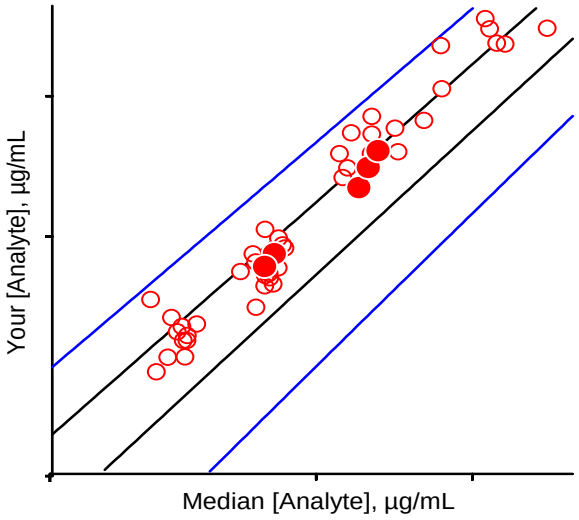
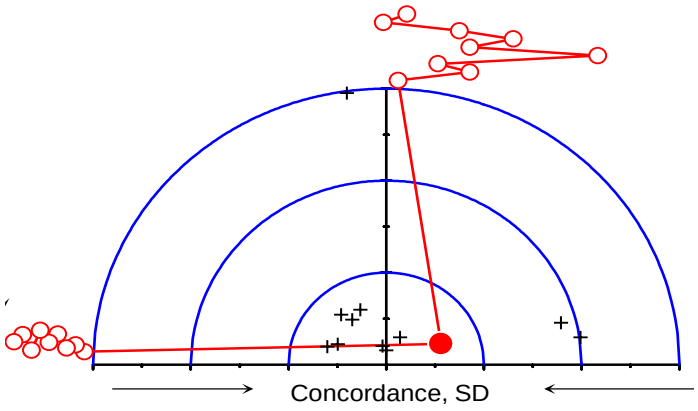
390	Fresh-frozen, native, single-donor	71#383
391	Fresh-frozen, native, multi-donor	65#356, 66#360

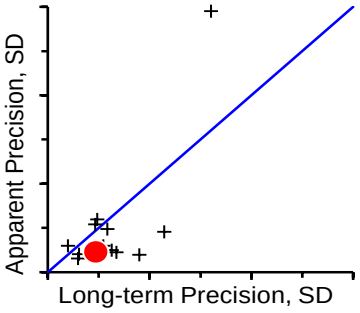
Individualized RR LXXII Report: FSV-BA

γ/β-Tocopherol, µg/mL









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

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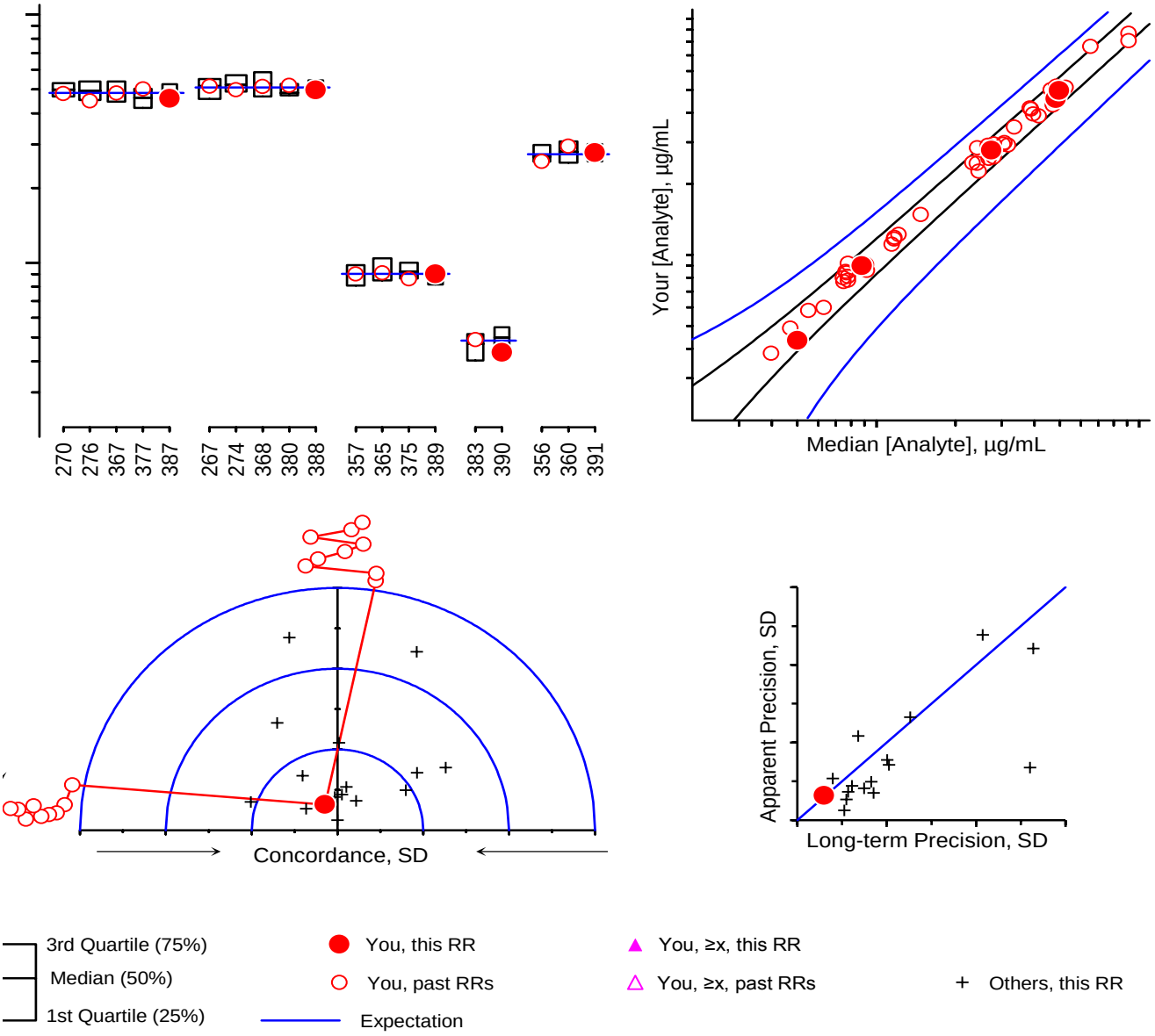
390	Fresh-frozen, native, single-donor	71#383
391	Fresh-frozen, native, multi-donor	65#356, 66#360

Individualized RR LXXII Report: FSV-BA

Total β-Carotene, µg/mL



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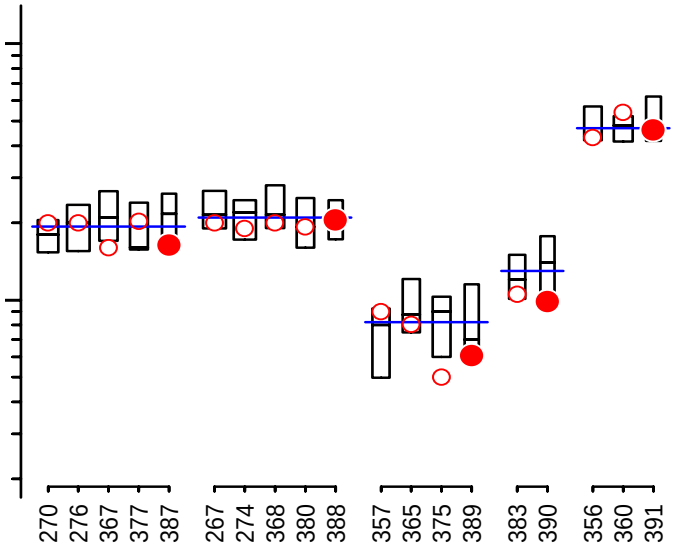
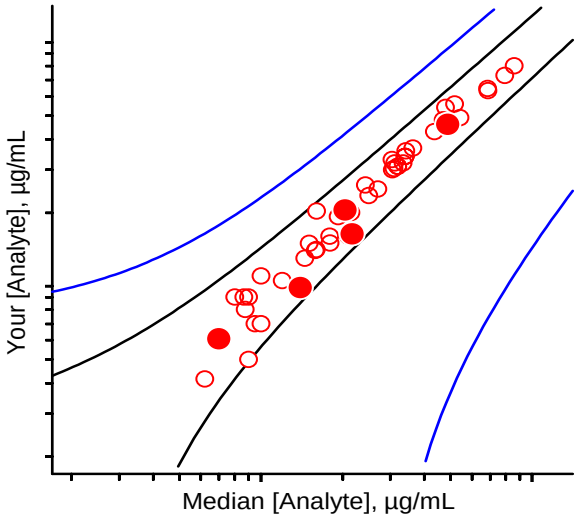
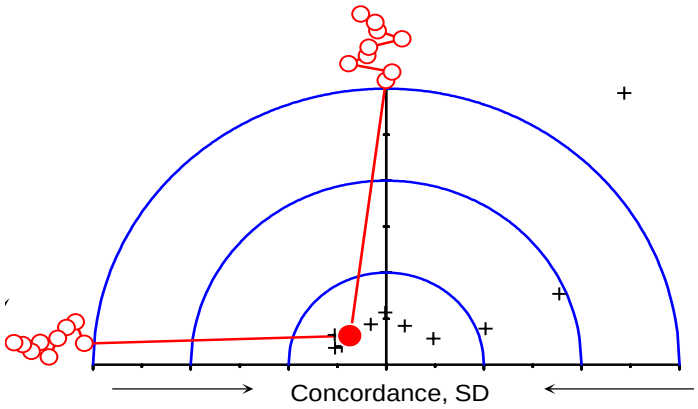
390	Fresh-frozen, native, single-donor	71#383
391	Fresh-frozen, native, multi-donor	65#356, 66#360

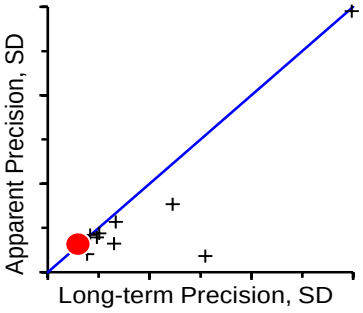
Individualized RR LXXII Report: FSV-BA

Total α-Carotene, µg/mL









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

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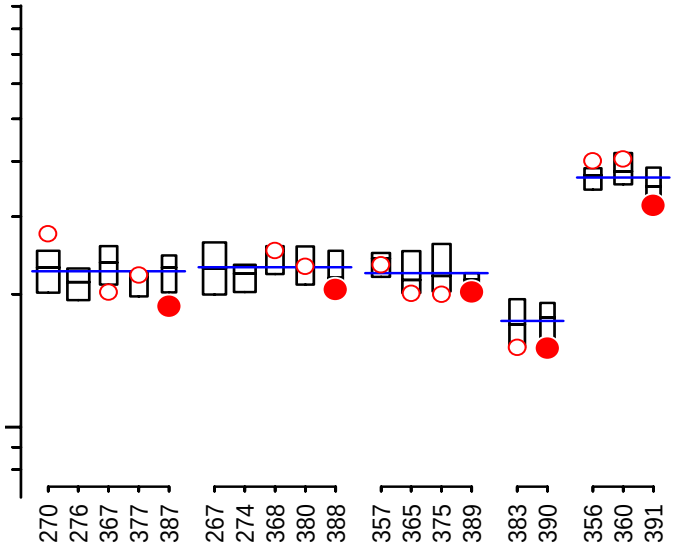
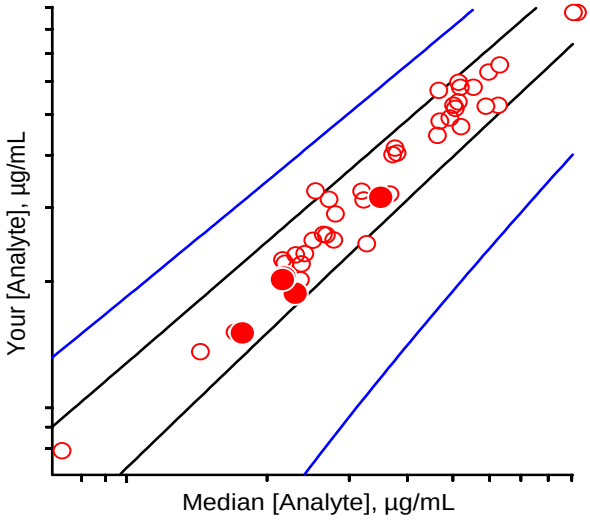
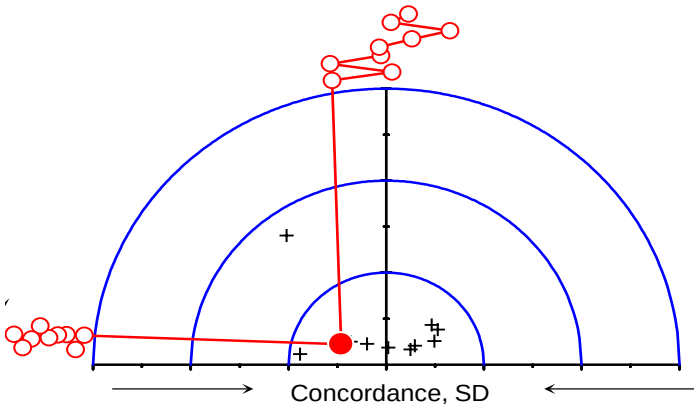
390	Fresh-frozen, native, single-donor	71#383
391	Fresh-frozen, native, multi-donor	65#356, 66#360

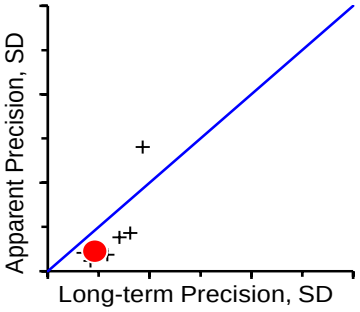
Individualized RR LXXII Report: FSV-BA

Total Lycopene, µg/mL









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

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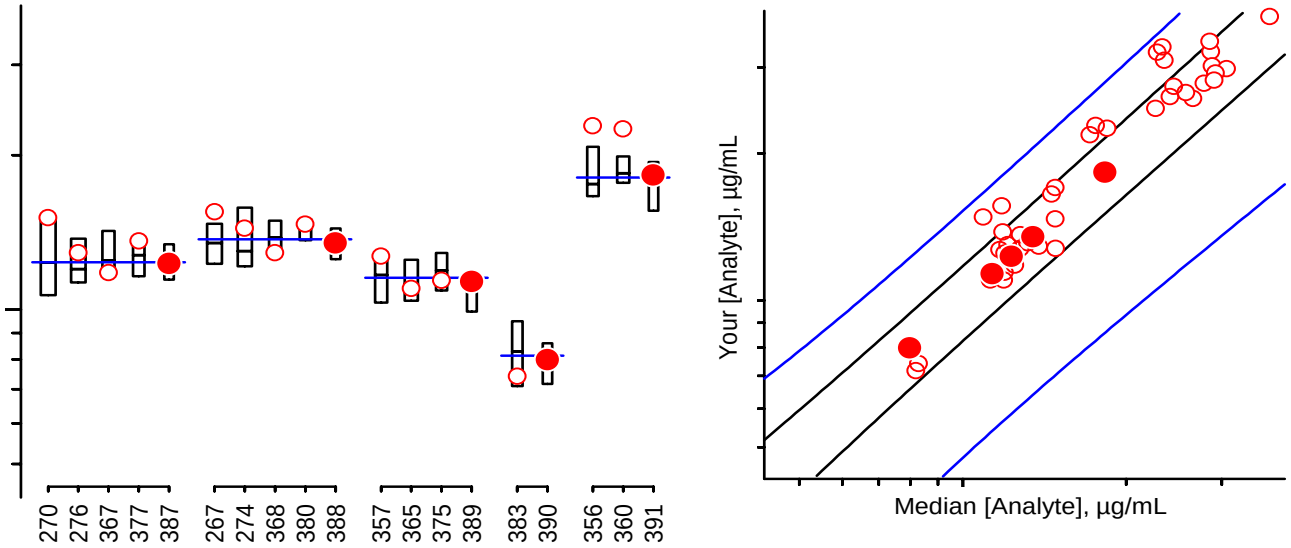
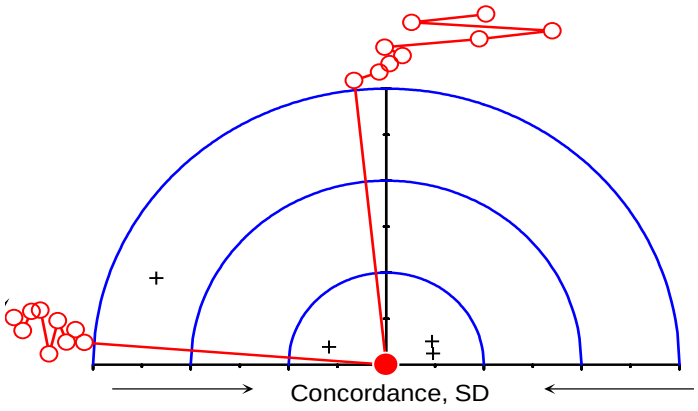
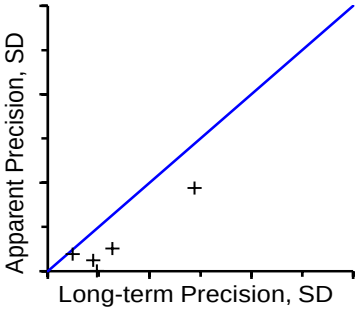
391	Fresh-frozen, native, multi-donor	65#356, 66#360

Individualized RR LXXII Report: FSV-BA

trans-Lycopene, µg/mL







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

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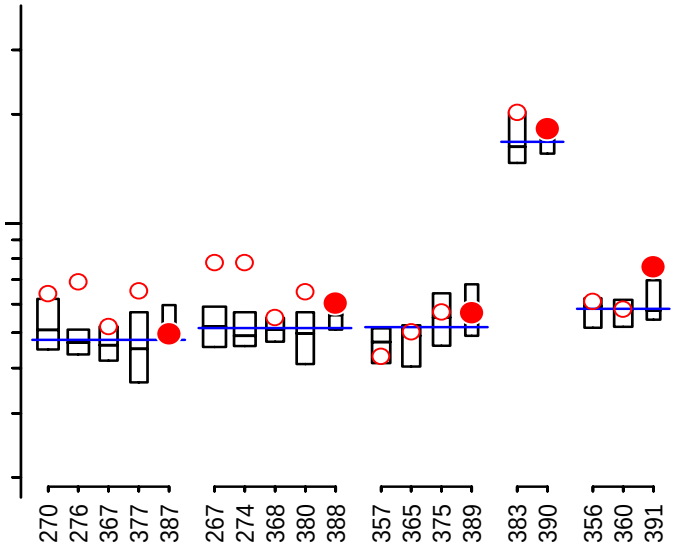
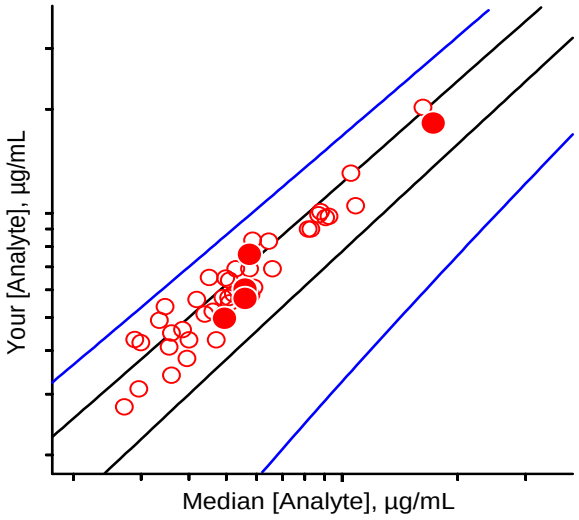
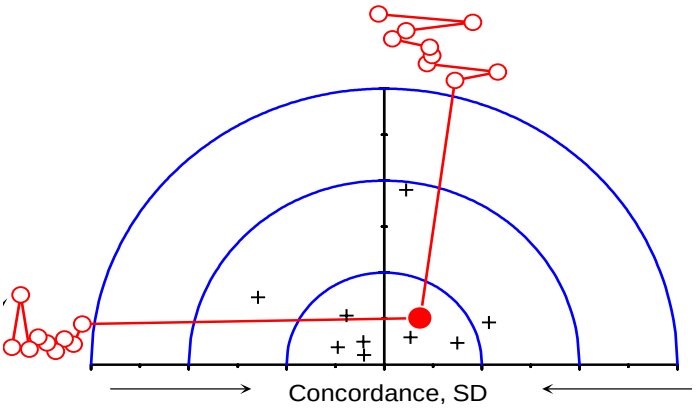
390	Fresh-frozen, native, single-donor	71#383
391	Fresh-frozen, native, multi-donor	65#356, 66#360

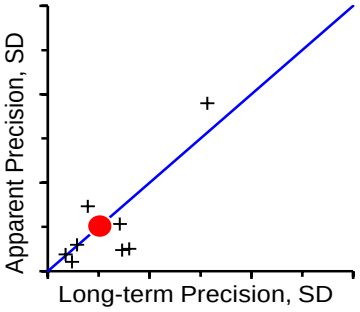
Individualized RR LXXII Report: FSV-BA

Total β-Cryptoxanthin, µg/mL









3rd Quartile (75%)
Median (50%)
1st Quartile (25%)
You, this RR
You, past RRs
Expectation
You, ≥x, this RR
You, ≥x, past RRs
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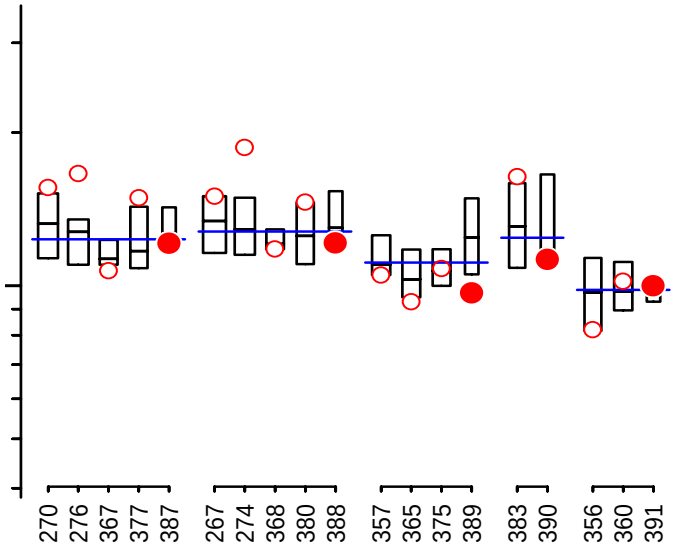
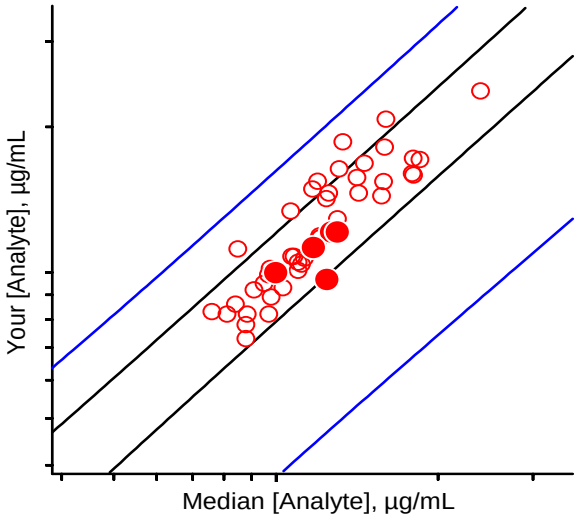
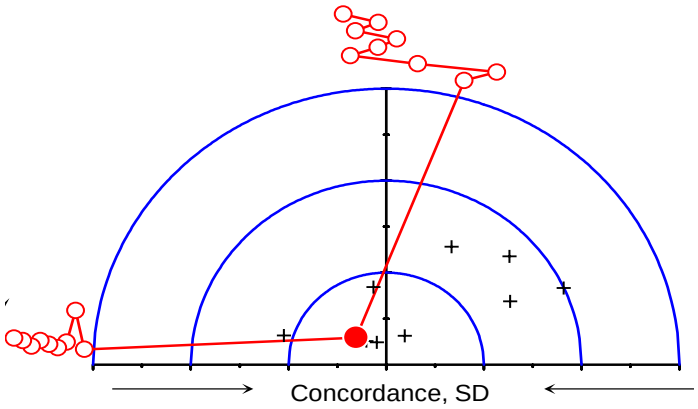
390	Fresh-frozen, native, single-donor	71#383
391	Fresh-frozen, native, multi-donor	65#356, 66#360

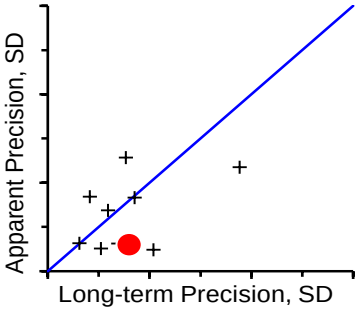
Individualized RR LXXII Report: FSV-BA

Total Lutein&Zeaxanthin, µg/mL









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

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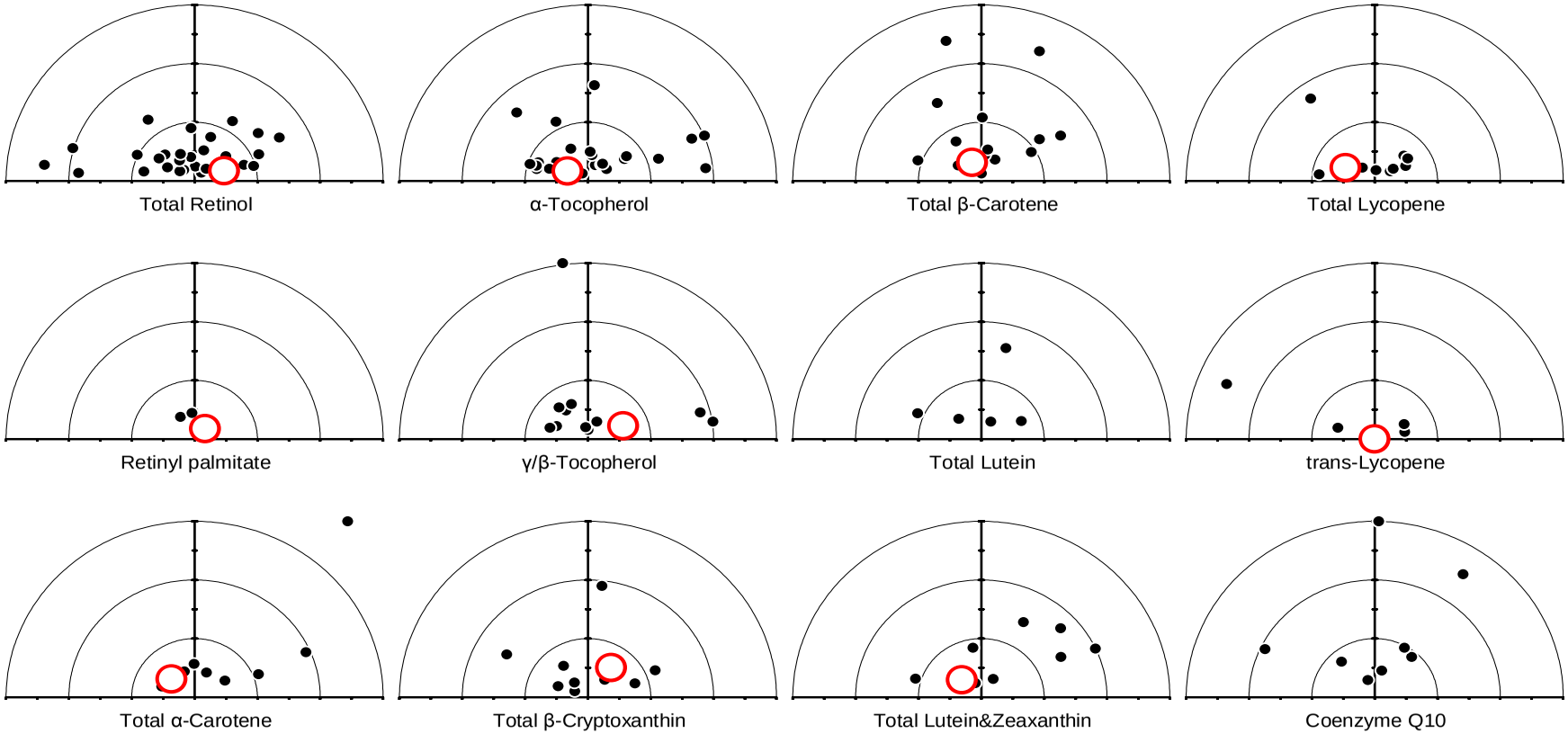
390	Fresh-frozen, native, single-donor	71#383
391	Fresh-frozen, native, multi-donor	65#356, 66#360

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



D13

Appendix E. Shipping Package Inserts for RR37

The following five items were included in each package shipped to an RR37 participant:

- Cover letter
- Protocol for Preparation and Analysis of the Ascorbic Acid Solid Control Material
- Preparation and Validation of Ascorbic Acid Solid Control Material Datasheet
- Analysis of Control Materials and Test Samples Datasheet
- Packing List and Shipment Receipt Confirmation Form

The cover letter, preparation protocol, and the two datasheets were enclosed in a sealed waterproof bag along with the samples themselves. The packing list was placed at the top of the shipping box, between the cardboard covering and the foam insulation.

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

June 25, 2012

Dear Colleague:

The samples within this package constitute Vitamin C Round Robin 37 (RR37) of the 2012 Micronutrients Measurement Quality Assurance Program. RR37 consists of four vials of frozen serum *test samples* (#371, #372 #373, and #374), one vial each of two frozen *control serum samples* (CS #3 and CS #4), and one vial of ascorbic acid *solid control material* (Control). Please follow the attached protocols when you prepare and analyze these samples. If you cannot prepare the *solid control* solutions gravimetrically, please prepare equivalent solutions volumetrically and report the exact volumes used. (Routine 0.5 g gravimetric measurements are generally 10-fold more accurate than routine 0.5 mL volumetric measurements.)

Please use the *control 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 either of your result is 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.

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 report your results (using the attached form) for RR37 by e-mail to david.duewer@nist.gov or fax to 301-977-0685 by **September 28, 2012**. 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 B Thomas

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

Enclosures: Protocols, Preparation and Analysis of Control Materials and Analysis of Test Samples
RR37 Report Form for Ascorbic Acid Solid Control Material Preparation
RR37 Report Form for Control Material and Test Sample Analyses

Micronutrient Measurement Quality Assurance Program for
Vitamin C

Please Read Through Completely BEFORE Analyzing Samples

Protocol for Preparation and Analysis of the Ascorbic Acid Solid Control Material

The *ascorbic acid solid control material* (in the amber vial) should be prepared and used in the following manner:

1) Prepare at least 500 mL of 5% mass fraction metaphosphoric acid (MPA) in distilled water. This solution will be referred to as the "Diluent" below.

2) Weigh 0.20 to 0.22 g of the ascorbic acid solid control material to 0.0001 g (if possible), dissolve it in the Diluent in a 100 mL volumetric flask, and dilute with the Diluent to the 100 mL mark. Weigh the amount of Diluent added to 0.1 g. Record the weights. The resulting material will be referred to as the "Stock Solution" below.

3) Prepare three dilute solutions of the Stock Solution as follows:

   Dilute Solution 1: Weigh 0.500 mL of the Stock Solution to 0.0001 g into a 100 mL volumetric flask; dilute with Diluent to the 100 mL mark. Record the weight.

   Dilute Solution 2: Weigh 0.250 mL of the Stock Solution to 0.0001 g into a 100 mL volumetric flask; dilute with Diluent to the 100 mL mark. Record the weight.

   Dilute Solution 3: Weigh 0.125 mL of the Stock Solution to 0.0001 g into a 100 mL volumetric flask; dilute with Diluent to the 100 mL mark. Record the weight.

4) Calculate and record the total ascorbic acid concentrations, [TAA], in these Dilute Solutions. If you follow the above gravimetric preparation directions, the [TAA] in μmol/L is calculated:

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

   For example, if you prepared the Stock Solution with 0.2000 g of solid ascorbic acid and 103.0 g of Diluent, then 0.5 mL of the Stock Solution should weigh (0.2+103)/200 = 0.52 g and $[\mathrm{TAA}]_{DS1}$ = (0.52 g)(0.2 g)·(56785 μmol/g·L)/(0.2 + 103 g) = 57.2 μmol/L. Likewise, 0.25 mL of the Stock Solution should weigh 0.26 g and $[\mathrm{TAA}]_{DS2}$ = 29.4 μmol/L and 0.125 mL should weigh 0.13 g and $[\mathrm{TAA}]_{DS3}$ = 14.2 μmol/L.

5) Measure the ultraviolet absorbance spectrum of Dilute Solution 1 against the Diluent as the blank using paired 1 cm path length cuvettes. Record the absorbance at 242, 243, 244, and 245 nm. Record the maximum absorbance (A_{max}) within this region. Record the wavelength (λ_{max}) at which this maximum occurs.

   The extinction coefficient ($E^{1\%}$) of ascorbic acid at λ_{max} (using a cell with a 1 cm path length) of Dilute Solution #1 can be calculated:

$$E^{1\%}\left(\frac{dL}{g \cdot cm}\right) = \frac{(A_{max}) \cdot ((g\ AA\ in\ Stock\ Solution) + (g\ Diluent\ in\ Stock\ Solution))}{(g\ Stock\ Solution\ in\ Dilute\ Solution\ 1) \cdot (g\ AA\ in\ Stock\ Solution)}$$

If your spectrophotometer is properly calibrated, λ_{max} should be between 243 and 244 nm and $E^{1\%}$ should be 550 ± 30 dL/g·cm. If they are not, you should recalibrate the wavelength and/or absorbance axes of your spectrophotometer and repeat the measurements.

6) Measure and record the concentration of total ascorbic acid in all three dilute solutions and in the 5% MPA Diluent in duplicate using *exactly* the same method that you will use for the serum control materials and test samples, including any enzymatic treatment. We recommend that you analyze these solutions in the following order: Diluent, Dilute Solution 1, Dilute Solution 2, Dilute Solution 3, Dilute Solution 3, Dilute Solution 2, Dilute Solution 1, Diluent.
   a) Compare the values of the duplicate measurements. *Are you satisfied that your measurement precision is adequate?*
   b) Compare the measured with the calculated [TAA] values. This is most conveniently done by plotting the measured values on the y-axis of a scatterplot against the calculated values on the x-axis. The line through the four {calculated, measured} data pairs should go through the origin with a slope of 1.0. *Are you satisfied with the agreement between the measured and calculated values?*

   Do **not** analyze the serum control materials or test samples until you are satisfied that your system is performing properly!

Once you have confirmed that your system is properly calibrated, analyze the serum controls CS #3 and CS #4 (see protocol below). The target range for CS #3 is **(15.5 ±1.6; 13.9 to 17.1) µmol/L** and the target range for CS #4 is **(46.1 ±4.6; 41.5 to 50.7) µmol/L**.

If either of your measured values is not within its target range, please review your sample preparation procedure and whether you followed *exactly* the same measurement protocol the solutions prepared from the solid control material as you used for these serum controls. If the protocols differ, please repeat from Step 6 using the proper protocol. If the proper protocol was used, your measurement system may not be suitable for MPA-preserved samples; please contact us at 301-975-3120 or jbthomas@nist.gov.

Do **not** analyze the test samples until you are satisfied that your system is performing properly and is suitable for the analysis of MPA-preserved serum!

Protocol for Analysis of the Serum Control Materials and Test Samples

The *serum control material* and *test samples* are in sealed ampoules. They were prepared by adding equal volumes of 10% MPA to spiked human serum. We have checked the samples for stability and homogeneity. Only the total ascorbic acid is stable. While these samples contain some dehydroascorbic acid, its content is variable. Therefore, only total ascorbic acid should be reported. The *serum control material* and *test samples* should be defrosted by warming at 20 °C for not more than 10 min otherwise some irreversible degradation may occur.

Each *serum test sample* contains between 0.0 and 80.0 µmol of total ascorbic acid/L of solution. The total ascorbic acid in each ampoule should be measured in duplicate. Please report your results in µmol/(L of the sample solution) rather than µmol/(L of serum NIST used to prepare the sample).

Participant #: _____ Date: _____

Vitamin C Round Robin 37

NIST Micronutrient Measurement Quality Assurance Program

Preparation and Validation of Ascorbic Acid Solid Control Material

STOCK SOLUTION

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

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

DILUTE SOLUTION 1

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

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

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

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

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

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

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

Wavelength of maximum absorbance..... _____ nm

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

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

DILUTE SOLUTION 2

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

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

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

DILUTE SOLUTION 3

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

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

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

Please return by **September 28, 2012**

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 37

NIST Micronutrient Measurement Quality Assurance Program

Analysis of Control Materials and Test Samples

Sample	Replicate 1	Replicate 2	Units
Dilute Solution 1			µmol/L of Dilute Solution
Dilute Solution 2			µmol/L of Dilute Solution
Dilute Solution 3			µmol/L of Dilute Solution
5% MPA Diluent			µmol/L of Diluent
CS # 3			µmol/L of Sample *Target: (15.5 ±1.6) µmol/L*
CS # 4			µmol/L of Sample *Target: (46.1 ±4.6) µmol/L*
Serum Test Sample #371			µmol/L of Sample
Serum Test Sample #372			µmol/L of Sample
Serum Test Sample #373			µmol/L of Sample
Serum Test Sample #374			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 28, 2012**

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 37

NIST Micronutrients Measurement Quality Assurance Program

Packing List and Shipment Receipt Confirmation Form

This box contains one vial each of the following **seven** VitC M^2QAP samples:

Label	Form
VitC #371	Liquid frozen (1:1 serum:10% MPA)
VitC #372	Liquid frozen (1:1 serum:10% MPA)
VitC #373	Liquid frozen (1:1 serum:10% MPA)
VitC #374	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)
Control	Solid AA

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

1) Date this shipment arrived: _____

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

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

4) Did the samples arrive frozen? Yes | No

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

6) When do you anticipate analyzing these samples? _____

Your prompt return of this information is appreciated.

The M^2QAP Gang

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

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

Appendix F. Final Report for RR37

The following five pages are the final report as provided to all participants:

- Cover letter.
- An information sheet that:
  - describes the contents of the “All-Lab” report,
  - describes the content of the “Individualized” report,
  - describes the nature of the test samples and details their previous distributions, if any, and
  - summarizes aspects of the study that we believe may be of interest to the participants.

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

December 7, 2012

Dear Colleague:

Enclosed is the summary report of the results for Round Robin 37 (RR37) 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.

RR37 consisted of four test samples (#371, #372,, #373, and #374), 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 first vitamin C round robin (RR38) of the 2013 MMQAP will be shipped **starting January 22, 2013.** Please contact us immediately if this schedule is problematic for your laboratory.

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
Analytical Chemistry Division
Material Measurement Laboratory

David L. Duewer, Ph.D.
Research Chemometrician
Analytical Chemistry Division
Material Measurement Laboratory

Enclosures

cc: L. C. Sander

The NIST MMQAP Vitamin C Round Robin 37 (RR37) report consists of:

Page	"Individualized" Report
1	Summary of your reported values for the nominal 55 mmol/L solution you prepared from the ascorbic acid solid control sample, the serum control sample, and the four serum test samples.
2	Graphical summary of your RR37 sample measurements.

Page	"All-Lab" Report
1	A tabulation of results and summary statistics for total ascorbic acid [TAA] in the RR37 samples and control/calibration solutions.

Serum-Based Samples. Two serum controls and four test samples were distributed in RR37.

CS#3	a (13.9 to 17.1) µmol/L material ampouled in late 2009
CS#4	a (41.5 to 50.7) µmol/L material ampouled in late 2009
S37:1	SRM 970 level 1, ampouled in mid-1998, previously distributed as an "Unknown" in RRs 11 to 16, 19, 20 23, 25, 29, 31, and 34
S37:2	Ampouled in late 2001, previously distributed in RRs 17, 18, 20, 22, 23, 27, and 31
S37:3	SRM 970 level 2, ampouled in mid-1998, previously distributed as an "Unknown" in RRs 11 to 15, 18, 20 22, 25, 29, and 36
S37:4	Ampouled in late 2001, previously distributed in RRs 16, 17, 20, 21, 23, 27, and 30

Results.

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

2) The Measured = a+b*Gravimetric calibration parameters for the control/calibration solutions (columns 10 to 13 of the All-Lab Report) indicate that the measurement systems for all participants are linear (R^2 close to 1 and the root-mean-square (RMS) residual close to 0.0) and well calibrated (intercepts close to 0 and slopes close to 1).

3) The Measured = p+q*Median regression parameters for samples S37:1 to S37:4 (columns 23 to 26 of the All-Lab Report) confirm the linearity of all measurement systems (R^2 close to 1 and RMS close to 0.0). However, the intercepts and slopes continue to indicate that there are systematic differences in the response of the various measurement procedures to the TAA in the 5% aqueous metaphosphoric acid (MPA) of the test samples.

4) The variability of the measured values after calibration to the control solutions (the final 6 columns of the All-Lab Report) continues to be as great or greater than that of the measured values themselves. The primary source of the systematic differences in response of the measurement procedures appears to be the presence of serum components in the sample matrix, not the MPA preservative. On this basis, we intend to drop the standard solution arm of the MMQAP Vitamin C Round Robin studies beginning with RR38.

Appendix G. "All-Lab Report" for RR37

The following single page is the "All-Lab Report" as provided to all participants, with two exceptions:

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

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

Micronutrients Measurement Quality Assurance Program for Total Ascorbic Acid
"Round Robin" 37 - Fall 2012

		Control / Calibration Samples											MPA	Dilute Solution 1			Samples																	
		Grav, µmol/L			Measured, µmol/L				Measured = a + b*Grav				Density	Spectrophotometry			Measured, µmol/L						Measured = p+q*Median				Calibrated, µmol/L					
Lab	Date	Dil:1	Dil:2	Dil:3	Dil:1	Dil:2	Dil:3	MPA	Inter	Slope	R^2	RMS	g/mL	λ_{max}	A_{max}	$E^{1\%}$	CS#3	CS#4	S37:1	S37:2	S37:3	S37:4	Inter	Slope	R^2	RMS	CS#3	CS#4	S37:1	S37:2	S37:3	S37:4
VC-MA	25/09/12	57.8	28.3	14.8	60.0	29.9	15.0	0.0	-0.03	1.04	1.000	0.4	1.036	243.	0.5510	541.6	16.5	49.2	8.0	20.5	27.8	44.0	-0.48	1.00	0.999	0.5	15.9	47.2	7.7	19.7	26.7	42.2
VC-MB	06/08/12	60.7	30.3	14.9	60.1	31.2	14.5	0.0	0.11	0.99	0.999	0.8	1.030	243.5	0.5990	560.3	12.6	46.5	8.8	21.7	26.8	44.7	0.20	0.99	0.999	0.7	12.6	46.6	8.7	21.7	26.9	44.8
VC-MC	20/08/12	60.6	30.6	15.2	61.7	31.3	15.8	0.0	0.15	1.02	1.000	0.2	1.024	244.	0.5952	558.0	16.0	48.4	8.6	22.6	28.2	48.9	-1.36	1.11	0.997	1.1	15.6	47.5	8.3	22.1	27.6	47.9
VC-MG	15/08/12	61.2	31.8	16.8	63.7	32.1	16.8	0.0	-0.43	1.04	1.000	0.6	1.026	243.6	0.6140	569.4	15.1	47.5	8.2	22.2	28.1	48.2	-1.50	1.10	0.998	0.8	14.9	46.0	8.3	21.8	27.4	46.7
VC-MH	12/09/12	60.1	30.2	14.9	59.7	29.4	14.7	0.0	-0.19	0.99	1.000	0.4	1.031	243.3	0.5933	560.8	14.6	45.7	7.4	19.7	25.4	44.7	-2.13	1.03	0.996	1.1	14.8	46.2	7.6	20.0	25.8	45.2
VC-MI	27/09/12	55.3	27.7	13.8	53.9	23.4	13.5	0.0	-0.69	0.97	0.994	2.2	1.031				15.2	43.6	9.1	20.2	27.8	44.9	-0.07	1.00	0.997	1.0	16.5	45.8	10.1	21.6	29.5	47.2
VC-MJ	29/08/12	55.1	28.1	15.1	55.5	26.8	13.4	0.7	-0.61	1.00	0.997	1.5	1.015	255[a]	0.340[a]	349.9[a]	22.2	53.0	10.9	25.9	31.7	50.1	2.09	1.08	0.999	0.6	22.7	53.3	11.5	26.4	32.2	50.5
VC-MN	12/09/12	57.8	29.2	13.9	59.7	30.2	13.5	0.0	-0.35	1.04	1.000	0.5	1.032	243.9	0.5694	559.7	15.1	43.2	8.5	21.5	26.6	43.3	0.44	0.96	0.999	0.5	14.9	41.9	8.5	21.0	25.9	42.1
VC-NM	10/08/12	56.1	29.4	13.8	58.2	30.2	14.3	0.0	-0.07	1.04	1.000	0.2	1.028	242.	0.5450	551.4	13.5	41.8	7.3	17.7	24.8	41.8	-1.75	0.96	0.996	1.1	13.1	40.4	7.1	17.1	23.9	40.4
	N	9	9	9	9	9	9	9					N	9	7	7	9	9	9	9	9	9					9	9	9	9	9	9
	Average	58.3	29.5	14.8	59.2	29.4	14.6	0.1					Average	1.028	243.3	0.5810	557.3	15.7	46.5	8.5	21.3	27.5	45.6					15.7	46.1	8.7	21.3	27.3	45.2
	SD	2.4	1.3	0.9	3.0	2.7	1.2	0.2					SD	0.006	0.7	0.0261	8.7	2.7	3.5	1.1	2.3	2.0	2.8					2.9	3.6	1.4	2.5	2.4	3.2
	Min	55.1	27.67	13.8	53.9	23.40	13.4	0.0					Min	1.015	242.0	0.5450	541.6	12.6	41.8	7.3	17.7	24.8	41.8					12.6	40.4	7.1	17.1	23.9	40.4
	%25	56.1	28.33	13.9	58.2	29.35	13.5	0.0					%25	1.026	243.2	0.5602	554.7	14.6	43.6	8.0	20.2	26.6	44.0					14.8	45.8	7.7	20.0	25.9	42.2
	Median	57.8	29.39	14.9	59.7	30.18	14.5	0.0					Median	1.030	243.5	0.5933	559.7	15.1	46.5	8.5	21.5	27.8	44.7					14.9	46.2	8.3	21.6	26.9	45.2
	%75	60.6	30.31	15.1	60.1	31.23	15.0	0.0					%75	1.031	243.8	0.5971	560.5	16.0	48.4	8.8	22.2	28.1	48.2					15.9	47.2	8.7	21.8	27.6	47.2
	Max	61.2	31.83	16.8	63.7	32.15	16.8	0.7					Max	1.036	244.0	0.6140	569.4	22.2	53.0	10.9	25.9	31.7	50.1					22.7	53.3	11.5	26.4	32.2	50.5
	MADe	3.7	1.6	0.4	2.2	1.6	1.4	0.0					eSD	0.0	0.6	0.0307	2.6	1.3	4.1	0.7	1.7	1.5	2.0					1.4	1.6	0.9	0.8	1.4	4.0
	CV	6	5	3	4	5	10						CV	0.33	0.26	5.2	0.5	9	9	8	8	5	5					10	3	11	4	5	9

a) 5% Trichloroacetic acid solution

Appendix H. Representative "Individualized Report" for RR37

Each participant in RR37 received an "Individualized Report" reflecting their reported results. The following two pages are the "Individualized Report" for participant "VC-MA".

Vitamin C "Round Robin" 37 Report: Participant VC-MA

Date	RR	Method	MPA Density g/mL	Dilute Solution 1 Spectrophotometry λ_{max}	A_{max}	$E^{1\%}$	Control/Calibration Solutions Y_{meas} = Inter + Slope* X_{grav} Inter	Slope	R^2	SEE
02/24/10	32	HPLC-EC	1.035	242.0	0.566	545.1	0.3	1.03	1.000	0.46
09/27/10	33	HPLC-EC	1.037	244.0	0.560	540.5	0.4	1.08	1.000	0.43
02/28/11	34	HPLC-EC	1.039	244.0	0.575	555.2	0.6	1.14	1.000	0.78
08/24/11	35	HPLC-EC	1.039	242.0	0.568	547.7	0.0	1.03	1.000	0.24
02/02/12	36	HPLC-EC	1.035	244.0	0.561	550.5	-0.4	1.05	1.000	0.55
09/25/12	37	HPLC-EC	1.035	243.0	0.551	541.6	0.0	1.04	1.000	0.41
		Mean	1.037	243.2	0.56	546.8			Pooled SEE	0.50
		SD	0.002	1.0	0.01	5.6				

[TAA] mmol/Lsample

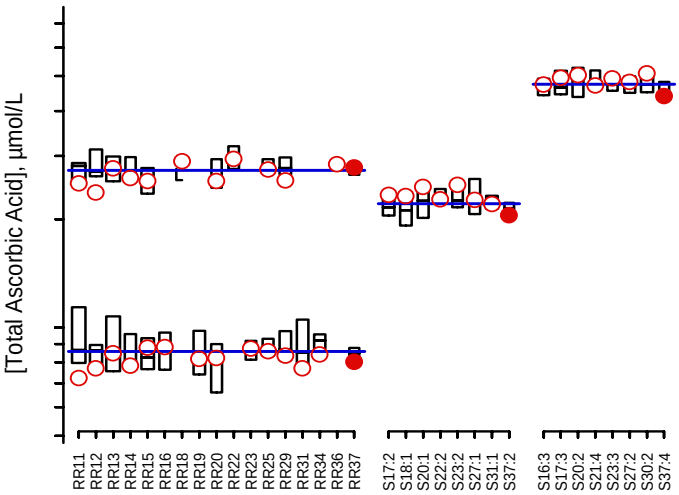
Date	RR	Sample	Rep_1	Rep_2	F_{adj}	Mean	SD_{dup}	N	Mean	SD_{repeat}	SD_{reprod}
09/23/98	11	S11:1	15.5	13.9	0.5	7.4	0.6	14	8.2	0.3	0.5
04/02/99	12	S12:1	14.5	15.8	0.5	7.6	0.5				
09/17/01	13	S13:1	8.4	8.5	1.0	8.5	0.1				
09/27/01	14	S14:3	8.0	7.7	1.0	7.8	0.2				
09/18/01	15	S15:1	8.9	8.7	1.0	8.8	0.1				
11/18/02	16	S16:1	8.8	8.8	1.0	8.8	0.0				
11/13/03	19	S19:4	7.8	8.6	1.0	8.2	0.5				
02/23/04	20	S20:3	8.3	8.1	1.0	8.2	0.1				
10/17/05	23	S23:4	8.6	8.8	1.0	8.7	0.1				
08/28/06	25	S25:1	8.7	8.5	1.0	8.6	0.2				
08/11/08	29	S29:2	8.3	8.4	1.0	8.3	0.1				
09/10/09	31	S31:3	7.3	8.1	1.0	7.7	0.5				
02/28/11	34	S34:1	8.5	8.3	1.0	8.4	0.1				
09/25/12	37	S37:1	**8.0**	**8.1**	1.0	8.0	0.0				
09/23/98	11	S11:2	50.7	47.7	0.5	24.6	1.1	12	26.7	0.5	1.8
04/02/99	12	S12:2	49.5	45.9	0.5	23.9	1.3				
09/17/01	13	S13:2	27.6	27.7	1.0	27.7	0.1				
09/27/01	14	S14:4	25.7	26.4	1.0	26.0	0.5				
09/18/01	15	S15:2	25.4	25.6	1.0	25.5	0.2				
03/20/03	18	S18:3	28.8	29.2	1.0	29.0	0.3				
02/23/04	20	S20:4	25.9	25.2	1.0	25.5	0.5				
03/08/05	22	S22:4	29.4	29.4	1.0	29.4	0.0				
08/28/06	25	S25:2	27.6	27.4	1.0	27.5	0.1				
08/11/08	29	S29:3	25.6	25.7	1.0	25.6	0.1				
02/02/12	36	S36:3	28.3	28.5	1.0	28.4	0.1				
09/25/12	37	S37:3	**27.9**	**27.8**	1.0	27.8	0.1				
12/12/02	17	S17:2	23.3	23.4	1.0	23.4	0.1	8	23.0	0.5	1.4
03/20/03	18	S18:1	22.7	23.7	1.0	23.2	0.7				
02/23/04	20	S20:1	25.1	24.1	1.0	24.6	0.7				
03/08/05	22	S22:2	22.7	22.7	1.0	22.7	0.0				
10/17/05	23	S23:2	25.5	24.4	1.0	24.9	0.8				
10/05/07	27	S27:1	22.9	22.4	1.0	22.6	0.4				
09/10/09	31	S31:1	21.5	22.6	1.0	22.1	0.7				
09/25/12	37	S37:2	**20.4**	**20.7**	1.0	20.5	0.2				
11/18/02	16	S16:3	49.9	44.9	1.0	47.4	3.5	8	48.3	1.3	2.2
12/12/02	17	S17:3	49.7	49.1	1.0	49.4	0.4				
02/23/04	20	S20:2	50.6	50.0	1.0	50.3	0.4				
09/13/04	21	S21:4	47.1	47.0	1.0	47.0	0.0				
10/17/05	23	S23:3	49.8	48.8	1.0	49.3	0.7				
10/05/07	27	S27:2	48.6	47.6	1.0	48.1	0.8				
03/03/09	30	S30:2	51.2	50.4	1.0	50.8	0.6				
09/25/12	37	S37:4	**43.9**	**44.1**	1.0	44.0	0.1				

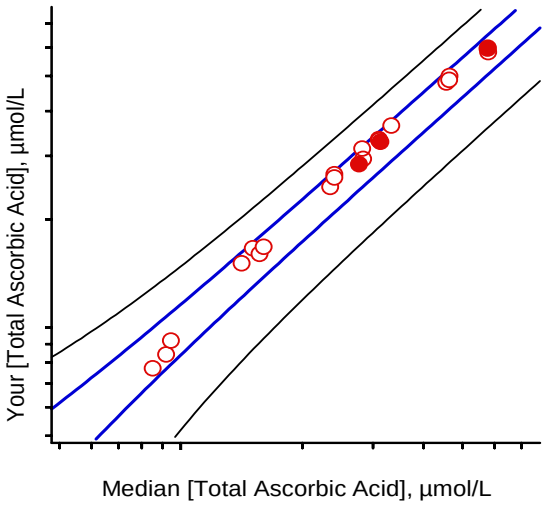
Please check our records against your records. Send corrections and/or updates to...

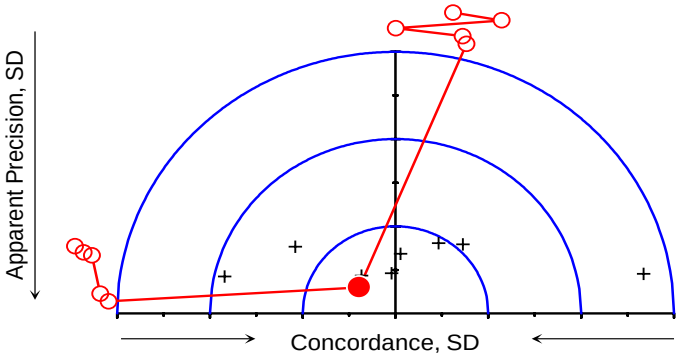
Micronutrients Measurement Quality Assurance Program
National Institute of Standards and Technology
Fax: (301) 977-0685
Email: david.duewer@nist.gov

Vitamin C "Round Robin" 37 Report: Participant VC-MA

Total Ascorbic Acid, µmol/mL







3rd Quartile (75%)
Median (50%)
1st Quartile (25%)

● You, this RR
○ You, past RRs

\+ Others, this RR

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

Sample	Comments
S37:1	SRM970 Lv I - distributed in RRs 11, 12, 13, 14, 15, 16, 19, 20, 23, 25, 29, 31, and 34
S37:2	Distributed in RRs 17, 18, 20, 22, 23, 27, and 31
S37:3	SRM970 Lv II - distributed in RRs 11, 12, 13, 14, 15, 18, 20, 22, 25, 29, and 36
S37:4	Distributed in RRs 16, 17, 20, 21, 23, 27, and 30