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NIST Micronutrients Measurement Quality Assurance Program Summer 2008 Comparability Studies

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

David L. Duewer Jeanice B. Thomas

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April, 2013



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

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Abstract

The National Institute of Standards and Technology coordinates the Micronutrients Measurement Quality Assurance Program (MMQAP) for laboratories that measure fat- and water-soluble vitamins and carotenoids in human serum and plasma. This report describes the design of and results for the Summer 2008 MMQAP measurement comparability improvement studies: 1) Round Robin LXIV Fat-Soluble Vitamins and Carotenoids in Human Serum and 2) Round Robin 29 Total Ascorbic Acid in Human Serum. The materials for both studies were shipped to participants in May 2008; participants were requested to provide their measurement results by September 12, 2008.

Keywords

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

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Introduction

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

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

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

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

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin LXIV comparability study (hereafter referred to as RR64) received two lyophilized and three liquid-frozen human serum test samples for analysis. Unless multiple vials were previously requested, participants received one vial of each serum. These sera were shipped on dry ice to participants in May 2008. The communication materials included in the sample shipment are provided in Appendix A.

Participants are requested to report values for all fat-soluble vitamin-related analytes that are of interest to their organizations. Not all participants report values for the target analytes, and many participants report values for non-target analytes.

The final report delivered to every participant in RR64 consists of three documents:

- A cover letter for the current study, a brief description of the other two documents, and a discussion of our analysis of the overall results that may be of broad interest. This cover letter is reproduced as Appendix B.
- The "All-Lab Report" that lists all of the reported measurement results, a number of consensus statistics for analytes reported by more than one participant, and the mean median and pooled SD from any prior distributions of the serum. This report also provides a numerical "score card" for each participant's measurement comparability for the more commonly reported analytes. This report is reproduced as Appendix C.

• An "Individualized Report" that graphically analyzes each participant's results for all analytes reported by at least five participants. This report also provides a graphical summary of their measurement comparability. The graphical tools used in this report are described in detail elsewhere [3]. An example "Individualized Report" is reproduced as Appendix D.

Round Robin 29: Vitamin C in Human Serum

Participants in the MMQAP Vitamin C in Human Serum Round Robin 29 comparability study (hereafter referred to as RR29) received four frozen serum test samples, one frozen control serum, and a solid ascorbic acid control material for analysis. Unless multiple vials were previously requested, participants received one vial of each material. These sample materials were shipped on dry ice to participants in May 2008. The communication materials included in the sample shipment are provided in Appendix E.

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

The final report delivered to every participant in RR29 consists of three documents:

- A cover letter for the current study, a brief description of the other two documents, and a
 discussion of our analysis of overall results that may be of broad interest. This cover letter is
 reproduced as Appendix F.
- The "All-Lab Report" that summarizes all of the reported measurement results and provides several consensus statistics. This report is reproduced as Appendix G.
- An "Individualized Report" that graphically analyzes each participant's results for TAA, including a graphical summary of their measurement comparability. The graphical tools used in this report are described in detail elsewhere [3]. An example "Individualized Report" is reproduced as Appendix H.

References

- 1 Duewer DL, Brown Thomas J, Kline MC, MacCrehan WA, Schaffer R, Sharpless KE, May WE, Crowell JA. NIST/NCI Micronutrients Measurement Quality Assurance Program: Measurement Repeatabilities and Reproducibilities for Fat-Soluble Vitamin-Related Compounds in Human Sera. Anal Chem 1997;69(7):1406-1413.
- 2 Margolis SA, Duewer DL. Measurement Of Ascorbic Acid in Human Plasma and Serum: Stability, Intralaboratory Repeatability, and Interlaboratory Reproducibility. Clin Chem 1996;42(8):1257-1262.
- 3 Duewer DL, Kline MC, Sharpless KE, Brown Thomas J, Gary KT, Sowell AL. Micronutrients Measurement Quality Assurance Program: Helping Participants Use Interlaboratory Comparison Exercise Results to Improve Their Long-Term Measurement Performance. Anal Chem 1999;71(9):1870-1878.

Appendix A. Shipping Package Inserts for RR64

The following three items were included in each package shipped to an RR64 participant:

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

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

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

May 12, 2008

Dear Colleague:

Enclosed are the samples (Sera 347-351) for the second fat-soluble vitamins and carotenoids in serum round robin study (Round Robin LXIV) for the fiscal year (FY) 08 NIST Micronutrients Measurement Quality Assurance Program. You will find one vial of each of three liquid-frozen serum samples and two lyophilized samples for analysis along with a form for reporting your results. When reporting your results, please submit one value for each analyte for a given serum sample. If a value is obtained below your limit of quantification, please indicate this result on the form by using NQ (Not Quantified). Results are due to NIST by September 12, 2008. Results received more than two weeks after the due date will not be included in the summary report for this round robin study. For your convenience, we have also included one vial of Serum 198 (which was distributed in four past round robin studies) that can be used as a control and to help validate your method. We have included the averaged assigned values for the measurands in this serum as well.

Lyophilized samples should be reconstituted with 1.0 mL of HPLC-grade water or equivalent. We recommend that dissolution be facilitated with 3 to 5 min agitation in an ultrasonic bath or at least 30 min at room temperature with intermittent swirling. (CAUTION: Vigorous shaking will cause foaming and possibly interfere with accurate measurement. The rubber stopper contains phthalate esters that may leach into the sample upon intermittent contact of the liquid sample with the stopper. These esters absorb strongly in the UV region and elute near retinol in most LC systems creating analytical problems.) Pipette a known volume of serum from the vial for analysis. The final volume of the reconstituted sample is greater than 1.0 mL. Water should not be added to the liquid-frozen samples.

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

Please mail or fax your results to:

Micronutrients Measurement Quality Assurance Program

NIST

100 Bureau Drive Stop 8392 Gaithersburg, MD 20899-8392

Fax: (301) 977-0685

If you have questions or comments regarding this study, please call me at (301) 975-3120; e-mail me at ibthomas@nist.gov; or mail/fax queries.to the above address.

Sincerely.

Jeanice Brown Thomas

Research Chemist

Analytical Chemistry Division

Chemical Science and Technology Laboratory

Enclosures



Reference Values for NIST Serum 198, µg/mL

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

- a Expected value; the average of the interlaboratory median results and mean of NIST results in M^2QAP RR31, RR35, RR43, and RR57
- b Approximate 95% uncertainty interval; the interval Value \pm U_{95} is believed with approximately 95% confidence to contain the true value of the analyte
- c Approximate 95% prediction interval; the interval Value $\pm P_{95}$ contains approximately 95% of valid measurements obtained with measurement systems similar to those in use in M²QAP RR31, RR35, RR43, and RR57

Round Robin LXIV: Human Sera NIST Micronutrients Measurement Quality Assurance Program

total retinol trans-retinol didehydroretinol retinyl palmitate α -tocopherol γ/β -tocopherol			
didehydroretinol retinyl palmitate α-tocopherol γ/β-tocopherol			
retinyl palmitate α-tocopherol γ/β-tocopherol			
α-tocopherol γ/β-tocopherol			
γ/β-tocopherol			
δ-tocopherol			
total β-carotene			
trans-β-carotene			
total cis-β-carotene			
total α-carotene			
total lycopene			
trans-lycopene			
total β-cryptoxanthin			
total α-cryptoxanthin			
total lutein			
total zeaxanthin			
total lutein&zeaxanthin			
total coenzyme Q10			
ubiquinol (QH ₂)			
ubiquinone (Qox)			
phylloquinone (K ₁)			
25-hydroxyvitamin D			
Other measurands?	 	 	

* we prefer µg/mL

Fax: 301-977-0685

Email: David.Duewer@NIST.gov

Were the liquid-frozen samples (349 to 351) frozen when received? Yes | No

Comments:

Mail: M²QAP NIST, Stop 8392 Gaithersburg, MD 20899-8392

Participant	#:	
ranticipant	#.	

Fat-Soluble Vitamins Round Robin LXIV NIST Micronutrients Measurement Quality Assurance Program

Packing List and Shipment Receipt Confirmation Form

This box contains: one vial each of the following five FSV M²QAP sera

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

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

|--|

- 2) Are all five sera vials intact? Yes | No If "No", which one(s) were damaged?
- 3) Was there any dry-ice left in cooler? Yes | No
- 4) Did the liquid frozen samples arrive frozen? Yes | No
- 5) At what temperature are you storing the serum samples? ____ °C
- 6) When do you anticipate analyzing these samples? _____

Your prompt return of this information is appreciated.

The M²QAP Gang

Appendix B. Final Report for RR64

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

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



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

November 20, 2008

Dear Colleague:

Enclosed is the summary report of the results for round robin LXIV (RR64) of the 2008 NIST Micronutrients Measurement Quality Assurance Program (M²QAP) for the fat-soluble vitamins and carotenoids in human serum. Included in this report are: 1) a summary of data and measurement comparability scores for all laboratories, 2) a detailed graphical analysis of your results; and 3) a graphical summary of your measurement comparability.

Your overall measurement comparability is summarized in the "Score Card" summary, page 6 of the All Lab Report. Combined results rated 1 to 3 are within 1 to 3 standard deviations of the assigned value, respectively; those rated 4 are >3 standard deviations from the assigned value. Similar information is presented graphically in the "target plots" that are the last page of your Individualized Report. If you have concerns regarding your laboratory's performance, please contact us for consultation.

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

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

Sincerely,

Jeanice Brown Thomas

Research Chemist

Analytical Chemistry Division

Chemical Science and Technology Laboratory

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

NST

The NIST M²QAP Round Robin LXIV (RR64) report consists of:

Page	"All Lab" Report
1-4	A listing of all results and statistics for all analytes.
5	A legend for the list of results and statistics.
6	The text Comparability Summary ("Score Card") of measurement performance.
Page	"Individualized" Report
	I .
1	Your values, the number of labs reporting values, and our assigned values.
1 2 to	Your values, the number of labs reporting values, and our assigned values. "Four Plot" summaries of your current and past measurement performance, one page for
1 2 to n	Your values, the number of labs reporting values, and our assigned values.

Samples. Five samples were distributed in RR64.

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

Results

- 1) <u>Sera Stability.</u> There was no significant change in the median level or measurement variability of any measurand in any of the lyophilized or fresh-frozen materials.
- 2) <u>Candidate SRM 1950, Metabolites in Human Plasma (#350).</u> As in RR63, no analysis problems were reported for this material. We anticipate this material being available for purchase mid-2009.
- 3) <u>SRM 968d Fat-Soluble Vitamins and Carotenoids in Human Serum (#351).</u> As in RR63, no analysis problems were reported for this material. It will be available for purchase in the near future.

Appendix C. "All-Lab Report" for RR64

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

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

The data summary in the "All-Lab Report" has been altered to ensure confidentiality of identification codes assigned to laboratories. The only attributed results are those reported by NIST. The NIST results are not used in the assessment of the consensus summary results of the study.

L 351	-	0.073																				,	0.142	0.086					4	0.073	0.083	0.142	19	9	0.084	0.028				0.083
5-Tocopherol, µg/ml	-	3 0.068																				7	01	8 0.082					3 3	8 0.068	3 0.075	3 0.082		6 5	0.07	7 0.009				0.053 0.075
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g/mL	0.431	0.438	0.419	0.464	0.410	0.428	0.387	0.385	0.526	0.430	0.420	0.464	0.447	0.425	≥0.430	20.592	0.442	0.394	0.430	0.370	0.450	0.483	0.401	0.404	0.363	0.360	>0.407	0.366	30	0.360	0.423	0.526	 	30	0.423	0.023	0.408	0.005	0.00	0.415
Total Retinol, µg/ml	0.665	0.679	0.673	0.746	0.600	0.644	0.620	0.606	0.738	0.690	0.650	0.633	0.648	0.656	≥0.630	20.353	0.606	0.648	0.690	0.570	0.700	0.724	0.608	0.605	0.565	0.580	>0.641	0.569	30	0.554	0.638	0.746	0.00 0	33	0.642	0.045	0.647	0.009	0.00	0.643
Total Re	0.633	0.680	0.630	969.0	0.590	0.607	0.553	0.545	0.702	0.660	0.620	0.739	0.633			20.424 2 0.661			0.620	0.550	0.660	0.085	0.676	0.593	0.552	0.530	> 0.60.3		30	0.530	0.623	0.739	11	33	0.608	0.043	0.602	0.030	0.027	0.612
347	g		0.355				0.385				0.350						0.342					0.447				0.360	>0.386					0.472		4	0.381	0.042			0.030	0.350
L	Ā		FSV-BC (FSV-BD (FSV-BF (FSV-BO			FSV-BR ≥	'VI	FSV-BU (FSV-CE						FSV-DQ FSV-DV	l	z			Max		Npast		SDpast (SNIST (NAV
4	S C	FS	FS FS	FS	ES E	S E	Š K	ξ	FS	S	ES 5	S S	FS	FS\	FS	ָר הַ	FS	FS	FSV	FS	Z G	נו ב	ξ K	FSV	FS	FS\	FS/	FS			Μ̈́			_	Medianpast	Ś	Mear		U)	

ycopene, µg/mL	349	0.229 0.332 0.170 0.125 0.229 0.237 0.138 0.108	0.335 0.356 0.184 0.153	0.023 0.234 0.241 0.140 0.114	0.320 0.327 0.210 0.150	0.039 0.200 0.239 0.119 0.105	0.288 0.297 0.149 0.090 0.272 0.279 0.147 0.128		8 8 8 8 0.200 0.200 0.237 0.119 0.090 0.266 0.288 0.148 0.120 0.335 0.356 0.210 0.153 0.047 0.050 0.025 0.020 18 17 17 16	8 8 8 0.250 0.264 0.146 0.114 0.045 0.048 0.019 0.018	
	347	0.034	0.035		0.039		0.033	20	8 0.023 0.034 0.039 0.007	8 0.038 0.008	73
ie, µg/mL	349	0.517 0.328 0.259 0.492 0.336 0.280	0.621 0.361 0.537 0.347 0.416 0.257 0.579 0.286	0.440 0.288 0.248 0.588 0.400 0.392 0.152 0.080 0.040	0.336 0.191 0.451 0.338 0.570 0.367 0.572 0.310	0.833 0.400 0.310	0.074 0.490 0.496 0.289 0.191	0.391 0.267 0.207	16 16 0.152 0.080 0.0 0.507 0.319 0.2 0.695 0.400 0.3 0.103 0.051 0.0	21 18 0.512 0.323 0.2 0.064 0.036 0.0	0.432 0.301 0.273 0.004 0.026 0.003 0.012 0.002 0.003
	_	0.079 0.446 0.071 0.474	0.076 0.591 0.075 0.520 0.066 0.396 15 0.057 0.446	8 0.069 0.428 0.070 0.504 13 0.177 0.155	0.048 0.086 0.071 0.070	0.030 0.618		21 0.160 0.250	16 16 16 004 0.048 0.155 009 0.073 0.464 022 0.177 0.616 005 0.008 0.082 56 11 18	15 24 21 009 0.071 0.483 003 0.021 0.054	10 nq 0.438 0.012 0.005
µg/mL		0.026 0.009	0.024 0.010 nq nq 0.025 0.007 0.020 0.015	0.024 0.008 0.026 0.005 0.014 0.013		0.022 <0.005	0.028 0.007 0.018 <0.009 0.026 0.007	0.011 0.021	18 16 0.011 0.004 0.024 0.009 0.004 0.022 17 56	17 15 0.027 0.009 0.005 0.003	0.032 0.010 0.003 0.001
Total α-Carotene	348 349 3	0.073 0.080 0.065 0.066	0.085 0.091 0.086 0.090 0.072 0.078 0.080 0.092	0.072 0.078 0.079 0.089 0.031 0.038	0.097 0.104 0.111 0.110 0.092 0.097 0.090 0.090	0.061 0.071	0.091 0.091 0.055 0.059 0.085 0.086	0.051 0.062	19 19 0.031 0.038 0.079 0.086 0.111 0.110 0.019 0.017 23 19	21 21 0.075 0.080 0.012 0.011	0.078 0.079 0.004 0.003 0.003 0.001
,			0.017 0.014 0.029	005 - 0.018 nd		0.003	0.021 0.016 0.020	0.013	0.0	4 24 0.005 0.019 0.002 0.006	0.002
ene, µg/r	349 350 3	0.004 0.005 0.004 0.004	bu bu bu	0.004 0.003 0.005	0.007 0.007 0.007		0.028 0.009 0.007 0.006 0.006		6 6 0.004 0.003 0.005 0.005 0.019 0.007 0.002 0.001 38 23	6 5 0.007 0.006 0.002 0.002	
ď	347	0.033	3 0.025 nq	0.038 0.006	0.033 0.007				3 7 6 3 0.022 0.004 6 5 0.033 0.007 8 8 0.084 0.020 9 9 0.007 0.003 45	7 0.027 0.011	
rans-β-Carotene, μg/ml	347 348 349 350 351	0.315 0.107 0.118 0.076 0.080 0.287 0.103 0.105 0.070 0.071	0.346 0.118 0.121 0.075 0.078	0.279 0.100 0.100 0.066 0.069 0.289 0.116 0.108 0.077 0.084	0.314 0.123 0.115 0.057 0.067 0.355 0.123 0.122 0.089		0.274 0.103 0.106 0.061 0.048		8 8 8 8 8 8 0.274 0.100 0.100 0.057 0.048 0.302 0.112 0.111 0.073 0.075 0.055 0.055 0.055 0.11 0.012 0.012 0.010 0.009 0.009 0.011 0 11 9 12 12	10 11 11 9 9 0.320 0.110 0.118 0.069 0.075 0.028 0.011 0.011 0.007	
F		0.084	0.0387 0.075 0.093 0.078 0.070 0.069	0.077 0.2 0.089 0.2 0.040		0.082 0.182 0.070	0.054 0.2 0.050 0.090	0.053	21 0.040 0.2 0.077 0.3 0.182 0.3 0.011 0.0	22 0.075 0.3 0.013 0.0	0.081 0.003 0.001
		0.073	0.068 10.0 0.064 C 0.087 C 0.075 C 0.074 C	0.072 C 0.083 C 0.090 C	ΛI	0.168	0.066 C	0.061	22 0.057 0.078 0.168 0.014 0.014	22 0.075 0.012	0.002
ne, µ		0.112	0.079 0.099 0.153 0.121 0.116	0.108 0.127 0.082	ΛI	0.201 0.140	0.113 0.114 0.135	0.088	22 0.079 0.122 0.201 0.018		0.129 0.004 0.006
Total β-C	348	0.107	0.084 0.099 0.146 0.117 0.107	0.108 0.136 0.070		0.197 0.031	0.112 0.106 0.132	0.052	22 0.031 0.115 0.197 0.017	24 0.114 0.016	0.005
	347	0.309	0.175 0.367 0.354 0.371 0.325	0.320 0.373 0.064		0.422	0.301 0.294 0.340	0.378	22 0.064 0.335 0.422 0.045	31 0.341 0.043	0.008
4	Lab	FSV-BB FSV-BB FSV-BC	FSV-BD FSV-BE FSV-BG FSV-BH FSV-BH FSV-BJ	FSV-BM FSV-BM FSV-BO FSV-BO FSV-BO		FSV-CE FSV-CE	FSV-CF FSV-CG FSV-CW FSV-CW FSV-CZ	FSV-DD FSV-DQ FSV-DV FSV-EF	Median Max SD SD	Npast Medianpast SDpast	Meannist Srep Shet

Total a-Cryptoxanthin, µg/mL Total Lutein, µg/mL	0.022 0.019 0.037 0.099 0.101 (0.025 0.084 0.088 0.058 0.045 0.045 0.013 0.046 0.036 0.045 0.045 0.024 0.071 0.074 0.061 0.049 0.023 0.071 0.079 0.062 0.054	0.002 0.013 0.013 0.004 0.007 0.033 0.077 0.079 0.064 0.059 0.021 0.072 0.073 0.066 0.057	0.029 0.086 0.075 0.072 0.098	0.021 0.055 0.054 0.047 0.030 0.026 0.079 0.080 0.069 0.058	0.010 0.098 0.101 0.091 0.077	3 3 1 11 11 11 11 11 11 0.013 0.004 0.005 0.045 0.030 0.032 0.022 0.019 0.037 0.099 0.010 0.098 0.091 0.098 0.005	6 5 6 5 7 12 14 14 12 12 0.006 0.024 0.026 0.001 0.006 0.006 0.001 0.002 0.001 0.002 0.001 0.002 0.001 0.002 0.001 0.002 0.001 0.002 0.001 0.002 0.001 0.002 0.001 0.002 0.001 0.002 0.001	0.002 0.003 0.001 0.004 0.003 0.005
Total β-Cryptoxanthin, μg/mL Total α-Crypto	BA 0.009 0.064 0.069 0.049 0.004 0.003 0.022 BB 0.010 0.052 0.053 0.036 0.040 0.003 0.022 BD BB BB 0.000 0.005 0.0	FSV-BG 0.009 0.063 0.069 0.040 0.050 FSV-BH nq 0.059 0.062 0.041 0.046 FSV-BI 0.008 0.051 0.053 0.033 0.036 FSV-BL rev.bb nq 0.037 0.045 0.026 0.032 FSV-BL FSV-BL	0.027 0.039 0.040 0.025 0.031 0.010 0.048 0.051 0.034 0.044 0.008 0.018 0.015 0.025 0.018	FSV-BS 0.016 0.075 0.080 0.041 0.056 FSV-BS 0.016 0.049 0.050 0.036 0.040 FSV-BU 0.011 0.048 0.047 0.032 0.034 FSV-BU 0.017 0.055 0.055 0.033 0.034 FSV-BW 70 0.052 0.055 0.033 0.034 FSV-CD 0.006 0.045 0.046 0.032 0.039 FSV-CD	FSV-CF FSV-CG 0.015 0.077 0.077 0.049 0.047 FSV-CI FSV-CM 0.005 0.051 0.053 0.031 0.036 FSV-CZ	FSV-DD FSV-DQ 0.016 0.035 0.056 0.034 0.033 FSV-EP	15 18 18 18 18 3 3 0.005 0.018 0.015 0.015 0.015 0.015 0.015 0.010 0.010 0.051 0.053 0.034 0.039 0.003 0.022 0.004 0.009 0.009 0.006 0.008 0.029 0.004 0.009 0.009 0.006 0.008 18 18 18 22	Npast 17 21 21 18 18 6 5 Median _{past} 0.012 0.052 0.055 0.036 0.040 0.006 0.024 0.0 SD _{past} 0.004 0.008 0.010 0.009 0.005 0.001 0.006 0.0 MeanNIST nq 0.049 0.049 0.034 0.033	, 0.000 0.002 0.005 0 0.001 0.001 0.004 0 0.001 0.003 0.006 0

Round Robin LXIV Laboratory Results

-		penzy	Coenzyme Q10, µg/ml), µg/m		Phy	/loquin	one (K	Phylloquinone (K1), ng/mL	nL 25.4
Lab	347	348	349	320	351	347	348	349	320	351
FSV-BA FSV-BB FSV-BC										
FSV-BE FSV-BF	0.306	0.764	0.791	0.850	0.786	0.534	0.492	0.466	0.526	0.280
FSV-BG FSV-BH										
FSV-BI		709		0.00						
FSV-BX	0.270	0.097	0.703	0.540	0.573					
FSV-BL										
FSV-BM										
FSV-BO										
FSV-BP										
FSV-BQ										
FSV-BR										
FSV-B3	0 127	0 223	0.252	0 340	0.354					
FSV-BU		0.443								
FSV-BV										
FSV-BW	0.356	0.783	0.787	0.633	0.602					
FSV-CD										
FSV-CE										
77-V07 GC-V07										
FSV-C	0.310	0.760	0.800	0.850	0.750	0.625	0.421	0.386	0.424	0.206
FSV-CW	0.109	0.310							!	
FSV-CZ	0.323	0.848		0.743	0.626					
FSV-DD										
FSV-DQ FSV-DV										
FSV-EE	0.285	0.634	0.653	0.700	0.496					
z :	æ (ω ;			ω ι	2 5	7 5	2 5	7 5	2 5
⊆ Z	0.109	0.223			0.207	0.53	0.42	0.39	0.42	0.21
Median	0.296	0.729	0.730	0.667	0.588	0.58	0.46	0.43	0.48	0.24
Max		0.848				0.03	0.49	74.0	0.53	0.28
ડે ફિ		22								
Noast	G	ľ.	ſĊ.	σ	σ					
Medianpast		0.701	0.751							
SDpast	0.018	0.096	0.104	0.166	0.122					
Meannist										
Srep										
SNIST										
NAV	0.296	0.729	0.730	0.667	0.588					
	0.00	3		0.200						

Analytes Reported By One Laboratory

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

Term	Legend
N	Number of (non-NIST) quantitative values reported for this analyte
Min	Minimum (non-NIST) quantitative value reported
Median	Median (non-NIST) quantitative value reported
Max	Maximum (non-NIST) quantitative value reported
SD	Standard deviation for (non-NIST) results: 0.741*(3rd Quartile - 1st Quartile)
CV	Coefficient of Variation for (non-NIST) results: 100*SD/Median
	Mark of N/a) for an anal PD/a)
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)
Moan	Mean of NIST results
_	
	NIST's within-vial pooled standard deviation
	NIST's among-vial pooled standard deviation
SNIST	Combined standard deviation for NIST analyses: $(S_{rep}^2 + S_{het}^2)^{0.5}$
NAV	NIST Assigned Value
147(0	= (Median + Mean _{NIST})/2 for analytes reported by NIST analyst(s)
NIALI	= Median for analytes reported by ≥ 5 labs but not NIST
NAU	NIST Assigned Uncertainty: (S ² + S _{btw} ²) ^{0.5}
	S is the maximum of (0.05*NAV, SD, S _{NIST} , eSD) and S _{btw} is the standard
	deviation between Median and Mean _{NIST} . The expected long-term SD, eSD,
	is defined in: Duewer et al. Anal Chem 1997;69(7):1406-1413.
	Not analyzed
- -	Not detected (i.e., no detectable peak for analyte)
nd	Detected but not quantitatively determined
nq ≤x	Concentration at or below the limit of quantification, x
≥x ≥x	Concentration at or below the limit of quantification, x Concentration greater than or equal to x
≥X 	Discrepant value: heterogeneous serum, damaged sample, malfunction, etc.
:	
italics	Not explicitly reported but calculated by NIST from reported values

Comparability Summary

Lab	TR	аТ	g/bT	bC	tbC	аC	TLy	TbX	TLu	ΤZ	L&Z
FSV-BA	1	1	1	1	1	1	1	2			1
FSV-BB	2	1	1	1	1	1	1	1	2	2	2
FSV-BC	1										
FSV-BD	1	1									
FSV-BE	2	1	1	3							
FSV-BF	1	1		1							
FSV-BG	1	1	1	2		1	1	1	1	1	1
FSV-BH	1	1	1	1	1	1	1	1	2	2	2
FSV-BI	1	1	1	1		1	1	1	1	1	1
FSV-BJ	1	1	1	1		2	1	1	1		
FSV-BK	3	1									
FSV-BL	1	1									
FSV-BM	1	1									
FSV-BN	2	2	1	1	1	1	1	3	1	1	1
FSV-BO	2	1	1	1	1	1	2	1	1	1	1
FSV-BP	1	2		4		2	4	3			3
FSV-BQ	1	2									
FSV-BR	1	3									
FSV-BS	4			1	1	2	2	2			1
FSV-BT	2	1	1	1	2	2	1	1	2	2	2
FSV-BU	1	2	1	1		1	1	1			1
FSV-BV	1	1	1	1		1	1	1			1
FSV-BW	1	1	1	1		1	2	1			1
FSV-CD	2	1	1	4		1		1			4
FSV-CE	1	4		3							
FSV-CF	2	1									
FSV-CG	1	2	2	1	2	1	1	2			2
FSV-CI	2	1	2	2		1			2	2	2
FSV-CW	1	1	1	1		1		1	1	2	1
FSV-CZ	2	2	3								
FSV-DD	2										
FSV-DQ		2	1	3		2	3	2	2	2	1
FSV-DV	1	2		1							
FSV-EE	2	1									
NIST	1	1	1	1		1	1	1	1	1	1
n	34	32	20	24	8	20	17	19	12	11	19
ا، رہ	TR	аТ	g/bT	bC	tbC	aC	TLy	TbX	TLu		L&Z
% 1	62	69	85	71	75	75	71	68	58	45	63
% 2	32	25	10	8	25	25	18	21	42	55	26
% 3	3	3	5	13	0	0	6	11	0	0	5
% 4	3	3	0	8	0	0	6	0	0	0	5

Label	Definition
Lab	Participant code
TR	Total Retinol
аТ	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
_	number of participants providing quantitative data
n o/ 1	number of participants providing quantitative data
% 1	Percent of CS = 1 (within 1 SD of medians)
% 2	Percent of CS = 2 (within 2 SD of medians)
% 3	Percent of CS = 3 (within 3 SD of medians)
% 4	Percent of CS = 4 (3 or more SD from medians)

"Comparability Score"

The Comparability Score (CS) of summarizes your measurement performance for a given measurand, relative to the consensus medians. CS is the average distance, in standard deviation units, that your measurement performance characteristics are from the consensus performance. CS is calculated when the number of quantitative values you reported for a measurand, N_{you} , is at least two and the measurand has been reported by 10 or more participants.

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

$$C = Concordanc \, e = \sum_{i}^{N_{you}} \frac{You_{i} - Median_{i}}{NAU_{i}} / N_{you}$$

$$AP = Apparent \ Precision = \sqrt{\sum_{i}^{N_{you}} \left(\frac{You_{i} - Median_{i}}{NAU_{i}}\right)^{2} / (N_{you} - 1)}$$

NAU = NIST Assigned Uncertainty, our estimate of the overall measurement standard deviation for each sample. The estimate includes serum heterogeneity, analytical repeatability, and among-participant reproducibility variance components.

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

Appendix D. Representative "Individualized Report" for RR64

Each participant in RR64 received an "Individualized Report" reflecting their reported results. Each report included a detailed analysis for analytes that were assayed by at least five participants. The following analytes met this criterion in RR64:

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

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

Individualized Round Robin LXIV Report: FSV-BA

Set 1 of 40

Summary

	Ser	um 347		Ser	17 Serum 348		348 Serum 349	um 349		Ser	49 Serum 350		0 Serum 351	1m 351	
Analyte		NAV	⊂	You	NAV	L	You	NAV	ے	You	NAV n	٦	You	NAV	⊆
Total Retinol	0.409	0.35	30	0.633	0.612	30	0.665	0.643	30	0.431	0.415	30	0.356	0.338	53
Retinyl Palmitate	0.09	0.12	6	0.1	0.1	6	0.1	0.1	6	0.02	0.02	7	0.03	0.01	2
α-Tocopherol 7.	7.11	6.83	31	9.04	10.11 31	31	9.59	10.81	31	7.78	8.25	31	5.80	5.94	59
γ/β-Tocopherol	1.351	1.333		1.629	1.753	19	1.742	1.816	19	1.733	1.792	19	1.480	1.448	18
ō-Tocopherol	0.081	0.075		0.046	0.046	က	0.053	0.053	က	0.075	0.075	က	0.080	0.083	4
Total β-Carotene	0.348	0.334	22	0.111	0.115	22	0.122	0.125	22	0.081	0.079	22	0.084	0.079	21
trans-β-Carotene	0.315	0.302		0.107	0.112	∞	0.118	0.111	∞	0.076	0.073	∞	0.080	0.075	∞
Total cis-β-Carotene	0.033	0.033		0.004	0.007	9	0.004	0.005	9	0.005	0.005	9		0.005	9
Total α-Carotene	0.016	0.017		0.073	0.079	19	0.080	0.083	19	0.026	0.028	18		0.009	16
Total Lycopene	0.079	0.073	16	0.446	0.451	16	0.517	0.469	16	0.328	0.310	16		0.268	16
trans-Lycopene	0.034	0.034		0.259	0.266	∞	0.302	0.288	∞	0.170	0.148	∞		0.120	∞
Total β-Cryptoxanthin	0.009	0.010	15	0.064	0.050	18	0.069	0.051	18	0.049	0.034	18		0.036	18
Total α-Cryptoxanthin	0.003	0.003	က	0.029	0.022	က	0.032	0.023	က	0.022	0.014	က		0.015	က
Total Lutein&Zeaxanthin	0.036	0.037	17	0.101	0.107	18	0.104	0.109	18	0.092	0.094	18		0.077	18

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

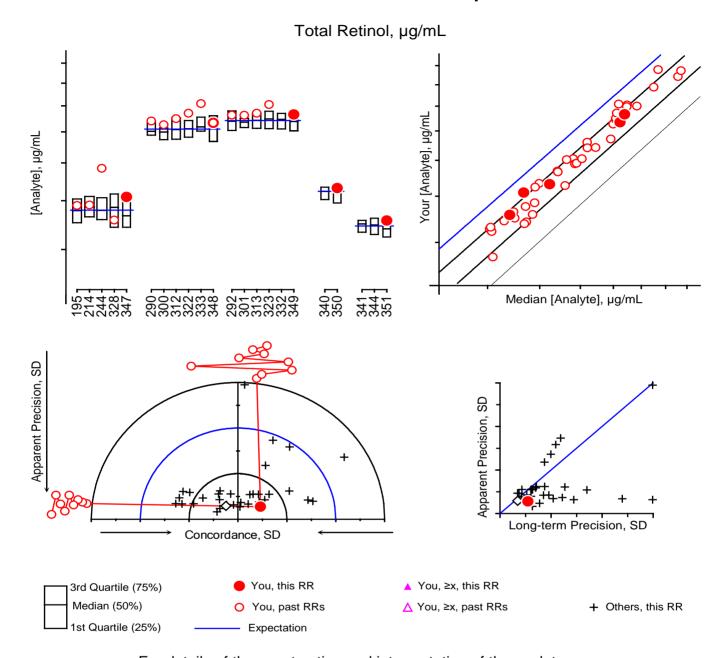
Please check our records against your records. Send corrections and/or updates to...

Micronutrients Measurement Quality Assurance Program National Institute of Standards and Technology

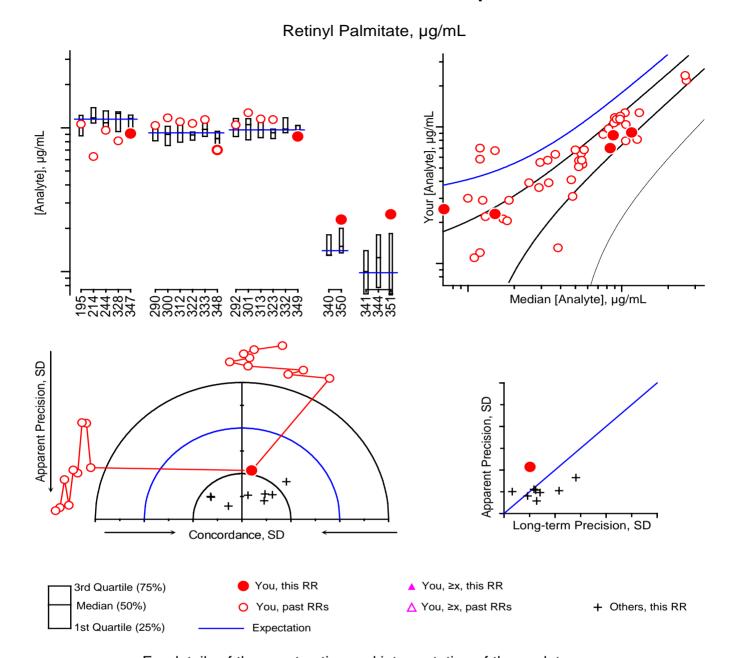
Gaithersburg, MD 20899-8392 USA 100 Bureau Drive Stop 8392

Tel: (301) 975-3935

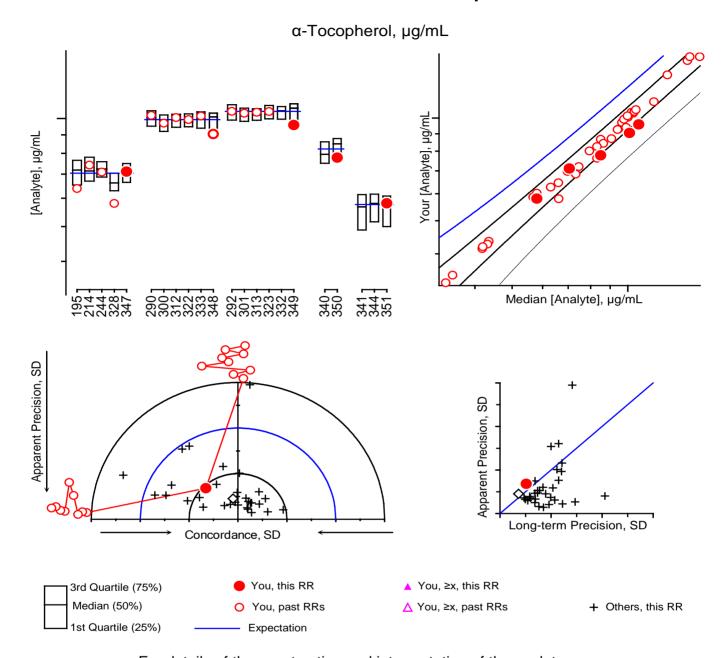
Fax: (301) 977-0685 Email: david.duewer@nist.gov



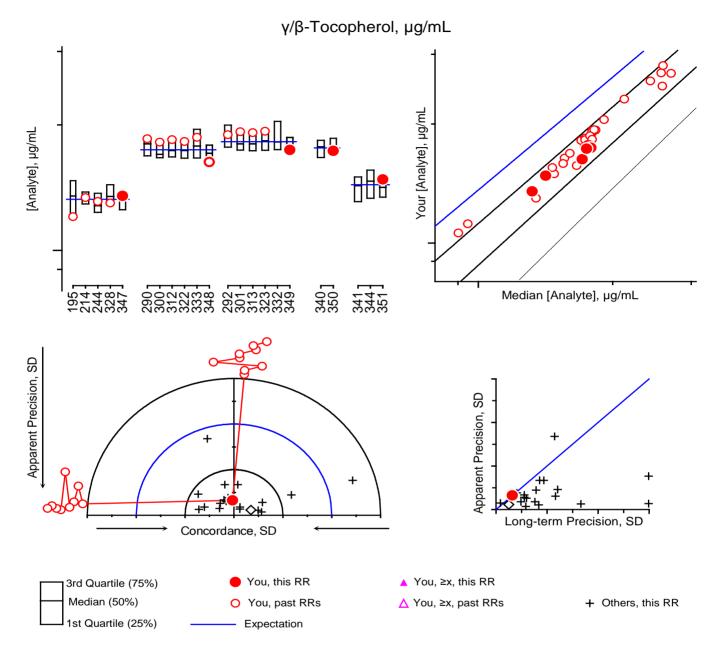
<u>Serum</u>	<u>History</u>	<u>Comments</u>
#347	31:#195, 35:#214, 43:#244, 60:#328	Lyophilized, multi-donor, aug(TR, RP,aT)
#348	53:#290, 55:#300,57:#312, 59:#322:RR59,61:#333	Lyophilized, native, single-donor
#349	53:#292, 55:#301,57:#313, 59:#323:RR59,61:#332	Fresh-frozen, native, single-donor
#350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



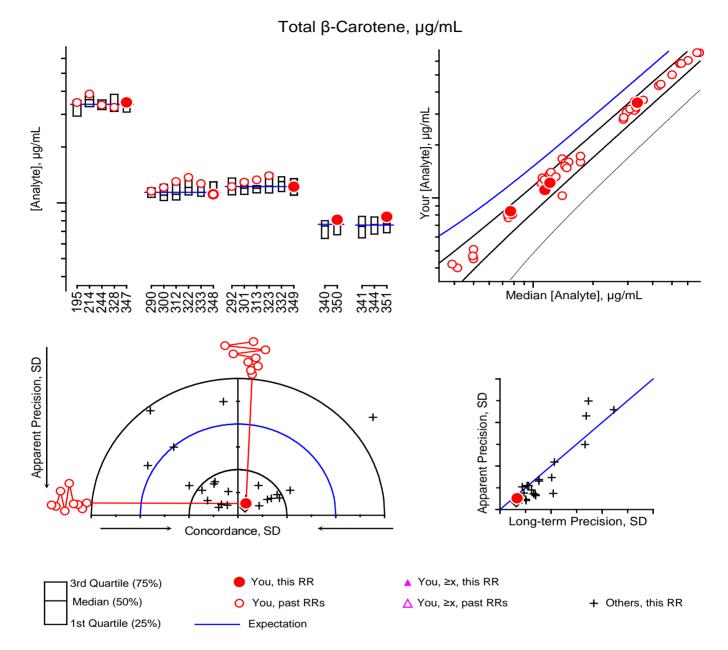
<u>Serum</u>	<u>History</u>	<u>Comments</u>
#347	31:#195, 35:#214, 43:#244, 60:#328	Lyophilized, multi-donor, aug(TR, RP,aT)
#348	53:#290, 55:#300,57:#312, 59:#322:RR59,61:#333	Lyophilized, native, single-donor
#349	53:#292, 55:#301,57:#313, 59:#323:RR59,61:#332	Fresh-frozen, native, single-donor
#350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



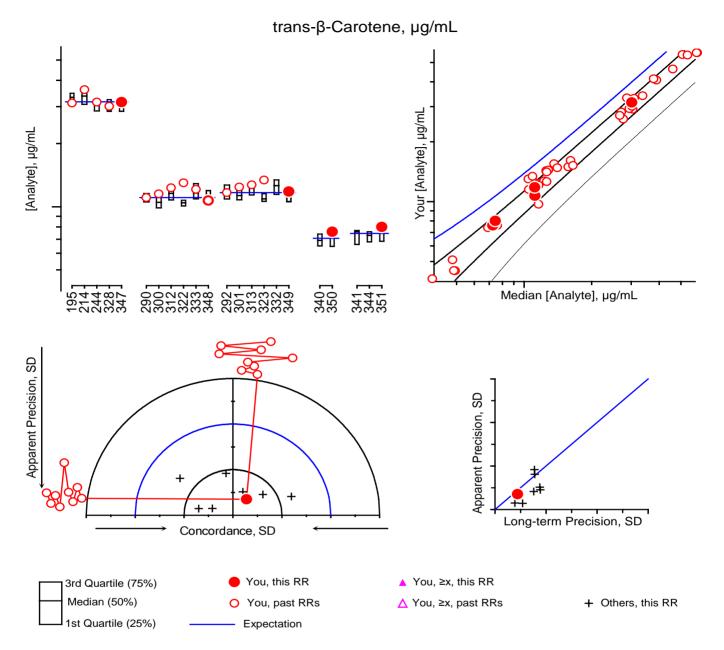
<u>Serum</u>	<u>History</u>	<u>Comments</u>
#347	31:#195, 35:#214, 43:#244, 60:#328	Lyophilized, multi-donor, aug(TR, RP,aT)
#348	53:#290, 55:#300,57:#312, 59:#322:RR59,61:#333	Lyophilized, native, single-donor
#349	53:#292, 55:#301,57:#313, 59:#323:RR59,61:#332	Fresh-frozen, native, single-donor
#350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



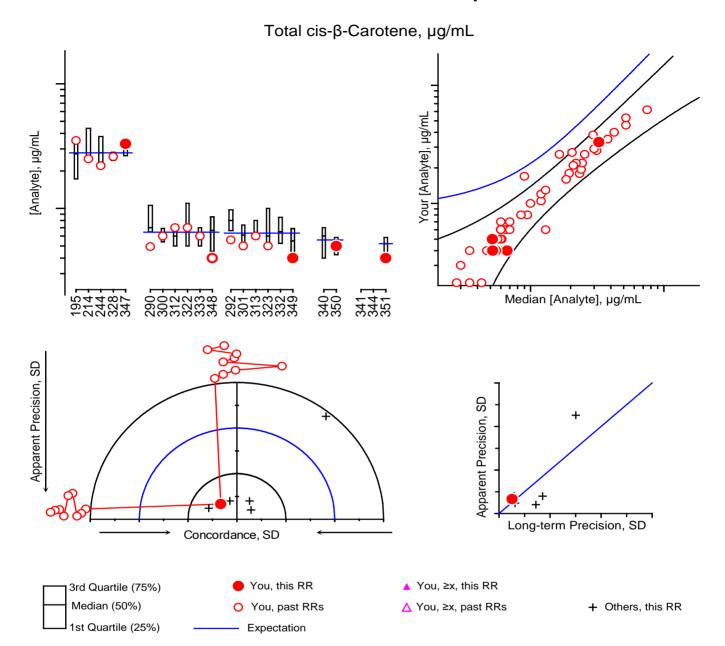
<u>Serum</u>	<u>History</u>	<u>Comments</u>
#347	31:#195, 35:#214, 43:#244, 60:#328	Lyophilized, multi-donor, aug(TR, RP,aT)
#348	53:#290, 55:#300,57:#312, 59:#322:RR59,61:#333	Lyophilized, native, single-donor
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#350	63:#340	Plasma, fresh-frozen, native, multi-donor
#351	63:#341, 63:#344	Fresh-frozen, native, multi-donor



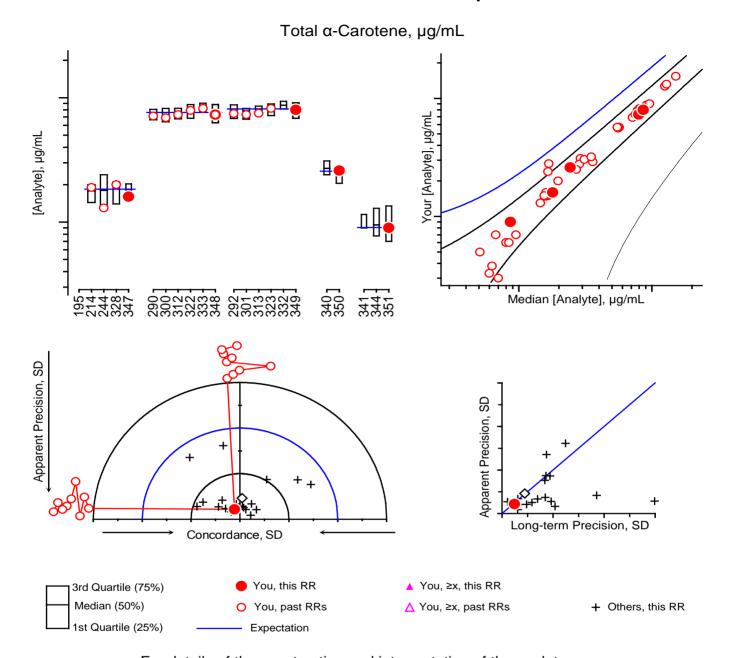
<u>Serum</u>	<u>History</u>	<u>Comments</u>
#347	31:#195, 35:#214, 43:#244, 60:#328	Lyophilized, multi-donor, aug(TR, RP,aT)
#348	53:#290, 55:#300,57:#312, 59:#322:RR59,61:#333	Lyophilized, native, single-donor
#349	53:#292, 55:#301,57:#313, 59:#323:RR59,61:#332	Fresh-frozen, native, single-donor
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#351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



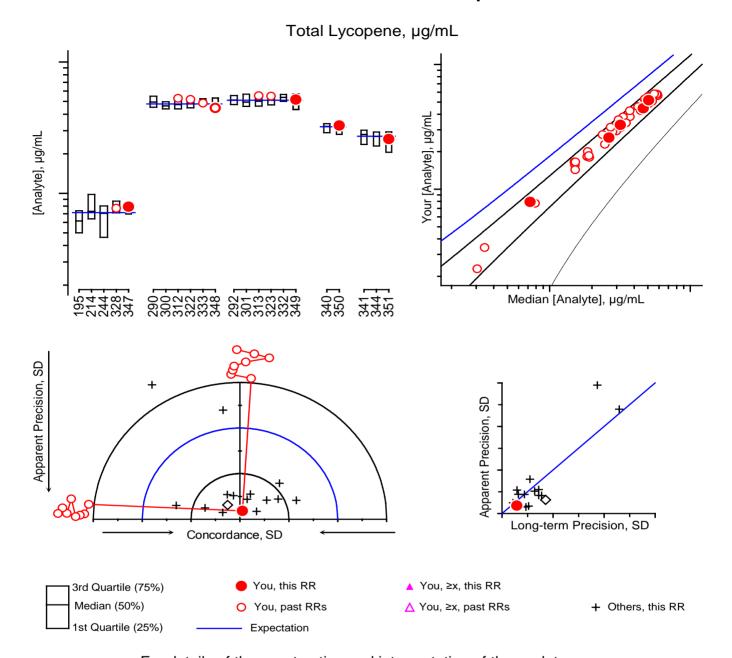
<u>Serum</u>	<u>History</u>	<u>Comments</u>
#347	31:#195, 35:#214, 43:#244, 60:#328	Lyophilized, multi-donor, aug(TR, RP,aT)
#348	53:#290, 55:#300,57:#312, 59:#322:RR59,61:#333	Lyophilized, native, single-donor
#349	53:#292, 55:#301,57:#313, 59:#323:RR59,61:#332	Fresh-frozen, native, single-donor
#350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



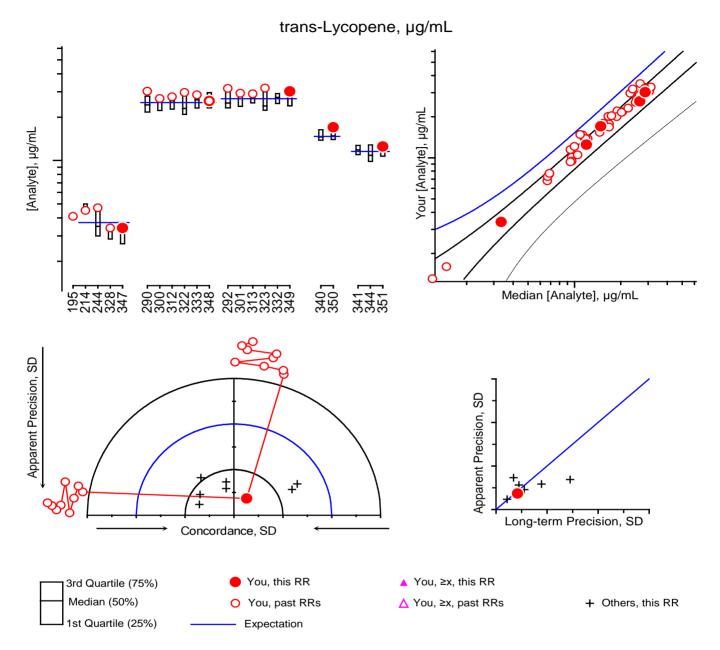
<u>Serum</u>	<u>History</u>	<u>Comments</u>
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#350	63:#340	Plasma, fresh-frozen, native, multi-donor
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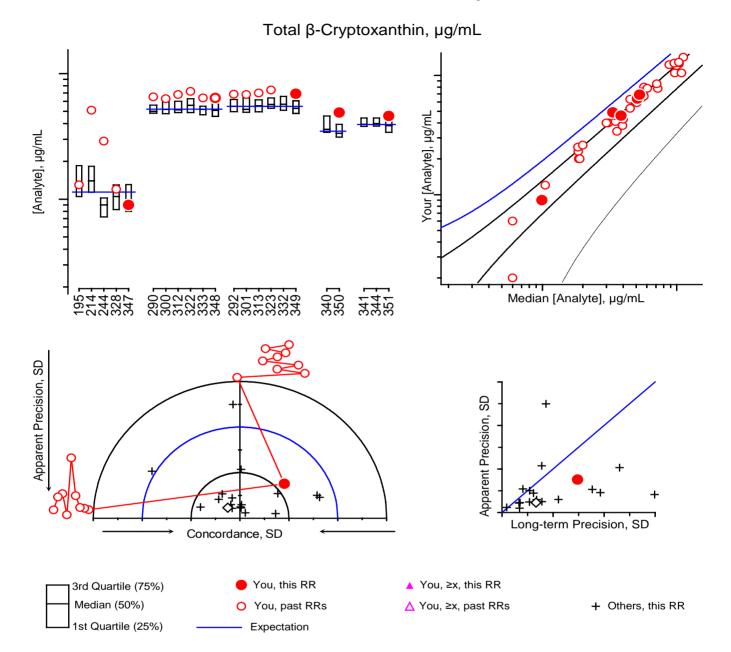


<u>Serum</u>	<u>History</u>	<u>Comments</u>
#347	31:#195, 35:#214, 43:#244, 60:#328	Lyophilized, multi-donor, aug(TR, RP,aT)
#348	53:#290, 55:#300,57:#312, 59:#322:RR59,61:#333	Lyophilized, native, single-donor
#349	53:#292, 55:#301,57:#313, 59:#323:RR59,61:#332	Fresh-frozen, native, single-donor
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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

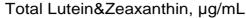
Individualized RR LXIV Report: FSV-BA

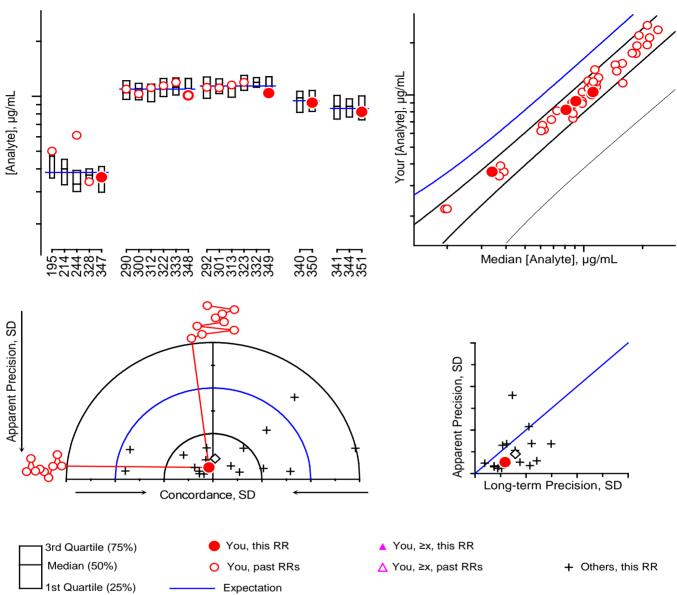


For details of the construction and interpretation of these plots, see: Duewer, Kline, Sharpless, Brown Thomas, Gary, Sowell. Anal Chem 1999;71(9):1870-8.

<u>Serum</u>	<u>History</u>	<u>Comments</u>
#347	31:#195, 35:#214, 43:#244, 60:#328	Lyophilized, multi-donor, aug(TR, RP,aT)
#348	53:#290, 55:#300,57:#312, 59:#322:RR59,61:#333	Lyophilized, native, single-donor
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Individualized RR LXIV Report: FSV-BA





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

<u>Serum</u>	<u>History</u>	<u>Comments</u>
#347	31:#195, 35:#214, 43:#244, 60:#328	Lyophilized, multi-donor, aug(TR, RP,aT)
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#350	63:#340	Plasma, fresh-frozen, native, multi-donor
#351	63:#341, 63:#344	Fresh-frozen, native, multi-donor

Coenzyme Q10

Total β-Cryptoxanthin

trans-β-Carotene

Individualized Report

Set 1 of 40

Appendix E. Shipping Package Inserts for RR29

The following five items were included in each package shipped to an RR29 participant:

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

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



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

May 12, 2008

Dear Colleague:

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

RR29 consists of four vials of frozen serum test samples (#15, #73, #90, and #111), one vial of frozen control serum (CS #2) and one vial of ascorbic acid solid control material (Control) Please follow the attached protocols when you prepare and analyze these samples. If you cannot prepare the solid control solutions gravimetrically, please prepare equivalent solutions volumetrically and report the exact volumes used. (Routine 0.5 g gravimetric measurements are generally 10-fold more accurate than routine 0.5 mL volumetric measurements.)

Please use the control serum to validate the performance of your measurement system <u>before</u> you analyze the *test samples*. The target value and \approx 95% confidence interval for target value and \approx 95% confidence interval for CS #2 is 28.1 \pm 1.0 μ mol/L of sample.

The report for RR28 was mailed May 2, 2008. If you find your results for RR28 unsatisfactory, we recommend that you obtain **Standard Reference Material (SRM) 970 Ascorbic Acid in Serum** to validate your methodology and value assign in-house control materials. This SRM may be purchased from the Standard Materials Reference Program at NIST (Tel: 301-975-6776, Fax: 301-948-3730, or e-mail: srminfo@nist.gov).

Please be aware that sample contact with any oxidant-contaminated surface (vials, glassware, etc.) may degrade your measurement system's performance (SA Margolis and È Park, "Stability of Ascorbic Acid in Solutions in Autosampler Vials", *Clinical Chemistry* **2001**, *47*(8), 1463-1464). You should suspect such degradation if you observe unusually large variation in replicate analyses.

If you have any questions or concerns about the Vitamin C Micronutrients Measurement Quality Assurance Program please contact Jeanice Brown Thomas at tel: 301-975-3120, fax: 301-977-0685, or e-mail: jbthomas@nist.gov.

We ask that you return your results for these RR29 samples before Sep 12, 2008. We would appreciate receiving your results as soon as they become available. Please use the attached form. Your results will be kept confidential.

Sincerely,

Jeanice Brown Thomas

Research Chemist

Analytical Chemistry Division

Chemical Science and Technology Laboratory

Enclosures: Protocols, Preparation and Analysis of Control Materials and Analysis of Test Samples

RR29 Report Form for Ascorbic Acid Solid Control Material Preparation

RR29 Report Form for Control Material and Test Sample Analyses



Micronutrient Measurement Quality Assurance Program for Vitamin C

Please Read Through Completely BEFORE Analyzing Samples

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

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

- 1) Prepare at least 500 mL of 5% mass fraction metaphosphoric acid (MPA) in distilled water. This solution will be referred to as the "Diluent" below.
- 2) Weigh 0.20 to 0.22 g of the ascorbic acid solid control material to 0.0001 g (if possible), dissolve it in the Diluent in a 100 mL volumetric flask, and dilute with the Diluent to the 100 mL mark. Weigh the amount of Diluent added to 0.1 g. Record the weights. The resulting material will be referred to as the "Stock Solution" below.
- 3) Prepare three dilute solutions of the Stock Solution as follows:
 - <u>Dilute Solution 1:</u> 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.
 - <u>Dilute Solution 2:</u> 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.
 - <u>Dilute Solution 3:</u> 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:

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

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

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:

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

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 <u>not</u> analyze the serum control materials or test samples until you are satisfied that your system is performing properly!

7) Once you have confirmed that your system is properly calibrated, analyze the serum control CS #2 (see protocol below). The target values for this materials is 28.1 ±1.0 µmol/L of sample. If your measured values are not close to this value, please review your sample preparation procedure and whether you followed *exactly* the same measurement protocol the solutions prepared from the solid control material as you used for these serum controls. If the protocols differ, please repeat from Step 6 using the proper protocol. If the proper protocol was used, your measurement system may not be suitable for MPA-preserved samples; please contact us at 301-975-3120 or jbthomas@NIST.gov.

Do <u>not</u> 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 <u>total ascorbic acid</u> should be reported. The *serum control material* and *test samples* should be defrosted by warming at 20 °C for not more than 10 min otherwise some irreversible degradation may occur.

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

Participant #:	Date:
----------------	-------

Vitamin C Round Robin 29

NIST Micronutrient Measurement Quality Assurance Program

Preparation and Validation of Ascorbic Acid Solid Control Material

STOCK SOLUTION

Mass of ascorbic acid in the Stock Solution	g
Mass of 5% MPA Diluent added to the 100 mL volumetric flask	g
DILUTE SOLUTION 1	
Mass of added stock solution (0.5 mL)	g
Mass of 5% MPA Diluent added to the 100 mL volumetric flask	g
Absorbance of Dilute Solution 1 at 242 nm	AU
Absorbance of Dilute Solution 1 at 243 nm	AU
Absorbance of Dilute Solution 1 at 244 nm	AU
Absorbance of Dilute Solution 1 at 245 nm	AU
Absorbance of Dilute Solution absorbance maximum	AU
Wavelength of maximum absorbance	nm
Calculated E ^{1%}	dL/g·cm
Calculated [TAA] _{DS1}	μmol/L
DILUTE SOLUTION 2	
Mass of added stock solution (0.25 mL)	g
Mass of 5% MPA Diluent added to the 100 mL volumetric flask	g
Calculated [TAA] _{DS2}	μmol/L
DILUTE SOLUTION 3	
Mass of added stock solution (0.125 mL)	g
Mass of 5% MPA Diluent added to the 100 mL volumetric flask	g
Calculated [TAA] _{DG2}	umol/L

Please return before 12-Sep-2008

Fax: 301-977-0685

Participant #:	Date:
----------------	-------

Vitamin C Round Robin 29 NIST Micronutrient Measurement Quality Assurance Program

Analysis of Control Materials and Test Samples

Sample	Replicate 1	Replicate 2	Units
Dilute Solution 1			μmol/L of Dilute Solution
Dilute Solution 2			μmol/L of Dilute Solution
Dilute Solution 3			 μmol/L of Dilute Solution
5% MPA Diluent			 μmol/L of Diluent
CS #2			μmol/L of Sample <i>Target</i> : 28.1 ±1.0 μmol/L
Serum Test Sample #15			μmol/L of Sample
Serum Test Sample #73			 μmol/L of Sample
Serum Test Sample #90			μmol/L of Sample
Serum Test Sample #111			μmol/L of Sample

Were samples frozen upon receipt? Yes | No

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

COMMENTS:

Fax: 301-977-0685

Vitamin C Round Robin 29 NIST Micronutrients Measurement Quality Assurance Program Packing List and Shipment Receipt Confirmation Form

This box contains one vial each of the following **six** VitC M²QAP samples:

Label	Form
VitC #15	Liquid frozen (1:1 serum:10% MPA)
VitC #73	Liquid frozen (1:1 serum:10% MPA)
VitC #90	Liquid frozen (1:1 serum:10% MPA)
VitC #111	Liquid frozen (1:1 serum:10% MPA)
CS #2	Liquid frozen (1:1 serum:10% MPA)
Control	Solid AA

- Please 1) Open the pack immediately
 - 2) Check that it contains one vial each of the above samples
 - 3) Check if the samples arrived frozen
 - 4) Store the samples at -20 °C or below until analysis
 - 5) Complete the following information
 - 6) Fax the completed form to us at 301-977-0685 (or email requested information to david.duewer@nist.gov)
- 1) Date this shipment arrived: _____
- 2) Are all of the vials intact? Yes | No If "No", which one(s) were damaged?
- 3) Was there any dry-ice left in cooler? Yes | No
- 4) Did the samples arrive frozen? Yes | No
- 5) At what temperature are you storing the samples? _____ °C
- 6) When do you anticipate analyzing these samples? _____

Your prompt return of this information is appreciated.

The M²QAP Gang

Appendix F. Final Report for RR29

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

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



UNITED STATES DEPARTMENT OF COMMERCE Mational Institute of Standards and Technology Geithersburg, Maryland 20899-

November 25, 2008

Dear Colleague:

Enclosed is the summary report of the results for Round Robin 29 (RR29) for the measurement of total ascorbic acid (TAA, ascorbic acid plus dehydroascorbic acid) in human serum. Included in this report are a summary of data for all laboratories and an individualized summary of your laboratory's measurement performance. The robust median is used to estimate the consensus value for all samples, the "median absolute deviation from the median" (MADe) is used to estimate the expected standard deviation, and the coefficient of variation (CV) is defined as 100×MADe/median.

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

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

Samples for the first vitamin C round robin (RR30) of the 2009 M²QAP will be shipped during the week of December 8, 2009. If you have questions or concerns regarding this report, please contact David Duewer at 301-975-3935; e-mail: david.duewer@nist.gov or me at 301-975-3120; e-mail: jbthomas@nist.gov; or fax: 301-977-0685.

Sincerely,

danice Brown Thomas

Research Chemist

Analytical Chemistry Division

Chemical Science and Technology Laboratory

Enclosures

Cc: L. C. Sander

D.L. Duewer



The NIST M²QAP Vitamin C Round Robin 29 (RR29) report consists of

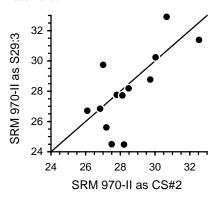
Page	"Individualized" Report
1	Summarizes your reported values for the nominal 55 mmol/L solution you prepared from the ascorbic acid solid control sample, the serum control sample, and the four serum test samples.
2	Graphical summary of your RR29 sample measurements.
Page	"All Lab" Report
1	A tabulation of results and summary statistics for Total Ascorbic Acid [TAA] in the RR29 samples and control/calibration solutions.

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

- CS#2 SRM 970 level 2, ampouled in mid-1998.
- S29:1 A "blank" stripped serum, ampouled in late 2001, previously distributed as sample S19:1 (RR19, 9/03), S21:1 (RR21, 9/04), S23:1 (RR23, 9/05), and S26:1 (RR26, 3/07).
- S29:1 SRM 970 level 1, ampouled in mid-1998.
- S29:2 SRM 970 level 2, ampouled in mid-1998.
- S29:4 Serum 111, ampouled in 1989. It was used in some early experiments but was first distributed in the current MMQAP as S25:3 (RR25, 9/06).

Results.

- 1) Nearly all participants who prepared the four 5% MPA control/calibration solutions (the three "Dilute Solutions" and the "Diluent") did so correctly. The criteria used to evaluate this success are: the density of the 5% MPA (≈1.03 gm/mL), the observed wavelength maximum of "Dilute Solution #1"(≈244 nm), the observed absorbance at that maximum (≈0.58 OD), the calculated E¹% #1"(≈560 dL/g·cm). On the evidence of MPA density, one participant prepared the solutions in 0% MPA.
- 2) The Measured = a+b*Gravimetric calibration parameters for the control/calibration solutions (columns 10 to 13 of the All Lab Report) indicate that the measurement systems for all participants are linear (R² close to 1 and RMS close to 0.0) and reasonably well calibrated (intercepts range from -1.2 to 1.5 and slopes range from 0.88 to 1.04).
- 3) The Measured = p+q*Median regression parameters for samples S29:1 to S29:4 (columns 23 to 26 of the All Lab Report) mostly confirm the linearity of the measurement systems (R² close to 1 and RMS close to 0.0). As in RR28, there appears to be no correlation between regression intercepts and slopes and those for gravimetric calibration, again implying the existence of serum matrix effects.
- 4) There is no evidence of sample degradation in either of the SRM 970 materials.
- 5) There is little evidence for any "attraction to the known-value" in the analysis of the control material. However, comparison of the SRM 970-II material analyzed as CS#2 and as S29:3 (see Youden plot to the right) provides direct evidence that most of the observed among-participant differences in reported value result from systematic biases among the measurement systems.



Appendix G. "All-Lab Report" for RR29

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

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

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

Micronutrients Measurement Quality Assurance Program for Total Ascorbic Acid "Round Robin" 29 - September 2008

	an	RMS	4.	0.2	1.0	1.8	1.1	9.0	6.0	1.6	1.9	1.1	6.0	1.8	3.0											
	p + q*Median	\mathbb{R}^2 RI	0.988	1.000	966.0	0.983	0.994	0.998	966.0	0.987	926.0	0.993	0.994	0.980	0.953											
	Ш						1.02 0.9	1.00 0.9	1.02 0.9																	
	Measured	er Slope	74 0.92	15 0.97	57 1.04	30 0.96	•	-		78 0.97	14 0.87	36 0.92	33 0.87	11 0.87	26 0.96											
	Me	:4 Inter	13.0 0.74	11.1 -0.15	10.3 -0.57	12.3 2.60	10.0	12.2 0.16	10.2 -0.45	17.5 4.78	12.8 5.14	16.9 7.36	11.5 0.83	8.0 -0.11	6.2 -1.26	13	11.7	3.1	Ī	6.2	10.2	.5	12.8	7.	2.0	17
Samples		3 S29:4		•	•	-	-					•	•		7	13		2.5			-				2.9	Ξ
Sar	I/L	2 \$29:3	3 25.6	0 26.8	6 28.8					7 31.4		4 32.9		0 24.5	4 26.	13 ,	.,					7 27.8				,
	Measured, µmol/l	\$29:2				2 9.8) 8.7	11.7	•	2 16.4		0.6	3 7.			7 2.7			8.1		2 9.8	2 16.4	0.1	_
	/leasure	S29:1	0.0	0.0	0.0		0.0	0.3	0.0	4.9	5.7	7.2		0.0	9.0	13	1.8				0.0			7.2		
	V	S29:1	0.0	0.0	0.0	4.2	0.0	0.3	0.0	4.9	5.7	7.2	0.0	0.0	9.0	13	1.8	2.7	,	0.0	0.0	0.0	4.2	7.2	0.0	
		CS#2	27.2	26.8	29.7	30.0	28.1	27.8	28.5	32.5	27.0	30.7	28.2	27.5	26.1	13	28.5	1.8		26.1	27.2	28.1	29.7	32.5	1.6	9
_	etry	E1%	553.2	542.1	551.8	590.3	555.3	6.799		342.3ª	551.0	526.4				80	554.8	18.6		526.4	548.8	552.5	528.5	590.3	9.8	1.8
Dilute Solution 1	Spectrophotometry	A _{max}	0.5670	0.5640	0.5460	0.5955	0.5980	0.6130		0.356 ^a 3.		0.5600				80	0.5788	0.0229		0.5460	0.5630	0.5768	0.5961	0.6130	0.0263	4.6
Dilute (Spectrop						243.8 0.5	244.1 0.6				243.7 0.5				80	3.6 0.5	0.5 0.0		243.0 0.5	243.0 0.5	243.8 0.5	244.0 0.5	244.1 0.6	0.4 0.0	0.17
		λmax	_	9 244	9 243.				7	7 254 ^a	9 244.								L							
MPA	Density	g/mL	1.036	1.029	1.029	1.031	1.029	1.032	1.032	1.027	0.999	1.038				10	1.028	0.011		0.999	1.029	1.030	1.032	1.038	Ö	0.25
	irav	RMS	9.0	9.0	0.8	1.3	4.1	0.2	1.0	0.5	0.7	4.0				Z	Average	SD	-	Min	%25	Median	%75	Max	eSD	<u></u>
	a + b*Grav	\mathbb{R}^2	1.000	1.000	0.999	0.998	0.998	1.000	0.999	1.000	0.999	1.000					á					_				
	Ш	Slope	1.03	1.03	1.04	1.01	1.03	1.02	0.92	0.98	0.88	1.00														
nples	Measured	Inter	0.34	-0.27	-0.73	0.58	-1.16	-0.13	0.91	0.37	1.53	0.77														
on San	/٦	MPA	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	1.0	0.8				10	0.2	0.4		0.0	0.0	0.0	0.0	1.0	0.0	
alibrati	I, µmol	Dil:3	15.5	14.2	13.4	14.6	14.8	15.4	14.5	15.0	15.4	15.3				10	14.8	0.7		13.4	14.6	14.9	15.3	15.5	9.0	4
Control / Calibration Samples	Measured, µmol/l	Dil:2	30.8	30.5	28.8	31.2	31.2	30.7	27.9	28.7	28.5	31.0				10	29.9	1.3		27.87	28.74	30.55	30.92	31.25	1.0	က
Con	Me	Dil:1		2.09	58.2	57.6	62.2	62.4	53.1	58.1	54.7	6.09				10	58.8	3.1		53.1	57.8	59.1	6.09	62.4	2.6	4
	J/L	Dil:3	14.8	14.6	14.3	14.2	17.0	15.3	13.9	14.8	14.9	14.9				10	14.9	0.8		13.9		14.8	14.9	17.0	0.4	3
	Grav, µmol/l	Dil:2	28.9	29.4	28.6	28.8	31.7	30.4	28.4	28.4	30.6	29.8				10	29.5	1.		28.39	28.69	29.12	30.24	31.71	1.0	က
	Gre	Dil:1	58.2	59.1	56.2	57.3	61.1	61.3	57.1	59.0	60.4	60.4					2	1.8			57.5	59.1	60.4	61.3	2.3	4
•		Date	11/08/08	20/06/08	20/06/08	19/09/08	21/08/08	19/06/08	10/09/08	22/08/08	12/06/08	80/60/80	10/09/08	25/06/08	27/08/08	Z	Average	SD	-	Min	%25	Median	%75	Max	MADe	C C
		Lab	VC-MA 1	VC-MB 2	VC-MC 2	VC-ME 1		VC-MH 1	VC-MI 1	VC-MJ 2	VC-MK 1	VC-MN 0	VC-MP 1	VC-MS 2	VC-MU 2		•									
		_	>	>	>	>	Ś	Ś		>	>	Ś	>	>	>											

Appendix H. Representative "Individualized Report" for RR29

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

03/09/06

08/28/06

24

25

HPLC-EC

HPLC-EC

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

Dilute Solution 1

Spectrophotometry

 $\boldsymbol{A}_{\text{max}}$

242.0 0.555 557.4

244.0 0.568

E^{1%}

586.7

Control/Calibration Solutions

 $Y_{meas} = Inter + Slope^* X_{grav}$

Inter Slope

1.13

0.95

0.2

8.0

 R^2

1.000

0.999

SEE

0.41

0.92

MPA

Density

g/mL

1.031

1.039

Method

08/28/06	25	HPLC-EC		1.039		242.0	0.555		0.8		0.999	0.92
03/20/07	26	HPLC-EC		1.033			0.573		0.3		1.000	0.31
10/05/07	27	HPLC-EC		1.032		242.0			-0.1		1.000	0.14
03/04/08	28	HPLC-EC		1.035		243.0			0.7		0.999	0.99
08/11/08	29	HPLC-EC		1.037		243.0		553.2	0.3	1.03	1.000	0.64
			Mean	1.035		243.0	0.57	561.8				0.57
			SD	0.003		0.9	0.01	12.6				0.34
			CV	0.28		0.37	1.2	2.2				
				r	ΓΔΔ1 m	mol/Ls	amnle					
Date	RR	Sample		Rep₁	Rep ₂	F _{adj}	Mean	SD_dup	N	Mean	SD_{repeat}	SD_{reprod}
03/08/05	22	CS#2		29.0	29.0	1.0	29.0	0.0	6		0.5	1.1
10/17/05	23	CS#2		29.4	30.5	1.0	30.0	0.0	0	20.4	0.5	1.1
03/09/06	24	CS#2		29.4	29.1	1.0	29.2	0.0				
08/28/06	25	CS#2		27.2	28.1	1.0	27.6	0.6				
10/05/07	27	CS#2		28.1	27.4	1.0	27.7	0.5				
08/11/08	29	CS#2		27.2	27.2	1.0	27.2	0.0				
00/11/00		00112	l	2112		1.0		0.0				
11/18/02	16	S16:2		0.0	0.1	1.0	0.0	0.0	4	0.0	0.0	0.0
11/13/03	19	S19:1		na	na	1.0			-			
09/13/04	21	S21:1		na	na	1.0						
10/17/05	23	S23:1		0.0	0.0	1.0	0.0	0.0				
03/20/07	26	S26:1		0.0	0.0	1.0	0.0	0.0				
08/11/08	29	S29:1		0.0	0.0	1.0	0.0	0.0				
			-									
11/18/02	16	S16:1		8.8	8.8	1.0	8.8	0.0	6	8.5	0.2	0.3
11/13/03	19	S19:4		7.8	8.6	1.0	8.2	0.5				
02/23/04	20	S20:3		8.3	8.1	1.0	8.2	0.1				
10/17/05	23	S23:4		8.6	8.8	1.0	8.7	0.1				
08/28/06	25	S25:1		8.7	8.5	1.0	8.6	0.2				
08/11/08	29	S29:2		8.3	8.4	1.0	8.3	0.1				
		_	Ī									
03/20/03	18	S18:3		28.8	29.2	1.0	29.0	0.3	5	27.4	0.3	1.8
02/23/04	20	S20:4		25.9	25.2	1.0	25.5	0.5				
03/08/05	22	S22:4		29.4	29.4	1.0	29.4	0.0				
08/28/06	25	S25:2		27.6	27.4	1.0	27.5	0.1				
08/11/08	29	S29:3		25.6	25.7	1.0	25.6	0.1				
08/28/06	25	S25:3		12.3	12.4	1.0	12.4	0.1	2	12.7	0.2	0.5
08/11/08	29	S29:4		12.8	13.3	1.0	13.0	0.3			J.L	0.0
			ı					3.9				

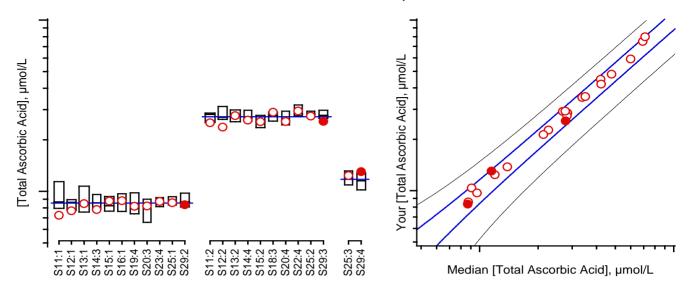
Please check our records against your records. Send corrections and/or updates to...

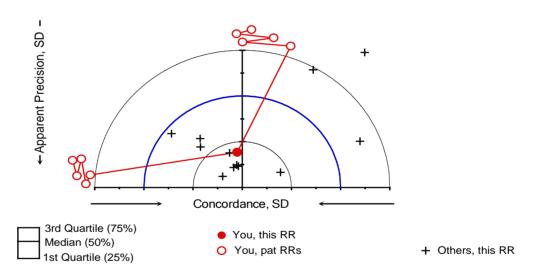
Micronutrients Measurement Quality Assurance Program National Institute of Standards and Technology 100 Bureau Drive Stop 8392 Gaithersburg, MD 20899-8392 USA

Fax: (301) 977-0685 Email: david.duewer@nist.gov

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

Total Ascorbic Acid, µmol/mL





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

<u>Sample</u> <u>Comments</u>

S29:1 VitC #15 (not displayed), serum blank previously distributed in RRs 16, 19, 21, 23, and 26

S29:2 VitC #73, previously distributed in RRs 11, 12, 13, 14, 15, 16, 19, 20, 23, and 25

S29:3 VitC #90, previously distributed in RRs 11, 12, 13, 14, 15, 18, 20, 22, and 25

S29:4 VitC #111, previously distributed in RR 25