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NIST Micronutrients Measurement Quality Assurance Program Summer 2005 Comparability Studies

Results for Round Robin LVIII Fat-Soluble Vitamins and Carotenoids in Human Serum and Round Robin 23 Ascorbic Acid in Human Serum

David L. Duewer Jeanice B. Thomas

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David L. Duewer Jeanice B. Thomas Chemical Sciences Division Materials Measurement Laboratory

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U.S. Department of Commerce *Cameron F. Kerry, 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 2005 MMQAP measurement comparability improvement studies: 1) Round Robin LVIII Fat-Soluble Vitamins and Carotenoids in Human Serum and 2) Round Robin 23 Total Ascorbic Acid in Human Serum. The materials for both studies were shipped to participants in May 2005; participants were requested to provide their measurement results by September 12, 2005.

Keywords

Human Serum Retinol, α-Tocopherol, γ-Tocopherol, Total and *Trans*-β-Carotene SRM 3276 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 LVIII: Fat-Soluble Vitamins and Carotenoids in Human Serum

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin LVIII comparability study (hereafter referred to as RR58) received two ampoules of SRM 3276 Carrot Extract in Sunflower Oil, two lyophilized human serum test samples, 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 2005. The "carrot oil" ampoules were shipped in the same package, but isolated from the dry ice. 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 RR58 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. Anonymized results for the "carrot oil" are also included. 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 23: Vitamin C in Human Serum

Participants in the MMQAP Vitamin C in Human Serum Round Robin 23 comparability study (hereafter referred to as RR23) received four frozen serum test samples, two frozen control sera, 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 2005. 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 RR23 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 RR58

The following four items were included in each package shipped to an RR58 participant:

- Cover letter
- Datasheet for SRM 3276 carrot Extract in Sunflower Oil
- Datasheet for serum samples
- Packing List and Shipment Receipt Confirmation Form

The cover letter and 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.



Dear Colleague:

Enclosed are two sets of samples for the second fat-soluble vitamins and carotenoids in serum round robin study (Round Robin LVIII) for the fiscal year (FY) 05 NIST Micronutrients Measurement Quality Assurance Program. The first set consists of two ampoules of Standard Reference Material (SRM) 3276 Carrot Extract in Sunflower Oil, which is being prepared as part of a collaborative effort among NIST, the Food and Drug Administration (FDA) and the National Institutes of Health's Office of Dietary Supplements (NIH/ODS). This material should be refrigerated upon receipt. The material was prepared by extracting carrots into sunflower oil and readily dissolves in ethanol. The "carrot oil" was spiked with 2,6-di-tert-butyl-4-methylphenol (BHT) to a concentration of approximately 670 mg/kg. SRM 2376 was ampouled under argon in amber ampoules. Each ampoule contains approximately 1 mL of material. This material is very viscous, therefore we recommend that you weigh out your sample for analysis rather than relying on an accurate volumetric reading from a pipet. Your results should therefore be best expressed as mass fractions (ug/g).

Please analyze a test portion from each of the two ampoules. We would like you to measure α -carotene, β -carotene (total, trans, and any other isomers that you are able to quantify), and tocopherols in this material. Also, feel free to report values for any other carotenoids that you see. When reporting your results, please submit one value for each analyte from each ampoule. Also, please submit a chromatogram of your carrot oil analyses and specify the chromatographic conditions used.

The second set (Sera 314 – 318) consists of one vial of each of three liquid-frozen and two lyophilized 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 12, 2005. Results received more than two weeks after the due date will not be included in the summary report for this round robin study. The feedback report concerning the study will be distributed in October.

Lyophilized samples should be reconstituted with 1.0 mL of HPLC-grade water or equivalent. Before reconstitution, samples should be allowed to stand at room temperature under subdued light until thawed. 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 (E 1% 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 for Round Robin LVIII and the carrot oil study to:

Micronutrients Measurement Quality Assurance Program NIST 100 Bureau Drive Stop 8392 Gaithersburg, MD 20899-8392 Fax: (301) 977-0685

NST

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

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

Round Robin LVIII: Carrot Oil NIST Micronutrients Measurement Quality Assurance Program

| Analyte | Ampoule 1 | Ampoule 2 | Units* |
|----------------------------|-----------|-----------|------------------|
| α -tocopherol | | | |
| γ/β -tocopherol | | | |
| δ -tocopherol | | | |
| total β-carotene | | | |
| trans-β-carotene | | | |
| total cis-β-carotene | | | |
| 9-cis-β-carotene | | | |
| 13-cis-β-carotene | | | |
| 15-cis-β-carotene | | | |
| total α -carotene | | | |
| trans- α -carotene | | | |
| Other measurands? | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | |
| | | | * we prefer μg/g |
| Method: | | | |
| | | | |
| | | | |

Comments:

Please attach representative chromatogram(s)!

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

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

| Analyte | 314 | 315 | 316 | 317 | 318 | Units* |
|-------------------------------|-----|-----|-----|-----|-----|--------|
| total retinol | | | | | | |
| trans-retinol | | | | | | |
| didehydroretinol | | | | | | |
| retinyl palmitate | | | | | | |
| lpha-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 (K₁) | | | | | | |
| 25-hydroxyvitamin D | | | | | | |
| Other measurands? | | | | | | |
| | | | | | | |
| | | | | | | |

 * we prefer $\mu g/mL$

Were the liquid frozen samples frozen when received? Yes | No

Comments:

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

Packing List and Shipment Receipt Confirmation Form

This box contains: two ampoules of **carrot oil** one vial each of the following five FSV M2QAP **sera**

| Serum | Form | Reconstitute? |
|-------|---------------|-----------------------------|
| #314 | Liquid frozen | No |
| #315 | Liquid frozen | No |
| #316 | Liquid frozen | No |
| #317 | Lyophilized | Yes (1 ml H ₂ O) |
| #318 | Lyophilized | Yes (1 ml H ₂ O) |

- Please 1) Open the pack immediately
 - 2) Check that it contains all of the above samples
 - 3) Check if the ampoules and vials are intact
 - 4a) Store the carrot oil at 4 °C until analysis
 - 4b) 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 both carrot oil ampoules and 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 sera arrive frozen? Yes | No
- 5a) At what temperature are you storing the **carrot oil** ampoules? °C
- 5b) 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

Mail: M²QAP

NIST, Stop 8392 Gaithersburg, MD 20899-8392

Appendix B. Final Report for RR58

The following eight 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 20898-

November 8, 2005

Dear Colleague:

Enclosed is the summary report of the results for round robin LVIII (RR58) of the 2005 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 comparabilities relative to the NIST assigned values. The NIST-assigned values are equally weighted means of the medians from this interlaboratory comparison exercise and the means from the analyses performed by NIST.

Data for evaluating laboratory performance in RR58 are provided in the comparability summary (Score Card) on page 6 of the All Lab Report. Laboratory comparability is summarized as follows: 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.

If you have concerns regarding your laboratory's performance, we suggest that you obtain and analyze a unit of SRM 968c, Fat-Soluble Vitamins, Carotenoids, and Cholesterol in Human Serum. If your measured values do not agree with the certified values, we suggest that you contact us for consultation.

Samples for the first 2006 QA interlaboratory exercise will be shipped starting the last week of November 2005. We will send you a reminder via e-mail or fax a week prior to shipment. It is critical that you carefully inspect all samples upon arrival and that you promptly confirm to us that they have arrived. We will replace samples (lost or damaged in shipment or miss-packaged by us) only for participants who report the problem within one calendar week after the package arrives.

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

Enclosures



The NIST M²QAP Round Robin LVIII (RR58) report consists of:

| Page | "All Lab" Report |
|-----------|--|
| 1-4 | A listing of all results and statistics for analytes reported by at least two laboratories. |
| 5a 5b | A list of results for the analytes reported by only one laboratory. A legend for the above two lists. |
| 6 | The text version of the "Comparability Summary" (or "Score Card"). |
| 7 | "Carrot Oil" results: mean values for all analytes reported by any participant. |
| Page | "Individualized" Report |
| 1 | Your values, the number of labs reporting values, and our assigned values. |
| 2 to n | "Four Plot" summaries of your current and past measurement performance, one page for each analyte you report that is also reported by at least 8 other participants. |
| n+1 | The "target" plot version of your "Comparability Summary" scores. |

Samples. The five sera below were distributed in RR58.

| Serum | Description | Prior Distributions |
|-------|---|--|
| 314 | Fresh-frozen, native, multi-donor serum prepared in 2005. This serum was expected to have moderate to high levels of many analytes. | None |
| 315 | Fresh-frozen, native, multi-donor serum prepared in 2005. This serum was prepared as a 77:110 volumetric blend of sera #314:#316. | None |
| 316 | Fresh-frozen, native, multi-donor serum prepared in 2005. This serum was expected to have low levels of most analytes. | None |
| 317 | Lyophilized blended serum, a 1:1 blend of the pools used to produce SRM 968c Levels I and II. | #248 RR44 (9/98) |
| 318 | Lyophilized blended serum with native carotenoid levels, augmented with <i>trans</i> -retinol and γ/β-tocopherol; SRM 968c Level I. | #248 RR44 (9/98), #258 RR46 (6/99), #263 RR47 (5/00), #280 RR51 (3/02), #304 RR46 (9/04) |

Results

- 1) <u>Sera Stability:</u> There was no significant change in the median level nor increase in the variability of any measurand in either sera #317 nor #318.
- 2) Relative Accuracy: Sera #314, #315, and #316 were prepared to evaluate the relative accuracy of your assays. Serum #314 was prepared from a serum pool known to have relatively high levels of many analytes. Serum #316 was prepared from a serum pool known to have relatively low levels of most analytes. Serum #315 was prepared as a 77:110 volumetric blend of the two pools. If your assay imprecision is small and the response is linear over the range from the low level of #316 to the

M²QAP Fat-Soluble Vitamin: Study LVIII Feedback

high level of #314, assuming that the blend is equal to the sum of its parts, then the result for #315 should be $77/187 \times \#314 + 110/187 \times \#316$. Likewise, serum #317 was prepared as a 1:1 volumetric blend of the pools used to produce SRM 968c Levels I (serum #318) and II (serum #309 in RR57). With the same caveats as above (and with greater uncertainty since serum #309 was analyzed in the previous RR), the result for #317 should be $1/2 \times \#309 + 1/2 \times \#318$.

The median values for *nearly* all analytes follow the expected behavior with both blended sera. For total β -carotene and total α -carotene, the observed medians for the blends are a bit above the line connecting the respective endpoints; however, they are definitively in-between the endpoints. However, the behavior of γ/β -tocopherol is unexpected: the median value for #315 is larger than either #314 or #316.

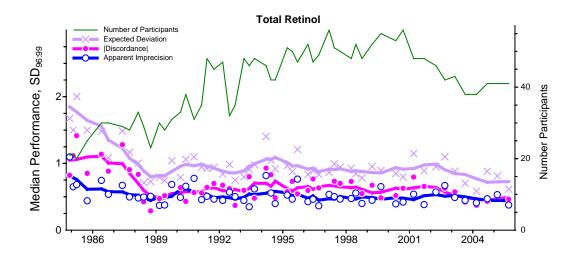
While the level of γ/β -tocopherol is relatively low in all three of these sera, it is well above the level where detection problems are expected. We obtain identical results with fluorescence detection as with absorbance and with peak height as with peak area, so it is unlikely to be an artifact of incomplete chromatographic separation or integration. We have also resolved the two isomers on a high-performance column and found that the ratio between the γ - and β -isomers is about the same in the two endpoints... and that *both* isomers are higher in #315 than in either endpoint. It really does look like the blending process either made the γ/β more readily extractable or that the relatively high level of α -tocopherol in #314 somehow suppresses the extraction of the γ/β . At any rate, we're puzzled. Other ideas and suggestions are most welcome.

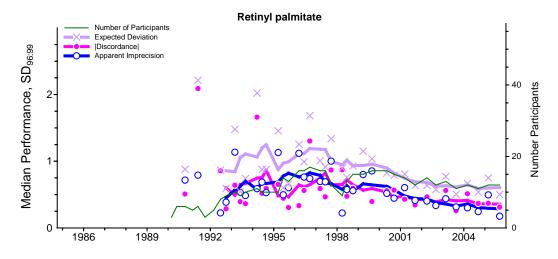
3) Community Measurement Performance Characteristics: The graphs on the following four pages summarize the among-participant concordance, expected apparent precision, expected overall comparability, and number of participants reporting values for all of the "generally-reported" analytes. These summary graphs are described in detail in: Duewer DL, Kline MC, Sharpless KE, Brown Thomas J. NIST Micronutrients Measurement Quality Assurance Program: Characterizing the measurement community's performance over time. Anal Chem 2000;72(17):4163-4170.

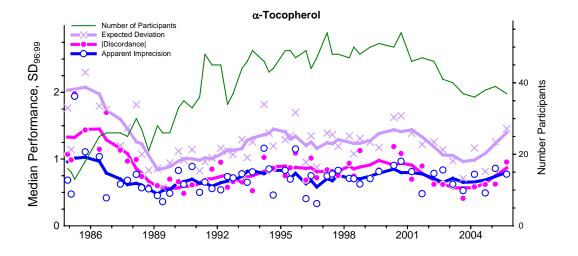
The following table summarizes our interpretation of the information in these graphics:

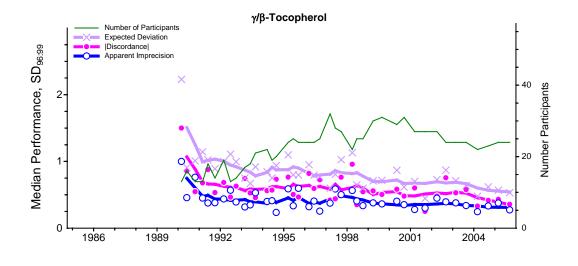
| Analyte | Comparability Relative to 2003 |
|-------------------------|--------------------------------|
| Total Retinol | Holding to slight improvement |
| Retinyl palmitate | Improving |
| α-Tocopherol | Declining after improvement |
| γ/β-Tocopherol | Holding to slight improvement |
| Total β-Carotene | Holding to slight decline |
| Total α-Carotene | Holding |
| Total Lycopene | Holding to slight decline |
| trans-Lycopene | Holding |
| Total β-Cryptoxanthin | Holding to slight decline |
| Total Lutein | Holding to slight improvement |
| Total Zeaxanthin | Holding |
| Total Lutein&Zeaxanthin | Holding |

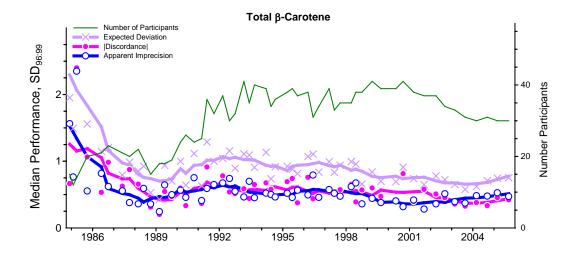
We intend to investigate possible causes for the loss of α -tocopherol comparability in the coming year.

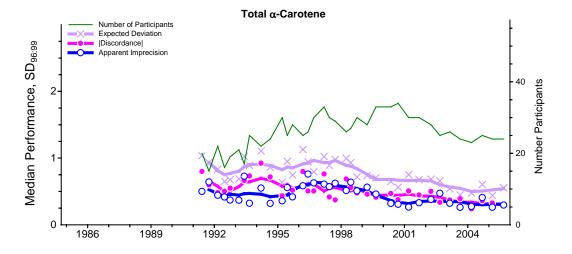


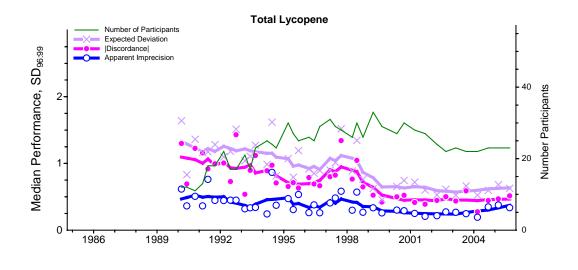


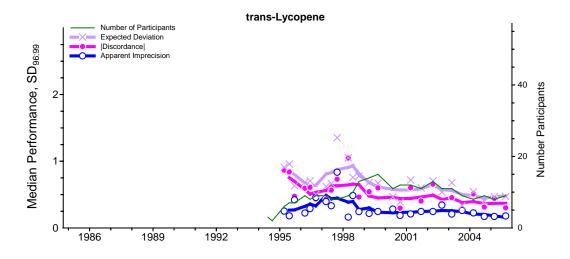


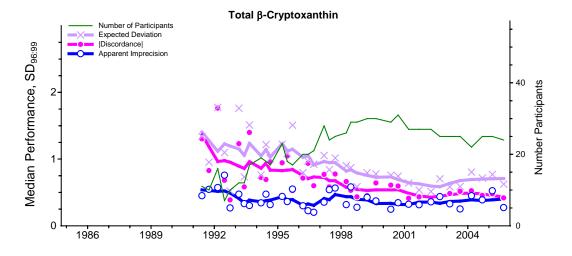


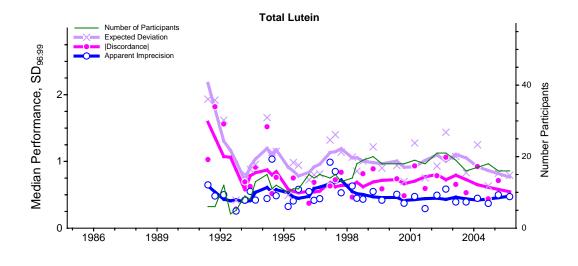


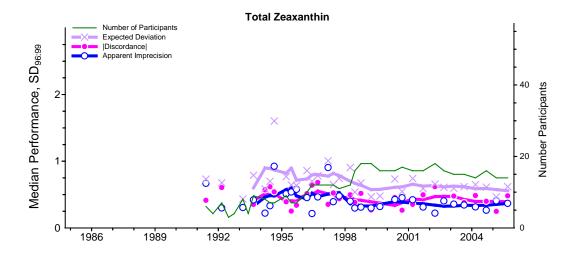


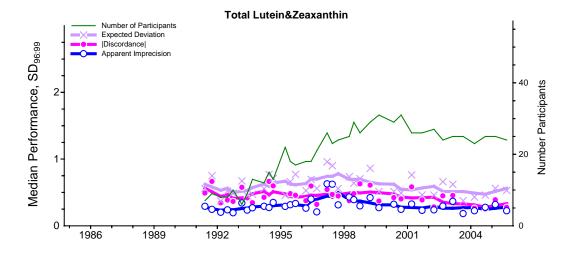












M²QAP Fat-Soluble Vitamin: Study LVIII Feedback

4) <u>Carrot oil observations/conclusions</u>: We appreciate the effort that all of you who evaluated the "carrot oil" ampoules put into your analyses! About half of you provided some results or reported that no results could be obtained using available methods.

We chose not to provide specific instructions for the analysis of this material to prevent biasing your results. We wanted to get an idea of the handling and measurement capabilities for this matrix in the hands of experienced analysts. Unsurprisingly, those of you who also participate in our "food" SRM value assignment studies reported more complete sets of results than those who do not. We were somewhat surprised that more of you did not investigate dissolving the material in ethanol (or whatever alcohol is most compatible with your chromatographic liquid phase). This simplistic approach generally has worked well for us.

Also surprisingly, the among-participant variation for the carotenoids was considerably smaller than for the tocopherols. We believe much of the scatter in the tocopherol results arises from incomplete chromatographic (baseline) separation... and the unusual and unexpected isomer ratios: only a trace of α -tocopherol level is present while both the δ - and γ -tocopherols are about 400 μ g/g. We strongly suspect that the carrier for the carrot-extract is **not** predominantly "sunflower oil" as stated by the supplier.

We intend to design a second study of this material, both to better characterize the material and to evaluate its potential for use as a calibrant. If we do ask you to evaluate this material again, we will provide detailed instructions.

Appendix C. "All-Lab Report" for RR58

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

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

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

Round Robin LVIII Serum Results All Values in µg/mL

| | | 0.120 | | | | | | 2 0.143 | | | | | | | | | | 0.124 | | 0108 | | 0 | bu s | 0.130 | | 3 0.005 | | | 0.010 | | 0.047 | 0.130 | 000 | 0.003 | |
|-------------------|---------|----------------------------|------------------------------|---------------------|-------------|-------|-----------------------|-------------|--------------|-----------|-------------|------------------------|-------|-------------|----------|----------------------|-------|------------------|-----------|---|-------|-------------|----------------------|-------------|-------|---------|-------------|-------------|----------------------|-------|----------------------------|-------------|---|-------------------------|-------------|
| ō-Tocopherol | 316 317 | 0.098 0.342 0.093 0.331 | | | | | | 0.132 0.312 | | | | | | | | | | 0.124 0.411 | | 0.086.0.419 | 50000 | 0.100 0.276 | nq 0.156 | 0.104 0.370 | | 7 8 | | 0.132 0.419 | 0.014 0.05/ 14 17 | 0 | 0.402 | 0.102 0.331 | 2 2 | 0.008 0.010 0.011 | 0 101 0 334 |
| δ-Toc | 315 3 | 0.113 0.0 0.103 0.0 | | | | | | 0.116 0.1 | | | | | | | | | | 0.128 0.1 | | 0 115 0 | 2 | 2 | u bu | 0.114 0.1 | | 7 | 0.115 0.1 | 0.128 0.1 | 0.006 5 | 0 | | 0.107 0.1 | 2000 | | 0 111 0 1 |
| | | 0.109 0 0.095 0 | | | | | | 0.133 0 | | | | | | | | | | 0.153 0 | | 0.101 | | 4 | bu | 0.102 0 | | 7 | 0.102 0 | 0.153 0 | 0.017 0 | 0 | | 0.105 0 | 2 0 010 0 | 0.006 0.012 0.012 | 0.103.0 |
| | 318 | 3.94 | 0 4.20 | | 7 3.53 | | | 6 3.30 | | | 9 5.71 | | | 5 4.78 | | 3.68 | | 7 3.85 3 3.86 | | 9 | 9 | 1 3.88 | | 1 3.67 | | 3 23 | | | 5 0.32 5 8 | | 6 3.83 2 0.34 | 4.00 | 2 2 | 0.08 | 3 07 |
| γ/β-Tocopherol | | 1.75 2.69 1.63 2.61 | 1.70 2.8 | 1.75 2.75 2.37 3.70 | | | | 1.44 2.26 | | | | 1.73 2.59 1.67 2.61 | | 1 74 2 75 | | 1.59 2.50 | | 1.67 2.87 | 2.42 2.9 | 175 274 | | 1.76 2.61 | | 1.61 2.51 | | 23 23 | | | 0.09 0.14 5 5 | 0 23 | 2.76 0.22 | 1.72 2.73 | 2 2 | 0.03 0.02 0.06 0.06 | 173 273 |
| γ/β-Το | 315 | 1.84 | 1.70 | 1.74 | 1.71 | 7/- | | 1.46 | | | 1.45 | 1.80 | 1.89 | 2.13 | 2 | 1.70 | | 1.74 | 2.28 | 1 70 | 2 | 1.81 | 2.01 | 1.66 | | 23 | 1.79 | 2.43 | 7. 8 | 0 | | 1.82 | 2 2 | 0.03 | 184 |
| | 브 | 7.20 1.50 7.71 1.32 | 8.00 8.40 7.80 1.30 | | 6.53 1.48 | | 7.32 | 7.09 1.18 | 6.85 7.51 | 6.10 | | 7.79 1.40 | | 7 82 1.69 | | 7.28 1.41 | | | 8.70 1.98 | 8 8 4 B 5 8 5 8 5 8 5 8 5 8 5 8 5 8 5 8 5 8 5 | | 7.31 1.42 | | 7.37 1.31 | _ | 36 23 | | | 8 U.T.S | 45 0 | 7.49 0.66 | 7.52 1.43 | 2 2 | | 7 54 1 46 |
| <u> </u> | | 11.17 7.: 12.10 7. | 12.00 8. 13.10 8. | | | | 10.77 7.3 11.30 7. | | | 9.50 6.10 | | 10.56 7.4 12.19 7. | | 10.98 6.1 | | 12.62 7.3 | | | 12.56 8. | 12.36.6. | | | 11.48 7. | 12.00 7.3 | | 36 | | | 8 0.58 8 8 | 46 | 11.55 7.49 1.05 0.66 | 12.21 | 2 0 12 0 | | |
| α-Tocopherol | 316 | 4.14 4.18 | 4.70 | 5.16 | 3.73 | 3.57 | 3.88 | 3.92 | 4.13 2.92 | 3.30 | | 4.49 | 4.37 | 4.10 | 4.07 | 4.18 | 5.20 | 2.19 | 5.03 | 2 73 | 4.10 | 4.35 | 4.03 | 4.18 | 5.40 | 36 | 4.18 | 5.40 | 0.30 | 0 0 | _ | 3.98 | 2 0 0 | | 807 |
| Γ-¤ | | 13 9.28 17 9.29 | 00 9.70 00 10.00 | | 9 8.53 | | | | | 80 8.00 | | 36 9.78 32 9.91 | | 8 8.77 | | 3 9.76 | | 92 8.14 | ~ | 0.05 | | 9.62 | | 5 9.63 | _ | 36 36 | | _ | 9.70 | 0 | | 9 9.45 | 2 | | 7 9 41 |
| | ш | 39 15.43 24 16.47 | 15.80 | | | | 14.21 | | 14.69 | 13.30 | | 15.86 | | | | 16.62 | 14.70 | | 17.34 | 10 80 | | 21 16.24 | 16.6 | 31 16.15 | 22.39 | 5 | | | 25 9 | 13 | 4 4 | 15.99 | 0 2 | 0.11 | _ |
| te | | 0.064 0.039 0.049 0.024 | | 0.049 0.041 | 0.055 0.035 | | | 0.048 0.025 | | | | | 0 | 0.066 0.041 | | 0.058 0.027 | | 0.054.0.033 | | 0.056.0.034 | | 0.040 0.021 | | 0.053 0.031 | | 12 12 | | | 0.006 0.008 11 25 | | 0.049 0.034 0.020 0.011 | | | | 0.054 0.034 |
| Retinyl Palmitate | 316 3 | 0.012 0.0 0.009 0.0 | | 0.024 0.0 | nd 0.0 | | | 0.007 0.0 | | | | | | ng bu | | 0.012 0.0 | | <0.01 | | 0 020 | | 0.004 0.0 | | 0.012 0.0 | | 8 | | 0.024 0.0 | 0.005 42 | 0 | öö | | | | 0.012.0.0 |
| Retinyl | 315 | 0.051 0 | | 0.056 | 0.058 | 400.0 | | 0.044 | | | | | 3 | 0.001 | | 0.072 0 | | 0.052 < | | 0.053 | | 0.040 | | 0.050 | | 12 | | | 11 | 0 | | | | | 0.052 |
| | | 0.104 | | 0.103 | 0.132 | 0.120 | | 0.093 | | | | | | 0.121 | | 0.139 | | 0 109 | | 0 110 | | 0.091 | | 0.100 | | 12 | 0.107 | | 0.015 | 0 | | | | | 0 104 |
| | 318 | | | | | | | | | 0.880 | 0.671 | | | | 0.880 | | | | | | | 0.827 | 1.099 | | | 9 (| 0.861 | 1.099 | 0 | | 0.839 | | 7 | | 0880 |
| etinol | 3 317 | | | | | | | | | 5 0770 | | | | | | 4 0.672 | | | | | | 4 0.644 | 0.797 | | | 9 9 | 2 0.681 | 8 0.797 | 0 0 | 0 0 | | | 2 | | 0 0 690 |
| trans-Retinol | 15 316 | | | | | | | | | 65 0 415 | | | | | | 32 | | | | | | 71 0.374 | 76 0.438 | | | 9 9 | 0.488 0.402 | 76 0.43 | 0 | 0 | | | 7 | | 71 0 410 |
| | 314 31 | | | | | | | | | 0.540.07 | 0.384 0.320 | | | | .570 0.5 | 0.572 0.5 | | | | | | 0.537 0.471 | 589 0.5 | | | 9 98 0 | 0.555 0.4 | .589 0.5 | 0 | 0 | | | 7 | | 0 540 0 471 |
| | 18 | 0.868 | 0.780 | 0.904 | 0.739 | 0.888 | 1.000 | 0.926 | 0.833 | 0.894 | | 0.866 | 1.089 | 0.830 | | 20.842 0 | 0.947 | 0.932 | 54 | 008 | 0.850 | | 0.835 >1.099 | 0.858 | 0.810 | 36 | | | 0.049 6 | 42 | 0.858 0.056 | 0.844 | 2000 | 0.010 | |
| | | | 0.610 | | | | 0.750 1 0.628 C | | | 0.737 0 | | 0.661 C 0.721 C | | 0.660 | | ≥0.672 ≥0 0.696 | | 0.919 0 | | 0 687 | _ | | 0.652 C >0.797 ≥1 | 0.674 C | | | | | 0.047 | | 0.662 C | 0.681 | 2 2 | | |
| Total Retinol | | | 0.360 | | _ | | 0.440 0.407 0 | | | 0.403 | | 0.355 (| | 0.400 | | ≥0.394 ≥C | | 0.374 (| | 7070 | | | 0.356 C | 0.386 (| | 36 | | | 0.023 | 0 | | 0.394 (| 0 005 | | 0.391 |
| Tota | | | 0.487 | | - × | | 0.550 (0.486 (| | | 0.539 (| | 0.458 (0.518 (| | | | ≥0.505 ≥C | | 490 | | 0.478 | | 0.471 | | 0.517 | | | | | 5 | 0 | | 0.496 | 2 00 0 | | 0 493 |
| | | | 0.538 (0.538) | | | | | 558 | 260 | 0.574 (| ı Ai | 0.536 (| | 0.555 | | ≥0.572 ≥(0.485 (| | | 0.541 | 0 546 | | 0.537 | , , | 0.568 (| | | | | 0.027 | 0 | | 0.557 (| 2 000 | | 0.551 |
| | | FSV-BA FSV-BB | | | | | | | FSV-BD (| | | | | | | FSV-CD × | | FSV-CG C | | FSV-CT | | FSV-DA (| | FSV-DI (| | Z | | | 3 S S | Npast | Medianpast SDpast | | NNIST | | |

Round Robin LVIII Serum Results All Values in µg/mL

| Total β-Carotene 314 315 316 317 318 0.604 0.279 0.040 0.314 0.177 0.601 0.282 0.042 0.318 0.16 | 316 317 0.040 0.314 0.042 0.318 | 7 314 318 | | 318 0.173 0.167 | 314 0.564 0.569 | trans-β-Carol 314 315 316 0.564 0.260 0.037 0.0569 0.267 0.039 | Carotene 16 317 137 0.291 139 0.300 | 318 0.161 0.157 | 314 31 0.040 0.0 | Total cis-β-Carotene 315 316 317 0.018 0.003 0.022 0.015 0.003 0.018 | Carotene 3 317 3 0.022 3 0.018 | 318 0.013 0.011 | Tota 314 315 0.127 0.057 0.109 0.051 | 316 0.006 0.005 | tene 317 318 0.056 0.015 0.051 0.011 | Tota 314 315 0.379 0.274 0.331 0.243 | Total Lycopene 115 316 317 274 0.165 0.425 243 0.150 0.372 | 318 0.360 0.309 | trans 314 315 0.201 0.138 0.166 0.113 | 316 0.076 0.062 | ane 317 318 0.203 0.179 0.162 0.140 |
|--|---|--|--|--|---|---|--|-----------------------|-----------------------------------|---|--|---------------------------------------|--|---|--|--|---|---|--|---------------------------------------|---|
| 0.631 0.281 0.040 0.350 0.192 0.654 0.317 0.034 0.373 0.186 0.736 0.355 0.079 0.425 0.195 0.685 0.313 0.039 0.370 0.190 0.653 0.295 0.039 0.348 0.177 0 0.558 0.244 0.036 0.281 0.153 0.593 0.267 0.037 0.319 0.161 | 0.040 0.350 0.192 0.034 0.373 0.186 0.079 0.425 0.195 0.039 0.370 0.190 0.036 0.281 0.153 0.037 0.319 0.161 | 0.350 0.192 0.373 0.186 0.425 0.195 0.370 0.190 0.653 0.295 0.039 0.348 0.177 0.281 0.153 0.319 0.161 | 0.192 0.186 0.195 0.190 0.153 0.161 | 0.653 0.295 0.039 0.348 0.177 | 0.348 0.177 | 0.348 0.177 | | 0 | 0.032 0.018 | 318 nq | | 0.023 0.013 | 0.127 0.059 0.120 0.062 0.122 0.053 0.120 0.052 0.119 0.052 | 0.008 0.013 <i>nq</i> 0.004 | 0.052 0.016 0.057 0.018 0.053 <i>nq</i> 0.049 0.018 | 0.331 0.256 0.411 0.340 0.276 0.186 0.285 0.189 | 0.150 0.405 0.197 0.499 0.114 0.291 0.121 0.280 | 0.371 0.405 0.246 0.244 0.319 | 0.234 0.172 | 0.103 | 0.252 0.207 |
| 0.558 0.282 0.042 0.323 0.163 0.517 0.242 0.037 0.293 0.150 0.058 0.212 0.043 0.276 0.139 0.279 0.350 nq 0.330 0.170 | 0.042 0.323 0.163 0.517 0.242 0.037 0.293 0.150 0.043 0.276 0.139 0.170 | 0.323 0.163 0.517 0.242 0.037 0.293 0.150 0.276 0.139 0.170 | 0.163 0.517 0.242 0.037 0.293 0.150 0.139 | 0.517 0.242 0.037 0.293 0.150 | 0.293 0.150 | 0.293 0.150 | | 0.0 |)44 0.C | 0.044 0.023 0.008 0.033 0.016 | 18 0.033 | | 0.119 0.055 0.132 0.057 0.112 0.029 | 0.010 nq nq | 0.057 0.017 0.056 0.010 0.030 nq | 0.289 0.227 0.333 0.204 0.403 0.375 | 0.134 0.335 0.173 0.351 0.217 0.420 | 0.294 0.296 0.292 | 0.154 0.1 | 0.112 0.069 0.174 0.154 | 174 0.154 |
| 20.621 20.284 20.084 20.343 20.202 0.621 0.284 0.084 0.343 0.202 0.666 0.304 0.044 0.351 0.199 0.554 0.284 0.042 0.326 0.183 0.0529 0.069 0.067 0.399 0.174 0.561 0.303 0.041 0.341 0.191 0.580 0.275 0.036 0.301 0.151 0.569 20.258 20.034 20.302 20.161 0.569 20.258 20.034 20.302 20.161 0.569 0.258 0.034 0.302 0.161 | 20.064 20.343 20.202 0.621 0.284 0.064 0.343 0.202 0.044 0.351 0.196 0.564 0.284 0.042 0.326 0.183 0.067 0.299 0.174 0.041 0.341 0.191 0.036 0.301 0.151 20.034 20.302 20.161 0.569 0.258 0.034 0.302 0.161 | 20.343 20.202 0.621 0.284 0.064 0.343 0.202 0.351 0.196 0.564 0.284 0.042 0.326 0.183 0.202 0.391 0.191 0.301 0.151 0.569 0.258 0.034 0.302 0.161 | 20.202 0.621 0.284 0.084 0.343 0.202 0.196 0.564 0.284 0.042 0.326 0.183 0.171 0.191 0.569 0.258 0.034 0.302 0.161 | 0.621 0.284 0.064 0.343 0.202 0.564 0.284 0.042 0.326 0.183 0.569 0.258 0.034 0.302 0.161 | 0.343 0.202 0.326 0.183 0.302 0.161 | 0.343 0.202 0.326 0.183 0.302 0.161 | 0.202 | ö | 0.044 0.0 | 0.020 0.00 | 0.003 0.026 0.013 | | 0.138 0.076 0.121 0.063 0.110 0.052 0.096 0.042 0.165 0.070 0.125 0.055 | 0.033 0.008 0.009 0.004 0.009 | 0.079 0.044 0.061 0.022 0.049 0.013 0.056 <i>nq</i> 0.054 0.013 | 0.497 0.359 0.328 0.250 0.352 0.272 0.414 0.291 0.288 0.206 | 0.257 0.538 0.153 0.375 0.189 0.408 0.167 0.445 0.125 0.324 | 0.493 0.370 0.354 0.405 0.273 | 0.161 0.124 | 0.076 | 0.184 0.186 0.186 0.150 0.139 |
| 0.729 0.298 0.045 0.337 0.169 0.684 0.310 0.050 0.292 0.171 | 0.045 0.337 0.050 0.292 | 0.337 | | | | | | | | | | | 0.163 0.073 | 0.014 | 0.070 0.022 | 0.526 0.319 | 0.150 0.451 | 0.336 | | | |
| 0.571 0.260 0.035 0.319 0.159 0.530 0.241 0.032 0.296 0.147 0.041 20.613 20.269 20.039 20.285 20.162 0.613 0.269 0.039 0.285 0.162 0.701 0.365 0.093 0.289 0.219 0.645 0.335 0.082 0.357 0.200 0.056 1.034 0.444 0.060 0.491 0.273 0.263 0.291 0.023 0.330 0.152 0.550 0.050 0 | 0.035 0.319 0.159 0.530 0.241 0.032 0.296 0.147 20.039 20.285 20.162 0.613 0.269 0.039 0.285 0.162 0.093 0.389 0.219 0.645 0.335 0.082 0.357 0.200 0.060 0.491 0.273 0.645 0.291 0.023 0.330 0.152 0.632 0.291 0.023 0.330 0.152 | 0.159 0.530 0.241 0.032 0.296 0.147 20.162 0.613 0.269 0.039 0.285 0.162 0.219 0.645 0.335 0.082 0.357 0.200 0.273 0.632 0.291 0.023 0.330 0.152 0.052 0.291 0.023 0.330 0.152 | 0.159 0.530 0.241 0.032 0.296 0.147 20.162 0.613 0.269 0.039 0.285 0.162 0.219 0.645 0.335 0.082 0.357 0.200 0.273 0.632 0.291 0.023 0.330 0.152 0.052 0.291 0.023 0.330 0.152 | 0.530 0.241 0.032 0.296 0.147 0.613 0.269 0.039 0.285 0.162 0.645 0.335 0.082 0.357 0.200 0.632 0.291 0.023 0.330 0.152 | 0.032 0.296 0.147 0.039 0.285 0.162 0.082 0.357 0.200 0.023 0.330 0.152 | 0.296 0.147 0.285 0.162 0.357 0.200 0.330 0.152 | 0.147 0.162 0.200 0.152 | 0:0 0:0 | 41 0.C 56 0.C | 0.019 0.003 | 03 0.023 | 0.012 | 0.145 0.065 0.132 0.060 0.135 0.065 0.143 0.069 | 0.006 <0.009 0.008 0.011 | 0.069 0.018 0.057 0.024 0.063 0.017 0.067 0.022 | 0.342 0.238 0.428 0.322 0.369 0.244 | 0.128 0.386 0.202 0.500 0.120 0.335 | 0.313 0.456 0.318 | 0.189 0.128 | 0.066 | 0.197 0.166 |
| 0.279 0.033 0.267 | 0.053 0.267 0.137 0.738 0.326 0.045 0.308 0.158 0.053 0.267 0.137 | 0.267 0.137 0.738 0.326 0.045 0.308 0.158 0.267 0.137 | 0.137 0.738 0.326 0.045 0.308 0.158 0.138 | 0.738 0.326 0.045 0.308 0.158 | 0.045 0.308 0.158 | 0.308 0.158 | 0.158 | 0.0 | | 0.026 0.006 0.032 0.019 | 0.032 | | 0.124 0.054 | 0.006 | 0.049 0.014 | 0.336 0.232 0.297 0.232 | 0.144 0.315 0.138 0.313 | 0.273 | 0.189 0.122 | 0.072 | 0.155 0.138 |
| 0.285 0.040 0.335 | 0.040 0.335 | 0.335 | | | | | | | | | | | | | | 0.300 0.230 | 0.160 | | | | |
| 0.806 0.287 0.006 0.294 0.130 25 25 24 25 12 12 12 12 18 8 0.279 0.212 0.006 0.267 0.313 0.173 0.591 0.274 0.039 0.305 0.147 0.039 0.054 0.286 0.042 0.323 0.173 0.591 0.277 0.039 0.365 0.161 0.043 1.034 0.044 0.093 0.491 0.273 0.738 0.357 0.292 0.057 0.094 0.005 0.022 0.053 0.024 0.005 0.028 0.017 0.007 16 11 24 12 12 9 9 12 9 10 16 16 | 0.006 0.294 0.130 | 0.294 0.130 | 0.130 12 | 12 12 12 12 12 12 12 12 12 12 12 12 12 1 | 12 12 12 12 0.023 0.285 0.147 0.039 0.305 0.161 0.082 0.357 0.202 0.005 0.005 0.028 0.017 12 9 10 | 12 12 0.285 0.147 0.305 0.161 0.357 0.202 0.028 0.017 9 10 | 12 0.147 0.161 0.202 0.017 | 0.03 | 8 2 0.0 3 0.0 7 0.0 6 | 8 7 0.015 0.003 0.020 0.003 0.029 0.011 0.004 0.003 21 100 | 7 8 03 0.018 03 0.025 1 0.033 03 0.007 | 8 0.011 0.013 0.003 23 | 23 23 0.096 0.029 0.125 0.057 0.165 0.076 0.012 0.008 10 15 | 0.004 0.008 0.008 0.003 0.004 | 0.048 0.016 23 20 20 0.030 0.010 0.056 0.017 0.079 0.044 0.007 0.004 12 26 | 23 23 0.276 0.186 0.336 0.243 0.526 0.375 0.059 0.041 17 17 | 0.157 0.292 23 23 0.114 0.280 0.150 0.375 0.257 0.538 0.025 0.073 17 19 | 0.247 23 0.244 0.313 0.061 | 9 9 0.148 0.098 0.166 0.122 0.234 0.172 0.026 0.012 15 10 | 9 0.055 0.069 0.103 0.007 | 9 9 0.150 0.132 0.174 0.154 0.252 0.207 0.030 0.030 |
| 0 0 0 31 30 0 0 0 14 14 0 0 0 0.317 0.173 0.298 0.160 0.047 0.022 0.039 0.017 | 0 31 30 0 0 14 14 0.317 0.173 0.298 0.160 0.047 0.022 0.039 0.017 | 31 30 0 0 14 14 0.317 0.173 0.298 0.160 0.047 0.022 0.039 0.017 | 30 0 0 0 14 14 0.173 0.298 0.160 0.022 0.039 0.017 | 0 0 0 14 14 0.298 0.160 0.039 0.017 | 0 14 14 0.298 0.160 0.039 0.017 | 14 14 0.298 0.160 0.039 0.017 | 14 0.160 0.017 | | 0 | 0 | 0 10 0.024 0.008 | 0 9 4 0.014 3 0.006 | 0 | 0 | 26 26 0.053 0.016 0.015 0.004 | 0 | 0 25 0.367 0.088 | 25 0.307 0.051 | 0 | 0 0 | 12 12 0.177 0.158 0.036 0.026 |
| 0.599 0.307 nq 0.315 0.172 0.509 0.256 nq 0.272 0.162 0.062 2 | nq 0.315 0.172 0.509 0.256 nq 0.272 0.162 2 2 2 2 2 2 2 0.013 0.003 0.024 0.009 0.014 0.005 0.001 0.005 0.005 0.001 0.001 0.002 0.001 0.004 0.005 0.001 0.004 0.005 0.004 0.005 0.004 0.005 0.014 0.005 0.014 0.005 0.014 0.005 0.014 0.005 0.014 0.005 0.014 0.005 0.014 0.005 0.014 0.005 0.014 0.005 0.014 0.005 0.014 0.005 0.014 0.005 0.014 0.005 0.014 0.005 0.014 0.005 0.014 0.005 | 0.172 0.509 0.256 nq 0.272 0.162 2 2 2 2 2 0.003 0.024 0.009 0.014 0.005 0.005 0.005 0.004 0.001 0.005 0.005 0.005 0.004 0.004 0.005 | 0.172 0.509 0.256 nq 0.272 0.162 2 2 2 2 2 0.003 0.024 0.009 0.014 0.005 0.005 0.005 0.004 0.001 0.005 0.005 0.005 0.004 0.004 0.005 | 0.509 0.256 | nq 0.272 0.162 2 2 0.014 0.005 0.001 0.002 0.014 0.005 | 0.272 0.162 2 2 0.014 0.005 0.001 0.002 0.014 0.005 | 0.162 2 0.005 0.002 0.005 | 0.090 | | 0.052 nq 2 0.022 0.014 0.026 | 0.042 0.003 0.005 0.006 | 0.010 2 0.003 0.007 0.007 | 0.118 0.058 2 2 0.005 0.005 0.001 0.005 0.005 0.007 | bu | 0.059 nq 2 0.005 0.000 0.005 | | | | | | |
| $\begin{array}{cccccccccccccccccccccccccccccccccccc$ | 0.041 0.318 0.173 0.550 0.266 0.039 0.289 0.162 0.010 0.047 0.027 0.085 0.033 0.008 0.040 0.019 | 0.318 0.173 0.550 0.266 0.039 0.289 0.162 0.047 0.027 0.085 0.033 0.008 0.040 0.019 | 0.027 0.550 0.266 0.039 0.289 0.162 0.027 0.085 0.033 0.008 0.040 0.019 | 0.550 0.266 0.039 0.289 0.162 0.085 0.033 0.008 0.040 0.019 | 0.289 0.162 0.040 0.019 | 0.289 0.162 0.040 0.019 | 0.162 | 0 0 | 0.066 0.0 | 0.036 0.003 | 03 0.033 03 0.015 | 0.011 | 0.121 0.057 0.035 0.017 | 0.008 | 0.057 0.016 | 0.335 0.241 | 0.152 0.374 0.039 0.082 | 0.311 | 0.166 0.122 0.029 0.021 | 0.069 | 0.174 0.154 0.031 0.030 |

Round Robin LVIII Serum Results All Values in µg/mL

| Coenzyme Q10 15 316 317 318 | | 6 0.535 0.817 0.599 | | | 1 0.608 0.735 0.580 | | | 9 0.696 0.935 0.747 | 0 0.460 0.650 0.640 0 0.440 1.080 1.110 | 0 0.570 0.720 0.600 | | 6 6 6 6 6 11 0.440 0.650 0.580 8 0.553 0.776 0.620 9 0.696 1.080 1.110 3 0.089 0.135 0.090 7 16 17 14 | | | |
|-------------------------------------|------------------|---|---|--|---|------------------|--|--|--|----------------------------|--|--|-------------------------------|------|---|
| 314 315 | | 0.612 0.656 | | | 0.049 0.521 | | | 0.656 0.819 | 0.610 0.530 0.850 0.690 | 0.560 0.580 0.570 | | 6 6 0.049 0.521 0.611 0.618 0.850 0.819 0.054 0.103 9 17 | | | |
| 318 | 0.087 | 0.088 0.077 0.073 0.069 | 0.088 0.083 0.083 | 0.085 0.089 0.091 0.089 | 0.059 | 0.092 | 0.087 | | 0.095 | | 0.103 | | 25 0.092 0.017 | | |
| anthin 317 | 0.104 | 0.100 0.111 0.082 0.078 | 0.108 0.081 0.160 | 0.097 0.086 0.098 0.093 | 0.062 | 0.104 | 0.132 | 0.090 0.069 0.098 | 0.108 | | 0.124 | 24 0.062 0.098 0.160 0.016 | 24 0.102 0.032 | | |
| n&Zeax 316 | 0.036 | 0.034 0.047 0.028 0.031 | 0.043 <i>nq</i> 0.030 | 0.047 0.033 0.037 0.037 | 0.027 0.052 | 0.036 | 0.048 | 0.041 nq 0.042 | 0.048 | | 0.095 | 22 0.027 0.039 0.095 0.010 | 0 | | |
| Total Lutein&Zeaxanthin 315 316 317 | 0.090 | 0.085 0.096 0.072 0.074 | 0.090 0.056 0.120 | 0.087 0.069 0.085 0.086 | 0.067 | 0.092 | 0.110 | 0.085 ≥0.054 0.086 | 0.097 | | 0.121 | 23 0.056 0.087 0.121 0.006 | 0 | | |
| 314 Tc | 0.149 | 0.140 0.142 0.125 0.720 | 0.153 0.172 0.230 | 0.140 0.115 0.155 0.145 | 0.124 | 0.167 | 0.184 | | 0.155 | | 0.214 | 23 0.115 0.145 0.230 0.021 | 0 | | |
| 318 | | 0.030 0.032 0.027 | 0.036 | 0.020 0.022 | 0.191 | | 0.032 | 0.026 0.019 0.027 | 0.037 | | 0.089 | 0.019 0.032 0.191 0.009 | 0.0 | 7 | |
| anthin 317 | _ | 3 0.036 3 0.028 4 0.023 | 5 0.038 | 3 0.020 | 0.136 0.066 0.030 0.076 0.073 0.035 0.027 0.022 0.033 0.191 | | 9 0.026 | | 1 0.033 | | 0.092 | 3 0.016 7 0.028 0 0.092 4 0.009 | 0.0 | 2 2 | |
| Total Zeaxanthin | _ | 9 0.018 7 0.013 3 0.014 | 0.015 | 0.016 0.008 | 7 0.022 | | 2 0.019 | 0.017 nq 1 0.017 | 0.021 | | 0.080 | 2 0.008 3 0.017 4 0.080 5 0.004 | | 2 | |
| (| | 7 0.029 4 0.017 0 0.018 | 0.025 9 nd | | 5 0.027 | | 0.022 | | 0.030 | | 0.084 | | | 2 | |
| 314 | | 0.027 | 0.040 | 0.031 | 0.03 | | 0.030 | | 0.041 | | 0.101 | | | | |
| 318 | | 7 0.066 4 0.041 5 0.042 3 0.062 | 0.052 | 3 0.063 | 3 0.073 | | 1 0.055 | | 5 0.058 | 3 0.050 | 2 0.014 | 5 0.014 5 0.052 7 0.073 8 0.011 | 17 1 0.055 3 0.013 | 2 2 | |
| stein 317 | _ | 9 0.097 5 0.054 7 0.055 6 0.073 | 0.057 | 3 0.063 | 0 0.076 | | 5 0.061 | 4 0.067 0.053 5 0.071 | 8 0.075 | 0.017 0.058 0.050 | 5 0.032 | 5 0.032 5 0.065 9 0.097 7 0.013 8 20 | ö ö | 2 | |
| Total Lutein 3 | _ | 5 0.039 5 0.015 6 0.017 8 0.026 | 5 0.028 6 <i>nq</i> | 1 0.023 | 6 0.03(| | 3 0.035 | | 7 0.028 | | 7 0.015 | 5 0.015 4 0.025 5 0.039 8 0.007 2 28 | | 2 | |
| 316 | | 6 0.085 1 0.055 0 0.056 8 0.068 | 3 0.065 3 0.056 | 8 0.051 | 90.0 | | 2 0.063 | 1 0.065 1 0.054 4 0.065 | 4 0.067 | 0.105 0.057 | 3 0.037 | 6 16 8 0.037 3 0.064 16 0.085 18 0.008 | 0 | 2 | |
| 314 | _ | 0.126 0.101 0.100 0.118 | 0.123 | 0.078 | 0.13 | | 0.102 | 0.111 | 0.114 | 0.10 | 0.113 | - | | | |
| in 318 | | | 1 0.024 | 8 0.019 | | | | 1 0.022 | 3 0.020 | | | 0.00 | 0.0 | | |
| toxanthi 317 | 2 0.02; | | 1 0.02 | 7 0.01 | | | | 9 0.02 | 9 0.01 | | | 6 6 6 6 7 0.016 9 0.020 2 0.023 12 0.002 | 0 6 0.018 0.002 | | |
| Total α-Cryptoxanthin 315 316 | 8 0.01 | | 5 0.01 | 3 0.00 | | | | 5 0.00 | 5 0.00 | | | 6 6 12 0.007 15 0.009 18 0.012 01 0.002 9 20 | | | |
| - | | | 0.019 0.015 0.011 0.021 | 0.020 0.013 0.007 0.018 | | | | 0.017 0.015 0.009 0.021 | 0.022 0.015 0.009 0.018 | | | 0.00 | | | |
| 314 | | | | | M Al | | | | | | | | | 2 | |
| in 318 | 5 0.08 | 5 0.078 2 0.068 3 0.099 5 0.065 3 0.070 | 0.057 0.076 0.053 0.065 0.110 0.072 | 5 0.074 5 0.074 5 0.074 7 0.067 | 0.053 | 3 0.066 | 3 0.093 | 0.076 7 0.058 9 0.069 | 0.059 0.073 0.039 0.070 | | 2 0.083 | 24 9 0.053 5 0.071 0 0.099 1 0.008 | 0.0 | | |
| toxanthi 317 | 0.06 | 5 0.055 3 0.042 7 0.083 3 0.046 5 0.053 | 5 0.057 5 0.053 0 0.110 | 2 0.076 2 0.055 0 0.055 2 0.047 | 5 0.040 | 0.058 | 3 0.078 | 5 0.061 2 0.047 5 0.059 | 7 0.059 | |) 0.067 | 24 24 24 0.020 0.039 0.053 0.035 0.055 0.071 0.059 0.110 0.099 0.005 0.011 0.008 | 0.0 | 2 | |
| Total β-Cryptoxanthin 315 316 | | 5 0.036 5 0.023 1 0.047 2 0.033 0 0.035 | 5 0.036 1 0.036 6 0.020 | 5 0.042 4 0.032 8 0.040 1 0.032 | 5 0.025 9 0.029 | 4 0.030 | 1 0.048 | 1 0.035 0 0.022 5 0.035 | 4 0.037 7 0.031 | | 1 0.059 | 24 24 24 24 0.035 0.035 0.020 0.039 0.053 0.050 0.045 0.035 0.050 0.010 0.099 0.005 0.011 0.008 0.005 0.011 0.008 0.01 0.009 0.005 0.011 0.008 0.008 0 | | 2 | |
| | 3 0.05 | 8 0.045 0 0.035 3 0.071 8 0.042 5 0.040 | 0 0.045 3 0.041 7 0.056 | 8 0.055 2 0.044 1 0.048 5 0.041 | 8 0.035 5 0.039 | 4 0.044 | 4 0.06 | 0 0.051 0 0.040 6 0.045 | 7 0.054 1 0.047 | | 2 0.06 | N 24 24 Min 0.045 0.035 dian 0.052 0.045 Max 0.093 0.071 SD 0.011 0.009 CV 20 20 | | 2 | |
| 314 | | FSV-BC FSV-BD FSV-BF FSV-BF FSV-BH FSV-BH 0.093 FSV-BH 0.048 FSV-BI 0.045 FSV-BK FSV-BK | 0.050 0.053 0.057 | FSV-BR FSV-BS 0.068 FSV-BT 0.052 FSV-BU 0.051 FSV-BV 0.045 | V 0.048 X 0.045 | 0.064 | F 0.07, | S 0.060 T 0.050 V 0.056 | Z A 0.067 B 0.061 | O IL = | V -/ 0.08 <u>2</u> | N 24 Min 0.045 Median 0.052 Max 0.093 SD 0.011 CV 20 | | | • |
| Lab | FSV-BA FSV-BB | FSV-BC FSV-BB FSV-BE FSV-BG FSV-BH FSV-BI FSV-BI FSV-BI | FSV-BM FSV-BO FSV-BP FSV-BQ | FSV-BR 0.068 FSV-BS 0.062 FSV-BU 0.051 FSV-BV 0.045 | FSV-BW FSV-BX | FSV-CC FSV-CD | FSV-CE FSV-CG FSV-CG 0.074 0.061 0.043 0.078 0.093 FSV-CI | FSV-CS 0.060 FSV-CT 0.050 FSV-CW 0.056 | FSV-CZ FSV-DA FSV-DB | FSV-DD FSV-DF FSV-DI | FSV-DV FSV-DW 0.082 0.064 0.059 0.067 0.083 | Min Median Max SD SD CV | Npast Medianpast SDpast | NIST | J |

Round Robin LVIII Serum Results All Values in µg/mL

| 314 | 315 | Ubiquinol 5 316 | | 317 | 318 | 314 | 315 | Ubiquinone | ne 317 | 318 | 314 PF | Phylloquinone (K1) x1000 | 316 | 317 | 318 |
|--|--|-------------------------|------------------------------|------------------------------|------------------------------|------------------------------|-------------------------------|------------------------------|------------------------------|------------------------------|------------------------------|-------------------------------|------------------------------|------------------------------|------------------------------|
| FSV-BA FSV-BE FSV-BE FSV-BE FSV-BE FSV-BH FSV-BH FSV-BH FSV-BH FSV-BN FS | | | | | | | | | | | 1.571 | 1.571 0.774 0.154 1.116 1.063 | 0.154 | | 1.063 |
| | nd 0.195 0.488 0.472 0.452 0.850 0.690 0.440 1.080 1.110 | 0.195 0.488 0.472 0.452 | .1 40 | . 080 | 0.452 | 0.049 | 0.049 0.326 0.120 0.263 0.127 | 0.120 | 0.263 | 0.127 | 1.180 | 1.180 0.520 0.100 0.770 0.670 | 0.100 | 0.770 | 0.670 |
| FSV-D1 FSV-DV FSV-DW | 0.150 0.370 | 70 0.5 | 40 0. | .585 | 0.540 0.585 0.600 | 0.310 | 0.310 0.220 0.050 0.160 0.105 | 0.050 | 0.160 | 0.105 | 0.890 | 0.890 0.490 0.220 0.730 0.740 | 0.220 | 0.730 | 0.740 |
| <u> </u> | 2 3 0.150 0.195 0.500 0.370 0.850 0.690 | | 3 0.440 0.488 0.540 | 3 0.472 0.585 1.080 | 3 0.452 0.600 1.110 | 2 0.049 0.180 0.310 | 2 0.220 0.273 0.326 | 2 0.050 0.085 0.120 | 2 0.160 0.212 0.263 | 2 0.105 0.116 0.127 | 3 0.890 1.180 1.571 | 3 0.490 0.520 0.774 | 3 0.100 0.154 0.220 | 3 0.730 0.770 1.116 | 3 0.670 0.740 1.063 |
| Neast Medianpast SDpast NIST NNIST Srep Shet SNIST | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| NAV NAU | 0.3 | 0.370 0.488 0.585 0.600 | .88 0. | 285 | 0.600 | | | | | | 1.180 | 1.180 0.520 0.154 0.770 0.740 | 0.154 | 0.770 | 0.740 |

Round Robin LVIII Serum Results All Values in µg/mL

Analytes Reported By One Laboratory

| Analyte | Code | 314 | 315 | 316 | 317 | 318 |
|---------------------|--------|--------|--------|--------|-------|-------|
| trans-Lutein | FSV-DA | 0.107 | 0.061 | 0.023 | 0.068 | 0.051 |
| 25-hydroxyvitamin D | FSV-BN | 0.0381 | 0.0200 | 0.0090 | | |
| Phytofluene | FSV-DA | 0.051 | 0.030 | 0.013 | 0.049 | 0.039 |
| Phytoene | FSV-DA | 0.047 | 0.028 | 0.024 | 0.048 | 0.024 |
| Retinyl stearate | FSV-DA | 0.040 | 0.012 | nd | 0.016 | 0.008 |
| 9-cis-Lycopene | FSV-BN | 0.083 | 0.065 | 0.041 | 0.103 | 0.086 |
| 13-cis-Lycopene | FSV-BN | 0.045 | 0.038 | 0.026 | 0.055 | 0.051 |
| 15-cis-Lycopene | FSV-BN | 0.016 | 0.022 | 0.008 | 0.014 | 0.013 |

Legend

| Term | Definition |
|--|---|
| N | Number of (non-NIST) quantitative values reported for this analyte |
| Min | Minimum (non-NIST) quantitative value reported |
| Median | Median (non-NIST) quantitative value reported |
| Max | Maximum (non-NIST) quantitative value reported |
| SD | Standard deviation for (non-NIST) results: 0.741*(3rd Quartile - 1st Quartile) |
| CV | Coefficient of Variation for (non-NIST) results: 100*SD/Median |
| N past | Mean of N(s) from past RR(s) |
| Medianpast | Mean of Median(s) from past RR(s) |
| SD _{past} | Pooled SD from past RR(s) |
| | |
| | Mean of all analyses (vials x duplicates) reported by a NIST analyst |
| | Number of total vials analyzed in duplicate by NIST analysts |
| | Within-vial pooled standard deviation |
| | Among-vial pooled standard deviation |
| Snist | Total standard deviation for NIST analyses: $(S_{rep}^2 + S_{het}^2)^{0.5}$ |
| NAV | NIST Assigned Value |
| 147.17 | = (Median + MeanNIST)/2 for analytes reported by NIST analyst(s) |
| | = Median for analytes reported by ≥ 10 labs but not NIST |
| NAU | NIST Assigned Uncertainty: (S ² + S _{btw} ²) ^{0.5} |
| | S is the maximum of (0.05*NAV, SD, SNIST, eSD) and Sbtw is the standard |
| | deviation between Median _{part} and Mean _{NIST} . The expected long-term SD, eSD, |
| | is defined in: Duewer, et al. Anal Chem 1997;69(7):1406-1413. |
| | |
| nd | Not detected (i.e., no detectable peak for analyte) |
| nq | Detected but not quantitatively determined |
| <x< td=""><td>Concentration at or below the limit of quantification, x</td></x<> | Concentration at or below the limit of quantification, x |
| ≥x | Concentration greater than or equal to x |

Round Robin LVIII "Score Card" for Serum Samples

Comparability Summary

| Lab | TR | аТ | g/bT | bC | tbC | aС | TLy | TbX | TLu | ΤZ | L&Z |
|--------|----|----|------|----|-----|----|-----|-----|-----|----|-----|
| FSV-BA | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 |
| FSV-BB | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 2 | 1 | 1 |
| FSV-BC | 1 | | | | | | | | | | |
| FSV-BD | 2 | 1 | | | | | | | | | 1 |
| FSV-BE | 1 | 2 | 1 | 1 | | | | | | | |
| FSV-BF | 1 | 1 | 1 | 1 | | 1 | 1 | 1 | | | 1 |
| FSV-BG | 1 | 2 | 1 | 3 | | 1 | 2 | 1 | 1 | 2 | 1 |
| FSV-BH | 1 | 2 | 4 | 1 | 1 | 1 | 1 | 3 | | | |
| FSV-BI | 2 | 2 | 1 | 1 | | 1 | 1 | 1 | 1 | 1 | 1 |
| FSV-BJ | 1 | 1 | 1 | 1 | | 1 | 1 | 1 | 2 | 4 | 4 |
| FSV-BK | 2 | 1 | | | | | | | | | |
| FSV-BL | 2 | 2 | | | | | | | 1 | | |
| FSV-BM | 1 | 1 | | | | | | | | | |
| FSV-BN | 1 | 1 | 2 | 1 | 1 | 1 | 1 | 1 | | | 3 |
| FSV-BO | 1 | 2 | | 2 | | 1 | 1 | 1 | 1 | 1 | 1 |
| FSV-BP | 1 | 3 | | 3 | | 2 | 2 | 3 | | | 1 |
| FSV-BQ | 1 | 3 | | | | | | | 1 | 2 | 2 |
| FSV-BR | 1 | 1 | | | | | | | 1 | 2 | 2 |
| FSV-BS | 4 | | 4 | 2 | 2 | 4 | 3 | 2 | 2 | 2 | 1 |
| FSV-BT | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | | | |
| FSV-BU | 1 | 1 | 1 | 2 | | 1 | 1 | 1 | | | |
| FSV-BV | 3 | 1 | 1 | 1 | | 1 | 1 | 1 | | | 2 |
| FSV-BW | 1 | 1 | 3 | 1 | | 1 | 1 | 1 | | | |
| FSV-BX | 1 | 1 | 1 | 1 | 1 | 1 | | 1 | | | 1 |
| FSV-CC | 1 | 1 | | | | | | | | | 1 |
| FSV-CD | 1 | 1 | 1 | 1 | | 2 | 2 | 1 | | | 1 |
| FSV-CE | 2 | 1 | | 1 | | | | | 1 | 1 | 1 |
| FSV-CF | 2 | 2 | | | | | | | | | 1 |
| FSV-CG | 3 | 3 | 1 | 1 | 1 | 1 | 1 | 2 | | | |
| FSV-CI | 1 | 1 | 1 | 1 | 1 | 1 | | | | | 2 |
| FSV-CS | 1 | 2 | 3 | 3 | 3 | 1 | 2 | 1 | | | |
| FSV-CT | | | | 4 | | | 1 | 1 | 1 | 1 | 1 |
| FSV-CW | 1 | 3 | 1 | 1 | 2 | 1 | | 1 | 1 | 1 | 1 |
| FSV-CZ | 1 | 1 | | 2 | | | | | | | |
| FSV-DA | 1 | 1 | 1 | 2 | 2 | 1 | 1 | 1 | | | |
| FSV-DB | 1 | 1 | 1 | 1 | | | 1 | 1 | 3 | 4 | 3 |
| FSV-DD | 3 | | | | | | | | | | |
| FSV-DF | 1 | | | | | | | | 1 | | |
| FSV-DI | 1 | 1 | 1 | 1 | | | 1 | | 1 | 1 | 1 |
| FSV-DV | 4 | 3 | | | | | | | | | |
| FSV-DW | 1 | 3 | | 3 | | 1 | 1 | 2 | | | |
| NIST | 1 | 1 | 1 | 1 | 1 | 1 | | | | | |
| n | 41 | 37 | 24 | 30 | 13 | 24 | 23 | 24 | 16 | 14 | 24 |

| | | | g/bT | | | | | | | | |
|-----|----|----|---------|----|----|----|----|----|----|----|----|
| % 1 | 73 | 62 | 79 4 | 67 | 69 | 88 | 78 | 79 | 75 | 57 | 71 |
| % 2 | 15 | 22 | 4 | 17 | 23 | 8 | 17 | 13 | 19 | 29 | 17 |
| % 3 | 7 | 16 | 8 | 13 | 8 | 0 | 4 | 8 | 6 | 0 | 8 |
| % 4 | 5 | 0 | 8 | 3 | 0 | 4 | 0 | 0 | 0 | 14 | 4 |

| 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 |
| | |
| n | number of participants providing quantitative data |
| % 1 | Percent of CS = 1 (within 1 SD of medians) |
| % 2 | Percent of CS = 2 (within 2 SD of medians) |
| % 3 | Percent of CS = 3 (within 3 SD of medians) |
| % 4 | Percent of CS = 4 (3 or more SD from medians) |
| | |
| | "Comparability Score" |

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

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

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

$$\text{AP} = \text{Apparent Precision} = \sqrt{\sum_{i}^{N_{you}} \!\! \left(\frac{\text{You}_i - \text{Median}_i}{\text{NAU}_i} \right)^2 / \!\! \left(\!\! N_{you} - 1 \! \right)}$$

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 RR58

Each participant in RR58 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 RR58:

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

The following 15 pages are the "Individualized Report" for the analytes evaluated by participant FSV-BA.

Individualized Round Robin LVIII Report: FSV-BA

Summary

| | | | |) | | | | | | | | | | | |
|-------------------------|-------|--------|----|-------|---------|-----|-------|-----------|----|-------|-------------|----|-------|-----------|--------------|
| | Seri | um 314 | | Ser | rum 315 | | Sen | Serum 316 | | Ser | 6 Serum 317 | | Seri | Serum 318 | |
| Analyte | You | NAV | ے | You | | | You | NAV | _ | You | NAV | _ | You | NAV | ⊆ |
| Total Retinol | 0.541 | 0.551 | 36 | 0.491 | 0.493 | 35 | 0.384 | 0.39 | 36 | 0.700 | 0.682 | 36 | 0.872 | 0.858 | 36 |
| Retinyl Palmitate | 0.10 | 0.10 | 12 | 0.1 | 0.1 | 12 | 0.0 | 0.0 | ∞ | 90.0 | 0.05 | 12 | 0.04 | 0.03 | 12 |
| α-Tocopherol | 15.43 | 16.07 | 36 | 9.28 | 9.41 | | 4.14 | 4.08 | 36 | 11.17 | 11.95 | | | 7.54 | 36 |
| γ/β-Tocopherol | 1.497 | 1.458 | 23 | 1.839 | 1.809 | 23 | 1.752 | 1.72 | 23 | 2.686 | 2.728 | 23 | | 3.945 | 23 |
| ō-Tocopherol | 0.109 | 0.103 | / | 0.113 | 0.111 | | 0.098 | 0.10 | 7 | 0.342 | 0.334 | ∞ | 0.120 | 0.126 | 7 |
| Total β-Carotene | 0.604 | 0.601 | 25 | 0.279 | 0.295 | 25 | 0.040 | 0.04 | 24 | 0.314 | 0.318 | | 0.173 | 0.173 | 25 |
| trans-β-Carotene | 0.564 | 0.550 | 12 | 0.260 | 0.266 | | 0.037 | 0.03 | 12 | 0.291 | 0.289 | 12 | 0.161 | 0.162 | 12 |
| Total cis-β-Carotene | 0.040 | 0.066 | ∞ | 0.018 | 0.036 | ∞ | 0.003 | 0.00 | 7 | 0.022 | 0.033 | | 0.013 | 0.011 | _∞ |
| Total α-Carotene | 0.127 | 0.121 | 23 | 0.057 | 0.057 | | 900.0 | 0.00 | 18 | 0.056 | 0.057 | | 0.015 | 0.016 | 20 |
| Total Lycopene | 0.379 | 0.335 | 23 | 0.274 | 0.241 | 23 | 0.165 | 0.15 | 23 | 0.425 | 0.374 | 23 | 0.360 | 0.311 | 23 |
| trans-Lycopene | 0.201 | 0.166 | တ | 0.138 | 0.122 | | 0.076 | 0.06 | 6 | 0.203 | 0.174 | 6 | 0.179 | 0.154 | 6 |
| Total β-Cryptoxanthin | 0.063 | 0.052 | 24 | 0.053 | 0.045 | . 4 | 0.041 | 0.03 | 24 | 0.065 | 0.055 | • | | 0.072 | 24 |
| Total α-Cryptoxanthin | 0.027 | 0.020 | 9 | 0.018 | 0.015 | 9 | 0.012 | 0.00 | 9 | 0.023 | 0.020 | 9 | | 0.021 | 9 |
| Total Lutein&Zeaxanthin | 0.149 | 0.144 | 23 | 0.09 | 60.0 | 23 | 0.04 | 0.04 | 22 | 0.104 | 0.098 | • | | 0.09 | 24 |

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...

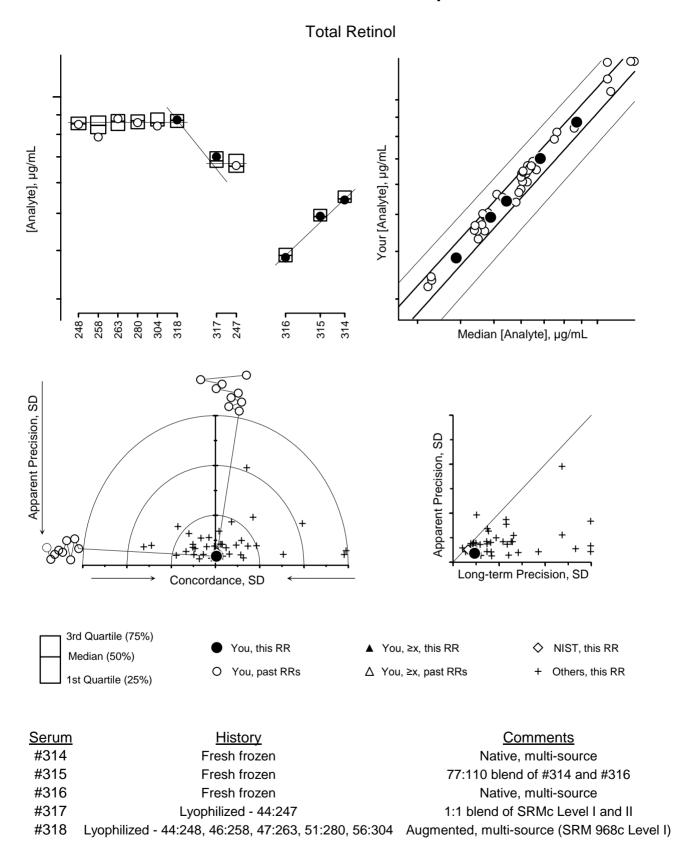
Micronutrients Measurement Quality Assurance Program National Institute of Standards and Technology 100 Bureau Drive Stop 8392 Gaithersburg, MD 20899-8392 USA

Tel: (301) 975-3935 Fax: (301) 977-0685 Email: david.duewer@nist.gov

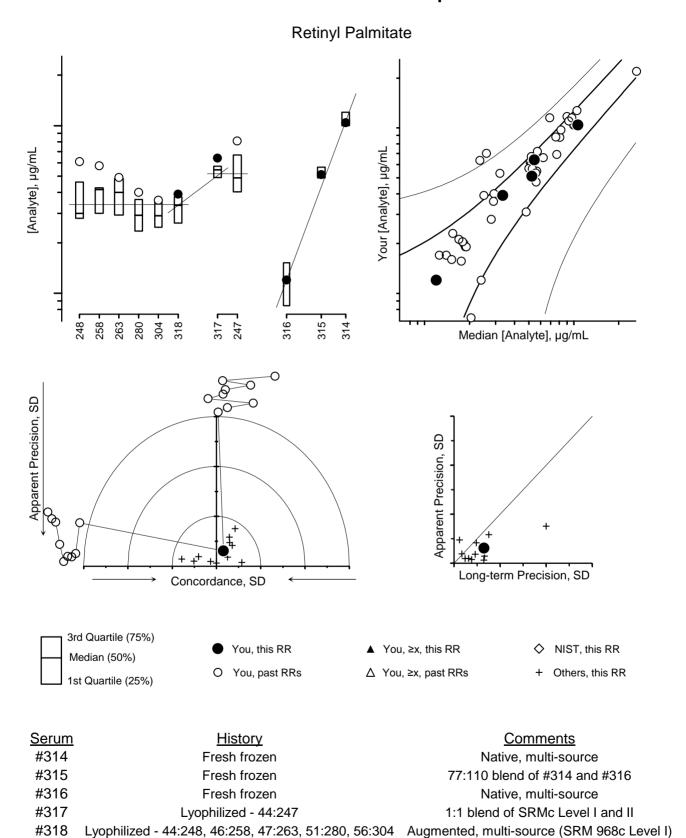
Email: david.duewer@nist.gov

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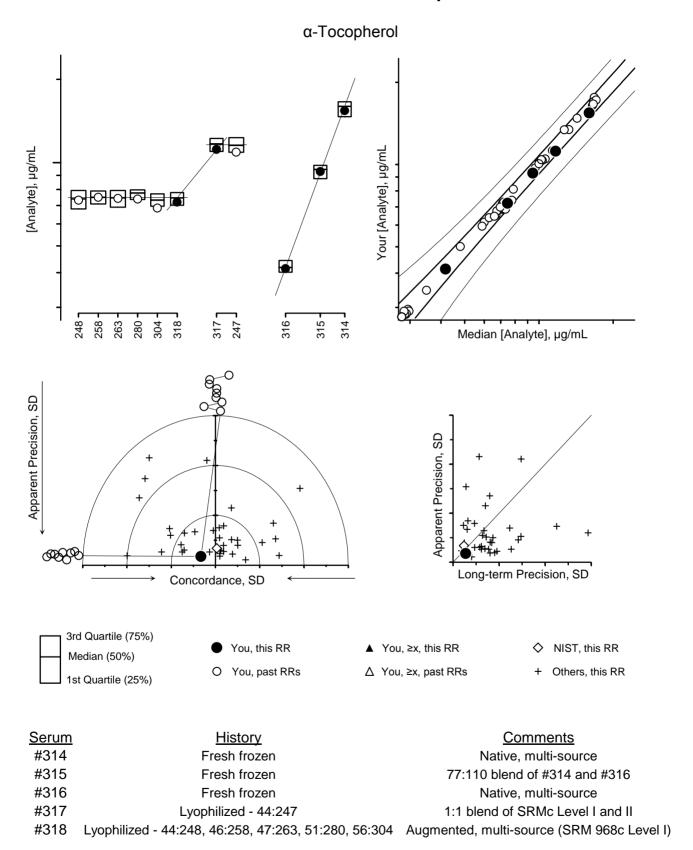
Individualized RR LVIII Report: FSV-BA

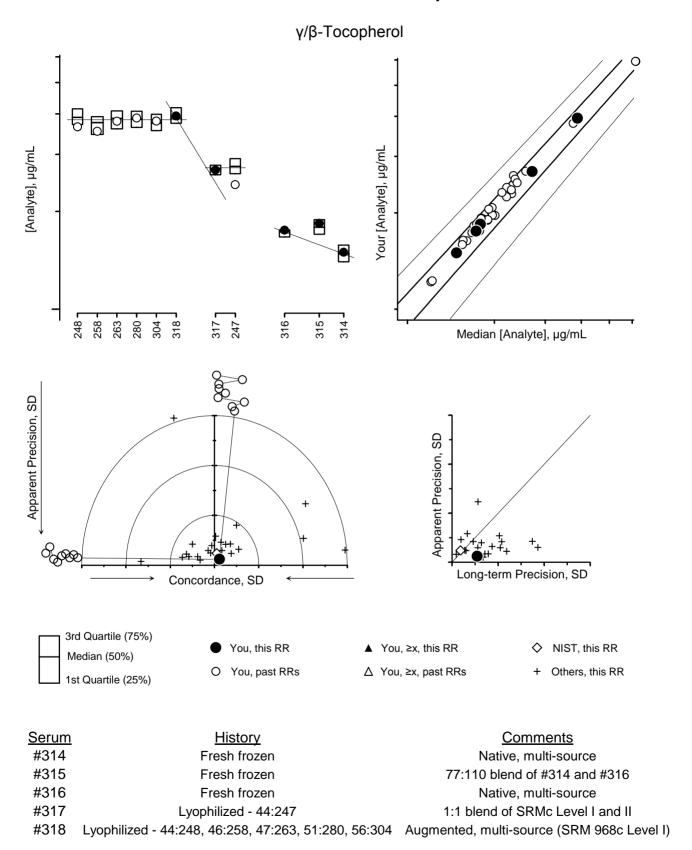


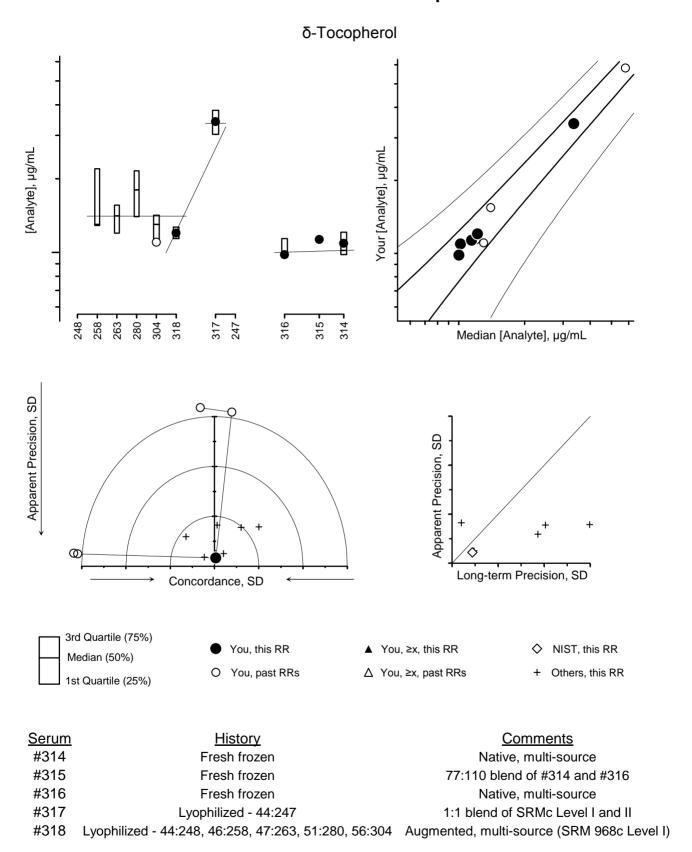
Individualized RR LVIII Report: FSV-BA

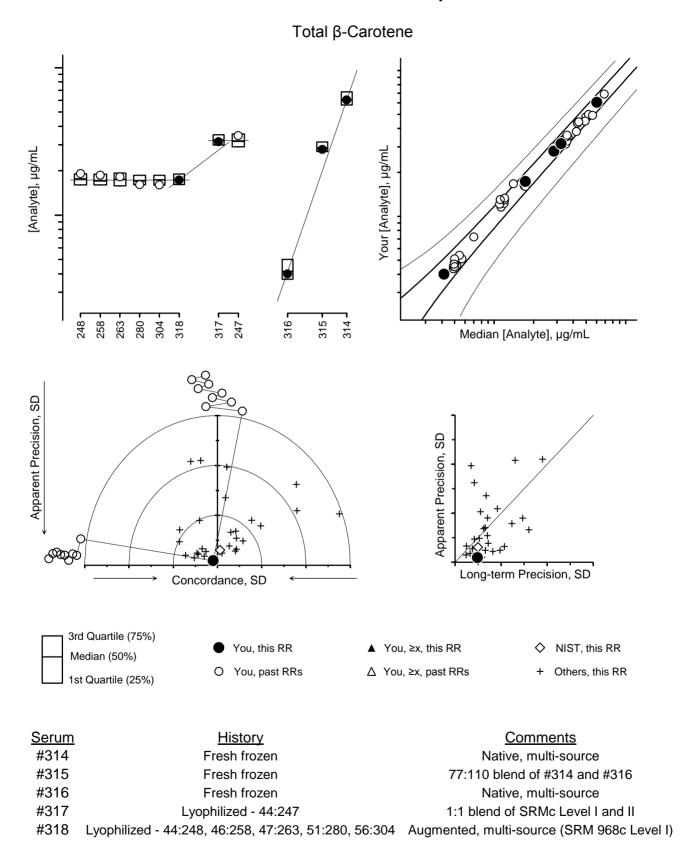


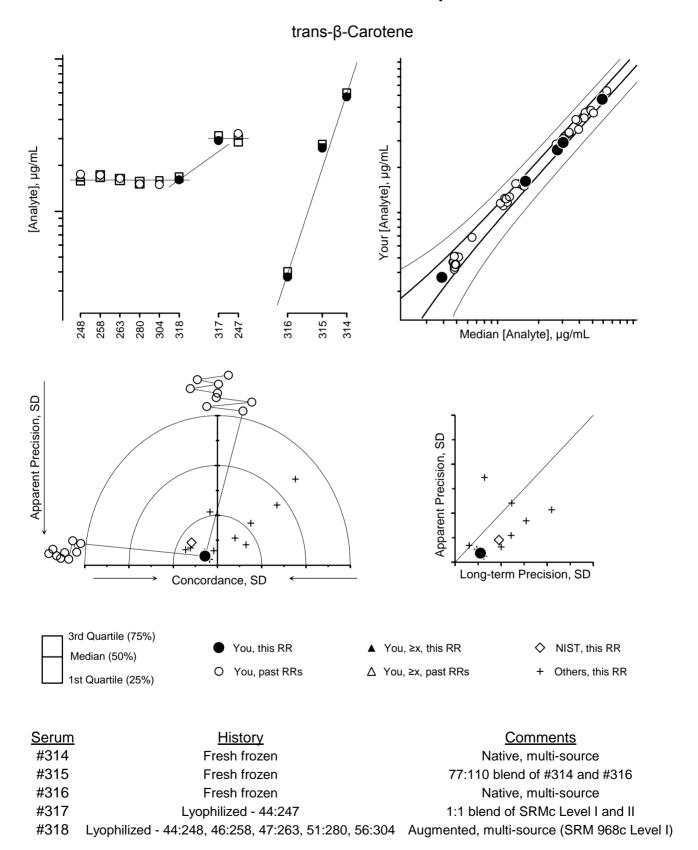
Individualized RR LVIII Report: FSV-BA

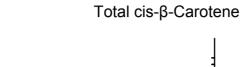


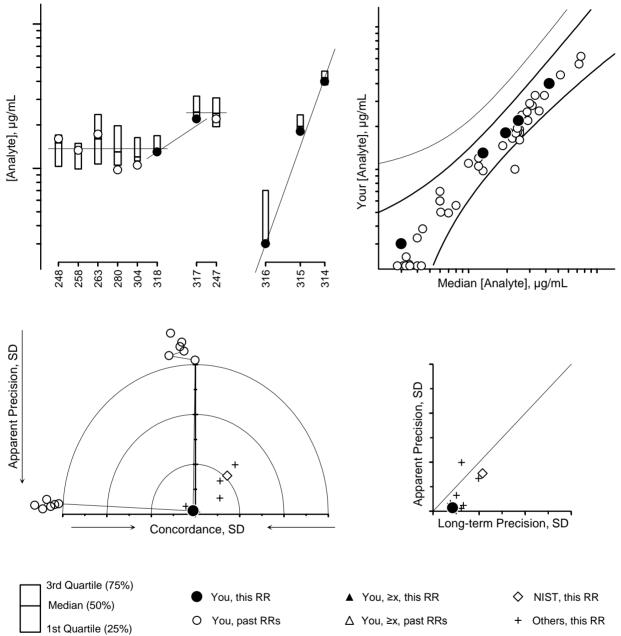




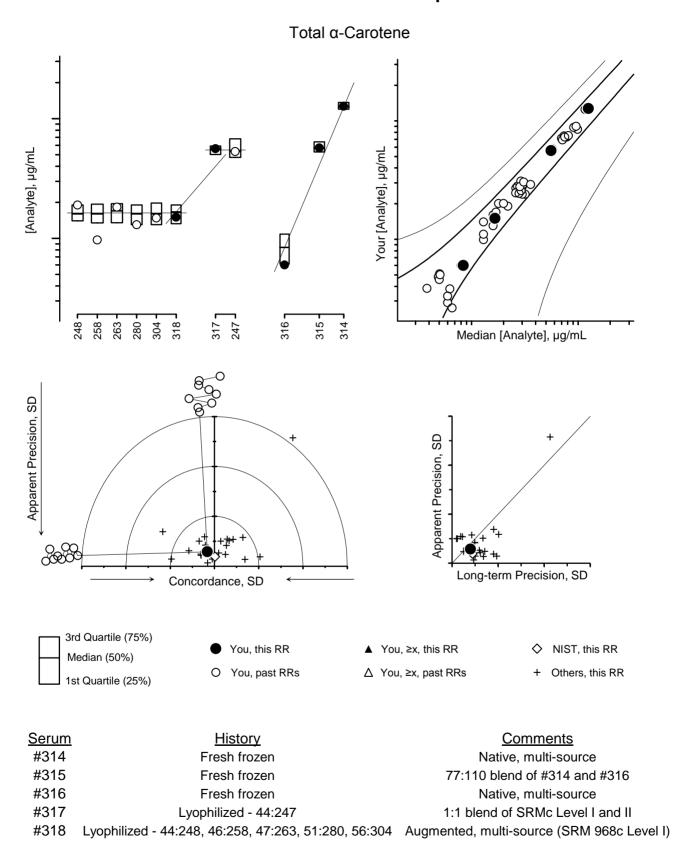


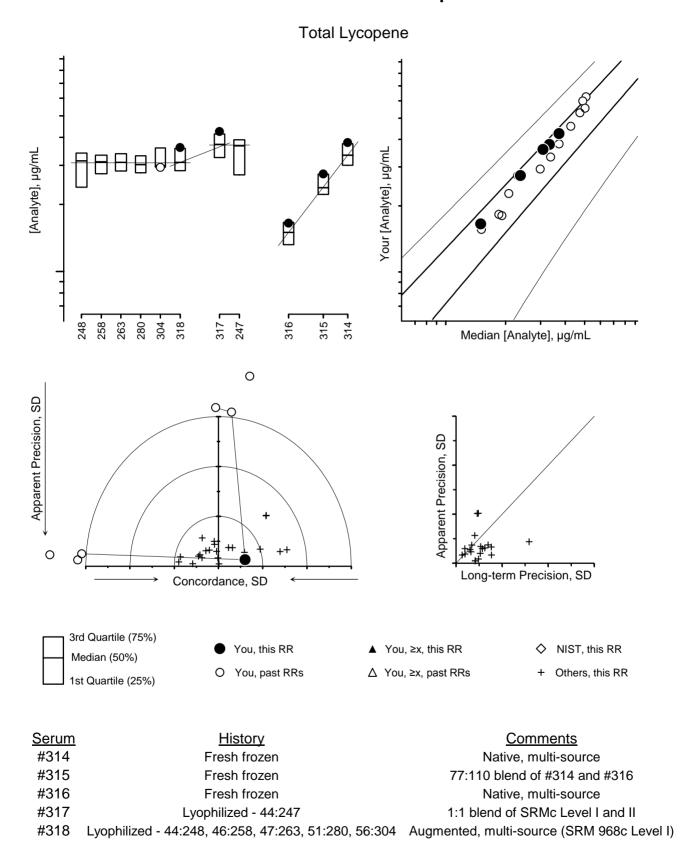


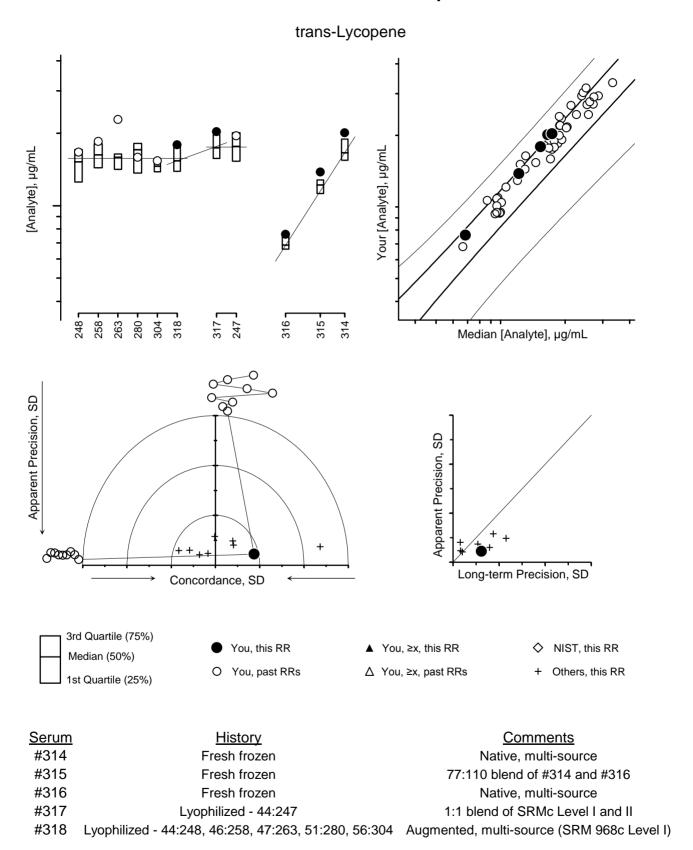




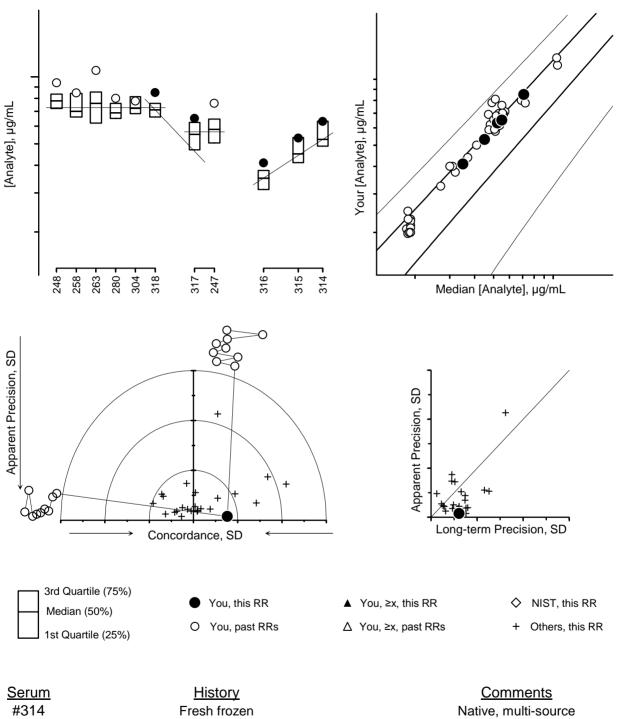
| Serum | <u>History</u> | <u>Comments</u> |
|-------|--|--|
| #314 | Fresh frozen | Native, multi-source |
| #315 | Fresh frozen | 77:110 blend of #314 and #316 |
| #316 | Fresh frozen | Native, multi-source |
| #317 | Lyophilized - 44:247 | 1:1 blend of SRMc Level I and II |
| #318 | Lyophilized - 44:248, 46:258, 47:263, 51:280, 56:304 | Augmented, multi-source (SRM 968c Level I) |



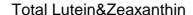


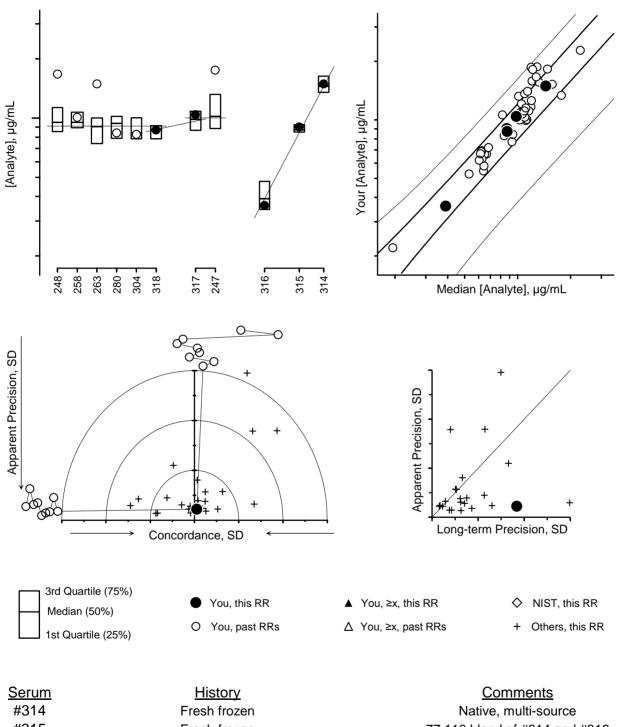




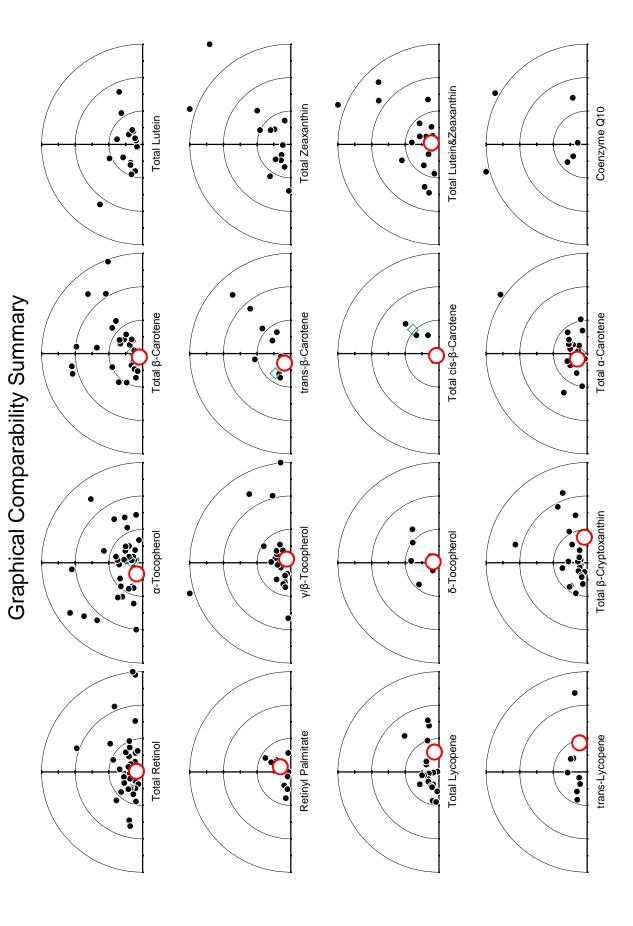


| <u>Serum</u> | <u>History</u> | <u>Comments</u> |
|--------------|--|--|
| #314 | Fresh frozen | Native, multi-source |
| #315 | Fresh frozen | 77:110 blend of #314 and #316 |
| #316 | Fresh frozen | Native, multi-source |
| #317 | Lyophilized - 44:247 | 1:1 blend of SRMc Level I and II |
| #318 | Lyophilized - 44:248, 46:258, 47:263, 51:280, 56:304 | Augmented, multi-source (SRM 968c Level I) |





Set 1 of 41



Appendix E. Shipping Package Inserts for RR23

The following five items were included in each package shipped to an RR23 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.

May 11, 2005

Dear Colleague:

The samples within this package constitute Vitamin C Round Robin 23 (RR23) of the fiscal year (FY) 05 Micronutrients Measurement Quality Assurance Program.

RR23consists of four vials of frozen serum test samples (#14, #31, #54, and #71), one vial of ascorbic acid solid control material (Control), and two vials of frozen serum control materials (Control #1 and Control #2). 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.)

The two serum control materials are a new component of the M^2QAP for Vitamin C. Please use these materials 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 Control #1 is 8.41 \pm 0.61 μ mol/L sample; the target value and \approx 95% confidence interval for Control #2 is 28.05 \pm 0.49 μ mol/L sample.

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.

The report for RR22 was mailed the week of April 14, 2005. If you find your results for RR22 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).

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 RR23 samples *before* **September 30, 2005**. We would appreciate receiving your results as soon as they become available. Please use the attached form. Your results will be kept confidential.

incerely,

Jeanige 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

RR22 Report Form for Ascorbic Acid Solid Control Material Preparation RR22 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 $[TAA]_{DS2} = 28.4 \text{ } \mu\text{mol/L}$ 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 calibrate 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: 301-975-3120 or Jeanice.BrownThomas@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 | #: |
|--------------------|----|
| | |

| D (| | |
|-------|--|--|
| Date: | | |
| Date | | |

Fax: 301-977-0685

Email: david.duewer@nist.gov

Vitamin C Round Robin 23

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] _{pga} | umol/I |

Please return by Sept 12, 2005

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

Vitamin C Round Robin 23 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 |
| Serum Control #1 | | | μmol/L of Sample Target: 8.5 ±0.5 μmol/L |
| Serum Control #2 | | | μmol/L of Sample Target: 28.1 ±1.0 μmol/L |
| Serum Test Sample #14 | | | μmol/L of Sample |
| Serum Test Sample #31 | | | μmol/L of Sample |
| Serum Test Sample #54 | | | μmol/L of Sample |
| Serum Test Sample #71 | | | μmol/L of Sample |
| | | | <u> </u> |

Were samples frozen upon receipt? Yes | No

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

COMMENTS:

Fax: 301-977-0685

Vitamin C Round Robin 23 NIST Micronutrients Measurement Quality Assurance Program Packing List and Shipment Receipt Confirmation Form

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

| Sample | Form |
|------------|-----------------------------------|
| VitC #14 | Liquid frozen (1:1 serum:10% MPA) |
| VitC #31 | Liquid frozen (1:1 serum:10% MPA) |
| VitC #54 | Liquid frozen (1:1 serum:10% MPA) |
| VitC #71 | Liquid frozen (1:1 serum:10% MPA) |
| Control #1 | Liquid frozen (1:1 serum:10% MPA) |
| Control #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 RR23

The following three 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.

November 8, 2005

Dear Colleague:

Enclosed is the summary report of the results for Round Robin 23 (RR23) 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 a summary of individual laboratory performance and interlaboratory accuracy and repeatability. 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 23 consisted of four *test samples* (#14, #31, #54, and #71), two *serum control materials*, 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 study of the 2006 M²QAP (RR24) will be shipped starting the last week of November 2005.

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

Research Chemist

Analytical Chemistry Division

Chemical Science and Technology Laboratory

Enclosures



The NIST M²QAP Vitamin C Round Robin 23 (RR23) report consists of

| Page | "Individualized" Report |
|------|--|
| | Summarizes your reported values for the nominal 55 mmol/L solution you prepared from the ascorbic acid solid control sample, the two serum control samples, and the four serum test samples. |
| 2 | Graphical summary of your RR23 sample measurements. |
| Page | "All Lab" Report |
| 1 | A tabulation of results and summary statistics for Total Ascorbic Acid [TAA] in the RR23 samples and control/calibration solutions. |

Serum-based Samples. Two serum controls and four unknowns were distributed in RR23.

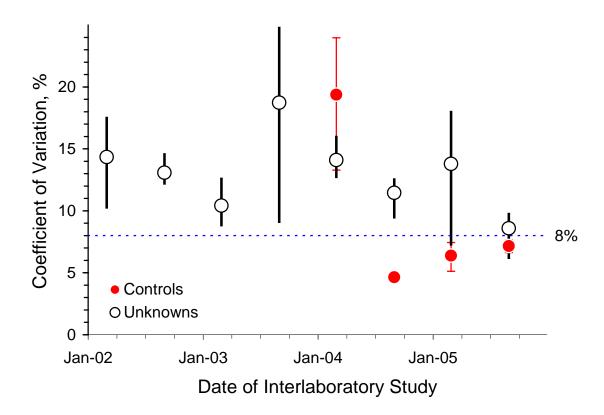
- CS1 SRM 970 level 1, ampouled in mid-1998.
- CS2 SRM 970 level 2, ampouled in mid-1998.
- S23:1 Serum 14, ampouled in late 2001, previously distributed as sample S19:1 (RR19, Sep-03) and S21:1 (RR21, Sep-04). A "blank" stripped serum.
- S23:2 Serum 31, ampouled in late 2001, previously distributed as sample S17:2 (RR17, Sep-02), S18:1 (RR18, Mar-03) S20:1 (RR20, Mar-04), and S22:1 (RR22, Mar-05). An augmented serum.
- S23:3 Serum 54, ampouled in late 2001, previously distributed as sample 16:3 (RR13, Mar-02), S17:3 (RR17, Sep-02), S20:2 (RR20, Mar-04), and S21:4 (RR21, Sep-04). An augmented serum.
- S23:4 Serum 71, SRM 970 level 1, ampouled in mid-1998. This material was distributed with identification in RR11 (Oct-98) and RR12 (Mar-99) and as samples S13-1 (RR13, Mar-00), S14-3 (RR14, Mar-01), S15:1 (RR15, Sep-01), S16:1 (RR16, Mar-02), S19:4 (Sep-03), and S20:3 (Mar-04). An augmented serum.

Results.

- 1) All participants who prepared the four control/calibration solutions (the three dilute solutions and the 5% MPA diluent) did so correctly. The criteria used to evaluate this success are: the density of the 5% MPA (\approx 1.03 gm/mL), the observed wavelength maximum of "Dilute Solution #1"(\approx 244 nm), the observed absorbance at that maximum (\approx 0.55 OD), the calculated E^{1%} #1"(\approx 550 dL/g·cm).
- 2) Judging from the calibration parameters calculated for the control/calibration solutions (intercepts close to 0.0 and slopes close to 1.0), the measurement systems for all participants are well calibrated.
- 3) There is no evidence of sample degradation with any of the samples distributed.
- 4) The following Figure displays the expected %relative standard deviation or "coefficient of variation" (CV) for the control and unknown samples as a function of time for the past four years. The expected CV is estimated by pooling the individual CVs of all non-blank samples in a given study. The individual sample CVs are estimated as 100×MADe/median. The "error bars" span from the lowest to the highest observed CVs.

While the data are as yet insufficient for any great confidence, the general reduction in the expected CV with time since the introduction of the control samples in 2004 suggests that analysis of the

controls helps control the observed variability of the unknown samples. Based on the convergence of the expected CV of the control materials with that for the unknowns, the currently achievable amonglaboratory CV for the analysis of total ascorbic acid is about 8%.



Appendix G. "All-Lab Report" for RR23

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" 22 - March 2005

| ı | | | ' | ~ | <u></u> | - | _ | _ | | _ | | | ì | 6 | _ | _ | 10 | ~ | ~ | _ | _ | ~ | - |
|-------------------------------|--------------------|--------------------|----------|----------|----------|----------|----------|----------|----------|----------|----------|----------|---|----|---------|--------|---------|---------|--------|---------|---------|--------|------|
| | | \$23:4 | 8.6 | 8.8 | 7.6 | 8.4 | 8.9 | 8.7 | 8.8 | 11.0 | 10.7 | | | 5 | 9.1 | 1.1 | 7.6 | 8.6 | 8.8 | 8.6 | 11.0 | 0.3 | 4 |
| | | S23:3 | 45.9 | 45.5 | 45.5 | 46.8 | 55.7 | 49.3 | 43.5 | 47.7 | 51.6 | | | 6 | 47.9 | 3.7 | 43.5 | 45.5 | 46.8 | 49.3 | 55.7 | 1.9 | 4 |
| | µmol/L | S23:2 | 23.5 | 23.5 | 20.5 | 21.5 | 26.0 | 23.4 | 19.8 | 24.0 | 21.6 | | | 6 | 22.6 | 1.9 | 19.8 | 21.5 | 23.4 | 23.5 | 26.0 | 5.6 | 11 |
| | Corrected, µmol/ | \$23:1 | 0.5 | 0.7 | 0.0 | 0.0 | 6.0 | 0.3 | 0.7 | 3.5 | 1.5 | | | 6 | 6.0 | 1.1 | 0.0 | 0.3 | 0.7 | 6.0 | 3.5 | 0.5 | 77 |
| | S | CS#2 | 28.1 | 30.0 | 29.1 | 26.5 | 31.3 | 27.5 | 28.5 | 29.6 | 32.1 | | | 6 | 29.2 | 1.8 | 26.5 | 28.1 | 29.1 | 30.0 | 32.1 | 1.5 | 2 |
| les | | CS#1 (| 9.2 | 9.8 | 8.1 | 7.7 | 9.0 | 8.3 | 9.0 | 10.2 | 10.0 | | | 6 | 8.9 | 0.8 | 7.7 | 8.3 | 9.0 | 9.2 | 10.2 | 1: | 12 |
| Samples | | S23:4 | 8.7 | 7.7 | 8.1 | 8.9 | 7.9 | 9.3 | 8.2 | 11.4 | 12.7 | 8.5 | | 10 | 9.1 | 1.6 | 7.7 | 8.1 | 9.8 | 9.5 | 12.7 | 0.8 | 10 |
| | | \$23:3 | 49.3 | 42.6 | 45.3 | 46.9 | 54.2 | 48.3 | 43.3 | 48.7 | 52.5 | 46.2 | | 10 | 47.7 | 3.7 | 42.6 | 45.5 | 47.6 | 49.1 | 54.2 | 3.0 | 9 |
| | µmol/L | S23:2 S | 24.9 | 21.7 | 20.7 | 21.8 | 24.8 | 23.4 | 19.3 | 24.6 | 23.3 | 21.6 | | 10 | 22.6 | 1.9 | 19.3 | 21.6 | 22.6 | 24.3 | 24.9 | 2.1 | 6 |
| | Measured, µmol/I | S23:1 S | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 1.3 | 0.0 | 3.8 | 3.8 | 0.0 | | 10 | 6.0 | 1.6 | 0.0 | 0.0 | 0.0 | 6.0 | 3.8 | 0.0 | |
| | Ž | CS#2 | 30.0 | 27.9 | 29.2 | 26.8 | 30.1 | 27.4 | 28.1 | 30.3 | 33.6 | 28.0 | | 10 | 29.1 | 2.0 | 26.8 | 27.9 | 28.6 | 30.0 | 33.6 | 1.9 | 7 |
| | | CS#1 (| 9.4 | 7.5 | 9.8 | 8.2 | 8.0 | 8.9 | 8.5 | 10.6 | 12.0 | 8.4 | | 10 | 9.0 | 1.4 | 7.5 | 8.2 | 8.5 | 9.3 | 12.0 | 0.7 | 8 |
| _ | 2 | E ^{1%} | 6.795 | 597.8 | 2.975 | 565.5 | 539.5 | 545.4 | | 343.9 | 567.5 | | | 80 | 538.0 | 80.5 | 343.9 | 543.9 | 566.5 | 570.1 | 597.8 | 23.2 | 4.1 |
| Dilute Solution 1 | Spectrophotometry | A _{max} | 0.5621 | 0.6247 | 0.5831 | 0.5798 | 0.5420 | 0.4538 | | 0.3680 | 0.5947 | | | 8 | 0.5385 | 0.0854 | 0.3680 | 0.5200 | 0.5710 | 0.5860 | 0.6247 | 0.0391 | 8.9 |
| Dilute (| Spectro | λ _{max} / | 244. 0 | | | 243.6 0 | | | | 254.4 0 | 244. 0 | | | 8 | 245.0 0 | 3.8 0 | 243.0 0 | 243.5 0 | | 244.0 0 | 254.4 0 | 0.3 | 0.12 |
| | ^ | | | | | | | | 52 | | | | | 6 | | 22 | | | | | | 1 | |
| MPA | Density | g/mL | 1.03 | 1.0 | 1.03 | 1.030 | 1.03 | 1.02 | 1.02 | 1.0 | 1.03 | | | | 1.029 | 0.005 | | | | | 1.035 | | 0.12 |
| | S | SEE | 1.5 | 0.9 | 1.0 | 0.7 | 1.0 | 1.1 | 0.7 | 0.3 | 0.4 | | | z | Average | SD | Min | %52 | Median | %75 | Max | eSD | ွဲ |
| | arameters | \mathbb{R}^2 | 0.998 | 0.999 | 0.999 | 1.000 | 0.999 | 0.998 | 0.999 | 1.000 | 1.000 | | | | ∢ | | | | _ | | | | |
| | Calibration Parar | Slope | 1.09 | 0.95 | 0.98 | 0.99 | 0.99 | 96.0 | 1.01 | 1.02 | 0.97 | | | | | | | | | | | | |
| ples | Calit | Inter | -0.59 | -0.66 | 0.63 | 0.58 | -0.87 | 0.95 | -0.67 | 0.20 | 2.25 | | | | | | | | | | | | |
| ın Sam | | MPA | 9.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.2 | 1.9 | | | 6 | 0.3 | 0.6 | 0.0 | 0.0 | 0.0 | 0.2 | 1.9 | 0.0 | |
| alibratic | mol/I | Dil:3 | 13.6 | 13.4 | 14.7 | 15.2 | 13.9 | 13.0 | 13.0 | 16.0 | 16.8 | | | 6 | 14.4 | 1.4 | 13.0 | 13.4 | 13.9 | 15.2 | 16.8 | 1.3 | 6 |
| Control / Calibration Samples | Measured, µmol/ | Dil:2 | 28.4 | 26.1 | 29.6 | 29.6 | 28.0 | 24.0 | 27.6 | 31.4 | 31.3 | | | 6 | 28.4 | 2.4 | 24.00 | 27.60 | 28.35 | 29.64 | 31.40 | 1.9 | 7 |
| Co | Š | Dil:1 | 61.3 | 56.3 | 56.4 | 67.3 | 56.1 | 45.9 | 57.4 | 61.8 | 60.1 | | | 6 | 57.0 | 4.7 | 45.9 | 56.3 | 57.4 | 60.1 | 61.8 | 1.9 | 3 |
| | J/Joi | Dil:3 | 13.7 | 15.1 | 14.2 | 14.1 | 15.8 | 11.6 | 14.1 | 15.8 | 14.5 | | | 6 | 14.3 | 1.3 | 11.6 | 14.1 | 14.2 | 15.1 | 15.8 | 0.7 | 5 |
| | Gravimetric, µmol/ | Dil:2 | 27.8 | 29.1 | 28.5 | 29.5 | 29.8 | 23.5 | 28.4 | 30.4 | 29.6 | | | 6 | 28.5 | 2.0 | 23.53 | 28.39 | 29.14 | 29.58 | 30.38 | 1.0 | 3 |
| | Gravim | Dil:1 | 56.2 | 59.3 | 57.4 | 58.2 | 57.0 | 47.2 | 57.2 | 8.09 | 59.5 | | | 6 | 57.0 | 3.9 | 47.2 | 57.0 | 57.4 | 59.3 | 8.09 | 1.8 | 3 |
| | 7 | | 17/10/05 | 25/05/05 | 20/90/70 | 20/00/02 | 22/09/05 | 03/10/05 | 12/09/05 | 24/06/05 | 20/60/60 | 12/08/05 | | z | Average | SD | Min | %25 | Median | %75 | Max | MADe | ટે |
| | | Date | | | Ŭ | | | _ | ` | | _ | • | | | Ave | | | | ž | | | 2 | |
| | | Lab | VC-MA | VC-ME | VC-MC | VC-ME | VC-MG | VC-Mh | VC-M | VC-MJ | VC-MK | VC-MP | | | | | | | | | | | |

Appendix H. Representative "Individualized Report" for RR23

Each participant in RR23 received an "Individualized Report" reflecting their reported results. The following two pages are the "Individualized Report" for participant "VC-MA".

Vitamin C "Round Robin" 23 Report: Participant VC-MA

Dilute Solution 1

Control/Calibration Solutions

Fax: (301) 977-0685

Email: david.duewer@nist.gov

MPA

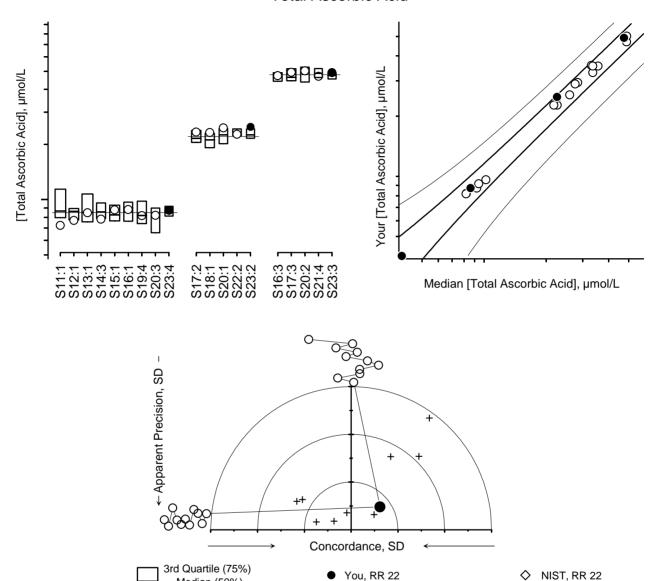
| | | | IVIPA | | | ie Solutio | | Control/Calibration Solutions | | | | | | |
|----------------------|----------|--------------|--------------|------------------|------------------------|--------------|-----------------|-------------------------------|-----------------------|----------------------|---------------------|--|--|--|
| | | | Density | | Spect | trophotoi | - | Y, | _{meas} = Int | er + Slope | * X _{grav} | | | |
| Date | RR | Method | g/mL | | λ_{max} | A_{max} | E ^{1%} | Inter | Slope | R^2 | SEE | | | |
| 03/20/03 | 18 | HPLC-EC | 1.026 | | 244.0 | 0.509 | 563.1 | -0.1 | 1.02 | 1.000 | 0.18 | | | |
| 11/13/03 | 19 | HPLC-EC | 1.026 | | 243.0 | 0.584 | 561.9 | 1.1 | 1.03 | 0.998 | 1.24 | | | |
| 02/23/04 | 20 | HPLC-EC | 1.031 | | 243.0 | 0.552 | 560.7 | -0.4 | 1.05 | 1.000 | 0.65 | | | |
| 09/13/04 | 21 | HPLC-EC | 1.030 | | 244.0 | 0.555 | 562.2 | -0.1 | 0.99 | 1.000 | 0.10 | | | |
| 03/08/05 | 22 | HPLC-EC | 1.034 | | 243.0 | 0.559 | 562.9 | 0.2 | 1.06 | 1.000 | 0.24 | | | |
| 10/17/05 | 23 | HPLC-EC | 1.030 | | 244.0 | 0.562 | 567.9 | -0.6 | 1.09 | 0.998 | 1.47 | | | |
| | | Mean | 1.029 | | 243.5 | 0.55 | 563.1 | | | | 0.65 | | | |
| | | SD | 0.003 | | 0.5 | 0.02 | 2.5 | | | | 0.58 | | | |
| | | CV | 0.29 | | 0.22 | 4.4 | 0.4 | | | | | | | |
| | | | | [TAA] r | nmol/Ls | ample | | | | | | | | |
| Date | RR | Sample | Rep₁ | Rep ₂ | F_{adj} | Mean | SD_{dup} | N | Mean | SD _{repeat} | SD_{reprod} | | | |
| 02/23/04 | 20 | CS#1 | 7.8 | 8.0 | 1.0 | 7.9 | 0.1 | 4 | 8.5 | 0.1 | 0.7 | | | |
| 09/13/04 | 21 | CS#1 | 8.1 | 7.9 | 1.0 | 8.0 | 0.1 | | | | | | | |
| 03/08/05 | 22 | CS#1 | 8.5 | 8.7 | 1.0 | 8.6 | 0.1 | | | | | | | |
| 10/17/05 | 23 | CS#1 | 9.3 | 9.5 | 1.0 | 9.4 | 0.1 | | | | | | | |
| 00/00/04 | 00 | 00.40 | 05.0 | 00.0 | 4.0 | 00.0 | 0.0 | | 07.0 | 0.0 | 4.0 | | | |
| 02/23/04 | 20 | CS#2 | 25.8 | 26.2 | 1.0 | 26.0 | 0.3 | 4 | 27.9 | 0.6 | 1.9 | | | |
| 09/13/04 03/08/05 | 21 22 | CS#2 CS#2 | 26.2 29.0 | 27.2 29.0 | 1.0 | 26.7 29.0 | 0.7 | | | | | | | |
| 10/17/05 | 23 | CS#2 CS#2 | 29.0 | | 1.0 | 30.0 | 0.0 | | | | | | | |
| 10/17/05 | 23 | C5#2 | 29.4 | 30.5 | 1.0 | 30.0 | 8.0 | | | | | | | |
| 11/13/03 | 19 | S19:1 | nd | nd | 1.0 | | | 1 | 0.0 | 0.0 | | | | |
| 09/13/04 | 21 | S21:1 | nd | nd | 1.0 | | | | | | | | | |
| 10/17/05 | 23 | S23:1 | 0.0 | 0.0 | 1.0 | 0.0 | 0.0 | | | | | | | |
| | | | | | | | | | | | | | | |
| 03/20/03 | 18 | S18:1 | 22.7 | 23.7 | 1.0 | 23.2 | 0.7 | 4 | 23.9 | 0.6 | 1.1 | | | |
| 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 | | | | | | | |
| 02/23/04 | 20 | S20:2 | 50.6 | 50.0 | 1.0 | 50.3 | 0.4 | 3 | 48.9 | 0.5 | 1.7 | | | |
| 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 | | | | | | | |
| | | | | | | _ | | | | | | | | |
| 11/13/03 | 19 | S19:4 | 7.8 | 8.6 | 1.0 | 8.2 | 0.5 | 3 | 8.4 | 0.3 | 0.4 | | | |
| 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 | | | | | | | |
| | | | | | | | | | | | | | | |

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

Vitamin C "Round Robin" 23 Report: Participant VC-MA





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.

You, RR 17-21

Others, RR 22

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

Median (50%)

1st Quartile (25%)

S23:1 Serum 14, a "blank" previously distributed in RRs 16, 19, 21, and 22

S23:2 Serum 31, previously distributed in RRs 17, 18, 20, and 22

S23:3 Serum 54, previously distributed in RRs 16, 17, 20, and 21

S23:4 SRM 970 Level 1, previously distributed in RRs 11, 12, 13, 14, 15, 16, 19, and 20