

**NISTIR 7880-23**

**NIST Micronutrients Measurement  
Quality Assurance Program  
Winter 2001  
Comparability Studies**

Results for Round Robin XLIX  
Fat-Soluble Vitamins and Carotenoids in Human Serum  
and Round Robin 14 Ascorbic Acid in Human Serum

David L. Duewer  
Sam A. Margolis (Retired)  
Katherine E. Sharpless  
Jeanice B. Thomas

<http://dx.doi.org/10.6028/NIST.IR.7880-23>



NISTIR 7880-23

**NIST Micronutrients Measurement  
Quality Assurance Program  
Winter 2001  
Comparability Studies**

Results for Round Robin XLIX  
Fat-Soluble Vitamins and Carotenoids in Human Serum  
and Round Robin 14 Ascorbic Acid in Human Serum

David L. Duewer  
Sam A. Margolis (Retired)  
Katherine E. Sharpless  
Jeanice B. Thomas  
*Chemical Sciences Division  
Materials Measurement Laboratory*

<http://dx.doi.org/10.6028/NIST.IR.7880-23>

July, 2013



U.S. Department of Commerce  
*Penny Pritzker, Secretary*

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

(This page intentionally blank)

## **Abstract**

The National Institute of Standards and Technology coordinates the Micronutrients Measurement Quality Assurance Program (MMQAP) for laboratories that measure fat- and water-soluble vitamins and carotenoids in human serum and plasma. This report describes the design of and results for the Winter 2001 MMQAP measurement comparability improvement studies: 1) Round Robin XLIX Fat-Soluble Vitamins and Carotenoids in Human Serum and 2) Round Robin 14 Total Ascorbic Acid in Human Serum. The materials for both studies were shipped to participants in January 2001. Participants in Round Robin XLIX were requested to provide their measurement results by March 16, 2001. Participants in Round Robin 14 were requested to provide their measurement results by May 12, 2001.

## **Keywords**

Human Serum

Retinol,  $\alpha$ -Tocopherol,  $\gamma$ -Tocopherol, Total and *Trans*- $\beta$ -Carotene  
Total Ascorbic Acid

## Table of Contents

<b>Abstract</b> .....	iii
<b>Keywords</b> .....	iii
<b>Table of Contents</b> .....	iv
<b>Introduction</b> .....	1
<b>Round Robin XLIX: Fat-Soluble Vitamins and Carotenoids in Human Serum</b> .....	1
<b>Round Robin 14: Vitamin C in Human Serum</b> .....	2
<b>References</b> .....	3
<b>Appendix A. Shipping Package Inserts for RR49</b> .....	A1
<b>Appendix B. Final Report for RR49</b> .....	B1
<b>Appendix C. “All-Lab Report” for RR49</b> .....	C1
<b>Appendix D. Representative “Individualized Report” for RR49</b> .....	D1
<b>Appendix E. Shipping Package Inserts for RR14</b> .....	E1
<b>Appendix F. Final Report for RR14</b> .....	F1
<b>Appendix G. “All-Lab Report” for RR14</b> .....	G1
<b>Appendix H. Representative “Individualized Report” for RR14</b> .....	H1

## Introduction

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

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

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

### **Round Robin XLIX: Fat-Soluble Vitamins and Carotenoids in Human Serum**

Participants in the MMQAP Fat-Soluble Vitamins and Carotenoids in Human Serum Round Robin XLIX comparability study (hereafter referred to as RR49) received four lyophilized and one liquid-frozen human serum test samples for analysis. Unless multiple vials were previously requested, participants received one vial of each serum. These sera were shipped on dry ice to participants in January 2001. 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 RR49 consists of three documents:

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

- An “Individualized Report” that graphically analyzes each participant’s results for selected analytes. 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 14: Vitamin C in Human Serum**

Participants in the MMQAP Vitamin C in Human Serum Round Robin 14 comparability study (hereafter referred to as RR14) received four frozen serum test samples 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 January 2001. The communication materials included in the sample shipment are provided in Appendix E.

The test 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 RR14 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 RR49**

The following three items were included in each package shipped to an RR49 participant:

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

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



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

January 16, 2001

Dear Colleague:

Happy New Year! Enclosed is the first set of samples for the quality assurance round robin exercise (Round Robin XLIX) for 2001. You will find one vial of each of one liquid-frozen and four lyophilized serum samples for analysis along with a form for reporting your results. When reporting your results, please submit one value for each analyte for a given serum sample. If an obtained value is below your limit of quantitation, please indicate this result on the form by using NQ (*Not Quantitated*). For analytes not measured, please leave a blank. Results are due to NIST by **March 16, 2001**. Results received 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 provided during the week of April 9, 2001.

Lyophilized samples should be reconstituted with 1.0 mL of HPLC-grade water or equivalent. We recommend that dissolution be facilitated with 3 to 5 min agitation in an ultrasonic bath or at least 30 min at room temperature with intermittent swirling. (CAUTION: Vigorous shaking will cause foaming and possibly interfere with accurate measurement. The rubber stopper contains phthalate esters that will 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). Liquid-frozen sample 271 should **not be** reconstituted.

For consistency, we request that laboratories use the following absorptivities (E 1% cm) in ethanol: retinol, 1843 at 325 nm; retinyl palmitate, 975 at 325 nm;  $\alpha$ -tocopherol, 75.8 at 292 nm;  $\gamma$ -tocopherol, 91.4 at 298 nm;  $\alpha$ -carotene, 2800 at 444 nm (in hexane);  $\beta$ -carotene, 2560 at 450 nm (in ethanol), 2592 at 452 nm (in hexane); lycopene, 3450 at 472 nm (in hexane).

Please mail or fax your results for Round Robin XLIX to:

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

If you have questions regarding this round robin exercise, please call me at (301) 975-3120; e-mail me at [jbthomas@nist.gov](mailto: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 XLIX**  
**NIST Micronutrients Measurement Quality Assurance Program**

Analyte	269	270	271	272	273	Units*
total retinol						
trans-retinol						
retinyl palmitate						
α-tocopherol						
γ-tocopherol						
δ-tocopherol						
total β-carotene						
trans-β-carotene						
total cis-β-carotene						
total α-carotene						
trans-α-carotene						
total lycopene						
trans-lycopene						
total β-cryptoxanthin						
total α-cryptoxanthin						
total lutein						
total zeaxanthin						
total lutein&zeaxanthin						
ubiquinone-10 (Q <sub>10</sub> )						
phylloquinone (K <sub>1</sub> )						
25-hydroxyvitamin D						
cholesterol						
Other analytes?						

\* we prefer µg/mL

Was serum 271 frozen when received? Yes | No

Mail: M<sup>2</sup>QAP  
 NIST, Stop 8392  
 Gaithersburg, MD 20899-8392

Please return results on-or-before  
 16-Mar-2001

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

Participant #: \_\_\_\_\_

Date: \_\_\_\_\_

**Round Robin XLIX**  
NIST Micronutrients Measurement Quality Assurance Program  
Packing List and Shipment Receipt Confirmation Form

The enclosed bubble-pack (should) contain one vial each of the following **five** sera:

<u>Serum</u>	<u>Form</u>	<u>Reconstitute?</u>
#269	Lyophilized	Yes (1 ml H <sub>2</sub> O)
#270	Lyophilized	Yes (1 ml H <sub>2</sub> O)
#271	Liquid frozen	No
#272	Lyophilized	Yes (1 ml H <sub>2</sub> O)
#273	Lyophilized	Yes (1 ml H <sub>2</sub> O)

- Please**
- 1) Open the pack immediately
  - 2) Check that it contains one vial each of the above samples
  - 3) Check if serum #271 arrived frozen
  - 4) Store the RR XLIX samples upright at -20 °C or below until analysis
  - 5) Complete the following information
  - 6) Fax the completed form to us at 301-977-0685  
(or email requested information to david.duewer@nist.gov)

1) Date this shipment arrived: \_\_\_\_\_

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

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

4) Did serum #271 arrive frozen? Yes | No

5) At what temperature are you storing the samples? \_\_\_\_\_ °C

6) When do you anticipate analyzing these samples? \_\_\_\_\_

Thank you in advance for your prompt response.

The M<sup>2</sup>QAP Gang

## **Appendix B. Final Report for RR49 and Folate Questionnaire**

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

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

The fifth page of this section is a questionnaire that was distributed in the same mailing as the final report. This questionnaire was intended to assess the level of interest in the measurement of folates in serum and plasma.



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

April 25, 2001

Dear Colleague:

Enclosed is the summary report of the results for Round Robin XLIX (RR 49) for fat-soluble vitamins and carotenoids. Included in this report are: a summary of data for all laboratories; the measurement comparability summary for evaluating laboratory performance; lyophilized vs. fresh-frozen commutability data, a summary of individual laboratory performance and interlaboratory accuracy and precision; and a summary of the NIST assigned value (NAV) vs. your laboratory value for the analytes that you measured. As in previous reports, 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 RR 49 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 deviation(s) 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.

The following are newly released or forthcoming publications. Reprints will be provided upon request.

***"Preparation and Value Assignment of Standard Reference Material 968c, Fat-Soluble Vitamins, Carotenoids, and Cholesterol in Human Serum,"*** Brown Thomas et al., *Clinica Chimica Acta* 305 (2001) 141-155.

***"The Stability of Ascorbic Acid in Autosampler Vials,"*** Margolis, S.A. and Park, E., accepted by *Clinical Chemistry*, March 2001.

The NIST Micronutrients Measurement Quality Assurance Workshop was held on April 4, 2001 as a symposium at the Experimental Biology (EB) meeting in Orlando, Florida. At the workshop, results from the past three interlaboratory comparison exercises (Round Robins 47-49), methodology updates, and program needs were discussed. There were 41 attendees at the workshop. As discussed at the session, the "better-than-expected" attendance was thought to be due to the workshop being held at the end of the conference instead of at the beginning. Since the EB meetings now begin a day earlier (on Saturday) than in the past, having the QA workshop at the end of the conference allows conferees to attend the EB meetings first and focus on the QA workshop afterwards. It was agreed that the next workshop will be held in conjunction with EB, which provides the appropriate audience, and will be held at the end of the conference. It was also suggested that our next QA workshop be held at EB 2003 in San Diego, CA. You will be notified about the next QA workshop as plans are made.

Most of the discussion for the afternoon session at the workshop concerned the measurement of folates in serum. Attendees expressed that quality assurance of folate measurements in serum and a folate-in-serum/plasma reference material are needed, and that they would like to start participating in round robin studies for folates in serum/plasma. To help us assess the level of interest in the measurement of folates in serum/plasma among the current QA program participants, **please return the enclosed questionnaire by May 31.**

The NIST logo, consisting of the letters 'NIST' in a bold, sans-serif font.

Another topic discussed at the workshop was the need to have more than one vitamin C round robin study so that laboratories can meet the requirements of the Clinical Laboratory Improvement Amendments (CLIA). Since there were not enough representatives from laboratories that routinely measure vitamin C present at the workshop, a decision about increasing the number of studies could not be made at that time. Again, we would appreciate your feedback regarding this issue by completing the enclosed questionnaire. Additionally, please be reminded that **SRM 970, Vitamin C in Frozen Human Serum**, is now available. This material can be purchased through the Standard Reference Materials Program at 301-975-6776; fax: 301-948-3730; or at [srminfo@nist.gov](mailto:srminfo@nist.gov).

If you have any questions regarding this report, please contact David Duewer at 301/975-3935; e-mail: [david.duewer@nist.gov](mailto:david.duewer@nist.gov), or me at 301/975-3120; e-mail: [jbthomas@nist.gov](mailto:jbthomas@nist.gov); fax: 301/977-0685.

Sincerely,



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

cc:

L. C. Sander  
S.A. Wise

Enclosures



The NIST M<sup>2</sup>QAP Round Robin XLIX (RR49) report consists of

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 10 other participants.
n+1	The “target” plot version of your “Comparability Summary” scores.
n+2	An experimental “Commutability” plot summarizing your measurements in the five liquid-frozen / lyophilized sample pairs distributed in RR47, RR48, and RR49.
Page	“All Lab” Report
1-4	A listing of all results and statistics for analytes reported by at least two laboratories
5	A list of results for the four analytes reported by only one laboratory. A legend for the above two lists
6	The “Comparability Summary” (or “Score Card”)

**Samples.** Five sera were distributed in RR49.

Serum #269 is a lyophilized material that has been distributed in three previous studies, as #193 in RR30 (3/94), #254 in RR45 (3/99), and #255 in RR46 (6/99). It was prepared from a native serum pool augmented with “retinol” that we now believe was highly enriched in *cis*-isomers. Up to 30% of the total retinol in this sample may be in the form of *cis*-retinols.

Serum #270 is a lyophilized material prepared from a native serum pool. The liquid-frozen partner of this sample was distributed as #267 in RR48. Sera #267 and #270 were prepared and aliquoted into vials as a single batch.

Serum #271 is a liquid-frozen material prepared from a native serum pool. The lyophilized partner of this sample was distributed as #266 in RR48. Sera #266 and #271 were prepared and aliquoted into vials as a single batch.

Serum #272 is a lyophilized material that has been distributed in two previous studies, as #223 and #225 in RR38 (9/96) and #228 in RR39 (3/97). It was prepared from a native serum pool augmented with retinyl palmitate.

Serum #273 is a lyophilized material that was distributed as #203 in RR33 (3/95). It was prepared from a native carotenoid-deficient serum augmented with retinol, retinyl palmitate, ( $\alpha$ ,  $\gamma$ ,  $\delta$ )-tocopherols, ( $\alpha$ ,  $\beta$ )-carotenes, and lycopene.

### Qualitative Observations.

- 1) Several participants noted the presence an “insoluble stringy clot” in Serum #271. As noted in the RR48 report for #266, we failed to re-filter the final blend of the serum pool used for this sample. As with its lyophilized partner, the resulting solids do **not** appear to have affected measurements.
- 2) One participant noted that what we’ve been reporting as  $\gamma$ -tocopherol is “really”  $\gamma$ - and/or  $\beta$ -tocopherol. We now denote this measurand as “ $\gamma/\beta$ -tocopherol.
- 3) All participants received their samples still frozen. We thank all of you who promptly confirmed receipt of the samples. Such prompt confirmation greatly simplifies our package delivery tracking and thus helps contain M<sup>2</sup>QAP costs. *We will be requesting similar confirmation in all future studies.*

## Quantitative Results

- 1) With the possible exception of zeaxanthin, there have been no changes in the median level of any measurand in sera #269 (7 years storage), #272 (5 years), or #273 (6 years). There is, however, evidence of increased variability in retinol and  $\alpha$ -tocopherol measurements, particularly in sera #272 and #273. We believe that this increased variability may be related to contamination of many of the lyophilized sera prepared in the past 6 years. We will discuss this more fully in the RR50 report.

As discussed in the RR45 report for serum #254, perhaps 30% of the retinol in Serum #269 is in the form of *cis*-isomers. While we did not intentionally spike this serum with *cis*-retinol, our methods indicate an unusually large retinol shoulder peak that is compatible with the presence of one or more of the *cis*-forms.

- 2) The distribution of reported “retinol-related measurand” interlaboratory values is bimodal, with approximately equal numbers centered at about 0.9  $\mu\text{g}/\text{mL}$  and 1.1  $\mu\text{g}/\text{mL}$ . Somewhat distressingly, many of your are **not** correctly specifying the form you are actually measuring. Many of the reported “Total Retinol” values are more compatible with being “*trans*-Retinol”; many of reported “*trans*-Retinol” values are more compatible with being “Total”.

If you report “Total Retinol” and your #269 value isn’t closer to 0.9  $\mu\text{g}/\text{mL}$  than to 1.1  $\mu\text{g}/\text{mL}$ , we suggest that you check your integration protocol to ensure that you are including *cis*-retinol isomers. If you report “*trans*-Retinol” and your #269 value isn’t closer to 1.1  $\mu\text{g}/\text{mL}$  than to 0.9  $\mu\text{g}/\text{mL}$ , we suggest that you confirm that your separation system does adequately resolve *cis*- and *trans*-retinol isomers. We intend to address this issue in the near future with explicitly *cis*-augmented samples.

- 3) A total of five matched {liquid-frozen, lyophilized} matrix pairs have now been distributed: {261,259} and {262,260} in RR47, {265,268} in RR48, and {266,271} and {270,267} in RR48 and RR49. Since these pairs were prepared at the same time from the same serum pools, the ratio of the values in the two matrices (liquid-frozen / lyophilized) is a direct assessment of measurement commutability between the two matrices. The last page of your “Individualized Report” summarizes the community’s and your individual commutability ratios.

We have not yet completed the detailed analysis of these data; however, a number of interesting results are already apparent:

The average (liquid-frozen / lyophilized) ratio is 0.94, essentially identical to the 0.95 expected from serum density consideration. There is no systematic variation in this ratio across measurands. While there is considerable variability about this average with most of the “minor” carotenoids, much of this variability in the ratios is a consequence of limited reporting precision at low measurand levels (e.g.,  $0.0034/0.0036 = 0.94$  but  $0.003/0.004 = 0.75$ ).

There are several participants with retinol and/or tocopherol commutability ratios that are consistently above or below 0.94, indicating a systematic difference between the matrices in their measurement systems. We believe that this arises from contamination of the lyophilized samples used rather than a lyophilization-specific matrix effect.

Note: since the {266,271} and {270,267} ratios involve measurements made half a year apart, the average of these two ratios (in logarithmic form) estimates commutability while the difference between the ratios estimates within-laboratory calibration differences between RR48 and RR49.

Lab number: \_\_\_\_\_

Phone: \_\_\_\_\_

Fax: \_\_\_\_\_

E-mail: \_\_\_\_\_

## Questionnaire

### NIST Micronutrients Measurement Quality Assurance Program

Please provide us with your input by completing this form and returning it by **May 31, 2001** to:

NIST Micronutrients Measurement Quality Assurance Program

NIST

100 Bureau Drive, Stop 8392

Gaithersburg, MD 20899-8392

**Fax: 301-977-0685**

\_\_\_\_\_ Folates in serum/plasma are currently measured in my organization by:

\_\_\_\_\_  
*Name of the contact*

\_\_\_\_\_  
*Phone*

\_\_\_\_\_  
*Fax*

\_\_\_\_\_  
*E-mail address*

\_\_\_\_\_ I would participate and be willing to pay for a second annual QA interlaboratory comparison study for vitamin C if it were available.

\_\_\_\_\_ I am required to comply with the Clinical Laboratory Improvement Amendments and/or another laboratory standards program for the measurement of:

vitamin A

carotenes

vitamin C

vitamin E

vitamin D

vitamin K<sub>1</sub>

coenzyme Q<sub>10</sub>

other (*please specify*) \_\_\_\_\_

Comments:

## **Appendix C. “All-Lab Report” for RR49**

The following eight 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 XLIX Laboratory Results

All Values in µg/mL

Lab	trans-Lutein					Total Zeaxanthin					trans-Zeaxanthin				
	269	270	271	272	273	269	270	271	272	273	269	270	271	272	273
FSV-BA															
FSV-BB						0.051	0.048	0.041	0.054	0.035					
FSV-BD															
FSV-BE															
FSV-BF															
FSV-BG						0.019	0.012	0.020	0.018	0.020					
FSV-BH						0.020	0.033	0.023	0.025	0.035					
FSV-BI						0.026	0.042	0.033	0.038	0.039					
FSV-BJ															
FSV-BK															
FSV-BL															
FSV-BM															
FSV-BN						0.017	0.027	0.022	0.023	0.032					
FSV-BO						0.020	0.024	0.018	0.019	0.027					
FSV-BP															
FSV-BQ															
FSV-BR															
FSV-BS															
FSV-BT						>0.028	>0.033	>0.038	>0.044	>0.041	0.028	0.033	0.038	0.044	0.041
FSV-BU															
FSV-BV															
FSV-BW															
FSV-BX						0.022	0.034	0.024	0.026	0.034					
FSV-CB						0.020	0.035	0.033	0.007	0.036					
FSV-CC															
FSV-CD															
FSV-CE															
FSV-CF															
FSV-CG						0.018	0.025	0.024	0.034	0.022					
FSV-CH															
FSV-CI															
FSV-CL															
FSV-CR															
FSV-CV															
FSV-CW	0.048	0.073	0.073	0.093	0.056	0.023	0.034	0.025	0.026	0.033					
FSV-CZ															
FSV-DB															
FSV-DD															
FSV-DF															
FSV-DI															
FSV-DQ						0.019	0.064	0.017	0.008	0.025					
FSV-DR															
FSV-DU															
FSV-EH	0.051	0.080	0.077	0.092	0.061	0.023	0.036	0.028	0.029	0.033					
FSV-EQ															
FSV-FB						0.020	0.037	0.016	0.012	0.030					
FSV-FJ															
N	2	2	2	2	2	13	13	13	13	13	1	1	1	1	1
Min	0.048	0.073	0.073	0.092	0.056	0.017	0.012	0.016	0.007	0.020					
Median	0.050	0.077	0.075	0.093	0.059	0.020	0.034	0.024	0.025	0.033	0.028	0.033	0.038	0.044	0.041
Max	0.051	0.080	0.077	0.093	0.061	0.051	0.064	0.041	0.054	0.039					
eSD						0.002	0.010	0.007	0.010	0.004					
eCV						12	31	28	42	13					
Npast	0	0	0	0	0	12	15	15	11	8	0	0	0	0	0
Medianpast						0.027	0.035	0.027	0.032	0.035					
SDpast						0.010	0.009	0.005	0.012	0.008					
NISTa						nq	nq	0.020	0.015	0.024					
NISTb	0.054	0.067	0.068	0.091	0.050	>0.029	>0.036	>0.026	>0.034	>0.033	0.029	0.036	0.026	0.034	0.033
NNIST	1	2	2	2	1			2	2	2	1	2	2	2	1
Mean	0.054	0.067	0.068	0.091	0.050			0.020	0.015	0.024	0.029	0.036	0.026	0.034	0.033
Srep	0.009	0.004	0.005	0.004	0.001			0.001	0.001	0.001	0.000	0.002	0.002	0.004	0.003
Shet	0.000	0.005	0.003	0.005	0.000			0.000	0.003	0.000	0.000	0.004	0.003	0.005	0.000
Sanl															
SNIST	0.009	0.006	0.006	0.007	0.001			0.001	0.003	0.001	0.000	0.004	0.004	0.007	0.003
NAV						0.020	0.034	0.022	0.020	0.028					
NAU						0.006	0.010	0.007	0.012	0.011					

# Round Robin XLIX Laboratory Results

All Values in µg/mL

Lab	Total Lutein&Zeaxanthin					Coenzyme Q10					Phylloquinone (K1) x1000				
	269	270	271	272	273	269	270	271	272	273	269	270	271	272	273
FSV-BA	0.106	0.156	0.158	0.182	0.132										
FSV-BB	0.113	0.149	0.138	0.172	0.099										
FSV-BD															
FSV-BE											0.38	0.91	0.38	0.42	0.16
FSV-BF	0.107	0.157	0.153	0.203	0.138										
FSV-BG	0.089	0.073	0.102	0.101	0.087										
FSV-BH	0.060	0.101	0.086	0.110	0.089										
FSV-BI	0.096	0.141	0.145	0.187	0.130										
FSV-BJ															
FSV-BK															
FSV-BL															
FSV-BM															
FSV-BN	0.060	0.104	0.098	0.118	0.087										
FSV-BO	0.079	0.134	0.126	0.224	0.107										
FSV-BP	0.080	0.120	0.110	0.130	0.006										
FSV-BQ															
FSV-BR															
FSV-BS															
FSV-BT	0.192	0.191	0.217	0.263	0.192										
FSV-BU	0.091	0.153	0.144	0.203	0.130										
FSV-BV	0.071	0.113	0.109	0.136	0.099										
FSV-BW															
FSV-BX	0.079	0.130	0.115	0.144	0.103										
FSV-CB	0.069	0.109	0.117	0.140	0.099										
FSV-CC															
FSV-CD	0.080	0.116	0.143	0.130	0.089										
FSV-CE															
FSV-CF															
FSV-CG	0.100	0.143	0.139	0.171	0.120										
FSV-CH						0.487	0.45	0.52	0.335	0.48					
FSV-CI															
FSV-CL	0.084	0.114	0.100	0.125	0.100										
FSV-CR															
FSV-CV	0.092	0.146	0.125	0.172	0.110	1.948	0.96	0.99	0.942	1.78					
FSV-CW	>0.071	>0.107	>0.098	>0.119	>0.089	0.910	0.83	0.95	0.570	1.07					
FSV-CZ						0.790	0.94	1.10	0.540	1.00					
FSV-DB	0.098	0.129	0.115	0.152	0.120										
FSV-DD															
FSV-DF															
FSV-DI						0.855	0.72	0.79	0.493	0.85	0.68	1.37	0.53	0.47	nd
FSV-DQ	0.066	0.271	0.106	0.080	0.058										
FSV-DR															
FSV-DU															
FSV-EH	>0.074	>0.116	>0.105	>0.121	>0.094										
FSV-EQ	0.017	0.196	0.183	0.245	0.178										
FSV-FB	0.072	0.100	0.081	0.112	0.074										
FSV-FJ															
N	22	22	22	22	22	5	5	5	5	5	2	2	2	2	1
Min	0.017	0.073	0.081	0.080	0.006	0.487	0.45	0.52	0.335	0.48	0.38	0.91	0.38	0.42	
Median	0.082	0.132	0.121	0.148	0.101	0.855	0.83	0.95	0.540	1.00	0.53	1.14	0.46	0.45	0.16
Max	0.192	0.271	0.217	0.263	0.192	1.948	0.96	1.10	0.942	1.78	0.68	1.37	0.53	0.47	
eSD	0.020	0.030	0.030	0.047	0.024	0.096	0.16	0.22	0.070	0.22					
eCV	25	22	25	32	24	11	20	23	13	22					
Npast	22	29	29	22	21	0	0	0	0	0	0	0	0	0	0
Medianpast	0.089	0.134	0.114	0.154	0.117										
SDpast	0.016	0.027	0.017	0.034	0.018										
NISTa	>0.055	>0.101	0.130	0.151	0.098										
NISTb	>0.083	>0.103	>0.093	>0.125	>0.082										
NNIST	1	2	4	4	3										
Mean	0.077	0.103	0.112	0.138	0.090										
Srep	0.000	0.005	0.007	0.008	0.004										
Shet	0.000	0.009	0.005	0.000	0.000										
Sanl			0.026	0.019	0.011										
SNIST	0.000	0.010	0.028	0.020	0.012										
NAV	0.080	0.118	0.117	0.143	0.096										
NAU	0.021	0.036	0.030	0.048	0.026										

# Round Robin XLIX Laboratory Results

## Analytes Reported By One Laboratory

Values in µg/mL

Analyte	Code	269	270	271	272	273
25-hydroxyvitamin D	FSV-BN	0.01	0.06	0.02	0.02	0.01
3'-dehydro-Lutein	FSV-BH	0.04	0.03	0.04	0.06	0.03
Phytofluene	FSV-CL	0.059	0.014	0.042	0.078	0.024
Total cis-Lutein&Zeaxanthin	FSV-BT	0.092	0.052	0.064	0.100	0.056
trans-Lutein&Zeaxanthin	FSV-BT	0.100	0.138	0.153	0.163	0.136
trans-β-Cryptoxanthin	NISTb	0.040	0.056	0.059	0.053	0.045

## Legend

Term	Definition
N	Number of (non-NIST) quantitative values reported for this analyte
Min	Minimum (non-NIST) quantitative value reported
Median	Median (non-NIST) quantitative value reported
Max	Maximum (non-NIST) quantitative value reported
SD	Adjusted median absolute deviation from the median of the non-NIST results
CV	Coefficient of Variation for (non-NIST) results: 100*SD/Median
$N_{past}$	Mean of N(s) from past RR(s)
$Median_{past}$	Mean of Median(s) from past RR(s)
$SD_{past}$	Pooled SD from past RR(s)
NIST	Mean of NIST results
NAV	NIST Assigned Value = (Median + NIST)/2 for analytes reported by NIST = Median for analytes reported by ≥ 5 labs but not NIST
NAU	NIST Assigned Uncertainty: $\sqrt{(S^2 + S_{btw}^2)}$ S is the maximum of (0.05*NAV, SD, $SD_{past}$ , eSD) and $S_{btw}$ is the standard deviation between Median and NIST. The expected long-term SD, eSD, is defined in: Duewer et al., Anal Chem 1997;69(7):1406-1413.
$N_{NIST}$	Number of total vials analyzed in duplicate by NIST analysts
$Mean_{NIST}$	Mean of the NIST-analyzed vial means
$S_{rep}$	Within-vial pooled standard deviation
$S_{het}$	Among-vial pooled standard deviation
$S_{anl}$	Between NIST analyst standard deviation
$S_{NIST}$	Total standard deviation for NIST analyses: $(S_{rep}^2 + S_{het}^2 + S_{anl}^2)^{0.5}$
<i>nd</i>	Not detected (i.e., no detectable peak for analyte)
<i>nq</i>	Detected but not quantitatively determined
<x	Concentration less than the limit of quantification, x
>x	Concentration greater than x
<i>italics</i>	Not explicitly reported but calculated by NIST from reported values

# Round Robin XLIX Laboratory Results

## Comparability Summary

Lab	TR	aT	g/bT	bC	tbC	aC	TLy	TbX	TLu	TZ	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	1	2	2
FSV-BD	1	1		1							
FSV-BE	1	1	1	1						2	
FSV-BF	1	1	1	2		1	1	1			
FSV-BG	1	1	1	1		2	1	1	1	2	
FSV-BH	1	1	1	1	1	1	1	2	1	3	1
FSV-BI	1	1	1	1		1	1	1	1	1	1
FSV-BJ	1	1	1	1		1	1	1	1	1	1
FSV-BK	1	2									
FSV-BL	3	4									
FSV-BM	1	2									
FSV-BN	1	1	2	1	1	2	1	1	1	1	
FSV-BO	1	1		1		1	2	1	2		
FSV-BP	1	1		1		1	3	1			1
FSV-BQ	1	1								2	1
FSV-BR	1	2									2
FSV-BS	1			3	3	2	1	1	2	1	1
FSV-BT	2	1	1	1	1	1	1	1	1		
FSV-BU	1	1	1	1		1	2	1		1	1
FSV-BV	2	1	1	1		1	1	2		2	4
FSV-BW	1	1	1	1		2	1				
FSV-BX	1	1	1	1	2	1		1	1		
FSV-CB	1	1		1		1	1	1	1	1	1
FSV-CC	1	1									
FSV-CD	1	1	1	1		1	1	1			
FSV-CE	1	1		1							1
FSV-CF	1	1									1
FSV-CG	1	2	1	1	1	1	1	1	2		1
FSV-CH	2	1	1	2		1	1				1
FSV-CI	2	1	2							1	1
FSV-CL	2	2	2	1		1	1	1			
FSV-CR	1	1									
FSV-CV	4	2	4	2		4	4				
FSV-CW	2	1	1	1	1	1	1	1	1		
FSV-CZ	1	4		2							
FSV-DB	1	1		1			2	1		2	3
FSV-DD	1										
FSV-DF	1										
FSV-DI	1	1	2	1	1				1		
FSV-DQ		4	4	4		2	4	2	3		3
FSV-DR	1	2		1							
FSV-DU	1	2		2	2						
FSV-EH	1	1	1	1	1	1	1	1	1	1	1
FSV-EQ	1	3		1	1	1	1	1			
FSV-FB	3	1		1	2	2	1	1	1	1	1
FSV-FJ	1	1	1	2		1					
NISTa	1	1	1	1	1	1	1	1	1	1	1
NISTb	1	1	1	1	1	1	1	1	1	1	1
n	48	46	27	38	16	30	28	27	19	16	26

Label	Definition
Lab	Participant code
TR	Total Retinol
aT	α-Tocopherol
g/bT	γ/β-Tocopherol
bC	Total β-Carotene
tbC	trans-β-Carotene
aC	Total α-Carotene
TLy	Total Lycopene
TbX	Total β-Cryptoxanthin
TLu	Total Lutein
TZ	Total Zeaxanthin
L&Z	Total Lutein & Zeaxanthin
n	number of participants providing quantitative data
% 1	Percent of CS = 1 (within 1 SD of medians)
% 2	Percent of CS = 2 (within 2 SD of medians)
% 3	Percent of CS = 3 (within 3 SD of medians)
% 4	Percent of CS = 4 (3 or more SD from medians)

### “Comparability Score”

The Comparability Score (CS) summarizes your measurement performance for a given analyte relative to the consensus medians in this study. CS is the average distance (in units of standard deviation) of your measurement performance characteristics from the consensus performance. CS is calculated when the number of quantitative values you reported,  $N_{you}$ , is at least two and at least six participants reported quantitative values for the analyte.

We define CS as follows:

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

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

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

NAU = NIST Assigned Uncertainty

For further details, please see

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

	TR	aT	g/bT	bC	tbC	aC	TLy	TbX	TLu	TZ	L&Z
% 1	81	74	78	79	75	77	79	89	79	69	73
% 2	13	17	15	16	19	20	11	11	16	25	15
% 3	4	2	0	3	6	0	4	0	5	6	8
% 4	2	7	7	3	0	3	7	0	0	0	4

## Appendix D. Representative “Individualized Report” for RR49

Each participant in RR49 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 RR49:

- Total Retinol
- *trans*-Retinol
- Retinyl Palmitate
- $\alpha$ -Tocopherol
- $\gamma/\beta$ -Tocopherol
- $\delta$ -Tocopherol
- Total  $\beta$ -Carotene
- *trans*- $\beta$ -Carotene
- Total *cis*- $\beta$ -Carotene
- Total  $\alpha$ -Carotene
- Total Lycopene
- *trans*-Lycopene
- Total  $\beta$ -Cryptoxanthin
- Total  $\alpha$ -Cryptoxanthin
- Total Lutein
- Total Zeaxanthin
- Total Lutein & Zeaxanthin
- Coenzyme Q10

The following 13 pages are the “Individualized Report” for the analytes evaluated by participant FSV-BA.

# Individualized Round Robin XLIX Report: FSV-BA

## Summary

Analyte	Serum 269			Serum 270			Serum 271			Serum 272			Serum 273		
	You	NAV	n	You	NAV	n	You	NAV	n	You	NAV	n	You	NAV	n
Total Retinol	1.252	0.979	36	0.688	0.630	36	0.495	0.449	36	0.564	0.491	36	1.350	1.204	36
Retinyl Palmitate	0.07	0.03	13	nd	0.0	12	0.1	0.1	14	0.12	0.07	14	0.07	0.06	14
α-Tocopherol	28.85	26.88	44	6.15	5.71	44	7.21	6.72	44	8.11	7.52	44	17.58	16.33	44
γ/β-Tocopherol	6.423	6.124	25	2.292	2.179	25	1.961	1.890	25	1.755	1.705	25	1.215	1.165	25
Total β-Carotene	0.347	0.358	32	0.480	0.452	32	0.331	0.314	32	0.165	0.154	32	0.051	0.052	31
trans-β-Carotene	0.316	0.305	14	0.460	0.420	14	0.323	0.288	14	0.155	0.150	14	0.051	0.048	14
Total cis-β-Carotene	0.033	0.035	10	0.023	0.025	10	0.010	0.022	10	0.011	0.011	10	nd	0.010	6
Total α-Carotene	0.014	0.013	24	0.020	0.015	23	0.029	0.030	24	0.027	0.030	24	0.028	0.028	23
Total Lycopene	0.624	0.465	27	0.274	0.206	27	0.425	0.329	27	0.598	0.484	27	0.227	0.194	26
trans-Lycopene	0.293	0.259	12	0.151	0.134	12	0.220	0.197	12	0.267	0.232	12	0.121	0.105	12
Total β-Cryptoxanthin	0.050	0.041	25	0.064	0.049	25	0.071	0.062	25	0.067	0.060	25	0.069	0.046	25
Total Lutein&Zeaxanthin	0.106	0.082	22	0.156	0.118	22	0.158	0.117	22	0.182	0.143	22	0.132	0.096	22

You : Your reported values for the listed analytes (micrograms/milliliter)

NAV : NIST Assigned Values, equal to (NIST's average-of-averages + this RR's median) / 2

n : Number of non-NIST laboratories reporting quantitative values for this analyte in this serum

nd : Not detected

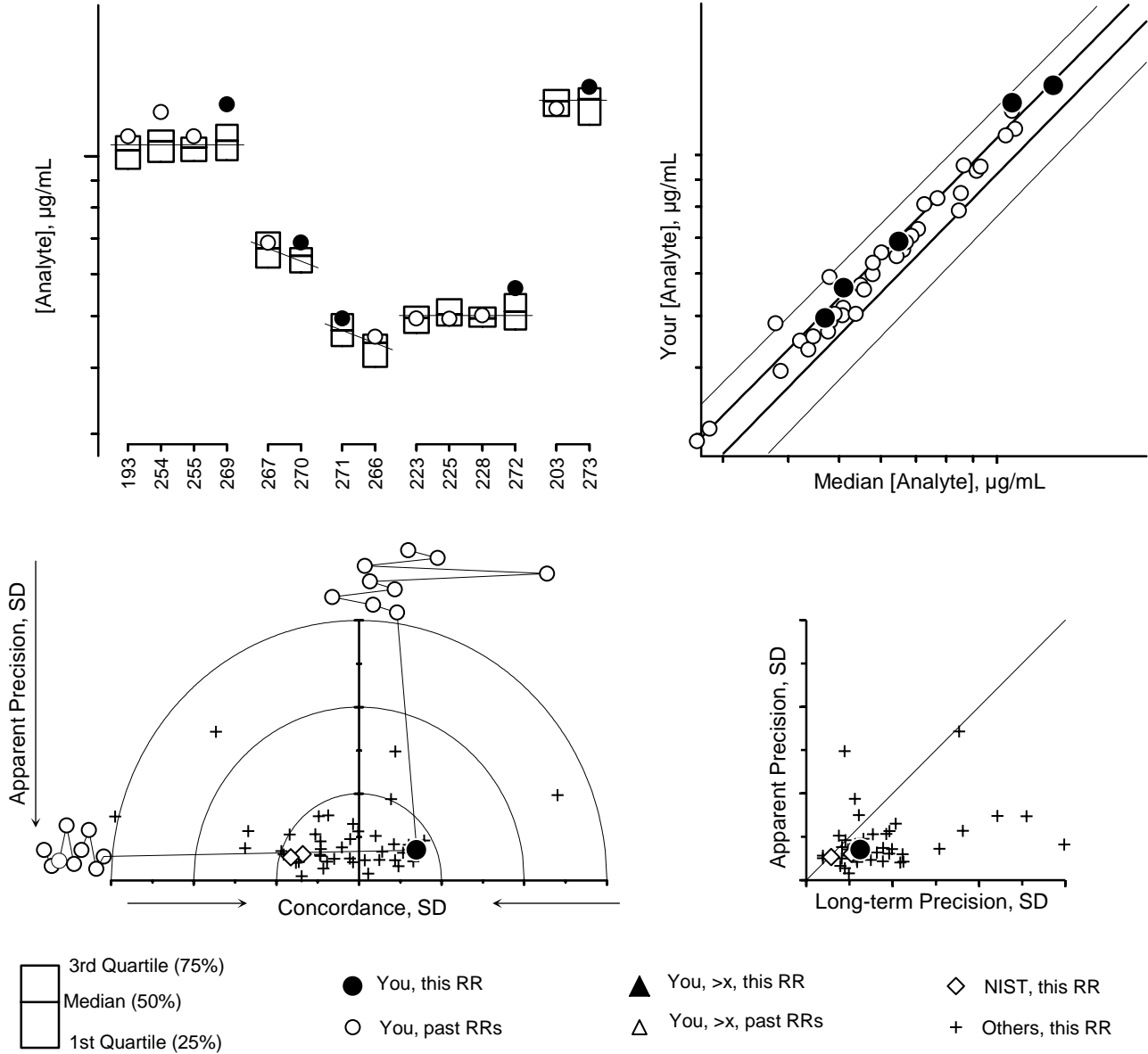
Please check our records against your records. Send corrections and/or updates to...

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

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

# Individualized RR XLIX Report: FSV-BA

## Total Retinol



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

### Serum

#269 #193 RR30 3/94, #254 RR45 3/99, #255 RR46 6/99  
 #270 Same pool as #267 (lq fz) RR48 9/00  
 #271 Same pool as #266 (ly) RR48 9/00  
 #272 #223 & #225 RR38 9/96, #228 RR39 3/97  
 #273 #203 RR33 3/95

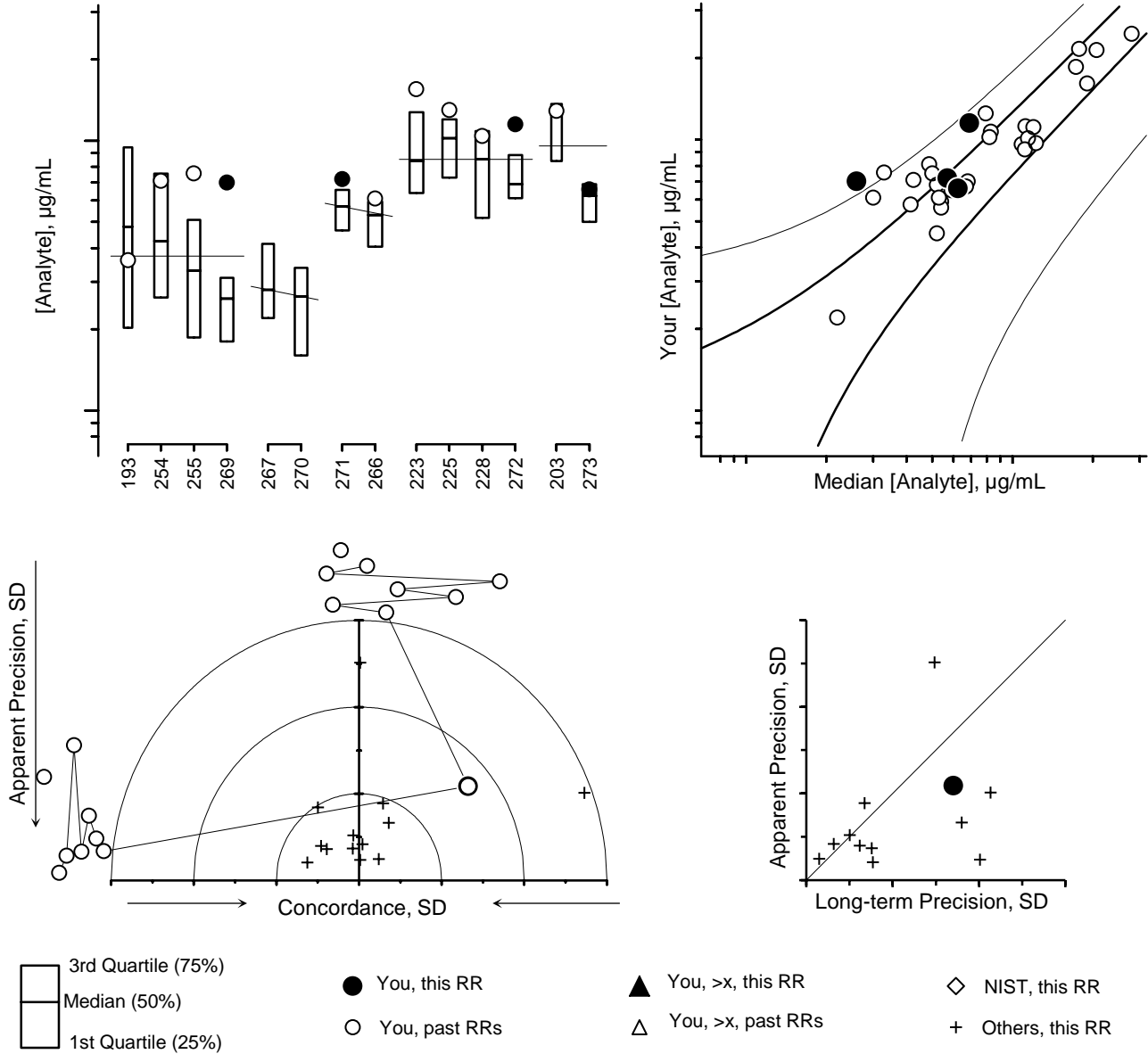
### History

### Comments

Lyophilized  
 Lyophilized  
 Fresh frozen  
 Lyophilized  
 Lyophilized

# Individualized RR XLIX Report: FSV-BA

## Retinyl Palmitate



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

Serum

History

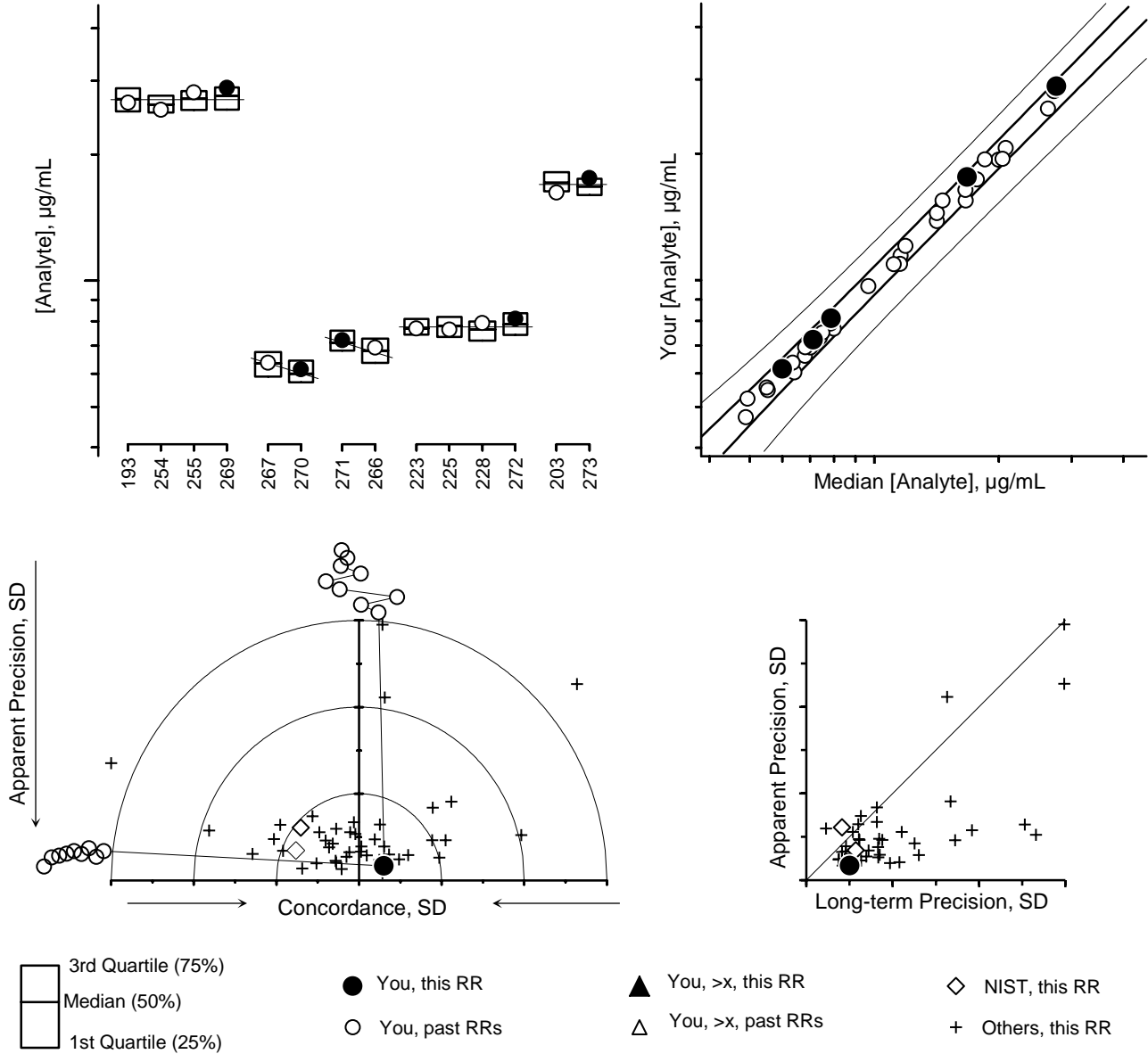
Comments

#269	#193 RR30 3/94, #254 RR45 3/99, #255 RR46 6/99	Lyophilized
#270	Same pool as #267 (lq fz) RR48 9/00	Lyophilized
#271	Same pool as #266 (ly) RR48 9/00	Fresh frozen
#272	#223 & #225 RR38 9/96, #228 RR39 3/97	Lyophilized
#273	#203 RR33 3/95	Lyophilized



# Individualized RR XLIX Report: FSV-BA

## α-Tocopherol



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

Serum

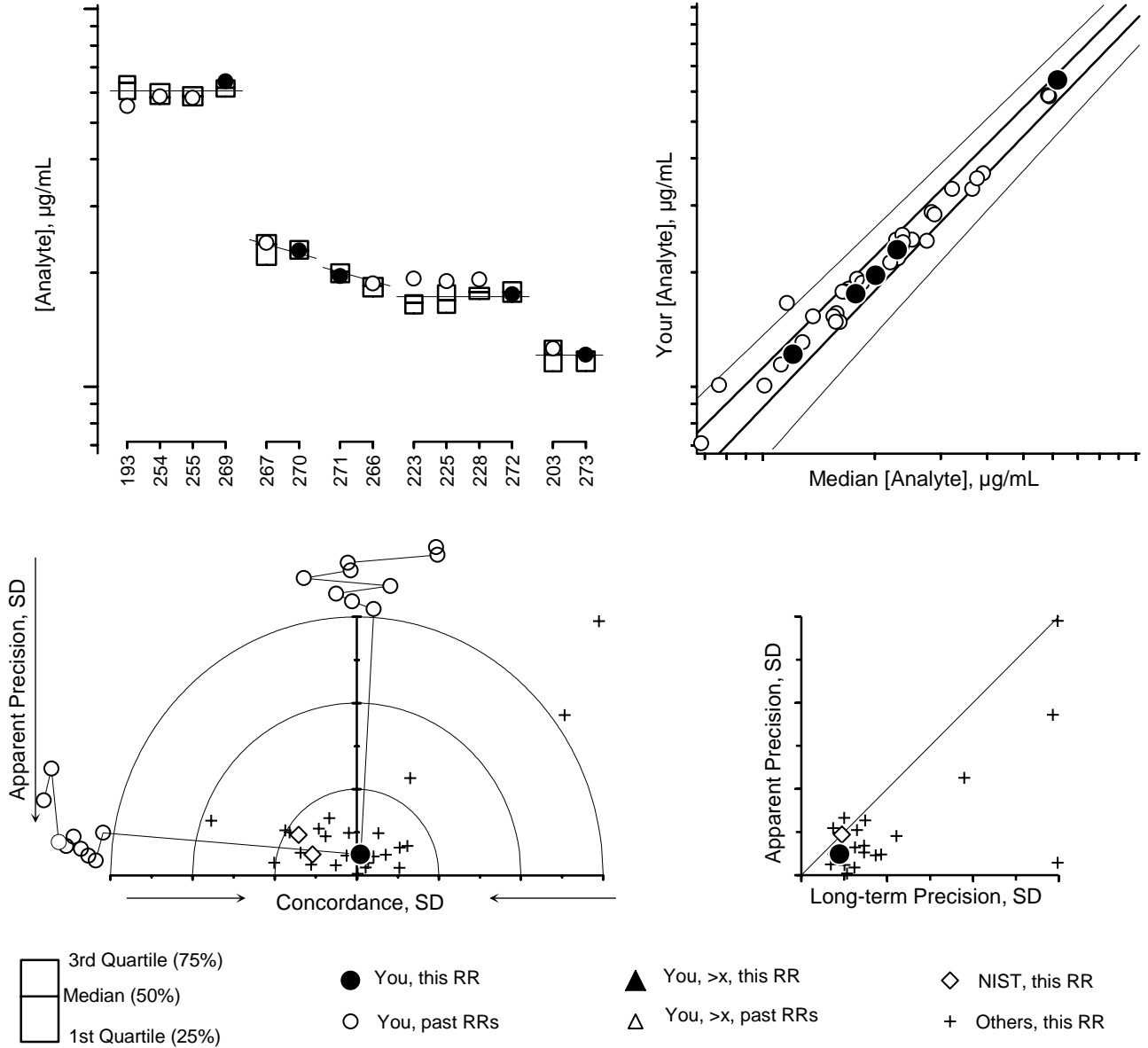
History

Comments

#269	#193 RR30 3/94, #254 RR45 3/99, #255 RR46 6/99	Lyophilized
#270	Same pool as #267 (lq fz) RR48 9/00	Lyophilized
#271	Same pool as #266 (ly) RR48 9/00	Fresh frozen
#272	#223 & #225 RR38 9/96, #228 RR39 3/97	Lyophilized
#273	#203 RR33 3/95	Lyophilized

# Individualized RR XLIX Report: FSV-BA

$\gamma/\beta$ -Tocopherol



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

Serum

History

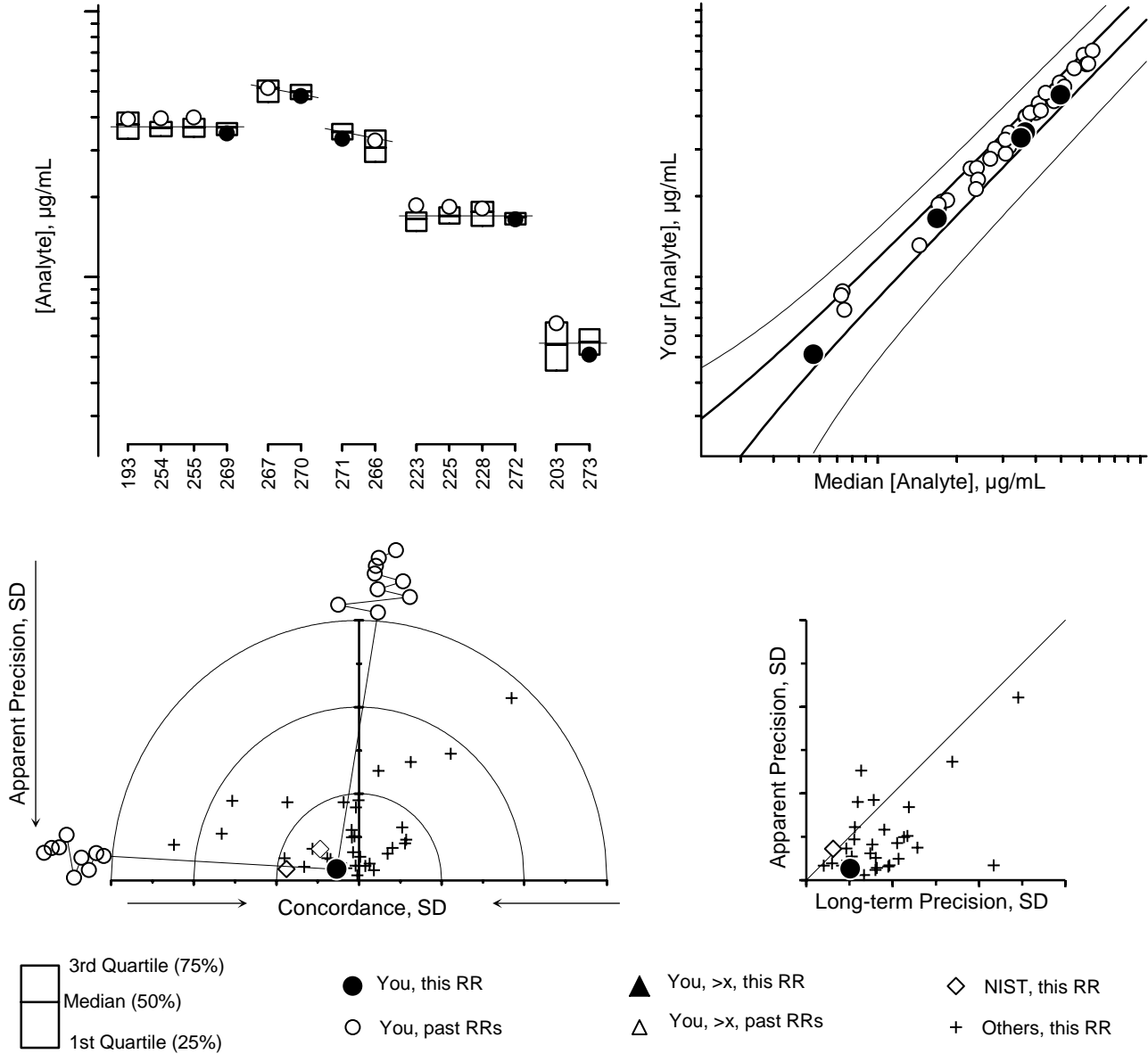
Comments

#269	#193 RR30 3/94, #254 RR45 3/99, #255 RR46 6/99
#270	Same pool as #267 (lq fz) RR48 9/00
#271	Same pool as #266 (ly) RR48 9/00
#272	#223 & #225 RR38 9/96, #228 RR39 3/97
#273	#203 RR33 3/95

Lyophilized
Lyophilized
Fresh frozen
Lyophilized
Lyophilized

# Individualized RR XLIX Report: FSV-BA

## Total $\beta$ -Carotene



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

Serum

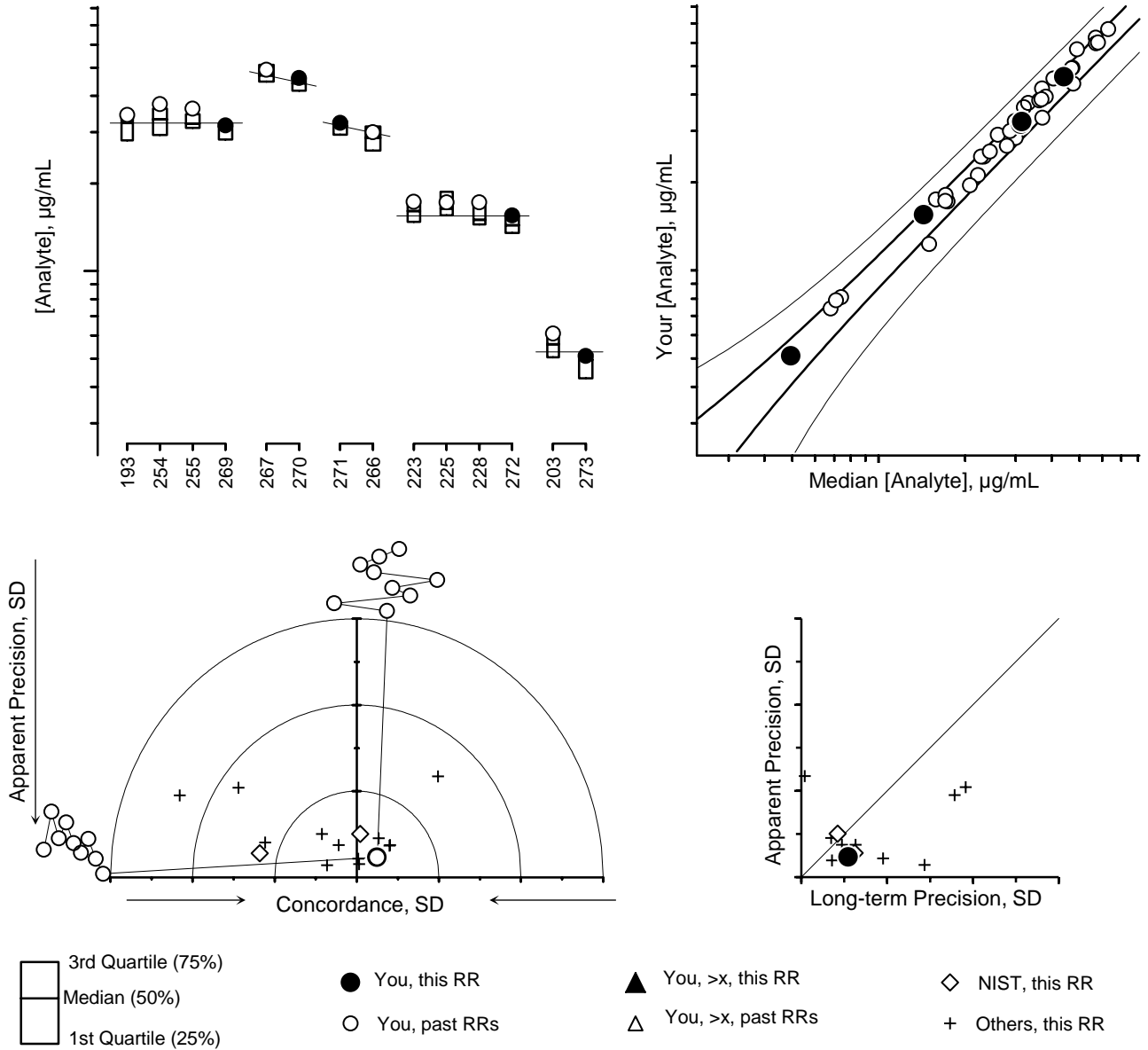
History

Comments

#269	#193 RR30 3/94, #254 RR45 3/99, #255 RR46 6/99	Lyophilized
#270	Same pool as #267 (lq fz) RR48 9/00	Lyophilized
#271	Same pool as #266 (ly) RR48 9/00	Fresh frozen
#272	#223 & #225 RR38 9/96, #228 RR39 3/97	Lyophilized
#273	#203 RR33 3/95	Lyophilized

# Individualized RR XLIX Report: FSV-BA

trans-β-Carotene



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

Serum

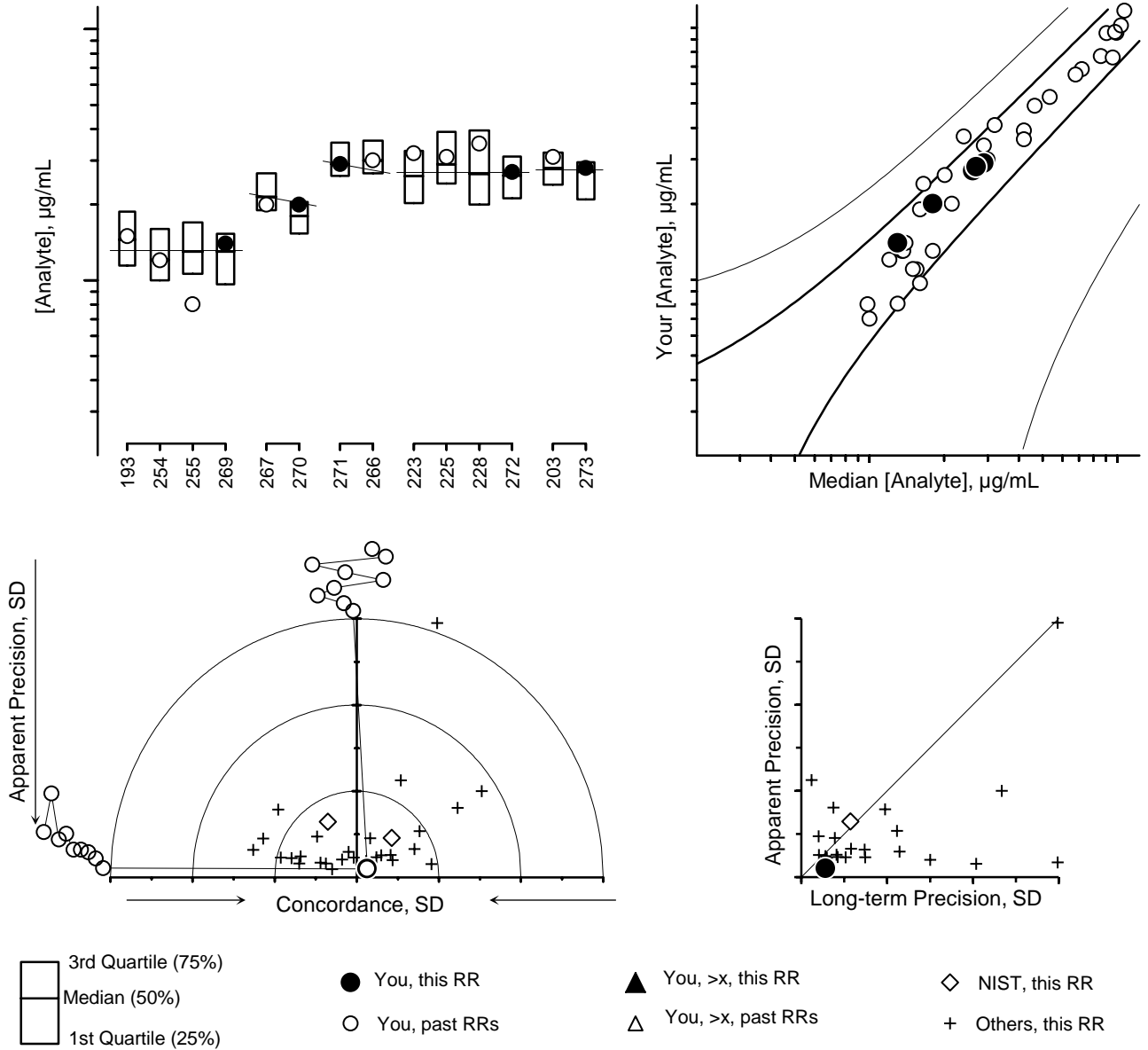
History

Comments

#269	#193 RR30 3/94, #254 RR45 3/99, #255 RR46 6/99	Lyophilized
#270	Same pool as #267 (lq fz) RR48 9/00	Lyophilized
#271	Same pool as #266 (ly) RR48 9/00	Fresh frozen
#272	#223 & #225 RR38 9/96, #228 RR39 3/97	Lyophilized
#273	#203 RR33 3/95	Lyophilized

# Individualized RR XLIX Report: FSV-BA

## Total $\alpha$ -Carotene



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

Serum

History

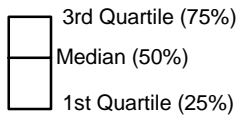
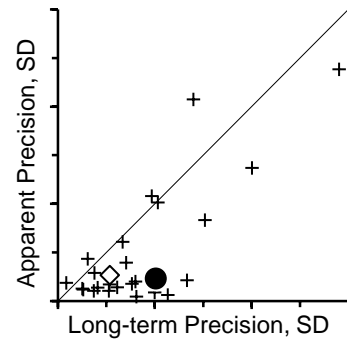
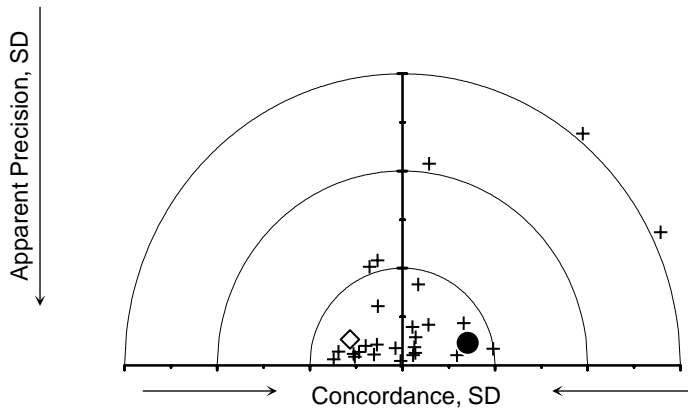
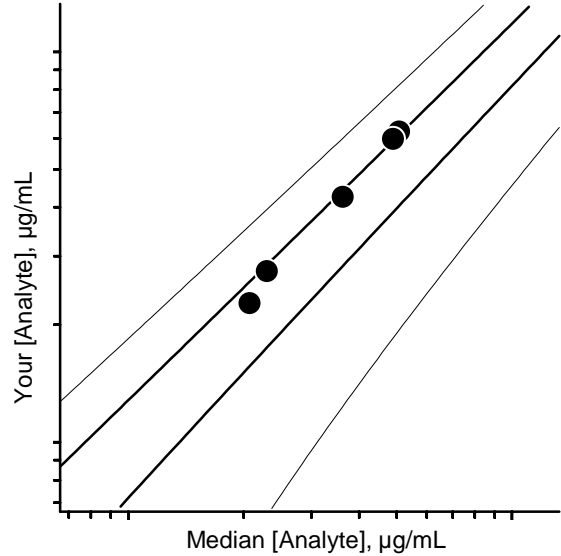
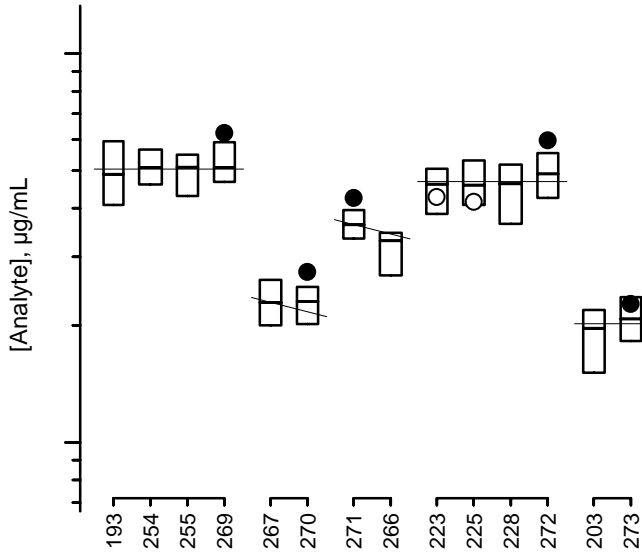
Comments

#269	#193 RR30 3/94, #254 RR45 3/99, #255 RR46 6/99
#270	Same pool as #267 (lq fz) RR48 9/00
#271	Same pool as #266 (ly) RR48 9/00
#272	#223 & #225 RR38 9/96, #228 RR39 3/97
#273	#203 RR33 3/95

Lyophilized
Lyophilized
Fresh frozen
Lyophilized
Lyophilized

# Individualized RR XLIX Report: FSV-BA

## Total Lycopene



● You, this RR  
○ You, past RRs

▲ You, >x, this RR  
△ You, >x, past RRs

◇ NIST, this RR  
+ Others, this RR

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

### Serum

#269 #193 RR30 3/94, #254 RR45 3/99, #255 RR46 6/99  
#270 Same pool as #267 (lq fz) RR48 9/00  
#271 Same pool as #266 (ly) RR48 9/00  
#272 #223 & #225 RR38 9/96, #228 RR39 3/97  
#273 #203 RR33 3/95

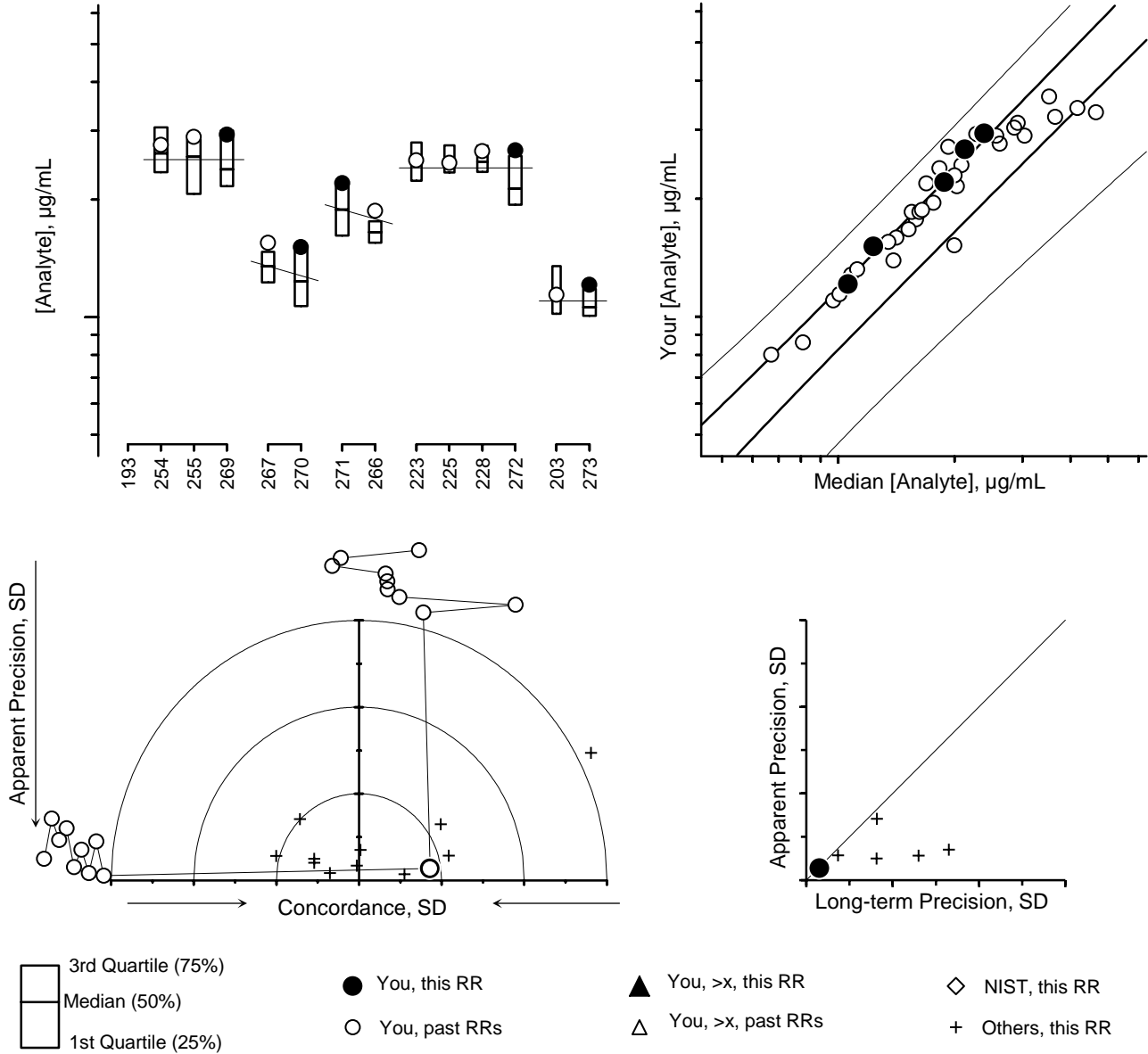
### History

### Comments

Lyophilized  
Lyophilized  
Fresh frozen  
Lyophilized  
Lyophilized

# Individualized RR XLIX Report: FSV-BA

trans-Lycopene



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

Serum

History

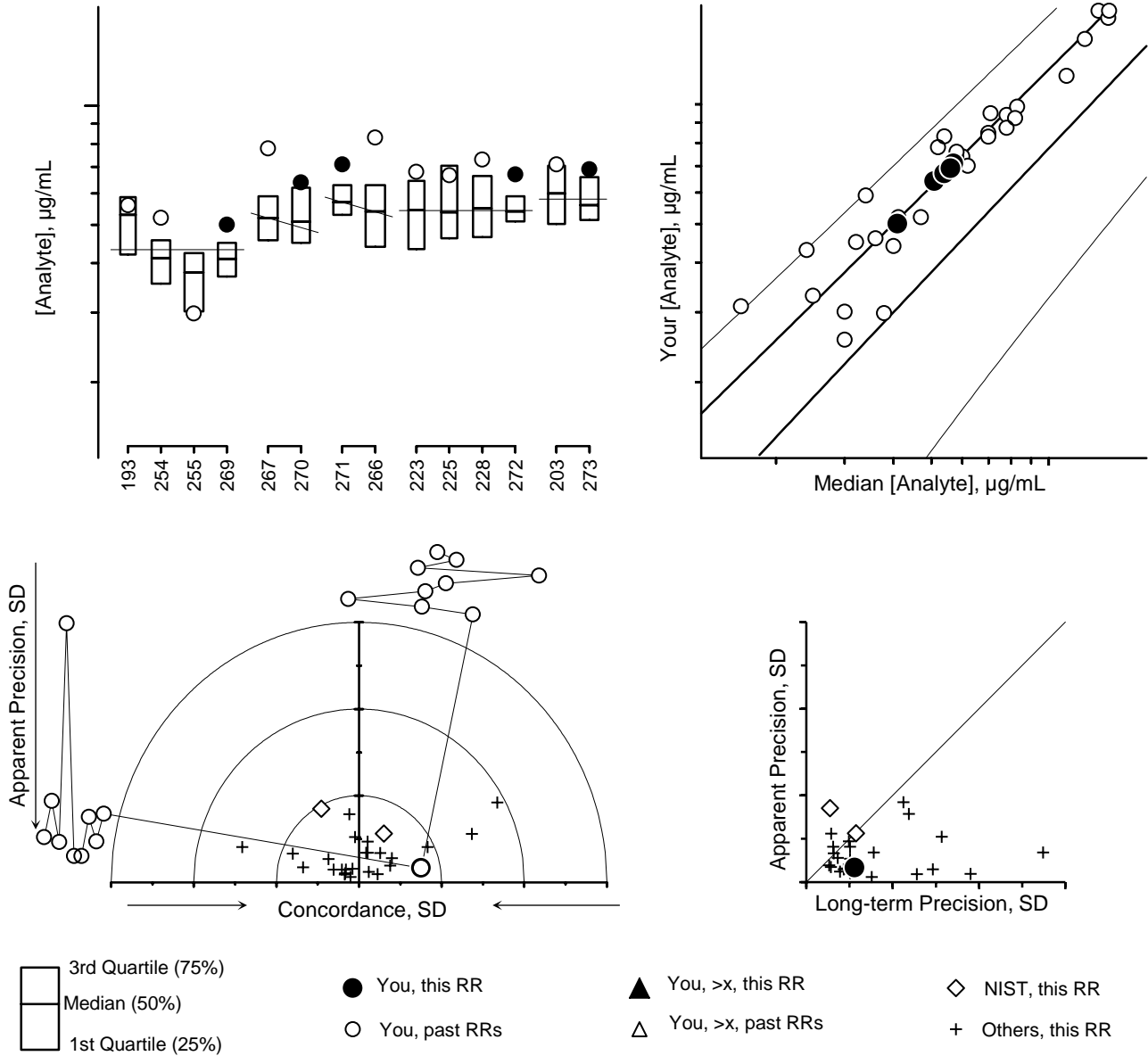
Comments

#269	#193 RR30 3/94, #254 RR45 3/99, #255 RR46 6/99
#270	Same pool as #267 (lq fz) RR48 9/00
#271	Same pool as #266 (ly) RR48 9/00
#272	#223 & #225 RR38 9/96, #228 RR39 3/97
#273	#203 RR33 3/95

Lyophilized
Lyophilized
Fresh frozen
Lyophilized
Lyophilized

# Individualized RR XLIX Report: FSV-BA

## Total $\beta$ -Cryptoxanthin



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

Serum

History

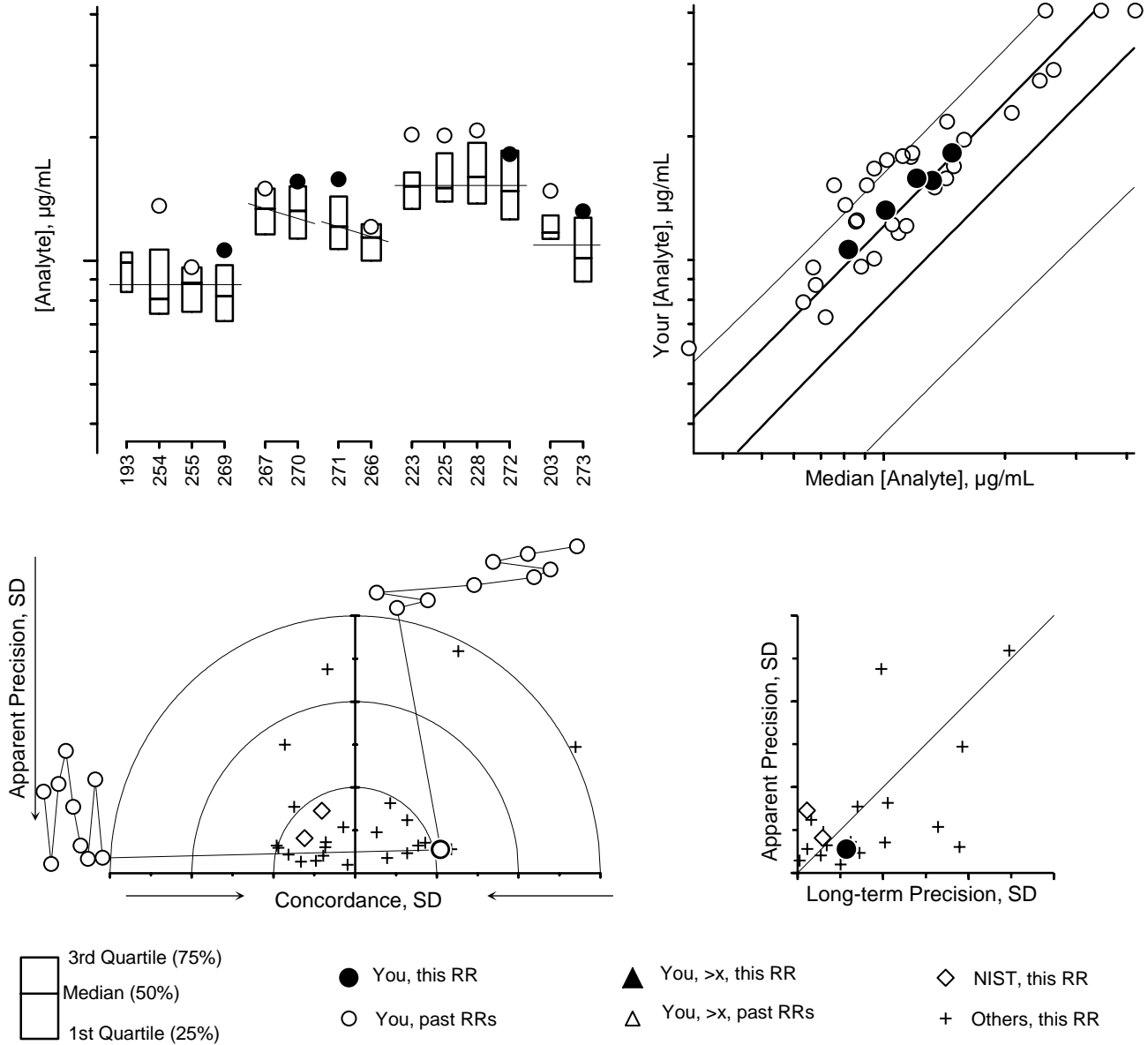
Comments

#269	#193 RR30 3/94, #254 RR45 3/99, #255 RR46 6/99	Lyophilized
#270	Same pool as #267 (lq fz) RR48 9/00	Lyophilized
#271	Same pool as #266 (ly) RR48 9/00	Fresh frozen
#272	#223 & #225 RR38 9/96, #228 RR39 3/97	Lyophilized
#273	#203 RR33 3/95	Lyophilized



# Individualized RR XLIX Report: FSV-BA

## Total Lutein&Zeaxanthin



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

Serum

History

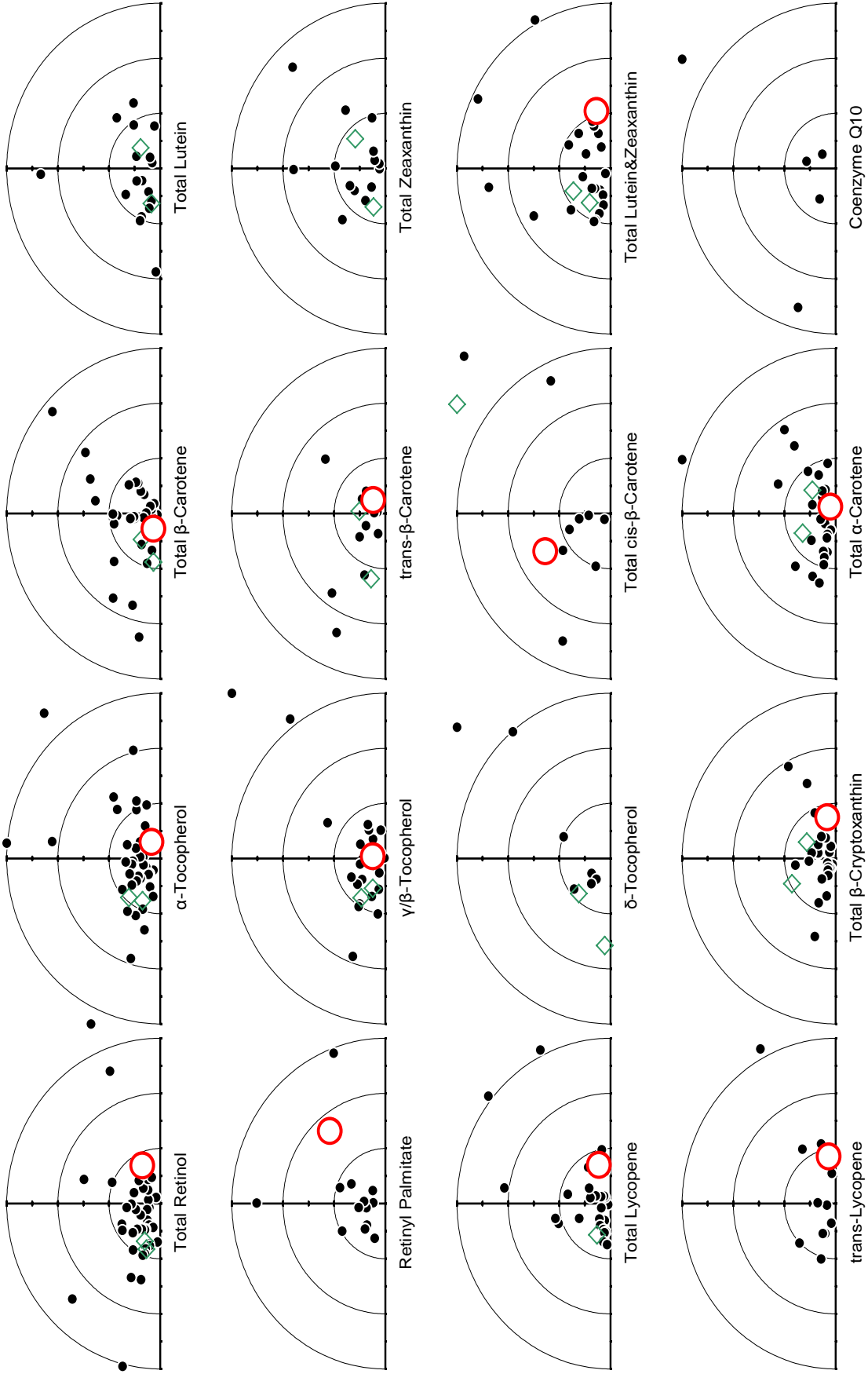
Comments

#269 #193 RR30 3/94, #254 RR45 3/99, #255 RR46 6/99  
 #270 Same pool as #267 (lq fz) RR48 9/00  
 #271 Same pool as #266 (ly) RR48 9/00  
 #272 #223 & #225 RR38 9/96, #228 RR39 3/97  
 #273 #203 RR33 3/95

Lyophilized  
 Lyophilized  
 Fresh frozen  
 Lyophilized  
 Lyophilized

# Individualized Round Robin XLIX Report: FSV-BA

## Graphical Comparability Summary



## **Appendix E. Shipping Package Inserts for RR14**

The following three items were included in each package shipped to an RR14 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

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.

We have no record, either electronic or hardcopy, of a packing list for the RR14 study.

January, 2001

Dear Colleague:

For the past 16 years the National Institute of Standards and Technology (NIST) has coordinated a Micronutrients Measurement Quality Assurance (QA) Program for laboratories making vitamin measurements in human serum. Frozen and/or freeze dried sera are sent to laboratories for analysis as an interlaboratory comparison exercise. Results are returned to NIST for data tabulation and evaluation. Value-assignment of the sample pools is based on the median of all the laboratory results, with confirmation based on measurements at NIST. We provide consultation and trouble-shooting regarding methods of analysis, and a certificate of participation in the QA program is issued at the end of each calendar year. We also host a micronutrient analysis QA workshop for fat-soluble vitamin, carotenoid, and ascorbic acid measurements in serum.

The enclosed set of samples constitute the round robin exercise for vitamin C (Round Robin XIV) for 2001. Four vials of frozen serum (*test samples*), and a vial of solid ascorbic acid (*a control sample*), are enclosed. Please follow the attached protocol when you analyze these samples.

Report your results using the attached form by **May 12, 2001**. We also request that you send us a representative chromatogram from the analysis of each sample and indicate whether peak height or peak area was used in the calculation of the ascorbic acid concentration. Your results will be kept confidential. Results received two weeks after the due date will not be included in the summary report of this round robin study. The summary report concerning this study will be provided near the end of June.

Please mail your results to:

Micronutrients Measurement Quality Assurance Program NIST  
100 Bureau Drive, Stop 8392  
Gaithersburg, MD 20899-8392

or Fax: 301.977.0685 E-mail: sam.margolis@nist.gov

If you have any questions or concerns please call me at 301.975.3137 or contact me by Fax or E-mail.

Thank you for your participation and we look forward to receiving your results.

Sincerely,

Sam A. Margolis, Ph.D.  
Research Chemist  
Analytical Chemistry Division  
Chemical Science and Technology Laboratory

Enclosures

## Protocol for analyzing samples

The *control sample* consists of a sample of solid ascorbic acid in an amber vial and should be used in the following manner (please record your weights on the attached report form):

1. Prepare 250 mL of 5% metaphosphoric acid (MPA) in distilled water.
2. Weigh **180-220 mg** of the solid ascorbic acid sample to 0.1 mg (if possible), dissolve it in 5% MPA in a 100 mL volumetric flask, and dilute to the 100 mL mark. **Weigh the amount of MPA solution that was added.** This will be referred to as the Stock Solution.
3. Prepare three dilute solutions of the Stock Solution as follows:

*Dilute Solution 1:* **Weigh** 0.500 mL of the stock solution into a 100 mL volumetric flask. Then dilute with 5% MPA solution to 100 mL mark and **weigh the amount of MPA solution that was added.**

*Dilute Solution 2:* **Weigh** 0.250 mL of the stock solution into a 100 mL volumetric flask. Then dilute with 5% MPA solution to 100 mL mark and **weigh the amount of MPA solution that was added.**

*Dilute Solution 3:* **Weigh** 0.125 mL of the stock solution into a 100 mL volumetric flask. Then dilute with 5% MPA solution to 100 mL mark and **weigh the amount of MPA solution that was added.**

4. Record the ultraviolet absorbance spectrum of *Dilute Solution 1* against 5% MPA solution as the blank using paired cuvettes. Record the wavelength in the region of 240-245 nm at which you observe the maximum absorbance and record the absorbance at that wavelength.
5. Record the absorbance of the sample at 242, 243 and 244 nm.
6. Measure the concentration of the ascorbic acid in all three dilute solutions and the 5% MPA diluent in duplicate along with the ampouled *test samples* using your usual methods.

The purpose of measuring the absorbance at the wavelength maximum is to check the concentration of your sample. If your spectrophotometer is properly calibrated, the maximum absorbance should be between 243 and 244 nm. If the concentration is correct, the molar extinction coefficient ( $E^{1\%}$ ) of ascorbic acid at this wavelength (using a cell with a 1 cm path length) should be close to  $550 \pm 30$  nm. The extinction coefficient of your solution can be calculated using the following equation:

$$E^{1\%} \text{ dl/g}\cdot\text{cm} = \frac{\text{Observed Absorbance}_{\lambda_{\text{max}}}}{\frac{(\text{g AA}/100 \text{ mL stock})(\text{g stock in } 100 \text{ mL dilute solution})}{(\text{g AA stock solution}) + (\text{g MPA solution in } 100 \text{ mL dilute solution } 1)}}$$

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

Each *test sample* should contain between **0** and **100** μmol of ascorbic acid/L of solution. The total ascorbic acid in each ampoule should be measured in duplicate by the method(s) used in your laboratory. Please report your results in μg/L of sample.

## REPORT OF ANALYSIS

NAME:

ADDRESS:

Telephone no.: \_\_\_\_\_

Fax no.: \_\_\_\_\_

**Method of Analysis:**

*Please note the type of method that you use.* \_\_\_\_\_

*Please attach representative chromatograms.*

Method used for calculating ascorbic acid concentration.

Peak height \_\_\_\_\_ Peak area \_\_\_\_\_

Manufacturer of ascorbic acid used to make in-house standards \_\_\_\_\_

Were samples frozen upon receipt? \_\_\_\_ Yes \_\_\_\_ No

Date of Analysis: \_\_\_\_\_

### PREPARATION OF STOCK SOLUTION AND DILUTED SOLUTIONS

#### ***STOCK SOLUTION***

Weight of ascorbic acid in the Stock Solution \_\_\_\_\_ mg

Weight of 5% MPA added to the 100 mL volumetric flask \_\_\_\_\_ g

#### **DILUTE SOLUTION 1**

Weight of added stock solution (0.5 mL) \_\_\_\_\_ mg

Weight of 5% MPA added to the 100 mL volumetric flask \_\_\_\_\_ g

Absorbance of Dilute Solution 1 at **243 nm** \_\_\_\_\_ AU

Absorbance of Dilute Solution 1 at **244 nm** \_\_\_\_\_ AU

Wavelength of maximum absorbance \_\_\_\_\_ nm

Calculated molar absorptivity \_\_\_\_\_ dL/g·cm

**DILUTE SOLUTION 2**

Weight of added stock solution (0.25 mL) \_\_\_\_\_ mg  
Weight of 5% MPA added to the 100 mL volumetric flask \_\_\_\_\_ g

**DILUTE SOLUTION 3**

Weight of added stock solution (0.125 mL) \_\_\_\_\_ mg  
Weight of 5% MPA added to the 100 mL volumetric flask \_\_\_\_\_ g

COMMENTS: (use other side if necessary)

Mail by May 12, 2001 to:  
Micronutrients Measurement Quality Program  
NIST, 100 Bureau Drive, Stop 8392  
Gaithersburg, MD 20899-8392

Fax: 301-977-0685 Micronutrients  
E-mail: sam.margolis@nist.gov



# REPORT OF ANALYSIS

## RESULTS ( $\mu\text{mol/L}$ of Sample)

### DILUTE SOLUTION 1

REPLICATE 1 \_\_\_\_\_  $\mu\text{mol/L}$  of dilute solution 1  
REPLICATE 2 \_\_\_\_\_  $\mu\text{mol/L}$  of dilute solution 1

### DILUTE SOLUTION 2

REPLICATE 1 \_\_\_\_\_  $\mu\text{mol/L}$  of dilute solution 2  
REPLICATE 2 \_\_\_\_\_  $\mu\text{mol/L}$  of dilute solution 2

### DILUTE SOLUTION 3

REPLICATE 1 \_\_\_\_\_  $\mu\text{mol/L}$  of dilute solution 3  
REPLICATE 2 \_\_\_\_\_  $\mu\text{mol/L}$  of dilute solution 3

### TEST SAMPLE #1

REPLICATE 1 \_\_\_\_\_  $\mu\text{mol/L}$  of Sample 1  
REPLICATE 2 \_\_\_\_\_  $\mu\text{mol/L}$  of Sample 1

### TEST SAMPLE #2

REPLICATE 1 \_\_\_\_\_  $\mu\text{mol/L}$  of Sample 2  
REPLICATE 2 \_\_\_\_\_  $\mu\text{mol/L}$  of Sample 2

### TEST SAMPLE #3

REPLICATE 1 \_\_\_\_\_  $\mu\text{mol/L}$  of Sample 3  
REPLICATE 2 \_\_\_\_\_  $\mu\text{mol/L}$  of Sample 3

### TEST SAMPLE #4

REPLICATE 1 \_\_\_\_\_  $\mu\text{mol/L}$  of Sample 4  
REPLICATE 2 \_\_\_\_\_  $\mu\text{mol/L}$  of Sample 4

Mail by May 12, 2001 to:  
Micronutrients Measurement Quality Program  
NIST, 100 Bureau Drive, Stop 8392  
Gaithersburg, MD 20899-8392

Fax: 301-977-0685 Micronutrients  
E-mail: sam.margolis@nist.gov

## **Appendix F. Final Report for RR14**

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

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



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

August 8, 2001

Dear Colleague:

Enclosed is the summary report of the results for Round Robin 14 (RR14) 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; the measurement comparability summary for evaluating laboratory performance; and a summary of individual laboratory performance and interlaboratory accuracy and precision. As in previous reports, the estimated standard deviations (eSD) for the measurements are defined as  $0.74 \times$  interquartile range and the estimate coefficients of variation (eCV) are defined as  $100 \times \text{eSD}/\text{median}$ . NIST data are not included in the statistical analysis.

RR14 consists of four unknowns and one solid reference ascorbic acid for control solutions. Details regarding the samples can be found in the enclosed report.

Data for evaluating laboratory performance in RR14 are provided in the comparability summary (Score Card) on page 1 of the "All Lab Report." Laboratory comparability is summarized as follows: results rated 1 to 3 are within 1 to 3 standard deviation(s) of the assigned values; 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 970, Vitamin C in Frozen Human Serum. SRM 970 can be purchased from the NIST SRM Office 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.

If you have any questions regarding this report, please contact David Duewer at [david.duewer@nist.gov](mailto:david.duewer@nist.gov), phone: 301-975-3935 or Sam Margolis at [sam.margolis@nist.gov](mailto:sam.margolis@nist.gov); phone: 301-975-3137; fax: 301-977-0685.

Sincerely,

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

Sam A. Margolis, Ph.D.  
Research Chemist  
Analytical Chemistry Division  
Chemical Science and Technology Laboratory

enclosure

The NIST M<sup>2</sup>QAP Vitamin C Round Robin 14 (RR14) report consists of

Page	“Individualized” Report
1	Summarizes your reported values for the nominal 55 mmol/L control solution and the SRM 970 Level 1 and 2 samples distributed in RR11 through RR14.
2	Graphical summary of your RR 14 sample measurements.
3	Graphical summary of your RR 14 control solution measurements.

Page	“All Lab” Report
1	A listing of all results and statistics for Total Ascorbic Acid [TAA] in the RR14 samples and control solutions, the density of the 5% metaphosphoric acid (MPA) used to prepare the control solutions, the maximum absorbance reported between 243 nm and 245 nm for control solution #1, and the molar extinction coefficient.

**Samples.** Four unknowns and a control material (ascorbic acid) were distributed in RR14. . Serum sample 1 was prepared (04/93) as sample 180a. Serum sample 2 was prepared (03/95) as sample 188b. Sera samples 3 and 4 were prepared (05/98) as SRM 970, Level 1 and Level 2, respectively. Each serum sample was prepared by adding ascorbic acid to a serum pool that was depleted of ascorbic acid.

#### Qualitative Observations.

- 1) Too few sets of data have been returned to place a great deal of faith in the statistical summaries. If the program is to continue, the number of active participants must be increased. Suggestions are more than welcome!
- 2) Many participants that reported “Not detected” for the “blank” (S14-2) sample had significantly non-zero intercepts for their external calibration curve. While these intercepts averaged about 1  $\mu\text{mol/L}$  (the expected value for [TAA] in S14-2), they ranged as high as 13  $\mu\text{mol/L}$ . Future exercises will request a “true” 5% MPA blank to help sort things out.
- 3) Almost all participants correctly prepared the control solutions, although one participant appears to be using about 3% MPA and one about 10%. While the actual concentration of MPA in the nominal 5% is not believed to be a critical parameter, these two participants reported the most extreme (low and high) molar extinction coefficients ( $E^{1\%}$ ). These differences may influence calibrant purity corrections.
- 4) Based upon 1% to 10% differences between reported and calculated  $E^{1\%}$  values, several participants based their calculations on volume rather than weight of stock solution delivered to control solution #1. In general, 0.500 g gravimetric measurements are more accurate than are 0.500 mL volumetric measurements. These differences may influence calibrant purity corrections.
- 5) All participants are now reporting “reasonable” maximum absorbance values ( $OD_{\text{max}}$ ) for the nominal 55  $\mu\text{mol/L}$  control solution #1. However, several participants report the location of maximal absorbance 1 nm to 2 nm offset from its nominal location of 244 nm. For most accurate results, spectrophotometers require periodic calibration of both their absorbance and wavelength axes.

#### Quantitative Results.

- 1) There is no sign of degradation (change in median [TAA] or increase in estimated standard deviation) in either SRM 970 Level 1 or Level 2 since their Certification in 1998. We will continue to periodically monitor these materials.

## **Appendix G. “All-Lab Report” for RR14**

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. 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.

Vitamin C 'Round Robin' 14  
Laboratory Results

Lab	Date	Method	Total Ascorbic Acid				[AA] <sub>nom</sub> ([AA] <sub>obs</sub> / [AA] <sub>cat(c)</sub> )				Ctrl1 Properties			
			S14:1	S14:2	S14:3	S14:4	14-Ctrl1	14-Ctrl2	14-Ctrl3	14-Ctrl3	D <sub>5</sub> %MPA	A <sub>max</sub>	OD <sub>max</sub>	E <sup>1</sup> %
			μmol/L solution	μmol/L solution	μmol/L solution	μmol/L solution	Nominal μmol/L solution	Nominal μmol/L solution	Nominal μmol/L solution	g/mL	nm	AU	dL/g·cm	
VC-MA	27/09/01	HPLC-EC	20.0	0.00	7.8	26.0	56.03	26.54	13.83	1.034	243.0	0.5409	548	
VC-MB	26/03/01	AO-OPD, Cobas Fara	23.0	3.60	9.8	30.2	57.54	29.32	15.38	1.033	245.0	0.5908	566	
VC-MC	02/05/01	HPLC-EC	26.0	6.32	11.4	33.0	60.08	30.72	14.60	1.032	243.0	0.5773	572	
VC-ME	17/05/01	Cobas Bio Fluoro	18.7	0.04	6.5	25.7	54.03	26.83	13.42	1.032	243.6	0.6280	568	
VC-MG	02/03/01	HPLC-UV-ionpair	18.0	0.00	9.1	27.5	55.35	24.49	10.91	1.032	243.6	0.5870	584	
VC-MH	11/04/01	HPLC-Fluor OPD	19.8	0.00	7.9		54.95	27.59	13.97	1.033	243.0	0.5461	569	
VC-MI	11/05/01	HPLC-UV	29.3	0.00		31.4	78.76	33.67	15.30	1.035				
VC-MO	15/02/01	HPLC-OPD	16.7	0.26	8.1	23.7	54.47	29.50	15.45	1.032	243.0	0.5617	556	
VC-MQ	05/06/01	HPLC-EC	18.7	1.49	7.9	24.8	53.78			1.043	242.0	0.5090	527	
VC-MR	13/03/01	HPLC-OPDA	20.5	0.00	9.0	28.5	57.17	28.67	15.25	1.032	244.0	0.5520	538	
VC-MS	13/04/01	HPLC-EC	19.0	0.00	7.9	25.7				1.031	244.0	0.5369	542	
VC-NH	23/04/01	HPLC-Fluor DAB	22.8		9.5		53.49	27.11	13.73	1.034	243.0	0.5215	565	
VC-NK	27/04/01	24DNPH	36.7	0.00	12.2	21.0	58.25	36.12	23.63	1.029	243.0	0.5470	543	
NIST	18/04/01	HPLC-EC	20.6	1.7	9.1	27.1	52.58	25.79	13.31	1.031	243.6	0.5643	572	
			13	12	12	11	12	11	11	13	12	12	12	
			Average	22.2	1.0	8.9	57.8	29.1	15.0	1.033	243.4	0.558	557	
			Min	16.7	0.00	6.5	53.49	24.49	10.91	1.029	242.0	0.509	527	
			%25	18.7	0.00	7.9	54.36	26.97	13.78	1.032	243.0	0.540	543	
			Median	20.0	0.00	8.6	55.69	28.67	14.60	1.032	243.0	0.550	561	
			%75	23.0	0.57	9.6	57.72	30.11	15.34	1.034	243.7	0.580	568	
			Max	36.7	6.32	12.2	78.76	36.12	23.63	1.043	245.0	0.628	584	
			eSD	3.0	0.00	1.1	2.60	2.71	1.15	0.001	0.44	0.030	18	
			eCV	15		13	5	9	8	0.1	0	5.4	3.2	

## **Appendix H. Representative “Individualized Report” for RR14**

Each participant in RR14 received an “Individualized Report” reflecting their reported results. The following three pages are the “Individualized Report” for participant “VC-MA”.

# Vitamin C 'Round Robin' 14 Report: Participant VC-MA

Date	Method	RR	Control	AA mg	MPA g	Stock mg	MPA g	[AA] $\mu\text{mol/L}$ Calc	[AA] $\mu\text{mol/L}$ Obs	A <sub>243</sub> OD	A <sub>244</sub> OD	A <sub>245</sub> OD	E <sup>1%</sup> <sub>max</sub> dL/gcm
09/23/98	HPLC-EC (Height)	11	55 $\mu\text{mol/L}$	200.0	103.09	526.0	102.45	57.8	6.1	0.0525	0.0527		0
04/02/99	HPLC-EC (Height)	12	55 $\mu\text{mol/L}$	215.0	103.10	517.1	102.52	61.1	53.1	0.0721	0.0721		67
09/17/01	HPLC-EC	13	55 $\mu\text{mol/L}$	200.8	103.16	508.0	102.58	56.0	55.4	0.5650	0.5232		572
10/02/01	HPLC-EC (Height)	14	55 $\mu\text{mol/L}$ , Ctrl-1	200.3	103.23	510.0	102.31	56.1	57.1	0.5409	0.5193		548

297  $\pm$ 305

Grand Average

Mean	SD <sub>repeat</sub>	SD <sub>reprod</sub>
7.8	0.3	0.5

[TAA]  $\mu\text{mol/L}$ <sub>sample</sub>

RR	Sample	Rep <sub>1</sub>	Rep <sub>2</sub>	Factor	Mean	SD <sub>dup</sub>
11	SRM Lv 1, A	15.5	13.9	0.5	7.4	0.6
11	SRM Lv 1, B	14.0	14.5	0.5	7.1	0.2
12	SRM Lv 1, A	14.5	15.8	0.5	7.6	0.5
12	SRM Lv 1, B	16.1	15.1	0.5	7.8	0.3
13	SRM Lv 1, S13-1	8.4	8.5	1.0	8.5	0.1
14	SRM Lv 1, S14-3	8.0	7.8	1.0	7.9	0.2

25.6	1.1	1.7
------	-----	-----

11	SRM Lv 2, A	50.7	47.7	0.5	24.6	1.1
11	SRM Lv 2, B	48.8	54.0	0.5	25.7	1.8
12	SRM Lv 2, A	49.5	45.9	0.5	23.9	1.3
12	SRM Lv 2, B	46.2	47.9	0.5	23.5	0.6
13	SRM Lv 2, S13-2	27.6	27.7	1.0	27.7	0.1
14	SRM Lv 2, S14-4	25.7	26.4	1.0	26.0	0.5

14	S14-1 (180B)	20.4	19.6	1.0	20.0	0.6
----	--------------	------	------	-----	------	-----

14	S14-2 (188B)			1.0	0.0	0.0
----	--------------	--	--	-----	-----	-----

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

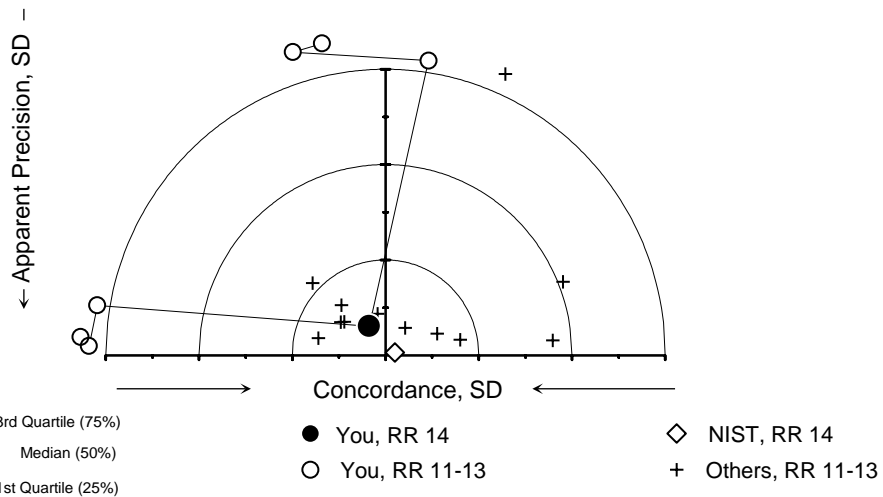
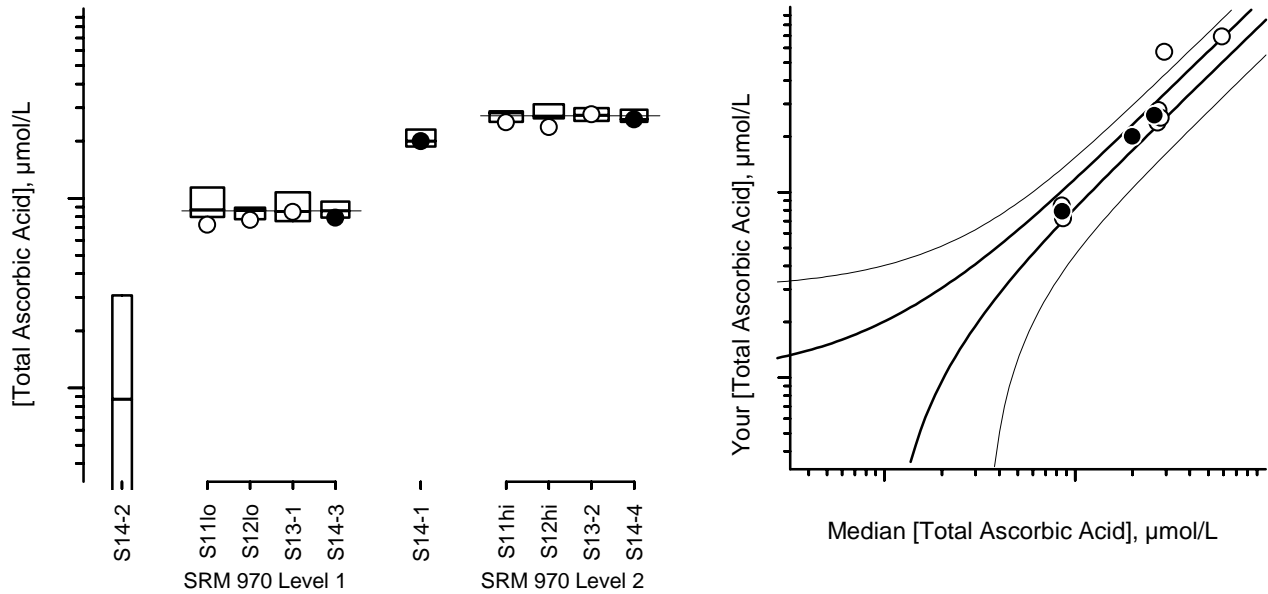
Micronutrients Measurement Quality Assurance Program  
 National Institute of Standards and Technology  
 100 Bureau Drive Stop 8392  
 Gaithersburg, MD 20899-8392 USA

Fax: (301) 977-0685  
 Email: sam.margolis@nist.gov



# Vitamin C 'Round Robin' 14 Report: Participant VC-MA

## Total Ascorbic Acid: Samples



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

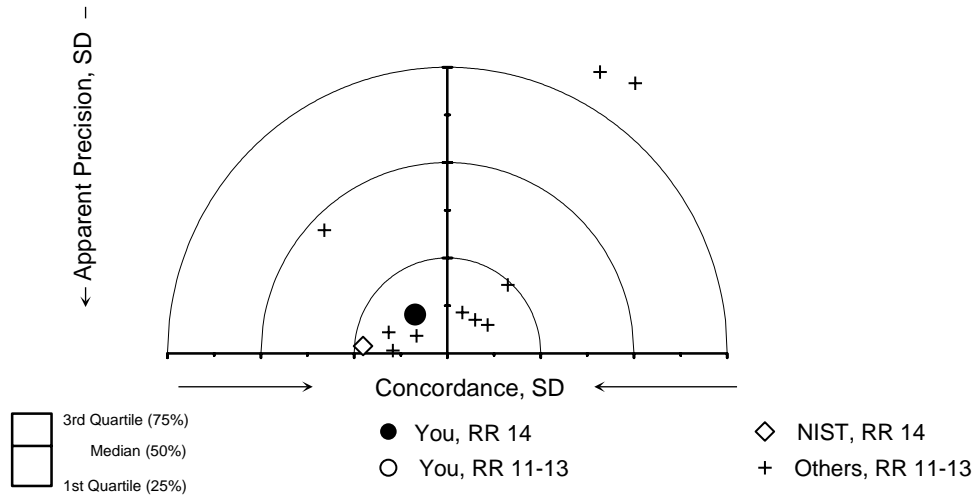
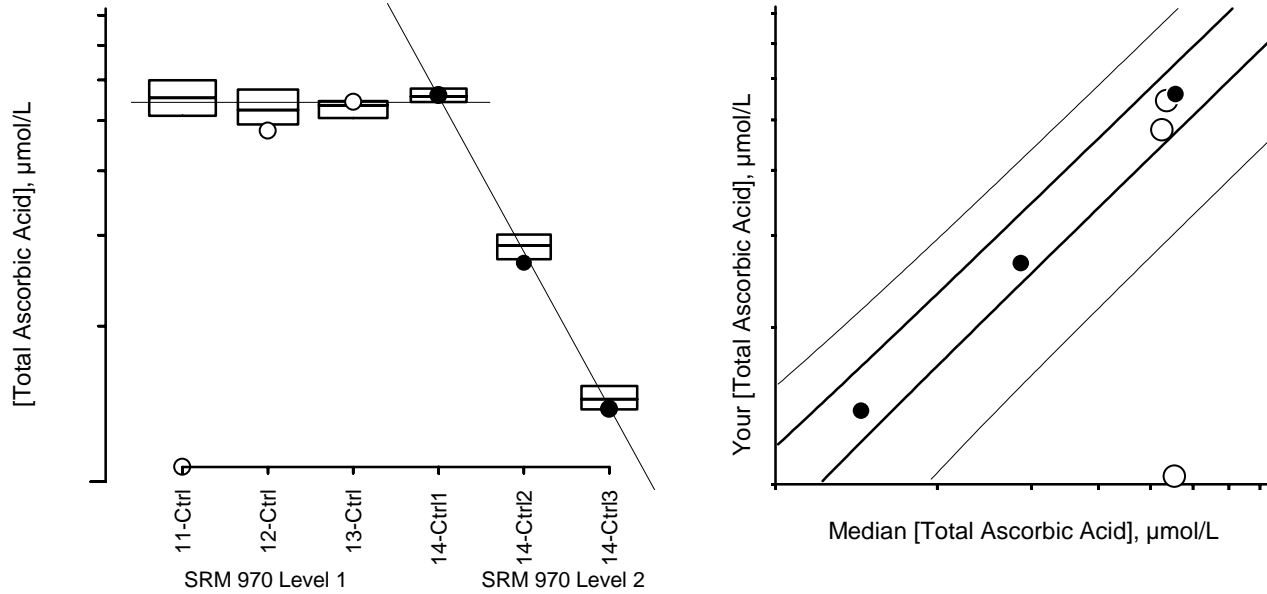
Sample

- S14-1 Serum 180B, augmented
- S14-2 Serum 188B, no augmentation
- S14-3 SRM 970 Level 1
- S14-4 SRM 970 Level 2

Comments

# Vitamin C 'Round Robin' 14 Report: Participant VC-MA

## Total Ascorbic Acid: Control Solutions



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

Sample

Comments

- 14Ctrl-1 Control Solution 1, nominally 55.0 µm/mL. Displayed Value = (Nominal)(Measured)/(Calculated)
- 14Ctrl-2 Control Solution 2, nominally 27.5 µm/mL. Displayed Value = (Nominal)(Measured)/(Calculated)
- 14Ctrl-3 Control Solution 3, nominally 13.75 µm/mL. Displayed Value = (Nominal)(Measured)/(Calculated)