Dear DSQAP participants,

Please find a copy of the report from Exercise C enclosed, we are happy to answer any questions you may have about the report and the exercise. We would like to thank the laboratories that sent additional method information; we are using the data to plan discussions for the next DSQAP workshop.

Do you have any suggestions for Exercise D? Based on the results from Exercise C, we would recommend repeating some water soluble vitamins, possibly the phytosterols and fatty acids. Do you see any problem areas in the report? Are there other analyte/matrix pairs that would help you? If so, please let us know in an e-mail to dsqap@nist.gov.

Best regards,
DSQAP Team

Dietary Supplement Quality Assurance Program
Exercise C
Final Data Report

Your laboratory code for this exercise: NIST

This report consists of several parts:

- **Overview**: a general description of the statistical treatment of the data, and how to read the plots.
- **Data Table**:, a table with your laboratory's individual results, the community results, and the NIST results.
- **Graphs**: a section that includes graphical representation of the data for the analytes tested in this exercise, points to consider when examining the data, and when appropriate, recommendations from exercise C.

As always, if you have any questions, please contact us.

OVERVIEW

STATISTICS:

Your individual data table and graphs contain information about your performance relative to the rest of the participants and relative to a target around the expected result.

INDIVIDUAL DATA TABLE

Section 1 of the data table contains your results, including your mean and standard deviation. Please check these and make sure that you agree with the data in the table.

Section 2 of the data table contains the community results, including the median value for each analyte, the MADe (a robust estimate of the standard deviation), and the minimum/maximum values reported for the analyte.

Section 3 of the data table contains the NIST results. In most cases, the value and the U95 confidence interval have been determined with two independent analytical methods. At least six samples have been tested with each of the methods and duplicate sample preparations from the sample package have been included allowing for the U95 to encompass homogeneity within and between packages.

GRAPHS

Two graphs are provided for each analyte in each sample, one which plots lab results for the sample vs. the participating labs (View 1), the second plots lab results for the sample vs. lab results for the control (View 2). Both views include the consensus values and the target values.

View 1

Individual laboratory data are plotted with the individual laboratory standard deviation. The black solid line represents the consensus median and the black dotted lines represent the consensus variability. The center of the region with the red hash marks represents our view of the "correct answer", it is bounded (+/-) two times the pooled standard deviation of the participants or two times the NIST uncertainty (whichever is larger), thus creating a target zone. With this view, it is relatively easy to determine if a laboratory falls within the target zone, it is also possible to compare where the target zone lies relative to the consensus values. In most cases, the target zone falls within the consensus values which is the expected result. One program goal is to bring the consensus values closer together and clustered around the target value.

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View 2

In this view the results reported for the sample are plotted vs. the results for the control. The red box represents the target values for the control (x-axis) and the sample (y-axis) and the black dotted lines represent the analogous information for the consensus values.

This view provides additional information to complement view 1. For example, if your values are low for both the control and sample (or high for both), you may have calibration issues. If your laboratory falls into this category, you may want to investigate how your calibrants are prepared as well as the purity of your calibrant material.

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National Institute of Standards and Technology
Analytical Chemistry Division

Analyte	Units	1. Your Results, Lab Code: NIST	1. Your Results, Lab Code: Mean	1. Your Results, Lab Code: s_{total}	2. Community Results: N	Median	MADe	Min	Max	3. NIST: Value	3. NIST: U_{95}
Arsenic	ng/g		350.00		13	368.000	34.594	254	2854	350.0	35.0
Cadmium	ng/g		14.00		13	16.00	4.91	12.7	129.1	14.00	1.40
Calcium	% mass fraction		0.490		13	0.498	0.018	0.5	0.6	0.4900	0.0130
Phosphorus	% mass fraction		0.378		10	0.392	0.016	0.3	2.4	0.378	0.004
Sodium	mg/kg		4150		11	4154	516	3437	6167	4150	140
Zinc	mg/kg		152.300		13	156.333	9.439	133.3	181.1	152.30	5.10
Retinol	mg/kg		16.10		10	15.39	1.70	5.6	22.4	16.10	1.30
Niacin	mg/kg		97.50		13	107.67	47.94	17.6	498.0	97.50	2.30
Vitamin B6	mg/kg		14.20		14	15.33	5.09	11.0	420.8	14.20	1.50
Campesterol	mg/g		0.12		6	0.08	0.02	0.07	0.10	0.12	0.00
β-sitosterol	mg/g		0.45		6	0.29	0.05	0.23	0.37	0.45	0.02
Stigmasterol	mg/g		0.05		6	0.04	0.01	0.03	0.05	0.05	0.00
Campesterol (solution)	mg/g		0.31		6	0.22	0.06	0.10	0.29	0.31	0.03
β-sitosterol (solution)	mg/g		1.15		6	1.08	0.32	0.55	1.42	1.15	0.01
Stigmasterol (solution)	mg/g		0.13		6	0.13	0.02	0.09	0.15	0.13	0.00
Linoleic Acid	mg/g		374.00		11	364.10	26.04	325.0	471.7	374.0	35.0
α-Linolenic Acid	mg/g		3.45		10	2.02	0.24	1.00	3.40	3.45	0.63
γ-Linolenic Acid	mg/g		251.00		11	236.87	15.02	222.0	332.7	251.0	24.0

Mean Average of all your reported values

s_{total} Overall standard deviation

N Number of quantitative values reported

Median Median of the reported values

MADe robust estimate of the standard deviation derived from the median absolute deviation (MAD)

Min, Max Minimum and maximum reported values

Value NIST-assessed value

U_{95} ±95% confidence interval about the assessed value

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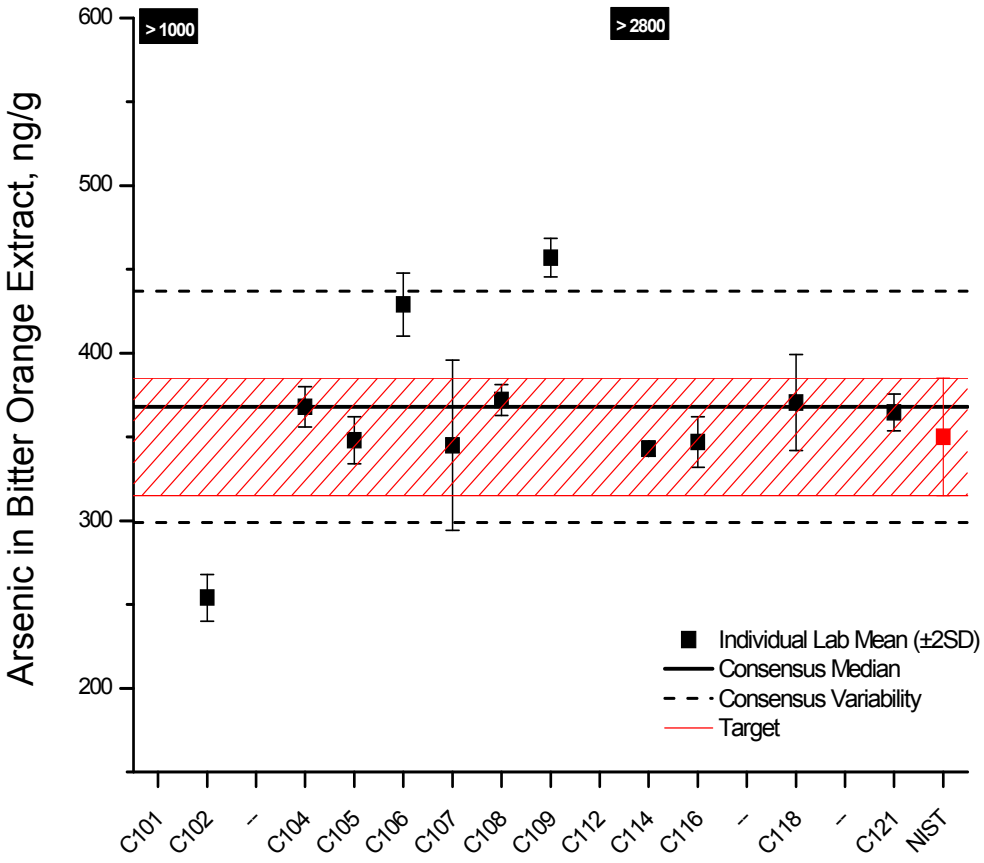
ARSENIC

Arsenic View 1



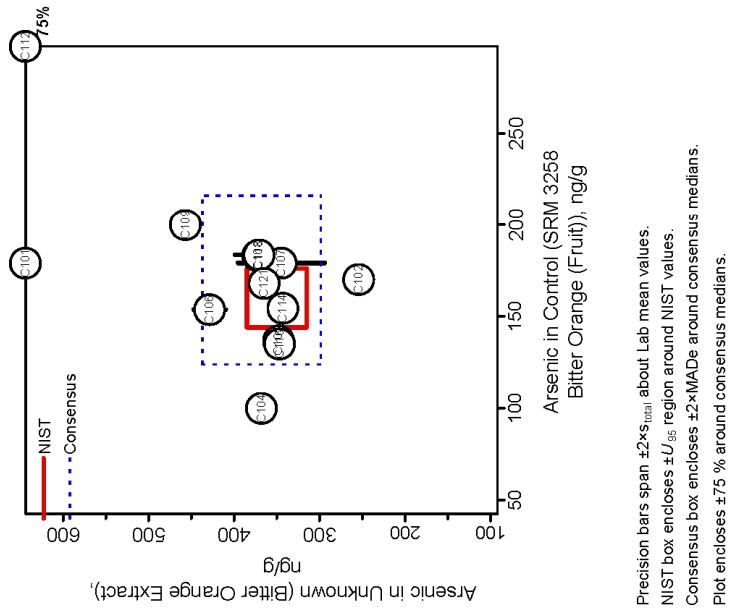
Return to Introduction

Arsenic View 2

Dietary Supplements Quality Assurance Program
December 2008 Study - Round C

Arsenic, ng/g

Lab	Control SRM 3258 B	Unknown: A1	Bitter Orange Extract B1	C1	Mean	s_{total}
C101	179.	1060.	970.	1080	1037	59
C102	170.	250.	250.	262	254	7
C104	100.	362.	374.	368	368	6
C105	136.7	346.0	342.8	353	348	7
C106	153.7	417.7	434.4	434	429	9
C107	179.	374.	330.	330	345	25
C108	183.	377.	371.	368	372	5
C109	200.	460	460	450	457	6
C112	1684.	3276.8	1934.5	3352	2854	797
C114	154.43	340.8	345.3	342	343	2
C116	135.	350.	338.	352	347	8
C118	183.5	386.7	359.5	365.6	370.57	14.30
C121	168.	365.	370.	359.	364.67	5.51
N	13				13	
Mean	278.95				606.73	222.0
Median	170.00				368.00	
MADe	23.08				34.59	69.19
%RSD	13.6				9.4	
NIST	160.000				350.00	
$\pm U_{95}$	16.000				35.000	

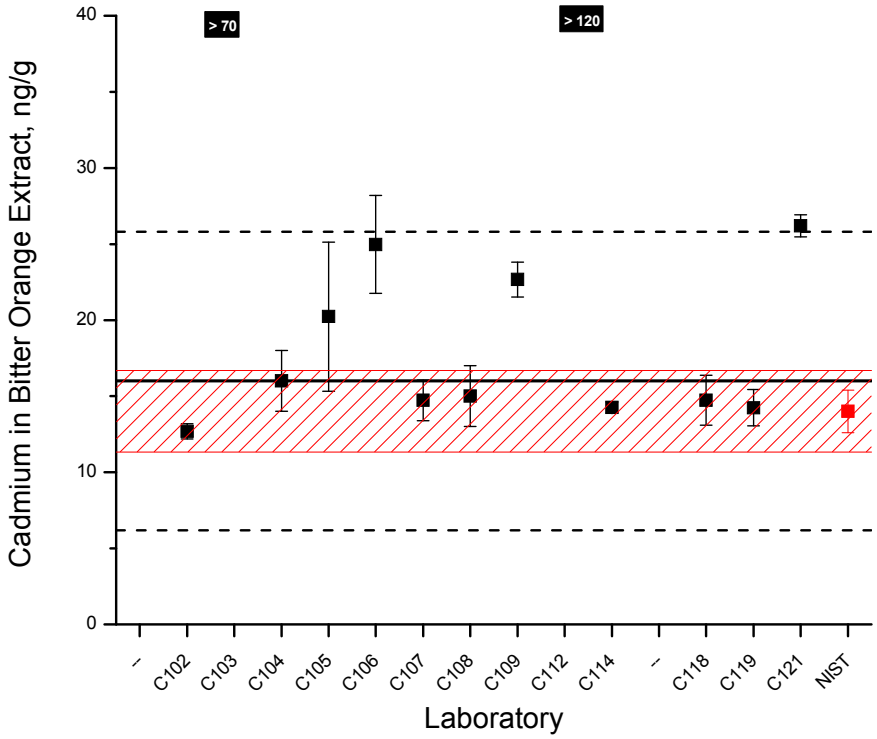


Precision bars span $\pm 2 \times s_{total}$ about Lab mean values.
NIST box encloses $\pm U_{95}$ region around NIST values.
Consensus box encloses ±2×MADe around consensus medians.
Plot encloses ±75 % around consensus medians.

Return to Introduction

CADMIUM

Cadmium View 1



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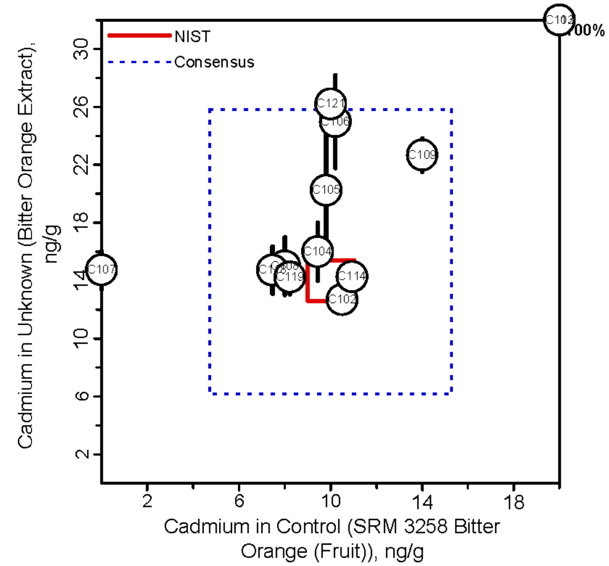
Cadmium View 2

Dietary Supplements Quality Assurance Program
May 2008 Study - Round B

Cadmium, ng/g

	Control	Unknown: Bitter Orange Extract				
Lab	SRM 3258 B	A1	B1	C1	Mean	s_{total}
C102	10.5	12.95	12.68	12.44	12.69	0.26
C103	70.	75.	72.	69.	72.00	3.00
C104	9.43	16.	17.	15.	16.00	1.00
C105	9.8	22.7	17.8	20.2	20.23	2.45
C106	10.2	23.38	26.59	24.98	24.98	1.61
C107		14.9	14.	15.3	14.73	0.67
C108	8.	15.	16.	14.	15.00	1.00
C109	14.	23.	22.	23.	22.67	0.58
C112	87.7	106.5	131.7	149.	129.07	21.37
C114	10.93	14.45	14.08	14.26	14.26	0.19
C118	7.46	15.49	13.86	14.83	14.73	0.82
C119	8.22	13.95	13.84	14.92	14.24	0.59
C121	10.	25.9	26.1	26.6	26.20	0.36
N	13				13	
Mean	20.17				30.52	6.07
Median	10.00				16.00	
MADe	2.64				4.91	9.81
%RSD	26.4				30.7	
NIST	10.000				14.000	
$\pm U_{95}$	1.000				1.400	

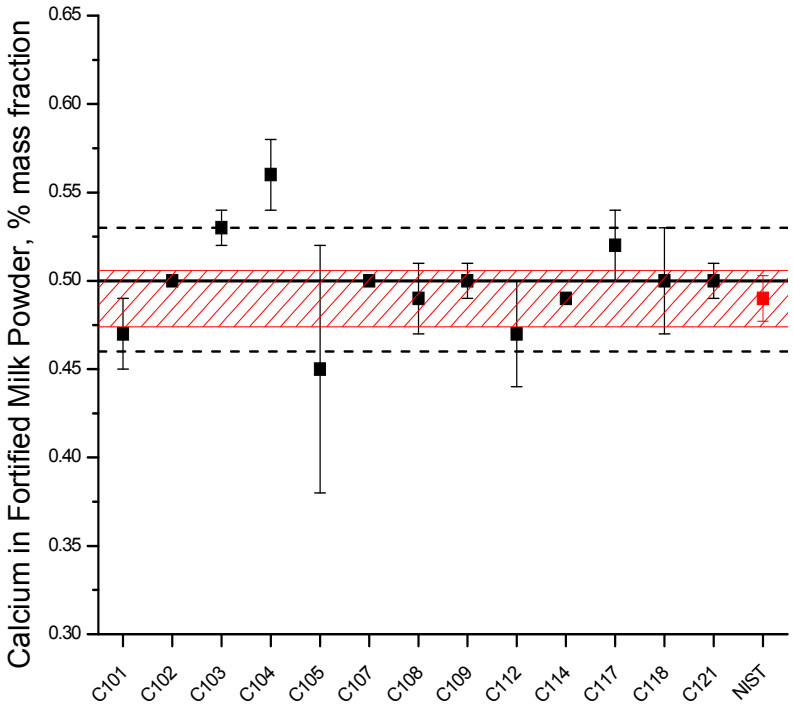


Precision bars span $\pm 2 \times s_{total}$ about Lab mean values.
NIST box encloses $\pm U_{95}$ region around NIST values.
Consensus box encloses ±2×MADe around consensus medians.
Plot encloses ±100 % around consensus medians.

Return to Introduction

CALCIUM

Calcium View 1



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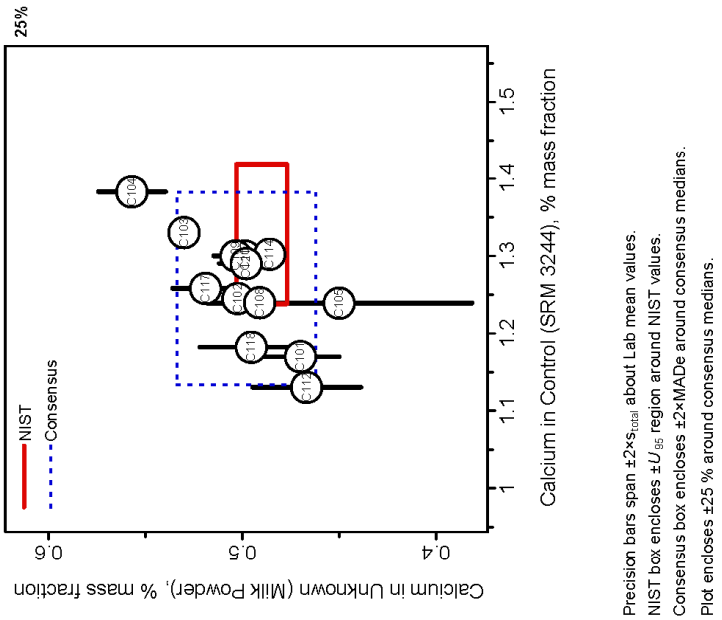
Calcium View 2

Dietary Supplements Quality Assurance Program

May 2008 Study - Round B

Calcium, % mass fraction

	Control	Unknown: Milk Powder				
Lab	SRM 3244	A1	B1	C1	Mean	s_{total}
C101	1.17	0.46	0.47	0.48	0.47	0.01
C102	1.25	0.5	0.5	0.5	0.50	0.00
C103	1.33	0.53	0.53	0.53	0.53	0.00
C104	1.38	0.55	0.55	0.57	0.56	0.01
C105	1.24	0.49	0.44	0.43	0.45	0.03
C107	1.3	0.5	0.5	0.5	0.50	0.00
C108	1.24	0.49	0.48	0.5	0.49	0.01
C109	1.3	0.5	0.51	0.5	0.50	0.01
C112	1.13	0.48	0.47	0.45	0.47	0.01
C114	1.3	0.49	0.48	0.49	0.49	0.00
C117	1.26	0.53	0.51	0.52	0.52	0.01
C118	1.18	0.5	0.5	0.48	0.50	0.01
C121	1.29	0.5	0.51	0.49	0.50	0.01
N	13				13	
Mean	1.26				0.50	0.01
Median	1.26				0.50	
MADe	0.06				0.02	0.04
%RSD	4.9				3.6	
NIST	1.328				0.490	
$\pm U_{95}$	0.090				0.013	



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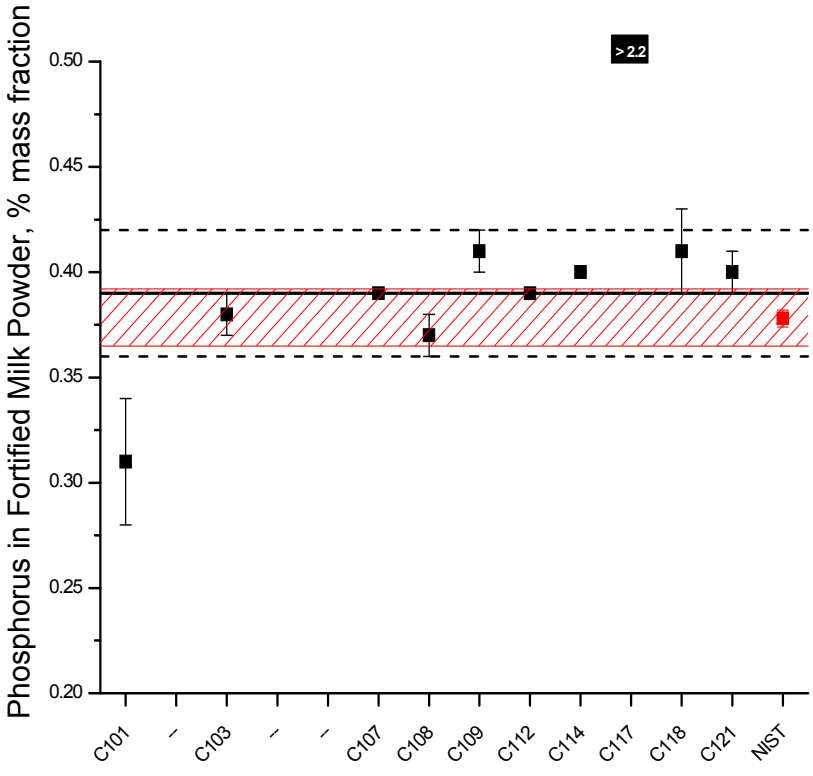
PHOSPHORUS

Phosphorus View 1



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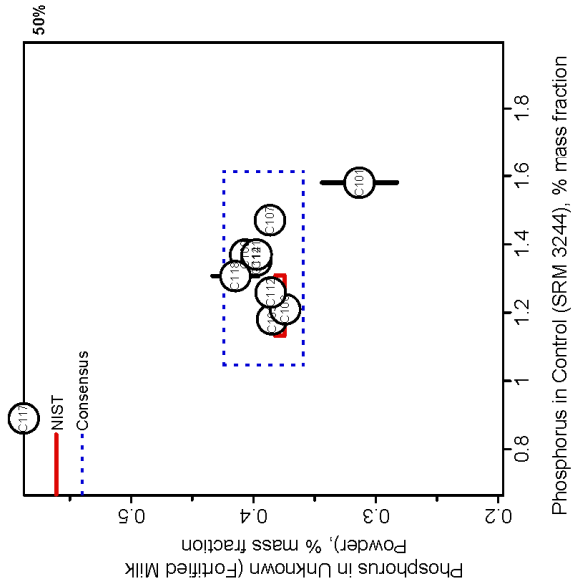
Phosphorus View 2

Dietary Supplements Quality Assurance Program
May 2008 Study - Round B

Phosphorus, % mass fraction

Lab	Control SRM 3244	Unknown: Fortified Milk Powder A1	B1	C1	Mean	s_{total}
C101	1.58	0.3	0.33	0.31	0.31	0.02
C103	1.18	0.38	0.39	0.39	0.38	0.00
C107	1.47	0.39	0.39	0.39	0.39	0.00
C108	1.21	0.37	0.37	0.38	0.37	0.01
C109	1.37	0.41	0.4	0.41	0.41	0.01
C112	1.26	0.39	0.38	0.39	0.39	0.00
C114	1.35	0.4	0.4	0.4	0.40	0.00
C117	0.89	2.67	2.58	1.86	2.37	0.44
C118	1.31	0.42	0.42	0.4	0.41	0.01
C121	1.37	0.4	0.4	0.39	0.40	0.00
N	10				10	
Mean	1.30				0.58	0.14
Median	1.33				0.39	
MADe	0.14				0.02	0.03
%RSD	10.6				4.2	
NIST	1.220				0.378	
$\pm U_{95}$	0.088				0.004	



Precision bars span $\pm 2 \times s_{total}$ about Lab mean values.
NIST box encloses $\pm U_{95}$ region around NIST values.
Consensus box encloses ±2×MADe around consensus medians.
Plot encloses ±50 % around consensus medians.

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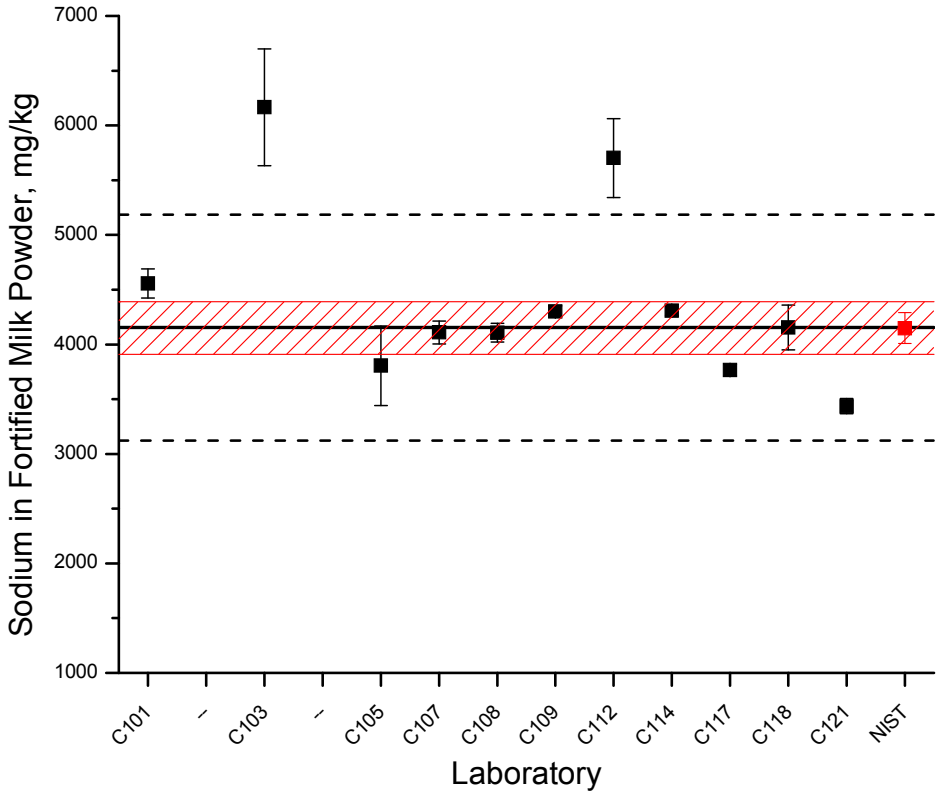
SODIUM

Sodium View 1



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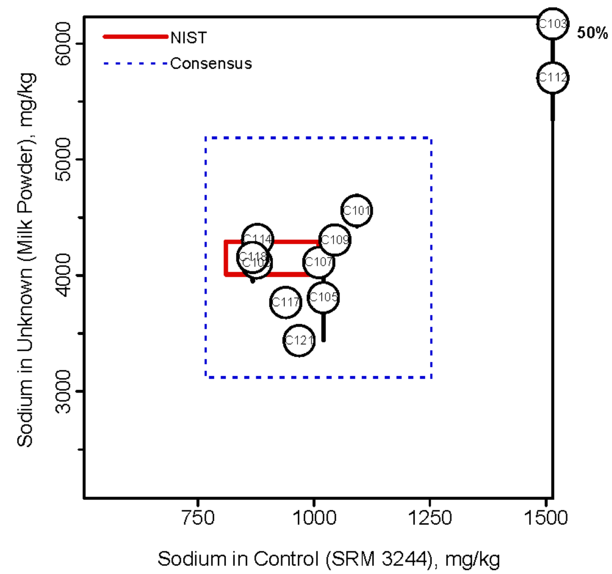
Sodium View 2

Dietary Supplements Quality Assurance Program
May 2008 Study - Round B

Sodium, mg/kg

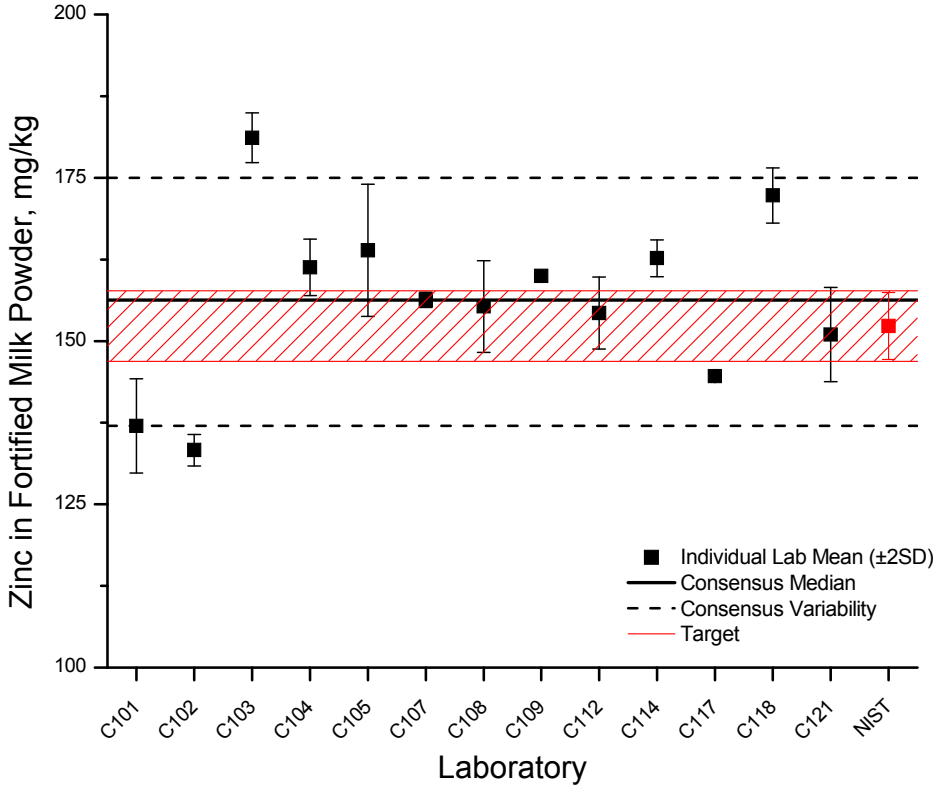
Lab	Control SRM 3244	Unknown: Milk Powder A1	B1	C1	Mean	s_{total}
C101	1092.	4500.	4540.	4630	4556.7	66.6
C103	2567.	5900.	6433.	6167	6166.7	266.5
C105	1019.8	3968.2	3610.3	3841	3806.4	181.4
C107	1010.	4170.	4080.	4080	4110.	52.
C108	876.	4140.	4059.	4121	4106.7	42.4
C109	1045.	4280.	4330.	4300	4303.3	25.2
C112	2472.5	5777.5	5832.5	5498	5702.5	179.7
C114	878.3	4322.	4318.	4283	4307.7	21.5
C117	938.74	3741.1	3793.2	3761	3765.2	26.3
C118	867.9	4215.	4212.	4036	4154.3	102.5
C121	968.	3410.0	3425.0	3476	3437.0	34.60
N	11				11	
Mean	1248.7				4401.5	120.1
Median	1010.0				4154.3	
MADe	121.6				515.8	1032
%RSD	12.0				12.4	
NIST	910.0				4150.0	
$\pm U_{95}$	100.0				140.0	



Precision bars span $\pm 2 \times s_{total}$ about Lab mean values.
NIST box encloses $\pm U_{95}$ region around NIST values.
Consensus box encloses ±2×MADe around consensus medians.
Plot encloses ±50 % around consensus medians.

Return to Introduction

Zinc View 1

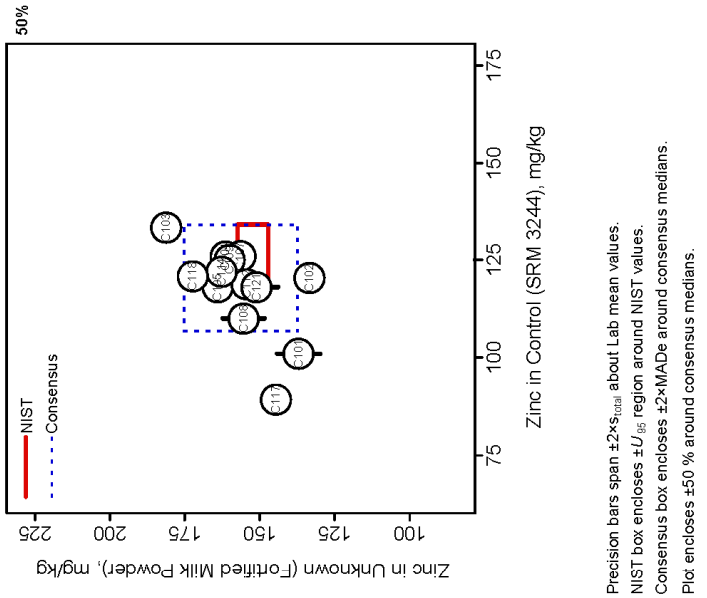


Return to Introduction

Zinc View 2

Dietary Supplements Quality Assurance Program
May 2008 Study - Round B

Zinc, mg/kg



	Control	Unknown: Fortified Milk Powder				
Lab	SRM 3244	A1	B1	C1	Mean	s_{total}
C101	101.	133.	138.	140.	137.	3.61
C102	120.4	134.7	132.9	132.4	133.3	1.21
C103	133.3	180.	183.3	180.	181.1	1.91
C104	125.85	158.9	162.3	162.9	161.3	2.16
C105	118.	169.4	159.4	163.	163.9	5.06
C107	126.	156.	157.	156.	156.3	0.58
C108	110.	155.	152.	159.	155.3	3.51
C109	125.	160.	160.	160.	160.	
C112	118.8	151.5	154.5	157.	154.3	2.75
C114	122.	162.5	164.2	161.4	162.7	1.41
C117	89.28	144.7	144.7	144.3	144.6	0.26
C118	120.8	173.9	173.1	169.9	172.3	2.12
C121	118.	147.	152.	154.	151.	3.61
N	13				13	
Mean	117.57				156.4	2.60
Median	120.40				156.3	
MADe	6.82				9.4	19
%RSD	5.7				6.0	
NIST	126.400				152.3	
$\pm U_{95}$	7.700				5.100	

Precision bars span $\pm 2 \times s_{total}$ about Lab mean values.
NIST box encloses $\pm U_{95}$ region around NIST values.
Consensus box encloses ±2×MADe around consensus medians.
Plot encloses ±50 % around consensus medians.

Return to Introduction

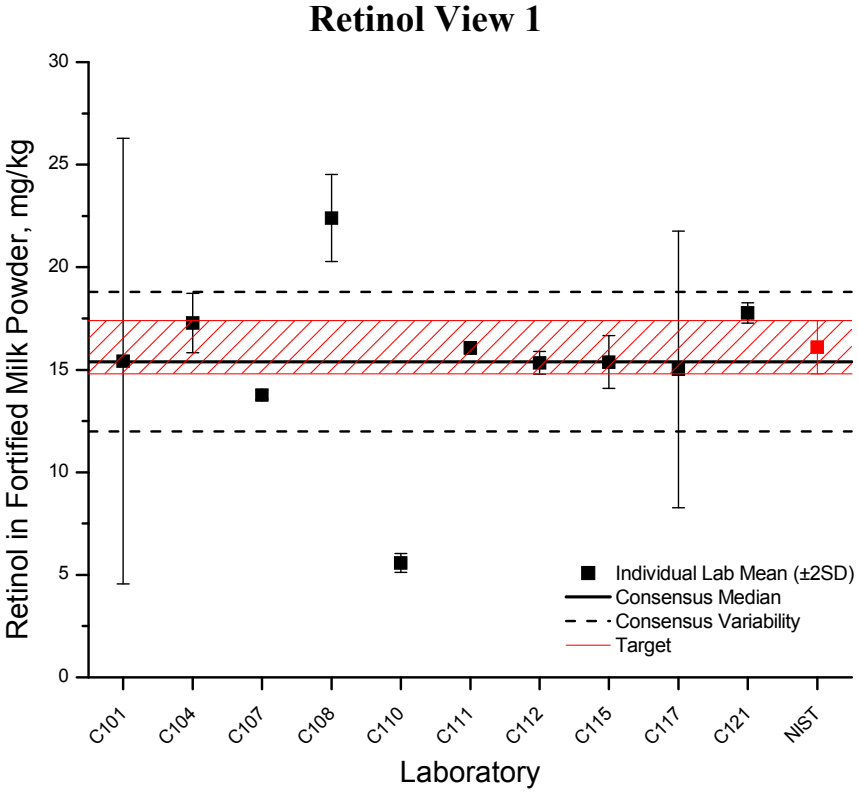
RETINOL

The fortified milk powders used for this exercise were fortified with fat-soluble vitamins, water soluble vitamins, elements, and fatty acids. The control and sample were related but not identical.

- Overall good results-only a few labs are outliers
- Several labs have higher standard deviations than expected (based on a pooled standard deviation)
- View 2 shows some evidence of calibration issues. Consider how the calibrants are made:
  - Are they traceable to a gravimetric number or a spectroscopic number?
  - Was the purity of the calibrant evaluated?
  - Did you see 13-*cis*-retinol and how was it factored in to your retinol calculation?

Recommendations:

- Check calibration method including traceability
- If your individual laboratory results had a large RSD, consider the addition of an internal standard
- Would you like to measure tocopherols in this material?



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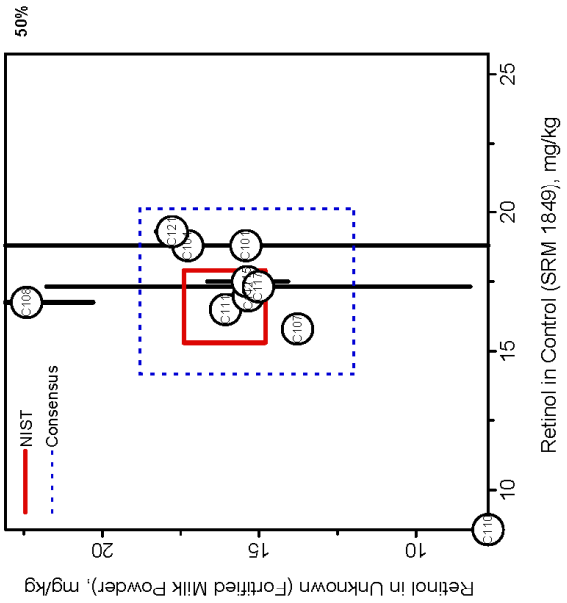
Retinol View 2

Dietary Supplements Quality Assurance Program

May 2008 Study - Round B

Retinol, mg/kg

Lab	Control SRM 1849	Unknown: Fortified Milk Powder A1	B1	C1	Mean	s_{total}
C101	18.8	18.9	9.16	18.2	15.42	5.43
C104	18.8	16.9	18.1	16.8	17.27	0.72
C107	15.8	13.7	13.9	13.7	13.77	0.12
C108	16.75	21.6	22.	23.6	22.40	1.06
C110	3.2	5.7	5.7	5.3	5.57	0.23
C111	16.5	15.9	16.1	16.2	16.07	0.15
C112	16.99	15.32	15.07	15.63	15.34	0.28
C115	17.5	15.1	14.9	16.1	15.37	0.64
C117	17.32	12.12	18.72	14.22	15.02	3.37
C121	19.3	18.	17.8	17.5	17.77	0.25
N	10				10	
Mean	16.10				15.40	2.08
Median	17.15				15.39	
MADe	1.49				1.70	
%RSD	8.7				11.1	
NIST	16.600				16.100	
$\pm U_{95}$	1.300				1.300	



Precision bars span $\pm 2 \times s_{total}$ about Lab mean values.
NIST box encloses $\pm U_{95}$ region around NIST values.
Consensus box encloses ±2×MADe around consensus medians.
Plot encloses ±50 % around consensus medians.

Return to Introduction

NIACIN AS NIACINAMIDE

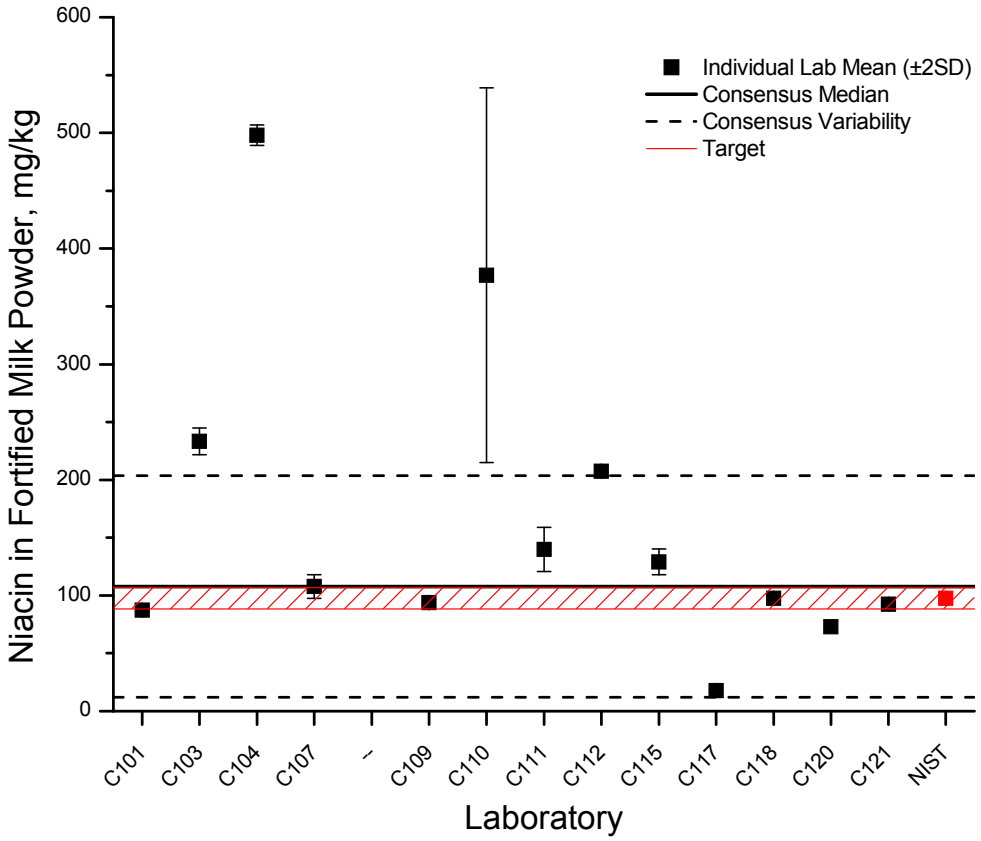
The fortified milk powder used for this exercise was fortified with fat-soluble vitamins, water soluble vitamins, elements, and fatty acids. The control was SRM 3244 Ephedra Containing Protein Powder. The level of Niacinamide in the control is approximately 2.5 times the level of niacinamide in the fortified milk powder.

- The results are significantly more scattered than we had expected and the scatter appears to be random (not related to method)
- Please check to be certain that you reported niacinamide. If you did not and you would like to submit amended data we are happy to accept it.

Recommendations:

- Check form of Niacin reported
- We suggest that we repeat this exercise with a more closely related control/unknown sample pair.

Niacin View 1



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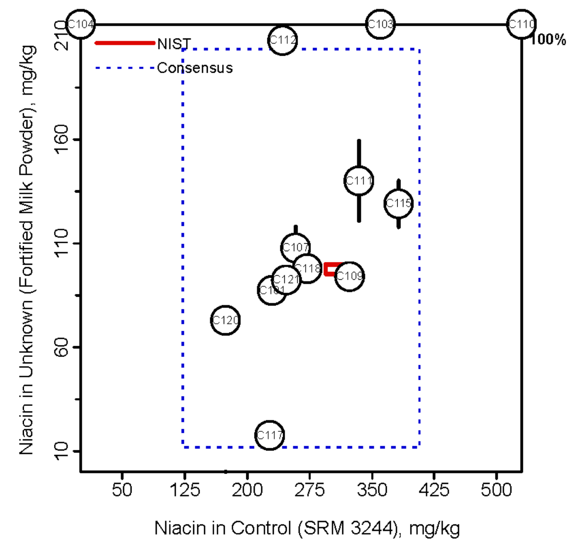
Niacin View 2

Dietary Supplements Quality Assurance Program
May 2008 Study - Round B

Niacin, mg/kg

Lab	Control SRM 3244	Unknown: Fortified Milk Powder A1	B1	C1	Mean	s_{total}
C101	230.	86.7	87.9	87.1	87.23	0.61
C103	360.	240.	230.	230.	233.3	5.8
C104		495.	496.	503.	498.	4.4
C107	258.	102.	109.	112.	107.7	5.1
C109	323.	94.6	94.8	92.3	93.9	1.4
C110	1014.	296.	377.	458.	377.	81.
C111	334.	141.	149.	130.	140.	9.5
C112	242.5	207.5	205.	210.	207.5	2.5
C115	382.	124.	128.	135.	129.	5.6
C117	227.	16.7	17.6	18.4	17.6	0.8
C118	272.	98.2	96.7	98.3	97.7	0.9
C120	174.	72.9			72.9	
C121	247.	90.6	94.8	91.5	92.30	2.21
N	12				13	
Mean	338.63				165.7	23.8
Median	265.00				107.7	
MADe	71.16				47.9	95.87
%RSD	26.9				44.5	
NIST	304.000				97.5	
$\pm U_{95}$	10.000				2.300	



Precision bars span $\pm 2 \times s_{total}$ about Lab mean values.
NIST box encloses $\pm U_{95}$ region around NIST values.
Consensus box encloses ±2×MADe around consensus medians.
Plot encloses ±100 % around consensus medians.

Return to Introduction

VITAMIN B_6 AS PYRIDOXINE HCL

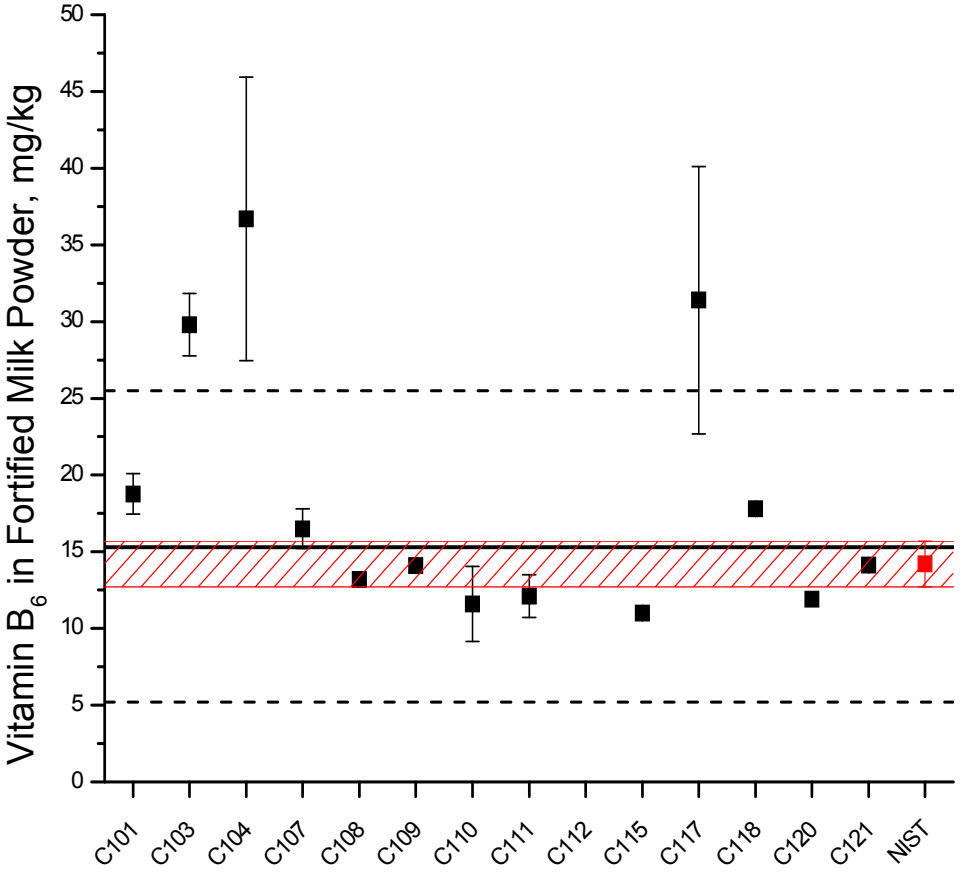
The fortified milk powder used for this exercise was fortified with fat-soluble vitamins, water soluble vitamins, elements, and fatty acids. The control was SRM 3244 Ephedra Containing Protein Powder. The level of Niacinamide in the control is approximately 2.5 times the level of niacinamide in the fortified milk powder.

- The results are significantly scattered, as with the niacinamide, the scatter does not appear to be related to the analytical measurement technique.
- Please check to be certain that you reported pyridoxine HCl. If you did not and you would like to submit amended data we are happy to accept it.

Recommendations:

- Check form of Pyridoxine HCl reported
- We suggest that we repeat this exercise with a more closely related control/unknown sample pair.

Vitamin B_6 View 1



Return to Introduction

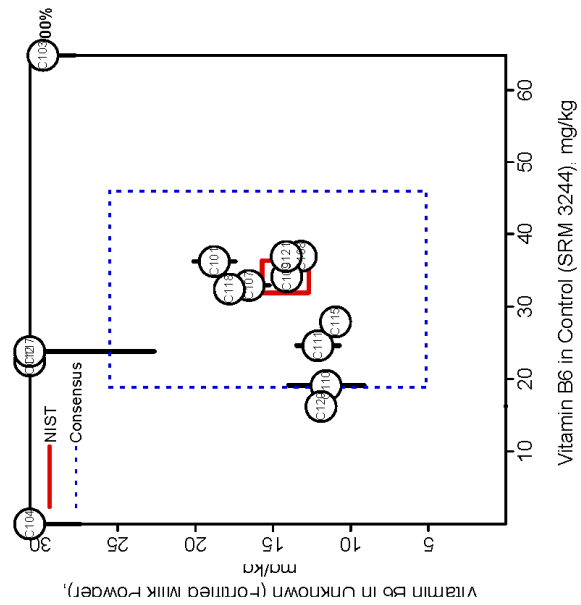
Vitamin B_6 View 2

Dietary Supplements Quality Assurance Program

May 2008 Study - Round B

Vitamin B6, mg/kg

Lab	Control SRM 3244	Unknown: Fortified Milk Powder A1	B1	C1	Mean	s_{total}
C101	36.2	19.1	19.2	18.	18.77	0.67
C103	620.	28.7	30.7	30.	29.8	1.01
C104		42.	34.	34.	36.7	4.6
C107	32.9	15.8	17.	16.8	16.5	0.6
C108	36.96	13.2	13.3	13.	13.2	0.2
C109	34.1	13.9	14.3	14.1	14.1	0.2
C110	19.1	11.3	10.5	12.9	11.6	1.2
C111	24.6	12.5	12.5	11.3	12.1	0.7
C112	22.5	417.5	420.	425.	420.8	3.8
C115	27.9	11.	11.	10.9	11.	0.1
C117	23.8	35.1	26.6	32.5	31.4	4.4
C118	32.4	17.9	17.7	17.8	17.8	0.1
C120	16.2	11.9			11.90	
C121	36.9	14.3	14.1	14.	14.13	0.15
N	13				14	
Mean	74.12				47.1	2.1
Median	32.40				15.3	
MADe	6.76				5.1	10.2
%RSD	20.9				33.2	
NIST	34.100				14.2	
$\pm U_{95}$	2.200				1.500	



Precision bars span $\pm 2 \times s_{total}$ about Lab mean values.
NIST box encloses $\pm U_{95}$ region around NIST values.
Consensus box encloses ±2×MADe around consensus medians.
Plot encloses ±100 % around consensus medians.

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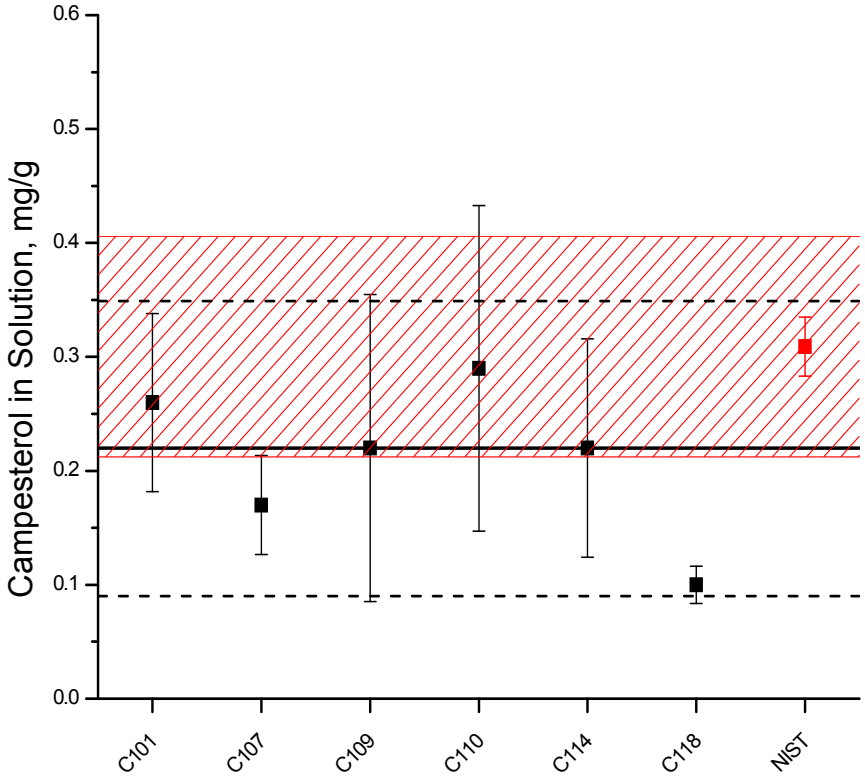
PHYTOSTEROL SOLUTIONS

The phytosterols solution was gravimetrically prepared mixture of campesterol, β-sitosterol, and stigmasterol (plus some impurities) in chloroform. It was made at a concentration that would be appropriate for a calibrant and required only derivatization. The average measurements of the sterol solution appeared to be close to the expected value; however the standard deviations were larger than expected. Most laboratories "hydrolyzed" the solution; it appears that this step introduces uncertainty into the measurement. Most labs report adding an internal standard immediately prior to the sample derivatization step, adding the internal standard at the earliest opportunity (i.e. prior to extraction and hydrolysis) would almost likely reduce the uncertainty introduced with the extended sample handling. One lab mentioned problems weighing the solution reproducibly, did others experience this?

- Results for phytosterols berries are significantly low (view 1) indicating:
  - Incomplete extraction
  - Incomplete hydrolysis

- However, view 2 shows better results for the control indicating:
  - Hydrolysis is relatively complete
  - Extraction is the issue yielding significantly low results

Campesterol in Phytosterol Solution View 1



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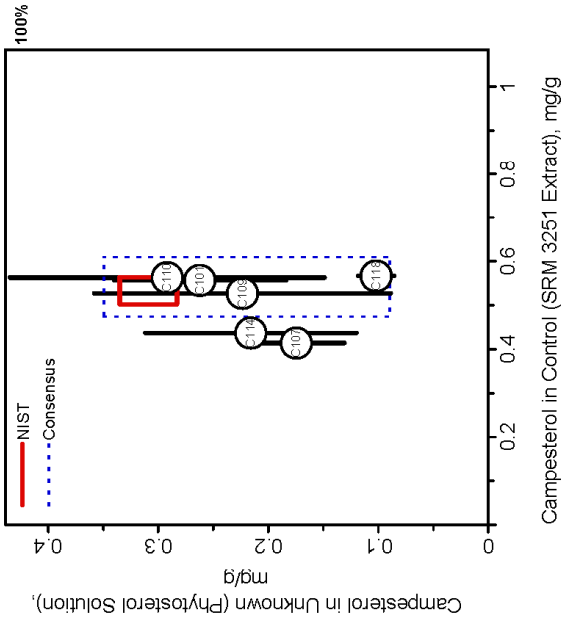
Campesterol in Phytosterol Solution View 2

Dietary Supplements Quality Assurance Program

May 2008 Study - Round B

Campesterol (solution), mg/g

	Control	Unknown: Phytosterol Solution				
Lab	SRM 3251 E	A1	B1	C1	Mean	s_{total}
C101	0.56	0.31	0.24	0.24	0.26	0.039
C107	0.41	0.15	0.18	0.19	0.17	0.022
C109	0.53	0.3	0.16	0.21	0.22	0.067
C110	0.56	0.37	0.24	0.27	0.29	0.072
C114	0.44	0.27	0.17	0.21	0.22	0.048
C118	0.57	0.11	0.09	0.11	0.10	0.01
N	6				6	
Mean	0.51				0.21	0.05
Median	0.54				0.22	
MADe	0.03				0.06	0.130
%RSD	6.3				29.6	
NIST	0.533				0.3090	
$\pm U_{95}$	0.031				0.0260	



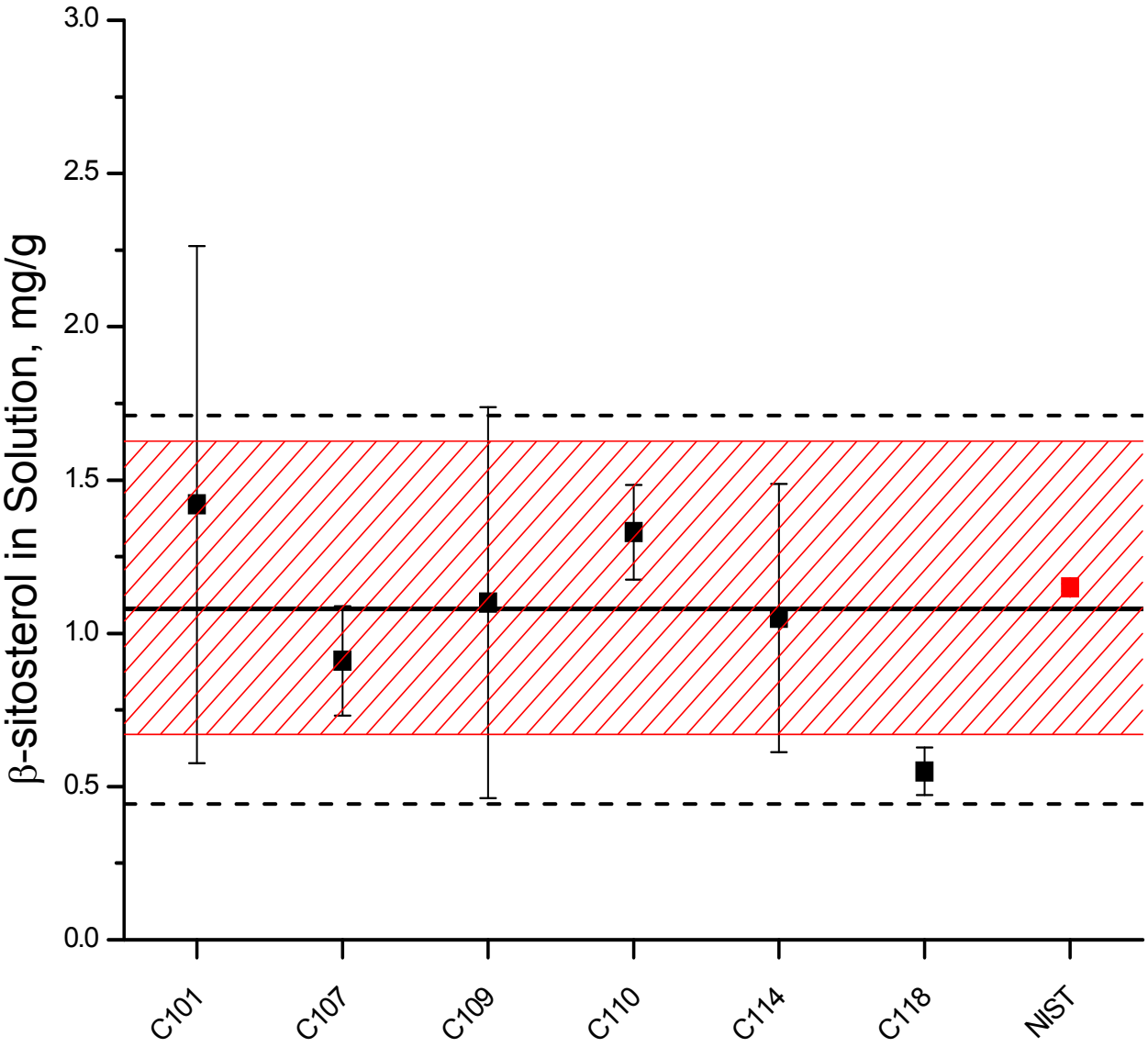
Precision bars span $\pm 2 \times s_{total}$ about Lab mean values.
NIST box encloses $\pm U_{95}$ region around NIST values.
Consensus box encloses ±2×MADe around consensus medians.
Plot encloses ±100 % around consensus medians.

Return to Introduction

β-SITOSTEROL IN SOLUTION

A discussion of the measurement of phytosterols in *Serenoa repens* extract and *Serenoa repens* fruit is included with the campesterol in solution graphs.

β-sitosterol in Solution View 1



Return to Introduction

β-sitosterol in Solution View 2

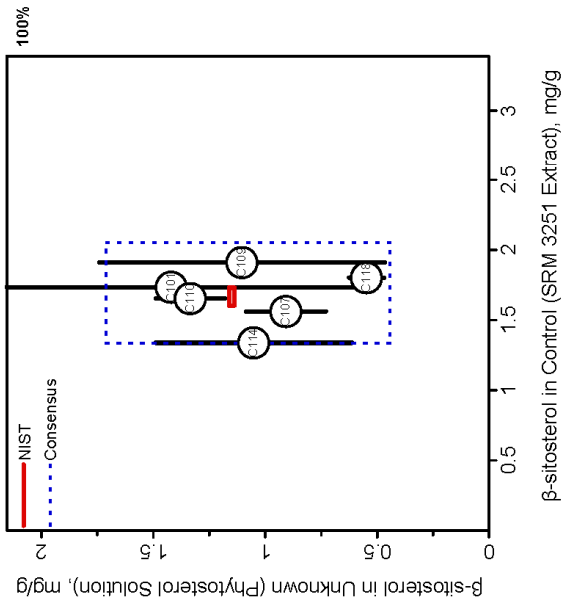
Dietary Supplements Quality Assurance Program

May 2008 Study - Round B

β-sitosterol (solution), mg/g

Lab	Control SRM 3251 E	Unknown: Phytosterol Solution A1	B1	C1	Mean	s_{total}
C101	1.73	1.91	1.18	1.17	1.42	0.42
C107	1.56	0.82	0.91	0.99	0.91	0.09
C109	1.91	1.45	0.82	1.05	1.10	0.32
C110	1.65	1.42	1.33	1.26	1.33	0.08
C114	1.34	1.28	0.84	1.03	1.05	0.22
C118	1.8	0.58	0.5	0.56	0.55	0.04

N	6		6	
Mean	1.67		1.06	0.24
Median	1.69		1.08	
MADe	0.18		0.32	0.633
%RSD	10.6		29.4	
NIST	1.666		1.149	
$\pm U_{95}$	0.064		0.012	



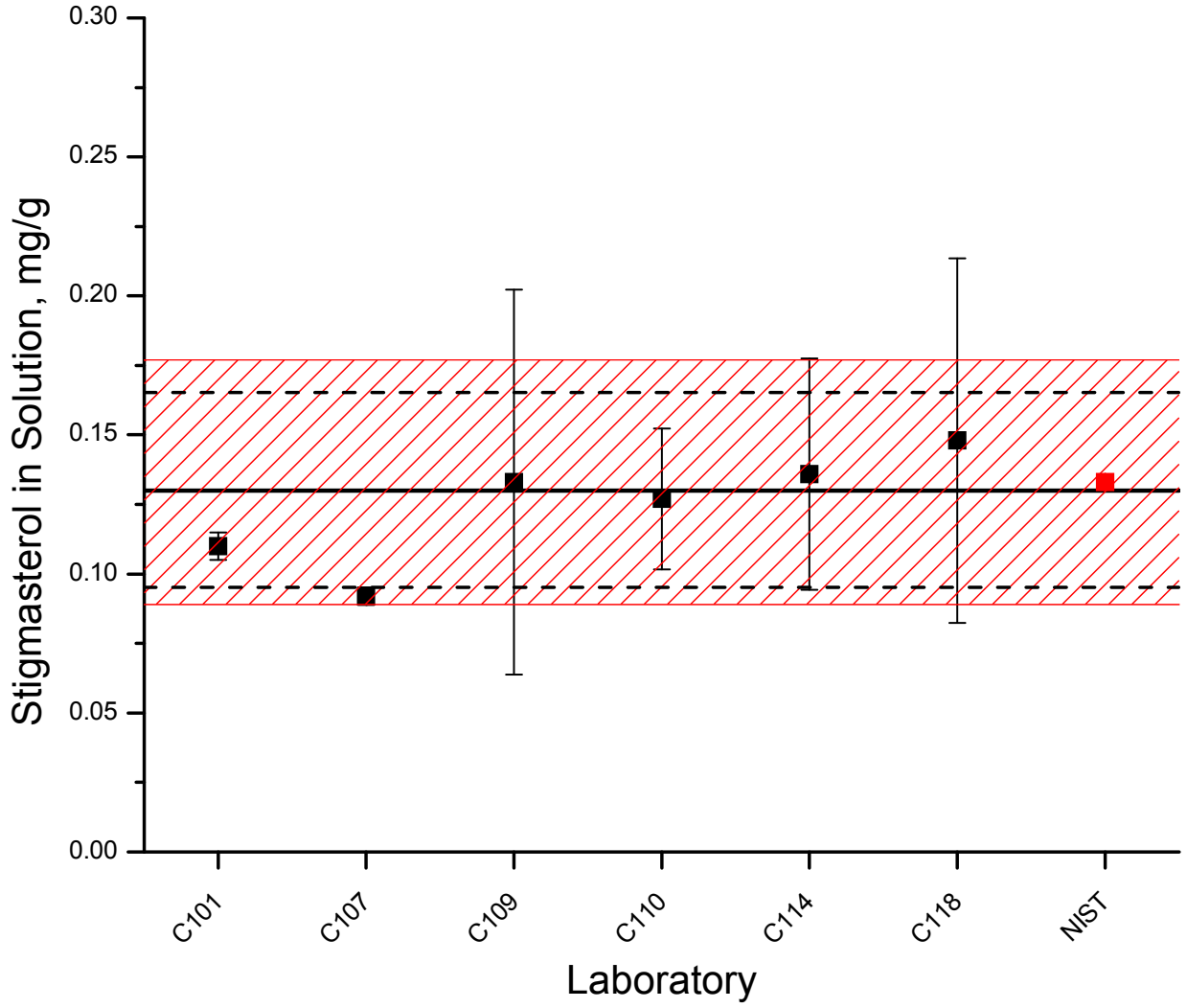
Precision bars span $\pm 2 \times s_{total}$ about Lab mean values.
NIST box encloses $\pm U_{95}$ region around NIST values.
Consensus box encloses ±2×MADe around consensus medians.
Plot encloses ±100 % around consensus medians.

Return to Introduction

STIGMASTEROL IN SOLUTION

A discussion of the measurement of phytosterols in *Serenoa repens* extract and *Serenoa repens* fruit is included with the campesterol in solution graphs.

Stigmasterol in Solution View 1



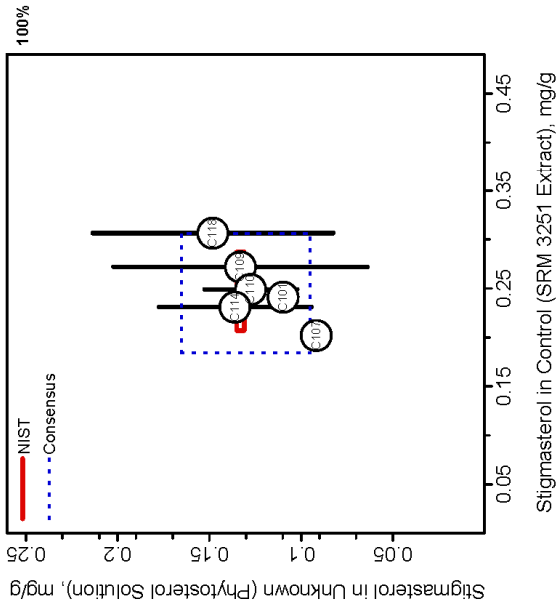
Return to Introduction

Stigmasterol in Solution View 2

Dietary Supplements Quality Assurance Program
May 2008 Study - Round B

Stigmasterol (solution), mg/g

	Control	Unknown: Phytosterol Solution				
Lab	SRM 3251 E	A1	B1	C1	Mean	s_{total}
C101	0.24	0.11	0.11	0.11	0.110	0.00
C107	0.2	0.09	0.09	0.09	0.092	0.00
C109	0.27	0.17	0.1	0.13	0.133	0.03
C110	0.25	0.14	0.13	0.12	0.127	0.01
C114	0.23	0.16	0.12	0.12	0.136	0.02
C118	0.31	0.15	0.18	0.11	0.148	0.03
N	6				6	
Mean	0.25				0.12	0.02
Median	0.25				0.130	
MADe	0.03				0.02	0.035
%RSD	12.4				13.5	
NIST	0.247				0.1330	
$\pm U_{95}$	0.040				0.0020	



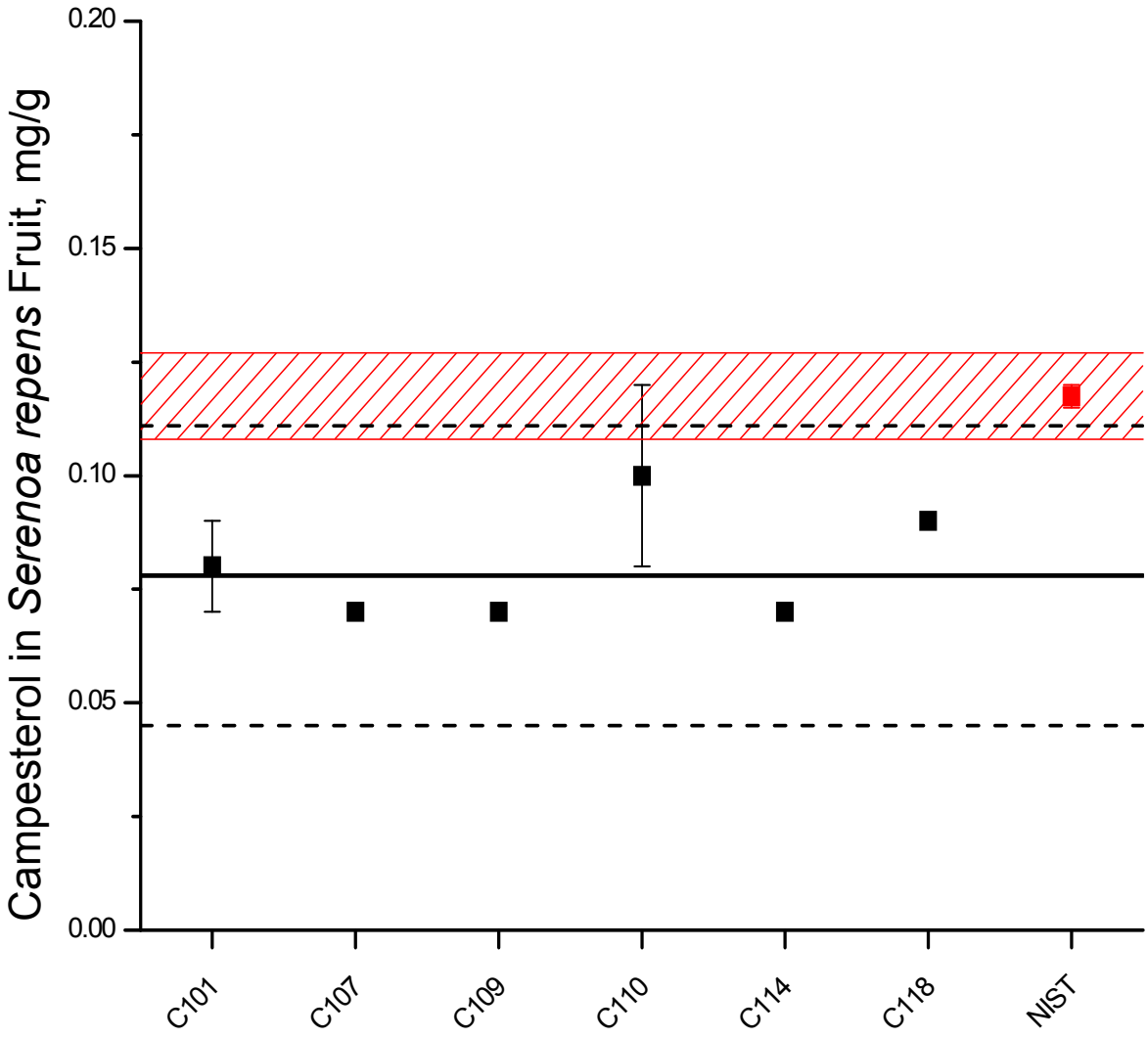
Precision bars span $\pm 2 \times s_{total}$ about Lab mean values.
NIST box encloses $\pm U_{95}$ region around NIST values.
Consensus box encloses ±2×MADe around consensus medians.
Plot encloses ±100 % around consensus medians.

Return to Introduction

CAMPESTEROL IN *SERENOA REPENS* FRUIT

A discussion of the measurement of phytosterols in *Serenoa repens* extract and *Serenoa repens* fruit is included with the campesterol in solution graphs.

Campesterol in *Serenoa repens* Fruit View 1



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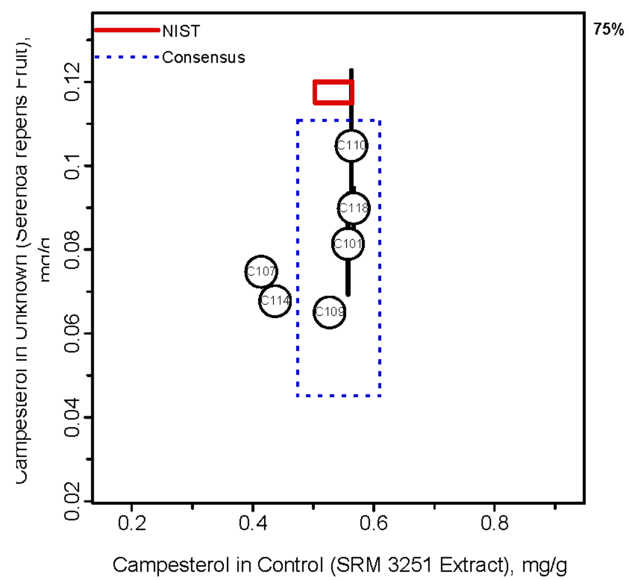
Campesterol in *Serenoa repens* Fruit View 2

Dietary Supplements Quality Assurance Program

May 2008 Study - Round B

Campesterol, mg/g

	Control	Unknown: Serenoa repens Fruit				
Lab	SRM 3251 E	A1	B1	C1	Mean	s_{total}
C101	0.56	0.08	0.09	0.08	0.08	0.006
C107	0.41	0.08	0.08	0.07	0.07	0.001
C109	0.53	0.06	0.06	0.07	0.07	0.002
C110	0.56	0.11	0.1	0.1	0.10	0.009
C114	0.44	0.07	0.07	0.07	0.07	0.002
C118	0.57	0.09	0.09	0.09	0.09	0.00
N	6				6	
Mean	0.51				0.08	0.00
Median	0.54				0.078	
MADe	0.03				0.016	0.033
%RSD	6.3				21.0	
NIST	0.533				0.1175	
$\pm U_{95}$	0.031				0.0025	



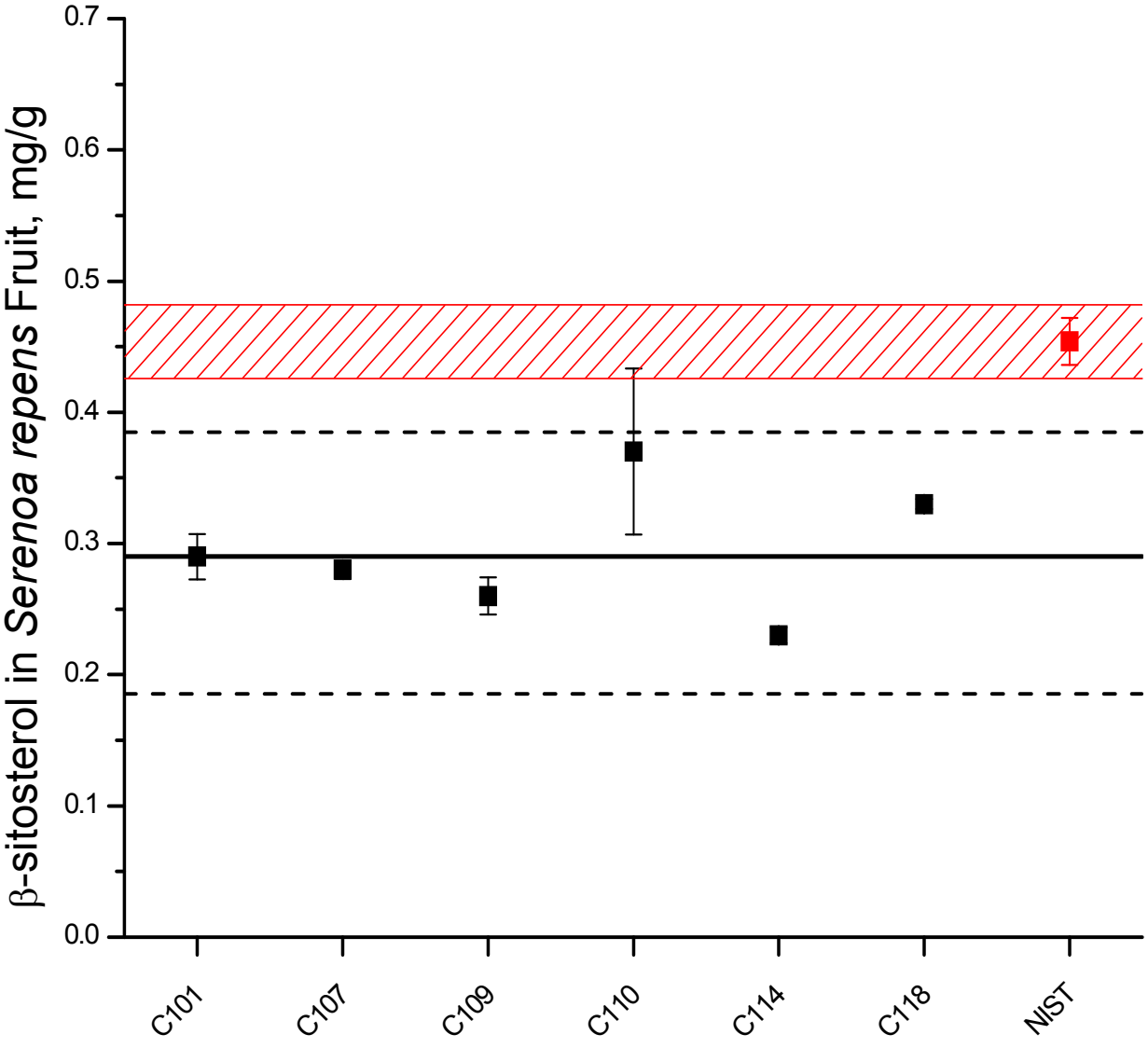
Precision bars span $\pm 2 \times s_{total}$ about Lab mean values.
NIST box encloses $\pm U_{95}$ region around NIST values.
Consensus box encloses ±2×MADe around consensus medians.
Plot encloses ±75 % around consensus medians.

[Return to Introduction]

β-SITOSTEROL IN *SERENOA REPENS* FRUIT

A discussion of the measurement of phytosterols in *Serenoa repens* extract and *Serenoa repens* fruit is included with the campesterol in solution graphs.

β-sitosterol in *Serenoa repens* Fruit View 1



Return to Introduction

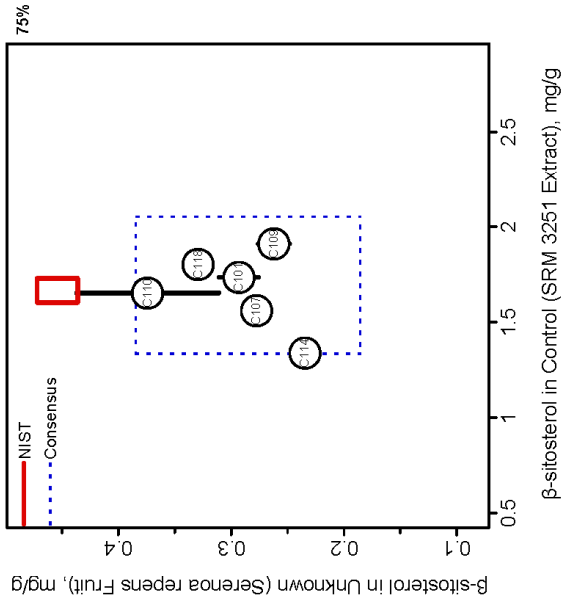
β-sitosterol in *Serenoa repens* Fruit View 2

Dietary Supplements Quality Assurance Program
May 2008 Study - Round B

β-sitosterol, mg/g

	Control	Unknown: Serenoa repens Fruit				
Lab	SRM 3251 E	A1	B1	C1	Mean	s_{total}
C101	1.73	0.29	0.3	0.29	0.29	0.01
C107	1.56	0.28	0.27	0.28	0.28	0.00
C109	1.91	0.26	0.26	0.27	0.26	0.01
C110	1.65	0.41	0.34	0.37	0.37	0.03
C114	1.34	0.24	0.23	0.24	0.23	0.00
C118	1.8	0.33	0.33	0.33	0.33	0.00

N	6	6	
Mean	1.67	0.29	0.01
Median	1.69	0.29	
MADe	0.18	0.05	0.100
%RSD	10.6	17.5	
NIST	1.666	0.454	
$\pm U_{95}$	0.064	0.018	



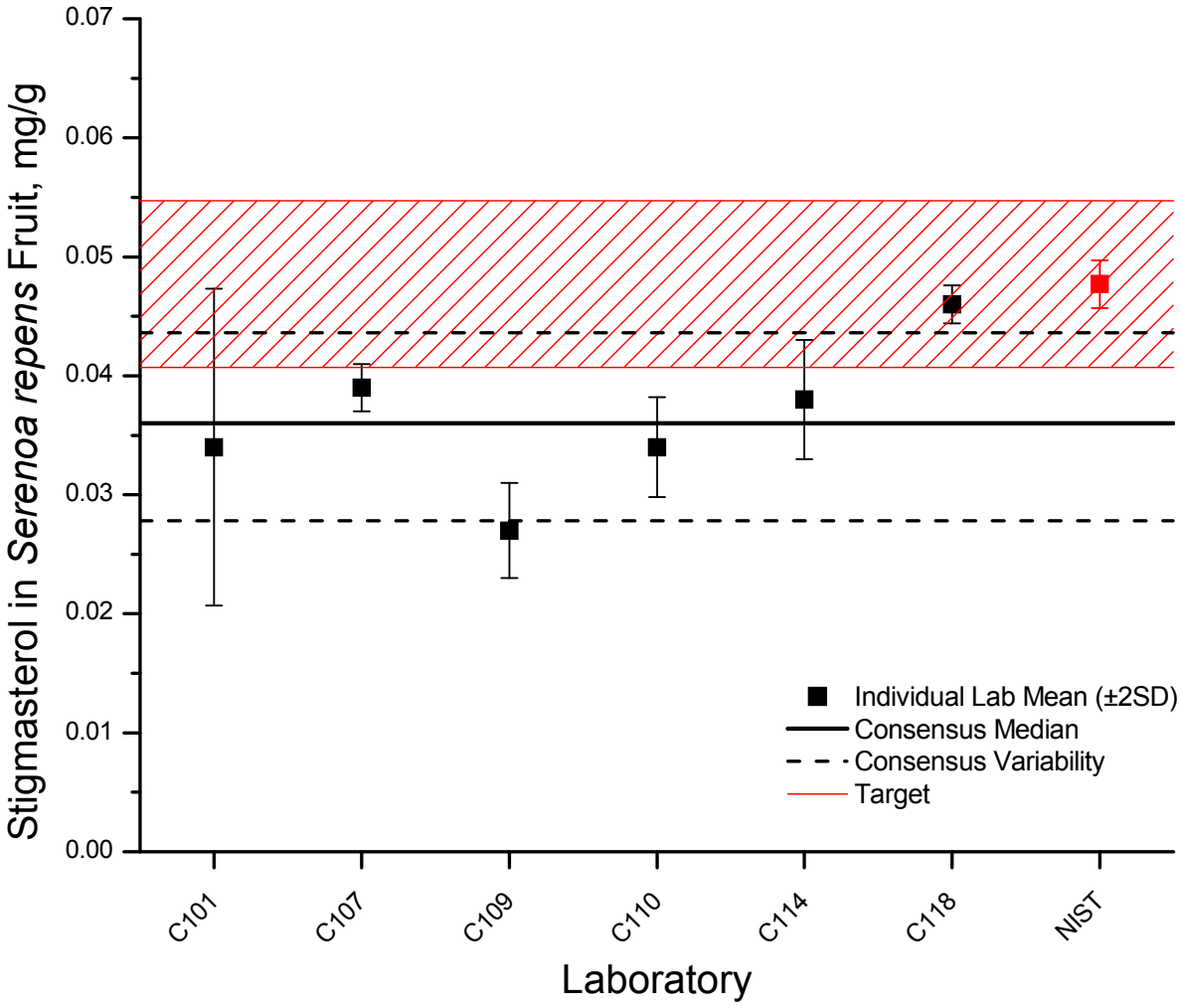
Precision bars span $\pm 2 \times s_{total}$ about Lab mean values.
NIST box encloses $\pm U_{95}$ region around NIST values.
Consensus box encloses ±2×MADe around consensus medians.
Plot encloses ±75 % around consensus medians.

Return to Introduction

STIGMASTEROL IN *SERENOA REPENS* FRUIT

A discussion of the measurement of phytosterols in *Serenoa repens* extract and *Serenoa repens* fruit is included with the campesterol in solution graphs.

Stigmasterol in *Serenoa repens* Fruit View 1



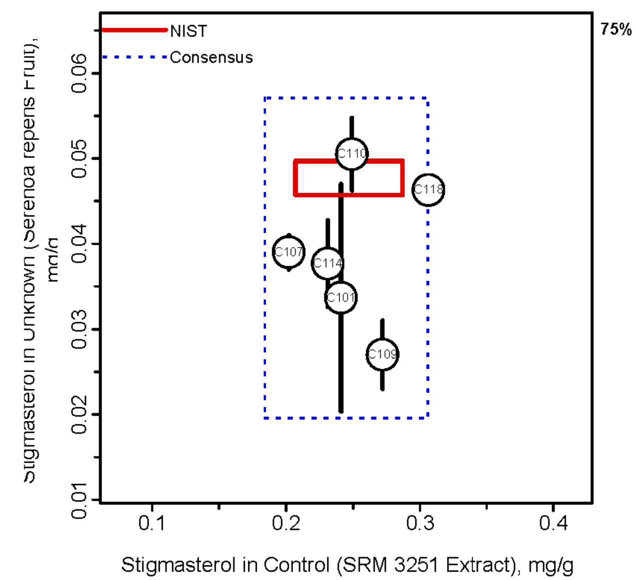
Return to Introduction

Stigmasterol in *Serenoa repens* Fruit View 2

Dietary Supplements Quality Assurance Program
May 2008 Study - Round B

Stigmasterol, mg/g

Lab	Control SRM 3251 E	Unknown: Serenoa repens Fruit A1	B1	C1	Mean	s_{total}
C101	0.24	0.03	0.04	0.04	0.034	0.01
C107	0.2	0.04	0.04	0.04	0.039	0.00
C109	0.27	0.03	0.03	0.03	0.027	0.00
C110	0.25	0.05	0.05		0.051	0.00
C114	0.23	0.04	0.04	0.04	0.038	0.00
C118	0.31	0.05	0.05	0.05	0.046	0.00
N	6				6	
Mean	0.25				0.04	0.00
Median	0.25				0.038	
MADe	0.03				0.01	0.019
%RSD	12.4				24.4	
NIST	0.247				0.0477	
$\pm U_{95}$	0.040				0.0020	



Precision bars span $\pm 2 \times s_{total}$ about Lab mean values.
NIST box encloses $\pm U_{95}$ region around NIST values.
Consensus box encloses ±2×MADe around consensus medians.
Plot encloses ±75 % around consensus medians.

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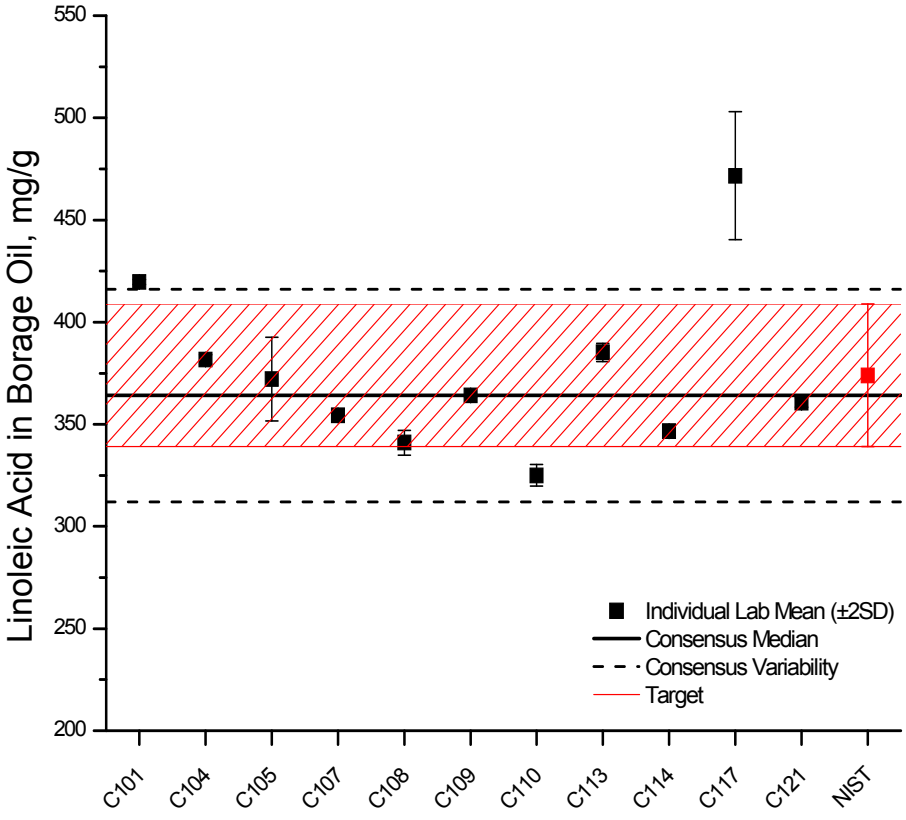
Fatty Acids

- View 1 indicates that in general the results for linoleic acid and γ-linoleic acid agree well with each other and the target
- View 1 indicates that the participants agree well on α-linoleic acid and are significantly different than the target values-this is under further investigation (we suspect that there is a stability issue).
- View 2 shows a correlation between results for the control and the unknown sample-in this case this cannot be definitively diagnosed as calibration issues; it could also be due to the derivatization of the sample. Would you like to investigate this further?

Recommendation:

- Measure Omega-3 and Omega-6 fatty acids in oils with more dissimilar ratios than the pair used in exercise C
- Perhaps include a solution to determine if the correlation between the control and unknown sample results are due to the sample preparation or calibration.

Linoleic Acid in Borage Oil View 1



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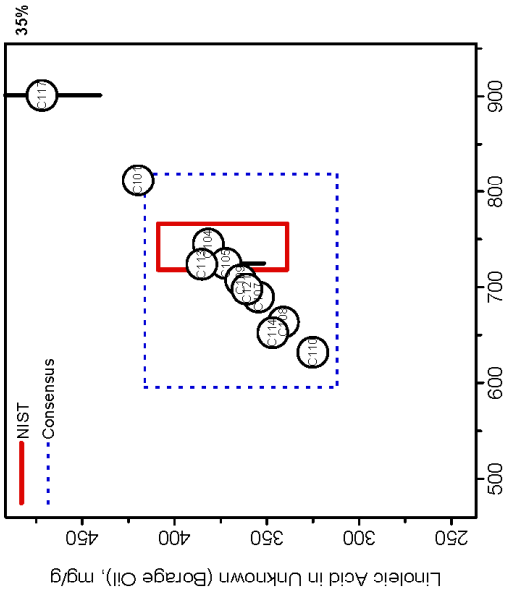
Linoleic Acid in Borage Oil View 2

Dietary Supplements Quality Assurance Program
May 2008 Study - Round B

Linoleic Acid, mg/g

Lab	Control SRM 3274 E	Unknown: Borage Oil A1	B1	C1	Mean	s_{total}
C101	812.	420.	419.	420.	419.7	0.6
C104	745.	383.	381.	381.	381.7	1.2
C105	724.73	360.9	374.5	381.	372.1	10.2
C107	689.6	355.3	355.3	352.7	354.4	1.5
C108	663.5	344.	341.	338.	341.	3.
C109	707.3	364.3	364.3	363.7	364.1	0.3
C110	632.	328.	324.	323.	325.	2.6
C113	724.	382.6	386.5	386.5	385.2	2.3
C114	652.5	345.4	348.2	346.4	346.7	1.4
C117	901.	486.	474.	455.	471.7	15.6
C121	698.	360.	360.	#####	360.67	1.15
N	11				11	
Mean	722.69				374.7	5.86
Median	707.30				364.1	
MADe	55.89				26.0	52.089
%RSD	7.9				7.2	
NIST	742.0				374.0	
$\pm U_{95}$	24.0				35.00	



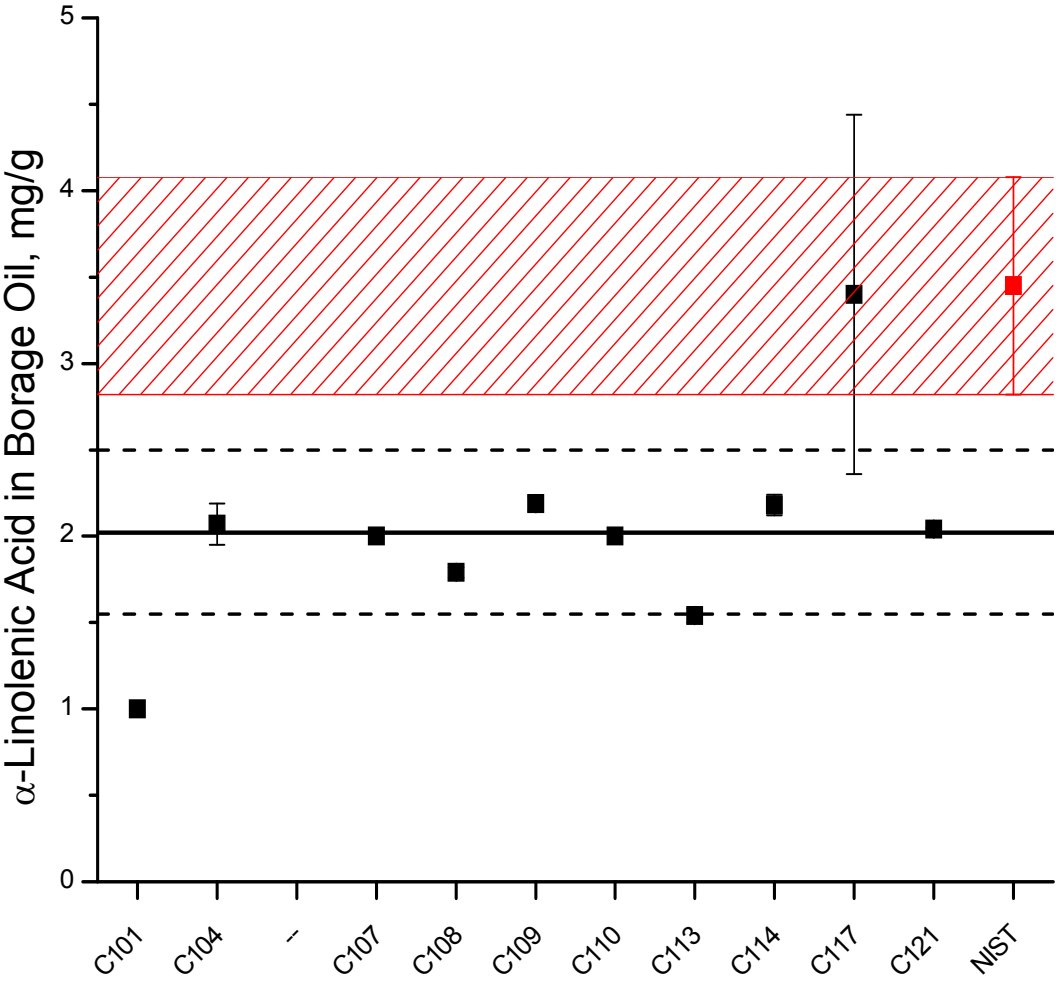
Precision bars span $\pm 2 \times s_{total}$ about Lab mean values.
NIST box encloses $\pm U_{95}$ region around NIST values.
Consensus box encloses ±2×MADe around consensus medians.
Plot encloses ±35 % around consensus medians.

Return to Introduction

α-LINOLENIC ACID

A discussion of the measurement of omega-3 and omega-6 fatty acids in botanical oils is included with the graphs of linoleic acid.

α-Linolenic Acid in Borage Oil View 1



Return to Introduction

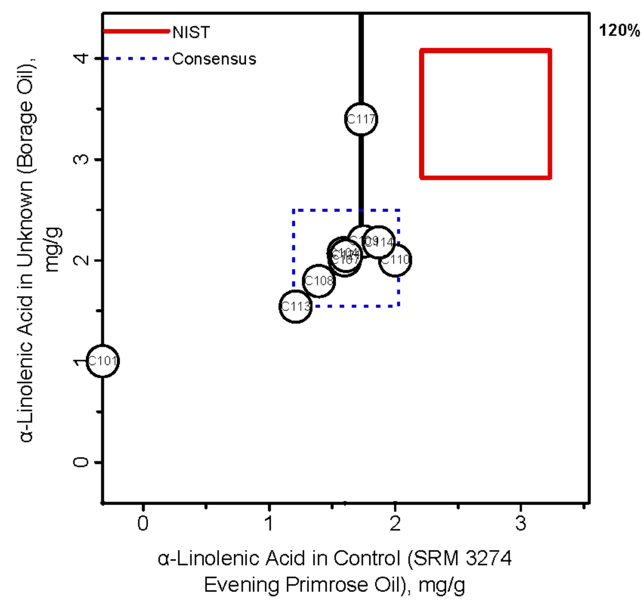
α-Linolenic Acid in Borage Oil View 2

Dietary Supplements Quality Assurance Program

May 2008 Study - Round B

α-Linolenic Acid

Lab	Control SRM 3274 E	Unknown: Borage Oil A1	B1	C1	Mean	s_{total}
C101		1.	1.	1.	1.00	
C104	1.59	2.14	2.02	2.05	2.07	0.062
C107	1.6	2.	2.	2.	2.00	
C108	1.4	1.81	1.78	1.79	1.79	0.015
C109	1.75	2.21	2.18	2.17	2.19	0.021
C110	2.	2.	2.	2.	2.00	
C113	1.21	1.52	1.54	1.56	1.54	0.020
C114	1.87	2.17	2.21	2.15	2.18	0.031
C117	1.73	3.77	3.62	2.8	3.40	0.522
C121	1.61	2.04	2.05	2.04	2.04	0.006
N	9				10	
Mean	1.64				2.02	0.17
Median	1.61				2.02	
MADe	0.21				0.24	0.474
%RSD	12.9				11.7	
NIST	2.7				3.45	
$\pm U_{95}$	0.5				0.63	



Precision bars span $\pm 2 \times s_{total}$ about Lab mean values.

NIST box encloses $\pm U_{95}$ region around NIST values.

Consensus box encloses ±2×MADe around consensus medians.

Plot encloses ±120 % around consensus medians.

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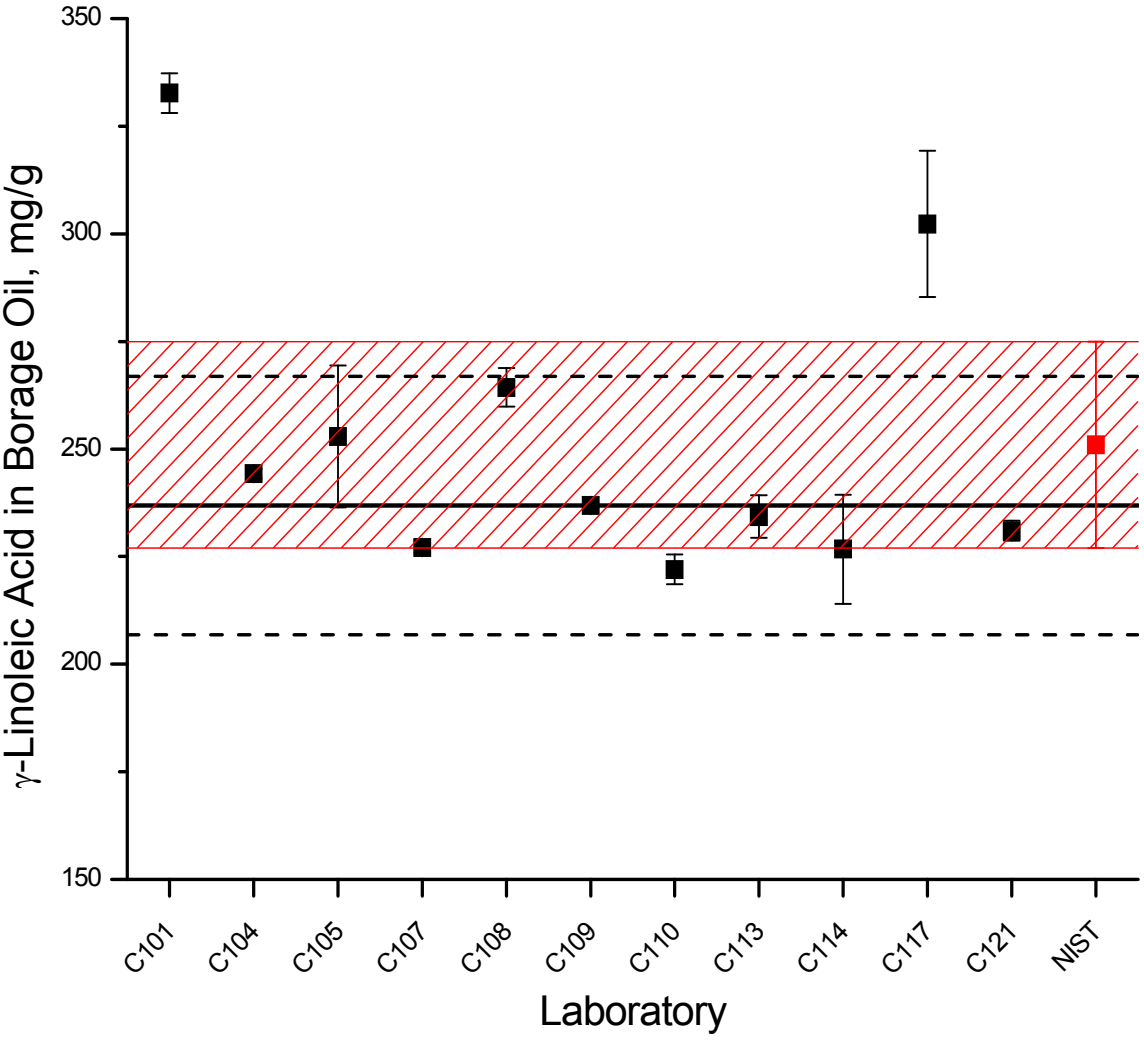
γ-LINOLENIC ACID

A discussion of the measurement of omega-3 and omega-6 fatty acids in botanical oils is included with the graphs of linoleic acid.

γ-Linolenic Acid in Borage Oil View 1



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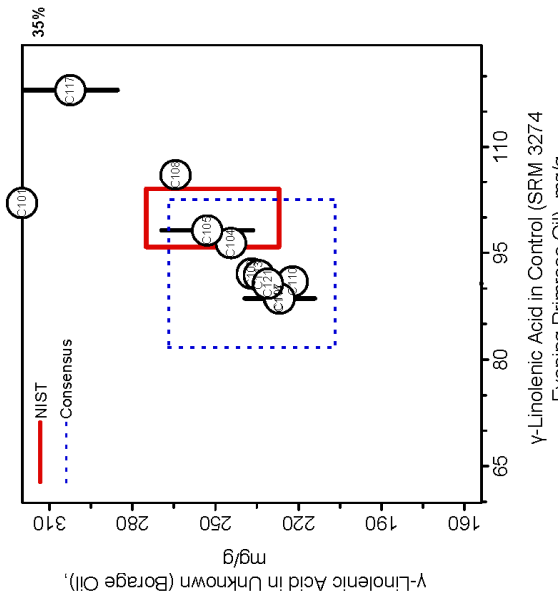
γ-Linolenic Acid in Borage Oil View 2

Dietary Supplements Quality Assurance Program
May 2008 Study - Round B

γ-Linolenic Acid, mg/g

Lab	Control SRM 3274 E	Unknown: Borage Oil A1	B1	C1	Mean	s_{total}
C101	102.	330.	334.	334.	332.7	2.31
C104	96.4	245.	244.	244.	244.3	0.58
C105	98.16	243.4	257.8	257.6	252.9	8.23
C107	88.8	227.6	227.3	226.5	227.1	0.57
C108	106.05	266.6	264.3	262.1	264.3	2.25
C109	92.1	237.	237.	236.6	236.9	0.23
C110	91.	224.	221.	221.	222.	1.73
C113	91.86	233.5	237.	232.4	234.3	2.45
C114	88.6	233.9	224.5	221.8	226.7	6.35
C117	118.	311.	302.	294.	302.3	8.50
C121	90.7	231.	231.	231.	231.	
N	11				11	
Mean	96.70				252.2	4.27
Median	92.10				236.9	
MADe	5.19				15.0	30.047
%RSD	5.6				6.3	
NIST	99.9				251.0	
$\pm U_{95}$	4.1				24.00	



Precision bars span $\pm 2 \times s_{total}$ about Lab mean values.
NIST box encloses $\pm U_{95}$ region around NIST values.
Consensus box encloses ±2×MADe around consensus medians.
Plot encloses ±35 % around consensus medians.

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