July 28, 2008

Dear Colleague:

Enclosed is your "Participation Certificate" for the Spring 2008 Dietary Supplement Quality Assurance Program (DSQAP) exercise. This certificate displays your results relative to the distribution of results reported by all participants. In addition, a one-page summary of individual, community, and NIST results has been included.

The individual data reports are listed for each analyte separately and are organized into three sections: 1) the individual laboratory/participant's results, 2) the overall community results, and 3) the NIST-assessed value and associated uncertainty. For your individual results, the mean and overall standard deviation are listed for each analyte for which a dataset was received. Since single measurements were made for each of three distributed unknown samples (Packets A, B, and C) during this Round B exercise, the overall standard deviation (s_{total}) is comprised of only the between-sample standard deviation ($s_{between}$) component. A measure of how comparable the individual laboratory/participant's result is to the overall community's performance for each analyte is represented as a Z-score. This is determined as the difference between your individual result and the median of the community result divided by the MADe (a robust estimate of the standard deviation derived from the median absolute deviation (MAD)) of the community result. The significance of the Z-score is as followed: $|Z| < 2$ is considered within the consensus value of the community result, $2 < |Z| < 3$ is considered as marginally different from the consensus value of the community result, and $|Z| > 3$ is considered as significantly different from the consensus value of the community result.

With the exception of sterols, a sufficient number of laboratories reported results for meaningful statistical analysis (N>8). Although statistical analysis is limited by lower participation for the determination of sterols, this study appears to indicate that methods of analysis need improvement. Is this an exercise that should be repeated since it appears to be problematic? Are there other analytes from this exercise or the pilot that you would like to see repeated?

The results of this exercise and materials for future exercises will be discussed during an informal half-day workshop at the AOAC meeting in Dallas. The workshop will be from 9-12 on Tuesday, September 23, 2008 in Cumberland J. It will be followed by a Technical Division on Reference Materials sponsored symposium, "Proficiency Testing and Reference Materials: Interrelations and Opportunities". If you would like to attend, you must register for the AOAC meeting,

http://www.aoac.org/meetings1/122nd_annual_mtg/main.htm. In addition, please e-mail DSQAP@nist.gov by September 2, 2008 if you plan to attend the workshop.

We appreciate your participation in this exercise. If you have any questions regarding the certificate or summary sheet please e-mail them to DSQAP@nist.gov. We would like to hear feedback on your needs for this program, please let us know what analytes/matricies are most useful.

We hope to see you in September.

Sincerely,

Catherine Rimmer, Ph.D.
Research Chemist
DSQAP Coordinator
(organic analytes)

Laura Wood
Biologist
DSQAP Coordinator
(inorganic analytes)

Katrice Lippa, Ph.D.
Research Chemist
DSQAP Data Analyst

National Institute of Standards and Technology
Analytical Chemistry Division

		1. Your Results, Lab Code: NIST						2. Community Results					3. NIST	
Analyte	Units	Mean	s_{within}	$s_{between}$	s_{total}	Z	sig	N	Median	MADe	Min	Max	Value	U_{95}
Arsenic	mg/kg	0.265	correction	1.000		0.2		13	0.257	0.035	0.201	0.334	0.265	0.032
Synephrine	mg/g	9.10	correction	1.00		1.8		8	8.46	0.36	4.77	8.78	9.10	0.30
Campesterol	mg/g	0.118	correction	1.000		6.4	**	5	0.084	0.005	0.080	0.138	0.118	0.005
Bsitosterol	mg/g	0.454	correction	1.000		59.4	**	5	0.293	0.003	0.249	0.462	0.454	0.036
Stigmasterol	mg/g	0.048	correction	1.000		0.8		5	0.044	0.004	0.041	0.094	0.048	0.004
Quercetin	mg/g	7.56	correction	1.00		-0.6		14	8.02	0.71	7.06	20.67	7.56	0.80
Kaempferol	mg/g	7.19	correction	1.00		0.8		14	6.79	0.52	6.29	23.47	7.19	1.40
Isorhamnetin	mg/g	1.90	correction	1.00		0.8		13	1.77	0.17	0.15	3.70	1.90	0.44
VitaminB1	mg/g	1.05	correction	1.00		0.2		21	1.01	0.25	0.63	1.80	1.05	0.20
VitaminB2	mg/g	1.27	correction	1.00		-0.1		21	1.29	0.20	0.91	2.15	1.27	0.28

Mean — Average of all your reported values

s_{within} — Pooled within-sample standard deviation (repeatability)

$s_{between}$ — Between-sample standard deviation, adjusted for s_{within}

s_{total} — Overall standard deviation (combines s_{within} and $s_{between}$)

Z — Z-score: (Mean - Median)/MADe

sig — Approximate significance of Z:

(blank) = within consensus value

* = marginally different from consensus value

** = significantly different from consensus value

N — Number of quantitative values reported

Median — Median of the reported values

MADe — Median Absolute Deviation from the Median / 0.674

Min, Max — Minimum and maximum reported values

Value — NIST-assessed value

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

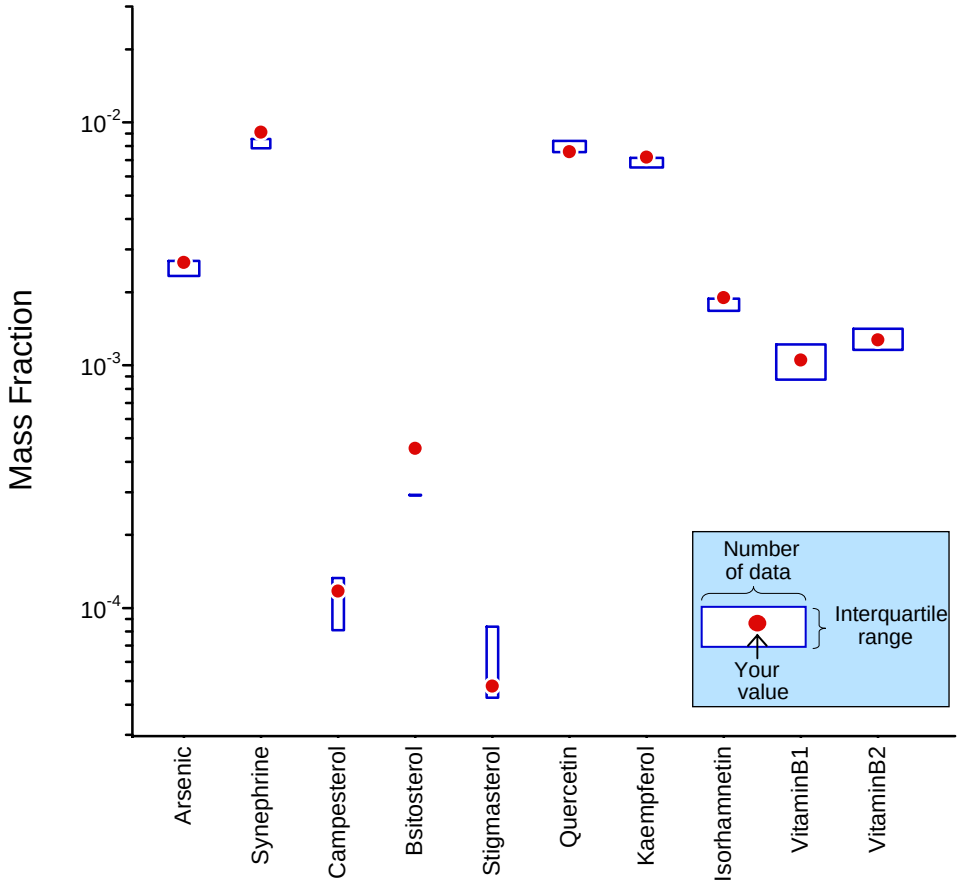
National Institute of Standards and Technology

Dietary Supplements Quality Assurance Program

NIST

Analytical Chemistry Division
National Institute of Standards and Technology

Participated in the May 2008 Interlaboratory Comparability Study



DSQAP Coordinator *DSQAP Coordinator* *DSQAP Data Analyst*

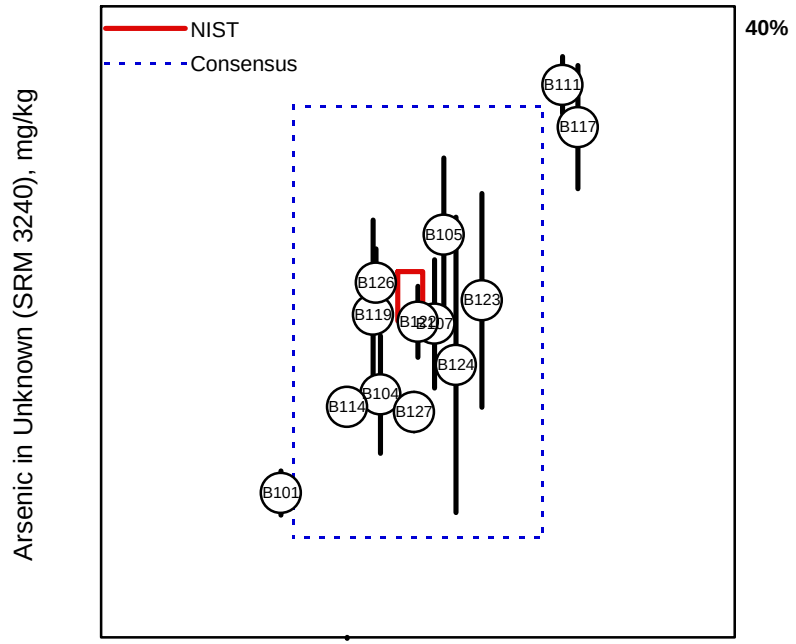
Chief, Analytical Chemistry Division

Dietary Supplements Quality Assurance Program

May 2008 Study - Round B

Arsenic, mg/kg

	Control	Unknown: SRM 3240				
Lab	SRM 3242	A1	B1	C1	Mean	s_{total}
B101	0.86	0.21	0.2	0.2	0.20	0.00
B104	0.99	0.23	0.23	0.24	0.23	0.01
B105	1.07	0.3	0.27	0.29	0.29	0.01
B107	1.06	0.24	0.26	0.26	0.26	0.01
B111	1.23	0.33	0.33	0.34	0.33	0.00
B114	0.95	0.23	0.23	0.23	0.23	
B117	1.25	0.31	0.33	0.32	0.32	0.01
B119	0.98	0.26	0.27	0.24	0.26	0.02
B122	1.04	0.25	0.26	0.26	0.26	0.01
B123	1.12	0.28	0.27	0.24	0.26	0.02
B124	1.09	0.24	0.22	0.27	0.24	0.02
B126	0.99	0.28	0.26	0.27	0.27	0.01
B127	1.04	0.22	0.23	0.23	0.23	0.00
N	13				13	
Mean	1.05				0.26	0.01
Median	1.04				0.26	
MADe	0.08				0.04	
%RSD	7.8				13.7	
NIST	1.0				0.3	
$\pm U_{95}$	0.0				0.0	



Precision bars span ±2×s_{total} about Lab mean values.

NIST box encloses ±U_{95} region around NIST values.

Consensus box encloses ±2×MADe around consensus medians.

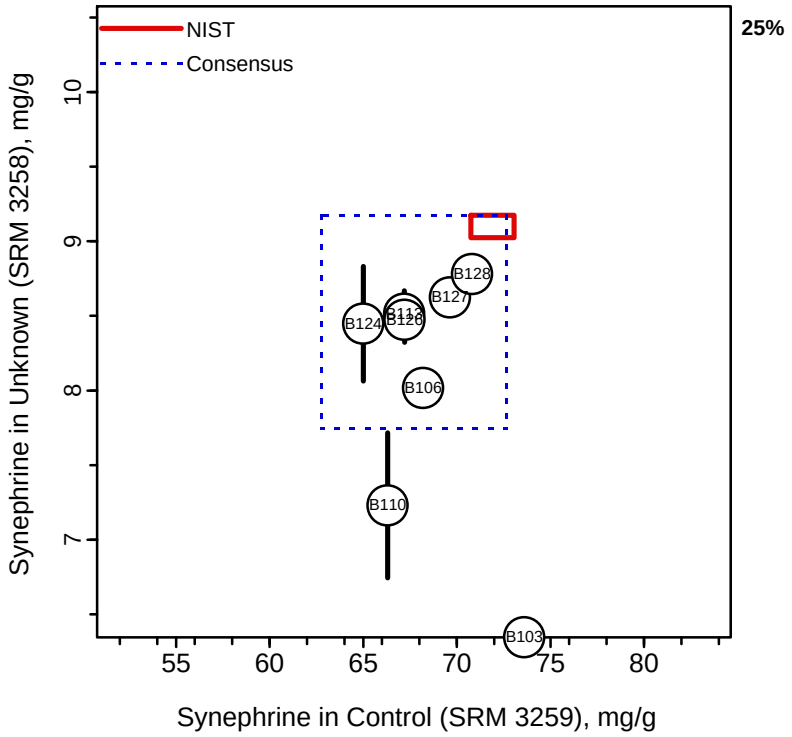
Plot encloses ±40 % around consensus medians.

Dietary Supplements Quality Assurance Program

May 2008 Study - Round B

Synephrine, mg/g

	Control	Unknown: SRM 3258				
Lab	SRM 3259	A1	B1	C1	Mean	s_{total}
B103	73.6	4.7	5.	4.6	4.77	0.21
B106	68.2	7.99	8.01	8.05	8.02	0.03
B110	66.3	7.51	7.1	7.08	7.23	0.24
B113	67.2	8.45	8.49	8.6	8.51	0.08
B124	65.	8.24	8.62	8.48	8.45	0.19
B126	67.21	8.43	8.43	8.56	8.47	0.08
B127	69.64	8.62	8.67	8.58	8.62	0.05
B128	70.82	8.75	8.75	8.85	8.78	0.06
N	8				8	
Mean	68.50				7.86	0.14
Median	67.71				8.46	
MADe	2.48				0.36	
%RSD	3.7				4.2	
NIST	71.9				9.1	
$\pm U_{95}$	1.2				0.1	



Precision bars span ±2×s_{total} about Lab mean values.

NIST box encloses ±U_{95} region around NIST values.

Consensus box encloses ±2×MADe around consensus medians.

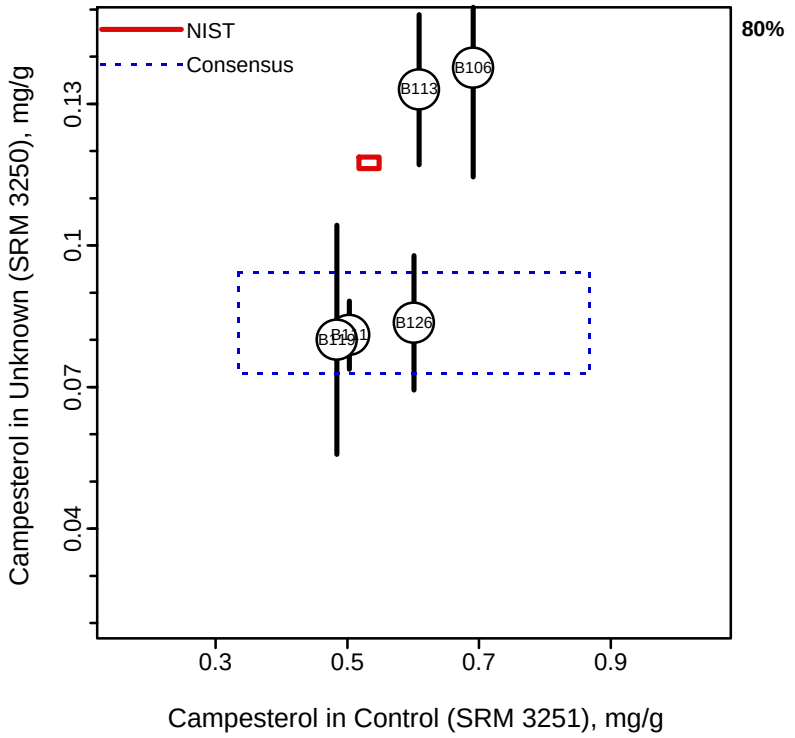
Plot encloses ±25 % around consensus medians.

Dietary Supplements Quality Assurance Program

May 2008 Study - Round B

Campesterol, mg/g

	Control	Unknown: SRM 3250				
Lab	SRM 3251	A1	B1	C1	Mean	s_{total}
B106	0.69	0.14	0.13	0.15	0.14	0.012
B111	0.5	0.08	0.08	0.08	0.08	0.004
B113	0.61	0.14	0.14	0.12	0.13	0.008
B119	0.48	0.09	0.07	0.08	0.08	0.012
B126	0.6	0.08	0.09	0.09	0.08	0.007
N	5				5	
Mean	0.58				0.10	0.01
Median	0.60				0.08	
MADe	0.13				0.01	
%RSD	22.1				6.4	
NIST	0.53				0.12	
$\pm U_{95}$	0.02				0.00	



Precision bars span ±2×s_{total} about Lab mean values.

NIST box encloses ±U_{95} region around NIST values.

Consensus box encloses ±2×MADe around consensus medians.

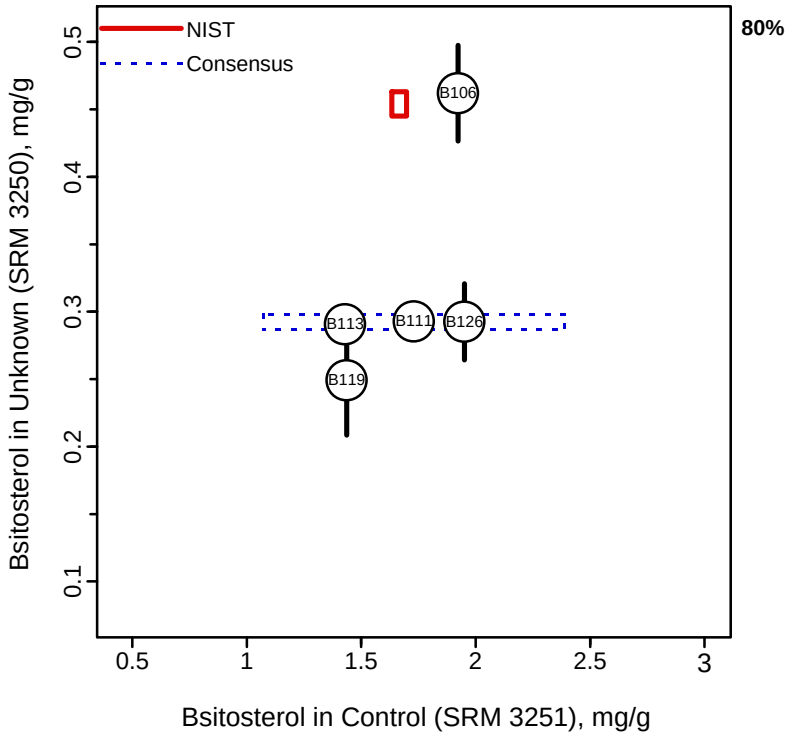
Plot encloses ±80 % around consensus medians.

Dietary Supplements Quality Assurance Program

May 2008 Study - Round B

Bsitosterol, mg/g

	Control	Unknown: SRM 3250				
Lab	SRM 3251	A1	B1	C1	Mean	s_{total}
B106	1.92	0.48	0.44	0.47	0.46	0.02
B111	1.73	0.3	0.29	0.29	0.29	0.00
B113	1.43	0.29	0.29	0.29	0.29	0.00
B119	1.44	0.27	0.23	0.25	0.25	0.02
B126	1.95	0.28	0.3	0.3	0.29	0.01
N	5				5	
Mean	1.69				0.32	0.01
Median	1.73				0.29	
MADe	0.33				0.00	
%RSD	19.0				0.9	
NIST	1.67				0.45	
$\pm U_{95}$	0.03				0.01	



Precision bars span ±2×s_{total} about Lab mean values.

NIST box encloses ±U_{95} region around NIST values.

Consensus box encloses ±2×MADe around consensus medians.

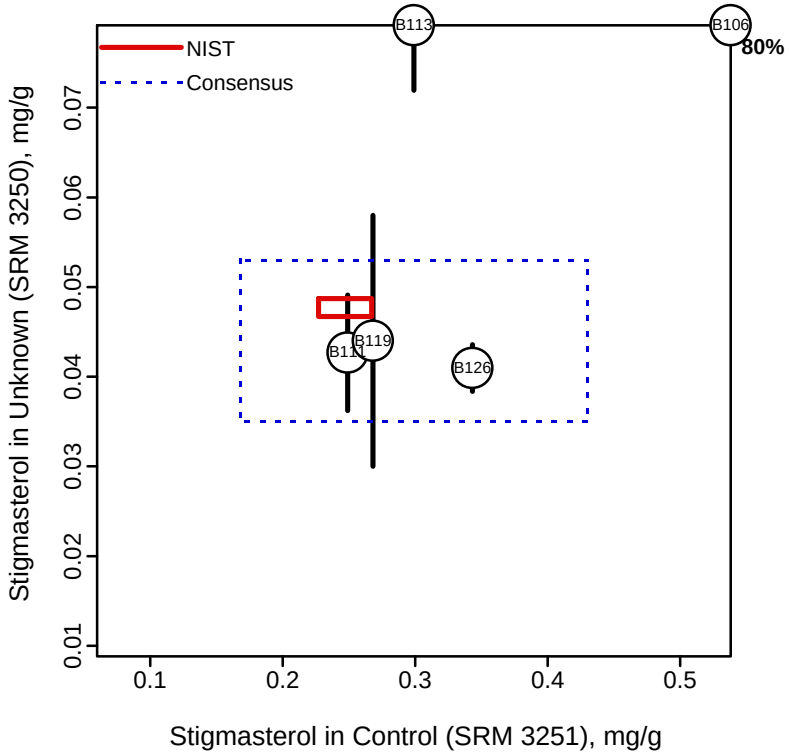
Plot encloses ±80 % around consensus medians.

Dietary Supplements Quality Assurance Program

May 2008 Study - Round B

Stigmasterol, mg/g

	Control	Unknown: SRM 3250				
Lab	SRM 3251	A1	B1	C1	Mean	s_{total}
B106	0.57	0.1	0.09	0.09	0.09	0.00
B111	0.25	0.04	0.04	0.05	0.04	0.00
B113	0.3	0.09	0.09	0.08	0.08	0.01
B119	0.27	0.05	0.04	0.04	0.04	0.01
B126	0.34	0.04	0.04	0.04	0.04	0.00
N	5				5	
Mean	0.35				0.06	0.00
Median	0.30				0.04	
MADe	0.07				0.00	
%RSD	21.9				10.2	
NIST	0.25				0.048	
$\pm U_{95}$	0.02				0.001	



Precision bars span ±2×s_{total} about Lab mean values.

NIST box encloses ±U_{95} region around NIST values.

Consensus box encloses ±2×MADe around consensus medians.

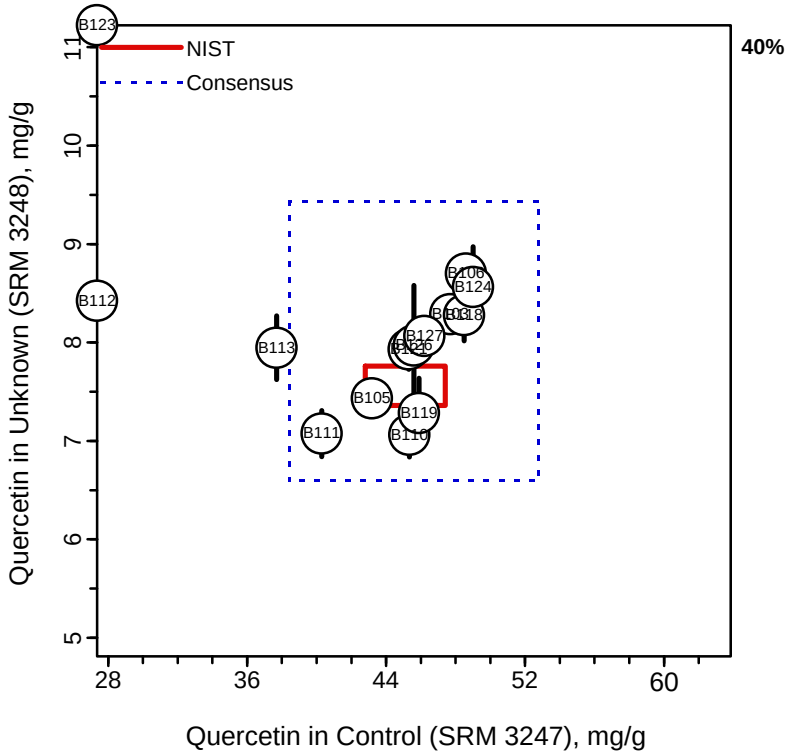
Plot encloses ±80 % around consensus medians.

Dietary Supplements Quality Assurance Program

May 2008 Study - Round B

Quercetin, mg/g

	Control	Unknown: SRM 3248				
Lab	SRM 3247	A1	B1	C1	Mean	s_{total}
B103	47.7	8.29	8.3	8.27	8.29	0.02
B105	43.19	7.45	7.42	7.42	7.43	0.02
B106	48.6	8.67	8.77	8.66	8.70	0.06
B110	45.35	7.04	7.18	6.96	7.06	0.11
B111	40.3	7.2	6.97	7.05	7.07	0.12
B112	1.56	8.36	8.46	8.45	8.42	0.05
B113	37.7	8.05	7.76	8.03	7.95	0.16
B118	48.49	8.14	8.3	8.39	8.28	0.13
B119	45.9	7.37	7.4	7.08	7.28	0.18
B121	45.3	7.91	8.04	7.84	7.93	0.10
B123		20.6	20.	21.4	20.67	0.70
B124	49.	8.34	8.74	8.61	8.56	0.20
B126	45.6	8.3	7.9	7.7	7.97	0.31
B127	46.2	8.1	8.1	8.	8.07	0.06
N	13				14	
Mean	41.91				8.83	0.23
Median	45.60				8.02	
MADe	3.57				0.71	
%RSD	7.8				8.8	
NIST	45.10				7.56	
$\pm U_{95}$	2.30				0.20	



Precision bars span ±2×s_{total} about Lab mean values.

NIST box encloses ±U_{95} region around NIST values.

Consensus box encloses ±2×MADe around consensus medians.

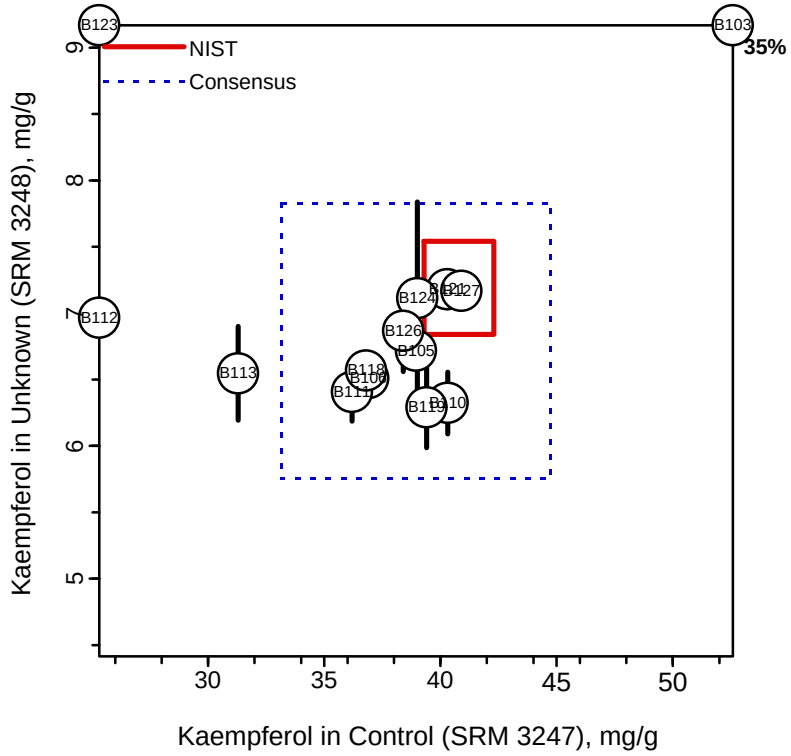
Plot encloses ±40 % around consensus medians.

Dietary Supplements Quality Assurance Program

May 2008 Study - Round B

Kaempferol, mg/g

	Control	Unknown: SRM 3248				
Lab	SRM 3247	A1	B1	C1	Mean	s_{total}
B103	57.7	10.	10.	10.1	10.03	0.06
B105	38.95	6.69	6.73	6.73	6.72	0.02
B106	36.9	6.53	6.51	6.49	6.51	0.02
B110	40.31	6.4	6.38	6.19	6.32	0.12
B111	36.2	6.48	6.28	6.46	6.41	0.11
B112	0.85	6.96	7.01	6.93	6.97	0.04
B113	31.3	6.71	6.36	6.57	6.55	0.18
B118	36.8	6.55	6.53	6.63	6.57	0.05
B119	39.4	6.34	6.41	6.12	6.29	0.15
B121	40.3	7.11	7.25	7.18	7.18	0.07
B123		23.5	22.9	24.	23.47	0.55
B124	39.	7.53	6.93	6.88	7.11	0.36
B126	38.4	6.9	7.	6.7	6.87	0.15
B127	40.9	7.2	7.2	7.1	7.17	0.06
N	13				14	
Mean	36.69				8.15	0.20
Median	38.95				6.79	
MADe	2.89				0.52	
%RSD	7.4				7.6	
NIST	40.80				7.19	
$\pm U_{95}$	1.50				0.35	



Precision bars span ±2×s_{total} about Lab mean values.

NIST box encloses ±U_{95} region around NIST values.

Consensus box encloses ±2×MADe around consensus medians.

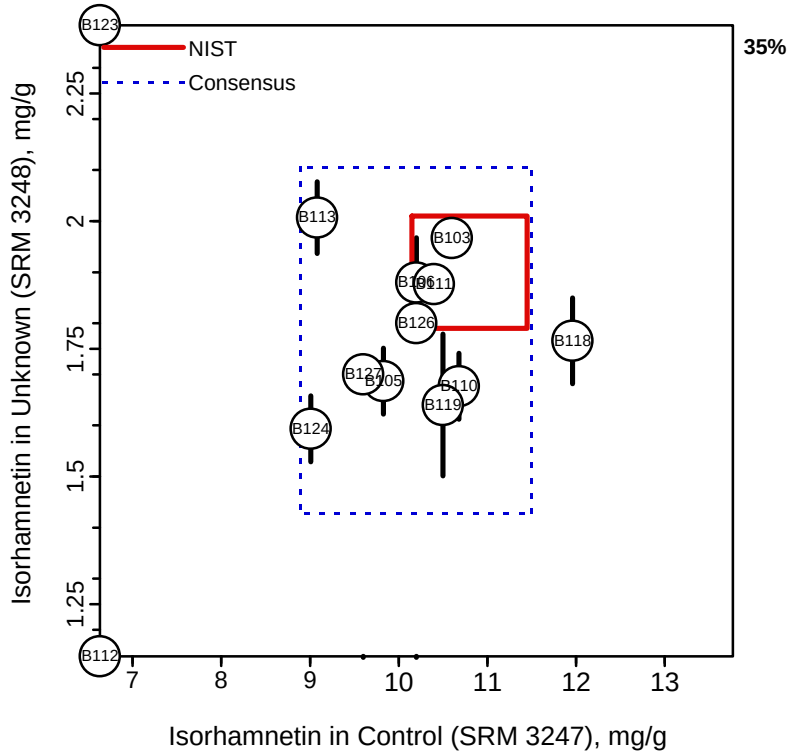
Plot encloses ±35 % around consensus medians.

Dietary Supplements Quality Assurance Program

May 2008 Study - Round B

Isorhamnetin, mg/g

	Control	Unknown: SRM 3248				
Lab	SRM 3247	A1	B1	C1	Mean	s_{total}
B103	10.6	1.95	1.97	1.98	1.97	0.02
B105	9.83	1.65	1.71	1.7	1.69	0.03
B106	10.2	1.83	1.9	1.91	1.88	0.04
B110	10.68	1.7	1.69	1.64	1.68	0.03
B111	10.4	1.89	1.86	1.88	1.88	0.02
B112	0.02	0.16	0.14	0.15	0.15	0.01
B113	9.08	2.04	1.97	2.01	2.01	0.04
B118	11.96	1.74	1.74	1.81	1.77	0.04
B119	10.5	1.6	1.72	1.6	1.64	0.07
B123		3.7	3.6	3.8	3.70	0.10
B124	9.01	1.63	1.57	1.58	1.59	0.03
B126	10.2	1.8	1.8	1.8	1.80	
B127	9.6	1.7	1.7	1.7	1.70	
N	12				13	
Mean	9.34				1.80	0.04
Median	10.20				1.77	
MADe	0.65				0.17	
%RSD	6.4				9.6	
NIST	10.80				1.90	
$\pm U_{95}$	0.65				0.11	



Precision bars span ±2×s_{total} about Lab mean values.

NIST box encloses ±U_{95} region around NIST values.

Consensus box encloses ±2×MADe around consensus medians.

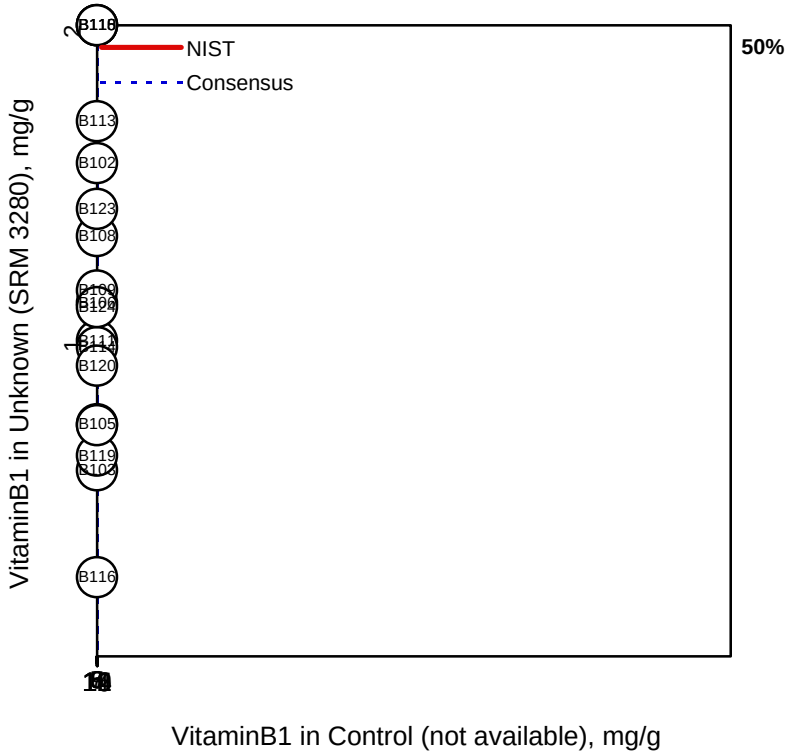
Plot encloses ±35 % around consensus medians.

Dietary Supplements Quality Assurance Program

May 2008 Study - Round B

VitaminB1, mg/g

	Control	Unknown: SRM 3280				
Lab	not available	A1	B1	C1	Mean	s_{total}
B102		1.31	1.28	1.28	1.29	0.02
B103		0.8	0.8	0.8	0.80	0.00
B105		0.82	0.9	0.9	0.87	0.05
B106		1.08	1.05	1.07	1.07	0.02
B108		1.16	1.17	1.19	1.17	0.02
B109		1.09	1.07	1.1	1.09	0.01
B110		1.81	1.86	1.73	1.80	0.07
B111		1.05	0.99	0.98	1.01	0.04
B113		1.36	1.33	1.38	1.36	0.03
B114		1.02	0.99	0.98	1.00	0.02
B115		1.96	1.27	1.43	1.55	0.36
B116		0.65	0.57	0.67	0.63	0.06
B118		1.52	1.53	1.65	1.57	0.07
B119		0.84	0.82	0.82	0.82	0.01
B120		0.95	0.99	0.96	0.97	0.02
B123		1.31	1.19	1.16	1.22	0.08
B124		1.05	1.05	1.08	1.06	0.02
B125		0.96	0.95	0.95	0.95	0.00
B126		0.87	0.84	0.81	0.84	0.03
B127		1.	1.	1.01	1.00	0.01
B128		0.87	0.88	0.86	0.87	0.01
N					21	
Mean					1.09	0.09
Median					1.01	
MADe	1.00				0.25	
%RSD	1.0				24.5	
NIST					1.05	
$\pm U_{95}$					0.05	



Precision bars span ±2×s_{total} about Lab mean values.

NIST box encloses ±U_{95} region around NIST values.

Consensus box encloses ±2×MADe around consensus medians.

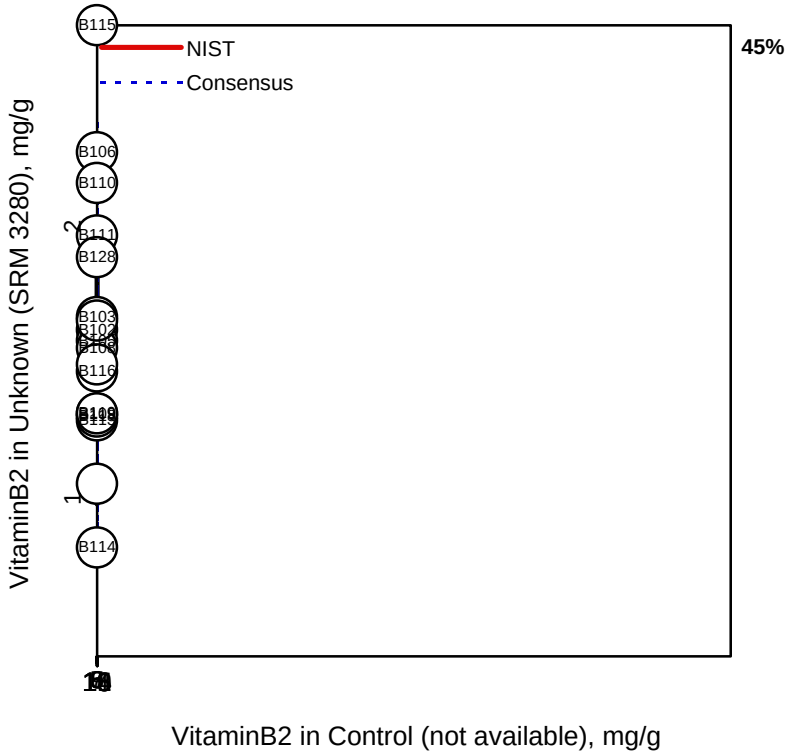
Plot encloses ±50 % around consensus medians.

Dietary Supplements Quality Assurance Program

May 2008 Study - Round B

VitaminB2, mg/g

	Control	Unknown: SRM 3280				
Lab	not available	A1	B1	C1	Mean	s_{total}
B102		1.3	1.32	1.31	1.31	0.01
B103		1.3	1.3	1.4	1.33	0.06
B105		1.3	1.28	1.29	1.29	0.01
B106		1.64	1.62	1.65	1.64	0.02
B108		1.27	1.27	1.29	1.28	0.01
B109		1.18	1.14	1.15	1.16	0.02
B110		1.52	1.64	1.58	1.58	0.06
B111		1.5	1.57	1.38	1.48	0.10
B113		1.13	1.11	1.19	1.14	0.04
B114		0.85	0.96	0.92	0.91	0.06
B115		2.47	2.05	1.94	2.15	0.28
B116		1.2	1.3	1.2	1.23	0.06
B118		1.14	1.16	1.16	1.15	0.01
B119		1.09	1.19	1.19	1.16	0.06
B120		1.01	1.02	1.	1.01	0.01
B123		1.43	1.45	1.36	1.42	0.05
B124		1.27	1.32	1.32	1.30	0.03
B125		1.45	1.44	1.44	1.44	0.01
B126		1.3	1.35	1.33	1.33	0.02
B127		1.24	1.24	1.26	1.25	0.01
B128		0.99	1.03	1.06	1.03	0.04
N					21	
Mean					1.31	0.07
Median					1.29	
MADe	1.00				0.20	
%RSD	1.0				15.4	
NIST					1.27	
$\pm U_{95}$					0.07	



Precision bars span ±2×s_{total} about Lab mean values.

NIST box encloses ±U_{95} region around NIST values.

Consensus box encloses ±2×MADe around consensus medians.

Plot encloses ±45 % around consensus medians.