Dietary Supplement Laboratory Quality Assurance Program
Exercise E
Final Data Report

October 13, 2010

Thank you for your participation in Exercise E of the Dietary Supplements Laboratory Quality Assurance Program. This exercise attracted a record signup of all exercises to date, and more importantly, had the highest percentage of data returned. The attached final report contains five sections: an overview of the data treatment and representation; graphs displaying the community performance; general recommendations from the exercise, your individual laboratory results, and certificates of participation.

Please check the data from your laboratory throughout both reports and make sure that it agrees with the data that you submitted. If you do not agree with the data in this report, please let us know so that we can correct it.

If you have any questions or suggestions, please let us know.

Best regards,

DSQAP Team

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INTRODUCTION

This report consists of several parts:

Overview of Data Treatment and Representation: a general description of the statistical treatment of the data and how to read the plots.

Data: a section that includes tables and graphical representations of the data for the analytes tested in this exercise, points to consider when examining the data and recommendations from Exercise E when appropriate.

- Nutritional Elements in Breakfast Cereal Overview
  - Iron in Cereals Containing Wheat, Table
  - Iron in Cereals Containing Rice, Table
  - Calcium Cereals Containing Wheat, Table
  - Calcium in Cereals Containing Rice, Table
  - Zinc in Cereals Containing Wheat, Table
  - Zinc in Cereals Containing Rice, Table
  - Nutritional Elements in Cereal A, Graphs

- Niacinamide in Fortified Breakfast Cereal
  - Data Table
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- Catechins in Green Tea Overview
  - Total Catechins
    - Data Table
    - Graphs
  - Catechin
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  - Epicatechin
    - Data Table
    - Graphs
  - Epicatechin Gallate
    - Data Table
    - Graphs
  - Epigallocatechin
    - Data Table
    - Graphs
  - Epigallocatechin Gallate
    - Data Table
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- Gallocatechin
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- Gallocatechin Gallate
  - Data Table
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- β-carotene in Multivitamin
  - total β-carotene
    - Data Table
    - Graphs
  - *trans*-β-carotene
    - Data Table
    - Graphs
  - 9-*cis*-β-carotene
    - Data Table
    - Graphs

- Aflatoxins in Peanuts
  - Total Aflatoxins
    - Data Table
    - Graphs
  - B_1
    - Data Table
    - Graphs
  - B_2
    - Data Table
    - Graphs
  - G_1
    - Data Table
    - Graphs
  - G_2
    - Data Table
    - Graphs

Recommendations and Question

Individual Laboratory Report

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OVERVIEW OF DATA TREATMENT AND REPRESENTATION

STATISTICS

Your individual data table and graphs contain information about your performance relative to that of the other participants in this exercise and relative to a target around the expected result.

DATA TABLES

The raw data for each analyte in each sample are provided in tabular form within each section. Please check your data for transcription errors.

GRAPHS

Two graphs are provided for each analyte in each sample, one which plots the results for the sample only (View 1) and one which plots the lab results for the sample against the lab results for the control (View 2). Both views include the consensus values and the target values (where appropriate).

View 1

In this view, 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 calculated as $2 \times$ MADe. The center of the region between the red lines represents the target zone (when available). The target value is the NIST certified value (or an approximation where appropriate) and is bounded by two times the pooled standard deviation ($2 \times s_{total,\ pooled}$) of the participants or the NIST uncertainty (U_{95}), whichever is larger, creating a target zone for "acceptable" performance. With this view, it is relatively easy to determine if a laboratory falls within the target zone and to compare where the target zone lies relative to the consensus zone. In most cases, the target zone falls within the consensus zone, which is the expected result. One program goal is to bring the consensus zone closer together and closer to the target value.

View 2

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

This view provides additional information to complement View 1. For example, if your values are low (or high) for both the control and sample, 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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INDIVIDUAL DATA TABLE

Section 1 of the data table contains your results, including your mean, standard deviation, and Z-score (when appropriate). Please check these and make sure that you agree with the mean and standard deviation reported in the table. Your individual lab code can be found in the upper left corner of each individual data table.

The significance of the Z-score is as follows:

- $|Z| < 2$ indicates that your result is considered to be within the community consensus value.
- $2 < |Z| < 3$ indicates that your result is considered to be marginally different from the community consensus value.
- $|Z| > 3$ indicates that your result is considered to be significantly different from the community consensus value.

A Z-score does not necessarily indicate whether or not your values are correct; instead, it is a reflection of how close your values are to the rest of the community.

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 and maximum values reported for the analyte.

Section 3 of the data table contains the NIST results. In most cases, the assigned value and the U_{95} 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 the U_{95} to encompass variability due to inhomogeneity within and between packages.

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NUTRITIONAL ELEMENTS IN BREAKFAST CEREALS

This study was different than the other studies in this exercise; in addition to providing laboratory comparability and proficiency information, it was also designed to show the effect of sample type and sample preparation on both within- and between-laboratory comparability of results. The results from the complete study will take longer to analyze, therefore only the comparability data are presented in this report. Participants will be notified of the extended results as soon as they are available.

Six cereal samples were sent with this study:

- Cereal A is SRM 3233 Fortified Breakfast Cereal, a ground, sieved wheat-based cereal.
- Cereal B is a ground wheat-based cereal.
- Cereal C is Cereal B, prior to grinding.
- Cereal D is a ground rice-based cereal.
- Cereal E is Cereal D prior to grinding.
- Cereal F is a hand crushed 1:1 mixture (by mass) of Cereals C and E.

Assuming that all of the samples are homogeneous, the results for the pairs of samples (B/C and D/E) should be identical and sample F is expected to be an approximate average of the two. The differences in variability between the ground and flake cereals will be examined to evaluate the sampling effects, and will be reported at a later date.

- Twenty-three laboratories enrolled in this exercise and received samples; 19 laboratories reported results (83 %).
- Since homogeneity was established only for sample A in this study, it is the only sample with target values. NIST is a blinded participant for the remaining samples in this study.
- Overall, the majority of laboratories are achieving relatively precise (< 3%) measurements that fall within the consensus range. The precision was better for Fe than for Ca and Zn.

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Iron in Breakfast Cereals Containing Wheat: DSQAP Exercise E

		Sample A: Ground Wheat Cereal SRM 3233					Sample B: Ground Wheat Cereal					Sample C: Flake Wheat Cereal				
	lab code	A1	A2	A3	A mean	A SD	B1	B2	B3	B Mean	B SD	C1	C2	C3	C Mean	C SD
Individual Results	E502	691	703	707	700	8	697	742	675	704	34	772	750	791	771	21
	E509	710	700	750	720	26	790	760	810	787	25	820	800	840	820	20
	E510	682	692	674	683	9	720	728	747	732	14	699	559	743	667	96
	E511	773	745	723	747	25	828	807	782	806	23	850	813	792	818	29
	E513	750	740	740	743	6	770	780	770	773	6	800	810	790	800	10
	E515	793	821	801	805	14	903	909	886	899	12	907	924	894	908	15
	E520	883	841	858	861	21	909	907	920	912	7	950	939	924	938	13
	E522	735	756	741	744	11	761	763	836	787	43	859	821	855	845	21
	E526	743	745	754	747	6	825	828	825	826	2	866	878	799	848	43
	E530	335	403	387	375	35	720	747	624	697	65	456	478	382	439	50
	E531	790	790	740	773	29	920	800	750	823	87	830	810	810	817	12
	E533	670	690	710	690	20	770	720	770	753	29	800	790	790	793	6
	E534	876	858	813	849	32	908	849	883	880	30	889	911	901	900	11
	E537	712	725	712	716	8	778	804	766	783	19	756	730	752	746	14
	E538	425	567	438	477	79	468	674	526	556	107	505	687	490	561	110
	E539	774	777	795	782	11	870	854	841	855	15	804	866	879	850	40
	E540	770	780	820	790	26	860	850	830	847	15	850	870	850	857	12
	E541	714	700	705	706	7	787	778	772	779	8	798	800	802	800	2
	E543	780	797	779	785	10	869	829	836	845	21	838	846	832	839	7
Community Results			Mean		718			Mean		789			Mean		788	
			Median		744			Median		787			Median		818	
			Maximum		861			Maximum		912			Maximum		938	
			Minimum		375			Minimum		556			Minimum		439	
			MADe		30			MADe		31			MADe		26	
			%RSD		4 %			%RSD		4 %			%RSD		3%	
			N		54			N		54			N		54	

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Iron in Breakfast Cereals Containing Rice: DSQAP Exercise E

		Sample D: Ground Rice Cereal					Sample E: Flake Rice Cereal					Sample F: Crushed Wheat and Rice Cereal Mix				
Individual Results	lab code	D1	D2	D3	D Mean	D SD	E1	E2	E 3	E Mean	E SD	F1	F2	F3	F Mean	F SD
	E502	498	477	492	489	11	483	499	501	494	10	597	625	609	610	14
	E509	450	460	450	453	6	420	460	400	427	31	620	600	590	603	15
	E510	432	466	480	460	25	383	352	417	384	33	528	485	486	500	25
	E511	524	523	502	516	12	506	514	498	506	8	632	676	654	654	22
	E513	520	500	520	513	12	500	500	500	500	0	650	670	660	660	10
	E515	487	468	462	472	13	447	512	502	487	35	612	631	625	623	10
	E520	563	568	585	572	12	592	578	561	577	16	695	701	713	703	9
	E522	487	489	501	492	8	494	499	478	490	11	657	655	646	653	6
	E526	503	515	502	507	7	496	505	503	501	5	672	660	669	667	6
	E530	471	415	497	461	42	356	342	297	332	31	267	389	384	347	69
	E531	520	530	490	513	21	470	570	460	500	61	710	740	670	707	35
	E533	460	450	470	460	10	480	460	460	467	12	560	620	570	583	32
	E534	521	554	544	540	17	533	520	557	537	19	755	704	731	730	26
	E537	527	514	510	517	9	510	519	511	513	5	646	623	613	627	17
	E538	447	491	383	440	54	459	486	434	459	26	524	573	474	524	49
	E539	572	520	448	513	62	558	610	544	571	35	734	747	720	734	14
	E540	570	550	550	557	12	550	540	540	543	6	690	720	710	707	15
	E541	498	523	504	508	13	503	512	489	501	12	677	691	688	685	7
	E543	521	541	510	524	16	528	520	553	534	17	737	749	729	738	10
Community Results			Mean		499			Mean		488			Mean		629	
			Median		508			Median		500			Median		653	
			Maximum		572			Maximum		577			Maximum		734	
			Minimum		440			Minimum		332			Minimum		347	
			MADe		18			MADe		20			MADe		23	
			%RSD		3.5 %			%RSD		4%			%RSD		3.5%	
			N		54			N		54			N		54	

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Calcium in Breakfast Cereals Containing Wheat: DSQAP Exercise E

		Sample A: Ground Wheat Cereal SRM 3233					Sample B: Ground Wheat Cereal					Sample C: Flake Wheat Cereal				
	lab code	A1	A2	A3	A mean	A SD	B1	B2	B3	B Mean	B SD	C1	C2	C3	C Mean	C SD
Individual Results	E502	37112	37379	37511	37334	203	35794	35476	35996	35756	262	35780	35165	35940	35628	409
	E509	34300	36000	35000	35100	854	34000	34000	34000	34000	0	34000	33000	34400	33800	721
	E510	33600	32440	32520	32853	648	32080	32300	31650	32010	331	30150	31630	30430	30737	786
	E511	37900	36200	35800	36633	1115	35500	33900	33900	34433	924	36000	34200	34300	34833	1012
	E513	38000	37000	36000	37000	1000	33000	34000	35000	34000	1000	36000	34000	34000	34667	1155
	E515	35072	35661	35224	35319	306	33307	33627	32863	33266	384	33627	34948	33893	34156	699
	E520	42381	41400	41404	41728	565	38599	38580	38976	38718	223	39407	39114	38156	38892	654
	E522	36956	37620	37182	37253	338	35195	35293	34721	35070	306	35381	34631	34561	34858	455
	E526	36150	36150	36250	36183	58	34300	34400	34550	34417	126	34800	34850	34700	34783	76
	E529	38930	39020	38510	38820	272	35030	34330	34760	34707	353	36750	37160	37230	37047	259
	E531	36640	42680	37400	38907	3290	37530	39700	34480	37237	2622	35560	38260	39780	37867	2137
	E533	37800	37500	37300	37533	252	35000	35000	35300	35100	173	34600	35800	35900	35433	723
	E534	39100	40100	39900	39700	529	38500	36100	36400	37000	1308	36300	38200	37600	37367	971
	E537	40593	41600	39662	40618	969	34427	34784	33287	34166	782	37622	37233	38693	37849	756
	E538	35176	38217	36412	36602	1529	32933	35809	34227	34323	1440	34204	36685	34032	34974	1485
	E539	36200	37100	36800	36700	458	35600	33800	35400	34933	987	35100	35200	36200	35500	608
	E540	36700	37300	37400	37133	379	34200	34500	34000	34233	252	34400	35000	35000	34800	346
	E541	41119	39251	40357	40242	939	37538	37768	38128	37811	297	39495	39750	40507	39917	526
	E543	37618	37161	36964	37248	336	34452	34624	34775	34617	162	34951	33970	34584	34502	496
Community Results			Mean		37537			Mean		35066			Mean		35728	
			Median		37193			Median		34570			Median		35204	
			Maximum		41728			Maximum		38718			Maximum		39917	
			Minimum		32853			Minimum		32010			Minimum		30737	
			MADe		812			MADe		507			MADe		1053	
			%RSD		2.2 %			%RSD		1.5 %			%RSD		3%	
			N		54			N		54			N		54	

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Calcium in Breakfast Cereals Containing Rice: DSQAP Exercise E

		Sample D: Ground Rice Cereal					Sample E: Flake Rice Cereal					Sample F: Crushed Wheat and Rice Cereal Mix				
	lab code	D1	D2	D3	D Mean	D SD	E1	E2	E 3	E Mean	E SD	F1	F2	F3	F Mean	F SD
Individual Results	E502	197	195	196	196	1	192	202	196	197	5	18156	18166	18456	18259	171
	E509	190	190	210	197	12	210	210	200	207	6	18100	16000	16000	16700	1212
	E510	202	197	187	195	7	258	183	257	233	43	16450	15900	16540	16297	346
	E511	202	190	196	196	6	202	190	204	199	8	17100	17700	18300	17700	600
	E513	180	180	180	180	0	180	180	180	180	0	17000	16000	17000	16667	577
	E515	232	236	232	233	2	227	245	243	238	10	12352	14391	12642	13128	1103
	E520	168	171	175	171	4	164	165	155	161	6	15934	15700	16727	16120	538
	E522	145	148	141	145	4	147	148	144	146	2	17307	16928	17529	17255	304
	E526	190	180	180	183	6	190	190	190	190	0	16250	16600	16450	16433	176
	E529	252	184	168	201	45	262	191	236	230	36	17100	18100	20050	18417	1500
	E531	250	290	250	263	23	200	240	200	213	23	16940	19330	17770	18013	1213
	E533	220	240	240	233	12	220	210	220	217	6	13700	19300	16200	16400	2805
	E534	244	221	207	224	19	228	203	219	217	13	21900	20800	19300	20667	1305
	E537	184	176	169	176	8	171	173	168	171	3	17582	17382	17128	17364	228
	E538	1343	1504	897	1248	314	1421	905	556	960	435	17684	19429	21203	19439	1760
	E539	174	145	144	154	17	285	434	451	390	91	20300	20800	18100	19733	1436
	E540	480	440	460	460	20	440	430	450	440	10	18400	18300	18100	18267	153
	E541	273	238	194	235	40	341	223	320	295	63	31997	23040	22759	25932	5254
	E543	272	282	293	282	11	275	280	284	279	5	21155	20199	22039	21131	920
Community Results			Mean		272			Mean		272			Mean		17933	
			Median		196			Median		217			Median		17532	
			Maximum		1248			Maximum		960			Maximum		25932	
			Minimum		145			Minimum		146			Minimum		13128	
			MADe		14			MADe		11			MADe		1263	
			%RSD		7 %			%RSD		5 %			%RSD		7 %	
			N		54			N		4873			N		54	

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Zinc in Breakfast Cereals Containing Wheat: DSQAP Exercise E

		Sample A: Ground Wheat Cereal SRM 3233					Sample B: Ground Wheat Cereal					Sample C: Flake Wheat Cereal				
	lab code	A1	A2	A3	A mean	A SD	B1	B2	B3	B Mean	B SD	C1	C2	C3	C Mean	C SD
Individual Results	E502	631	630	632	631	1	720	719	710	716	6	713	712	722	716	5
	E509	540	540	560	547	12	620	600	610	610	10	820	800	840	820	20
	E510	502	552	556	536	30	569	563	631	587	38	554	446	565	522	66
	E511	630	602	595	609	19	695	666	664	675	17	714	675	676	688	22
	E513	540	530	530	533	6	570	570	580	573	6	590	590	580	587	6
	E515	647	652	646	648	3	716	747	707	723	21	742	768	758	756	13
	E520	683	670	673	675	7	725	730	735	730	5	755	740	735	743	10
	E522	614	627	619	620	7	689	693	681	688	6	710	699	689	699	11
	E526	620	602	601	608	11	677	684	677	679	4	702	716	710	709	7
	E529	593	581	589	588	6	691	689	678	686	7	688	675	682	682	7
	E530	588	596	587	590	5	677	674	671	674	3	658	664	618	647	25
	E531	630	710	690	677	42	750	800	830	793	40	620	710	720	683	55
	E533	580	570	570	573	6	660	610	680	650	36	730	690	700	707	21
	E534	578	585	582	582	4	640	609	618	622	16	583	615	605	601	16
	E537	615	621	620	619	3	665	665	651	660	8	665	650	643	653	11
	E538	554	599	578	577	23	602	637	620	620	17	599	656	627	627	28
	E539	612	613	613	613	1	691	677	685	684	7	673	706	726	702	27
	E540	640	660	660	653	12	700	710	710	707	6	710	720	710	713	6
	E541	554	551	554	553	2	609	609	611	610	1	624	622	626	624	2
	E543	624	625	628	626	2	694	690	700	694	5	703	691	693	696	6
Community Results			Mean		602			Mean		668			Mean		678	
			Median		608			Median		675			Median		688	
			Maximum		677			Maximum		793			Maximum		820	
			Minimum		533			Minimum		573			Minimum		522	
			MADe		9			MADe		10			MADe		19	
			%RSD		1.5 %			%RSD		1.5 %			%RSD		2.8 %	
			N		57			N		57			N		57	

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Zinc in Breakfast Cereals Containing Rice: DSQAP Exercise E

		Sample D: Ground Rice Cereal					Sample E: Flake Rice Cereal					Sample F: Crushed Wheat and Rice Cereal Mix				
Individual Results	lab code	A1	A2	A3	A mean	A SD	B1	B2	B3	B Mean	B SD	C1	C2	C3	C Mean	C SD
	E502	23.5	23.5	23.7	23.6	0.1	23.7	23.7	23.6	23.7	0.1	394.0	399.9	396.1	396.6	3.0
	E509	19.0	19.0	20.0	19.3	0.6	19.0	19.0	20.0	19.3	0.6	330.0	310.0	300.0	313.3	15.3
	E510	19.8	20.4	21.2	20.5	0.7	18.9	15.5	18.6	17.7	1.9	290.4	285.7	276.4	284.2	7.1
	E511	20.9	19.7	20.0	20.2	0.6	20.6	19.6	20.5	20.2	0.6	352.0	359.0	361.0	357.3	4.7
	E513	18.0	18.0	19.0	18.3	0.6	19.0	18.0	18.0	18.3	0.6	330.0	300.0	330.0	320.0	17.3
	E515	26.0	26.0	26.0	26.0	0.0	25.0	25.0	26.0	25.3	0.6	277.0	327.0	293.0	299.0	25.5
	E520	6.9	8.7	8.3	7.9	1.0	7.0	8.2	6.4	7.2	0.9	311.0	308.0	331.0	316.7	12.5
	E522	19.4	19.4	19.4	19.4	0.0	19.6	19.7	19.5	19.6	0.1	358.0	354.0	361.0	357.7	3.5
	E526	20.7	20.6	20.4	20.6	0.2	20.1	20.0	20.5	20.2	0.3	341.0	346.0	341.0	342.7	2.9
	E529	17.3	11.2	12.0	13.5	3.3	19.1	18.9	19.0	19.0	0.1	348.0	358.0	393.0	366.3	23.6
	E530	20.8	26.1	23.1	23.3	2.7	22.2	20.4	19.9	20.8	1.2	325.4	312.0	319.0	318.8	6.7
	E531	20.0	20.0	20.0	20.0	0.0	20.0	20.0	20.0	20.0	0.0	450.0	410.0	420.0	426.7	20.8
	E533	30.0	30.0	30.0	30.0	0.0	30.0	30.0	30.0	30.0	0.0	280.0	380.0	320.0	326.7	50.3
	E534	17.7	17.0	16.0	16.9	0.9	17.0	16.0	17.0	16.7	0.6	388.0	326.0	360.0	358.0	31.0
	E537	20.4	20.3	19.9	20.2	0.3	20.4	19.9	19.8	20.0	0.3	323.0	307.0	300.0	310.0	11.8
	E538	20.5	21.4	20.0	20.6	0.7	19.7	21.9	19.7	20.4	1.3	378.7	429.9	366.4	391.6	33.7
	E539	20.2	19.4	17.4	19.0	1.4	23.6	25.9	25.9	25.1	1.3	433.0	439.0	377.0	416.3	34.2
	E540	37.0	40.0	38.0	38.3	1.5	44.0	43.0	35.0	40.7	4.9	420.0	420.0	410.0	416.7	5.8
	E541	18.0	19.0	19.0	18.7	0.6	18.0	21.0	19.0	19.3	1.5	354.0	356.0	363.0	357.7	4.7
	E543	20.2	20.5	22.6	21.1	1.3	21.0	21.3	22.1	21.5	0.6	462.0	427.0	456.0	448.3	18.7
Community Results			Mean		21			Mean		21			Mean		351	
			Median		20			Median		20			Median		357	
			Maximum		38			Maximum		41			Maximum		427	
			Minimum		8			Minimum		7			Minimum		284	
			MADe		1			MADe		1			MADe		19	
			%RSD		4.2 %			%RSD		4.3 %			%RSD		5.2 %	
			N		57			N		57			N		57	

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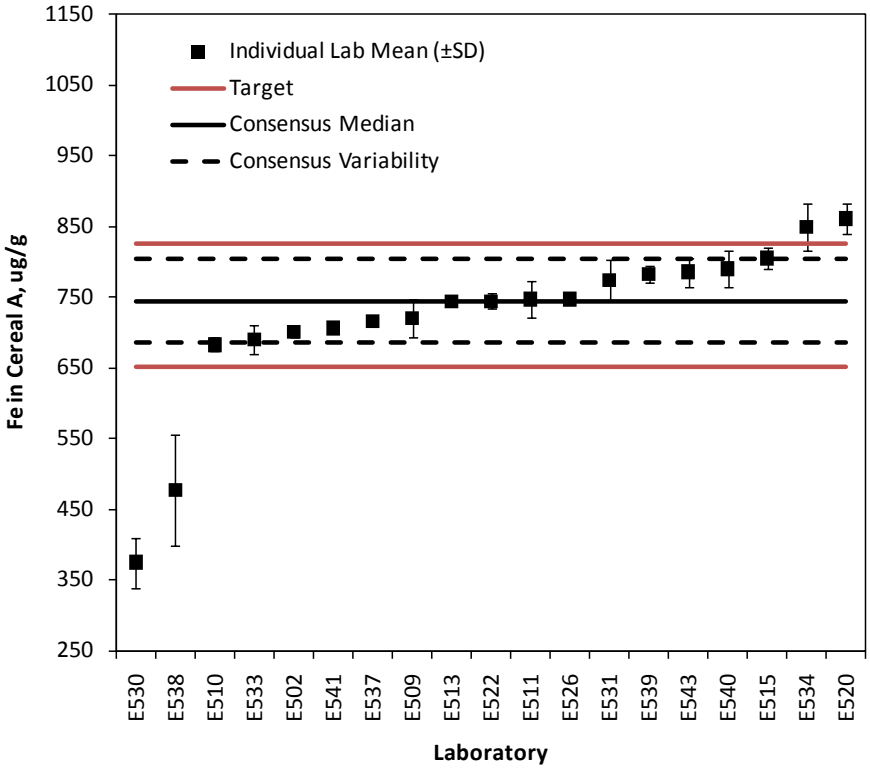
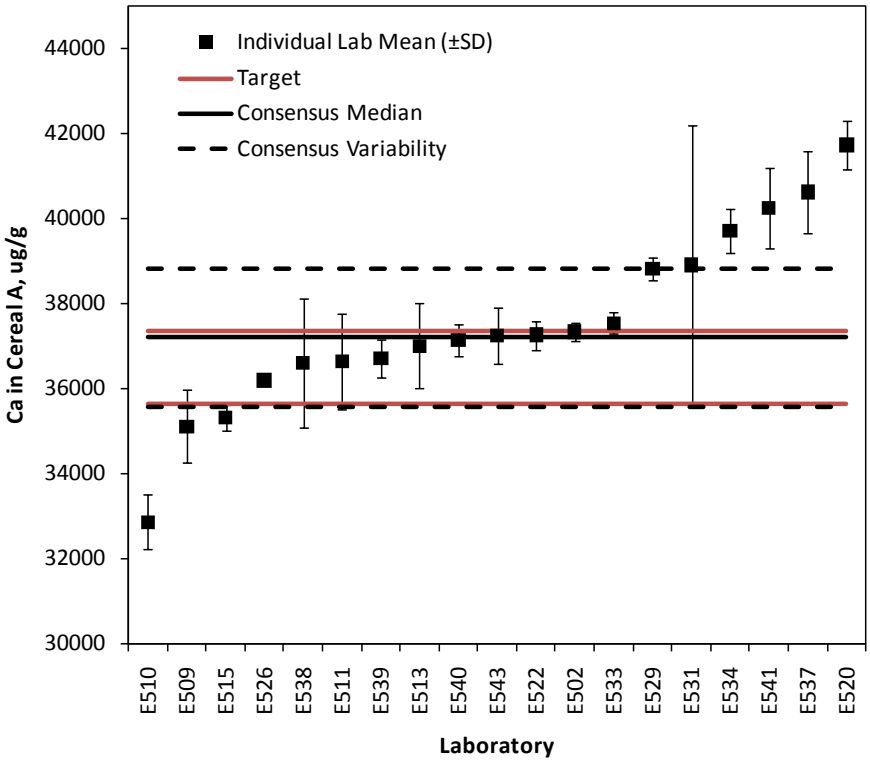
NUTRITIONAL ELEMENTS IN BREAKFAST CEREALS





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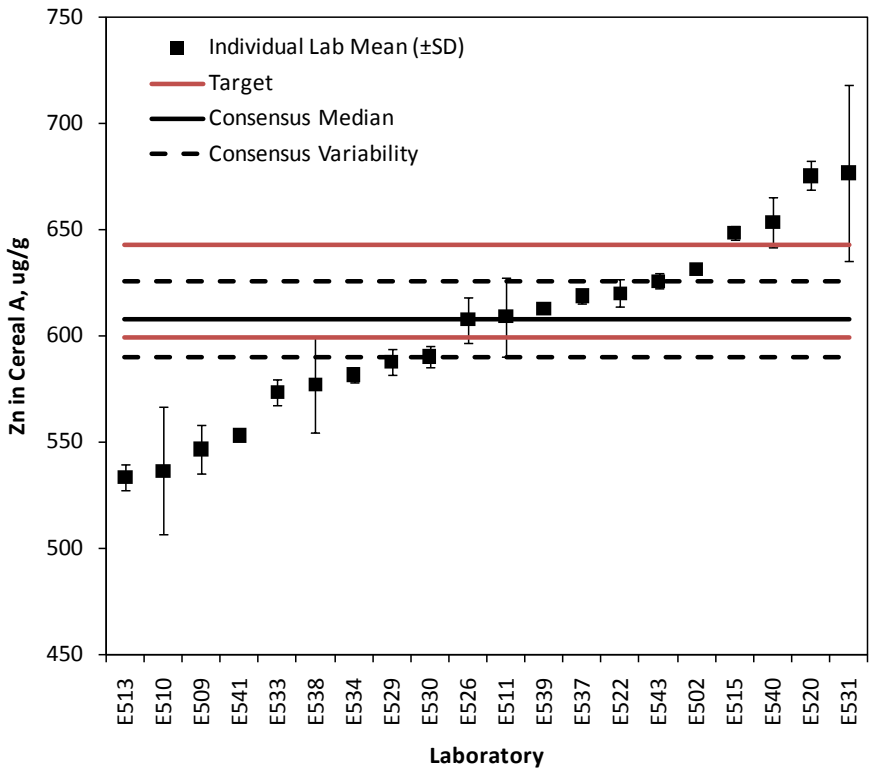

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NIACINAMIDE IN FORTIFIED BREAKFAST CEREAL

Exercise E is the third exercise in which participants have measured niacinamide. The focus on niacinamide arose because all previous DSQAP water-soluble vitamin studies demonstrated surprisingly scattered results. Relative to the other water-soluble vitamins, niacinamide is relatively stable and in relatively high abundance, thus it is a good candidate for methodological problem solving. In Exercise C, for the measurement of niacinamide in infant formula (SRM 1849) with Ephedra-Containing Protein Powder (SRM 3244) as a control, laboratory performance was poor, with fewer than half of the laboratories reporting results within the a relatively wide target area. A study in Exercise D paired an infant formula with SRM 1849 Infant/Adult Nutritional Formula as a control. Participants in Exercise D were also provided with a vial containing 500 mg of USP Niacinamide for use as a calibrant. The results in Exercise D demonstrated significant improvement, highlighting the importance of the selection of control and calibration materials.

As a final arm of the study, a very different sample matrix was sent for the measurement of niacinamide. In Exercise E, a ground breakfast cereal was sent with candidate SRM 3233 Fortified Breakfast Cereal as a control material.

- Seventeen laboratories enrolled in this exercise and received samples; 14 laboratories reported results (82 %).
- The consensus value was tighter for the unknown cereal than the control and there were more outliers for the measurement of the control than for the unknown.
- Examination of the two dimensional data plot shows that (with the exception of the significant outliers) results are on a diagonal line indicating that there may be a significant amount of variation in the calibrant materials used by different laboratories for the measurement of niacinamide.

Recommendations:

- Always be sure to characterize/qualify calibration materials and look for impurities in the materials.
- The use of well-paired control/sample materials is important.

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Niacin in Wheat Based Breakfast Cereals

		Control SRM 3233		Sample Ground Wheat Breakfast Cereal				
	Lab Code	Mean	SD	Sample A	Sample B	Sample C	Mean	SD
Individual Results	E505	841	16	711	836	771	773	63
	E510	688	136	399	715	827	647	222
	E511	851	18	869	878	836	861	22
	E514	793	11	917	922	918	919	3
	E515	746	14	951	957	977	962	14
	E520	837	82	675	769	718	721	47
	E524	749	2	836	843	837	839	4
	E525	765	18	859	865	860	861	3
	E531	760	15	846	852	846	848	3
	E533	760	4	854	854	853	854	1
	E534	906	2	784	785	788	786	2
	E536	753	3	861	861	863	862	1
	E538	784	7	919	904	901	908	10
	E541	791	23	838	710	800	783	66
Community Results						Mean		830
						Median		851
						Maximum		962
						Minimum		647
						MADe		10
						%RSD		1 %
						N		42

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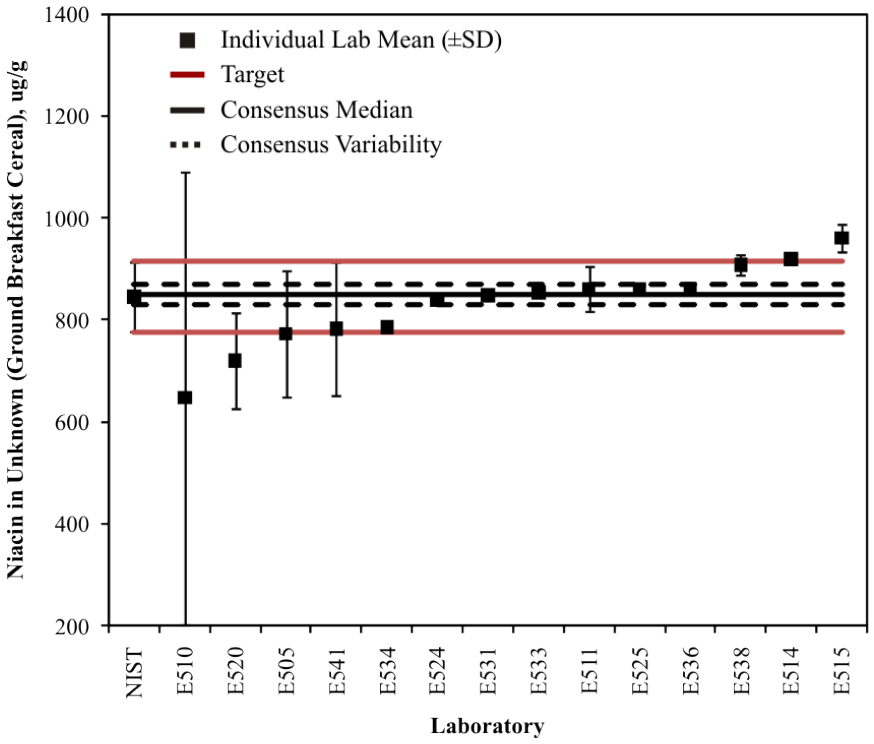
NIACINAMIDE IN FORTIFIED BREAKFAST CEREAL

VIEW 1



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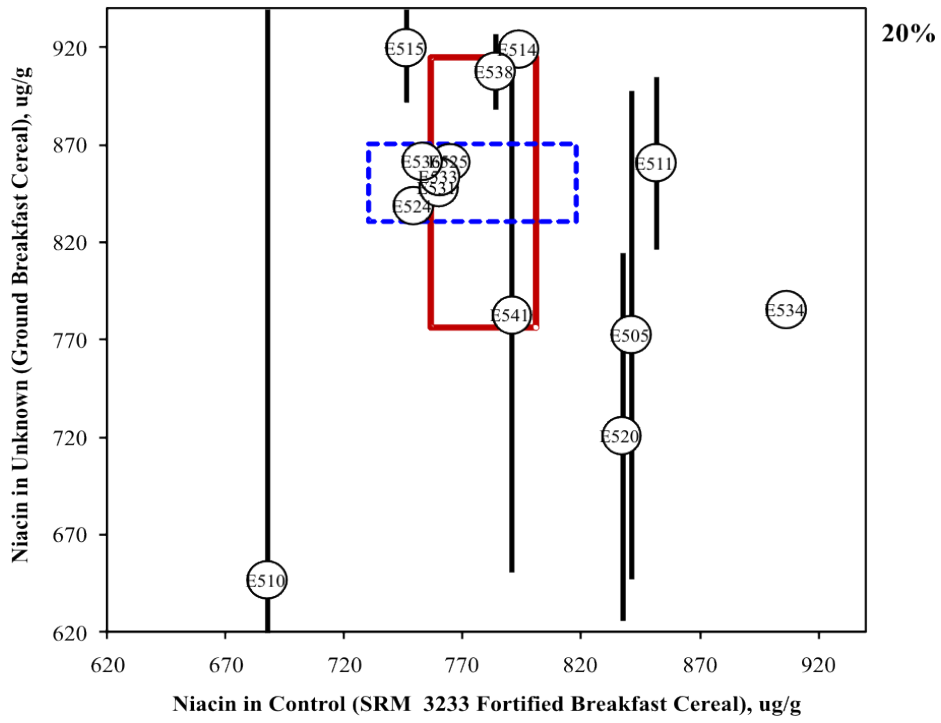

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CATECHINS IN GREEN TEA

SRM 3256 Green Tea-Containing Tablets was used as the sample with SRM 3255 *Camellia sinesis* Extract as a control material. The reporting spreadsheet specifically asked for epicatechin, epicatechin gallate (ECG), epigallocatechin (EGC), epigallocatechin gallate (EGCG), gallocatechin (GC), and gallocatechin gallate (GCG), however, it was understood that not all methods separate all of the compounds that calibration standards are not necessarily available for each of the analytes.

The data for the green tea study were more scattered than the other studies in this exercise. In order to effectively determine the source of this scatter, it is necessary to investigate chromatograms (are analytes completely resolved from each other?) and to further investigate calibration and extraction schemes. The control values are in much better agreement than the sample values; as the control does not require extraction, this points to possible extraction biases for the study overall.

- Eighteen laboratories signed up for this exercise and received samples; 14 laboratories reported data (78 %).
- Laboratories varied greatly on performance from analyte to analyte. For example, the reported results for one analyte might be right on target but the results for another analyte might be very high.
- Is it possible that extraction methods are causing epimerization? Several labs were low on epicatechin and high on catechin.
- Does epimerization matter for your purposes, or is the measurement of total catechins sufficient?
- Almost all laboratories reported high values for EGCG, and the consensus zone was entirely above the target zone. Is another compound coeluting with EGCG and causing a bias to higher values?
- A total catechin estimation was made as the sum of the reported values for all the catechins for each laboratory. Overall the results were more reassuring, with the consensus range falling in the center of the target zone for the sample. The two-dimensional data plot indicates a strong possibility of a calibration issue.

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Total Catechins in Green Tea

		Control SRM 3255		Green Tea-Containing Tablet (SRM 3256)				
	Lab Code	Mean	SD	Sample A	Sample B	Sample C	Mean	SD
Individual Results	E501	704	5	158	170	170	166	7
	E505	645	49	116	116	116	116	0
	E507	684	12	140	138	139	139	1
	E510	544	42	107	102	103	104	3
	E511	722	3	171	172	171	171	1
	E512	649	15	130	130	106	122	14
	E515	615	3	129	131	130	130	1
	E519	574	3	115	114	114	114	0
	E528	732	4	167	164	166	166	2
	E529	696	1	162	160	161	161	1
	E533	672	42	151	148	151	150	2
	E534	723	7	166	171	163	167	4
	E535	973	7	205	203	203	204	2
	E537	666	61	145	147	145	145	1
Community Results						Mean		147
						Median		148
						Maximum		204
						Minimum		104
						MADe		2
						%RSD		1.4 %
						N		42

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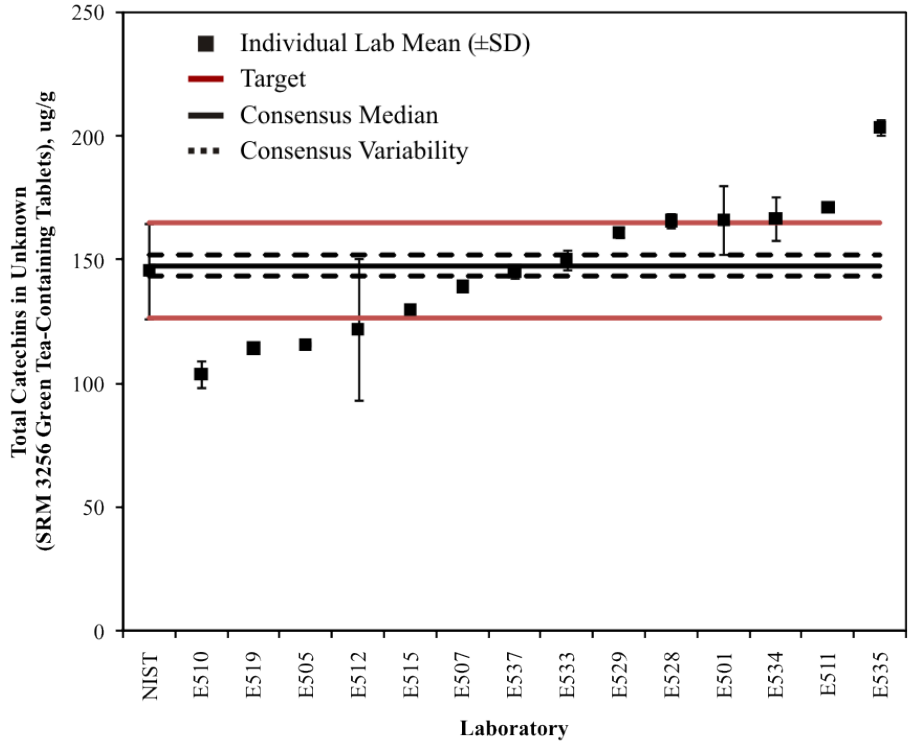
TOTAL CATECHINS IN GREEN TEA

VIEW 1



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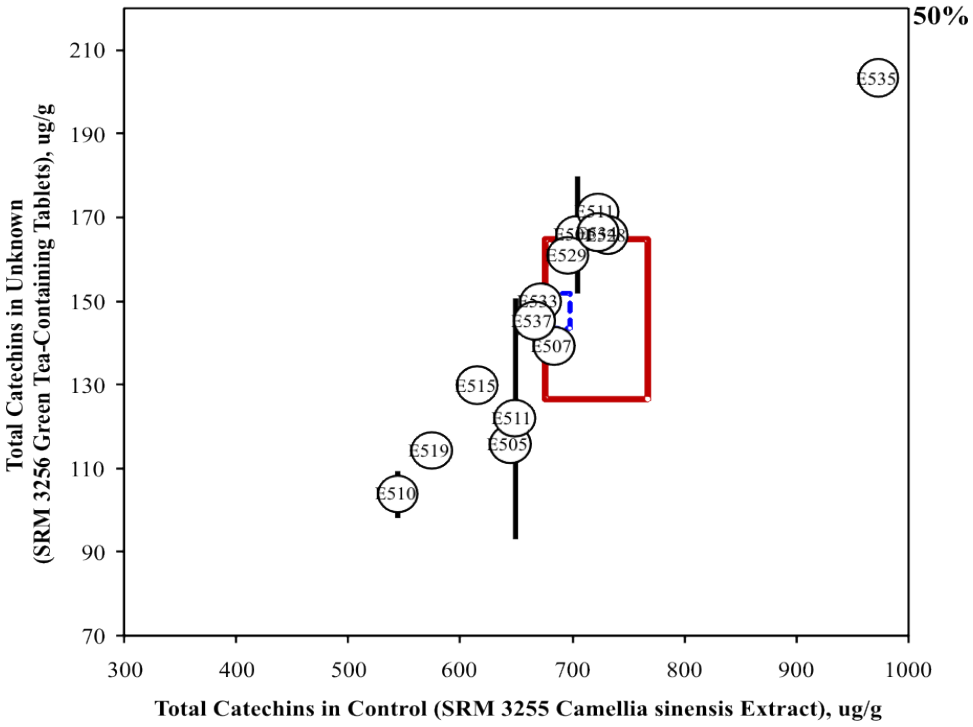

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Catechin in Green Tea

		Control SRM 3255		Green Tea-Containing Tablet (SRM 3256)				
	Lab Code	Mean	SD	Sample A	Sample B	Sample C	Mean	SD
Individual Results	E501	20	0	8	10	10	9	1
	E505	11	1	6	6	6	6	0
	E507	8	0					
	E510	9	1	3	2	2	2	0
	E511	8	0	2	2	2	2	0
	E512	10	1	3	3	3	3	0
	E515	9	0	3	3	3	3	0
	E519	9	1	3	3	3	3	0
	E528	9	0	2	2	2	2	0
	E529	8	0	3	3	3	3	0
	E533	10	2	5	5	4	5	0
	E534	9	0	2	2	2	2	0
	E535	10	0	3	3	3	3	0
	E537	48	64	3	3	3	3	0
Community Results						Mean		4
						Median		3
						Maximum		9
						Minimum		2
						MADe		0
						%RSD		5 %
						N		39

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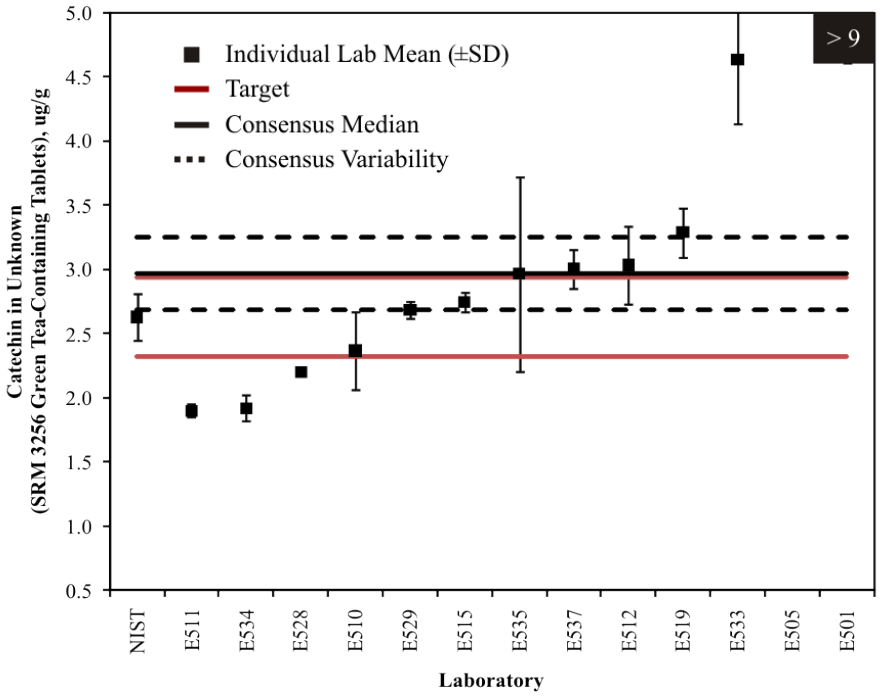
CATECHIN IN GREEN TEA

VIEW 1



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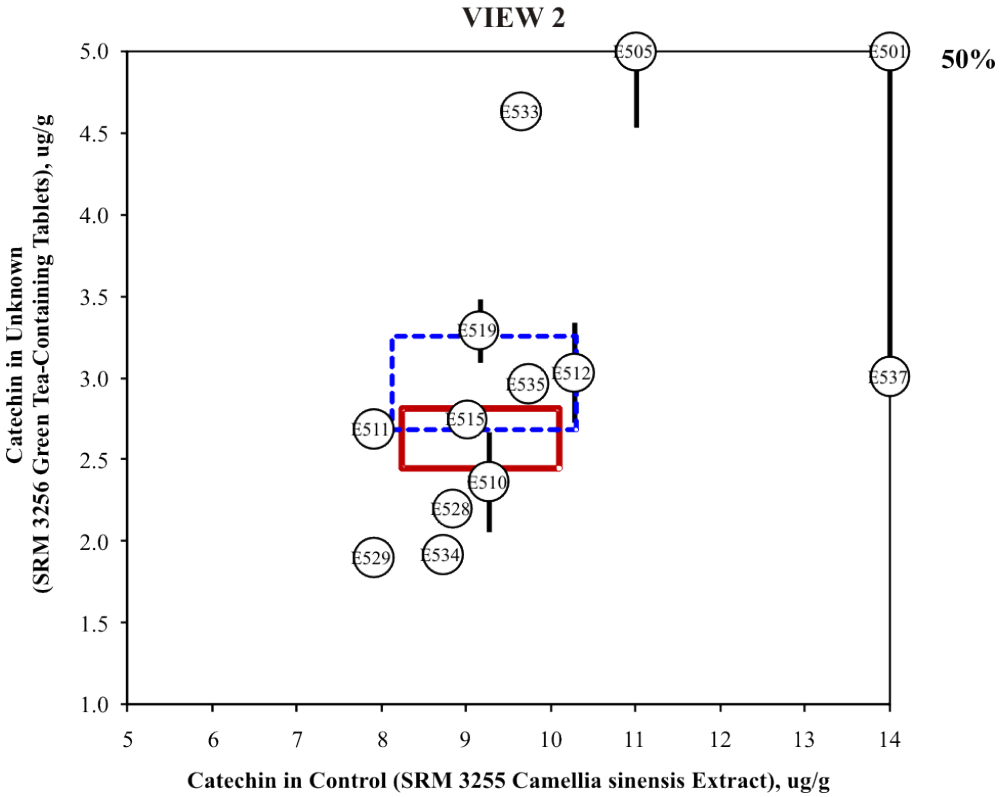

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Epicatechin in Green Tea

		Control SRM 3255		Green Tea-Containing Tablet (SRM 3256)				
	Lab Code	Mean	SD	Sample A	Sample B	Sample C	Mean	SD
Individual Results	E501	57	1	13	15	15	14	1
	E505	48	4	14	14	14	14	0
	E507	47	5	11	11	10	11	0
	E510	38	3	12	11	11	11	0
	E511	47	0	12	12	12	12	0
	E512	47	1	10	11	10	10	1
	E515	45	0	17	17	17	17	0
	E519	44	0	11	11	11	11	0
	E528	48	1	11	11	11	11	0
	E529	40	1	14	13	14	14	0
	E533	94	39	9	10	11	10	1
	E534	45	0	15	16	15	15	1
	E535	48	1	12	11	12	12	0
	E537	43	2	11	11	11	11	0
Community Results						Mean		12
						Median		11
						Maximum		17
						Minimum		10
						MADe		0
						%RSD		5 %
						N		42

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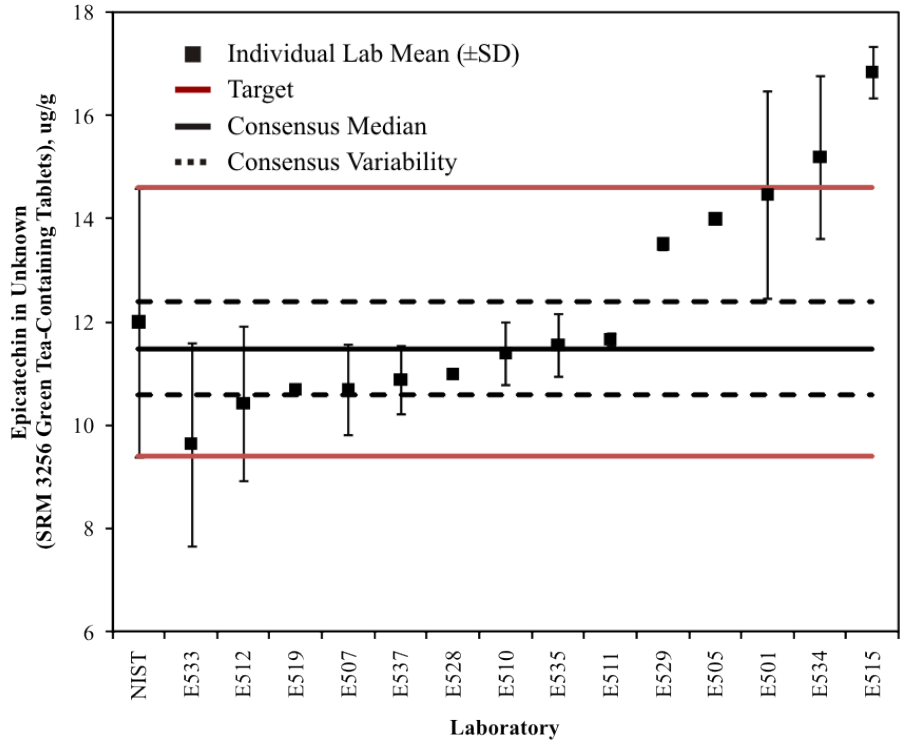
EPICATECHIN IN GREEN TEA

VIEW 1



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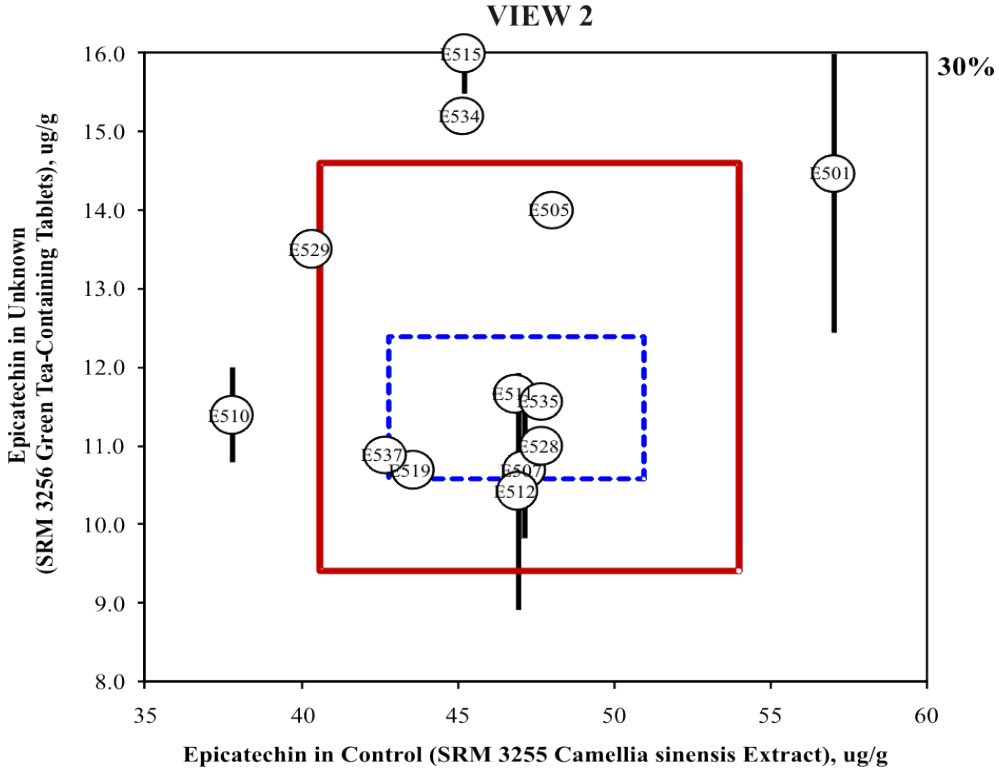

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Epicatechin Gallate in Green Tea

		Control SRM 3255		Green Tea-Containing Tablet (SRM 3256)				
	Lab Code	Mean	SD	Sample A	Sample B	Sample C	Mean	SD
Individual Results	E501	123.7	0.6	24.2	28.0	26.9	26.4	2.0
	E505	90.3	6.8	15.0	15.0	15.0	15.0	0.0
	E507	92.3	1.0	18.0	17.9	18.0	18.0	0.1
	E510	91.7	6.5	16.9	16.3	16.5	16.6	0.3
	E511	103.7	0.6	20.5	20.7	20.6	20.6	0.1
	E512	90.3	5.5	17.1	18.0	17.6	17.6	0.5
	E515	92.9	0.6	19.3	19.4	19.2	19.3	0.1
	E519	99.8	1.1	20.3	20.1	20.4	20.3	0.2
	E528	90.3	0.6	18.0	18.0	18.0	18.0	0.0
	E529	86.9	1.2	17.2	17.1	16.7	17.0	0.3
	E533	109.3	1.5	27.3	26.7	27.4	27.1	0.4
	E534	101.0	1.0	20.4	21.0	20.0	20.5	0.5
	E535	74.0	0.0	15.4	14.2	15.7	15.1	0.8
	E537	89.3	0.3	19.8	20.5	20.5	20.3	0.4
Community Results						Mean		19.4
						Median		18.7
						Maximum		27.1
						Minimum		15.0
						MADe		0.4
						%RSD		2.3 %
						N		42

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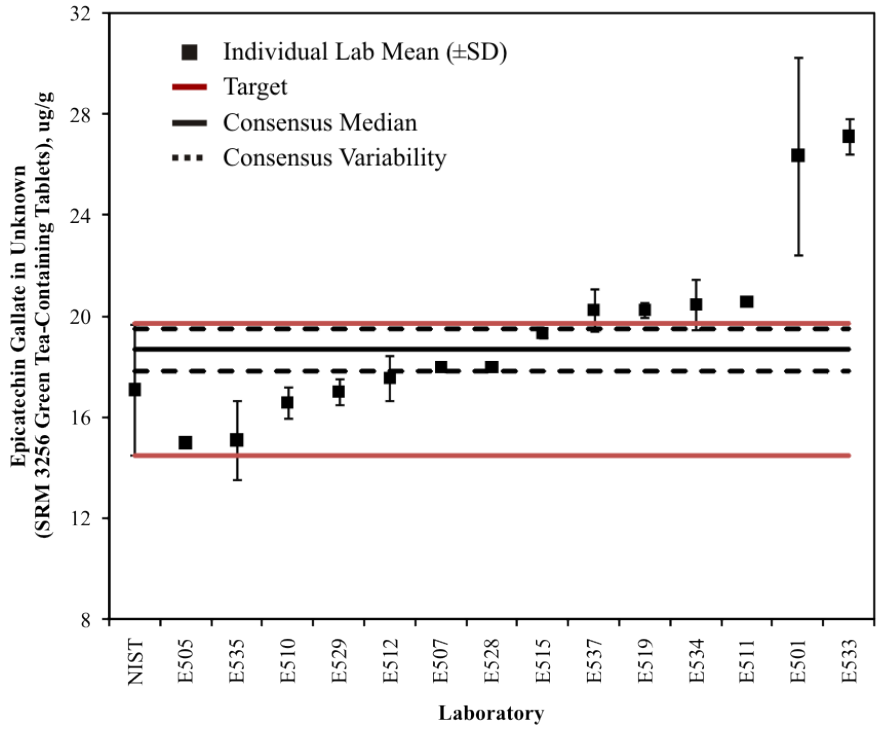
EPICATECHIN GALLATE (ECG) IN GREEN TEA

VIEW 1



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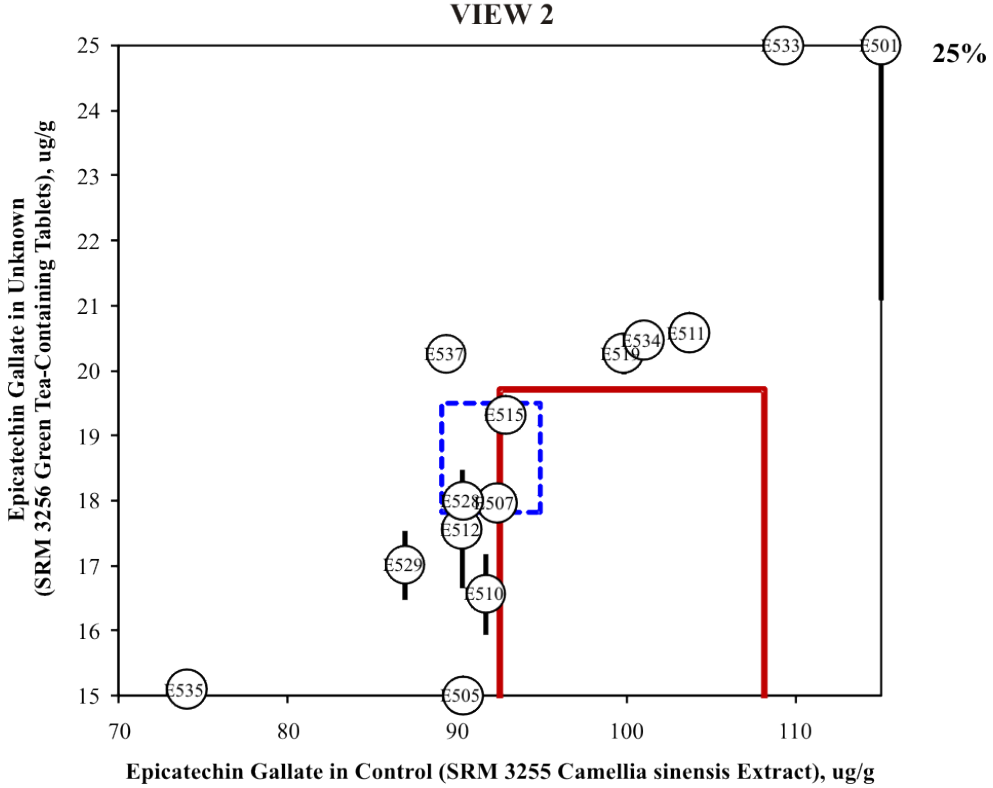

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Epigallocatechin in Green Tea

		Control SRM 3255		Green Tea-Containing Tablet (SRM 3256)				
	Lab Code	Mean	SD	Sample A	Sample B	Sample C	Mean	SD
Individual Results	E501	85	1	30	34	33	32	2
	E505	22	3	6	6	7	6	1
	E507	85	5	29	29	29	29	0
	E511	82	1	31	31	31	31	0
	E512	93	2	29	28	21	26	4
	E528	77	1	29	29	29	29	0
	E529	92	1	38	37	38	38	0
	E533	63	1	28	27	28	28	0
	E534	87	1	32	33	31	32	1
	E535	72	2	22	22	22	22	0
	E537	77	3	28	28	27	28	0
Community Results						Mean		27
						Median		29
						Maximum		38
						Minimum		6
						MADe		1
						%RSD		2 %
						N		33

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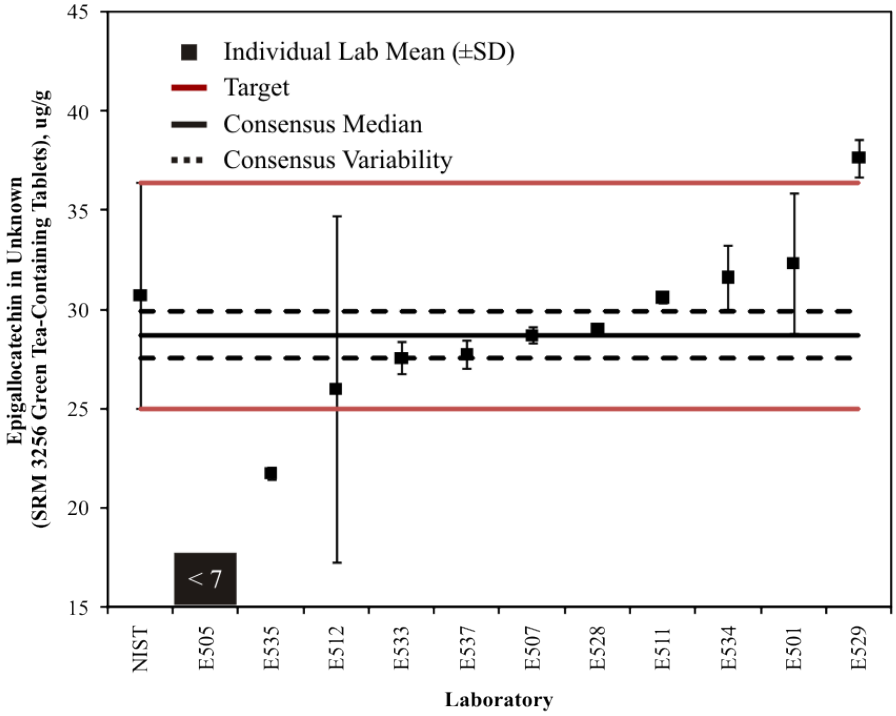
EPIGALLOCATECHIN (EGC) IN GREEN TEA

VIEW 1



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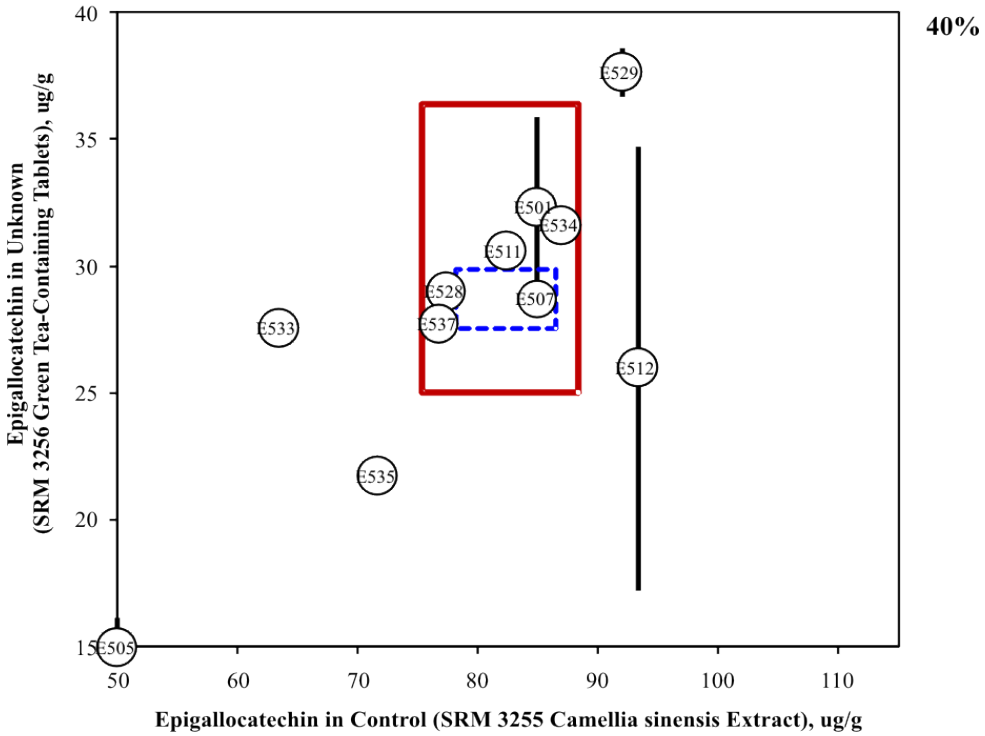

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Epigallocatechin Gallate in Green Tea

		Control SRM 3255		Green Tea-Containing Tablet (SRM 3256)				
	Lab Code	Mean	SD	Sample A	Sample B	Sample C	Mean	SD
Individual Results	E501	418.3	3.1	81.6	83.9	84.6	83.4	1.6
	E505	424.3	31.8	67.0	67.0	66.0	66.7	0.6
	E507	404.2	4.7	74.8	74.4	75.1	74.8	0.3
	E510	346.9	27.4	61.7	58.8	59.5	60.0	1.5
	E511	424.3	1.5	84.5	85.2	85.0	84.9	0.4
	E512	408.0	8.7	71.0	70.0	54.0	65.0	9.5
	E515	425.0	2.3	82.7	83.4	83.0	83.0	0.4
	E519	421.7	1.2	80.3	79.8	80.1	80.1	0.3
	E528	442.7	2.1	90.0	88.0	89.0	89.0	1.0
	E529	431.7	1.7	84.2	83.5	84.0	83.9	0.4
	E533	383.2	3.1	78.9	76.9	78.2	78.0	1.0
	E534	440.3	4.0	88.4	91.0	86.9	88.8	2.1
	E535	710.7	3.1	141.0	141.0	139.0	140.3	1.2
	E537	409.2	0.9	83.5	83.9	83.2	83.5	0.4
Community Results						Mean		82.9
						Median		83.2
						Maximum		140.3
						Minimum		60.0
						MADe		1.2
						%RSD		1.4 %
						N		42

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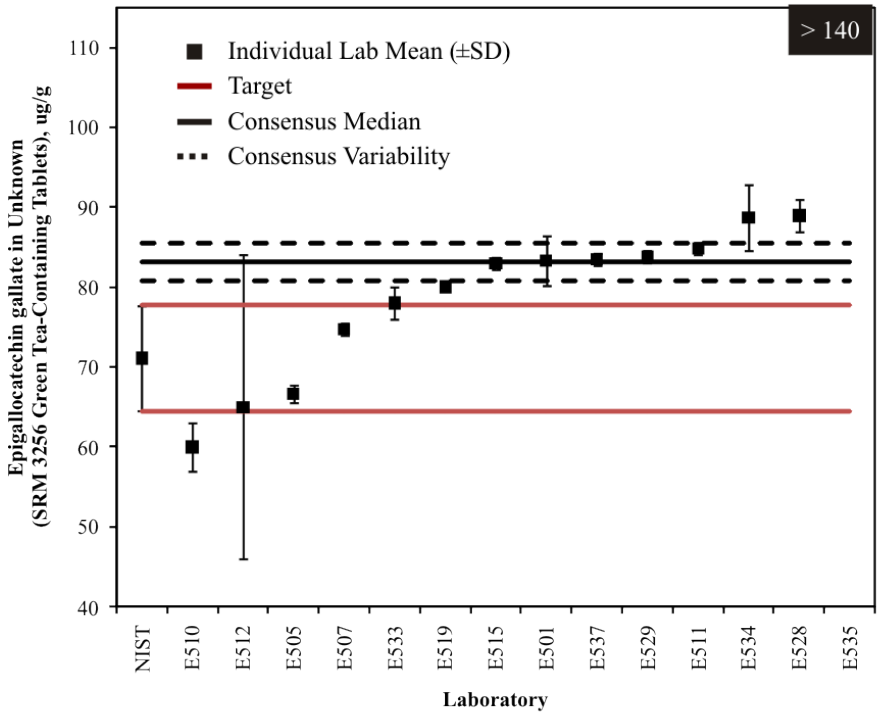
EPIGALLOCATECHIN GALLATE (EGCG) IN GREEN TEA

VIEW 1



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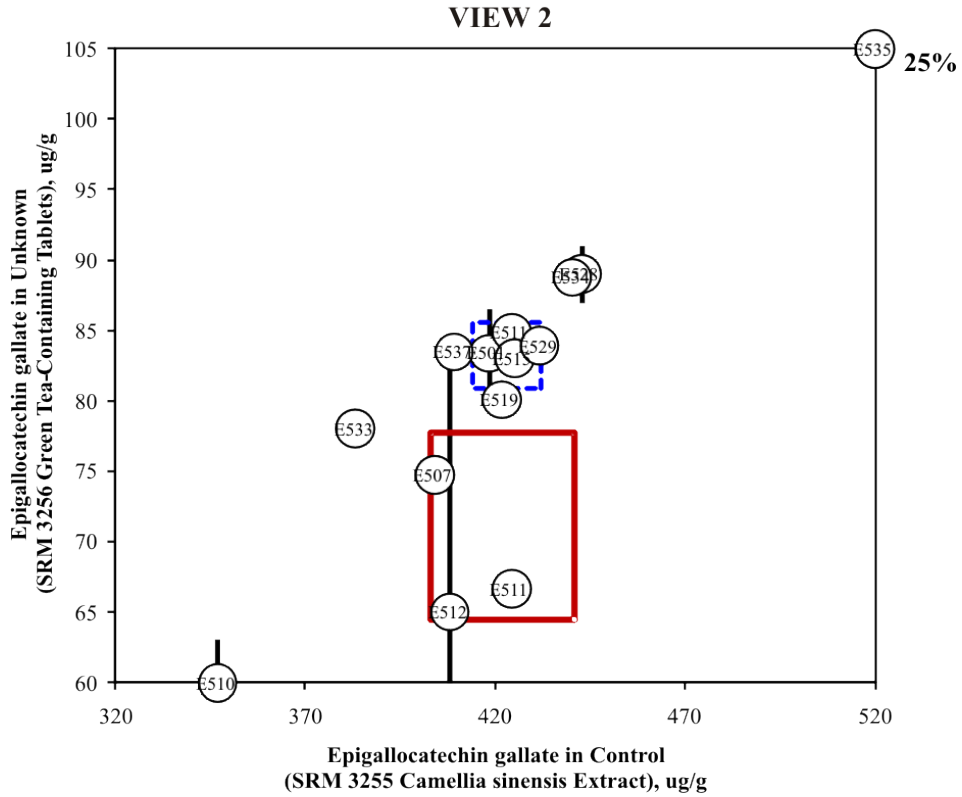

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Gallocatechin in Green Tea

		Control SRM 3255		Green Tea-Containing Tablet (SRM 3256)				
Individual Results	Lab Code	Mean	SD	Sample A	Sample B	Sample C	Mean	SD
	E505	6.3	0.6	2.0	2.0	2.0	2.0	0.0
	E510	22.5	1.8	8.6	7.6	8.0	8.1	0.5
	E511	13.0	0.1	14.3	14.5	14.1	14.3	0.2
	E528	21.3	0.6	8.0	8.0	8.0	8.0	0.0
	E535	17.2	0.7	5.9	5.9	5.8	5.9	0.1
Community Results						Mean		7.6
						Median		8.0
						Maximum		14.3
						Minimum		2.0
						MADe		0.1
						%RSD		1 %
						N		15

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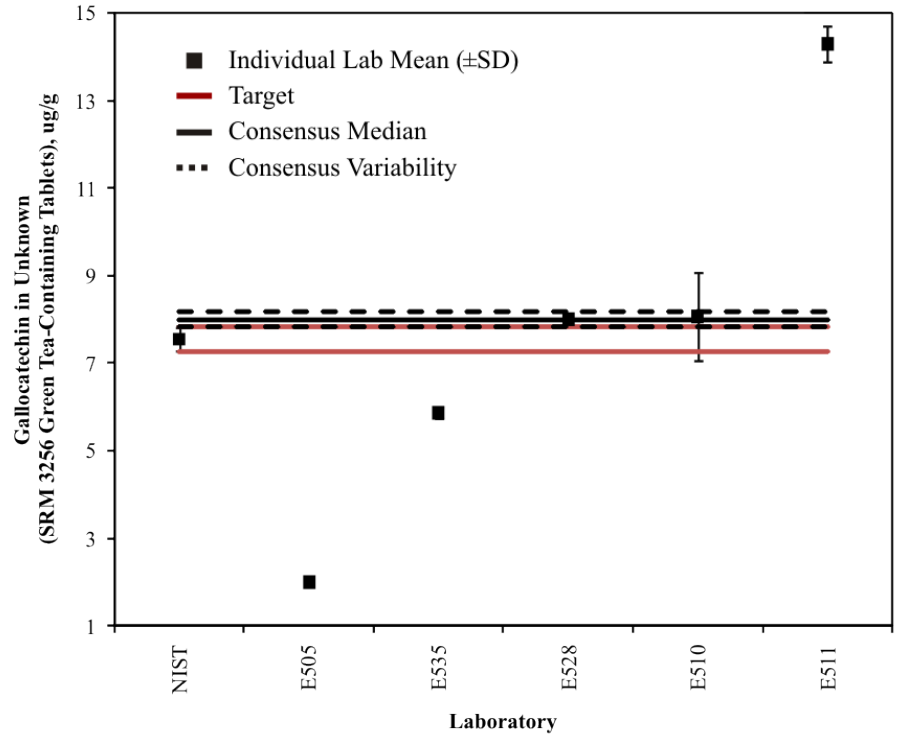
GALLOCATECHIN (GC) IN GREEN TEA

VIEW 1



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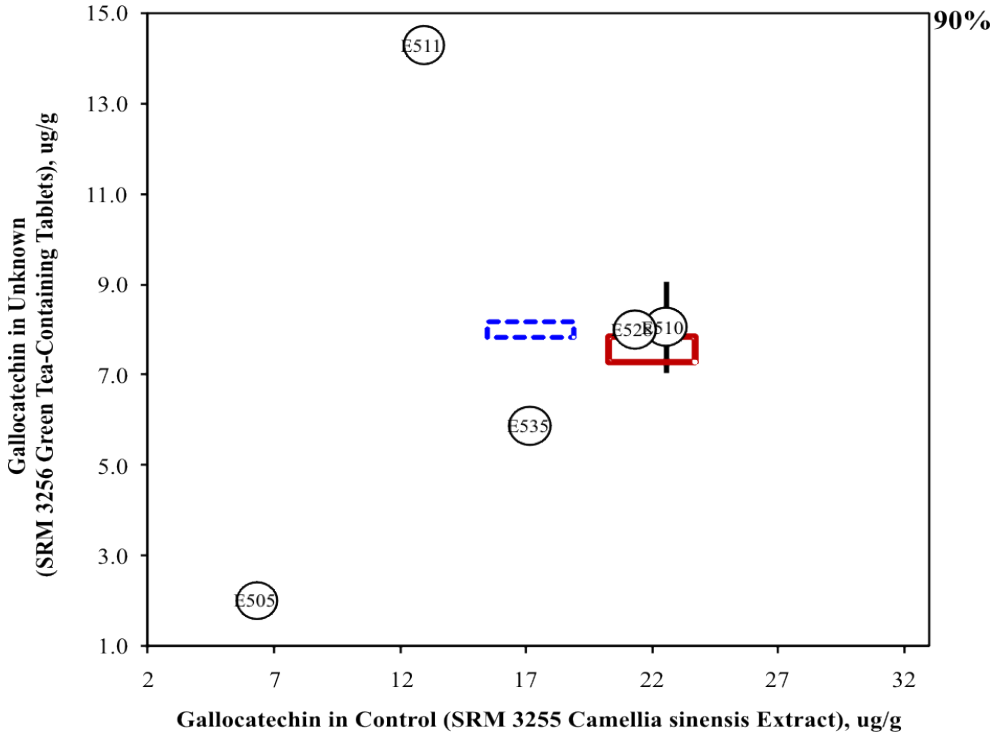

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Gallocatechin Gallate in Green Tea

		Control SRM 3255		Green Tea-Containing Tablet (SRM 3256)				
	Lab Code	Mean	SD	Sample A	Sample B	Sample C	Mean	SD
Individual Results	E505	42.7	3.1	6.0	6.0	6.0	6.0	0.0
	E507	47.6	0.5	7.2	7.1	7.2	7.2	0.1
	E510	35.8	2.9	5.5	5.5	5.6	5.5	0.1
	E511	44.4	0.2	7.4	7.5	7.5	7.5	0.1
	E515	43.2	0.1	7.9	7.9	7.8	7.9	0.1
	E528	43.3	0.6	9.0	8.0	9.0	8.7	0.6
	E529	37.4	0.1	6.3	6.3	6.1	6.2	0.1
	E533	12.3	0.7	3.0	2.9	3.0	2.9	0.1
	E534	41.2	0.4	8.6	8.8	8.5	8.6	0.2
	E535	42.3	1.5	6.1	5.8	6.0	6.0	0.2
Community Results						Mean		6.6
						Median		6.7
						Maximum		8.7
						Minimum		2.9
						MADe		0.1
						%RSD		1.3 %
						N		30

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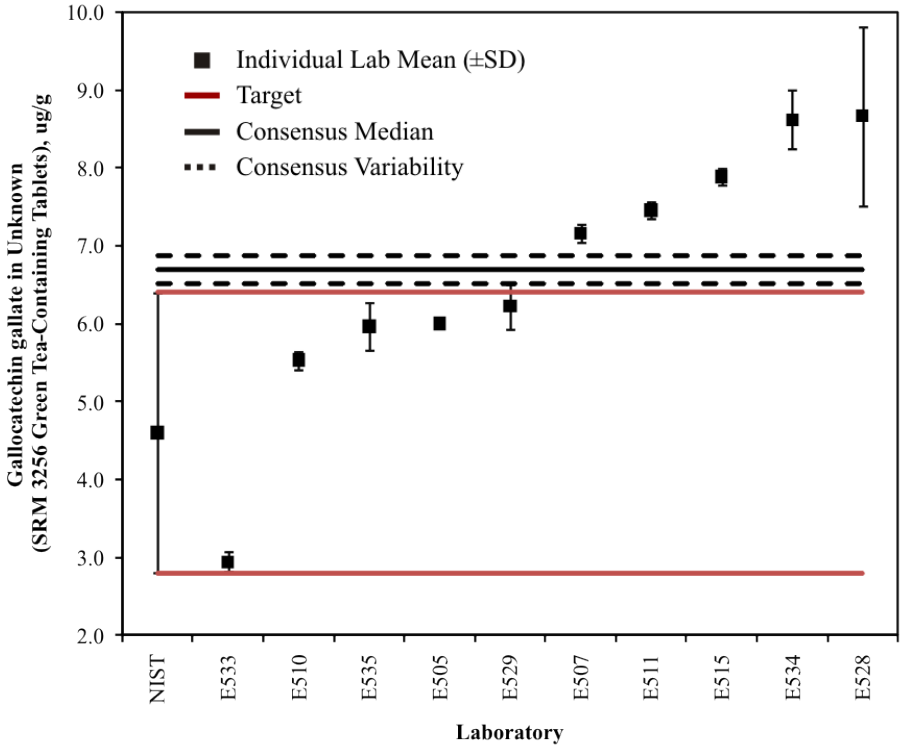
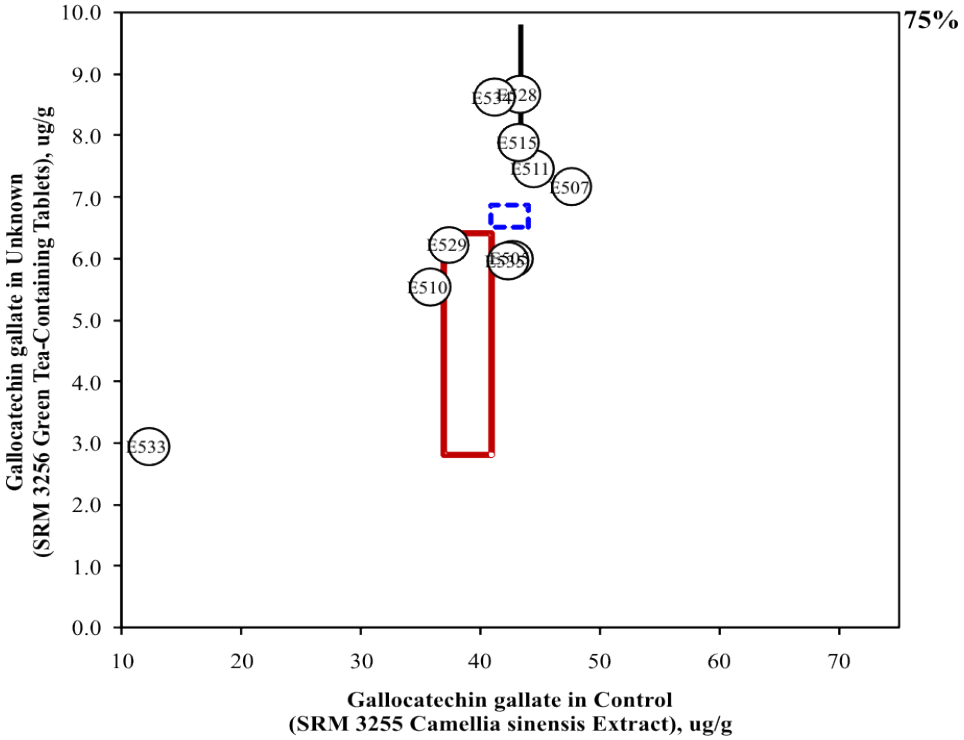
GALLOCATECHIN GALLATE (GCG) IN GREEN TEA

VIEW 1



VIEW 2



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β-CAROTENE IN MULTIVITAMIN TABLETS

Exercise D included a study of β-carotene in botanical oils. Unfortunately, during the course of the study it was discovered that the β-carotene in the carrot extract in oil was degrading rapidly, making the interpretation of the results close to impossible. In Exercise E, SRM 3280 Multivitamin/Multielement Tablets were sent to participants as the sample and SRM 3251 *Serenoa repens* Extract was sent as a control. The amount of total β-carotene in the multivitamin tablet is approximately 10 times the amount in the control material and the extraction of the β-carotene from the multivitamin tablet is significantly more challenging than SRM 3251.

- Twenty laboratories enrolled in this exercise and received samples; 14 laboratories reported results (70 %).
- Due to the extraction methods required for the multivitamin tablet, the total β-carotene is the best measure for laboratory comparability.
- Most laboratories that were not in the target range reported total β-carotene values that were high for the control and low for the sample.
- 53 % of the laboratories that reported data were within the target range for the measurement of β-carotene in the sample.

Recommendations:

- The low values reported for the sample are likely due to extraction problems, either an incomplete extraction or the β-carotene isomerized or degraded, leading to a lower value.
- Check molar absorptivity used for the determination of calibrant concentration. If different laboratories are using different values, there is the potential for wide variation in the results.

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Total β-carotene in Multivitamin

		Control SRM 3251		Sample Multivitamin Tablet (SRM 3280)				
	Lab Code	Mean	SD	Sample A	Sample B	Sample C	Mean	SD
Individual Results	E501	43			45	35	38	6
	E505	39	0	470	430	450	450	20
	E511	54	1	391	375	402	389	14
	E515	48	0	607	551	585	581	28
	E518	47	0	24	18	23	21	3
	E520	61	0	340	307	316	321	17
	E529	60	4	828	729	717	758	61
	E533	47	3	515	542	491	516	26
	E534	45	2	473	455	458	462	10
	E536	50	1	166	165	173	168	4
	E537	69	3	500	447	590	512	72
	E538	128	12	458	456	484	466	15
	E540	63	1	491	461	481	478	15
	E542	64						
Community Results						Mean		397
						Median		462
						Maximum		758
						Minimum		21
						MADe		23
						%RSD		5 %
						N		39

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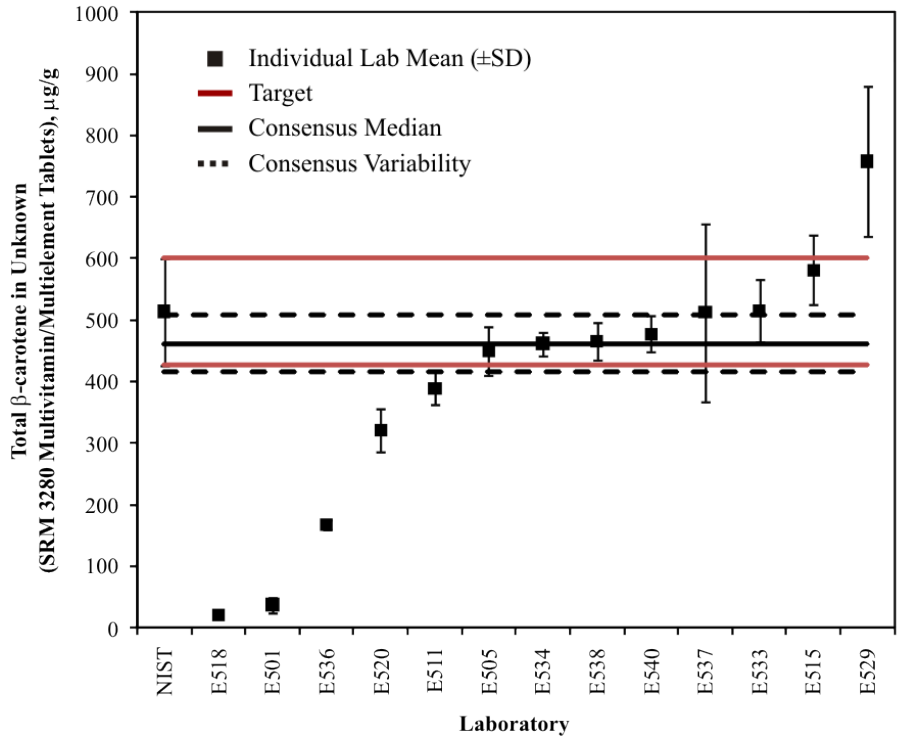
TOTAL β-CAROTENE IN MULTIVITAMIN TABLETS

VIEW 1



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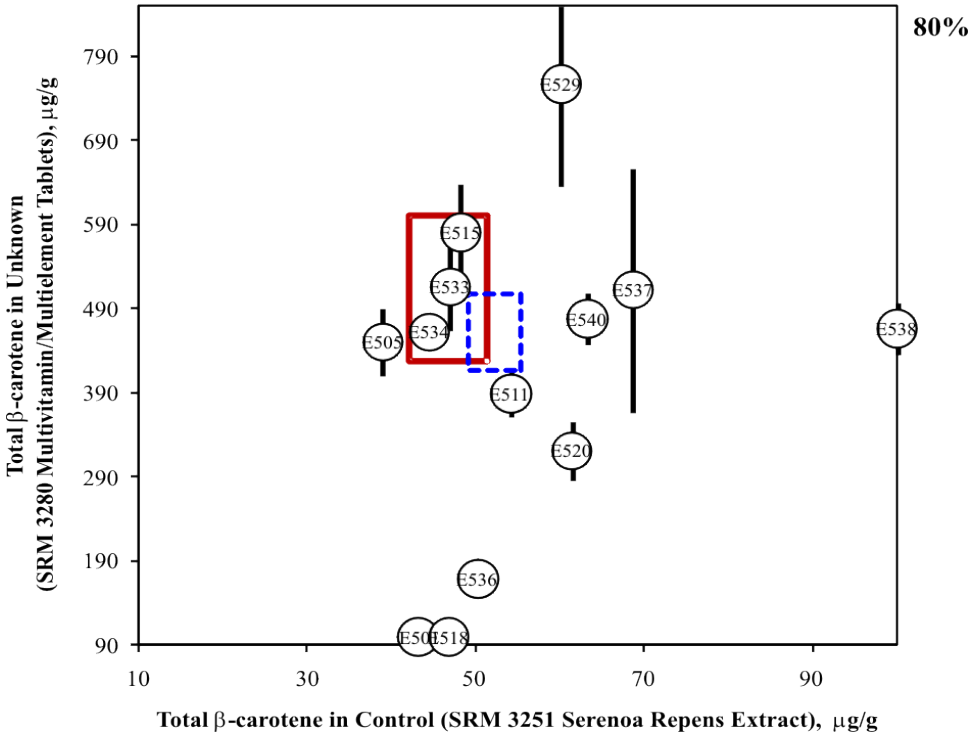

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All *trans* β-carotene in Multivitamin

		Control SRM 3251		Sample Multivitamin Tablet (SRM 3280)				
Individual Results	Lab Code	Mean	SD	Sample A	Sample B	Sample C	Mean	SD
	E505	32	0	390	360	370	373	15
	E511	48	1	386	365	393	381	15
	E518	39	0	17	13	17	16	2
	E520	49	0	297	267	274	280	16
	E529	49	3	711	627	616	651	52
	E533	39	2	366	375	338	360	19
	E537	54	2	500	447	590	512	72
	E542			127	106	152	128	23
Community Results						Mean		397
						Median		462
						Maximum		758
						Minimum		21
						MADe		23
						%RSD		5 %
						N		39

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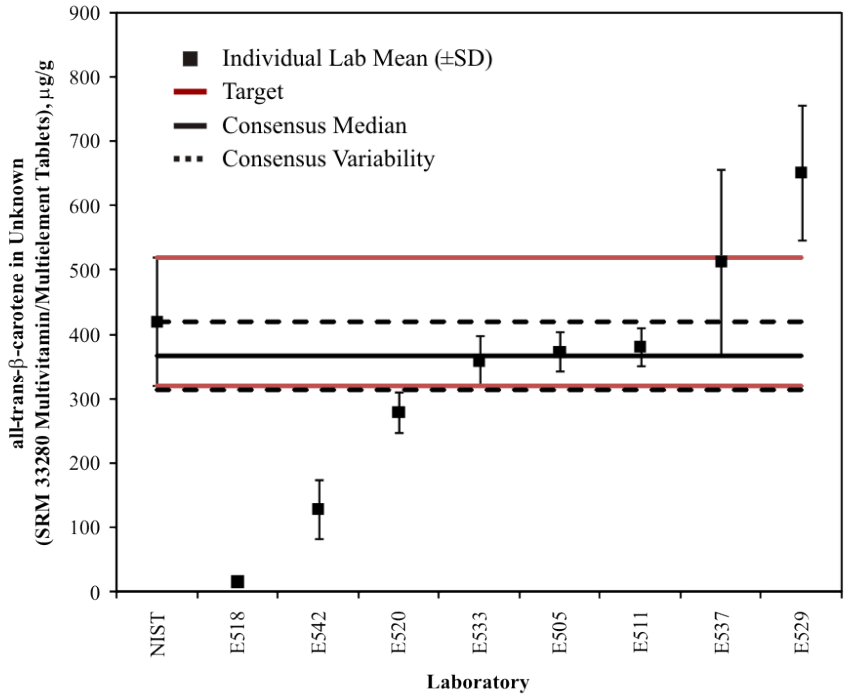
trans-β-CAROTENE IN MULTIVITAMIN TABLETS

VIEW 1



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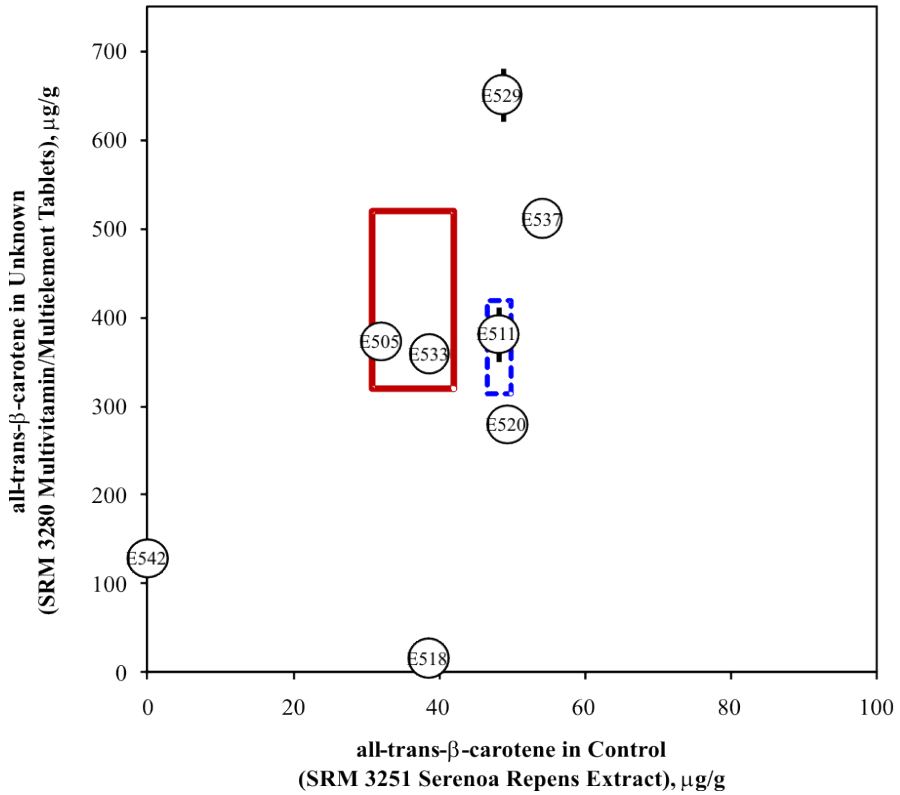

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9-*cis* -β-carotene in Multivitamin

		Control SRM 3251		Sample Multivitamin Tablet (SRM 3280)				
	Lab Code	Mean	SD	Sample A	Sample B	Sample C	Mean	SD
Individual Results	E505	4	0	11	12	12	12	1
	E511	6	0	5	10	9	8	3
	E518	6	0	2	1	2	2	0
	E520	7	0					
	E529	12	2	117	101	101	106	9
	E533	12	3	149	167	153	156	9
	E537	15	1					
Community Results						Mean		57
						Median		12
						Maximum		156
						Minimum		2
						MADe		4
						%RSD		34 %
						N		15

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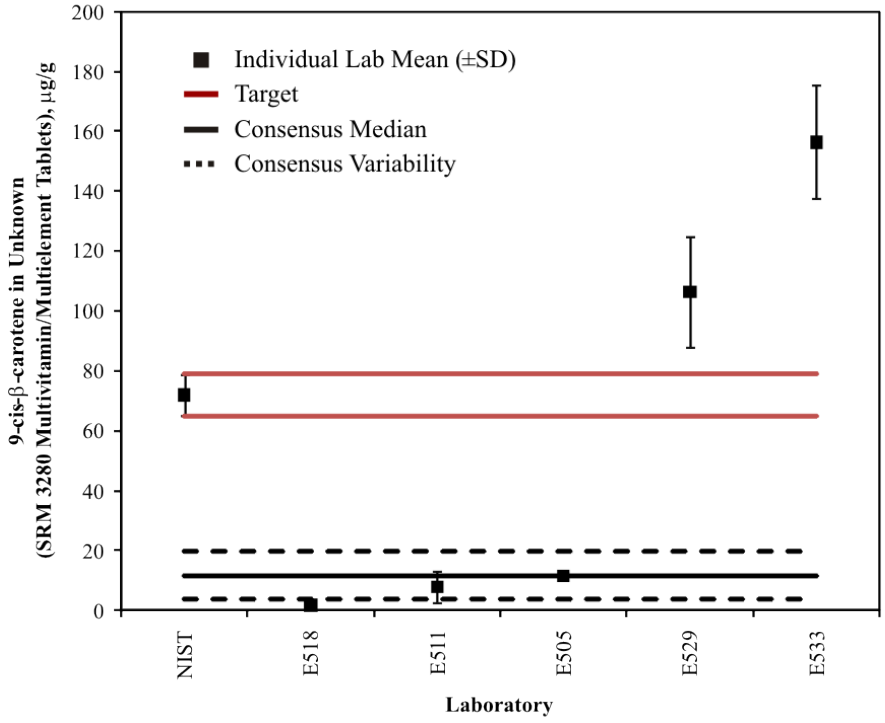
9-*cis*-β-CAROTENE IN MULTIVITAMIN TABLETS

VIEW 1



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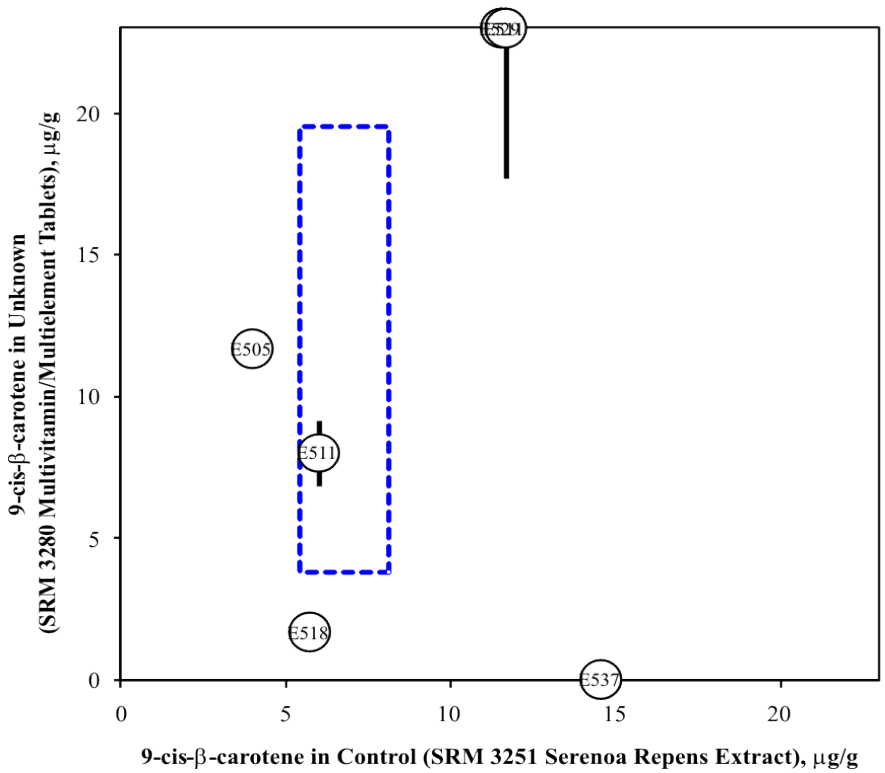

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AFLATOXINS IN PEANUT PRODUCTS

The Good Manufacturing Processes (GMPs), established by the US Food and Drug Administration (FDA), now apply to all dietary supplement manufacturers and specify the necessity of testing for contaminants and toxins as well as active/marker compounds. For this reason, the DSQAP will be including more toxins and contaminant studies in future exercises.

Ground whole peanuts were sent as a sample with SRM 2387 Peanut Butter as a control. The samples were purchased from The Food and Environment Research Agency's Food Analysis Performance Assessment Scheme (FAPAS) in the UK. They were analyzed by 65 laboratories between March 2009 and April 2009. The levels of aflatoxins B_1 and B_2 in SRM 2387 were measured as part of an interlaboratory intercomparison exercise. Aflatoxins G_1 and G_2 were not measured in the SRM.

- Seven laboratories enrolled in this exercise and received samples; 5 laboratories reported results (71 %).
- All five laboratories that provided data for aflatoxin B_1 performed well.
- All four laboratories that provided data for aflatoxin B_2 performed well.
- The three laboratories that provided data for aflatoxins G_1 and G_2 performed well.
- The total aflatoxins were reported by the five laboratories and all performed well.

Considerations:

- This study did not receive the number of participants we anticipated. What toxins and contaminants are most important for your laboratories?

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Total Aflatoxins in Peanuts

		Control SRM 2387		Peanuts				
	Lab Code	Mean	SD	Sample A	Sample B	Sample C	Mean	SD
Individual Results	E504	5.5	0.3	11.0	11.4	11.1	11.2	0.2
	E510	2.4	0.1	11.8	11.8	11.2	11.6	0.3
	E511	4.1	0.1	8.8	12.5	9.2	10.2	2.0
	E521	5.2	0.2	8.1	7.4	9.8	8.4	1.2
	E523	6.3	0.3	11.7	12.8		12.3	0.8
	E534	5.1	0.4	8.6	7.6	9.0	8.4	0.7
	E537	7.9	0.3	8.3	8.9		8.6	0.4
Community Results						Mean		10.1
						Median		10.2
						Maximum		12.3
						Minimum		8.4
						MADe		1.1
						%RSD		11 %
						N		19

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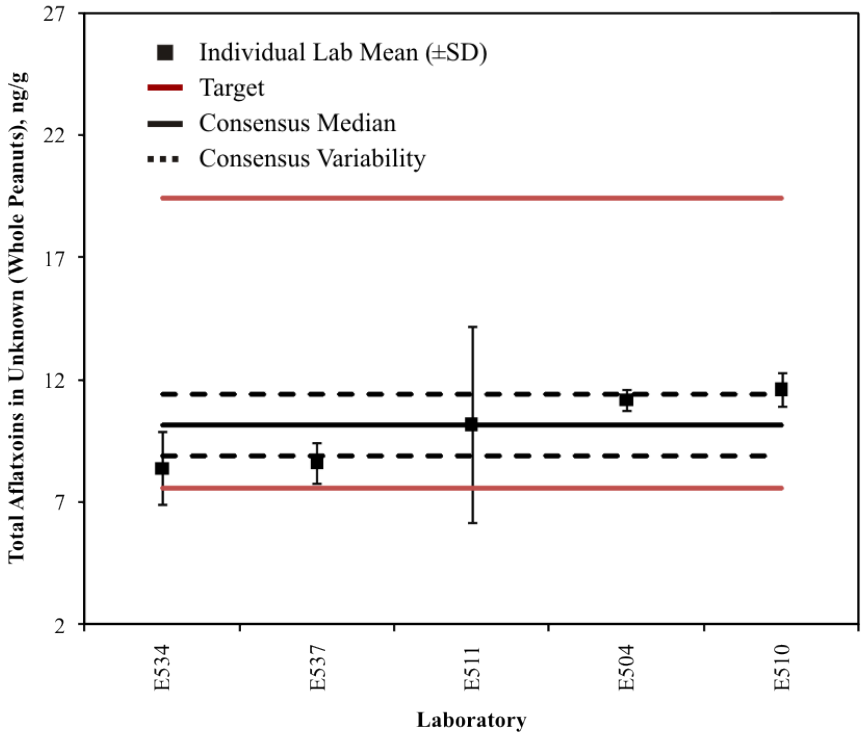
TOTAL AFLATOXINS IN PEANUT PRODUCTS

VIEW 1



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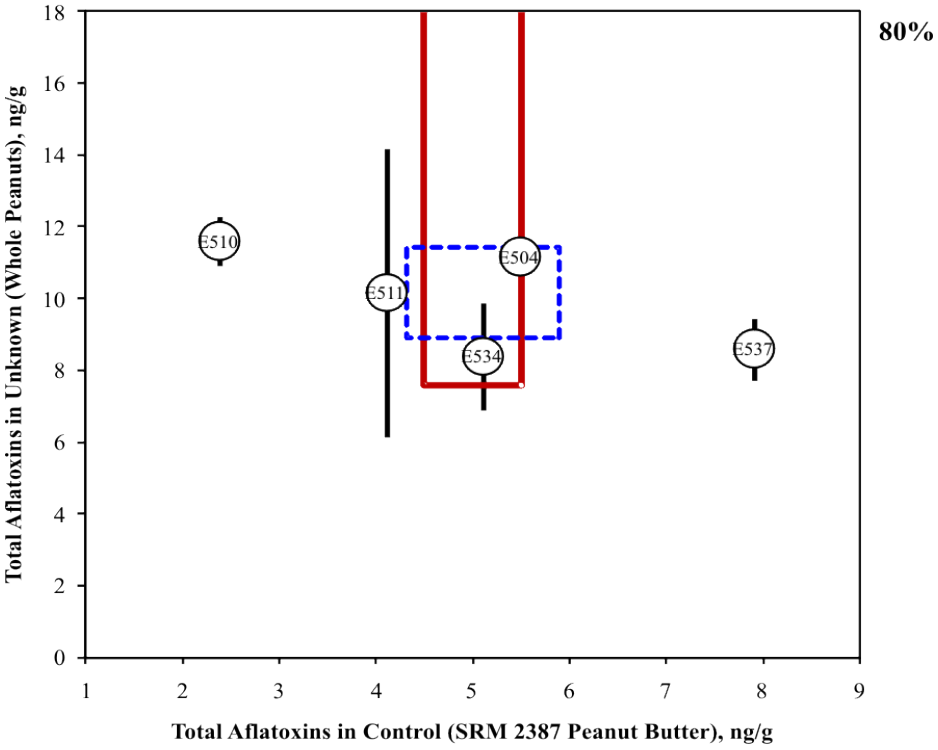

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Aflatoxin B1 in Peanuts

		Control SRM 2387		Peanuts				
Individual Results	Lab Code	Mean	SD	Sample A	Sample B	Sample C	Mean	SD
	E504	4.7	0.2	6.2	6.5	5.7	6.1	0.4
	E510	1.8	0.1	6.5	6.6	6.4	6.5	0.1
	E511	3.5	0.1	3.9	6.6	4.7	5.0	1.4
	E521	4.6	0.2	4.7	4.1	5.7	4.8	0.8
	E523	5.3	0.2	6.3	6.8		6.6	0.4
	E534	4.1	0.3	4.1	3.7	4.3	4.0	0.3
	E537	7.9	0.3	5.6	6.2		5.9	0.4
Community Results						Mean		5.6
						Median		5.9
						Maximum		6.6
						Minimum		4.0
						MADe		0.5
						%RSD		9.2 %
						N		19

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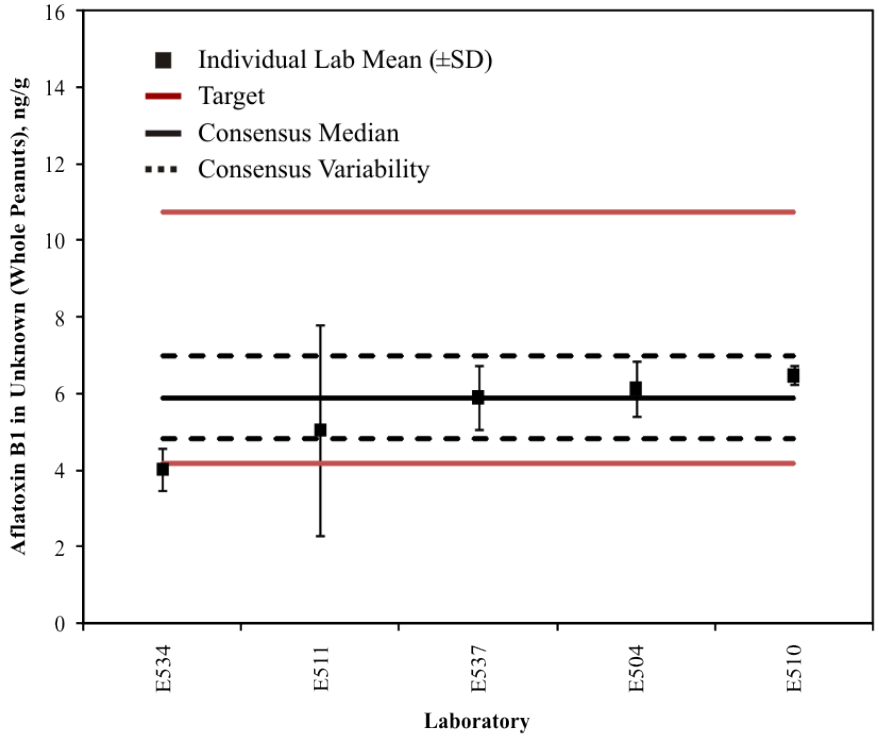
AFLATOXIN B_1 IN PEANUT PRODUCTS

VIEW 1



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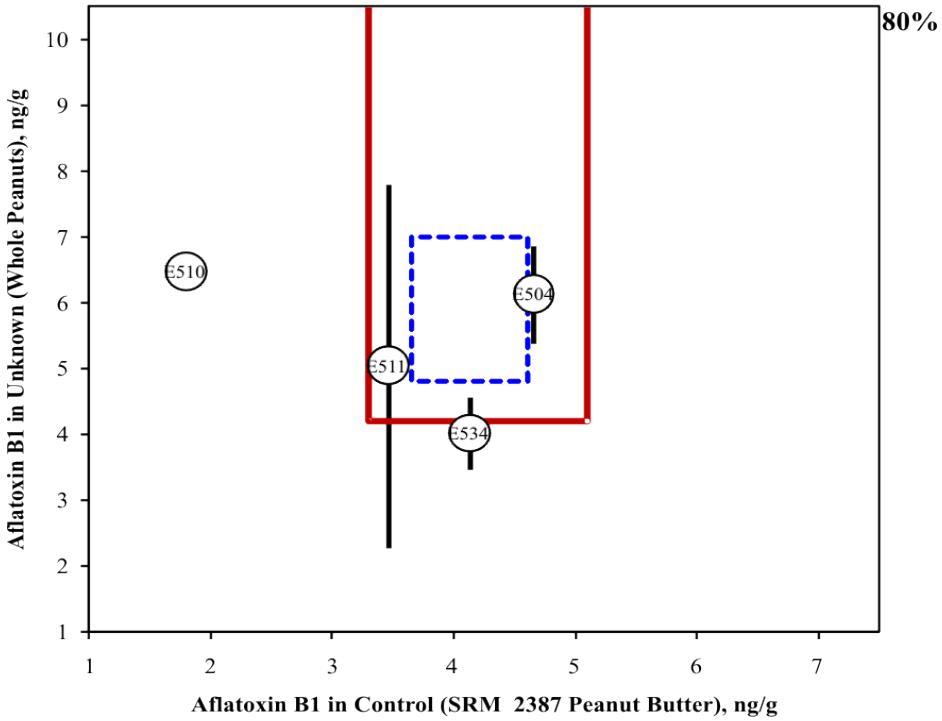

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Aflatoxin B2 in Peanuts

		Control SRM 2387		Peanuts				
Individual Results	Lab Code	Mean	SD	Sample A	Sample B	Sample C	Mean	SD
	E504	0.8	0.1	1.8	1.7	1.8	1.8	0.1
	E510	0.5	0.0	1.6	1.6	1.5	1.6	0.1
	E511	0.6	0.0	1.1	1.8	1.3	1.4	0.3
	E521	0.7	0.0	1.1	1.0	1.3	1.1	0.1
	E523	1.0	0.0	1.6	1.8		1.7	0.1
	E534	0.8	0.1	1.4	1.2	1.6	1.4	0.2
Community Results						Mean		1.5
						Median		1.5
						Maximum		1.8
						Minimum		1.1
						MADe		0.2
						%RSD		14 %
						N		17

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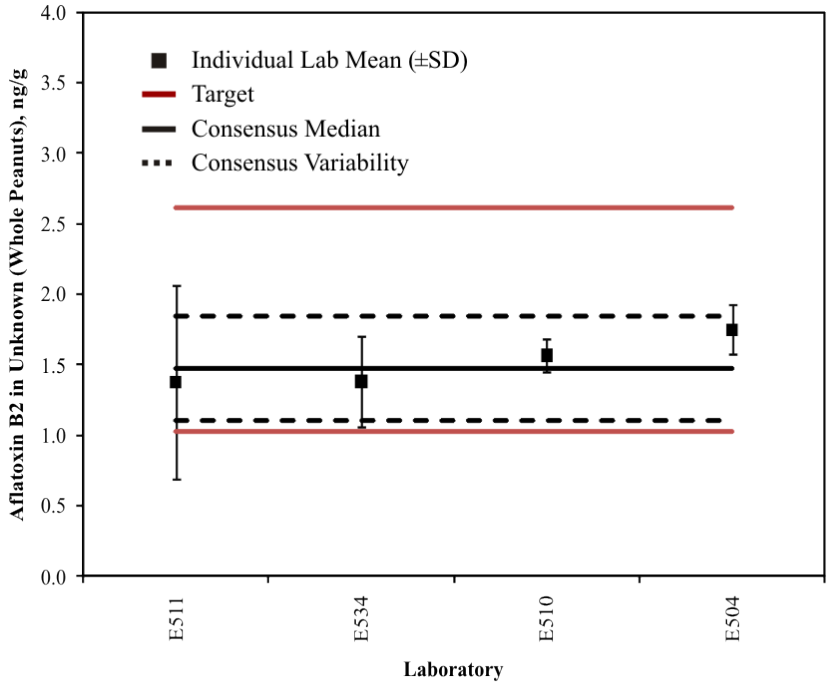
AFLATOXIN B_2 IN PEANUT PRODUCTS

VIEW 1



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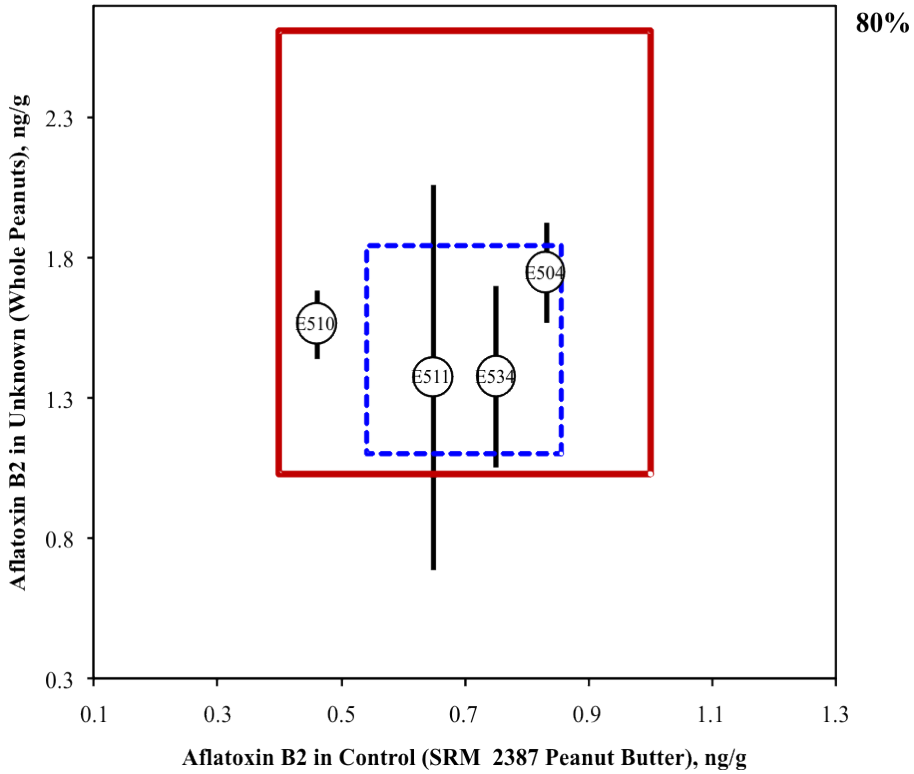

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Aflatoxin G1 in Peanuts

		Control SRM 2387		Peanuts				
Individual Results	Lab Code	Mean	SD	Sample A	Sample B	Sample C	Mean	SD
	E504			1.7	2.0	2.1	1.9	0.2
	E510	0.1	0.0	2.3	2.3	2.1	2.2	0.1
	E521			1.6	1.6	1.9	1.7	0.2
	E523			2.3	2.6		2.5	0.2
	E534	0.2	0.0	1.6	1.4	1.6	1.5	0.1
Community Results						Mean		2.0
						Median		1.9
						Maximum		2.5
						Minimum		1.5
						MADe		0.3
						%RSD		14 %
						N		14

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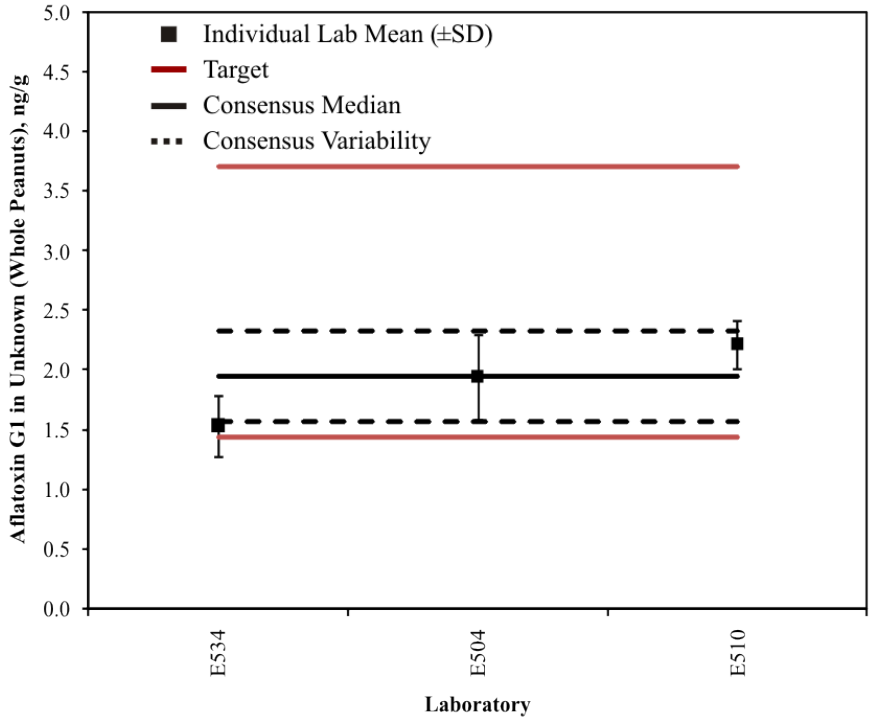
AFLATOXIN G_1 IN PEANUT PRODUCTS

VIEW 1



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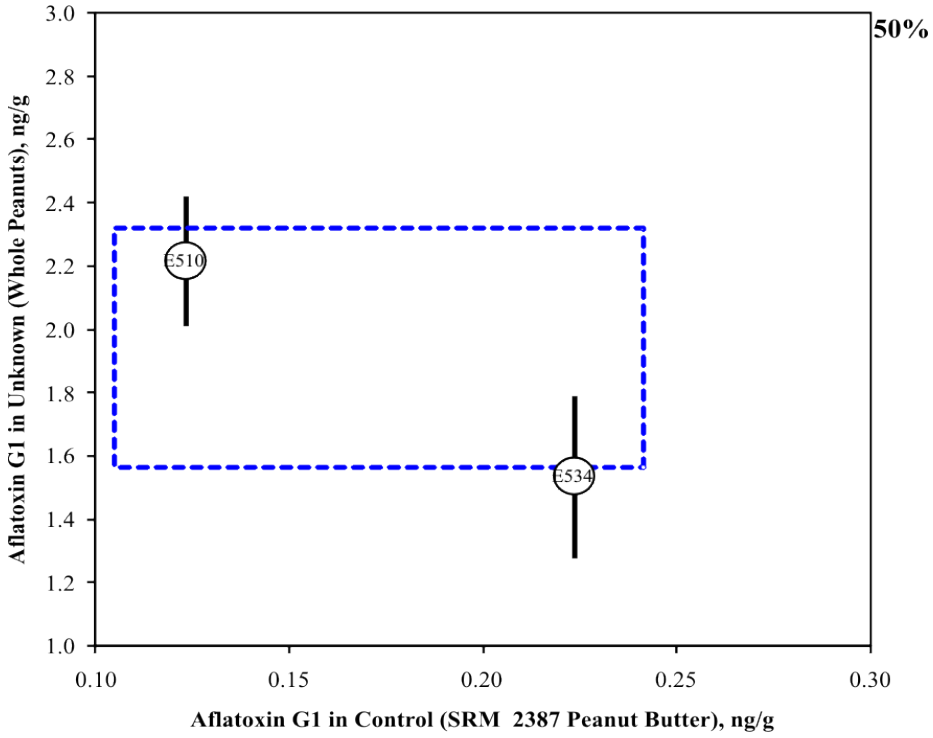

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Aflatoxin G2 in Peanuts

		Control SRM 2387		Peanuts				
Individual Results	Lab Code	Mean	SD	Sample A	Sample B	Sample C	Mean	SD
	E504			1.2	1.3	1.5	1.4	0.1
	E510			1.5	1.3	1.2	1.3	0.1
	E521			0.7	0.7	0.9	0.8	0.1
	E523			1.5	1.6		1.6	0.1
	E534	0.0	0.0	1.6	1.2	1.6	1.4	0.2
Community Results						Mean		1.3
						Median		1.4
						Maximum		1.6
						Minimum		0.8
						MADe		0.2
						%RSD		12.5 %
						N		14

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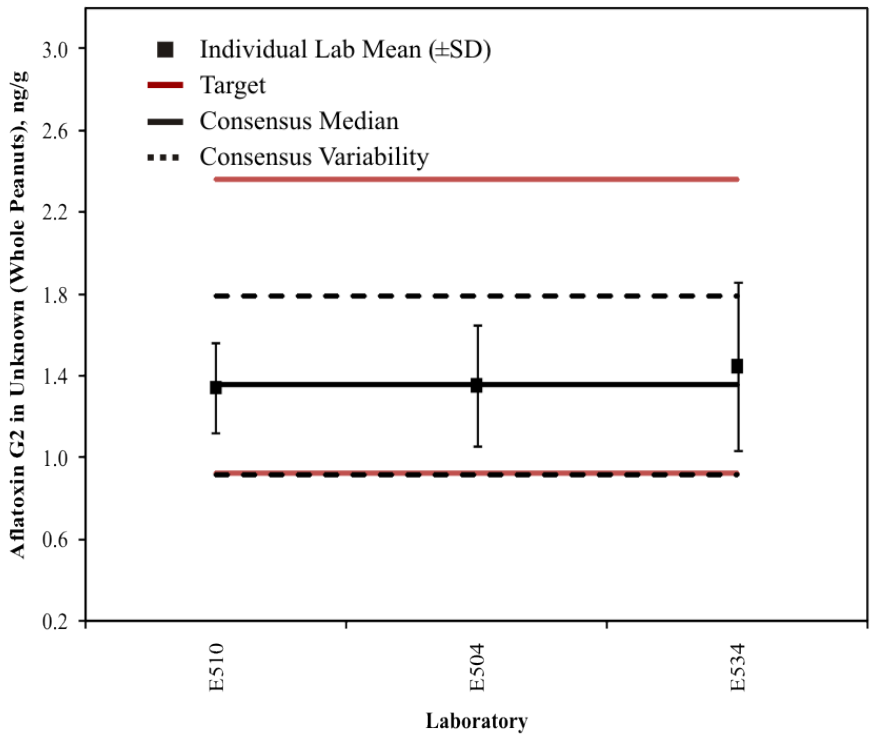
AFLATOXIN G_2 IN PEANUT PRODUCTS

VIEW 1



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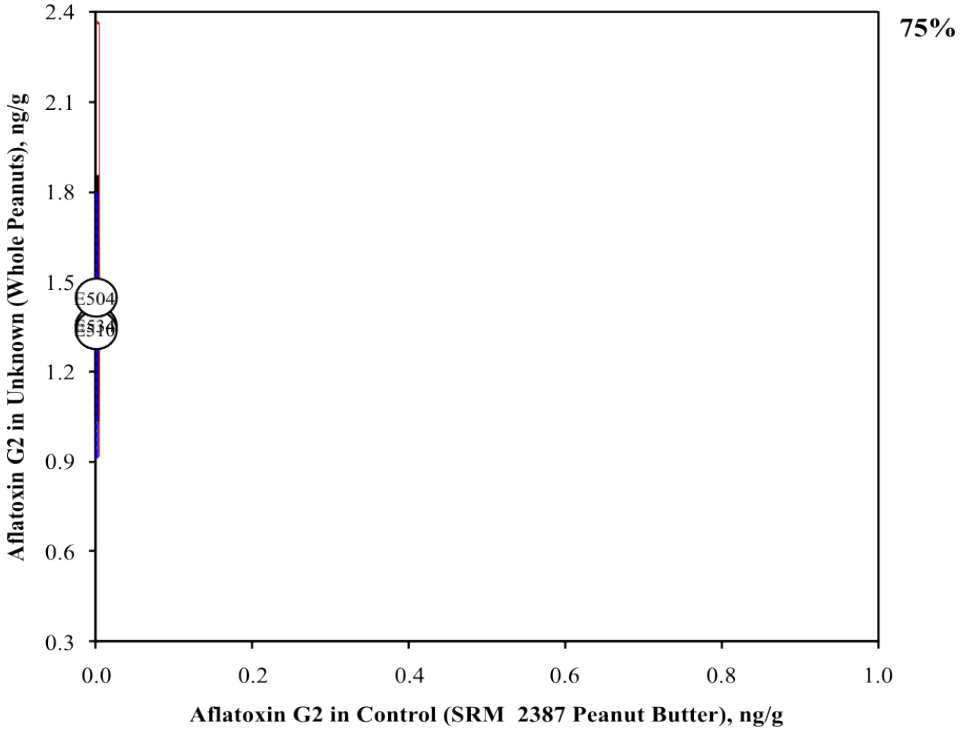

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RECOMMENDATIONS

There were several global issues that we noticed while reviewing the data; please consider these recommendations as they may help improve measurement accuracy and precision.

When a target range is provided for a control, the information should be used as a quality self check. The target value should NOT be used to determine a scaling factor. The matrices are often significantly different between the sample and control. Analytes may be extracted differently from the two materials, thus the adjustment factor for recovery in the control would not be valid for the sample.

Sample cleanup with solid-phase extraction or filters may be useful prior to analysis. However, the cleanup method should always be tested with a calibrant during method development to ensure that the analytes of interest are not binding to the cleanup substrate. Organic analytes are especially likely to bind to nylon or other polymer-based filters.

Evaluation of the purity and stability of calibration materials is very important; this point is highlighted by the measurement of niacinamide in Exercises C, D, and E. There are several factors to consider when evaluating calibration materials:

- Does the labeled purity make sense or are there a number of “extra” peaks in the chromatogram that are unaccounted for?
- Are you storing crystalline materials properly? (are light-sensitive compounds stored away from light? Do you store materials cold or under inert gases? Do you bring them to room temperature in desiccators?)
- How long does your laboratory store calibration materials? Have you evaluated the stability under your storage conditions?

QUESTION

Related to catechins in green tea and also β-carotene, do epimers and isomers matter to your laboratory or is it more important for you to measure the total analyte accurately and precisely?

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