NISTIR 8233

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https://doi.org/10.6028/NIST.IR.8233

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March 2019

U.S. Department of Commerce
Wilbur L. Ross, Jr., Secretary

National Institute of Standards and Technology
Walter Copan, NIST Director and Undersecretary of Commerce for Standards and Technology

ABSTRACT

The Marine Mammal Health and Stranding Response Program, part of the National Oceanic and Atmospheric Administration's office of Protected Resources, requested the National Institute of Standards and Technology (NIST) to investigate the potential for false-positive identification of polycyclic aromatic hydrocarbons (PAHs) in blubber sampled from marine mammals. Such samples can contain total concentrations of persistent organic pollutants (POPs) in the mg/kg range that could potentially interfere with measurement and identification of PAHs. The major goals for this study were to (1) determine if laboratories can quantify PAHs amended to blubber samples containing typical POP levels, and (2) determine if false-positive detections of PAHs commonly occur in such samples. Five participating laboratories were each provided with five samples to measure PAHs according to their standard protocols. Samples represented PAH-amended and unamended extracts from high and low POP marine mammal blubber, respectively, and a solution containing only PAH compounds. Results from this study indicate (1) PAHs were able to be differentiated from POPs co-occurring in the sample, and (2) false-positive detections for PAHs were rare and at or near the reporting limit.

Introduction

Polycyclic aromatic hydrocarbons (PAHs) have two or more fused aromatic rings with a pair of carbon atoms shared between rings. Some PAHs occur naturally in coal, crude oil, and gasoline, or can originate from natural sources such as burning, natural seeps of petroleum or coal, and volcanic activities. PAHs are found in the ambient air in the gas-phase and sorbed to aerosols. Major anthropogenic sources of PAHs include combustion and industrial activities especially those related to refining and use of petroleum products. PAHs are also a component in marine petroleum spills and exert toxic impacts on protected species such as marine mammals through inhalation, dermal adsorption, or ingestion of contaminated food. The degree of alkylation of PAHs and other aspects have allowed PAHs to be used as indicators of petroleum spills in the marine environment.

The request from NOAA's Marine Mammal Health and Stranding Program (MMHSRP) focused on potential false-positive detection of PAHs in marine mammal blubber during routine analysis to support their activities. Marine mammal blubber often has high (mg/kg) concentrations of persistent organic pollutants (POPs; mainly organochlorine pesticides and polychlorinated biphenyls) that may interfere with measurement of PAHs by gas chromatography unit resolution mass spectrometry (GC-MS). In addition, higher vertebrates are adept at metabolizing and excreting PAHs, making recent reports of PAHs in marine mammal tissues questionable. The current study was designed to address possible misidentification and therefore misreporting of PAHs in the presence of other POPs. The goals were twofold: (1) can participating laboratories differentiate PAHs from a POP-dominated background in marine mammal blubber samples; and

(2) do POPs in marine mammal blubber result in false-positive detection of PAHs. Results from this study will help inform the MMHSRP regarding the validity of using PAH data derived from the analysis of marine mammal blubber.

Methods and Calculations:

Sample Preparation

SRM 1945 "Organics in Whale Blubber" and Control Material IV (CM-IV; Kucklick et al. 2010) were chosen for this work, both representing cryohomogenized blubber collected from stranded long finned pilot whales (*Globicephala melas*). SRM 1945 represented a "low" POP background while Control Material IV represented a "high" POP background containing approximately 3 mg/kg and 60 mg/kg total organohalogen compounds, respectively (see Kucklick et al. 2010). Ten aliquots (ca. 3 g each) of each material were extracted by pressurized fluid extraction (ASE; Dionex, Salt Lake City, UT) each yielding approximately 60 mL of extract in dichloromethane (DCM). Extraction conditions were as follows, repeated for three cycles (one-third of the solvent each time): cell temperature 100 °C, equilibration 5 min, static time 5 min, cell pressure was 13.8 MPa. Pooled 600 mL (10 x 60 mL) extracts were concentrated to approximately 40 mL in a Turbovap (Biotage, Charlotte, NC). Total extractable organics (TEO; mainly lipids) were determined by subsampling the extract (by mass), evaporating the solvent, and weighing the residue. TEO was 21.7 % (mass fraction) for the SRM 1945 extract and 20.3 % (mass fraction) for CM-IV extract.

Extracts were divided into two 20 mL aliquots of solution (mass known). Aliquots were diluted with DCM to a final volume of 40 mL, yielding two 40 mL aliquots for both SRM 1945 and CM-IV solutions. One extract each of SRM 1945 and CM-IV were amended with 1.5 mL of SRM 1491a "Methyl-Substituted Polycyclic Aromatic Hydrocarbons in Toluene" and 0.5 mL of SRM 2260a "Aromatic Hydrocarbons in Toluene" (Table 2). This resulted in a suite of four blubber extracts, one with high POPs background amended with PAHs, one with high POPs background and no PAHs, one with low POPs background amended with PAHs, and one with low POPs background and no PAHs. A fifth solution containing only PAHs, absent any matrix effects and other POPs, was created (hereafter called Control Solution) by amending 40 mL of toluene with 2 mL of SRM 1491a and 0.5 mL of SRM 2260a. Each solution was vortexed and separated into 13 aliquots of approximately 3 mL each (mass known). Extracts and control were pipetted into 8 mL hexane rinsed vials. Once all extracts were aliquoted, they were stored at -20 °C until use or shipment to participating laboratories.

Expected values for concentrations of PAHs in the resulting Control Solution and blubber extracts were calculated using the certificate of analysis mass fraction (ng/g) times the mass of the material used and divided by the mass of the total solution created (Table 1 and Table 2). Mean and standard deviation (SD) of reported measurements were calculated for each analyte by

extract type and laboratory. Results were visualized for each extract type (Control, Spiked, and Unspiked) in a manner similar to the classic z-score framework. By doing so, each value was standardized to the calculated expected value instead of self-referencing to observations. Expected values of each analyte were subtracted from the mean reported value and the difference divided by the standard uncertainty (U) of the expected value, resulting in an expression of how many standard uncertainties from the expected value was the mean reported value. This allows visualization of all PAH analytes in the same space, resulting in a graphical comparison of measurements across laboratories for all analytes (Figures 1-3) for a given extract type. Laboratory ID Code is plotted into this space, and boxplots representing all measurement means per analyte are overlaid for reference across participating laboratories. For the Control Solution and amended blubber extracts, the horizontal line around zero represents the expected value +/- 3U. For the unamended extracts, the solid line at zero represents the mean reported detection limit, with framing dashed lines representing +/- 3 standard deviations; only detected analytes are presented. Tables providing mean and standard deviation of values measured by each lab (Table 3) and across labs for each solution (Tables 4-6) are provided.

Results and Discussion:

Five laboratories participated in the exercise, contributing a total of six data sets. Analytes targeted by the study were not always reported by every participant due to differences in analytical protocols. Within lab measurement variability is reported as percent relative standard deviation (% RSD). In extracts where PAHs were present, 21 of 25 PAHs exceeded an RSD of 10% for within laboratory measurements for at least one extract (Table 3). All measurements from Lab 5 (18 analytes reported) were within 10% RSD. By this metric, Lab 5 demonstrated the most consistent measurement set, while lab 4 demonstrated the least precision among participants. Analytes commonly exceeding 10% RSD within laboratories were dominated by low molecular weight methylated PAHs (Table 3). Analytes exceeding 30% RSD within laboratories were uncommon and included anthracene, benzo[*k*]fluoranthene, chrysene + triphenylene, and phenanthrene. Coelution of benzo[*j*]fluoranthene with benzo[*k*]fluoranthene and of chrysene with triphenylene are known common issues that may contribute to imprecise reporting.

The precision of mean measurements across laboratories (Tables 2-4) indicated that there was general agreement among compounds identified. Mean reported measurements among participating laboratories are presented graphically relative to expected values in Figures 1-3 (see methods), demonstrating the lack of agreement between laboratories when scaled to the expected uncertainty range of the provided extracts. Analyte-wise z-score and accuracy plots were also produced (Appendix D) across laboratories for each extract; the limited number of participating laboratories in the current study poses a challenge for interpreting z-scores with

such varied data, so a comparison against expected values is also provided; Figures 1-3 should be read as extract-wise summaries of Appendix D.

Participating laboratories were able to differentiate PAHs from the POP-dominated background in blubber. Measurement of PAHs in POPs-dominated samples may include infrequent false positives (Figure 3, Table 6), but false positives observed in the present study were close to the reporting limit and may arise from low-level laboratory contamination. Common sources of PAH contamination include laboratory evaporation gases, solvents and those that may arise when processing samples in laboratories with high ambient air concentrations of PAHs. In this study, false positives were reported for anthracene, benzo[e]pyrene, perylene, and pyrene (Figure 3, Table 6). Lab 6 reported phenanthrene in a single replicate in Extract 2 (SRM1945 unamended) and anthracene and pyrene in Extract 4 (CM-IV, unspiked). Lab 4 reported benzo[*e*]pyrene and perylene in Extract 4 (CM-IV, unspiked). Laboratories 1, 2, 3 and 5 did not report PAHs in the unspiked extracts. From this study, it is not possible to tell if PAH detections in the unspiked extracts originated from interferences from POPs or from laboratory contamination. It is possible that laboratories not reporting PAHs had better resolution of PAHs from POPs that were also in the sample. This is suggested by the results from laboratory 4 where more PAHs were detected in the extract with the higher concentration of POPs than in the extract derived from SRM 1945. For laboratory 6, the PAH detected in the highest concentration (phenanthrene) in unspiked extracts was observed in Extract 2 (SRM 1945). If POP interference caused this detection then this compound should have also been detected in Extract 4, as POP concentrations are much greater in CM-IV. Consequently, this detection suggests that laboratory contamination may have resulted in the detection. We recommend that laboratories analyzing PAHs in marine mammal blubber use GC columns that are specific for PAH analysis (e.g. 50% phenyl dimethylpolysiloxane columns) and run adequate blanks during sample processing to monitor for any potential laboratory contamination during sample preparation and clean up.

Conclusions:

Results from control samples demonstrated that participating laboratories can identify PAHs by GC-MS in marine mammal blubber even in the presence of high concentrations of POPs (Figure 1, Table 4). Despite using several different clean up and instrumental methods, participating laboratories were able to identify and quantify the amended PAHs in the low and high POP background samples (Figure 2, Table 5a,b). Quantification of several low-molecular weight and alkylated PAHs (e.g. methyl-phenanthrene and methyl-anthracene) was more variable than expected. Higher relative variability may be due to the loss of more volatile lower molecular weight PAHs during sample preparation. Generally, PAHs in the unamended samples (extracts 2 and 4) were below the reporting limits of most laboratories with the exceptions discussed above.

Taken in context, participating laboratories were able to differentiate PAHs from a POPs-dominated background in marine mammal blubber extracts and to quantify PAH concentrations. Investigation of possible reasons for a general lack of agreement for quantitation results of certain analytes was beyond the scope of the current study.

Disclaimer: Certain commercial equipment, instruments, or materials are identified in this paper to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

References:

Kucklick, J. R., Schantz, M. M., Pugh, R. S., Porter, B. J., Poster, D. L., Becker, P. R., Rowles, T. K., Leigh, S., and Wise, S. A., ***"Marine mammal blubber reference and control materials for use in the determination of halogenated organic compounds and fatty acids,"*** Anal. Bioanal. Chem., 397, 423-432 (2010).

List of Analytes of Interest in the Interlaboratory Analytical Comparison Study

Parent PAHs:

anthracene
benzo[*a*]fluoranthene
benzo[*a*]pyrene
benzo[*b*]fluoranthene
benzo[*e*]pyrene
benzo[*ghi*]perylene
benzo[*j*]fluoranthene
benzo[*k*]fluoranthene
chrysene
dibenz[*a,h*]anthracnene
fluoranthene
indeno[1,2,3-*cd*]pyrene
perylene
phenanthrene
pyrene
triphenylene

Alkylated PAHs:

1-methylphenanthrene
1,7-dimethylphenanthrene
2-methylanthracene
2-methylphenanthrene
3-methylphenanthrene
4H-cyclopenta[*def*]phenanthrene
9-methylphenanthrene

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Table 1. Mass of SRMs spiked into extracts

	1.5 mL SRM 1941a	0.5mL SRM 2260	2.0 mL SRM 1941a
Extract 1 SRM1945	1.26917g	0.43838 g	
Extract 3 CMIV	1.28372 g	0.43079 g	
Control		0.44209g	1.72437 g

Table 2. Certified mass fractions for SRM 1491a and 2260a and calculated expected values of PAH components in extracts sent to participating laboratories

SRM	Compound	Mass Fraction (µg/g)			ng/g (mass frac * 1000)			Control (ng/g)			1945 spiked (ng/g)			CMIV Spiked (ng/g)		
1491a	1-methylphenanthrene	2.24	+/-	0.03	2243	+/-	28	131.3	+/-	1.64	58.28	+/-	0.73	58.50	+/-	0.73
1491a	1,7-dimethylphenanthrene	1.96	+/-	0.03	1962	+/-	27	114.8	+/-	1.58	50.98	+/-	0.70	51.17	+/-	0.70
1491a	2-methylanthracene	1.36	+/-	0.01	1355	+/-	10	79.32	+/-	0.59	35.21	+/-	0.26	35.34	+/-	0.26
1491a	2-methylphenanthrene	2.40	+/-	0.02	2396	+/-	18	140.3	+/-	1.05	62.25	+/-	0.47	62.49	+/-	0.47
1491a	3-methylphenanthrene	2.13	+/-	0.01	2134	+/-	10	124.9	+/-	0.59	55.45	+/-	0.26	55.66	+/-	0.26
2260a	4H-cyclopenta[*def*]phenanthrene	2.32	+/-	0.11	2320	+/-	110	34.82	+/-	1.65	20.82	+/-	0.99	20.31	+/-	0.96
1491a	9-methylphenanthrene	2.29	+/-	0.02	2288	+/-	19	133.9	+/-	1.11	59.45	+/-	0.49	59.68	+/-	0.50
2260a	anthracene	3.74	+/-	0.05	3736	+/-	54	56.07	+/-	0.81	33.53	+/-	0.48	32.70	+/-	0.47
2260a	benzo[*a*]fluoranthene	2.28	+/-	0.06	2279	+/-	64	34.20	+/-	0.96	20.45	+/-	0.57	19.95	+/-	0.56
2260a	benzo[*a*]pyrene	4.71	+/-	0.17	4710	+/-	170	70.69	+/-	2.55	42.27	+/-	1.53	41.23	+/-	1.49
2260a	benzo[*b*]fluoranthene	7.86	+/-	0.10	7860	+/-	100	118.0	+/-	1.50	70.54	+/-	0.90	68.80	+/-	0.88
2260a	benzo[*e*]pyrene	4.56	+/-	0.05	4561	+/-	54	68.45	+/-	0.81	40.93	+/-	0.48	39.92	+/-	0.47
2260a	benzo[*ghi*]perylene	5.67	+/-	0.07	5669	+/-	69	85.08	+/-	1.04	50.88	+/-	0.62	49.62	+/-	0.60
2260a	benzo[*j*]fluoranthene	4.15	+/-	0.10	4145	+/-	97	62.21	+/-	1.46	37.20	+/-	0.87	36.28	+/-	0.85
2260a	benzo[*k*]fluoranthene	3.44	+/-	0.04	3444	+/-	36	51.69	+/-	0.54	30.91	+/-	0.32	30.14	+/-	0.32
2260a	chrysene	4.62	+/-	0.11	4620	+/-	110	69.33	+/-	1.65	41.46	+/-	0.99	40.44	+/-	0.96
2260a	dibenz[*a,h*]anthracnene	4.56	+/-	0.06	4555	+/-	63	68.36	+/-	0.95	40.88	+/-	0.57	39.87	+/-	0.55
2260a	fluoranthene	8.32	+/-	0.09	8324	+/-	87	124.9	+/-	1.31	74.70	+/-	0.78	72.86	+/-	0.76
2260a	indeno[1,2,3-*cd*]pyrene	4.43	+/-	0.03	4425	+/-	30	66.41	+/-	0.45	39.71	+/-	0.27	38.73	+/-	0.26
2260a	perylene	4.43	+/-	0.05	4430	+/-	45	66.48	+/-	0.68	39.76	+/-	0.40	38.77	+/-	0.39
2260a	phenanthrene	11.6	+/-	0.12	11570	+/-	120	173.6	+/-	1.80	103.8	+/-	1.08	101.3	+/-	1.05
2260a	pyrene	8.95	+/-	0.08	8949	+/-	83	134.3	+/-	1.25	80.31	+/-	0.74	78.33	+/-	0.73
2260a	triphenylene	4.12	+/-	0.16	4120	+/-	160	61.83	+/-	2.40	36.97	+/-	1.44	36.06	+/-	1.40
	chrysene + triphenylene	8.74	+/-	0.27	8740	+/-	270.00	131.2	+/-	4.05	78.44	+/-	2.42	76.50	+/-	2.36
	benzo[*k*]fluoranthene + benzo[*j*]fluoranthene	7.59	+/-	0.13	7589	+/-	133.00	113.9	+/-	2.00	68.11	+/-	1.19	66.42	+/-	1.16

Table 3. Summary of results as reported by participating laboratories

a. **Results reported by Participating Laboratory 1**

	Control			Extract 1			Extract 2			Extract 3			Extract 4		
	ng/g			ng/g			ng/g			ng/g			ng/g		
Analyte	AVE	STD	RSD%	AVE	STD	RSD%	AVE	STD	RSD%	AVE	STD	RSD%	AVE	STD	RSD%
1-methylphenanthrene	59.0	6.24	10.6	39.9	1.31	3.29	< 4.74			36.6	4.10	11.2	< 4.69		
1,7-dimethylphenanthrene	54.3	0.58	1.06	28.2	0.40	1.42	< 10.0			28.0	0.38	1.35	< 9.9		
2-methylanthracene	25.2	3.82	15.2	25.0	1.16	4.63	< 7.83			22.2	3.22	14.5	< 7.75		
2-methylphenanthrene	54.7	8.08	14.8	41.0	1.74	4.24	< 5.62			37.3	5.75	15.4	< 5.56		
3-methylphenanthrene	59.7	9.07	15.2	46.0	2.40	5.22	< 4.23			42.1	7.34	17.4	< 4.18		
4H-cyclopenta[*def*]phenanthrene	22.1	2.32	10.5	21.7	1.31	6.04	< 1.07			18.8	2.50	13.3	< 1.06		
9-methylphenanthrene	64.3	7.64	11.9	44.8	1.86	4.15	< 5.29			41.1	5.45	13.3	< 5.23		
anthracene	53.7	7.36	13.7	31.5	0.93	2.95	< 1.72			28.6	2.24	7.81	< 1.70		
benzo[*a*]fluoranthene	33.0	1.55	2.60	39.6	0.56	1.41	< 1.05			38.2	0.80	2.10	< 1.04		
benzo[*a*]pyrene	70.9	0.87	2.64	21.1	0.81	3.84	< 2.48			19.8	1.08	5.43	< 2.45		
benzo[*b*]fluoranthene	119	0.62	0.88	44.7	1.56	3.49	< 3.97			42.6	0.32	0.75	< 3.93		
benzo[*e*]pyrene	71.7	0.58	0.48	77.8	1.42	1.82	< 2.10			76.8	1.52	1.98	< 2.08		
benzo[*ghi*]perylene	90.7	1.15	1.61	45.2	0.85	1.88	< 2.61			44.8	0.62	1.39	< 2.58		
benzo[*j*]fluoranthene	59.8	2.10	2.32	59.4	1.65	2.78	< 1.58			55.7	1.00	1.80	< 1.57		
benzo[*k]* fluoranthene	57.0	0.80	1.41	37.5	0.49	1.31	< 1.87			36.4	0.85	2.33	< 1.85		
chrysene	71.6	5.97	8.34	27.6	1.31	4.72	< 2.13			27.6	3.36	12.2	< 2.10		
dibenz[*a,h*]anthracnene	74.5	1.41	1.89	46.6	1.67	3.57	< 10.7			44.3	0.60	1.36	< 10.6		
fluoranthene	118	1.53	1.30	76.4	1.72	2.26	< 3.83			72.4	0.78	1.08	< 3.79		
indeno[1,2,3-*cd*]pyrene	69.5	0.96	1.39	43.7	1.15	2.64	< 2.04			41.3	0.71	1.72	< 2.01		
perylene	67.1	1.91	2.85	44.2	1.34	3.04	< 2.04			41.5	0.45	1.09	< 2.02		
phenanthrene	110	2.65	2.41	66.0	1.73	2.62	< 5.32			65.3	3.79	5.79	< 5.27		
pyrene	129	1.53	1.19	83.2	1.78	2.14	< 4.12			79.8	1.04	1.30	< 4.07		
triphenylene	42.7	2.91	6.82	36.1	1.53	4.23	< 1.90			32.5	1.73	5.34	< 1.88		

b. Results reported by Participating Laboratory 2

	Control			Extract 1			Extract 2			Extract 3			Extract 4		
	ng/g			ng/g			ng/g			ng/g			ng/g		
Analyte	AVE	STD	RSD%	AVE	STD	RSD%	AVE	STD	RSD%	AVE	STD	RSD%	AVE	STD	RSD%
1-methylphenanthrene	70.2	7.66	10.9	46.1	2.97	6.44	< 4.31			41.8	1.99	4.75	< 4.26		
1,7-dimethylphenanthrene	50.7	2.22	4.39	24.0	1.04	4.35	< 3.26			24.1	0.92	3.84	< 3.22		
2-methylanthracene	30.3	3.54	11.7	24.6	3.46	14.0	< 2.25			20.5	1.70	8.32	< 2.23		
2-methylphenanthrene	78.0	10.18	13.1	51.9	2.92	5.62	< 5.24			45.4	3.19	7.03	< 5.19		
3-methylphenanthrene	119.3	15.18	12.7	82.0	6.04	7.36	< 10.8			77.1	5.30	6.88	< 10.7		
4H-cyclopenta[*def*]phenanthrene	31.6	2.44	7.72	24.4	1.29	5.28	< 1.07			23.2	2.63	11.3	< 1.06		
9-methylphenanthrene	73.5	7.89	10.7	48.3	2.99	6.19	< 9.53			43.7	2.35	5.36	< 9.43		
anthracene	46.4	3.05	6.58	27.7	0.97	3.51	< 1.72			24.6	2.44	9.94	< 1.70		
benzo[*a*]fluoranthene	74.4	0.46	1.26	22.0	1.48	6.74	< 1.95			19.7	1.05	5.34	< 1.93		
benzo[*a*]pyrene	127	1.46	1.96	48.4	2.25	4.65	< 2.17			45.5	1.38	3.03	< 2.14		
benzo[*b*]fluoranthene	70.8	0.85	0.67	83.4	3.56	4.28	< 3.62			81.1	1.43	1.76	< 3.58		
benzo[*e*]pyrene	94.4	0.90	1.27	45.9	1.84	4.00	< 2.10			44.8	1.30	2.90	< 2.08		
benzo[*ghi*]perylene	64.1	1.46	1.54	61.0	2.76	4.52	< 6.84			57.0	1.53	2.69	< 6.77		
benzo[*j*]fluoranthene	36.4	0.66	1.02	42.1	1.66	3.95	< 1.91			40.8	0.84	2.05	< 1.89		
benzo[*k*]fluoranthene	53.1	1.26	2.37	34.8	1.13	3.24	< 1.58			33.5	1.00	2.99	< 1.57		
chrysene + triphenylene	141	1.87	1.33	90.5	3.70	4.09	< 4.02			83.9	2.81	3.35	< 3.98		
dibenz[*a,h*]anthracnene	97.5	5.31	5.45	33.6	1.22	3.63	< 2.10			34.2	4.98	14.6	< 2.07		
fluoranthene	74.5	1.87	2.51	49.0	2.19	4.48	< 10.4			45.6	1.35	2.95	< 10.3		
indeno[1,2,3-*cd*]pyrene	129	1.74	1.35	83.3	3.06	3.67	< 2.04			77.8	2.69	3.45	< 2.01		
perylene	61.1	2.60	4.26	40.3	2.08	5.18	< 2.04			38.6	1.07	2.77	< 2.02		
phenanthrene	69.6	2.46	3.54	45.9	2.06	4.50	< 5.32			42.6	0.95	2.24	< 5.27		
pyrene	181	1.15	0.64	116	2.81	2.42	< 4.12			110	5.49	5.00	< 4.07		

c. Results reported by Participating Laboratory 3

	Control			Extract 1			Extract 2			Extract 3			Extract 4		
	ng/g			ng/g			ng/g			ng/g			ng/g		
Analyte	AVE	STD	RSD%	AVE	STD	RSD%	AVE	STD	RSD%	AVE	STD	RSD%	AVE	STD	RSD%
1-methylphenanthrene	86.5	11.0	12.7	84.4	3.16	3.74	<0.38			70.4	9.11	13.0	<0.71		
2-methylanthracene	38.8	10.5	27.1	25.9	1.14	4.39	<0.49			21.9	5.27	24.1	<2.50		
2-methylphenanthrene	62.5	5.94	9.50	82.5	3.50	4.24	<2.50			66.4	10.0	15.0	<2.51		
3-methylphenanthrene	108	1.51	1.40	118	4.46	3.79	<0.50			97.4	13.7	14.1	<0.86		
4H-cyclopenta[*def*]phenanthrene	24.3	0.34	1.42	33.3	1.45	4.36	<0.50			31.4	0.85	2.71	<0.97		
9-methylphenanthrene	76.4	6.16	8.06	84.4	3.76	4.45	<0.45			70.8	10.2	14.3	<0.85		
anthracene	38.4	3.75	9.76	35.5	1.51	4.26	<0.22			33.4	0.52	1.55	<0.43		
benzo[*a*]fluoranthene	38.6	0.28	1.40	20.7	0.57	2.73	<1.28			19.5	0.33	1.69	<2.15		
benzo[*a*]pyrene	67.5	0.27	0.70	40.8	0.64	1.58	<0.61			40.1	0.42	1.05	<1.06		
benzo[*b*]fluoranthene	36.1	1.06	1.57	71.2	1.16	1.62	<1.45			69.3	0.81	1.17	<2.45		
benzo[*e*]pyrene	44.7	6.45	17.9	43.1	0.77	1.79	<0.43			42.1	0.67	1.60	<0.73		
benzo[*ghi*]perylene	39.7	1.91	4.28	46.5	1.60	3.44	<0.30			43.7	0.37	0.85	<0.48		
benzo[*j*]fluoranthene	19.7	0.22	0.54	43.7	0.67	1.54	<0.25			43.1	0.44	1.03	<0.42		
benzo[*k*] fluoranthene	27.5	1.77	6.44	31.2	0.31	0.99	<0.58			30.2	0.76	2.51	<0.98		
chrysene + triphenylene	81.2	1.85	2.28	85.4	1.39	1.63	<0.36			82.9	0.88	1.06	<0.67		
dibenz[*a,h*]anthracnene	33.7	8.80	26.1	36.1	0.54	1.49	<0.60			33.8	0.58	1.72	<0.60		
fluoranthene	75.3	2.45	3.25	76.2	1.35	1.77	<0.74			72.9	1.36	1.87	<1.00		
indeno[1,2,3-*cd*]pyrene	33.0	9.35	28.3	32.2	1.51	4.69	<0.41			31.6	1.10	3.47	<0.65		
perylene	33.6	0.50	1.49	37.1	0.58	1.57	<0.60			36.0	0.32	0.90	<0.78		
phenanthrene	112	3.55	3.18	112	2.11	1.89	<1.81			99.2	16.3	16.4	<3.37		
pyrene	78.3	5.04	6.44	83.4	1.13	1.36	<0.24			78.7	1.21	1.54	<0.60		

d. Results reported by Participating Laboratory 4

	Control			Extract 1			Extract 2			Extract 3			Extract 4		
	ng/g			ng/g			ng/g			ng/g			ng/g		
Analyte	AVE	STD	RSD%	AVE	STD	RSD%	AVE	STD	RSD%	AVE	STD	RSD%	AVE	STD	RSD%
1-methylphenanthrene	27.7	3.25	11.7	27.1	0.75	2.77	DL			27.6	1.84	6.68	DL		
1,7-dimethylphenanthrene	28.6	3.66	12.8	30.4	1.00	3.29	DL			31.7	0.81	2.56	DL		
2-methylanthracene	46.4	7.81	16.8	53.1	7.23	13.6	DL			43.6	12.1	27.8	DL		
2-methylphenanthrene															
3-methylphenanthrene	45.1	4.41	9.78	26.3	2.26	8.61	DL			30.9	4.67	15.1	DL		
4H-cyclopenta[*def*]phenanthrene															
9-methylphenanthrene	39.7	3.44	8.67	27.1	5.59	20.6	DL			25.8	2.92	11.3	DL		
anthracene	31.1	4.63	14.9	29.7	9.82	33.1	DL			40.6	2.14	5.28	DL		
benzo[*a*]fluoranthene															
benzo[*a*]pyrene	31.6	4.76	15.1	15.7	3.70	23.7	DL			18.3	1.65	9.02	DL		
benzo[*b*]fluoranthene	45.0	9.47	21.1	46.8	2.94	6.30	DL			48.1	9.08	18.9	DL		
benzo[*e*]pyrene	30.8	2.77	8.99	16.1	2.89	18.0	DL			23.6	3.10	13.1	24.7	5.75	23.3
benzo[*ghi*]perylene	41.1	1.66	4.04	13.0	2.29	17.6	DL			6.5		*	DL		
benzo[*j*]fluoranthene															
benzo[*k]* fluoranthene	31.3	13.8	43.9	17.4	8.05	46.4	DL			30.1	17.0	56.5	DL		
chrysene + triphenylene	49.6	16.4	33.1	27.4	4.13	15.1	DL			29.3	3.52	12.0	DL		
dibenz[*a,h*]anthracnene	50.6	2.86	5.66	27.0	4.09	15.2	DL						DL		
fluoranthene	59.6	2.68	4.49	65.9	4.51	6.85	DL			69.4	2.17	3.12	DL		
indeno[1,2,3-*cd*]pyrene	43.2	1.08	2.51	14.4	1.69	11.7	DL						DL		
perylene	35.6	0.66	1.86	18.0	2.35	13.1	DL			15.5		*	8.46		*
phenanthrene	65.0	7.01	10.8	78.4	2.20	2.80	DL			88.7	4.77	5.38	DL		
pyrene	50.8	4.43	8.72	65.7	7.14	10.9	DL			72.7	7.55	10.4	11.6	2.60	22.3

DL-value below detection limit; * - reported only one detect of three replicate samples

e. Results reported by Participating Laboratory 5

	Control			Extract 1			Extract 2			Extract 3			Extract 4		
	ng/g			ng/g			ng/g			ng/g			ng/g		
Analyte	AVE	STD	RSD%	AVE	STD	RSD%	AVE	STD	RSD%	AVE	STD	RSD%	AVE	STD	RSD%
1-methylphenanthrene	147	2.08	1.41	66.6	2.99	4.48	<0.53			65.7	2.33	3.54	<0.55		
1,7-dimethylphenanthrene	133	0.58	0.43	64.9	1.79	2.76	<0.53			65.3	1.12	1.72	<0.55		
2-methylanthracene	51.6	0.75	1.46	28.3	0.65	2.30	<0.29			29.9	0.71	2.38	<0.30		
2-methylphenanthrene															
3-methylphenanthrene	141	1.53	1.08	67.5	0.55	0.82	<0.55			67.3	1.25	1.86	<0.57		
4H-cyclopenta[*def*]phenanthrene															
9-methylphenanthrene	150	2.08	1.38	69.9	1.23	1.76	<0.55			70.0	1.32	1.89	<0.57		
anthracene	63.4	0.98	1.55	37.5	0.76	2.02	<0.53			36.2	0.90	2.49	<0.56		
benzo[*a*]fluoranthene															
benzo[*a*]pyrene	73.3	1.57	2.14	41.3	2.06	4.97	<0.50			39.0	0.90	2.30	<0.50		
benzo[*b*]fluoranthene	134	4.16	3.10	78.0	2.90	3.71	<0.50			71.5	0.53	0.74	<0.50		
benzo[*e*]pyrene	76.7	1.38	1.80	45.0	1.61	3.57	<0.50			43.4	1.80	4.14	<0.50		
benzo[*ghi*]perylene	83.6	0.81	0.97	34.0	1.76	5.18	<0.50			32.3	0.57	1.76	<0.49		
benzo[*j*]fluoranthene + benzo[*k*]fluoranthene	120	1.53	1.28	66.1	4.49	6.80	<0.51			61.6	1.22	1.97	<0.50		
chrysene + triphenylene	49.6	13.4	27.0	27.4	3.37	12.3	<0.50			29.3	2.88	9.81	<0.50		
dibenz[*a,h*]anthracnene	107	0.58	0.54	64.7	2.86	4.41	<0.50			58.1	0.66	1.13	<0.50		
fluoranthene	144	1.00	0.69	88.5	2.90	3.28	<0.54			86.3	2.46	2.85	<0.56		
indeno[1,2,3-*cd*]pyrene	65.6	0.93	1.42	39.4	1.10	2.80	<0.51			35.4	0.21	0.59	<0.50		
perylene	62.9	1.65	2.63	37.6	1.25	3.33	<0.51			35.2	0.68	1.94	<0.50		
phenanthrene	182	2.08	1.14	111	3.21	2.89				107	2.08	1.95			
pyrene	148	0.58	0.39	93.1	1.93	2.07	<0.53			91.4	2.05	2.24	<0.56		

f. Results reported by Participating Laboratory 6

	Control			Extract 1			Extract 2			Extract 3			Extract 4		
	ng/g			ng/g			ng/g			ng/g			ng/g		
Analyte	AVE	STD	RSD%	AVE	STD	RSD%	AVE	STD	RSD%	AVE	STD	RSD%	AVE	STD	RSD%
1-methylphenanthrene	**62.7**	0.76	1.21	30.6	4.24	13.9				29.5	3.06	10.4	0.00		
1,7-dimethylphenanthrene															
2-methylanthracene															
2-methylphenanthrene															
3-methylphenanthrene															
4H-Cyclopenta[*def*]phenanthrene															
9-methylphenanthrene															
anthracene	37.0	1.18	3.19	21.6	3.90	18.1	<0.01			19.8	1.53	7.72	0.13	0.23	173
benzo[*a*]fluoranthene															
benzo[*a*]pyrene	35.5	0.68	1.92	24.9	3.70	14.9	<0.03			27.7	2.31	8.33	<0.03		
benzo[*b*]fluoranthene	96.1	3.55	3.70	78.0	11.1	14.2	<0.03			79.4	6.15	7.74	<0.03		
benzo[*e*]pyrene															
benzo[*ghi*]perylene	45.3	3.16	6.97	31.9	0.87	2.73	<0.04			34.8	4.93	14.2	<0.04		
benzo[*j*]fluoranthene															
benzo[*k]* fluoranthene	33.6	1.80	5.37	21.2	3.53	16.7	<0.03			23.3	3.72	16.0	<0.03		
chrysene															
dibenz[*a,h*]anthracnene															
fluoranthene	84.4	3.55	4.21	53.2	7.78	14.6				52.2	5.96	11.4	0.00		
indeno[1,2,3-*cd*]pyrene															
perylene															
phenanthrene	117	2.87	2.45	70.5	13.6	19.3	4.10	4.00	97.7	51.4	15.6	30.4	0.00		
pyrene	92.3	3.07	3.32	58.7	7.50	12.8				59.7	5.28	8.85	0.37	0.64	173
triphenylene															

Figure 1. Control Solution (NIST SRM 1491a + SRM 2260a in Iso-Octane) agreement with calculated expected values. Mean mass fraction reported for each analyte (per lab, corresponding number) expressed as the number of standard uncertainties (SU) away from calculated expected values. The line around zero represents the 99% confidence interval around the expected value. Refer to Table 4 below for a summary of reported values from participating laboratories.

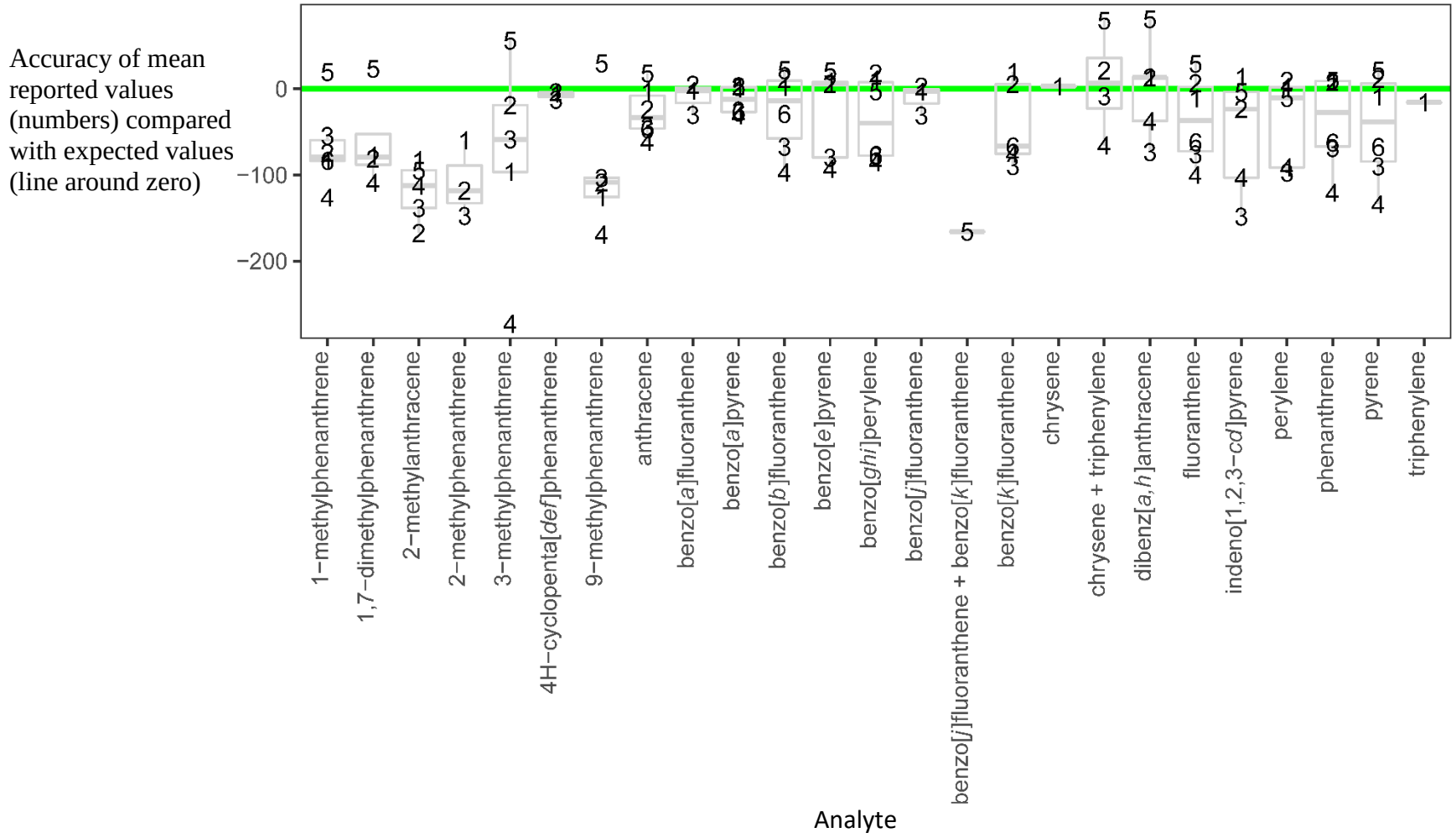


Table 4. Summary of PAH measurements (ng/g; $\bar{x} \pm 1\sigma$) from participating laboratories for the Control Solution (NIST SRM 1491a + SRM 2260a in Iso-Octane)

Control	Calculated Expected Value	Lab 1	Lab 2	Lab 3	Lab 4	Lab 5	Lab 6
1-methylphenanthrene	131 ± 2	62.7 ± 7.1	70.2 ± 7.7	86.5 ± 11.0	27.7 ± 3.3	147 ± 2	62.7 ± 0.8
1,7-dimethylphenanthrene	115 ± 2	53.3 ± 0.6	50.7 ± 2.2	*NM*	28.6 ± 3.7	133 ± 1	*NM*
2-methylanthracene	79.3 ± 0.6	55.2 ± 14.8	30.3 ± 3.5	38.8 ± 10.5	46.4 ± 7.8	51.6 ± 0.8	*NM*
2-methylphenanthrene	140 ± 1	109 ± 35	78.0 ± 10.2	62.5 ± 5.9	*NM*	*NM*	*NM*
3-methylphenanthrene	125 ± 1	96.7 ± 31.9	119 ± 15	108 ± 2	45.1 ± 4.4	141 ± 2	*NM*
4H-cyclopenta[*def*]phenanthrene	34.8 ± 1.7	30.0 ± 3.7	31.6 ± 2.4	24.3 ± 0.3	*NM*	*NM*	*NM*
9-methylphenanthrene	134 ± 1	64.0 ± 7.2	73.5 ± 7.9	76.4 ± 6.2	39.7 ± 3.4	150 ± 2	*NM*
anthracene	56.1 ± 0.8	54.9 ± 3.6	46.4 ± 3.1	38.4 ± 3.8	31.1 ± 4.6	63.4 ± 1.0	37.0 ± 1.2
benzo[*a*]fluoranthene	34.2 ± 1.0	33.0 ± 0.9	36.4 ± 0.5	19.7 ± 0.3	*NM*	*NM*	*NM*
benzo[*a*]pyrene	70.7 ± 2.6	70.9 ± 0.6	74.4 ± 1.5	38.6 ± 0.3	31.6 ± 4.8	73.3 ± 1.6	35.5 ± 0.7
benzo[*b*]fluoranthene	118 ± 2	119 ± 1	127 ± 1	67.5 ± 1.1	45.0 ± 9.5	134 ± 4	96.1 ± 3.6
benzo[*e*]pyrene	68.5 ± 0.8	71.7 ± 1.2	70.8 ± 0.9	36.1 ± 6.4	30.8 ± 2.8	76.7 ± 1.4	*NM*
benzo[*ghi*]perylene	85.1 ± 1.0	90.7 ± 2.1	94.4 ± 1.5	44.7 ± 1.9	41.1 ± 1.7	83.6 ± 0.8	45.3 ± 3.2
benzo[*j*]fluoranthene	62.2 ± 1.5	59.8 ± 1.6	64.1 ± 0.7	39.7 ± 0.2	*NM*	*C*	*NM*
benzo[*k*]fluoranthene	51.7 ± 0.5	57.0 ± 0.8	53.1 ± 1.3	27.5 ± 1.8	31.3 ± 13.8	*C*	33.6 ± 1.8
benzo[*j*]fluoranthene+benzo[*k*]fluoranthene	220 ± 1	*S*	*S*	*S*	*S*	120 ± 2	*S*
chrysene	69.3 ± 1.7	71.6 ± 6.0	*C*	*C*	*C*	*C*	*NM*
triphenylene	61.8 ± 2.4	42.7 ± 2.9	*C*	*C*	*C*	*C*	*NM*
chrysene + triphenylene	85.9 ± 1.1	*S*	97.5 ± 5.3	81.2 ± 1.9	49.6 ± 16.4	130 ± 3	*NM*
dibenz[*a,h*]anthracene	68.4 ± 0.9	74.5 ± 1.4	74.5 ± 1.9	33.7 ± 8.8	50.6 ± 2.9	107 ± 1	*NM*
Fluoranthene	125 ± 1	118 ± 2	129 ± 2	75.3 ± 2.5	59.6 ± 2.7	144 ± 1	84.4 ± 3.6
indeno[1,2,3-*cd*]pyrene	66.4 ± 0.5	69.5 ± 1.0	61.1 ± 2.6	33.0 ± 9.4	43.2 ± 1.1	65.6 ± 0.9	*NM*
perylene	66.5 ± 0.7	67.1 ± 1.9	69.6 ± 2.5	33.6 ± 0.5	35.6 ± 0.7	62.9 ± 1.7	*NM*
phenanthrene	174 ± 2	182 ± 5	181 ± 1	112 ± 4	65.0 ± 7.0	182 ± 2	117 ± 3
pyrene	134 ± 1	129 ± 2	141 ± 2	78.3 ± 5.0	50.8 ± 4.4	148 ± 1	92.3 ± 3.1

C – analytes combined; NM – not measured; S – analytes separated

Figure 2. Spiked Extract of NIST SRM 1945 and CM-IV with SRM 1491 + SRM 2260a mean concentrations of each analyte (per lab, corresponding number) compared with nominal spike values (The line around zero represents the 99% confidence interval around the expected value). Refer to Table 5a,b below for a summary of reported data from participating laboratories.

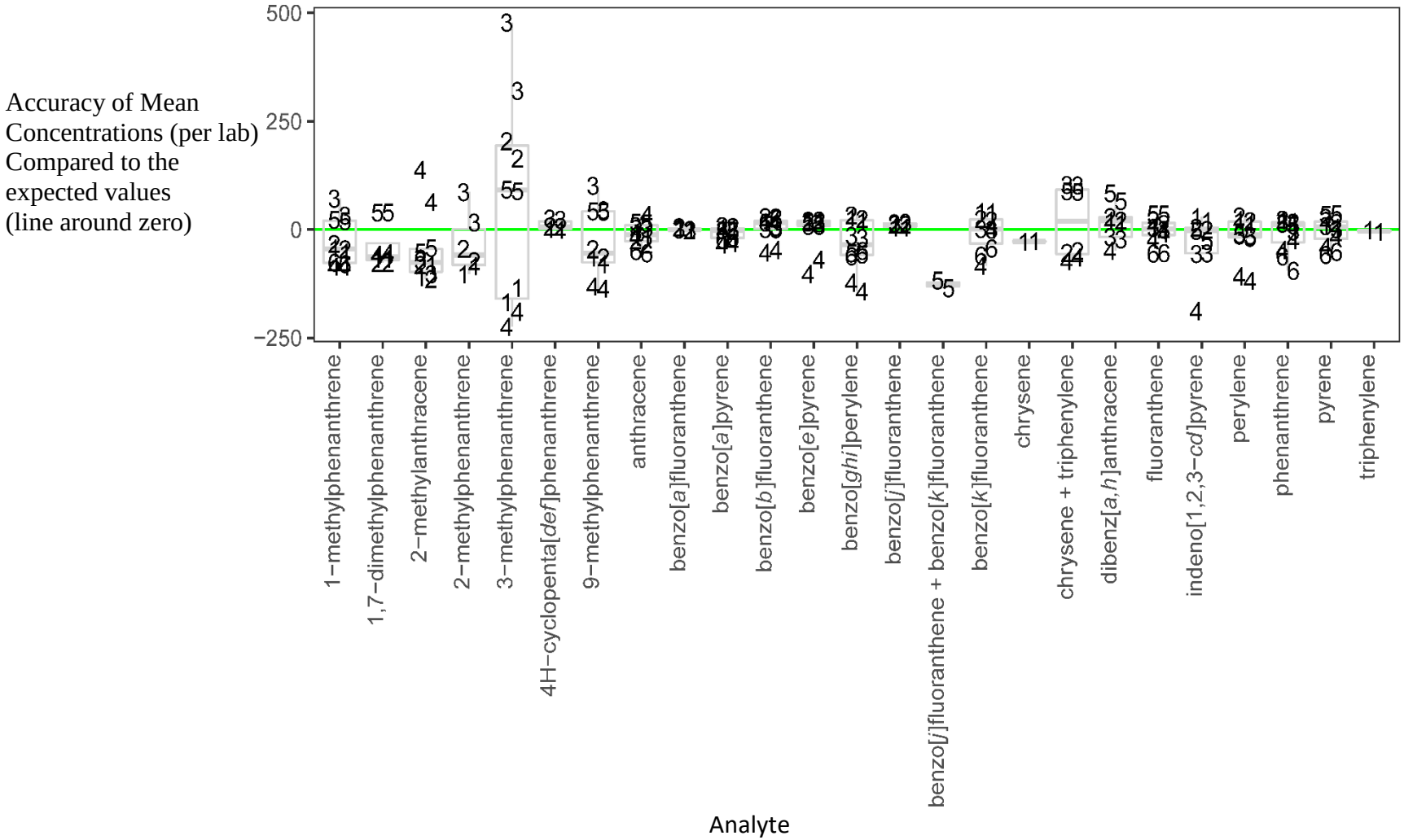


Table 5a. Summary of PAH measurements (ng/g; $\bar{x} \pm 1\sigma$) from participating laboratories for the extract of NIST SRM 1945 spiked with SRM 1491 + SRM 2260a.

Extract 1 (SRM 1945)	Spike Level	Lab 1	Lab 2	Lab 3	Lab 4	Lab 5	Lab 6
1-methylphenanthrene	58.3 ± 0.7	42.7 ± 1.8	46.1 ± 3.0	84.4 ± 3.2	27.1 ± 0.8	66. 6± 3.0	30.6 ± 4.2
1,7-dimethylphenanthrene	51.0 ± 0.7	27.5 ± 0.4	24.0 ± 1.0	*NM*	30.4 ± 1.0	64.9 ± 1.8	*NM*
2-methylanthracene	35.2 ± 0.3	21.0 ± 2.1	24.6 ± 3.5	25.9 ± 1.1	53.1 ± 7.2	28.3 ± 0.7	*NM*
2-methylphenanthrene	62.3 ± 0.5	38.1 ± 3.4	51.9 ± 2.9	82.5 ± 3.5	*NM*	*NM*	*NM*
3-methylphenanthrene	55.4 ± 0.3	33.7 ± 3.1	82.0 ± 6.0	118 ± 4	26.3 ± 2.3	67.5 ± 0.6	*NM*
4H-cyclopenta[*def*]phenanthrene	20.8 ± 1.0	21.4 ± 1.0	24.4 ± 1.3	33.3 ± 1.5	*NM*	*NM*	*NM*
9-methylphenanthrene	59.4 ± 0.5	43.5 ± 2.1	48.3 ± 3.0	84.4 ± 3.8	27.1 ± 5.6	69.9 ± 1.2	*NM*
anthracene	33.5 ± 0.5	31.5 ± 0.9	27.7 ± 1.0	35.5 ± 1.5	29.7 ± 9.8	37.5 ± 0.8	21.6 ± 3.9
benzo[*a*]fluoranthene	20.5 ± 0.6	21.1 ± 0.8	22.0 ± 1.5	20.7 ± 0.6	*NM*	*NM*	*NM*
benzo[*a*]pyrene	42.3 ± 1.5	44.7 ± 1.6	48.4 ± 2.3	40.8 ± 0.6	15.7 ± 3.7	41.3 ± 2.1	24.9 ± 3.7
benzo[*b*]fluoranthene	70.5 ± 0.9	77.8 ± 1.4	83.4 ± 3.6	71.2 ± 1.2	46.8 ± 2.9	78.0 ± 2.9	78.0 ± 11.1
benzo[*e*]pyrene	40.9 ± 0.5	45.2 ± 0.9	45.9 ± 1.8	43.1 ± 0.8	16.1 ± 2.9	45.0 ± 1.6	*NM*
benzo[*ghi*]perylene	50.9 ± 0.6	59.4 ± 1.7	61.0 ± 2.8	46.5 ± 1.6	13.0 ± 2.3	34.0 ± 1.8	31.9 ± 0.9
benzo[*j*]fluoranthene	37.2 ± 0.9	39.6 ± 0.6	42.1 ± 1.7	43.7 ± 0.7	*NM*	*C*	*NM*
benzo[*k*]fluoranthene	30.9 ± 0.3	37.5 ± 0.5	34.8 ± 1.1	31.2 ± 0.3	17.4 ± 8.1	*C*	21.2 ± 3.5
benzo[*j*]fluoranthene+benzo[*k*]fluoranthene	97.5 ± 0.5	*S*	*S*	*S*	*S*	66.1 ± 4.5	*S*
chrysene	41.5 ± 1.0	27.6 ± 1.3	*C*	*C*	*C*	*C*	*NM*
triphenylene	37.0 ± 1.4	36.1 ± 1.5	*C*	*C*	*C*	*C*	*NM*
chrysene + triphenylene	51.4 ± 0.7	*S*	33.6 ± 1.2	85.4 ± 1.4	27.4 ± 4.1	80.6 ± 3.8	*NM*
dibenz[*a,h*]anthracene	40.9 ± 0.6	46.6 ± 1.7	49.0 ± 2.2	36.1 ± 0.5	27.0 ± 4.1	64.7 ± 2.9	*NM*
Fluoranthene	74.7 ± 0.8	76.4 ± 1.7	83.3 ± 3.1	76.2 ± 1.3	65.9 ± 4.5	88.5 ± 2.9	53.2 ± 7.8
indeno[1,2,3-*cd*]pyrene	39.7 ± 0.3	43.7 ± 1.2	40.3 ± 2.1	32.2 ± 1.5	14.4 ± 1.7	39.4 ± 1.1	*NM*
perylene	39.8 ± 0.4	44.2 ± 1.3	45.9 ± 2.1	37.1 ± 0.6	18.0 ± 2.4	37.6 ± 1.3	*NM*
phenanthrene	104 ± 1	115 ± 3	116 ± 3	112 ± 2	78.4 ± 2.2	111 ± 3	70.5 ± 13.6
pyrene	80.3 ± 0.7	83.2 ± 1.8	90.5 ± 3.7	83.4 ± 1.1	65.7 ± 7.1	93.1 ± 1.9	58.7 ± 7.5

Table 5b. Summary of PAH measurements (ng/g; $\bar{x} \pm 1\sigma$) from participating laboratories for the extract of CM-IV spiked with SRM 1491 + SRM 2260a.

Extract 3 (CM-IV)	Spike Level	Lab 1	Lab 2	Lab 3	Lab 4	Lab 5	Lab 6
1-methylphenanthrene	58.5 ± 0.7	39.2 ± 4.9	41.8 ± 2.0	70. 4± 9.1	27. 6± 1.8	65. 7± 2.3	29.5 ± 3.1
1,7-dimethylphenanthrene	51.2 ± 0.7	27.4 ± 0.4	24.1 ± 0.9	*NM*	31.7 ± 0.8	65.3 ± 1.1	*NM*
2-methylanthracene	35.3 ± 0.3	24.8 ± 8.3	20.5 ± 1.7	21.9 ± 5.3	43.6 ± 12.1	29.9 ± 0.7	*NM*
2-methylphenanthrene	62.5 ± 0.5	42.7 ± 9.8	45.4 ± 3.2	66.4 ± 10.0	*NM*	*NM*	*NM*
3-methylphenanthrene	55.7 ± 0.3	38.1 ± 9.0	77.1 ± 5.3	97.4 ± 13.7	30.9 ± 4.7	67.3 ± 1.2	*NM*
4H-cyclopenta[*def*]phenanthrene	20.3 ± 1.0	20.0 ± 2.9	23.2 ± 2.6	31.4 ± 0.9	*NM*	*NM*	*NM*
9-methylphenanthrene	59.7 ± 0.5	39.9 ± 5.4	43.7 ± 2.3	70.8 ± 10.2	25.8 ± 2.9	70.0 ± 1.3	*NM*
anthracene	32.7 ± 0.5	28.6 ± 2.2	24.6 ± 2.4	33.4 ± 0.5	40.6 ± 2.1	36.2 ± 0.9	19.8 ± 1.5
benzo[*a*]fluoranthene	19.9 ± 0.6	19.8 ± 1.1	19.7 ± 1.1	19.5 ± 0.3	*NM*	*NM*	*NM*
benzo[*a*]pyrene	41.2 ± 1.5	42.6 ± 0.3	45.5 ± 1.4	40.1 ± 0.4	18.3 ± 1.7	39.0 ± 0.9	27.7 ± 2.3
benzo[*b*]fluoranthene	68.8 ± 0.9	76.8 ± 1.5	81.1 ± 1.4	69.3 ± 0.8	48.1 ± 9.1	71.5 ± 0.5	79.4 ± 6.2
benzo[*e*]pyrene	39.9 ± 0.5	44.8 ± 0.6	44.8 ± 1.3	42.1 ± 0.7	23.6 ± 3.1	43.4 ± 1.8	*NM*
benzo[*ghi*]perylene	49.6 ± 0.6	55.7 ± 1.0	57.0 ± 1.5	43.7 ± 0.4	6.47 *NA*	32.3 ± 0.6	34.8 ± 4.9
benzo[*j*]fluoranthene	36.3 ± 0.8	38.2 ± 0.8	40.8 ± 0.8	43.1 ± 0.4	*NM*	*C*	*NM*
benzo[*k*]fluoranthene	30.1 ± 0.3	36.4 ± 0.9	33.5 ± 1.0	30.2 ± 0.8	30.1 ± 17.0	*C*	23.3 ± 3.7
benzo[*j*]fluoranthene+benzo[*k*]fluoranthene	97.8 ± 0.5	*S*	*S*	*S*	*S*	61.6 ± 1.2	*S*
chrysene	40.4 ± 1.0	27.6 ± 3.4	*C*	*C*	*C*	*C*	*NM*
triphenylene	36.1 ± 1.4	32.5 ± 1.7	*C*	*C*	*C*	*C*	*NM*
chrysene + triphenylene	50.1 ± 0.6	*S*	34.2 ± 5.0	82.9 ± 0.9	29.3 ± 3.5	78.3 ± 1.0	*NM*
dibenz[*a,h*]anthracene	39.9 ± 0.6	44.3 ± 0.6	45.6 ± 1.3	33.8 ± 0.6	*NM*	58.1 ± 0.7	*NM*
Fluoranthene	72.9 ± 0.8	72.4 ± 0.8	77.8 ± 2.7	72.9 ± 1.4	69.4 ± 2.2	86.3 ± 2.5	52.2 ± 6.0
indeno[1,2,3-*cd*]pyrene	38.7 ± 0.3	41.3 ± 0.7	38.6 ± 1.1	31.6 ± 1.1	*NM*	35.4 ± 0.2	*NM*
perylene	38.8 ± 0.4	41.5 ± 0.5	42.6 ± 1.0	36.0 ± 0.3	15.5 *NA*	35.2 ± 0.7	*NM*
phenanthrene	101 ± 1	110 ± 3	110 ± 5	99.2 ± 16.3	88.7 ± 4.8	107 ± 2	51.4 ± 15.6
pyrene	78.3 ± 0.7	79.8 ± 1.0	83.9 ± 2.8	78.7 ± 1.2	72.7 ± 7.6	91.4 ± 2.1	59.7 ± 5.3

C- analytes combined; NM – not measured; S – analytes separate

Figure 3. Unspiked: Extract of NIST SRM 1945 and CM-IV without PAHs (a) Mean concentrations of each analyte (per lab, corresponding number) compared with mean detection limits (line around zero +/- 3 SD) of reported analytes. Lab 4 and Lab 6 only reported findings in unspiked samples (refer to Table 6 below).

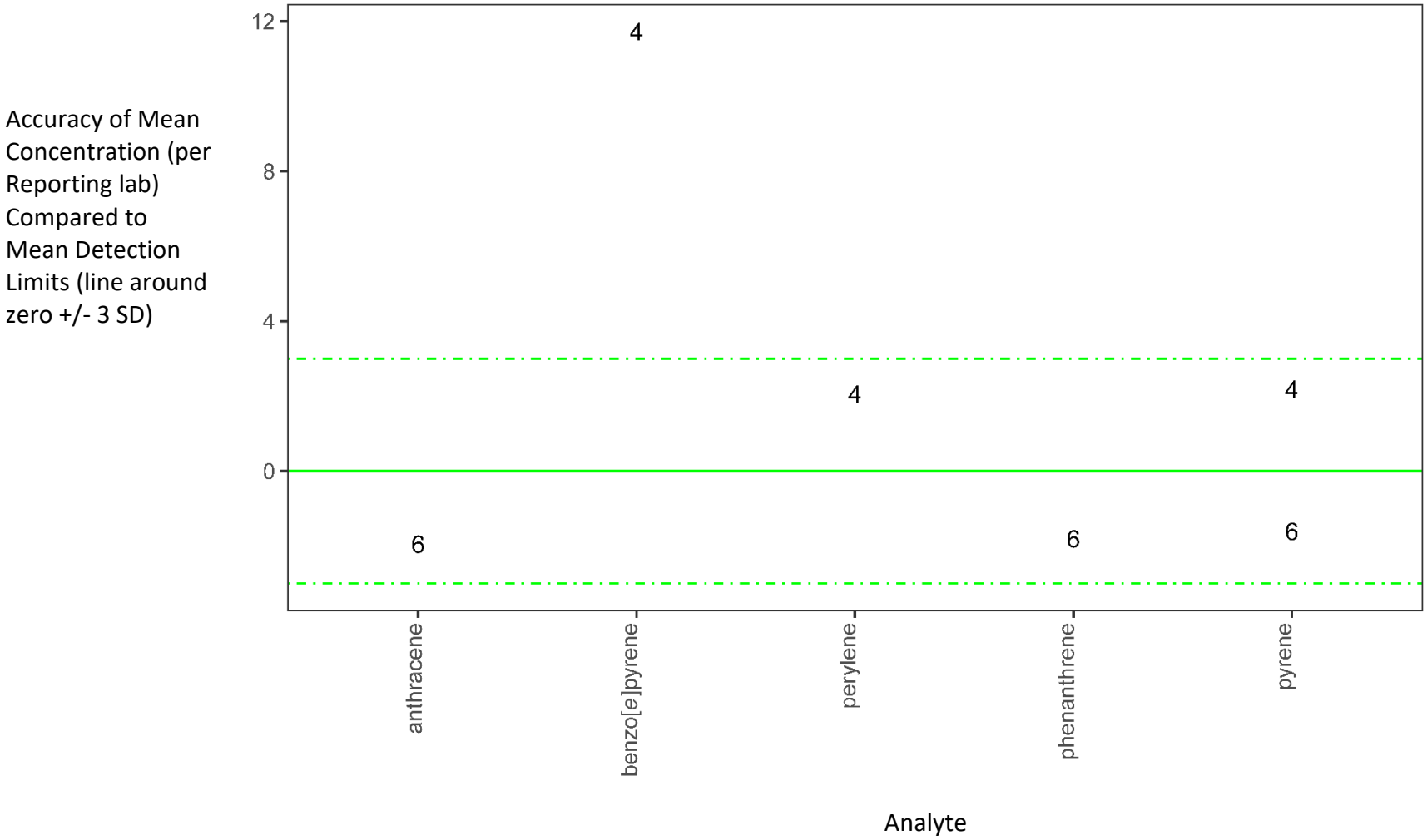


Table 6. Summary of PAH measurements (ng/g; $\bar{x} \pm 1\sigma$) detected by participating laboratories in the unspiked extracts of NIST SRM 1945 and CM-IV.

Extract 2 SRM 1945 Unspiked	Detection Limits	Lab 1	Lab 2	Lab 3	Lab 4	Lab 5	Lab 6
anthracene	1.77 ± 1.68	<5.03 ± 0.52	<1.95 ± 0.2	<0 252 ± 0 029	BDL	<0.693 ± 0.203	<0.01 *NA*
benzo[*e*]pyrene	3.96 ± 3.54	<8.63 ± 0.9	<8.09 ± 0.84	<0 411 ± 0 014	BDL	<0.668 ± 0.208	*NM*
perylene	4.38 ± 3.96	<8.67 ± 0.9	<9.88 ± 1.03	<0 600 ± 0 007	BDL	<0.674 ± 0.209	*NM*
phenanthrene	19.2 ± 14.0	<22.3 ± 2.3	<40.1 ± 4.2	<2 06 ± 0 22	BDL	*NM*	6.15 ± 2.62
pyrene	5.14 ± 5.94	<5.71 ± 0.59	<16.5 ± 1.7	<0 225 ± 0 012	BDL	*NM*	*NR*

Extract 4 CM-IV Unspiked	Detection Limits	Lab 1	Lab 2	Lab 3	Lab 4	Lab 5	Lab 6
anthracene	1.59 ± 1.93	<4.42 ± 0.05	<1.72 ± 0.02	<0.252 ± 0.029	BDL	<0.608 ± 0.048	0.400 *NA*
benzo[*e*]pyrene	4.45 ± 4.12	<7.59 ± 0.08	<7.11 ± 0.07	<0.411 ± 0.014	24.7 ± 5.8	<0.551 ± 0.051	*NM*
perylene	4.96 ± 4.57	<7.62 ± 0.08	<8.69 ± 0.09	<0.600 ± 0.007	8.46 *NA*	<0.556 ± 0.051	*NM*
phenanthrene	19.4 ± 17.0	<19.6 ± 0.2	<35.2 ± 0.4	<2.06 ± 0.22	BDL	*NM*	*NR*
pyrene	5.77 ± 6.87	<5.02 ± 0.05	<14.5 ± 0.1	<0.225 ± 0.012	11.6 ± 2.6	*NM*	1.10 *NA*

BDL – below detection limit; C – analytes combined; NA – not applicable; NM – not measured; NR – not reportable; S – analytes separated

APPENDIX D

Charts of Analyzed Polycyclic Aromatic Hydrocarbons Results by Sample

See Tables 3 through 6 for results reported as <number, detection limit, etc.

For plot 1:

Solid line: Median value

Dotted line: z = ± 1, i. e., 25 % from median value

Dotted/dashed line: z = ± 2, i. e., 50 % from median value

Dashed line: z = ± 3, i. e., 75 % from median value

For plot 2:

Solid line: Nominal expected/target value

Dotted line: 95 % confidence interval (CI)

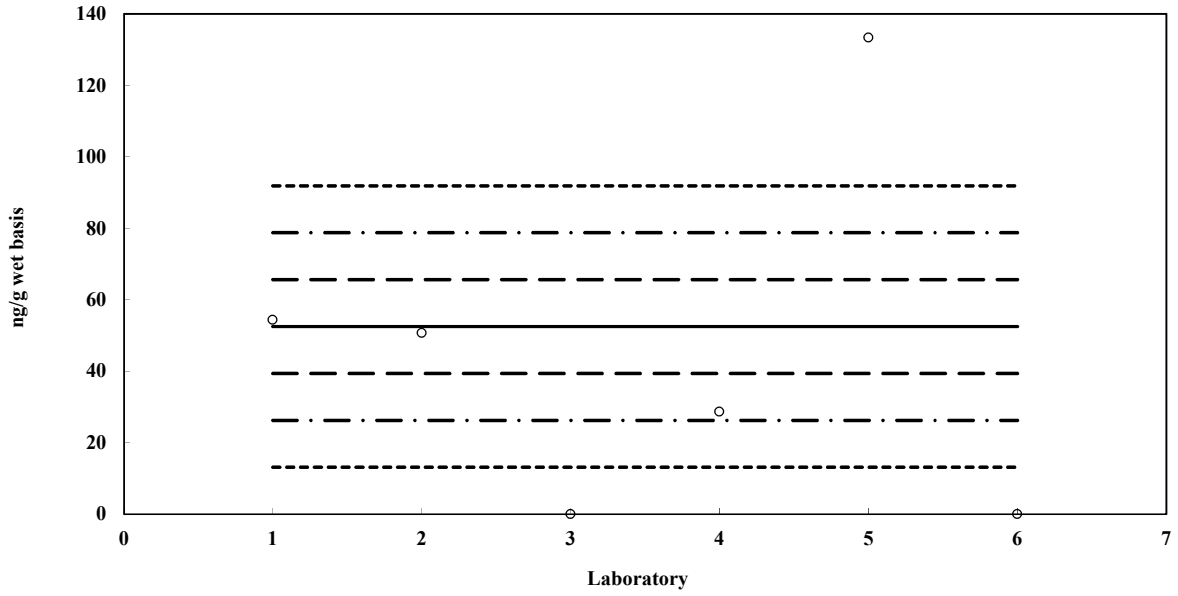
Dashed line: 30 % from 95 % confidence interval (CI)

Note: The numbers added to the charts are the values reported that are off the scale of the chart.

1,7-Dimethylphenanthrene
Median value = 52.5 ng/g SD = 28.9 ng/g 95% CI = ± 53.8 ng/g (wet basis)
Reported Results: 4
SRMs 2260a and 1491a

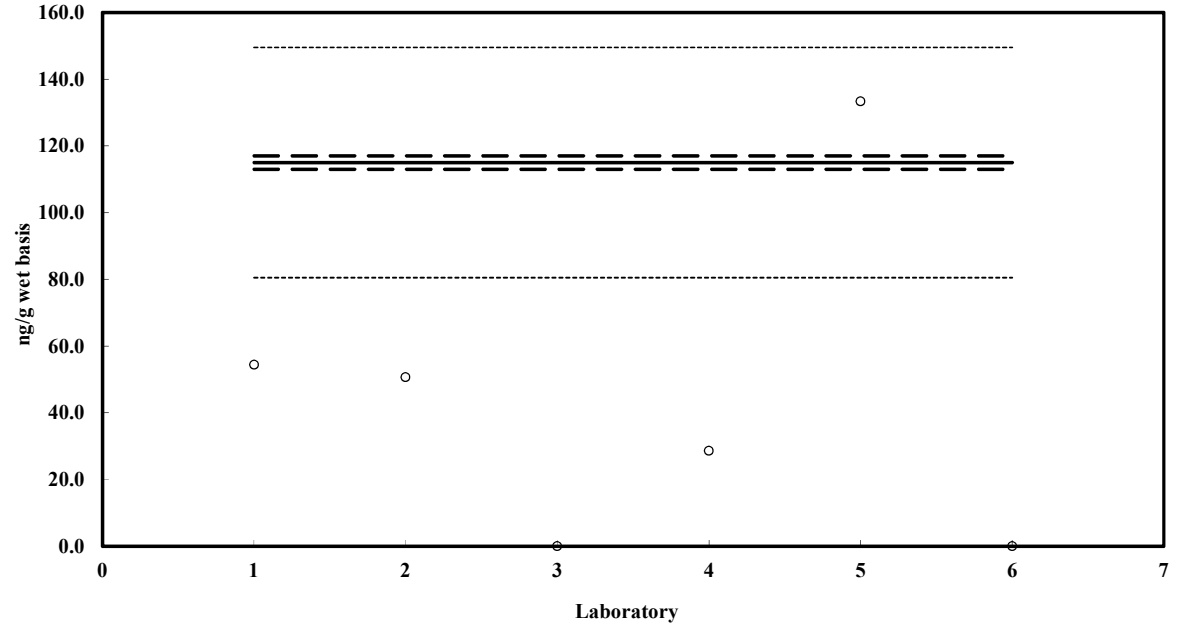
Control Sample

Median Value
± 1 Z
± 2 Z
± 3 Z

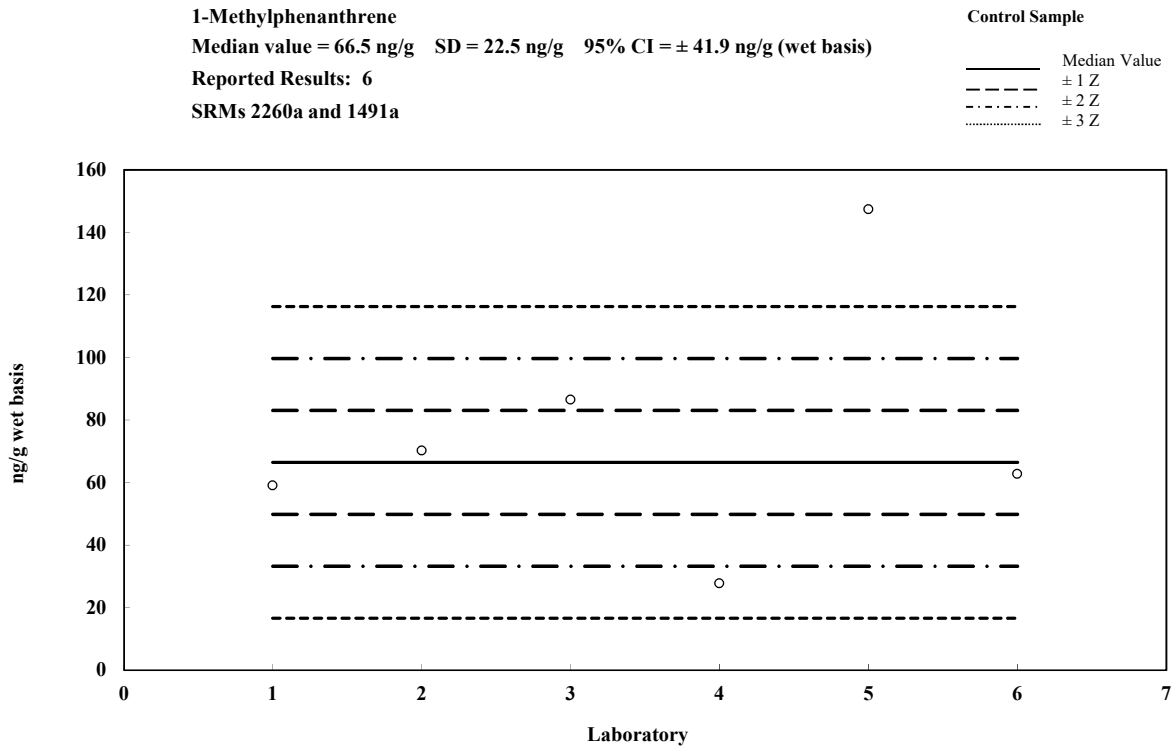


1,7-Dimethylphenanthrene
Value = 115 ± 2 ng/g (wet basis)
Reported Results: 4
SRMs 2260a and 1491a

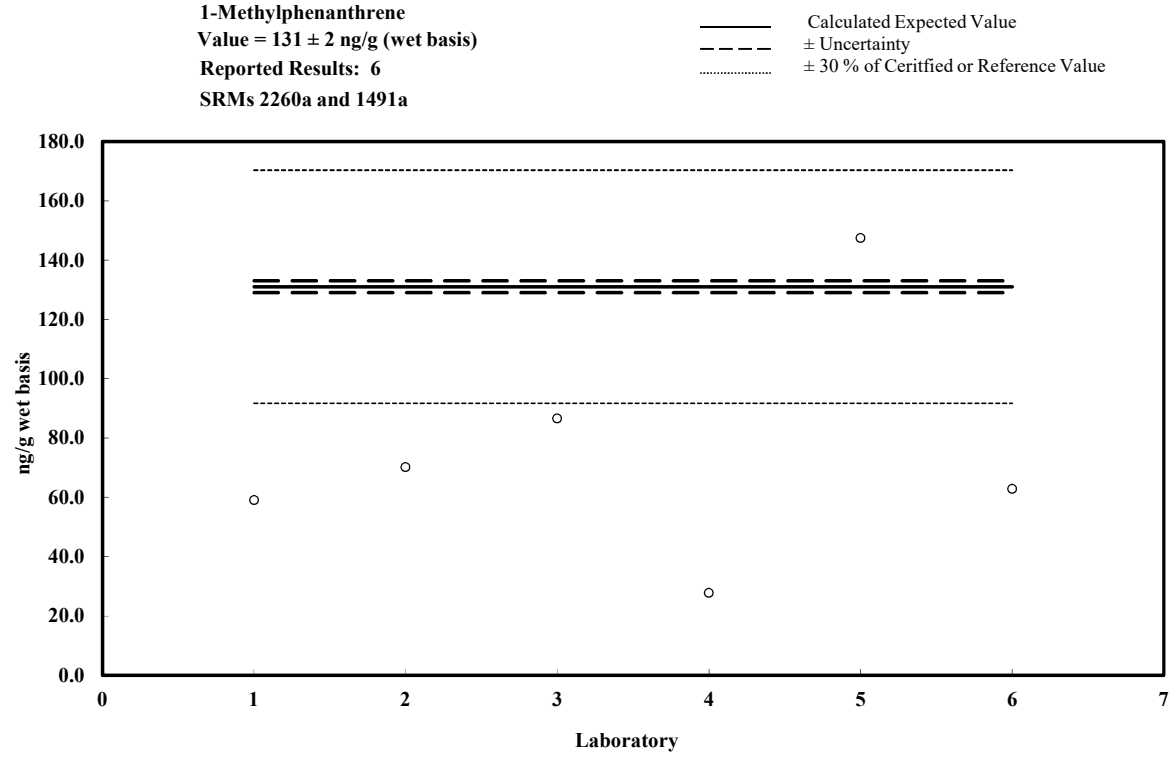
Calculated Expected Value
± Uncertainty
± 30 % of Ceritfied or Reference Value



1-Methylphenanthrene
Median value = 66.5 ng/g SD = 22.5 ng/g 95% CI = ± 41.9 ng/g (wet basis)
Reported Results: 6
SRMs 2260a and 1491a



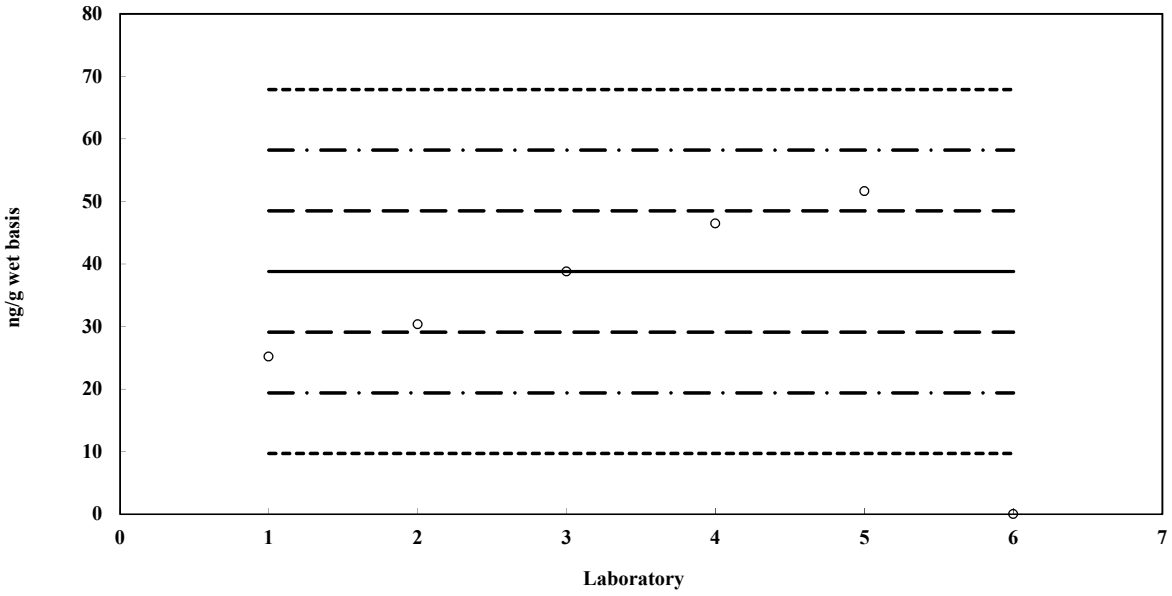
1-Methylphenanthrene
Value = 131 ± 2 ng/g (wet basis)
Reported Results: 6
SRMs 2260a and 1491a



2-Methylanthracene
Median value = 38.8 ng/g SD = 16.1 ng/g 95% CI = ± 29.9 ng/g (wet basis)
Reported Results: 5
SRMs 2260a and 1491a

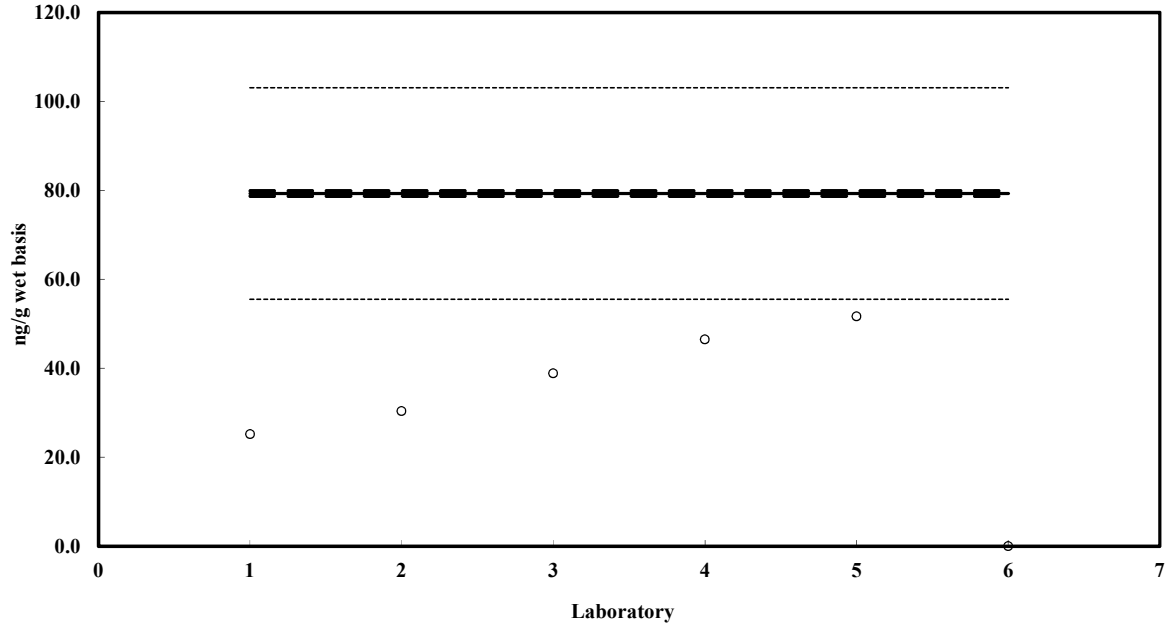
Control Sample

Median Value
± 1 Z
± 2 Z
± 3 Z

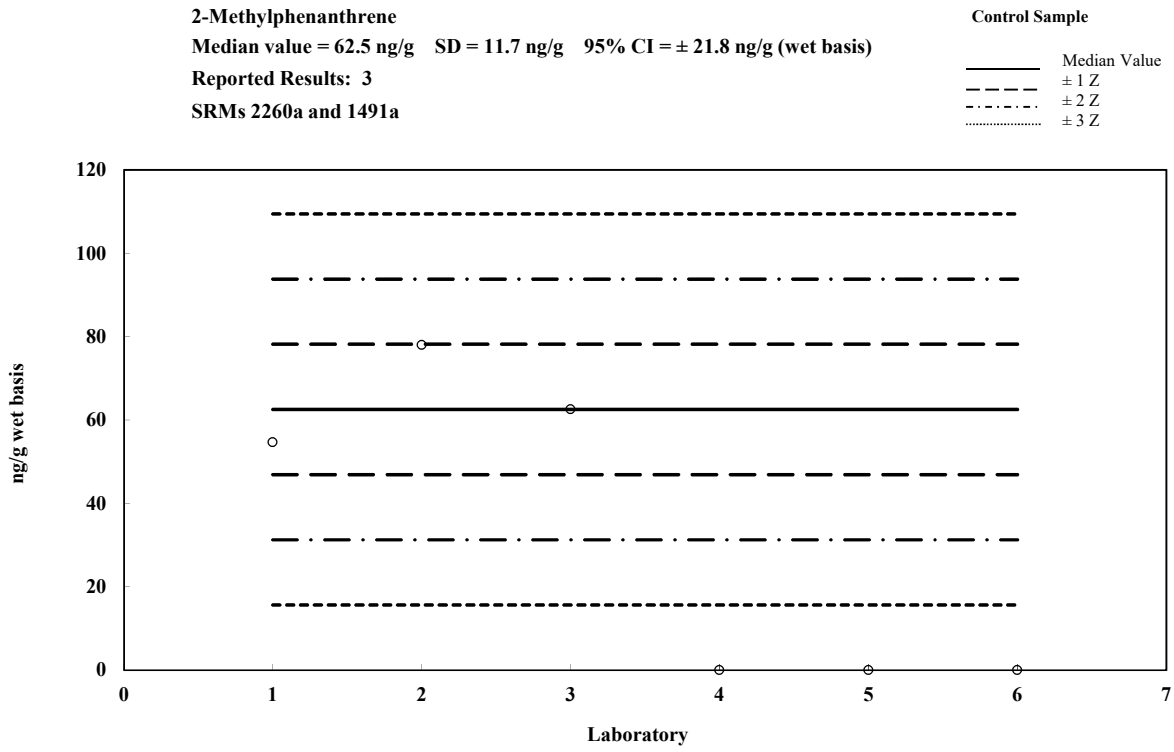


2-Methylanthracene
Value = 79 ± 1 ng/g (wet basis)
Reported Results: 5
SRMs 2260a and 1491a

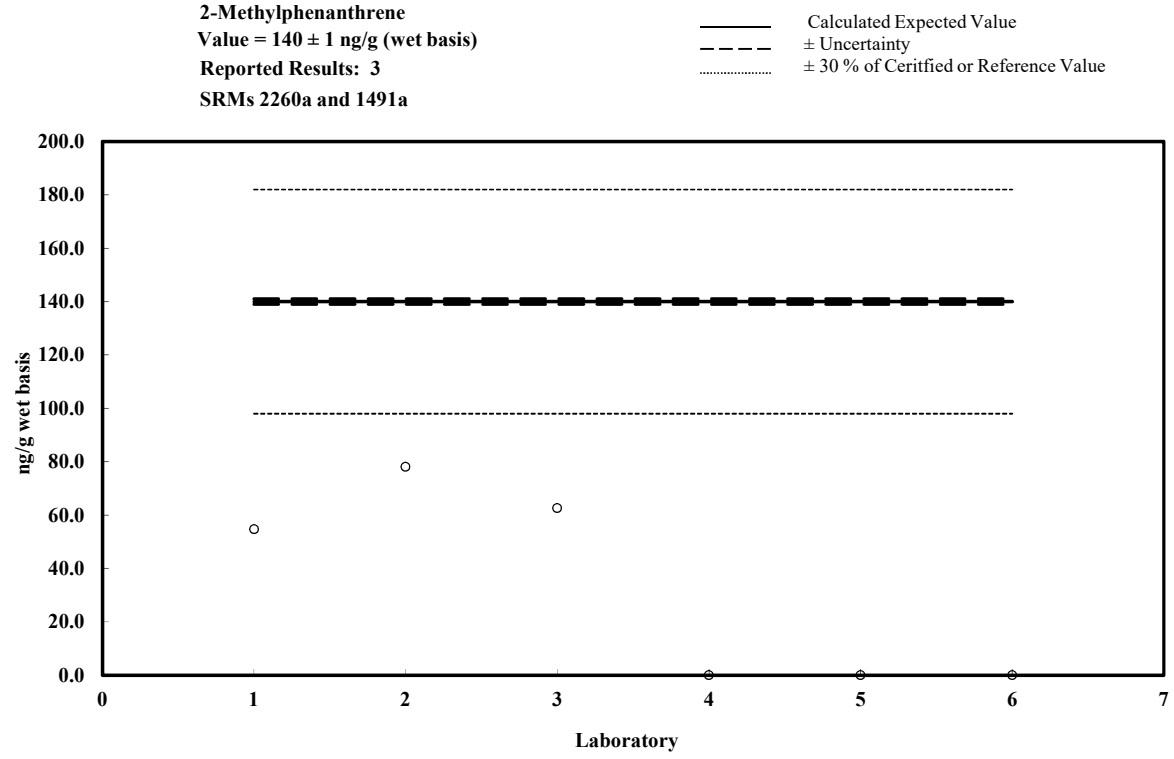
Calculated Expected Value
± Uncertainty
± 30 % of Ceritfied or Reference Value



2-Methylphenanthrene
Median value = 62.5 ng/g SD = 11.7 ng/g 95% CI = ± 21.8 ng/g (wet basis)
Reported Results: 3
SRMs 2260a and 1491a



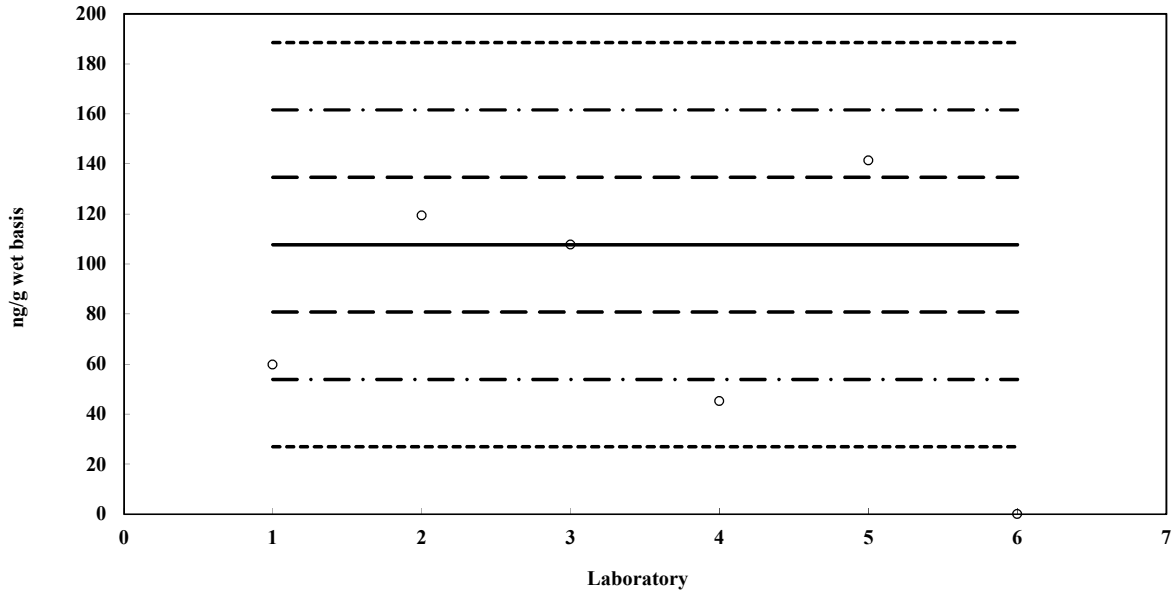
2-Methylphenanthrene
Value = 140 ± 1 ng/g (wet basis)
Reported Results: 3
SRMs 2260a and 1491a



3-Methylphenanthrene
Median value = 107.7 ng/g SD = 59.7 ng/g 95% CI = ± 111 ng/g (wet basis)
Reported Results: 5
SRMs 2260a and 1491a

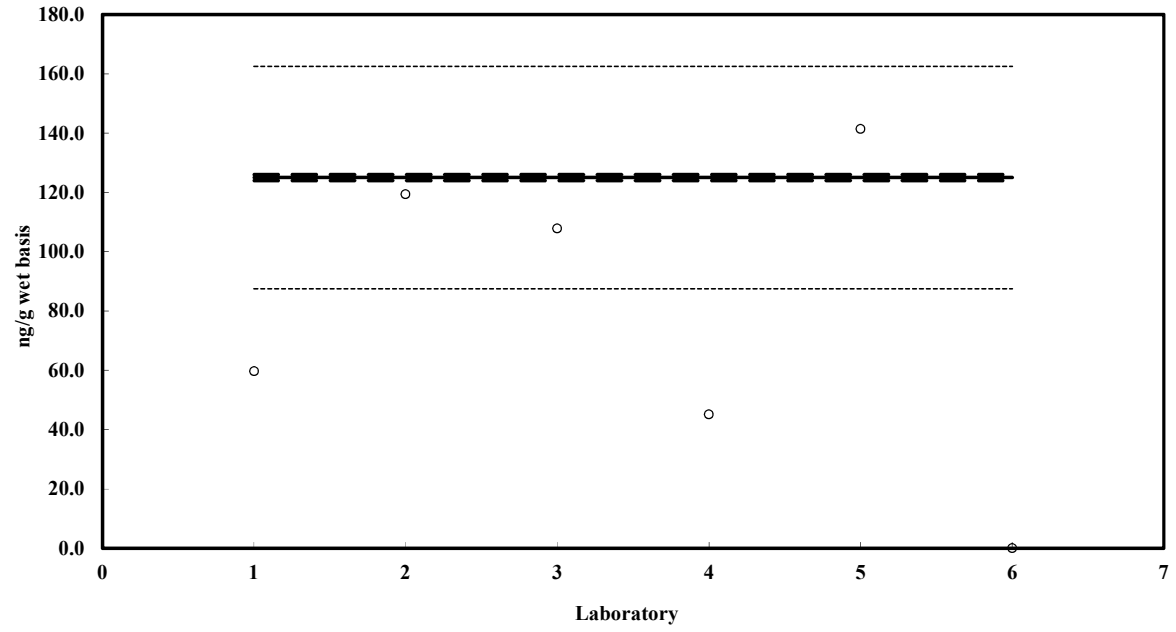
Control Sample

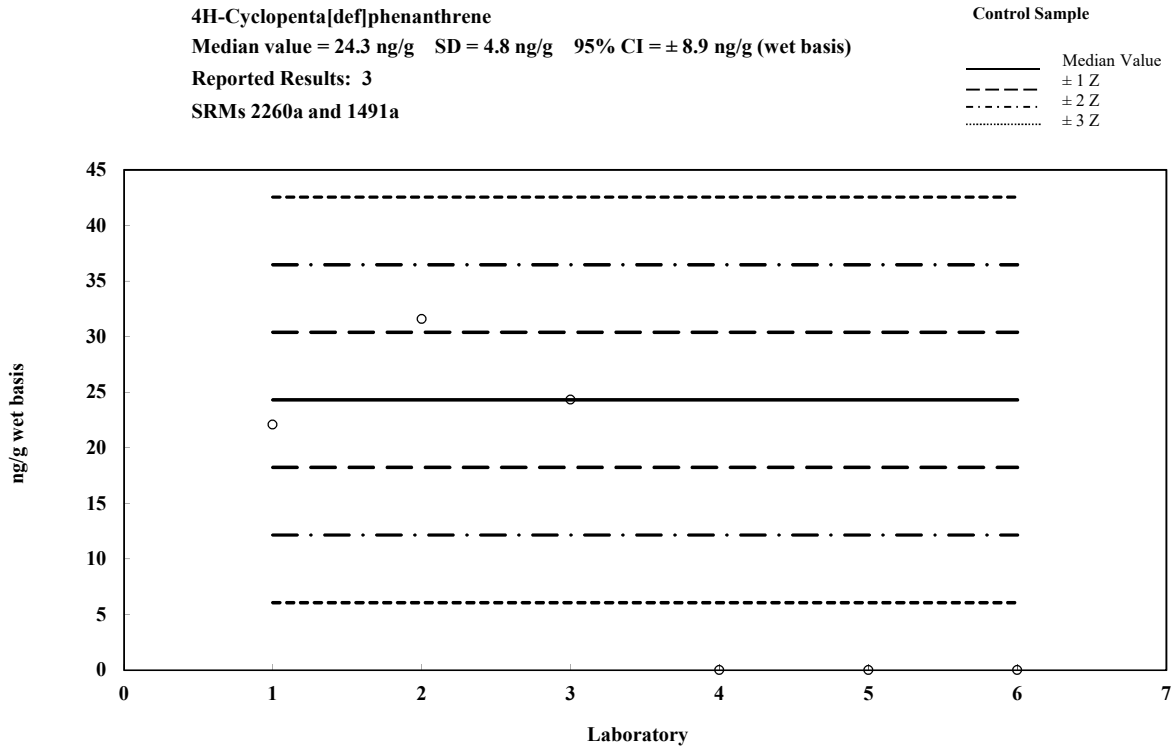
Median Value
± 1 Z
± 2 Z
± 3 Z

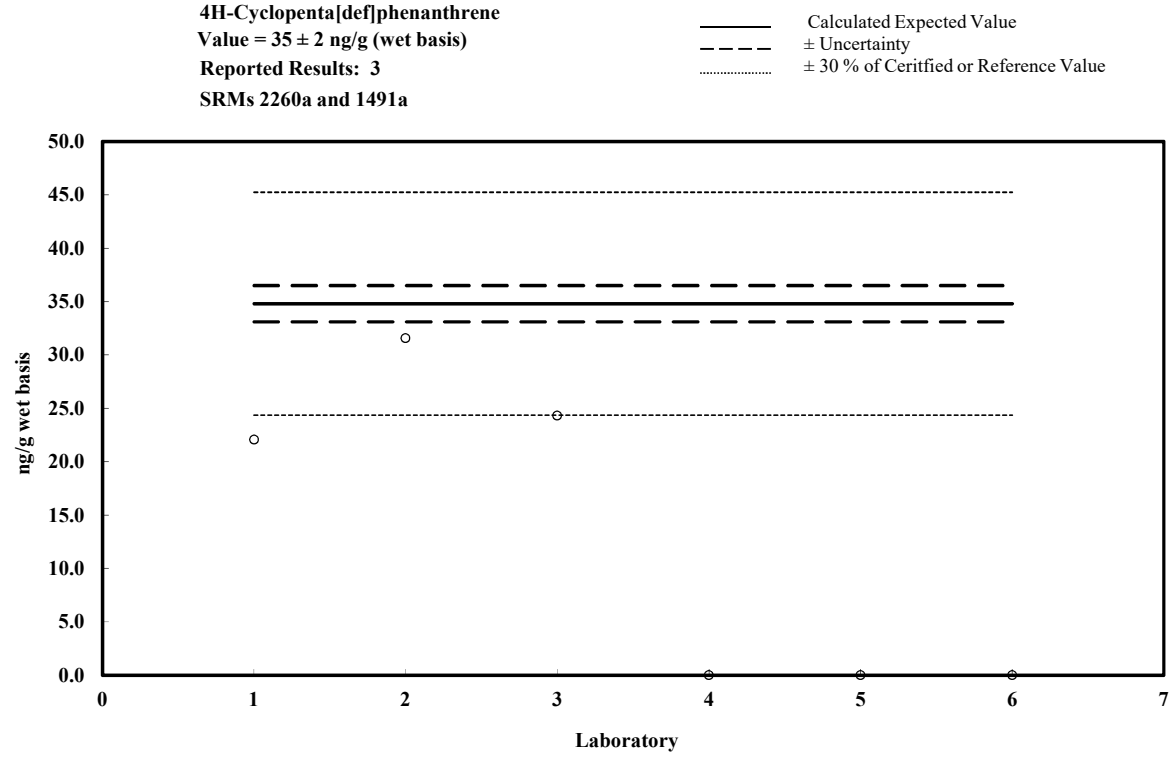


3-Methylphenanthrene
Value = 125 ± 1 ng/g (wet basis)
Reported Results: 5
SRMs 2260a and 1491a

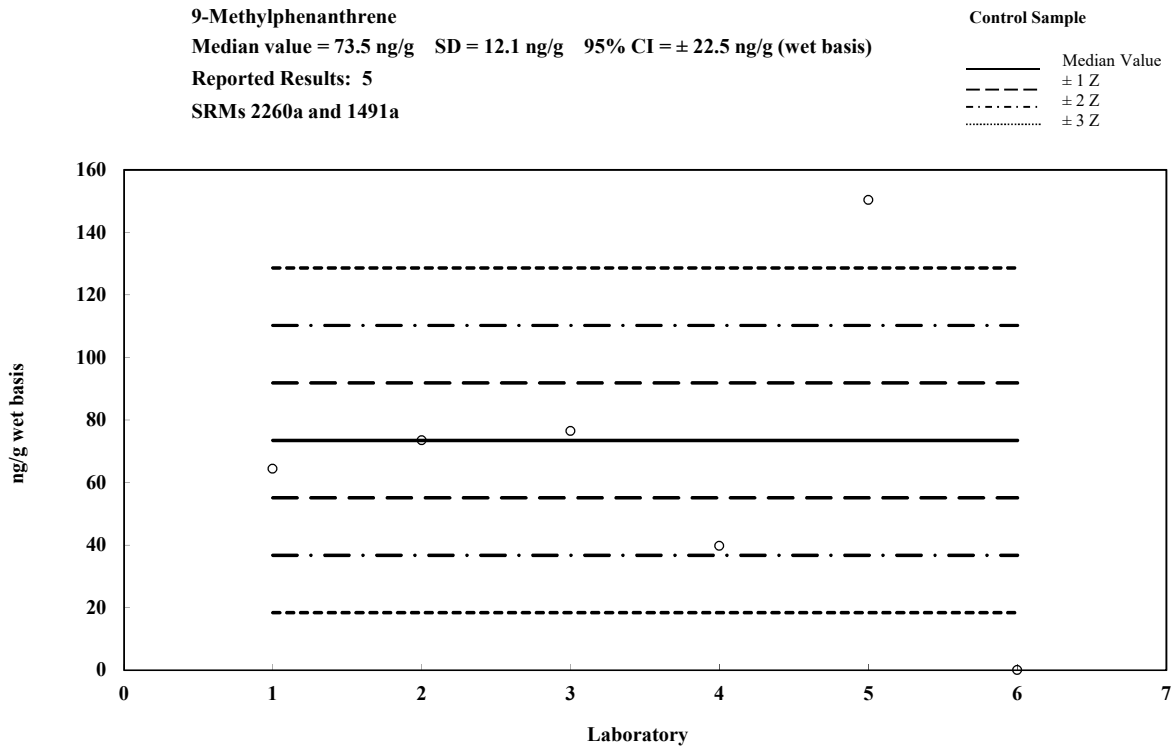
Calculated Expected Value
± Uncertainty
± 30 % of Ceritfied or Reference Value



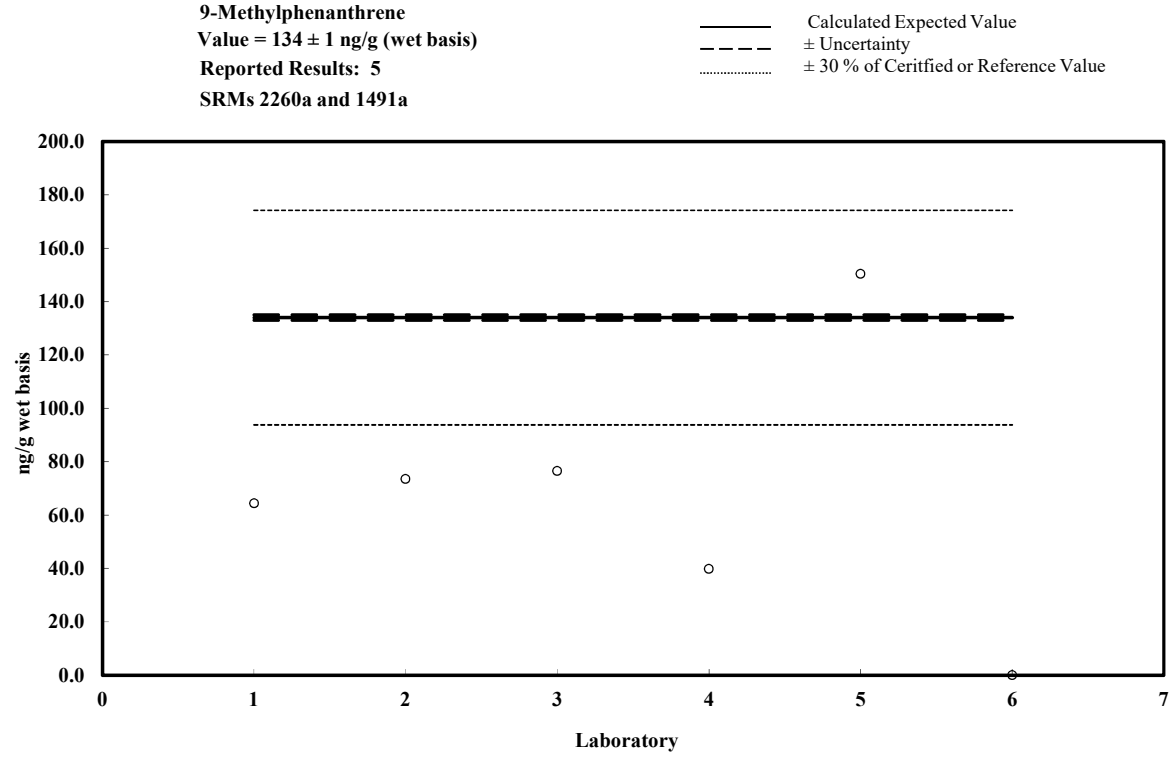




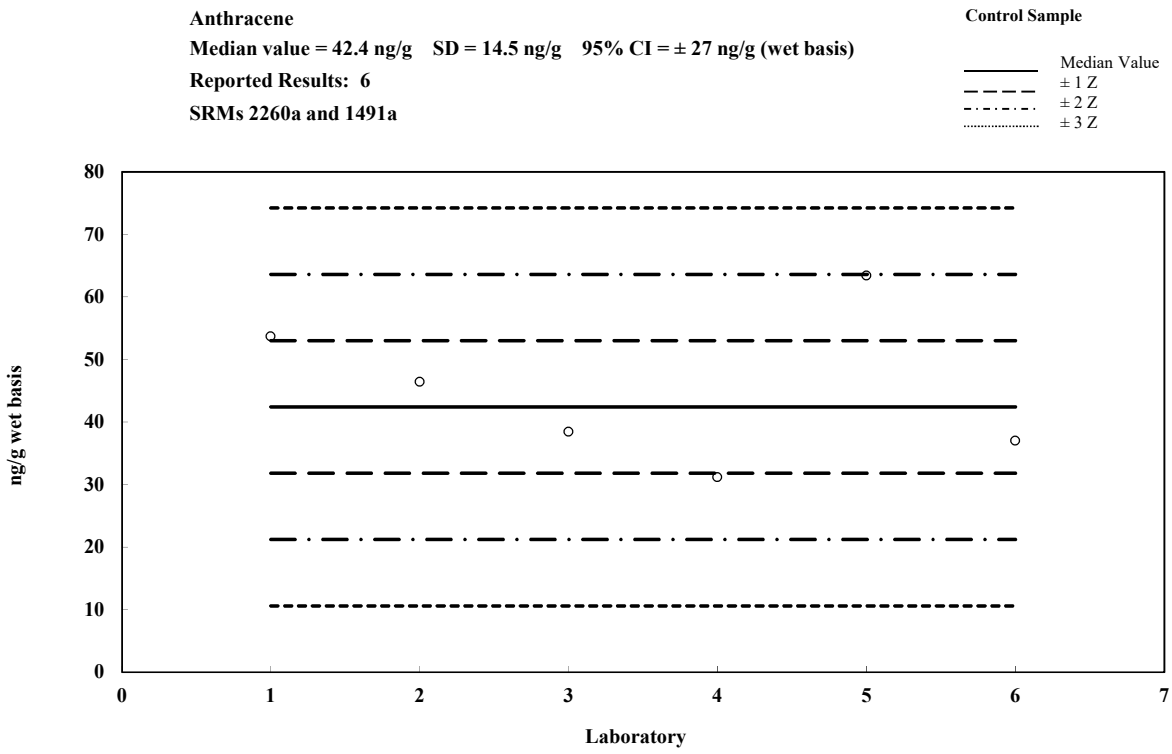
9-Methylphenanthrene
Median value = 73.5 ng/g SD = 12.1 ng/g 95% CI = ± 22.5 ng/g (wet basis)
Reported Results: 5
SRMs 2260a and 1491a



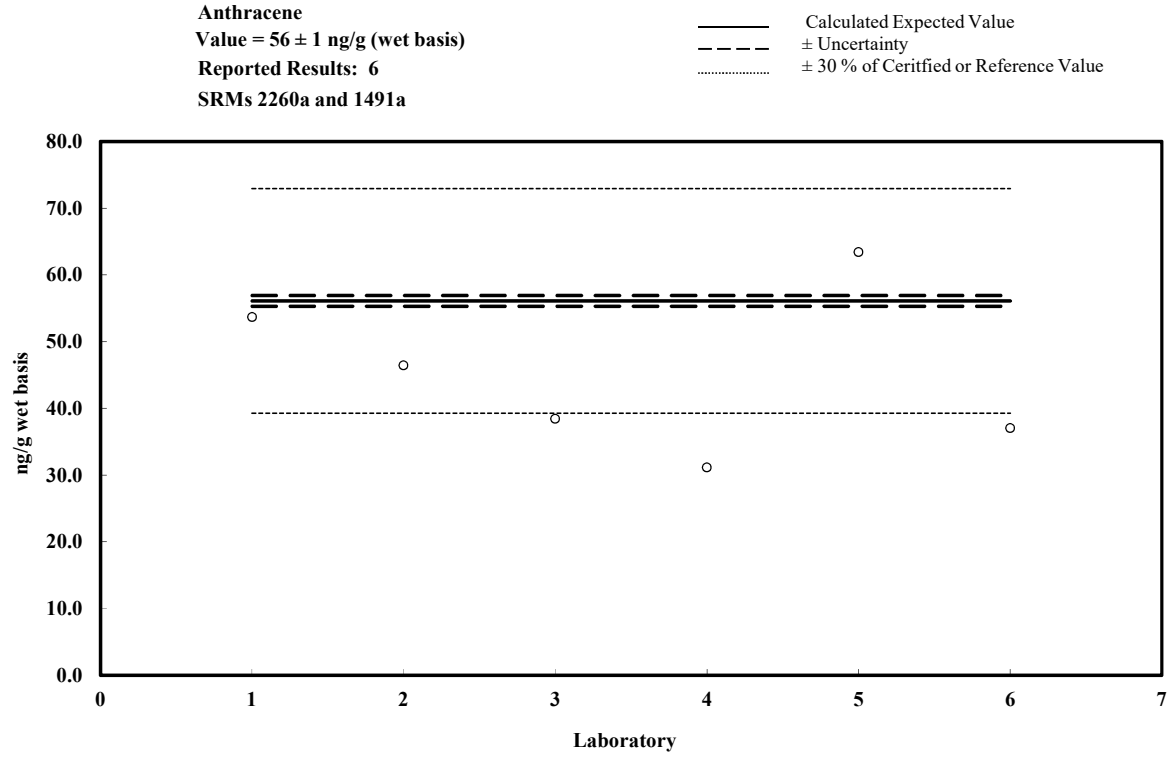
9-Methylphenanthrene
Value = 134 ± 1 ng/g (wet basis)
Reported Results: 5
SRMs 2260a and 1491a



Anthracene
Median value = 42.4 ng/g SD = 14.5 ng/g 95% CI = ± 27 ng/g (wet basis)
Reported Results: 6
SRMs 2260a and 1491a



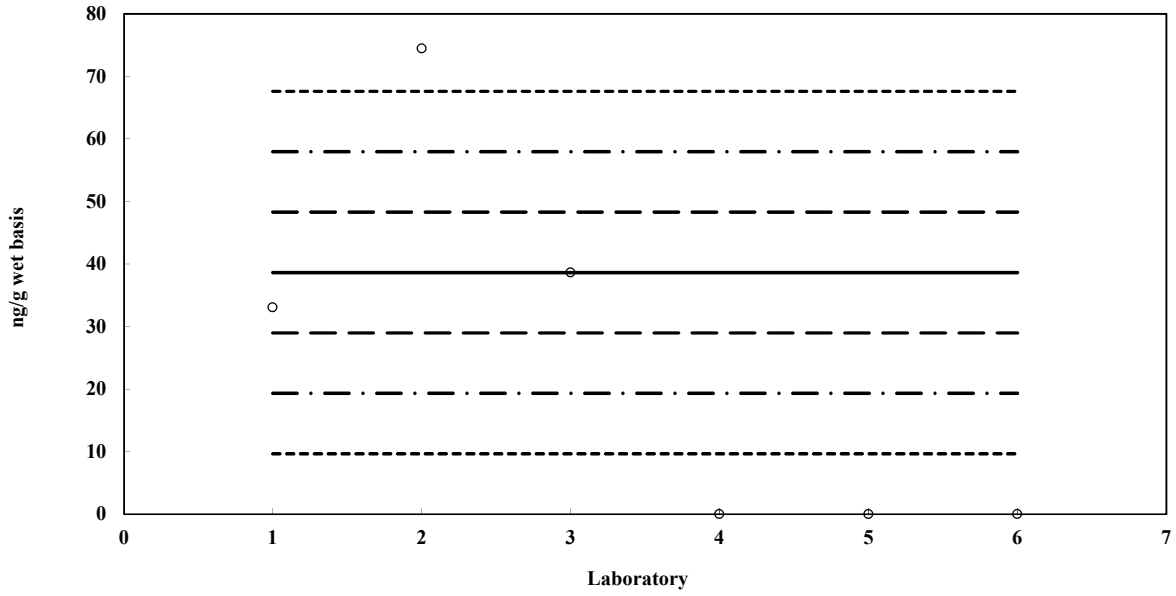
Anthracene
Value = 56 ± 1 ng/g (wet basis)
Reported Results: 6
SRMs 2260a and 1491a



Benzo[a]fluoranthene
Median value = 38.6 ng/g SD = 20.7 ng/g 95% CI = ± 38.5 ng/g (wet basis)
Reported Results: 3
SRMs 2260a and 1491a

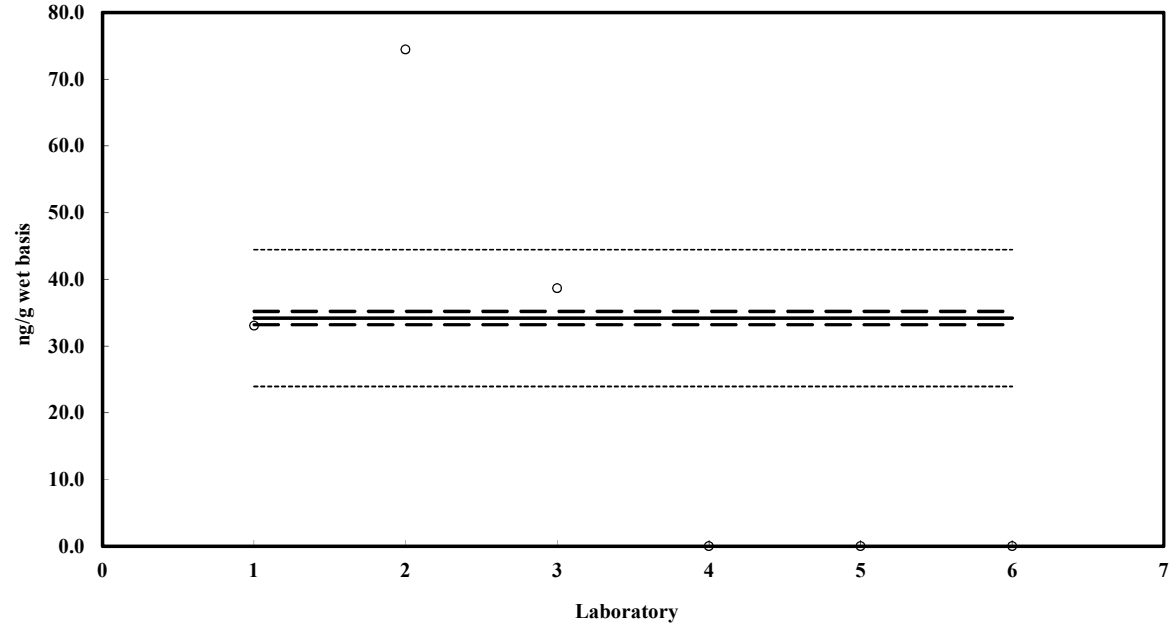
Control Sample

Median Value
± 1 Z
± 2 Z
± 3 Z



Benzo[a]fluoranthene
Value = 34 ± 1 ng/g (wet basis)
Reported Results: 3
SRMs 2260a and 1491a

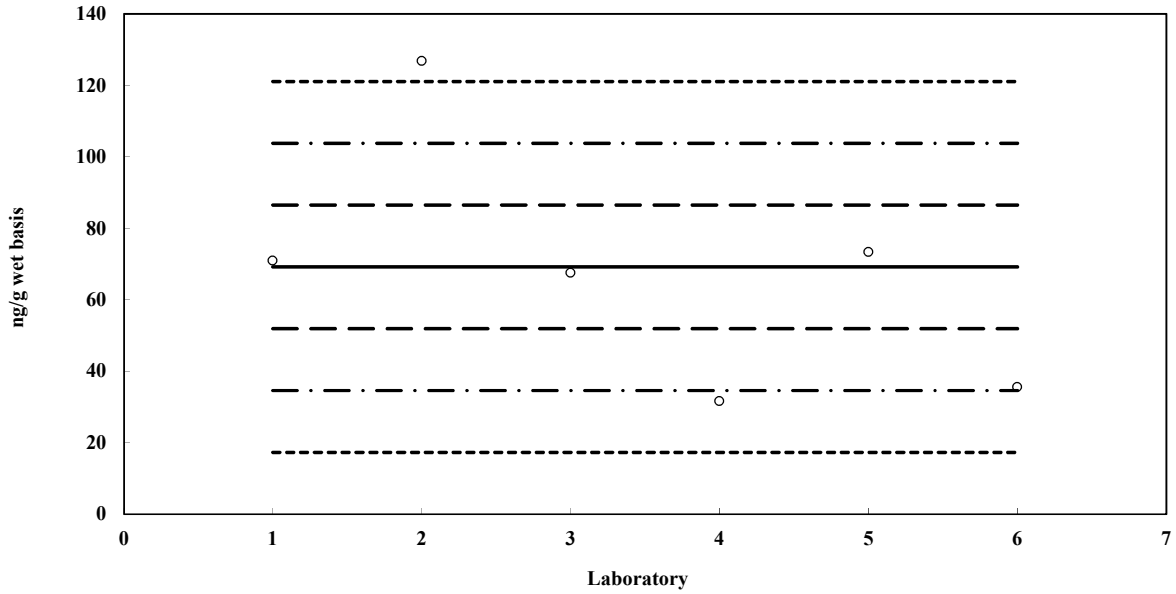
Calculated Expected Value
± Uncertainty
± 30 % of Ceritfied or Reference Value



Benzo[a]pyrene
Median value = 69.2 ng/g SD = 29.2 ng/g 95% CI = ± 54.3 ng/g (wet basis)
Reported Results: 6
SRMs 2260a and 1491a

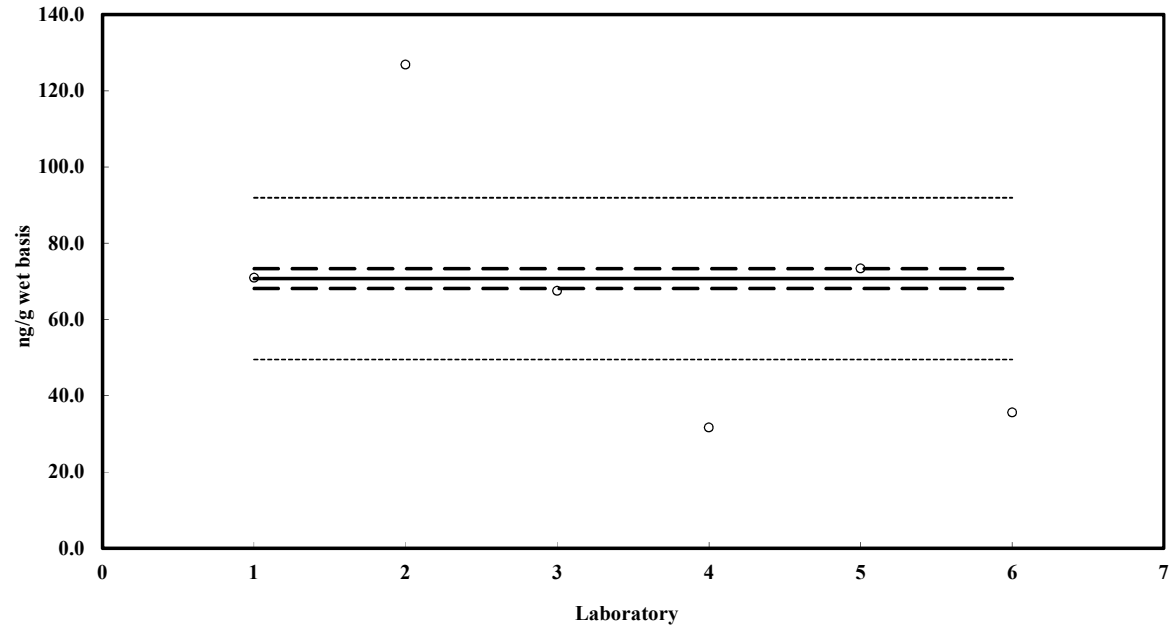
Control Sample

Median Value
± 1 Z
± 2 Z
± 3 Z

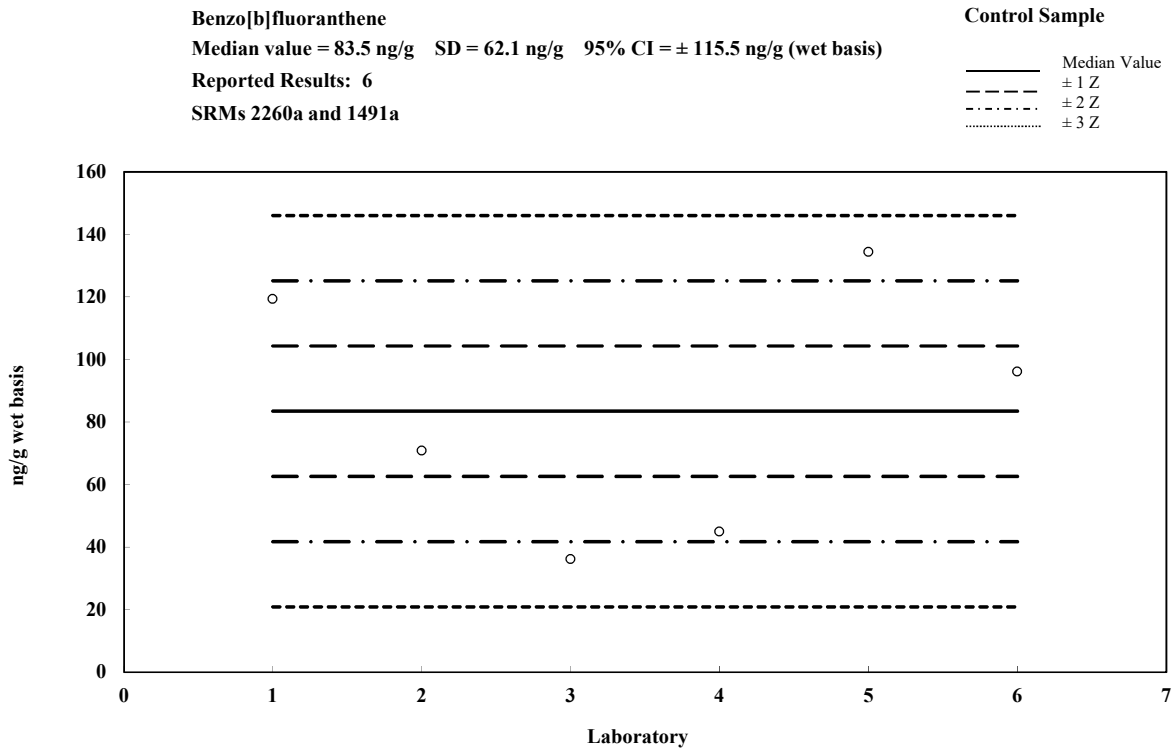


Benzo[a]pyrene
Value = 71 ± 3 ng/g (wet basis)
Reported Results: 6
SRMs 2260a and 1491a

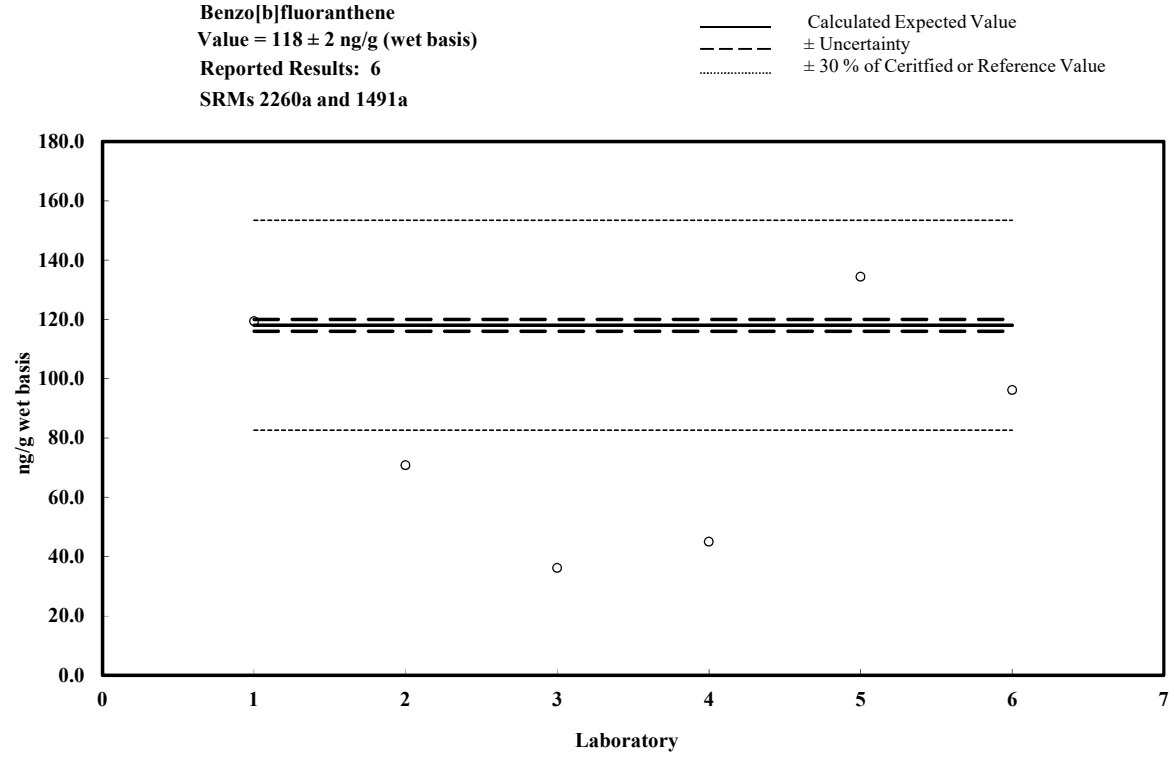
Calculated Expected Value
± Uncertainty
± 30 % of Ceritfied or Reference Value



Benzo[b]fluoranthene
Median value = 83.5 ng/g SD = 62.1 ng/g 95% CI = ± 115.5 ng/g (wet basis)
Reported Results: 6
SRMs 2260a and 1491a



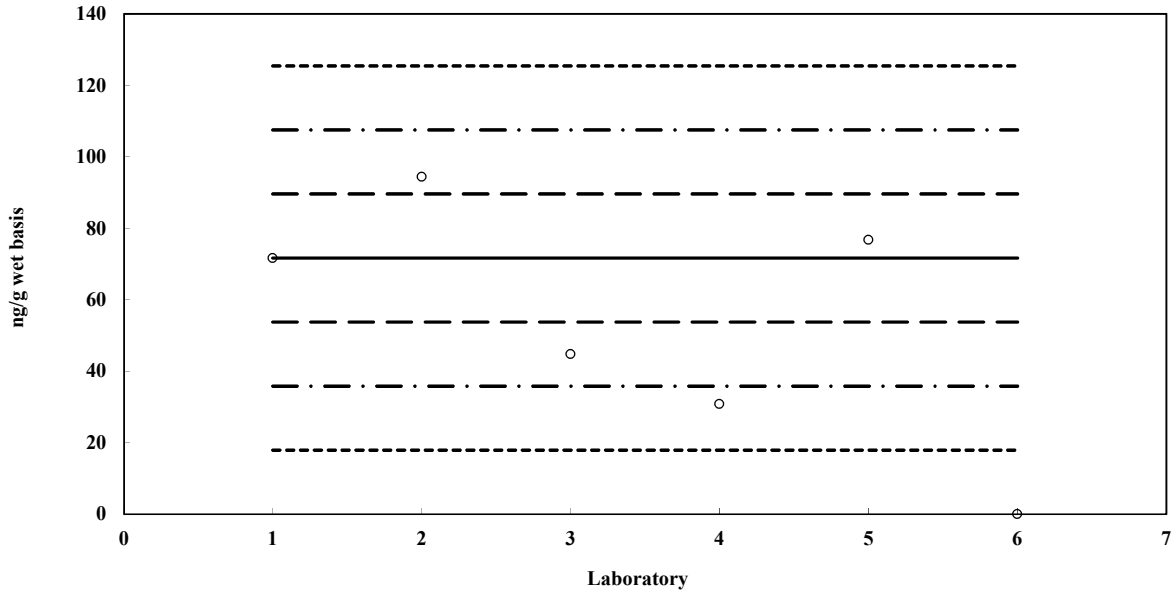
Benzo[b]fluoranthene
Value = 118 ± 2 ng/g (wet basis)
Reported Results: 6
SRMs 2260a and 1491a



Benzo[e]pyrene
Median value = 71.7 ng/g SD = 32 ng/g 95% CI = ± 59.5 ng/g (wet basis)
Reported Results: 5
SRMs 2260a and 1491a

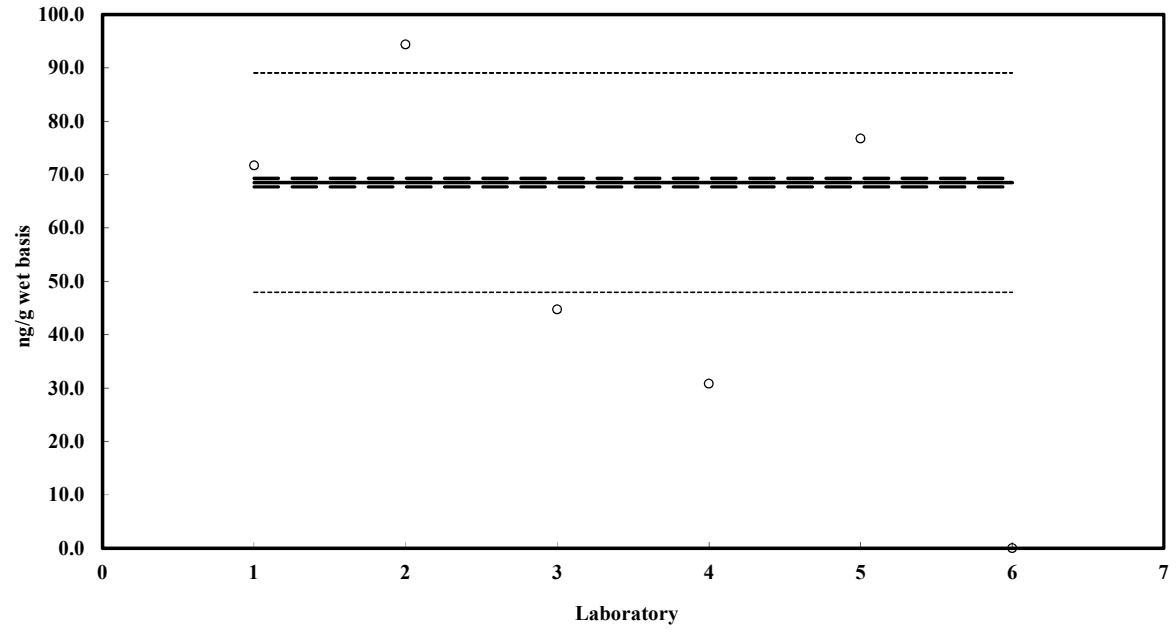
Control Sample

Median Value
± 1 Z
± 2 Z
± 3 Z



Benzo[e]pyrene
Value = 69 ± 1 ng/g (wet basis)
Reported Results: 5
SRMs 2260a and 1491a

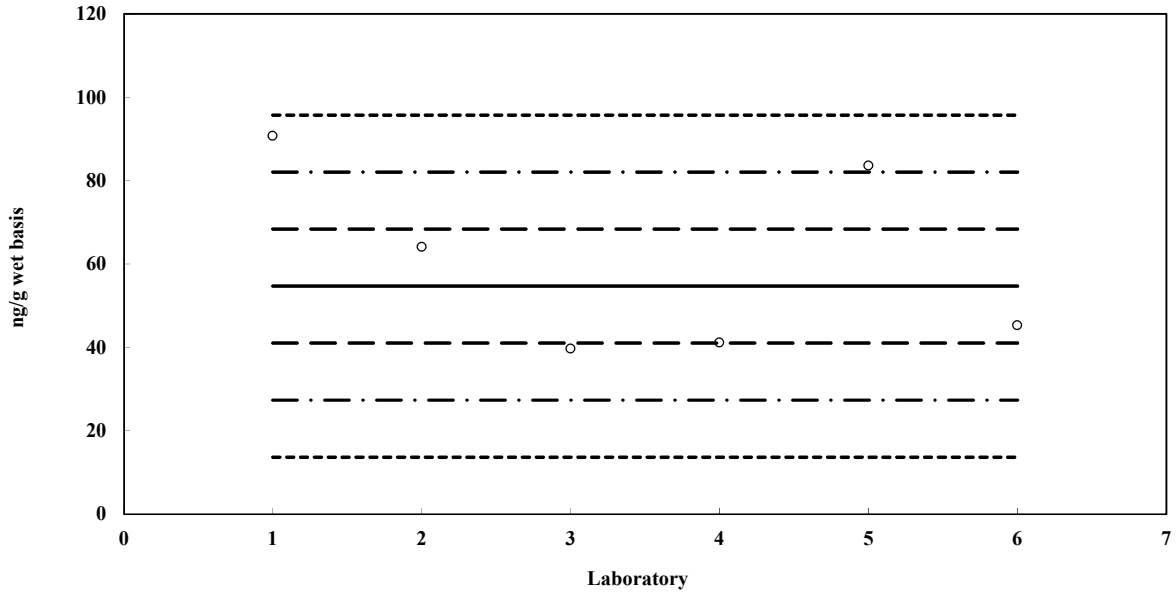
Calculated Expected Value
± Uncertainty
± 30 % of Ceritfied or Reference Value



Benzo[ghi]perylene
Median value = 54.7 ng/g SD = 36.5 ng/g 95% CI = ± 67.9 ng/g (wet basis)
Reported Results: 6
SRMs 2260a and 1491a

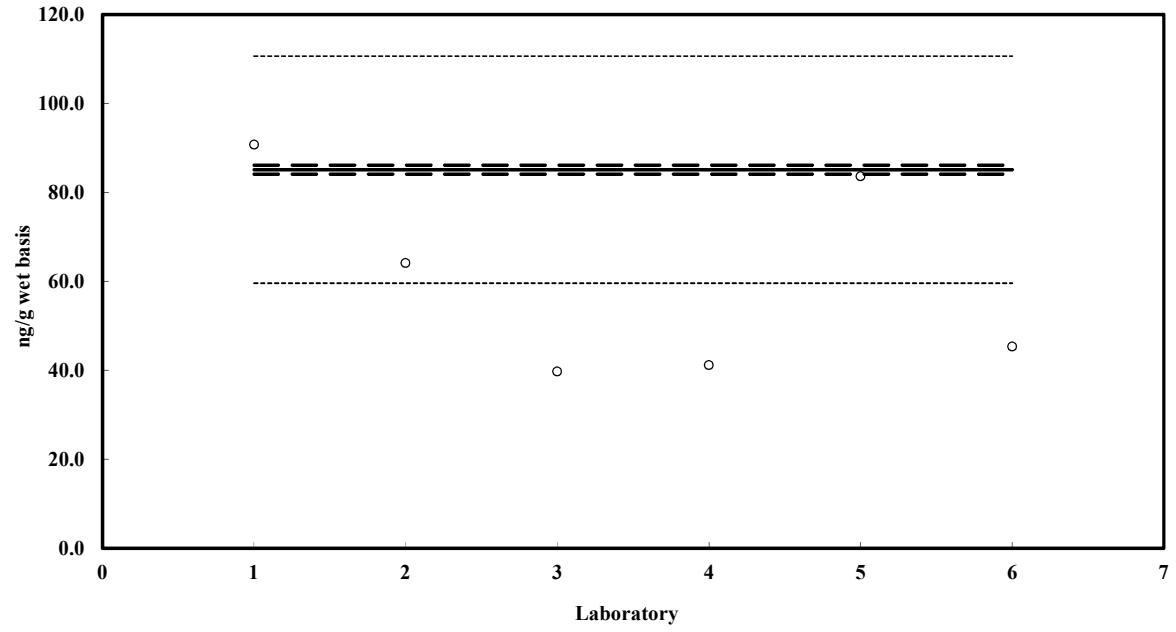
Control Sample

Median Value
± 1 Z
± 2 Z
± 3 Z

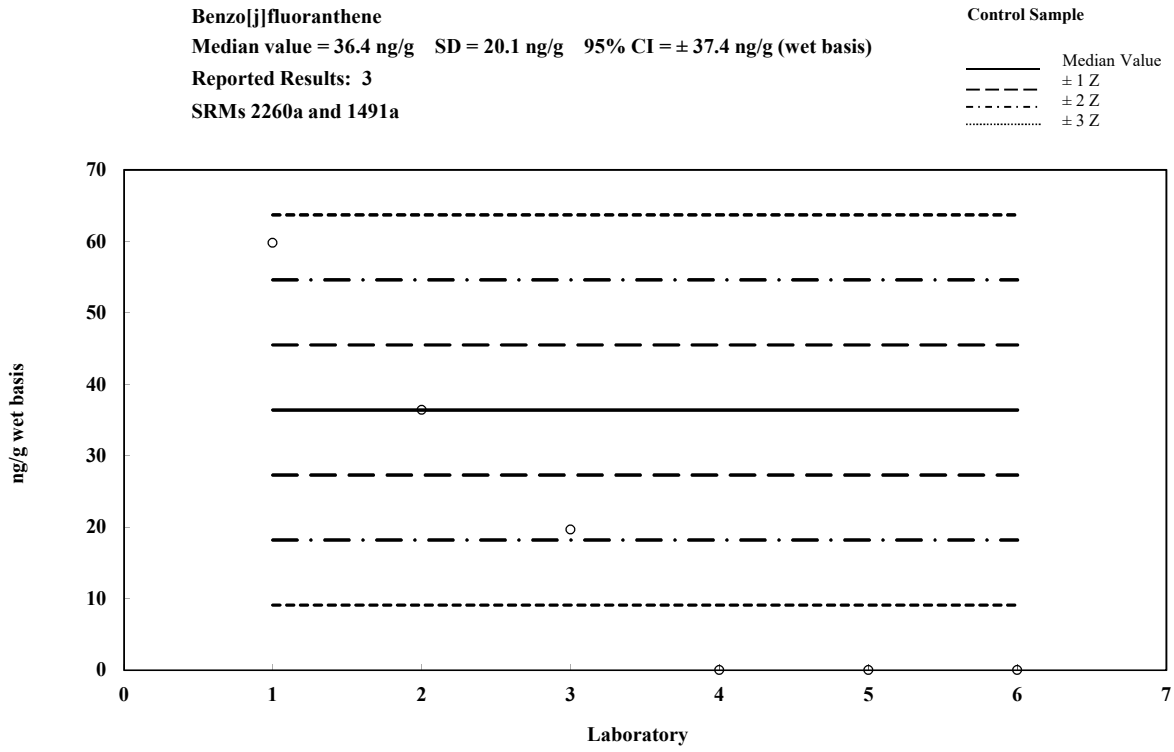


Benzo[ghi]perylene
Value = 85 ± 1 ng/g (wet basis)
Reported Results: 6
SRMs 2260a and 1491a

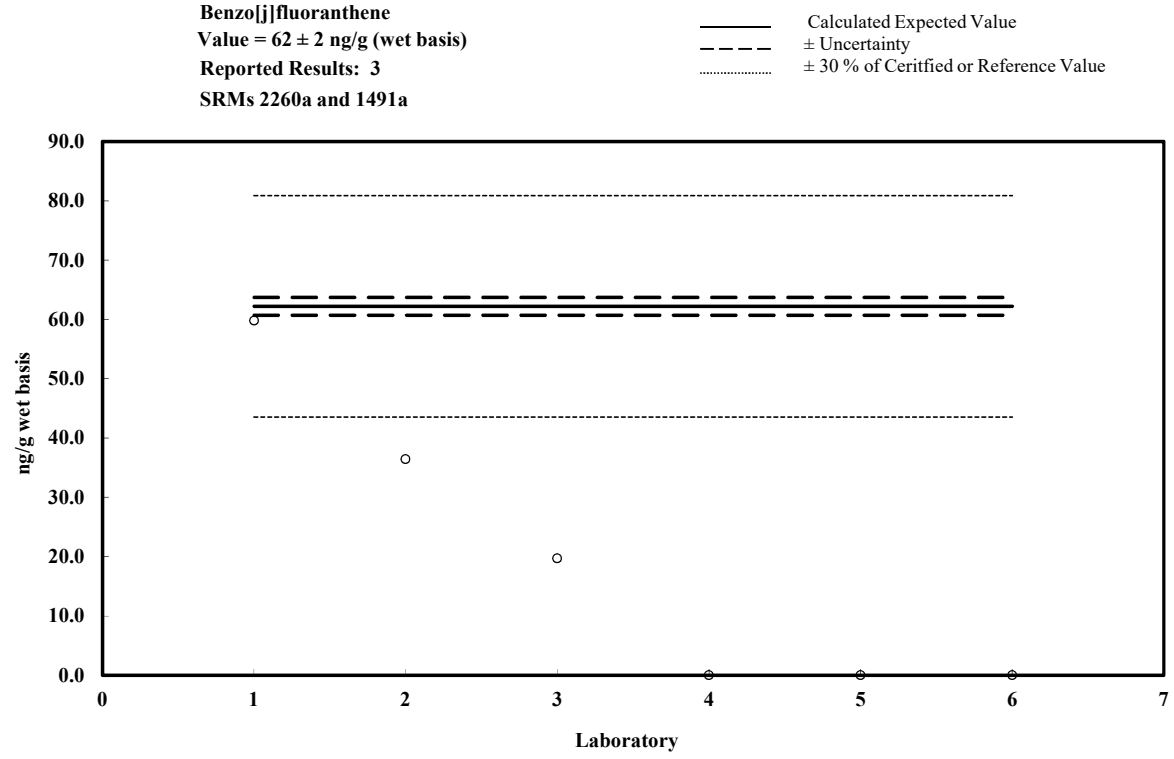
Calculated Expected Value
± Uncertainty
± 30 % of Ceritfied or Reference Value



Benzo[j]fluoranthene
Median value = 36.4 ng/g SD = 20.1 ng/g 95% CI = ± 37.4 ng/g (wet basis)
Reported Results: 3
SRMs 2260a and 1491a



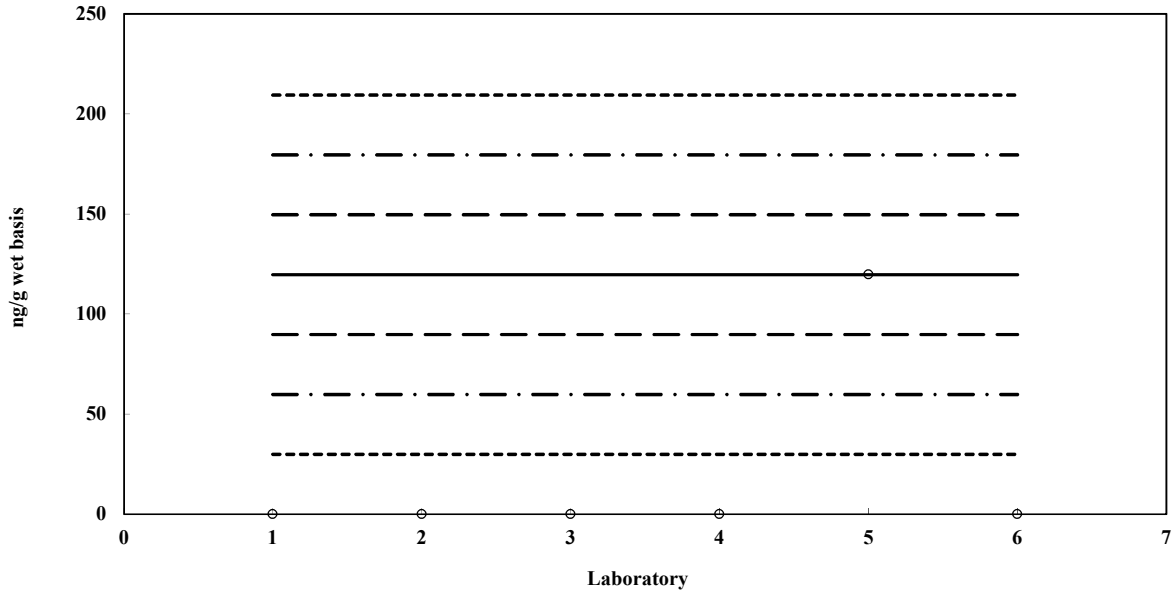
Benzo[j]fluoranthene
Value = 62 ± 2 ng/g (wet basis)
Reported Results: 3
SRMs 2260a and 1491a



Benzo[k]fluoranthene + Benzo[j]fluoranthene
Median value = 119.7 ng/g SD = 0 ng/g 95% CI = ± 0 ng/g (wet basis)
Reported Results: 1
SRMs 2260a and 1491a

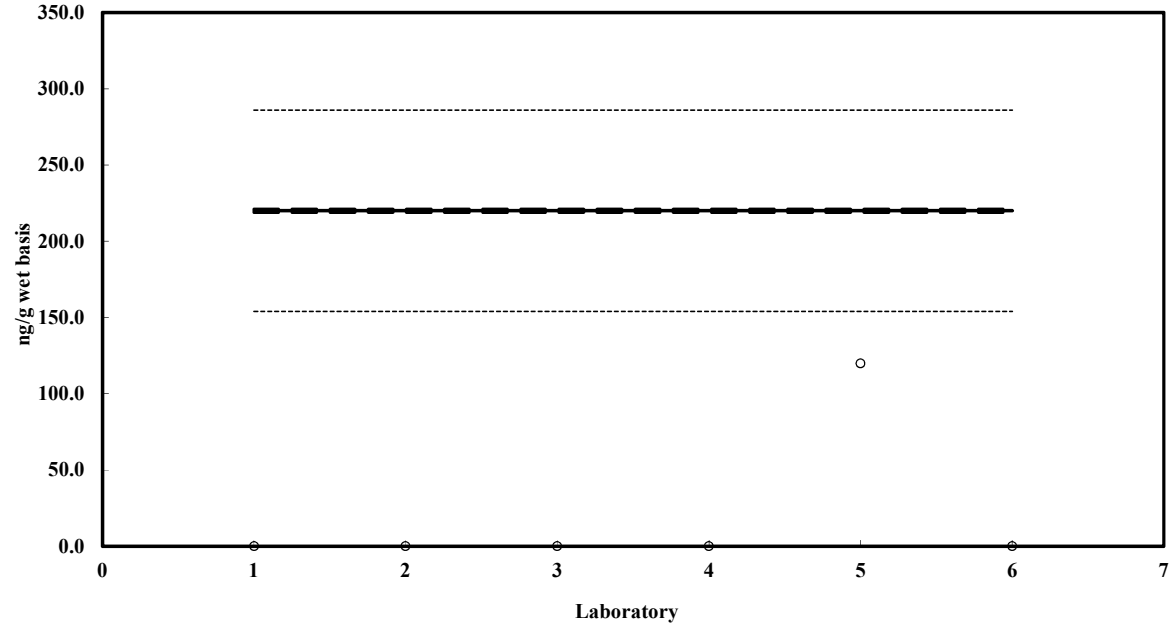
Control Sample

Median Value
± 1 Z
± 2 Z
± 3 Z

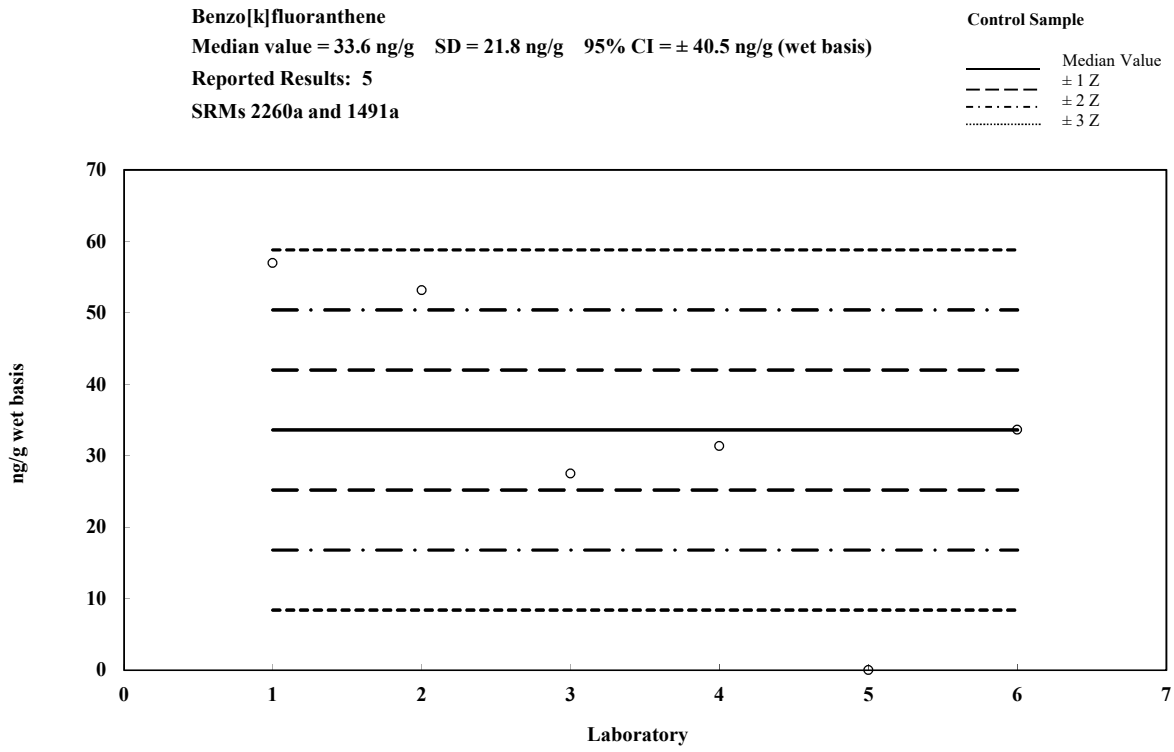


Benzo[k]fluoranthene + Benzo[j]fluoranthene
Value = 220 ± 1 ng/g (wet basis)
Reported Results: 1
SRMs 2260a and 1491a

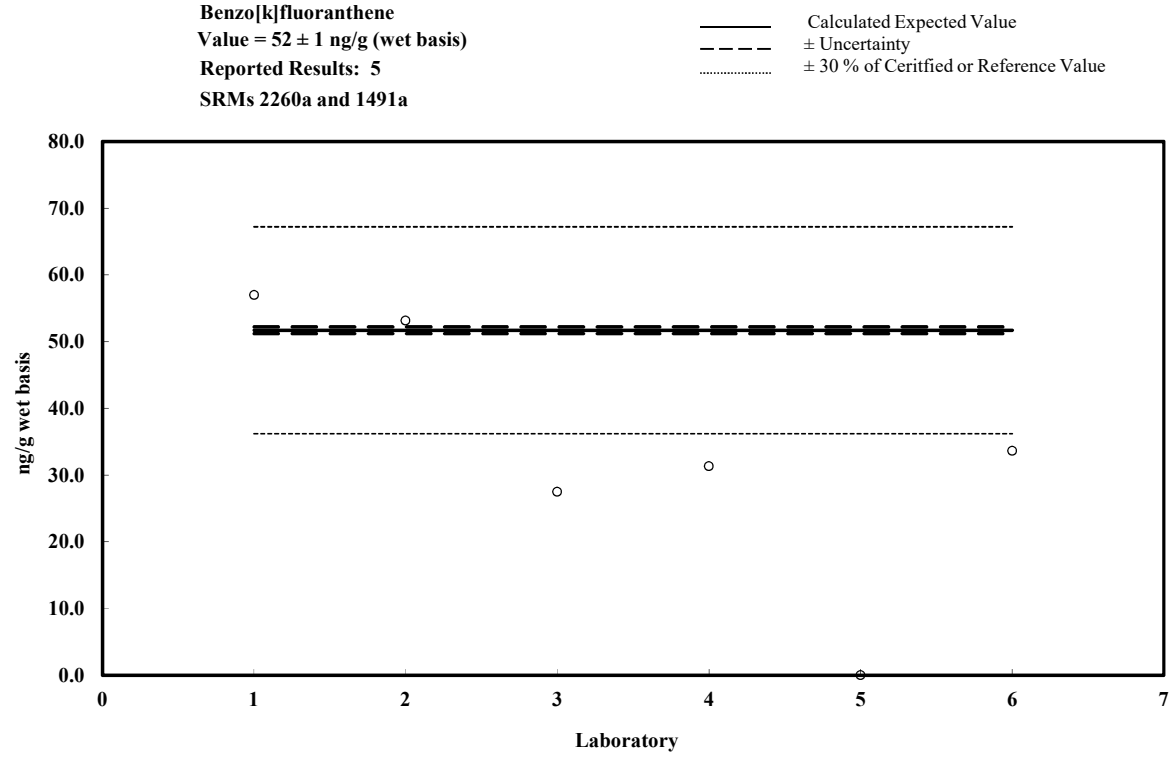
Calculated Expected Value
± Uncertainty
± 30 % of Ceritfied or Reference Value



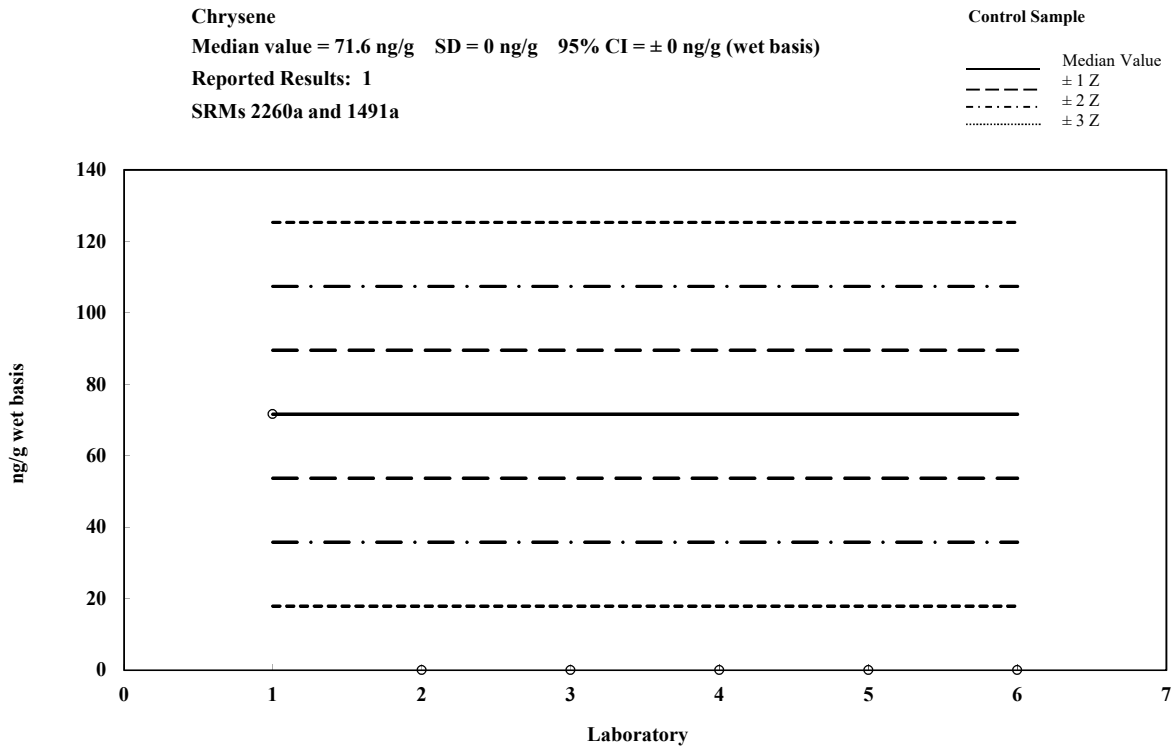
Benzo[k]fluoranthene
Median value = 33.6 ng/g SD = 21.8 ng/g 95% CI = ± 40.5 ng/g (wet basis)
Reported Results: 5
SRMs 2260a and 1491a



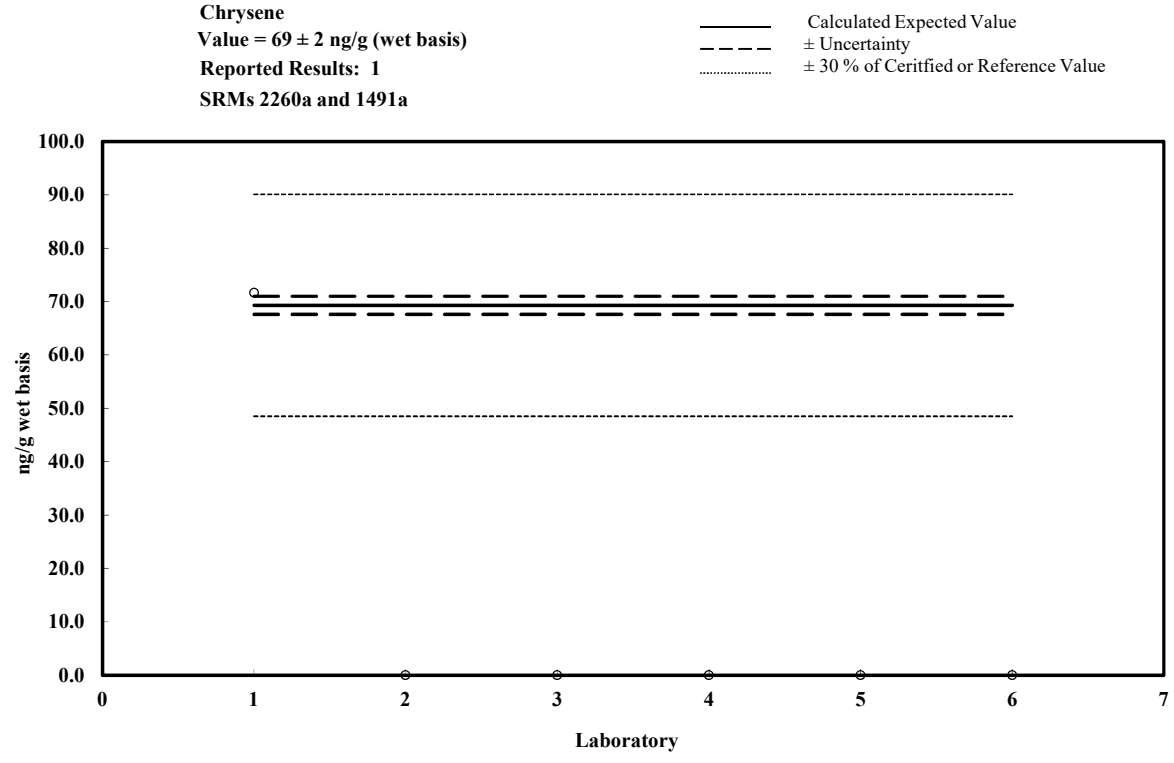
Benzo[k]fluoranthene
Value = 52 ± 1 ng/g (wet basis)
Reported Results: 5
SRMs 2260a and 1491a



Chrysene
Median value = 71.6 ng/g SD = 0 ng/g 95% CI = ± 0 ng/g (wet basis)
Reported Results: 1
SRMs 2260a and 1491a

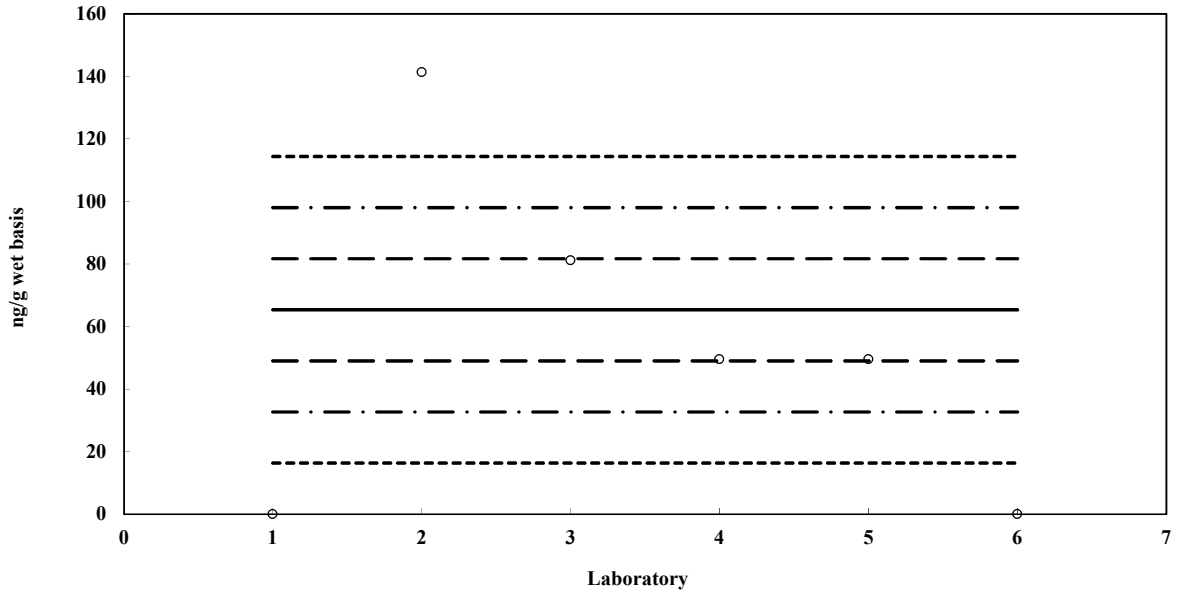


Chrysene
Value = 69 ± 2 ng/g (wet basis)
Reported Results: 1
SRMs 2260a and 1491a



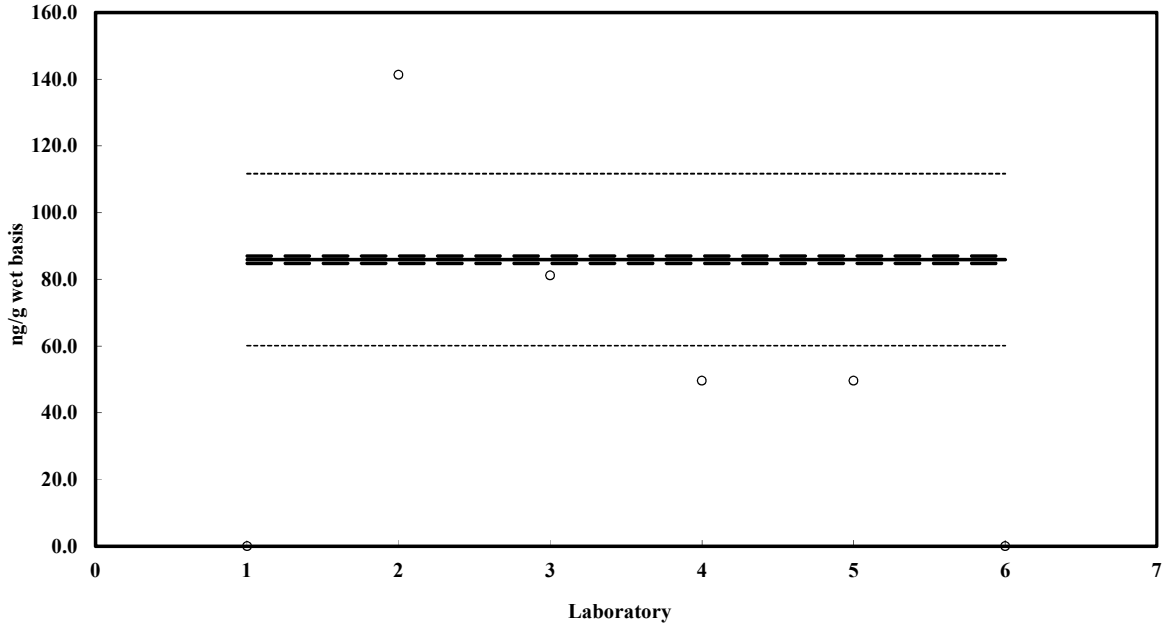
Chrysene+Triphenylene
Median value = 65.4 ng/g SD = 46.7 ng/g 95% CI = ± 86.9 ng/g (wet basis)
Reported Results: 4
SRMs 2260a and 1491a

Control Sample
Median Value
± 1 Z
± 2 Z
± 3 Z



Chrysene+Triphenylene
Value = 86 ± 1 ng/g (wet basis)
Reported Results: 4
SRMs 2260a and 1491a

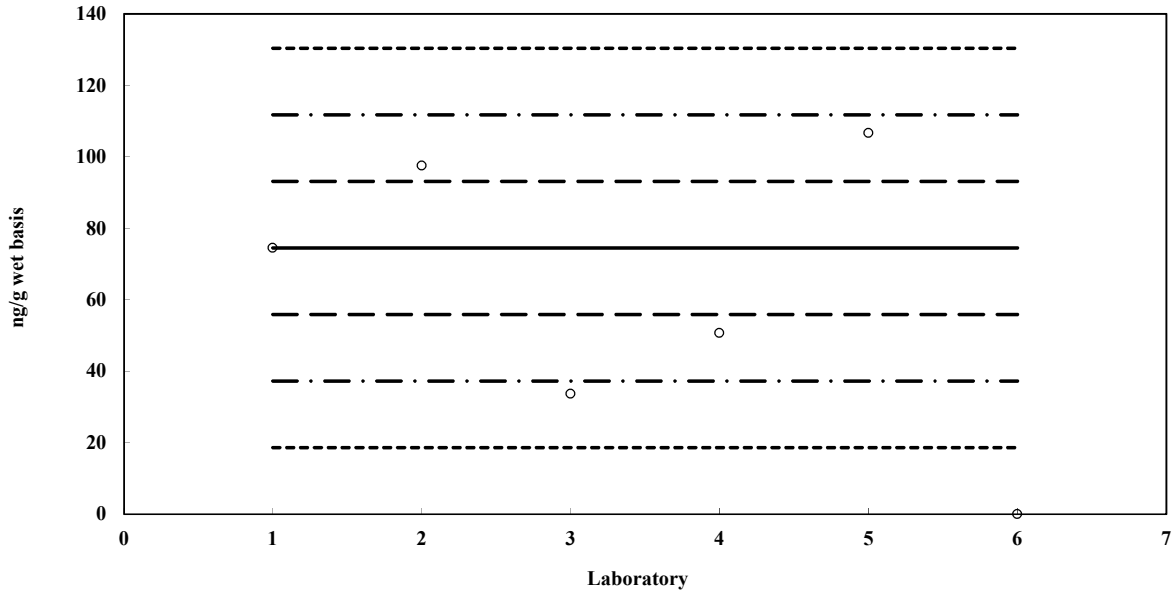
Calculated Expected Value
± Uncertainty
± 30 % of Ceritfied or Reference Value



Dibenz[a,h]anthracene
Median value = 74.5 ng/g SD = 46.9 ng/g 95% CI = ± 87.2 ng/g (wet basis)
Reported Results: 5
SRMs 2260a and 1491a

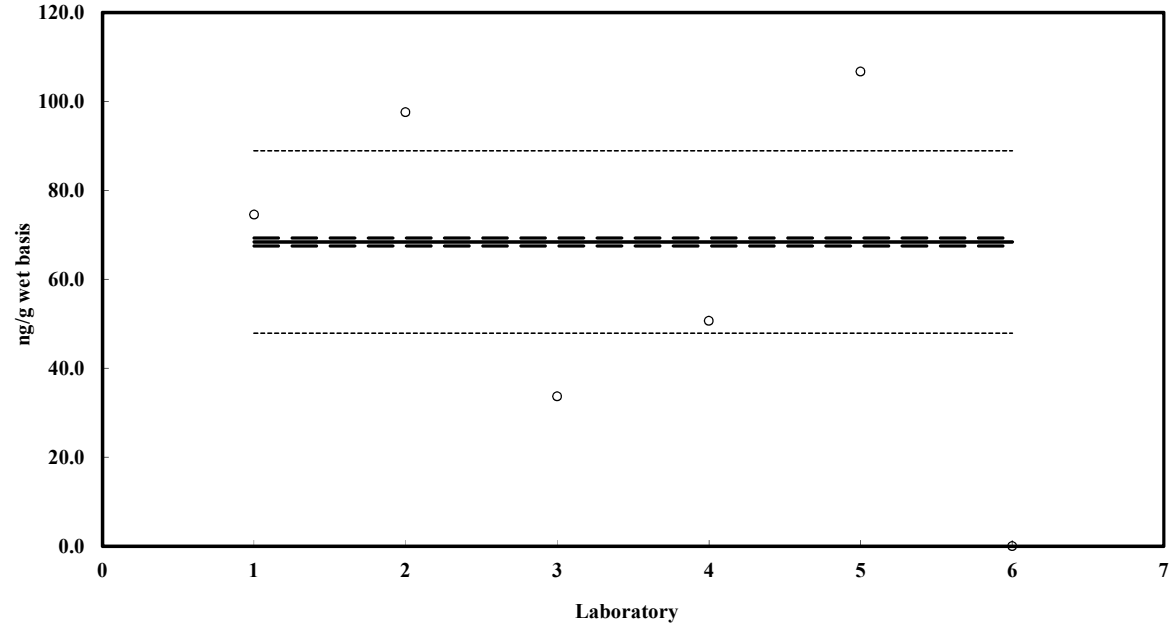
Control Sample

Median Value
± 1 Z
± 2 Z
± 3 Z



Dibenz[a,h]anthracene
Value = 68 ± 1 ng/g (wet basis)
Reported Results: 5
SRMs 2260a and 1491a

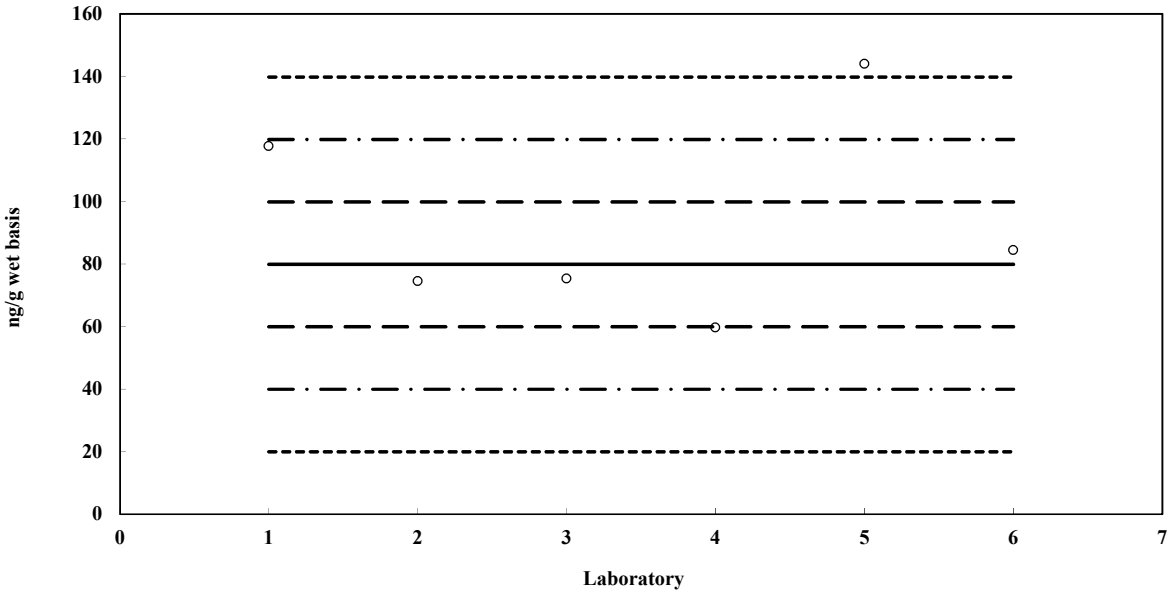
Calculated Expected Value
± Uncertainty
± 30 % of Ceritfied or Reference Value



Fluoranthene
Median value = 79.9 ng/g SD = 34.6 ng/g 95% CI = ± 64.4 ng/g (wet basis)
Reported Results: 6
SRMs 2260a and 1491a

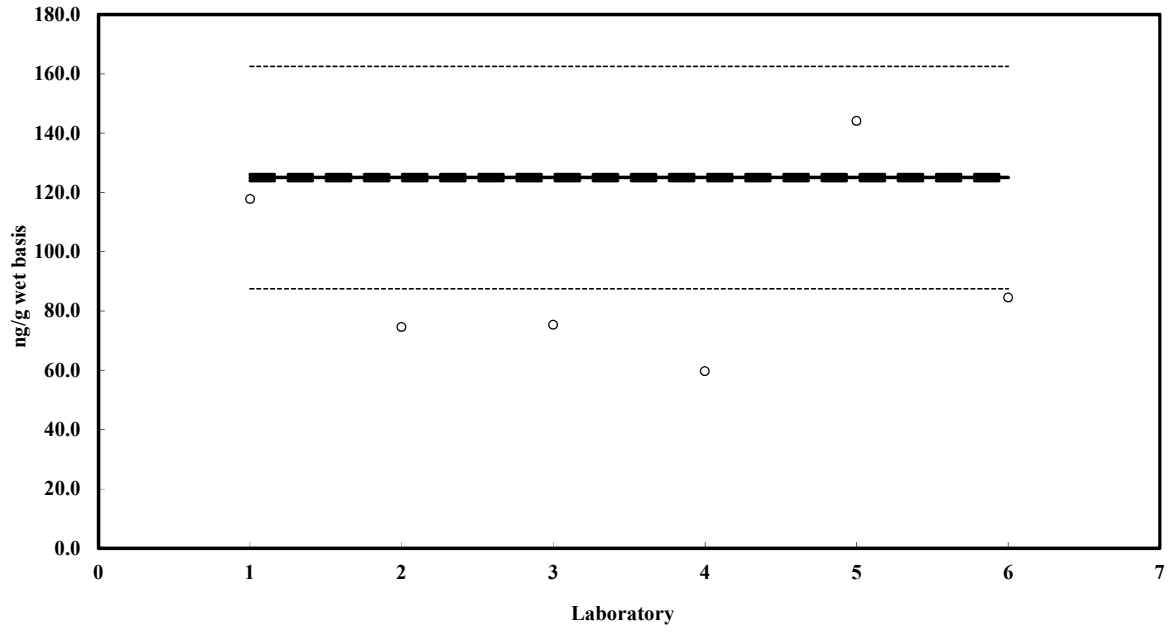
Control Sample

Median Value
± 1 Z
± 2 Z
± 3 Z



Fluoranthene
Value = 125 ± 1 ng/g (wet basis)
Reported Results: 6
SRMs 2260a and 1491a

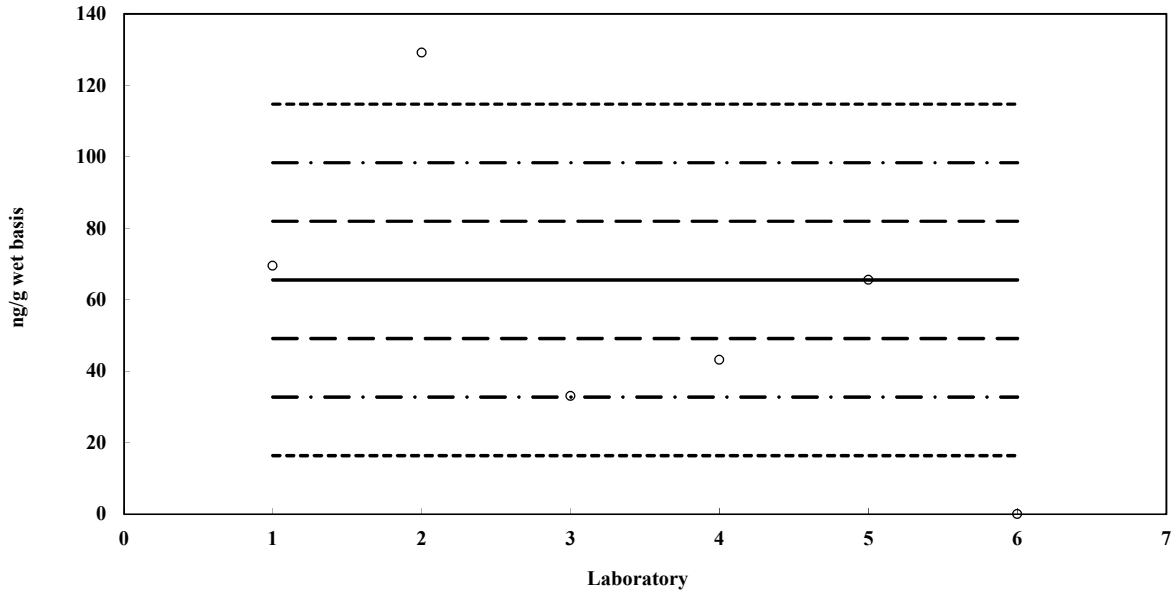
Calculated Expected Value
± Uncertainty
± 30 % of Ceritfied or Reference Value



Indeno[1,2,3-cd]pyrene
Median value = 65.6 ng/g SD = 26.3 ng/g 95% CI = ± 48.9 ng/g (wet basis)
Reported Results: 5
SRMs 2260a and 1491a

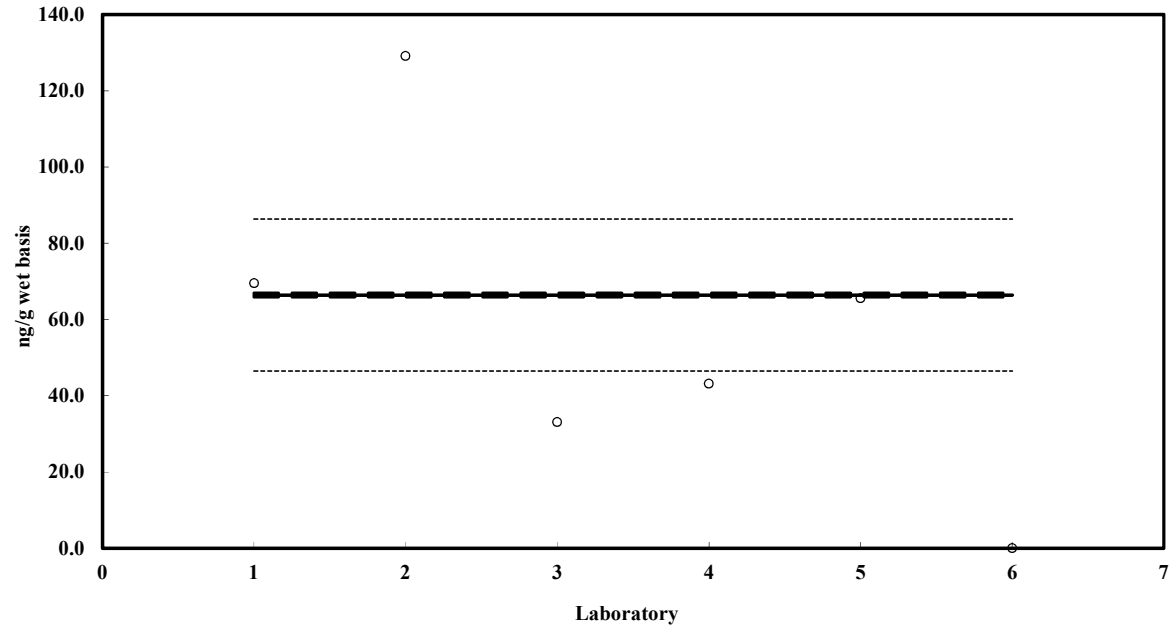
Control Sample

Median Value
± 1 Z
± 2 Z
± 3 Z



Indeno[1,2,3-cd]pyrene
Value = 66 ± 1 ng/g (wet basis)
Reported Results: 5
SRMs 2260a and 1491a

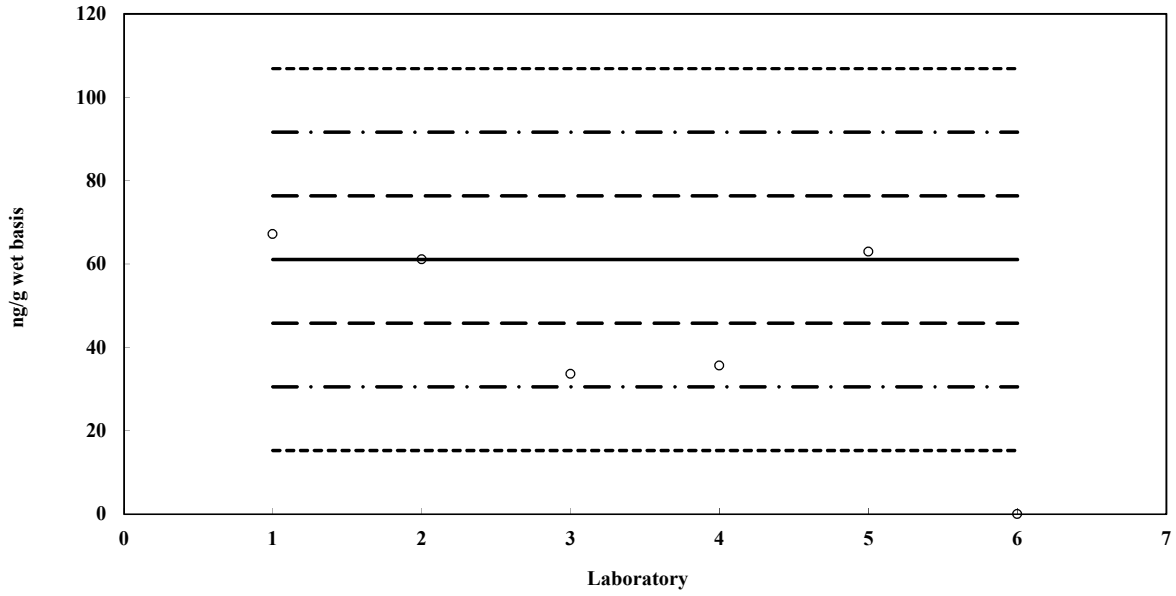
Calculated Expected Value
± Uncertainty
± 30 % of Ceritfied or Reference Value



Perylene
Median value = 61.1 ng/g SD = 27.3 ng/g 95% CI = ± 50.8 ng/g (wet basis)
Reported Results: 5
SRMs 2260a and 1491a

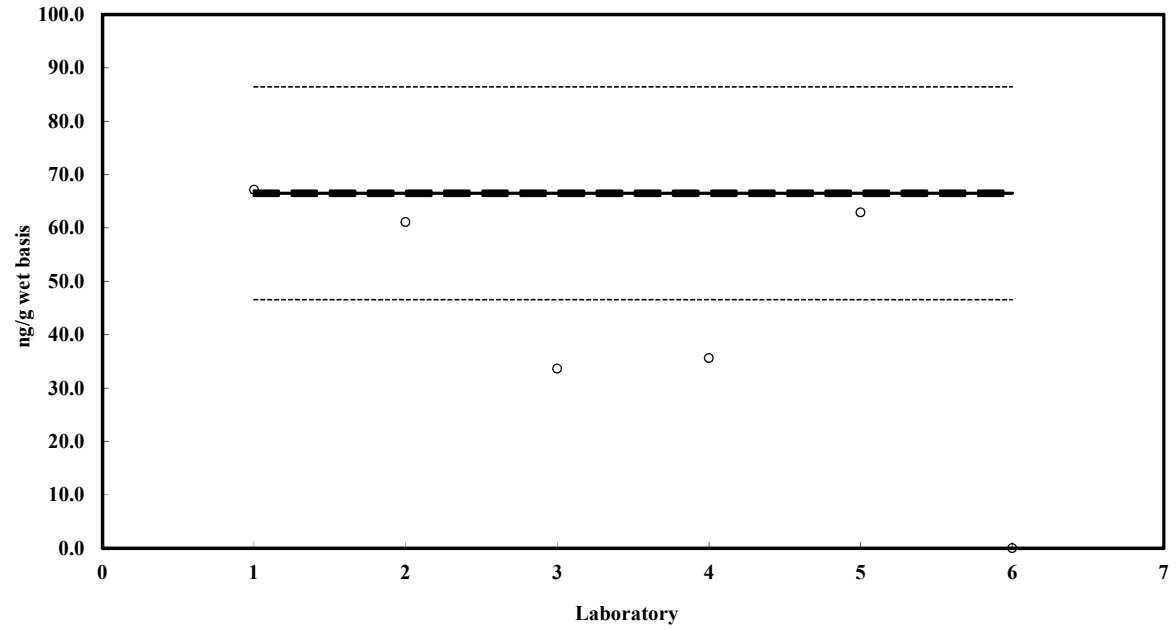
Control Sample

Median Value
± 1 Z
± 2 Z
± 3 Z



Perylene
Value = 67 ± 0 ng/g (wet basis)
Reported Results: 5
SRMs 2260a and 1491a

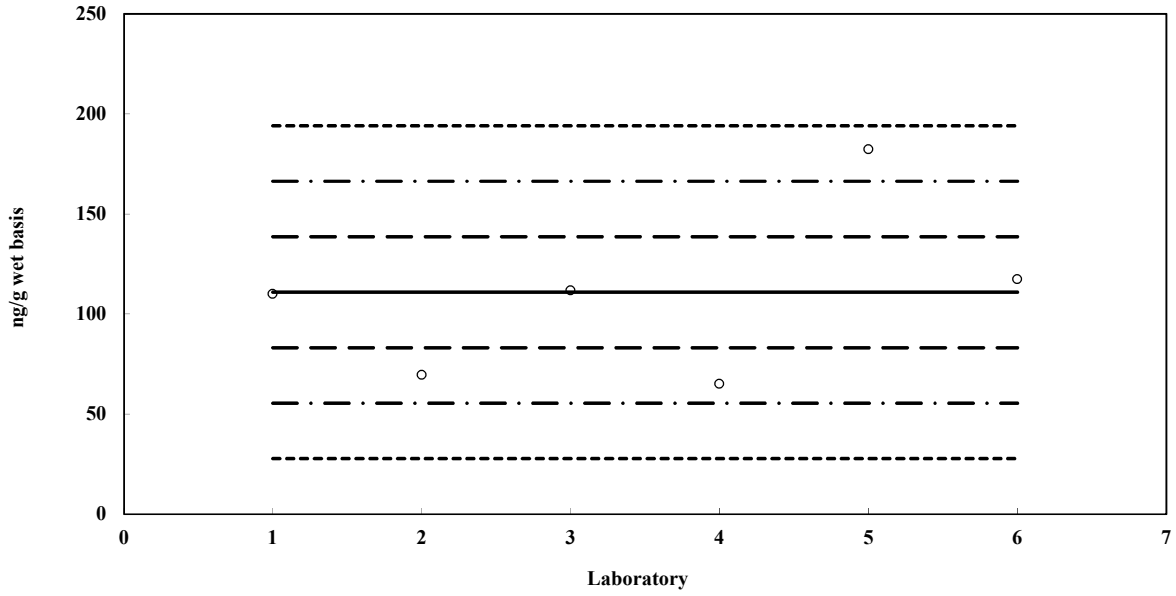
Calculated Expected Value
± Uncertainty
± 30 % of Ceritfied or Reference Value



Phenanthrene
Median value = 110.9 ng/g SD = 36.3 ng/g 95% CI = ± 67.5 ng/g (wet basis)
Reported Results: 6
SRMs 2260a and 1491a

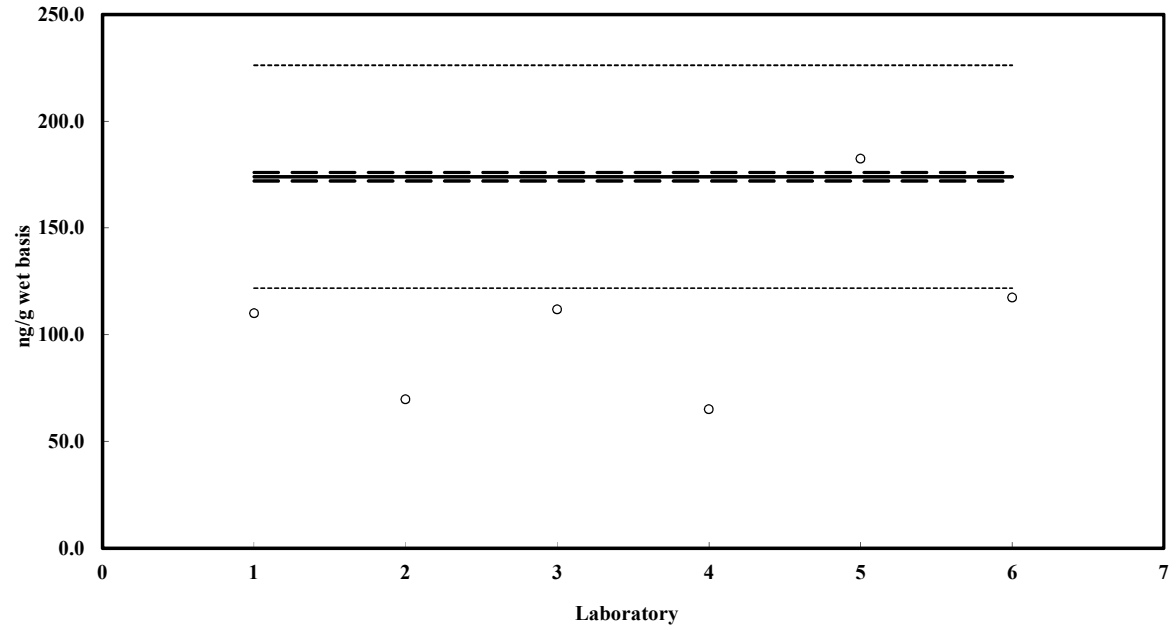
Control Sample

Median Value
± 1 Z
± 2 Z
± 3 Z



Phenanthrene
Value = 174 ± 2 ng/g (wet basis)
Reported Results: 6
SRMs 2260a and 1491a

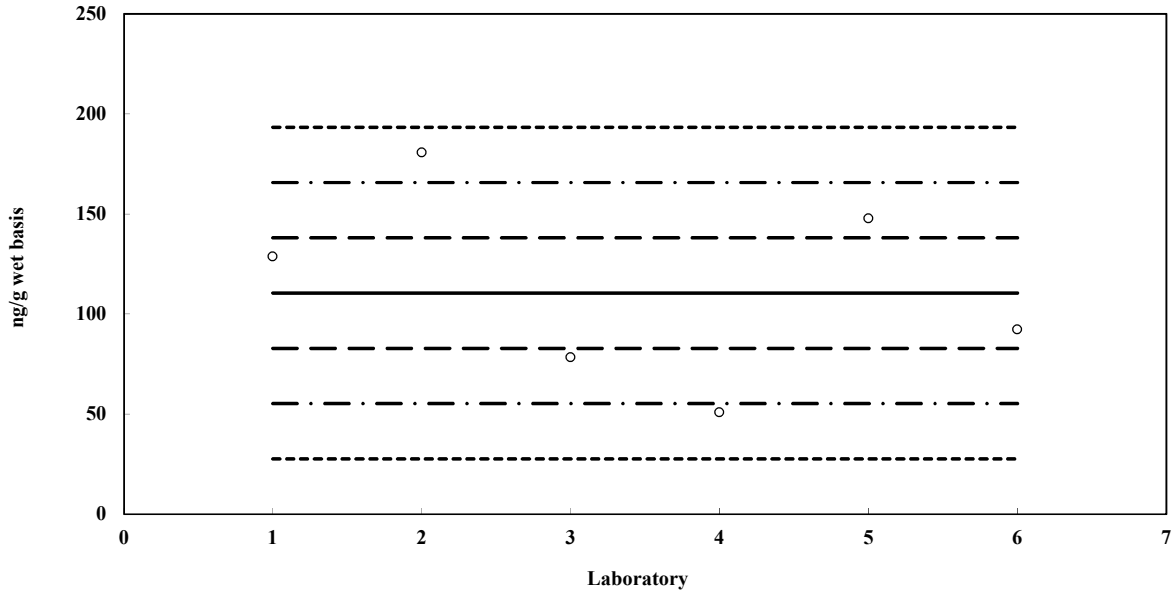
Calculated Expected Value
± Uncertainty
± 30 % of Ceritfied or Reference Value



Pyrene
Median value = 110.5 ng/g SD = 61.1 ng/g 95% CI = ± 113.6 ng/g (wet basis)
Reported Results: 6
SRMs 2260a and 1491a

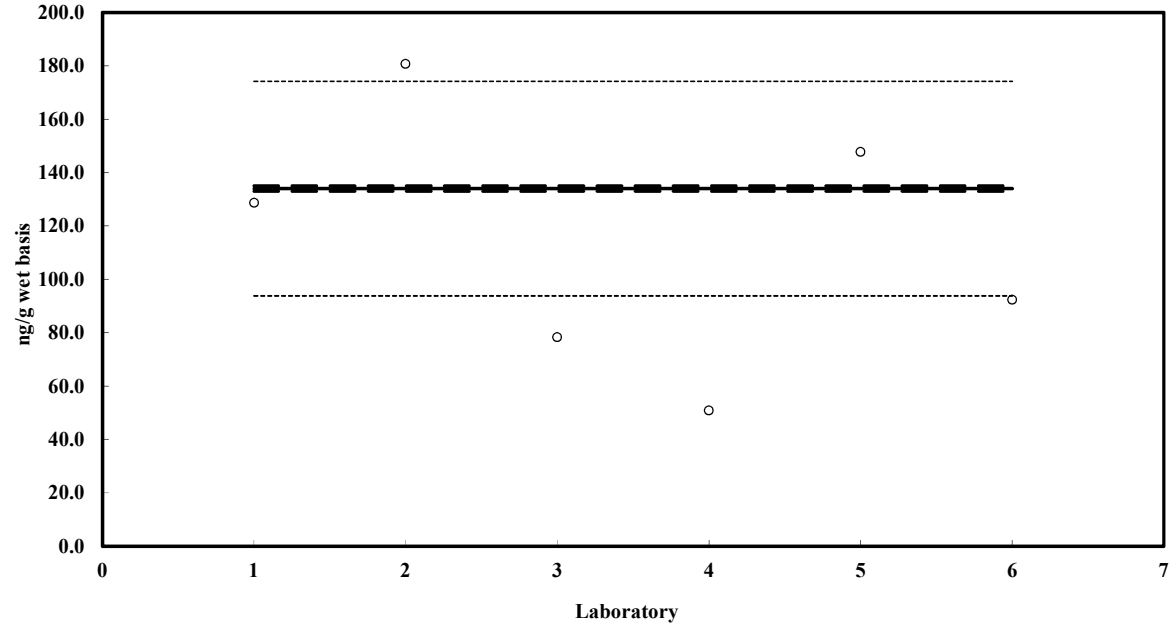
Control Sample

Median Value
± 1 Z
± 2 Z
± 3 Z

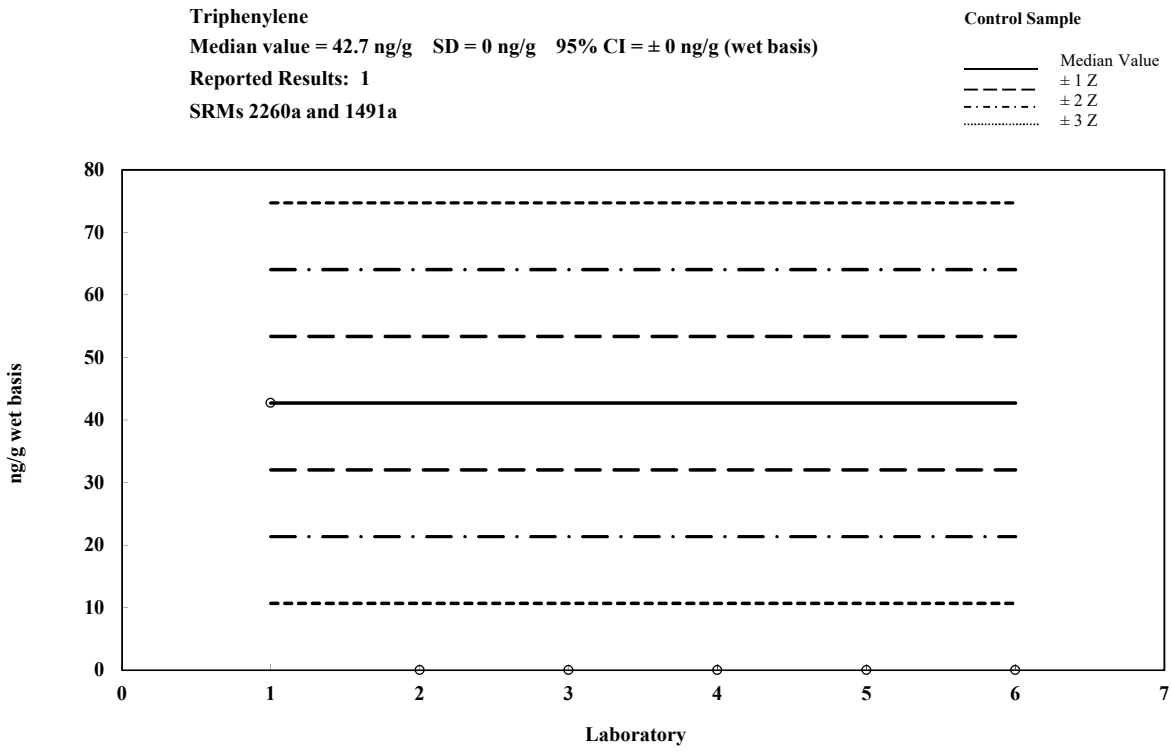


Pyrene
Value = 134 ± 1 ng/g (wet basis)
Reported Results: 6
SRMs 2260a and 1491a

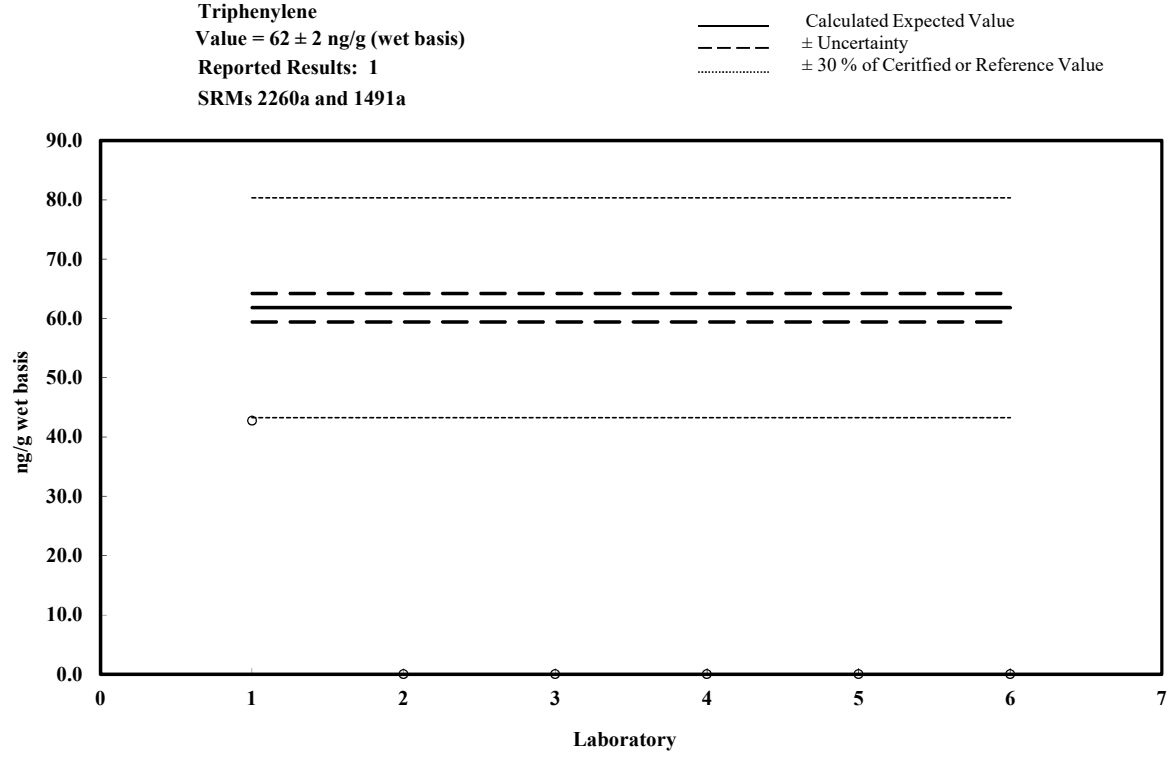
Calculated Expected Value
± Uncertainty
± 30 % of Ceritfied or Reference Value



Triphenylene
Median value = 42.7 ng/g SD = 0 ng/g 95% CI = ± 0 ng/g (wet basis)
Reported Results: 1
SRMs 2260a and 1491a

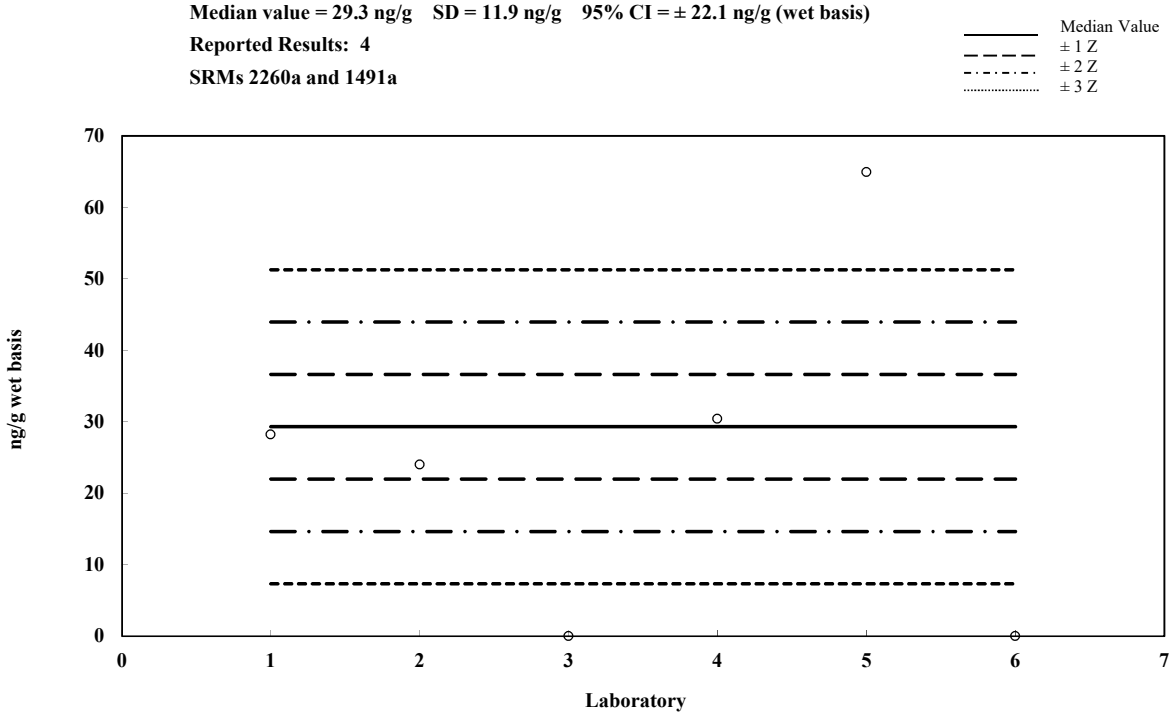


Triphenylene
Value = 62 ± 2 ng/g (wet basis)
Reported Results: 1
SRMs 2260a and 1491a

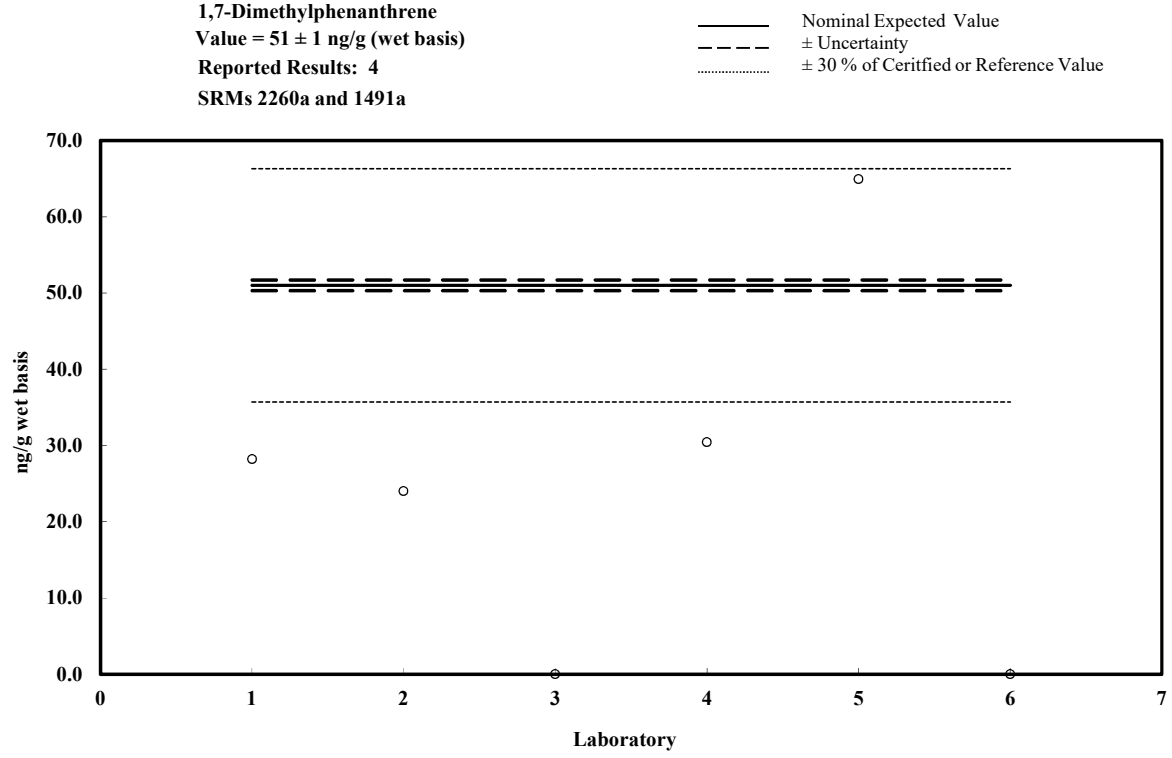


1,7-Dimethylphenanthrene
Median value = 29.3 ng/g SD = 11.9 ng/g 95% CI = ± 22.1 ng/g (wet basis)
Reported Results: 4
SRMs 2260a and 1491a

Extract 1

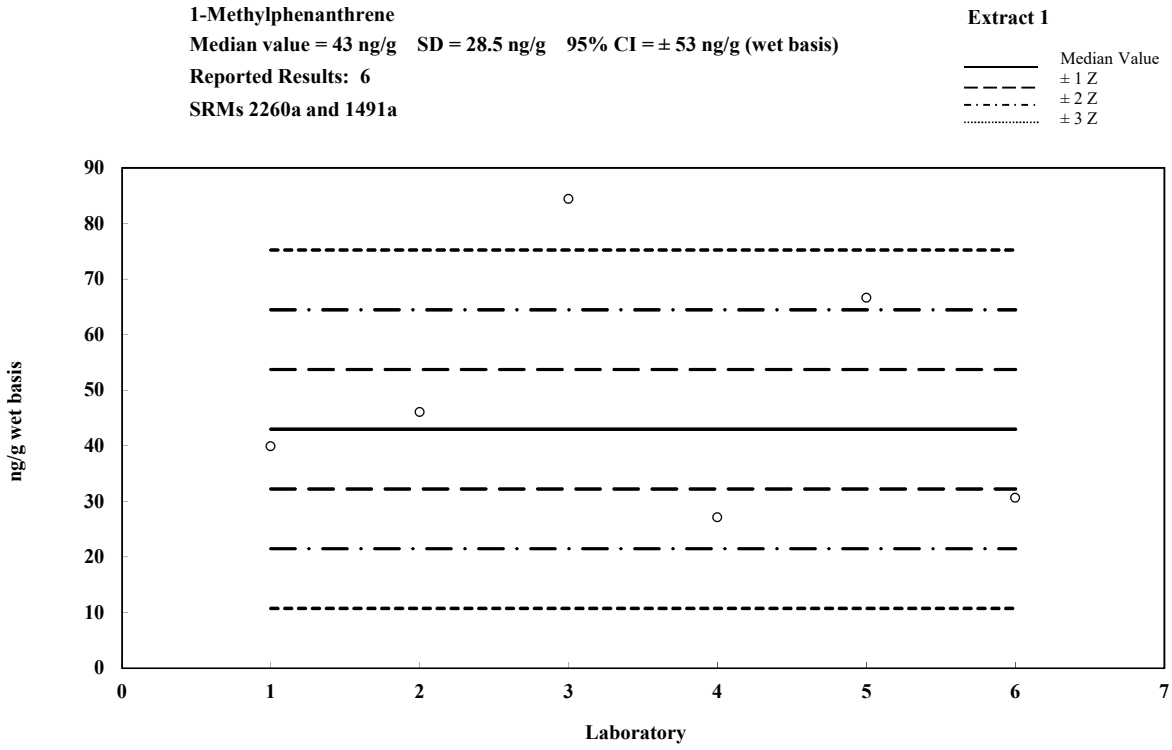


1,7-Dimethylphenanthrene
Value = 51 ± 1 ng/g (wet basis)
Reported Results: 4
SRMs 2260a and 1491a

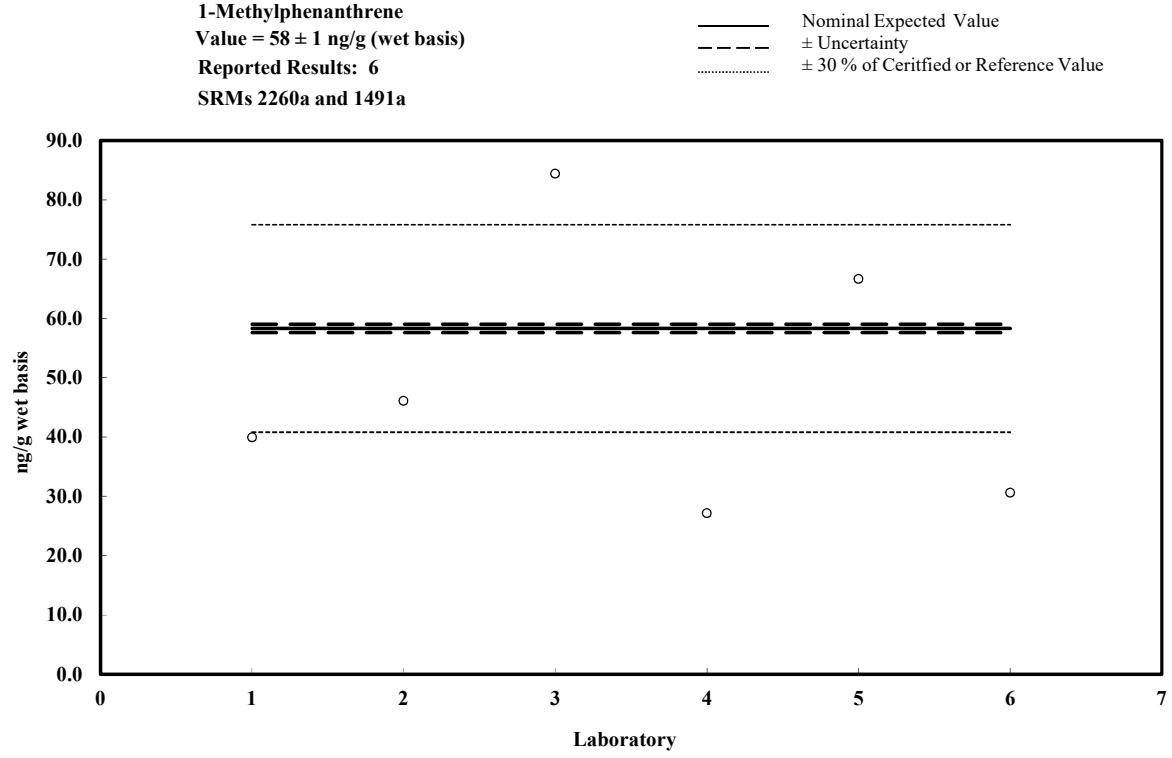


1-Methylphenanthrene
Median value = 43 ng/g SD = 28.5 ng/g 95% CI = ± 53 ng/g (wet basis)
Reported Results: 6
SRMs 2260a and 1491a

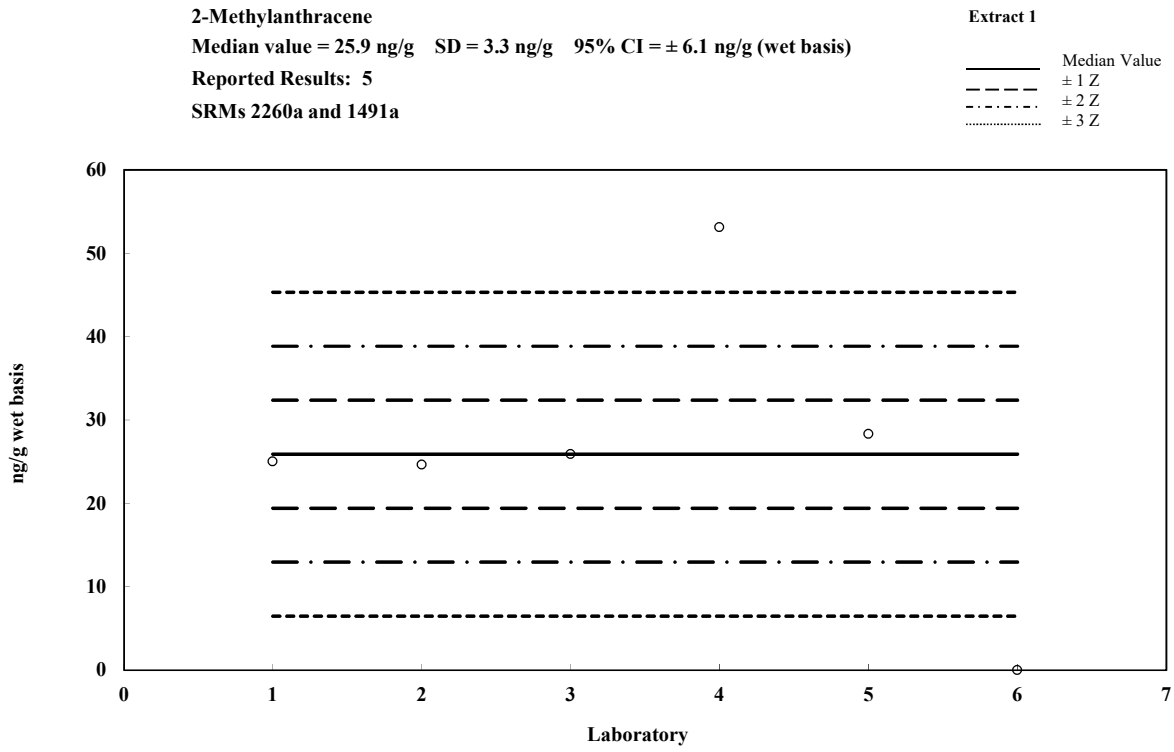
Extract 1



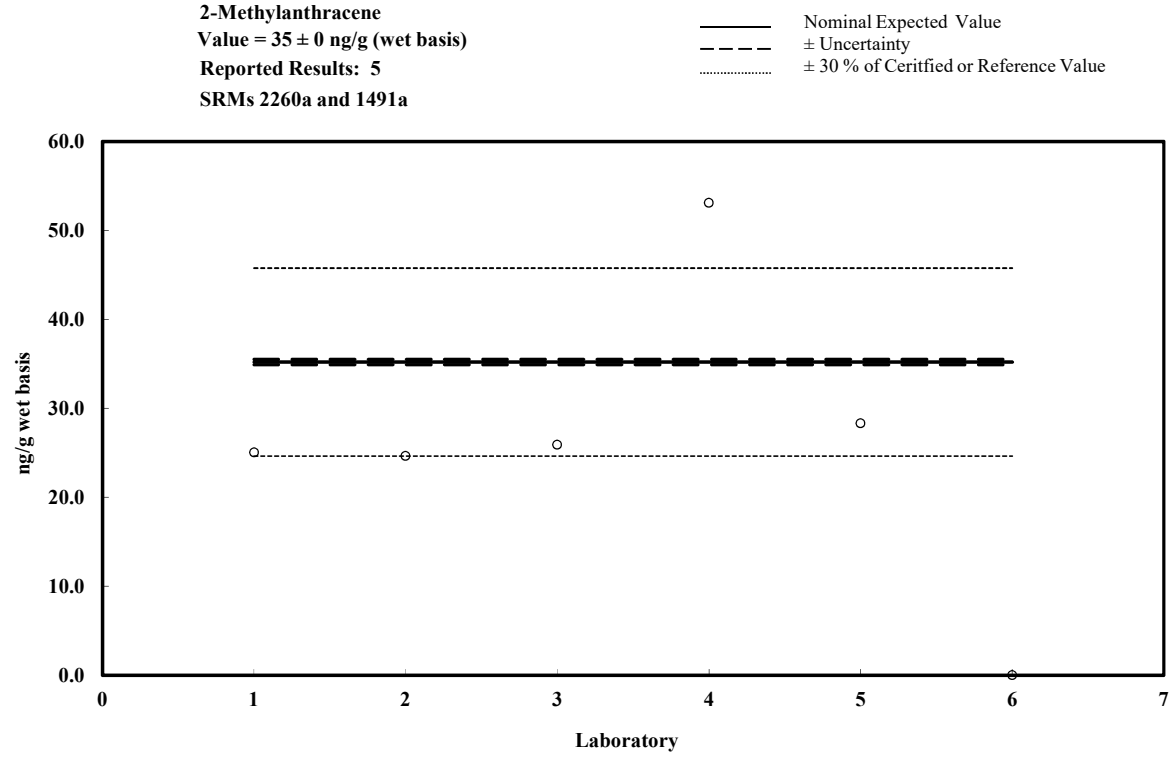
1-Methylphenanthrene
Value = 58 ± 1 ng/g (wet basis)
Reported Results: 6
SRMs 2260a and 1491a



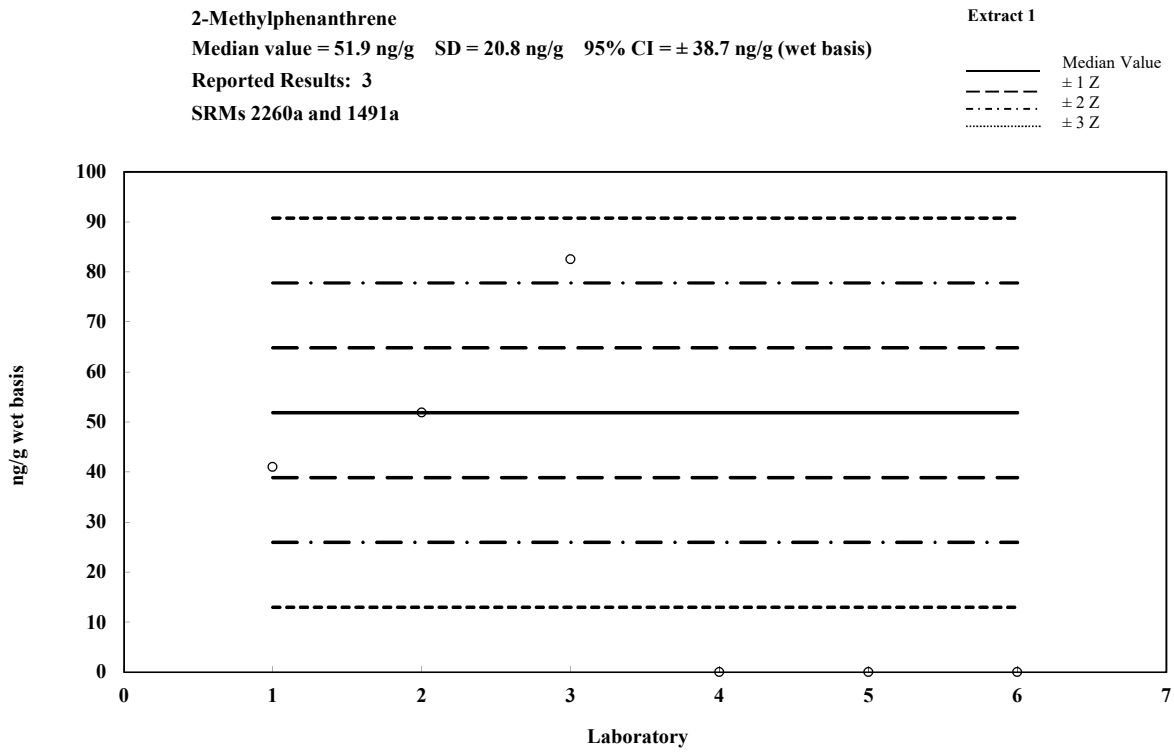
2-Methylanthracene
Median value = 25.9 ng/g SD = 3.3 ng/g 95% CI = ± 6.1 ng/g (wet basis)
Reported Results: 5
SRMs 2260a and 1491a



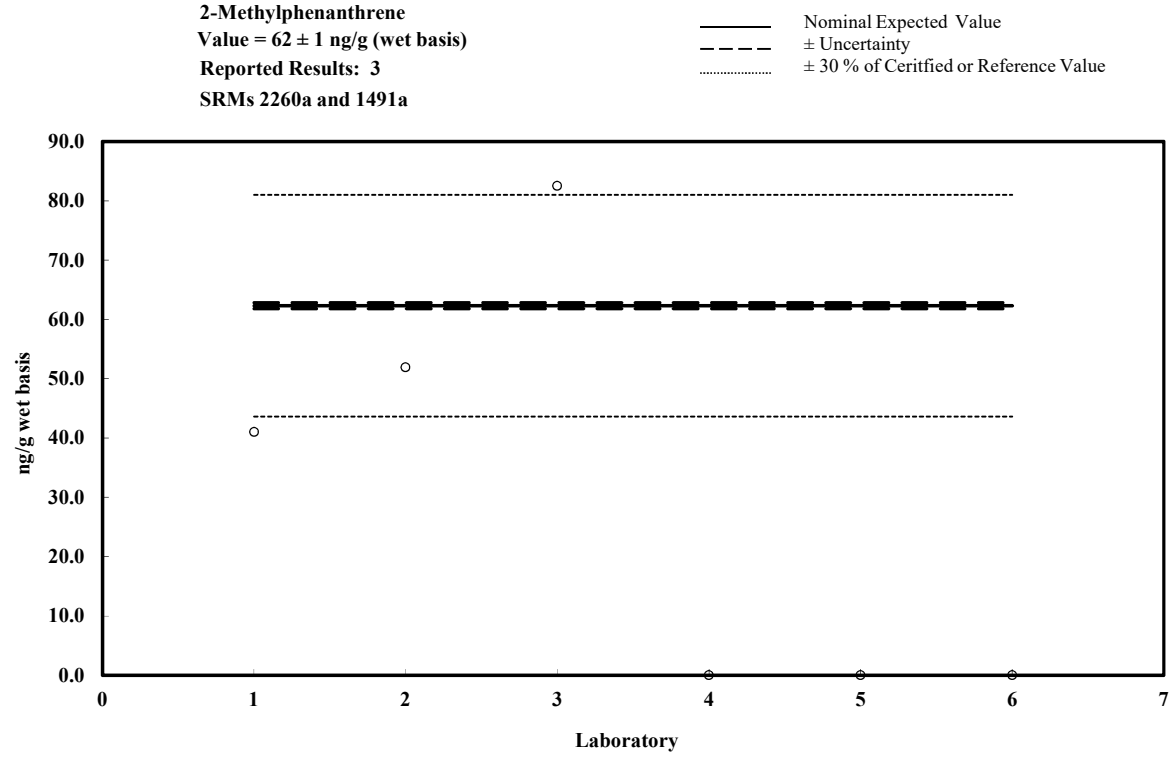
2-Methylanthracene
Value = 35 ± 0 ng/g (wet basis)
Reported Results: 5
SRMs 2260a and 1491a



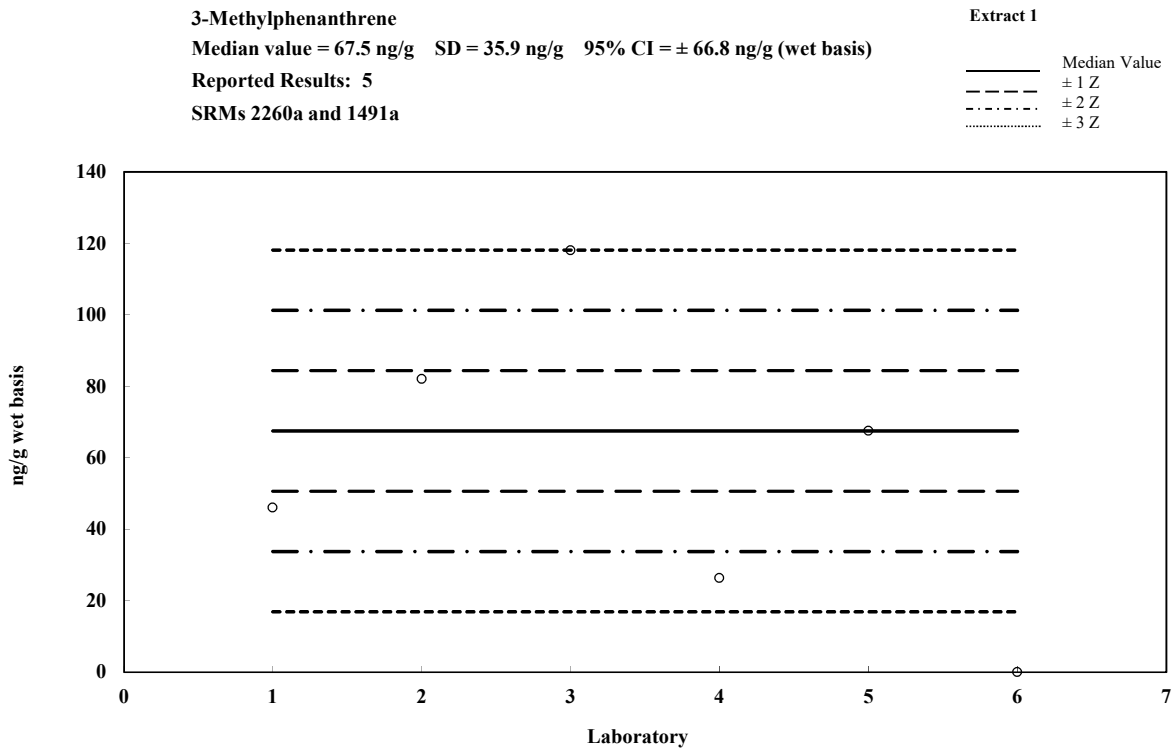
2-Methylphenanthrene
Median value = 51.9 ng/g SD = 20.8 ng/g 95% CI = ± 38.7 ng/g (wet basis)
Reported Results: 3
SRMs 2260a and 1491a



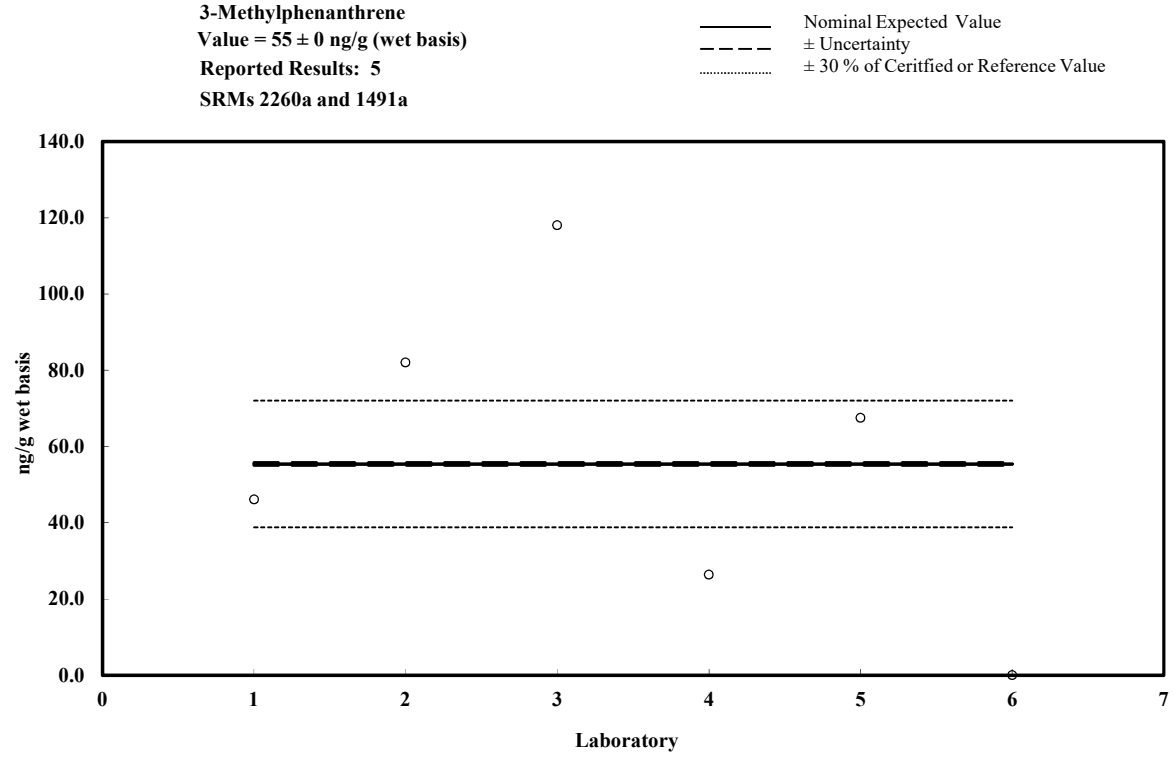
2-Methylphenanthrene
Value = 62 ± 1 ng/g (wet basis)
Reported Results: 3
SRMs 2260a and 1491a

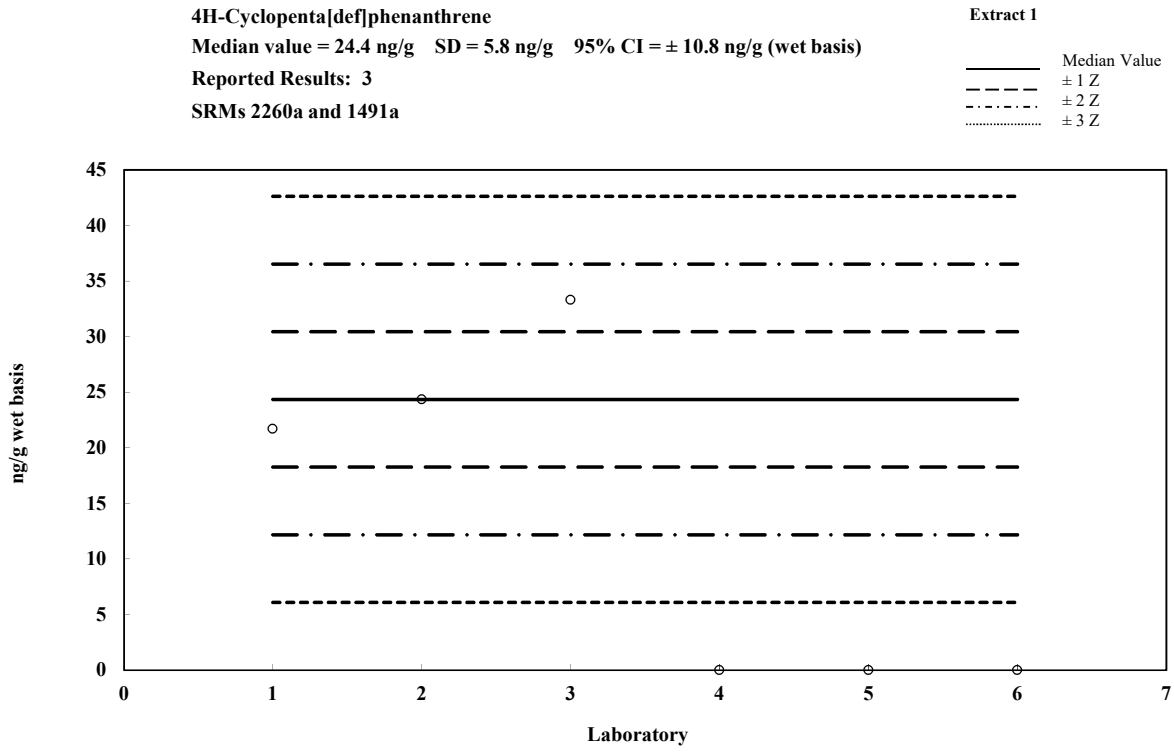


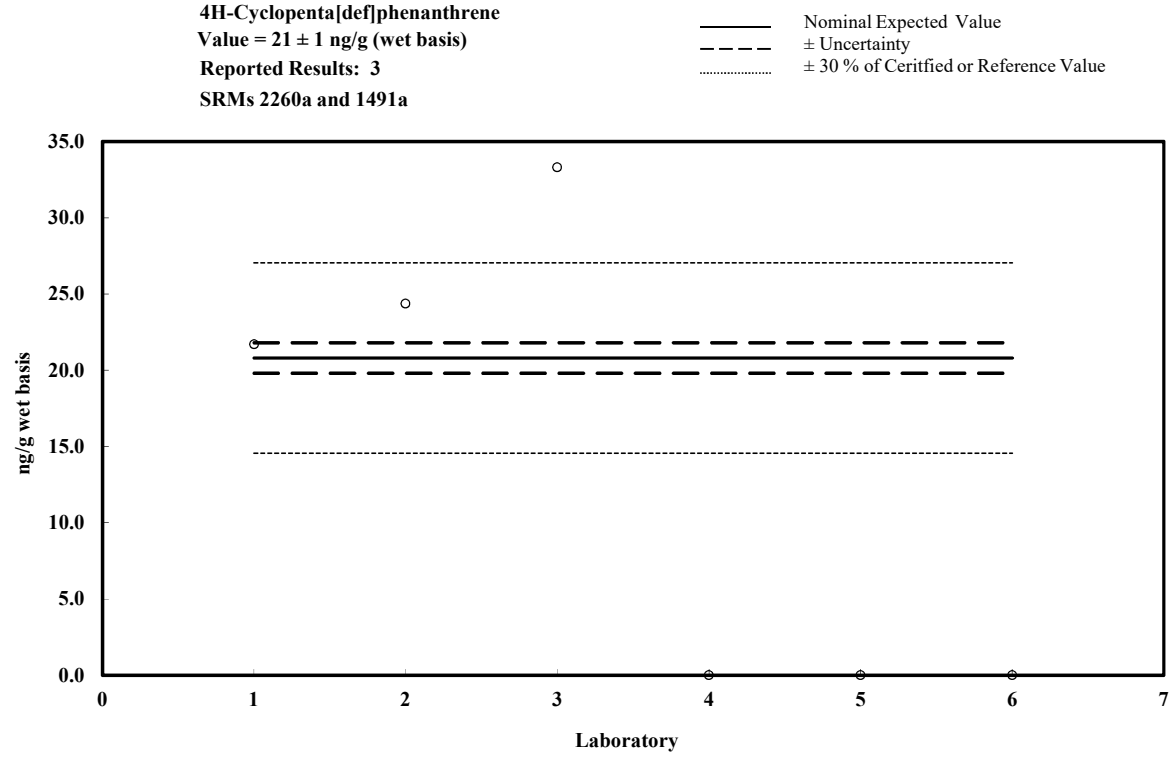
3-Methylphenanthrene
Median value = 67.5 ng/g SD = 35.9 ng/g 95% CI = ± 66.8 ng/g (wet basis)
Reported Results: 5
SRMs 2260a and 1491a



3-Methylphenanthrene
Value = 55 ± 0 ng/g (wet basis)
Reported Results: 5
SRMs 2260a and 1491a





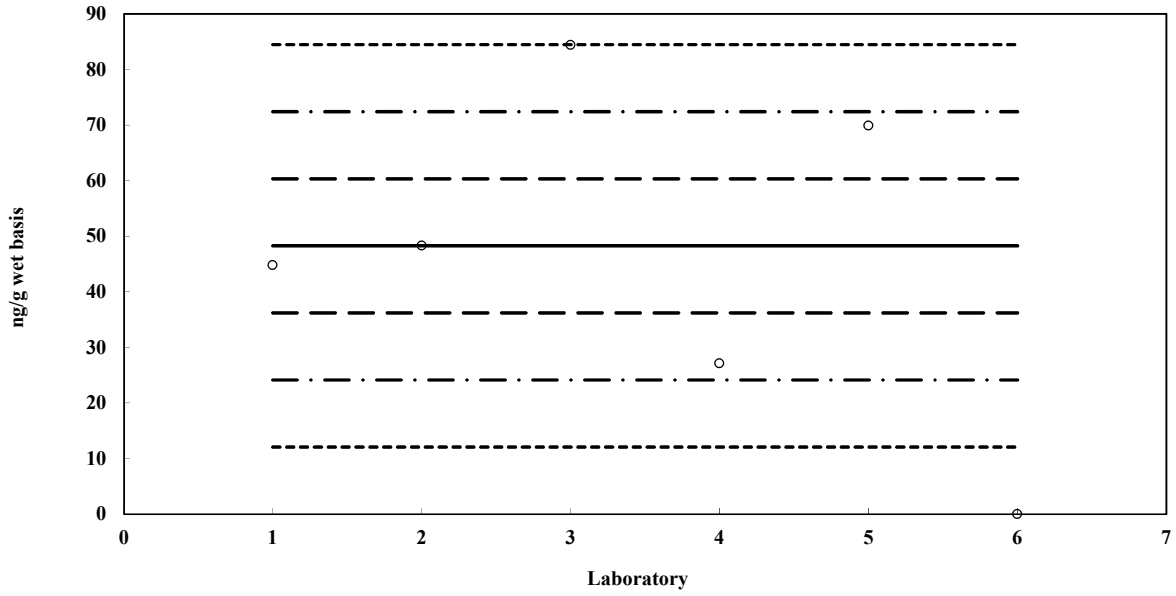


9-Methylphenanthrene **Extract 1**

Median value = 48.3 ng/g SD = 25.1 ng/g 95% CI = ± 46.7 ng/g (wet basis)

Reported Results: 5

SRMs 2260a and 1491a

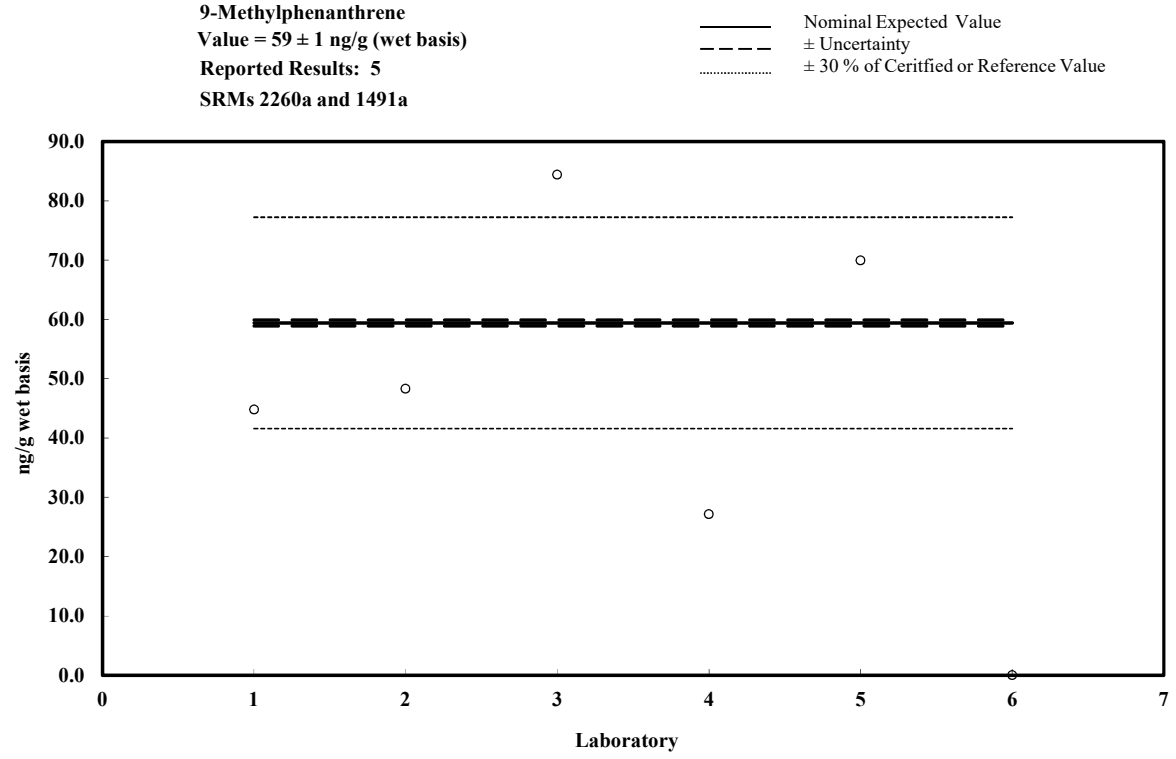


9-Methylphenanthrene

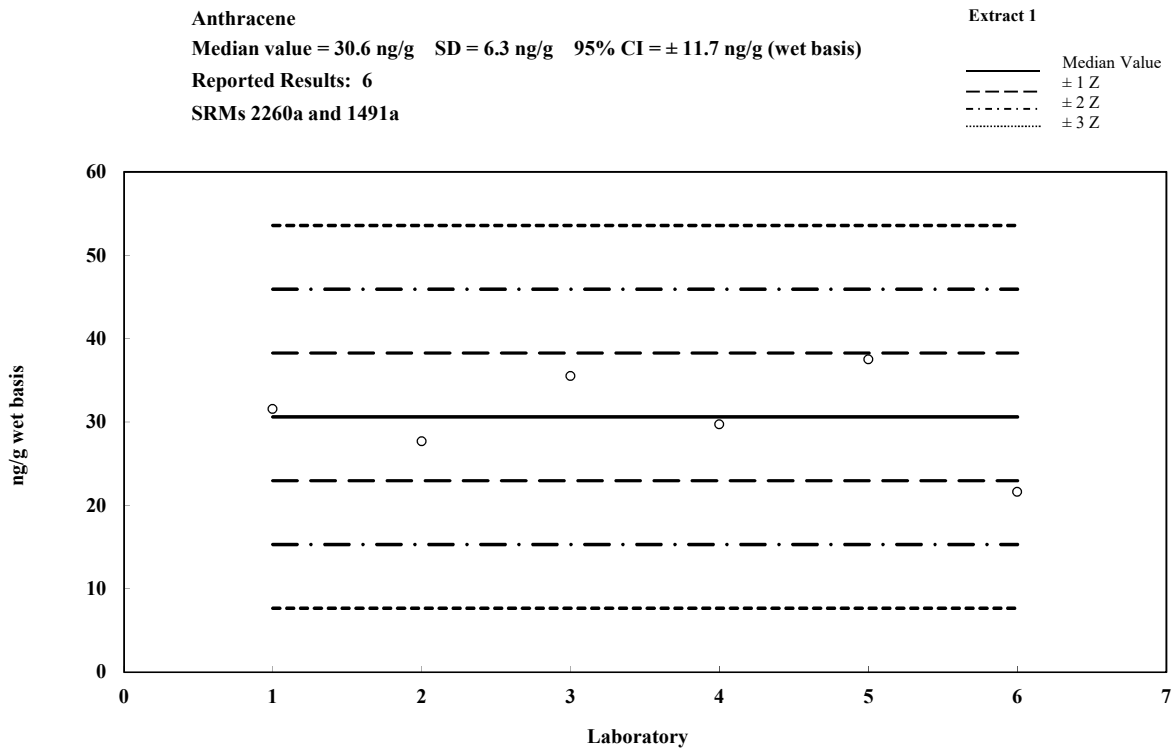
Value = 59 ± 1 ng/g (wet basis)

Reported Results: 5

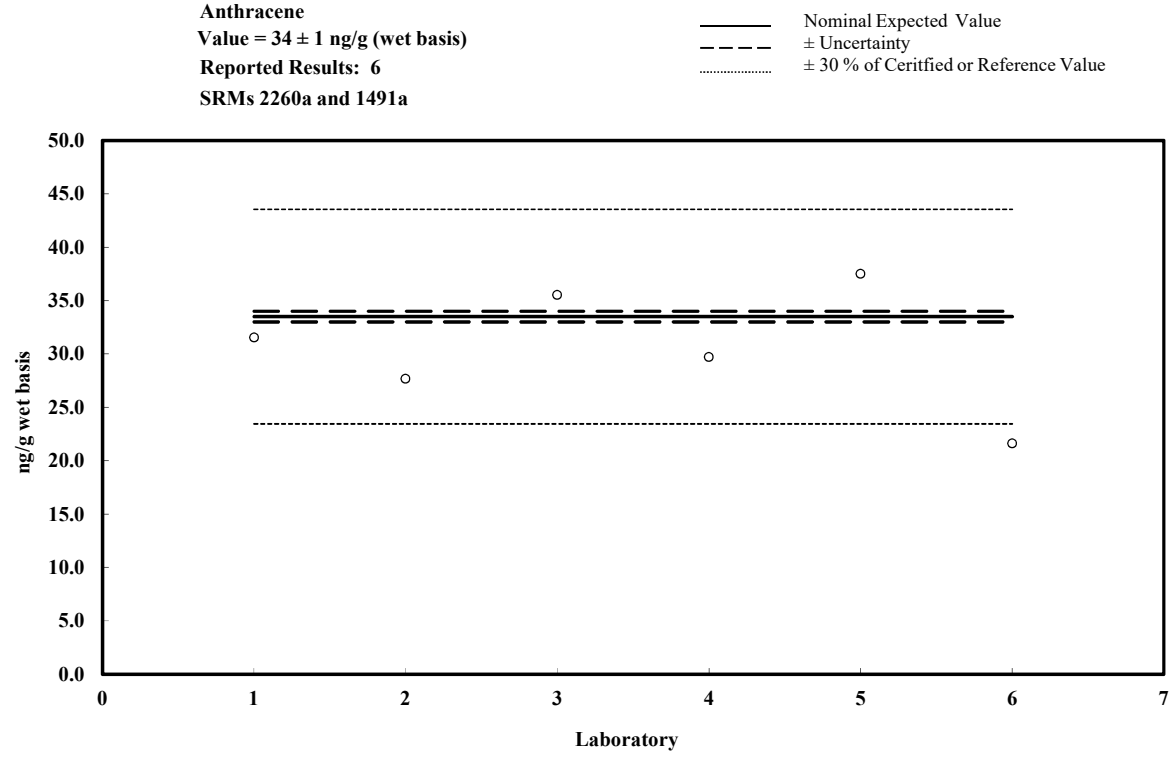
SRMs 2260a and 1491a



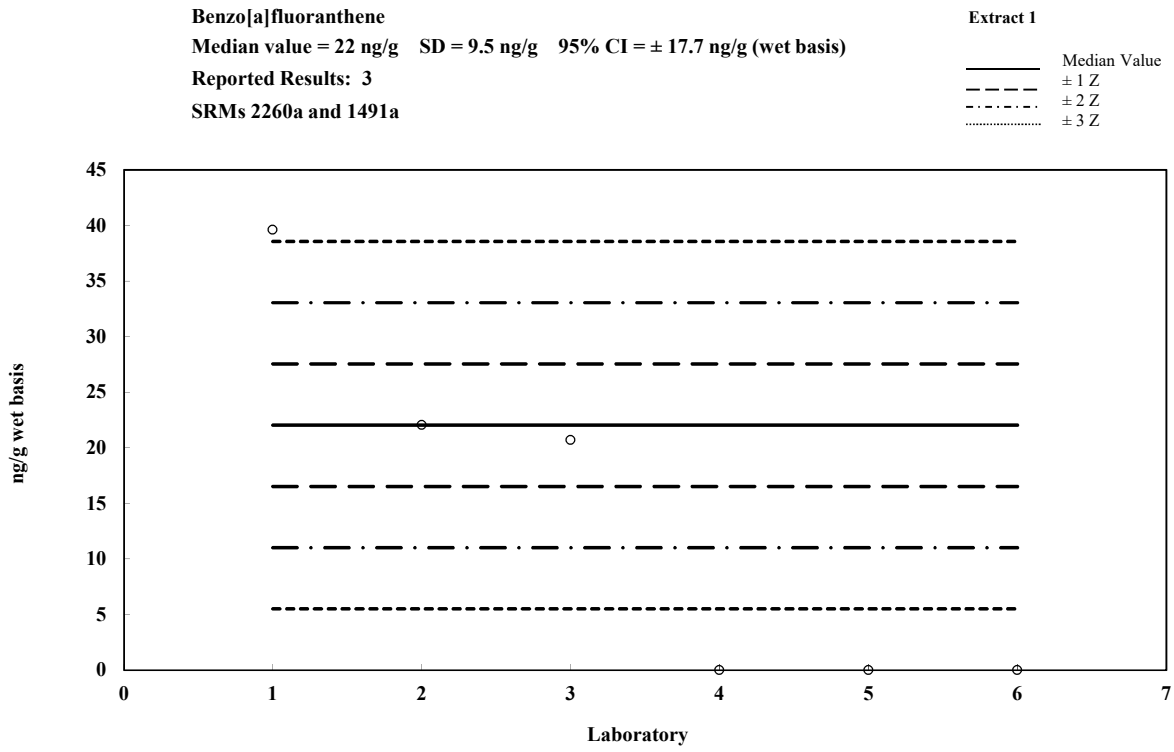
Anthracene
Median value = 30.6 ng/g SD = 6.3 ng/g 95% CI = ± 11.7 ng/g (wet basis)
Reported Results: 6
SRMs 2260a and 1491a



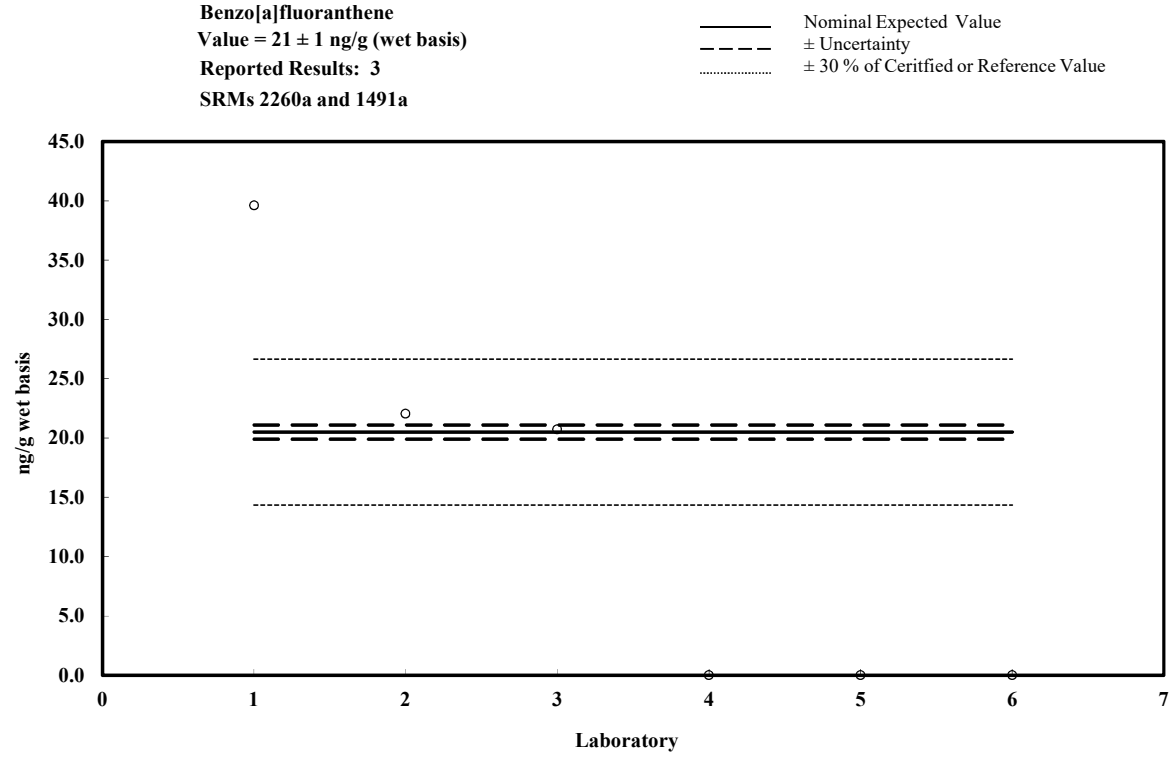
Anthracene
Value = 34 ± 1 ng/g (wet basis)
Reported Results: 6
SRMs 2260a and 1491a

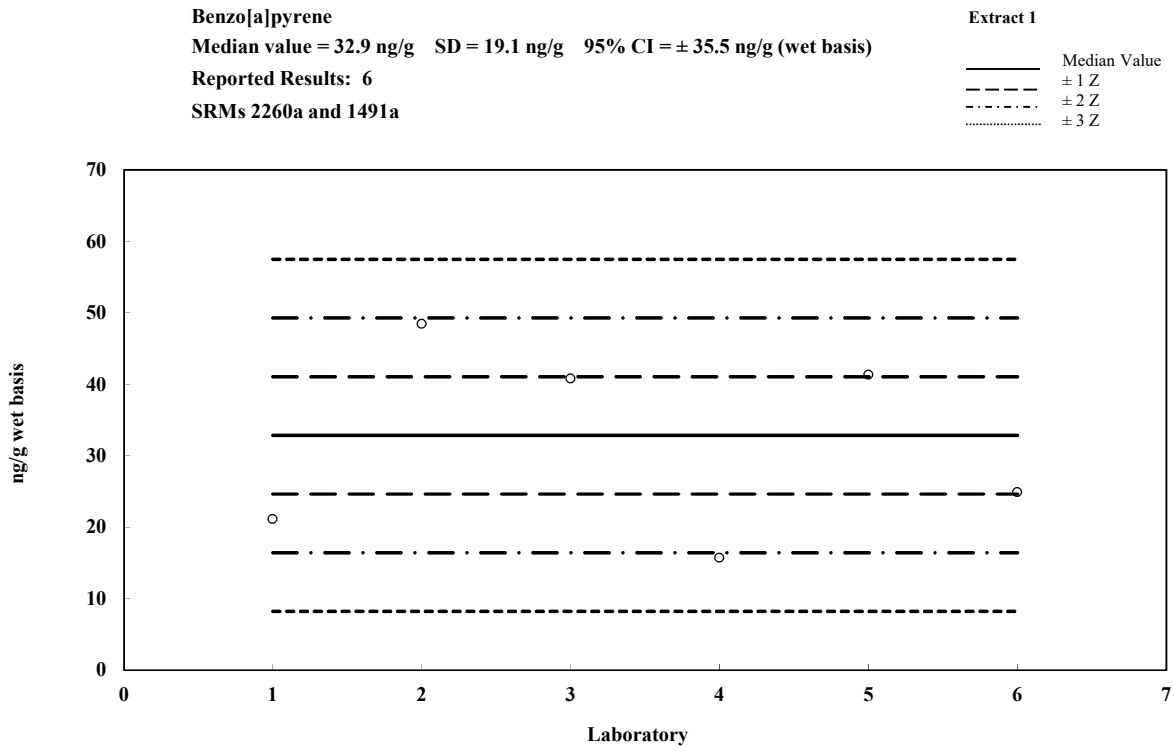


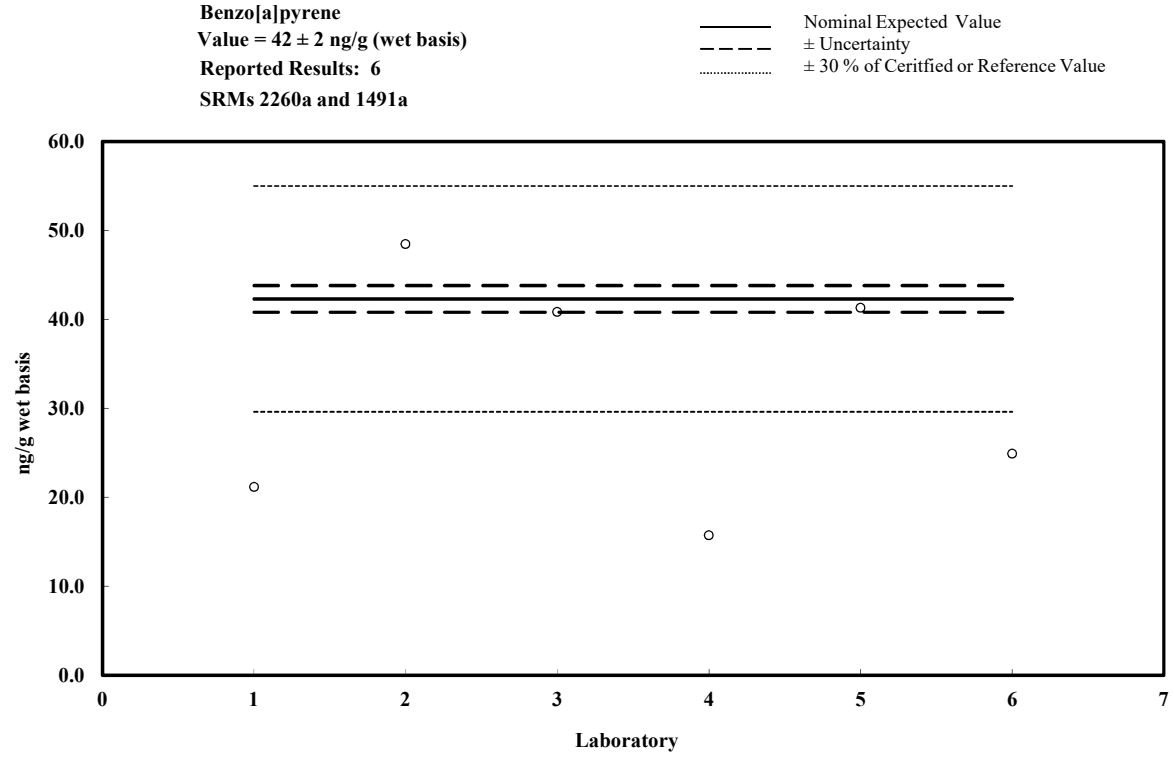
Benzo[a]fluoranthene
Median value = 22 ng/g SD = 9.5 ng/g 95% CI = ± 17.7 ng/g (wet basis)
Reported Results: 3
SRMs 2260a and 1491a



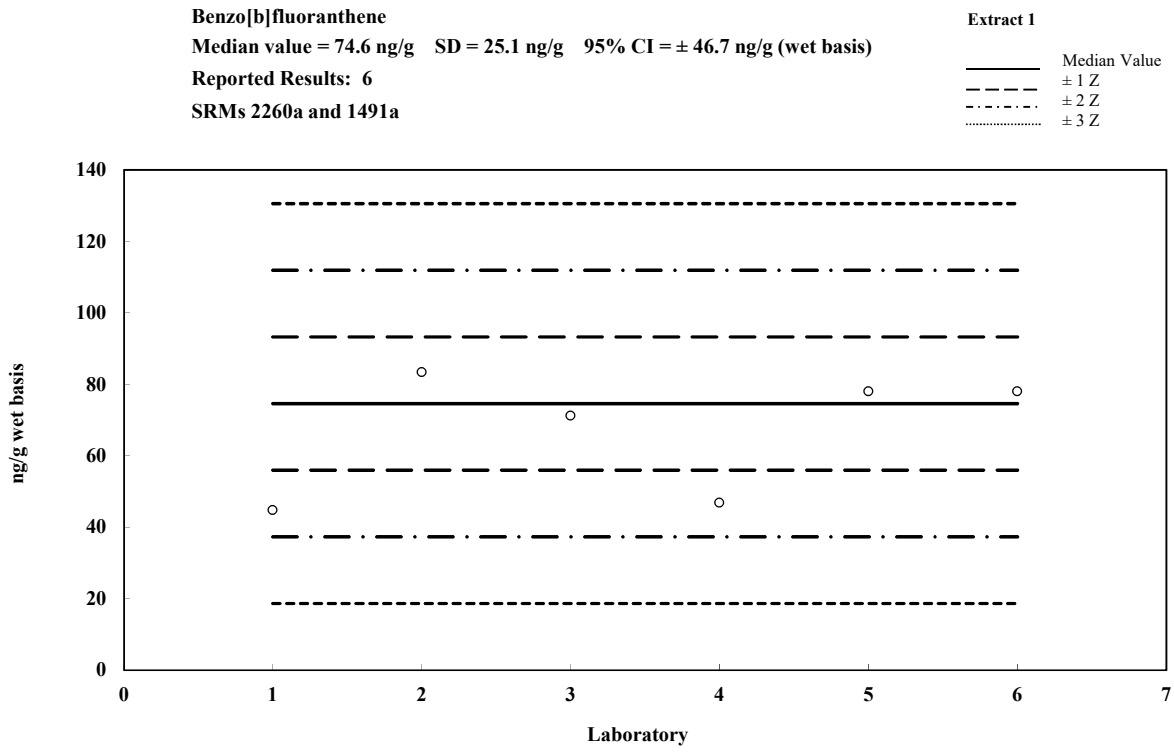
Benzo[a]fluoranthene
Value = 21 ± 1 ng/g (wet basis)
Reported Results: 3
SRMs 2260a and 1491a



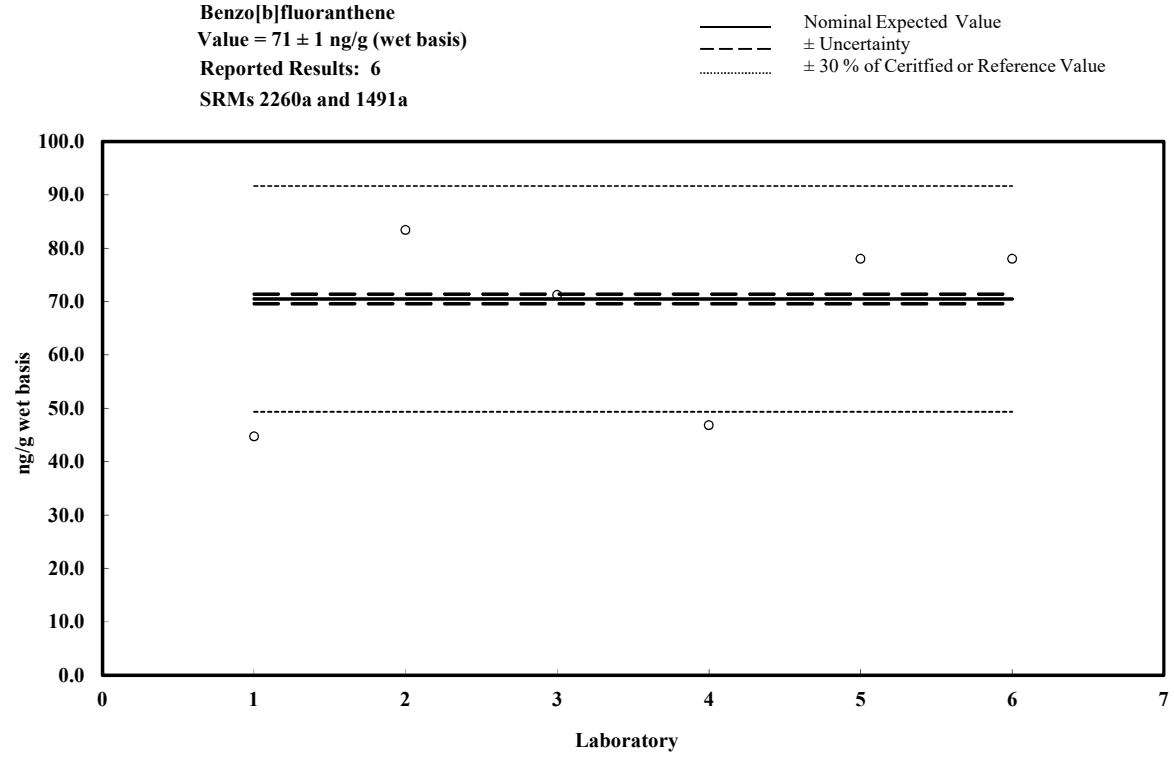




Benzo[b]fluoranthene
Median value = 74.6 ng/g SD = 25.1 ng/g 95% CI = ± 46.7 ng/g (wet basis)
Reported Results: 6
SRMs 2260a and 1491a



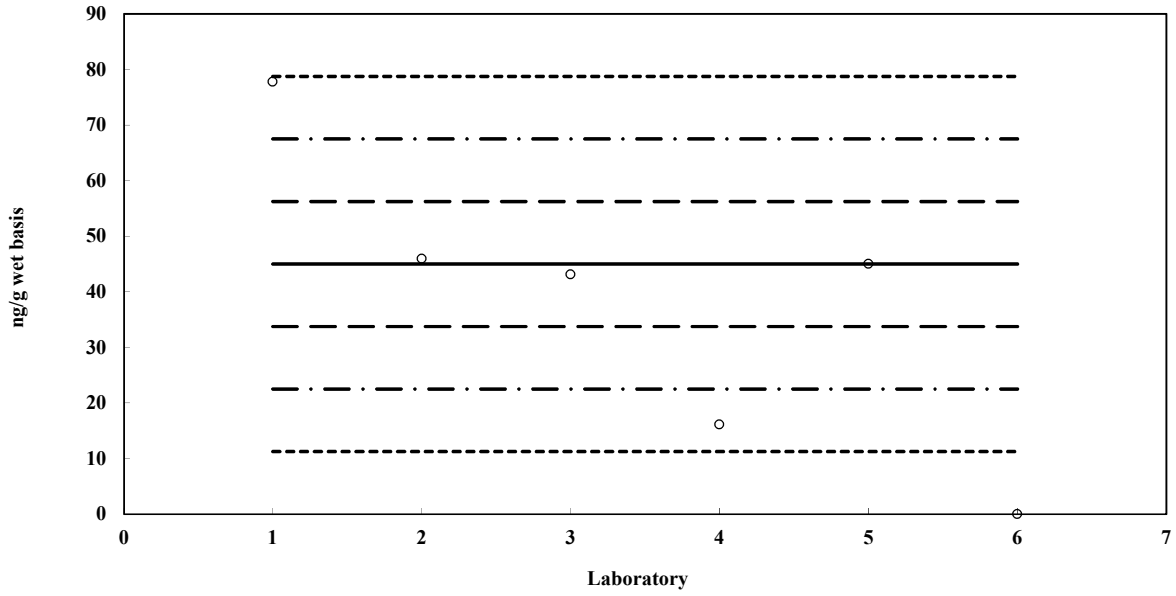
Benzo[b]fluoranthene
Value = 71 ± 1 ng/g (wet basis)
Reported Results: 6
SRMs 2260a and 1491a



Benzo[e]pyrene
Median value = 45 ng/g SD = 2.8 ng/g 95% CI = ± 5.2 ng/g (wet basis)
Reported Results: 5
SRMs 2260a and 1491a

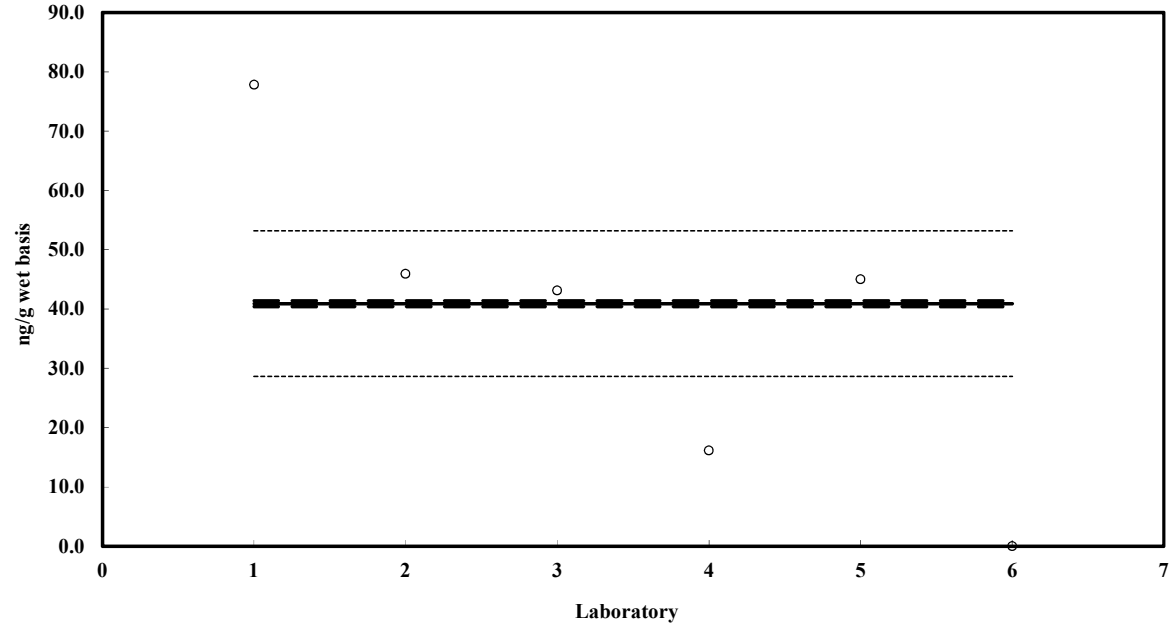
Extract 1

Median Value
± 1 Z
± 2 Z
± 3 Z

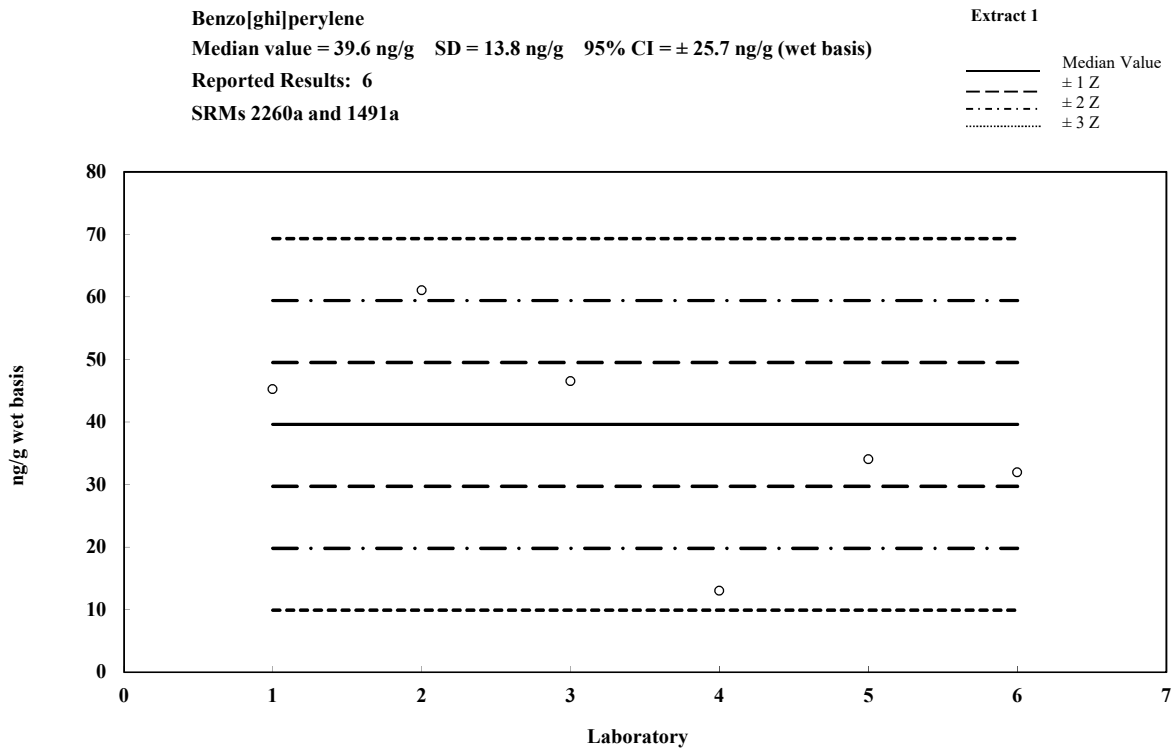


Benzo[e]pyrene
Value = 41 ± 1 ng/g (wet basis)
Reported Results: 5
SRMs 2260a and 1491a

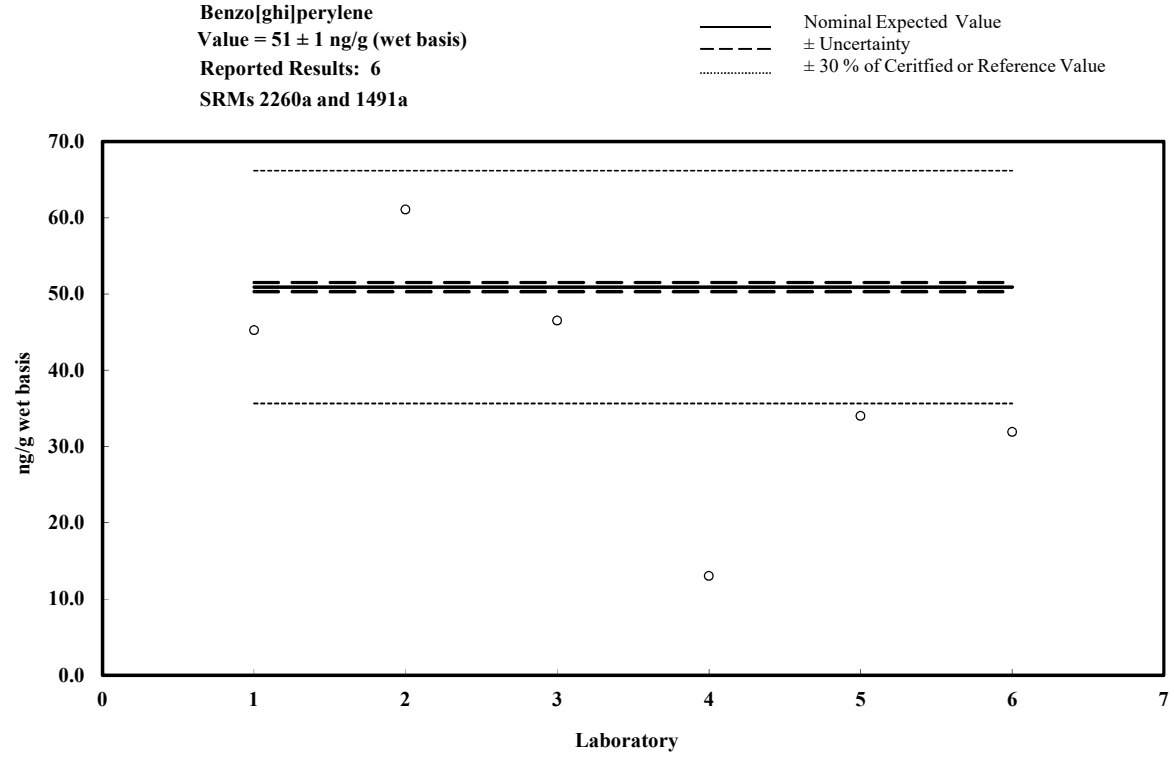
Nominal Expected Value
± Uncertainty
± 30 % of Ceritfied or Reference Value



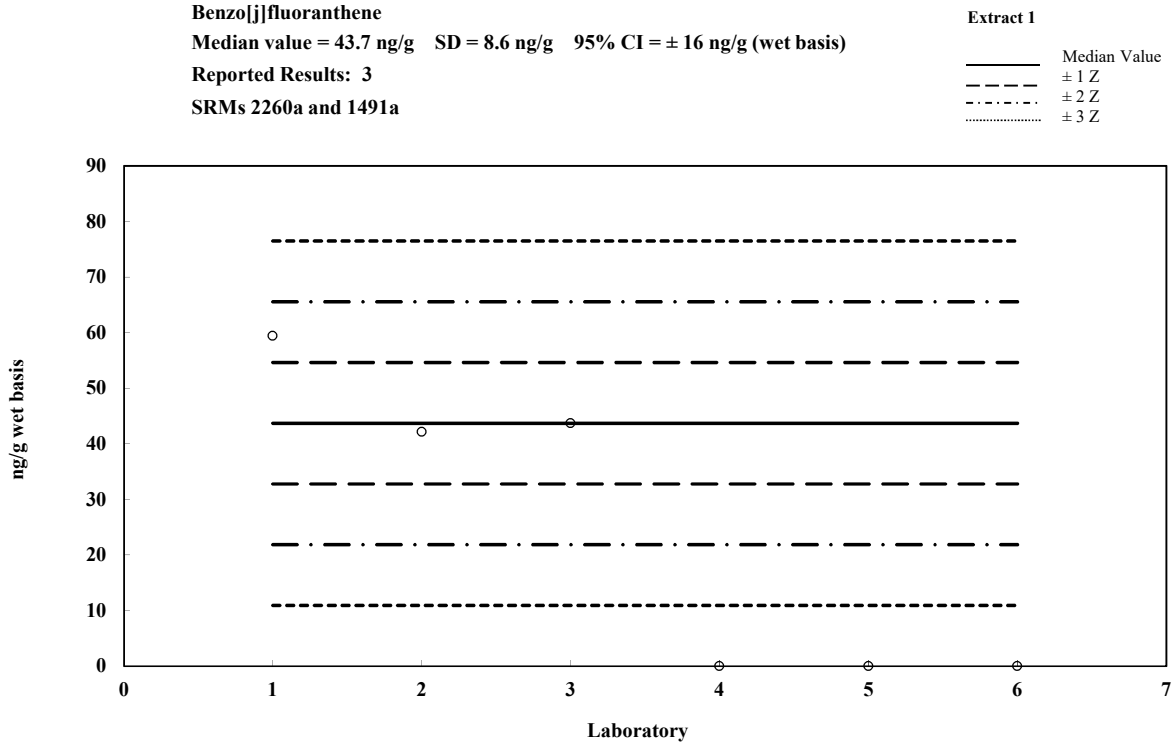
Benzo[ghi]perylene
Median value = 39.6 ng/g SD = 13.8 ng/g 95% CI = ± 25.7 ng/g (wet basis)
Reported Results: 6
SRMs 2260a and 1491a



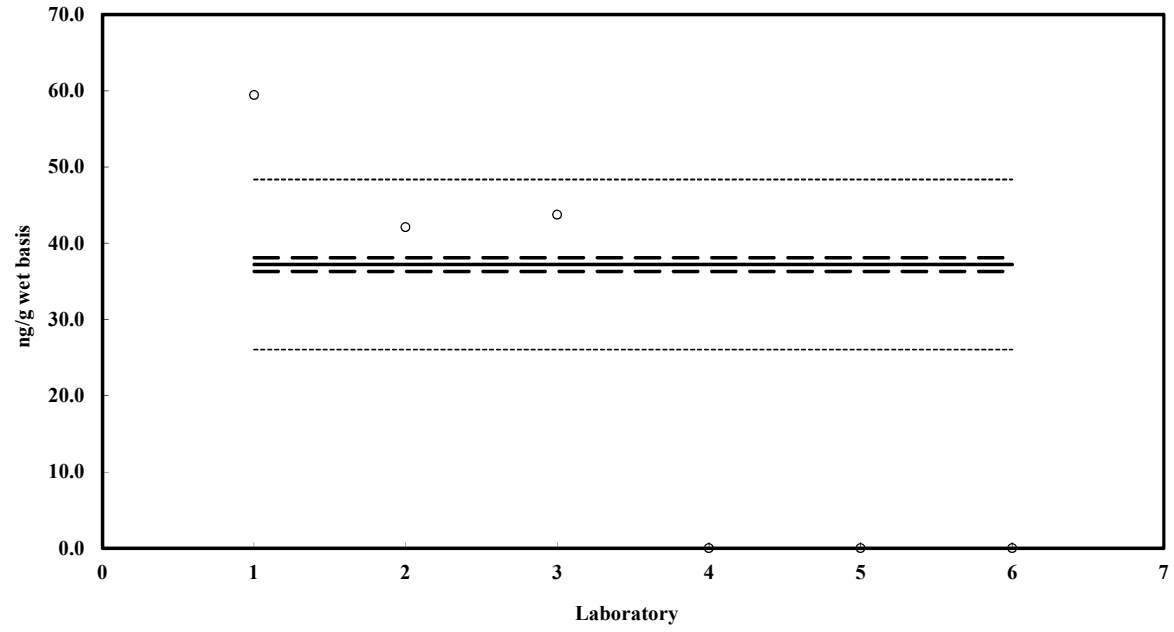
Benzo[ghi]perylene
Value = 51 ± 1 ng/g (wet basis)
Reported Results: 6
SRMs 2260a and 1491a



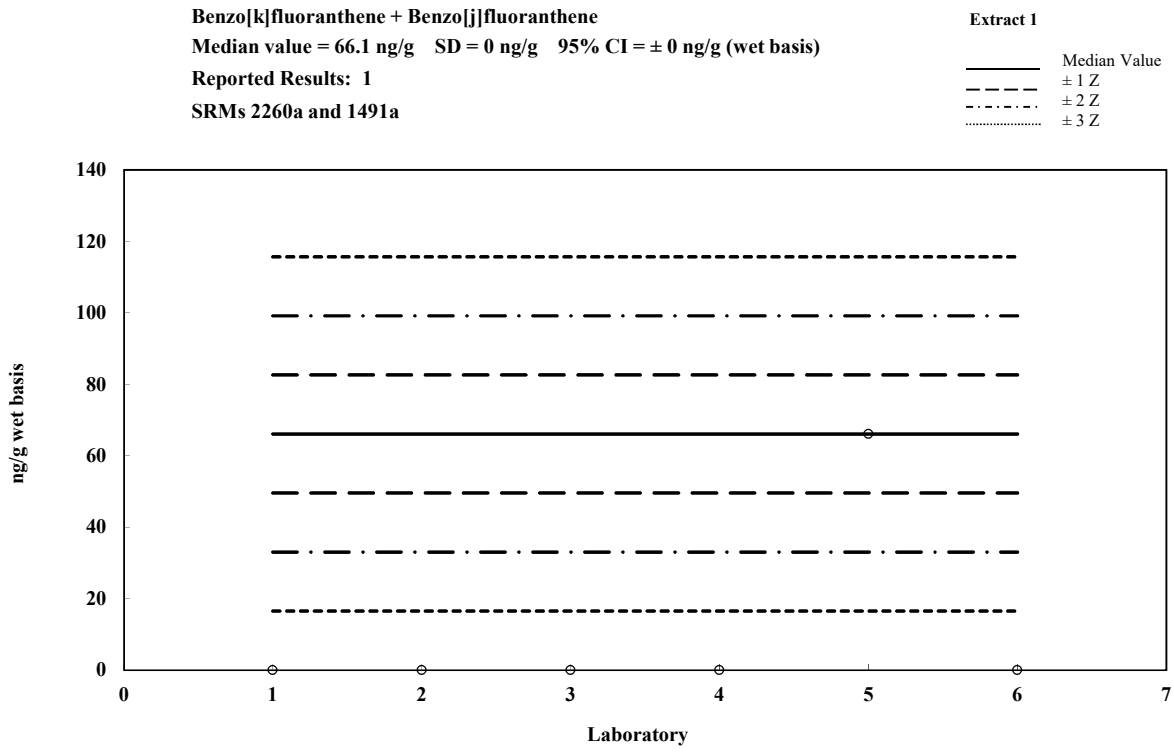
Benzo[j]fluoranthene
Median value = 43.7 ng/g SD = 8.6 ng/g 95% CI = ± 16 ng/g (wet basis)
Reported Results: 3
SRMs 2260a and 1491a



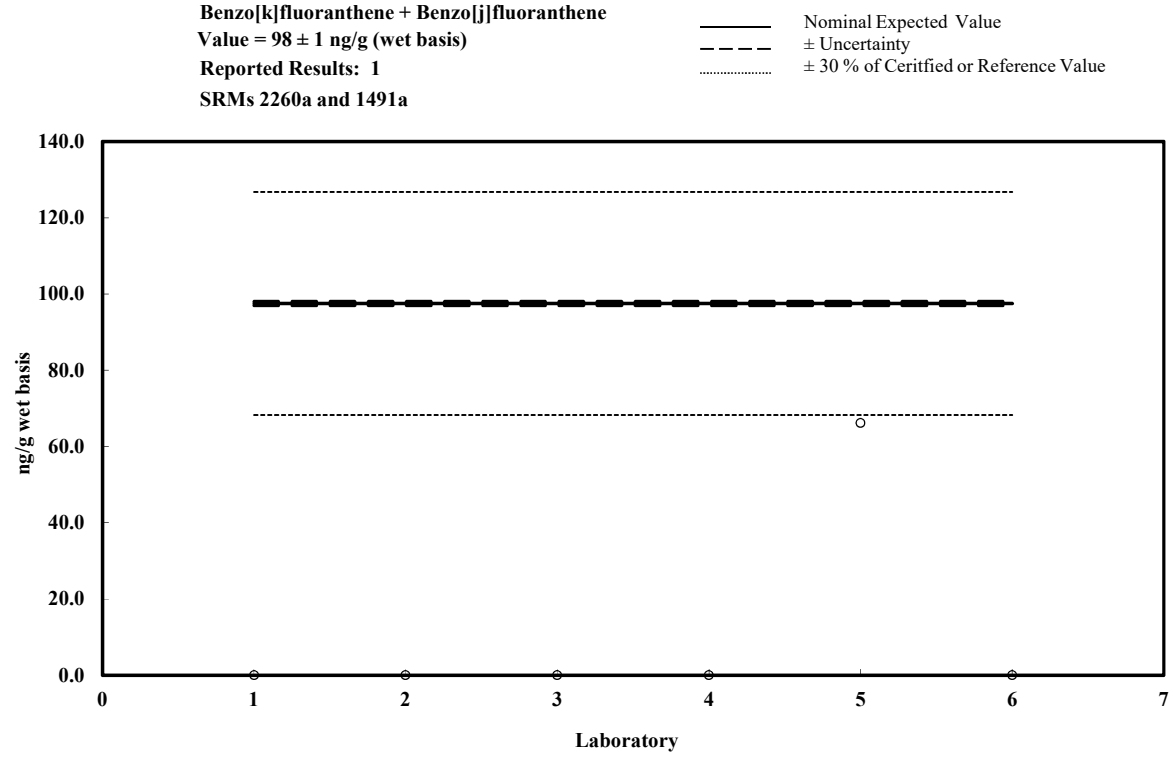
Benzo[j]fluoranthene
Value = 37 ± 1 ng/g (wet basis)
Reported Results: 3
SRMs 2260a and 1491a



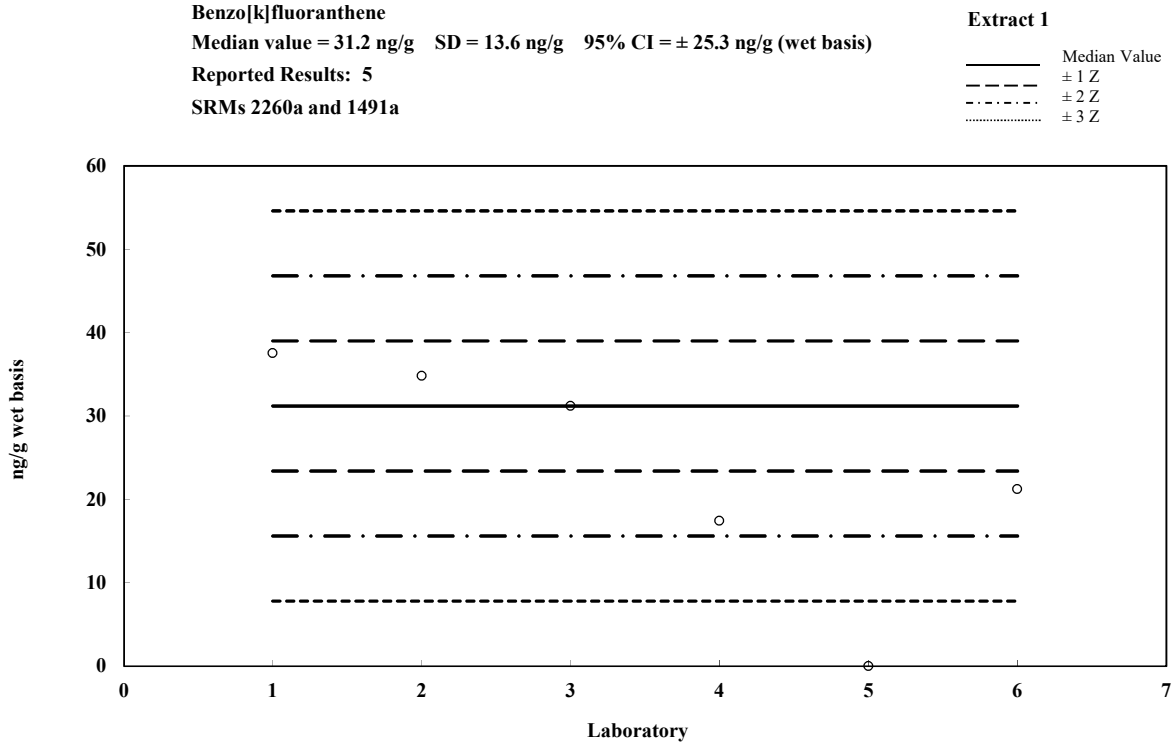
Benzo[k]fluoranthene + Benzo[j]fluoranthene
Median value = 66.1 ng/g SD = 0 ng/g 95% CI = ± 0 ng/g (wet basis)
Reported Results: 1
SRMs 2260a and 1491a



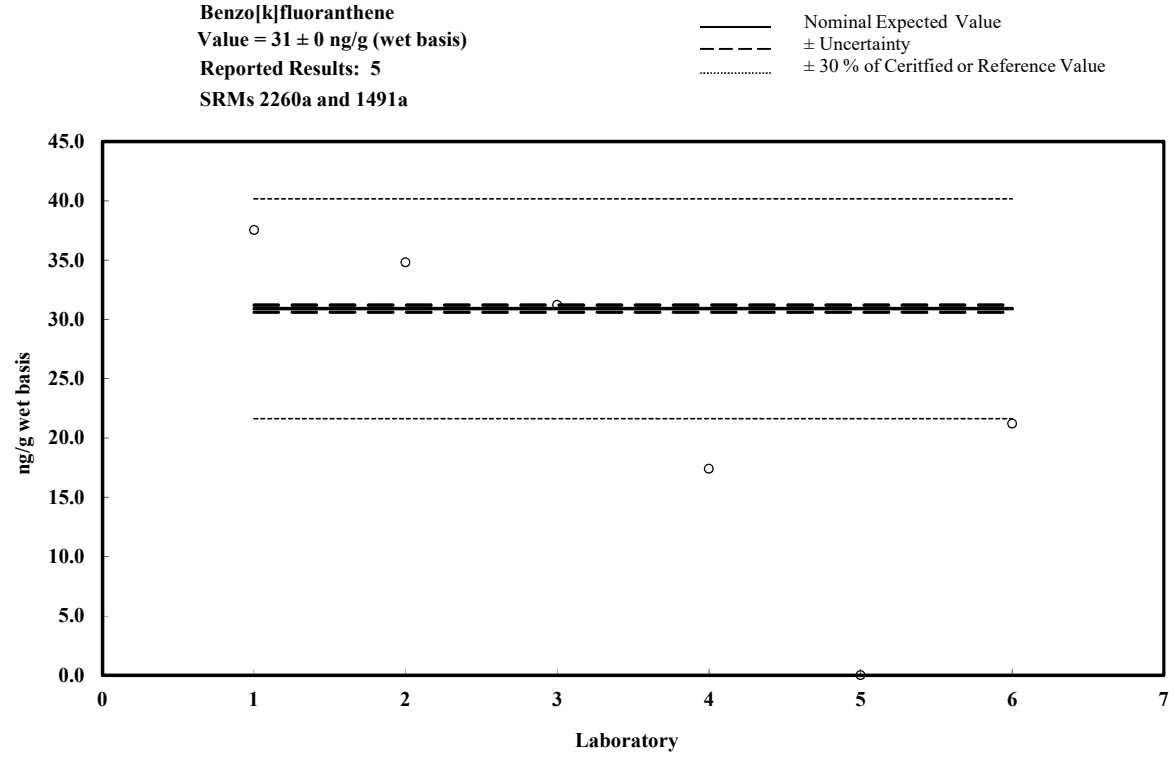
Benzo[k]fluoranthene + Benzo[j]fluoranthene
Value = 98 ± 1 ng/g (wet basis)
Reported Results: 1
SRMs 2260a and 1491a



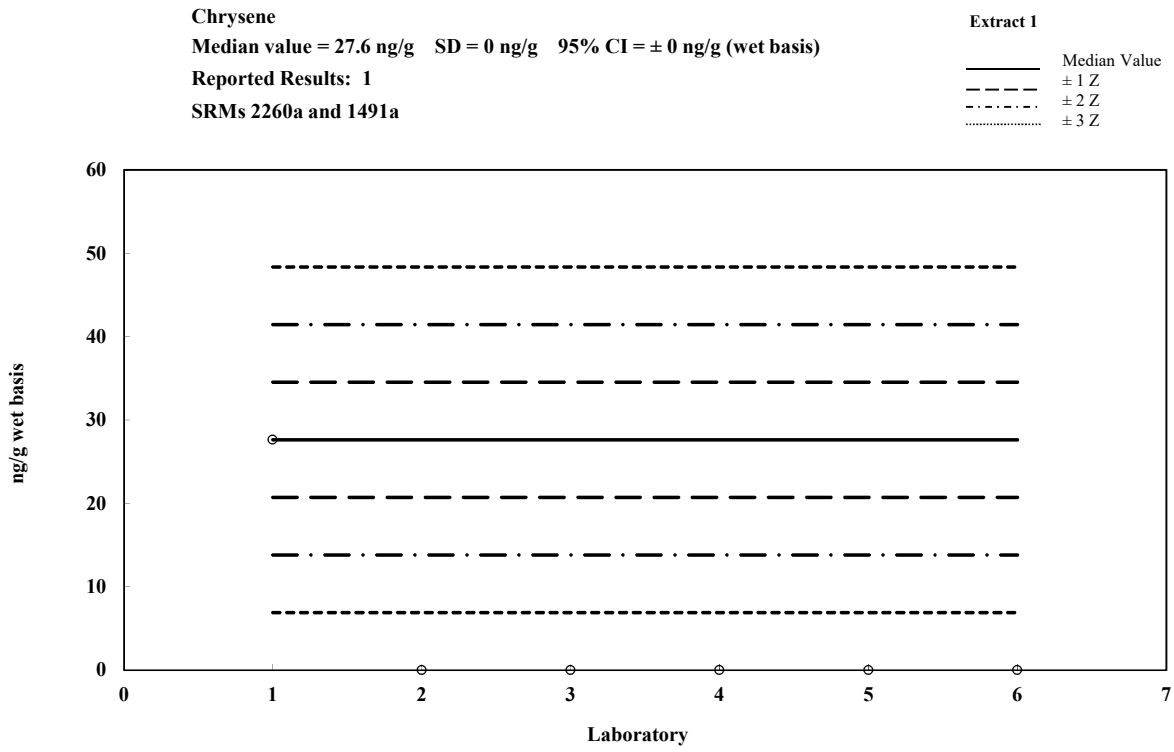
Benzo[k]fluoranthene
Median value = 31.2 ng/g SD = 13.6 ng/g 95% CI = ± 25.3 ng/g (wet basis)
Reported Results: 5
SRMs 2260a and 1491a



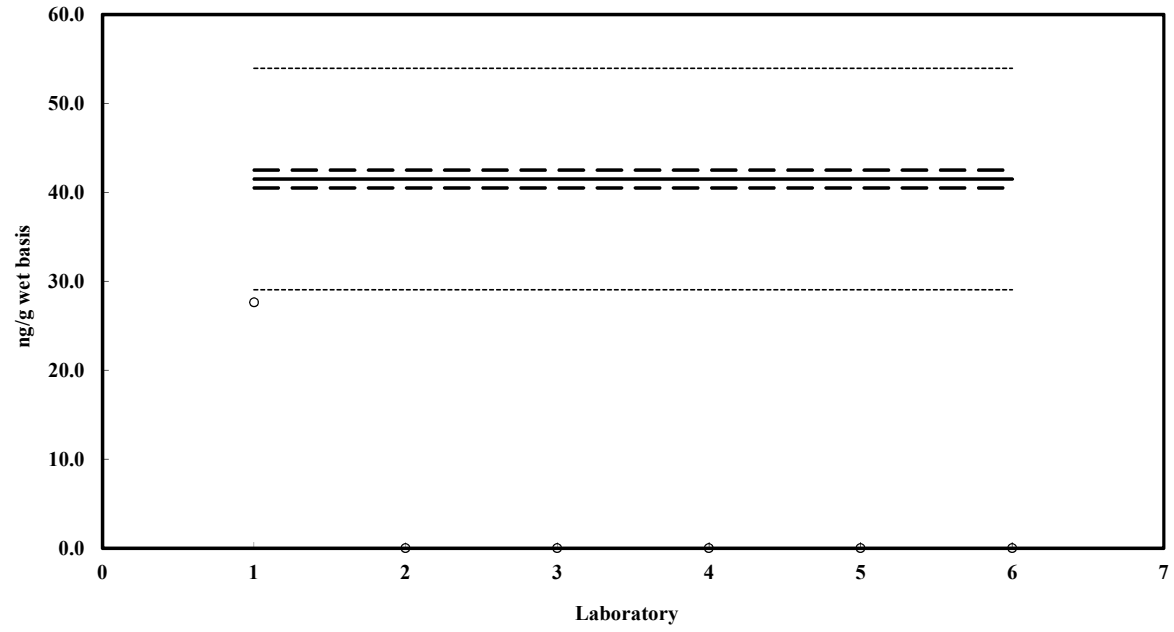
Benzo[k]fluoranthene
Value = 31 ± 0 ng/g (wet basis)
Reported Results: 5
SRMs 2260a and 1491a

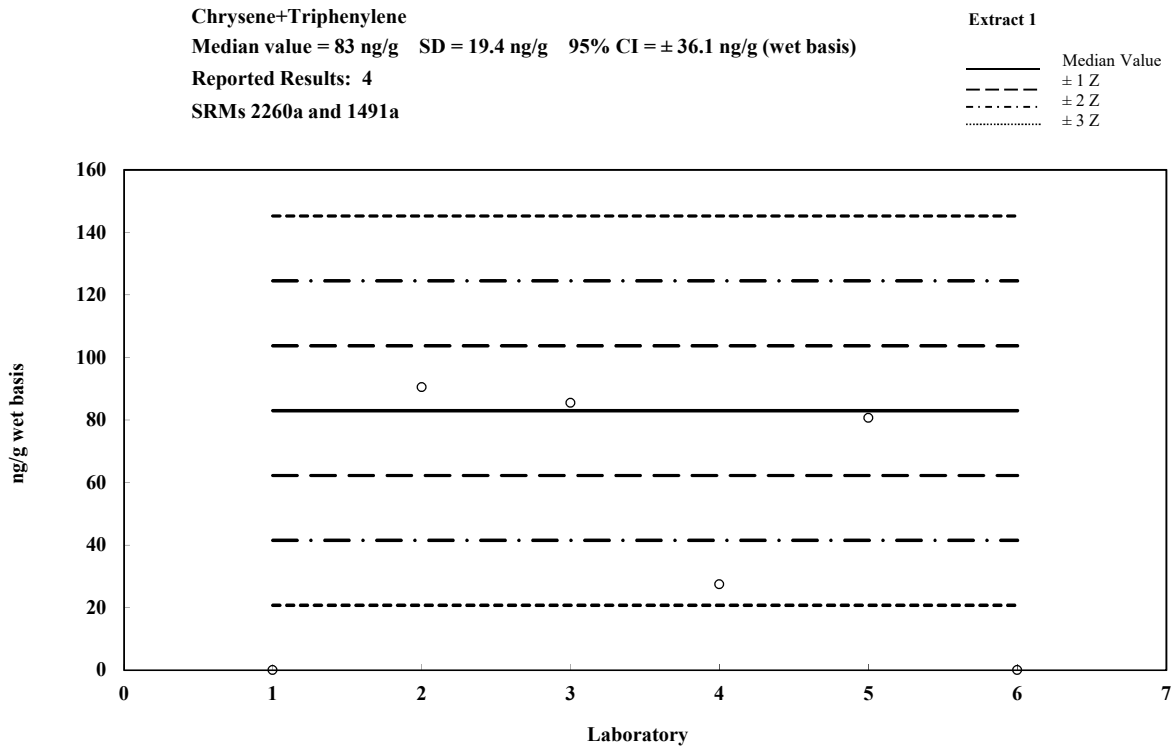


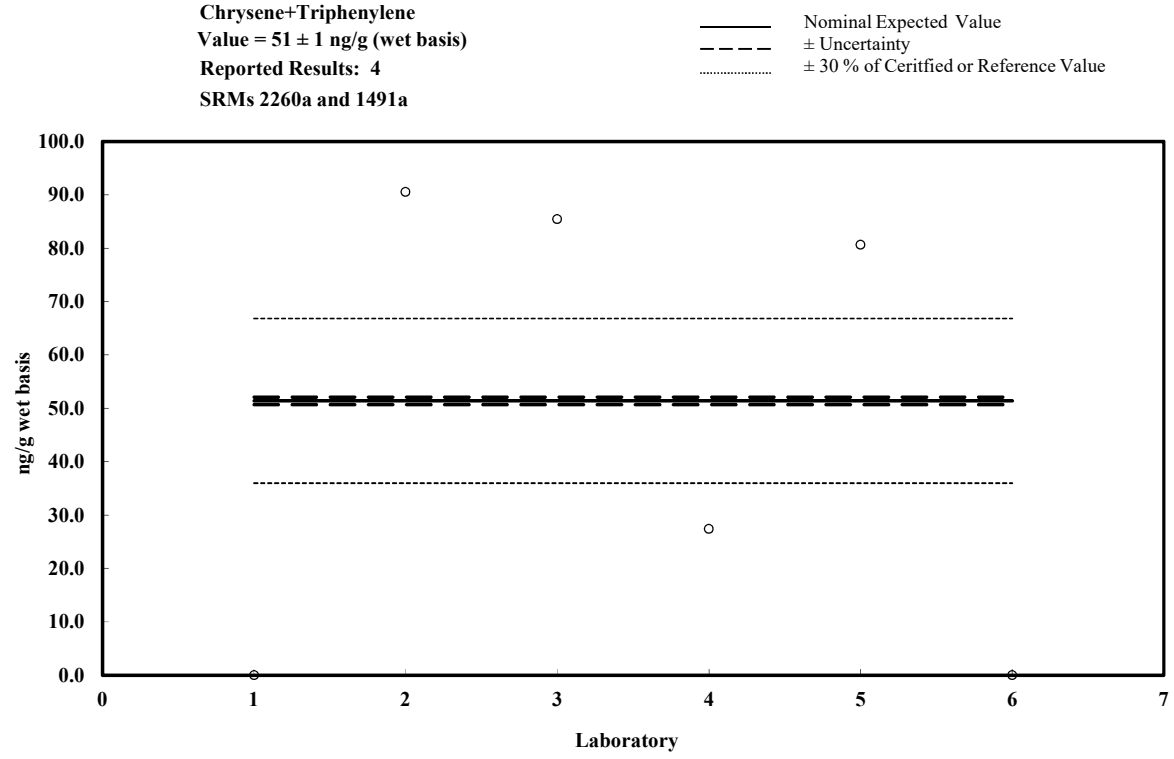
Chrysene
Median value = 27.6 ng/g SD = 0 ng/g 95% CI = ± 0 ng/g (wet basis)
Reported Results: 1
SRMs 2260a and 1491a



Chrysene
Value = 42 ± 1 ng/g (wet basis)
Reported Results: 1
SRMs 2260a and 1491a





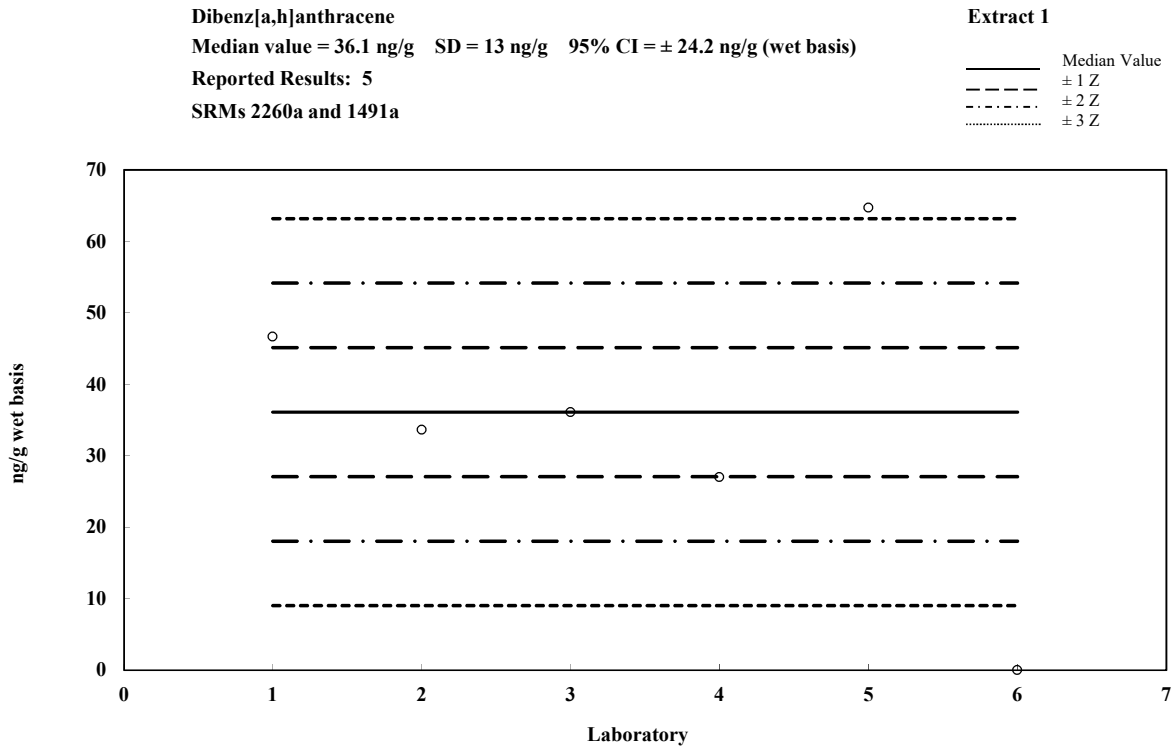


Dibenz[a,h]anthracene **Extract 1**

Median value = 36.1 ng/g SD = 13 ng/g 95% CI = ± 24.2 ng/g (wet basis)

Reported Results: 5

SRMs 2260a and 1491a

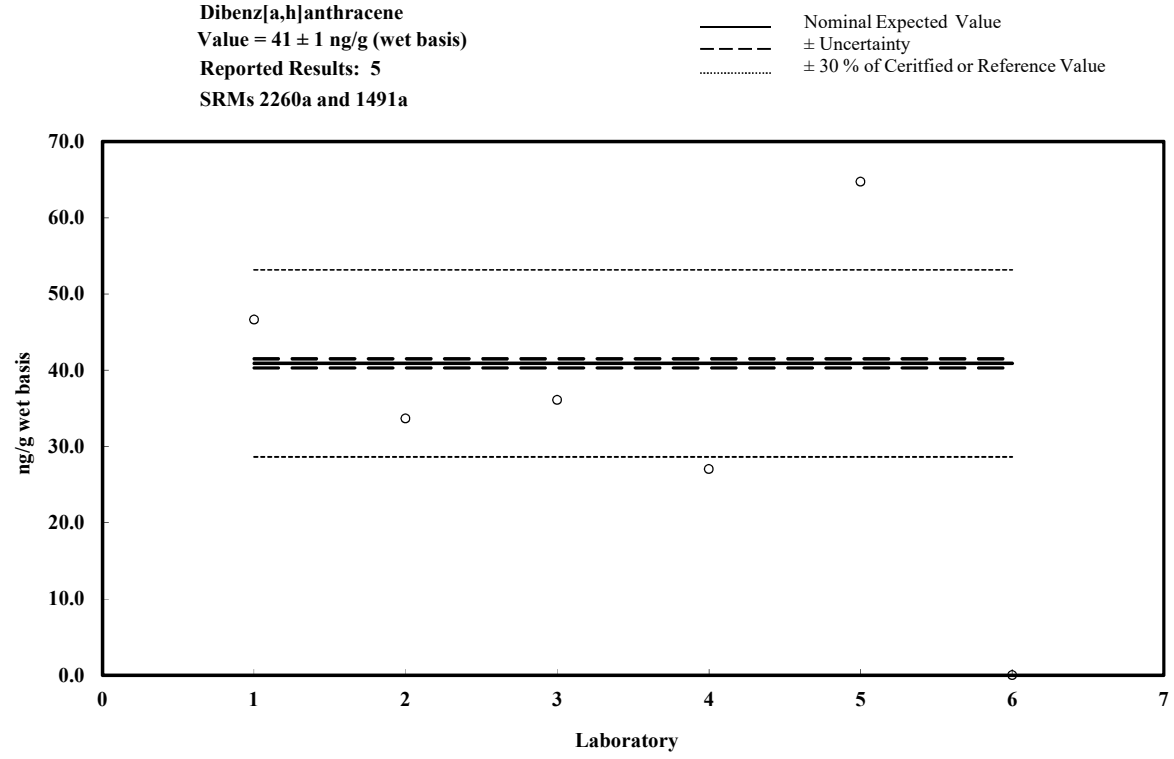


Dibenz[a,h]anthracene

Value = 41 ± 1 ng/g (wet basis)

Reported Results: 5

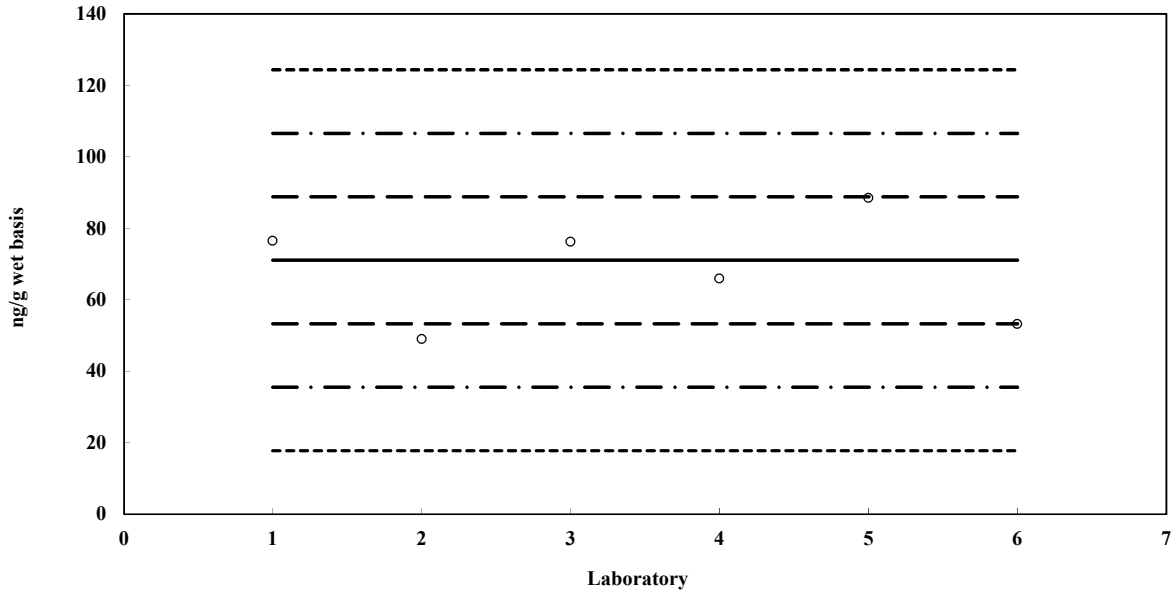
SRMs 2260a and 1491a



Fluoranthene
Median value = 71.1 ng/g SD = 20 ng/g 95% CI = ± 37.2 ng/g (wet basis)
Reported Results: 6
SRMs 2260a and 1491a

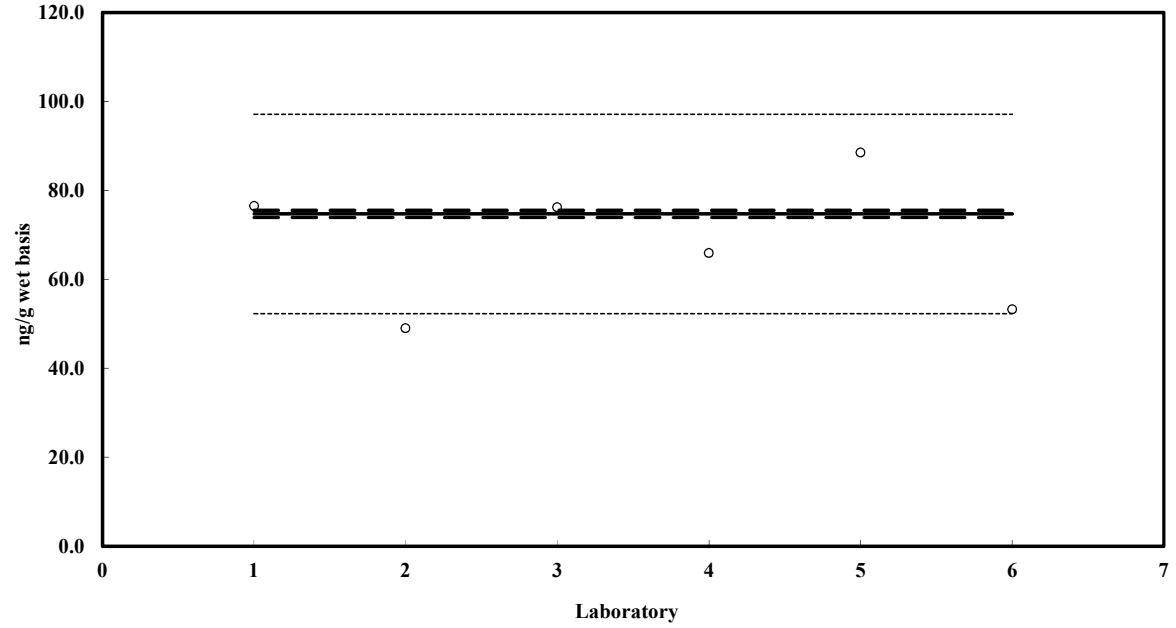
Extract 1

Median Value
± 1 Z
± 2 Z
± 3 Z



Fluoranthene
Value = 75 ± 1 ng/g (wet basis)
Reported Results: 6
SRMs 2260a and 1491a

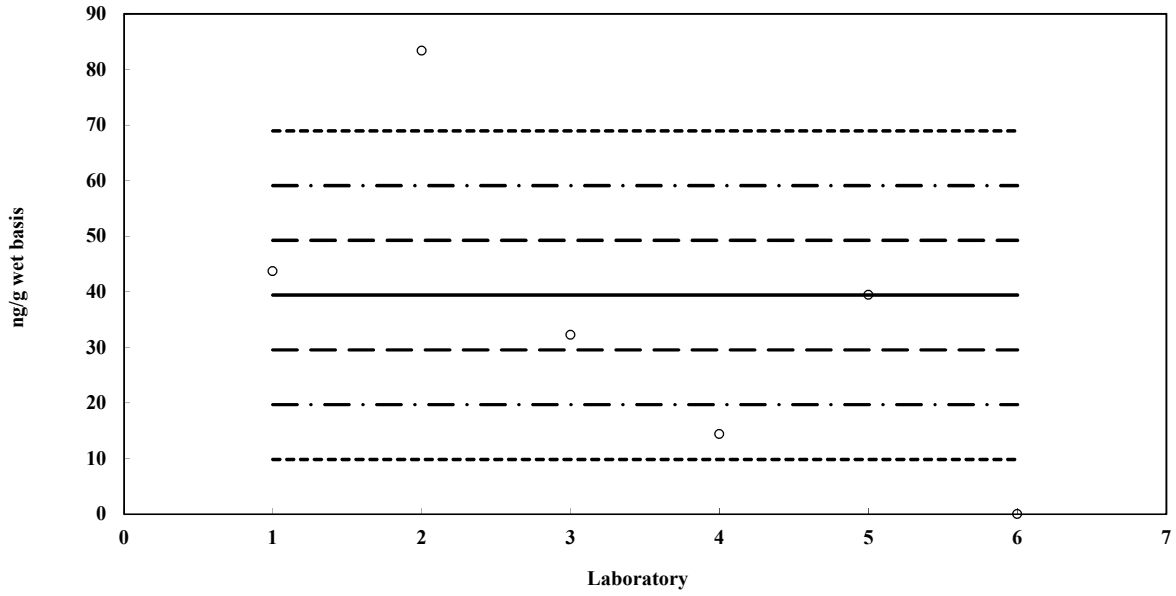
Nominal Expected Value
± Uncertainty
± 30 % of Ceritfied or Reference Value



Indeno[1,2,3-cd]pyrene
Median value = 39.4 ng/g SD = 11.5 ng/g 95% CI = ± 21.4 ng/g (wet basis)
Reported Results: 5
SRMs 2260a and 1491a

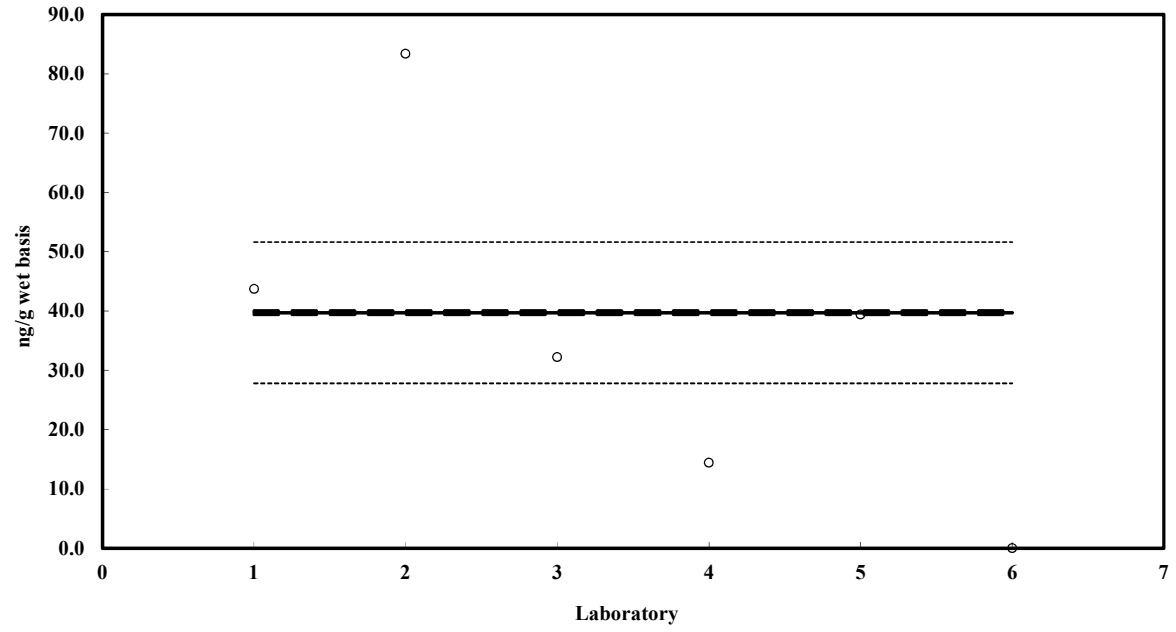
Extract 1

Median Value
± 1 Z
± 2 Z
± 3 Z



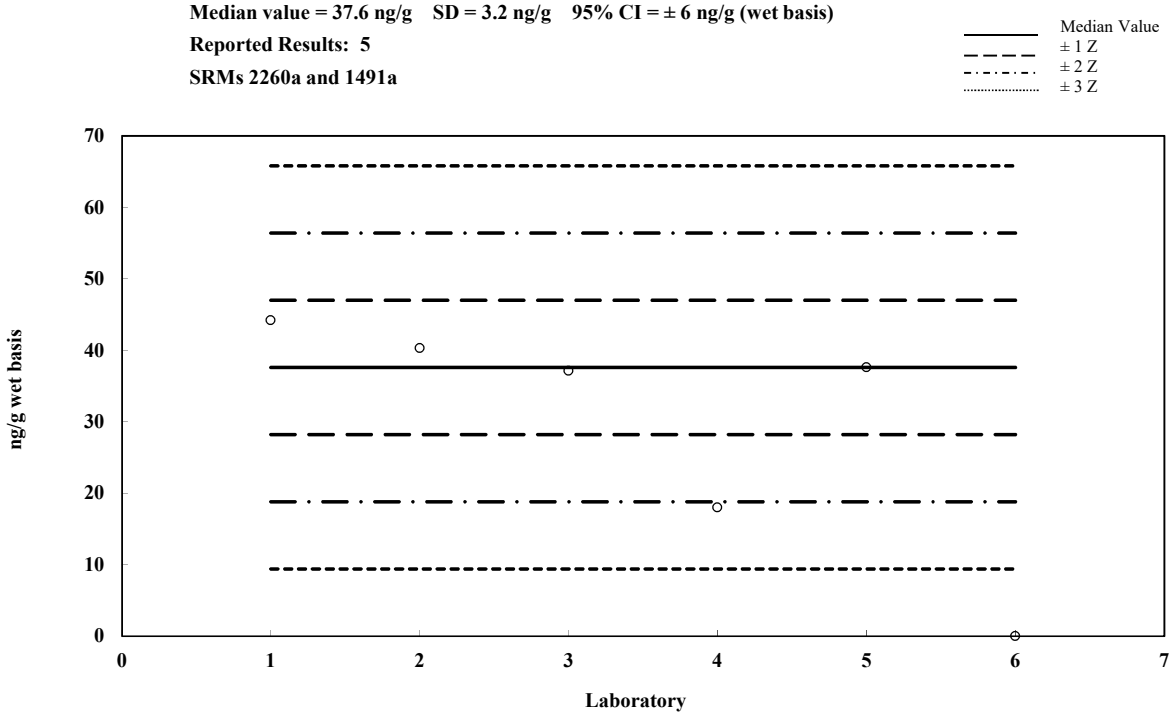
Indeno[1,2,3-cd]pyrene
Value = 40 ± 0 ng/g (wet basis)
Reported Results: 5
SRMs 2260a and 1491a

Nominal Expected Value
± Uncertainty
± 30 % of Ceritfied or Reference Value

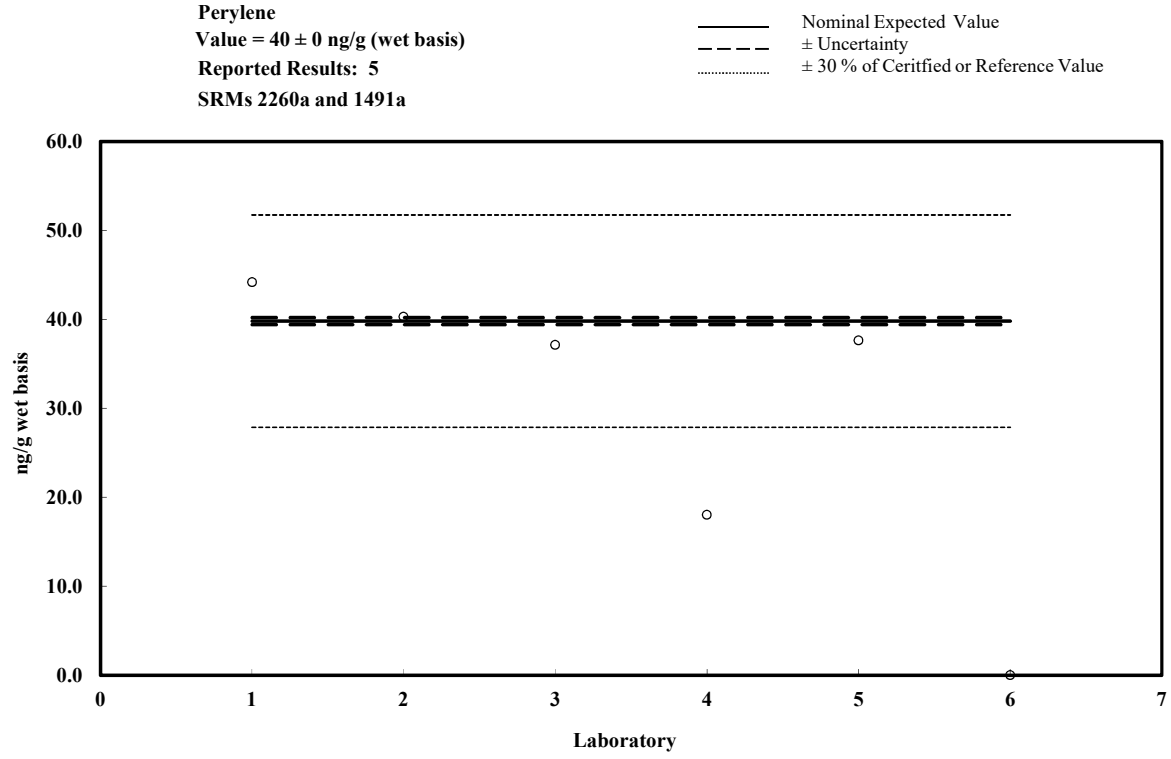


Perylene
Median value = 37.6 ng/g SD = 3.2 ng/g 95% CI = ± 6 ng/g (wet basis)
Reported Results: 5
SRMs 2260a and 1491a

Extract 1



Perylene
Value = 40 ± 0 ng/g (wet basis)
Reported Results: 5
SRMs 2260a and 1491a

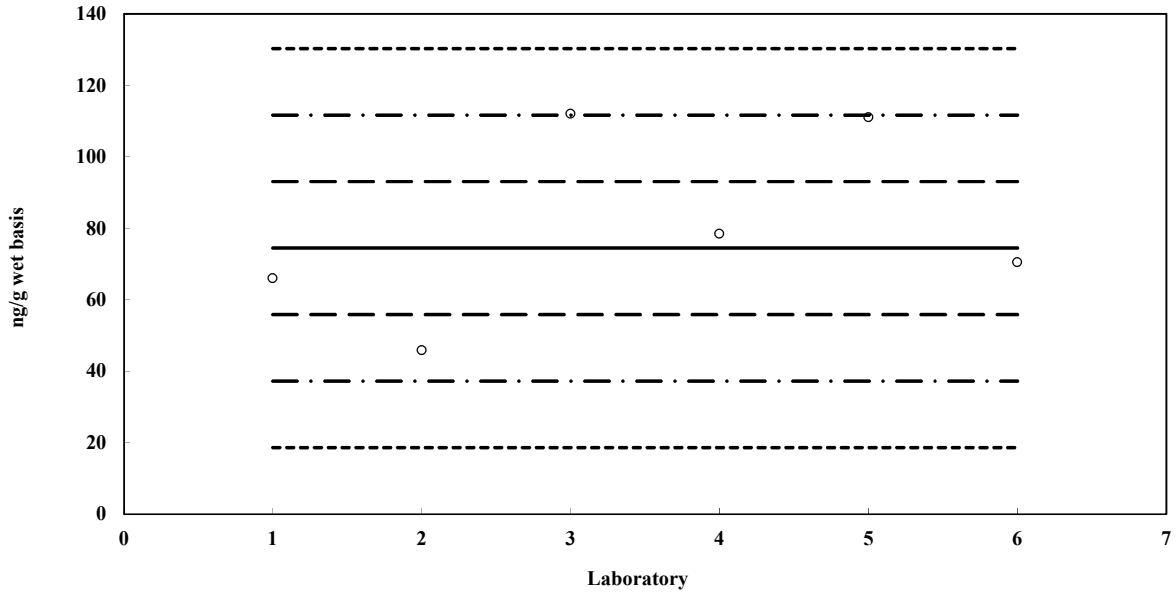


Phenanthrene **Extract 1**

Median value = 74.5 ng/g SD = 35.7 ng/g 95% CI = ± 66.4 ng/g (wet basis)

Reported Results: 6

SRMs 2260a and 1491a

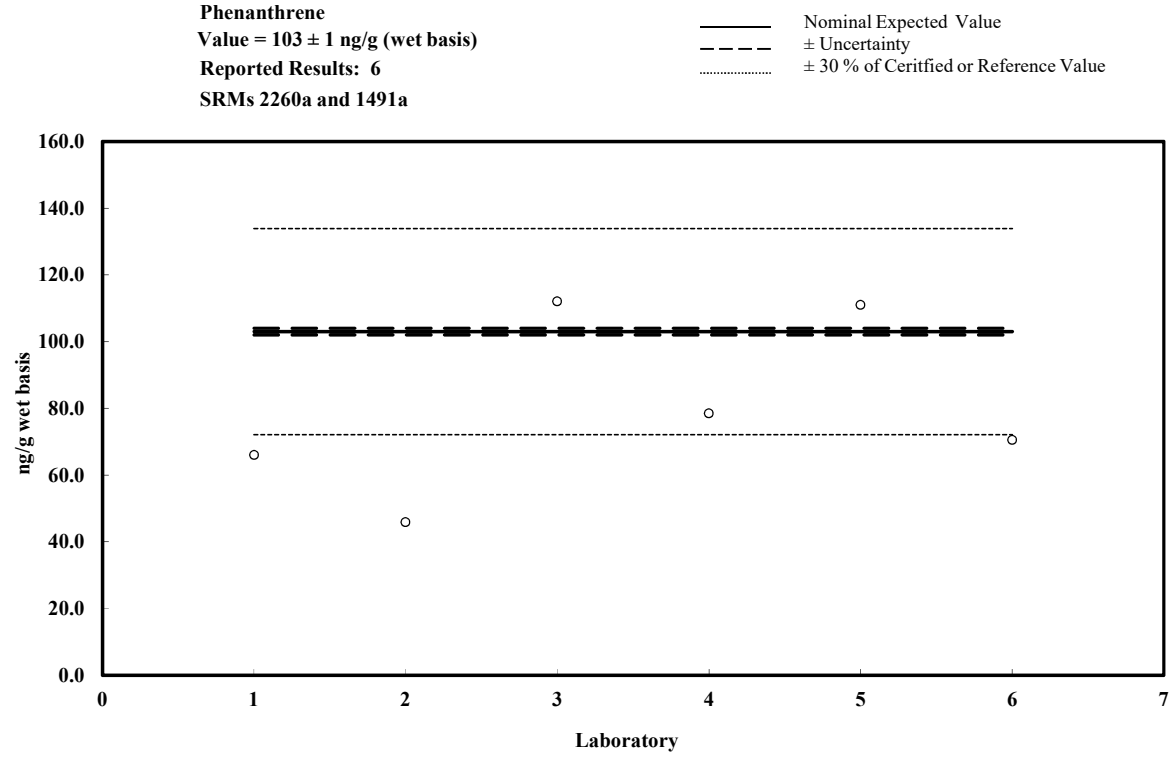


Phenanthrene

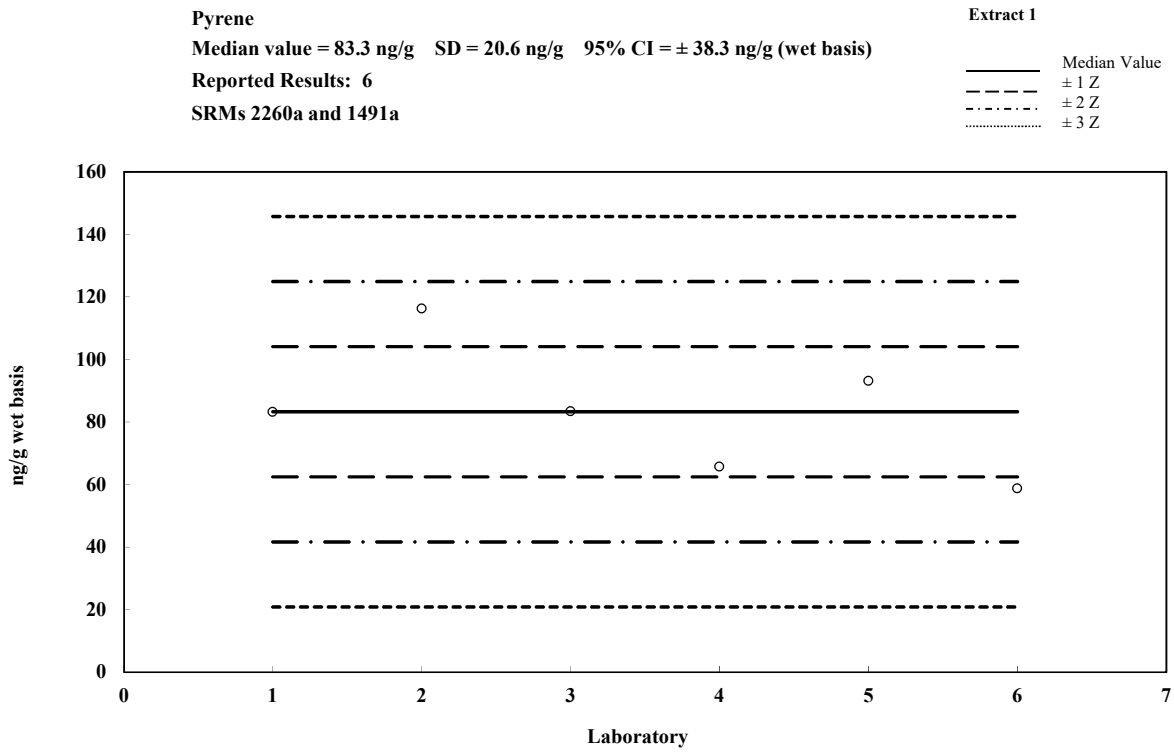
Value = 103 ± 1 ng/g (wet basis)

Reported Results: 6

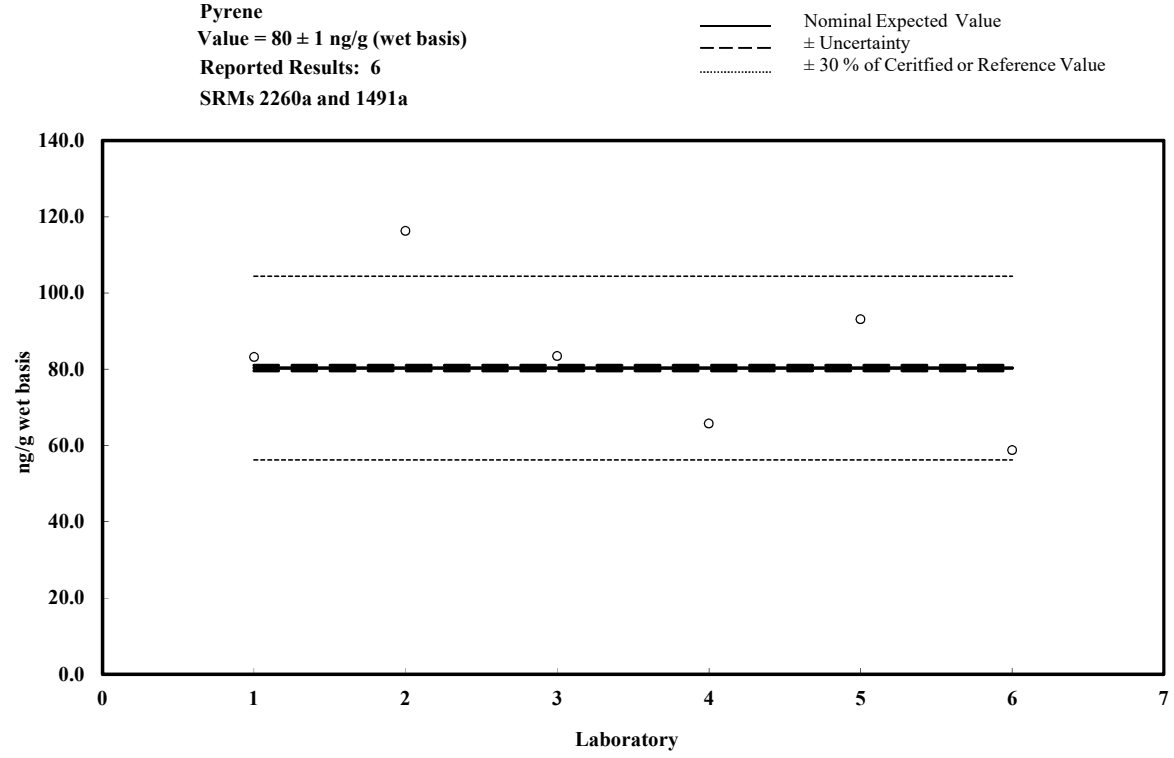
SRMs 2260a and 1491a



Pyrene
Median value = 83.3 ng/g SD = 20.6 ng/g 95% CI = ± 38.3 ng/g (wet basis)
Reported Results: 6
SRMs 2260a and 1491a

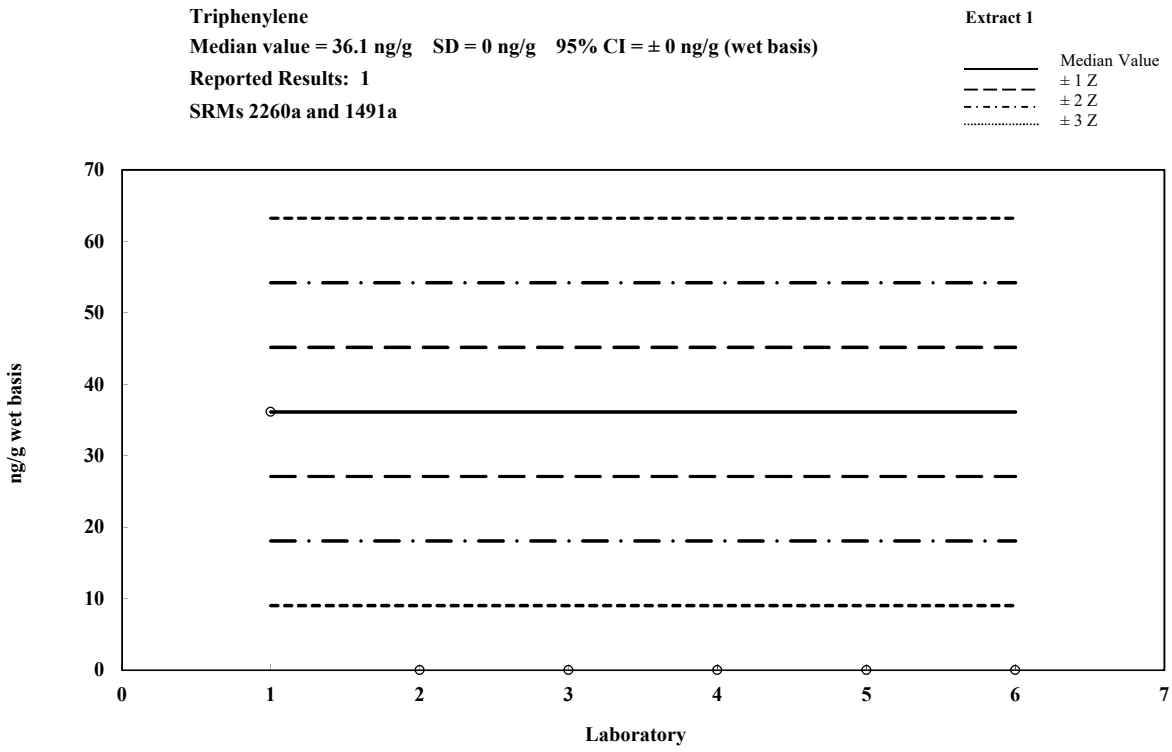


Pyrene
Value = 80 ± 1 ng/g (wet basis)
Reported Results: 6
SRMs 2260a and 1491a

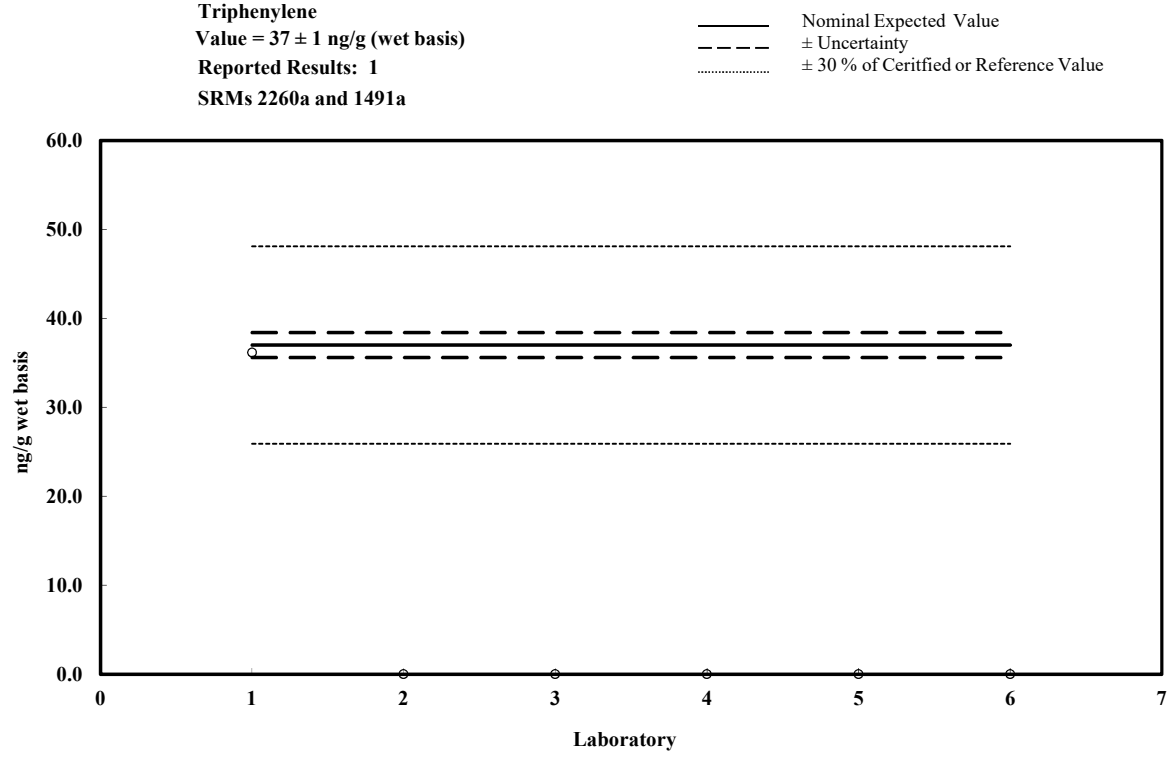


Triphenylene
Median value = 36.1 ng/g SD = 0 ng/g 95% CI = ± 0 ng/g (wet basis)
Reported Results: 1
SRMs 2260a and 1491a

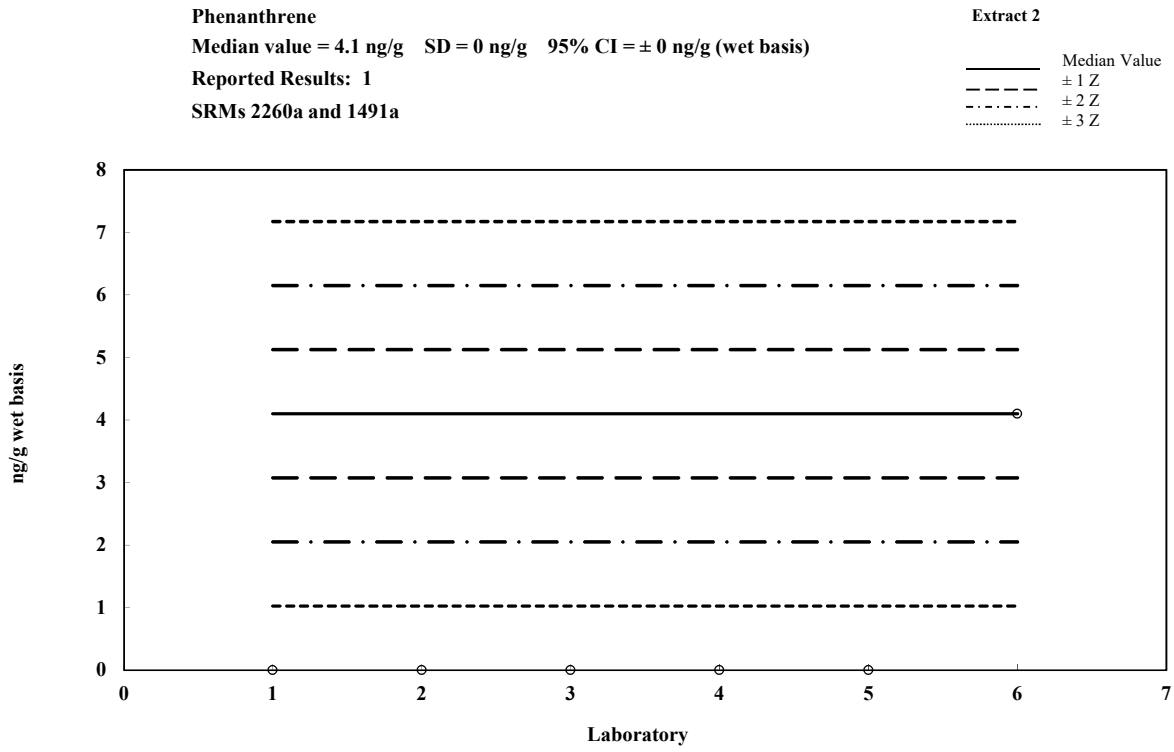
Extract 1



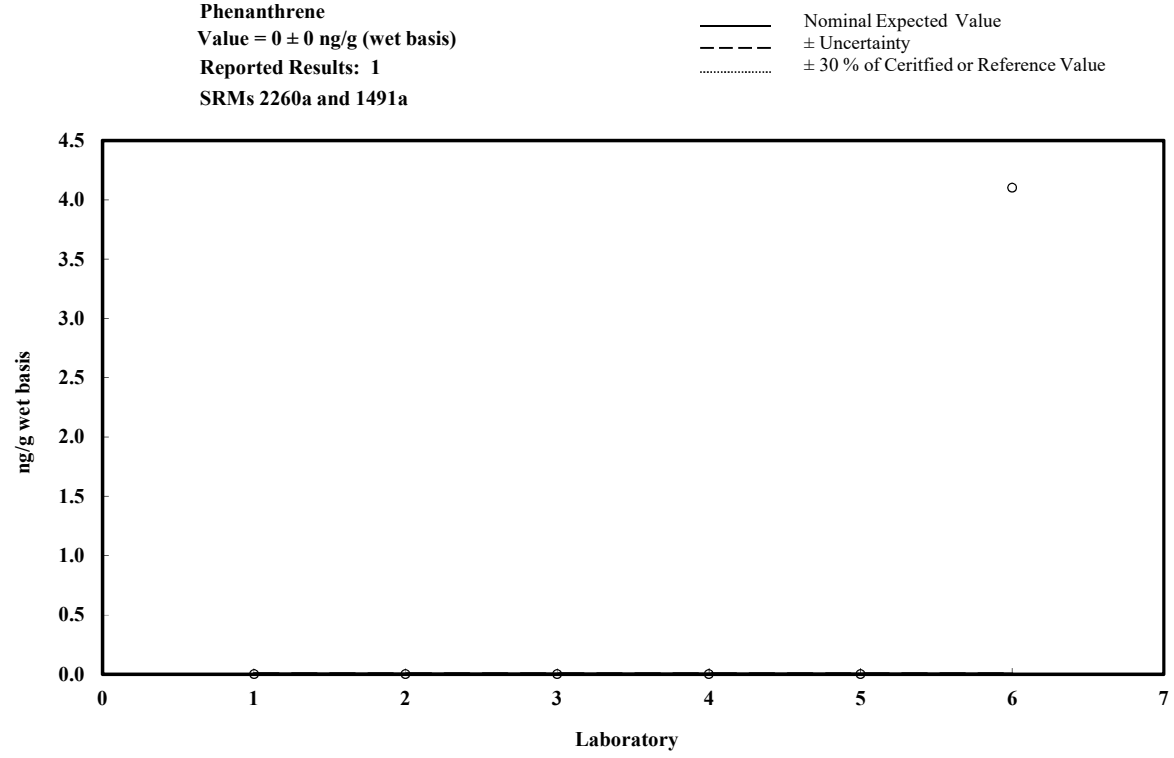
Triphenylene
Value = 37 ± 1 ng/g (wet basis)
Reported Results: 1
SRMs 2260a and 1491a



Phenanthrene
Median value = 4.1 ng/g SD = 0 ng/g 95% CI = ± 0 ng/g (wet basis)
Reported Results: 1
SRMs 2260a and 1491a

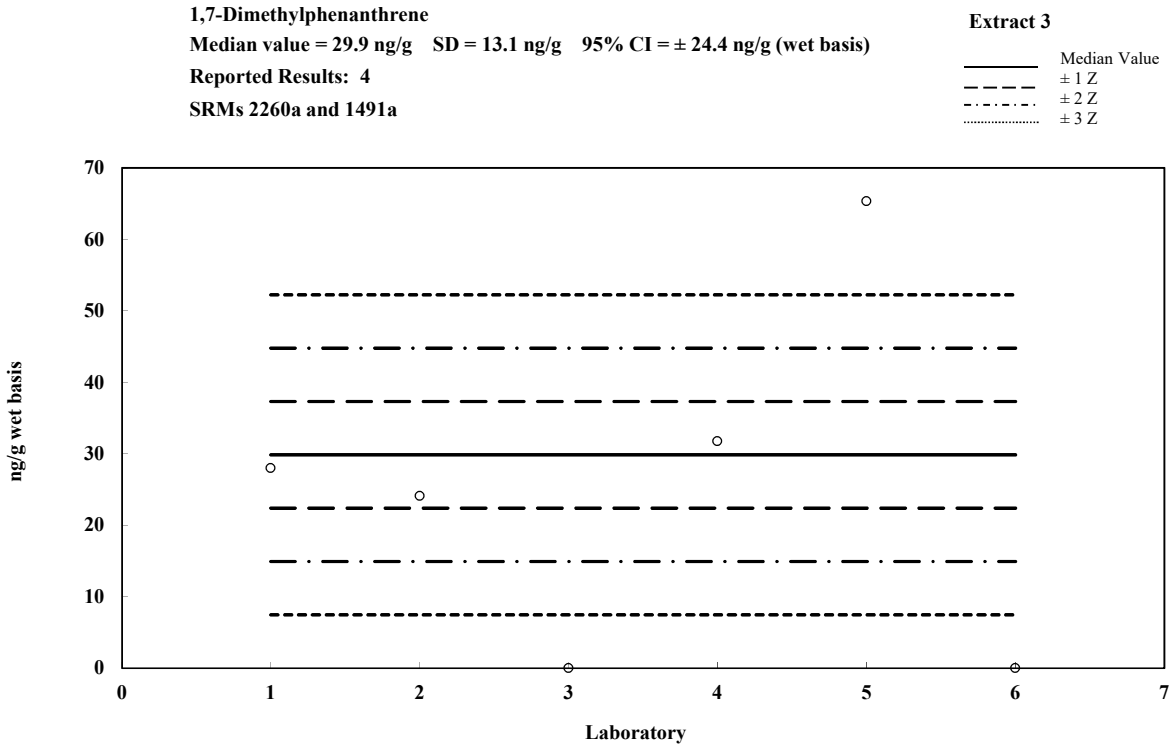


Phenanthrene
Value = 0 ± 0 ng/g (wet basis)
Reported Results: 1
SRMs 2260a and 1491a

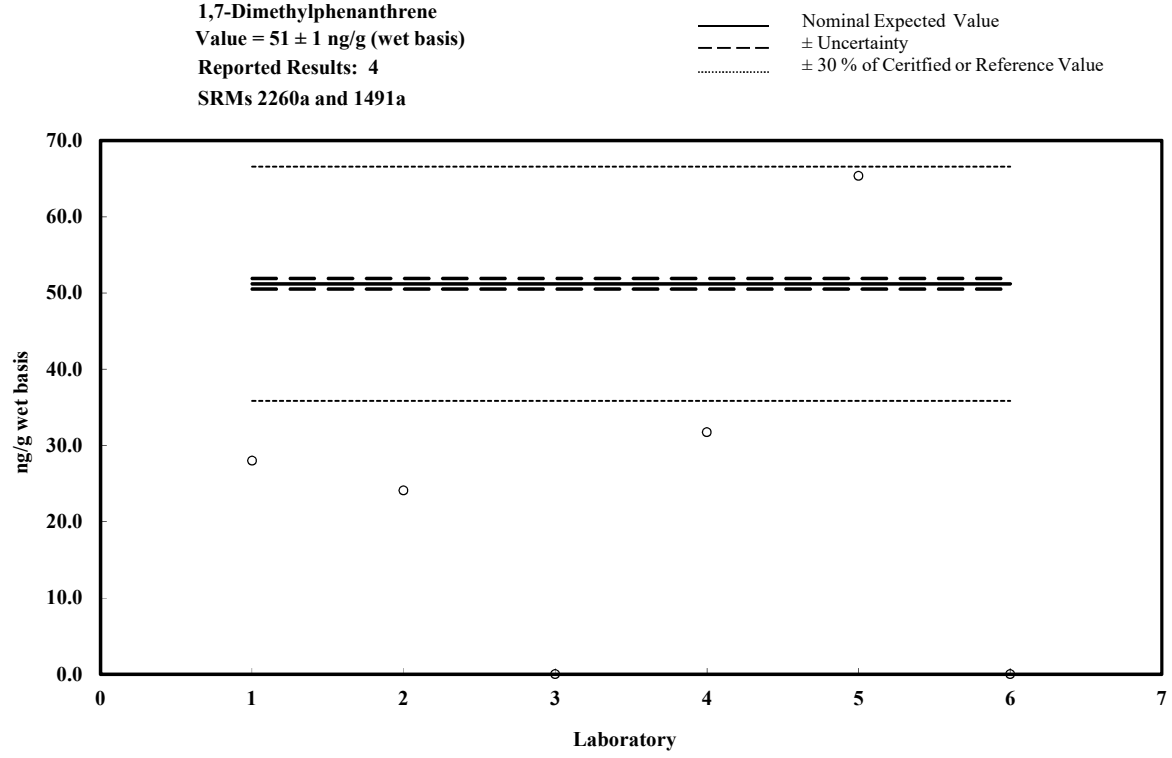


1,7-Dimethylphenanthrene
Median value = 29.9 ng/g SD = 13.1 ng/g 95% CI = ± 24.4 ng/g (wet basis)
Reported Results: 4
SRMs 2260a and 1491a

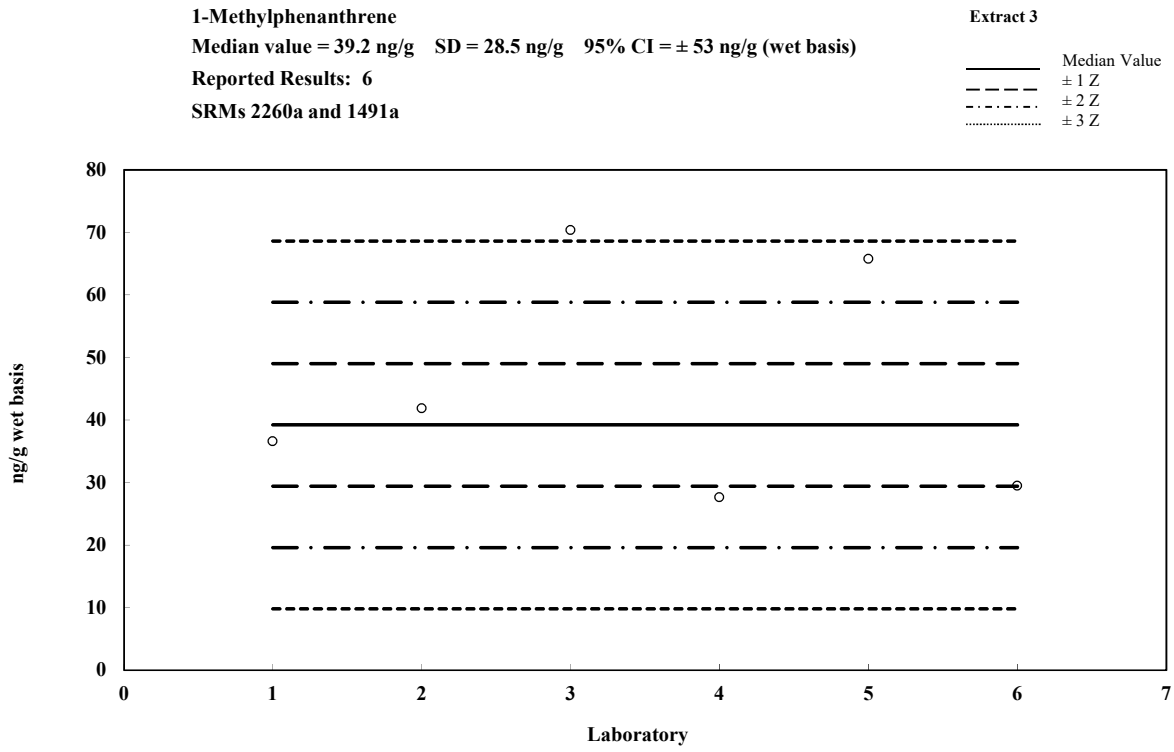
Extract 3



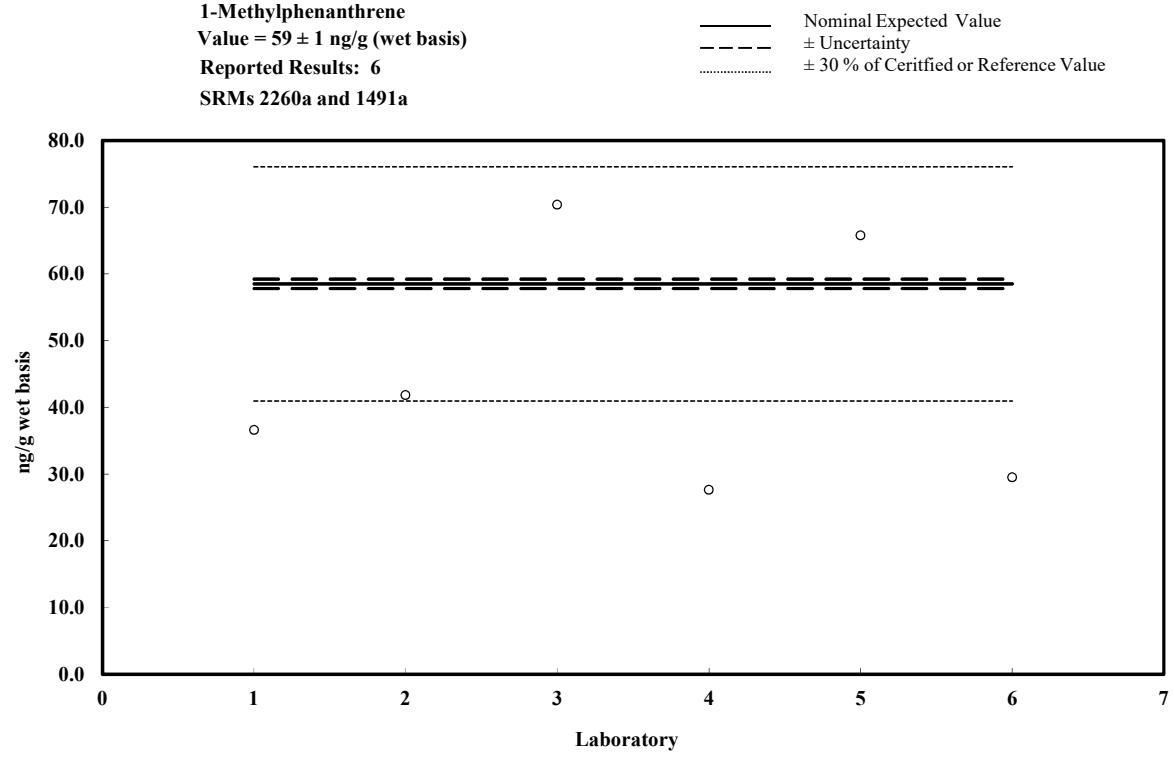
1,7-Dimethylphenanthrene
Value = 51 ± 1 ng/g (wet basis)
Reported Results: 4
SRMs 2260a and 1491a



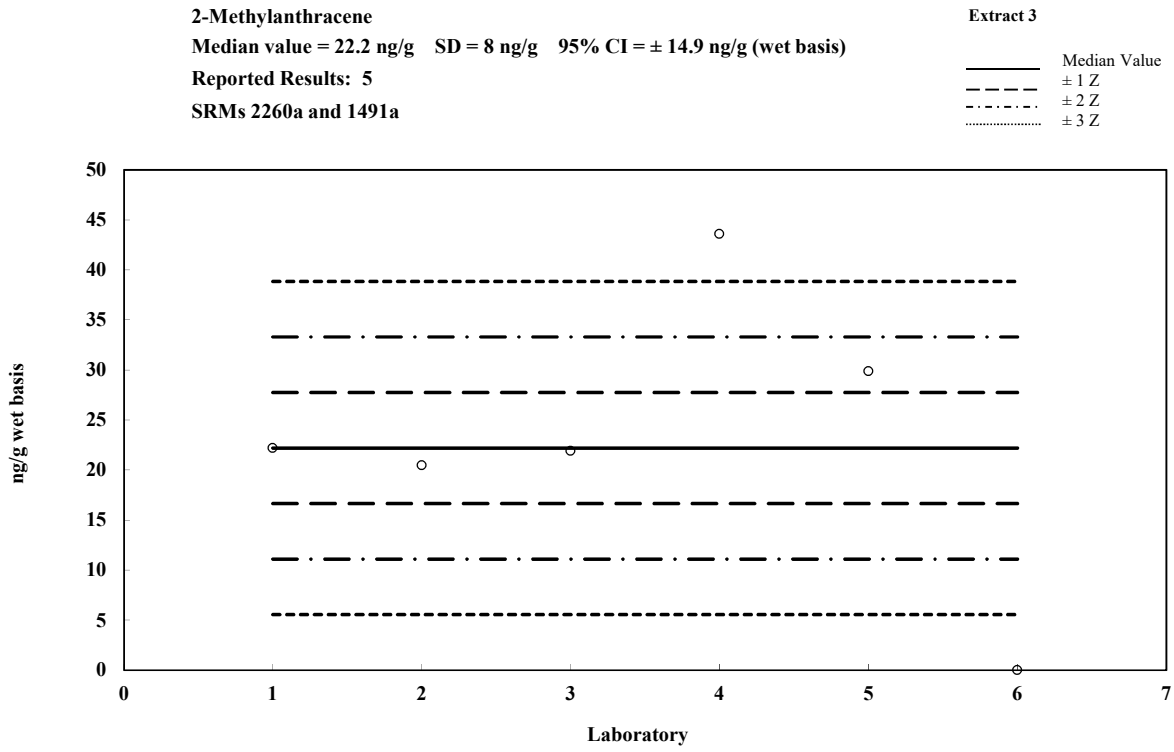
1-Methylphenanthrene
Median value = 39.2 ng/g SD = 28.5 ng/g 95% CI = ± 53 ng/g (wet basis)
Reported Results: 6
SRMs 2260a and 1491a



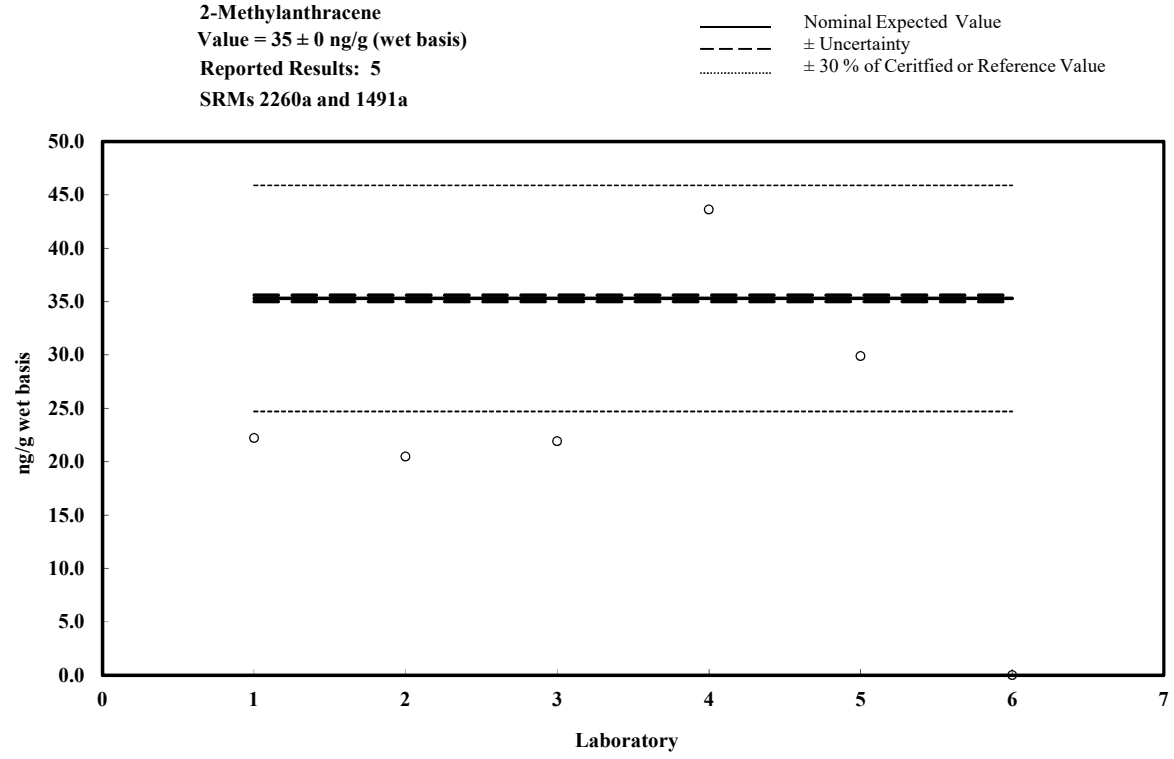
1-Methylphenanthrene
Value = 59 ± 1 ng/g (wet basis)
Reported Results: 6
SRMs 2260a and 1491a



2-Methylanthracene
Median value = 22.2 ng/g SD = 8 ng/g 95% CI = ± 14.9 ng/g (wet basis)
Reported Results: 5
SRMs 2260a and 1491a

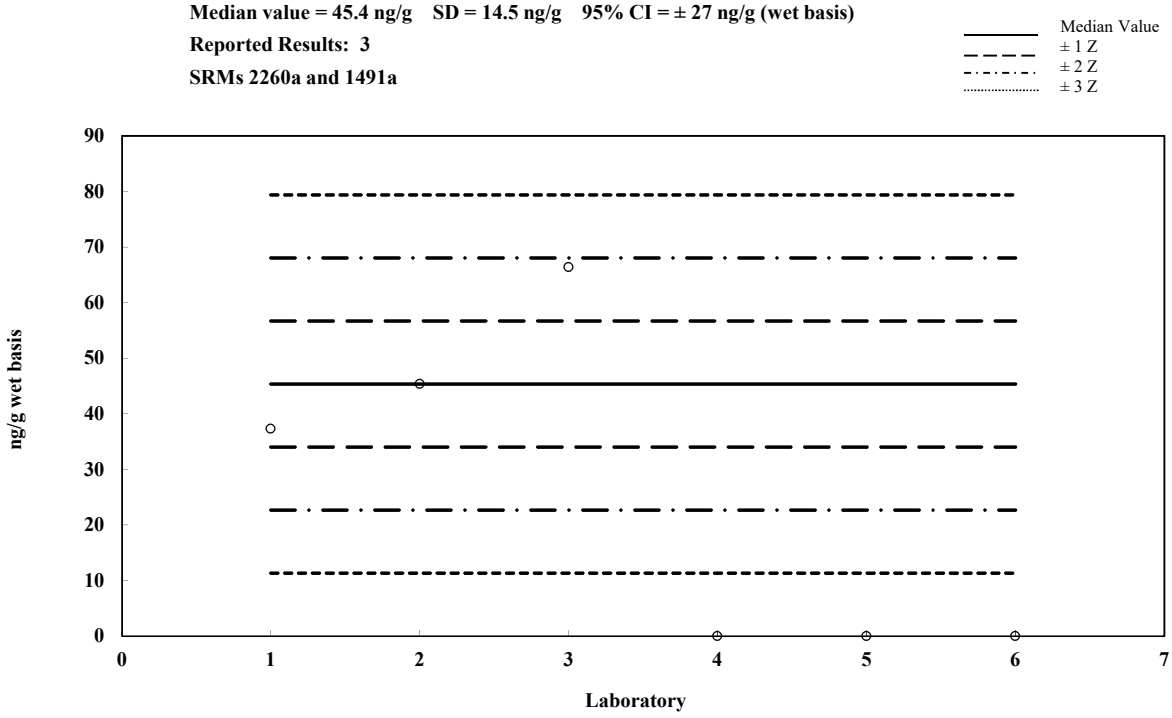


2-Methylanthracene
Value = 35 ± 0 ng/g (wet basis)
Reported Results: 5
SRMs 2260a and 1491a

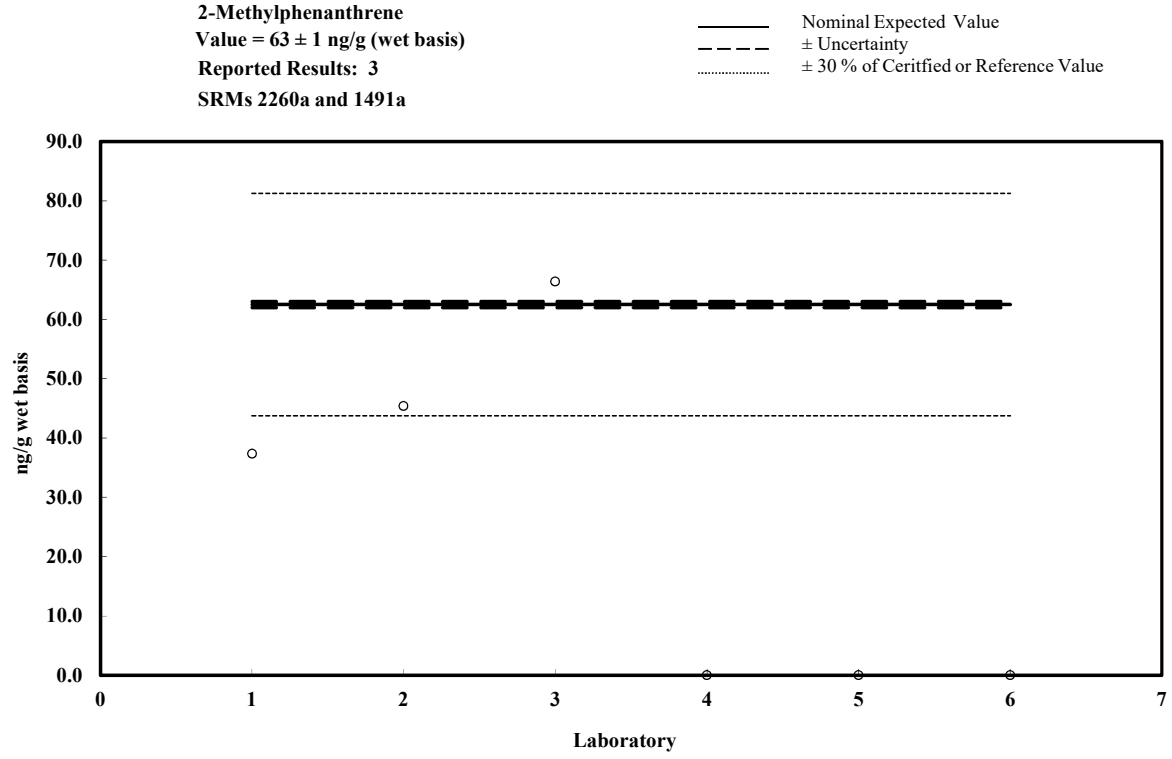


2-Methylphenanthrene
Median value = 45.4 ng/g SD = 14.5 ng/g 95% CI = ± 27 ng/g (wet basis)
Reported Results: 3
SRMs 2260a and 1491a

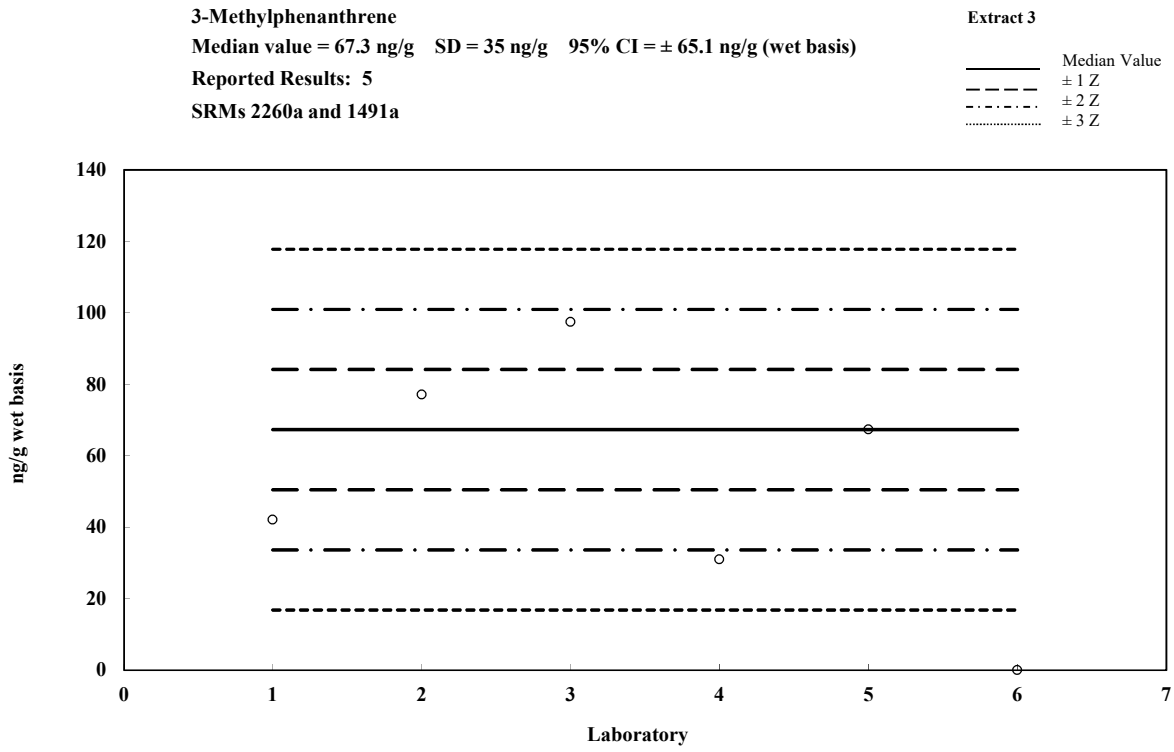
Extract 3



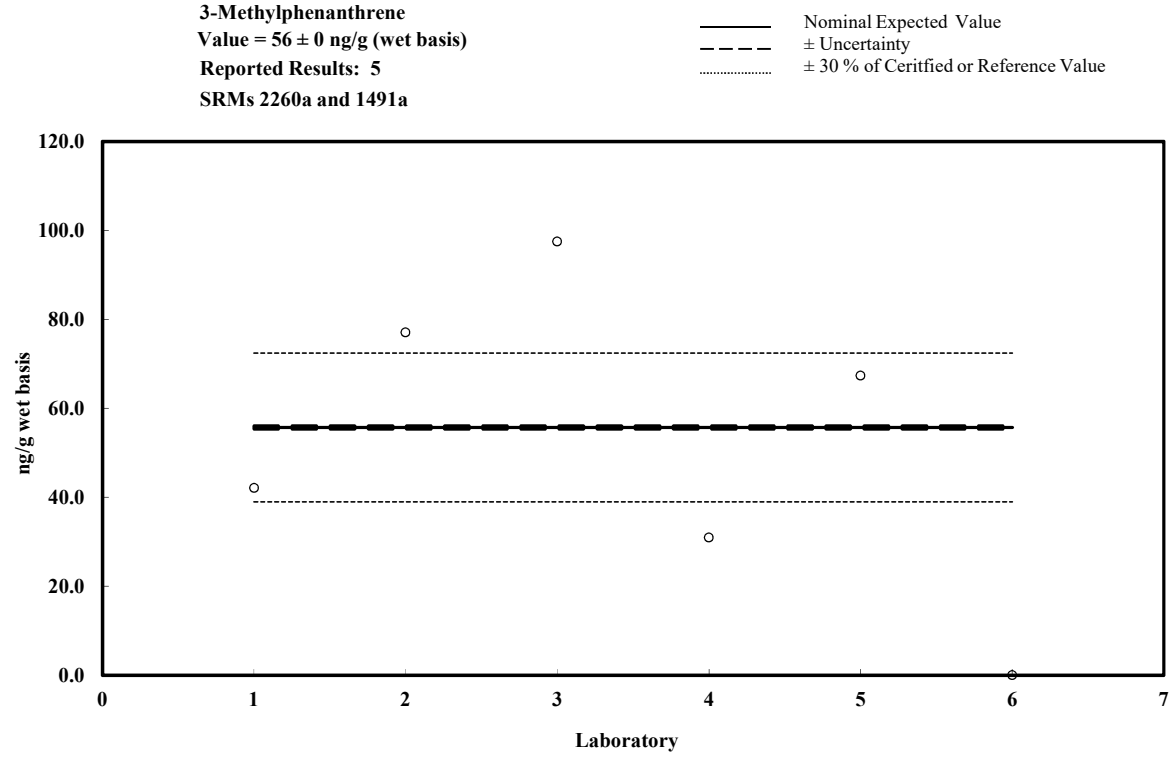
2-Methylphenanthrene
Value = 63 ± 1 ng/g (wet basis)
Reported Results: 3
SRMs 2260a and 1491a



3-Methylphenanthrene
Median value = 67.3 ng/g SD = 35 ng/g 95% CI = ± 65.1 ng/g (wet basis)
Reported Results: 5
SRMs 2260a and 1491a



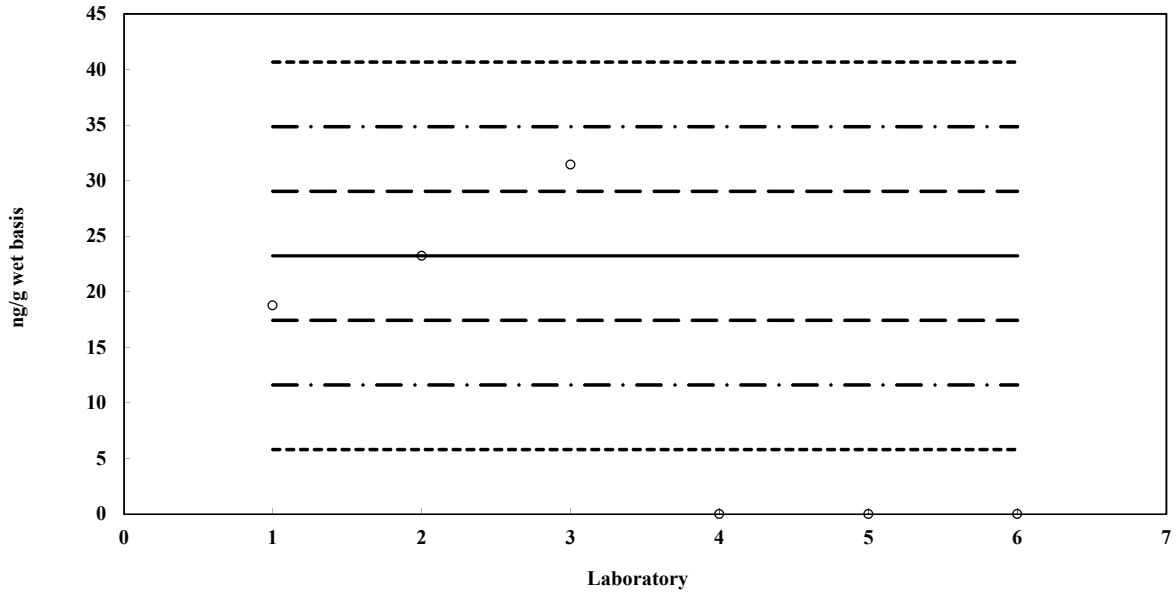
3-Methylphenanthrene
Value = 56 ± 0 ng/g (wet basis)
Reported Results: 5
SRMs 2260a and 1491a



4H-Cyclopenta[def]phenanthrene
Median value = 23.2 ng/g SD = 6.3 ng/g 95% CI = ± 11.7 ng/g (wet basis)
Reported Results: 3
SRMs 2260a and 1491a

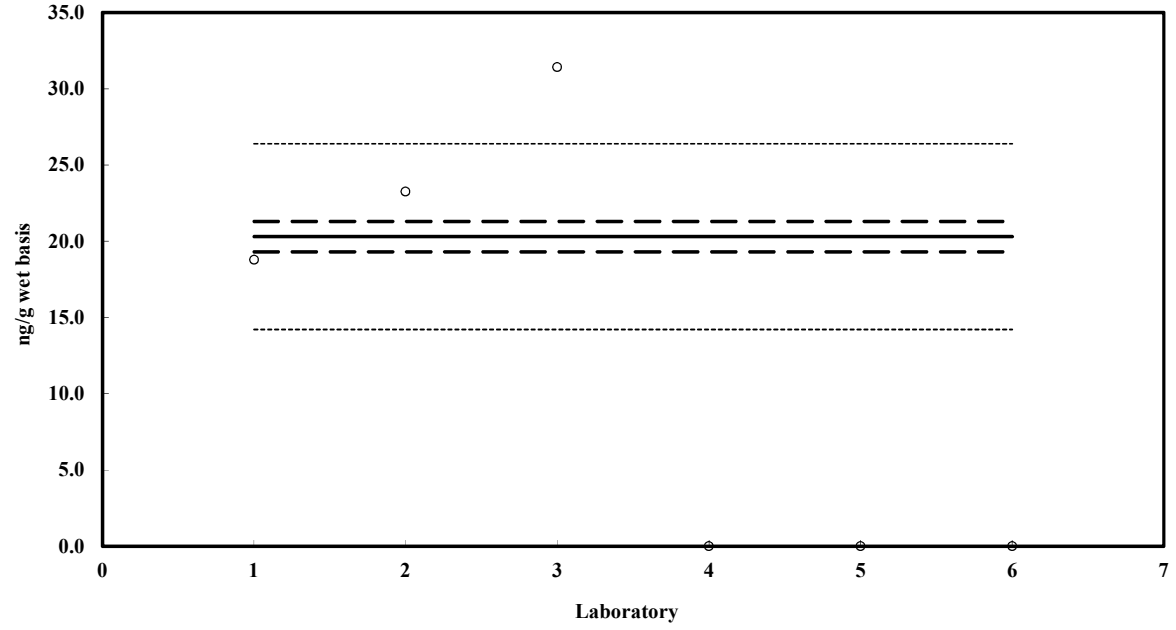
Extract 3

Median Value
± 1 Z
± 2 Z
± 3 Z

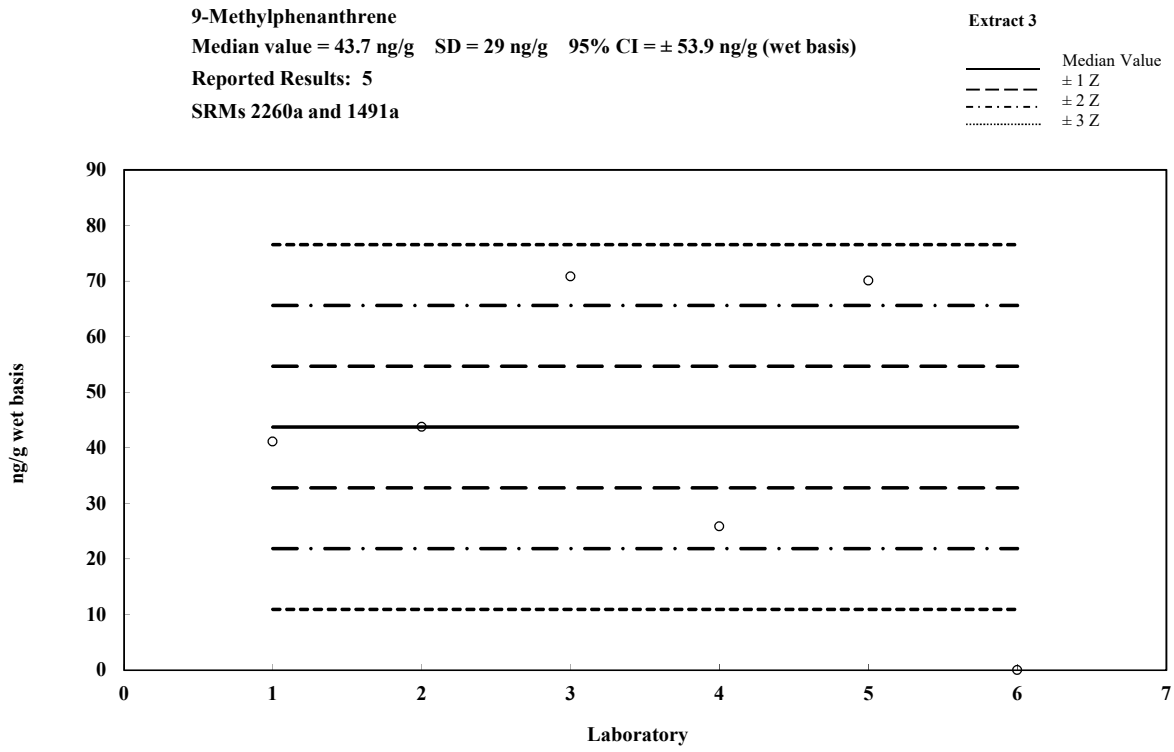


4H-Cyclopenta[def]phenanthrene
Value = 20 ± 1 ng/g (wet basis)
Reported Results: 3
SRMs 2260a and 1491a

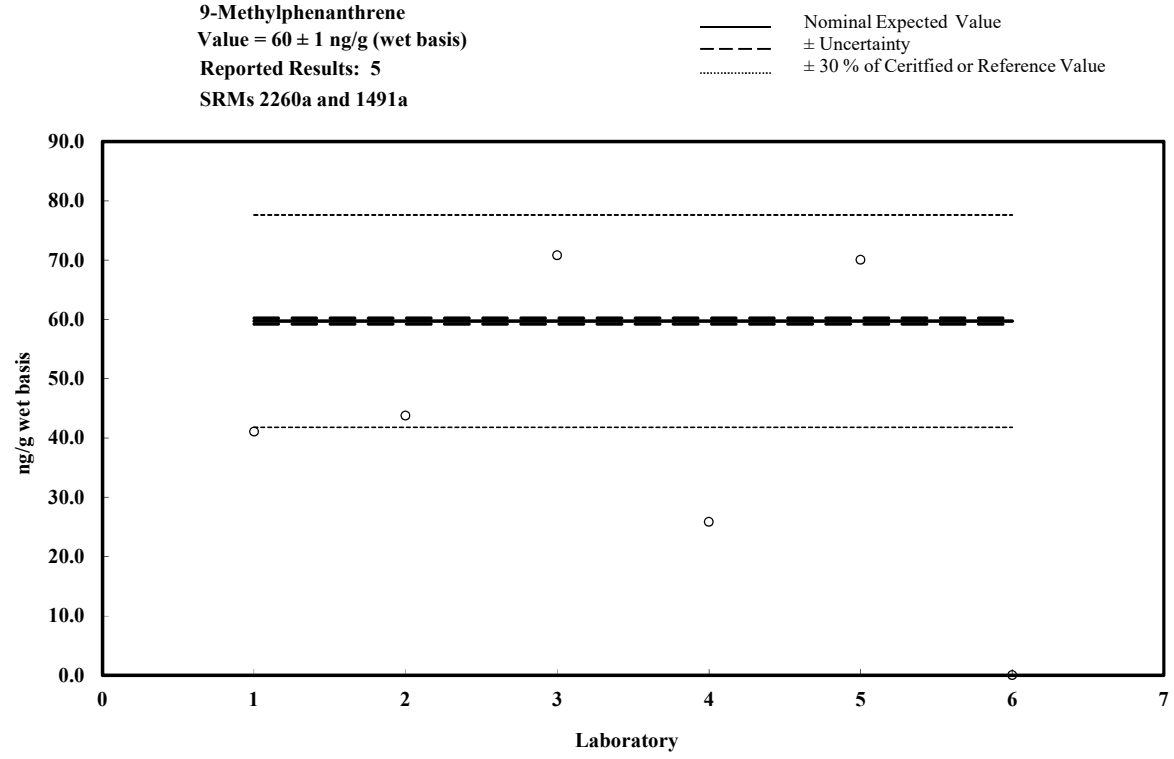
Nominal Expected Value
± Uncertainty
± 30 % of Ceritfied or Reference Value

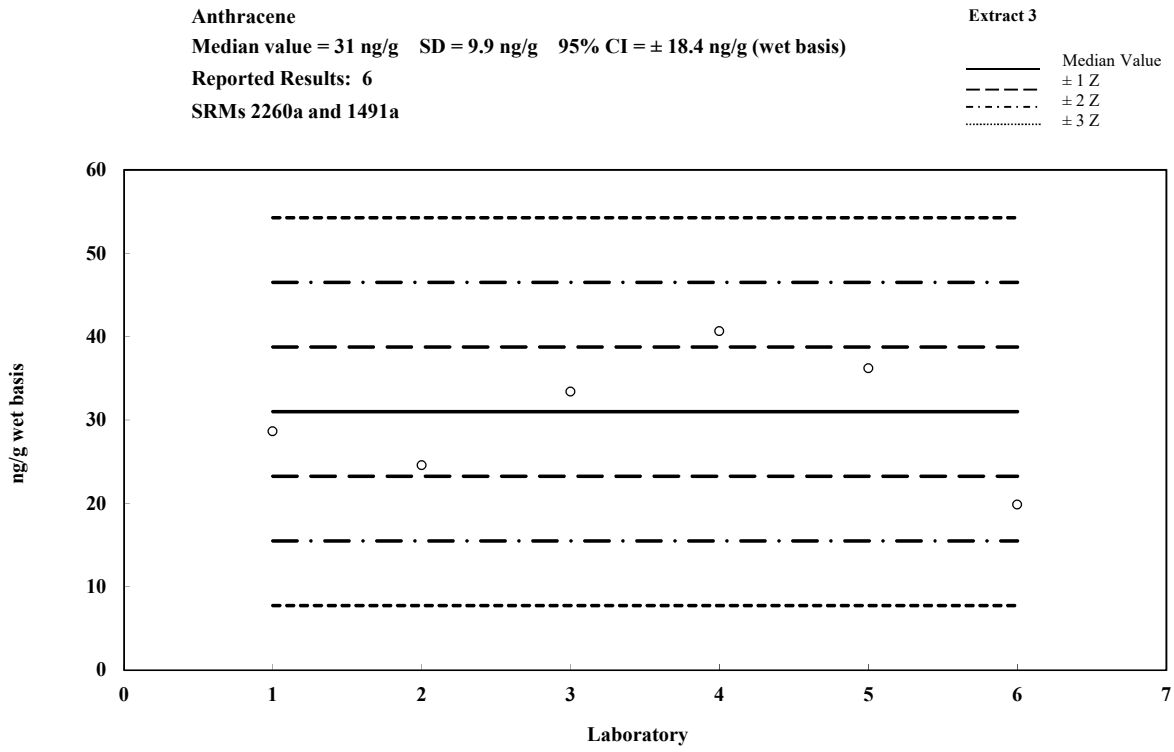


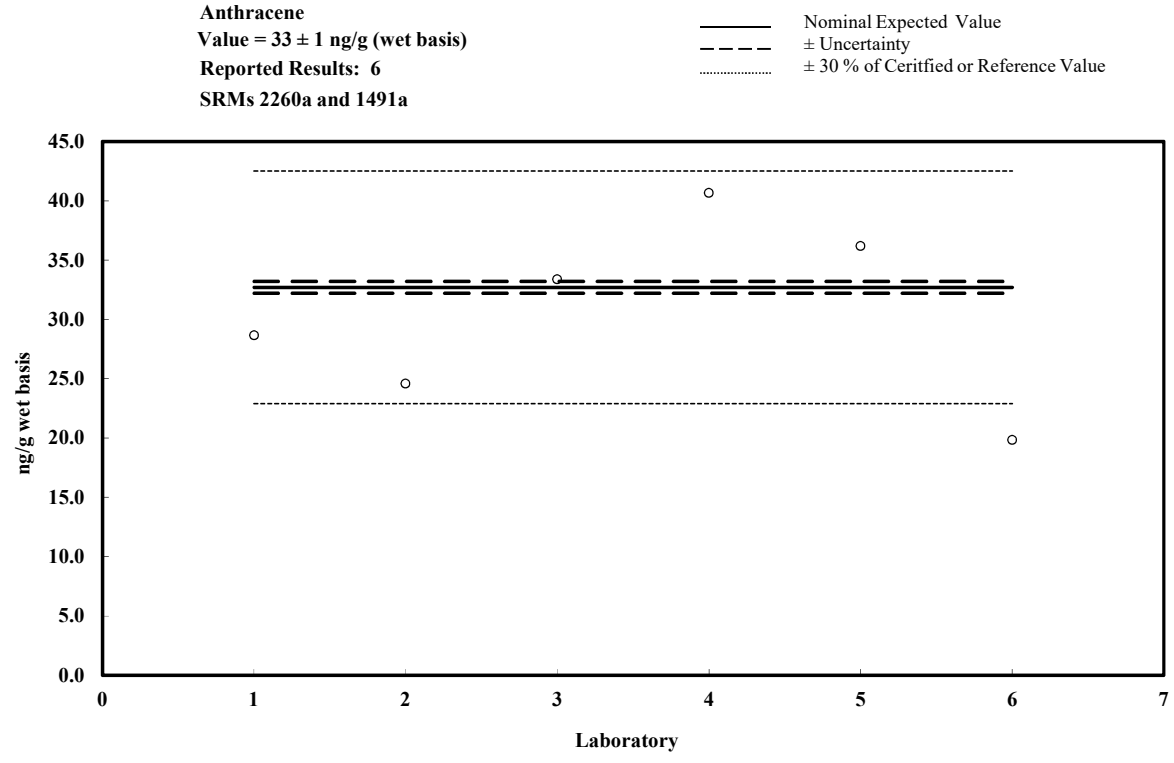
9-Methylphenanthrene
Median value = 43.7 ng/g SD = 29 ng/g 95% CI = ± 53.9 ng/g (wet basis)
Reported Results: 5
SRMs 2260a and 1491a



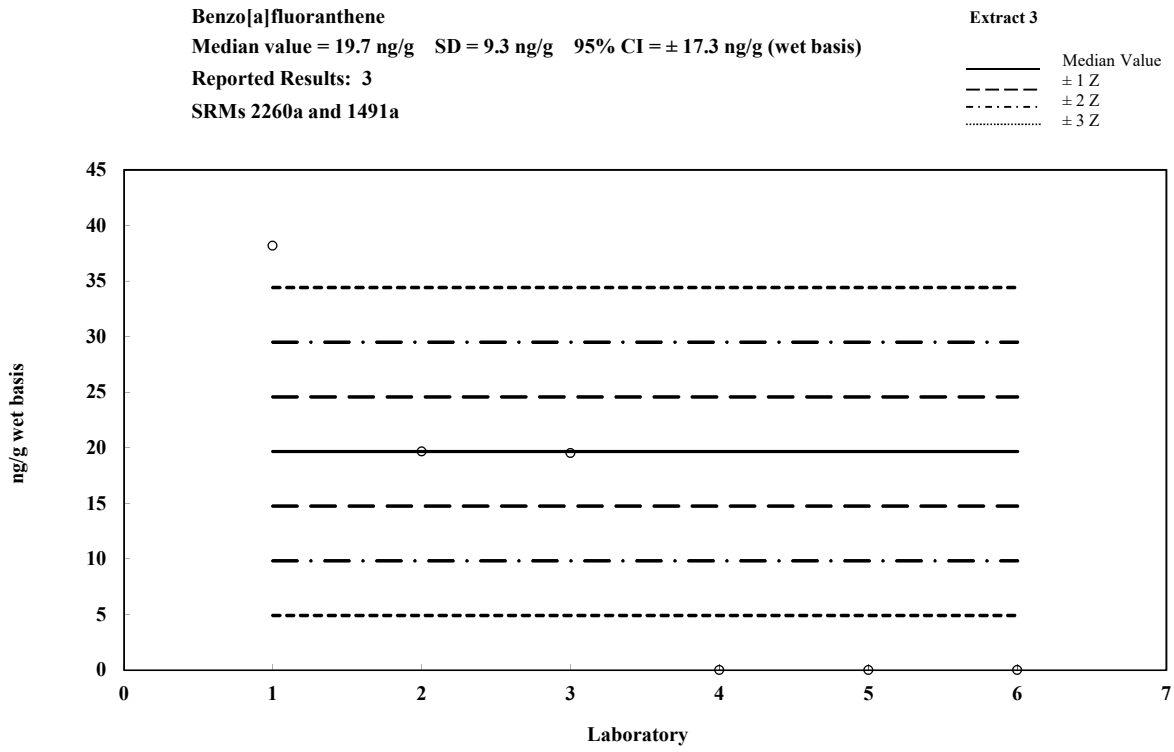
9-Methylphenanthrene
Value = 60 ± 1 ng/g (wet basis)
Reported Results: 5
SRMs 2260a and 1491a



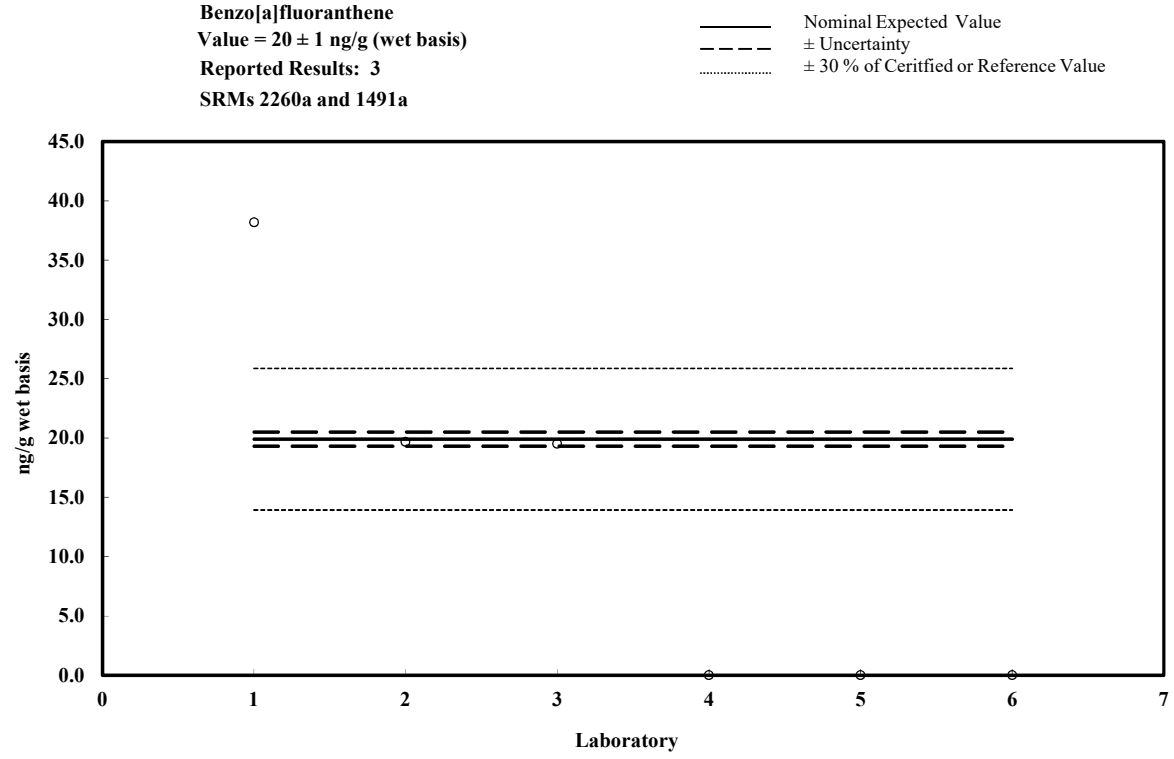




Benzo[a]fluoranthene
Median value = 19.7 ng/g SD = 9.3 ng/g 95% CI = ± 17.3 ng/g (wet basis)
Reported Results: 3
SRMs 2260a and 1491a



Benzo[a]fluoranthene
Value = 20 ± 1 ng/g (wet basis)
Reported Results: 3
SRMs 2260a and 1491a

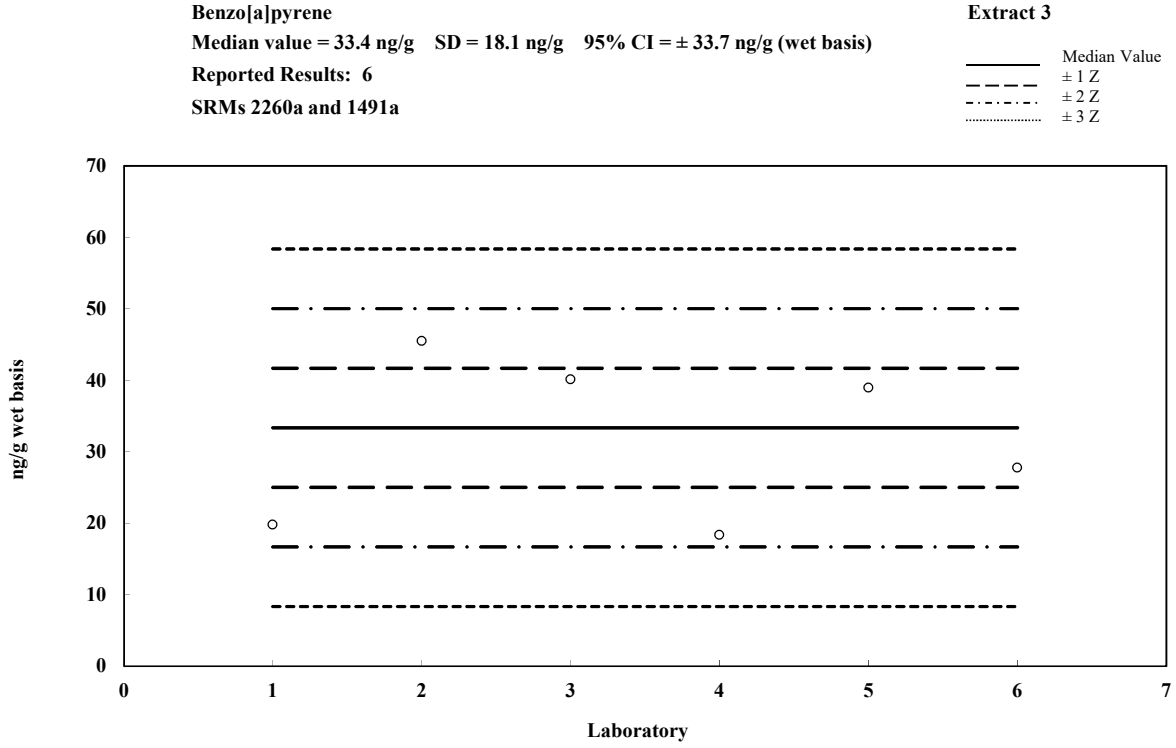


Benzo[a]pyrene **Extract 3**

Median value = 33.4 ng/g SD = 18.1 ng/g 95% CI = ± 33.7 ng/g (wet basis)

Reported Results: 6

SRMs 2260a and 1491a

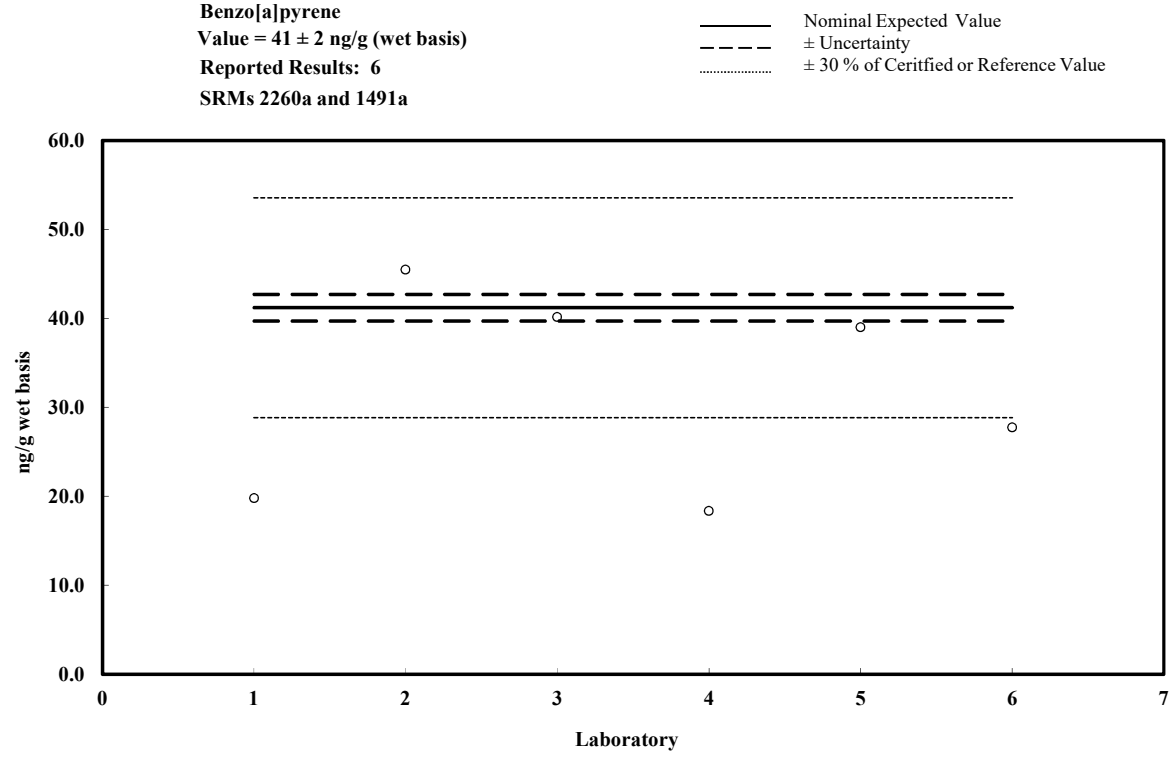


Benzo[a]pyrene

Value = 41 ± 2 ng/g (wet basis)

Reported Results: 6

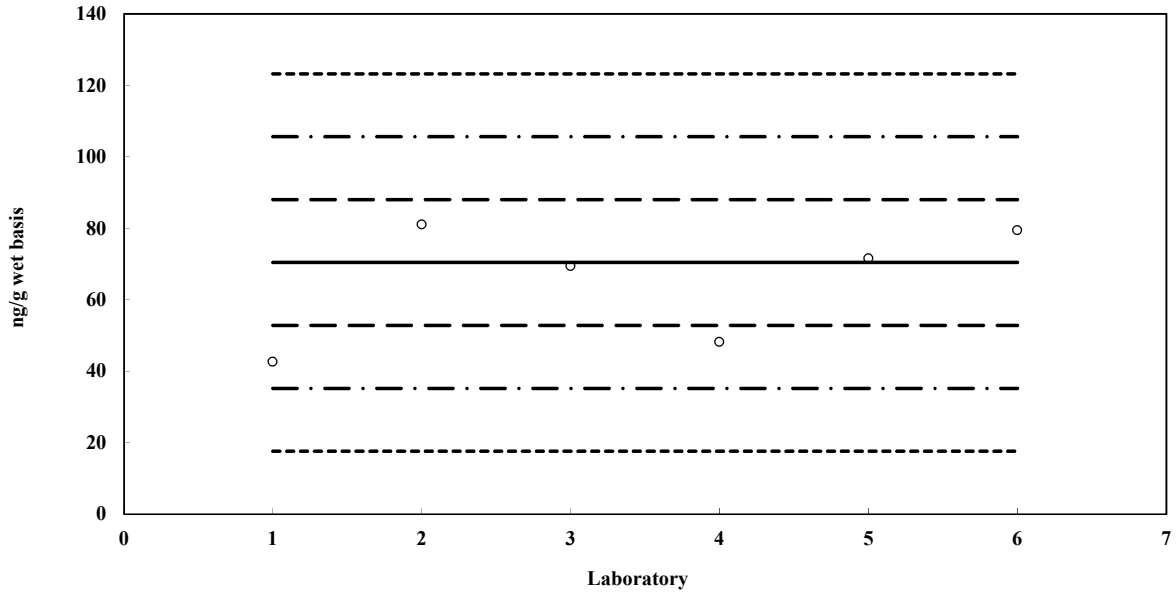
SRMs 2260a and 1491a



Benzo[b]fluoranthene
Median value = 70.4 ng/g SD = 24 ng/g 95% CI = ± 44.6 ng/g (wet basis)
Reported Results: 6
SRMs 2260a and 1491a

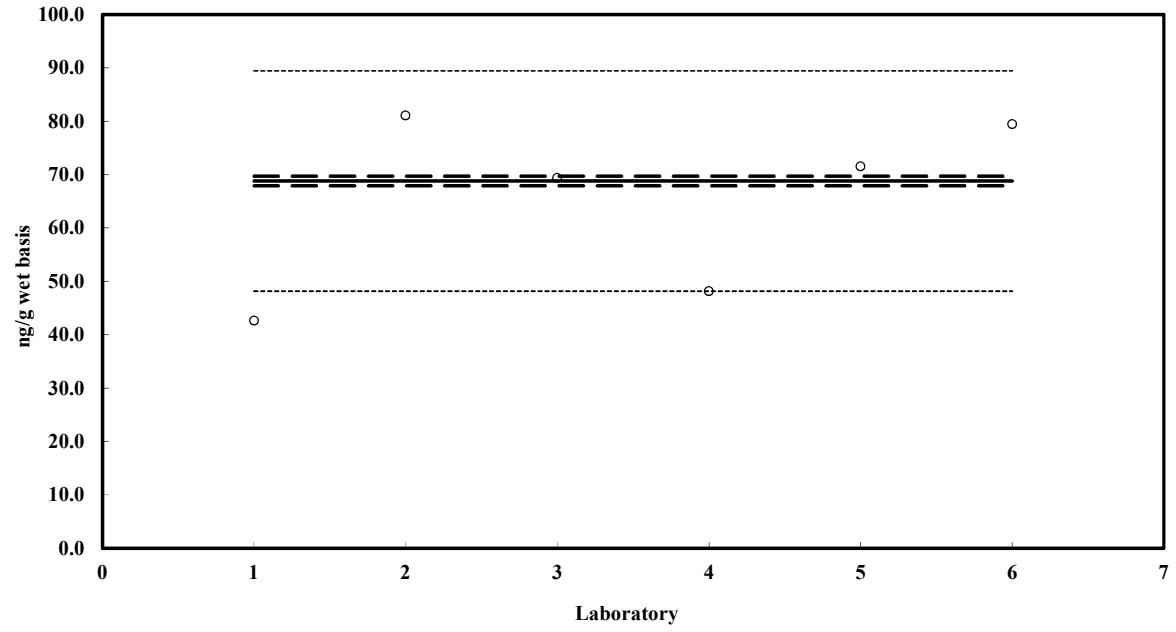
Extract 3

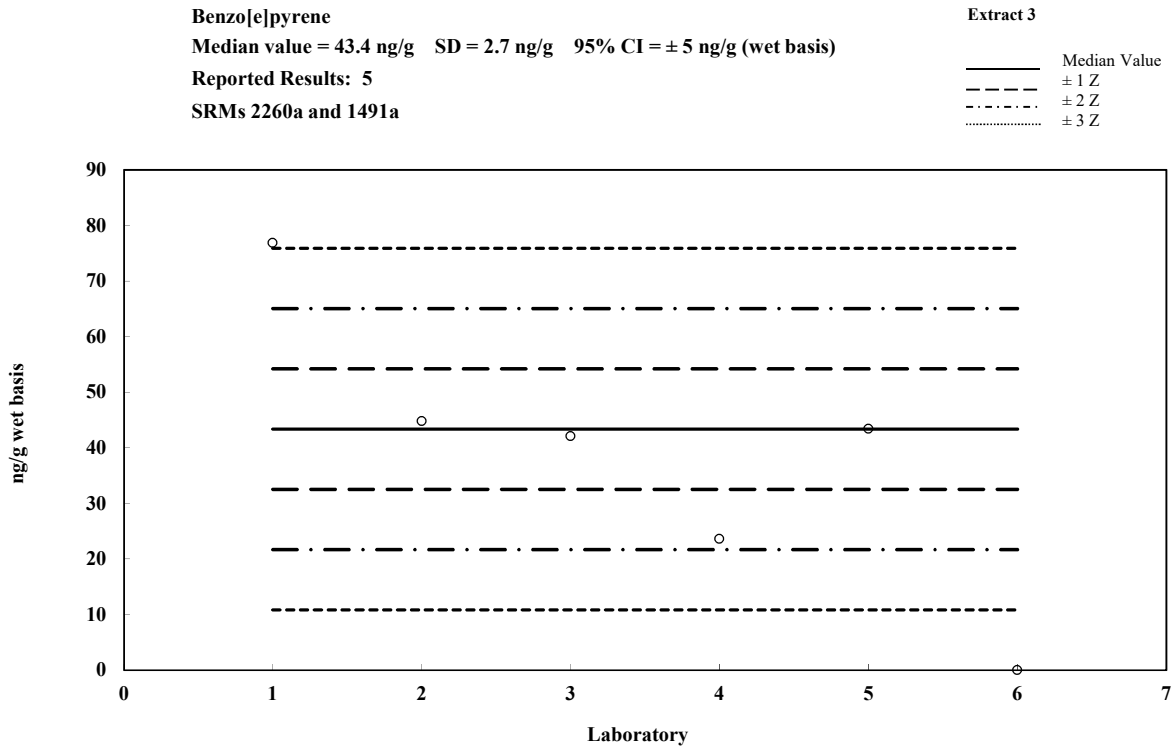
Median Value
± 1 Z
± 2 Z
± 3 Z

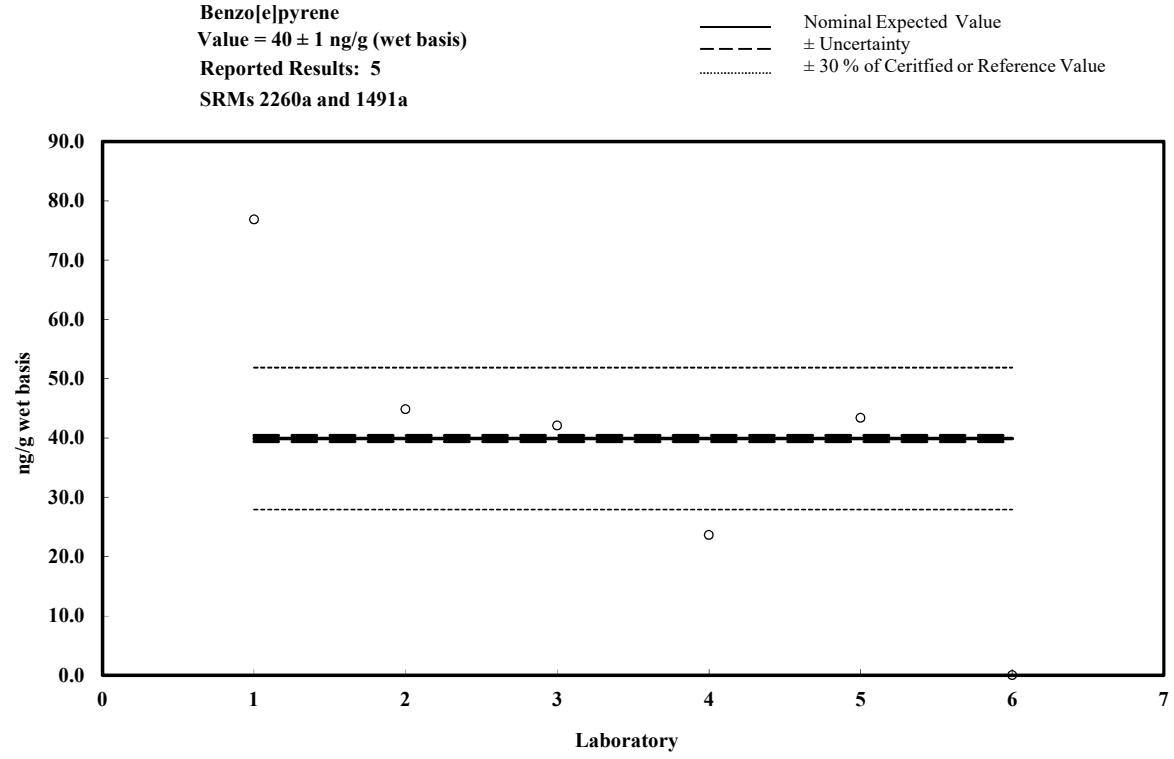


Benzo[b]fluoranthene
Value = 69 ± 1 ng/g (wet basis)
Reported Results: 6
SRMs 2260a and 1491a

Nominal Expected Value
± Uncertainty
± 30 % of Ceritfied or Reference Value

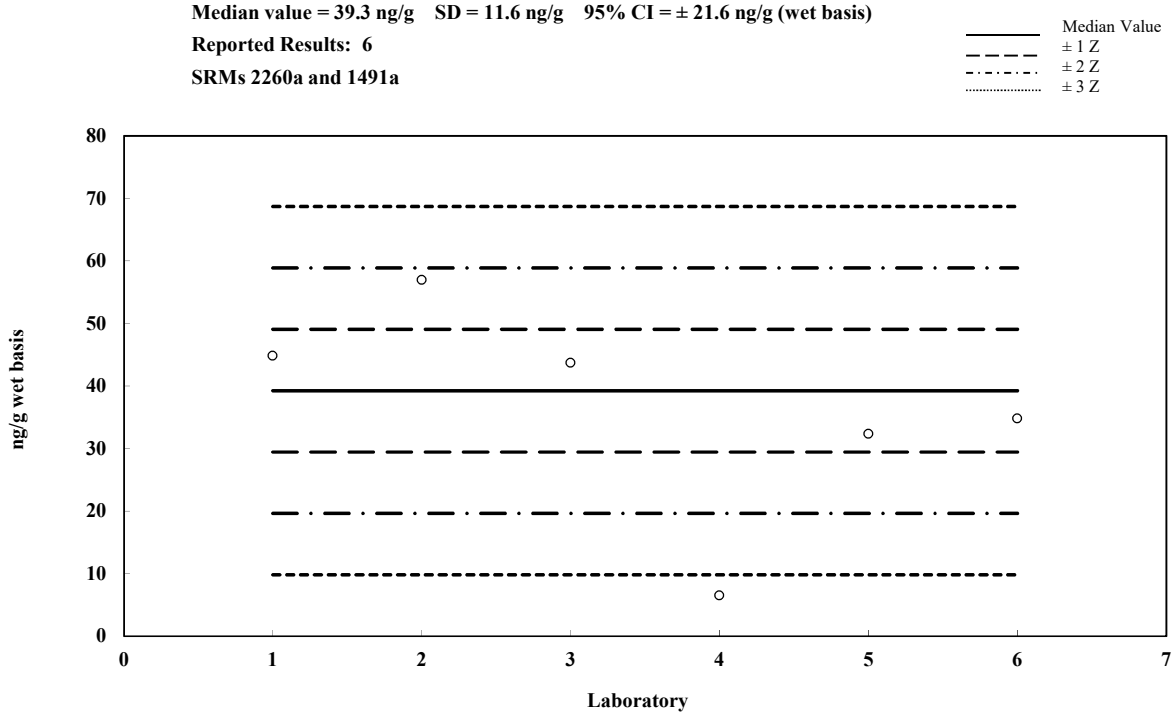




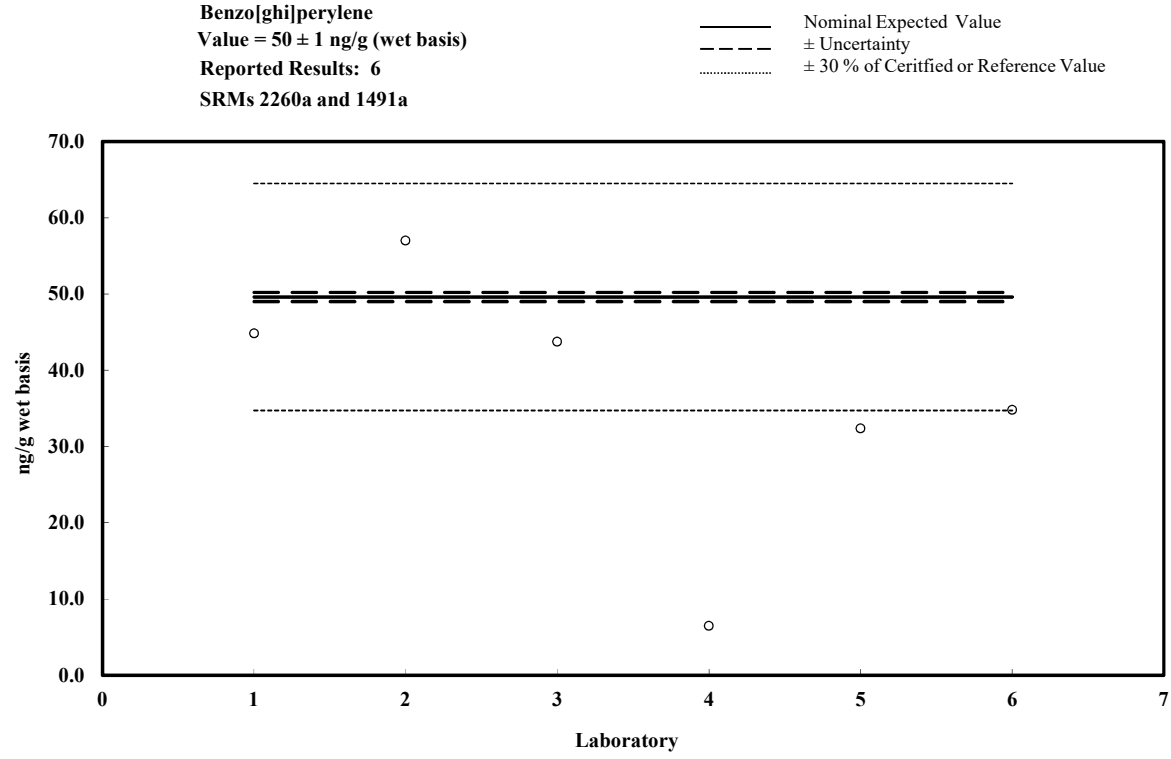


Benzo[ghi]perylene
Median value = 39.3 ng/g SD = 11.6 ng/g 95% CI = ± 21.6 ng/g (wet basis)
Reported Results: 6
SRMs 2260a and 1491a

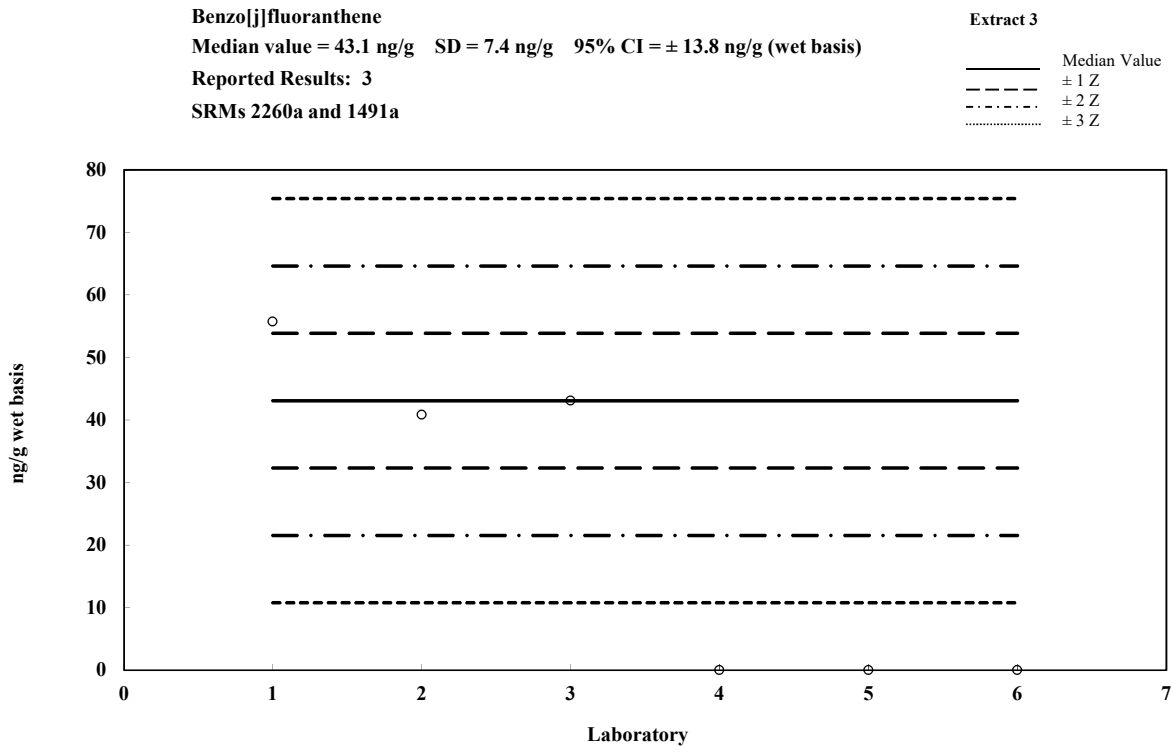
Extract 3



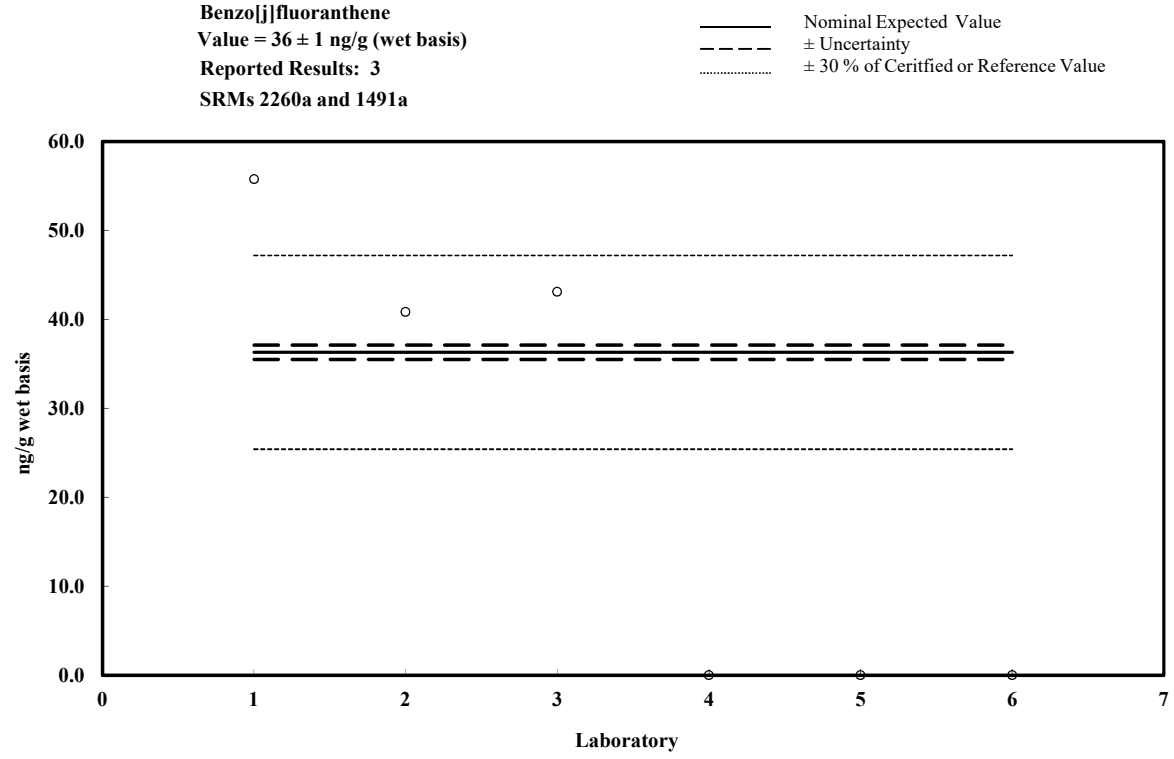
Benzo[ghi]perylene
Value = 50 ± 1 ng/g (wet basis)
Reported Results: 6
SRMs 2260a and 1491a



Benzo[j]fluoranthene
Median value = 43.1 ng/g SD = 7.4 ng/g 95% CI = ± 13.8 ng/g (wet basis)
Reported Results: 3
SRMs 2260a and 1491a

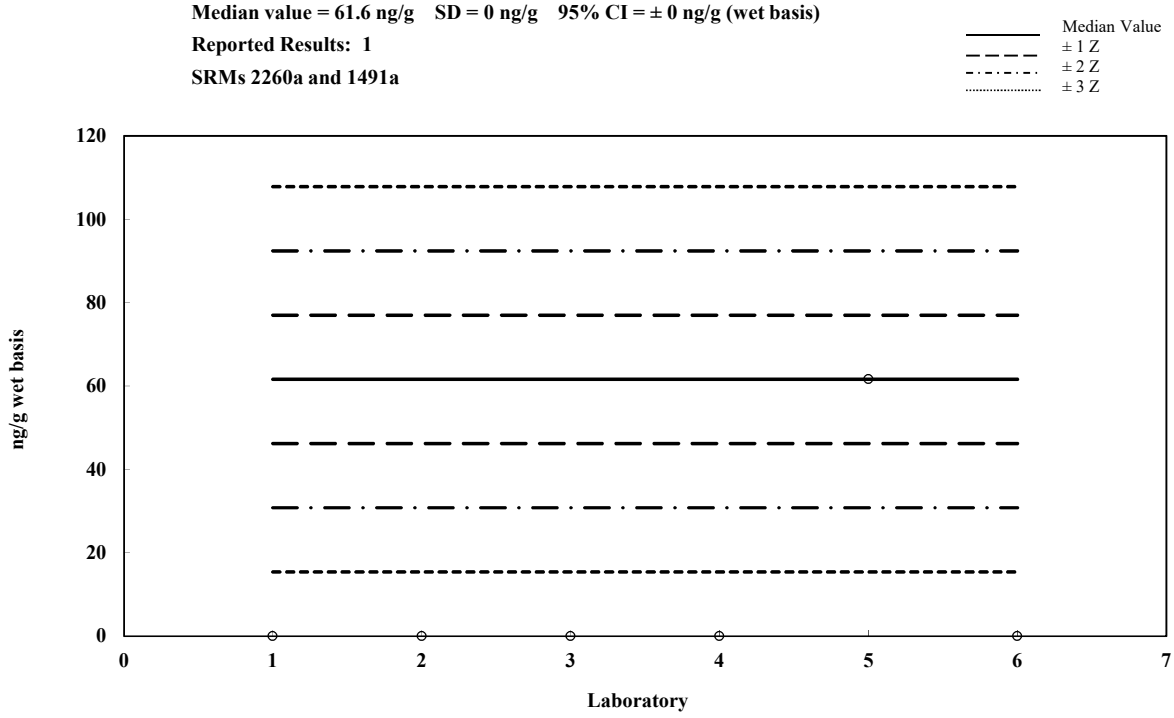


Benzo[j]fluoranthene
Value = 36 ± 1 ng/g (wet basis)
Reported Results: 3
SRMs 2260a and 1491a

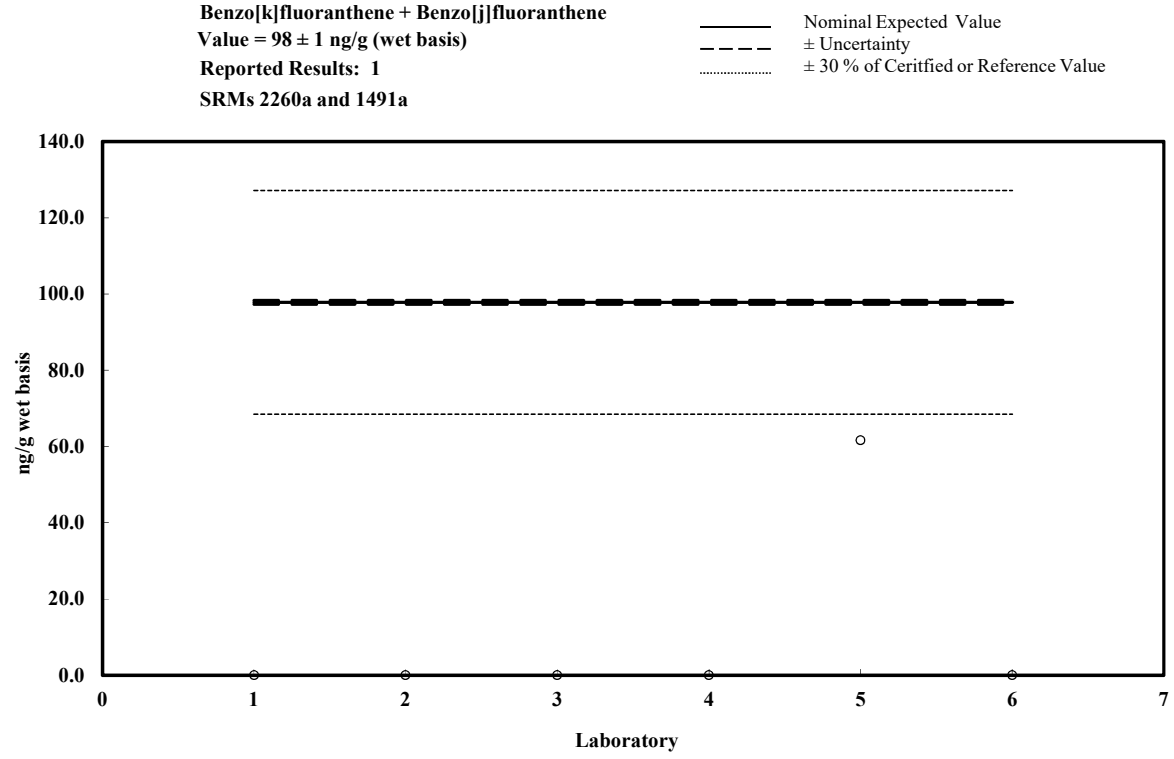


Benzo[k]fluoranthene + Benzo[j]fluoranthene
Median value = 61.6 ng/g SD = 0 ng/g 95% CI = ± 0 ng/g (wet basis)
Reported Results: 1
SRMs 2260a and 1491a

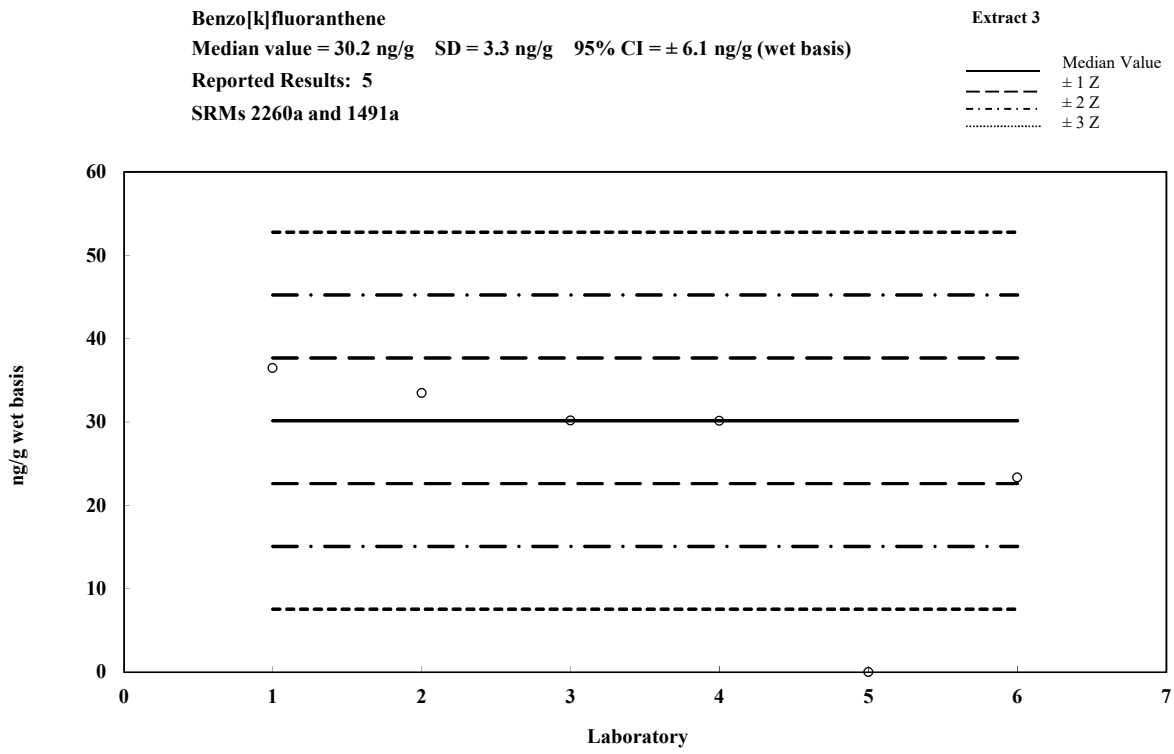
Extract 3



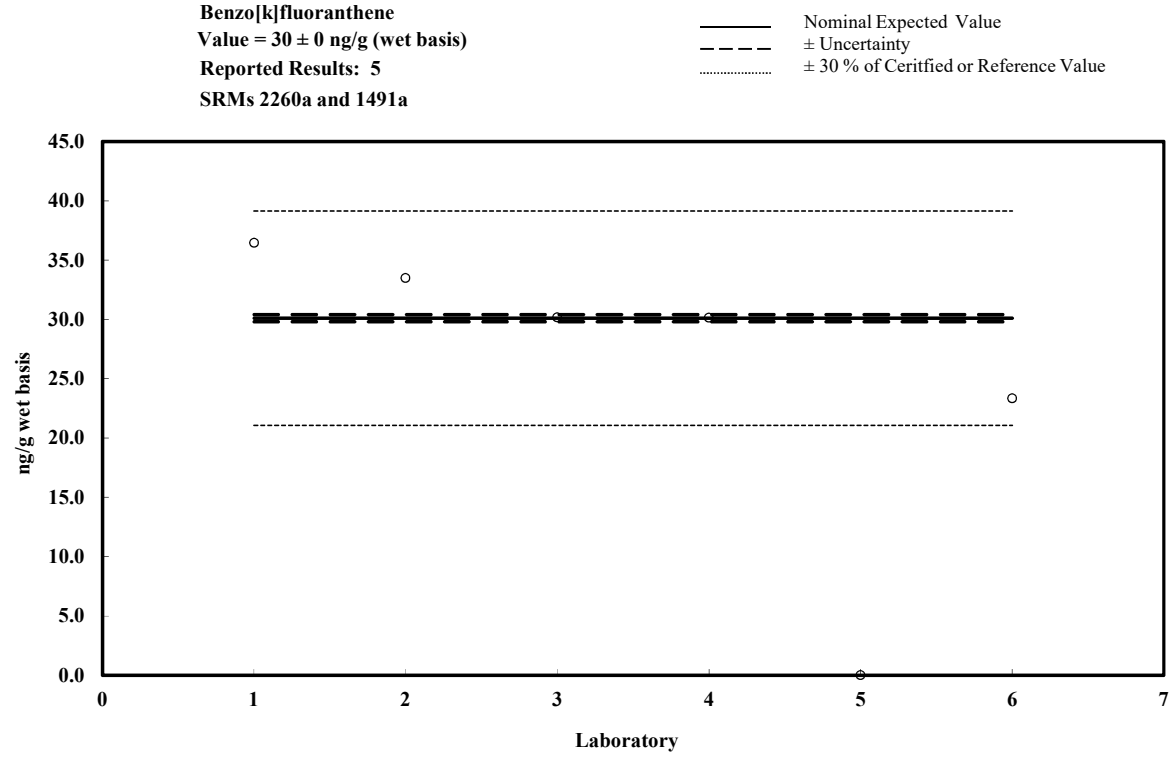
Benzo[k]fluoranthene + Benzo[j]fluoranthene
Value = 98 ± 1 ng/g (wet basis)
Reported Results: 1
SRMs 2260a and 1491a



Benzo[k]fluoranthene
Median value = 30.2 ng/g SD = 3.3 ng/g 95% CI = ± 6.1 ng/g (wet basis)
Reported Results: 5
SRMs 2260a and 1491a

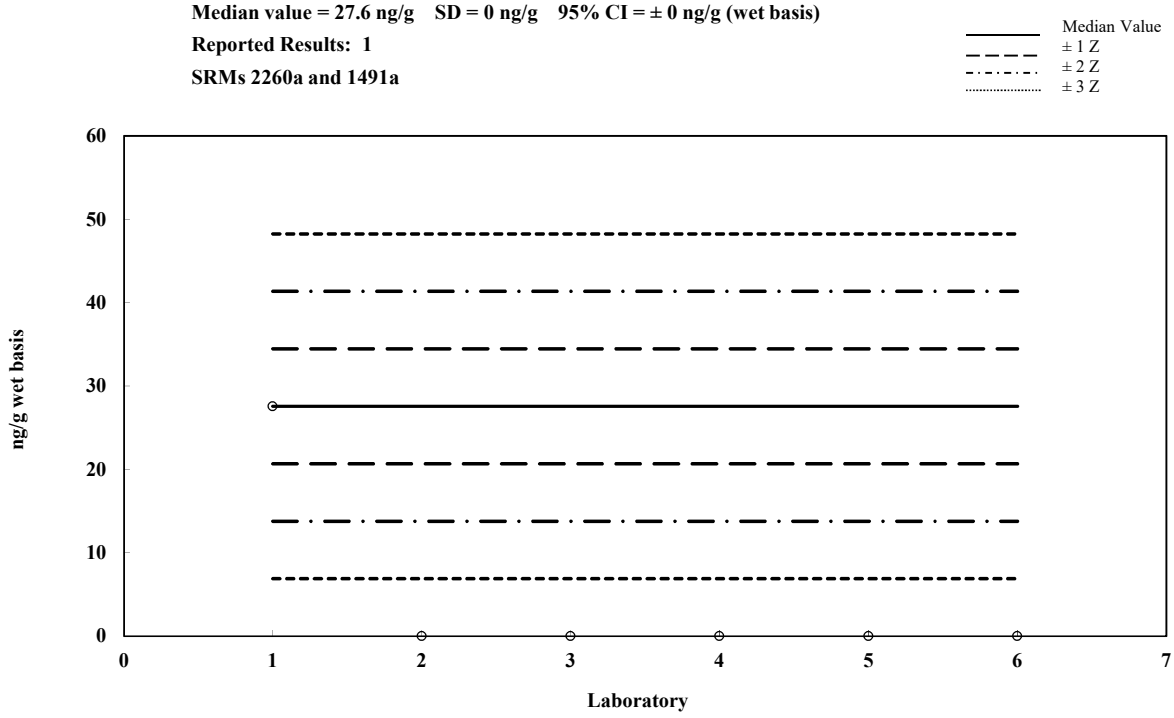


Benzo[k]fluoranthene
Value = 30 ± 0 ng/g (wet basis)
Reported Results: 5
SRMs 2260a and 1491a

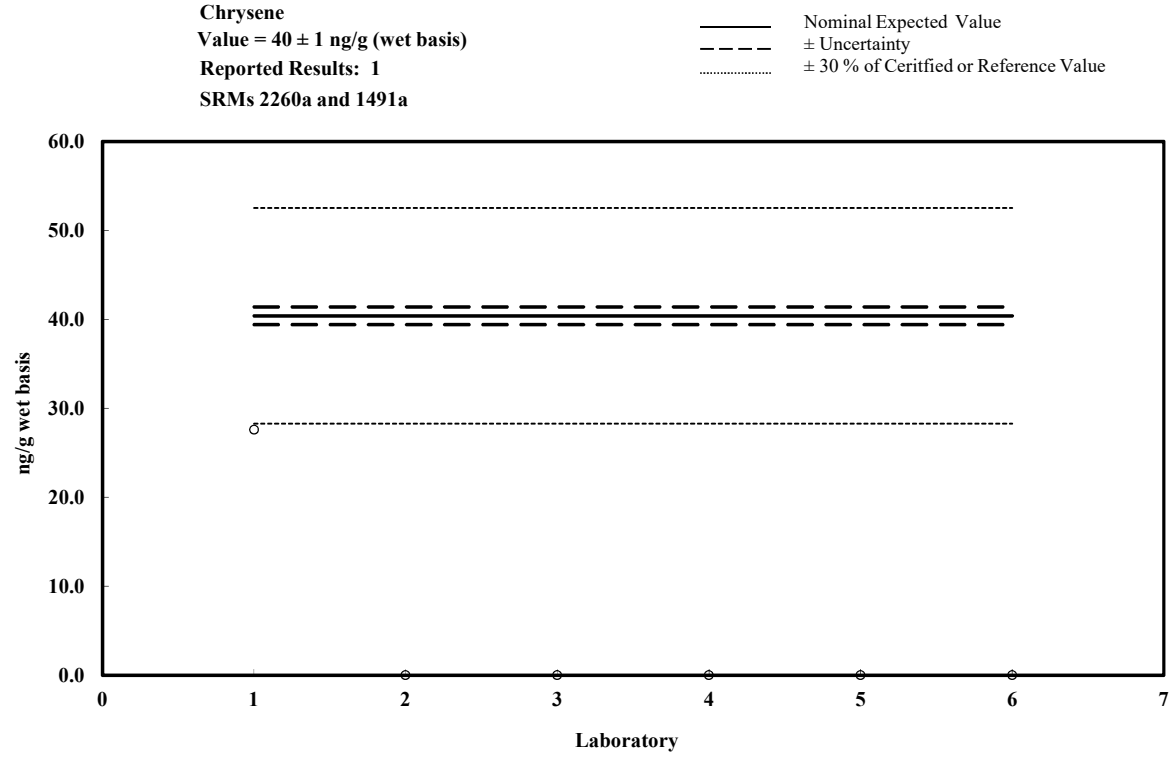


Chrysene
Median value = 27.6 ng/g SD = 0 ng/g 95% CI = ± 0 ng/g (wet basis)
Reported Results: 1
SRMs 2260a and 1491a

Extract 3



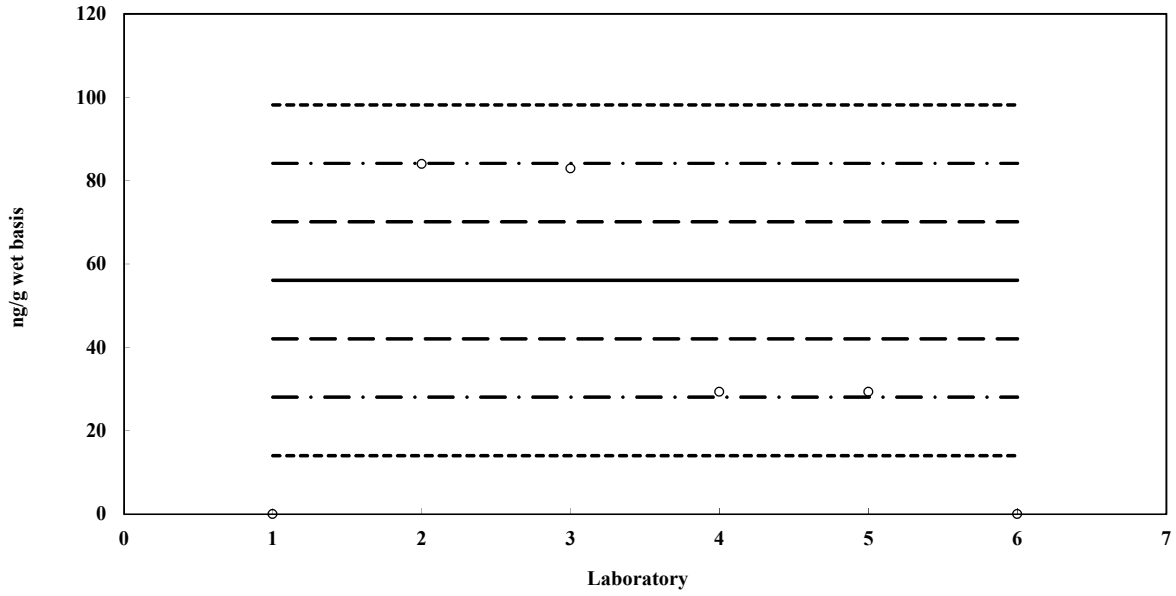
Chrysene
Value = 40 ± 1 ng/g (wet basis)
Reported Results: 1
SRMs 2260a and 1491a



Chrysene+Triphenylene
Median value = 56.1 ng/g SD = 53.9 ng/g 95% CI = ± 100.3 ng/g (wet basis)
Reported Results: 4
SRMs 2260a and 1491a

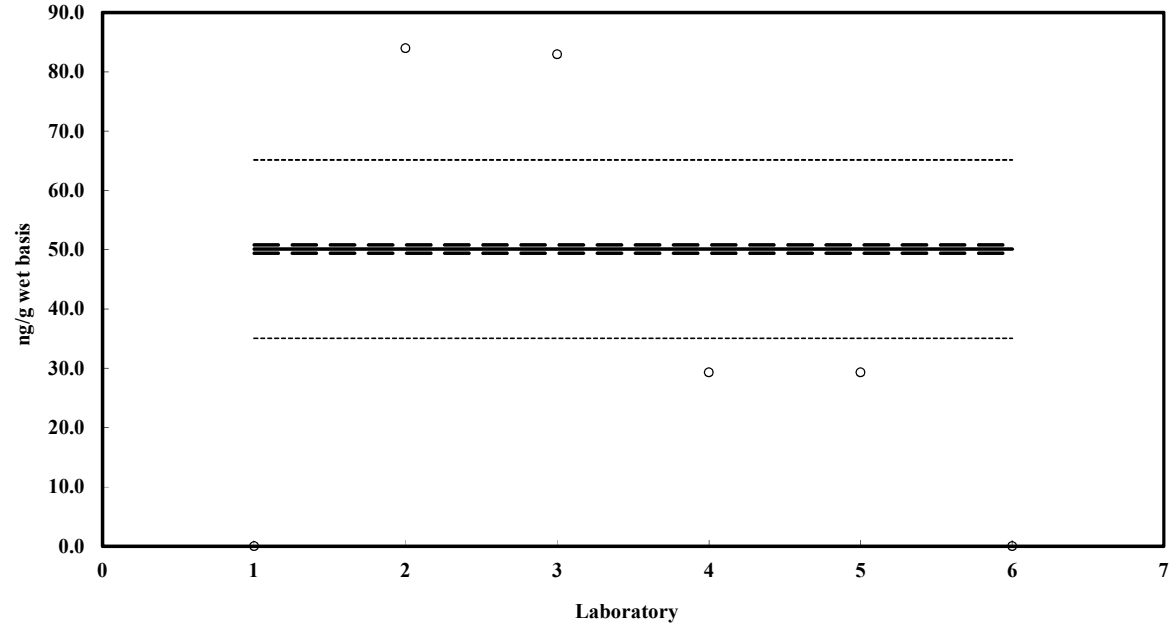
Extract 3

Median Value
± 1 Z
± 2 Z
± 3 Z

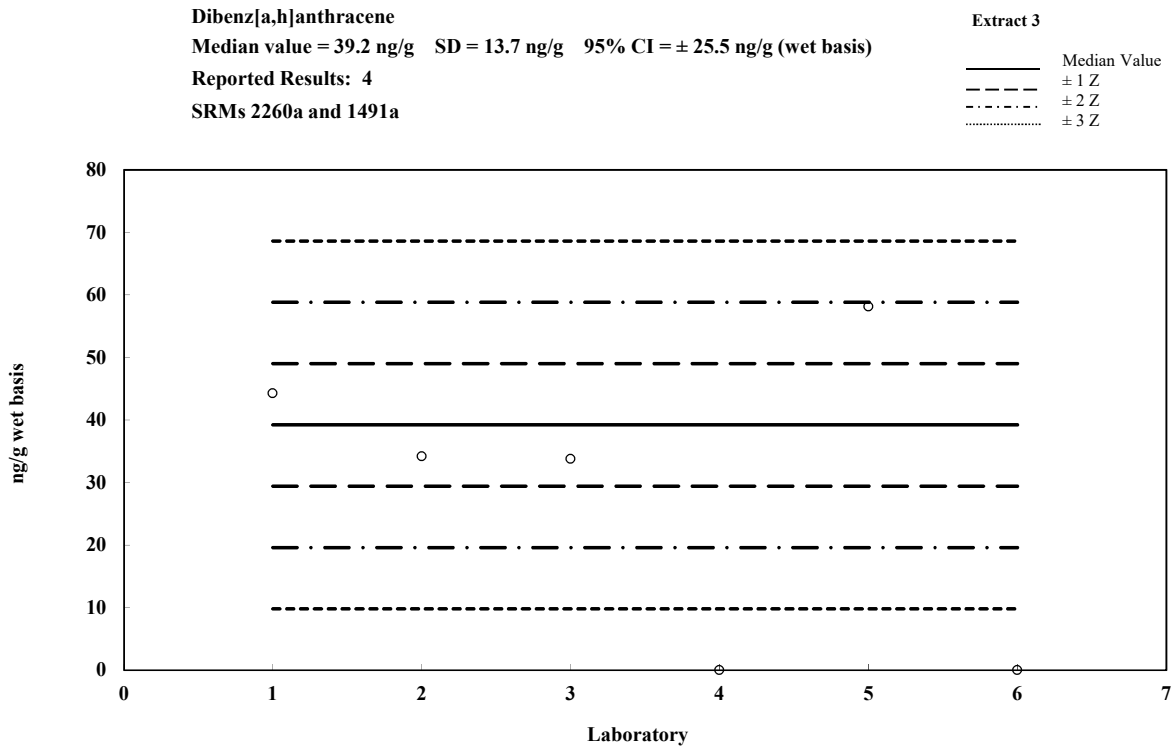


Chrysene+Triphenylene
Value = 50 ± 1 ng/g (wet basis)
Reported Results: 4
SRMs 2260a and 1491a

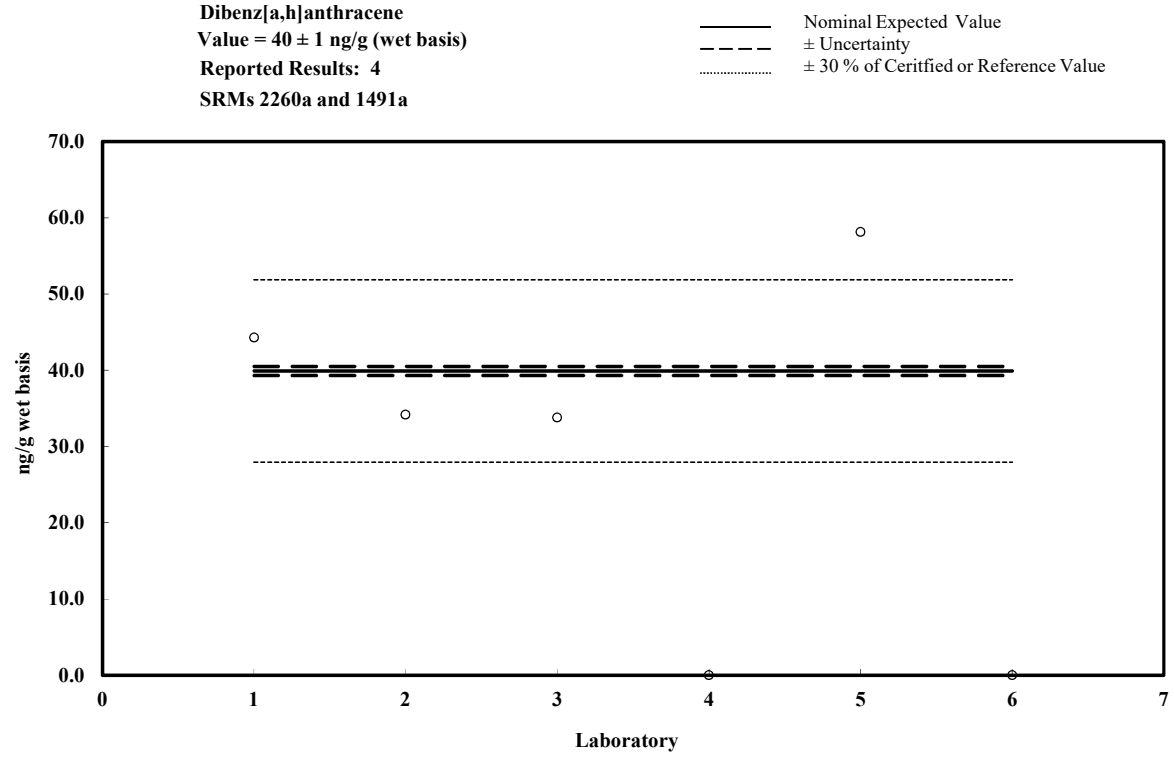
Nominal Expected Value
± Uncertainty
± 30 % of Ceritfied or Reference Value



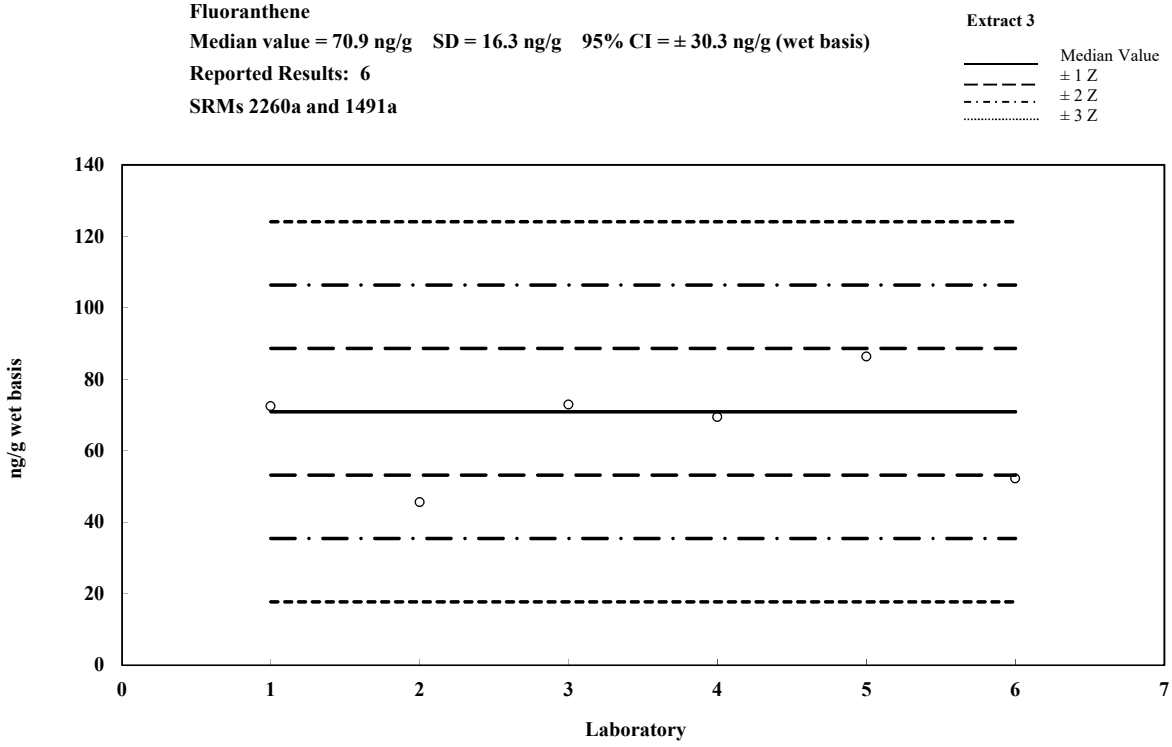
Dibenz[a,h]anthracene
Median value = 39.2 ng/g SD = 13.7 ng/g 95% CI = ± 25.5 ng/g (wet basis)
Reported Results: 4
SRMs 2260a and 1491a



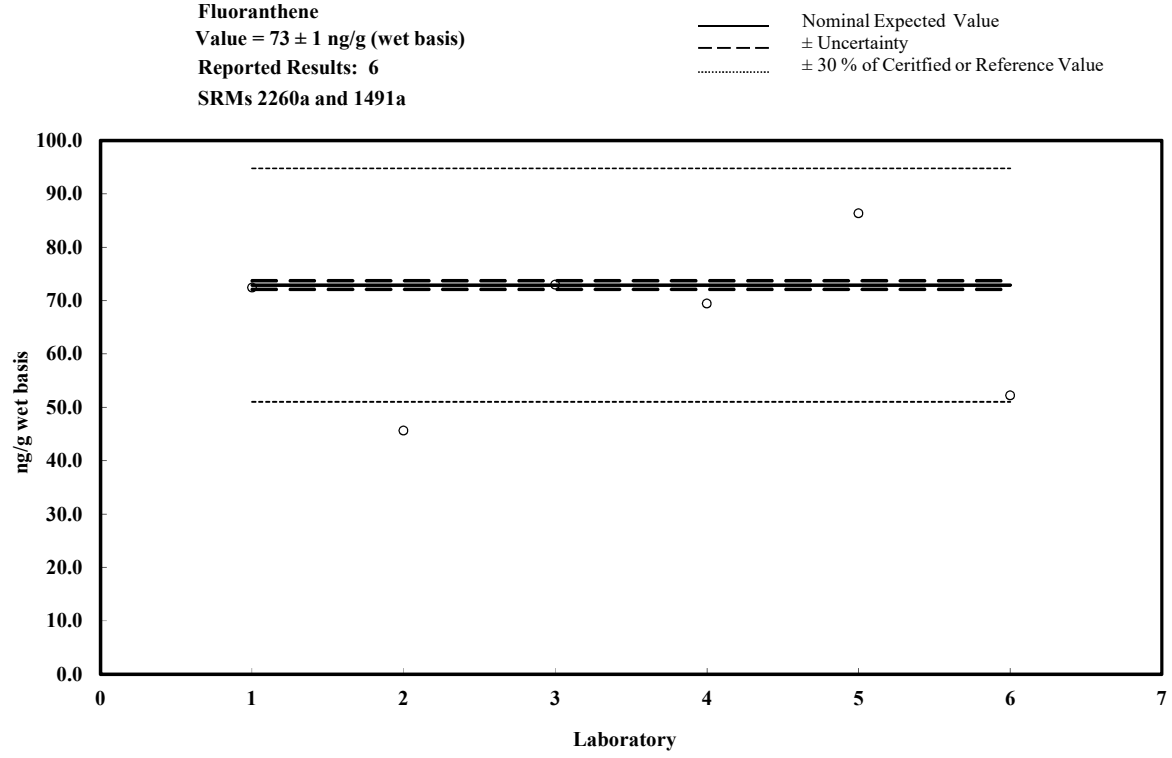
Dibenz[a,h]anthracene
Value = 40 ± 1 ng/g (wet basis)
Reported Results: 4
SRMs 2260a and 1491a



Fluoranthene
Median value = 70.9 ng/g SD = 16.3 ng/g 95% CI = ± 30.3 ng/g (wet basis)
Reported Results: 6
SRMs 2260a and 1491a



Fluoranthene
Value = 73 ± 1 ng/g (wet basis)
Reported Results: 6
SRMs 2260a and 1491a



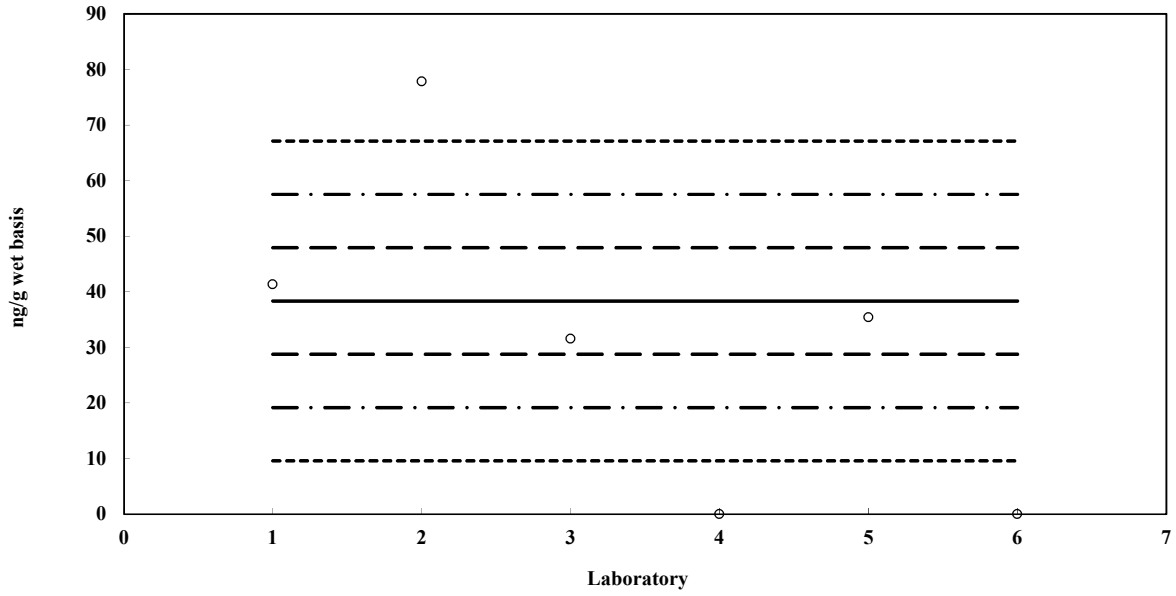
Indeno[1,2,3-cd]pyrene **Extract 3**

Median value = 38.4 ng/g SD = 16 ng/g 95% CI = ± 29.8 ng/g (wet basis)

Reported Results: 4

SRMs 2260a and 1491a

Median Value
± 1 Z
± 2 Z
± 3 Z



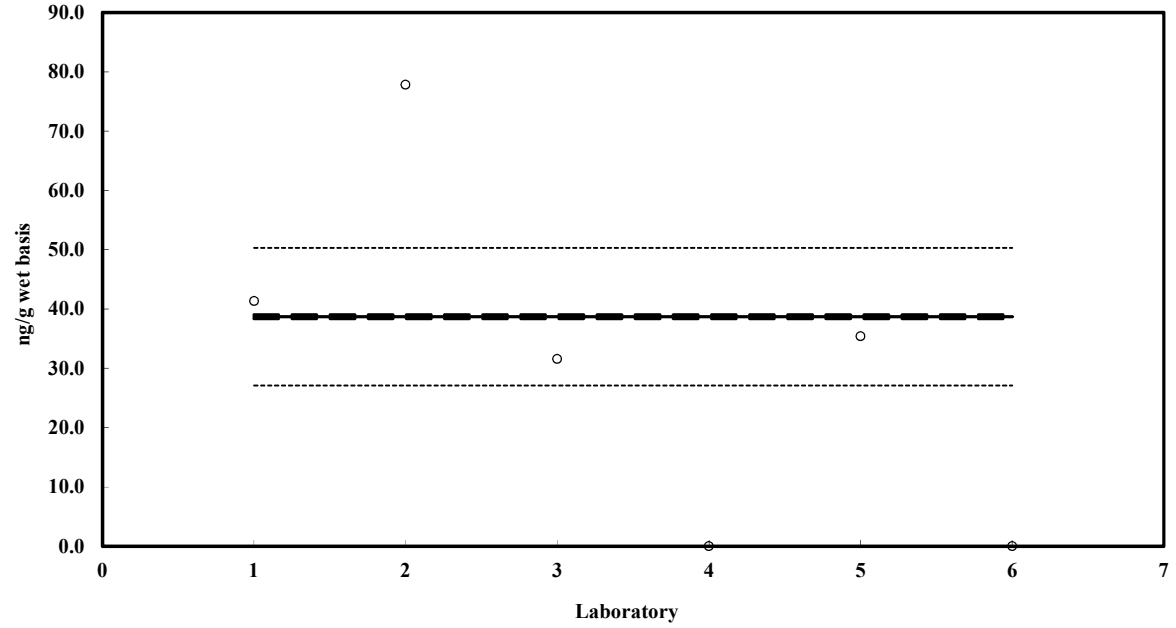
Indeno[1,2,3-cd]pyrene

Value = 39 ± 0 ng/g (wet basis)

Reported Results: 4

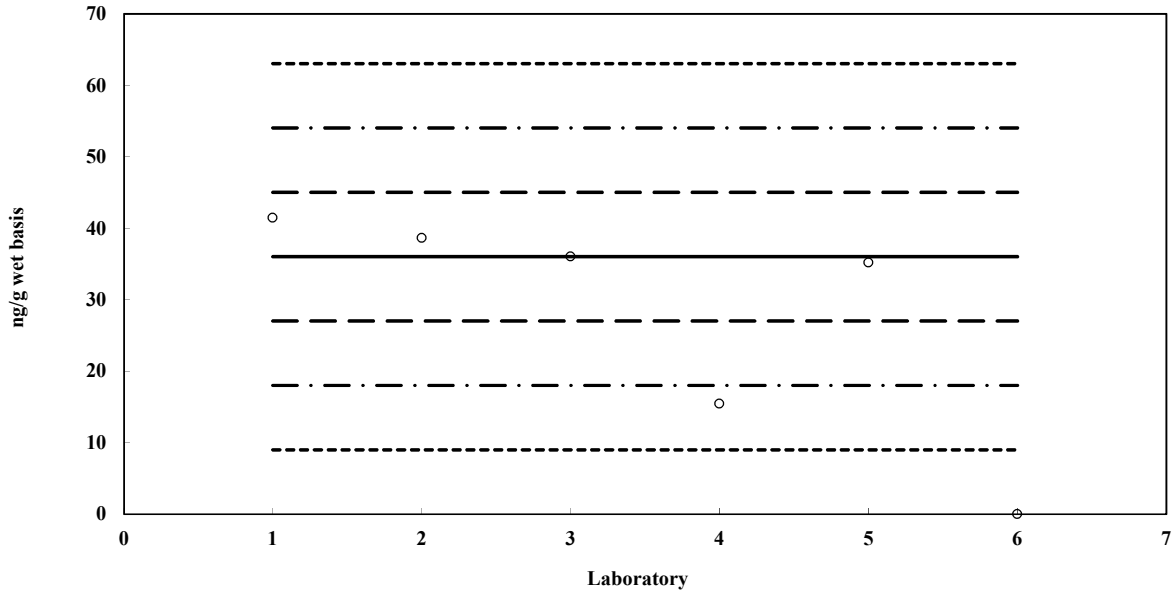
SRMs 2260a and 1491a

Nominal Expected Value
± Uncertainty
± 30 % of Ceritfied or Reference Value

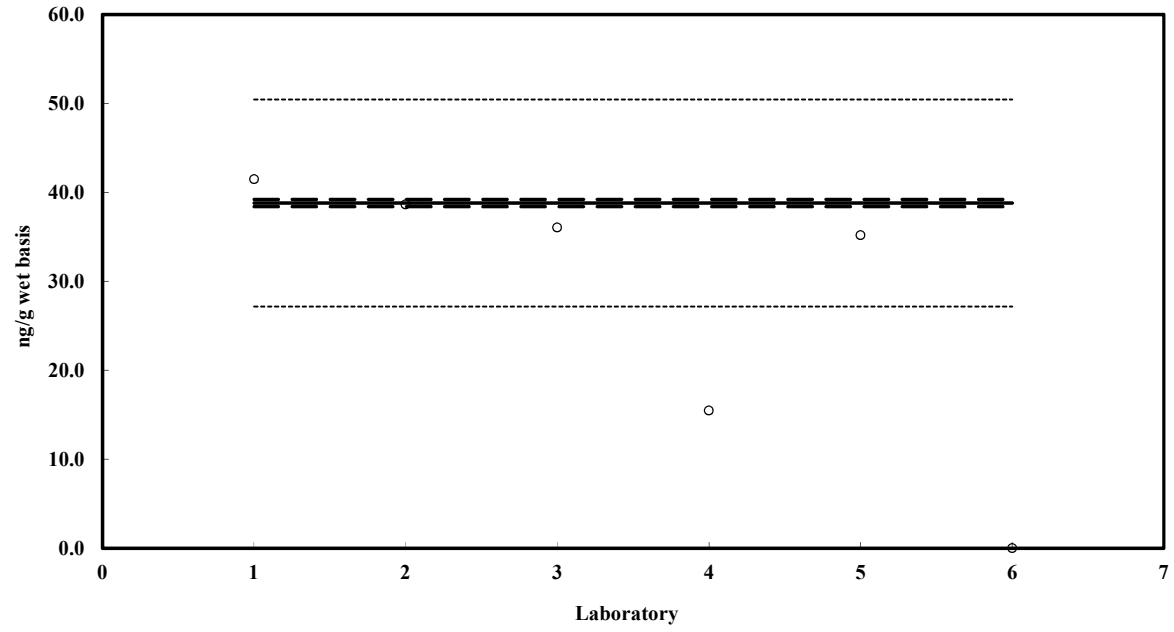


Perylene
Median value = 36 ng/g SD = 3.5 ng/g 95% CI = ± 6.5 ng/g (wet basis)
Reported Results: 5
SRMs 2260a and 1491a

Extract 3



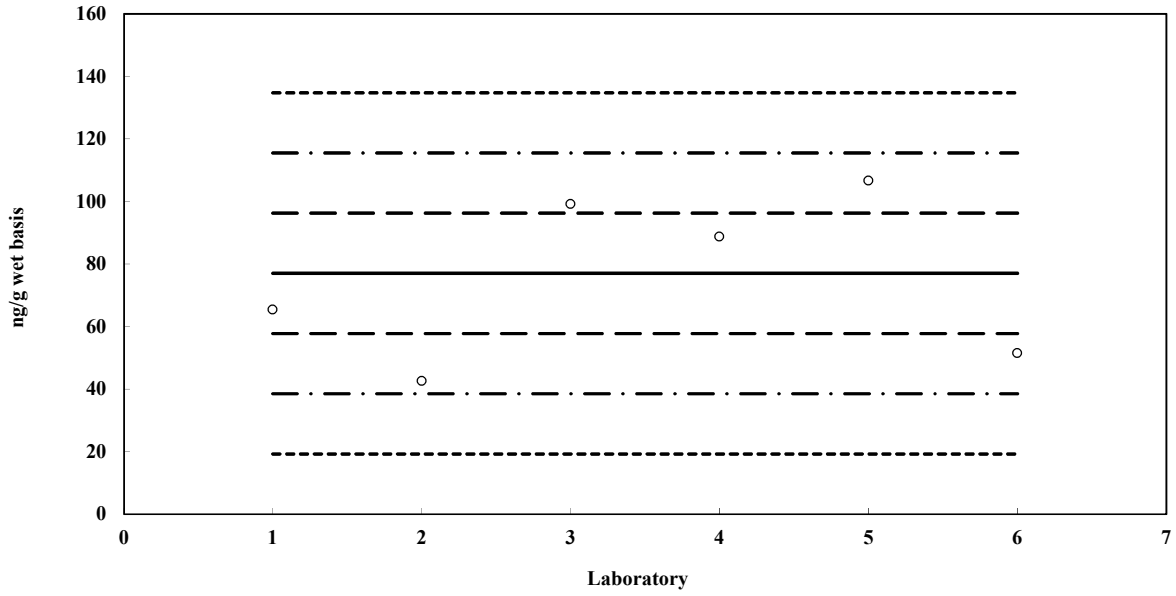
Perylene
Value = 39 ± 0 ng/g (wet basis)
Reported Results: 5
SRMs 2260a and 1491a



Phenanthrene
Median value = 77 ng/g SD = 41.7 ng/g 95% CI = ± 77.6 ng/g (wet basis)
Reported Results: 6
SRMs 2260a and 1491a

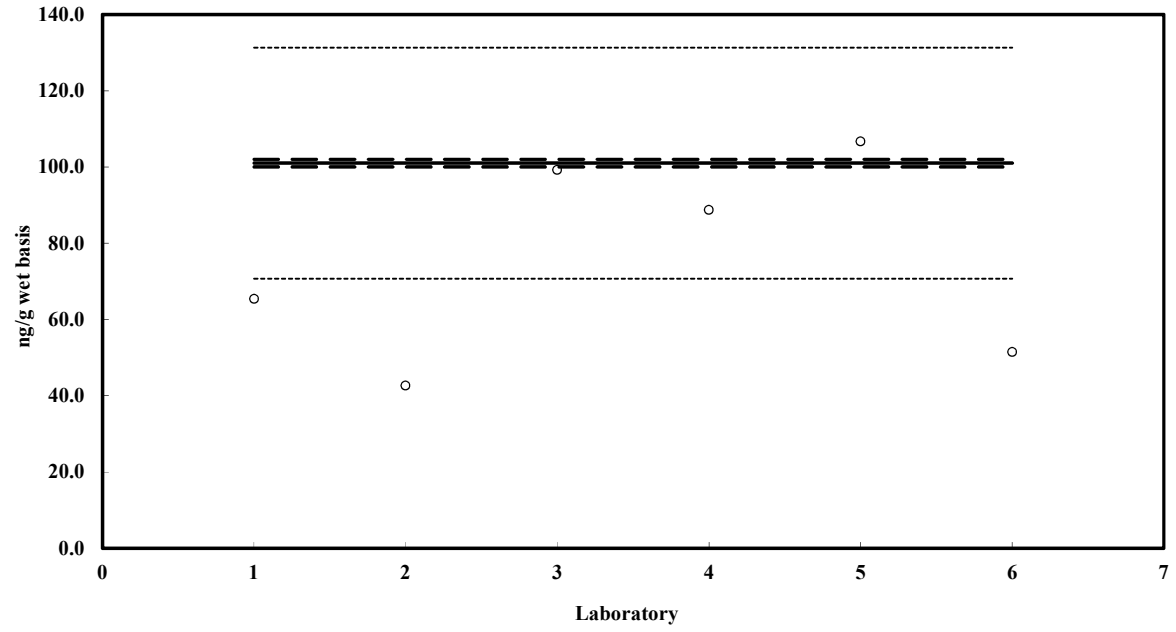
Extract 3

Median Value
± 1 Z
± 2 Z
± 3 Z



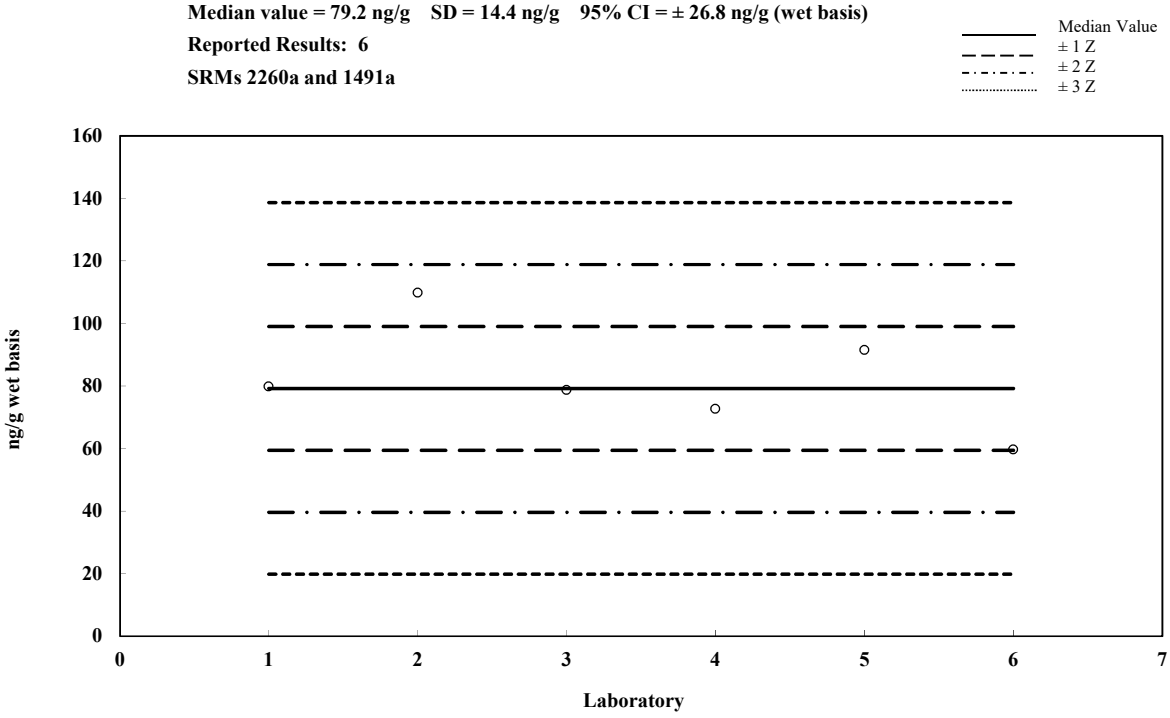
Phenanthrene
Value = 101 ± 1 ng/g (wet basis)
Reported Results: 6
SRMs 2260a and 1491a

Nominal Expected Value
± Uncertainty
± 30 % of Ceritfied or Reference Value

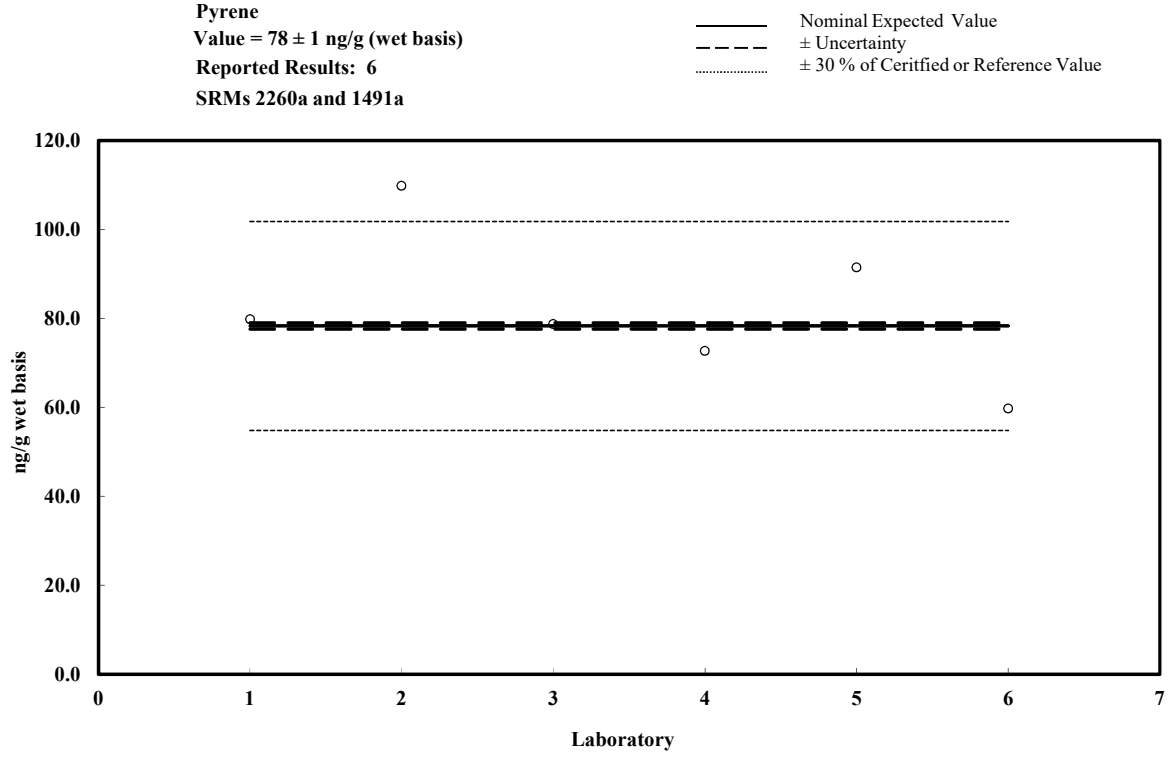


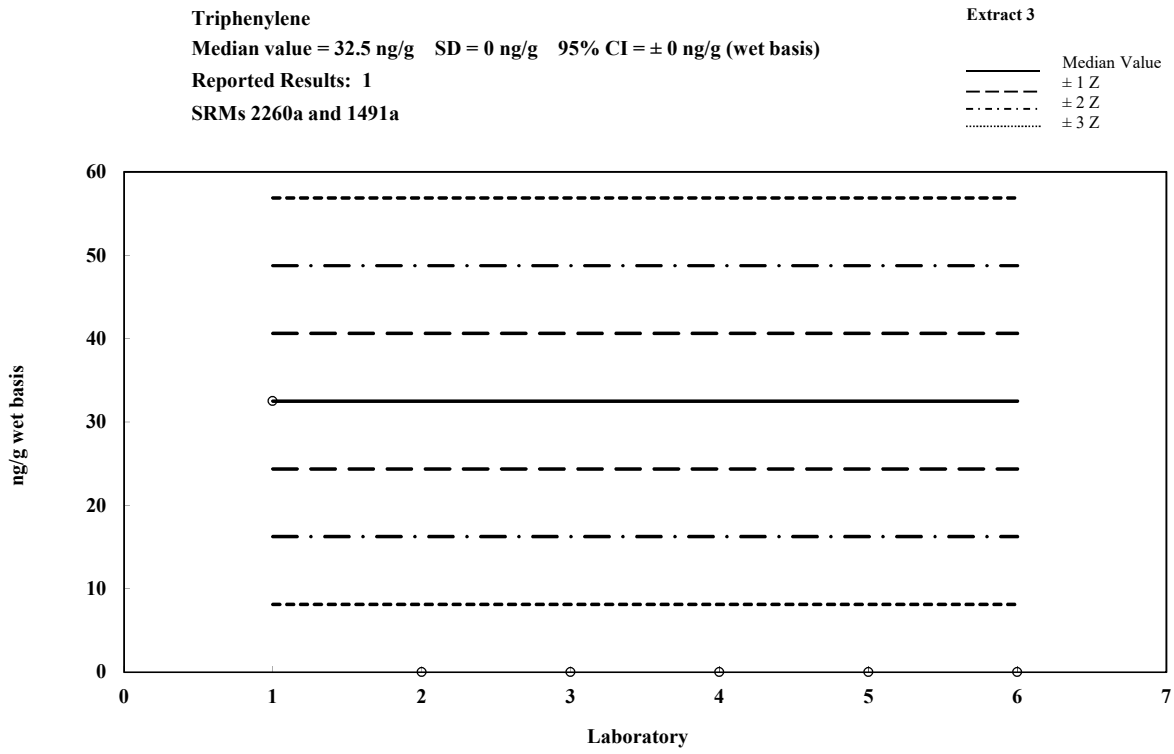
Pyrene
Median value = 79.2 ng/g SD = 14.4 ng/g 95% CI = ± 26.8 ng/g (wet basis)
Reported Results: 6
SRMs 2260a and 1491a

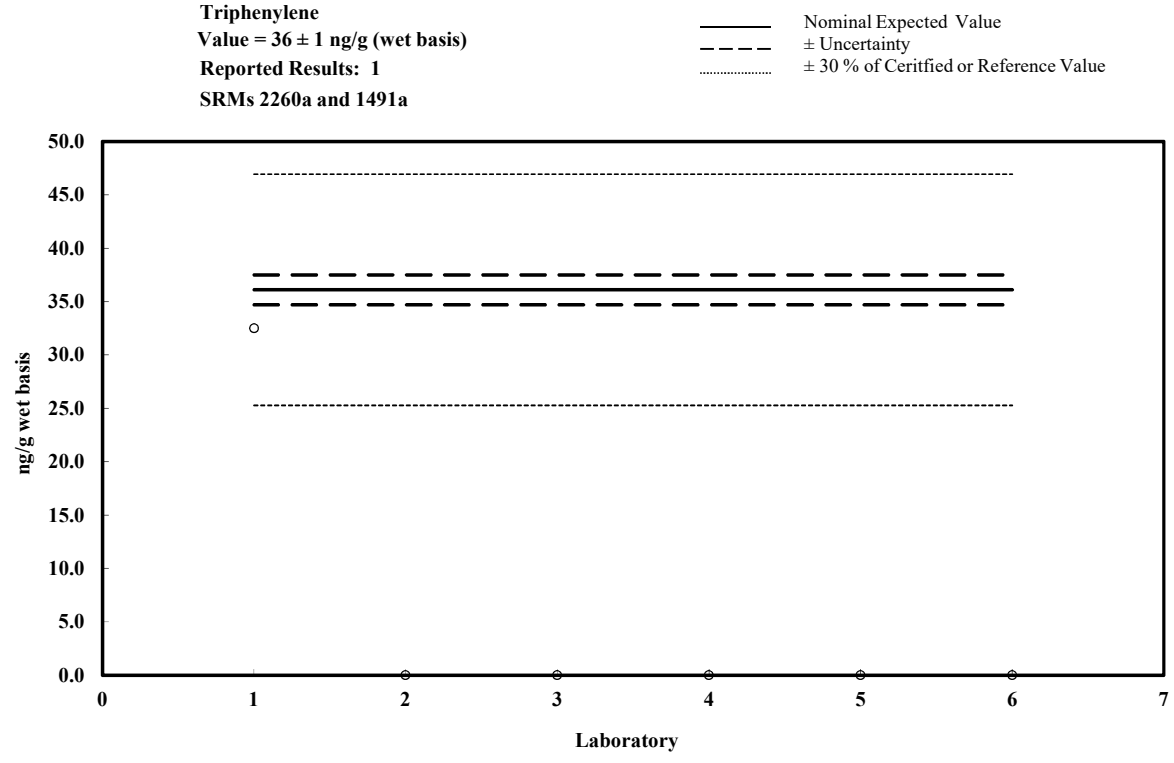
Extract 3



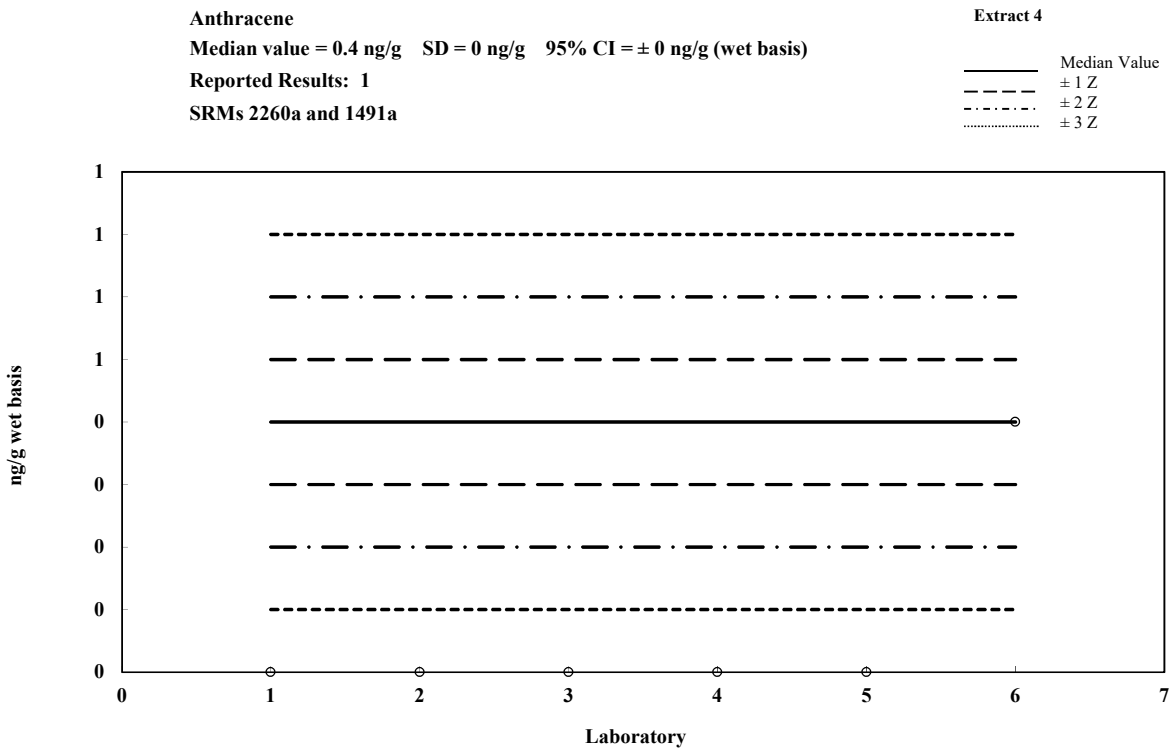
Pyrene
Value = 78 ± 1 ng/g (wet basis)
Reported Results: 6
SRMs 2260a and 1491a



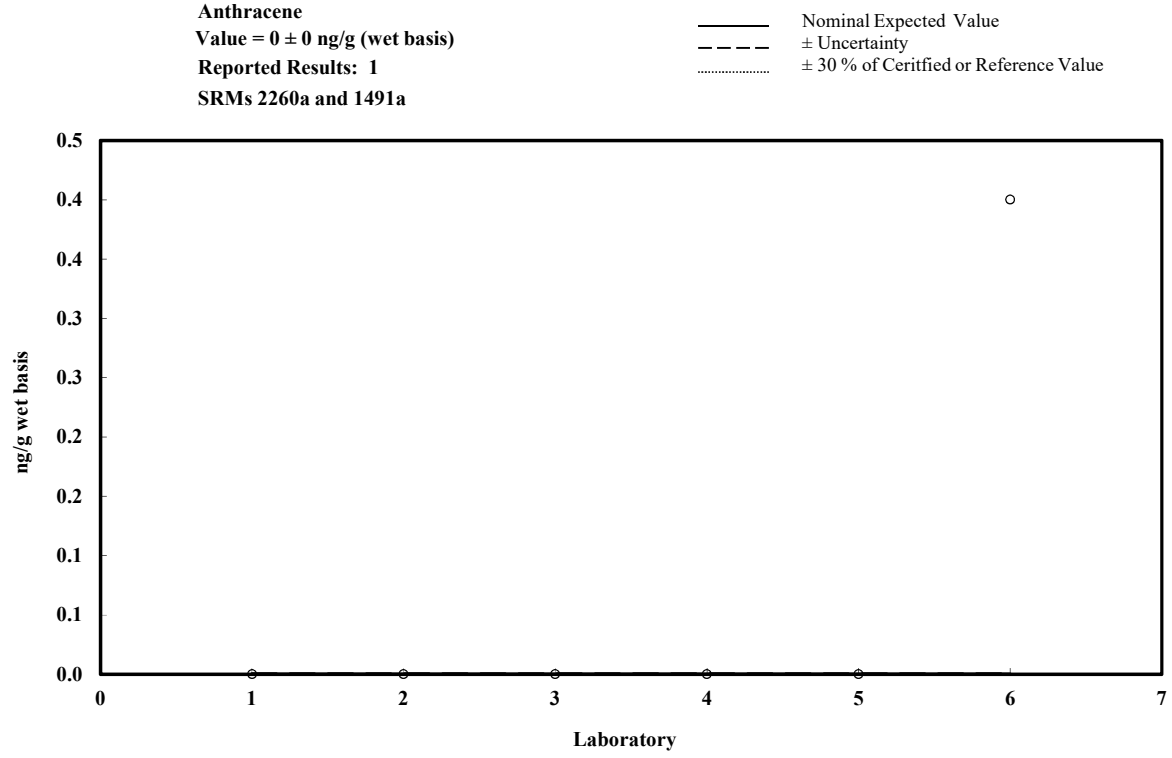




Anthracene
Median value = 0.4 ng/g SD = 0 ng/g 95% CI = ± 0 ng/g (wet basis)
Reported Results: 1
SRMs 2260a and 1491a



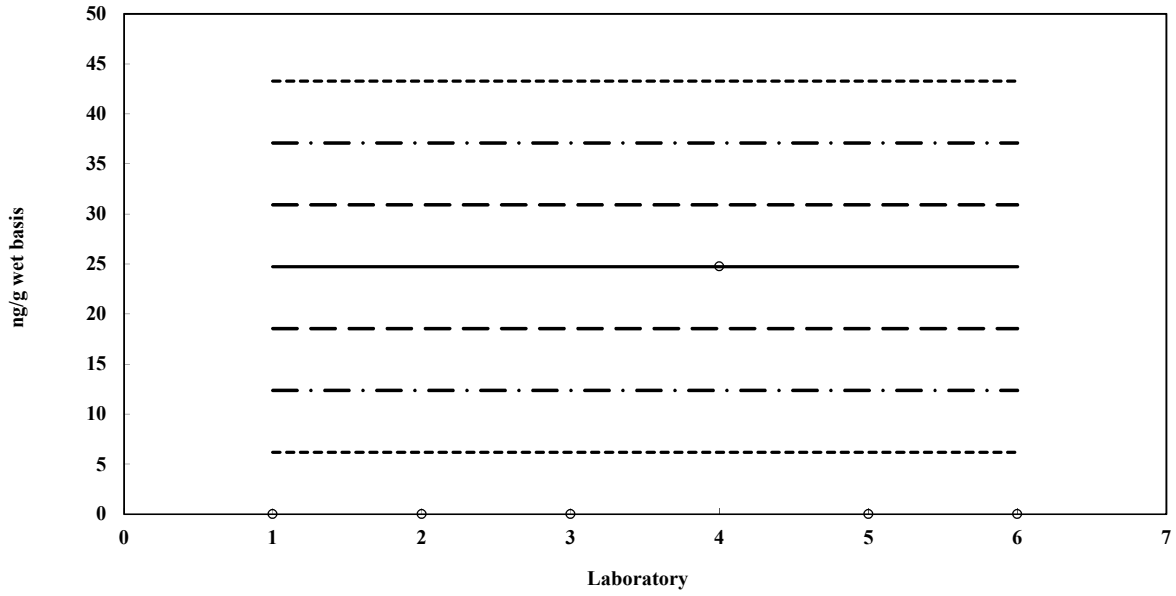
Anthracene
Value = 0 ± 0 ng/g (wet basis)
Reported Results: 1
SRMs 2260a and 1491a



Benzo[e]pyrene
Median value = 24.7 ng/g SD = 0 ng/g 95% CI = ± 0 ng/g (wet basis)
Reported Results: 1
SRMs 2260a and 1491a

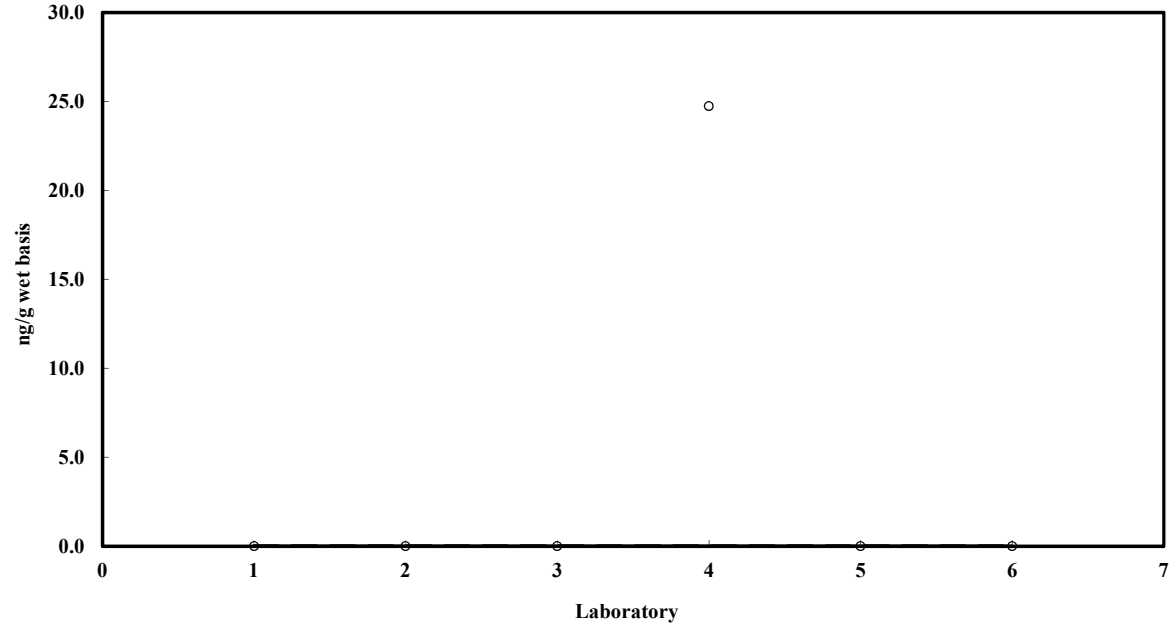
Extract 4

Median Value
± 1 Z
± 2 Z
± 3 Z



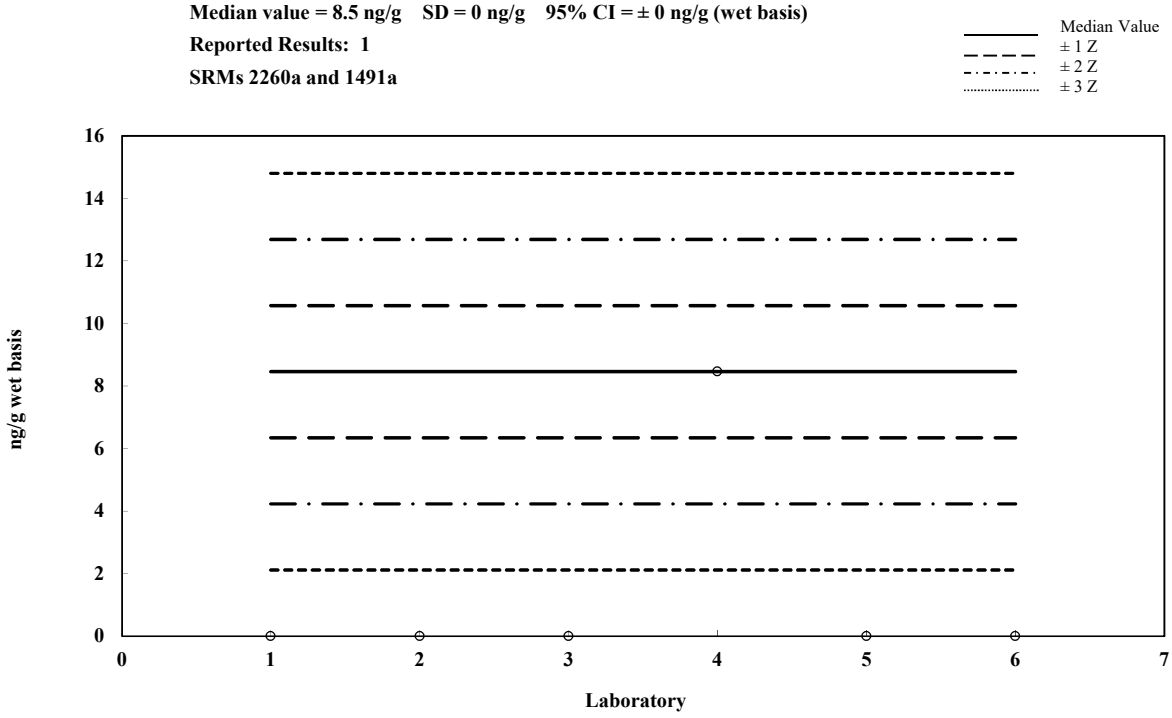
Benzo[e]pyrene
Value = 0 ± 0 ng/g (wet basis)
Reported Results: 1
SRMs 2260a and 1491a

Nominal Expected Value
± Uncertainty
± 30 % of Ceritfied or Reference Value

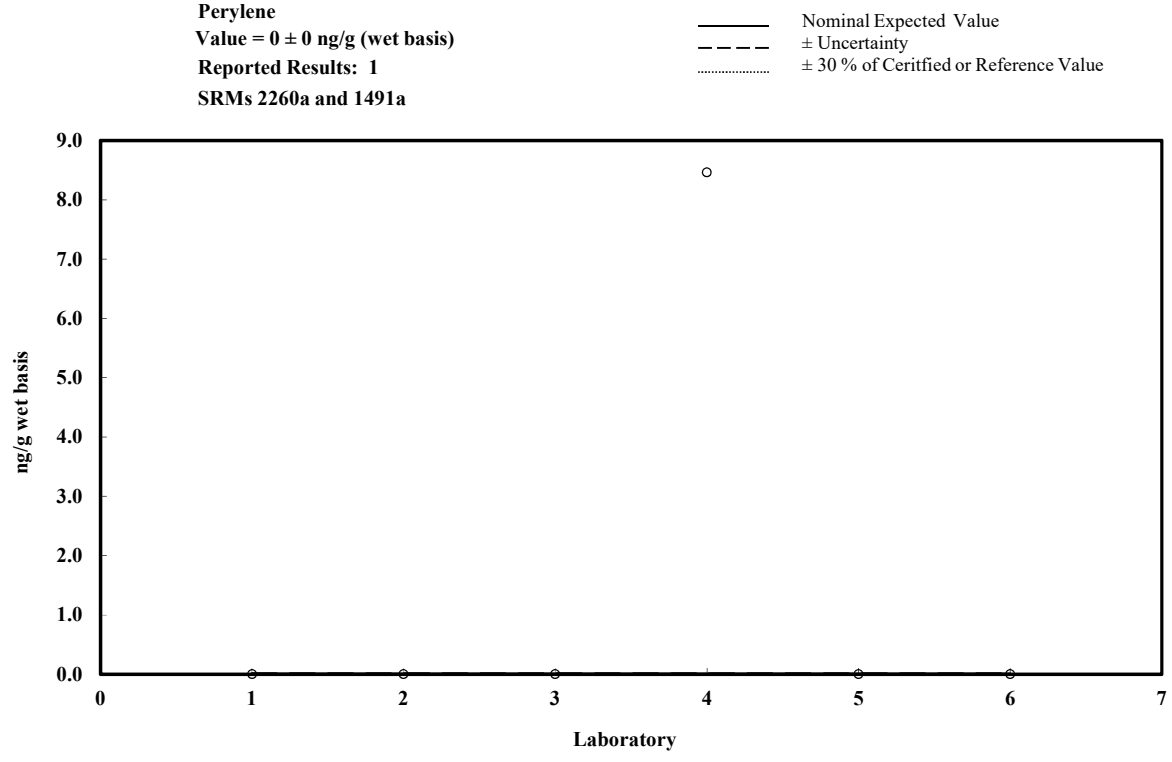


Perylene
Median value = 8.5 ng/g SD = 0 ng/g 95% CI = ± 0 ng/g (wet basis)
Reported Results: 1
SRMs 2260a and 1491a

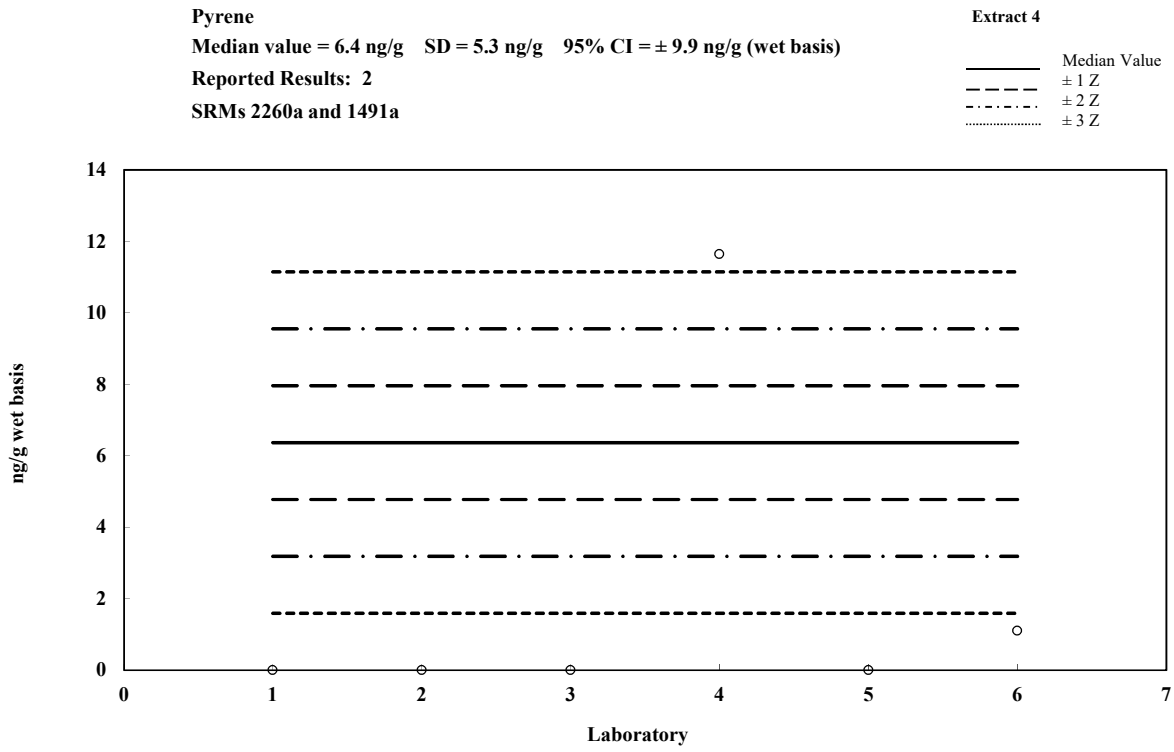
Extract 4



Perylene
Value = 0 ± 0 ng/g (wet basis)
Reported Results: 1
SRMs 2260a and 1491a



Pyrene
Median value = 6.4 ng/g SD = 5.3 ng/g 95% CI = ± 9.9 ng/g (wet basis)
Reported Results: 2
SRMs 2260a and 1491a



Pyrene
Value = 0 ± 0 ng/g (wet basis)
Reported Results: 2
SRMs 2260a and 1491a