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Description and Results of the 2005 NIST/NOAA Interlaboratory Comparison Exercise Program for Organic Contaminants in Marine Mammal Tissues

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U.S. Department of Commerce
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DISCLAIMER

Certain commercial equipment or instruments are identified in this report to specify adequately the experimental procedures. Such identification does not imply recommendations or endorsement by the National Institute of Standards and Technology nor does it imply that the equipment or instruments are the best available for the purpose.

ABSTRACT

The National Institute of Standards and Technology (NIST), in support of the National Oceanic and Atmospheric Administration's Marine Mammal Health and Stranding Response Program (NOAA/MMHSRP), conducts annual interlaboratory comparison exercises for the determination of chlorinated pesticides, polychlorinated biphenyl congeners (PCBs), and trace elements in marine mammal tissues. These exercises provide one mechanism for laboratories to evaluate their measurement quality and comparability for these constituents in marine mammal tissues. In the 2005 exercise, 4 and 17 laboratories participated in determining the concentrations of selected fatty acids and PCBs and organochlorine pesticides, respectively, in a homogenized blubber control material "Marine Mammal Quality Assurance Exercise Homogenate VII" (Homogenate VII) and Standard Reference Material (SRM) 1945 Organics in Whale Blubber. This report includes the results reported by the participating laboratories, combined consensus data results, and summary statistics for each analyte in the samples. The numerical indices used to assess laboratory performance are also discussed.

INTRODUCTION

Laboratories measuring organic contaminants in the marine environment must assess the accuracy and precision of their measurements. Quality control of measurements made on marine environmental samples is vital to the accurate assessment of marine pollution and its effects on wildlife and human health. NIST aims to improve the quality of analytical measurements of organic contaminants in marine and environmental matrices by developing improved analytical methods, producing NIST Standard Reference Materials (SRMs) and other control materials, and conducting annual interlaboratory comparison exercises.

Through the NIST National Marine Analytical Quality Assurance Program and with support from the NOAA Marine Mammal Health and Stranding Response Program (MMHSRP), NIST conducts interlaboratory comparison activities to include analyses of marine mammal tissues. The 2005 NIST/NOAA Interlaboratory Comparison Exercise Program for Organic Contaminants in Marine Mammal Tissues was modeled after previous exercises (e.g., Schantz et al., 1996; Schantz et al., 2002; Kucklick et al., 2002, Kucklick et al., 2006). The current exercise was designed to help laboratories assess data comparability and quality relative to other groups providing measurements of organochlorine contaminants in marine mammal tissues and to link these important measurements to a national metrology laboratory. The results of the exercises presented in this report should be useful for both assessing current methodology and reducing the variability of contaminant data reported on marine mammals. Future exercises will allow for the assessment of analytical data quality over time. This report summarizes the 2005 organic contaminant exercise including methods used for analysis, data reported by the laboratories on the intercomparison materials, and numerical indices used to assess laboratory performance. A report describing the 2005 trace element results of this exercise is also available (Christopher et al. 2006).

MATERIALS USED IN THE 2005 EXERCISE

The 2005 NIST/NOAA Interlaboratory Comparison Exercise for Organochlorines in Marine Mammal Tissues (2005 MMQA) used two materials sent out to 24 laboratories. Twenty-one data sets from 18 laboratories were submitted for this exercise (Table 1). Seventeen data sets were submitted for organohalogen compounds and four for fatty acid compounds. Participants were asked to make three measurements each on two materials: SRM 1945 "Organics in Whale Blubber" and MMQA-VII (Homogenate VII), the "unknown." The unknown material for the 2005 Organic Intercomparison Exercise was blubber from a male Blainville's beaked whale (*Mesoplodon densirostris*) that stranded in North Carolina, USA. The tissues were homogenized and blended (Zeisler *et al.,* 1983). At a NIST facility in Charleston, South Carolina, the blubber tissue was stored in a liquid nitrogen (LN_2) vapor phase freezer at –150 °C until prepared for this exercise. One bottle of this material containing approximately 10 g along with one bottle of SRM 1945 were sent either on dry ice or using a liquid nitrogen-cooled biological dry shipper via overnight express to each participating laboratory.

Exercise Requirements and Target Analytes

A suite of analytes was chosen for the exercise based on those tested in prior exercises (Schantz et al., 1996; Schantz et al., 2002; Kucklick et al., 2002; Kucklick et al., 2006) and several

additional analytes were included to broaden this list (Table 2a). In addition to the compounds listed in Table 2a, participants were requested to provide, if possible, values for polybrominated diphenyl ether (PBDE) congeners, coplanar polychlorinated biphenyl (PCB) congeners, total toxaphene and toxaphene congeners, chlorinated dioxins and furans and fatty acids (Table 2b). Laboratories were requested to make triplicate measurements of these compounds in each of the materials and to report their data using a data template provided by NIST. Results from the exercise were discussed during a workshop held in conjunction with the 2005 Society of Marine Mammology Biennial meeting in San Diego, California on December 13, 2005.

EVALUATION OF THE EXERCISE RESULTS

Determination of Laboratory Means and Consensus Values for Homogenate VII

Each laboratory reported the results of their analyses (Sample 1, Sample 2, and Sample 3) and the mean for each laboratory was calculated. The consensus value was calculated after comparing the results from an individual laboratory's analysis of SRM 1945 to the certified or reference value for the target organohalogens compounds given on the Certificate of Analysis for SRM 1945. If the value of a compound from a given laboratory for SRM 1945 was within 30 % of the reference value, the laboratory's result for the unknown sample was included in the calculation of the consensus value for the Homogenate VII. The median value for each compound was then calculated (Tables 3-5).

For the fatty acid portion of the exercise, the consensus value was not derived after screening the data as above. The 2005 exercise was only the second time participants were asked to measure fatty acids in SRM 1945 or an unknown; the first time was the 2003 exercise (Kucklick et al., 2006). The fatty acid consensus values for SRM 1945 from the 2003 exercise only consisted of results from three laboratories hence we did not fell it appropriate to use a consensus value based on such a small number of observations to screen data from the 2005 exercise.

Reported Results

Laboratories were assigned a random numerical identification code to shield the identity of the participants with the exception of NIST which was laboratories 1, 2, and 3. The same code was used for both materials. The results from the analysis of Homogenate VII and SRM 1945 are summarized in Tables 3 through 10. Appendix A shows the tabulated results from the individual laboratories for both materials and the results are shown graphically in Appendices B, C, and D. Appendix E gives the methods used for analysis by each laboratory and Appendix F shows data for additional analytes notes.

Table 1: Laboratories Participating in the 2005 NIST/NOAA Interlaboratory Comparison Exercise Program for Organic Contaminants.

Mississippi State Chemical Lab Mississippi State Mississippi State, Mississippi USA	Geochemical and Environmental Research Group Texas A&M University USA
California Animal Health and Food Safety Lab Toxicology UC Davis, School of Veterinary Medicine Davis, California USA	University of Pennsylvania Toxicology Laboratory Philadelphia, Pennsylvania USA
NOAA/National Ocean Service Hollings Marine Laboratory Charleston, South Carolina USA	Center for Marine Environmental studies, Ehime University Ehime, Japan Japan
NOAA/National Marine Fisheries Service Highlands, New Jersey USA	NOAA/National Marine Fisheries Service Northwest Fisheries Science Center Seattle, Washington USA (three data sets)
Laboratory of Environmental Toxicology The Norwegian School of Veterinary Science Oslo Norway	NIST Hollings Marine Laboratory Charleston, South Carolina USA
NOAA/National Ocean Service Center for Coastal Environmental Health and Biomolecular Research Charleston, South Carolina USA	NIST Gaithersburg, Maryland USA (two data sets)
Toxicological Center University of Antwerp (UA) Wilrijk Belgium	Mote Marine Laboratory Sarasota, Florida USA
Veterinary Medical Center Michigan State University East Lansing, Michigan USA	NOAA Auke Bay Laboratory Juneau, Alaska USA
Centre for Environment, Fisheries and Aquaculture Science CEFAS Burnham Laboratory Essex UK	Department of Biology University of Ottawa Ottawa, Ontario Canada

Table 2a: Target Analytes for the NIST/NOAA Interlaboratory Comparison Exercise Program for Organic Contaminants in Marine Mammal Tissues.

Pesticides	PCB Congeners	Substitution
2,4'-DDT	18	2,2',5
4,4'-DDT	28	2,4,4'
2,4'-DDE	31	2,4',5
4,4'-DDE	44	2,2',3,5'
2,4'-DDD	49	2,2',4,5'
4,4'-DDD	52	2,2',5,5'
HCB	66	2,3',4,4'
α-HCH	87	2,2',3,4,5'
γ-HCH	95	2,2',3,5',6l
β-HCH	99	2,2',4,4',5
heptachlor epoxide	101	2,2',4,5,5'
cis-chlordane	105	2,3,3',4,4'
trans-chlordane	118	2,3',4,4',5
oxychlordane	128	2,2',3,3',4,4'
cis-nonachlor	132	2,2',3,3',4,6'
trans-nonachlor	138	2,2',3,4,4',5'
dieldrin	149	2,2',3,4',5',6
Mirex	151	2,2',3,5,5',6
	153	2,2',4,4',5,5'
	156	2,3,3',4,4',5
	170	2,2',3,3',4,4',5
	180	2,2',3,4,4',5,5'
	183	2,2',3,4,4',5',6
	187	2,2',3,4',5,5',6
	194	2,2',3,3',4,4',5,5'
	195	2,2',3,3',4,4',5,6
	201	2,2',3,3',4,5,5',6'
	206	2,2',3,3',4,4',5,5',6
	209	2,2',3,3',4,4',5,5',6,6'

Table 2b: Optional Analytes for the NIST/NOAA Interlaboratory Comparison Exercise Program for Organic Contaminants in Marine Mammal Tissues.

Compound	Substitution	Compound	Substitution
Polybrominated Diphenyl Ethers		**Toxaphene**	
PBDE 47	2,2',4,4'	Total toxaphene	
PBDE 99	2,2',4,4',5	Toxaphene Congener 26	2-endo,3-exo,5-endo,6-exo,8,8,10,10-Octachlorobornane
PBDE 100	2,2',4,4,'6	Toxaphene Congener 50	2-endo,3-exo,5-endo,6-exo,8,8,9,10,10-Nonachlorobornane
PBDE 153	2,2',4,4',5,5'	Toxaphene Congener 62	2,2,5,5,8,9,9,10,10-Nonachlorobornane
PBDE 154	2,2',4,4',5,6'		
		Coplanar PCBs	
Fatty Acids		PCB 77	3,3',4,4'
Lauric acid	C12:0	PCB 126	3,3',4,4',5
Myristic acid	C14:0	PCB 169	3,3',4,4',5,5'
Pentadecanoic acid	C15:0		
		Chloro Dioxins and	
Palmitic acid	C16:0	**Furans**	
		Hexabromocyclododecane	
Margaric acid	C17:0	Isomers (α-, β-, and γ)	
Stearic acid	C18:0		
Arachidic acid	C20:0		
Palmitoleic acid	C16:1(n-7)		
Vaccenic acid	C18:1(n-7)		
Oleic acid	C18:1(n-9)		
Elaidic acid	C18:1(n-9)		
	C20:1(n-7)		
Gondoic	C20:1(n-9)		
Gadoleic acid	C:20:1(n-11)		
Erucic acid	C22:1(n-9)		
Cetoleic	C22:1(n-11)		
Nervonic acid	C24:1(n-9)		
Linoleic acid	C18:2(n-6)		
a-Linolenic acid	C18:3(n-3)		
g-linolenic acid	C18:3(n-6)		
Stearidonic acid	C18:4(n-3)		
Homo-gamma-linoleic acid	C20:2(n-6)		
Homo-alpha-linolenic acid	C20:3(n-3)		
Arachidonic acid	C20:4(n-6)		
EPA	C20:5(n-3)		
	C22:2(n-6)		
DPA	C22:5(n-3)		
DHA	C22:6(n-3)		

Assignment of z-and p-scores

Performance Scores: Different programs have different data quality needs. The acceptability of the results submitted by a laboratory will be decided by the individual program(s) for which the laboratory provides data. Typically, the program will use these exercise results in conjunction with the laboratory's performance in the analysis of certified reference materials and/or control materials, and of other quality assurance samples. These exercise results are shown in a number of ways in this report to aid in the evaluation of data quality.

IUPAC guidelines (IUPAC 1993) describe the use of "z-scores" and "p-scores" for assessment of accuracy and precision in interlaboratory comparison exercises, such as described in this report. These indices assess the difference between the result of the laboratory and the exercise assigned value, and can be used, with caution, to compare performance on different analytes and on different materials.

Accuracy Assessment (z-score):

$$z = \text{bias estimate} / \text{performance criterion}$$

or

$$z = (x - X)/\sigma$$

where x is the individual laboratory result, X is the "Exercise Assigned Value," and σ is the target value for the standard deviation. As described in the IUPAC guidelines, the choice of σ is dependent upon the data quality objective of a particular program. It can be fixed or determined by reference to validated methodology (*e.g.,* the calculated σ from the exercise data, see Tables 3 through 5). The fixed performance criterion is more useful in the comparison of a laboratory's performance on different materials, while the use of the actual variation may be more useful within a given exercise, for example, if the determination of a particular analyte is more problematic than usual.

The z-scores calculated using both approaches and applied to each laboratory's data are given in Appendix A. The same criterion was adopted for use in this exercise as was used in the former NIST/NOAA/NS&T program, where the target standard deviation was set to 25 % of the exercise assigned value. The z-scores for the Homogenate VII represent 25 % of the assigned value so that z = +1 is the assigned value plus 25 %, z = -1 is the assigned value minus 25 % and so forth. z-scores are also calculated based on the standard deviation of an analyte in the unknown material such that z = +1 is one "exercise standard deviation" higher than the assigned value and z = -1 is one "exercise standard deviation" lower than the assigned value and so forth. From a scientific point of view, IUPAC does not recommend the classification of z-scores, but does allow for such classification, *e.g.,*

$|z| \leq 2$ Satisfactory

$2 \leq |z| \leq 3$ Questionable

$|z| \geq 3$ Unsatisfactory

The tables in Appendix A summarize the results and performance indices including the number of analytes that fall within each category for each laboratory.

Precision Assessment (p-score):

$$p = \sigma_{lab} / \sigma_{target} \approx CV_{lab} / CV_{target}$$

where σ_{lab} and σ_{target} are variance estimates for the individual laboratory and the target variance, respectively. The CV_{lab} is the coefficient of variance (or ratio of standard deviation to the mean), while the CV_{target} is a target value chosen by the participants. During the workshop that accompanied this exercise, a target CV of 15 % was agreed upon, which is the same value used by other NIST run exercise programs (Schantz et al., 1996; Schantz et al., 2002; Kucklick et al., 2002; Kucklick et al., 2006). Note that the precision that p describes is that which occurs within a batch of analyses. Between-batch variance is likely larger and was not assessed in this exercise.

RESULTS AND DISCUSSION

Summarized results are shown in Tables 3 through 10 for the compounds listed in Tables 2a and 2b. Tabular results for individual laboratories are given in Appendix A, graphical results are given in Appendices B, C and D, and methods used by individual laboratories along with additional data and notes are given in Appendices E and F, respectively. Seventeen data sets were submitted for the organohalogen portion and four data sets were submitted for the fatty acid portion of the exercise. Fourteen data sets were submitted for pesticides, 12 for PCB congeners, and eight for the requested PBDE congeners. All except one laboratory had participated in the exercise before. For the 2003 exercise, 23 data sets were submitted for the organohalogen portion (PCB congeners and organochlorine pesticides) of the exercise and six data sets were submitted for the fatty acid portion (Kucklick et al., 2006).

The laboratories used a variety of methods to analyze the samples (Appendix E). For extraction, eight laboratories used pressurized fluid extraction, four used Soxhlet extraction, three used assisted extraction either with sonication or homogenization, two used liquid/liquid extraction, and one laboratory used column elution. Most laboratories (15) used internal standards, four laboratories used external standards, and one laboratory used a combination of internal and external standards. For detection, the most commonly used technique was gas chromatography-mass spectrometry (GC-MS; 10 laboratories), followed by GC-electron capture detection (GC-ECD; seven laboratories), GC-flame ionization detection (GC-FID; one lab for fatty acids), liquid chromatography diode array detection (one lab), and a combination of GC-MS and GC-ECD depending on compound class (one lab). All laboratories employing GC used capillary columns ranging in length form 25 m to 60 m. For organohalogen analysis, the most commonly used column was a 5 % phenyl methyl polysiloxane phase.

Based on the consensus values, Homogenate VII had considerably higher concentrations of total pesticides, PCBs, and PBDE congeners relative to SRM 1945 (Tables 3-8; Appendices B through D). The factor differences between Homogenate VII and SRM 1945 (i.e., Homogenate VII concentration/SRM 1945 concentration) were 9.7, 6.7, and 2.7 for the total organochlorine pesticides, total PCB congeners, and PBDE congeners, respectively, listed on Tables 3-8. The difference may be due to habitat, diet, or gender; SRM 1945 was collected from a mature female pilot whale while Homogenate VII was collected from a male Blainvilles's beaked whale. Concentrations of organohalogens are lower in female whales that have had calves relative to male or juvenile toothed whales (e.g., Tuerk et al. 2005).

The relative scatter of reported values seemed similar among compounds with some exceptions. The reported values for lipid (See Appendix C) were less than for the organohalogen compounds. PCB congener 201 showed somewhat of a bimodal distribution of values (Appendix A). For SRM 1945, the certified value for this compound is 16.8 ng/g ± 1.3 ng/g. However, four laboratories reported values averaging about 65 ng/g for this compound. It is likely that the laboratories were using the Zell and Ballschmiter nomenclature (Guitart et al., 1993) and are actually reporting the value for IUPAC congener 199 that has a reference value of 84.8 ng/g ± 2.2 ng/g in SRM 1945. For the 2005 exercise, eight laboratories submitted data for the PBDEs which is three more laboratories than in 2003 (Kucklick et al., 2006). While several laboratories generally had good agreement with the certified values for the PBDE congener values in SRM 1945, a few labs had difficulty consistently obtaining values within 25 % of the certified value indicating that PBDE congener determination is still challenging for some laboratories (Appendix D).

This is the second exercise in which fatty acid analysis was requested. The values for individual fatty acids in SRM 1945 were on average within 25 % of the median of the 2003 results. For comparison, the median values for PCB congeners and organochlorine pesticides in SRM 1945 for the 2005 exercise were on average within 9 % of the certified values. The median fatty acid results from the 2003 and 2005 exercises will be compiled by NIST and listed on the Certificate of Analysis for SRM 1945.

CONCLUSIONS

Twenty-one laboratories submitted data for the two exercise materials on a wide variety of organic constituents including PCBs, pesticides, fatty acids, and PBDE congeners. The repeated participation of many laboratories in the exercise suggests that the exercise has been a useful tool for assessing their analytical performance. A new exercise will be conducted in 2007 using SRM 1945 and a new unknown material. The exercise coordinators plan to vary the type of control material used to include other species of interest such as a delphinid. NIST is somewhat constrained on the choice of the material as a fairly large quantity (≈ 1 kg) is needed, and this amount of material is not available on a routine basis. SRM 1945 will continue to be the control material used in this exercise because it is the only marine mammal tissue available with certified and reference values for organochlorine compounds. During the meeting for participants held in conjunction with the Society of Marine Mammology biennial meeting, several laboratories indicated that a marine mammal plasma, serum, or whole blood should be

included as a matrix for the next exercise. NIST is attempting to obtain blood materials for this purpose. So far the attempts at obtaining a large enough pool of materials for the exercise have not been successful, however these attempts are continuing. It is likely that the blubber and blood exercises will not take place concurrently due to these difficulties.

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Table 3: Median organochlorine pesticide and lipid concentrations in Homogenate VII (Blainville's beaked whale; ng/g wet mass) reported by each laboratory. The values in bold were not used to derive the consensus value (see text for explanation).

Compound	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	Overall Median	1 SD	n	Consensus Median	1 SD	n
2,4'-DDT	572	1096	**1473**	**433**	333	295	523	**1011**		**2250**		**1573**			317		**1826**	792	670	12	428	304	6
4,4'-DDT	2624	2427	2511	2320	2343	1432	**3227**	3170	2076	**4023**		**4149**		**1112**	1553		**5021**	2469	1108	14	2343	534	9
2,4'-DDE	102.3	86.8	80.3	76.6		72.9	66.2	82.4							58.0			78.4	13.4	8	78.4	13	8
4,4'-DDE	8694	6265	**9927**	7660	8630	6520	7143	**10370**	6969	5507		7166		2538	5703	8720	**1055**	7143	2515	15	7056	1733	12
2,4'-DDD	111	73.5	84.9	96.6	125	**110**	116	198		**670**		75.0			47.7			110	175	11	97	43	9
4,4'-DDD	1502	471	464	1323	1473	1076		**2490**	1248	1650		1361		760	983	**1940**	**1286**	1305	544	14	1286	412	10
HCB	314	267	277	251	314	**288**	267	279	205			258			208		**181**	267	42	12	267	37	10
alpha-HCH				4.59				6.25				3.66			3.50			4.12	1.3	4.0	4.12	1.3	4
beta-HCH				3.27			13.8					5.79		7.03				6.41	4.5	4			
gamma-HCH							**17.0**	**5.61**				0.87			3.37			4.49	7.1	4	2.12	1.8	2
Heptachlor Epoxide		48.8	49.6	59.2		**63.8**	**51.1**							16.3	**69.7**		**89.4**	55.1	21	8	49.2	19	4
Cis-Chlordane	88.6	87.4	**93.4**	32.0		29.7	17.8	47.8				61.8		86.9	51.0		**94.9**	61.8	29	11	49.4	26	8
Trans-Chlordane	7.79	19.9		7.62		7.14	**16.8**	**41.2**		**1430**		23.5					**80.9**	19.9	469	9	7.79	7.9	5
Oxychlordane	275	350	321	265		240	252	232				275		155	237			258	53	10	258	53	10
Cis-Nonachlor	218	232	242	256		**198**		248				**672**			227	209		232	149	9	232	17	7
Trans-Nonachlor	1830	1888	1851	1410		1494	1360	1397				1737			1353	**116**		1452	515	10	1494	230	9
Dieldrin	164	175	208	210		**235**		303	**100**	**617**				75.0	128	**420**		208	158	11	175	72	7
Mirex	200	165	177	187		128		237.0		**363**		**234**			**155**		**190**	188	65.2	10	177	27	5
Lipid	80.8	79.7	93.5	70.5	87.7	81.7	88.3	73.6	82.0	79.6	78.0	89.3	86	77.4	84.8		86.2	81.8	6.1	16	81.8	6	16

Table 4: Median PCB congener concentrations in Homogenate VII (Blainville's beaked whale; ng/g wet mass) reported by each laboratory. The values in bold were not used to derive the consensus value (see text for explanation).

Congener(s)	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	Overall Median	1 SD	n	Consensus Median	1 SD	n
18	8.39	4.75	3.46			**8.40**	30.8		**32.0**						**4.2**		**6.84**	7.62	12	8	6.57	13	4
28	23.2	18.6	18.5	15.3			17.7	22.7	**19.3**			20.0	25.0		14.4		**5.6**	18.6	5.3	11	18.6	3.6	9
31		1.76		2.69			**3.0**					16.4			0.0		0.80	2.69	6.5	5	1.76	0.7	3
44	50.2	43.7	44.6	48.5		**47.7**	55.3	46.2	**49.3**						44.1		**6.3**	47.0	13.5	10	46.2	4.2	7
49	88.7	89.1	86.0	92.7		**96.7**	**98.8**	85.0	101						69.6		**25.8**	88.9	22.1	10	88.7	10	7
52	332	284	276	269		222	270	271	262			259.2	344		282		56	271	72	12	271	34	11
66/95*							**380**											380		1			
87		226	**220**	201				195									**40**	201	77	5	201	16	3
99	558	460	490	426		477	465	466				483	324		352		234	465	92	11	465	68	11
101 (+90)	843	715	**753**	609	**2953**			653	645.0			579	465		1070		**178**	653	729	11	649	185	8
105	89.2	95.8	95.7	83.0	90.3	76.1	**112**	84.2	87.0			104.9	92		177		**9.5**	90.3	35	13	90.3	27	11
118	536	695	704	670	813		636	**878**	610			583	553		645		**151**	640	179	12	640	82	10
128	129	148	157	155	144		**157**		173			152	144		171		**74**	152	27	11	150	14	10
132								**1907**							**127**		**29.5**	127	1057	3			0
138 (+163+164)	1902	1375	1388	1563	1377		1217	1273	1326			1507	1062		1670		**343**	1376	381	12	1377	230	11
149	833	743	761	741		859	697	605	636			653	793		720		**185**	731	175	12	741	80	11
151	330	251	277	261		262	277	210	242			294			254		**0**	261	32	10	261	32	10
153	**2496**		**2534**	1970	2207		1387		1831			1977	1703		1320		**542**	1900	600	10	1831	324	7
156	76.3	105	102	94.4	75.9	90.6	102	88.9	**92.7**			112	76.6		155			93.5	22	12	98.2	24	10
170 (+190)	307	286	301			291	281	306	334			305	257		248		**72**	291	70	11	296	25	10
180	908	830	909	834	890	800	827	915	890			1023	540		616		**222**	834	212	13	862	133	12
183	249	233	254	236		199	**259**	249	222			199	206		242		**82**	234	48	12	234	21	10
187	969	794	800	720		648	635	699	747			**478**	572		609		**252**	674	180	12	710	116	10
194	136	121	127	128		**149**	**161**	110	135			149	76.9		**162**		**30**	131	38	12	127	22	8
195	36.1	41.5	35.8	33.7		37.5	77.0	30.9							84		**9.67**	36.1	23	9	36.8	21	8
201	50.8	47.2	45.2	**276**		**276**	**272**	40.8							**292**		**94**	94.0	119	9	46.2	4.2	4
206	78.8	81.7	84.6	79.3		63.9	84.4	68.3				92.4			131		**27.0**	80.5	26	10	81.7	19	9
209	34.0	26.6	25.3	23.9		18.1	**35.0**	22.2				36.9			43		**9.5**	26.0	9.8	10	26.0	8.3	8
66	56.4	83.1	77.2	73.4		**87.3**		68.5	103			246			107		**55.0**	80.2	55	10	77.2	61	9
95	290	333	357	335		250							255		326			326	42	7	308	39	6

Table 5: Median PBDE congener concentrations in Homogenate VII (Blainville's Beaked whale; ng/g wet mass) reported by each laboratory. The values in bold were not used to derive the consensus value (see text for explanation).

Compound	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	Median	1 SD	n	Consensus Median	1 SD	n
PBDE 47	160			153		174	143				119	103	121		122			132	24	8	132	24	8
PBDE 99				26.3		**43.5**	26.6				21.7	19.2	22.2		**76.0**			26.3	20	7	22.2	3.2	5
PBDE 100	49.9			39.7		**44.8**	40.8				41.4	25.6	34.6		28.9			40.2	8.1	8	39.7	8.2	7
PBDE 153	17.1			15.4		**26.1**	16.1				13.1	12.6	12.6		**35.3**			15.8	8.1	8	14.2	2.0	6
PBDE 154	54.7			37.5		**61.6**	44.6				35.9	28.0	33.4		**17.6**			36.7	14	8	36.7	9.4	6

Table 6: Median organochlorine pesticide and lipid concentrations in SRM 1945 determined by each laboratory (ng/g wet mass and percent (mass fraction), respectively).

Compound	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	Median	1 SD	± 95% CI	n	Reference Value	Uncertainty	Comment
2,4'-DDT	97.7	94.9	134	57.5	85.4	85.3	101	130.7		220		178			71.0		266	99	64	36	12	90.9	13.5	certified value
4,4'-DDT	255	220	239	169	229	196	362	251	216	440		372		383	180		370	245	89	46	14	233	8.02	certified value
2,4'-DDE	16.8	12.5	11.1	14.2		17.4	14.3	14.2							17.3			14.2	2.3	1.6	8	14.2	1.39	certified value
4,4'-DDE	519	470	851	516	477	482	509	687	452	407		510		355	412	454	85.7	477	162	82	15	497	19.5	certified value
2,4'-DDD	22.0	17.9	21.2	18.5	24.7	53.2	25.1	21.7		93.3		18.0			19.7			21.7	23	14	11	19.55	1.18	certified value
4,4'-DDD	120	126	86.7	117	114	152		199	96.0	153		102		78.2	106	160	167	119	35	18	14	119.9	4.87	certified value
HCB	30.9	28.6	32.6	27.3	30.6	41.3	37.1	29.9	26.7			28.6			25.9		40.3	30.3	5.2	2.9	12	30.6	1.45	certified value
alpha-HCH	21.1			16.1				17.6				13.9		7.30	13.0		20.3	16.1	4.7	3.5	7	16.9	1.41	certified value
beta-HCH				5.02			11.2	10.4				2.0		5.03	6.33			5.68	3.5	2.8	6			
gamma-HCH	4.81			2.95			107	8.3				2.5			3.37		3.85	3.85	39	29	7	3.18	0.01	certified value
Heptachlor Epoxide		10.7	10.3	10.0		23.9	17.9	15.4						9.57	16.3		14.0	14.0	4.7	3.1	9	10.7	0.09	certified value
Cis-Chlordane	58.4	47.1	81.1	51.0		43.5	45.7	50.2				52.0		24.8	41.7		22.7	47.1	16	9.3	11	48.1	1.58	certified value
Trans-Chlordane	12.4	11.8	5.65	11.2		11.1	20.2	27.0		145		15.1			12.7		70.7	12.7	42	25	11	11.8	0.54	certified value
Oxychlordane	19.0	21.6	23.8	17.4		18.7	21.5	24.8				20.7		21.2	17.3			21.0	2.5	1.6	10	21.2	1.06	certified value
Cis-Nonachlor	36.3	48.0	55.0	50.7		62.5		53.0				115			52.7	46.0		52.7	22	15	9	45.8	3.29	certified value
Trans-Nonachlor	197	193	241	162		173	170	197				189			153	93.5		181	38	24	10	197.5	15.5	certified value
Dieldrin	52.7	53.4	52.5	47.6		67.0		58.8	33.5	103				52.0	41.3	81.5		52.7	19	12	11	50.1	4.05	certified value
Mirex	34.9	27.7	37.4	32.6		39.5		43.0		127		44.7			18.7		55.4	38.5	30	19	10	31.0	3.35	certified value
Lipid	71.2	72.2	72.0	61.9	52.4	68.8	74.1	54.0	73.3	63	69.3	76.5	75.2	64.9	72.3		75.0	71.6	6.6	3.2	16	71.9	1.27	reference value

Table 7: Median PCB congener concentrations in SRM 1945 determined by each laboratory (ng/g wet mass).

Congener	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	Median	1 SD	± 95% CI	n	Reference Value	Uncertainty	Comment
18	5.19	4.81	3.49			8.43	5.16	3.38							2.57		8.16	4.99	2.2	1.5	8	4.35	0.56	certified value
28	13.5	12.5	9.91	10.5			13.8	14.8				10.5	12.4		11.5		5.68	11.9	2.6	1.6	10	13.1	1.07	certified value
31	2.79	3.26	3.94	3.23			6.42					7.70	2.27		4.38		3.00	3.26	1.8	1.2	9	3.56	0.36	certified value
44	11.3	11.2	11.3	12.8		16.9	13.0	13.5							10.9		4.05	11.3	3.4	2.2	9	12.1	0.51	certified value
49	18.3	18.3	18.5	18.8		28.3	25.3	18.2	17.5						16.9		10.8	18.3	4.7	2.9	10	18.3	0.04	certified value
52	43.0	42.2	39.8	38.1		36.3	47.5	38.3	31.0			38.4	43.9		34.4		18.5	38.3	7.5	4.2	12	40.7	1.30	certified value
66/95*							64.0																	
87		18.8	28.2	26.0				22.8									12.6	22.8	6.2	5.4	5	20.6	2.55	certified value
99	65.0	56.5	68.2	56.8		69.7	63.8	58.8				67.3	44.0		58.6		49.2	58.8	8.1	4.8	11	58.5	5.17	certified value
101 (+90)	98.0	71.6	104.1	78.2	456			78.8	79.3			81.3	62.3		79.4		43.8	79.3	115	68	11	78.2	12.4	certified value
105	26.7	26.5	30.8	28.1	31.6	31.0	37.7	24.7	32.3			31.5	29.5		25.3		10.8	29.5	6.3	3.4	13	28.6	1.16	certified value
118	68.9	75.2	98.9	84.5	96.1		88.1	102.9	79.7			81.5	74.5		67.1		42.6	80.6	16	9.3	12	76.5	2.87	certified value
128	22.2	22.9	24.8	23.7	20.0		34.8	22.7	28.3			23.3	23.4		26.5		22.8	23.3	3.8	2.2	12	23.0	1.06	certified value
132								224.0							35.4		9.4	35.4	117	133	3	21.1	4.75	reference value
138 (+163+164)	182	143	166	189	144		165	137	156			170	133		173		84	161	28	16	12	146	12.9	certified value
149	103	95.0	99.2	84.1		98.3	95.5	70.1	80.7			69.6	95.7		74.1		39.6	89.6	18	10	12	89.0	6.94	certified value
151	32.3	26.8	32.5	27.8		31.7	32.2	21.1	26.7			32.5			26.2			27.8	9.5	5.6	11	28.6	1.33	certified value
153	300		334	249	279	0.0	201		215			245	211		181		109	230	64	40	10	228	9.75	certified value
156	10.2	11.0	14.0	12.3	8.02	15.8	14.6	10.9	16.0			13.4	10.2		12.6			12.4	2.5	1.4	12	11.4	0.95	certified value
170 (+190)	43.9	43.4	46.9			47.1	40.3	44.1	49.0			44.3	39.1		33.0		25.6	43.9	6.9	4.1	11	42.6	2.18	certified value
180	148	133	169	143	141	141	165	157	160			153	100		107		73.1	143	28	15	13	138.3	9.70	certified value
183	39.3	35.1	44.7	39.9		37.4	49	42.3	38.7			37.7	36.6		30.5		21.3	38.2	7.0	4.0	12	38.04	1.83	certified value
187	150	113	146	116		117	130	116	124			83.9	103		97.0		64.0	116	24	14	12	121.5	11.1	certified value
194	64.6	47.2	60.3	55.7		78.8	77.9	50.3	61.0			60.3	37.7		32.0		29.9	58.0	16	9.0	12	53.5	5.18	certified value
195	14.0	12.6	11.5	10.3		15.4	11.6	12.5							15.3		5.23	12.5	3.1	2.0	9	14.3	2.17	certified value
201	16.7	16.2	15.8	80.2		24.9	87.5	13.1							53.7		43.0	24.9	29	18.9	9	16.8	1.30	certified value
206	50.5	43.8	54.3	48.5		39.8	51.8	44.0				52.9			35.9		25.5	46.3	9.0	5.6	10	44.9	4.23	certified value
209	18.7	18.9	20.5	18.1		13.3	25.5	19.0				19.8			14.6		10.4	18.8	4.2	2.6	10	17.2	1.86	certified value
66	22.3	22.9	26.7	22.2		30.5		21.8	26.7			25.4			22.2		18.2	22.6	3.5	2.1	10	22.4	0.51	certified value
95	33.3	33.5	47.9	39.6		39.3		35.0					33.0		39.8			37.1	5.1	3.5	8	33.9	0.51	certified value

Table 8: Median PBDE congener concentrations in SRM 1945 determined by each laboratory (ng/g wet mass).

Congener	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	Median	1 SD	± 95% CI	n	Reference Value	Uncertainty	Comment
PBDE 47	39.7			37.0		51.5	40.9				35.5	37.8	36.4		32.3			37.4	5.7	4.0	8	39.6	0.18	certified value
PBDE 99				15.4		29.4	17.6				16.5	16.5	16.4		46.9			16.5	12	8.7	7	18.9	2.32	certified value
PBDE 100	12.0			8.44		16.4	9.51				10.3	9.24	9.10		9.39			9.45	2.6	1.8	8	10.3	1.13	certified value
PBDE 153	8.01			7.46		17.3	7.16				8.33	8.38	7.50		12.6			8.17	3.6	2.5	8	8.34	0.55	certified value
PBDE 154	10.9			11.3		30.8	12.3				12.4	13.0	10.9		8.26			11.8	7.0	4.9	8	13.3	1.73	certified value

Table 9: Results from the analysis of Homogenate VII (Blainville's beaked whale) for fatty acids. Values are in percent (mass fraction). Fatty acids not requested for the exercise are given in Appendix F.

Compound (Synonyms)			Laboratory					
			18	19	20	21	Median	1 SD
Lauric acid	Dodecanoic acid	C12:0	0.082	0.080	0.059	0.092	0.078	0.01
Myristic acid	Tetradecanoic acid	C14:0	0.72	1.51	1.19	1.72	1.28	0.44
Pentadecanoic acid	Pentadecanoic acid	C15:0	0.075	0.073	0.052	0.084	0.071	0.01
Palmitic acid	Hexadecanoic acid	C16:0	0.98	2.44	1.78	2.64	1.96	0.75
Margaric acid	Heptadecanoic acid	C17:0	0.058	0.050	0.043	0.071	0.055	0.01
Stearic acid	Octadecanoic acid	C18:0	0.65	0.71	0.48	0.89	0.68	0.17
Arachidic acid	Eicosanoic acid	C20:0	0.082	0.12	0.054	0.16	0.10	0.05
Palmitoleic acid	(Z)-9-Hexadecanoic acid	C16:1(n-7)	3.04	3.09	2.34	3.54	3.00	0.49
Vaccenic acid	(Z)-11-Octadecenoic acid	C18:1(n-7)	1.31	1.25	0.99	1.49	1.26	0.21
Oleic acid	(Z)-9-Octadecanoic acid	C18:1(n-9)	18.3	13.4	8.00	15.2	13.7	4.3
Elaidic acid	(E)-9-Octadecenoic acid	C18:1(n-9)						
	(Z)-13-eicosenoic acid	C20:1(n-7)		0.26	0.25	0.35	0.29	0.06
Gondoic	(Z)-11-eicosenoic acid	C20:1(n-9)	6.09	7.09	4.62	8.17	6.49	1.51
Gadoleic acid	(Z)-9-Eicosenoic acid	C:20:1(n-11)	5.91	5.04	3.32	5.69	4.99	1.17
Erucic acid	(Z)-13-Docosenoic acid	C22:1(n-9)	0.73	0.73	0.42	1.11	0.75	0.28
Cetoleic	(Z)-11-docosenoic acid	C22:1(n-11)	5.94	4.64	3.06	5.41	4.76	1.26
Nervonic acid	(Z)-15-Tetracosenoic acid	C24:1(n-9)	0.19	0.15	0.08	0.20	0.16	0.05
Linoleic acid	(Z,Z)-9,12-Octadecadienoic acid	C18:2(n-6)	0.47	0.41	0.03	0.54	0.36	0.23
a-Linolenic acid	(Z,Z,Z)-9,12,15-Octadecatrienoic acid	C18:3(n-3)	0.54	0.083		0.17	0.26	0.24
g-linolenic acid	(Z,Z,Z)-6,9,12-Octadecatetraenoic acid	C18:3(n-6)	0.011	0.010		0.022	0.014	0.01
Stearidonic acid	(Z,Z,Z,Z,)-6,9,12,15-Octadecatetraenoic acid	C18:4(n-3)	0.079	0.143		0.090	0.10	0.03
Homo-gamma-linoleic acid	(Z,Z)-11,14-Eicosadienoic acid	C20:2(n-6)	0.15	0.067		0.17	0.13	0.06
Homo-alpha-linolenic acid	(Z,Z,Z)-11,14,17-Eicosatrienoic acid	C20:3(n-3)		0.030		0.066	0.048	0.03
Arachidonic acid	(Z,Z,Z,Z)-5,8,11,14-Eicosatetraenoic acid	C20:4(n-6)	0.13	0.12	0.04	0.18	0.12	0.06
EPA	(Z,Z,Z,Z,Z)-5,8,11,14,17-Eicosapentaenoic acid	C20:5(n-3)	0.38	0.31	0.11	0.51	0.33	0.17
	(Z,Z)-13,16-Docosadienoic acid	C22:2(n-6)	0.031			0.033	0.032	
DPA	(Z,Z,Z,Z,Z)-7,10,13,16,19-Docosapentaenoic acid	C22:5(n-3)	0.29	0.20	0.08	0.47	0.26	0.17
DHA	(Z,Z,Z,Z,Z,Z)-4,7,10,13,16,19-Docosahexaenoic Acid	C22:6(n-3)	0.86	0.57	0.10	0.85	0.59	0.36

Table 10: Results from the analysis of SRM 1945 for fatty acids. Values are in percent (mass fraction). Fatty acids not requested for the exercise are given in Appendix F. 2003 values are the median of results from thee laboratories.

Compound (Synonyms)			Laboratory						2003 Values	
			18	19	20	21	Median	1 SD	Median	1 SD
Lauric acid	Dodecanoic acid	C12:0	0.19	0.15	0.13	0.19	0.16	0.03	0.20	0.09
Myristic acid	Tetradecanoic acid	C14:0	0.87	3.13	2.71	3.63	2.59	1.20	3.44	1.2
Pentadecanoic acid	Pentadecanoic acid	C15:0	0.25	0.30	0.23	0.36	0.28	0.06	0.36	0.08
Palmitic acid	Hexadecanoic acid	C16:0	1.22	7.51	5.96	7.41	5.52	2.96	7.13	3.5
Margaric acid	Heptadecanoic acid	C17:0	0.16	0.22	0.16	0.25	0.20	0.05	0.25	0.09
Stearic acid	Octadecanoic acid	C18:0	0.83	1.25	1.11	1.43	1.16	0.25	1.36	0.38
Arachidic acid	Eicosanoic acid	C20:0	0.069	0.10	0.075	0.14	0.10	0.03	0.11	0.02
Palmitoleic acid	(Z)-9-Hexadecanoic acid	C16:1(n-7)	5.08	5.75	4.50	6.46	5.45	0.85	6.47	1.8
Vaccenic acid	(Z)-11-Octadecenoic acid	C18:1(n-7)	1.59	1.80	1.61	2.00	1.75	0.19	2.04	0.7
Oleic acid	(Z)-9-Octadecanoic acid	C18:1(n-9)	17.8	13.9	10.9	15.2	14.4	2.83	16.7	2.5
Elaidic acid	(E)-9-Octadecenoic acid	C18:1(n-9)							0.21	0.08
	(Z)-13-eicosenoic acid	C20:1(n-7)		0.24	0.18	0.27	0.23	0.04		
Gondoic	(Z)-11-eicosenoic acid	C20:1(n-9)	4.24	4.67	3.54	5.27	4.43	0.73	5.04	1.9
Gadoleic acid	(Z)-9-Eicosenoic acid	C:20:1(n-11)	3.05	2.94	2.48	3.25	2.93	0.33	1.41	1.6
Erucic acid	(Z)-13-Docosenoic acid	C22:1(n-9)	0.47	0.51	0.44	0.80	0.56	0.17	0.70	0.20
Cetoleic	(Z)-11-docosenoic acid	C22:1(n-11)	5.14	4.61	3.32	5.17	4.56	0.86	4.12	
Nervonic acid	(Z)-15-Tetracosenoic acid	C24:1(n-9)	0.29	0.22	0.13	0.30	0.24	0.08	0.30	
Linoleic acid	(Z,Z)-9,12-Octadecadienoic acid	C18:2(n-6)	0.75	0.75	0.11	0.91	0.63	0.36	0.91	0.30
a-Linolenic acid	(Z,Z,Z)-9,12,15-Octadecatrienoic acid	C18:3(n-3)	0.35	0.32		0.40	0.36	0.04	0.62	0.21
g-linolenic acid	(Z,Z,Z)-6,9,12-Octadecatetraenoic acid	C18:3(n-6)	0.018	0.020		0.026	0.021	0.004	0.02	0.01
Stearidonic acid	(Z,Z,Z,Z,)-6,9,12,15-Octadecatetraenoic acid	C18:4(n-3)	0.21	0.23		0.20	0.21	0.02	0.18	
Homo-gamma-linoleic acid	(Z,Z)-11,14-Eicosadienoic acid	C20:2(n-6)	0.22	0.17		0.030	0.14	0.10	0.24	0.09
Homo-alpha-linolenic acid	(Z,Z,Z)-11,14,17-Eicosatrienoic acid	C20:3(n-3)	0.14	0.13		0.19	0.16	0.03	0.18	
Arachidonic acid	(Z,Z,Z,Z)-5,8,11,14-Eicosatetraenoic acid	C20:4(n-6)	0.23	0.22	0.08	0.31	0.21	0.10	0.30	
EPA	(Z,Z,Z,Z,Z)-5,8,11,14,17-Eicosapentaenoic acid	C20:5(n-3)	1.09	1.12	0.54	1.27	1.00	0.32	1.44	0.32
	(Z,Z)-13,16-Docosadienoic acid	C22:2(n-6)	0.02			0.04	0.029	0.01	0.018	0.01
DPA	(Z,Z,Z,Z,Z)-7,10,13,16,19-Docosapentaenoic acid	C22:5(n-3)	1.00	0.88	0.48	0.94	0.83	0.23	1.05	0.3
DHA	(Z,Z,Z,Z,Z,Z)-4,7,10,13,16,19-Docosahexaenoic Acid	C22:6(n-3)	3.34	3.70	0.88	4.33	3.06	1.51	4.42	1.5

Appendix A

Tabular results of PCB congener, pesticide, PBDE congener, and lipid data reported by all laboratories.

Appendix B

Graphical results of PCB congener and lipid data reported by all laboratories. The Z-scores for Homogenate VII represent 25 % of the assigned value so that z = +1 is the assigned value plus 25 %, z = -1 is the assigned value minus 25 % and so forth. Error bars are ± 1 standard deviation.

Appendix C

Graphical results of pesticide and lipid data reported by all laboratories. The Z-scores for Homogenate VII represent 25 % of the assigned value so that z = +1 is the assigned value plus 25 %, z = -1 is the assigned value minus 25 % and so forth. Error bars are ± 1 standard deviation.

Appendix D

Graphical results of PBDE congener data reported by all laboratories. The Z-scores for Homogenate VII represent 25 % of the assigned value so that z = +1 is the assigned value plus 25 %, z = -1 is the assigned value minus 25 % and so forth. Error bars are ± 1 standard deviation.

Appendix E

Tabular summary of methods used for analysis by each laboratory.

Appendix F

Additional analyte data and notes reported by individual laboratories.