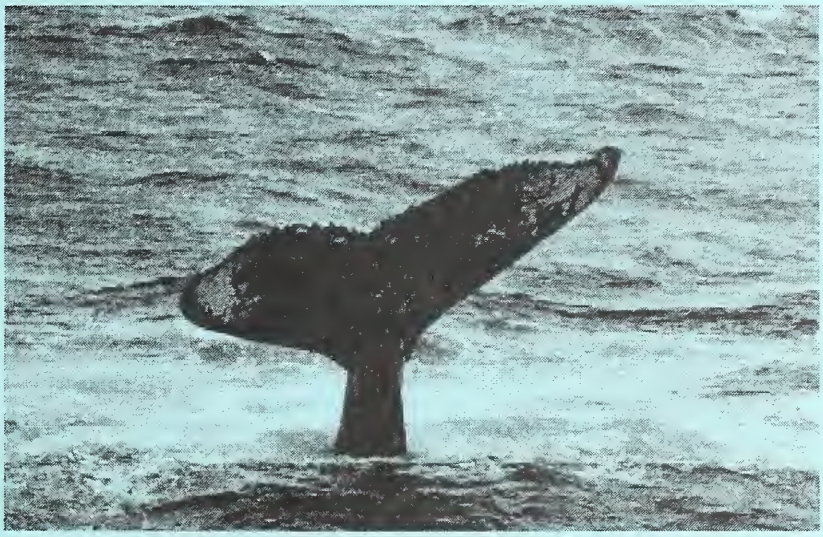
NISTIR 6279

NATIONAL MARINE MAMMAL TISSUE BANK AND QUALITY ASSURANCE PROGRAM:
Protocols, Inventory, and Analytical Results



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NATIONAL INSTITUTE OF STANDARDS
AND TECHNOLOGY
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CONTENTS

LIST OF TABLES

LIST OF FIGURES

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DISCLAIMER

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NATIONAL MARINE MAMMAL TISSUE BANK AND QUALITY ASSURANCE PROGRAM: Protocols, Inventory, and Analytical Results

INTRODUCTION

Over the last two decades, environmental specimen banking has gained international recognition as an important component of long-term environmental monitoring. Specimen banking enables future investigators to extend their research into the past, thereby increasing their time-line of study. Banking also provides specimens that may be used for future verification of analytical results, thus contributing to the quality assurance of environmental monitoring data. The U.S. National Biomonitoring Specimen Bank (NBSB) was developed by and is maintained at the National Institute of Standards and Technology (NIST), Gaithersburg, Maryland. This banking system is the direct result of a pilot Environmental Specimen Bank Program initiated in 1979 by NIST in cooperation with the U.S. Environmental Protection Agency (U.S. EPA) to determine the feasibility of long-term storage of environmental samples (Wise and Zeisler, 1984). The specimen type selected for this pilot program was human liver tissue. The NBSB currently provides for the long-term storage of well-documented and preserved specimens that represent several different types of environmental matrices (Wise and Koster, 1995).

The largest portion of the NBSB inventory comes from the marine ecosystem and a very important part of this inventory consists of tissue samples collected from marine mammals (Becker et al., 1997b). Tissues from marine mammals have been collected and banked in the NBSB since 1987 as a result of collaboration with several organizations, including the National Oceanic and Atmospheric Administration (NOAA), the U.S. Fish and Wildlife Service (USFWS), the U.S. Geological Survey Biological Resources Division (USGS-BRD), the New England Aquarium (Boston, Massachusetts), the Marine Mammal Center (Sausalito, California), the North Slope Borough Department of Wildlife Management (Barrow, Alaska), and the Natural Resources Division of Kawerak, Inc. (Nome, Alaska).

Major financial support for the archival of marine mammal specimens at the NBSB has been provided from two sources, the Alaska Marine Mammal Tissue Archival Project (AMMTAP) and the Marine Mammal Health and Stranding Response Program (MMHSRP). The AMMTAP began in 1987 as a collaboration between NIST and NOAA's National Ocean Service (NOS) with funding from the U.S. Department of the Interior, Minerals Management Service (MMS). The AMMTAP is now conducted by the U.S. Geological Survey's Biological Resources Division in cooperation with NOAA's National Marine Fisheries Service (NMFS) and NIST. The purpose of the AMMTAP is to collect and store for long time periods (decades) tissue samples collected from marine mammals in Alaska. Of primary interest are specimens from regions under consideration for offshore oil and gas development or coastal mining. The principle source of these specimens is from Alaska Native subsistence hunts, which allows for the collection of fresh specimens immediately following death of the animal. Detailed descriptions of this project have been published (Becker et al., 1988; 1993). NIST has also published reports describing collection and archival protocols, presenting inventory of specimens, and presenting and interpreting analytical results for Alaska marine mammals sampled

under AMMTAP (Becker et al., 1988; Becker et al., 1991; Becker et al., 1992; Becker et al., 1995b; Koster et al., 1994).

Through NMFS sponsorship, the National Marine Mammal Tissue Bank (NMMTB) began in 1987 as part of the NBSB for collecting and banking non-Alaska marine mammal tissue specimens. In 1992, the NMMTB was formally established by the Marine Mammal Health and Stranding Response Act (Public Law 102-587). In addition to specifically establishing the NMMTB, the Act places requirements on marine mammal stranding networks, reponse to unusual mortality events, and the establishment and maintenance of databases. NMFS implemented the Act by instituting the Marine Mammal Health and Stranding Response Program (MMHSRP). The MMHSRP includes stranding networks, response to unusual mortality events, information management, and biomonitoring. The biomonitoring component consists of real-time measurement of tissue contaminants, the NMMTB, and quality assurance. Although originally separate projects, NIST considers the NMMTB and the AMMTAP specimen bank as being essentially the same program conducted as part of the NBSB of marine mammal specimens.

In addition to the NMMTB, NIST administers and coordinates the quality assurance program for chemical analysis of marine mammal tissues as a component of the MMHSRP. Although the protocols and specimen inventory for Alaska marine mammal specimens collected by AMMTAP have been published in numerous reports, no comparable reports have been published for marine mammal specimens collected outside Alaska for the MMHSRP. The intent of this report is to describe the NMMTB and the quality assurance program, to present the protocols used by the NMMTB, to present the inventory of specimens for the non-Alaska animals, and to present analytical results from specimens analyzed as part of the routine NIST banking procedures and as part of the quality assurance program. Although the intent is to focus on the animals from outside Alaska, some interpretation of the analytical data requires reference to data previously reported by NIST for the AMMTAP. Future reports on the activities of NIST relative to the banking of marine mammal tissues and quality assurance will combine data and results for all of the U.S., including those collected in Alaska under AMMTAP.

NATIONAL MARINE MAMMAL TISSUE BANK (NMMTB)

BACKGROUND

Primarily as a result of the 1987-88 mass mortality of bottlenose dolphins (*Tursiops truncatus*) on the east coast of the U.S. and the lack of baseline data on the levels of anthropogenic contaminants in marine mammals of the U.S., the National Marine Mammal Tissue Bank (NMMTB) was established in 1989 at the NBSB in conjunction with the NMFS Office of Protected Resources. The protocols developed for collecting, preparing, and storing tissue samples for the NMMTB were based on protocols previously used for the AMMTAP. In the case of the NMMTB, however, sample sources were restricted to stranded animals and incidental catches of marine mammals during commercial fishing operations.

To evaluate the practical aspects of the collection protocols for both incidental catches and strandings, NIST initiated a demonstration project. As part of this project, collections were started in 1990 on the northeast coast of the U.S. in cooperation with scientists at the New England Aquarium (NEA), Boston, Massachusetts. The species sampled were harbor porpoise, *Phocoena phocoena*, from incidental catches, and pilot whales, *Globicephala melas*, from mass strandings. Two tissues (liver and blubber) were identified as the primary tissues to be banked (Becker et al. 1994).

The collection of samples from animals taken incidentally during commercial fishing operations is feasible due to the Fisheries Conservation and Management Act (FCMA), which requires observers to be on board all foreign fishing vessels operating in U.S. waters, and because of the 1988 Amendments to the Marine Mammal Protection Act (MMPA), which requires observers to be on U.S. fishing vessels. These observers, under the authority of NMFS, are instructed to identify animals suitable for providing tissues to the NMMTB.

During the demonstration project, potential animals for sampling were identified by the NOAA observers. These animals were each placed in an insulated bag cooled by pumped sea water. Upon arrival at port, the insulated bag was filled with ice to keep the animal cold until it reached the New England Aquarium. The observer called the New England Aquarium to arrange a pickup, and after verification of an acceptable animal, aquarium staff notified NIST staff, who then traveled to Boston within 24 hours to collect the samples.

Also during the demonstration phase, samples were collected from pilot whales when a mass stranding occurred at Hyannis, Massachusetts in December 1990. Again, once NEA staff had verified the animals were satisfactory for sampling, NIST staff were notified and immediately proceeded to the stranding site to collect samples.

Both of these sampling exercises provided good quality tissue samples and proved the feasibility of the protocol. Based on the results of this demonstration project, the collection of tissues was continued and expanded into other regions of the U.S. (Lillestolen et al., 1993; Becker et al., 1994).

Based on legislation enacted in 1992, the NMMTB program was expanded and directed toward facilitating the collection and dissemination of data on marine mammal health and health trends in marine mammal populations in the wild, correlating these trends with available data on physical, chemical, and biological environmental parameters, and coordinating effective responses to unusual marine mammal mortalities (Becker et al., 1994). This program, now known as the Marine Mammal Health and Stranding Response Program (MMHSRP), is coordinated by the NMFS in cooperation with the USFWS. Specimen banking is one component of this larger program (Becker et al., 1994). The MMHSRP is focused on animal health assessment, real-time contaminant monitoring, specimen banking, response to marine mammal strandings and mass mortalities, quality assurance/quality control of analytical results, and establishment and management of a nationwide database on the health of marine mammal populations in the U.S. Both the specimen banking (NMMTB) and quality assurance programs are administered by NIST.

GOAL OF THE NMMTB

In support of the MMHSRP, the goal of the NMMTB is to establish and maintain a resource of selected marine mammal tissues for the purpose of:

1. Providing samples for future retrospective analyses for new analytes of interest;
2. Providing samples for future analyses using improved analytical techniques; and
3. Providing a resource of samples that have been collected and stored in a systematic and well-documented manner for comparing results over time to identify whether environmental trends exist.

Sample collection and archival protocols were based on those developed by the AMMTAP for Alaska species (Becker et al., 1988; 1991). The development of criteria for selection of types of tissues, species, and individual animals to be sampled for the NMMTB are discussed below.

SAMPLE SELECTION CRITERIA

Tissues

In collaboration with the New England Aquarium, a protocol was developed to collect two primary tissues (liver and blubber) from a limited number of representative "indicator" species from strandings or incidental catches. As the program progressed, kidney tissue was included as a primary tissue.

Tissues selected for collection and archival in the NMMTB follow criteria that are based on the philosophy and approach developed for the NBSB (Wise & Zeisler, 1984):

1. A minimum of two 150 g samples can be obtained from the tissue (this amount is adequate to provide sufficient material to allow multiple analyses over a long time period);
2. The tissue will provide a homogeneous sample that is representative of the organ;
3. The sample is conducive to precise anatomical description (the location can easily be identified so all samples are taken from the same site);
4. The tissue is adaptable to sampling techniques (titanium and Teflon materials are used for handling the tissue to minimize contamination; additionally tissue contact is limited to clean dust-free surfaces);
5. The tissue has a potential for concentrating both inorganic and organic contaminants.

Based on these criteria, liver was selected at the "NMMTB Team of Scientists" planning meeting in Monterey, CA (December, 1989) as the primary tissue for collection and storage in the NMMTB. Liver is a major site for detoxifying chemical contaminants and has sufficient lipid content to make it a suitable accumulator of both organic and inorganic contaminants. The second tissue selected for inclusion in the NMMTB was blubber, which due to its high lipid content, concentrates organic contaminants to relatively high levels. Because of the role of kidneys in the bioaccumulation of some heavy metals (e.g., cadmium), kidney tissue was later included. As the program grows it is

anticipated that other tissues types may be selected for inclusion in the NMMTB.

When samples are taken for the NMMTB, additional samples of other tissues and fluids are collected, according to a "standardized general protocol" developed by the MMHSRP (Becker et al., 1994). Although these ancillary samples are retained by the facility of the collector, they are reported to the NMMTB to provide additional valuable information related to the samples held in long-term storage. Also prior to 1997, additional samples for the real-time contaminant monitoring component of the MMHSRP were collected from the animals sampled for the NMMTB. Now the samples collected for the specimen bank are cryogenically homogenized and subsamples routinely provided to the biomonitoring component for real-time analysis, therefore, expanding the baseline data on the banked specimens.

Species

The NMMTB protocol was originally designed for the collection of liver and blubber from a limited number of representative "indicator" species. Criteria for species selection were based on the following considerations (from Becker et al., 1994):

1. Available source of a large number of animals on a regular (ideally annually) basis;
2. Representatives of both coastal and pelagic species are included;
3. Representatives of both cetaceans and pinnipeds are included;
4. Potential for accumulating anthropogenic contaminations to relative high levels;
5. Consideration of the trophic position in the food web (bottom feeder, pelagic fish feeder, pelagic plankton feeder); and
6. Relative value as food or subsistence species for Native Americans.

The three sources of animals to be sampled for the tissue bank are animals that periodically strand on the coast (particularly those that mass strand), those that are taken incidentally during commercial fishing, and species that are taken during Native American subsistence hunts. At the 1989 Monterey meeting mentioned previously, two species of marine mammals were selected as target species for the "Demonstration Phase" of the NMMTB program implemented in the North Atlantic: pilot whales and harbor porpoises. In conjunction with the New England Aquarium, the harbor porpoise was the first species used to test the collection protocol. Regional indicator species identified at the Monterey meeting for specimen banking are listed in Table 1 (also see Becker et al., 1994).

Individual Animals

For the Demonstration Phase of the NMMTB, selection criteria had been developed, in conjunction with Greg Early of the New England Aquarium, for the identification of individuals of the preferred species that were suitable for sampling. Acceptable animals were identified on fishing vessels by NMFS observers using the following criteria:

1. The animal appears to be "normal" and "healthy";
2. The animal is not bloated;
3. There is no obvious scavenger damage;
4. The body cavity is intact;

Table 1. Indicator species for the National Marine Mammal Tissue Bank

Species	Region	Habitat	Source
Harbor Porpoise, *Phocoena phocoena*	Northeast U.S. Pacific U.S.	Coastal	Incidental
Dall Porpoise, *Phocoenoides dalli*	Pacific U.S.	Pelagic	Incidental
Bottlenose Dolphin, *Tursiops truncatus*	Southeast U.S. including Gulf of Mexico	Coastal	Strandings
Atlantic White-sided Dolphin, *Lagenorhynchus acutus*	Northeast U.S.	Coastal & Pelagic	Strandings
Beluga Whale, *Delphinapterus leucas*	Bering Sea and Arctic Ocean	Coastal	Subsistence
Pilot Whale, *Globicephala melas*	Northeast U.S. Southeast U.S. including Gulf of Mexico	Pelagic	Strandings
Bowhead Whale, *Balaena mysticetus*	Bering Sea and Arctic Ocean	Coastal	Subsistence
Harbor Seal, *Phoca vitulina*	Northeast U.S. Pacific U.S. including Gulf of Alaska	Coastal	Incidental Subsistence
Ringed Seal, *Phoca hispida*	Bering Sea and Arctic Ocean	Coastal	Subsistence
Northern Fur Seal, *Callorhinus ursinus*	Pacific U.S. including Gulf of Alaska	Pelagic	Subsistence
California Sea Lion, *Zalophus californianus*	Pacific U.S.	Coastal	Strandings Incidental[1]
Polar Bear, *Ursus maritimus*	Bering Sea and Arctic Ocean	Coastal & Pelagic	Subsistence

5. The animal is immediately put into the insulated body bag with circulating sea water to maintain the animal at sea water temperature or is otherwise maintained at cold temperatures without freezing;
6. The post-mortem time elapse is not more than 24 hours when the sample arrives at port.

The marine mammal was not frozen prior to sampling. Sampling of the animal occurred within 12 to 18 h after arrival in port. This time requirement was evaluated during the Demonstration Phase and found to be workable. Animals involved in mass strandings were evaluated using the same criteria. Since it was not possible to put the animal on ice and transport it to the New England Aquarium, sampling was carried out in the field and the protocol reflects the alternative procedures for animals sampled during strandings.

Specimen Access Policy

To establish control over the release of banked specimens for research purposes, formal access policies are required. For marine mammal specimens maintained by NIST in the NBSB the following items are required: (1) a justification for the use of banked specimens rather than real-time specimens, (2) demonstrated quality assurance procedures to assure the quality of analytical results, and (3) an agreement by the researcher to provide all analytical results to the NBSB for inclusion in the sample documentation. More details on access to specimens are found in Becker et al. (1991, 1994) and a copy of the NMMTB access policy is found in Appendix A of this volume.

CHEMICAL ANALYSIS QUALITY ASSURANCE PROGRAM

Within the MMHSRP, analytical results for chemical contaminants in marine mammals are generated by both the contaminant monitoring and specimen banking components. The contaminant monitoring component involves real-time analysis of samples, which includes analyses of samples collected specifically for real-time analysis and not banking, as well as subsamples of banked specimens. The contaminant monitoring component is conducted by NMFS's Northwest Fisheries Science Center (NWFSC) Environmental Conservation Division. To assess the accuracy and comparability of results among NWFSC, NIST, and other laboratories, NIST in collaboration with NMFS administers a quality assurance (QA) program for analytical measurements of contaminants in marine mammal tissues. This QA program, described by Wise (1993), consists of (1) preparation, analysis, and distribution of marine mammal tissue control materials; (2) interlaboratory comparison exercises among laboratories involved in marine mammal tissue analyses; and (3) development of Standard Reference Materials (SRMs) for use in the analysis of marine mammal tissues.

The QA program performs a major function in maintaining the quality of data resulting from the analysis of NMMTB specimens. Scientists requesting specimens from the bank for retrospective studies must demonstrate their analytical capabilities through appropriate QA activities, including participation in the NIST-administered QA program. In addition, NMFS requires that all researchers analyzing marine mammal tissues for contaminants under NMFS funding be participants in this program. This requirement ensures that the analytical results from marine mammal monitoring and research programs are of high quality and comparable to those generated in the monitoring and banking components of the MMHSRP.

METHODS

SPECIMEN COLLECTION PROTOCOLS

Two subsamples (samples A and B) are routinely collected for each of the tissue types for each animal sampled. These subsamples are ca. 150 g, wet mass, each. Standard protocols developed by NIST for the collection and archival of marine mammal tissues have been specifically designed to: (1) provide sufficient material (ca.300 g) for multiple analyses for different analytes; (2) minimize the possibility of sample loss by storing duplicate portions (subsamples A and B) in separate freezers; (3) control collection, processing, and storage procedures and equipment to minimize inadvertent contamination during sample handling and ensure sample integrity; (4) provide cryogenic storage conditions (-150 °C) to ensure sample stability over relatively long periods of time (i.e., decades); and (5) maintain a sample tracking system with all data resulting from sample analysis, sample collection history, and other data on the individual animals (e.g., necropsy reports), in order to maintain a complete database on the species sampled (Becker et al., 1988; 1991; 1994). A detailed description of the NMMTB sampling protocols and procedures are provided in Appendix B of this volume.

Archival Protocol

Specimen Log-in and Storage

Samples are received at NIST and transported to the NBSB facility. The shipping containers are unpacked and samples are inspected for any packaging problems and for unsuitable temperatures. Sample Data Forms (shown in Appendix C) and samples are compared to ensure that they correspond and that all information has been included. The samples are assigned a NBSB number and are permanently archived in the liquid nitrogen storage space.

When samples are placed in the permanent freezer, storage location is entered into the specimen storage form. The samples are placed in cylindrical cardboard tubes (6.0 cm diameter and 63.5 cm length); each tube will hold up to five samples. These samples will remain stored in the liquid nitrogen freezer at about -150 °C until they are requested for analysis. Information describing the animal is maintained, both in hard copy and in a computer database, as part of the documentation for each sample in the specimen bank.

The duplicate samples of each tissue (samples A and B) are stored in different liquid nitrogen freezers to provide additional security. Sample A is intended for long-term storage while sample B is available for any analyses as requested (refer to the Specimen Access Policy in Appendix A).

Specimen Preparation: Cryogenic Homogenization

Of the two subsamples of each tissue collected, material selected for analysis is taken from subsample B. This subsample, ca. 150 g, wet mass, is divided into aliquots using a cryogenic grinding and homogenization procedure specifically designed to maintain cryogenic conditions during the operation (Zeisler et al., 1983). This reduces the potential loss of volatile compounds and avoids degradation of the sample due to thawing and refreezing. The procedure provides a homogeneous wet frozen powder with greater than 90% of the particles less than 0.46 mm in diameter and with subsampling errors due to inhomogeneity estimated at less than 2% for trace elements (Langland et al., 1983).

Analytical Methods

Inorganic Analysis: Determination of Trace and Major Elements in Selected Tissues

Although trace element concentrations have been periodically determined for liver, kidney, and muscle, liver is the tissue that is routinely analyzed because it is the principle organ that can provide the best measurement for the largest number of elements. The NIST has relied on several analytical procedures involving collaboration between several investigators and organizations. The principle approach uses instrumental neutron activation analysis (INAA), a multi-element analytical technique that can provide data on a large number of trace elements using only a limited amount of sample. INAA is routinely used to measure 37 elements in the NMMTB specimens (Na, Mg, Al, Cl, K, Ca, Sc, V, Mn, Fe, Co, Cu, Zn, As Se, Br, Rb, Sr, Mo, Ag, Cd, Sn, Sb, I, Cs, Ba, La, Ce, Sm, Eu, Tb, Hf, Ta, Au, Hg, Th, and U). This method consists of exposing samples and standards to a neutron

field to produce radioactivity and measuring the energy and amount of the resulting radiation. Many of the stable nuclides of elements comprising the sample undergo neutron capture which, for many elements, results in the formation of radioactive product nuclides. The gamma ray emissions from the resulting nuclides are collected using a germanium detector. The energy of the gamma ray indicates from which element the product nuclide was formed and the amount of radiation emitted is proportional to the concentration of that element.

The INAA approach used by the program has been previously described (Becker et al., 1995a; Becker et al., 1995b; Mackey et al., 1995; Mackey et al., 1996). In preparation of homogenized subsamples for INAA, aliquots of the wet frozen powder are lyophilized at a pressure of 1 Pa for 5 days during which the temperature is gradually increased from -20 °C to 5 °C. The dried powder is weighed into two 200-mg aliquots and each aliquot is formed into a disk using a commercial stainless steel die and hydraulic press. The disks are packaged individually in acid-washed linear polyethylene (LPS) film. Since Hg analysis can not be performed on these packaged disks due to permeation of volatile Hg into the film during irradiation, two 100-mg aliquots of the powder are placed in acid-washed quartz vials. The vials are flash frozen in liquid nitrogen prior to sealing to avoid any evaporative losses of elemental Hg or Hg compounds.

For each analysis, aliquots of powdered SRMs are packaged in the same way and are included in the analysis scheme for the purpose of quality control. Standards consisting of known amounts of each element are deposited onto filter papers, which are formed into disks so that counting geometry is consistent between samples, controls, and standards. Analysis of SRM 1577a Bovine Liver and, beginning in 1991, a QA pilot whale liver tissue homogenate (Wise et al., 1993) is included with all multi-element INAA measurements. Analyses of SRM 2710 Montana Soil and SRM 1571 Orchard Leaves are included with all Hg measurements.

The irradiation and counting times for INAA are chosen to optimize the number of elements that can be determined and the detection limits for each. All irradiations are done at the NIST Reactor at a reactor power of 15 MW, which corresponds to a neutron fluence rate of ca. 2.0×10^{13} $cm^{-2}s^{-1}$. The samples, standards, and controls are subject to two irradiations, the first for 120 s and the second for 16 h. Approximately 90 s after the first irradiation, the samples are counted for 5 min to determine elements for which the product nuclides possess very short half-lives (Mg, Al, Cl, Ca, V, Cu, Br, and I). After several hours of decay, samples are counted again for 20 min to determine nuclides that possess half-lives on the order of a few hours (Na, K, Mn). Each sample and control material is then repackaged in clean LPS film and irradiated a second time with a decay time of ca. 6 days. Each sample is then counted for 4 h to determine concentrations of As, Mo, Cd, Sb, La, Sm, Au, and U. After a decay time of 1 to 2 months, samples are counted for 8 h to determine Sc, Fe, Co, Zn, Se, Rb, Sr, Ag, Sb, Cs, Ba, Ce, Eu, Tb, Hf, Ta, Hg, and Th. Quantitation is based on comparison with standards. The ratio of the amount of activity induced in the standard at the end of the irradiation per unit mass of element (A_o/g) is calculated and this value or standard constant is used to determine the amount of the element present in the sample based on the amount of activity measured in the sample. Spectral and data reduction for all samples were performed using a μVAX 3400 computer with Nuclear Data software.

Other analytical techniques have been used to provide data on elements that are not routinely measured by INAA (e.g., Pb and Ni) and to provide quality control data for selected elements (Cu, Zn, and Hg) by comparing data from two different analytical techniques. Ni and Pb were determined at the Institute of Applied Physical Chemistry, Research Center of Jülich, Germany, using differential pulse and square wave voltammetry and previously published procedures (Ostapczuk et al., 1986) after high pressure ashing digestion with nitric acid (Würfels et al., 1989). Hg concentrations were determined using cold vapor atomic absorption spectrometry (CVAAS) as described elsewhere (May and Stoeppler, 1984; Zeisler et al., 1993). Other techniques at NIST and collaborating institutions are used to provide additional data as needed.

Organic Analysis: Determination of Polychlorinated Biphenyls (PCBs) and Chlorinated Pesticides in Selected Tissues

Blubber is routinely analyzed for lipophilic compounds because it is the tissue that commonly has the highest concentration levels of these substances. Measurements for determining chlorinated hydrocarbons are commonly performed in the Analytical Chemistry Division at NIST. The NIST methodology has been described in detail elsewhere (Becker et al., 1995b; Schantz et al., 1993; Schantz et al., 1995). Briefly, the cryogenically homogenized blubber samples (2 to 3 g) were mixed with sodium sulfate and Soxhlet extracted using dichloromethane. The majority of lipids were removed by size exclusion chromatography (SEC) and then polychlorinated biphenyl (PCB) and pesticide fractions were isolated by normal-phase liquid chromatography (LC) on a semi-preparative-scale aminopropylsilane column. The fractions were analyzed by gas chromatography with electron capture detection (GC-ECD) using a 60 m x 0.25 mm i.d. 5% mole fraction phenyl methylpolysiloxane capillary column with helium as the carrier gas. The samples that have concentrations reported for both PCB 66 and PCB 95 were also analyzed by GC-ECD using 50 m x 0.25 mm i.d. 5% mole fraction phenyl methylpolysiloxane with 10% mole fraction methyl-C-18 (C-18) column.

RESULTS AND DISCUSSION

SPECIMENS ARCHIVED IN THE NMMTB

The inventory of marine mammal specimens in the NMMTB (as of December 31, 1997) collected by both the AMMTAP and the MMHSRP includes over 918 specimens collected from 332 animals representing 22 species (Table 2). Species sampling by region and collection rates are shown in Fig. 1. Although detailed inventories for the AMMTAP have been published (Becker et al., 1992; Koster et al., 1994), no such inventories have been published for animals sampled outside of Alaska. Summary and detailed inventories of the non-Alaska animals are therefore provided in this volume (Tables 3 and 4; Appendix D). Future inventories of marine mammal tissues published by the NBSB will combine all specimens, Alaskan and non-Alaskan.

Of the 918 marine mammal tissue specimens archived in the NMMTB, 234 were collected by the MMHSRP from 92 animals (12 species) outside of Alaska. These include tissue specimens from 60 individuals representing six species of cetaceans and 32 individuals representing six species of pinnipeds. Cetaceans include: fin whale (*Balaenoptera physalus*), pilot whale (*Globicephela melas* or *melaena*), harbor porpoise (*Phocoena phocoena*), common dolphin (*Delphinus delphis*), white-sided dolphin (*Lagenorhynchus acutus*), and rough-toothed dolphin (*Steno bredanensis*). Pinniped species include: ringed seal (*Phoca hispida*), harbor seal (*P. vitulina*), harp seal (*P. groenlandica*), grey seal (*Halichoerus grypus*), hooded seal (*Cystophora cristata*), and California sea lion (*Zalophus californianus*) .

The summaries of the NMMTB inventory in Tables 3 and 4 provide information on the status of the banked specimens. Table 3 indicates specimens for which Sample B is homogenized and ready for analysis, while Table 4 indicates which specimens have been analyzed by NIST. Less than half of the banked specimens have been analyzed for chlorinated hydrocarbons and inorganic constituents, i.e., the liver and blubber specimens from pilot whales, harbor porpoise, and white-sided dolphins. Baseline concentration values for as many as 37 trace elements have been measured in banked liver specimens from these three species and published (Mackey et al., 1995; Becker et al., 1997a; Becker et al., 1997b). Many of these elements are not analyzed routinely by conventional analytical techniques used in monitoring programs. In addition to these papers, the relationship between Hg, Se, and Ag in the liver of pilot whales has been discussed (Becker et al., 1995a), and the baseline concentrations of 33 PCB congeners, DDT compounds, *alpha*- and *gamma*-HCH, HCB, heptachlor epoxide, oxychlordane, *cis*-chlordane, *trans*-chlordane, *cis*-nonachlor, *trans*-nonachlor, and dieldrin in the banked blubber specimens of pilot whales, harbor porpoise, and white-sided dolphins were recently described in Becker et al. (1997b).

Table 2. Species, sampling region, and location and number of individual animals sampled for marine mammal specimens archived in the NMMTB as of December 31, 1997. These include animals sampled by both the AMMTAP and the MMHSRP

Species	Region	Location	Number of Individuals
Ceteceans:			
Bowhead whale (*Balaena mysticetus*)	Arctic	Alaska	52
Fin whale (*Balaenoptera physalus*)	North Atlantic	Massachusetts	1
Pilot whale (*Globicephala melas*)	North Atlantic	Massachusetts	12
Harbor porpoise (*Phocoena phocoena*)	North Atlantic	Massachusetts	12
	North Pacific	Washington	1
White-sided dolphin (*Lagenorhynchus acutus*)	North Atlantic	Massachusetts	10
Common dolphin (*Delphinus delphis*)	North Atlantic	Massachusetts	9
Rough-toothed dolphin (*Steno bredanensis*)	Gulf of Mexico	Florida	15
Beluga whale (*Delphinapterus leucas*)	Arctic	Alaska	24
	North Pacific	Alaska	21
Pinnipeds:			
Harbor seal (*Phoca vitulina*)	North Atlantic	Massachusetts	3
	North Pacific	Alaska	10
Ringed seal (*P. hispida*)	Arctic	Alaska	39
	Bering Sea	Alaska	20
	North Atlantic	Maine	1
Spotted seal (*P. largha*)	Bering Sea	Alaska	2
Harp seal (*P. groenlandica*)	North Atlantic	Massachusetts	4
Grey seal (*Halichoerus grypus*)	North Atlantic	Massachusetts	3
Hooded seal (*Cystophora cristata*)	North Atlantic	Massachusetts	3
Bearded seal (*Erignathus barbatus*)	Arctic	Alaska	3
	Bering Sea	Alaska	7
Northern elephant seal (*Mirounga angustirostris*)	North Pacific	Alaska	1
Steller sea lion (*Eumetopias jubatus*)	North Pacific	Alaska	3
	Bering Sea	Alaska	1
Northern fur seal (*Callorhinus ursinus*)	North Pacific/Bering Sea	Alaska	21
California sea lion (*Zalophus californianus*)	North Pacific	California	18
Pacific walrus (*Odobenus rosmarus rosmarus*)	Bering Sea	Alaska	20
Fissipeds:			
Polar bear (*Ursus maritimus*)	Arctic	Alaska	25
	Bering Sea	Alaska	3
Sea otter (*Enhydra lutris*)	North Pacific	Alaska	3
TOTAL NUMBER OF ANIMALS			332

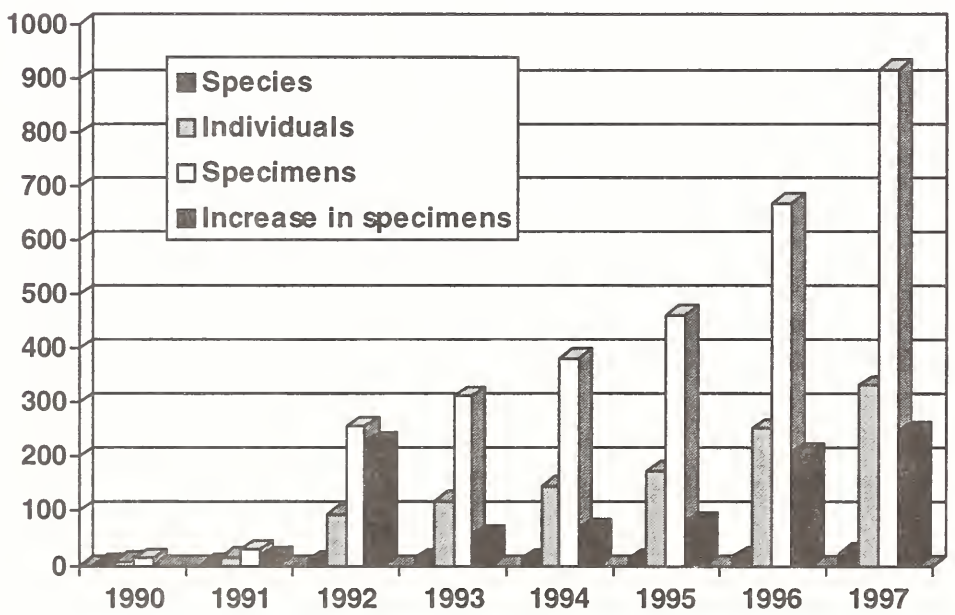

Fig. 1. National Marine Mammal Tissue Bank: regional sampling and collection rate

Table 3. Marine mammals sampled outside of Alaska for the NMMTB (O - samples archived; X - subsample B homogenized and divided into aliquots for analyses).

Species	Sex	Field ID	Region	Date	Tissue[1] L	K	B	M
Pilot Whale	F	MH90-597Gm	N. Atlantic	1990	X		X	
Pilot Whale	F	MH90-599Gm	N. Atlantic	1990	X		X	
Pilot Whale	F	MH90-600Gm	N. Atlantic	1990	X		X	
Pilot Whale	F	MH90-617Gm	N. Atlantic	1990	X		X	
Pilot Whale	F	MH90-624Gm	N. Atlantic	1990	X		X	
Pilot Whale	F	MH91-602Gm	N. Atlantic	1991	X		X	
Pilot Whale	F	MH91-603Gm	N. Atlantic	1991	X		O	
Pilot Whale	M	MH91-604Gm	N. Atlantic	1991	X		X	
Pilot Whale	M	MH91-605Gm	N. Atlantic	1991	X		O	
Pilot Whale	M	NMFS #3069	N. Atlantic	1996	X	X	X	
Pilot Whale	F	NMFS #1609	N. Atlantic	1996	X	X	X	
Pilot Whale	F	NMFS #2090	N. Atlantic	1996	X	X	X	
Harbor Porpoise	M	MH90-553Pp	N. Atlantic	1990	X		X	
Harbor Porpoise	F	MH91-424Pp	N. Atlantic	1991	X		X	
Harbor Porpoise	F	MH91-461Pp	N. Atlantic	1991	X		X	
Harbor Porpoise	F	MH91-505Pp	N. Atlantic	1991	X		X	
Harbor Porpoise	M	MH91-504Pp	N. Atlantic	1991	X		X	
Harbor Porpoise	F	MH92-477Pp	N. Atlantic	1992	X		O	
Harbor Porpoise	F	MH92-575Pp	N. Atlantic	1992	X		X	
Harbor Porpoise	M	MH92-597Pp	N. Atlantic	1992	X		O	
Harbor Porpoise	M	PJG-126	N. Pacific	1993	O		O	
Harbor Porpoise	F	MH93-496Pp	N. Atlantic	1993	O		O	
Harbor Porpoise	F	MH97-462Pp	N. Atlantic	1997	O		O	
Harbor Porpoise	F	GM-97-19	N. Atlantic	1997	O	O	O	
Harbor Porpoise	F	GM-97-27	N. Atlantic	1997	O	O	O	
White-sided Dolphin	F	MH93-445La	N. Atlantic	1993	X		O	
White-sided Dolphin	F	MH93-446La	N. Atlantic	1993	X		O	
White-sided Dolphin	M	MH93-460La	N. Atlantic	1993	X		X	
White-sided Dolphin	F	MH93-461La	N. Atlantic	1993	X		O	
White-sided Dolphin	M	MH94-611La	N. Atlantic	1994	O		O	
White-sided Dolphin	M	MH96-408La	N. Atlantic	1996	O		O	
White-sided Dolphin	M	MH97-425La	N. Atlantic	1997	O		O	
White-sided Dolphin	M	MH97-506La	N. Atlantic	1997	O		O	
White-sided Dolphin	F	MH97-507La	N. Atlantic	1997	O		O	
White-sided Dolphin	F	MH97-534La	N. Atlantic	1997	O	O	O	

Tissue[1]
L - Liver B - Blubber
K - Kidney M - Muscle

Table 3 (cont.) Marine mammals sampled outside of Alaska for the NMMTB (O - samples archived; X - subsample B homogenized and divided into aliquots for analyses).

Species	Sex	Field ID	Region	Date	Tissue[1] L	K	B	M
Common Dolphin	F	NMFS #3960	N. Atlantic	1996	X	X	X	
Common Dolphin	M	NMFS #3694	N. Atlantic	1996	X	X	X	
Common Dolphin	M	NMFS #3069	N. Atlantic	1997	X	X	X	
Common Dolphin	F	MH97-594Dd	N. Atlantic	1997	X	X	X	
Common Dolphin	F	MH97-596Dd	N. Atlantic	1997	X	X	X	
Common Dolphin	F	MH97-597Dd	N. Atlantic	1997	X	X	X	
Common Dolphin	F	MH97-598Dd	N. Atlantic	1997	X	X	X	
Common Dolphin	F	MH97-599Dd	N. Atlantic	1997	X	X	X	
Common Dolphin	F	MH97-600Dd	N. Atlantic	1997	X	X	X	
Rough-toothed Dolphin	M	RTDL-001	G. of Mexico	1997	O	O	O	
Rough-toothed Dolphin	M	RTDL-002	G. of Mexico	1997	O	O	O	
Rough-toothed Dolphin	F	RTDL-003	G. of Mexico	1997	O	O	O	
Rough-toothed Dolphin	M	RTDL-004	G. of Mexico	1997	O	O	O	
Rough-toothed Dolphin	M	RTDL-005	G. of Mexico	1997	O	O	O	
Rough-toothed Dolphin	M	RTDL-006	G. of Mexico	1997	O	O	O	
Rough-toothed Dolphin	F	RTDL-007	G. of Mexico	1997	O	O	O	
Rough-toothed Dolphin	F	RTDL-008	G. of Mexico	1997	O	O	O	
Rough-toothed Dolphin	F	RTDL-009	G. of Mexico	1997	O	O	O	
Rough-toothed Dolphin	F	RTDL-010	G. of Mexico	1997	O	O	O	
Rough-toothed Dolphin	F	RTDL-011	G. of Mexico	1997	O	O	O	
Rough-toothed Dolphin	M	RTDL-012	G. of Mexico	1997	O	O	O	
Rough-toothed Dolphin	F	RTDL-013	G. of Mexico	1997	O	O	O	
Rough-toothed Dolphin	M	RTDL-014	G. of Mexico	1997	O	O	O	
Rough-toothed Dolphin	F	RTDL-015	G. of Mexico	1997	O	O	O	
Fin Whale	F	MH-97-531Bp	N. Atlantic	1997			O	
Hooded Seal	F	MH-97-428Cc	N. Atlantic	1997	O		O	
Hooded Seal	M	MH-97-455Cc	N. Atlantic	1997	O		O	
Hooded Seal	M	MH-97-461Cc	N. Atlantic	1997	O		O	
Harp Seal	M	MH-97-460Pg	N. Atlantic	1997	O		O	
Harp Seal	F	MH-97-469Pg	N. Atlantic	1997	O		O	
Harp Seal	M	MH-97-438Pg	N. Atlantic	1997	O		O	
Harp Seal	M	MH-97-629Pg	N. Atlantic	1997	O	O	O	
Harbor Seal	F	MH-97-430Pv	N. Atlantic	1997	O		O	
Harbor Seal	M	MH-97-503Pv	N. Atlantic	1997	O		O	
Harbor Seal	M	MH-97-576Pv	N. Atlantic	1997	O	O	O	
Ringed Seal	M	MH-97-431Ph	N. Atlantic	1997	O		O	

Tissue[1]
L - Liver B - Blubber
K - Kidney M - Muscle

Table 3 (cont.) Marine mammals sampled outside of Alaska for the NMMTB (O - samples archived; X - subsample B homogenized and divided into aliquots for analyses).

Species	Sex	Field ID	Region	Date	Tissue[1] L	K	B	M
California Sea Lion	F	CSL-2203	N. Pacific	1993	O	O	O	
California Sea Lion	M	CSL-2217	N. Pacific	1993	O	O	O	
California Sea Lion	M	CSL-2881	N. Pacific	1993	O	O	O	
California Sea Lion	M	CSL-3067	N. Pacific	1993	O	O	O	
California Sea Lion	M	CSL-3101	N. Pacific	1996	O	O	O	
California Sea Lion	M	CSL-2203	N. Pacific	1996	O	O	O	O
California Sea Lion	M	CSL-3101	N. Pacific	1996	O	O	O	
California Sea Lion	M	CSL-3105	N. Pacific	1996	O	O	O	
California Sea Lion	M	CSL-2203	N. Pacific	1996	O	O	O	
California Sea Lion	F	CSL-3127	N. Pacific	1997	O	O	O	
California Sea Lion	M	CSL-3153	N. Pacific	1997	O	O	O	
California Sea Lion	M	CSL-3264	N. Pacific	1993	O	O	O	
California Sea Lion	M	CSL-3306	N. Pacific	1993	O	O	O	
California Sea Lion	M	CSL-3314	N. Pacific	1993	O	O	O	
California Sea Lion	M	CSL-3321	N. Pacific	1993	O	O	O	
California Sea Lion	M	CSL-3377	N. Pacific	1996	O	O	O	
California Sea Lion	M	CSL-3449	N. Pacific	1996	O	O	O	
California Sea Lion	F	CSL-3438	N. Pacific	1996	O	O	O	
Grey Seal	M	MH-97-484Hg	N. Atlantic	1997	O		O	
Grey Seal	F	MH-97-491Hg	N. Atlantic	1997	O		O	
Grey Seal	M	MH-97-493Hg	N. Atlantic	1997	O		O	

Tissue[1]
L - Liver B - Blubber
K - Kidney M - Muscle

Table 4. Inventory of NMMTB specimens (non-Alaska) that have been analyzed at NIST.

Species/Tissue Type	Specimens Archived	Organics	Inorganics
Pilot Whale (*Globicephala melas*)			
Liver	12	-	9
Kidney	3	-	-
Blubber	12	7	-
Harbor Porpoise (*Phocoena phocoena*)			
Liver	13	-	6
Kidney	2	-	-
Blubber	13	5	-
White-Sided Dolphin (*Lagenorhynchus acutus*)			
Liver	10	-	4
Kidney	1	-	-
Blubber	10	1	-
Common Dolphin (*Delphinus delphis*)			
Liver	9	-	-
Kidney	9	-	-
Blubber	9	-	-
Rough-toothed Dolphin (*Steno bredanensis*)			
Liver	15	-	-
Kidney	15	-	-
Blubber	15	-	-
Fin Whale (*Balaenoptera physalus*)			
Blubber	1	-	-
California Sea Lion (*Zalophus californianus*)			
Liver	18	-	-
Kidney	18	-	-
Blubber	18	-	-
Muscle	1	-	-
Hooded Seal (*Cystophora cristata*)			
Liver	3	-	-
Blubber	3	-	-
Harp Seal (*Phoca groenlandica*)			
Liver	4	-	-
Kidney	1	-	-
Blubber	4	-	-
Harbor Seal (*Phoca vitulina*)			
Liver	3	-	-
Kidney	1	-	-
Blubber	3	-	-
Ringed Seal (*Phoca hispida*)			
Liver	1	-	-
Blubber	1	-	-
Grey Seal (*Halichoerus grypus*)			
Liver	3	-	-
Blubber	3	-	-
Total	234	13	19

Heavy metals and Other Elements

For many of the trace elements in marine mammal tissues, little is known of what concentrations are within the normal ranges for a particular species. Many researchers analyze tissues for a few essential elements, such as Zn, Cu, or Se, and/or for potentially toxic elements, such as Cd, Hg, or Pb. The use of INAA typically provides data for 30 to 40 elements, many of which are not routinely measured by researchers using more conventional analytical methods. Concentrations for 30 to 40 elements were determined using INAA in liver tissues from nine pilot whales, six harbor porpoises, and four white-sided dolphins from the North Atlantic archived in the NMMTB. The resulting data are found in Appendix E. The concentration ranges, mean values, and standard deviations for selected elements in the livers of the three species are presented in Table 5. Comparison of the concentrations of these elements in the livers of these three species with values for beluga and bowhead whale liver tissues archived in the NMMTB are presented in Figs. 2 through 5.

Concentrations of K, Cl, Na, Mg, Ca, Br, and Rb in liver tissues are generally consistent from animal to animal and from species to species (Figs. 2 and 3). The relative standard deviations of the mean values for these elements range from 5 % to 25 %. The biological half-lives for most of these elements are relatively short, so that one would not expect to find much animal-animal variability. Concentrations of Cs vary much more from animal to animal than those of the other electrolytes. The variation for Cs may be due to the much lower abundance of Cs in the marine environment than other electrolytes and probably is not metabolically regulated.

Concentrations of the essential trace elements, such as Cu, Zn, and Se, are generally characterized by a relatively narrow range of values within a species and, for many elements, the ranges are similar from one species to another (Figs. 2 and 4). The relative standard deviations for the mean values ranged from 15 % to 45 % within a species for all elements except Se, for which the relative standard deviations ranged from 50 % to 70 %. The relatively narrow range of values and similarity from species to species probably indicates that these elements are well regulated and may indicate relatively short biological half-lives. The wider range of values for Se is due to the accumulation of this trace element as the animal ages. Concentrations of Se are generally higher in the pilot and beluga whale livers than in the livers of any of the other cetaceans (Fig. 4).

The nonessential, potentially toxic elements, such as Cd and Hg, show the greatest variability with concentration ranges often spanning several orders of magnitude (Fig. 4). These elements accumulate in liver tissue as the animal ages, so that the observed levels probably reflect the magnitude and duration of exposure to the element. Both Cd and Hg can occur in relatively high concentrations in the livers of marine mammals, depending upon the species and the type of prey on which they feed. Although the livers of beluga whales had Cd concentrations at relatively low levels, their Hg levels were high. As a comparison, the bowhead whales were found to have very low Hg concentrations and relatively high Cd levels (Mackey et al., 1996).

Three other elements show the same pattern of widely varying concentrations within and between species, i.e., V, Se, and Ag (Figs. 4 and 5). The V levels in livers of the marine mammals from Alaska span two orders of magnitude with concentrations ranging from 0.02 µg/g to 1 µg/g wet mass (Fig. 5 and Mackey et al., 1996). Levels in the livers of mammals from the North Atlantic are generally near or below the detection limit. This appears to be a regional difference and is

Table 5. Concentration ranges and mean ± one standard deviation values ($\mu g \cdot g^{-1}$, wet mass) of heavy metals and other elements in liver tissues of three cetacean species from the North Atlantic archived in the NMMTB.

	Pilot Whale *(Globicephala melas)* n = 9	*Harbor Porpoise* *(Phocoena phocoena)* n = 6	*White-sided Dolphin* *(Lagenorhynchus acutus)* n = 4
Na	1260 - 1620 1480 ± 152	1200 - 1700 1430 ± 167	1110 - 1260 1190 ± 65
Mg	81 - 150 120 ± 24	128 - 267 204 ± 51	134 - 162 148 ± 14
Cl	1630 - 2230 1810 ± 196	1590 - 2030 1810 ± 1820	1360 - 1530 1470 ± 80
K	1640 - 2640 2190 ± 306	2130 - 3310 2680 ± 458	2770 - 3150 2900 ± 176
Ca	24 - 68 48 ± 12	29.7 - 63.3 48.8 ± 12	41 - 58 50.5 ± 7
V	≤0.01 (ND)[a] - 0.02 0.02 (n=2)	≤0.01 (ND) - 0.02 n = 1, 0.02	≤0.01 (ND) - 0.06 n = 1, 0.06
Mn	1.78 - 2.98 2.45 ± 0.41	2.68 - 5.15 4.49 ± 0.93	3.1 - 4.1 3.7 ± 0.5
Fe	144 - 806 388 ± 209	226 - 562 388 ± 116	66 - 297 179 ± 100
Co	0.007 - 0.015 0.012 ± 0.003	0.004 - 0.010 0.006 ± 0.002	0.010 - 0.0160 0.013 ± 0.003
Cu	1.13 - 3.8 2.83 ± 0.81	3.84 - 15.3 8.87 ± 3.97	3.18 - 8.06 6.33 ± 2.15
Zn	28.3 - 51.1 38.2 ± 7.65	25.3 - 38.2 28.4 ± 4.9	30.9 - 50.7 41.6 ± 9.9
As	≤0.4 (ND) - 1.17 0.45 ± 0.33	0.18 - 0.58 0.34 ± 0.13	0.179 - 0.415 0.297 ± 0.105
Se	1.59 - 28.5 12.8 ± 9.2	1.11 - 4.23 2.04 ± 1.19	2.22 - 9.05 5.31 ± 2.98

Table 5 (cont.) Concentration ranges and mean ± one standard deviation values ($\mu g \cdot g^{-1}$, wet mass) of heavy metals and other elements in liver tissues of three cetacean species from the North Atlantic archived in the NMMTB.

	Pilot Whale (Globicephala melas) n = 9	*Harbor Porpoise (Phocoena phocoena)* n = 6	*White-sided Dolphin (Lagenorhynchus acutus)* n = 4
Br	Na[b]	11.75 - 16.38; n = 4 14.1 ± 2.1; n = 4	12.4 - 16.6 14.0 ± 1.8
Rb	1.44 - 2.1 1.89 ± 0.2	1.04 - 1.73 1.38 ± 0.28	≤1 (ND) - 2.7 n = 1, 2.7
Ag	0.013 - 0.333 0.16 ± 0.11	0.148 - 0.757 0.398 ± 0.256	0.27 - 1.50 0.75 ± 0.53
Cd	2.78 - 14.28 8.48 ± 3.14	≤0.4 (ND) - 0.51 n = 1, 0.51	0.24 - 0.886 0.42 ± 0.29
Cs	≤0.002(ND)-0.010 0.006 ± 0.002	0.033 - 0.050 0.11 - 0.019	0.027 - 0.042 0.032 ± 0.006
Hg	1 - 83 43.2 ± 36.4	2.19 - 43.4, n = 3 28.1 ± 22.5; n = 3	1.01 - 22.8 9.06 ± 12.0

[a]ND indicates that the levels are below the detection limit; quantitative determination was not possible.
[b]NA indicates that analyses were not performed.

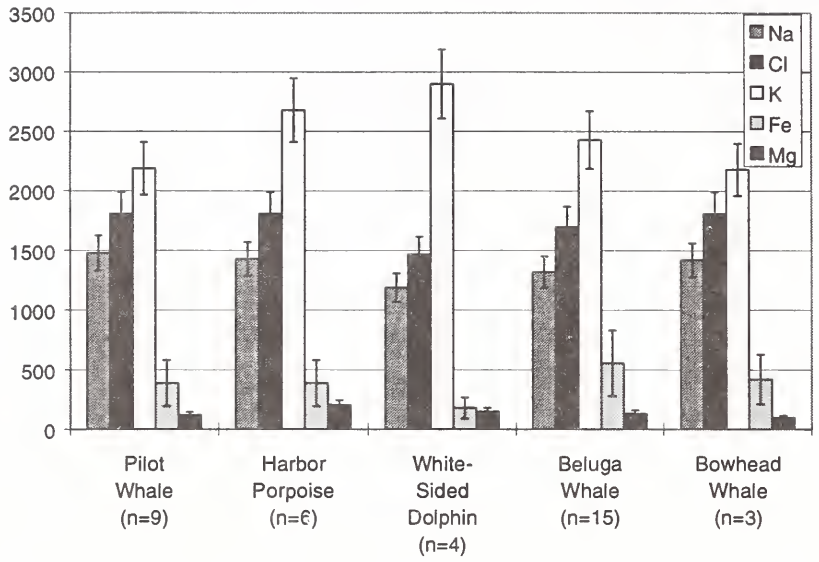

Fig. 2. Mean concentrations of Na, Cl, K, Fe, and Mg in the livers of five cetacean species archived in the NMMTB. Each error bar represents the mean ± one standard deviation. Values are expressed as $\mu g \cdot g^{-1}$ wet mass.

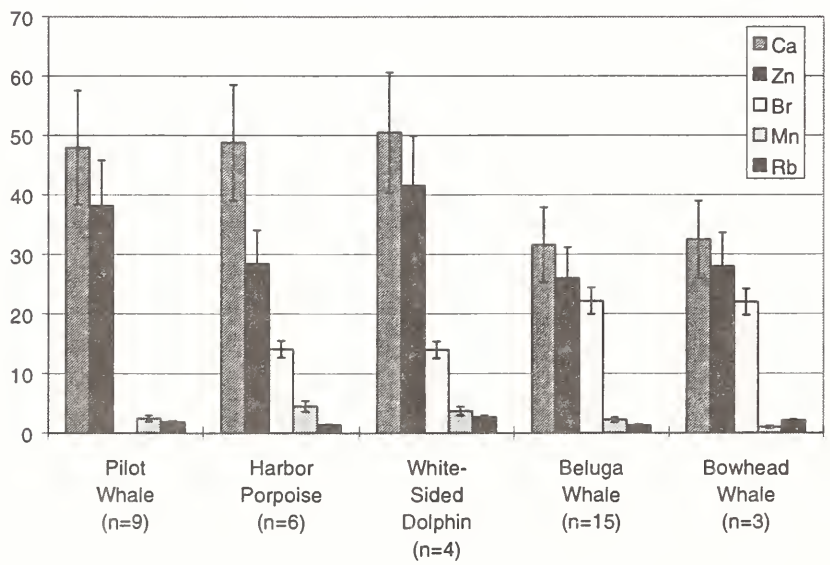

Fig. 3. Mean concentrations of Ca, Zn, Br, Mn, and Rb in the livers of five cetacean species archived in the NMMTB. Each error bar represents the mean ± one standard deviation. Values are expressed as $\mu g \cdot g^{-1}$ wet mass.

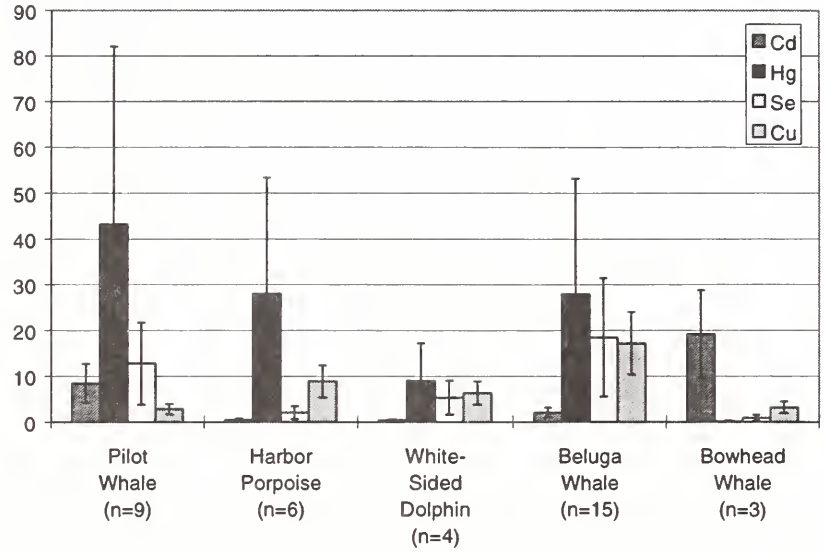

Fig. 4. Mean concentrations of Cd, Hg, Se, and Cu in the livers of five cetacean species archived in the NMMTB. Each error bar represents the mean ± one standard deviation. Values are expressed as $\mu g \cdot g^{-1}$ wet mass.

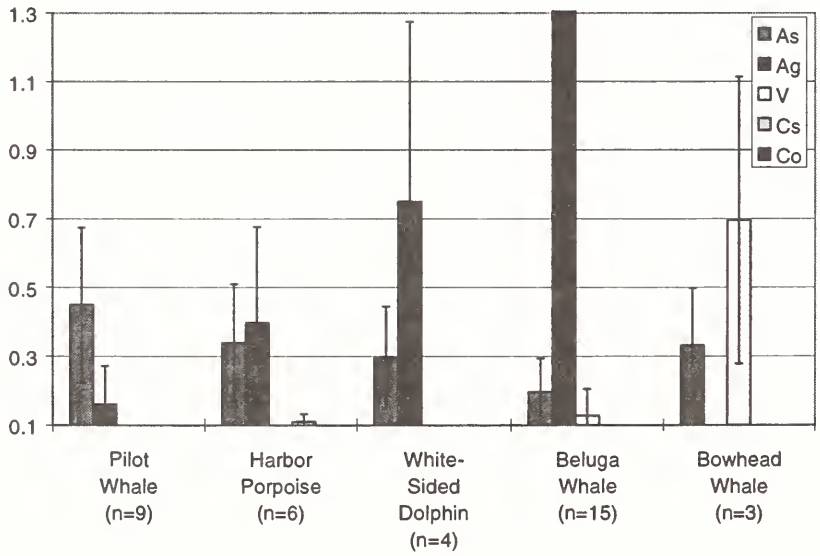

Fig. 5. Mean concentrations of As, Ag, V, Cs, and Co in the livers of five cetacean species archived in the NMMTB. Each error bar is the mean ± one standard deviation. Values are expressed as $\mu g \cdot g^{-1}$ wet mass.

discussed in detail below. Many researchers have reported a positive correlation between hepatic Se and Hg concentrations for several different species of animals. Hepatic Se exhibits positive linear correlation with hepatic Hg for the NBSB marine mammals as well (Mackey et al., 1995). Although hepatic Cd has sometimes been reported as being positively correlated with Se in marine mammals, no such relationship has been found so far in the NBSB specimens.

For most of the marine mammal species in this study, Ag concentrations in livers are also correlated with Se and Hg. For example, whether using multiple regression, simple linear regression, or Spearman's rank correlation, both Ag and total Hg have been reported to be strongly correlated with Se in the livers of beluga whales and pilot whales banked in the NBSB (Becker et al., 1995a). Selenium may have a role in the detoxification of Hg in the liver, either by association with the metal binding proteins or by directly binding to Hg. These correlations between hepatic Ag, Se, and Hg concentrations may also be simply an indication of accumulation of all three elements with age for the animal or may indicate that there exists a more direct biochemical relationship. Physiological mechanisms involving the interaction of Ag and Se have been shown for other species of mammals and may also function in these marine mammals. The possible biochemical relationships have been previously discussed in detail (Becker et al., 1995a). Silver concentrations generally range from 0.01µg/g to 0.1 µg/g wet mass in liver tissues of all of the marine mammals studied, with the exception of beluga whales. Levels in the liver tissues of the beluga whales are generally orders of magnitude greater than those found in other species, with concentrations ranging from 10 µg/g to 107 µg/g wet mass. The source or cause of these comparatively elevated levels of Ag in the beluga whale livers has not been identified.

Results of INAA of marine mammal liver tissues will be helpful in determining whether concentrations are increasing over time or whether bioaccumulation is occurring for a given species within a given organ. One measure of bioaccumulation is to determine whether concentrations increase with the age of the animal. Positive correlations with age were observed for Cd, V, Se, Ag, and Hg in livers of beluga whales (Mackey et al., 1996). These elements accumulate in beluga whale liver tissue with age. Because age information was not available for any of the other species, animal length was used as an indication of relative age of the animals. For some species, e.g., pilot whales (Kasuya et al., 1988) and beluga whales (Doidge, 1990), there is a known relationship between the length and the age of the animal.

The elements for which there were positive correlations between hepatic concentrations and animal age or length are listed in Table 6. For several species, the number of animals for which data are available is still too few to determine whether the observed correlations are significant. However, it is clear that hepatic Hg, Se, and Ag increase over time for most of these mammals and that V concentrations increase with age for the Alaska marine mammals (Mackey et al., 1996). Bioaccumulation of V has also been reported in livers of harbor seals and grey seals (*Halichoerus grypus*) from the Swedish coast (Frank et al., 1992). Hepatic levels of Cd were correlated with age or length only for the beluga whales and ringed seals (Mackey et al., 1996).

Although uptake and bioaccumulation of Hg, Se, Ag, V, and Cd are determined by many factors, the diet of the animals probably plays a major role. The possible role of prey in the bioaccumulation of trace elements in these marine mammals has been previously discussed (Becker et al., 1995a; Mackey et al, 1995; Mackey et al., 1996); however, additional research is needed to

determine whether the concentration levels found in the liver tissues for these marine mammals reflect levels that would be present naturally or whether the levels reflect the influence of anthropogenic inputs into the marine environment.

Table 6. Trace elements that accumulate in livers of cetaceans.

Species	Elements
Alaska:	
Beluga Whale (*Delphinapterus leucas*)	V, Se, Ag, Cd, Hg
Bowhead Whale (*Balaena mysticetus*)	V, Se, Ag, Hg
North Atlantic:	
Pilot Whale (*Globicephala melas*)	Se, Ag, Hg
Harbor Porpoise (*Phocoena phocoena*)	Se
White-sided Dolphin (*Lagenorhynchus acutus*)	Hg

A major difference between the trace element concentrations of the livers from the Alaskan cetaceans as compared to those from the North Atlantic animals (i.e., pilot whales, harbor porpoises, and white-sided dolphins) is the level of V (Fig. 9). As mentioned previously, V is present at measurable levels in the livers of all of the Alaskan marine mammals, whereas for most of the North Atlantic animals, hepatic V concentrations are near or below the detection limit of 0.01 μg/g. This apparent geographic difference in vanadium is discussed in Mackey et al. (1996).

Chlorinated Persistent Organic Compounds

The concentrations of PCB congeners and chlorinated pesticides have been measured by NIST in the blubber of two cetacean species from the North Atlantic (pilot whale and harbor porpoise) archived in the NMMTB. The resulting data are found in Appendix F and summary statistics are presented in Tables 7 and 8. Both of these North Atlantic cetaceans had higher concentrations of PCBs and DDT in their blubber than the beluga whales that have been sampled by the program in several locations in Alaska, i.e., Point Lay and Point Hope in the Arctic Ocean and Cook Inlet in the subarctic Gulf of Alaska (Fig. 6). The harbor porpoise had the highest levels of PCBs and the pilot whale had the highest concentration of total DDT (s-DDT).

Of the 33 congeners composing the "total PCBs" (s-PCB), as identified in Table 7, the following (in descending order) had consistently the highest concentration values in both species: 153, 138/163/164, 187, 180, 149, 118, 99, 101/90, 52, 95, 170/190, 66, 105, and 44. Table 8 gives the range of values for each of these congeners found in the two species. Although the mean concentration value of 101/90 was about the same in both species (i.e., ca.600 $ng \cdot g^{-1}$), the variation

was greater in the pilot whales (i.e., one standard deviation was 85% of the mean as compared to 47% for the harbor porpoise).

The concentrations of these congeners in the blubber of the two North Atlantic species are compared in Fig. 7 with the concentrations measured by the program in beluga whales from the Alaska Arctic and subarctic (Cook Inlet). Although PCB 153 occurred at higher concentrations in all of the animals, it contributed the most to the s-PCB levels in harbor porpoise (21%) as compared to the other two species (10% to 14%). This congener is relatively resistant to metabolic breakdown and remains stable in animal tissues as compared to the other congeners. PCB 138/163/164 appeared to contribute more to the total PCB concentrations in harbor porpoise and pilot whale than was the case for the beluga whales (13% to 16% for the former two species, 8 - 9% for the beluga whales). Contributions of PCB 118 and 149 were basically the same for all three species, while PCB 105 and 44 were substantially less in the harbor porpoise (0.6% and 0.3%, respectively) as compared to the pilot and beluga whales (1% to 3% for both species and both congeners).

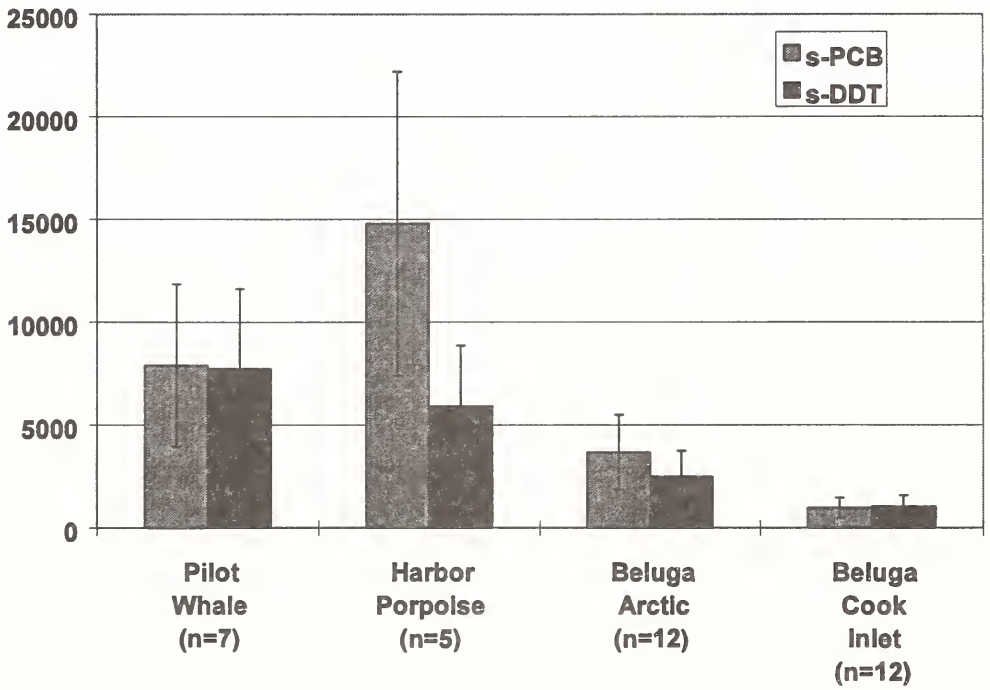

Fig. 6. Mean concentrations of PCBs and DDT in the blubber of three cetacean species archived in the NMMTB. s-PCB is the sum of 33 congeners (18, 28, 31, 44, 49, 52, 66, 87, 95, 99, 101/90, 105, 110/77, 118, 128, 138/163/164, 149, 151, 153, 156, 170/190, 180, 183, 187, 194, 195, 206, and 209). s-DDT is the sum of 2,4'-DDE, 4,4'DDE; 2,4'-DDD, 4,4'-DDD, 2,4'-DDT, and 4,4'-DDT. Each error bar represents the mean ± one standard deviation. Values are expressed as ng·g $^{-1}$ wet mass.

Table 7. Concentration ranges and mean ± one standard deviation values ($ng \cdot g^{-1}$, wet mass) of chlorinated hydrocarbons in fat tissue (blubber) of two species of cetaceans from the North Atlantic archived in the NMMTB.

	Pilot Whale, *Globicephala melas* n = 7	Harbor Porpoise, *Phocoena phocoena* n = 5
s-PCB[a]	2030 - 17200 7900 ± 5330	7420 - 22600 14800 ± 6200
s-DDT[b]	1708 - 13035 7748 ± 4701	4690 - 11200 7280 ± 2870
4,4'-DDE	942 - 7118 3847 ± 2128	1880 - 4900 3260 ± 1250
HCB	43 - 465 223 ± 179	223 - 1070 515 ± 353
α-HCH	20 - 62.2 32.6 ± 14.0	232 - 708 406 ± 199
γ-HCH	3.42 - 20 7.86 ± 5.74	42.2 - 152 81.2 ± 41.8
cis-chlordane	68.3 - 366 223 ± 137	397 - 1640 822 ± 539
cis-nonachlor	39.3 - 125 82.2 ± 60.6	325 - 697 511 ± 263
trans-nonachlor	280 - 2420 1340 ± 901	1920 - 3420 2660 ± 558
oxychlordane	26.7 - 270 143 ± 106	183 - 801 424 ± 228
heptachlor epoxide	14.6 - 138 81.1 ± 48.8	196 - 564 304 ± 158
dieldrin	56.8 - 604 262 ± 240	658 - 1450 963 ± 294

[a]Sum of 33 congeners: 18, 28, 31, 44, 49, 52, 66, 87, 95, 99, 101/90, 105, 110/77, 118, 128, 138/163/164, 149, 151, 153, 156, 170/190, 180, 183, 187, 194, 195, 206, 209

[b]Sum of 2,4'-DDE; 4,4'-DDE; 2,4'-DDD; 4,4'-DDD; 2,4'-DDT; 4,4'-DDT

Table 8. Concentration ranges and mean ± one standard deviation values ($ng \cdot g^{-1}$, wet mass) of major PCB congeners in fat tissue (blubber) of two species of cetaceans from the North Atlantic archived in the NMMTB.

	Pilot Whale, *Globicephala melas* n = 7	Harbor Porpoise, *Phocoena phocoena* n = 5
44	17 - 188 84 ± 70.1	28.6 - 101 46.1 ± 30.9
52	43 - 982 377 ± 380	336 - 1541 739 ± 479
66	27.5 - 404 174 ± 161	106 - 648 291 ± 212
95	45.6 - 621 297 ± 239	291 - 1430 786 ± 429
99	38 - 1260 470 ± 488	180 - 1920 861 ± 698
101/90	43.9 - 1320 611 ± 519	217 - 907 591 ± 277
105	22.6 - 397 150 ± 51	20.9 - 168 93 ± 59
118	86.4 - 1256 502 ± 414	351 - 1010 710 ± 270
138/163/164	172 - 2600 1030 ± 821	1030 - 3770 2360 ± 1120
149	124 - 1210 545 ± 381	643 - 1890 1180 ± 533
153	246 - 2670 1120 ± 820	1747 - 5600 3170 ± 1530
180	270 - 1200 564 ± 301	450 - 2120 982 ± 663
170/190	85.6 - 346 197 ± 88	156 - 953 354 ± 337
187	232 - 1300 582 ± 353	446 - 2440 1174 ± 772

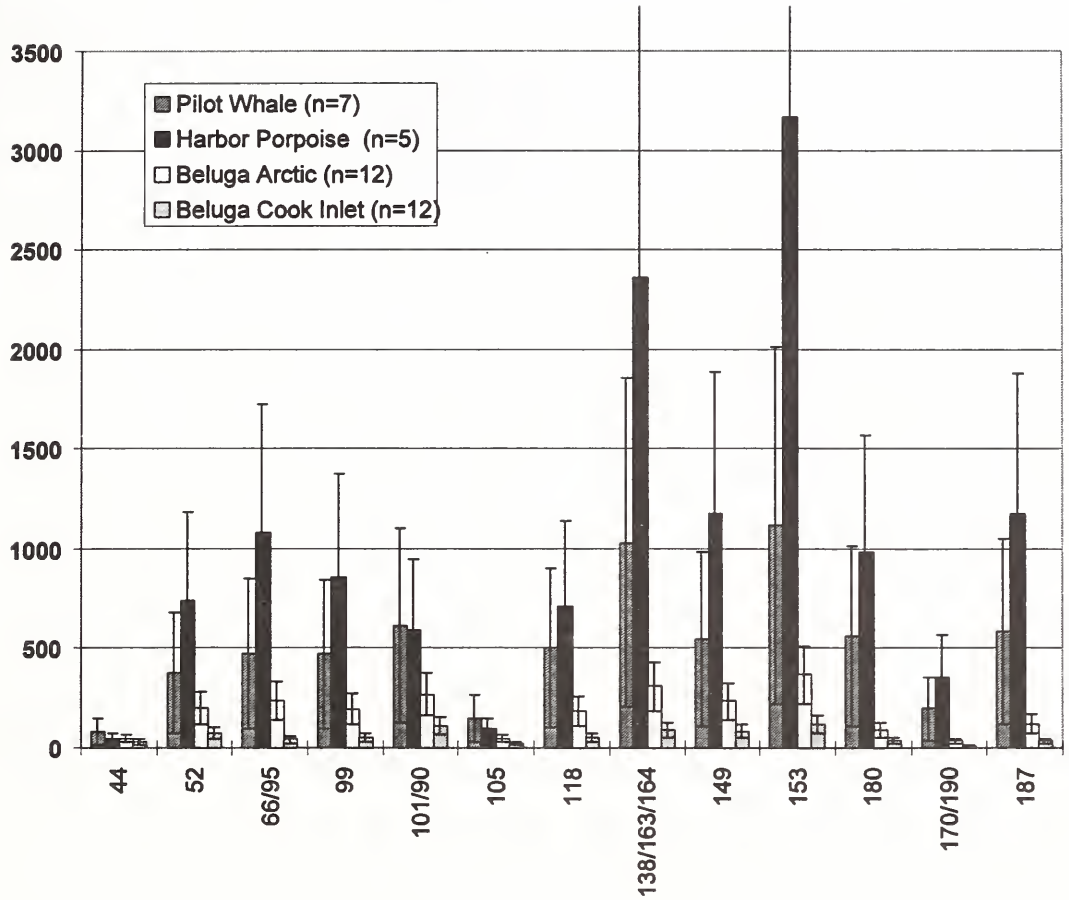

Fig. 7. Mean concentrations of PCB congeners with the highest values measured in the blubber of three cetacean species archived in the NMMTB. These congeners had consistently the highest concentration values of the 33 PCB congeners reported for these animals Each error bar represents the mean ± one standard deviation. Values are expressed as $ng \cdot g^{-1}$ wet mass.

The concentrations of chlordane compounds (*cis*-nonachlor, *trans*-nonachlor, *cis*-chlordane, oxychlordane, and heptachlor epoxide) in the blubber of the two North Atlantic cetaceans are compared in Fig. 8 to values for Arctic and subarctic belugas sampled by the program. *Trans*-nonachlor is the chlordane compound that usually dominates the total chlordane levels of marine mammals. As was the case for PCBs, the chlordane compounds occurred at higher concentrations in the harbor porpoise than in either the pilot or beluga whale, and substantially higher levels of *trans*-nonachlor occurred in the blubber of the pilot whale than in the beluga whales.

As was the case for the chlordane compounds, dieldrin, α-HCH, and γ-HCH (lindane) occurred in higher concentrations in the harbor porpoise than in the pilot or beluga whales (Fig. 9). This was particularly so for dieldrin and α-HCH. Unlike these compounds, hexachlorobenzene (HCB) occurred at higher concentrations in the blubber of beluga whales from the Arctic than in either of the North Atlantic species, and this compound occurred at higher concentrations in the beluga whales

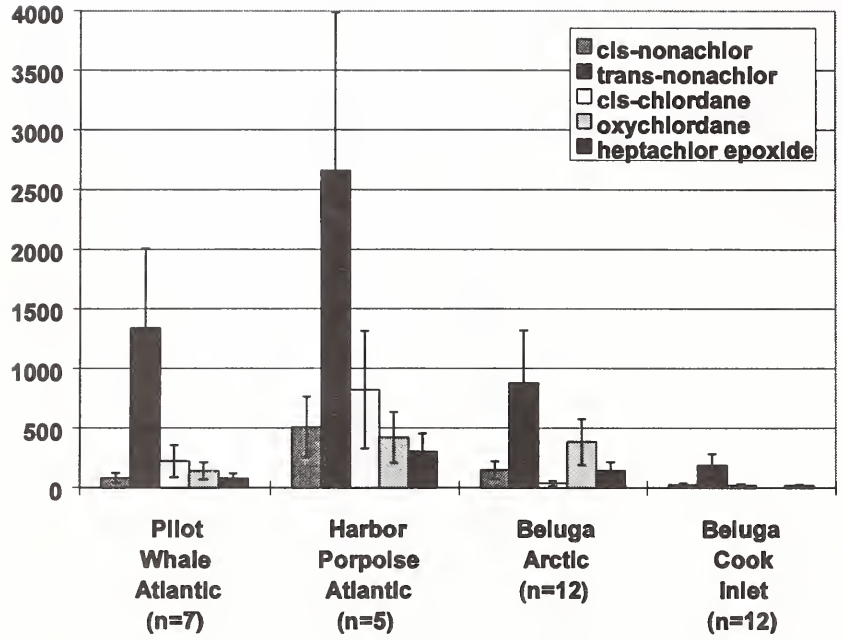

Fig. 8. Mean concentrations of chlordane compounds in the blubber of three cetacean species archived in the NMMTB. Each error bar represents the mean ± one standard deviation. Values are expressed as $ng \cdot g^{-1}$ wet mass.

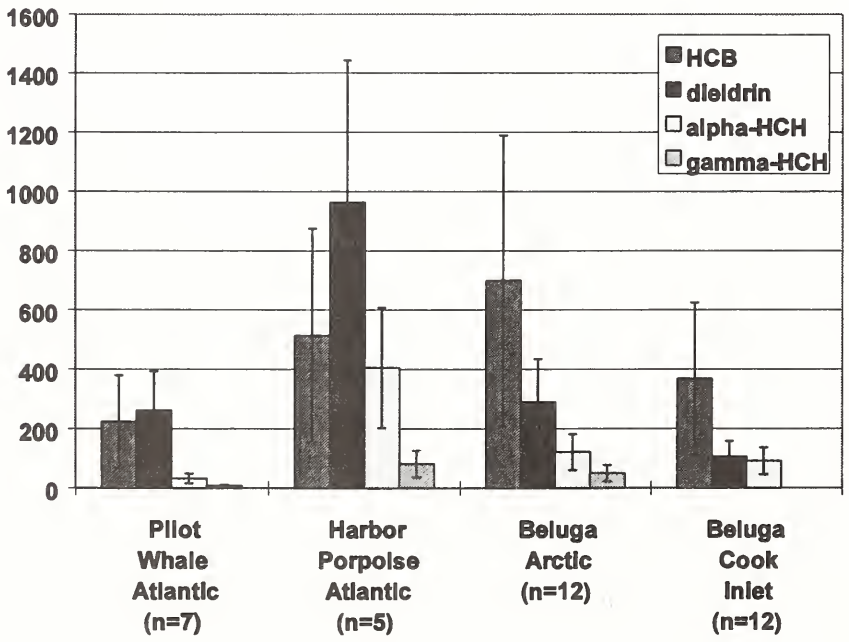

Fig. 9. Mean concentrations of HCB, dieldrin, *alpha*-HCH, and *gamma*-HCH (lindane) in the blubber of three cetacean species archived in the NMMTB. Each error bar represents the mean ± one standard deviation. Values are expressed as $ng \cdot g^{-1}$ wet mass.

from Cook Inlet than in the pilot whales from the North Atlantic. This is consistent with the "Global Fractionation and Polar Condensation" theory of Wania and Mackay in which the atmospheric transport and global deposition of persistent organic pollutants has been related to the log octanol-air partition coefficient, subcooled liquid vapor pressure, and temperature of condensation of the individual compounds. Based on these factors, HCB has relatively high mobility, and has been identified as a compound that is preferentially deposited and accumulated in polar latitudes (Wania & Mackay, 1996).

CHEMICAL ANALYSIS QUALITY ASSURANCE PROGRAM

Preparation and Analysis of Control Materials

Control materials, which are similar to the matrices being analyzed, are analyzed with the regular samples and the results monitored to determine whether the analytical procedures are in control. The first control materials developed for the program were derived from liver and blubber tissues collected from pilot whales (*Globicephala melaena*) stranded in 1990 on Cape Cod, Massachusetts. Approximately 2 kg of each tissue were used to prepare tissue homogenates for use as analytical control materials and in interlaboratory comparison exercises. These homogenates are fresh frozen samples similar to marine mammal tissue samples routinely analyzed, rather than freeze-dried matrices typically used as control or reference materials. The tissues were cryogenically pulverized and homogenized in Teflon mills to provide frozen powder-like materials (Zeisler et al., 1983). These two control materials were analyzed at NIST to determine concentrations of trace elements (*Whale Liver Homogenate I*) and organic contaminants *(Whale Blubber Control Material*) and to assess sample homogeneity (Wise, 1993; Wise et al., 1993). The analytical data for the *Whale Liver Homogenate I* are presented in Appendix E.4 and for the *Whale Blubber Control Material* in Appendix F.3.

The concentrations of 39 trace elements were determined in the *Whale Liver Homogenate I* using INAA at NIST, coupled with differential pulse and square wave stripping voltammetry, performed at the Research Center of Jülich. Hg and methyl-Hg were also determined at Jülich using cold vapor atomic absorption (CVAA) spectroscopy (for Hg) and ion-exchange chromatography plus CVAA (for methyl-Hg). The analyses at Jülich provided results for two elements not analyzed by INAA (Ni and Pb), data for methyl-Hg, and a comparison of results for five elements (Co, Cu, Zn, Cd, and Hg) measured by different techniques. The results for these five elements using INAA, voltammetry, and CVAA were in good agreement. The data resulting from these analyses, as well as a description of the analytical techniques and interpretation of the results, are provided in Wise et al. (1993). A copy of this paper is found in Appendix G.

The analyses of the *Whale Blubber Control Material* were conducted at NIST using GC-ECD and gas chromatography-mass spectrometry (GC-MS) to determine 30 PCB congeners and 16 chlorinated pesticides. Six aliquots of the control material were analyzed in duplicate with GC-ECD using two different columns (DB-5 and C-18) and GC-MS using a DB-5 column, thus giving results using three different approaches. The results obtained from the three methods were generally in good agreement and discrepancies could be attributed to the separation of coeluting congeners due to differences in selectivity of the GC columns or the measurement of coeluting congeners due to

the selectivity of the mass spectrometric detection. The resulting data and recommended values for the PCBs and pesticide concentrations in this control material are found in Wise et al. (1993), which is reproduced in this volume as Appendix G.

Both the pilot whale liver homogenate (*Whale Liver Homogenate I*) and the *Whale Blubber Control Material* are available for use by laboratories as control materials during the routine analyses of marine mammal tissues. In addition, a second liver homogenate control material is being developed from liver tissue collected from beluga whales taken in Alaska native subsistence hunts in 1996. Based on experience gained in the preparation and analyses of *Whale Liver Homogenate I*, this second homogenate (*Whale Liver Homogenate II*) will be the basis for the development of a whale liver SRM, with certified trace element values (see discussion in the SRM section, below).

In 1996, manatee blubber was collected by Scott Wright, Florida Department of Environmental Protection, during the manatee die-off of that year and shipped to NIST, where it was prepared as a control material and readied for distribution to laboratories investigating the cause(s) of this mortality. Although the manatee deaths were found to be caused by a biotoxin and this material was not distributed, several kg of the homogenate are stored at the NMMTB for other future analyses, if needed.

Interlaboratory Comparison Exercises

The major activity to determine and improve the comparability of analytical results among NIST, NWFSC, and others are interlaboratory comparison exercises using samples of marine mammal tissues. The primary participants have been NIST and NWFSC; however, participation by other laboratories is increasing. After each intercomparison exercise, analysts from NIST and NWFSC meet to discuss the results and ways to improve accuracy and analytical comparability. To date these meetings have been informal, but as the program expands and more laboratories participate in the exercises, they may become more formal.

Table 9 lists the exercises conducted during the period 1991-97. The first exercise to determine comparability of analytical data was conducted in 1991-92 and consisted of the distribution of the liver and blubber control materials to NWFSC and several other laboratories for analysis, i.e., Department of Fisheries and Oceans (DFO) Canada in Winnipeg, the Geochemical and Environmental Research Group (GERG) at Texas A&M University, and the Department of Analytical and Environmental Chemistry at the University of Ulm in Germany.

In 1992, three laboratories (NIST, DFO Canada in Winnipeg, and the Department of Analytical and Environmental Chemistry at the University of Ulm in Germany) analyzed blubber subsamples from four to six beluga whales, the pilot whale blubber control material, and SRM 1588 Organics in Cod Liver Oil in an interlaboratory exercise to compare results of analyses for PCB congeners and chlorinated pesticides. These three laboratories had been collaborating on a routine basis in the analysis of samples collected through the AMMTAP. The analytical methods used by these three laboratories are described by Schantz et al. (1996). To eliminate differences in sources of calibration solutions, all three labs used common solutions to prepare calibration standards. Different internal standards and volume correction standards were used by each laboratory. Each laboratory used its preferred methods of extraction (Soxhlet extraction at NIST, ball-milling extraction at DFO, and

column extraction at Ulm). All three laboratories used a size exclusion chromatography cleanup, but with different columns, to remove the lipid material from the extracts and separated the PCB congeners from the majority of the pesticides as part of the cleanup procedures. GC-ECD was used by all three laboratories for the final analysis employing different columns. The results of this exercise were published by Schantz et al. (1996).

Results from the three laboratories for the chlorinated hydrocarbons in the *Whale Blubber Control Material* were in good agreement (Appendix F.3 and Table 10). NIST results for PCB 138, 153, and 180, which are the three congeners with the highest concentrations, were about 25% higher than the results from the two other laboratories. Although this may have been due to nonlinearity of the detectors, the chlorinated hydrocarbons with the highest concentration in this sample, 4,4'-DDE, exhibited between-laboratory variation of < 2%.

Table 9. Interlaboratory comparison exercises conducted for the Marine Mammal Health and Stranding Response Program during 1991-97.

Dates	Analytes	Matrix	Objective	Participants
1991-92	PCBs	whale blubber	lab comparability	NIST; NWFSC; DFO Canada;
	Cl pesticides[1]			GERG; Univ. of Ulm
	Elements	whale liver CM[2]	analytical control	NIST; NWFSC
1992-93	PCBs	whale blubber;	lab comparability	NIST; DFO Canada;
	Cl pesticides[1]	whale blubber CM[2]		Univ. of Ulm
1993-94	PCBs	whale blubber;	SRM development	NIST; NWFSC; DFO Canada;
	Cl pesticides[1]	whale blubber CM[2]		GERG; Arthur D. Little; NW Aquatic Sciences; Univ. of Ulm
1993-97	Elements	whale & seal liver	sample comparability	NIST; NWFSC
	PCBs	whale & seal blubber		
	Cl pesticides[1]			
	Elements	whale liver CM[2]	analytical control	NIST; NWFSC
1997-98	Elements	whale liver	lab comparability & control material development	NIST; NWFSC; Texas A&M Univ., Veterinary College

[1]Chlorinated Pesticides
[2]Control Material

Table 10. Results ($ng \cdot g^{-1}$, wet mass) of the interlaboratory comparison exercise for selected PCB congeners and chlorinated pesticides in Whale Blubber Quality Assurance (QA) Material. From Schantz et al. (1996).

Compound		NIST[a]	DFO[b]	Ulm[c]
PCB 18	(2,2',5-Trichlorobiphenyl)	6.12 ± 0.86	<5.1	NA[d]
PCB 28	(2,4,4'-Trichlorobiphenyl)	20.2 ± 0.5	29.9 (8.6)	26 (2)
PCB 31	(2,4',5-Trichlorobiphenyl)	2.59 ± 0.14	NA[d]	inf [d]
PCB 44	(2,2'3,5'-Tetrachlorobiphenyl)	17.8 ± 1.9	19.9 (2.2)	35 (1)
PCB 49	(2,2',4,5'-Tetrachlorobiphenyl)	34.8 ± 0.5	48.2 (12.9)	57 (2)
PCB 52	(2,2',5,5'-Tetrachlorobiphenyl)	103 ± 10	86.5 (5.9)	120 (1)
PCB 66	(2,3',4,4'-Tetrachlorobiphenyl)	50.4 ± 8.5	[202 (12)][e]	61 (2)
PCB 87	(2,2',3,4,5'-Pentachlorobiphenyl)	66.7 ± 0.7	NA	NA
PCB 95	(2,2',3,5',6-Pentachlorobiphenyl)	50.1 ± 0.1	[202 (12)][e]	143 (2)
PCB 99	(2,2'4,4',5-Pentachlorobiphenyl)	inf	162 (11)	159 (2)
PCB 101	(2,2'4,5,5'-Pentachlorobiphenyl)	261 ± 17	231 (15)	248 (3)
PCB 105	(2,3,3'4,4'-Pentachlorobiphenyl)	88.9 ± 13.0	61.5 (3.0)	82 (2)
PCB 110	(2,3,3',4'6-Pentachlorobiphenyl)	61.2 ± 0.8	52.8 (3.7)	47 (3)
PCB 118	(2,3',4,4'5-Pentachlorobiphenyl)	267 ± 25	217 (9)	217 (4)
PCB 128	(2,2',3,3',4,4'-Hexachlorobiphenyl)	99.0 ± 0.7	inf	77 (2)
PCB 138	(2,2',3,4,4',5'-Hexachlorobiphenyl)	664 ± 8	489 (24)	506 (8)
PCB 149	(2,2',3,4',5',6-Hexachlorobiphenyl)	372 ± 6	274 (14)	288 (5)
PCB 151	(2,2',3,5,5',6-Hexachlorobiphenyl)	111 ± 2	94.7 (5.4)	107 (2)
PCB 153	(2,2',4,4',5,5'-Hexachlorobiphenyl)	870 ± 9	582 (33)	618 (8)
PCB 156	(2,3,3',4,4',5-Hexachlorobiphenyl)	38.2 ± 0.7	47.9 (3.2)	39 (2)
PCB 170	(2,2',3,3',4,4',5-Heptachlorobiphenyl)	156 ± 1	129 (6)	118 (4)
PCB 180	(2,2',3,4,4',5,5'-Heptachlorobiphenyl)	483 ± 9	322 (17)	372 (4)
PCB 183	(2,2',3,4,4',5',6'-Heptachlorobiphenyl)	147 ± 1	109 (3)	117 (0)
PCB 187	(2,2'3,4',5,5',6-Heptachlorobiphenyl)	357 ± 8	317 (17)	NA
PCB 194	(2,2',3,3',4,4',5,5'-Octachlorobiphenyl)	69.8 ± 2.6	59.2 (3.5)	NA
PCB 195	(2,2',3,3',4,4',5,6-Octachlorobiphenyl)	16.3 ± 0.8	17.1 (6.9)	20 (1)
PCB 201	(2,2',3,3',4,5',6,6'-Octachlorobiphenyl)	22.7 ± 0.7	25.4 (16.2)	NA
PCB 206	(2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl)	29.0 ± 0.4	23.6 (1.1)	NA
PCB 209	(Decachlorobiphenyl)	7.98 ± 0.10	<0.9	NA

Table 10 (Cont.) Results (ng·g-[1], wet mass) of the interlaboratory comparison exercise for selected PCB congeners and chlorinated pesticides in Whale Blubber Quality Assurance (QA) Material. From Schantz et al. (1996).

Compound	NIST[a]	DFO[b]	Ulm[c]
2,4'-DDE	53.4 ± 7.4	49.4 (1.4)	62 (4)
4,4'-DDE	2032 ± 46	2076 (92)	1993 (20)
2,4'-DDD	58.4 ± 3.5	63.2 (3.1)	63 (2)
4,4'-DDD	260 ± 31	313 (7)	346 (18)
2,4'-DDT	222 ± 11	293 (16)	363 (12)
4,4'-DDT	651 ± 26	494 (19)	800 (55)
Hexachlorobenzene	36.9 ± 1.1	40.1 (3.3)	41 (3)
g-hexachlorohexane (HCH)	3.58 ± 0.35	3.7 (0.2)	<10
a-HCH	20.8 ± 2.6	22.9 (1.5)	22 (0.5)
b-HCH	NA	6.5 (1.6)	<10
Heptachlor epoxide	32.5 ± 2.7	28.3 (1.5)	NA
Oxychlordane	73.5 ± 0.8	66.2 (1.4)	72 (2)
cis-Chlordane	107 ± 2	81.8 (2.7)	82 (1)
cis-Nonachlor	111 ± 3	150 (5)	137 (5)
trans-Nonachlor	648 ± 25	503 (19)	600 (9)
Dieldrin	115 ± 3	95.1 (3.6)	90 (4)
Mirex	74.5 ± 0.9	33.0 (9.9)	83 (4)

[a] NIST values are the means of the results from three analytical methods with uncertainties expressed as 95% confidence intervals (see Wise et al., 1993). Six subsamples were analyzed using each of the three analytical methods.

[b] Five subsamples analyzed, one from each of three bottles and two from one bottle. Concentration value is the mean value, and the numbers in parentheses are one standard deviation of a single measurement.

[c] One subsample from each of the three bottles analyzed. Concentration value is the mean value, and the numbers in parentheses represent one standard deviation of a single measurement.

[d] inf = interference and NA = not analyzed.

[e] Values in [ ] indicate known coelution of two or more congeners; PCB 66 coeluted with PCB 95.

[f] PCB 163 (2,3,3',4',5,6-hexachlorobiphenyl) coeluted with PCB 138 for each of the methods.

As was the case for the *Whale Blubber Control Material*, the analytical data of the three laboratories were generally in good agreement for the beluga whale blubber and SRM 1588 (i.e., the concentrations of 95% of the compounds measured agreed within 25%). The ECD calibrations and the chromatographic separations on the different columns probably explain the majority of the differences observed among the three laboratories.

In 1993, two intercomparison exercises were initiated. The first exercise focused on determining PCB congeners and chlorinated pesticides in blubber tissue using the blubber homogenate prepared as a proposed SRM (see discussion below). This material was distributed to NWFSC and several other laboratories for analysis (i.e., DFO Canada, GERG, Northwestern Aquatic Sciences in Newport, OR, Arthur D. Little in Cambridge, MA, and University of Ulm). The second QA activity involved a "sample split" between NIST and NWFSC of blubber and liver samples from three marine mammals for both inorganic and organic analyses. These tissue samples were from specimens collected as part of the tissue banking and monitoring components of the program. In the past, for many of the liver and blubber specimens in the NMMTB, similar tissue samples were collected using NWFSC protocols and shipped to NWFSC for analysis as part of the monitoring effort. For this intercomparison exercise, NIST homogenized the selected liver and blubber samples from the tissue bank and provided a subsample of each of the homogenates to NWFSC. NIST and NWFSC analyzed subsamples of the tissue homogenates from the bank (liver for trace elements and blubber for PCBs and pesticides). The NWFSC analyzed the "monitoring" liver and blubber samples from the same animals. This provided an assessment of interlaboratory comparability on the same tissue homogenates and the comparability of the analyses of banked specimens and the monitoring specimens collected from the same animals. This QA activitiy was an informal exercise only; the results have not been published.

In 1997, an interlaboratory comparison exercise on trace elements in beluga whale liver sample splits was initiated between NIST, NWFSC, and the Department of Veterinary Anatomy and Public Health at Texas A&M University. All three laboratories are involved in analyzing marine mammal tissues collected in the Beaufort and Chukchi seas of Alaska. The latter laboratory is under contract to the North Slope Borough Department of Wildlife Management (NSB-DWM), Barrow, AK, to analyze tissues for heavy metals from a wide variety of Alaska wildlife used for food in villages of the North Slope Borough. It is at the request of the NSB-DWM that the Texas A&M laboratory be a participant in the marine mammal quality assurance program. For this exercise, sample splits of beluga whale liver tissues collected during the 1996 subsistence hunts at Point Lay, AK, were provided to the participants, as well as the pilot whale liver control material (*Whale Liver Homogenate I*) and a new beluga whale liver control material (*Whale Liver Homogenate II*) which is being considered as a candidate for the development of a marine mammal liver SRM (see discussion below).

Development of Standard Reference Materials (SRMs)

One of the goals of the Marine Mammal QA program is to develop certified reference materials (CRMs) for use in validating analytical measurements of trace elements and organic contaminants in marine mammal tissues. At the beginning of the program, there were several CRMs available from NIST, the National Research Council Canada (Ottawa, Canada), or the Community Bureau of Reference (Brussels, Belgium) for inorganic contaminants in marine tissues including oyster tissue,

mussel tissue, fish muscle and liver tissue, and lobster tomalley. However, for organic contaminants such as PCB congeners and chlorinated pesticides, there were only mussel tissue and cod fish liver oil. Because of this lack of organic reference materials, blubber was selected as the first priority tissue and liver as the second priority tissue for development of SRMs as part of the Marine Mammal QA Program.

SRM 1945, Organics in Whale Blubber.

The experience gained from the preparation and analysis of the pilot whale blubber control material (Wise et al., 1993) was used to develop SRM 1945 Organics in Whale Blubber, a certified material that can be used for validating measurements of organic contaminants in marine mammal blubber. Before the development of SRM 1945 (certified in 1994), there were only two biological matrix SRMs available from NIST with certified and noncertified concentrations for PCBs and chlorinated pesticides: SRM 1588 Organics in Cod Liver Oil and SRM 1974 Organics in Mussel Tissue (*Mytilus edulis*). SRM 1588, which serves as an excellent surrogate for a tissue extract with a high lipid content, has certified concentrations for 5 PCB congeners and 10 chlorinated pesticides and noncertified values for 20 additional PCB congeners and 4 additional chlorinated pesticides (Schantz et al 1992). The mussel tissue SRM has noncertified concentrations for 13 PCB congeners and 9 chlorinated pesticides.

In September 1991 approximately 15 kg of blubber were collected from a stranding of pilot whales on Cape Cod, MA. The material was cryogenically pulverized and homogenized in the same manner as described for the control materials. The resulting frozen blubber homogenate was analyzed using three different analytical techniques based on GC-ECD on two stationary phases with different selectivity for the separation of PCB congeners and GC-MS. The results of these three techniques were in good agreement and were combined to provide certified concentrations for 27 PCB congeners (Table 11) and 15 chlorinated pesticides (Table 12). Noncertified values for two additional PCB congeners (PCB 28 and 31) and chlorinated pesticides (dieldrin and b-HCH) are also available.

Analytical data for the certification of PCBs and chlorinated pesticides in SRM 1945 was published by Schantz et al. (1995), a copy of which is provided in Appendix H. SRM 1945, which represents the most highly characterized natural-matrix SRM with respect to these organic compounds, will complement the other frozen tissue SRM (SRM 1974 Mussel Tissue) by providing concentrations that are generally a factor of 10 to 100 times higher for the PCB congeners and chlorinated pesticides. Solvent extraction of the whale blubber produces an oil matrix similar to the cod liver oil SRM; however, the concentrations of PCB congeners and pesticides in SRM 1945 are generally 2 to 3 times lower than the NIST cod liver oil SRM except for the PCB congeners with higher degrees of chlorination, which have concentrations similar to the cod liver oil.

The low relative uncertainties associated with the majority of analyte concentrations (5 to 10%) and the extensive list of certified analytes makes SRM 1945 a valuable resource for validating analytical methods for the determination of PCB congeners and chlorinated pesticides in marine mammal blubber and other high lipid-containing materials. A copy of the Certificate of Analysis for SRM 1945 Organics in Whale Blubber is found in Appendix I.

Table 11. Certified concentrations of PCB congeners in SRM 1945. From Certificate of Analysis, SRM 1945 Organics in Whale Blubber, June 19, 1994, NIST, Gaithersburg, MD.

Polychlorinated Biphenyls[a]		Concentration ($ng \cdot g^{-1}$ wet mass)[b]		
PCB 18	(2,2',5-Trichlorobiphenyl)	4.48	±	0.88
PCB 44	(2,2'3,5'-Tetrachlorobiphenyl)	12.2	±	1.4
PCB 49	(2,2',4,5'-Tetrachlorobiphenyl)	20.8	±	2.8
PCB 52	(2,2',5,5'-Tetrachlorobiphenyl)	43.6	±	2.5
PCB 66	(2,3',4,4'-Tetrachlorobiphenyl)	23.6	±	1.6
PCB 87	(2,2',3,4,5'-Pentachlorobiphenyl)	16.7	±	1.4
PCB 95	(2,2',3,5',6-Pentachlorobiphenyl)	33.8	±	1.7
PCB 99	(2,2'4,4',5-Pentachlorobiphenyl)	45.4	±	5.4
PCB 101	(2,2'4,5,5'-Pentachlorobiphenyl)	65.2	±	5.6
90	(2,2'3,4',5-Pentachlorobiphenyl)			
PCB 105	(2,3,3'4,4'-Pentachlorobiphenyl)	30.1	±	2.3
PCB 110	(2,3,3',4'6-Pentachlorobiphenyl)	23.3	±	4.0
PCB 118	(2,3',4,4'5-Pentachlorobiphenyl)	74.6	±	5.1
PCB 128	(2,2',3,3',4,4'-Hexachlorobiphenyl)	23.7	±	1.7
PCB 138	(2,2',3,4,4',5'-Hexachlorobiphenyl)	131.5	±	7.4
163	(2,3,3',4',5,6-Hexachlorobiphenyl)			
164	(2,3,3',4',5',6-Hexachlorobiphenyl)			
PCB 149	(2,2',3,4',5',6-Hexachlorobiphenyl)	106.6	±	8.4
PCB 151	(2,2',3,5,5',6-Hexachlorobiphenyl)	28.7	±	5.2
PCB 153	(2,2',4,4',5,5'-Hexachlorobiphenyl)	213	±	19
PCB 156	(2,3,3',4,4',5-Hexachlorobiphenyl)	10.3	±	1.1
PCB 170	(2,2',3,3',4,4',5-Heptachlorobiphenyl)	40.6	±	2.6
190	(2,3,3',4,4',5,6-Heptachlorobiphenyl)			
PCB 180	(2,2',3,4,4',5,5'-Heptachlorobiphenyl)	106.7	±	5.3
PCB 183	(2,2',3,4,4',5',6'-Heptachlorobiphenyl)	36.6	±	4.1
PCB 187	(2,2'3,4',5,5',6-Heptachlorobiphenyl)	105.1	±	9.1
PCB 194	(2,2',3,3',4,4',5,5'-Octachlorobiphenyl)	39.6	±	2.5
PCB 195	(2,2',3,3',4,4',5,6-Octachlorobiphenyl)	17.7	±	4.3
PCB 201	(2,2',3,3',4,5',6,6'-Octachlorobiphenyl)	16.96	±	0.89
PCB 206	(2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl)	31.1	±	2.7
PCB 209	(Decachlorobiphenyl)	10.6	±	1.1

[a] When two or more congeners are known to coelute, the PCB congener listed first is the major component and the additional congeners may be present as minor components. The quantitative results are based on the response of the congener listed first.

[b] The certified values are weighted means of results from three analytical techniques as described by Schiller and Eberhardt (1991). The uncertainty is based on a 95% confidence interval for the true concentration and includes an allowance for differences between the analytical methods used.

Table 12. Certified concentrations of chlorinated pesticides in SRM 1945. From Certificate of Analysis, SRM 1945 Organics in Whale Blubber, June 19, 1994, NIST, Gaithersburg, MD.

Chlorinated Pesticides	Concentration ($ng \cdot g^{-1}$ wet mass)[a]		
Hexachlorobenzene	32.9	±	1.7
α-HCH	16.2	±	3.4
γ-HCH	3.30	±	0.81
Heptachlor epoxide	10.8	±	1.3
Oxychlordane	19.8	±	1.9
Mirex	28.9	±	2.8
cis-Chlordane (a-Chlordane)	46.9	±	2.8
cis-Nonachlor	48.7	±	7.6
trans-Nonachlor	231	±	11
2,4'-DDE	12.28	±	0.87
4,4'-DDE	445	±	37
2,4'-DDD	18.1	±	2.8
4,4'-DDD	133	±	10
2,4'-DDT	106	±	14
4,4'-DDT	245	±	15

[a] The certified values are weighted means of results from three analytical techniques as described by Schiller and Eberhardt (1991). The uncertainty is based on a 95% confidence interval for the true concentration and includes an allowance for differences between the analytical methods used.

Proposed Whale Liver SRM

At present the only liver tissue SRM available from NIST is SRM 1577b, Bovine Liver. For many elements, the concentration levels found in the whale liver control material are similar to the levels found in SRM 1577b. However, for As, Se, Ag, Cd, and Hg, which are elements associated with toxic effects, the concentrations in the liver control material are much greater (orders of magnitude in some cases) than the concentrations in the bovine liver SRM. The concentrations of these elements are comparable to the levels that have been observed in liver tissues of other marine mammals (Becker et al. 1992). Thus, for analyses of these environmentally important elements in marine mammal tissues, a whale liver reference material is a more appropriate material than SRM 1577b.

Another advantage to the proposed whale liver SRM similar to the liver control material homogenate would be the frozen tissue matrix. The majority of trace element tissue reference materials available from NIST and other producers of reference materials are distributed as lyophilized (freeze-dried), defatted extracts, or partially cooked matrices. The physical characteristics of these materials are,

in many instances, significantly different from the sample matrix actually analyzed, thus the value of these materials as quality control samples is often limited. NIST has previously issued a frozen tissue matrix SRM certified for organic contaminants, SRM 1974 Organics in Mussel Tissue (*Mytilus edulis*), which has noncertified concentrations for 36 trace elements. The availability of a frozen whale liver tissue SRM with certified concentrations for trace elements would be of great use to inorganic analysts involved in the analysis of marine mammal tissues.

In the summer of 1996, liver tissue (30 kg) was collected from beluga whales taken in an Alaska Native subsistence hunt at Point Lay, AK, and transported to NIST for use as a potential marine mammal liver SRM. A subsample of this material (5 kg) is now being used as a control material (*Whale Liver Homogenate II*). It was distributed in 1997 to three laboratories participating in an interlaboratory comparison exercise on trace elements in marine mammal liver, i.e., NIST, NWFSC, and Texas A&M University Department of Veterinary Anatomy and Public Health. This exercise is described above and is listed in Table 8.

CONCLUSIONS

Of the 760 tissue specimens archived in the NMMTB over the last few years, only a small part of this collection has been analyzed. Although limited, this database has been used for MMHSRP program development and for determining baseline concentrations of trace elements and chlorinated hydrocarbons in the banked specimens. The banked marine mammal specimens are proving to be very useful in establishing a database on organic compounds and inorganic substances in animals from widely separate geographical areas. Many of these species feed at the same trophic level and occupy similar habitats, even though they occur in very different climatic regions. As the database continues to grow, it will provide a valuable resource for investigations of input of anthropogenic contaminants to the marine environment as well as determining the "normal" levels of many naturally occurring, essential and nonessential (and sometimes toxic) elements.

The Quality Assurance program is an important part of the Marine Mammal Health and Stranding Response Program's effort to obtain reliable information on contaminant levels in marine mammals. Reference materials derived from this program and the interlaboratory comparison exercises are providing an important means of ensuring the comparability and accuracy of analytical results being generated by laboratories analyzing marine mammal tissues.

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APPENDIX A

NMMTB SPECIMEN ACCESS POLICY

(From Becker et al., 1994)

The goal of the National Marine Mammal Tissue Bank (NMMTB) is to maintain quality controlled marine mammal tissues to permit retrospective analyses for the purpose of determining environmental trends and conducting analyses using new and innovative analytical techniques. The NMFS Office of Protected Resources, Silver Spring, Maryland, has the responsibility for managing the NMMTB. NMMTB tissues are banked in the National Institute of Standards and Technology's National Biomonitoring Specimen Bank, Gaithersburg, Maryland.

Duplicate samples (denoted A and B) of ca.150 g for each tissue specimen are banked in the NMMTB. Subsamples of the "B" samples can be homogenized and aliquoted into approximately 20 subsamples of 6 to 8 g, each, which are then available for scientific research. The "A" samples will remain as bulk samples and will only be homogenized after all portions from the corresponding "B" samples are depleted and sufficient justification exists to homogenize the remaining material. Thus, 50% of each specimen is available to the scientific community for research and scientific evaluations consistent with the goals of the NMMTB and 50% is intended for long-term storage as a more permanent archive for posterity.

The homogenized subsamples of "B" samples are divided into three categories. Category 1 constitutes 40% of the homogenized material and is available to the scientific community for research that is consistent with the goals of the NMMTB. Formal requests for access to these tissues must be made as described below. Category 2 consists of 50% of the homogenized material and is reserved for use by the contributing agencies. A procedure for accessing these samples will be established by the contributing agencies and the NMMTB. Category 3 is the remaining 10% of the homogenized material, which is reserved for baseline analyses of the specimens to monitor storage stability, to compare with analyses from real-time monitoring programs, and to conduct research investigations within the NMMTB program.

If an "A" sample is eventually homogenized, the resulting subsamples are divided into the following four categories. Category 1 consists of 25% of the material and is available to the scientific community as described above. Category 2 consists of 25% of the material and is reserved for use by the contributing agencies. Category 3 consists of 10% of the material for use within the NMMTB programs as described above. Category 4 is the remaining 40%, which is intended as a permanent archive that will not be utilized unless high priority needs exist; the determination of this need will be made by an advisory committee to the NMMTB. Combining the "A" and "B" samples, the specimen allocations for each use are as follows: Category 1 = 32.5%, Category 2 = 37.5%, Category 3 = 10%, and Category 4 = 20%. For clarification, the relative amounts of each category are illustrated in the following table.

Formal requests for banked tissues in Category 1 must be submitted to the Director of the NMFS Office of Protected Resources. The request will be reviewed by an informal review committee consisting of no less than three individuals. The review committee will be convened by the Director

and shall have representation from both the government and academic/private sector. At least one member of the review committee shall be an expert in the field that is related to the proposed research activity. The Director will make the final decision based on the advice provided by the review committee.

Relative Amounts of Tissue for Each NMMTB Category

Use	Sample A = 150 g		Sample B = 150 g	
	Weight (g)	Portion of Sample	Weight (g)	Portion of Sample
Scientific Community	37.5	25%	60	40%
Contributing Agency	37.5	25%	75	50%
Long-Term Archival	60	40%	0	0%
Baseline Analysis	15	10%	15	10%

Requests for samples from the NMMTB must include a clear and concise statement of the proposed work and be consistent with the goals of the NMMTB. The following specific information should be included in the request for samples:

1. Name of principal investigator and affiliated research or academic organization,
2. Specific tissue sample and quantity desired,
3. Explanation of proposed research to be conducted,
4. Justification for use of banked tissue,
5. Research facility where analyses will be conducted,
6. Analytical quality control procedures to be used,
7. Estimated date for completion of research, and schedule/date of subsequent reports,
8. Agreement that all results/findings be reported to the NMMTB,
9. Agreement that credit and acknowledgment will be given to NMFS and the NMMTB for use of banked tissues.

Of particular importance in determining whether a sample request will be granted is the justification that samples from the NMMTB are required to accomplish the research and that suitable samples to accomplish the goals of the proposed research could not be obtained from other sources. The NMMTB is not intended to be used as a readily accessible source of marine mammal tissue for any researcher needing specimens, but only for research requiring banked samples from the past.

An informal review process will also be set up for banked tissues withdrawn from Category 2; however, the final decision on Category 2 samples will be made by the contributing agency. Use of Category 3 tissues will be determined by NMFS and NIST. Category 4 tissues cannot be withdrawn until all tissues from the other categories have been depleted. When Category 4 tissues are withdrawn, the same review process will be followed as described for Category 1 tissues with the final determination made by the Director.

Formal requests shall be submitted to the following address:

Director
Office of Protected Resources (F/PR)
NMFS
U.S. Department of Commerce
1315 East-West Highway
Silver Spring, MD 20910

REFERENCE

Becker, P.R., D. Wilkinson, and T.I. Lillestolen, Marine Mammal Health and Stranding Response Program: Program Development Plan, **NOAA Technical Memorandum NMFS-OPR-94-2**, National Oceanic and Atmospheric Administration, Silver Spring, Maryland (1994).

APPENDIX B

SAMPLING PROTOCOLS AND PROCEDURES

(From Becker et al., 1994)

B.1. Tissue Collection

The collection protocols for the NMMTB are modifications of those used by the NBSB for the collection of human liver samples (Harrison et al., 1981; Zeisler et al., 1983a), protocols used by NOAA's National Status and Trends program for the collection of fish, bivalves, and sediment samples (Lauenstein, 1986; Lauenstein and Young, 1986) and protocols used by NOAA's Alaska Marine Mammal Tissue Archival Project (Becker et al., 1988). Within the limitations imposed by the conditions under which collections are made, the intent of the protocol is to obtain fresh, well-defined tissue samples uncontaminated by extraneous sources of trace elements and organic compounds, and to package and transport these samples as quickly as possible under conditions that eliminate or minimize tissue degradation prior to storage. Sample storage and inventory procedures are in accordance with those routinely performed at the NBSB, including specimen storage under liquid nitrogen vapor at -150 °C.

B.1.1. Materials Required

The following materials are used for collecting marine mammal tissue samples. These materials, except where noted, will be provided by the NBSB.

Bytac sheeting
Valeron sheeting;
Dry shippers (LN_2 cooled) with shipping containers;
*LN_2 in container for freezing samples on site;
Lab coats (disposable);
Insulated gloves, safety glasses, and tongs for handling the LN_2 and frozen samples;
*Balance for weighing samples;
Surgical scissors, forceps;
Labels for exterior of sample jars;
Tape for securing exterior jar labels;
Lid labels;
Shipping labels;
NBSB Sampling Data Forms.

In addition, the collection of two replicates of a single sample requires:

4 pairs non-talced vinyl gloves,
1 35.6 cm x 15.2 cm (14" x 16") Teflon FEP (Fluorinated ethylene propylene) bag or sheet to provide a clean working surfaces,
1 30.5 cm x 30.5 cm (12" x 12") Teflon FEP bag for transporting samples from the field to the processing facilities,

2 Teflon jars (180 mL, 49 mm diameter, 120 mm length) with lid and jar labels,
*1 4L bottle containing high purity distilled (HP) water or best available water for rinsing samples,
1 Titanium blade/Teflon TFE (Tetrafluoroethylene) handle knife, and
*1 4L bottle high grade ethanol for rinsing knife.

*Supplied by facility performing the necropsy.

B.1.2. Sample Collection Protocol

The sample collection protocol consists of three stages: tissue removal from the individual animal, tissue processing to obtain representative samples, and packaging/shipping of the samples to the archive. The division of the protocol into stages is used as an aid in organizing and simplifying the collection procedures.

Stage I, tissue removal, may occur under conditions of limited control. Procedures for tissue removal will vary depending upon the group of mammals being sampled, but should be the same for individuals of the same species.

Stage II, tissue processing, will occur under laboratory conditions where possible; this includes shipboard laboratories; however, the processing of tissues might vary depending on the availability of laboratory facilities near the collection site and the tissue type to be sampled.

Stage III, sample packaging and shipping, should be relatively standard for all tissues and should not vary.

Standard forms for recording information pertinent to the sample collections are presented in Appendix C.

Stage I, Tissue Removal

1. Basic Considerations for all Sampling

   a. The size of the tissue sample removed from an animal should be sufficient to provide two subsamples of 150 to 200 g each for archival. This quantity of material is required to provide sufficient material for long-term storage as well as aliquots for periodic analysis.

   b. The anatomical location of the tissue removal is specified in order to maintain consistency and comparability between the same tissue types.

   c. Clean, non-talced vinyl gloves are used by all personnel involved in sample removal and handling. Caution must be taken throughout the procedures to reduce the risk of chemical contamination of the sample. Contamination may originate from the individual performing the work (do not smoke during the procedure), the atmosphere, the skin of

the animal, the instruments used in the dissection, and any chemicals that happen to be in the area where the work is being performed.

d. No animal will be considered a candidate for sampling if tissues cannot be collected within the specified time after death and if handling of the animal has not followed protocol criteria or can not be documented.

2. Procedures for Cetaceans (Fay, et al. 1979):

   a. Record information on location, time taken, and other pertinent data on the individual animal on the NBSB Sampling Data Form (Appendix C)

   b. Make and record all measurements required on the Sampling Data Form.

   c. With the animal lying on its side, confirm the gender.

   d. Using a stainless steel instrument, make an incision through the skin and blubber on the dorsal side, anterior to the dorsal fin and posterior to the blow hole and measure the blubber thickness (mm).

3. Remove the tissue samples as soon as possible after opening the body cavity. Opening of the body cavity and initial cutting of the skin to expose adipose and muscle tissue should be performed with high quality stainless steel dissection tools previously rinsed in the high purity water and ethanol.

   a. **Adipose Tissue (Blubber)**. The anatomical site of blubber removal will depend on the distribution of fat layers on the animal; therefore, it will be rather species specific.

      For cetaceans, make an incision anterior to the dorsal fin and posterior to the blow hole with a stainless steel implement. Make two more cuts perpendicular to the first incision near the fin and blow hole respectively on the left side of the animal (i.e., a three-sided flap of skin is cut). Using the titanium knife and grasping a corner of the blubber with hemostat forceps, separate a section of blubber from the muscle layer that is below the blubber. After the blubber is separated from the muscle, separate the blubber from the skin in the same manner. A total of 300 g of blubber are required for storage. Place the blubber in a clean Teflon bag for immediate transport to the processing area.

   b. **Liver**. Note the general appearance of the liver before removal, including any unusual coloration, texture, shape, etc.

      For cetaceans: preferably using a stainless steel instrument, remove the entire liver and place it in a clean Teflon bag. However, if the entire liver can not be removed, use a titanium knife to remove a 150 to 200 g section from both sides of the medial indentation at the distal end of the liver and place in a clean Teflon bag for immediate transport to the processing area.

c. **Kidney**. Note the general appearance of the kidney before removal, including any unusual coloration, texture, shape, etc.

(1) For the smaller species (seals and sea otter): Remove both kidneys from the animal and place in a clean Teflon bag for immediate transport to the processing area. Both kidneys are required in order to provide a total sample size of 300 g. (Note the left and the right kidney). Attachments of the kidney may be cut using surgical scissors that have been previously rinsed. If the kidney membrane is ruptured, the sample is not acceptable.

(2) For the larger species (sea lion, seal, and cetaceans): Remove the left kidney from the animal and place in a clean Teflon bag for immediate transport to the processing area. Attachments of the kidney may be cut using surgical scissors that have been previously rinsed. If the kidney membrane is ruptured, the sample is not acceptable.

If the entire kidney is too large to be transported back to the processing area, remove a 300 to 400 g section from the posterior end of the left kidney using the titanium knife and place in a clean Teflon bag for transport.

4. Following the removal of the NBSB samples, any additional samples and measurements that are taken should be included on the NBSB Sampling Data Form.

Stage II, Tissue Processing

1. Tissue processing should take place within a laboratory facility under the cleanest conditions available. At a minimum, the tissue processing should take place in a covered and enclosed area free of obvious sources of contamination such as cigarette smoke, fuel oil fumes and smoke, laboratory formaldehyde, etc. The processing area of the laboratory should be cleaned to remove dust and the working surfaces covered with Teflon surface sheeting.

2. Only titanium knives are to be used to cut samples during Stage II. Two knife cleaning procedures are presented in Sections B.2.1 and B.2.2. Cleaning Procedure I can be performed at the processing site. Procedure II will be performed at the NIST facilities, or other suitable laboratories.

3. If tissue sections are taken for histological work, this information is recorded in the appropriate space on the data reporting forms. The results of the histological analysis should be reported to the NBSB. Any change in the physical location of the histological slides should be reported to the NBSB. Investigators who intend to dispose of histological slides after analysis are encouraged to transfer these materials to the NBSB, where they will be cataloged, cross-referenced to archived tissue samples, and maintained in the NIST archives.

4. **Liver**

a. Tare the weight of two pre-cleaned Teflon jars, record that weight, and label the jar for the sample.

b. Remove the sample from the Teflon bag. Holding the sample over a sink, rinse the surface of the specimen with HP water. Pour approximately 100 mL or more of the HP water from the bottle over the specimen to wash off blood and other fluid. Rub the specimen with gloved hand, if necessary, to remove blood, etc. Allow the specimen to drain for several minutes. Place the rinsed specimen on a clean Teflon sheet.

c. If the whole liver is transported to the processing facility, remove 300 g from the distal end of both sides of the medial indentation using a titanium knife. Dissect the 300 g portion into two equal parts and place one section in jar A and the other section in jar B. Each sample will fit in a Teflon jar with a volume of 180 mL (49 mm diameter and 120 mm length).

d. If the liver sample is 300 g to 400 g, use the titanium knife to divide the specimen into two equal samples (Sample A and Sample B) of 150 g to 200 g each and trim the edges that came in contact with other materials. Each sample must fit in a Teflon jar with a volume of 180 mL (49 mm diameter and 120 mm length).

e. After the sample has been dissected and placed in the Teflon jar, weigh the jar and record the tared weight.

f. When the work on the sample has been completed, rinse the titanium knife with the HP water and rub with the gloved fingers to remove all blood and fluids from the knife before they have time to dry. Rinse the knife with ethanol and air dry.

g. Continue to Stage III, Tissue Packaging and Shipping.

5. **Kidney**

a. Tare the weights of two Teflon jars.

b. Remove the specimen from the Teflon bag, rinse the surface of each kidney with HP water. Pour approximately 100 mL or more of the water over each kidney to wash off blood and other fluid. Rub the specimen with gloved hand, if necessary, to remove blood, etc. Allow the kidneys to drain for several minutes.

c. In some cases each kidney will approach the weight of the required specimen size for each subsample (150 g) and no subsampling will be necessary. The right kidney will provide Sample A and the left kidney will provide Sample B. If subsampling is required (or if cutting of the sample is necessary to fit the sample into the jar), use the titanium knife to remove a 150 g section from the kidney. Each sample will fit in a Teflon jar with a volume of 180 mL (49 mm diameter and 120 mm length).

d. If the left whole kidney was collected from the animal or if samples of kidney tissue were collected, using the titanium knife, remove two subsamples of 150 g each from the kidney sample and trim the edges that came in contact with other materials. Each subsample will fit in a Teflon jar with a volume of 180 mL (49 mm diameter and 120 mm length).

e. After the sample has been dissected and placed in the Teflon jar, weigh the jar and record the tared weight.

f. Rinse the titanium knife with the high purity water and rub with the gloved fingers to remove all blood and fluids from the knife before they have time to dry. Rinse the knife with ethanol and air dry.

g. Continue to Stage III, Tissue Packaging and Shipping.

6. **Blubber**

a. Tare the weight of two pre-cleaned Teflon jars, record those weights, and label the jars for the sample.

b. Remove the specimen from the Teflon bag; pour approximately 100 mL or more of HP water over the specimen to wash off blood and other fluid. Allow the specimen to drain for several minutes.

c. Use the titanium knife to remove any remaining portions of muscle or skin attached to the blubber and to trim the edges that were cut with the stainless steel implements. Also, any tissue that came in contact with a contaminant, i. e., animal hair, hemostats, etc., should be excised and discarded. Excise a 300 g to 400 g section from the specimen.

d. Using a clean titanium knife divide the specimen into two equal samples (Sample A and Sample B) of 150-200 g each. Each sample will fit in a Teflon jar with a volume of 180 mL (49 mm diameter and 120 mm length).

e. After the sample has been dissected and placed in the Teflon jar, weigh the jar and record the tared weight.

f. Rinse the titanium knife with HP water and rub with the gloved fingers to remove all blood and fluids from the knife before they have time to dry. Rinse the knife with ethanol and air dry.

g. Continue to Stage III, Tissue Packaging and Shipping.

Stage III, Tissue Packaging and Shipping

1. Record the sample weights on the NBSB Sampling Data Form and sample labels. Affix the sample label to the side of each of the two jars with wide tape, which must be wrapped on itself at least three times, and place the lid label in the recessed slot on the jar lid.

2. Freeze each sample by immersing in LN_2 for 10 minutes. If LN_2 is not immediately available, freeze sample in freezer at -70 °C or lower.

Liquid nitrogen should not be stored in sealed containers. Personnel handling LN_2 are cautioned to wear boots, cuffless trousers, non-absorbent aprons, loose insulating gloves, and safety glasses.

3. Prior to shipping, the LN_2 dry shipper should be filled with liquid nitrogen for at least 6 hours. This is required to fully saturate the absorbent inside the shipper. Pour off the excess LN_2 and place the frozen samples in the shipper.

4. Once the dry shipper is full, transport it to the NIST; do not store the samples in intermediate freezers.

5. Double check the NBSB Sampling Data Forms for completeness and accuracy. Any deviations or modifications of the protocol must be noted on the sampling form.

6. Place a copy of the completed forms in the shipping container that holds the dry shipper.

7. Samples should be shipped to NIST within 48 hours or as soon as possible after sample collection using 24 hour express package service to:

National Institute of Standards and Technology
Clopper Road and Quince Orchard Road
Building 235, Room B125
Gaithersburg, Maryland 20899
Attn: Barbara Porter (301) 975-6291

The dry shippers must not contain excess LN_2 when shipped, i.e., all LN_2 must be absorbed or poured off. Maximum holding time for the shippers is 10 to 12 days. Shipping expenses will be borne by NIST. Do not ship late in the week, i.e., Thursday or Friday, or before holidays, unless special arrangements have been made with the shipping service and NIST.

8. The Specimen Bank personnel should be notified by telephone as soon as possible after the specimens are shipped:

Barb Porter	(301) 975-6291 or
Steve Wise	(301) 975-3112

B.2. Titanium Knife Cleaning Procedure

There are two cleaning procedures for the titanium knives and other titanium reusable implements: the first is to be completed after processing a sample, and the second is used after sharpening or after excessive contamination. The titanium knives should be sharpened only with the silicon carbide stone provided and only these knives should be sharpened with the stone.

B.2.1 Procedure I

After placing tissue sections in bags and before leaving the sample preparation area, the knife should be rinsed using HP water. While rinsing and with gloved hands, run fingers and lint-free cloths over the blade and handle of the knife to help remove any adhering blood or tissue. This is best done before any fluid or tissue has a chance to dry on the knife. In the laboratory, the knife should be rinsed again, as above, with water and then with ethanol. The knife should then be placed on a clean surface (do not touch the blade) and allowed to air dry, preferably in a laminar flow hood. The knife should then be placed in a Teflon bag for storage and transport to the next sampling site. The implement should at no time be touched with ungloved hands.

B.2.2. Procedure II

This procedure should be applied after excessive contamination of the implements and always after a knife is sharpened. Rinse the implement as described in cleaning procedure I. In the laboratory, the implement is placed in a clean container and covered with 99.5% ethyl alcohol for 1 h to 2 h. The implement is then covered with HP water overnight. The implement is covered with a 1:10 dilution of hydrochloric acid for 30 min (made up using one part hydrochloric acid and ten parts HP water). The implement is rinsed with HP water then covered with 1:10 dilution of nitric acid for 30 min (made up using one part nitric acid and ten parts HP water). The implement is again rinsed with HP water. The knife may be disassembled to clean if necessary. The implement is removed from the washing container and placed on a clean surface to air dry, preferably under a HEPA filter. Only the knife handle should touch the drying surface. The clean, dry implement should be stored in a Teflon bag.

B.3. References

Becker, P.R., D. Wilkinson, and T.I. Lillestolen, Marine Mammal Health and Stranding Response Program: Program Development Plan, **NOAA Technical Memorandum NMFS-OPR-94-2**, National Oceanic and Atmospheric Administration, Silver Spring, Maryland (1994).

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Fay, F.H., L.M. Shults, and R.A. Dieterich, A Field Manual of Procedures for Postmortem Examination of Alaskan Marine Mammals, **IMS Report Number 79-1** and **IAB Occasional Publication on Northern Life No. 3**, University of Alaska, Fairbanks, Alaska (1979).

Harrison, S.H., R. Zeisler, and S.A. Wise, Editors, Pilot Program for the National Environmental Specimen Bank - Phase I, **EPA-600/1-025**, Health Effects Research Laboratory, USEPA, Research Triangle Park, North Carolina (1981).

Lauenstein, G.G., National Status and Trends Program for Environmental Quality Benthic Surveillance Project: Specimen Banking Project, Ocean Assessments Division, Office of Oceanography and Marine Assessment, National Ocean Service, NOAA, Rockville, Maryland (1986).

Lauenstein, G.G., National Status and Trends Program for Environmental Quality Benthic Surveillance Project: Specimen Banking Project, Ocean Assessments Division, Office of Oceanography and Marine Assessment, National Ocean Service, NOAA, Rockville, Maryland (1986).

Zeisler, R., J.K. Langland, and J.K. Harrison, Cryogenic homogenization procedure for biological tissues, *Anal. Chem.* **60**, 2760-2765 (1983).

APPENDIX C

NBSB SAMPLING DATA FORM

(for the NMMTB)

NATIONAL BIOMONITORING SPECIMEN BANK

Sampling Data

National Marine Mammal Tissue Bank

Animal ID Number _____ Species _____

Sample Source _____

Collection Site ID _____ Lat. _____ Long. _____

Time of Death: day mo yr [ ][ ][ ] hr [ ] Method of Collection _____

Intermediate Storage (temp/remarks) _____

_____

Necropsy Location _____

Sample Type: ☐ Liver ☐ Kidney ☐ Muscle ☐ Blubber ☐ Other _____

Time of Collection: day mo yr [ ][ ][ ] hr [ ] Collected by _____

Intermediate Storage (temp/remarks) _____

_____

Time of Preparation: day mo yr [ ][ ][ ] hr [ ] Processor _____

Time of LN_2 Freezing [ ][ ][ ] [ ]

Time Shipped From Site: [ ][ ][ ] [ ] Shipper _____

Time Received at Archive: [ ][ ][ ] [ ] Receiver _____

Protocol: ☐ Standard ☐ Modified (Please note modification below)

Remarks:

Animal ID Number _____

Condition: ☐ Alive ☐ Freshly dead Other (explain) _____

Sex: ☐ Male ☐ Female Total length ☐ Total weight ☐

Baleen/Tooth counts (erupted or total) UL/LL ☐ UR/LR ☐

Specify Unit of Measurements

Snout to melon	☐	Snout to center of anus	☐
Snout to blow hole	☐	Snout to center of genital aperture	☐
Snout to center of eye	☐	Snout to ant. insertion of flipper	☐
Snout to ant. insertion of fin	☐	Flipper length	☐
Snout to fin tip	☐	Flipper width	☐
Snout to fluke notch	☐	Fluke width	☐
Snout to caudal end of ventral grooves	☐	Fin height	☐

Girth: Axillary ☐ Max (location) ☐ Anal ☐

Blubber thickness (location)

dorsal ☐ lateral ☐ ventral ☐

Parasites (location)

external _____

Internal _____

Stomach contents _____

_____

Reproductive condition: Preg./Lactating/Fetus length _____

Gonad wt. L/R left ☐ right ☐

Animal ID Number _____

Method of Collection _____

Age _____ Method of Age Estimation _____

General Comments:

General Appearance of Individual:

General Appearance of Organs:

Histological Samples:

Individual/Organization _____

Final Destination _____

Tissues Sampled _____

Sample Weights:	A		B			A		B	
Liver		g.		g.	Blubber		g.		g.
Kidney		g.		g.	Other		g.		g.

Prepared by: _____
Name (print)

_____
Signature

A copy of this form should be shipped with samples to:

National Institute of Standards and Technology
Quince Orchard Road
Bldg. 235, Rm B165
Gaithersburg, MD 20899

Attn: Barbara Porter
(301) 975-6291

APPENDIX D

NMMTB SPECIMEN INVENTORY

A detailed inventory of tissue specimens collected for the NMMTB is presented on the following pages. Since the NBSB inventories of tissue specimens for Alaska animals collected by the AMMTAP have been previously published (Becker et al., 1992; Koster et al., 1994), the following inventory does not include Alaska animals. The NMMTB inventory includes the following information: species, meristic information on the animals sampled, sampling location, additional samples taken by other researchers, and the location of these additional samples, as well as other miscellaneous information regarding the individual samples or animal sampled. Those specimens for which one subsample has been homogenized and divided into aliquots for analysis are also identified. The format used to present this data is explained below.

NMMTB Sample Inventory Format

Animal ID No.	**Field ID No.**	**Sampling Date**	**Acquired From**	**Location**	**Latitude/Longitude**
Sex	**Age**	**Total Length** in cm	**Total Weight** in kg	**Fluke Width** in cm	**Axillary Girth** in cm	**Blubber Thickness** in cm
NBSB Samples	**NBSB ID**	**Subsample A weight**	**Subsample B weight**	**Histology**	**Sample Homogenization**
tissue type	number	in grams	in grams	yes or no	date homogenized

Additional samples collected for other researchers:
Organization and ID number, if appropriate
Present location of samples: list of samples

REFERENCES

Becker, P.R., S.A. Wise, M.M. Schantz, B.J. Koster, and R. Zeisler, Alaskan Marine Mammal Tissue Archival Project: Sample Inventory and Results of Analyses of Selected Samples for Organic Compounds and Trace Elements, **NISTIR 4731**, National Institute of Standards and Technology, Gaithersburg, Maryland (1992)

Koster, B.J., S.A. Wise, and P.R. Becker, Alaska Marine Mammal Tissue Archival Project: Specimen Inventory, **NISTIR 5462**, National Institute of Standards and Technology, Gaithersburg, Maryland (1994).

Inventory Contents

PILOT WHALE (*Globicephala melas*)

ANIMAL ID NO.	FIELD ID. NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Gm-001	MH90-597Gm	12 Dec 90	Stranding		Hyannis, MA	
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F		428		50.2x2	105.4x2	5.0
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM1L003	175	158			30 May 91
Blubber	NM1B004	110	110			30 Jun 97

ADDITIONAL SAMPLES COLLECTED:

Blubber, muscle, ovaries (right and left), liver, kidney, heart, and lung for pesticide analyses
Heart for DNA aging

PILOT WHALE (*Globicephala melas*)

ANIMAL ID NO.	FIELD ID. NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Gm-002	MH90-599GM	12 Dec 90	Stranding		Hyannis, MA	
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F		413		47x2	100x2	4.5
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM1L005	142	179		yes	8 May 92
Blubber	NM1B006	106	118			25 Mar 92

ADDITIONAL SAMPLES COLLECTED:

Blubber, muscle, liver, kidney, heart, lung, and spleen for pesticide analyses
Blubber, muscle, liver, kidney, heart, lung, and spleen for histology

PILOT WHALE *(Globicephala melas)*

ANIMAL ID NO.	FIELD ID. NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Gm-003	MH90-600Gm	12 Dec 90	Stranding		Hyannis, MA	
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F		439		52.4x2	109x2	4.3
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM1L007	158	157			13 May 92
Blubber	NM1B008	93	114			1 Apr 92

ADDITIONAL SAMPLES COLLECTED:

Blubber, muscle, ovary (left), liver, kidney, and heart for pesticide analyses
Heart for DNA aging
Serum profile at New England Aquarium

PILOT WHALE *(Globicephala melas)*

ANIMAL ID NO.	FIELD ID. NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Gm-004	MH90-617Gm	12 Dec 90	Stranding		Hyannis, MA	
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F		375		44x2	96x2	3.7
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM1L009	144	133			6 Jun 91
Blubber	NM1B010	112	122			8 Aug 91

ADDITIONAL SAMPLES COLLECTED:

Blubber, muscle, ovaries (right and left), liver, kidney, heart, and lung for pesticide analyses
Heart for DNA aging

PILOT WHALE *(Globicephala melas)*

ANIMAL ID NO.	FIELD ID. NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Gm-005	MH90-624Gm	12 Dec 90	Stranding		Hyannis, MA	
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F		387		46.8x2	100.4x2	5.9
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM1L011	169	158		yes	21 May 92
Blubber	NM1B012	119	125			2 Apr 92

ADDITIONAL SAMPLES COLLECTED:

Blubber, muscle, liver, kidney, heart, and lung for pesticide analyses
Blubber, muscle, stomach, ovary (left), liver, kidney, heart, and lung for histology

PILOT WHALE *(Globicephala melas)*

ANIMAL ID NO.	FIELD ID. NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Gm-006	MH91-602Gm	10 Sept 91	Stranding		Wellfleet, MA	
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F		274		29x2	69x2	2.6
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM1L021	171.9	173.8			4 May 92
Blubber	NM1B022	141	143.2			19 Mar 92

ADDITIONAL SAMPLES COLLECTED:

Eye removal for URI, Dr Winn

PILOT WHALE *(Globicephala melas)*

ANIMAL ID NO.	FIELD ID. NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Gm-007	MH91-603Gm	10 Sept 91	Stranding		Wellfleet, MA	
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F		420		47x2	104x2	4.7
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM1L023	164	160.5			30 Apr 92
Blubber	NM1B024	123.7	136.2			

ADDITIONAL SAMPLES COLLECTED:

Eye removal for URI, Dr. Winn
Animal used at NIST for SRM 1945

PILOT WHALE *(Globicephala melas)*

ANIMAL ID NO.	FIELD ID. NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Gm-008	MH91-604Gm	10 Sept 91	Stranding		Wellfleet, MA	
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M		300		35x2	90x2	3.5
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM1L025	174.1	170.9			5 May 92
Blubber	NM1B026	140.3	135.2			23 May 92

ADDITIONAL SAMPLES COLLECTED:

Eye removal for URI, Dr Winn
Animal used at NIST for SRM 1945

PILOT WHALE *(Globicephala melas)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Gm-009	MH91-605Gm	10 Sept 91	Stranding		Wellfleet, MA	
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M		314		33x2	81x2	3.2
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM1L027	163.2	174.4			30 Dec 93
Blubber	NM1B028	108.2	133.8			

ADDITIONAL SAMPLES COLLECTED:

Eye removal for URI, Dr Winn
Animal used at NIST for SRM 1945

PILOT WHALE *(Globicephala melas)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Gm-010	NMFS #3069	5 July 96	Drift net set		George' Bank Canyon, Atlantic Ocean	67°20'/40°14'
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M		210	136	52	107	17
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM6L061	140.3	109.2			23 Sept 96
Kidney	NM6K062	140.3	119.3			26 Sept 96
Blubber	NM6B063	50.6	62.9			27 Sept 96

ADDITIONAL SAMPLES COLLECTED:

Skull to Smithsonian
Tissue taken for contaminants studies for several agencies
Tissue taken for histopathology studies for several agencies
Parasites taken for Teri Rowles
Video and photographs taken
Lipid samples taken for analysis

PILOT WHALE *(Globicephala melas)*

ANIMAL ID NO.	FIELD ID. NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Gm-011	NMFS #1609	9 Jul 96	Drift net set		George's Bank Canyon, Atlantic Ocean	39°53'/69°28'
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F		324	189.2	71	88x2	44.0
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM6L064	112.8	124.2		yes	30 Sept 96
Kidney	NM6K065	102.2	100.2		yes	4 Oct 96
Blubber	NM6B066	83.6	83.3			8 Oct 96

ADDITIONAL SAMPLES COLLECTED:

Skull to Smithsonian
Tissue taken for contaminant studies for several agencies
Tissue taken for histopathology studies for several agencies
Parasites taken for Teri Rowles
Muscle taken for T Williams

PILOT WHALE *(Globicephala melas)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Gm-012	NMFS # 2090	9 Jul 96	Drift net set		George's Bank Canyon, Atlantic Ocean	39°53'/69°28'
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F		376			98x2	43.0
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM6L067	109.9	134.3		yes	10 Oct 96
Kidney	NM6K068	145.2	136.3		yes	11 Oct 96
Blubber	NM6B069	80.3	77.04			16 Oct 96

ADDITIONAL SAMPLES COLLECTED:

Skull to Smithsonian
Tissue taken for contaminants studies for several agencies
Tissue taken for histopathology studies for several agencies
Parasites taken for Teri Rowles
Urine and biopsy from multiple locations for Woodshole, Moore
Muscle for T Williams

HARBOR PORPOISE *(Phocoena phocoena)*						
ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Pp-001	MH90-553Pp	3 Oct 90	Incidental catch		Portland, ME	43°40' 70°15'
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M	8-10	145	47	38	88	1.1
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM1L001	161	147		yes	8 May 91
Blubber	NM1B002	128	123			21 Aug 91
ADDITIONAL SAMPLES COLLECTED:						
Histology and teeth to New England Aquarium Skeleton to Smithsonian Tissue for toxicology and histology						

HARBOR PORPOISE *(Phocoena phocoena)*						
ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Pp-002	MH91-424Pp	29 Jan 91	Incidential catch		Sciduate Town Pier	
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F	Juvenile	120	31.78	27	71	2
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM1L013	168.4	170.3			20 Apr 92
Blubber	NM1B014	152	138.9			11 Mar 92
ADDITIONAL SAMPLES COLLECTED:						
Tissue samples taken by Jennifer Hogan for NMFS, NWFSC, ECD, Seattle Tissue samples taken by John Nicholas for Woodshole, MA						

HARBOR PORPOISE *(Phocoena phocoena)*						
ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Pp-003	MH91-461Pp	19 Mar 91	Incidental catch		Portland, ME	
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F	3	132	38.59	31	84	2.6
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM1L015	129.7	141.9			23 May 91
Blubber	NM1B016	170.7	131.7			16 Sept 91
ADDITIONAL SAMPLES COLLECTED:						

HARBOR PORPOISE *(Phocoena phocoena)*						
ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Pp-004	MH91-505Pp	9 May 91	Incidental catch		Portland, ME	
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F	1	109	23.61	31	67.5	2.4
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM1L017	171.7	159.7			23 Apr 92
Blubber	NM1B018	148.1	140.9			12 Mar 92
ADDITIONAL SAMPLES COLLECTED:						

HARBOR PORPOISE *(Phocoena phocoena)*						
ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Pp-005	MH91-504Pp	9 May 91	Incidental catch		Portland, ME	
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M	1	121.5	29.51	28	72	2.2
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM1L019	178.6	174.7			29 Apr 92
Blubber	NM1B020	122.9	134.3			13 Mar 92
ADDITIONAL SAMPLES COLLECTED:						
Blubber tissue for Distribution Study at NMFS, Seattle, WA						

HARBOR PORPOISE *(Phocoena phocoena)*						
ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Pp-006	MH92-477Pp	14 Apr 92	Incidental catch		Nantastket Beach	
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F		111	39.50	30	79	2.2
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM1L029	107.7	161.1			25 Oct 94
Blubber	NM1L030	107.7	120.4			
ADDITIONAL SAMPLES COLLECTED:						

HARBOR PORPOISE *(Phocoena phocoena)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Pp-007	MH92-575Pp	20 Oct 92	Incidental catch		Portsmouth, NH	13415, 25812*
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F	1	123	34.05	34	85	2.75
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM2L031	128.5	106.6			25 Jan 93
Blubber	NM2B032	80.6				20 May 93

ADDITIONAL SAMPLES COLLECTED:

* Loran readings

HARBOR PORPOISE *(Phocoena phocoena)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Pp-008	MH92-597Pp	11 Dec 92	Incidental catch		Boothbay, ME	
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M	3	141	49.94	36	85	3
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM2L033	113.14	122.67			30 Sept 94
Blubber	NM2B034	96.4	107.72			

ADDITIONAL SAMPLES COLLECTED:

HARBOR PORPOISE *(Phocoena phocoena)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Pp-009	PJG-126	29 Apr 93	Stranding		Seattle, WA	
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M		152.7	40	36.4	74	1.5
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM3L043	80.78	63.92		yes	
Blubber	NM3B044	81.13	63.96			

ADDITIONAL SAMPLES COLLECTED:

Skull, skeleton, teeth, stomach contents, reproductive organs, blubber, muscle, liver, kidney, heavy metal samples, subcellular genetics, histology, skin, and dorsal fin
Lung for histology and toxicology

HARBOR PORPOISE *(Phocoena phocoena)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Pp-010	MH93-496Pp	13 May 93	Incidental catch		Sea Brook, NH Gulf of Maine	
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F		128	39	32	84	
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM3L045	141.4	140.7			
Blubber	NM3B046	120.7	123.1			

ADDITIONAL SAMPLES COLLECTED:

Photos taken
Liver and blubber for NMFS, NWFSC, ECD, Seattle

HARBOR PORPOISE *(Phocoena phocoena)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Pp-011	MH-97-462Pp	7 Mar 97	Stranding		Winthrop, MA	42°22.84'/70°58.21'
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M	July	128.0	29.5	28	68	2.0
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM7L107	152.2	158.4			
Blubber	NM7B108	148.9				

ADDITIONAL SAMPLES COLLECTED:

Esophagus, lung, and bladder for histology to New England Aquarium

HARBOR PORPOISE *(Phocoena phocoena)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Pp-012	GM-97-19	18 Aug 97	Weir Entanglement		Grand Manan, NB, Canada Gulf of Maine	
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F		123.0	39.4	33.0	80.0	26
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM7L143	92.0	104.0		yes	
Kidney	NM7K144	87.0	114.0		yes	
Blubber	NM7B145	71.0	63.0			

ADDITIONAL SAMPLES COLLECTED:

GM-97-19
Blubber, brain, liver, and muscle for toxicology

HARBOR PORPOISE *(Phocoena phocoena)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Pp-013	GM-97-27Pp	26 Aug 97	Weir Entanglement		Grand Manan, NB, Canada	
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F	Immature	118.0	28.0	26.5	75.5	2.0
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM7L146	90.0	68.0		yes	
Kidney	NM7K147	52.0	70.0		yes	
Blubber	NM7B148	63.0	72.0			

ADDITIONAL SAMPLES COLLECTED:

GM-97-27
Stomach and histology taken

WHITE-SIDED DOLPHIN *(Lagenorhynchus acutus)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
La-001	MH93-445La	23 Mar 93	Stranding		Barnstable, MA	41°44'/70°20'
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F	Adult	191	82	50	94	1.8
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM3L035	131.8	127.97		yes	3 Jan 94
Blubber	NM3B036	82.37	112.93			

ADDITIONAL SAMPLES COLLECTED:

Blubber, muscle, heart, liver, and lung for pesticide analyses
Liver, kidney, brain, lung, adrenal, reproductive organs, lymph nodes, thyroid, and thymus for histology

WHITE-SIDED DOLPHIN *(Lagenorhynchus acutus)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
La-002	MH93-446La	23 Mar 93	Stranding		Barnstable, MA	41°44'/70°20'
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F	Adult	185	79	44	96	1.8
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM3L037	133.26	141.14		yes	6 Jan 94
Blubber	NM3B038	108.5	101.98			

ADDITIONAL SAMPLES COLLECTED:

Blubber, muscle, heart, liver, kidney, and lung for pesticide analyses
Liver, kidney, brain, lung, adrenal, and lymph nodes for histology

WHITE-SIDED DOLPHIN *(Lagenorhynchus acutus)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
La-003	MH93-460La	Apr 93	Stranding		Wellfleet, MA	41°55'20"/70°03'15"
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M		260	190	70	130	2
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM3L039	151.14	165.3		yes	19 May 93
Blubber	NM3B040	115.44	117.6			19 May 93

ADDITIONAL SAMPLES COLLECTED:

Blubber, muscle, heart, liver, kidney, brain, and stomach contents for pesticide analyses
Muscle, heart, liver, kidney, brain, lung, adrenal, reproductive organs, stomach, lymph node, pancreas, and testes for histology
Lung for ISO
Blubber, muscle, heart, liver, kidney, and brain to NMFS
Heart and skin for DNA

WHITE-SIDED DOLPHIN *(Lagenorhynchus acutus)*						
ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
La-004	MH93-461La	Apr 93	Stranding		Wellfleet, MA	41°55'/70°03'
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F		193	73	56	98	1.75
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM3L041	148.88	150.85			7 Jan 94
Blubber	NM3B042	112.08	100.75			

ADDITIONAL SAMPLES COLLECTED:

Blubber, heart, kidney, and brain for pesticide analyses
Blubber, muscle, heart, liver, kidney, brain to NMFS
Liver, skin for DNA

WHITE-SIDED DOLPHIN *(Lagenorhynchus acutus)*						
ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
La-005	MH94-611La	Dec 94	Stranding		Eastham, MA	
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M	Juv	140	99.8	36	104	2
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM4L053	141	142		yes	
Blubber	NM4B054	142	142			

ADDITIONAL SAMPLES COLLECTED:

Blubber, muscle, liver, kidney, and brain for pesticide analyses
Liver, kidney, brain, adrenal, reproductive organs, skin, lymph nodes, and stomach for histology
Brain for ISO

WHITE-SIDED DOLPHIN *(Lagenorhynchus acutus)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
La-006	MH-96-408La	20 Jan 96	Stranding		Wellfleet,MA	41°56/70°01
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M	Adult	228	163	66	124	1.75
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM6L055	150.1	167.3		yes	
Blubber	NM6B056	108.0	97.8			

ADDITIONAL SAMPLES COLLECTED:

WHITE-SIDED DOLPHIN *(Lagenorhynchus acutus)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
La-007	MH97-425La	2 Feb 97	Stranding		Brewster,MA	41°46'/70°03'
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M	Juv	156	~100lbs	36	82	1.5
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM7L095	146.1	144.4		yes	
Blubber	NM7B096	100.7	99.8		yes	

ADDITIONAL SAMPLES COLLECTED:

WHITE-SIDED DOLPHIN *(Lagenorhynchus acutus)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
La-008	MH-97-506La	28 May 97	Stranding		Duxbury, MA	42°03.5/70°40.0
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M		173	56.7	50	94	2.2
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM7L129	162.10	175.96		yes	
Blubber	NM7B130	128.64	115.74			

ADDITIONAL SAMPLES COLLECTED:

Histology taken for New England Aquarium
Entire head taken

WHITE-SIDED DOLPHIN *(Lagenorhynchus acutus)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
La-009	MH97-507La	28 May 97	Stranding		Duxbury, MA	42°03.5/70°40.0
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F	Adult	188	113.4	34	108	2.2
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM7L131	176.94	146.28		yes	
Blubber	NM7B132	118.18	119.20			

ADDITIONAL SAMPLES COLLECTED:

Histology taken for New England Aquarium
Teeth taken for age determination

WHITE-SIDED DOLPHIN *(Lagenorhynchus acutus)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
La-010	MH97-534La	12 Aug 97	stranding		Great Island, MA	41°55'.492/70°04'.213
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F	adult	235		61	119.4	0.8
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM7L133	173.0	155.8			
Kidney	NM7K134	152.5	168.4			
Blubber	NM7B135	83.7	73.7			

ADDITIONAL SAMPLES COLLECTED:

Histology taken for WHOI - Armed Forces Institute of Pathology

ROUGH-TOOTHED DOLPHIN *(Steno bredanensis)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Sb-001	RTDL-001	15 Dec 97	Stranding		St Joseph's State Park, FL	29°45.2/85°25'
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M		183		44	95	
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM8L678	149.0	147.6			
Kidney	NM8K679	129.0	113.4		yes	17 Feb 98
Blubber	NM8B680	79.7	77.5			28 Jan 98

ADDITIONAL SAMPLES COLLECTED:

Field No - GW97SB-85
Adrenals, kidney, aorta, duodenum, pancreas, lung, stomach, and colon for histology to NMFS Maimi Lab, Ruth Ewing
Teeth for age determination to NMFS Miami Lab, Ruth Ewing
Head to Smithsonian, Charlie Potter
Skin and reproductive organs to NOS Charleston Lab, Wayne McFee
Melon and muscle to NMFS, NWFSC, ECD, Seattle
Stomach to Sea World

ROUGH-TOOTHED DOLPHIN *(Steno bredanensis)*						
ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Sb-002	RTDL-002	15 Dec 97	Stranding		St Joseph's State Park, FL	29°45.2/85°25'
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M		229		56	113	
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM8L681	146.8	144.4		yes	
Kidney	NM8K682	146.8	141.4		yes	18 Feb 98
Blubber	NM8B683	121.9	140.6			30 Jan 98
ADDITIONAL SAMPLES COLLECTED:						
Field No - GW97SB-23 Adrenals, liver, kidney, heart, pancreas, spleen, and lung for histology and teeth for age determination to NMFS Miami Lab, Ruth Ewing Head to Smithsonian ,Charlie Potter Skin and reproductive organs to NOS Charleston Lab, Wayne McFee Stomach to Sea World Melon and muscle to NMFS, NWFSC, ECD, Seattle						

ROUGH-TOOTHED DOLPHIN *(Steno bredanensis)*						
ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Sb-003	RTDL-003	15 Dec 97	Stranding		St Joseph's State Park, FL	29°45.2/85°25'
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F		235			120	
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM8L684	154.7	127.7		yes	
Kidney	NM8K685	116.1	141.6			
Blubber	NM8B686	66.8	93.3			2 Feb 98
ADDITIONAL SAMPLES COLLECTED:						
Field No - GW97SB-03 Adrenals, liver, perirenal lymph nodes, pancreas, and spleen for histology and teeth for age determination to NMFS Miami Lab, Ruth Ewing Head to Smithsonian, Charlie Potter Skin and stomach to Charleston NOS Lab, Wayne McFee Stomach to Sea World Melon and muscle to NMFS, NWFSC, ECD, Seattle						

ROUGH-TOOTHED DOLPHIN *(Steno bredanensis)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Sb-004	RTDL-004	15 Dec 97	Stranding		St Joseph's State Park, FL	29°45.2/85°25'
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M		199		48	88	1.4
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM8L687	139.8	148.1			
Kidney	NM8K688	122.1	125.2		yes	
Blubber	NM8B689	75.4	76.5			5 Feb 98

ADDITIONAL SAMPLES COLLECTED:

Field No - GW97SB-19
Pancreas, kidneys, testicles, spleen, bladder, mesenteric lymph nodes, stomach, duodenum, prescapular lymph nodes, lung, and associated lung lymph nodes for histology and teeth for age determination and to NMFS Miami Lab, Ruth Ewing
Head to Smithsonian ,Charlie Potter
Skin and reproductive organs to NOS Charleston Lab, Wayne McFee
Stomach to Sea World
Melon and muscle to NMFS, NWFSC, ECD, Seattle

ROUGH-TOOTHED DOLPHIN *(Steno bredanensis)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Sb-005	RTDL-005	15 Dec 97	Stranding		St Joseph's State Park, FL	29°45.2/85°25'
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M		235		63	126	
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM8L690	143.7	105.9		yes	
Kidney	NM8K691	107.3	117.7		yes	
Blubber	NM8B692	115.9	105.9			6 Feb 98

ADDITIONAL SAMPLES COLLECTED:

Field No - GW97SB-16
Liver, testes, and kidneys for histology and teeth for age determination to NMFS Miami Lab, Ruth Ewing
Head to Smithsonian, Charlie Potter
Skin and reproductive organs to Charleston NOS Lab, Wayne McFee
Stomach to Sea World
Melon and muscle to NMFS, NWFSC, ECD, Seattle

ROUGH-TOOTHED DOLPHIN *(Steno bredanensis)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Sb-006	RTDL-006	15 Dec 97	Stranding		St Joseph's State Park, FL	29°45.2/85°25'
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M		207		51	97	
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM8L693	157.7	172.8		yes	
Kidney	NM8K694	115.6	138.7		yes	
Blubber	NM8B695	54.5	69.1			9 Feb 98

ADDITIONAL SAMPLES COLLECTED:

Field No - GW97SB-84
Adrenals, spleen, lungs, lymph nodes, liver, kidney, and aorta for histology and teeth for age determination to NMFS Miami Lab, Ruth Ewing
Head to Smithsonian, Charlie Potter
Skin and reproductive organs to Charleston NOS Lab, Wayne McFee
Stomach to Sea World
Melon and muscle to NMFS, NWFSC, ECD, Seattle

ROUGH-TOOTHED DOLPHIN *(Steno bredanensis)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Sb-007	RTDL-007	15 Dec 97	Stranding		St Joseph's State Park, FL	29°45.2/85°25'
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F		241.3		23	116	
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM8L696	162.6	151.0		yes	
Kidney	NM8K697	111.8	121.7		yes	
Blubber	NM8B698	73.0	89.6			11 Feb 98

ADDITIONAL SAMPLES COLLECTED:

Field No - GW97SB-05
Adrenals, liver, kidney, pulmonary artery, pancreas, spleen, lung, aorta, and stomach for histology to NMFS Miami Lab, Ruth Ewing
Teeth for age determination to NMFS Miami Lab, Ruth Ewing
Fetus, skin, and reproductive organs to Charleston NOS Lab, Wayne McFee
Head to Smithsonian, Charlie Potter
Stomach to Sea World
Melon and muscle to NMFS, NWFSC, ECD, Seattle

ROUGH-TOOTHED DOLPHIN *(Steno bredanensis)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Sb-008	RTDL-008	15 Dec 97	Stranding		St Joseph's State Park, FL	29°45.2/85°25'
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F		221		58.5	108	
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM8L699	154.8	157.5		yes	
Kidney	NM8K700	135.8	130.3		yes	
Blubber	NM8B701	104.7	107.1			13 Feb 98

ADDITIONAL SAMPLES COLLECTED:

Field No - GW 97SB-86
Adrenal, liver, kidney, mesenteric lymph nodes, pancreas, spleen, bladder, lung, aorta, colon, and duodenum for histology to NMFS Miami Lab, Ruth Ewing
Teeth for age determination to NMFS Miami Lab, Ruth Ewing
Head to Smithsonian, Charlie Potter
Skin and reproductive organs to Charleston NOS Lab, Wayne McFee
Stomach to Sea World
Melon and muscle to NMFS, NWFSC, ECD, Seattle

ROUGH-TOOTHED DOLPHIN *(Steno bredanensis)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Sb-009	RTDL-009	15 Dec 97	Stranding		St Joseph's State Park, FL	29°45.2/85°25'
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F		228		56	114	
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM8L702	146.7	143.2			
Kidney	NM8K703	154.5	149.3		yes	
Blubber	NM8B704	99.3	94.7			12 Jan 98

ADDITIONAL SAMPLES COLLECTED:

Field No - GW97SB-02
Adrenals, spleen, kidney, lung lymph nodes, pancreas, bladder, and ovaries for histology to NMFS Miami Lab, Ruth Ewing
Teeth for age determination to NMFS Miami Lab, Ruth Ewing
Head to Smithsonian, Charlie Potter
Skin and reproductive organs to Charleston NOS Lab, Wayne McFee
Stomach to Sea World
Melon and muscle to NMFS, NWFSC, ECD, Seattle

ROUGH-TOOTHED DOLPHIN *(Steno bredanensis)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Sb-010	RTDL-010	15 Dec 97	Stranding		St Joseph's State Park, FL	29°45.2/85°25'
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F		209		55	102	
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM8L705	151.8	151.3		yes	
Kidney	NM8K706	127.5	147.0		yes	
Blubber	NM8B707	90.1	100.7			14 Jan 98

ADDITIONAL SAMPLES COLLECTED:

Field No - GW97SB-01
Liver, lung, kidney, and aorta for histology and teeth for age determination to NMFS Miami Lab, Ruth Ewing
Head to Smithsonian, Charlie Potter
Skin and reproductive organs to Charleston NOS Lab, Wayne McFee
Stomach to Sea World
Melon and muscle to NMFS, NWFSC, ECD, Seattle

ROUGH-TOOTHED DOLPHIN *(Steno bredanensis)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Sb-011	RTDL-011	15 Dec 97	Stranding		St Joseph's State Park, FL	29°45.2/85°25'
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F		201.9		47	100	
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM8L708	162.2	147.0			
Kidney	NM8K709	147.4	126.3		yes	
Blubber	NM8B710	90.8	110.3			20 Jan 98

ADDITIONAL SAMPLES COLLECTED:

Field No - GW97SB-06
Adrenals, spleen, bladder, pulmonary lymph nodes, pancreas, kidney, ovaries, lung, heart, and stomach for histology to NMFS Miami Lab, Ruth Ewing
Teeth for age determination to NMFS Miami Lab, Ruth Ewing
Head to Smithsonian, Charlie Potter
Skin and reproductive organs to Charleston NOS Lab, Wayne McFee
Stomach to Sea World
Melon and muscle to NMFS, NWFSC, ECD, Seattle

ROUGH-TOOTHED DOLPHIN *(Steno bredanensis)*						
ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Sb-012	RTDL-012	15 Dec 1997	Stranding		St Joseph's State Park, FL	29°45.2/85°25'
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M		135.9		35	226.1	3.8
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM8L711	155.7	172.0		yes	
Kidney	NM8K712	144.1	126.3			
Blubber	NM8B713	84.7	108.0			16 Jan 98
ADDITIONAL SAMPLES COLLECTED:						
Field No - GW97SB-81 Lung, liver, adrenals, bladder, thymus, testes, pancreas, and stomach for histology and teeth for age determination to NMFS Maimi Lab, Ruth Ewing Head to Smithsonian, Charlie Potter Skin and reproductive organs to Charleston NOS Lab, Wayne McFee Stomach to Sea World Melon and muscle to NMFS, NWFSC, ECD, Seattle						

ROUGH-TOOTHED DOLPHIN *(Steno bredanensis)*						
ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Sb-013	RTDL-013	15 Dec 1997	Stranding		St Joseph's State Park, FL	29°45.2/85°25'
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F		251.5		65.4	345.4	
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM8L714	132.2	143.2		yes	
Kidney	NM8K715	125.0	108.0		yes	
Blubber	NM8B716	130.4	110.1			21 Jan 98
ADDITIONAL SAMPLES COLLECTED:						
Field No - GW 97SB-82 Adrenal, liver, ovary, lung, pancreas, kidney, pulmonary artery, stomach, pulmonary lymph nodes for histology and teeth for age determination to NMFS Miami Lab, Ruth Ewing Head to Smithsonian, Charlie Potter Skin and reproductive organs to Charleston NOS Lab, Wayne McFee Stomach to Sea World Melon and muscle to NMFS, NWFSC, ECD, Seattle						

ROUGH-TOOTHED DOLPHIN *(Steno bredanensis)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Sb-014	RTDL-014	15 Dec 1997	Stranding		St Joseph's State Park, FL	29°45.2/85°25'
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M		58.4		41.9	266.7	
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM8L717	141.3	144.6			
Kidney	NM8K718	141.3	131.7		yes	
Blubber	NM8B719	119.5	131.7			23 Jan 98

ADDITIONAL SAMPLES COLLECTED:

Field No - GW97SB-07
Pancreas, spleen, kidney, left ventricle, lung, diaphram, trachea, colon, intestines, stomach, thymus and teeth to NMFS Miami Lab, Ruth Ewing
Head to Smithsonian, Charlie Potter
Skin and reproductive organs to Charleston NOS Lab, Wayne McFee
Stomach to Sea World
Melon and muscle to NMFS, NWFSC, ECD, Seattle

ROUGH-TOOTHED DOLPHIN *(Steno bredanensis)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Sb-015	RTDL-015	15 Dec 1997	Stranding		St Joseph's State Park, FL	29°45.2/85°25'
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F		176.5		38.7	228.6	
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM8L720	163.6	160.7		yes	
Kidney	NM8K721	141.3	118.7		yes	
Blubber	NM8B722	94.7	64.6			27 Jan 98

ADDITIONAL SAMPLES COLLECTED:

Field No - GW97SB-83
Pancreas, bile duct, kidney, aorta, liver, spleen, lung, stomach, and teeth to NMFS Miami Lab, Ruth Ewing
Head to Smithsonian, Charlie Potter
Skin and reproductive organs to Charleston NOS Lab, Wayne McFee
Stomach to Sea World
Melon and muscle to NMFS, NWFSC, ECD, Seattle

CALIFORNIA SEA LION *(Zalophus californianus)*						
ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Zc-001	CSL-2203	30 Sept 93	Stranding		Morro Bay	38°51' 122°32'
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F	12 y	166	71		98.8	1.1
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM3L047	153	170		yes*	
Kidney	NM3K048	111	154			
Blubber	NM3B049	82	91			
ADDITIONAL SAMPLES COLLECTED:						
*Complete set of all organs, brain, and reproductive tract Hermit crabs from stomach, lumbar and spinal vertebra to archive at the Marine Mammal Center, Sausalito, CA Bile, kidney, blubber, brain, and liver to NMFS, NWFSC, ECD, Seattle Liver, kidney, muscle, brain, intestine, heart, and lung to the University of California at Davis Tooth for age determination and kidney smear to the Marine Mammal Center, Sausalito, CA Lung, liver, bronchus, bladder for microbiology						

CALIFORNIA SEA LION *(Zalophus californianus)*						
ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Zc-002	CSL-2217	27 Oct 93	Stranding		Casper Beach	38°51' 122°32'
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M	8 y	175	69		88.7	0.7
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM3L050	162	151		yes*	
Kidney	NM3K051	153	173			
Blubber	NM3B052	89	70			
ADDITIONAL SAMPLES COLLECTED:						
*Complete set of all organs Liver, kidney, blubber, brain, testes to NMFS, NWFSC, ECD, Seattle Tooth for aging and kidney smear to the Marine Mammal Center, Sausalito, CA Liver, lung for microbiology Stomach content to Museum of Vertebrate Zoology						

CALIFORNIA SEA LION *(Zalophus californianus)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Zc 003	CSL2881	5 May 96	Stranding		Monterey, CA	
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M	12 y	242.5	128.6			5.5
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM6L057	110				
Kidney	NM6K058	150				
Blubber	NM6B059	144	134			
Muscle	NM6M060	180				

ADDITIONAL SAMPLES COLLECTED:

Toxicology to NMFS, NWFSC, ECD, Seattle
Lung, heart, thyroid, spleen, liver, gall bladder, adrenals, kidney, urinary bladder, gonad, submandibular gland for histology , photos, tooth for aging, and X-rays at the Marine Mammal Center, Sausalito, CA

CALIFORNIA SEA LION *(Zalophus californianus)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Zc 004	CSL 3067	20 Aug 96	Stranding		Sausalito, CA	
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M		126	199.6		143	12
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM6L076	140.1	128.6		yes	
Kidney	NM6K077	166.6	186.4		yes	
Blubber	NM6L076	177.5	186.4			

ADDITIONAL SAMPLES COLLECTED:

Lung, liver, kidney, ureter, urinary bladder, urethra, adrenals, thyroid, heart, lymph nodes, stomach, and spleen for histology to the University of California at Davis
Serum sample stored at the Marine Mammal Center, Sausalito, CA

CALIFORNIA SEA LION *(Zalophus californianus)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Zc 005	CSL3101	29 Oct 96	Stranding		Sausalito, CA	
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M	subadult	188	104		101	2.5
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM6L079	124	130		yes	
Kidney	NM6K080	131	134		yes	
Blubber	NM6B081	149	147			

ADDITIONAL SAMPLES COLLECTED:

Lung, heart, thyroid, spleen, liver, adrenal, kidney, and urinary bladder at the Marine Mammal Center, Sausalito, CA

CALIFORNIA SEA LION *(Zalophus californianus)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Zc 006	CSL 3096	26 Oct 96	Stranding		Pebble Beach,CA	
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M	Adult	210	139		115	0.9
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM6L082	160	153		yes	
Kidney	NM6K083	200	195		yes	
Blubber	NM6B084	185	191			
Muscle	NM6M085	160	191			

ADDITIONAL SAMPLES COLLECTED:

Tooth collected for aging to the Marine Mammal Center, Sausalito, CA
Lung, trachea, heart, salivary gland, esophagus, stomach, duodenum , jejunum, ileum, colon, pancreas, spleen, gall bladder, adrenals, kidney, ureter, and urinary bladder for histology to University of California at Davis

CALIFORNIA SEA LION *(Zalophus californianus)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Zc 007	CSL3103	6 Nov 96	Stranding		Monterey Dunes Colony, CA	
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M	7	164	78		92	1.8
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM6L089	171	148		yes	
Kidney	NM6K087	158	166		yes	
Blubber	NM6B088	150	172			

ADDITIONAL SAMPLES COLLECTED:

Lung, heart, pulmonary artery, thyroid, stomach, ileum, colon, spleen, liver, adrenal, kidney, and urinary bladder for histology at the Marine Mammal Center, Sausalito, CA

CALIFORNIA SEA LION *(Zalophus californianus)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Zc 008	CSL 3105	11 Nov 96	Stranding		Aquatic Park, San Francisco, CA	
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M	8.5	221	144		119	2.2
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM6L089	178	179		yes	
Kidney	NM6K090	143	165		yes	
Blubber	NM6B091	172	177			

ADDITIONAL SAMPLES COLLECTED:

Lung, heart, aorta, salivary gland, thyroid, jejunum, ileum, colon, pancreas, spleen, gall bladder, liver, adrenals, kidney, ureter, urinary bladder, gonad, and sublumbar lymph node for histology to the University of California at Davis

CALIFORNIA SEA LION *(Zalophus californianus)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Zc 009	CSL3110	5 Dec 96	Stranding		Monterey County, Delonte, CA	
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M	9	167	220		124	29
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM7L092	139	170.3		yes	
Kidney	NM7K093	163	163		yes	
Blubber	NM6B094	169	181			

ADDITIONAL SAMPLES COLLECTED:

Thyroid, stomach, ileum, colon, liver, kidney, urinary bladder, tongue, esophagus, lymph nodes, gallbladder, and testicle for histology at the University of California at Davis
Teeth collected for age determination by the Marine Mammal Center, Sausalito, CA

CALIFORNIA SEA LION *(Zalophus californianus)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Zc 010	CSL 3127	28 Apr 97	Stranding		Corcoran Lagoon, CA	
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F	10	165	63.5		88	0.5
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM7L113	160	164		yes	
Kidney	NM7K114	158	172		yes	
Blubber	NM7B115	152	154			

ADDITIONAL SAMPLES COLLECTED:

Lung,heart,aorta,thyroid,stomach,duodenum,colon,spleen,liver,adrenal.kidney,urinary bladder, ovary, uterus, eyes, brain, muscle, colonic lymph node to the University of California at Davis for histology
Tooth taken for age determination to the Marine Mammal Center, Sausalito, CA
Radiographs taken
Lung and liver for microbiology

CALIFORNIA SEA LION *(Zalophus californianus)*						
ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Zc 011	CSL3153	22 May 97	Stranding		Sonoma Co., CA	
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M	14	208	134		120	1.9
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM7L124	159	154		yes	
Kidney	NM7K125	173	174		yes	
Blubber	NM7B126	175	134			
ADDITIONAL SAMPLES COLLECTED:						
Thyroid, stomach, ileum, colon, liver, kidney, urinary bladder, lymph nodes, testicle, lung, heart, pancreas, spleen, adrenals, and muscle for histology and teeth for age determination to the Marine Mammal Center, Sausalito, CA Skull and pelt to museum at Pt Logos						

CALIFORNIA SEA LION *(Zalophus californianus)*						
ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Zc 012	CSL3264	7 Aug 97	Stranding			
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M	3	141	39		81	14
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM7L137	140.7	166.9			
Kidney	NM7K138	151.2	152.5		yes	
Blubber	NM7B139	169.8	141.7			
ADDITIONAL SAMPLES COLLECTED:						

CALIFORNIA SEA LION *(Zalophus californianus)*						
ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Zc 013	CSL3306	2 Sept 97	Stranding			
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M	≥5	198	135		127	0.8
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM7L140	153.0	163.3		yes	
Kidney	NM7K141	162.3	173.4		yes	
Blubber	NM7B142	170.2	170.3			
ADDITIONAL SAMPLES COLLECTED:						
All except brain taken for histology for the Marine Mammal Center, Sausalito, CA						

CALIFORNIA SEA LION *(Zalophus californianus)*						
ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Zc 014	CSL3314	7 Nov 97	Stranded			
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M	~3	162	70		92	12
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM8L149	166.0	166.6		yes	
Kidney	NM8K150	152.1	148.1		yes	
Blubber	NM8B151	151.8	166.6			
ADDITIONAL SAMPLES COLLECTED:						
Lung, heart, tonsil, thyroid, salivary gland, stomach, ileum, liver, kidney, adrenals, urinary bladder, and spleen for histology and teeth for age determination to the Marine Mammal Center, Sausalito, CA						

CALIFORNIA SEA LION *(Zalophus californianus)*						
ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Zc 015	CSL3321	7 Nov 97	Stranding			
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M		217	167		140	26
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM8L152	160.3	165.9		yes	
Kidney	NM8K153	160.7	151.3		yes	
Blubber	NM8B154	177.1	154.9			
ADDITIONAL SAMPLES COLLECTED:						
Lung, heart, liver, gallbladder, adrenals, kidney, colon, ileum, urinary bladder, thyroid, spleen, lymph glands, salivary gland, esophagus, and testicle for histology and teeth for age determination to the Marine Mammal Center, Sausalito, CA						

CALIFORNIA SEA LION *(Zalophus californianus)*						
ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Zc 016	CSL3377	7 Nov 97	Stranding			
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M	~4	160	72		97	
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM8L155	161.7	170.4		yes	
Kidney	NM8K156	157.4	170.3		yes	
Blubber	NM8B157	165.2	181.7			
ADDITIONAL SAMPLES COLLECTED:						
Liver, kidney, heart, lung, adrenals, thyroid, esophagus, urinary bladder, and gonad for histology and teeth for age determination to the Marine Mammal Center, Sausalito, CA						

CALIFORNIA SEA LION *(Zalophus californianus)*						
ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Zc 017	CSL3449	2 Dec 97	Stranding		Fanshell Beach, Monterey Co, CA	
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M	Adult	224	190		132	31
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM8L198	173.1	160.8		yes	
Kidney	NM8K159	160.5	160.8		yes	
Blubber	NM8B160	166.3	186.1			
ADDITIONAL SAMPLES COLLECTED:						
Lung, heart, salivary gland,thyroid,tonsil,stomach,ileum,pancreas,spleen,liver,gallbladder,adrenal,kidney,ureter,urinary bladder, gonads, eye, and lymph nodes for histology and teeth for age determination for the Marine Mammal Center, Sausalito, CA						

CALIFORNIA SEA LION *(Zalophus californianus)*						
ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Zc 018	CSL3438	14 Nov 97	stranding		Santa Cruz, CA	
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F	adult	147	66		94	
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM8L194	142.9	186.1		yes	
Kidney	NM8K195	154.4	174.3		yes	
Blubber	NM8B196	171.0	160.8			
ADDITIONAL SAMPLES COLLECTED:						
Lung,heart,thyroid,tonsil,tongue,stomach,duodenum,colon,pancreas,spleen,liver,adrenal,kidney,urinary bladder, gonads, uterus, brain, and lymph nodes for histology to the Marine Mammal Center, Sausalito, CA						

COMMON DOLPHIN *(Delphinus delphis)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Dd-001	NMFS # 3960	10 Jul 96	Drift net set		George's Bank Canyon, Atlantic Ocean	39°54'/69°31'
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F		168			89.5	14.0
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM6L070	160.8	116.7		yes	18 Oct 96
Kidney	NM6K071	73.2	98.7		yes	23 Oct 96
Blubber	NM6B072	91.3	63.9			28 Oct 96

ADDITIONAL SAMPLES COLLECTED:

Skull to Smithsonian
Tissue taken for contaminant studies for several agencies
Tissue taken for histopathology studies for several agencies

COMMON DOLPHIN *(Delphinus delphis)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Dd-002	NMFS # 3694	10 Jul 96	Drift net set		George's Bank Canyon, Atlantic Ocean	39°54'/69°31'
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M		208	117.9	23.5x2	106.0	18.0
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM6L073	119.1	119.4		yes	30 Oct 96
Kidney	NM6K074	127.9	124.6		yes	1 Nov 96
Blubber	NM6B075	87.3	95.7			1 Nov 96

ADDITIONAL SAMPLES COLLECTED:

Skull to Smithsonian
Tissue taken for contaminant studies for several agencies
Tissue taken for histopathology for several agencies
Parasites for Teri Rowles

COMMON DOLPHIN *(Delphinus delphis)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Dd-003	NMFS # 3069	5 Jul 96	Drift net set		George's Bank Canyon, Atlantic Ocean	67°20'/40°14'
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M		210	136.0	52	107	17
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM8L161	103.4	109.2		yes	23 Sept 96
Kidney	NM6K062	140.3	119.3		yes	26 Sept 96
Blubber	NM6B063	50.6	62.9			27 Sept 96

ADDITIONAL SAMPLES COLLECTED:

Skull to Smithsonian
Tissue taken for contaminant studies for several agencies
Tissue taken for histopathology for several agencies
Parasites for Teri Rowles

COMMON DOLPHIN *(Delphinus delphis)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Dd-004	MH97-594Dd	16 Nov 97	stranding		North Falmouth,MA	39°69.5/70°37.22
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F	adult	220	113.4	48	106	1.5
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM8L161	171.2	156.7			
Kidney	NM8K162	138.9	171.7			
Blubber	NM8B163	93.2	147.9			

ADDITIONAL SAMPLES COLLECTED:

Histology taken for WHOI - Armed Forces Institute of Pathology

COMMON DOLPHIN *(Delphinus delphis)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Dd-005	MH97-596Dd	16 Nov 97	Stranding		North Falmouth, MA	39°69.5/70°37.22
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F	mature	215	113.4	50	115	1.5
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM8L164	153.8	136.5			
Kidney	NM8K165	144	151.8			
Blubber	NM8B169	120.9	126.7			

ADDITIONAL SAMPLES COLLECTED:

Histology for WHOI - Armed Forces Institute of Pathology

COMMON DOLPHIN *(Delphinus delphis)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Dd-006	MH97-597Dd	16 Nov 97	stranding		North Falmouth,MA	39°69.5/70°37.22
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F	mature	198		50	104	18mm
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM8L167	163.6	160.7		yes	
Kidney	NM8K168	158.8	160.2		yes	
Blubber	NM8B169	160.1	114.8			

ADDITIONAL SAMPLES COLLECTED:

Histology taken for WHOI - Armed Forces Institute of Pathology
Stomach (whole) to WHOI

COMMON DOLPHIN *(Delphinus delphis)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Dd-007	MH97-598Dd	16 Nov 97	stranding		North Falmouth, MA	39°69.5/70°37.22
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F	mature	211	113.4	47	92	1.7
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM8L173	173.4	134.8		yes	
Kidney	NM8K171	160.2	145.0		yes	
Blubber	NM8B172	146.6	121.6			

ADDITIONAL SAMPLES COLLECTED:

WHOI ID #Dd97-348
Histology taken for WHOI - Armed Forces Institute of Pathology
Stomach (whole) to WHOI

COMMON DOLPHIN *(Delphinus delphis)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Dd-008	MH97-599Dd	16 Nov 97	stranding		North Falmouth,MA	39°69.5/70°37.22
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F	mature	208		54	109	25mm
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM8L173	159.4	160.3		yes	
Kidney	NM8K174	160.4	134.8		yes	
Blubber	NNM8B175	128.2	156.3		yes	

ADDITIONAL SAMPLES COLLECTED:

WHOI ID #97-348
Histology taken for WHOI - Armed Forces Institute of Pathology
Stomach (whole) to WHOI

COMMON DOLPHIN *(Delphinus delphis)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Dd-009	MH97-600Dd	16 Nov 97	stranding		North Falmouth, MA	39°69.5/70°37.22
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F	adult	208	113.4	50	118	2.1
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM8L176	163.6	163.0		yes	
Kidney	NM8K177	163.8	156.1		yes	
Blubber	NM8B178	136.2	129.8		yes	

ADDITIONAL SAMPLES COLLECTED:

WHOI ID #Dd-97-350
Histology taken for WHOI - Armed Forces Institute of Pathology
Stomach (whole) to WHOI

HOODED SEAL *(Cystophora cristata)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Cc-001	MH-97-428Cc	4 Feb 97	Stranding		Hull, MA	42°17.46'/70°52.45'
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F	Juv	104			70	1.75
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM7L097	111.9	145.9		yes	
Blubber	NM7B098	121.0	122.1			

ADDITIONAL SAMPLES COLLECTED:

Bladder, uterus, kidney, liver, spleen, heart, and adrenals to New England Aquarium for histology
Jaw taken for aging

HOODED SEAL *(Cystophora cristata)*						
ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Cc-002	MH-97-455Cc	5 Mar 97	Stranding		Gloucester,MA	42°38.32'/70°41.13'
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M	Juv	108			74	1.3
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM7B104	147.1	141.7		yes	
Blubber	NM7B104	121.3	158.3			
ADDITIONAL SAMPLES COLLECTED:						
Lung, adrenals, liver, and kidney to New England Aquarium for histology						

HOODED SEAL *(Cystophora cristata)*						
ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Cc-003	MH-97-461Cc	8 Mar 97	Stranding		Plymouth,MA	41°58'/70°40'
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M	Juv	108	38.6		75	2
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM7L109	160.4	135.1		yes	
Blubber	NM7B110	157.7	159.1			
ADDITIONAL SAMPLES COLLECTED:						
Lung, liver, kidney, and adrenals to New England Aquarium for histology						

HARBOR SEAL *(Phoca vitulina)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Pv-001	MH-97-430Pv	5 Feb 97	Stranding		Nantucket,MA	41°17'/70°05'
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F	Juv	85	39.6		56	1
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM7L099	151	121.8		yes	
Blubber	NM7B100	146.2	144.8			

ADDITIONAL SAMPLES COLLECTED:

Samples for histology taken by New England Aquarium

HARBOR SEAL *(Phoca vitulina)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Pv-002	MH097-503Pv	26 May 97	Stranding		Rockport, MA	42°40'/70°37'
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M	Adult	144	45.4		88	2.5
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM7L127	160.21	140.99		yes	
Blubber	NM7B128	149.50	149.20			

ADDITIONAL SAMPLES COLLECTED:

Samples for histology taken by New England Aquarium
Tooth taken for aging

HARBOR SEAL *(Phoca vitulina)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Pv-003	MH-97-576Pv	9 Nov 97	Stranding		Sciduate, MA	42°11.147'/70°43'.098
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M	7 mo	94	24.9		47	1.5
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM8L187	118.3	120.4			
Kidney	NM8K185	156.5				
Blubber	NM8B186	111.3				

ADDITIONAL SAMPLES COLLECTED:

Samples for histology taken by New England Aquarium

RINGED SEAL *(Phoca hispida)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Ph-001	MH-97-431Ph	2/6/97	Stranding		Corea,ME	44°23.96'/67°58.36'
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M	Juv	75	10.5		55	2
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM7L101	107.2	137.8		yes	
Blubber	NM7B102	129.2	158.7			

ADDITIONAL SAMPLES COLLECTED:

Samples for histology taken by New England Aquarium

HARP SEAL *(Phoca groenlandica)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Pg-001	MH-97-460Pg	3/7/97	Stranding		Quincy,MA	42°16'/71°00'
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M	young adult	154	65.7		90	2
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM7L105	128.1	149.2		yes	
Blubber	NM7B106	136.7	161.0			

ADDITIONAL SAMPLES COLLECTED:

Lung, liver, adrenals, and kidney for histology to New England Aquarium

HARP SEAL *(Phoca groenlandica)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Pg-002	MH-97-469Pg	18 Mar 97	Stranding		Beverly,MA	42°33'/70°51'
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F	1 yr	104	31.8		80	2.0
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM7L111	166.8	161.1			
Blubber	NM7B112	154.4	153.4			

ADDITIONAL SAMPLES COLLECTED:

Jaw for aging

HARP SEAL *(Phoca groenlandica)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Pg-003	MH-97-483Pg	8 April 97	Stranding		Sciduate, MA	42°12.661'/70°43'.789
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M	Juv	117	36.3		88	2.0
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM7L116	151.47	168.84		yes	
Blubber	NM8B184	157.47	148.28			

ADDITIONAL SAMPLES COLLECTED:

Histology taken for New England Aquarium
GI tract contents collected
Tooth taken for aging

HARP SEAL *(Phoca groenlandica)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Pg-004	MH-97-629Pg	22 Dec 97	stranding		Revere, MA	42°25.8/70°59.0
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M	+5	155	74.8		100	2.5
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM8L182	162.0	156.9			
Kidney	NM8B183	160.8	163.0			
Blubber	NM8B184	156.0	156.1			

ADDITIONAL SAMPLES COLLECTED:

Histology to New England Aquarium
Reproductive organs, lymph nodes, and lung for virus isolation

GREY SEAL *(Halichoerus grypus)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Hg-001	MH-97-484Hg	8 April 97	Stranding		Nantucket, MA	41°22'/70°01'
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M	Juv	106	34.0		67	2.0
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM7L118	154.77	165.38		yes	
Blubber	NM7B118	164.67	152.27			

ADDITIONAL SAMPLES COLLECTED:

Histology taken for New England Aquarium

GREY SEAL *(Halichoerus grypus)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Hg-002	MH-97-491Hg	10 May 97	Stranding		Nantucket, MA	41°22'/70°01'
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F	Juv	102	31.8		74	1.5
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM7L120	153.13	170.78		yes	
Blubber	NM7B121	164.34	150.78			

ADDITIONAL SAMPLES COLLECTED:

Histology taken for New England Aquarium

GREY SEAL *(Halichoerus grypus)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Hg-003	MH-97-493Hg	11 May 97	Stranding		Nantucket, MA	
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
M	Juv	104	34.0		80	2.0
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver	NM7L122	162.97	141.19		yes	
Blubber	NM7B123	149.16	151.94			

ADDITIONAL SAMPLES COLLECTED:

Histology taken for New England Aquarium

FIN WHALE *(Balaenoptera physalus)*

ANIMAL ID NO.	FIELD ID NO.	DATE	ACQUIRED FROM		LOCATION	LAT / LONG
Bp-001	MH-97-531Bp	4 Aug 97	Stranding		Eastham, MA	41°52.520/70°00.569
SEX	AGE (y)	TOTAL LENGTH (cm)	TOTAL WEIGHT (kg)	FLUKE WIDTH (cm)	AXILLARY GIRTH (cm)	BLUBBER THICKNESS (cm)
F	adult	13140		360		4.8
NBSB SAMPLES	NBSB ID NO.	WEIGHT OF SUBSAMPLES			HISTOLOGY	SAMPLE HOMOGENIZED
		A (g)	B (g)			
Liver						
Kidney						
Blubber	NM8B136	159.1	148.8		yes	

ADDITIONAL SAMPLES COLLECTED:

Histology taken for New England Aquarium and Armed Forces Institute of Pathology

APPENDIX E

ANALYTICAL DATA FOR INORGANIC CONSTITUENTS

Table E.1. Concentrations of inorganic constituents in pilot whale (*Globicephala melas*) liver samples (mg/kg, wet mass) - Sheet I

Animal ID No.	Sampling Site	NIST ID No.	Subsample ID	Na	Mg	Al	Cl	K	Ca	Sc
MH-90-597Gm	NEA[e]	NM1L003	B015-A	1278 ± 10[a]	-	-	-	2415 ± 42	-	≤0.00012
			B015-B	1246 ± 10	127 ± 14	0.252 ± 0.048	1601 ± 14	2351 ± 42	25 ± 4	≤0.00013
			B015-C	1282 ± 10	145 ± 11	0.335 ± 0.160	1660 ± 14	2413 ± 42	22 ± 4	0.000095 ± 0.000037
MH-90-599Gm	NEA	NM1L005	B003-A	1628 ± 11	141 ± 17	≤0.205	1778 ± 16	2067 ± 44	61 ± 5	≤0.00010
			B003-B	1614 ± 11	158 ± 20	≤0.199	1773 ± 16	2092 ± 47	49 ± 6	≤0.00011
MH-90-600Gm	NEA	NM1L007	B003-A	1244 ± 8	143 ± 16	0.208 ± 0.112	1636 ± 15	2448 ± 46	42 ± 5	≤0.00012
			B003-B	1232 ± 8	133 ± 17	0.135 ± 0.113	1639 ± 15	2418 ± 45	57 ± 4	≤0.00012
MH-90-617Gm	NEA	NM1L009	B015-A	1561 ± 12	128 ± 14	0.144 ± 0.040	1783 ± 16	1980 ± 38	38 ± 5	≤0.00011
			B015-B	1551 ± 12	125 ± 13	0.106 ± 0.031	1772 ± 16	1938 ± 37	41 ± 10	≤0.00011
			B015-C	1564 ± 12	128 ± 16	0.203 ± 0.055	1787 ± 16	2075 ± 38	47 ± 4	≤0.00011
MH-90-624Gm	NEA	NM1L011	B003-A	1465 ± 10	118 ± 17	0.193 ± 0.069	1632 ± 15	2347 ± 45	34 ± 5	≤0.00019
			B003-B	1487 ± 10	121 ± 15	0.324 ± 0.079	1647 ± 15	2341 ± 43	45 ± 6	≤0.00019
MH-91-602Gm	NEA	NM1L021	B003-A	1605 ± 10	84 ± 14	0.309 ± 0.054	1919 ± 17	2260 ± 39	68 ± 5	≤0.00007
			B003-B	1579 ± 10	105 ± 15	≤0.186	1888 ± 17	2255 ± 48	68 ± 5	≤0.00007
MH-91-603Gm	NEA	NM1L023	B003-A	1593 ± 10	89 ± 14	≤0.182	1978 ± 17	2305 ± 47	48 ± 6	≤0.00008
			B003-B	1581 ± 10	110 ± 15	≤0.203	1956 ± 17	2223 ± 41	63 ± 6	≤0.00009
MH-91-604Gm	NEA	NM1L025	B003-A	1614 ± 11	125 ± 16	0.214 ± 0.068	2231 ± 20	2653 ± 46	42 ± 5	≤0.00008
			B003-B	1619 ± 11	136 ± 18	≤0.219	2226 ± 20	2506 ± 48	56 ± 6	≤0.00009
MH-91-605Gm	NEA	NM1L027	B003-A	1277 ± 6	192 ± 14	≤0.210	1512 ± 16	2811 ± 48	47 ± 5	≤0.0002
			B003-B	1282 ± 6	173 ± 13	≤0.165	1535 ± 12	2772 ± 46	58 ± 5	≤0.0002

[a] All elements determined by instrumental neutron activation analysis (INAA) except as noted; the uncertainties associated with the INAA results are due to counting statistics, including propagated uncertainties of the standard.
[b] Samples analyzed by voltammetry
[c] Samples analyzed by cold vapor atomic absorption spectrometry (CVAAS)
[d] Determined using ion-exchange chromatography and CVAAS
[e] New England Aquarium, Boston, MA
ND Not detectable

Table E.1. Concentrations of inorganic constituents in pilot whale (*Globicephala melas*) liver samples (mg/kg, wet mass) - Sheet II

Animal ID No.	Subsample ID	V	Mn	Fe	Co	Ni	Cu	Zn
NM1L003	B015-A	-	-	383 ± 3	0.01170 ± 0.00048		-	35.7 ± 0.3
	B015-B	0.0077 ± 0.0035	2.52 ± 0.05	384 ± 3	0.01108 ± 0.00048		3.66 ± 0.13	36.0 ± 0.3
	B015-C	≤0.0093	2.61 ± 0.04	375 ± 3	0.01192 ± 0.00045		4.06 ± 0.50	35.0 ± 0.3
	B011				0.00530 ± 0.00015[b]	0.03048 ± 0.00075[b]	4.05 ± 0.02[b]	34.2 ± 1.5[b]
NM1L005	B003-A	≤0.014	2.12 ± 0.04	223 ± 3	0.01257 ± 0.00034		2.72 ± 0.69	51.1 ± 0.4
	B003-B	≤0.014	2.13 ± 0.08	224 ± 2	0.01243 ± 0.00024		3.26 ± 0.63	51.1 ± 0.3
	B010				0.00694 ± 0.00069[b]	0.05950 ± 0.00370[b]	2.55 ± 0.09[b]	73.2 ± 0.9[b]
NM1L007	B003-A	≤0.014	1.83 ± 0.04	800 ± 7	0.00948 ± 0.00052		1.91 ± 0.47	42.3 ± 0.3
	B003-B	≤0.014	1.73 ± 0.07	812 ± 7	0.00955 ± 0.00045		2.32 ± 0.37	41.9 ± 0.3
NM1L009	B015-A	0.0176 ± 0.0039	2.65 ± 0.05	250 ± 2	0.01428 ± 0.00043		2.72 ± 0.21	36.3 ± 0.3
	B015-B	0.0258 ± 0.0054	2.69 ± 0.06	242 ± 2	0.01383 ± 0.00040		2.66 ± 0.47	35.5 ± 0.3
	B015-C	0.0134 ± 0.0038	2.65 ± 0.04	249 ± 2	0.01482 ± 0.00042		2.74 ± 0.17	36.4 ± 0.3
	B011				0.01541 ± 0.00073[b]	0.03741 ± 0.00188[b]	2.94 ± 0.05[b]	33.6 ± 0.2[b]
NM1L011	B003-A	≤0.013	1.93 ± 0.04	611 ± 5	0.01041 ± 0.00038		1.02 ± 0.60	48.7 ± 0.4
	B003-B	≤0.013	2.03 ± 0.03	615 ± 5	0.01059 ± 0.00038		1.24 ± 0.39	49.1 ± 0.3
NM1L021	B003-A	≤0.013	2.58 ± 0.05	143 ± 2	0.00636 ± 0.00023		2.77 ± 0.47	36.6 ± 0.3
	B003-B	≤0.013	2.51 ± 0.06	145 ± 2	0.00691 ± 0.00021		3.63 ± 0.60	36.6 ± 0.3
	B010				ND[b]	0.03834 ± 0.00175[b]	2.87 ± 0.13[b]	45.1 ± 0.2[b]
NM1L023	B003-A	0.022 ± 0.005	3.03 ± 0.05	365 ± 3	0.01463 ± 0.00041		3.29 ± 0.35	28.5 ± 0.2
	B003-B	0.020 ± 0.005	2.93 ± 0.04	365 ± 3	0.01496 ± 0.00038		3.99 ± 0.39	28.1 ± 0.3
	B010				0.01016 ± 0.00121[b]	0.04183 ± 0.00169[b]	2.97 ± 0.07[b]	36.8 ± 0.7[b]

[a] All elements determined by instrumental neutron activation analysis (INAA) except as noted; the uncertainties associated with the INAA results are due to counting statistics, including propagated uncertainties of the standard.

[b] Samples analyzed by voltammetry.

[c] Samples analyzed by cold vapor atomic absorption spectrometry (CVAAS)

[d] Determined using ion-exchange chromatography and CVAAS.

[e] New England Aquarium, Boston, MA

ND Not detectable

Table E.1. Concentrations of inorganic constituents in pilot whale (*Globicephala melas*) liver samples (mg/kg, wet mass) - Sheet III

Animal ID No.	Subsample ID	V	Mn	Fe	Co	Ni	Cu	Zn
NM1L025	B003-A	≤0.015	2.69 ± 0.04	273 ± 3	0.01447 ± 0.00034		3.35 ± 0.61	33.9 ± 0.3
	B003-B	≤0.016	2.70 ± 0.04	271 ± 3	0.01439 ± 0.00032		2.46 ± 0.58	33.5 ± 0.3
	B010				ND[b]	0.02039 ± 0.00076[b]	3.34 ± 0.15[b]	39.8 ± 1.5[b]
NM1L027	B003-A	≤0.013	3.44 ± 0.04	154 ± 4	0.01530 ± 0.00060		≤1.318	41.9 ± 0.2
	B003-B	≤0.011	3.38 ± 0.05	154 ± 4	0.01420 ± 0.00060		≤1.237	40.9 ± 0.2

[a] All elements determined by instrumental neutron activation analysis (INAA) except as noted; the uncertainties associated with the INAA results are due to counting statistics, including propagated uncertainties of the standard.
[b] Samples analyzed by voltammetry.
[c] Samples analyzed by cold vapor atomic absorption spectrometry (CVAAS)
[d] Determined using ion-exchange chromatography and CVAAS.
[e] New England Aquarium, Boston, MA
ND Not detectable

Table E.1. Concentrations of inorganic constituents in pilot whale (*Globicephala melas*) liver samples (mg/kg, wet mass) - Sheet IV

Animal ID No.	Subsample ID	As	Se	Br	Rb	Sr	Mo	Ag	Cd	Sn
NM1L003	B015-A	0.305 ± 0.013	14.92 ± 0.12	-	2.11 ± 0.04	≤0.8	≤0.57	0.290 ± 0.006	9.49 ± 0.34	≤1.8
	B015-B	0.288 ± 0.012	14.87 ± 0.12	-	2.14 ± 0.05	≤0.8	≤0.51	0.284 ± 0.006	9.36 ± 0.31	≤1.8
	B015-C	0.304 ± 0.012	14.67 ± 0.12	-	2.08 ± 0.04	≤0.8	≤0.47	0.284 ± 0.005	9.69 ± 0.30	≤1.7
	B011							9.04 ± 0.07[b]		
NM1L005	B003-A	1.183 ± 0.027	13.63 ± 0.09	18.2 ± 0.4	1.45 ± 0.03	≤0.6	≤0.85	0.189 ± 0.004	5.99 ± 0.25	≤1.2
	B003-B	1.159 ± 0.016	13.23 ± 0.09	17.8 ± 0.4	1.43 ± 0.06	≤0.7	≤0.52	0.191 ± 0.004	6.61 ± 0.26	≤1.0
	B010								7.59 ± 0.16[b]	
NM1L007	B003-A	0.291 ± 0.013	28.93 ± 0.19	16.8 ± 0.4	2.09 ± 0.06	≤0.9	0.96 ± 0.33	0.207 ± 0.005	8.03 ± 0.24	≤1.8
	B003-B	0.283 ± 0.014	27.99 ± 0.19	15.5 ± 0.4	1.83 ± 0.06	≤0.7	1.29 ± 0.39	0.201 ± 0.004	7.70 ± 0.22	≤1.4
NM1L009	B015-A	0.405 ± 0.014	7.90 ± 0.06	-	1.86 ± 0.03	≤0.7	≤0.47	0.111 ± 0.004	8.08 ± 0.29	≤1.6
	B015-B	0.417 ± 0.014	7.68 ± 0.06	-	1.84 ± 0.04	≤0.7	≤0.43	0.116 ± 0.004	8.59 ± 0.29	≤1.5
	B015-C	0.397 ± 0.013	7.96 ± 0.07	-	1.85 ± 0.04	≤0.7	≤0.41	0.112 ± 0.004	8.52 ± 0.27	≤1.5
	B011								8.61 ± 0.35[b]	
NM1L011	B003-A	0.271 ± 0.015	9.58 ± 0.07	16.5 ± 0.4	1.93 ± 0.29	≤1.2	0.36 ± 0.13	0.135 ± 0.005	7.70 ± 0.22	≤1.2
	B003-B	0.267 ± 0.021	9.58 ± 0.07	17.3 ± 0.4	2.04 ± 0.27	≤1.2	≤0.36	0.131 ± 0.005	7.72 ± 0.19	≤1.1
NM1L021	B003-A	≤0.037	1.58 ± 0.01	17.7 ± 0.4	1.79 ± 0.03	≤0.5	≤0.29	0.015 ± 0.002	3.06 ± 0.21	≤1.0
	B003-B	≤0.036	1.60 ± 0.01	18.5 ± 0.4	1.70 ± 0.03	≤0.5	≤0.32	0.011 ± 0.002	2.49 ± 0.16	≤0.9
	B010								3.48 ± 0.04[b]	
NM1L023	B003-A	0.238 ± 0.015	25.89 ± 0.17	19.9 ± 0.4	1.95 ± 0.04	≤0.6	≤0.42	0.339 ± 0.004	14.26 ± 0.39	≤1.2
	B003-B	0.212 ± 0.015	25.22 ± 0.17	20.1 ± 0.4	1.77 ± 0.04	≤0.6	≤0.49	0.326 ± 0.004	14.31 ± 0.36	≤1.2
	B010								17.29 ± 0.17[b]	

[a] All elements determined by instrumental neutron activation analysis (INAA) except as noted; the uncertainties associated with the INAA results are due to counting statistics, including propagated uncertainties of the standard.
[b] Samples analyzed by voltammetry.
[c] Samples analyzed by cold vapor atomic absorption spectrometry (CVAAS)
[d] Determined using ion-exchange chromatography and CVAAS.
[e] New England Aquarium, Boston, MA
ND Not detectable

Table E.1. Concentrations of inorganic constituents in pilot whale (*Globicephala melas*) liver samples (mg/kg, wet mass) - Sheet V

Animal ID No.	Subsample ID	As	Se	Br	Rb	Sr	Mo	Ag	Cd	Sn
NM1L025	B003-A	≤0.041	2.85 ± 0.02	20.9 ± 0.5	2.06 ± 0.04	≤0.5	≤0.31	0.033 ± 0.002	10.58 ± 0.32	≤1.1
	B003-B	0.062 ± 0.014	2.79 ± 0.02	21.5 ± 0.5	2.10 ± 0.04	≤0.6	≤0.42	0.025 ± 0.002	11.24 ± 0.36	≤1.1
	B010								11.57 ± 0.41[b]	
NM1L027	B003-A	0.027 ± 0.006	1.49 ± 0.02	15.4 ± 0.6	≤3.4	≤1.6	≤0.13	0.014 ± 0.004	3.171 ± 0.126	≤1.0
	B003-B	0.036 ± 0.005	1.49 ± 0.02	15.7 ± 0.4	3.14 ± 0.84	≤1.5	≤0.13	0.021 ± 0.004	3.048 ± 0.125	≤1.5

[a] All elements determined by instrumental neutron activation analysis (INAA) except as noted; the uncertainties associated with the INAA results are due to counting statistics, including propagated uncertainties of the standard.
[b] Samples analyzed by voltammetry.
[c] Samples analyzed by cold vapor atomic absorption spectrometry (CVAAS)
[d] Determined using ion-exchange chromatography and CVAAS.
[e] New England Aquarium, Boston, MA
ND Not detectable

Table E.1. Concentrations of inorganic constituents in pilot whale (*Globicephala melas*) liver samples (mg/kg, wet mass) - Sheet VI

Animal ID No.	Subsample ID	Sb	I	Cs	Ba	La	Ce	Sm
NM1L003	B015-A	0.00099 ± 0.00029	-	0.0058 ± 0.0011	≤12	≤0.0054	0.0858 ± 0.0087	≤0.0020
	B015-B	0.00117 ± 0.00033	0.35 ± 0.17	0.0044 ± 0.0011	≤ 9.9	≤0.0047	0.0953 ± 0.0092	≤0.0018
	B015-C	≤0.00128	≤0.48	0.0073 ± 0.0012	≤ 8.8	≤0.0044	0.0639 ± 0.0088	≤0.0017
NM1L005	B003-A	≤0.0010	≤0.726	0.0095 ± 0.0008	≤ 5.7	≤0.004	≤0.019	≤0.0019
	B003-B	≤0.0012	0.767 ± 0.219	0.0103 ± 0.0025	≤ 4.8	≤0.004	≤0.023	≤0.0019
NM1L007	B003-A	0.0021 ± 0.0003	≤0.692	0.0084 ± 0.0013	≤ 5.2	≤0.004	≤0.034	≤0.0021
	B003-B	≤0.0060	≤0.679	0.0085 ± 0.0010	≤ 4.5	≤0.004	≤0.028	≤0.0021
NM1L009	B015-A	0.00173 ± 0.00035	≤0.51	0.0057 ± 0.0010	≤ 8.8	≤0.0047	0.0320 ± 0.0064	≤0.0017
	B015-B	≤0.00107	≤0.49	0.0057 ± 0.0010	≤ 7.8	≤0.0043	0.0263 ± 0.0062	≤0.0016
	B015-C	0.00204 ± 0.00036	≤0.50	0.0055 ± 0.0011	≤ 7.0	≤0.0041	0.0385 ± 0.0071	0.00056 ± 0.00035
NM1L011	B003-A	≤0.0016	≤0.685	≤0.0022	≤ 4.1	≤0.003	≤0.018	≤0.0018
	B003-A	≤0.0012	≤0.653	≤0.0021	≤ 3.1	≤0.003	≤0.017	≤0.0019
NM1L021	B003-A	0.0005 ± 0.0003	≤0.688	0.0042 ± 0.0005	≤ 5.9	≤0.004	≤0.012	≤0.0017
	B003-B	≤0.0009	≤0.532	0.0052 ± 0.0005	≤ 4.5	≤0.005	≤0.012	≤0.0016
NM1L023	B003-A	≤0.0014	0.607 ± 0.200	0.0045 ± 0.0006	≤ 7.2	≤0.005	≤0.022	≤0.0024
	B003-B	≤0.0003	0.335 ± 0.209	0.0037 ± 0.0006	≤ 5.9	≤0.005	≤0.025	≤0.0022
NM1L025	B003-A	≤0.0006	≤0.742	0.0054 ± 0.0006	≤ 6.1	≤0.004	≤0.013	≤0.0019
	B003-B	≤0.0005	0.960 ± 0.251	0.0040 ± 0.0006	≤ 6.0	≤0.005	≤0.014	≤0.0021
NM1L027	B003-A	≤0.003	≤0.65	0.0083 ± 0.0011	≤ 4	≤0.003	≤0.031	≤0.0006
	B003-B	≤0.004	≤0.64	0.0083 ± 0.0011	≤ 4	≤0.003	≤0.022	≤0.0006

[a] All elements determined by instrumental neutron activation analysis (INAA) except as noted; the uncertainties associated with the INAA results are due to counting statistics, including propagated uncertainties of the standard.
[b] Samples analyzed by voltammetry.
[c] Samples analyzed by cold vapor atomic absorption spectrometry (CVAAS).
[d] Determined using ion-exchange chromatography and CVAAS.
[e] New England Aquarium, Boston, MA
ND Not detectable

Table E.1. Concentrations of inorganic constituents in pilot whale (*Globicephala melas*) liver samples (mg/kg, wet mass) - Sheet VII

Animal ID No.	Subsample ID	Eu	Tb	Hf	Ta	Au	Hg	Methyl-Hg	Pb	Th	U
NM1L003	B015-A	≤0.00029	≤0.00079	0.00014 ± 0.00012	≤0.00030	≤0.064	43.63 ± 0.09			≤0.0014	≤0.11
	B015-B	≤0.00030	≤0.00083	≤0.00032	≤0.00031	0.092 ± 0.036				≤0.0014	≤0.095
	B015-C	≤0.00028	≤0.00078	≤0.00031	≤0.00029	0.093 ± 0.036				≤0.0014	≤0.88
	B011						47.96 ± 2.25[c]	1.55[d]	0.0884 ± 0.0013[b]		
NM1L005	B003-A	≤0.0007	≤0.0010	≤0.0009	≤0.00049	≤0.00026	62.08 ± 0.47			≤0.0015	≤0.043
	B003-B	≤0.0006	≤0.0011	≤0.0010	≤0.00044	≤0.00024				≤0.0019	≤0.042
	B010						27.46 ± 1.25[c]	2.16[d]	0.1065 ± 0.0030[b]		
NM1L007	B003-A	≤0.0011	≤0.0015	≤0.0012	≤0.00089	≤0.00025	114 ± 6			≤0.0022	≤0.048
	B003-B	≤0.0006	≤0.0013	≤0.0011	≤0.00070	≤0.00023	110 ± 8			≤0.0018	≤0.047
NM1L009	B015-A	≤0.00025	≤0.00070	≤0.00028	≤0.00023	≤0.053	20.95 ± 0.04			≤0.0012	≤0.91
	B015-B	≤0.00024	≤0.00039	≤0.00027	≤0.00021	≤0.049				0.00051 ± 0.00042	≤0.84
	B015-C	≤0.00025	≤0.00069	≤0.00028	≤0.00022	≤0.045				≤0.0013	≤0.79
	B011						20.71 ± 1.18[c]	1.15[d]	≤0.024[b]		
NM1L011	B003-A	≤0.0008	≤0.0011	≤0.0010	≤0.00059	≤0.00022	33.56 ± 0.33			≤0.0016	≤0.040
	B003-B	≤0.0006	≤0.0009	≤0.0009	≤0.00038	≤0.00019	30.45 ± 0.36			≤0.0018	≤0.040
NM1L021	B003-A	≤0.0006	≤0.0008	≤0.0007	≤0.00032	≤0.00026	0.98 ± 0.05			≤0.0012	≤0.040
	B003-B	≤0.0005	≤0.0008	≤0.0007	≤0.00030	≤0.00022	1.12 ± 0.06			≤0.0013	≤0.036
	B010						0.28 ± 0.02[c]	0.10[d]	0.0333 ± 0.0022[b]		
NM1L023	B003-A	≤0.0007	≤0.0009	≤0.0008	≤0.00055	≤0.00032	82.79 ± 0.42			≤0.0014	≤0.054
	B003-B	≤0.0006	≤0.0009	≤0.0009	≤0.00057	≤0.00028				≤0.0015	≤0.049
	B010						44.37 ± 0.10[c]	1.65[d]	0.1279 ± 0.0026[b]		

[a] All elements determined by instrumental neutron activation analysis (INAA) except as noted; the uncertainties associated with the INAA results are due to counting statistics, including propagated uncertainties of the standard.
[b] Samples analyzed by voltammetry.
[c] Samples analyzed by cold vapor atomic absorption spectrometry (CVAAS).
[d] Determined using ion-exchange chromatography and CVAAS.
[e] New England Aquarium, Boston, MA
ND Not detectable

Table E.1. Concentrations of inorganic constituents in pilot whale (*Globicephala melas*) liver samples (mg/kg, wet mass) - Sheet VIII

Animal ID No.	Subsample ID	Eu	Tb	Hf	Ta	Au	Hg	Methyl-Hg	Pb	Th	U
NM1L025	B003-A	≤0.0006	≤0.0009	≤0.0008	≤0.00042	≤0.00028	6.49 ± 0.13			≤0.0013	≤0.042
	B003-B	≤0.0006	≤0.0009	≤0.0008	≤0.00043	≤0.00029				≤0.0014	≤0.046
	B010						4.26 ± 0.15[c]	0.51[d]	0.0428 ± 0.0015[b]		
NM1L027	B003-A	≤0.0002	≤0.0013	≤0.004	≤0.0016	≤0.00099	3.27 ± 0.03			≤0.021	≤0.08
	B003-B	≤0.0006	≤0.0012	≤0.003	≤0.0015	≤0.00098	2.98 ± 0.04			≤0.020	≤0.08

[a] All elements determined by instrumental neutron activation analysis (INAA) except as noted; the uncertainties associated with the INAA results are due to counting statistics, including propagated uncertainties of the standard.
[b] Samples analyzed by voltammetry.
[c] Samples analyzed by cold vapor atomic absorption spectrometry (CVAAS).
[d] Determined using ion-exchange chromatography and CVAAS.
[e] New England Aquarium, Boston, MA
ND Not detectable

Table E.2. Concentrations of inorganic constituents in harbor porpoise (*Phocoena phocoena*) liver samples (mg/kg, wet mass) - Sheet I

Animal ID No.	Sampling Site	NIST ID No.	Subsample ID	Na	Mg	Al	Cl	K	Ca	Sc	V
MH-90-553Pp	NEA[c]	NM1L001	B014-A	1429 ± 11[a]	188 ± 14	0.331 ± 0.046	1586 ± 14	2201 ± 39	32 ± 5	≤0.000096	0.0283 ± 0.0044
			B014-B	1444 ± 11	179 ± 14	0.357 ± 0.041	1592 ± 14	2209 ± 39	29 ± 4	≤0.000099	0.0202 ± 0.0036
			B014-C	1446 ± 11	176 ± 16	0.339 ± 0.168	1589 ± 14	2119 ± 38	28 ± 4	≤0.00010	0.0158 ± 0.0031
MH-91-424Pp	NEA	NM1L013	B003-D	1381 ± 11	288 ± 11	≤0.27	1906 ± 23	2789 ± 49	49 ± 7	≤0.00009	≤0.013
			B003-E	1424 ± 11	256 ± 11	≤0.16	1873 ± 23	2738 ± 52	42 ± 7	≤0.00008	≤0.013
			B004-A	1449 ± 11	243 ± 10	≤0.20	1957 ± 24	2942 ± 51	55 ± 6	≤0.00008	≤0.014
			B004-B	1336 ± 10	280 ± 12	≤0.63	1791 ± 22	2635 ± 43	42 ± 4	≤0.00008	≤0.014
MH-91-461Pp	NEA	NM1L015	B015-A	1500 ± 12	114 ± 16	1.800 ± 0.089	1918 ± 17	2155 ± 38	61 ± 17	0.000124 ± 0.000033	≤0.0127
			B015-B	1502 ± 12	128 ± 15	0.231 ± 0.052	1932 ± 17	2141 ± 41	66 ± 8	≤0.00011	≤0.0106
			B015-C	1509 ± 12	142 ± 14	0.254 ± 0.049	1938 ± 17	2089 ± 39	63 ± 4	≤0.00011	≤0.0100
MH-91-505Pp	NEA	NM1L017	B003-A	1698 ± 11	244 ± 25	0.251 ± 0.085	2030 ± 18	3243 ± 58	53 ± 5	≤0.00010	≤0.015
			B003-C	1707 ± 11	222 ± 22	0.143 ± 0.070	2029 ± 18	3370 ± 57	51 ± 6	≤0.00011	≤0.017
MH-91-504Pp	NEA	NM1L019	B003-A	1198 ± 8	167 ± 14	≤0.162	1632 ± 15	2984 ± 46	46 ± 5	≤0.00008	≤0.012
			B003-B	1191 ± 8	188 ± 23	≤0.161	1562 ± 12	2969 ± 45	38 ± 5	≤0.00008	≤0.012
MH-92-575Pp	NEA	NM2L031	B001-A	1372 ± 11	236 ± 12	≤0.16	1843 ± 22	2738 ± 44	60 ± 6	≤0.00007	≤0.012
			B001-B	1365 ± 11	246 ± 10	≤0.16	1856 ± 23	2723 ± 45	58 ± 5	≤0.00006	≤0.011

[a] All elements determined by instrumental neutron activation analysis (INAA) except as noted; the uncertainties associated with the INAA results are due to counting statistics, including propagated uncertainties of the standard.
[b] Samples analyzed by voltammetry.
[c] Samples analyzed by cold vapor atomic absorption spectrometry (CVAAS).
[d] Determined using ion-exchange chromatography and CVAAS.
[e] New England Aquarium, Boston, MA
ND Not detectable

Table E.2. Concentrations of inorganic constituents in harbor porpoise (*Phocoena phocoena*) liver samples (mg/kg, wet mass) - Sheet II

NIST ID No.	Subsample ID	Mn	Fe	Co	Ni	Cu	Zn	As	Se	Br	Rb
NM1L001	B014-A	4.42 ± 0.06	220 ± 2	0.00863 ± 0.00040	-	8.28 ± 0.53	25.29 ± 0.20	0.316 ± 0.020	4.141 ± 0.037	-	1.021 ± 0.026
	B014-B	4.55 ± 0.07	230 ± 2	0.01248 ± 0.00040	-	8.28 ± 0.52	26.22 ± 0.21	0.302 ± 0.018	4.284 ± 0.038	-	1.091 ± 0.029
	B014-C	4.46 ± 0.05	228 ± 2	0.00944 ± 0.00042	-	7.26 ± 0.52	26.02 ± 0.21	0.287 ± 0.014	4.268 ± 0.038	-	1.077 ± 0.029
NM1L013	B003-D	5.23 ± 0.25	569 ± 5	0.0054 ± 0.0004	-	10.90 ± 0.54	28.69 ± 0.25	0.373 ± 0.014	1.123 ± 0.018	16.3 ± 0.8	1.603 ± 0.065
	B003-E	5.14 ± 0.22	563 ± 5	0.0045 ± 0.0004	-	9.73 ± 0.56	28.30 ± 0.25	0.337 ± 0.027	1.105 ± 0.017	17.4 ± 0.8	1.487 ± 0.059
	B004-A	5.33 ± 0.24	559 ± 5	0.0054 ± 0.0004	-	10.13 ± 0.56	27.86 ± 0.24	0.338 ± 0.027	1.116 ± 0.017	16.1 ± 0.8	1.530 ± 0.060
	B004-B	4.90 ± 0.24	555 ± 5	0.0060 ± 0.0004	-	10.20 ± 0.80	27.99 ± 0.24	0.390 ± 0.031	1.109 ± 0.017	15.7 ± 0.7	1.497 ± 0.055
	B010			ND	0.04264 ± 0.00053[b]	11.88 ± 0.40[b]	33.05 ± 1.07[b]				
NM1L015	B015-A	2.67 ± 0.05	322 ± 3	0.00597 ± 0.00037	-	5.81 ± 0.61	25.22 ± 0.20	0.593 ± 0.014	2.070 ± 0.021	-	1.031 ± 0.033
	B015-B	2.69 ± 0.06	324 ± 3	0.00660 ± 0.00041	-	5.89 ± 0.09	25.27 ± 0.20	0.564 ± 0.015	2.080 ± 0.022	-	1.043 ± 0.034
	B015-C	2.69 ± 0.04	327 ± 3	0.00644 ± 0.00036	-	6.26 ± 0.55	25.52 ± 0.20	0.573 ± 0.014	2.129 ± 0.021	-	1.056 ± 0.033
	B011			0.00644 ± 0.00023[b]	0.02818 ± 0.00023[b]	7.70 ± 0.37[b]	25.43 ± 0.23[b]				
NM1L017	B003-A	5.15 ± 0.06	451 ± 4	0.00497 ± 0.00038	-	10.43 ± 0.88	38.2 ± 0.3	0.273 ± 0.016	2.31 ± 0.02	14.1 ± 0.5	1.55 ± 0.04
	B003-C	5.14 ± 0.05	451 ± 4	0.00551 ± 0.00037	-	9.41 ± 0.76	38.2 ± 0.3	0.298 ± 0.014	2.29 ± 0.02	16.4 ± 0.5	1.54 ± 0.06
	B010			ND	0.01882 ± 0.00074[b]	7.45 ± 0.10[b]	39.1 ± 0.7[b]				
NM1L019	B003-A	4.56 ± 0.05	413 ± 4	0.00429 ± 0.00030	-	3.96 ± 0.52	25.1 ± 0.2	0.332 ± 0.014	1.36 ± 0.01	16.2 ± 0.4	1.68 ± 0.04
	B003-B	4.66 ± 0.05	423 ± 4	0.00478 ± 0.00033	-	3.71 ± 0.58	25.5 ± 0.2	0.345 ± 0.013	1.39 ± 0.01	10.1 ± 0.5	1.77 ± 0.04
	B010			0.00291 ± 0.00026[b]	0.05656 ± 0.00238[b]	2.91 ± 0.21[b]	29.9 ± 0.3[b]				
NM2L031	B001-A	4.86 ± 0.21	352 ± 3	0.0032 ± 0.0003	-	15.12 ± 0.49	27.95 ± 0.24	0.183 ± 0.015	1.116 ± 0.017	11.6 ± 0.6	1.343 ± 0.046
	B001-B	4.94 ± 0.19	346 ± 3	0.0028 ± 0.0003	-	15.45 ± 0.73	27.40 ± 0.24	-	1.109 ± 0.017	11.9 ± 0.7	1.340 ± 0.042

[a] All elements determined by instrumental neutron activation analysis (INAA) except as noted; the uncertainties associated with the INAA result are due to counting statistics, including propagated uncertainties of the standard.

[b] Samples analyzed by voltammetry.

[c] Samples analyzed by cold vapor atomic absorption spectrometry (CVAAS).

[d] Determined using ion-exchange chromatography and CVAAS.

[e] New England Aquarium, Boston, MA

ND Not detectable

Table E.2. Concentrations of inorganic constituents in harbor porpoise (*Phocoena phocoena*) liver samples (mg/kg, wet mass) - Sheet III

NIST ID No.	Subsample ID	Sr	Mo	Ag	Cd	Sn	Sb	I	Cs	Ba	La
NM1L001	B014-A	≤0.69	≤1.1	0.1646 ± 0.0042	1.04 ± 0.37	≤1.6	0.0059 ± 0.0006	≤0.47	0.0589 ± 0.0015	≤25	≤0.010
	B014-B	≤0.69	0.51 ± 0.22	0.1721 ± 0.0042	≤0.85	≤1.6	0.0058 ± 0.0006	0.23 ± 0.14	0.0606 ± 0.0015	≤19	≤0.0082
	B014-C	≤0.71	≤0.66	0.1666 ± 0.0041	≤0.67	≤1.6	0.0076 ± 0.0006	≤0.49	0.0628 ± 0.0015	≤14	≤0.0065
NM1L013	B003-D	≤1.551	≤0.48	0.591 ± 0.008	≤0.59	≤2.23	0.0067 ± 0.0011	≤0.57	0.0103 ± 0.0004	≤13	≤0.007
	B003-E	≤2.211	≤0.45	0.585 ± 0.008	≤0.26	≤2.21	0.0069 ± 0.0013	≤0.57	0.0101 ± 0.0005	≤4	≤0.003
	B004-A	≤1.376	≤0.51	0.571 ± 0.007	≤0.29	≤2.01	0.0067 ± 0.0012	≤0.55	0.0105 ± 0.0005	≤2	≤0.002
	B004-B	≤1.411	≤0.76	0.581 ± 0.007	≤0.30	≤2.05	0.0062 ± 0.0016	≤0.56	0.0100 ± 0.0005	≤2	≤0.002
	B010				0.055 ± 0.002[b]						
NM1L015	B015-A	≤0.67	0.36 ± 0.13	0.5185 ± 0.0063	0.44 ± 0.14	≤1.5	0.0029 ± 0.0004	≤0.67	0.0344 ± 0.0013	≤6.2	≤0.0036
	B015-B	≤0.71	≤0.34	0.5180 ± 0.0063	0.56 ± 0.13	≤1.5	0.0031 ± 0.0005	0.72 ± 0.20	0.0310 ± 0.0012	≤5.7	≤0.0035
	B015-C	≤0.67	≤0.32	0.5230 ± 0.0062	0.54 ± 0.16	≤1.4	0.0030 ± 0.0004	0.23 ± 0.14	0.0332 ± 0.0013	≤5.1	≤0.0032
	B011				0.77 ± 0.03[b]						
NM1L017	B003-A	≤0.8	≤0.48	0.759 ± 0.006	≤0.48	≤1.56	≤0.0016	≤0.845	0.0539 ± 0.0014	≤7.6	≤0.005
	B003-C	≤0.8	≤0.55	0.755 ± 0.007	≤0.43	≤1.46	≤0.0015	≤0.857	0.0535 ± 0.0013	≤6.1	≤0.005
	B010				0.063 ± 0.001[b]						
NM1L019	B003-A	≤0.6	≤0.38	0.147 ± 0.003	≤0.37	≤1.28	0.0051 ± 0.0007	≤0.676	0.0494 ± 0.0011	≤5.6	≤0.004
	B003-B	≤0.6	≤0.44	0.148 ± 0.003	≤0.34	≤1.17	0.0053 ± 0.0007	≤0.584	0.0497 ± 0.0011	≤4.7	≤0.003
	B010				0.055 ± 0.0003[b]						
NM2L031	B001-A	≤1.219	≤0.42	0.218 ± 0.005	≤0.53	≤1.82	0.0036 ± 0.0012	≤0.49	0.0081 ± 0.0004	≤12	≤0.010
	B001-B	≤1.163	-	0.208 ± 0.004	-	≤1.72	0.0031 ± 0.0008	≤0.49	0.0081 ± 0.0003	-	-

[a] All elements determined by instrumental neutron activation analysis (INAA) except as noted; the uncertainties associated with the INAA results are due to counting statistics, including propagated uncertainties of the standard.
[b] Samples analyzed by voltammetry.
[c] Samples analyzed by cold vapor atomic absorption spectrometry (CVAAS).
[d] Determined using ion-exchange chromatography and CVAAS.
[e] New England Aquarium, Boston, MA
ND Not detectable

Table E.2. Concentrations of inorganic constituents in harbor porpoise (*Phocoena phocoena*) liver samples (mg/kg, wet mass) - Sheet IV

NIST ID No.	Subsample ID	Ce	Sm	Eu	Tb	Hf	Ta	Au	Hg	Methyl-Hg	Pb	Th	U
NM1L001	B014-A	0.0233 ± 0.0059	≤0.0034	≤0.00022	≤0.00070	≤0.00028	≤0.00023	≤0.12	15.38 ± 0.03	-	-	≤0.0012	≤0.20
	B014-B	0.0180 ± 0.0066	≤0.0028	≤0.00023	≤0.00071	≤0.00028	≤0.00024	≤0.098	-	-	-	≤0.0012	≤0.16
	B014-C	0.0241 ± 0.0067	≤0.0022	≤0.00023	≤0.00072	≤0.00029	≤0.00024	≤0.076	-	-	-	≤0.0013	≤0.13
	B011								11.63 ± 0.23[c]	1.16[d]	-		
NM1L013	B003-D	≤0.0087	≤0.002	≤0.0002	≤0.0013	≤0.0015	≤0.0012	≤0.003	43.40 ± 0.41	-	-	≤0.0023	≤0.08
	B003-E	≤0.0087	≤0.003	≤0.0002	≤0.0012	≤0.0015	≤0.0013	≤0.003	-	-	-	≤0.0024	≤0.09
	B004-A	≤0.0080	≤0.002	≤0.0002	≤0.0011	≤0.0014	≤0.0012	≤0.001	0.67 ± 0.01	-	-	≤0.0022	≤0.07
	B004-B	≤0.0082	≤0.002	≤0.0002	≤0.0012	≤0.0014	≤0.0012	≤0.002	0.71 ± 0.01	-	-	≤0.0023	≤0.07
	B010								0.48 ± 0.03[c]	0.29[d]	0.0144 ± 0.0011[b]		
NM1L015	B015-A	≤0.0128	≤0.0014	≤0.00024	≤0.00063	≤0.00028	≤0.00026	≤0.041	1.77 ± 0.01	-	-	≤0.0012	≤0.71
	B015-B	≤0.0135	≤0.0014	≤0.00025	≤0.00066	≤0.00029	≤0.00027	≤0.040	-	-	-	≤0.0012	≤0.68
	B015-C	≤0.0129	≤0.0013	≤0.00023	≤0.00062	≤0.00028	≤0.00026	0.238 ± 0.080	-	-	-	≤0.0012	≤0.63
	B011								1.03 ± 0.07[c]	0.32[d]	0.0220 ± 0.0011[b]		
NM1L017	B003-A	≤0.018	≤0.0022	≤0.0009	≤0.0011	≤0.0011	≤0.00077	≤0.00032	38.63 ± 0.36	-	-	≤0.0018	≤0.051
	B003-C	≤0.018	≤0.0021	≤0.0010	≤0.0011	≤0.0012	≤0.00075	≤0.00028	-	-	-	≤0.0019	≤0.047
	B010								0.72 ± 0.05[c]	0.43[d]	0.0163 ± 0.0007[b]		
NM1L019	B003-A	≤0.015	≤0.0018	≤0.0007	≤0.0009	≤0.0009	≤0.00055	≤0.00025	2.24 ± 0.08	-	-	≤0.0016	≤0.040
	B003-B	≤0.015	≤0.0017	≤0.0006	≤0.0009	≤0.0009	≤0.00054	≤0.00022	2.13 ± 0.07	-	-	≤0.0016	≤0.038
	B010								1.24 ± 0.01[c]	0.67[d]	0.0262 ± 0.0003[b]		
NM2L031	B001-A	≤0.0072	≤0.002	≤0.0002	≤0.0010	≤0.0012	≤0.0009	≤0.003	0.58 ± 0.01	-	-	≤0.0019	≤0.07
	B001-B	≤0.0069	-	≤0.0002	≤0.0010	≤0.0011	≤0.0009	-	0.54 ± 0.01	-	-	≤0.0019	≤0.08

[a] All elements determined by instrumental neutron activation analysis (INAA) except as noted; the uncertainties associated with the INAA results are due to counting statistics, including propagated uncertainties of the standard.

[b] Samples analyzed by voltammetry.

[c] Samples analyzed by cold vapor atomic absorption spectrometry (CVAAS).

[d] Determined using ion-chromatography and CVAAS.

[e] New England Aquarium, Boston, MA

ND Not detectable

Table E.3 Concentrations of inorganic constituents in white-sided dolphin (*Lagenorhynchus acutus*) liver samples (mg/kg, wet mass) - Sheet I

Animal ID No.	Sampling Site	NIST ID No.	Subsample ID	Na	Mg	Al	Cl	K	Ca	Sc
MH-93-445La	NEA[b]	NM3L035	B003-A	1183 ± 5	142 ± 13	≤0.193	1535 ± 12	2819 ± 52	48 ± 5	≤0.0002
			B003-B	1182 ± 5	138 ± 12	≤0.132	1512 ± 12	2725 ± 46	55 ± 4	≤0.0002
MH-93-446La	NEA	NM3L037	B003-A	1195 ± 5	134 ± 12	≤0.145	1511 ± 16	2734 ± 45	38 ± 4	≤0.0002
			B003-B	1212 ± 5	133 ± 12	≤0.139	1546 ± 12	2815 ± 43	44 ± 4	≤0.0002
MH-93-460La	NEA	NM3L039	B001-A	1111 ± 5	152 ± 13	1.05 ± 0.09	1481 ± 16	3147 ± 52	54 ± 4	≤0.0002
			B001-B	1104 ± 5	163 ± 12	0.48 ± 0.07	1451 ± 11	3146 ± 47	49 ± 4	≤0.0002
MH-93-461La	NEA	NM3L041	B003-A	1269 ± 6	163 ± 12	≤0.145	1360 ± 15	2940 ± 46	57 ± 4	≤0.0002
			B003-B	1259 ± 6	161 ± 17	≤0.142	1353 ± 10	2872 ± 45	59 ± 5	≤0.0001

[a] All elements determined by instrumental neutron activation analysis (INAA) except as noted; the uncertainties associated with the INAA results are due to counting statistics, including propagated uncertainties of the standard.
[b] New England Aquarium, Boston, MA
ND Not detectable

Table E.3 Concentrations of inorganic constituents in white-sided dolphin (*Lagenorhynchus acutus*) liver samples (mg/kg, wet mass) - Sheet II

NIST ID No.	Subsample ID	V	Mn	Fe	Co	Cu	Zn
NM3L035	B003-A	≤0.015	3.39 ± 0.07	219 ± 3	0.0109 ± 0.0006	6.89 ± 0.50	35.6 ± 0.1
	B003-B	≤0.010	3.32 ± 0.05	215 ± 3	0.0113 ± 0.0007	6.89 ± 0.66	35.6 ± 0.1
NM3L037	B003-A	≤0.010	3.15 ± 0.07	304 ± 4	0.0103 ± 0.0005	2.79 ± 0.66	31.4 ± 0.1
	B003-B	≤0.010	3.04 ± 0.05	289 ± 4	0.0093 ± 0.0006	3.58 ± 0.74	30.3 ± 0.1
NM3L039	B001-A	0.061 ± 0.008	4.12 ± 0.06	137 ± 4	0.0143 ± 0.0007	6.53 ± 0.62	50.5 ± 0.2
	B001-B	0.058 ± 0.007	4.11 ± 0.04	137 ± 6	0.0149 ± 0.0007	7.83 ± 0.65	50.9 ± 0.2
NM3L041	B003-A	≤0.010	4.14 ± 0.07	62 ± 3	0.0168 ± 0.0006	7.84 ± 0.70	49.3 ± 0.1
	B003-B	≤0.009	4.11 ± 0.04	69 ± 4	0.0151 ± 0.0010	8.27 ± 0.77	49.3 ± 0.3

[a] All elements determined by instrumental neutron activation analysis (INAA) except as noted; the uncertainties associated with the INAA results are due to counting statistics, including propagated uncertainties of the standard.

[b] New England Aquarium, Boston, MA

ND Not detectable

Table E.3 Concentrations of inorganic constituents in white-sided dolphin (*Lagenorhynchus acutus*) liver samples (mg/kg, wet mass) - Sheet III

NIST ID No.	Subsample ID	As	Se	Br	Rb	Sr	Mo	Ag	Cd
NM3L035	B003-A	0.200 ± 0.007	3.849 ± 0.033	14.9 ± 0.4	3.24 ± 0.84	≤1.4	0.171 ± 0.046	1.50 ± 0.01	0.248 ± 0.062
	B003-B	0.157 ± 0.006	3.750 ± 0.031	18.2 ± 0.5	2.16 ± 0.49	≤1.3	0.189 ± 0.048	1.51 ± 0.02	0.351 ± 0.065
NM3L037	B003-A	0.259 ± 0.008	2.242 ± 0.030	12.0 ± 0.4	≤0.96	≤1.4	0.132 ± 0.054	0.27 ± 0.01	0.287 ± 0.081
	B003-B	0.231 ± 0.007	2.192 ± 0.030	12.8 ± 0.4	≤0.83	≤1.1	≤0.104	0.27 ± 0.01	≤0.281
NM3L039	B001-A	0.406 ± 0.013	8.978 ± 0.103	13.3 ± 0.4	≤1.03	≤1.2	0.476 ± 0.075	0.69 ± 0.01	0.894 ± 0.105
	B001-B	0.424 ± 0.007	9.121 ± 0.105	14.9 ± 0.4	≤1.01	≤1.0	0.301 ± 0.051	0.70 ± 0.01	0.830 ± 0.063
NM3L041	B003-A	0.311 ± 0.016	6.186 ± 0.073	13.1 ± 0.3	≤0.90	≤1.1	0.280 ± 0.057	0.53 ± 0.01	0.187 ± 0.068
	B003-B	0.385 ± 0.014	6.167 ± 0.056	13.1 ± 0.4	≤0.81	≤0.9	0.260 ± 0.057	0.54 ± 0.01	0.299 ± 0.072

[a] All elements determined by instrumental neutron activation analysis (INAA) except as noted; the uncertainties associated with the INAA results are due to counting statistics, including propagated uncertainties of the standard.

[e] New England Aquarium, Boston, MA

ND Not detectable

Table E.3 Concentrations of inorganic constituents in white-sided dolphin (*Lagenorhynchus acutus*) liver samples (mg/kg, wet mass) - Sheet IV

NIST ID No.	Subsample ID	Sn	Sb	I	Cs	Ba	La	Ce	Sm
NM3L035	B003-A	≤2.2	≤0.004	≤0.57	0.0298 ± 0.0011	≤4	≤0.003	≤0.022	≤0.0005
	B003-B	≤2.1	≤0.005	≤0.42	0.0300 ± 0.0010	≤3	≤0.003	≤0.013	≤0.0005
NM3L037	B003-A	≤2.1	≤0.003	≤0.42	0.0275 ± 0.0012	≤4	≤0.003	≤0.021	≤0.0006
	B003-B	≤1.6	≤0.003	≤0.76	0.0270 ± 0.0014	≤3	≤0.002	≤0.016	≤0.0005
NM3L039	B001-A	≤1.8	≤0.003	≤0.67	0.0291 ± 0.0014	≤3	≤0.004	≤0.020	≤0.0009
	B001-B	≤2.0	≤0.003	≤0.43	0.0300 ± 0.0016	≤3	≤0.002	≤0.029	≤0.0006
NM3L041	B003-A	≤1.9	≤0.003	≤0.40	0.0428 ± 0.0015	≤3	≤0.003	≤0.029	≤0.0006
	B003-B	≤1.9	≤0.003	≤0.42	0.0402 ± 0.0016	≤4	≤0.003	≤0.026	≤0.0008

[a] All elements determined by instrumental neutron activation analysis (INAA) except as noted; the uncertainties associated with the INAA results are due to counting statistics, including propagated uncertainties of the standard.

[b] New England Aquarium, Boston, MA

ND Not detectable

Table E.3 Concentrations of inorganic constituents in white-sided dolphin (*Lagenorhynchus acutus*) liver samples (mg/kg, wet mass) - Sheet V

NIST ID No.	Subsample ID	Eu	Tb	Hf	Ta	Au	Hg	Th	U
NM3L035	B003-A	≤0.0002	≤0.0011	≤0.004	≤0.0017	0.00468 ± 0.0004	6.537 ± 0.056	≤0.009	≤0.08
	B003-B	≤0.0002	≤0.0013	≤0.004	≤0.0017	0.00432 ± 0.0005	7.303 ± 0.062	≤0.009	≤0.07
NM3L037	B003-A	≤0.0002	≤0.0010	≤0.005	≤0.0017	≤0.00189	2.919 ± 0.037	≤0.009	≤0.08
	B003-B	≤0.0002	≤0.0010	≤0.004	≤0.0016	≤0.00079	3.076 ± 0.034	≤0.007	≤0.07
NM3L039	B001-A	≤0.0004	≤0.0012	≤0.006	≤0.0022	≤0.00273	30.970 ± 0.169	≤0.009	≤0.10
	B001-B	≤0.0003	≤0.0012	≤0.006	≤0.0019	≤0.00213	32.313 ± 0.177	≤0.009	≤0.07
NM3L041	B003-A	≤0.0002	≤0.0011	≤0.005	≤0.0018	≤0.00091	19.572 ± 0.114	≤0.009	≤0.08
	B003-B	≤0.0002	≤0.0012	≤0.005	≤0.0019	≤0.00106	20.246 ± 0.121	≤0.009	≤0.09

[a] All elements determined by instrumental neutron activation analysis (INAA) except as noted; the uncertainties associated with the INAA results are due to counting statistics, including propagated uncertainties of the standard.
[c] New England Aquarium, Boston, MA
ND Not detectable

Table E.4. Pilot whale (*Globicephala melas*) liver quality assurance material (mg/kg, wet mass) - Sheet I

Bottle ID No.	Subsample ID	Na	Mg	Al	Cl	K	Ca	Sc	Ti	V
226	A	1269 ± 9	136 ± 6	≤0.24	1740 ± 18	2612 ± 62	50 ± 5	≤0.000087	≤7.1	≤0.017
	B	1288 ± 9	128 ± 6	0.73 ± 0.09	1740 ± 18	2791 ± 63	56 ± 6	≤0.000089	≤7.5	≤0.019
	C	1234 ± 9	139 ± 5	≤0.29	1664 ± 18	2575 ± 61	37 ± 5	≤0.000091	≤9.5	≤0.022
	D	1306 ± 9	143 ± 6	0.58 ± 0.07	1763 ± 18	2735 ± 63	40 ± 5	≤0.000088	≤7.2	≤0.018
	E	1304 ± 9	153 ± 8	≤1.31	1767 ± 18	2681 ± 62	58 ± 8	≤0.000088	≤14	≤0.049
	F	1242 ± 9	140 ± 6	0.95 ± 0.07	1703 ± 17	2523 ± 59	42 ± 5	≤0.000084	≤6.8	≤0.017
118	A	1272 ± 9	154 ± 6	1.44 ± 0.09	1730 ± 18	2636 ± 61	55 ± 7	≤0.000084	≤8.2	≤0.021
	B	1227 ± 9	-	-	-	2545 ± 62	-	≤0.00011	-	-
	C	1274 ± 9	130 ± 6	1.02 ± 0.08	1747 ± 18	2606 ± 62	45 ± 5	≤0.000085	≤9.8	≤0.023
129	A	1256 ± 9	-	-	-	2652 ± 62	-	≤0.000077	-	-
	B	1251 ± 9	141 ± 6	≤0.24	1708 ± 17	2589 ± 57	52 ± 5	≤0.000080	≤8.4	≤0.020
	C	1222 ± 9	132 ± 6	≤0.28	1667 ± 17	2602 ± 61	49 ± 6	≤0.000086	≤7.5	≤0.019
157	A	1238 ± 9	-	-	-	2591 ± 62	-	≤0.000085	-	-
	B	1240 ± 9	144 ± 9	8.05 ± 0.12	1716 ± 18	2542 ± 58	37 ± 5	≤0.000084	≤9.6	≤0.023
	C	1219 ± 8	130 ± 5	≤0.22	1670 ± 17	2473 ± 57	42 ± 5	≤0.000084	≤6.7	≤0.017
186	A	1266 ± 9	-	-	-	2694 ± 59	-	≤0.000081	-	-
	B	1284 ± 9	126 ± 5	≤0.23	1754 ± 18	2671 ± 59	36 ± 5	≤0.000084	≤6.6	≤0.017
	C	1297 ± 9	147 ± 6	≤0.19	1763 ± 18	2703 ± 61	44 ± 5	≤0.000080	≤5.1	≤0.015
230	A	1286 ± 9	136 ± 6	≤0.23	1760 ± 18	2757 ± 67	53 ± 6	≤0.000090	≤5.8	≤0.017
	B	1291 ± 9	136 ± 6	≤0.21	1749 ± 18	2715 ± 61	40 ± 5	≤0.000082	≤5.3	≤0.015

Table E.4. Pilot whale (*Globicephala melas*) liver quality assurance material (mg/kg, wet mass) - Sheet II

Bottle ID No.	Subsample ID	Cr	Mn	Fe	Co	Cu	Zn	As	Se	Rb
226	A	0.0258 ± 0.0052	2.925 ± 0.067	439 ± 3	0.01317 ± 0.00031	3.30 ± 0.59	32.26 ± 0.25	0.520 ± 0.015	11.063 ± 0.086	1.940 ± 0.039
	B	0.0348 ± 0.0065	2.876 ± 0.066	440 ± 4	0.01353 ± 0.00032	4.37 ± 0.72	32.39 ± 0.25	0.517 ± 0.017	11.239 ± 0.087	1.994 ± 0.037
	C	0.0327 ± 0.0051	2.857 ± 0.059	440 ± 4	0.01361 ± 0.00032	2.41 ± 0.64	32.36 ± 0.25	0.503 ± 0.014	11.124 ± 0.086	2.061 ± 0.050
	D	0.0235 ± 0.0053	2.910 ± 0.058	435 ± 3	0.01366 ± 0.00032	2.83 ± 0.62	32.13 ± 0.25	0.558 ± 0.016	11.096 ± 0.086	1.915 ± 0.046
	E	0.0285 ± 0.0064	2.881 ± 0.055	444 ± 4	0.01344 ± 0.00033	2.98 ± 1.34	33.01 ± 0.25	0.505 ± 0.016	10.860 ± 0.084	2.162 ± 0.056
	F	0.0319 ± 0.0049	2.789 ± 0.062	444 ± 4	0.01401 ± 0.00030	2.84 ± 0.62	32.67 ± 0.25	0.514 ± 0.019	11.190 ± 0.087	2.000 ± 0.046
118	A	0.0556 ± 0.0056	2.737 ± 0.068	444 ± 4	0.01400 ± 0.00032	2.51 ± 0.66	32.58 ± 0.25	0.531 ± 0.014	11.009 ± 0.086	2.038 ± 0.036
	B	0.0625 ± 0.0052	2.681 ± 0.070	439 ± 3	0.01384 ± 0.00036	-	32.36 ± 0.25	0.556 ± 0.014	11.142 ± 0.087	2.024 ± 0.038
	C	0.0503 ± 0.0057	2.895 ± 0.039	434 ± 3	0.01346 ± 0.00033	3.14 ± 0.61	31.59 ± 0.24	0.605 ± 0.022	10.833 ± 0.084	1.915 ± 0.043
129	A	0.0529 ± 0.0060	2.816 ± 0.078	438 ± 3	0.01348 ± 0.00034	-	31.98 ± 0.25	0.505 ± 0.013	10.787 ± 0.084	2.095 ± 0.047
	B	0.0431 ± 0.0053	2.660 ± 0.069	430 ± 3	0.01324 ± 0.00030	2.58 ± 0.53	31.61 ± 0.24	0.508 ± 0.014	10.783 ± 0.084	1.892 ± 0.035
	C	0.0828 ± 0.0069	2.811 ± 0.064	430 ± 3	0.01355 ± 0.00031	3.34 ± 0.73	31.66 ± 0.24	0.507 ± 0.017	10.840 ± 0.084	1.947 ± 0.036
157	A	0.0221 ± 0.0044	2.661 ± 0.078	439 ± 3	0.01362 ± 0.00031	-	32.31 ± 0.25	0.526 ± 0.014	10.744 ± 0.084	2.014 ± 0.040
	B	0.0308 ± 0.0049	2.603 ± 0.058	428 ± 3	0.01323 ± 0.00029	3.03 ± 0.66	31.71 ± 0.24	0.543 ± 0.014	10.724 ± 0.083	1.952 ± 0.036
	C	0.0495 ± 0.0059	2.663 ± 0.059	431 ± 3	0.01380 ± 0.00032	2.09 ± 0.62	31.97 ± 0.25	0.497 ± 0.019	10.895 ± 0.085	2.006 ± 0.043
186	A	0.0423 ± 0.0057	2.882 ± 0.077	440 ± 3	0.01393 ± 0.00033	-	32.19 ± 0.25	0.496 ± 0.014	11.063 ± 0.066	2.047 ± 0.042
	B	0.0548 ± 0.0052	2.868 ± 0.062	439 ± 3	0.01305 ± 0.00030	2.78 ± 0.51	32.04 ± 0.25	0.533 ± 0.013	10.753 ± 0.084	1.970 ± 0.033
	C	0.0401 ± 0.0048	2.821 ± 0.057	435 ± 3	0.01292 ± 0.00031	3.74 ± 0.63	31.70 ± 0.24	0.572 ± 0.022	10.797 ± 0.084	1.953 ± 0.043
230	A	0.0352 ± 0.0059	2.902 ± 0.067	444 ± 3	0.01364 ± 0.00036	2.73 ± 0.59	32.67 ± 0.25	0.526 ± 0.014	11.254 ± 0.087	2.021 ± 0.040
	B	0.0440 ± 0.0061	2.885 ± 0.053	443 ± 4	0.01351 ± 0.00030	3.31 ± 0.62	32.54 ± 0.25	0.565 ± 0.014	11.092 ± 0.086	1.986 ± 0.037

Table E.4. Pilot whale (*Globicephala melas*) liver quality assurance material (mg/kg, wet mass) - Sheet III

Bottle ID No.	Subsample ID	Sr	Mo	Ag	Cd	Sn	Sb	I	Cs
226	A	≤0.79	≤0.41	0.1866 ± 0.0049	8.75 ± 0.20	≤1.5	≤0.063	≤0.96	0.00404 ± 0.00089
	B	≤0.81	≤0.42	0.1879 ± 0.0048	8.64 ± 0.19	≤1.5	≤0.058	≤0.99	0.00547 ± 0.00078
	C	≤0.84	≤0.44	0.1785 ± 0.0053	8.56 ± 0.21	≤1.5	≤0.087	≤1.3	0.00776 ± 0.00084
	D	≤0.54	≤0.48	0.1827 ± 0.0052	8.78 ± 0.24	≤1.5	≤0.083	≤0.99	0.00714 ± 0.00075
	E	≤0.81	≤0.41	0.1844 ± 0.0049	8.52 ± 0.22	≤1.5	≤0.051	≤1.1	0.00549 ± 0.00067
	F	≤0.77	≤0.40	0.1900 ± 0.0047	8.95 ± 0.20	≤1.4	≤0.040	≤0.90	0.00588 ± 0.00084
118	A	≤0.76	≤0.31	0.1863 ± 0.0047	8.56 ± 0.21	≤1.6	≤0.058	≤1.05	0.00671 ± 0.00081
	B	≤0.78	≤0.52	0.1831 ± 0.0039	8.18 ± 0.26	≤1.6	≤0.163	-	0.00593 ± 0.00079
	C	≤0.78	≤0.50	0.1793 ± 0.0046	8.39 ± 0.24	≤1.6	≤0.043	≤1.25	0.00463 ± 0.00071
129	A	≤0.77	≤0.49	0.1778 ± 0.0047	8.26 ± 0.25	≤1.6	≤0.143	-	0.00585 ± 0.00065
	B	≤0.73	≤0.39	0.1801 ± 0.0048	8.44 ± 0.21	≤1.6	≤0.070	≤1.14	0.00536 ± 0.00085
	C	≤0.79	≤0.31	0.1803 ± 0.0053	8.58 ± 0.21	≤1.6	≤0.040	≤0.96	0.00589 ± 0.00080
157	A	≤0.77	≤0.47	0.1808 ± 0.0052	8.26 ± 0.24	≤1.5	≤0.126	-	0.00449 ± 0.00093
	B	≤0.76	≤0.49	0.1791 ± 0.0052	8.24 ± 0.23	≤1.5	≤0.109	≤1.24	0.00698 ± 0.00100
	C	≤0.77	≤0.41	0.1793 ± 0.0049	8.45 ± 0.21	≤1.5	≤0.035	≤0.92	0.00794 ± 0.00082
186	A	≤0.73	≤0.38	0.1773 ± 0.0046	8.39 ± 0.19	≤1.5	≤0.058	-	0.00485 ± 0.00080
	B	≤0.76	≤0.42	0.1837 ± 0.0047	8.66 ± 0.22	≤1.4	≤0.091	≤0.92	0.00618 ± 0.00082
	C	≤0.74	≤0.39	0.1726 ± 0.0045	8.20 ± 0.20	≤1.6	≤0.030	≤0.70	0.00624 ± 0.00078
230	A	≤0.82	≤0.47	0.1853 ± 0.0055	8.33 ± 0.24	≤1.5	≤0.118	≤0.80	0.00708 ± 0.00103
	B	≤0.75	≤0.41	0.1760 ± 0.0044	8.84 ± 0.22	≤1.5	≤0.082	≤0.73	0.00679 ± 0.00079

Table E.4. Pilot whale (*Globicephala melas*) liver quality assurance material (mg/kg, wet mass) - Sheet IV

Bottle ID No.	Subsample ID	Ba	La	Ce	Sm	Eu	Tb	Hf	Ta	Au	Th	U
226	A	≤4.6	≤0.0032	≤0.016	≤0.0019	≤0.00060	≤0.00065	≤0.00077	≤0.00055	≤0.0002	≤0.0012	≤0.043
	B	≤4.4	≤0.0031	≤0.016	≤0.0019	≤0.00061	≤0.00066	≤0.00079	≤0.00056	≤0.0002	≤0.0012	≤0.044
	C	≤5.7	≤0.0036	≤0.017	≤0.0019	≤0.00063	≤0.00068	≤0.00080	≤0.00056	≤0.0002	≤0.0012	≤0.044
	D	≤5.8	≤0.0036	≤0.016	≤0.0021	≤0.00060	≤0.00066	≤0.00078	≤0.00055	≤0.0003	≤0.0012	≤0.049
	E	≤4.2	≤0.0030	≤0.016	≤0.0020	≤0.00061	≤0.00064	≤0.00078	≤0.00056	≤0.0002	≤0.0012	≤0.043
	F	≤3.6	≤0.0027	≤0.015	≤0.0020	≤0.00057	≤0.00062	≤0.00074	≤0.00053	≤0.0002	≤0.0011	≤0.043
118	A	≤3.8	≤0.0028	≤0.015	≤0.0019	≤0.00062	≤0.00069	≤0.00074	≤0.00054	≤0.0002	≤0.0011	≤0.047
	B	≤8.9	≤0.0045	≤0.015	≤0.0020	≤0.00063	≤0.00070	≤0.00100	≤0.00058	≤0.0003	≤0.0015	≤0.050
	C	≤7.9	≤0.0030	≤0.015	≤0.0026	≤0.00061	≤0.00070	≤0.00076	≤0.00056	≤0.0003	≤0.0011	≤0.055
129	A	≤7.3	≤0.0041	≤0.014	≤0.0017	≤0.00056	≤0.00064	≤0.00068	≤0.00050	≤0.0003	≤0.0010	≤0.04[illegible]
	B	≤4.8	≤0.0031	≤0.014	≤0.0017	≤0.00059	≤0.00066	≤0.00071	≤0.00054	≤0.0002	≤0.0010	≤0.040
	C	≤3.2	≤0.0025	≤0.016	≤0.0015	≤0.00062	≤0.00068	≤0.00076	≤0.00057	≤0.0002	≤0.0011	≤0.033
157	A	≤7.3	≤0.0040	≤0.015	≤0.0018	≤0.00062	≤0.00069	≤0.00075	≤0.00057	≤0.0003	≤0.0011	≤0.045
	B	≤6.8	≤0.0039	≤0.015	≤0.0020	≤0.00061	≤0.00067	≤0.00074	≤0.00054	≤0.0003	≤0.0011	≤0.048
	C	≤3.3	≤0.0026	≤0.015	≤0.0021	≤0.00059	≤0.00064	≤0.00074	≤0.00054	≤0.0002	≤0.0011	≤0.045
186	A	≤4.2	≤0.0029	≤0.014	≤0.0017	≤0.00058	≤0.00065	≤0.00070	≤0.00054	≤0.0002	≤0.0010	≤0.039
	B	≤5.7	≤0.0035	≤0.015	≤0.0018	≤0.00060	≤0.00066	≤0.00073	≤0.00052	≤0.0002	≤0.0011	≤0.042
	C	≤3.0	≤0.0024	≤0.014	≤0.0021	≤0.00056	≤0.00060	≤0.00070	≤0.00051	≤0.0002	≤0.0011	≤0.044
230	A	≤7.1	≤0.0040	≤0.016	≤0.0019	≤0.00065	≤0.00072	≤0.00079	≤0.00059	≤0.0003	≤0.0012	≤0.046
	B	≤5.4	≤0.0034	≤0.015	≤0.0018	≤0.00060	≤0.00066	≤0.00073	≤0.00053	≤0.0002	≤0.0011	≤0.041

APPENDIX F

ANALYTICAL DATA FOR SELECTED ORGANIC COMPOUNDS

F.1. Chlorinated Hydrocarbons: Harbor Porpoise Blubber

F.2. Chlorinated Hydrocarbons: Pilot Whale Blubber

F.3. Quality Control in Organic Analysis: Comparison of Data from NIST, Department of Fisheries and Oceans Canada, and University of Ulm, Germany

Table F.1. Concentrations of PCB congeners and chlorinated pesticides in harbor porpoise (*Phocoena phocoena*) blubber (ng/g, wet mass). The uncertainty value, in parenthesis, is expressed as the standard deviation of a single measurement. - Sheet I

Sample ID	% Lipid	Method	PCB 18	PCB 28	PCB 31	PCB 44	PCB 49	PCB 52	PCB 66	PCB 87	PCB 95	PCB 99
NM1B002	83 (3)	ECD[a]	18.0 (4.9)	13.3 (1.7)	9.55 (0.89)	28.8 (2.2)	-	563 (50)	1376[c] (55)	79.4 (0.8)	1376[c] (55)	381
		MS[b]	18.1 (1.2)	13.3 (0.8)	8.92 (0.72)	28.3 (1.1)	-	539 (33)	648 (17)	82.6 (1.1)	722 (22)	372
NM1B014	83.5	ECD[a]	77.6	9.28	≤6	90.2	159	1474	232 173	1506	1975	
		MS[b]	76.2	≤9	≤9	112	142	1608	211 193	1427	1873	
NM1B016	82 (3)	ECD[a]	25.4 (2.6)	8.02 (0.28)	3.01 (0.66)	29.1 (1.7)	-	340 (20)	590[c] (24)	49.9 (0.9)	590[c] (24)	186
		MS[b]	23.9 (1.2)	8.11 (0.31)	3.15 (0.41)	28.5 (0.8)	-	331 (16)	308 (21)	46.8 (0.7)	291 (19)	173
NM1B018	78.4	ECD[a]	40.0	14.4	≤6	35.9	92.7	488	112 96.6	547	685	
		MS[b]	38.8	13.3	≤8	34.1	97.3	451	106 99.4	555	666	
NM1B020	76.9	ECD[a]	41.3	12.0	≤6	39.2	109	768	163 150	932	1227	
		MS[b]	39.6	13.5	≤8	35.1	94.8	824	182 144	933	1071	

[a] Gas chromatography with electron capture detection
[b] Gas chromatography with mass selective detection
[c] PCB 66 and PCB 95 coelute on the DB-5 column but are differentiated by the mass spectrometer

Table F.1. Concentrations of PCB congeners and chlorinated pesticides in harbor porpoise (*Phocoena phocoena*) blubber (ng/g, wet mass). The uncertainty value, in parenthesis, is expressed as the standard deviation of a single measurement. - Sheet II

Sample ID	Method	PCB 101/90	PCB 105	PCB 110/77	PCB 118	PCB 128	PCB 138/163/164	PCB 149	PCB 151	PCB 153	PCB 156	PCB 170/190
NM1B002	ECD	406 (9)	169 (8)	29.1 (0.3)	679 (35)	375 (45)	2063 (84)	1154 (44)	705 (46)	3238 (97)	97.3 (1.4)	958 (28)
	MS	395 (6)	166 (9)	28.3 (0.5)	666 (11)	364 (37)	2001 (33)	1111 (39)	721 (16)	3239 (61)	104 (3)	948 (15)
NM1B014	ECD	910	22.4	52.5	984	236	3756	1909	655	5845	≤6	242
	MS	904	19.4	53.9	887	221	3790	1875	632	5345	≤9	265
NM1B016	ECD	214 (10)	88.1 (4.3)	14.9 (0.3)	354 (11)	147 (18)	1025 (46)	635 (22)	242 (7)	1760 (77)	19.5 (0.8)	197 (2)
	MS	220 (7)	83.9 (3.1)	15.3 (0.2)	348 (8)	141 (8)	1034 (13)	651 (9)	221 (8)	1734 (37)	18.8 (0.5)	199 (11)
NM1B018	ECD	715	57.3	27.9	582	184	1788	722	253	1981	≤6	156
	MS	701	55.1	23.8	569	163	1708	717	245	2001	≤9	155
NM1B020	ECD	758	133	37.2	1052	274	3443	1500	464	3308	≤6	206
	MS	690	135	35.9	974	238	3018	1577	479	3259	≤9	212

Table F.1. Concentrations of PCB congeners and chlorinated pesticides in harbor porpoise (*Phocoena phocoena*) blubber (ng/g, wet mass). The uncertainty value, in parenthesis, is expressed as the standard deviation of a single measurement. - Sheet III

Sample ID	Method	PCB 180	PCB 183	PCB 187	PCB194	PCB195	PCB 206	PCB 209	2,4'-DDE	4,4'-DDE	2,4'-DDD	4,4'-DDD	2,4'-DDT
NM1L002	ECD	2121 (42)	809 (78)	2442 (102)	75.1 (1.2)	11.1 (0.3)	112 (2)	43.4 (5.0)	71.2 (9.5)	1900 (62)	116 (8)	993 (39)	734 (32)
	MS	2111 (37)	834 (18)	2431 (62)	72.7 (1.8)	10.9 (0.4)	115 (4)	41.3 (1.7)	72.5 (1.5)	1852 (53)	121 (5)	1004 (51)	727 (22)
NM1B014	ECD	1062	212	1185	97.8	52.3	37.5	7.92	421	4970	279	3494	287
	MS	919	217	1242	104	53.7	37.1	8.82	419	4840	261	3341	259
NM1L016	ECD	648 (23)	159 (12)	672 (16)	18.1 (0.3)	≤5	26.5 (1.6)	5.50 (0.19)	69.7 (4.0)	2557 (234)	95.3 (1.5)	793 (44)	192 (4)
	MS	621 (14)	166 (15)	661 (12)	17.5 (0.2)	≤5	26.0 (0.8)	≤8	68.3 (0.9)	2512 (99)	99.7 (2.1)	782 (31)	199 (5)
NM1B018	ECD	451	92.1	450	52.4	27.3	25.3	4.27	291	2828	67.6	1177	16.0
	MS	448	106	442	55.0	30.1	24.3	≤7	271	2764	66.1	1132	15.3
NM1B020	ECD	712	185	1110	77.5	40.7	35.9	11.6	526	4148	115	3454	10.5
	MS	730	209	1106	71.9	39.5	36.0	11.1	501	4212	111	3399	11.3

Table F.1. Concentrations of PCB congeners and chlorinated pesticides in harbor porpoise (*Phocoena phocoena*) blubber (ng/g, wet mass). The uncertainty value, in parenthesis, is expressed as the standard deviation of a single measurement. - Sheet IV

Sample ID	Method	4,4'-DDT	HCB	*gamma*-HCH	*alpha*-HCH	heptachlor epox.	oxychlordane	*cis*-chlordane	*cis*-nonachlor	*trans*-nonachlor	dieldrin	mirex
NM1B002	ECD	2066 (422)	229 (58)	42.9 (8.2)	235 (47)	353 (73)	361 (105)	1130 (222)	706 (39)	2642 (533)	1494 (299)	104 (4)
	MS	2164 (94)	217 (11)	41.4 (1.5)	228 (14)	344 (16)	355 (42)	1090 (161)	688 (24)	2314 (101)	1409 (106)	99.6 (1.1)
NM1B014	ECD	1973	1078	154	715	568	809 1665	-	3450	899	≤9	
	MS	1934	1062	149	701	559	793 1606	-	3399	878	≤9	
NM1B016	ECD	1010 (15)	246 (16)	80.2 (3.5)	499 (16)	210 (12)	187 (8)	433 (13)	331 (10)	1943 (232)	884 (48)	33.4 (0.2)
	MS	995 (32)	229 (15)	79.2 (1.7)	505 (15)	209 (6)	179 (5)	421 (10)	319 (11)	1902 (51)	861 (22)	32.6 (0.6)
NM1B018	ECD	895	621	64.3	328	208	367	405	-	2585	665	≤9
	MS	881	664	66.2	331	199	365	389	-	2515	651	≤10
NM1B020	ECD	1127	409	67.6	255	202	418	542	-	2952	960	≤9
	MS	1101	393	66.0	264	191	410	538	-	2912	934	≤9

Table F.2. Concentrations of PCB congeners and chlorinated pesticides in pilot whale (*Globicephala melas*) blubber (ng/g, wet mass). The uncertainty value, in parenthesis, is expressed as the standard deviation of a single measurement. - Sheet I

Sample ID	% Lipid	Method	PCB 18	PCB 28	PCB 31	PCB 44	PCB 49	PCB 52	PCB 66	PCB 87	PCB 95	PCB 99	PCB 101/90
NM1B004	77 (3)	ECD[a]	6.65 (0.17)	15.5 (1.9)	11.4 (1.9)	22.7 (0.5)	-	43.9 (4.2)	79.6[c] (1.2)	23.9 (0.5)	79.6[c] (1.2)	38.8 (1.9)	43.6 (1.1)
		MS[b]	6.62 (0.25)	14.9 (0.9)	10.2 (0.4)	22.3 (0.9)	-	42.1 (1.1)	35.2 (2.8)	21.5 (0.6)	45.6 (1.8)	37.2 (0.6)	44.2 (0.6)
NM1B006	78.1	ECD[a]	≤7	8.48	≤7	16.8	24.3	66.5	26.9	38.2	94.4	170	341
		MS[b]	≤9	≤9	≤9	17.2	22.7	64.1	27.5	33.8	99.4	167	314
NM1B008	77.1	ECD[a]	≤6	47.5	≤6	76.2	111	280	160	158	310	293	473
		MS[b]	≤8	44.7	≤8	72.8	105	273	141	156	277	253	492
NM1B010	78 (3)	ECD[a]	7.53 (0.18)	8.34 (0.95)	10.9 (0.4)	23.6 (1.1)	-	88.6 (2.0)	105[c] (6)	35.8 (1.8)	105[c] (6)	87.7 (1.8)	93.6 (0.8)
		MS[b]	8.13 (0.27)	8.12 (0.33)	10.5 (0.4)	22.7 (0.6)	-	79.9 (1.3)	44.3 (1.9)	33.6 (0.8)	63.1 (2.1)	88.1 (0.8)	95.2 (1.0)
NM1B012	78.9	ECD[a]	≤6	47.4	≤6	96.7	153	371	195	210	483	440	760
		MS[b]	≤8	46.0	≤8	101	144	365	187	199	426	390	757
NM1B022	82.3	ECD[a]	32.8	141	52.3	180	311	815	397	269	609	1349	1212
		MS[b]	34.7	132	51.0	196	296	823	404	277	548	1178	1281
NM1B026	81.7	ECD[a]	36.1	123	38.9	179	415	965	396	401	692	1029	1292
		MS[b]	34.5	132	36.1	149	436	1000	382	379	621	1065	1351

[a] Gas chromatography with electron capture detection
[b] Gas chromatography with mass selective detection
[c] PCB 66 and PCB 95 coelute on the DB-5 column but are differentiated by the mass spectrometer

Table F.2. Concentrations of PCB congeners and chlorinated pesticides in pilot whale (*Globicephala melas*) blubber (ng/g, wet mass). The uncertainty value, in parenthesis, is expressed as the standard deviation of a single measurement.- Sheet II

Sample ID	Method	PCB 105	PCB 110/77	PCB 118	PCB 128	PCB 138/163/164	PCB 149	PCB 151	PCB 153	PCB 156	PCB 170/190	PCB 180
NM1B004	ECD	22.8 (0.9)	38.1 (0.4)	86.1 (1.1)	33.3 (0.8)	175 (5)	126 (18)	43.3 (0.3)	258 (4)	25.8 (0.8)	85.2 (1.8)	272 (18)
	MS	22.2 (0.4)	37.4 (0.3)	86.8 (0.8)	32.3 (0.3)	169 (5)	122 (5)	44.1 (0.5)	234 (9)	26.2 (0.5)	85.8 (0.8)	268 (9)
NM1B006	ECD	30.2	38.7	124	92.0	424	201	72.4	433	49.5	161	395
	MS	29.4	36.3	131	99.6	447	182	70.8	504	46.8	164	384
NM1B008	ECD	140	99.1	502	103	981	603	199	963	43.6	238	532
	MS	121	99.0	482	111	1084	612	188	1008	44.1	205	504
NM1B010	ECD	55.8 (2.8)	34.5 (0.4)	205 (5)	45.2 (0.4)	441 (12)	322 (17)	107 (5)	694 (49)	45.9 (0.9)	176 (3)	493 (5)
	MS	54.9 (0.5)	33.7 (0.5)	211 (3)	44.8 (0.3)	431 (6)	313 (10)	111 (4)	688 (17)	44.8 (1.3)	179 (5)	485 (7)
NM1B012	ECD	217	83.5	732	179	1343	794	267	1598	62.4	269	652
	MS	204	86.8	710	163	1419	846	241	1642	59.3	259	633
NM1B022	ECD	229	282	646	303	1196	581	283	1015	61.1	128	459
	MS	184	286	594	292	1158	508	242	1250	64.1	117	426
NM1B026	ECD	390	177	1248	48.2	2639	1184	318	2533	65.1	363	1195
	MS	404	177	1264	44.9	2558	1229	332	2798	64.7	330	1198

Table F.2. Concentrations of PCB congeners and chlorinated pesticides in pilot whale (*Globicephala melas*) blubber (ng/g, wet mass). The uncertainty value, in parenthesis, is expressed as the standard deviation of a single measurement.- Sheet III

Sample ID	Method	PCB 183	PCB 187	PCB194	PCB195	PCB 206	PCB 209	2,4'-DDE	4,4'-DDE	2,4'-DDD	4,4'-DDD	2,4'-DDT
NM1L004	ECD	87.7 (0.6)	231 (1)	97.9 (1.9)	≤5	29.4 (1.3)	19.4 (3.2)	41.5 (0.8)	938 (42)	30.6 (2.8)	121 (8)	132 (1)
	MS	88.5 (1.1)	233 (5)	99.1 (0.9)	≤8	28.1 (0.4)	20.1 (0.9)	43.9 (0.5)	947 (31)	33.0 (1.1)	117 (3)	141 (3)
NM1B006	ECD	94.3	298	82.3	40.1	48.2	12.8	126	2395	45.2	282	295
	MS	99.1	309	80.4	38.6	46.9	11.7	132	2458	44.1	292	287
NM1B008	ECD	111	544	99.7	49.3	80.3	33.8	492	4152	128	751	657
	MS	91.4	572	101.6	44.1	78.9	34.8	474	4420	139	762	665
NM1L010	ECD	181 (5)	475 (13)	102 (1)	18.9 (1.4)	38.8 (1.2)	9.62 (0.37)	75.4 (0.8)	2571 (281)	47.7 (1.5)	196 (9)	283 (12)
	MS	184 (6)	469 (15)	105 (3)	22.2 (0.8)	37.8 (1.0)	9.91 (0.4)	76.1 (0.7)	2456 (101)	44.8 (2.1)	198 (7)	273 (8)
NM1B012	ECD	140	648	85.3	40.4	43.3	9.38	760	7084	219	1097	1000
	MS	132	702	88.2	43.9	42.3	10.2	771	7151	211	1005	991
NM1B022	ECD	173	543	96.7	50.2	31.4	3.72	444	5841	201	2529	1086
	MS	171	515	93.7	48.3	29.6	≤8	417	5609	189	2495	1078
NM1B026	ECD	358	1271	155	72.2	50.3	9.06	283	4552	240	3089	1799
	MS	342	1339	143	74.8	49.2	9.87	280	4682	233	3002	1694

Table F.2. Concentrations of PCB congeners and chlorinated pesticides in pilot whale (*Globicephala melas*) blubber (ng/g, wet mass). The uncertainty value, in parenthesis, is expressed as the standard deviation of a single measurement.- Sheet IV

Sample ID	Method	4,4'DDT	HCB	*gamma*-HCH	*alpha*-HCH	heptachlor epox.	oxychlordane	*cis*-chlordane	*cis*-nonachlor	*trans*-nonachlor	dieldrin	mirex
NM1B004	ECD	440 (12)	53.1 (0.8)	4.53 (0.20)	28.4 (1.8)	15.1 (0.9)	26.9 (0.1)	70.0 (4.8)	40.2 (3.0)	283 (20)	77.8 (4.4)	61.7 (0.2)
	MS	434 (11)	52.6 (0.4)	4.41 (0.50)	27.9 (0.5)	14.6 (0.5)	26.5 (0.5)	66.6 (3.1)	38.4 (2.1)	277 (8)	76.9 (1.2)	62.3 (0.5)
NM1B006	ECD	573	52.5	3.42	19.2	14.0	41.6	74.7	-	405	66.7	62.1
	MS	604	50.8	≤5	20.8	15.1	39.9	76.2	-	435	63.7	61.3
NM1B008	ECD	1416	221	5.85	24.2	81.6	147	236	-	1355	404	77.4
	MS	1430	220	≤5	22.7	79.5	151	228	-	1491	398	76.1
NM1B010	ECD	756 (24)	43.2 (1.7)	4.26 (0.17)	27.2 (0.4)	21.3 (0.3)	48.8 (0.8)	114 (3)	128 (6)	624 (16)	102 (5)	74.9 (1.5)
	MS	744 (15)	42.8 (1.4)	4.31 (0.11)	27.1 (0.7)	20.8 (0.6)	49.2 (0.3)	110 (3)	122 (3)	612 (10)	109 (6)	74.1 (0.9)
NM1B012	ECD	1946	337	8.46	31.7	135	206	368	-	1959	551	60.9
	MS	1902	321	9.13	30.9	141	202	349	-	1903	498	56.9
NM1B022	ECD	2132	413	19.4	62.7	124	258	371	-	2289	615	≤9
	MS	2093	389	20.6	61.6	117	271	361	-	2243	594	≤10
NM1B026	ECD	3124	471	8.88	37.4	120	275	355	-	2441	574	≤9
	MS	3091	459	8.14	34.9	118	266	349	-	2394	561	≤9

Table F.3. Concentrations of PCB congeners and chlorinated pesticides in interlaboratory comparison exercise among NIST, DFO, and Ulm on pilot whale (*Globicephala melas*) QA blubber (ng/g wet mass) - Sheet I

Sample ID	PCB 18	PCB 28	PCB 31	PCB 44	PCB 49	PCB 52	PCB 66	PCB 87	PCB 95	PCB 99
NIST[a]	6.12 ± 0.86	20.2 ± 0.5	2.59 ± 0.14	17.8 ± 1.9	34.8 ± 0.5	103 ± 10	50.4 ± 8.5	66.7 ± 0.7	50.1 ± 0.1	254 ± 4
DOF[b]	≤5.1	29.9 (8.6)	NA[d]	19.9 (2.2)	48.2 (12.9)	86.5 (5.9)	≤1.6	NA	202 (12)	162 (11)
Ulm[c]	NA	26 (2)	25 (1)	35 (1)	57 (2)	120 (1)	61 (2)	NA	143 (2)	159 (2)

[a] NIST values are the means of the results from all three methods with uncertainties expressed as 95% confidence intervals.
[b] Five subsamples analyzed, one from each of three bottles and two from one bottle.
[c] Subsamples from each of three bottles analyzed.
[d] NA = not analyzed

Table F.3. Concentrations of PCB congeners and chlorinated pesticides in interlaboratory comparison exercise among NIST, DFO, and Ulm on pilot whale (*Globicephala melas*) QA blubber (ng/g wet mass) - Sheet II

Sample ID	PCB 101	PCB 105	PCB 110	PCB 118	PCB 128	PCB 138	PCB 149	PCB 151	PCB 153	PCB 156	PCB 170
NIST[a]	261 ± 17	88.9 ± 13.0	[91.2 ± 0.8][e]	267 ± 25	99.0 ± 0.7	664 ± 8	372 ± 6	111 ± 2	870 ± 9	38.2 ± 0.7	156 ± 1
DOF[b]	231 (15)	61.5 (3.0)	52.8 (3.7)	217 (9)	4.77 (3.36)	489 (24)	274 (14)	94.7 (5.4)	582 (33)	47.9 (3.2)	129 (6)
Ulm[c]	248 (3)	82 (2)	47 (3)	217 (4)	77 (2)	506 (8)	288 (5)	107 (2)	618 (8)	39 (2)	118 (4)

[a] NIST values are the means of the results from all three methods with uncertainties expressed as 95% confidence intervals.
[b] Five subsamples analyzed, one from each of three bottles and two from one bottle.
[c] Subsamples from each of three bottles analyzed.
[e] Values in [] indicate known coelution of two or more congeners; PCB 77 (3,3',4,4'-tetrachlorobiphenyl) coeluted with PCB 110.

Table F.3. Concentrations of PCB congeners and chlorinated pesticides in interlaboratory comparison exercise among NIST, DFO, and Ulm on pilot whale (*Globicephala melas*) QA blubber (ng/g wet mass) - Sheet III

Sample ID	PCB 180	PCB 183	PCB 187	PCB194	PCB195	PCB 201	PCB 206	PCB 209	2,4'-DDE	4,4'-DDE
NIST[a]	483 ± 9	147 ± 1	357 ± 8	69.8 ± 2.6	16.3 ± 0.8	22.7 ± 0.7	29.0 ± 0.4	7.98 ± 0.10	53.4 ± 7.4	2032 ± 46
DOF[b]	322 (17)	109 (3)	317 (17)	59.2 (3.5)	17.1 (6.9)	25.4 (16.2)	23.6 (1.1)	≤0.9	49.4 (1.4)	2076 (92)
Ulm[c]	372 (4)	117 (0)	NA[d]	NA	20 (1)	NA	NA	NA	62 (4)	1993± (20)

[a] NIST values are the means of the results from all three methods with uncertainties expressed as 95% confidence intervals.
[b] Five subsamples analyzed, one from each of three bottles and two from one bottle.
[c] Subsamples from each of three bottles analyzed.
[d] NA = not analyzed.

Table F.3. Concentrations of PCB congeners and chlorinated pesticides in interlaboratory comparison exercise among NIST, DFO, and Ulm on pilot whale (*Globicephala melas*) QA blubber (ng/g wet mass) - Sheet IV

Sample ID	2,4'-DDD	4,4'DDD	2,4'DDT	4,4'-DDT	HCB	*gamma*-HCH	*alpha*-HCH	*beta*-HCH	heptachlor epox.	oxychlordane
NIST[a]	58.4 ± 3.5	260 ± 31	222 ± 11	651 ± 26	36.9 ± 1.1	3.58 ± 0.35	20.8 ± 2.6	NA[d]	32.5 ± 2.7	73.5 ± 0.8
DOF[b]	63.2 (3.1)	313 (7)	293 (16)	494 (19)	40.1 (3.3)	3.7 (0.2)	22.9 (1.5)	6.5 (1.6)	28.3 (1.5)	66.2 (1.4)
Ulm[c]	63 (2)	346 (18)	363 (12)	800 (55)	41 (3)	≤10	22 (0.5)	≤10	NA	72 (2)

[a] NIST values are the means of the results from all three methods with uncertainties expressed as 95% confidence intervals.
[b] Five subsamples analyzed, one from each of three bottles and two from one bottle.
[c] Subsamples from each of three bottles analyzed.
[d] NA = not analyzed.

Table F.3. Concentrations of PCB congeners and chlorinated pesticides in interlaboratory comparison exercise among NIST, DFO, and Ulm on pilot whale (*Globicephala melas*) QA blubber (ng/g wet mass) - Sheet V

Sample ID	*cis*-chlordane	*cis*-nonachlor	*trans*-nonachlor	dieldrin	mirex
NIST[a]	107 ±2	111 ± 3	648 ± 25	115 ± 3	74.5 ± 0.9
DOF[b]	81.8 (2.7)	150 (5)	503 (19)	95.1 (3.6)	33.0 (9.9)
Ulm[c]	82 (1)	137 (5)	600 (9)	90 (4)	83 (4)

[a] NIST values are the means of the results from all three methods with uncertainties expressed as 95% confidence intervals.
[b] Five subsamples analyzed, one from each of three bottles and two from one bottle.
[c] Subsamples from each of three bottles analyzed.
[d] NA = not analyzed.

APPENDIX G

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Development of frozen whale blubber and liver reference materials for the measurement of organic and inorganic contaminants

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Summary. Fresh frozen homogenates of pilot whale blubber and liver tissue were prepared for use as control materials for the determination of organic and inorganic contaminants in marine mammal tissue analyses. The blubber material was analyzed to determine 30 polychlorinated biphenyl congeners and 16 chlorinated pesticides using gas chromatography with electron capture detection and gas chromatography-mass spectrometry. A total of 39 trace elements and methylmercury were determined in the liver homogenate using instrumental neutron activation analysis, voltammetry, and cold vapor atomic absorption spectroscopy. The preparation and analysis of these two tissue materials are part of the development of marine mammal tissue reference materials.

Introduction

Because of increasing concerns about the effects of marine pollution on the health of marine mammals, the National Oceanic and Atmospheric Administration (NOAA) is developing a broad program consisting of four components: stranding networks, the National Marine Mammal Tissue Bank, contaminant monitoring, and quality assurance of chemical measurements. Details concerning the development of this program have been published recently by Lillestolen et al. [1]. As part of the monitoring and tissue banking efforts, a number of laboratories are involved in the analysis of marine mammal tissues to measure the levels of various trace elements and organic contaminants. In addition numerous other laboratories world-wide are involved in similar analyses of marine mammal tissues for research and other international monitoring programs. To assess the accuracy and comparability of results among various laboratories for marine mammal tissue analyses, NOAA and the National Institute of Standards and Technology (NIST) have initiated a quality assurance (QA) program for analytical measurements of contaminants in marine mammal tissues. This QA program consists of (1) preparation, analysis, and distribution of marine mammal tissue control materials; (2) interlaboratory comparison exercises among NIST and other laboratories participating in NOAA projects involving marine mammal tissue analyses; and (3) development of Standard Reference Materials (SRMs) for use in the analysis of marine mammal tissues.

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In December 1990 approximately 2 kg each of blubber and liver were collected from stranded pilot whales at Hyannis, Cape Cod (USA). The blubber and liver samples were used to prepare tissue homogenates for use as analytical control materials and in interlaboratory comparison exercises for organic and inorganic analyses, respectively. The tissues were cryogenically pulverized and homogenized in Teflon mills to provide frozen, powder-like materials [2]. The liver and blubber samples from the two whales were homogenized in 150-g batches and then combined in a Teflon bag and thoroughly mixed to prepare the final control material homogenates. The homogenates were subsampled with a Teflon scoop into clean glass jars with Teflon-lined lids (~ 15–20 g per jar). Approximately 120 jars of the blubber homogenate and 170 jars of the liver homogenate were prepared. These materials are stored at −80 °C. These homogenates are fresh frozen samples, similar to marine mammal tissue samples routinely analyzed, rather than freeze-dried matrices. These two control materials were analyzed at NIST to determine concentrations of organic contaminants and trace elements and to assess homogeneity.

A high priority in the NOAA Marine Mammal QA Program is the comparability of organic analytical measurements specifically for polychlorinated biphenyl (PCB) congeners and chlorinated pesticides. The blubber control material was analyzed using gas chromatography (GC) on two stationary phases with different selectivity for PCB congener separations and with two different GC detectors, i.e., electron capture detection (ECD) and mass spectrometry (MS). A total of 30 PCB congeners and 16 chlorinated pesticides were measured in the blubber control. The whale liver homogenate was analyzed at NIST using instrumental neutron activation analysis (INAA) to determine 38 trace elements. Differential pulse and square wave stripping voltammetry and cold vapor atomic absorption spectrometry (CVAAS) were performed at KFA Jülich to provide results for additional elements (Ni and Pb) as well as Co, Cu, Zn, Cd, and Hg, which were also measured by INAA.

Experimental section

Determination of PCB congeners and chlorinated pesticides

The basic sample preparation procedure for the analysis of marine mammal blubber samples has been described previously by Schantz et al. [3]. Subsamples of the blubber

homogenate from three different bottles and three portions from one bottle were analyzed. The methylene chloride extracts of the blubber samples were cleaned up using size exclusion chromatography (SEC) on a preparative-scale divinylbenzene-polystyrene column followed by a normal-phase liquid chromatography (LC) procedure on a semi-preparative aminopropylsilane column to isolate two fractions containing (1) the PCB congeners and lower polarity chlorinated pesticides (i.e., 4,4′-DDE, 2,4′-DDE, and mirex) and (2) the more polar chlorinated pesticides. The two fractions were analyzed using three different approaches: (1) GC-ECD on a 5% phenyl-substituted methylpolysiloxane phase (DB-5) fused silica capillary column (0.25 mm × 60 m, 0.25 μm film thickness; J&W Scientific, Inc., Folsom, CA, USA); (2) GC-ECD on a 5% phenyl-substituted methylpolysiloxane (CP-SIL 8) with 10% methyl-C-18 incorporated phase (C-18) fused silica capillary column (0.25 mm × 60 m, 0.25 μm film thickness; Chrompack International, Middelburg, The Netherlands); and (3) GC with mass selective detection (GC-MS) on a DB-5 column similar to the one used for the GC-ECD analyses. Two aliquots of SRM 1588, Organics in Cod Liver Oil, (NIST, Gaithersburg, MD, USA) were included as quality control samples during the analyses of the whale blubber samples. Response/recovery solutions were prepared by gravimetrically diluting SRM 2261 [Chlorinated Pesticides in Hexane (Nominal Concentration 2 μg/mL)], SRM 2262 [Chlorinated Biphenyl Congeners in 2,2,4-Trimethylpentane (Nominal Concentration 2 μg/mL)] (NIST, Gaithersburg, MD, USA), and two additional solutions containing 11 PCB congeners and four pesticides, respectively, and adding a known amount of the internal standard solution. The response/recovery solutions were processed in the same manner as the blubber samples. The pilot whale blubber was found to contain 14.8 ng/g of PCB 198; therefore, results determined using PCB 198 as the internal standard were corrected to account for the natural presence of this congener.

Trace element determination by INAA

INAA was performed to determine the trace element content and to assess the variability of trace element concentrations in the liver tissue control material. Six jars of the liver homogenate were randomly selected and freeze dried at 1 Pa, −20 °C shelf temperature and −50 °C condenser temperature for five days. Six portions from one jar and three portions from each of the other five jars were used for the analysis. Each portion weighing ~ 200 mg was formed into a disk-shaped pellet and packaged in linear polyethylene film for irradiation. Standards consisting of solutions dried on filter paper and two portions of SRM 1577a, Bovine Liver (NIST, Gaithersburg, MD) and NIES #6 Mussel Tissue (National Institute of Environmental Studies, Tsukuba, Japan) were prepared in the same manner. For the determination of Hg, portions of ~ 100 to 200 mg of the freeze-dried powder were sealed in acid washed quartz tubing. This type of sample packaging is required for the determination of Hg because Hg vapor formed during irradiation may pass through polyethylene film. The standards used for Hg consisted of aqueous Hg solutions contained in sealed quartz vials. The standard solutions in quartz were flash frozen in liquid nitrogen before sealing to avoid evaporative losses.

The INAA procedure used to analyze the whale liver control material has been described in detail elsewhere [4, 5, 6]. Irradiation, decay, and counting times were chosen to optimize the number of elements determined and the detection limits for each. All irradiations were carried out at the NIST Reactor at a reactor power of 15 MW which corresponds to a fluence rate of approximately 2×10^{13} n $\cdot$ cm$^{-2} \cdot$ s^{-1}. Two irradiations, the first for 120 s and the second for 16 h, were used. Approximately 90 s after the first irradiation, the samples were counted for 5 min to determine elements for which the product nuclides possess very short half-lives (Mg, Al, Cl, Ca, Ti, V, Cu, I). After several hours of decay, samples are counted again for 20 min to determine nuclides that possess half-lives on the order of a few hours (Na, K, and Mn). After the second irradiation and a decay time of approximately 6 days, each sample was counted for 4 h to determine concentrations of As, Cd, Sb, La, Sm, and Au; after a decay time of 1 to 2 months, the samples were counted to determine concentrations of Sc, Cr, Fe, Co, Zn, Se, Rb, Sr, Mo, Ag, Sb, Cs, Ba, Ce, Eu, Tb, Hf, Ta, and Th. For the determination of Hg, packaged in quartz vials and irradiated for 3 h at a reactor power of 15 MW, corresponding to a fluence rate of approximately 7.7×10^{13} n $\cdot$ cm$^{-2} \cdot$ s^{-1}. These samples were counted for 8 h after a decay time of 2 months. All data reduction and analysis were accomplished using a μVAX computer and Nuclear Data NAA software programs.

Voltammetry and cold vapor AAS

Differential pulse and square wave stripping voltammetry was performed at KFA Jülich to determine Co, Ni, Zn, Cu, Cd, and Pb. The voltammetric determinations were carried out according to previously published procedures for biological and environmental samples [7], after high pressure ashing digestion with nitric acid [8]. Cold vapor atomic absorption spectrometry (CVAAS) to determine Hg and methylmercury was also performed at KFA Jülich. CVAAS was performed after wet digestion in completely closed quartz vessels for total Hg content [9]. The methyl-mercury (methyl-Hg) was extracted from the sample by 6 mol/l HCl and separated on an anion exchange column [10]. After UV digestion, the Hg was determined in the eluate by CVAAS. The reported values for CVAAS and voltammetry are based on two sample dissolutions from three jars of freeze-dried tissue, which were each analyzed in duplicate. Two bottles of frozen material were analyzed for Hg in addition to the three bottles of freeze-dried material.

Results and discussion

PCB congeners and chlorinated pesticides

For the determination of organic contaminants, a list of 39 PCB congeners and 19 chlorinated pesticides was targeted for measurement, i.e., those analytes in SRMs 2261 and 2262 plus additional analytes typically found in marine mammal tissues. The blubber extracts were analyzed using two gas chromatographic columns with different selectivity (DB-5 and C-18) and two different GC detectors (electron capture and mass spectrometric). These three approaches have been compared previously for the analysis of SRM 1588 and two other fish oil reference materials [11, 12]. The results of the techniques for the determination of PCB congeners and chlorinated pesticides in the blubber control material are summarized in Tables 1 and 2, respectively. Results in Tables 1 and 2 are reported on a wet weight basis; however,

Table 1. Concentrations (μg/kg wet weight) of select PCB congeners in whale blubber QA material[a]

Congener	GC-ECD (DB-5)	GC-ECD (C-18)	GC-MS (DB-5)	Recommended value[b]
PCB 18 (2,2′,5-Trichlorobiphenyl)	6.34 (0.23)	5.69 (0.20)	6.40 (0.32)	6.12 ± 0.86
PCB 28 (2,4,4′-Trichlorobiphenyl)	19.4 (3.0)	20.0 (0.6)	20.4 (0.8)	20.2 ± 0.5
PCB 31 (2,4′,5-Trichlorobiphenyl)	2.62 (0.24)	2.54 (0.17)	2.61 (0.20)	2.59 ± 0.14
PCB 44 (2,2′,3,5′-Tetrachlorobiphenyl)	17.2 (1.5)	18.5 (0.9)	17.6 (1.6)	17.8 ± 1.9
PCB 49 (2,2′,4,5′-Tetrachlorobiphenyl)	34.9 (0.8)	34.5 (0.7)	35.1 (0.7)	34.8 ± 0.5
PCB 52 (2,2′,5,5′-Tetrachlorobiphenyl)	104 (6)	104 (6)	100 (5)	103 ± 10
PCB 66 (2,3′,4,4′-Tetrachlorobiphenyl)	[104 (6)][c]	48.7 (4.5)	52.2 (2.7)	50.4 ± 8.5
PCB 87 (2,2′,3,4,5′-Pentachlorobiphenyl)	66.7 (1.3)	66.3 (1.2)	67.3 (1.5)	66.7 ± 0.7
PCB 95 (2,2′,3,5′,6-Pentachlorobiphenyl)	[104 (6)][c]	50.3 (0.9)	50.0 (1.0)	50.1 ± 0.1
PCB 99 (2,2′,4,4′,5-Pentachlorobiphenyl)	256 (10)	250 (11)	257 (10)	254 ± 4
PCB 101 (2,2′,4,5,5′-Pentachlorobiphenyl)	257 (21)	259 (16)	266 (19)	261 ± 17
PCB 105 (2,3,3′,4,4′-Pentachlorobiphenyl)	88.2 (6.1)	87.6 (7.8)	84.5 (6.0)	88.9 ± 13.0
PCB 110 (2,3,3′,4′,6-Pentachlorobiphenyl)	[110 (7)][d]	91.0 (2.0)	91.1 (1.7)	91.2 ± 0.8
PCB 118 (2,3′,4,4′,5-Pentachlorobiphenyl)	265 (17)	270 (18)	267 (21)	267 ± 25
PCB 128 (2,2′,3,3′,4,4′-Hexachlorobiphenyl)	100 (2.1)	99.1 (1.7)	98.7 (2.3)	99.0 ± 0.7
PCB 138 (2,2′,3,4,4′,5′-Hexachlorobiphenyl)	668 (22)	671 (19)	656 (24)	664 ± 8
PCB 149 (2,2′,3,4′,5′,6-Hexachlorobiphenyl)	372 (9)	374 (9)	373 (10)	372 ± 6
PCB 151 (2,2′,3,5,5′,6-Hexachlorobiphenyl)	112 (4)	109 (3)	111 (2)	111 ± 2
PCB 153 (2,2′,4,4′,5,5′-Hexachlorobiphenyl)	872 (21)	864 (16)	880 (22)	870 ± 9
PCB 156 (2,3,3′,4,4′,5-Hexachlorobiphenyl)	37.7 (2.7)	39.0 (1.3)	37.8 (1.2)	38.2 ± 0.7
PCB 170 (2,2′,3,3′,4,4′,5-Heptachlorobiphenyl)	155 (4)[e]	157 (4)	155 (3)	156 ± 1
PCB 180 (2,2′,3,4,4′,5,6-Heptachlorobiphenyl)	492 (34)	479 (12)	481 (24)	483 ± 9
PCB 183 (2,2′,3,4,4′,5′,6-Heptachlorobiphenyl)	144 (3)	148 (2)	148 (4)	147 ± 1
PCB 187 (2,2′3,4′,5,5′,6-Heptachlorobiphenyl)	358 (19)	353 (10)	362 (16)	357 ± 8
PCB 194 (2,2′,3,3′,4,4′,5,5′-Octachlorobiphenyl)	70.4 (2.3)	68.7 (2.6)	70.4 (2.6)	69.8 ± 2.6
PCB 195 (2,2′,3,3′,4,4′,5,6-Octachlorobiphenyl)	16.3 (0.4)	16.4 (0.6)	16.3 (0.7)	16.3 ± 0.8
PCB 200 (2,2′,3,3′,4,5′,6,6′-Octachlorobiphenyl)	22.3 (2.0)	23.4 (1.1)	22.4 (1.2)	22.7 ± 0.7
PCB 206 (2,2′,3,3′,4,4′,5,5′,6-Nonachlorobiphenyl)	28.9 (1.3)	29.1 (1.0)	29.1 (1.1)	29.0 ± 0.4
PCB 209 (Decachlorobiphenyl)	8.00 (0.28)	7.99 (0.17)	7.35 (2.17)	7.98 ± 0.10

[a] Six aliquots of the whale blubber QA material were analyzed in duplicate; concentration value is the mean value, and number in parentheses are one standard deviation of a single measurement.

[b] Recommended values are the means of the results from all three methods with uncertainties expressed as 95% confidence intervals.

[c] PCB 66 and PCB 95 coelute under the conditions used.

[d] PCB 77 and PCB 110 coelute under the conditions used.

[e] PCB 170 and PCB 190 (2,2′,3′,4,4′,5,6-heptachlorobiphenyl) coelute on the DB-5 column; however, PCB 190 has little or no contribution based on the GC-ECD (C-18) results since PCB 170 and PCB 190 are separated on this column

Table 2. Concentrations (μg/kg wet weight) of selected chlorinated pesticides in whale blubber QA material[a]

Compound	GC-ECD (DB-5)	GC-ECD (C-18)	GC-MS (DB-5)	Recommended value[b]
2,4′-DDE	53.3 (4.6)	54.1 (3.8)	52.7 (4.3)	53.4 ± 7.4
4,4′-DDE	1710 (130)	1750 (140)	1875 (291)	1750 ± 64
2,4′-DDD	56.5 (5.5)	59.7 (3.5)	59.1 (3.2)	58.4 ± 3.5
4,4′-DDD	270 (22)	250 (27)	259 (16)	260 ± 31
2,4′-DDT	219 (6)	225 (5)	223 (12)	222 ± 11
4,4′-DDT	646 (32)	651 (36)	656 (20)	651 ± 26
Hexachlorobenzene	35.6 (2.7)	36.1 (2.8)	35.3 (2.3)	36.9 ± 1.1
γ-HCH	3.71 (0.20)	3.43 (0.22)	3.61 (0.27)	3.58 ± 0.35
α-HCH	22.1 (2.8)	19.6 (1.8)	20.8 (2.1)	20.8 ± 2.6
Heptachlor epoxide	32.7 (3.7)	32.8 (3.5)	32.0 (2.4)	32.5 ± 2.7
Oxychlordane	73.3 (1.6)	74.3 (1.9)	73.1 (1.0)	73.5 ± 0.8
cis-Chlordane	107 (6)	110 (5)	105 (3)	107 ± 2
cis-Nonachlor	112 (5)	112 (8)	108 (7)	111 ± 3
trans-Nonachlor	638 (23)	650 (23)	654 (12)	648 ± 25
Dieldrin	116 (7)	114 (7)	116 (4)	115 ± 3
Mirex	74.5 (2.6)	74.3 (2.0)	74.3 (3.1)	74.5 ± 0.9

[a] Six aliquots of the whale blubber QA material were analyzed in duplicate; concentration value is the mean value, and numbers in parentheses are one standard deviation of a single measurement.

[b] Recommended values are the means of the results from all three methods with uncertainties expressed as 95% confidence intervals

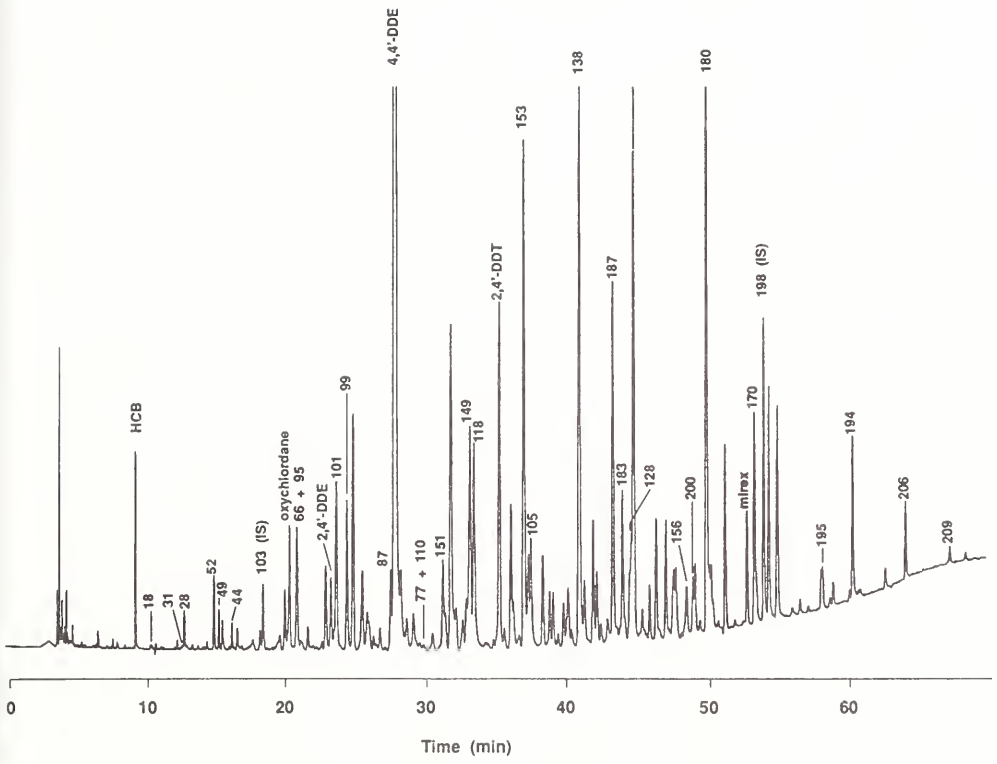

Fig. 1. GC-ECD analysis of the PCB and lower polarity pesticide fraction from the whale blubber control material on a DB-5 column

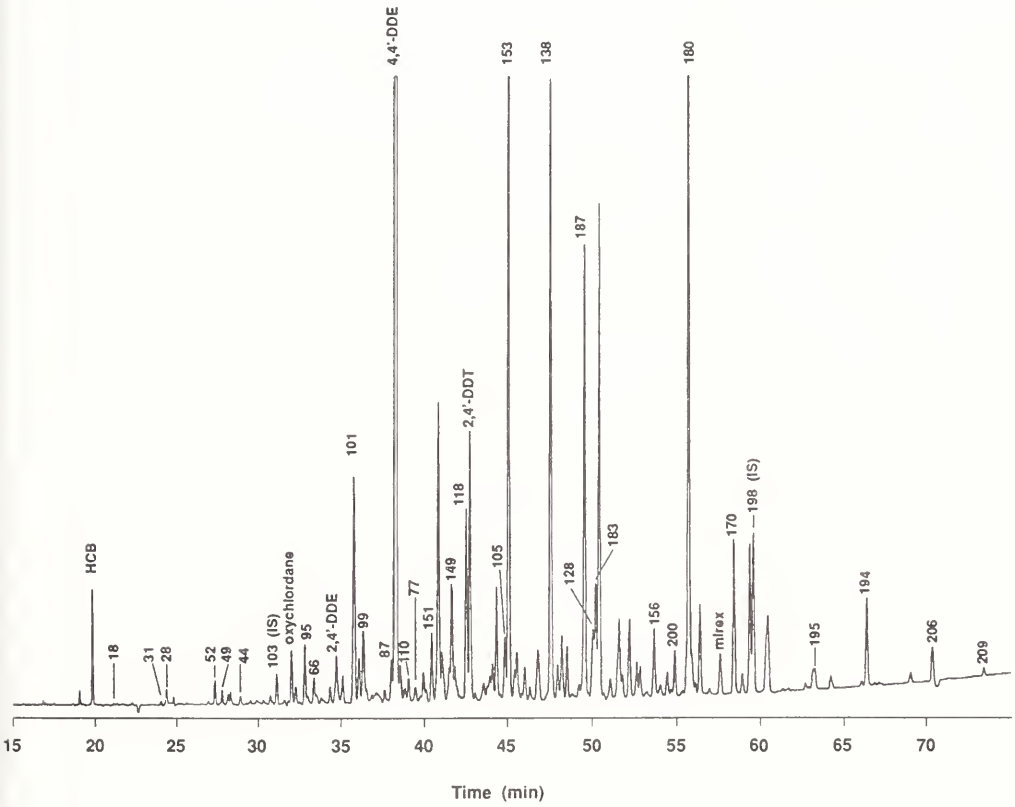

Fig. 2. GC-ECD analysis of the PCB and lower polarity pesticide fraction from the whale blubber control material on a C-18 column

for conversion to a fat extractable basis, 76 ± 2% of the material was extractable in methylene chloride. Chromatograms for the GC-ECD analyses of the PCB and lower polarity pesticide fraction on the two different columns are shown in Figs. 1 and 2. The chromatogram from the GC-ECD analysis of the more polar pesticide fraction on the DB-5 column is shown in Fig. 3.

Using the approach described above, 30 PCB congeners and 16 chlorinated pesticides were measured in the blubber samples (see Tables 1 and 2). Concentrations are not reported for the remaining nine PCB congeners (PCBs 1, 8, 29, 50, 104, 126, 154, 169, and 188), heptachlor, and aldrin since they were not detected in the pilot whale sample. The concentration of β-HCH is also not reported since it is not

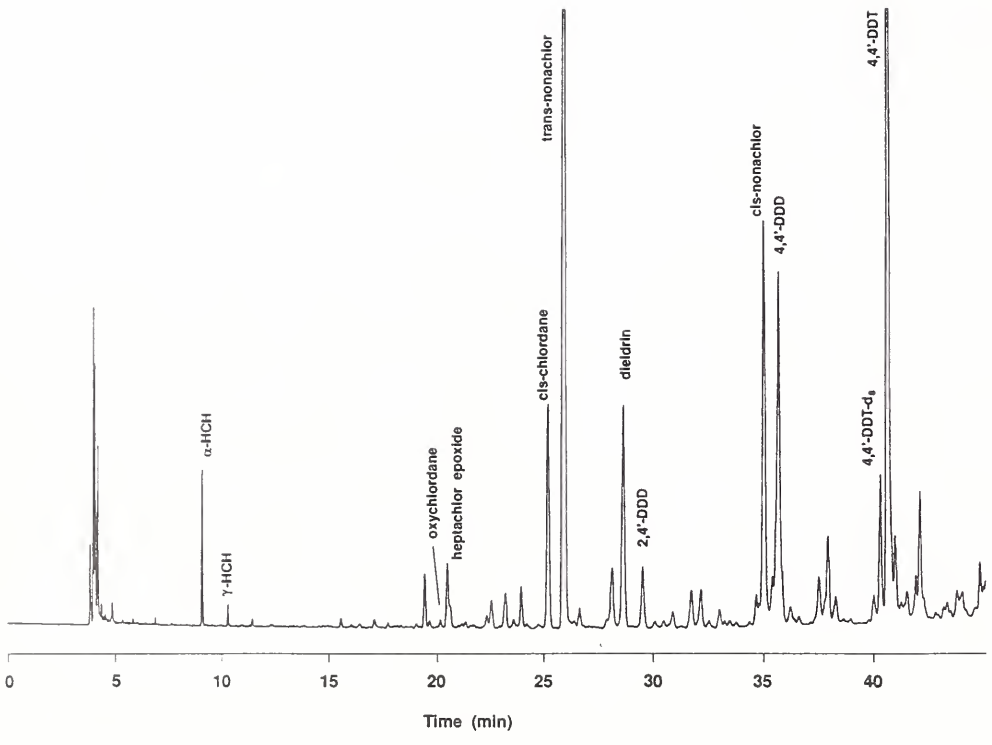

Fig. 3. GC-ECD analysis of the polar pesticide fraction from the whale blubber control material on a DB-5 column

recovered during the normal-phase LC cleanup procedure. The results obtained from the three different methods are generally in good agreement and discrepancies can be attributed to the separation of coeluting congeners due to differences in selectivity of the GC columns or the measurement of coeluting congeners due to the selectivity of the mass spectrometric detection. Recommended concentrations for the PCB congeners and chlorinated pesticides in the blubber homogenate were determined based on the mean of the results from the three methods with uncertainties expressed as 95% confidence intervals. PCB congener measurements are typically performed using a stationary phase similar to the DB-5. However several pairs of congeners of interest are typically not separated on the DB-5 column (e.g., PCB 66/PCB 95, PCB77/PCB 110, and PCB 170/190). These pairs were separated using the C-18 phase which has selectivity similar to the C-8 polysiloxane phase particularly for the di-through hepta-chlorobiphenyls [13, 14]. Ballschmiter et al. [15] and de Boer et al. [16] recently characterized the C-18 phase for PCB congener selectivity. The predominant PCB congeners in the pilot whale blubber control material are the hexachlorobiphenyl and heptachlorobiphenyl congeners (PCB 138, PCB 149, PCB 153, PCB 180, and PCB 187) at levels of 360-870 μg/kg.

Trace elements

The average concentration for each element determined in the liver homogenate is listed in Table 3. Although measurements were performed on dried portions, the concentrations are reported as mg/kg of wet weight; the average ratio of dry to wet weight for the six bottles of the liver homogenate was 0.273. Quantitative evaluation was possible for 18 of the 38 elements assayed in the pilot whale liver tissue. For the remaining 20 elements, a limit of detection (L_D, as defined in reference [17]) is listed for each element in Table 4. The mean values listed in Table 3 represent either a weighted or an unweighted mean (as indicated). The overall estimated analytical uncertainty for each element determined by INAA is listed in Table 3; this value is $ts/\sqrt{n}$ with an additional 2% combined in quadrature.

To assess the homogeneity of the material, the observed sample to sample variations determined by INAA were compared with the uncertainty associated with counting statistics combined in quadrature with an additional 2%, the estimate of the uncertainty associated with irradiation and counting geometry variations. For 13 of the 18 elements measured (Na, Mg, Cl, K, Mn, Fe, Co, Cu, Zn, Se, Rb, Ag, and Cd), the uncertainties associated with counting statistics account for the variation in concentration from sample to sample. For the remaining five elements (Al, Ca, Cr, As and Cs), sample to sample variations were greater than could be accounted for by this source of error. Results for two elements, Cr and Al, had particularly large variations. Chromium concentrations measured in the liver tissue control material ranged from 0.022 to 0.083 mg/kg. However Cr concentrations measured in the NIES Mussel Tissue agreed well with certified values, i.e., 0.59 ± 0.03 and 0.56 ± 0.03 mg/kg for two samples of NIES Mussel compared to the certified value of 0.63 ± 0.07 mg/kg. Chromium was not determined for NIST SRM 1577a since this material is known to be inhomogeneous with respect to Cr. One source of Cr contamination may be the linear polyethylene film in which the samples are packaged. Zeisler et al. [18] have reported that Cr present in the polyethylene bags is transferred to the sample during irradiation and may account for 8 ± 2 ng of Cr per g of sample. However, in addition to being too low to account for the observed variations, the Cr content of the film is usually uniform, whereas Cr contamination of the whale liver tissue material appears to be inhomogeneous. A preliminary study has indicated that the Teflon disk mill used for cryogenic homogenization of the tissue introduces Cr contamination at levels of ~ 0.03 to 0.08 mg/kg, comparable to the levels measured in the pilot whale liver tissue. It is likely that Cr contamination is a result of the cryogenic milling process.

Table 3. Concentrations (mg/kg wet weight) for pilot whale liver control material

	INAA			Voltammetry/CVAAS	
Element	Mean ± uncertainty[a]		1s (Mean)[b]	Mean[c]	1s (Mean)[b]
Na	1260	± 30	(6)		
Mg	138	± 5	(2)		
Cl	1730	± 40	(10)		
K	2640	± 70	(20)		
Ca	46.0	± 2.7	(1.8)		
Cr	0.042	± 0.031[d]	(0.003)		
Mn	2.81	± 0.079	(0.023)		
Fe	438	± 10	(1)		
Co	0.014[e]	± 0.0006	(0.00037)	0.010	(0.003)
Ni				0.0268	(0.0045)
Cu	2.96[e]	± 0.20	(0.137)	3.13	(0.15)
Zn	32.2	± 0.7	(0.1)	32.1	(3.5)
As	0.529	± 0.017	(0.006)		
Se	11.0	± 0.3	(0.1)		
Rb	2.00	± 0.06	(0.015)		
Ag	0.181[e]	± 0.005	(0.0009)		
Cd	8.51[e]	± 0.22	(0.050)	8.60	(0.30)
Cs	0.00604	± 0.0004	(0.00024)		
Hg	28.2	± 1.1	(0.6)	26.5	(1.5)
Methyl-Hg				1.36	(0.13)
Pb				0.50[f]	(0.41)

[a] Overall estimated analytical uncertainty at the 95% confidence level. This value is $ts/\sqrt{n}$ with an additional 2% combined in quadrature; number of samples (n) analyzed was 16 or 20 except for Hg where n = 7.
[b] 1s is the standard deviation of the mean.
[c] Result is the mean value from three bottles, analyzed in duplicate (n = 6).
[d] Cr concentration is inhomogeneous; see discussion in text.
[e] Result is the weighted mean.
[f] Measured concentrations ranged from 0.25 to 1.4 mg/kg

Table 4. Limits of detection (L_D in mg/kg wet weight) for pilot whale liver control material as determined by INAA

Element	L_D	Element	L_D
Al	≤ 0.2–8[a]	La	≤ 0.003
Sc	≤ 0.08	Ce	≤ 0.02
Ti	≤ 8	Sm	≤ 0.02
V	≤ 0.02	Eu	≤ 0.0006
Sr	≤ 0.8	Tb	≤ 0.0007
Mo	≤ 0.4	Hf	≤ 0.0008
Sn	≤ 2	Ta	≤ 0.0005
Sb	≤ 0.08	Au	≤ 0.0002
I	≤ 1	Th	≤ 0.001
Ba	≤ 5	U	≤ 0.004

[a] Range of values determined

For 10 of the 16 portions assayed for Al, the levels were too low to be detected while the values for the remaining six portions ranged from 0.6 to 8 mg/kg. The results for Al measured in the NIES Mussel were in agreement with literature values (249 ± 1 and 259 ± 1 mg/kg compared to an information value of 220 mg/kg), but results were lower than the information value for SRM 1577a (0.91 ± 0.22 and 1.59 ± 0.21 mg/kg compared to 2 mg/kg). There appears to be inhomogeneous Al contamination in this material. Possible sources of Al contamination are the glass in which the material is stored and sand that may have contaminated the tissue during the collection of the samples. For the As results, an additional 3.5% uncertainty would be required to account for the observed sample to sample variation; for Ca and Cs results an additional 5% and 7.5%, respectively, would be required. Concentrations measured in the two reference materials for Ca and As agree well with the certified values, and the concentration for Cs in NIES Mussel Tissue agrees with results from previous measurements. The source of the additional uncertainty for these elements in the pilot whale material is not known but may be due to material inhomogeneity for these elements.

The whale liver material was also analyzed using voltammetry to determine Co, Ni, Cu, Zn, Cd, and Pb and using CVAAS to determine Hg. These analyses provided results for two additional elements (i.e., Ni and Pb) and a comparison of different analytical techniques for five elements. The results of these analyses are also summarized in Table 3. The results for the different techniques were in good agreement for Cu, Zn, Cd, and Hg. The results for Pb ranged from 0.25 to 1.4 mg/kg which may be an indication of the difficulty of measuring such low levels of Pb by this technique or an indication of potential inhomogeneity for this element. Methyl-Hg was found at 1.36 mg/kg, which represents 4.8% of the total Hg concentration in the liver homogenate. Methyl-Hg was determined in both fresh frozen and freeze-dried samples to see whether losses occurred during freeze drying, and the results were found to be comparable.

Marine mammal tissue SRM development

One of the goals of the NOAA Marine Mammal QA program is to develop SRMs for use in validating analytical measurements of trace elements and organic contaminants in marine mammal tissues. Blubber was selected as the first priority tissue and liver as the second priority tissue for development of SRMs.

SRM 1945, Whale Blubber. In September 1991 approximately 15 kg of blubber was collected from another stranding of pilot whales on Cape Cod. This blubber material was cryogenically pulverized and homogenized in the same manner as described for the control materials. This frozen blubber homogenate will be issued as SRM 1945, Whale Blubber. Currently there are two biological matrix SRMs available from NIST with certified and noncertified concentrations for PCBs and chlorinated pesticides: SRM 1588, Organics in Cod Liver Oil, and SRM 1974, Organics in Mussel Tissue *(Mytilus edulis).* SRM 1588, which serves as an excellent surrogate for a tissue extract with a high lipid content, has certified concentrations for five PCB congeners and ten chlorinated pesticides and recently noncertified values for 20 additional PCB congeners and 4 additional chlorinated pesticides have been provided to make this SRM the most extensively characterized natural matrix material available with respect to PCB congeners and pesticide content [2, 11]. The mussel tissue SRM has noncertified concentrations for 13 PCB congeners and 9 chlorinated pesticides. SRM 1945 is currently being analyzed using the same approach as described for the analysis of the blubber control material (i.e., GC-ECD on columns of differing selectivity and GC-MS) to provide certified concentrations for 30-40 PCB congeners and chlorinated pesticides. The blubber SRM will complement the other tissue SRM (SRM 1974 Mussel Tissue) by providing concentrations that are generally a factor of 20-100 higher for the PCB congeners and chlorinated pesticides. Solvent extraction of the whale blubber produces an oil matrix similar to the cod liver oil SRM; however the concentrations of PCB congeners and pesticides in the whale blubber are typically 2-3 times higher than the NIST cod liver oil SRM particularly for the PCB congeners with higher degrees of chlorination.

Proposed Whale Liver SRM. At present the only liver tissue SRM available from NIST is SRM 1577a, Bovine Liver. For many elements, the concentration levels found in the whale liver QA material are similar to the levels found in SRM 1577a, as shown in Table 5. However, for As, Se, Ag, Cd and Hg, which are elements associated with toxic effects, the concentrations in the QA material are much greater (orders of magnitude in some cases) than the concentrations in the bovine liver SRM. The concentrations of these elements are comparable with the levels that have been observed in liver tissues of other marine mammals [6, 19]. Thus, for analyses of these environmentally important elements in marine mammal tissues, a whale liver reference material would be a more appropriate material than SRM 1577a.

Another potential use of whale liver reference material would be as a control material in the analyses of human liver tissues. Because of the potential health risks associated with the handling of human tissues, it is highly unlikely that a human liver tissue reference material will be available in the near future. The range of concentrations for selected trace elements in human liver samples are compared in Table 5

Table 5. Concentrations of trace elements in whale liver QA material compared to human liver and bovine liver (SRM 1577a)

	Concentration (mg/kg wet weight)				
	Pilot whale liver	Human liver			Bovine liver
Element	QA material	Median	Range		SRM 1577a[a]
Na	1260	1091	185	- 1890	729
Mg	138	146	100	- 195	180
Al	≤ 0.2-8	2	0.2	- 31.1	
Cl	1730	1370	760	- 2100	840
K	2640	2520	1380	- 3040	2990
Ca	46				36
Sc	≤ 0.08	0.00057	0.0001	- 0.0024	
V	≤ 0.02				0.0297
Cr	0.042				
Mn	2.81	1.2	0.6	- 2.03	2.97
Fe	438	230	43	- 510	58-2
Co	0.014	0.042	0.0135	- 0.0605	0.063
Ni	0.0268	0.037	≤ 0.010	- 0.1	
Cu	2.96	6.3	3.5	- 10.8	47.4
Zn	32.2	53	27.8	- 96.1	36.9
As	0.529	0.012	≤ 0.001	- 0.097	0.0141
Se	11.0	0.5	0.35	- 0.65	0.213
Rb	2	7	3.54	- 13.5	3.75
Sr	≤ 0.8				0.0414
Mo	≤ 0.4	0.57	0.21	- 1.23	1.05
Ag	0.181	0.0098	≤ 0.005	- 0.018	0.012
Cd	8.51	1.6	0.2	- 4.96	0.132
Sn	≤ 2	0.35	0.135	- 0.712	
Sb	≤ 0.08				0.001
I	≤ 1				
Cs	0.00604	0.0095	0.002	- 0.02	
Ba	≤ 5				
La	≤ 0.003	0.045	0.0007	- 0.16	
Ce	≤ 0.02	0.006	≤ 0.00144	- 0.221	
Sm	≤ 0.02				
Eu	≤ 0.0006				
Tb	≤ 0.0007				
Hf	≤ 0.0008				
Ta	≤ 0.0005				
Hg	28.2	0.086	0.026	- 0.47	0.0012
Pb	0.50	0.55	0.12	- 1.66	0.0405
Th	≤ 0.001				
U	≤ 0.004				0.00021

[a] Concentration values for SRM 1577a have been converted to a wet weight basis assuming a dry to wet weight ratio of 0.3

with the concentrations in the whale liver QA material and SRM 1577a (converted to a wet weight basis). For Cl and Fe, the concentrations found in the whale liver QA material are comparable to the upper end of the range of values that have been observed in human liver, whereas the levels present in SRM 1577a compare with the lower end of this range. For Co and Cu, levels present in the whale liver QA material are closer to the lower end of the range of concentrations found in human liver tissue, whereas the Cu level in SRM 1577a is much greater than is found in human tissue and the Co level is comparable with the median level in human liver. For the determination of Cl, Fe, Co and Cu, the use of both SRM 1577a and a whale liver reference material would provide a

check for both upper and lower concentration levels. For toxic pollutant elements such as As, Ag, Cd, and Hg, the concentrations in the pilot whale liver are higher by factors of about 2 to 50. Therefore a whale liver reference material would have concentrations of these elements that might be similar to individuals with occupational exposure to these elements.

Another advantage of a proposed whale liver SRM similar to the QA liver homogenate would be the frozen tissue matrix. The majority of trace element tissue reference materials available from NIST and other producers of reference materials are distributed as lyophilized (freeze-dried) matrices. The physical characteristics of these materials are, in many instances, significantly different from the sample matrix actually analyzed, thus the value of these materials as quality control samples is often limited. NIST has previously issued a frozen tissue matrix SRM certified for organic contaminants, SRM 1974, organics in Mussel Tissue *(Mytilus edulis),* which has noncertified concentrations for 36 trace elements. The availability of a frozen whale liver tissue SRM with certified concentrations for trace elements would be of great use to inorganic analysts.

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APPENDIX H

SCHANTZ ET AL. (1995) *ANAL. CHEM.* 67 : 901-910

Certification of Polychlorinated Biphenyl Congeners and Chlorinated Pesticides in a Whale Blubber Standard Reference Material

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A Standard Reference Material (SRM) made from whale blubber has been developed for the validation of methods used for the determination of polychlorinated biphenyl (PCB) congeners and chlorinated pesticides. This material, which is a frozen blubber tissue homogenate, was analyzed using three different analytical techniques. These techniques were based on gas chromatography with electron capture detection on two stationary phases with different selectivity for the separation of PCB congeners and gas chromatography with mass spectrometric detection. The results from these three techniques were in good agreement and were combined to provide certified concentrations for 27 PCB congeners and 15 chlorinated pesticides.

Since 1989 the National Institute of Standards and Technology (NIST) has issued two natural-matrix Standard Reference Materials (SRMs) that have certified concentrations for polychlorinated biphenyl (PCB) congeners, SRM 1588 (Organics in Cod Liver Oil)[1,2] and SRM 1939 (Polychlorinated Biphenyls (Congeners) in River Sediment A).[3-5] At NIST, the results from at least two "chemically independent" analytical techniques are typically used to determine the certified concentrations of the analytes. When only one analytical technique is used, the concentrations are generally reported as noncertified or information values. SRM 1588 has certified concentrations for 5 PCB congeners and 10 chlorinated pesticides which were determined using gas chromatography with electron capture detection (GC-ECD) and gas chromatography with mass spectrometric detection (GC/MS). Two independent sample "cleanup" procedures were used, but the same type of nonpolar capillary column was used for both GC analyses, making the techniques less independent than desired. SRM 1939 has certified concentrations for three PCB congeners determined using GC-ECD on a nonpolar phase and using dual-column GC/MS on a nonpolar phase followed by a liquid crystalline phase. These two techniques were considered suitably independent; however, only a limited number of PCB congeners were determined with the dual-column GC/MS technique.

The goal was to implement more independent analytical techniques and to expand the number of PCB congeners and chlorinated pesticides with certified concentrations in future SRMs. Therefore, different analytical techniques for the measurement of PCB congeners and chlorinated pesticides were evaluated for the analysis of five existing reference materials (SRM 1588, SRM 1941,[6,7] SRM 1974,[8,9] and two Certified Reference Materials available from the Community Bureau of Reference, CRM 349 (Chlorobiphenyls in Cod Liver Oil) and CRM 350 (Chlorobiphenyls in Mackerel Oil)).[10] These five materials were analyzed using three GC columns with different selectivity and two different GC detectors, ECD and MS. Using these techniques, concentrations for additional PCB congeners and chlorinated pesticides were reported for these reference materials, and the basis for two independent techniques for the certification of future SRMs was established. The first SRM certified using this approach was SRM 1945, Organics in Whale Blubber.

SRM 1945 was developed in response to the needs of the National Oceanic and Atmospheric Administration (NOAA) Marine Mammal Quality Assurance (QA) Program for use in the analysis of marine mammal tissues for PCB congeners and chlorinated pesticides.[11,12] SRM 1945, which is a fresh frozen sample similar to marine mammal tissue samples routinely analyzed in monitoring programs, was analyzed using techniques similar to those described above: GC-ECD on a 5% phenyl-substituted methylpolysiloxane phase and on a new dimethylpolysiloxane phase containing 50% methyl C-18 and GC/MS on a

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5% phenyl-substituted methylpolysiloxane phase. Certified concentrations were determined for 27 PCB congeners and 15 chlorinated pesticides, and noncertified concentrations were determined for two additional PCB congeners and two additional chlorinated pesticides. The approach and the results for the certification of the PCB congeners and chlorinated pesticides in SRM 1945 are described in this paper. A similar approach was also used to certify PCB congeners and chlorinated pesticides in a new marine sediment SRM (SRM 1941a). The details of the measurements for SRM 1941a are described elsewhere.[13]

EXPERIMENTAL SECTION

SRM Preparation. The whale blubber used to prepare this SRM was collected from an adult female pilot whale (*Globicephala melaena*) which was stranded on Cape Cod, MA, in September 1991. The blubber tissue was placed in Teflon bags, frozen on dry ice, shipped to NIST, and placed in a liquid nitrogen freezer (−150 °C). At NIST, any remaining skin was removed using a titanium knife and the blubber was again placed in Teflon bags and immediately returned to a liquid nitrogen freezer. The frozen whale blubber was pulverized using a cryogenic grinding procedure described previously.[14] The pulverized material was then thoroughly mixed with a Teflon paddle. Subsamples (10−15 g) of the whale blubber homogenate were aliquoted into precooled glass bottles. The bottles of SRM 1945 have been stored at −80 °C since preparation.

Determination of PCB Congeners and Chlorinated Pesticides. SRM 1945 was analyzed for selected PCB congeners and chlorinated pesticides using GC-ECD and GC/MS following the general approach described previously.[10,15] GC-ECD analyses were performed on two columns with different selectivities for the separation of PCB congeners: a 5% phenyl-substituted methylpolysiloxane stationary phase and a dimethylpolysiloxane phase containing 50% methyl C-18.

For the GC-ECD analyses, duplicate samples of 2−3 g (wet weight) of blubber from each of 10 randomly selected bottles were mixed with approximately 100 g of precleaned sodium sulfate and Soxhlet extracted for 18 h using approximately 200 mL of methylene chloride. The extracts were concentrated, and the majority of the lipid and biogenic material was removed using size exclusion chromatography (SEC) on a preparative-scale divinylbenzene/polystyrene column (10 μm particle size, 10 nm pore size). The eluent was concentrated, and the SEC fractionation was repeated. Normal-phase liquid chromatography on a semipreparative-scale aminopropylsilane column was then used to isolate two fractions containing (1) the PCB congeners and lower polarity chlorinated pesticides and (2) the more polar chlorinated pesticides. GC-ECD analyses of the two fractions were performed in duplicate on two columns: 0.25 mm × 60 m fused silica capillary column with a 5% phenyl-substituted methylpolysiloxane phase (0.25 μm film thickness; DB-5, J&W Scientific, Folsom, CA) and a 0.32 mm × 100 m fused silica capillary column

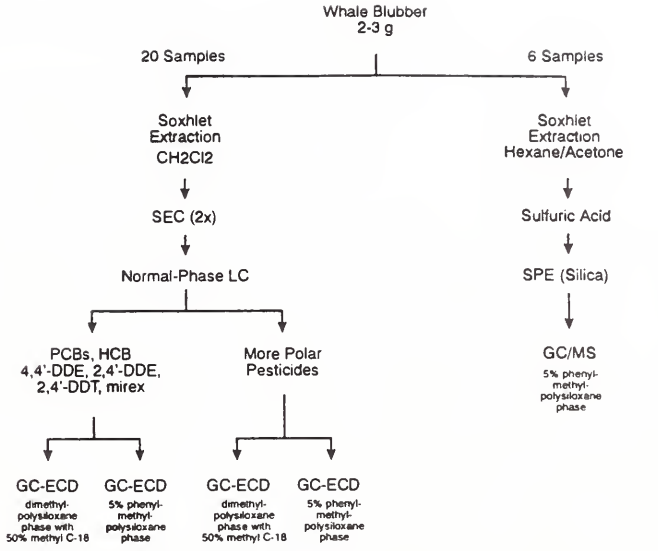

Figure 1. Analytical scheme for the determination of PCB congeners and chlorinated pesticides in SRM 1945 using different analytical techniques.

with a dimethylpolysiloxane phase containing 50% methyl C-18 (0.1 μm film thickness; CP Sil 5 C18 CB, Chrompack International, Middelburg, The Netherlands).

For the GC/MS analyses, a 2−3 g (wet weight) sample from each of six randomly selected bottles was mixed with approximately 100 g of precleaned sodium sulfate and Soxhlet extracted for 18 h using approximately 200 mL of 1:1 hexane/acetone (v/v). The extracts were concentrated and treated with concentrated sulfuric acid to remove the lipid interferences. A silica solid-phase extraction column was then used to remove the polar interferences in the extracts. The GC/MS analyses were performed in duplicate using a 0.25 mm i.d. × 60 m fused silica capillary column with a 5% phenyl-substituted methylpolysiloxane phase (0.25 μm film thickness; DB-5 MS, J&W Scientific). For the measurement of the PCB congeners, selected ions were monitored for each of the 10 degrees of chlorination (two ions per degree of chlorination, generally the major molecular ions). For the chlorinated pesticides, two selected ions were monitored, one for quantitation and the other for confirmation.

Two PCB congeners, which are not significantly present in the blubber extract (PCB 103 and PCB 198),[16,17] octachloronaphthalene, endrin, and perdeuterated 4,4′-DDT were added to the whale blubber homogenate prior to extraction for use as internal standards for quantification purposes. Calibration response factors for the analytes relative to the internal standards were determined by analyzing aliquots of the following: SRM 2261 (Concentrated Chlorinated Pesticides in Hexane), SRM 2262 (Concentrated PCB Congeners in Iso-octane), gravimetrically prepared solutions of additional analytes not contained in SRMs 2261 and 2262, and the internal standards.

Experimental Design. Experiments for each technique were designed so that all sources of possible random error in the measurements would be replicated to reduce the bias they could cause. Samples from multiple bottles, which were selected according to a stratified random sample of all bottles produced, were analyzed so that possible material heterogeneity would be

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Table 1. Summary of Analytical Results for the Determination of PCB Congeners in SRM 1945

polychlorinated biphenyls[a]		concentration (μg/kg wet wt)		
PCB no.	compound	GC-ECD[b] (CP Sil 5 C18 CB column)	GC-ECD[b] (DB-5 column)	GC/MS[c] (DB-5 column)
18	2,2′,5-trichlorobiphenyl	4.91(0.32)	3.78(0.30)	4.76(0.33)
28	2,4,4′-trichlorobiphenyl	12.16(0.91)	14.14(0.47)	[16.52][d](0.38)
31	2,4′,5-trichlorobiphenyl	2.55(0.23)	3.70(0.24)	[16.52][d](0.38)
44	2,2′,3,5′-tetrachlorobiphenyl	12.25(0.64)	13.17(0.74)	11.25(0.94)
49	2,2′,4,5′-tetrachlorobiphenyl	18.54(0.84)	22.64(0.74)	21.28(0.95)
52	2,2′,5,5′-tetrachlorobiphenyl	45.1(2.5)	42.8(2.9)	42.4(2.1)
66	2,3′,4,4′-tetrachlorobiphenyl	23.2(1.8)	[59.7][e](2.8)	23.7(1.9)
87	2,2′,3,4,5′-pentachlorobiphenyl	16.25(0.68)	17.71(0.91)	16.66(0.47)
95	2,2′,3,5′,6-pentachlorobiphenyl	34.1(1.7)	[59.7][e](2.8)	33.6(1.6)
99	2,2′,4,4′,5-pentachlorobiphenyl	41.5(2.9)	47.3(2.8)	47.3(2.3)
101	2,2′,4,5,5′-pentachlorobiphenyl	61.6(3.3)	66.3(4.3)	68.3(2.3)
90	2,2′,3,4′,5-pentachlorobiphenyl			
105	2,3,3′,4,4′-pentachlorobiphenyl	31.6(2.4)	30.0(2.4)	29.8(1.3)
110	2,3,3′,4′,6-pentachlorobiphenyl	25.9(2.1)	[36.1][f](3.3)	20.7(1.6)
118	2,3′,4,4′,5-pentachlorobiphenyl	74.9(4.7)	75.6(4.8)	71.7(3.7)
128	2,2′,3,3′,4,4′-hexachlorobiphenyl	23.2(2.2)	24.6(2.1)	22.9(1.5)
138	2,2′,3,4,4′,5′-hexachlorobiphenyl	131.7(8.0)[g]	127.7(9.0)	134.2(5.7)
163	2,3,3′,4′,5,6-hexachlorobiphenyl			
164	2,3,3′,4′,5′,6-hexachlorobiphenyl			
149	2,2′,3,4′,5′,6-hexachlorobiphenyl	101.6(6.6)	106.6(7.5)	110.6(4.7)
151	2,2′,3,5,5′,6-hexachlorobiphenyl	32.9(2.3)	26.7(2.4)	26.7(1.2)
153	2,2′,4,4′,5,5′-hexachlorobiphenyl	214(15)	201(16)	215(11)
156	2,3,3′,4,4′,5-hexachlorobiphenyl	9.76(0.69)	10.93(0.66)	10.07(0.72)
170	2,2′,3,3′,4,4′,5-heptachlorobiphenyl	39.3(2.6)[h]	40.2(2.6)	42.4(1.9)
190	2,3,3′,4,4′,5,6-heptachlorobiphenyl			
180	2,2′,3,4,4′,5,5′-heptachlorobiphenyl	105.5(6.2)	108.8(6.7)	109.5(4.9)
183	2,2′,3,4,4′,5′,6-heptachlorobiphenyl	39.5(2.9)	36.0(2.8)	34.4(1.8)
187	2,2′,3,4,5,5′,6-heptachlorobiphenyl	111.7(7.0)	103.1(6.7)	100.9(5.1)
194	2,2′,3,3′,4,4′,5,5′-octachlorobiphenyl	39.2(2.4)	41.3(2.6)	39.4(1.8)
195	2,2′,3,3′,4,4′,5,6-octachlorobiphenyl	14.0(1.3)	20.2(1.2)	19.0(1.2)
201	2,2′,3,3′,4,5′,6,6′-octachlorobiphenyl	16.9(1.1)	17.2(1.1)	16.48(0.82)
206	2,2′,3,3′,4,4′,5,5′,6-nonachlorobiphenyl	32.9(1.9)	30.7(1.9)	29.7(1.6)
209	decachlorobiphenyl	10.75(0.87)	11.16(0.94)	9.95(0.85)

[a] PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell[16] and later revised by Schulte and Malisch[17] to conform with IUPAC rules. When two or more congeners are known to coelute, the PCB congener listed first is the major component and the additional congeners may be present as minor components. The quantitative results are based on the response of the congener listed first. [b] Duplicate samples from 10 bottles extracted, and each extract analyzed in duplicate. Concentrations are the average, and numbers in parentheses are one standard deviation of a single measurement. The same extracts analyzed by GC-ECD on the C-18 column were also analyzed using the DB-5 column. [c] Samples from six bottles extracted and analyzed in duplicate. The concentration is the average, and the number in parentheses is one standard deviation of a single measurement. [d] Numbers in brackets indicate known coelution of two or more congeners. PCB 28 and 31 coelute under the GC/MS conditions used. [e] Numbers in brackets indicate known coelution of two or more congeners. PCB 66 and 95 coelute under the GC-ECD (DB-5 column) conditions used. [f] Numbers in brackets indicate known coelution of two or more congeners. PCB 77 (3,3′,4,4′-tetrachlorobiphenyl) and PCB 110 coelute under the GC-ECD (DB-5 column) conditions used. [g] PCB 164 is separated from PCB 138 and PCB 163 when the CP Sil 5 C18 column is used. [h] PCB 190 is separated from PCB 170 when the CP Sil 5 C18 column is used.

less likely to bias the technique results and possible fill-sequence trends could be detected. Four calibration solutions were prepared for each analytical technique so that the effect of bias caused by possible errors in nominal concentrations would be reduced. Samples were extracted and cleaned on more than 1 day for each method, and the extracts were analyzed on at least 4 days by each technique. This was incorporated in the design on the basis of the assumption that any differences between days are random with common variance and a mean of zero. Under this assumption, the method results will be less biased when averaged over more than 1 day. By replicating days in the design, we could also test for and quantify any possible variability between days.

Homogeneity Assessment. The homogeneity of SRM 1945 was assessed by analyzing duplicate samples of 2–3 g each from each of 10 randomly selected bottles as described in the previous section for the GC-ECD analyses. No statistically significant differences between bottles were observed for the PCB congeners or chlorinated pesticides at the 3 g sample size; therefore, because no bottle differences were detected, the material was assumed to be homogeneous. However, differences between samples within a bottle were found to be statistically significant for many compounds. Since the same sample could not be prepared twice, it was not possible to statistically separate the variability due to sample preparation from variability due to material heterogeneity. Because it is well understood that the sample preparation can introduce variability between samples and this SRM was carefully blended to be as homogeneous as possible, it was assumed that any between-sample variability was due to sample preparation and that material variability, if it existed, was negligible.

Nonvolatile Extractable Weight Determination. The percent of nonvolatile extractable material, primarily lipids, was determined for each sample after Soxhlet extraction (a total of 26 samples). The extract was evaporatively concentrated to approximately 20 mL (weight known), and an aliquot of 90 μL (weight known) was placed on an aluminum pan. The extract on the pan was air-dried, and the weight of the dried extract was noted. The ratio of weights (×100) is the percent of nonvolatile extractable material. The percent nonvolatile extractable material is 74.29 ± 0.45 (95% confidence interval for the mean).

Table 2. Summary of Analytical Results for the Determination of Chlorinated Pesticides in SRM 1945

compound	concentration (μg/kg wet wt) GC-ECD[a] (CP Sil 5 C18 CB column)	GC-ECD[a] (DB-5 column)	GC/MS[b] (DB-5 column)
2,4′-DDE	12.46(0.57)	11.69(0.70)	12.27(0.54)
4,4′-DDE	421(31)	453(25)	440(34)
2,4′-DDD	20.0(1.3)	16.2(1.4)	17.96(0.93)
4,4′-DDD	128(10)	132.4(9.1)	138.3(6.2)
2,4′-DDT	113.0(7.0)	98(10)	101.6(6.3)
4,4′-DDT	238(46)		246(12)
hexachlorobenzene	32.2(2.1)	33.1(2.2)	33.8(1.8)
γ-HCH	3.90(0.37)	2.63(0.37)	3.37(0.24)
α-HCH	18.6(1.2)	14.4(1.3)	15.3(1.0)
β-HCH	nd[c]	nd[c]	8.04(0.71)
heptachlor epoxide	10.74(0.87)	10.1(1.2)	11.2(1.0)
oxychlordane	20.6(1.5)	20.7(1.0)	18.61(0.83)
cis-chlordane	47.2(3.2)	46.0(3.4)	47.2(2.5)
cis-nonachlor	54.2(3.2)	47.8(3.1)	44.3(2.7)
trans-nonachlor	229(16)	233(16)	232(10)
dieldrin	39.4(3.0)	35.5(3.1)	nd[d]
mirex	30.1(2.3)	29.6(2.3)	26.8(2.2)

[a] Duplicate samples from 10 bottles extracted, and each extract analyzed in duplicate. Concentrations are the average, and numbers in parentheses are one standard deviation of a single measurement. The same extracts analyzed by GC-ECD on the C-18 column were also analyzed using the DB-5 column. [b] Samples from six bottles extracted and analyzed in duplicate The concentration is the average, and the number in parentheses is one standard deviation of a single measurement. [c] nd, not determined since β-HCH is not recovered from the normal-phase LC cleanup step. [d] nd, not determined since dieldrin is destroyed in the sulfuric acid step.

RESULTS AND DISCUSSION

The certification of natural-matrix environmental SRMs at NIST is based on the agreement of results from two or more different analytical techniques. Since 1980 NIST has certified natural-matrix SRMs for polycyclic aromatic hydrocarbons (PAHs) using GC/MS or GC with flame ionization detection as the first technique and liquid chromatography with fluorescence detection as the second independent technique. For PCB congener and chlorinated pesticide analysis, however, GC is the only suitable separation technique for the levels routinely found in environmental samples. The analysis of PCB/pesticide mixtures on a single GC column may lead to the overestimation of some compounds due to coelution.[18] When SRM 1588 (Organics in Cod Liver Oil) was certified in 1989,[1] GC-ECD analysis and GC/MS analysis were performed using similar columns, both containing a 5% phenyl-substituted methylpolysiloxane phase. Due to the similarity of the columns used and the possibility of coelution problems, only five PCB congeners were certified in SRM 1588. In 1990, selected PCB congeners were certified in SRM 1939 (Polychlorinated Biphenyl Congeners in River Sediment) using GC-ECD on the 5% phenyl-substituted methylpolysiloxane phase and a dual-column GC/MS method on a 5% phenyl-substituted methylpolysiloxane phase with heart cutting of specific portions of the chromatogram onto a liquid crystalline (SB-Smectic) phase.[3,4] Only three PCB congeners, two trichlorobiphenyl congeners and one tetrachlorobiphenyl congener, were certified in this material due to the thermal instability of the liquid crystalline phase.[19]

To find methods for expanding the list of certified PCB congeners, the use of GC-ECD analysis was investigated by use of three columns containing phases of different selectivity: a 5% phenyl-substituted methylpolysiloxane (DB-5), a 14% cyanopropylphenyl-substituted methylpolysiloxane (DB-1701), and a 5% phenyl-substituted methylpolysiloxane with 10% methyl C-18 and GC/MS analysis on the 5% phenyl-substituted methylpolysiloxane phase (DB-5 MS).[10] These methods were used to quantify additional PCB congeners in SRM 1588, SRM 1941, SRM 1974, CRM 349, and CRM 350. This study demonstrated that the 5% phenyl-substituted methylpolysiloxane phase containing 10% methyl C-18 provided a useful complement to the PCB congener separations obtained with the 5% phenyl-substituted methylpolysiloxane phase with resolution of PCB 15 from PCB 18, PCB 95 from PCB 66, PCB 90 from PCB 101, and PCB 190 from PCB 170, pairs that normally coelute on the 5% phenyl-substituted methylpolysiloxane phase. De Boer et al.[20] compared the retention characteristics of 51 PCB congeners on the 5% phenyl-substituted methylpolysiloxane phase containing 10% methyl C-18 and six other phases. The 14% cyanopropylphenyl-substituted methylpolysiloxane phase separated PCB 90 from PCB 101 and enhanced the separation between PCB 153 and PCB 105 as compared to the 5% phenyl-substituted methylpolysiloxane phase. Additionally, mass spectrometric detection allowed the quantification of several coeluting congener pairs with different levels of chlorination, e.g., PCB 15/18 and PCB 95/66.

Based on that study, the analytical scheme shown in Figure 1 was selected for certification of PCB congeners and chlorinated pesticides in SRM 1945. Two different solvent systems (methylene chloride and *n*-hexane/acetone) were used for the Soxhlet extraction, and two different procedures were used for the cleanup and isolation of the PCB congeners and chlorinated pesticides. One set of samples was analyzed by GC-ECD on the 5% phenyl-substituted methylpolysiloxane phase, and then the same sample extracts were analyzed on a commercially available dimethylpolysiloxane phase with 50% methyl C-18, which has selectivity similar to the experimental 5% phenyl-substituted methylpolysiloxane phase with 10% methyl C-18 phase used in the previous study.[10] The dimethylpolysiloxane phase containing 50% methyl C-18 has been characterized in detail by Ballschmiter et al.[21] A second set of samples was analyzed by GC/MS on the 5% phenyl-substituted methylpolysiloxane MS phase. The 14% cyanopropylphenyl-substituted methylpolysiloxane phase was not incorporated in the scheme since it does not provide any unique separations compared to the dimethylpolysiloxane phase containing 50% methyl C-18.

The dimethylpolysiloxane phase containing 50% methyl C-18 and the 5% phenyl-substituted methylpolysiloxane phase have different selectivity for the PCB separations. In addition to the separation of critical congener pairs mentioned above, other minor congeners demonstrate different selectivity. Based on the differences in sample preparation, separation selectivity, and detection selectivity, the assumption was made that each method has different sources of bias. Therefore, if the results from the different methods are in good agreement, it suggests that none of the methods was significantly biased.

Even though these analytical procedures have some similarities (i.e., nonpolar solvent extraction and GC separations) and are

(18) Rahman, M. S.; Bowadt, S.; Larsen, B. *J. High Resolut. Chromatogr.* **1993**, *16*, 731–735.

(19) Ballschmiter, K.; Bacher, R.; Mennel, A.; Fischer, R.; Riehle, U.; Swerev, M. *J. High Resolut. Chromatogr.* **1992**, *15*, 260–270.

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(21) Ballschmiter, K.; Mennel, A.; Buyten, J. *Fresenius J. Anal. Chem.* **1993**, *346*, 396–402.

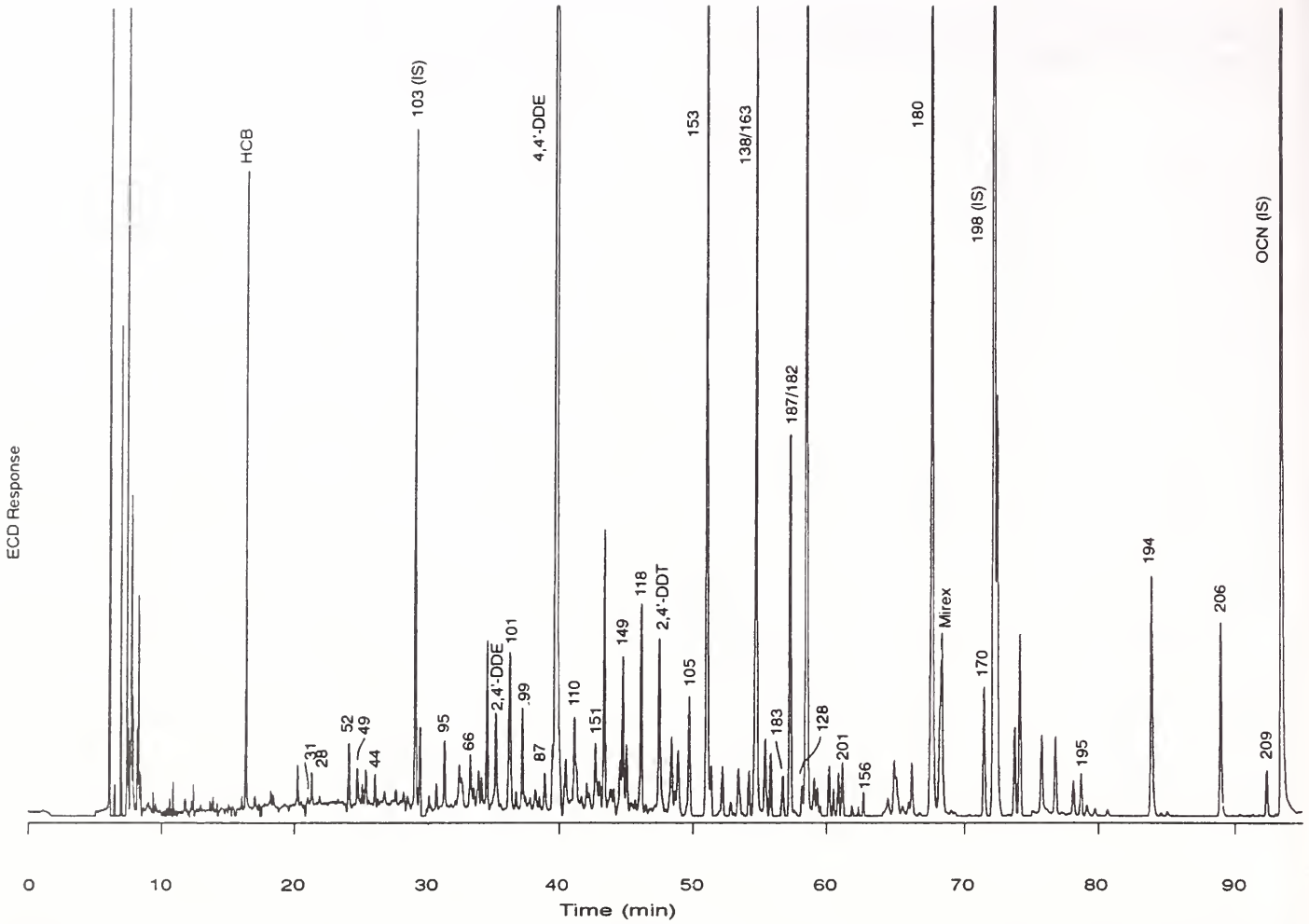

Figure 2. GC-ECD analysis of the PCB and lower polarity pesticide fraction isolated from SRM 1945 on a 100 m × 0.32 mm CP Sil 5 C18 CB column.

therefore not totally independent, this approach (based on differences in GC separation and detection selectivity) is appropriate for SRM certification given the current state of the art for the measurement of PCB congeners and pesticides. Other groups, including the Community Bureau of Reference (BCR), National Research Council of Canada (NRCC), and International Atomic Energy Agency (IAEA), have issued certified reference materials for PCBs and pesticides based on measurements from round robin studies involving a number of laboratories.[22–25] However, the certified values from round robin studies typically are subject to the same potential biases due to method similarities among the participating laboratories.

The results obtained for 29 PCB congeners and 17 chlorinated pesticides from the analysis of SRM 1945 using the three analytical techniques are summarized in Tables 1 and 2, respectively. Representative chromatograms for the PCB and lower polarity pesticide fraction from the GC-ECD analyses on the dimethylpolysiloxane phase containing 50% methyl C-18 and on the 5% phenyl-substituted methylpolysiloxane phase are shown in Figures 2 and 3, respectively. The GC/MS single ion monitoring traces for the tetrachloro-, pentachloro-, hexachloro-, and heptachlorobiphenyl congeners are shown in Figure 4. A representative chromatogram for the more polar pesticide fraction from the GC-ECD analyses on the 5% phenyl-substituted methylpolysiloxane phase is shown in Figure 5.

Differences between Methods. The dimethylpolysiloxane phase containing 50% methyl C-18 did separate the following pairs of PCB congeners that coelute on the 5% phenyl-substituted methylpolysiloxane phase: PCB 15/18, PCB 66/95, PCB 90/101, PCB 77/110, and PCB 170/190. It did not separate the PCB 138/163 pair but did separate PCB 164 from this pair. In this sample, however, the concentrations of PCB 15, PCB 90, PCB 164, and PCB 190 are not significant based on the GC-ECD analysis on the dimethylpolysiloxane phase containing 50% methyl C-18 (See Table 1 and discussion below). For the congeners of interest in this study, there were no additional significant coelution problems on the dimethylpolysiloxane phase containing 50% methyl C-18. GC/MS analysis discriminates between coeluting peaks with differences of an odd number of chlorines (i.e., 1,3, 5, and so on). Therefore, of the congener pairs listed above, MS detection discriminates between PCB 15/18, PCB 66/95, and PCB 77/110. Again, the concentration of PCB 15 appeared to be negligible based on the GC/MS analysis.

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(23) Marine Sediment Reference Materials for Polycyclic Aromatic Hydrocarbons HS-3, HS-4, HS-5, HS-6; Marine Sediment Reference Materials for Polychlorinated Biphenyls CS-1, HS-1, HS-2. National Research Council of Canada, Ottawa, ON, Canada, **1988**.

(24) Catalog for Analytical Quality Control Services, International Atomic Energy Agency, Vienna, Austria, 1994; pp 1–3.

(25) Griepink, B.; Wells, D. E.; Frias Ferreira, M. Report EUR 11520 EN Cod Liver Oil CRM No 349 and Mackerel Oil CRM No 350, Community Bureau of Reference, Brussels, Belgium, 1988.

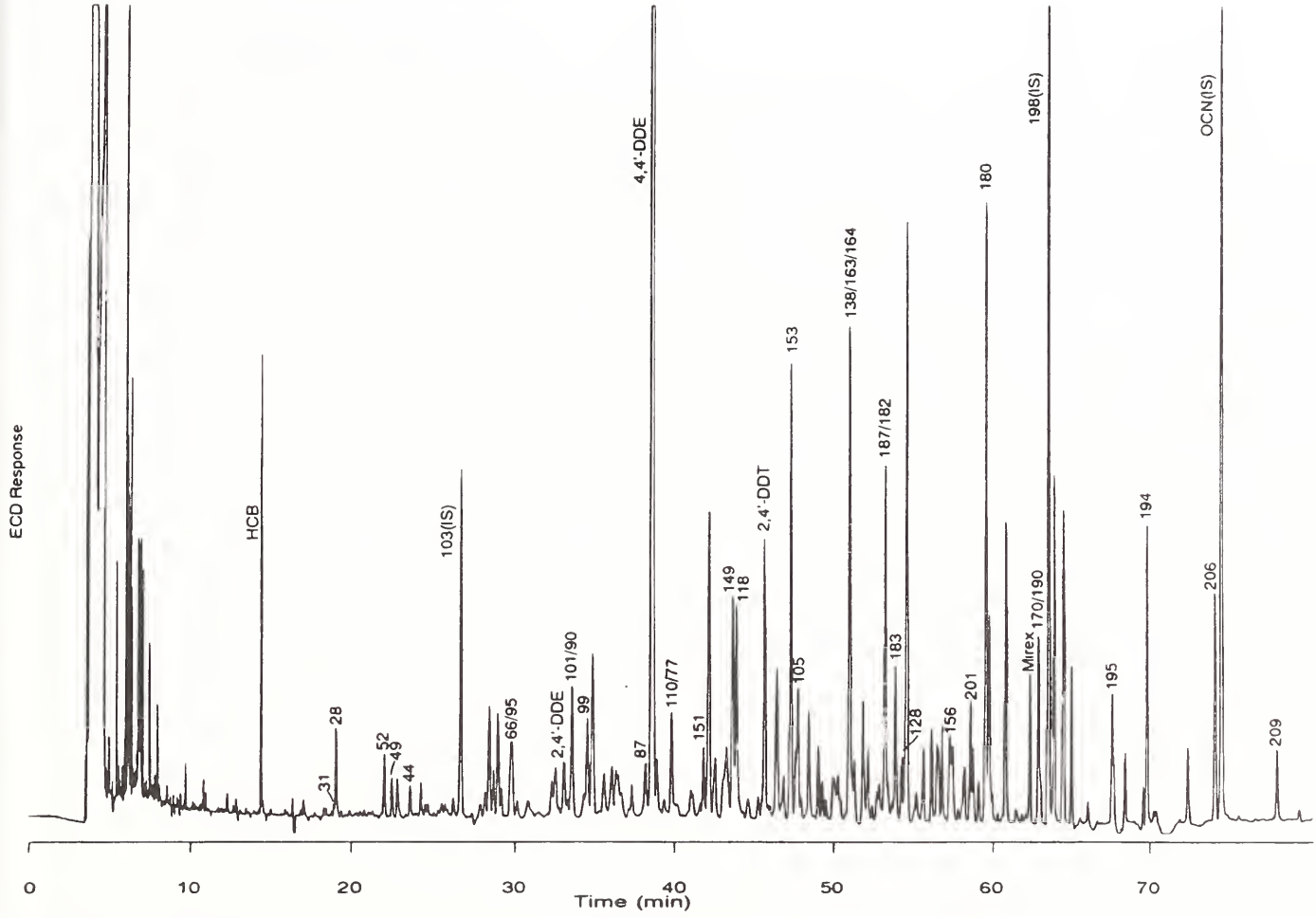

Figure 3. GC-ECD analysis of the PCB and lower polarity pesticide fraction isolated from SRM 1945 on a 60 m × 0.25 mm DB-5 column.

As mentioned above, PCB 66 (2,3′,4,4′-tetrachlorobiphenyl) and PCB 95 (2,2′,3,5′,6-pentachlorobiphenyl) coelute on the 5% phenyl-substituted methylpolysiloxane phase (see Table 1). However, the dimethylpolysiloxane phase containing 50% methyl C-18 separates PCB 95 from PCB 66, and GC/MS selectively measures PCB 66 (tetrachloro substitution) in the presence of PCB 95 (pentachloro substitution). In SRM 1945, the concentration of PCB 66 is approximately 70% that of PCB 95.

PCB 101 (2,2′,4,5,5′-pentachlorobiphenyl) and PCB 90 (2,2′,3,4′,5-pentachlorobiphenyl) are separated on the dimethylpolysiloxane phase containing 50% methyl C-18 but coelute on the 5% phenyl-substituted methylpolysiloxane phase and are not distinguishable by MS detection. The concentration of PCB 90 is less than 10% that of PCB 101 in this sample, as indicated by the concentration of 61.6 μg/kg wet wt for PCB 101 determined using the dimethylpolysiloxane phase containing 50% methyl C-18 (Table 1) compared to the concentrations of 66.3 and 68.3 μg/kg wet wt (Table 3) for the combined PCB 101/90 as determined on the 5% phenyl-substituted methylpolysiloxane phase (both ECD and MS).

PCB 110 (2,3,3′,6-pentachlorobiphenyl) coelutes with the "planar" congener PCB 77 (3,3′,4,4′-tetrachlorobiphenyl) when the 5% phenyl-substituted methylpolysiloxane phase is used. The planar non-ortho-substituted PCB congeners are of particular interest due to their enhanced toxicity.[26] The planar PCB congeners were not quantified in this study, but work is ongoing to isolate and quantify these compounds.[27] PCB 110 and PCB 77 are separated by use of the dimethylpolysiloxane phase containing 50% methyl C-18 and are distinguishable by MS detection. The concentration of PCB 77 could be as high as 35% of the concentration of PCB 110 in this sample based on the results in Table 1; however, this is unlikely. Other workers[28] have found a lower percentage of PCB 77 relative to PCB 110 in narwhal and beluga whale blubber. The planar non-ortho-substituted PCB congeners should only be quantified after isolation from the complex mixture of PCB congeners.

PCB 138 (2,2′,3,4,4′-hexachlorobiphenyl), PCB 163 (2,3,3′,5,6-hexachlorobiphenyl), and PCB 164 (2,3,3′,6-hexachlorobiphenyl) coelute when the 5% phenyl-substituted methylpolysiloxane phase is used. When the dimethylpolysiloxane phase containing 50% methyl C-18 is used, however, PCB 164 is separated from this trio. Based on the results in Table 1, the concentration of PCB 164 is negligible. Recently, Hillery et al.[29] quantified PCB 164 in SRM 1945 (2.4% of the combined concentration of PCB 138 and PCB 163) using a novel cyanobiphenyl stationary phase. This stationary phase separates PCB 138 from PCB 163 and PCB 164.

PCB 170 (2,2′,3,3′,4,4′,5-heptachlorobiphenyl) and PCB 190 (2,3,3′,4,4′,5,6-heptachlorobiphenyl) are not separated on the 5% phenyl-substituted methylpolysiloxane phase and are not distinguishable by MS detection. This pair is separated on the

(26) Ballschmiter, K; Bacher, R; Mennel, A; Fischer, R; Riehle, U; Swerev, M. *J. High Resolut. Chromatogr.* **1992**, *15*, 260–270.

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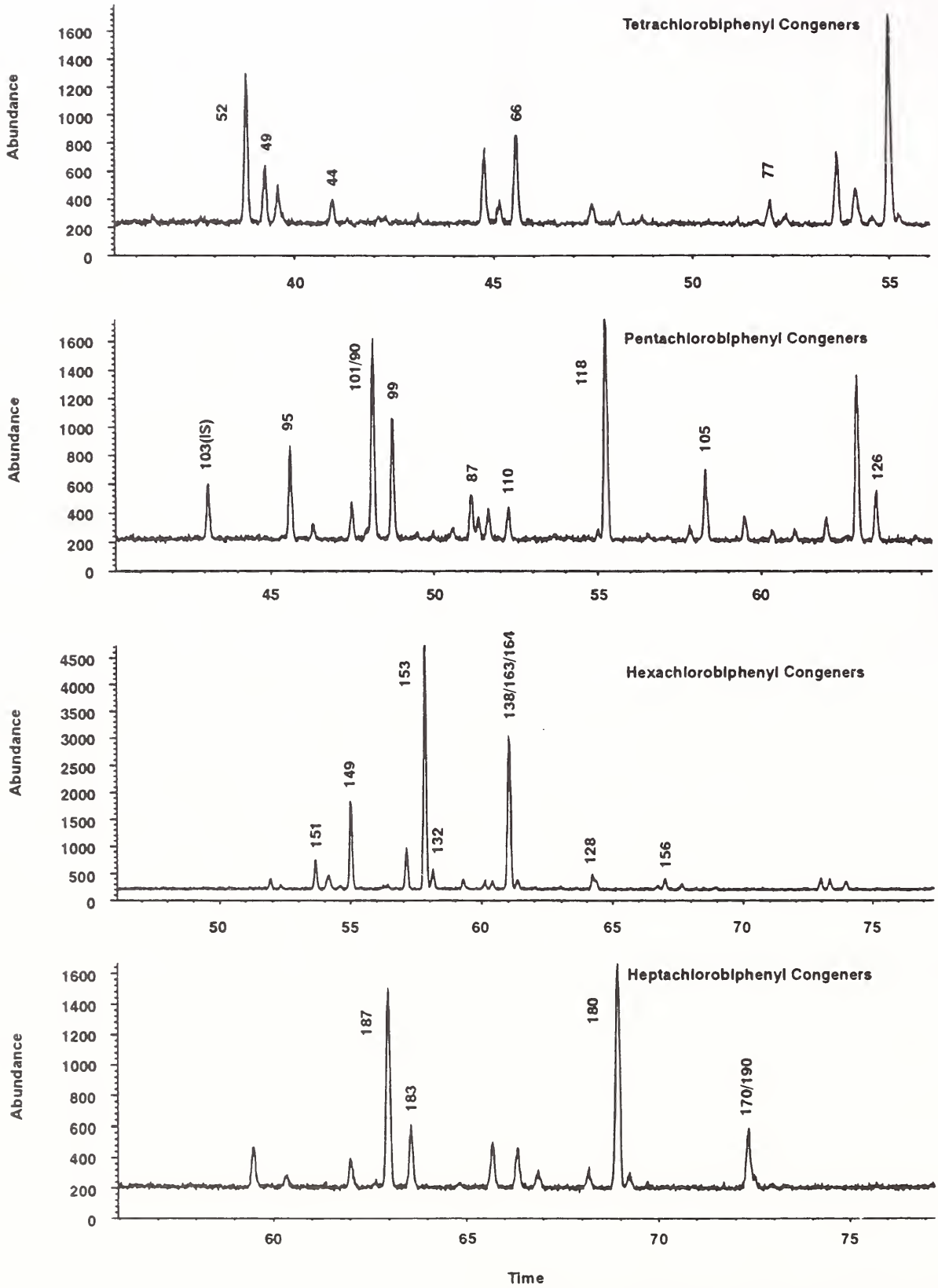

Figure 4. GC/MS analysis of the PCB and lower polarity pesticide fraction isolated from SRM 1945 on a 60 m × 0.25 mm DB-5 MS column: (A) tetrachlorobiphenyl congeners monitored at ion 289.90, (B) pentachlorobiphenyl congeners monitored at ion 323.90, (C) hexachlorobiphenyl congeners monitored at ion 359.85, and (D) heptachlorobiphenyl congeners monitored at ion 393.80.

dimethylpolysiloxane phase containing 50% methyl C-18; however, the concentration of PCB 190 appears to be less than 3% of the concentration of PCB 170 in SRM 1945.

No effects on the PCB congeners were observed from the sulfuric acid cleanup used for the samples analyzed by GC/MS. Kannan et al.[30] also did not observe any effects during acid or alkali treatments on the PCB congeners extracted from harbor seal (*Phoca vitulina*) blubber. For the chlorinated pesticides, there were only two significant differences among the analytical techniques. Dieldrin was destroyed during the sulfuric acid cleanup, and β-HCH was not recovered from the normal-phase liquid chromatographic cleanup step.

(30) Kannan, N.; Petrick, G.; Schultz-Bull, D. E.; Duinker, J. C. *J. Chromatogr.* **1993**, *642*, 425–434.

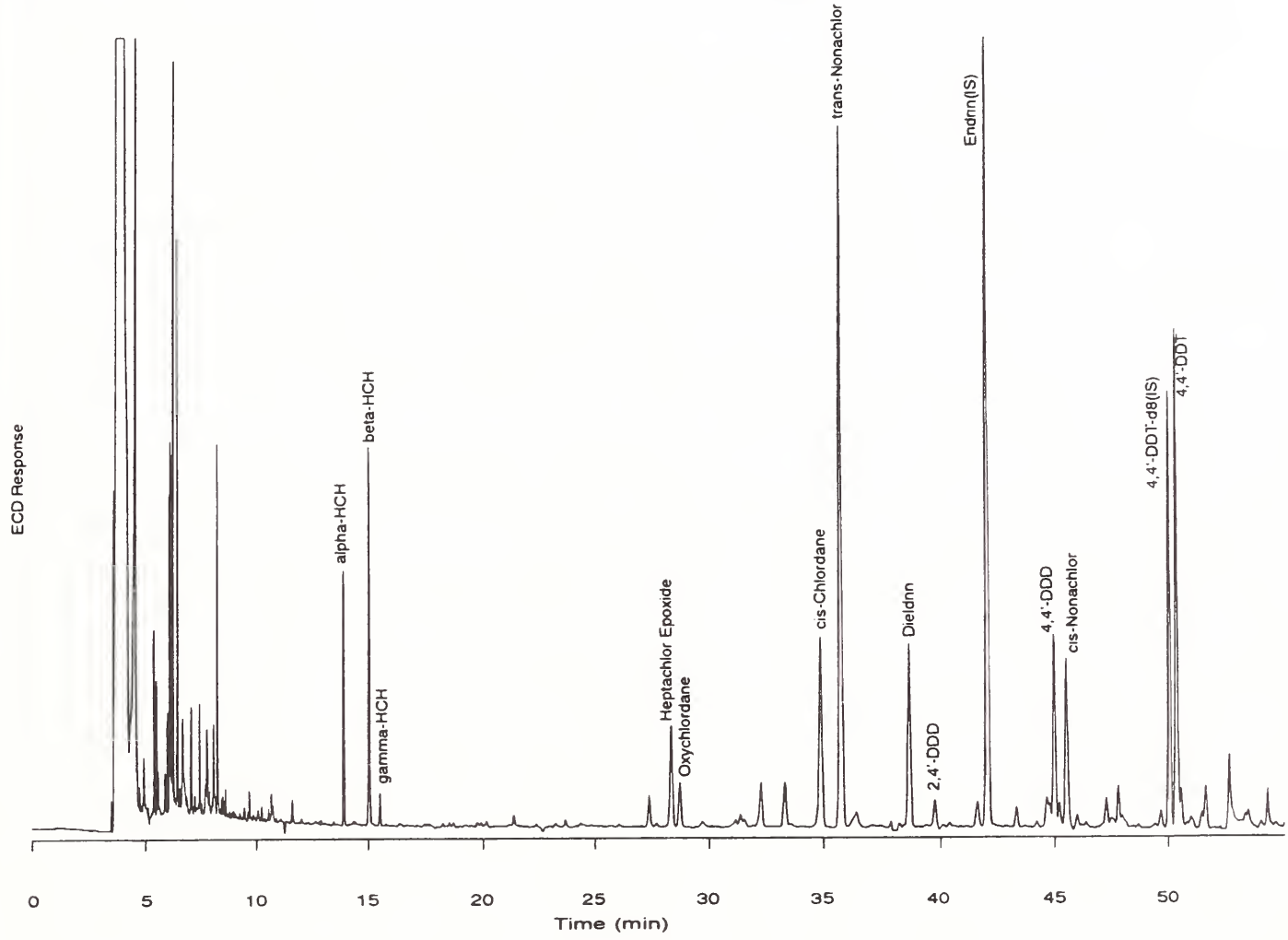

Figure 5. GC-ECD analysis of the more polar pesticide fraction isolated from SRM 1945 on a 60 m × 0.25 mm DB-5 column.

Statistical Assessment of Within-Technique Results. To compare results among the three techniques, the mean concentration and its precision for each technique were estimated. The experiments were designed to maximize the amount of independent information about the mean concentration for each analyte.

First, the uncertainty resulting from the calibration was determined. Although multiple measurements were made on each calibration solution, an average calibration was used. For each technique, four solutions bracketing the concentration of the analyte in the blubber were made, and a straight-line calibration was used. Random error in measuring the instrumental responses for the calibration solutions caused uncertainty in the final concentration; however, this uncertainty does not show up in the variability of the measured concentrations. Therefore, it was incorporated explicitly by propagation of errors.

Next, an analysis of variance was performed on the individual PCB or chlorinated pesticide concentrations calculated using the calibration curve for each technique. For the GC-ECD techniques, 10 samples were extracted in duplicate and cleaned up over 4 days. For the GC/MS technique, six samples were extracted and cleaned up over 2 days. Duplicate injections, over multiple days, were made on each sample. The analysis of variance was checked for effects due to extraction day (not significant), injection order (not significant), carousel tray (not significant), sample (significant for some cases), and interactions among these effects. Since two separate columns had been used to measure the same extracts for the GC-ECD data, data for both columns were included in the same analysis of variance. Based on the model, the appropriate variance of the mean was computed for each analyte and each technique.

A significant effect in the analysis of variance is illustrated in Figure 6. Shown are the concentrations for PCB 99 measured by GC-ECD (5% phenyl-substituted methylpolysiloxane phase) demonstrating a sample effect. The sample effect suggests that the variability introduced into the measurement process by the sample preparation step is greater than that introduced by the instrumental measurements. It also means that the two injection results from a single sample are not statistically independent, so that the variance of the mean is not the "raw" variance of the observations divided by the number of observations. This would cause underestimation of the variance. For example, if there are significant sample differences, the appropriate variance is the variance of the sample averages divided by the number of samples.

Finally, for each technique the variability in the calibration was combined with the variability in the mean concentration from the analysis of variance to determine the total variance of the mean and its degrees of freedom.

Certified Concentrations. The certified concentrations are shown in Tables 3 and 4 for the PCB congeners and chlorinated pesticides, respectively. These concentrations are weighted means of the results from two or more analytical techniques, as

Table 3. Certified Concentrations of PCB Congeners in SRM 1945

PCB no.	polychlorinated biphenyls[a] compound	concentration (μg/kg wet wt)[b]
18	2,2′,5-trichlorobiphenyl	4.48 ± 0.88
44	2,2′,3,5′-tetrachlorobiphenyl	12.2 ± 1.4
49	2,2′,4,5′-tetrachlorobiphenyl	20.8 ± 2.8
52	2,2′,5,5′-tetrachlorobiphenyl	43.6 ± 2.5
66	2,3′,4,4′-tetrachlorobiphenyl	23.6 ± 1.6
87	2,2′,3,4,5′-pentachlorobiphenyl	16.7 ± 1.4
95	2,2′,3,5′,6-pentachlorobiphenyl	33.8 ± 1.7
99	2,2′,4,4′,5-pentachlorobiphenyl	45.4 ± 5.4
101	2,2′,4,5,5′-pentachlorobiphenyl	65.2 ± 5.6
90	2,2′,3,4′,5-pentachlorobiphenyl	
105	2,3,3′,4,4′-pentachlorobiphenyl	30.1 ± 2.3
110	2,3,3′,4′,6-pentachlorobiphenyl	23.3 ± 4.0
118	2,3′,4,4′,5-pentachlorobiphenyl	74.6 ± 5.1
128	2,2′,3,3′,4,4′-hexachlorobiphenyl	23.7 ± 1.7
138	2,2′,3,4,4′,5′-hexachlorobiphenyl	131.5 ± 7.4
163	2,3,3′,4′,5,6-hexachlorobiphenyl	
164	2,3,3′,4′,5′,6-hexachlorobiphenyl	
149	2,2′,3,4′,5′,6-hexachlorobiphenyl	106.6 ± 8.4
151	2,2′,3,4,4′,5-hexachlorobiphenyl	28.7 ± 5.2
153	2,2′,4,4′,5,5′-hexachlorobiphenyl	213 ± 19
156	2,3,3′,4,4′,5-hexachlorobiphenyl	10.3 ± 1.1
170	2,2′,3,3′,4,4′,5-heptachlorobiphenyl	40.6 ± 2.6
190	2,3,3′,4,4′,5,6-heptachlorobiphenyl	
180	2,2′,3,4,4′,5,5′-heptachlorobiphenyl	106.7 ± 5.3
183	2,2′,3,4,4′,5′,6-heptachlorobiphenyl	36.6 ± 4.1
187	2,3′,3,4,5,5′,6-heptachlorobiphenyl	105.1 ± 9.1
194	2,2′,3,3′,4,4′,5,5′-octachlorobiphenyl	39.6 ± 2.5
195	2,2′,3,3′,4,4′,5,6-octachlorobiphenyl	17.7 ± 4.3
201	2,2′,3,3′,4,5′,6,6′-octachlorobiphenyl	16.96 ± 0.89
206	2,2′,3,3′,4,4′,5,5′,6-nonachlorobiphenyl	31.1 ± 2.7
209	decachlorobiphenyl	10.6 ± 1.1

[a] PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell[16] and later revised by Schulte and Malisch[17] to conform with IUPAC rules. When two or more congeners are known to coelute, the PCB congener listed first is the major component and the additional congeners may be present as minor components. The quantitative results are based on the response of congener listed first. [b] The certified values are weighted means of results from three analytical techniques as described by Schiller and Eberhardt.[31] The uncertainty is based on a 95% confidence interval for the true concentration and includes an allowance for differences among the analytical methods used.

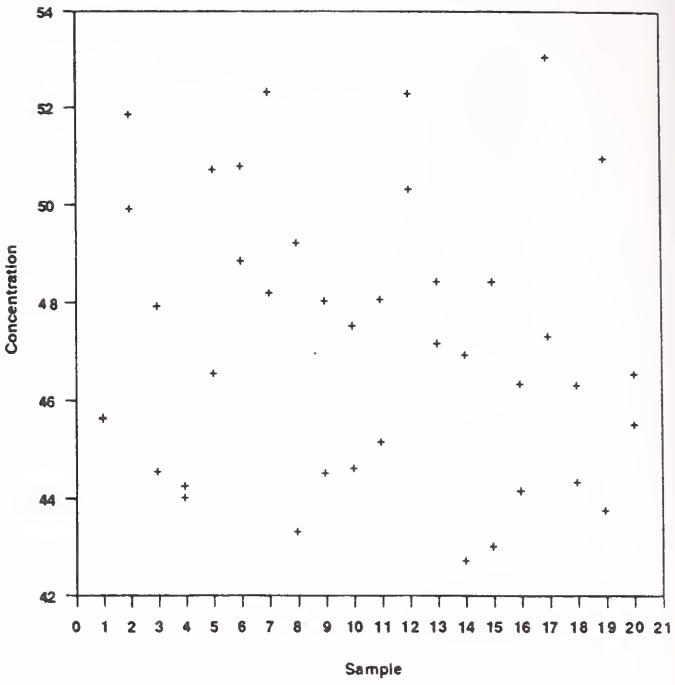

Figure 6. Concentration of PCB 99 determined using GC-ECD (DB-5 column) for 20 different samples analyzed in duplicate.

Table 4. Certified Concentrations of Chlorinated Pesticides in SRM 1945

chlorinated pesticides	concentration (μg/kg wet wt)[a]
hexachlorobenzene	32.9 ± 1.7
α-HCH	16.2 ± 3.4
γ-HCH	3.30 ± 0.81
heptachlor epoxide	10.8 ± 1.3
oxychlordane	19.8 ± 1.9
mirex	28.9 ± 2.8
cis-chlordane (α-chlordane)	46.9 ± 2.8
cis-nonachlor	48.7 ± 7.6
trans-nonachlor	231 ± 11
2,4′-DDE	12.28 ± 0.87
4,4′-DDE	445 ± 37
2,4′-DDD	18.1 ± 2.8
4,4′-DDD	133 ± 10
2,4′-DDT	106 ± 14
4,4′-DDT	245 ± 15

[a] The certified values are weighted means of results from three analytical techniques as described by Schiller and Eberhardt.[31] The uncertainty is based on a 95% confidence interval for the true concentration and includes an allowance for differences among the analytical methods used.

described by Schiller and Eberhardt.[31] The weights depend on the relative precision and agreement among the methods. If the methods are in good agreement, the weights are inversely proportional to the variances of the technique means. However, if the techniques do not agree well, the weights are approximately equal. The algorithm used by Schiller and Eberhardt[31] provides a "gray scale" for the weights between these two extremes, determined by the observed data.

Figure 7 illustrates two examples (PCB 18 and PCB 138) of the weighting scheme used to determine the certified values. For PCB 18, the weighting factors are nearly equal because of disagreement of the mean results from the three techniques. For PCB 138, where results from all three techniques agree well, the results from GC-ECD, dimethylpolysiloxane phase containing 50% methyl C-18, are weighted more heavily because of their higher precision. Although this has little effect on the certified value, since the technique means agree well, the unequal weighting reduces the total uncertainty. After the weighting factors were determined, the variances of the technique means and their degrees of freedom were combined to determine the variance and degrees of freedom of the weighted mean. Because this variance does not include information on between-technique differences, an allowance was also included for the observed between-technique difference. A 95% confidence interval for the weighted mean, with an allowance for differences between the techniques, is the uncertainty associated with the certified value.

There are 27 PCB congeners and 15 chlorinated pesticides certified in SRM 1945. This is a substantially larger number of PCB congeners and chlorinated pesticides than have been certified in any previous natural-matrix SRMs. The relative uncertainties for the certified concentrations range from 5 to 25% with the majority in the 5–10% range. The same analytical approach was also recently used to certify 21 PCB congeners and 6 pesticides

(31) Schiller, S. B.; Eberhardt, K. R. *Spectrochim. Acta* **1991**, *46B*, 1607–1613.

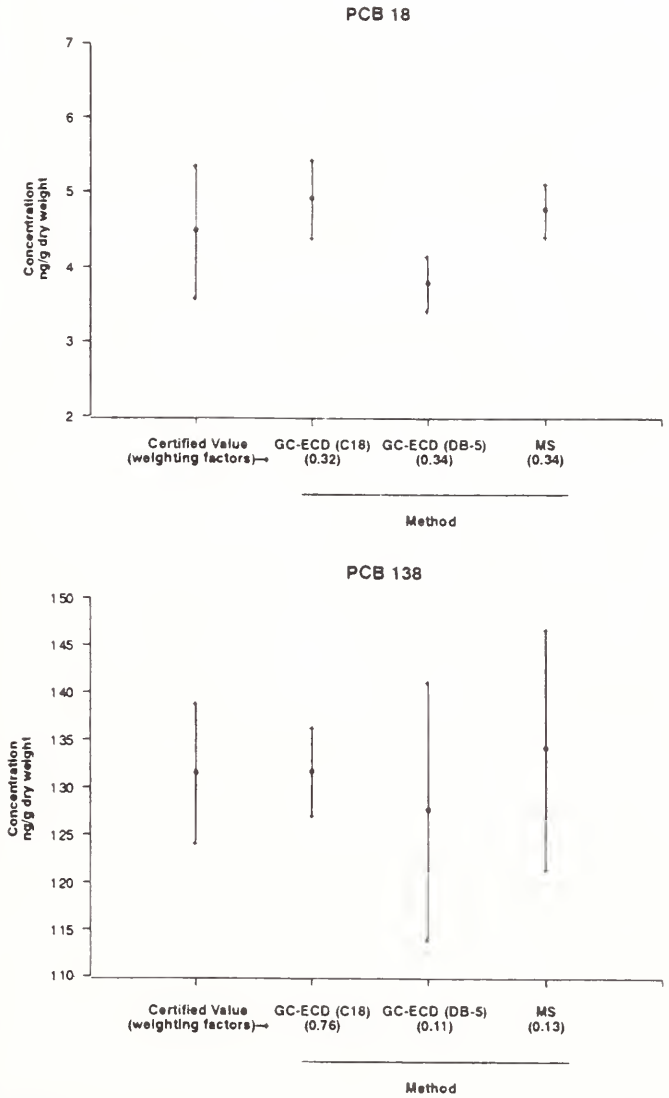

Figure 7. Graphical representation of results for PCB 18 and PCB 138 from three different analytical techniques and the weighting factors used in combining the results to obtain certified values.

in a sediment reference material (SRM 1941a, Organics in Marine Sediment).[13]

Noncertified Concentrations. Noncertified concentrations were determined for two additional PCB congeners and two additional chlorinated pesticides as shown in Table 5. The two PCB congeners, PCB 28 and PCB 31, were not separated during the GC/MS analysis. Dieldrin was destroyed during the sulfuric acid cleanup step, and β-HCH was not recovered from the normal-phase liquid chromatographic cleanup step, resulting in only one technique for each of these compounds.

CONCLUSIONS

The combination of results from three analytical techniques allowed the determination of certified concentrations for 27 PCB congeners and 15 chlorinated pesticides in SRM 1945, which represents the most highly characterized natural-matrix SRM with respect to PCB congeners and chlorinated pesticides. The relative uncertainties associated with the majority of the analyte concentrations were between 5 and 10%. The extensive list of analytes and the low uncertainties for the certified values will make SRM 1945 a valuable reference material for use in the validation of analytical methods for the determination of PCB congeners and chlorinated pesticides in marine mammal blubber and other high lipid-containing samples.

Table 5. Noncertified Concentrations of PCB Congeners and Chlorinated Pesticides in SRM 1945

	concentration (μg/kg wet wt)[b]
polychlorinated biphenyls[a]	
PCB 28	14.1 ± 1.4[c]
PCB 31	3.12 ± 0.69[d]
chlorinated pesticides	
dieldrin	37.5 ± 3.9[d]
β-HCH	8.0 ± 1.4[c]

[a] PCB congeners are numbers according to the scheme proposed by Ballschmiter and Zell[16] and later revised by Schulte and Malisch[17] to conform with IUPAC rules. [b] Concentrations are the weighted mean from the results of the indicated techniques. Uncertainties for the measurements are a 95% confidence interval for the mean for PCB 28 and β-HCH and a 95% confidence interval including an allowance for differences among the analytical methods used for PCB 31 and dieldrin. [c] Concentration determined by GC-ECD on DB-5 column. [d] Concentration determined by GC-ECD on C-18 column and DB-5 column. [e] Concentration determined by GC/MS.

ACKNOWLEDGMENT

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APPENDIX I

CERTIFICATE OF ANALYSIS FOR STANDARD REFERENCE MATERIAL (SRM) 1945 ORGANICS IN WHALE BLUBBER

National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 1945

Organics in Whale Blubber

Standard Reference Material (SRM) 1945 is a frozen whale blubber homogenate intended for use in evaluating analytical methods for the determination of selected polychlorinated biphenyl (PCB) congeners and chlorinated pesticides. A unit of SRM 1945 consists of two screw-capped glass bottles, each containing approximately 15 g of frozen whale blubber homogenate.

Certified values are provided in Tables 1 and 2 for the concentrations of 27 PCB congeners and 15 chlorinated pesticides, respectively, which are naturally present in the whale blubber. The certified values for the PCB congeners and chlorinated pesticides are based on the results obtained from the analyses of this material using different extraction/sample preparation procedures and analytical techniques based on gas chromatography with electron capture detection (GC-ECD) on two stationary phases of different selectivity and on gas chromatography-mass spectrometry (GC-MS).

Noncertified values are provided in Appendix A for two additional PCB congeners and two additional chlorinated pesticides. Summaries of the analytical results obtained by using the different analytical techniques during the certification are provided in Appendices B and C for the PCB congeners and chlorinated pesticides, respectively.

Expiration of Certification: This certification is valid within the specified uncertainty limits for three years from the date of shipment from NIST. In the event that the certification should become invalid before then, purchasers will be notified by NIST. Please return the attached registration form to facilitate notification.

The collection, preparation, and certification of SRM 1945 were supported in part by the Marine Mammal Health and Stranding Response Program of the Office of Protected Resources, National Marine Fisheries Service, National Oceanic and Atmospheric Administration (NOAA). The whale blubber material used for SRM 1945 was collected with the assistance of G. Early of the New England Aquarium (Boston, MA).

Collection and preparation of the SRM were performed by B.J. Koster and S.A. Wise of the NIST Organic Analytical Research Division and by M.P. Cronise, C.N. Fales, J.T. Fort, G.V. Proulx, and T.P. Shuggars of the NIST Standard Reference Materials Program. Analytical measurements were performed in the NIST Organic Analytical Research Division by M.M. Schantz.

Statistical design of the experimental work and evaluation of the data were provided by L.M. Oakley and S.B. Schiller of the NIST Statistical Engineering Division.

The coordination of the technical measurements leading to certification was under the direction of M.M. Schantz and S.A. Wise.

The technical and support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by T.E. Gills.

Gaithersburg, MD 20899
June 16, 1994

Thomas E. Gills, Chief
Standard Reference Materials Program

(over)

NOTICE AND WARNING TO USERS

Storage: SRM 1945 is provided as a frozen tissue homogenate in glass bottles. The tissue homogenate should not be allowed to thaw prior to subsampling for analysis. This material has been stored at NIST at -80 °C (or lower) since it was prepared. SRM 1945 should be stored at temperatures less than -80 °C, if possible, since the validity of the certified values at higher temperatures is unknown.

Handling: This material is a frozen tissue homogenate; after extended storage at temperatures of -25 °C or higher, or if allowed to warm, it will lose its powder-like form. For the handling of this material during sample preparation, the following procedures and precautions are recommended. If weighing relatively large quantities, remove a portion from the bottle and reweigh the bottle to determine the weight of the subsample. (Avoid heavy frost buildup on the containers by rapid handling and wiping of the containers prior to weighting.) Transfer subsamples to another container for weighing, using a pre-cooled thick-walled glass container rather than a thin-walled plastic container to minimize heat transfer to the sample. If possible, use a cold work space, e.g., an insulated container with dry ice or liquid nitrogen coolant on the bottom and pre-cooled implements, such as Teflon-coated spatulas, for transferring the powder. Normal biohazard safety precautions for the handling of biological tissues should be exercised.

Use: Subsamples of this SRM for analysis should be withdrawn from bottles immediately after opening and used without delay for the certified values listed in Tables 1 and 2 to be valid within the stated uncertainties. The bottles should be tightly closed immediately after removal of the subsamples and the remaining material may be frozen for use in later analyses. The concentrations of constituents in SRM 1945 are reported on a wet weight basis.

PREPARATION AND ANALYSIS

SRM Preparation: The whale blubber used to prepare this SRM was collected from an adult female pilot whale which was stranded on Cape Cod, MA in September, 1991. The blubber tissue was placed in Teflon bags, frozen on dry ice, shipped to NIST, and placed in a liquid nitrogen freezer (-150 °C). At NIST any remaining skin was removed using a titanium knife, and the blubber was again placed in Teflon bags and immediately returned to a liquid nitrogen freezer. Approximately 15 kg of whale blubber were prepared for use as the SRM. The frozen whale blubber was pulverized in batches of approximately 150 g each using a cryogenic grinding procedure described previously [1]. The total 15 kg of pulverized material was then combined and thoroughly mixed with a Teflon paddle. Subsamples (10-15 g) of the whale blubber homogenate were aliquoted into pre-cooled glass bottles and stored at -80 °C.

Determination of PCBs and Chlorinated Pesticides: SRM 1945 was analyzed for selected PCB congeners and chlorinated pesticides using GC-ECD and GC-MS following the general approach described previously [2,3]. For the GC-ECD analyses, duplicate samples of 2-3 g (wet weight) blubber from ten randomly selected bottles were mixed with approximately 100 g of pre-cleaned sodium sulfate and Soxhlet extracted for 18 h using approximately 200 mL of methylene chloride. The extracts were concentrated, and the majority of the lipid and biogenic material was removed using size exclusion chromatography (SEC) on a preparative-scale divinylbenzene-polystyrene column (10 μm particle size, 10 nm pore size). The eluent was concentrated, and the SEC fractionation was repeated. Normal-phase liquid chromatography on a semi-preparative-scale aminopropylsilane column was then used to isolate two fractions containing (1) the PCB congeners and lower polarity chlorinated pesticides and (2) the more polar chlorinated pesticides. GC-ECD analyses of the two fractions were performed on two columns of different selectivity for PCB separations: 0.25 mm i.d. x 60 m fused silica capillary column with a 5% phenyl-substituted methylpolysiloxane phase (0.25 μm film thickness) (DB-5, J&W Scientific, Folsom, CA) and a 0.32 mm i.d. x 100 m fused silica capillary column with a dimethylpolysiloxane phase containing 50% methyl C-18 (0.1 μm film thickness) (CP Sil 5 C18 CB, Chrompack International, Middelburg, The Netherlands).

For the GC-MS analyses, additional samples of 2-3 g (wet weight) from six randomly selected bottles were mixed with approximately 100 g of pre-cleaned sodium sulfate and Soxhlet extracted for 18 h using approximately 200 mL of 1:1 *n*-hexane/acetone (v/v). The extracts were concentrated and treated with concentrated sulfuric acid to remove the lipid interferences. A silica solid phase extraction column was then used to remove the polar interferences in the extracts. The GC-MS analyses were performed using a 0.25 mm i.d. x 60 m fused silica capillary with a 5% phenyl-substituted methylpolysiloxane phase (0.25 μm film thickness) (DB-5 MS, J&W Scientific, Folsom, CA).

Two PCB congeners, which are not significantly present in the blubber extract (PCB 103 and PCB 198), as well as octachloronaphthalene, endrin, and perdeuterated 4,4'-DDT were added to the whale blubber homogenate prior to extraction for use as internal standards for quantification purposes [4,5]. Calibration response factors for the analytes relative to the internal standards were determined by analyzing aliquots of SRMs 2261 (Concentrated Chlorinated Pesticides in Hexane), SRM 2262 (Concentrated PCB Congeners in Iso-octane), gravimetrically prepared solutions of additional analytes not contained in SRMs 2261 and 2262, and the internal standards.

Representative chromatograms for the PCB and lower polarity pesticide fraction from the GC-ECD analyses on the C-18 and DB-5 columns are shown in Figures 1 and 2, respectively. The GC-MS single ion monitoring chromatograms for the tetrachloro-, pentachloro-, hexachloro-, and heptachlorobiphenyl congeners are shown in Figure 3. A representative chromatogram for the more polar pesticide fraction from the GC-ECD analyses on the DB-5 column is shown in Figure 4.

Homogeneity Assessment: The homogeneity of SRM 1945 was assessed by analyzing duplicate samples of 2-3 g each from 10 randomly selected bottles as described in the previous section for the GC-ECD analyses. No statistically significant differences between bottles were observed for the PCB congeners or chlorinated pesticides at the 3-g sample size.

Non-volatile Extractable Weight Determination: The percent of non-volatile extractable material was determined for each sample after Soxhlet extraction (a total of 26 samples). The extract was evaporatively concentrated to approximately 20 mL (weight known), and an aliquot of 90 μL (weight known) was placed on an aluminum pan. The extract on the pan was air dried, and the weight of the dried extract was noted. The ratio of weights (x 100) is the percent of non-volatile extractable material. The percent non-volatile extractable material is 74.29 $\pm$ 0.45 (95% confidence interval for the mean).

Table 1. Certified Concentrations of PCB Congeners in SRM 1945

Polychlorinated Biphenyls[a]			Concentration (μg/kg wet weight)[b]		
PCB	18	(2,2',5-Trichlorobiphenyl)	4.48	±	0.88
PCB	44	(2,2',3,5'-Tetrachlorobiphenyl)	12.2	±	1.4
PCB	49	(2,2',4,5'-Tetrachlorobiphenyl)	20.8	±	2.8
PCB	52	(2,2',5,5'-Tetrachlorobiphenyl)	43.6	±	2.5
PCB	66	(2,3',4,4'-Tetrachlorobiphenyl)	23.6	±	1.6
PCB	87	(2,2',3,4,5'-Pentachlorobiphenyl)	16.7	±	1.4
PCB	95	(2,2',3,5',6-Pentachlorobiphenyl)	33.8	±	1.7
PCB	99	(2,2',4,4',5-Pentachlorobiphenyl)	45.4	±	5.4
PCB	101	(2,2',4,5,5'-Pentachlorobiphenyl)	65.2	±	5.6
	90	(2,2',3,4',5-Pentachlorobiphenyl)			
PCB	105	(2,3,3',4,4'-Pentachlorobiphenyl)	30.1	±	2.3
PCB	110	(2,3,3',4',6-Pentachlorobiphenyl)	23.3	±	4.0
PCB	118	(2,3',4,4',5-Pentachlorobiphenyl)	74.6	±	5.1
PCB	128	(2,2',3,3',4,4'-Hexachlorobiphenyl)	23.7	±	1.7
PCB	138	(2,2',3,4,4',5'-Hexachlorobiphenyl)	131.5	±	7.4
	163	(2,3,3',4',5,6-Hexachlorobiphenyl)			
	164	(2,3,3',4',5',6-Hexachlorobiphenyl)			
PCB	149	(2,2',3,4',5',6-Hexachlorobiphenyl)	106.6	±	8.4
PCB	151	(2,2',3,4,4',5-Hexachlorobiphenyl)	28.7	±	5.2
PCB	153	(2,2',4,4',5,5'-Hexachlorobiphenyl)	213	±	19
PCB	156	(2,3,3',4,4',5-Hexachlorobiphenyl)	10.3	±	1.1
PCB	170	(2,2',3,3',4,4',5-Heptachlorobiphenyl)	40.6	±	2.6
	190	(2,3,3',4,4',5,6-Heptachlorobiphenyl)			
PCB	180	(2,2',3,4,4',5,5'-Heptachlorobiphenyl)	106.7	±	5.3
PCB	183	(2,2',3,4,4',5',6-Heptachlorobiphenyl)	36.6	±	4.1
PCB	187	(2,2',3,4,5,5',6-Heptachlorobiphenyl)	105.1	±	9.1
PCB	194	(2,2',3,3',4,4',5,5'-Octachlorobiphenyl)	39.6	±	2.5
PCB	195	(2,2',3,3',4,4',5,6-Octachlorobiphenyl)	17.7	±	4.3
PCB	201	(2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl)	16.96	±	0.89
PCB	206	(2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl)	31.1	±	2.7
PCB	209	(Decachlorobiphenyl)	10.6	±	1.1

[a] PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [4] and later revised by Schulte and Malisch [5] to conform with IUPAC rules. When two or more congeners are known to coelute, the PCB congener listed first is the major component and the additional congeners may be present as minor components. The quantitative results are based on the response of congener listed first.

[b] The certified values are weighted means of results from three analytical techniques as described by Schiller and Eberhardt [6]. The uncertainty is based on a 95% confidence interval for the true concentration, and includes an allowance for differences between the analytical methods used.

Table 2. Certified Concentrations of Chlorinated Pesticides in SRM 1945

Chlorinated Pesticides	Concentration (μg/kg wet weight)[a]
Hexachlorobenzene	32.9 ± 1.7
α-HCH	16.2 ± 3.4
γ-HCH	3.30 ± 0.81
Heptachlor epoxide	10.8 ± 1.3
Oxychlordane	19.8 ± 1.9
Mirex	28.9 ± 2.8
cis-Chlordane (α-Chlordane)	46.9 ± 2.8
cis-Nonachlor	48.7 ± 7.6
trans-Nonachlor	231 ± 11
2,4'-DDE	12.28 ± 0.87
4,4'-DDE	445 ± 37
2,4'-DDD	18.1 ± 2.8
4,4'-DDD	133 ± 10
2,4'-DDT	106 ± 14
4,4'-DDT	245 ± 15

[a] The certified values are weighted means of results from three analytical techniques as described by Schiller and Eberhardt [6]. The uncertainty is based on a 95% confidence interval for the true concentration, and includes an allowance for differences between the analytical methods used.

SUPPLEMENTAL INFORMATION

Noncertified Quantitative Values: Appendices A through C contain supplementary analytical results obtained during the course of the certification of SRM 1945. Noncertified concentration values are listed in Appendix A for additional PCB congeners and chlorinated pesticides. These values are the results obtained by the measurement technique indicated and may include unrecognized bias; therefore, they are provided for information only. NIST does not recommend that this information be used for calibration, bias evaluation, or similar purposes for which certified values are used. Appendices B and C contain summaries of the analytical results obtained using the various analytical techniques for the determination of PCB congeners and chlorinated pesticides, respectively.

APPENDIX A

Noncertified Concentrations of PCB Congeners and Chlorinated Pesticides in SRM 1945

NOTE: The noncertified values listed below, have not been confirmed by an independent analytical technique as required for certification. Although bias has not been evaluated for the procedures used, the noncertified concentrations should be useful for comparison with results obtained using similar procedures (i.e., Soxhlet extraction and GC-ECD or GC-MS analyses on similar columns).

Polychlorinated Biphenyls[a]	Concentration (μg/kg wet weight)[b]
PCB 28	14.1 ± 1.4[c]
PCB 31	3.12 ± 0.69[d]
Chlorinated Pesticides	
Dieldrin	37.5 ± 3.9[d]
β-HCH	8.0 ± 1.4[e]

[a] PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [4] and later revised by Schulte and Malisch [5] to conform with IUPAC rules.

[b] Concentrations are the weighted mean from the results of the indicated techniques. Uncertainties for the measurements are a 95% confidence interval for the mean for PCB 28 and β-HCH and a 95% confidence interval including an allowance for differences between the analytical methods used for PCB 31 and dieldrin.

[c] Concentration determined by GC-ECD on DB-5 column.

[d] Concentration determined by GC-ECD on C-18 column and DB-5 column.

[e] Concentration determined by GC-MS.

APPENDIX B

Summary of Analytical Results for the Determination of PCB Congeners in SRM 1945

Polychlorinated Biphenyls[a]			Concentration (μg/kg wet weight)					
			GC-ECD[b] (C-18 Column)		GC-ECD[b] (DB-5 Column)		GC-MS[c] (DB-5 Column)	
PCB	18	(2,2,5-Trichlorobiphenyl)	4.91	(0.32)	3.78	(0.30)	4.76	(0.33)
PCB	28	(2,4,4'-Trichlorobiphenyl)	12.16	(0.91)	14.14	(0.47)	[16.52][d]	(0.38)
PCB	31	(2,4',5-Trichlorobiphenyl)	2.55	(0.23)	3.70	(0.24)	[16.52][d]	(0.38)
PCB	44	(2,2',3,5'-Tetrachlorobiphenyl)	12.25	(0.64)	13.17	(0.74)	11.25	(0.94)
PCB	49	(2,2',4,5'-Tetrachlorobiphenyl)	18.54	(0.84)	22.64	(0.74)	21.28	(0.95)
PCB	52	(2,2',5,5'-Tetrachlorobiphenyl)	45.1	(2.5)	42.8	(2.9)	42.4	(2.1)
PCB	66	(2,3',4,4'-Tetrachlorobiphenyl)	23.2	(1.8)	[59.7][e]	(2.8)	23.7	(1.9)
PCB	87	(2,2',3,4,5'-Pentachlorobiphenyl)	16.25	(0.68)	17.71	(0.91)	16.66	(0.47)
PCB	95	(2,2',3,5',6-Pentachlorobiphenyl)	34.1	(1.7)	[59.7][e]	(2.8)	33.6	(1.6)
PCB	99	(2,2',4,4',5-Pentachlorobiphenyl)	41.5	(2.9)	47.3	(2.8)	47.3	(2.3)
PCB	101	(2,2',4,5,5'-Pentachlorobiphenyl)	61.6	(3.3)	66.3	(4.3)	68.3	(2.3)
	90	(2,2',3,4',5-Pentachlorobiphenyl)						
PCB	105	(2,3,3',4,4'-Pentachlorobiphenyl)	31.6	(2.4)	30.0	(2.4)	29.8	(1.3)
PCB	110	(2,3,3',4',6-Pentachlorobiphenyl)	25.9	(2.1)	[36.1][f]	(3.3)	20.7	(1.6)
PCB	118	(2,3',4,4',5-Pentachlorobiphenyl)	74.9	(4.7)	75.6	(4.8)	71.7	(3.7)
PCB	128	(2,2',3,3',4,4'-Hexachlorobiphenyl)	23.2	(2.2)	24.6	(2.1)	22.9	(1.5)
PCB	138	(2,2',3,4,4',5'-Hexachlorobiphenyl)	131.7	(8.0)	127.7	(9.0)	134.2	(5.7)
	163	(2,3,3',4',5,6-Hexachlorobiphenyl)						
	164	(2,3,3',4',5',6-Hexachlorobiphenyl)						
PCB	149	(2,2',3,4',5',6-Hexachlorobiphenyl)	101.6	(6.6)	106.6	(7.5)	110.6	(4.7)
PCB	151	(2,2',3,5,5',6-Hexachlorobiphenyl)	32.9	(2.3)	26.7	(2.4)	26.7	(1.2)
PCB	153	(2,2',4,4',5,5'-Hexachlorobiphenyl)	214	(15)	201	(16)	215	(11)
PCB	156	(2,3,3',4,4',5-Hexachlorobiphenyl)	9.76	(0.69)	10.93	(0.66)	10.07	(0.72)

APPENDIX B (Continued)

Summary of Analytical Results for the Determination of PCB Congeners in SRM 1945

Polychlorinated Biphenyls[a]			Concentration (μg/kg wet weight)					
			GC-ECD[b] (C-18 Column)		GC-ECD[b] (DB-5 Column)		GC-MS[c] (DB-5 Column)	
PCB	170	(2,2',3,3',4,4',5-Heptachlorobiphenyl)	39.3	(2.6)	40.2	(2.6)	42.4	(1.9)
	190	(2,3,3',4,4',5,6-Heptachlorobiphenyl)						
PCB	180	(2,2',3,4,4',5,5'-Heptachlorobiphenyl)	105.5	(6.2)	108.8	(6.7)	109.5	(4.9)
PCB	183	(2,2',3,4,4',5',6-Heptachlorobiphenyl)	39.5	(2.9)	36.0	(2.8)	34.4	(1.8)
PCB	187	(2,2'3,4,5,5',6-Heptachlorobiphenyl)	111.7	(7.0)	103.1	(6.7)	100.9	(5.1)
PCB	194	(2,2',3,3',4,4',5,5'-Octachlorobiphenyl)	39.2	(2.4)	41.3	(2.6)	39.4	(1.8)
PCB	195	(2,2',3,3',4,4',5,6-Octachlorobiphenyl)	14.0	(1.3)	20.2	(1.2)	19.0	(1.2)
PCB	201	(2,2',3,3',4,5',6,6'-Octachlorobiphenyl)	16.9	(1.1)	17.2	(1.1)	16.48	(0.82)
PCB	206	(2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl)	32.9	(1.9)	30.7	(1.9)	29.7	(1.6)
PCB	209	(Decachlorobiphenyl)	10.75	(0.87)	11.16	(0.94)	9.95	(0.85)

[a] PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [4] and later revised by Schulte and Malisch [5] to conform with IUPAC rules. When two or more congeners are known to coelute, the PCB congener listed first is the major component and the additional congeners may be present as minor components. The quantitative results are based on the response of the congener listed first.

[b] Duplicate samples from ten bottles were extracted, and each extract was analyzed in duplicate. Concentrations are the average, and numbers in parentheses are one standard deviation of a single measurement. The same extracts analyzed by GC-ECD on the C-18 column were also analyzed using the DB-5 column.

[c] Samples from six bottles were extracted and analyzed in duplicate. Concentrations are the average, and numbers in parentheses are one standard deviation of a single measurement.

[d] Numbers in [ ] indicate known coelution of two or more congeners. PCB 28 and 31 coelute under the GC-MS conditions used.

[e] Numbers in [ ] indicate known coelution of two or more congeners. PCB 66 and 95 coelute under the GC-ECD (DB-5 column) conditions used.

[f] Numbers in [ ] indicate known coelution of two or more congeners. PCB 77 (3,3',4,4'-Tetrachlorobiphenyl) and 110 coelute under the GC-ECD (DB-5 column) conditions used.

APPENDIX C

Summary of Analytical Results for the Determination of Chlorinated Pesticides in SRM 1945

Compound	Concentration (μg/kg wet weight) GC-ECD[a] (C-18 Column)		GC-ECD[a] (DB-5 Column)		GC-MS[b] (DB-5 Column)	
2,4'-DDE	12.46	(0.57)	11.69	(0.70)	12.27	(0.54)
4,4'-DDE	421	(31)	453	(25)	440	(34)
2,4'-DDD	20.0	(1.3)	16.2	(1.4)	17.96	(0.93)
4,4'-DDD	128	(10)	132.4	(9.1)	138.3	(6.2)
2,4'-DDT	113.0	(7.0)	98	(10)	101.6	(6.3)
4,4'-DDT	238	(46)			246	(12)
Hexachlorobenzene	32.2	(2.1)	33.1	(2.2)	33.8	(1.8)
γ-HCH	3.90	(0.37)	2.63	(0.37)	3.37	(0.24)
α-HCH	18.6	(1.2)	14.4	(1.3)	15.3	(1.0)
β-HCH	ND[c]		ND[c]		8.04	(0.71)
Heptachlor epoxide	10.74	(0.87)	10.1	(1.2)	11.2	(1.0)
Oxychlordane	20.6	(1.5)	20.7	(1.0)	18.61	(0.83)
cis-Chlordane	47.2	(3.2)	46.0	(3.4)	47.2	(2.5)
cis-Nonachlor	54.2	(3.2)	47.8	(3.1)	44.3	(2.7)
trans-Nonachlor	229	(16)	233	(16)	232	(10)
Dieldrin	39.4	(3.0)	35.5	(3.1)	ND[d]	
Mirex	30.1	(2.3)	29.6	(2.3)	26.8	(2.2)

[a] Duplicate samples from ten bottles were extracted, and each extract was analyzed in duplicate. Concentrations are the average, and numbers in parentheses are one standard deviation of a single measurement. The same extracts analyzed by GC-ECD on C-18 column were also analyzed using the DB-5 column.

[b] Samples from six bottles were extracted and analyzed in duplicate. Concentrations are the average, and numbers in parentheses are one standard deviation of a single measurement.

[c] ND = not determined since β-HCH is not recovered from the normal-phase LC clean-up step.

[d] ND = not determined since dieldrin is destroyed in the sulfuric acid step.

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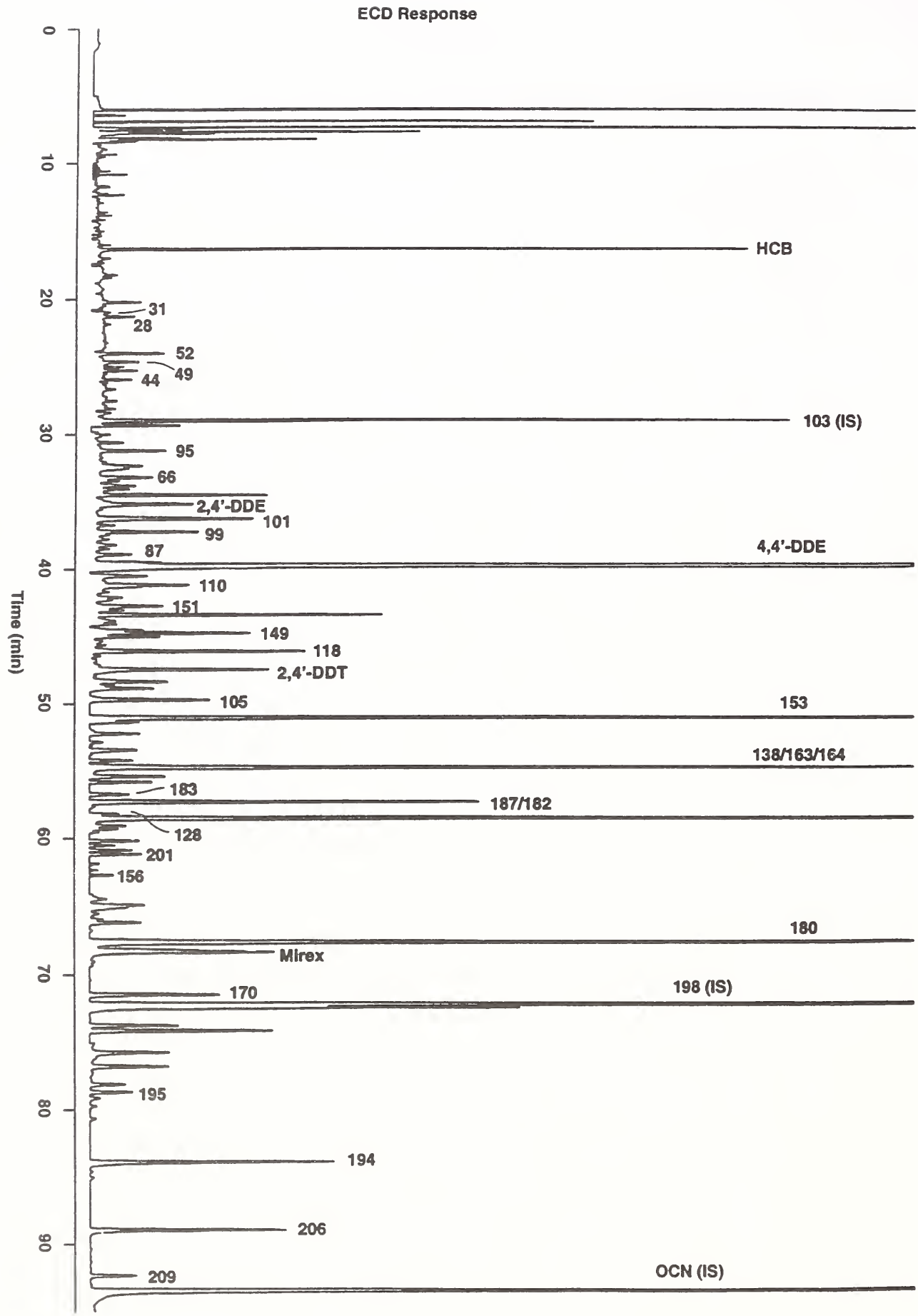

Figure 1. GC-ECD analysis of the PCB and lower polarity pesticide fraction isolated from SRM 1945 (Organics in Whale Blubber) on a 0.32 mm i.d. x 100 m C-18 column.

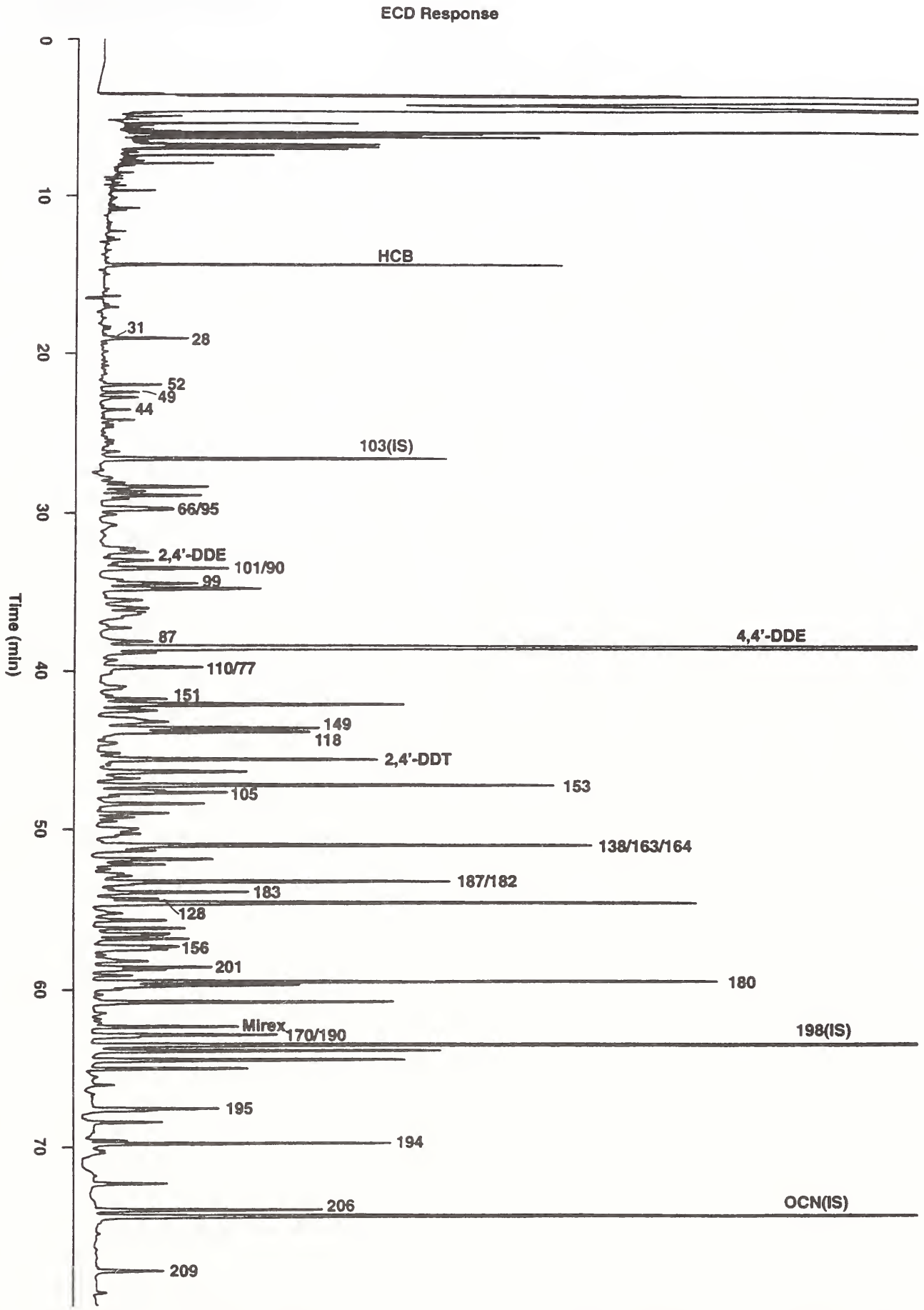

Figure 2. GC-ECD analysis of the PCB and lower polarity pesticide fraction isolated from SRM 1945 (Organics in Whale Blubber) on a 0.25 mm i.d. x 60 m DB-5 column.

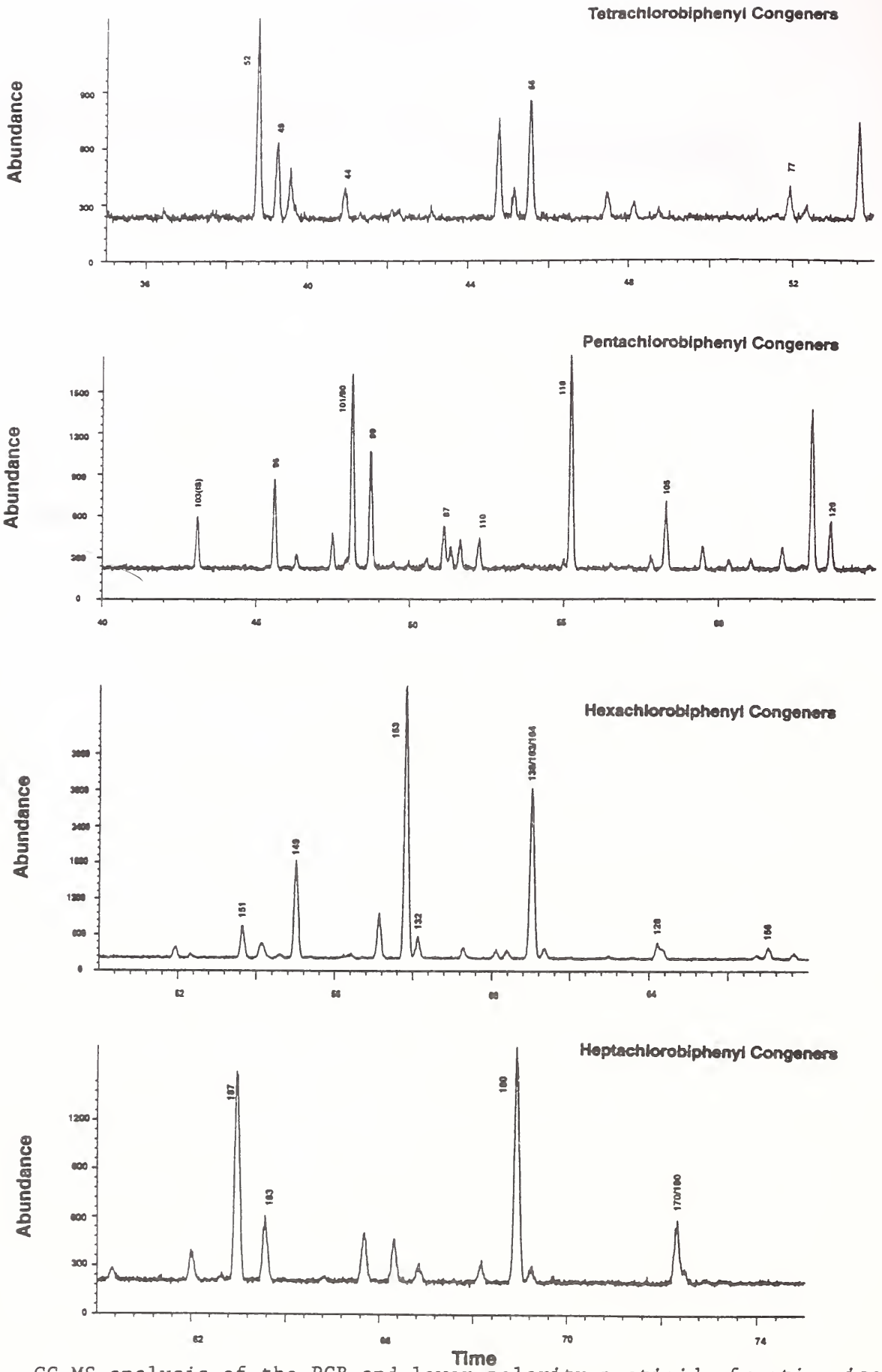

Figure 3. GC-MS analysis of the PCB and lower polarity pesticide fraction isolated from SRM 1945 (Organics in Whale Blubber) on a 0.25 mm i.d. x 60 m DB-5 column. (A) tetrachlorobiphenyl congeners monitored at ion 289.90, (B) pentachlorobiphenyl congeners monitored at ion 323.90, (C) hexachlorobiphenyl congeners monitored at ion 359.85, and (D) heptachlorobiphenyl congeners monitored at ion 393.80.

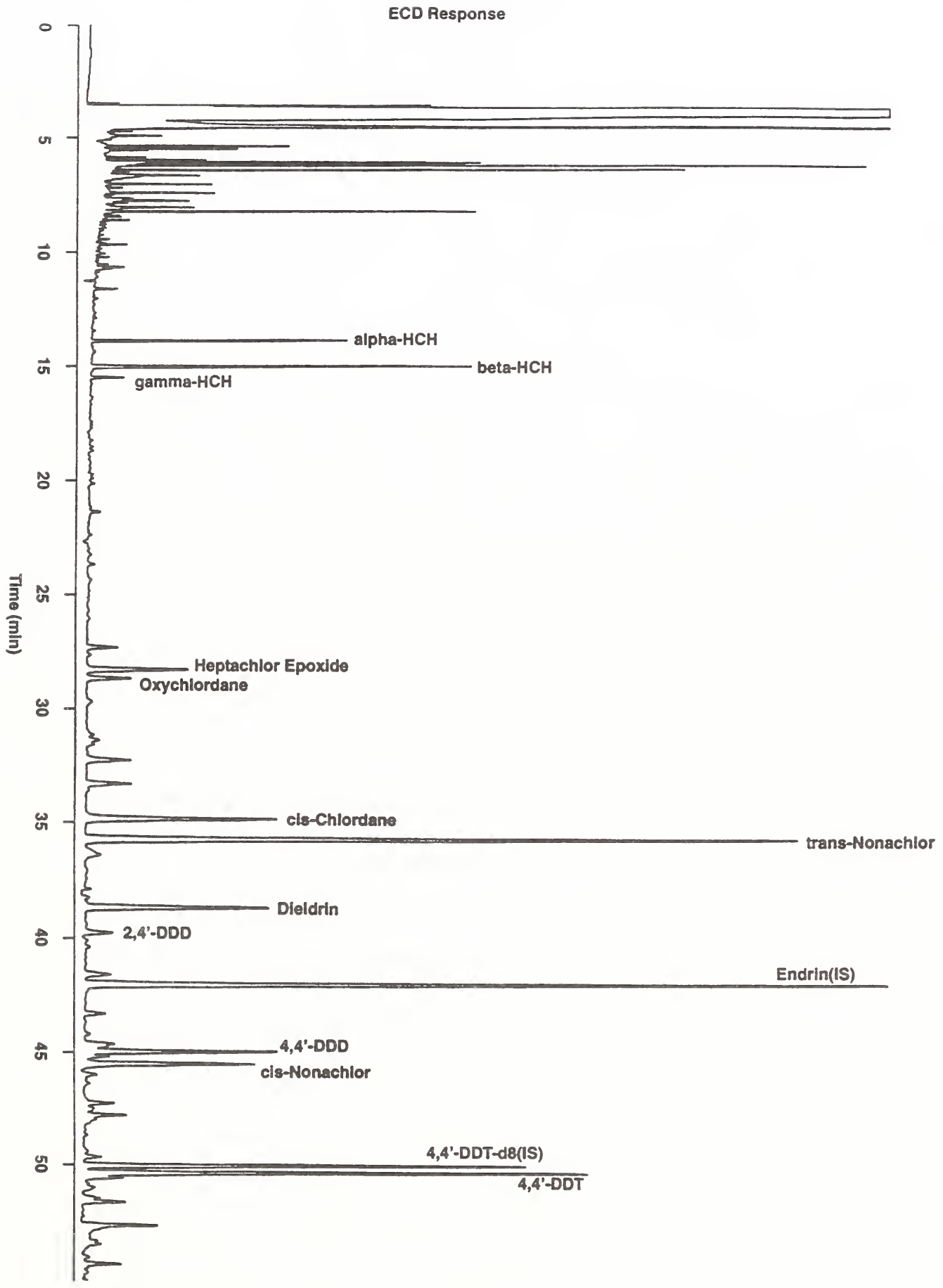

Figure 4. GC-ECD analysis of the more polar pesticide fraction isolated from SRM 1945 (Organics in Whale Blubber) on a 0.25 mm i.d. x 60 m DB-5 column.