

## Texas Department of State Health Services Division for Regulatory Services Environmental and Consumer Safety Section Policy/Standards/Quality Assurance Unit Seafood and Aquatic Life Group Survey Team

## Standard Operating Procedures and Quality Control/Assurance Manual

TEXAS DEPARTMENT OF STATE HEALTH SERVICES SEAFOOD AND AQUATIC LIFE GROUP 1100 WEST 49<sup>TH</sup> STREET AUSTIN, TEXAS 78756

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#### 1.0 Mission

The mission of the Seafood and Aquatic Life Group Survey Team is to protect consumers and recreational fishers from disease or chemical contaminants found in fish and other aquatic organisms harvested from Texas' lakes, rivers, and bays or near shore state waters.

#### 2.0 Goal

The goal of the Seafood and Aquatic Life Group Survey Team is to provide scientifically valid and defensible data for characterizing public health risks associated with the consumption of fish or shellfish.

#### 3.0 Introduction

Chemical Contamination of aquatic resources has occurred since the Industrial Revolution began in the early 1800s. Environmental concentrations of chemical contaminants have increased from the time of the Industrial revolution to today due to intensifying urbanization, industrial development, and use of new agricultural chemicals [1]. However, increased awareness of aquatic pollution along with stronger environmental laws has decreased the concentrations of some chemical contaminants in aquatic resources over the past thirty (30) years. Chemical contaminants affecting our aquatic resources today come from a variety of sources including permitted point source discharges (e.g. industrial and municipal facilities), accidental spills, and nonpoint sources (e.g. agricultural practices, resource extraction, urban runoff, in-place sediment contamination, groundwater recharge, vehicle exhaust, and atmospheric deposition from various combustion and incineration processes). Typical chemical contaminants from these pollution sources may include heavy metals, pesticides, polychlorinated biphenyls (PCBs), or other complex volatile and semi-volatile organic compounds.

As chemical contaminants reach surface waters, they become part of aquatic food chains. Some aquatic organisms easily absorb contaminants from water, sediments, or from other aquatic organisms; if absorption of contaminants is not immediately balanced by excretion, the concentration of contaminants in the organism may exceed the concentration in the surrounding waters or foods, a process known as bioconcentration. Some fish have no physiological mechanisms for removing contaminants from their bodies. Continued absorption of contaminants without concomitant excretion results in accumulation of the substance in tissues, a process called bioaccumulation [2]. It follows from the process of bioaccumulation that older, larger fish may contain higher levels of contaminants than younger, smaller fish. Understanding the dynamic process of bioconcentration and bioaccumulation is very important in protecting humans and other organisms from the adverse effects of chemical exposure, and it has become a critical consideration in the regulation of chemicals; thus, fish and shellfish tissue contaminant monitoring serves as an important indicator for chemical contamination of our aquatic environments.

The Texas Department of State Health Services (DSHS) is charged under the Health and Safety Code, Chapter 436, Texas Aquatic Life Act to declare a body of public water a prohibited area if a sanitary, chemical, or bacteriological survey indicates aquatic life is unfit for human consumption [3]. To carry out this charge, the DSHS Seafood and Aquatic Life Group monitor's chemical contaminant levels in fish and shellfish from Texas' lakes, rivers, bays, or near shore state waters to determine the public health risks associated with consumption of these food sources. This manual was developed to provide a standardized format for collecting, processing, and maintaining quality fish and shellfish chemical contaminant data. It also serves as a reference or guidance document for environmental specialists and biologists on fish and shellfish collection and processing techniques for chemical contaminant studies. The quality of our fish and shellfish sampling techniques determines the usefulness and reliability of the data for determining the public health risks associated with consumption of fish and shellfish. This manual is based, in part, on the procedures established by the *U.S. EPA Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories, Volume 2* [1] and guidance from the State of Texas' Toxic Substances Coordinating Committee (TSCC) and Fish Sampling Advisory Subcommittee.

## 4.0 Study Design

DSHS conducts intensive fish and shellfish studies to determine size specific levels of chemical contamination in fish and shellfish, to assess geographic extent of chemical contamination, and to characterize the human health risk associated with the consumption of environmentally contaminated fish and shellfish.

Prior to initiating an intensive study, DSHS staff develops a detailed sampling plan that clearly defines the following seven (7) major components: study objectives, site selection, target species and size class, target analytes, sample collection time, sample type, and sample size. This section describes each of the seven (7) major components and provides guidance for developing a detailed sampling plan.

#### Study Objectives

The primary objective of DSHS fish and shellfish tissue chemical contaminant studies is to characterize the human health risk associated with the consumption of fish or shellfish and to make the appropriate risk management decisions based on the public health risks calculated through the risk characterization process.

#### Site Selection

Sample sites are selected based on the assessment and professional judgment of the following seven (7) factors by DSHS staff: 1) location of point source pollution, 2) fishing pressure (e.g. sport, commercial, and subsistence), 3) public access, 4) distribution of fish and shellfish habitat, 5) review of relevant water, sediment, and tissue data (e.g. Tier 1-screening studies or historical fish and shellfish tissue sampling), 6) assessment of watershed activities and potential nonpoint source pollution inputs, and 7) site accessibility. When study resources are limited, sample sites must be selected to target fish and shellfish harvest areas suspected of having the highest levels of chemical contamination and posing the greatest potential human health risk from consumption of fish and shellfish.

Studies designed to evaluate the effects of point-source pollution must have sample sites located at, or just downstream of, the discharge point, and include sample sites that are located in areas assumed minimally impacted from the point source discharge. This study design identifies extremes of bioaccumulation, ranging from presumed undisturbed reference sites to sites where existing data or the presence of potential pollutant sources suggest significant chemical contamination and allows DSHS to characterize the risk from consumption of chemically contaminated fish and shellfish for the entire study area.

Studies designed to reevaluate existing fish and shellfish tissue chemical contamination problems (i.e. consumption advisories and bans) must select sample sites based on the location of historical sample sites. This approach allows temporal and spatial data comparison and reevaluation of the existing human health risk management actions.

#### Target Species and Size Class Selection

The 1993 United States Environmental Protection Agency (USEPA) Fish Contaminant Workgroup developed freshwater and estuarine/marine ecosystems target species lists for state contaminant monitoring programs assessing human consumption concerns [1]. The target species lists were developed based on review of species used in the following national monitoring programs: National Study of Chemical Residues in Fish (USEPA), National Dioxin Study (USEPA), National Pesticide Monitoring Program (United States Fish and Wildlife Service (USFWS)), National Contaminant Biomonitoring Program (USFWS), National Water Quality Assessment Program (United States Geological Survey (USGS)), and on a review of fish and shellfish species cited in state fish and shellfish consumption advisories or bans. The criteria used to select target species were similar in all monitoring programs reviewed by the workgroup. However, the priority given to each criterion may vary depending on the monitoring program objectives. According to the 1993 USEPA Fish Contaminant Workgroup, the three most important criterion for selecting target fish and shellfish for state contaminant monitoring programs assessing the human health risks associated with consumption of fish or shellfish were that species have the potential to bioaccumulate high concentrations of chemical contaminants, species were commonly consumed in the study area and were of commercial, recreational, or subsistence fishing value, and that species have a wide geographic distribution [1]. In addition to the three primary criteria for target species selection, it is also important that the target species be easy to identify taxonomically because of species-specific differences in bioaccumulation potential, and it is both practical and cost-effective to sample target species that are abundant, easy to capture, and large enough to provide adequate tissue samples for chemical analyses.

Sampling of two distinct ecological groups of fish (i.e. bottom-feeders and predators) as target species in freshwater and estuarine/marine systems is recommended. By sampling fish from different ecological groups, it allows a fish and shellfish tissue study to monitor ecological group-specific habitats, feeding strategies, and physiological factors. These ecological group-specific factors may contribute to differences in bioaccumulation of chemical contaminants. Table 1 and Table 2 list the recommended target species for Texas freshwater and estuarine and marine environments, respectively. Preferred target species are labeled for freshwater and estuarine and marine environments (Table 1 and Table 2 foot notes). If the preferred or recommended target species are unavailable at the selected study site(s), collect available species as sample specimens. For reevaluation of previously studied water bodies, target species selected must be, the same or similar to target species collected in previous studies.

Fish and shellfish sample specimens collected must be of harvestable size as defined by Texas Parks and Wildlife Department (TPWD) freshwater and saltwater fishing statewide regulations [4]. When harvestable size fish or shellfish are unavailable, fish or shellfish of any size may be selected as sample specimens (refer to the *Sample Type* section for more details on selecting and processing small fish or shellfish sample specimens).

Common Name	Scientific Name	Length Limit <sup>2</sup>
Predatory Species		
Largemouth bass <sup>1</sup>	Micropterus salmoides	≥ 14"
Walleye	Stizostedion vitreum	no length limit
Longnose gar	Lepisosteus osseus	no length limit
Sunfish species	Lepomis sp.	no length limit.
Flathead catfish <sup>1</sup>	Pylodictus olivaris	≥18"
White crappie <sup>1</sup>	Pomoxis annularis	≥ 10"
Black crappie <sup>1</sup>	Pomoxis nigromaculatus	≥ 10"
Freshwater drum <sup>1</sup>	Aplodinotus grunniens	no length limit
White bass <sup>1</sup>	Morone chrysops	≥ 10"
Striped bass <sup>1</sup>	Morone saxatilis	≥18"
Hybrid striped bass <sup>1</sup>	Morone saxatilis x chrysops	≥18"
Blue Tilapia	Tilapia aurea	no length limit
<b>Bottom Feeding Specie</b>	<u>s</u>	
Channel catfish <sup>1</sup>	Ictalurus punctatus	≥12"
Blue catfish <sup>1</sup>	Ictalurus furcatus	≥12"
Common carp <sup>1</sup>	Cyprinus carpio	no length limit
River carpsucker	Carpiodes carpio	no length limit
Smallmouth buffalo	Ictiobus bubalus	no length limit
Redhorse species	Moxostoma sp.	no length limit
<sup>1</sup> Indicates preferred targe	et species	

#### Table 1. Freshwater Target Species

<sup>1</sup> Indicates preferred target species. <sup>2</sup> These are statewide regulations enforced by TPWD. Length limits may vary by water body due to special regulations. Exceptions to the statewide regulations are published in TPWD Outdoor Annual [4].

Common Name	Scientific Name	Length Limit <sup>2</sup>
Predatory Species		
Red drum <sup>1</sup>	Sciaenops ocellatus	20" - 28"
Spotted seatrout <sup>1</sup>	Cynoscion nebulosus	≥15"
Southern flounder <sup>1</sup>	Paralichthys lethostigma	≥14"
Sand trout	Cynoscion arenarius	no length limit
King mackerel	Scomberomorus cavalla	≥27"
<b>Bottom Feeding Species</b>		
Atlantic croaker	Micropogonias undulatus	no length limit
Black drum <sup>1</sup>	Pogonias cromis	14" - 30"
Sheepshead <sup>1</sup>	Archosargus probatocephalus	≥12"
Gafftopsail catfish <sup>1</sup>	Bagre marinus	$\geq 14''$
Hardhead catfish	Arius felis	no length limit
<b>Oyster Species</b>		
Eastern oyster	Crassostrea virginica	≥ 3"
Shrimp Species		
Brown shrimp <sup>1</sup>	Penaeus aztecus	no length limit
White shrimp <sup>1</sup>	Penaeus setiferus	no length limit
Pink shrimp	Penaeus duorarum	no length limit
Crab Species		
Blue crab <sup>1</sup>	Callinectes sapidus	≥ 5"
Stone crab	Mennipe mercenaria	$\geq$ 2.5" claw length
		(right claw only)

#### ring and Marina Target Species Table 2 $\mathbf{T}$

<sup>1</sup> Indicates preferred target species. <sup>2</sup> These are statewide regulations enforced by TPWD. Length limits may vary by water body due to special regulations. Exceptions to the statewide regulations are published in TPWD Outdoor Annual [4].

#### Sample Type

Individual fish tissue fillet samples (skin-off fillet) are required, which allow for a more detailed analysis of size versus contaminant concentration when developing a risk characterization. However, small target fish species and shellfish composite samples are appropriate to meet the 200-gram minimum size requirements for laboratory analyses. For composite samples, the smallest specimen in the composite must be at least 75% of the total length of the largest specimen in the composite. Composite samples must consist of specimens from the same species and consist of two to five fish, four to six crabs, and an appropriate number of oysters to ensure that 200-gram minimum size requirement for laboratory analyses are met.

#### Target Analyte Selection

The appropriate target analyte selection is essential to the adequate protection of fish and shellfish consumers [1]. The USEPA has developed a list of recommended target analytes for fish and shellfish chemical contaminant studies from a review of the following information: pollutants analyzed in several national and regional fish contaminant monitoring programs, pesticides with active registrations, contaminants that have triggered states to issue fish and shellfish consumption advisories or bans, and published literature on the chemistry and health effects of potential contaminants [1]. DSHS has developed a list of target analytes based on EPA's recommendations, chemical contaminants previously identified in water quality, sediment, and fish tissue studies, and guidance from the state of Texas' Toxic Substances Coordinating Committee (TSCC) and the Fish Sampling Advisory Subcommittee [Appendix 1]. The target analyte list is divided into sections by chemical contaminant type: metals, pesticides, polychlorinated biphenyls (PCBs), volatile organic compounds (VOCs), semivolatile organic compounds (SVOCs), and Dioxin/Furans.

Fish tissue chemical contaminant studies are usually a result of the discovery of specific chemical contaminants during water quality monitoring, sediment monitoring, or identification of pollution sources. Ideally, a fish tissue contaminant study should look at all target analytes because this provides the greatest amount of information for fishers; however, financial resources available to complete the laboratory analyses are usually limited. Thus, DSHS uses a watershed-based approach to select target analytes [1]. This approach takes into consideration the following target analyte selection and prioritization factors: land use categories (i.e. rural, agricultural, suburban/urban, and industrial) [1; table 4-3], as well as geological characteristics, identified point source pollution, national pollution trends, available environmental data (i.e. fish tissue screening studies, water quality and sediment data), and financial resources. The watershed- based approach gives the highest priority to chemical contaminants that are widely dispersed nationally, relatively inexpensive to analyze, and assigns priority selection to target analytes based on the target analyte selection factors.

All DSHS fish tissue studies also adhere to the following target analyte selection guideline: for every five (5) fish or shellfish tissue samples collected for a study one (1) full scan analysis (i.e. metals, pesticides, PCBs, dioxins and furans, SVOCs, and VOCs analyses) is completed for a selected fish or shellfish tissue sample. In the event that DSHS is the first to conduct a fish or shellfish tissue chemical contaminant study of a water body, DSHS maximizes the number of full scans based on the available budget for a study to fully evaluate the extent of chemical contamination.

#### Sampling Times

Fish and shellfish tissue samples may be collected at any time during the year, with emphasis placed on the time of year when the target species are effectively sampled and most frequently harvested for consumption.

#### Sample Sizes

Sample sizes are generally subjectively determined by DSHS from the following guidelines: tissue sample analysis budget, size of water body, and if historical data is available, the use of a power of statistical test defined by the USEPA [1] may be used to determine the appropriate sample size for a fish and shellfish tissue study.

### 5.0 Sample Collection

#### Scientific Collection Permit

A scientific collection permit issued by TPWD must be obtained from TPWD and taken into the field when collecting samples. The TPWD law enforcement office nearest to the study area must be notified at least 24 hours prior to sampling. The scientific collection permit must be renewed annually.

#### Methods of Collection

The collection method chosen will depend on the target species sought and the conditions at the collection site. Both active and passive methods may be used, depending on which is the most cost and time effective. The primary fish collection methods available are electrofishing (active) and gill netting (passive). Passive capture techniques, such as gill nets, crab traps, trap nets, trot lines, etc.; can be used as long as gear is checked frequently to avoid sample deterioration. If needed, hook and line may be used as a collection method. Fish not selected for analysis must be released at the site of collection. Fish selected for analysis must be placed in a live well or immediately placed in a clean ice chest and iced. All ice chests must be scrubbed with detergent and rinsed with tap water, distilled water, or ambient water between uses.

#### Electrofishing

#### **Gear Requirements**

- **Boat**: All electrofishing boats must have a semi- V or flat bottom aluminum hull that has sufficient bow deck size to accommodate one dipper. The boat must have a bow rail waist height constructed of metal extending to the rear of the bow deck. Outboard motor and options must be selected based on boat size and use. (e.g. river-small water body boat or reservoir-bay boat). The reservoir-bay boat must be a minimum of 18 feet in length and equipped with a minimum of an 80 hp outboard motor with electric start and power trim/tilt. The river-small water body boat must be a minimum of 12 feet in length.
- **Pulsator and Generator**: The electrofishing boat must be equipped with a Smith Root Model GPP 2.5, 5.0, or 7.5. The model chosen must be based on the boat size and specifications.
- **Booms and Arrays**: The electrofishing boat must have booms constructed of PVC pipe or fiberglass. Boom design must be based on boat size and use. The reservoir-bay boat must have booms 7-8 feet in length mounted from the corners of the bow. The booms must be adjustable for height and direction. The river small-water body boat must have a "T" boom design mounted from the center of the bow rail. The boom must be adjustable for height. Boom length may be determined by user(s) not to exceed 8 feet in length and width. The electrofishing unit must be equipped with Smith Root SAA-6 adjustable anode arrays. The boat hull must be wired to act as the cathode.

#### • Safety Equipment and Procedures:

- 1) The electrofishing unit must be equipped with a Smith Root single or dual foot operated safety switch.
- 2) The electrofishing unit generator must be grounded to the hull.
- 3) Bow and boat floor decking must have a non-skid surface.
- 4) Dip nets must have fiberglass handles.
- 5) Dippers shall wear rubber-soled shoes or rubber boots and lineman gloves (1,000-volt minimum rated).
- 6) The electrofishing boat operator-driver and dipper(s) shall wear hearing protection.
- 7) All staff of the SALG Survey Team must be trained in Cardiopulmonary Resuscitation (CPR) and First Aid.
- 8) All staff of the SALG Survey Team and volunteers or observers must be familiar with electrofishing safety procedures.
- **Sample Activities:** Electrofishing should be conducted in shoreline areas accessible by the electrofishing boat. Sampling areas within a designated sample site should be selected based on the availability of suitable fish habitat to optimize sampling efficiency.

#### **Gill Netting**

#### **Gear Requirements**

• **Gill Net Size/Design:** No standard gill net size is required. Gill net length, depth, mesh size, and twine size may vary depending on study objectives and/or size of target species sought. However, the recommended gill net size for reservoir and bay sampling ranges from 120 to 300-ft by 8-ft deep consisting of single or multiple panels of square mesh monofilament ranging in size from 2-3 inches. If multiple mesh sizes are chosen, the gill net should be divided into mesh panels of equal length and depth. The float and bottom lines must be constructed of foam core and #30 lead core, respectively.

Length	Depth	Mesh Size	Twine Size
		2.0" (40')	#139
120'	8'	2.5" (40')	#139
		3.0" (40')	#139

#### • Gill net specification examples:

Length	Depth	Mesh Size	Twine Size
300'	8'	2.5" (300')	#139

• **Related Equipment:** At least two (2) floating buoys, not less than 6 inches in length, width, and height must be attached with poly or nylon rope to the both ends of the float line of each net, and at least two (2) weights must be attached to the lead core line, one weight at each end of the net. An alternate method may be used to secure gill nets in shallow water. This method requires the use of stakes or poles to stake the lead core line to reservoir or estuary bottom. All gill nets must be identified by an identification tag affixed to the gill net float line near the buoy or identification written on the buoy surface. Wording on the buoy and/or tag must be as follows:

#### Texas Department of State Health Services Seafood and Aquatic Life Group Scientific Permit Number SPR-0890-247

• **Sample Activities:** Gill nets should be set in the late afternoon—fished overnight and retrieved the next day. Overnight gill net sets allow the nets to be fished during two low light periods optimizing fish catch. Suitable fish habitat should be located and seasonal fish movement should be accounted for when selecting areas to set gill nets.

#### Crab Traps

#### **Gear Requirements**

- **Trap Size/Design:** May not exceed 18 cubic feet. Traps are constructed of #20 gauge coated aluminum wire for protection against corrosion. Square construction panels with two openings and a bait containing section. The trap lid should be constructed of a degradable panel by affixing untreated twine or small wire.
- **Related Equipment:** One (1) floating buoy, not less than 6 inches in length, width, and height must be attached with poly or nylon rope to the crab trap. All crab traps must be identified by an identification tag affixed to the line attaching the buoy to the crab trap or identification written on the buoy surface. Wording on the buoy and/or tag must be as follows:

#### Texas Department of State Health Services Seafood and Aquatic Life Group Scientific Permit Number SPR-0890-247

- **Bait:** Bait for crab traps must be fresh/frozen and may be caught from additional sampling activities such as gill nets or other acceptable methods. All left over bait must be properly discarded or frozen.
- **Trap Storage:** Crab traps must be washed after use with potable water and air-dried. Crab traps must be stored inside a covered area or shed to ensure the integrity of the trap.
- Sample Activities: Crab traps must be placed with the openings for entrapment portion toward the bottom with approximately 2 4 ft of water covering the trap. Set the traps in shallow waters during the summer and at deeper depths in winter for best results. As a precaution, do not place traps within 200 yards of a navigation channel. This will prevent possible damage and/or loss of equipment caused by commercial vessel traffic.

#### Sample Collection Precautions

A reasonable effort must be made to minimize sample handling and to avoid potential sources of contamination (e.g. sample cross contamination, grease, and/or gasoline contamination from sampling equipment).

## 6.0 Sample Collection Data Requirements & Documentation

All field collection data must be recorded on the *DSHS SALG Fish and Shellfish Tissue Collection Data Form* (Appendix 2). A different form must be used for each sample site and/or date of collection. Follow all instructions described in Table 3; Table 4; and Table 5 for recording data.

Data Type	Documentation Instruction
Water body	Record name of water body sampled. (e.g. Fosdic Lake; Trinity River)
Site Name	Record geographic reference description of selected sample site. (e.g. 2 miles south of Highway 887 bridge, State Land Tract 225, or Fish Point.
Site Code Record using numeric code to identify sample sites within a water body. (e.g. 01, 02,03)	
Date	Record two digits for month, day, and year, respectively (e.g. 08/23/03)
Time	Record using a 24-hour clock (e.g. 1500 = 3:00 p.m.)
GPS Coordinates	Record latitude and longitude to the nearest second or decimal equivalent
Weather (Wth)	Record weather conditions using the corresponding representative weather condition code: Clear (CL), Partly Cloudy (PC), Cloudy (CY), Fog (FG), Rain (RN), and Hazy (HZ)
Wind Direction (Dir)	Record wind direction: N, S, E, W, NE, NW, SE, or SW
Wind Speed	Record estimated wind speed in miles per hour.
Air Temperature	Record air temperature to the nearest degree °C
Water Temperature	Record water temperature to the nearest degree °C
Salinity	Record salinity to the nearest tenth (00.0) in parts per thousand (ppt)
рН	Record ph in standard pH units (1-14) to the nearest tenth (00.0)
Specific Conductance	Record specific conductance in micromhos to the nearest tenth (00000.0)
Collector(s)	Record names or initials of the fish or shellfish tissue sample collectors
Hydrologic Conditions	Tidal Movement: record tidal movement as incoming tide, outgoing tide, or slack tide.         http://www.srh.noaa.gov/hgx/marine.htm         River Flow: record river flow in cubic feet per second (cfs).         http://waterdata.usgs.gov/tx/nwis/sw         Reservoir Level: record current reservoir elevation level in feet mean sea level (msl).         http://waterdata.usgs.gov/tx/nwis/sw         or http://waterdata.usgs.gov/tx/nwis/sw
Observations	Record any pertinent observations (i.e. environmental or weather related abnormalities; fish abnormalities including deformities, wounds, or infections, etc)

 Table. 3 Sample Site Data Requirements

## Table 4. TCEQ Surface Water Quality Monitoring Site Field Descriptions

Data Type	Documentation Instruction
TCEQ Region <sup>1</sup>	Record TCEQ region code. (The TCEQ region in which a sample site is located)
TCEQ Station <sup>1</sup>	Record TCEQ station id. (Unique identifier assigned by TCEQ to a sampling station)
TCEQ Segment <sup>1</sup>	Record TCEQ segment id. (A code assigned to a classified stream segment)
TCEQ Sequence <sup>1</sup>	Record TCEQ stream sequence no. (A code assigned by TCEQ to a sampling site, placing it in sequence with all other sampling sites in a river basin)

<sup>1</sup> Only required for TCEQ grant funded projects.

## Table 5. Fish and Shellfish Tissue Sample Data Requirements

Data Type	Documentation Instruction
Sample Identification	Record sample number(s) using a three (3) letter number code for identifying fish or shellfish tissue samples (e.g. JPR-1 = Joe Pool Reservoir sample number 1). Code letters may be found in <i>Water body Codes for Texas Public Waters</i> (Appendix 3). All samples should be numbered sequentially in order of processing. If a new reservoir is sampled create a three (3)-letter water body code and update <i>Water body Codes for Texas Public Waters</i> .
Sample Date Collected	Record date fish or shellfish tissue sample was collected.
Sample Date Processed	Record date fish or shellfish tissue sample was processed.
Tissue Analysis	Record type(s) of chemical analysis performed for each fish or shellfish tissue sample. (e.g. Metals = As, Cd, Cu, Hg, Pb, Se, and Zn; Pesticides; Polychlorinated Biphenyls (PCBs); Semi-volatile organic compounds (SVOCs); Volatile organic compounds (VOCs); and Dioxins/Furans. Full Scan = Metals; Pesticides; PCBs; Dioxins/ Furans; SVOCs; and VOCs)
Gear Type	Record method of collection (i.e. electrofishing = (ES); gill net (GN); crab trap (CT); hook & line (HL); and trot line (TL)
EPA Species Code	Record the three (3)-digit EPA species code. Species codes may be found in the DSHS and EPA Species Code List (Appendix 4).
DSHS Species Code	Record three (3) letter species code. Species Codes may be found in the <i>DSHS and EPA Species Code List</i> (Appendix 4).
Number	Record number of fish or shellfish in tissue sample.
Composite Sample Average Length & Weight	Record the length in millimeters (mm) and weight in grams (g) for each sample included in the composite sample.
Length	Measure and record total length in millimeters (mm) for each fish tissue sample.
Weight	Weigh and record the weight in grams (g) for each fish tissue sample.

#### 7.0 Sample Processing, Handling, and Storage Procedures

#### Fish Fillet Sample Processing, Handling, and Storage

#### **Processing Equipment Requirements**

- Large plastic cutting board
- Heavy duty aluminum foil
- (1) fillet knives
- De-ionized water
- (2) steel fillet gloves
- Ziploc® freezer bags
- Large heavy duty trash bags
- Paper towels or tech wipes

#### **Data Requirements**

- Measure each fish and record length in millimeters (mm).
- Weigh each fish and record weight in grams (g).
- Record any unusual deformities, wounds, or infections observed.
- **Optional** remove otoliths from fish samples for age analysis.

#### **Sample Container Labeling Requirements**

• The sample containers (i.e. Ziploc® freezer bags) must be labeled with a waterproof marker including date, sample number, sample length, sample weight, and collection location (if not included as part of the sample number).

#### **Fillet Processing Instructions**

A fish fillet is defined as a longitudinal slice of de-boned, skin-off fish muscle tissue originating from the mid-dorsal line of the fish. The fillet tissue sample may be removed from either side of the fish. The belly muscle and any dark muscle tissue should not be separated from the light muscle tissue. Bones still present in the muscle tissue after filleting must be carefully removed. For large fish samples, a posterior and anterior portion of the fillet must be collected. For small fish samples the right and left side fillets may be combined to meet the tissue sample size required by the DSHS Laboratory or designated contract laboratory ( $\geq 200$  g for full scan analysis and  $\geq 50$  g for mercury only analysis). When right and left side fillets will not meet the required fish tissue sample size, a composite sample composed of the same species and similar size specimens—the smallest specimen in the composite must be at least 75% of the total length of the largest specimen in the composite sample— may be used as a fish tissue sample. If a sufficient amount of fish tissue is obtained from the right or left side of the fish sample, the opposite side may be retained as a duplicate or backup tissue sample. All fish tissue samples must be filleted with the skin removed unless consumption patterns indicate that a species is eaten with the skin on, in which case the skin will be left on the fillet. If the skin is left on the fillet, the scales must be removed by scraping the scales from the skin with the edge of

a knife.

Samples must be filleted on a plastic cutting board covered with heavy-duty aluminum foil. Aluminum foil must be replaced between each sample to prevent sample cross-contamination. Care must be exercised to avoid contamination from inadvertent puncture of the internal organs. If the fillet is contaminated with materials released from puncture of the internal organs, the fillet may be eliminated as a sample specimen or must be rinsed with deionized water and blotted dry with a clean, unused paper towel. A notation must be recorded on the data form regarding this procedure. The fillet knife must be rinsed with de-ionized water between each fish sample. For composite samples, the same foil may be used until preparation of all specimens in the composite is complete or the foil is damaged. All samples (individual or composite) must be double wrapped with heavy-duty aluminum foil and placed in an appropriately labeled Ziploc® plastic freezer bag. Standard Ziploc® plastic bags without a zipper mechanism must be used for all fish tissue samples. Do not use masking tape or wrapping tape, since the tissue sample may be contaminated if the foil or bag is damaged. Do not use the easy zip style plastic freezer bags with the sliding zipper mechanism to store an individual or composite fish tissue sample, as these bags do not seal completely and may allow leakage, possibly contaminating the fish tissue sample. Following proper packaging of the fish tissue sample, the sample must be immediately placed on wet ice and transferred to the SALG freezer as soon as possible. The freezer must be locked to maintain chain-of-custody requirements. Fish tissue samples must remain frozen until delivered to the designated laboratory.

#### Crab Sample Processing, Handling, and Storage

- The total width of the carapace shall be measured from the tip of one lateral spine to the tip of the opposite lateral spine and recorded to the nearest millimeter (mm).
- The crabs must be "backed" (by pulling away the carapace), and the internal organs must be removed.
- Crab samples must be double wrapped with aluminum foil and placed in a Ziploc® plastic bag that has been labeled following the directions for sample container labeling outlined in *Fish Fillet Sample Processing, Handling, and Storage*.
- Sealed bags must be immediately placed on wet ice and transferred to the SALG freezer as soon as possible. The freezer must be locked to maintain chain-of-custody requirements. Crab samples must remain frozen until delivered to the designated laboratory.

#### Shrimp, Crawfish, and Prawn Sample Processing, Handling, and Storage

- The weight to the nearest gram, the number of specimens in the sample and the estimated size (count per pound) must be recorded.
- The entire shrimp, crawfish, or prawn must be used for the sample.
- Samples must be double wrapped with aluminum foil and placed in a Ziploc® plastic bag that has been labeled following the directions for sample container labeling outlined in *Fish Fillet Sample Processing, Handling, and Storage*.
- Sealed bags must be immediately placed on wet ice and transferred to the SALG freezer as soon as possible. The freezer must be locked to maintain chain-of-

custody requirements. Samples must remain frozen until delivered to the designated laboratory.

#### Oyster, Clam, and Mussel Sample Processing, Handling, and Storage

- Shells must be opened and the meat cut loose and dropped directly into a glass jar. The sample must include the "liquor" that is inside the shell.
- All samples must be placed in glass jars, which have been rinsed with distilled water and allowed to air dry. Lids must have Teflon liners or be lined with aluminum foil.
- The number of shellfish in the composite sample and the average shell length must be calculated and recorded.
- The glass jar must be labeled with a waterproof marker including date, sample number, and analyses requested.
- Sealed jar(s) must be immediately placed on wet ice and transferred to the SALG freezer as soon as possible. The freezer must be locked to maintain chain-of-custody requirements. Samples must remain frozen until delivered to the designated laboratory.

## 8.0 Tissue Sample Holding Times

Analyte	Matrix	Preservation	Holding Time <sup>1</sup>
Mercury	Tissue (fillets and edible portions)	Freeze at $\leq 20 ^{\circ}\text{C}$	28 days
Other metals	Tissue (Fillets and edible portions	Freeze at $\leq 20 ^{\circ}\text{C}$	6 months
Organics	Tissue (Fillets and edible portions	Freeze at $\leq 20 ^{\circ}\text{C}$	1 year
Lipids	Tissue (Fillets and edible portions	Freeze at $\leq 20 ^{\circ}\text{C}$	1 year

Table 6. Holding Times for Fish and Shellfish Tissues<sup>1</sup>

<sup>1</sup>Maximum holding times recommended by the EPA (1995i)

## 9.0 Chain-of-Custody and Tissue Sample Shipping Procedures

#### Chain-of-Custody Procedures

The DSHS SALG Chain-of-Custody Record Form (Appendix 5) must be used to provide SALG with information about physical control of the tissue samples, from collection until arrival at the laboratory. The Chain-of-Custody Record Form must be used for all tissue samples, with the exceptions noted below. Follow all instructions described in Table 7 for completing the DSHS SALG Chain-of-Custody Record Form.

Table 7.	Chain-of-Custody	v Record Form	Instructions
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	Documentation Instruction
Account Number	Record purchase order number or account number assigned by the DSHS or contract laboratory for the requested tissue analyses.
Project Contract Number	Record project contract number established for project.
Tissue Sample Collection Date	Record tissue collection date. (two digits for month, day, and year, respectively (Ex. 08/23/03)).
Water body	Record name of water body sampled.
Deliver/Ship To	Record name of contract laboratory.
Relinquished by	Record agency/company name individual is employed by, print name, sign name, and record date and time (24-hour clock) tissue samples are relinquished to the shipper.
Accepted by Shipper	Record agency/company name individual is employed by, print name, sign name, and record date and time (24-hour clock) tissue samples are accepted by the shipper.
Accepted by Laboratory	Record agency/company name individual is employed by, print name, sign name, and record date and time (24-hour clock) tissue samples are accepted by the laboratory.
Sample Identification	Record sample number using a three (3) letter number code (defined in Table 5). Record species name or DSHS species code for the tissue sample.
Chemical Analysis	Check the appropriate square for the requested analysis: pesticides, PCBs, SVOCs, VOCs, and Dioxins and circle the metal(s) requested for analyses.

- All tissue samples must be stored in a secure (locked) SALG freezer until shipment or delivery to the designated laboratory. If possible, the tissue samples must remain in SALG personnel custody until relinquished to the laboratory.
- Samples submitted to laboratories must be logged in and tracked by the DSHS Chain-of-Custody Record Form (Appendix 5). The DSHS or designated laboratory assigns an account number to identify a group of tissue samples or single project. The account number must be set up prior to the delivery of the tissue samples.
- (Optional) Samples submitted to the DSHS Laboratory or designated contract laboratory by SALG personnel under contract with the Texas Commission on Environmental Quality (TCEQ) may be logged in using the *TCEQ Surface Water Quality Monitoring Program LPS Form*. This form may serve as the chain-of-custody record for TCEQ-contracted tissue samples submitted to the DSHS Laboratory or contract laboratory. This form also identifies the tissue sample until analyses and computations are completed. The DSHS or designated laboratory assigns an account number to identify a group of tissue samples or single project. The account number must be set up prior to the delivery of the tissue samples.

#### Tissue Sample Shipping Procedures

- The sample collector(s), or other designated SALG personnel, are responsible for preparing tissue sample shipments and ensuring that all chain-of-custody requirements are followed. The following procedures must be followed for shipping all tissue samples:
- The *DSHS SALG Chain-of-Custody Record Form* must be completed as described in Table 7. This form or other authorized forms must also accompany the tissue sample shipment until the contract laboratory receives the shipment and the samples are logged in by the contract laboratory.
- Prior to delivery, the contract laboratory must be contacted and notified of the expected tissue sample shipment arrival time.
- The contract laboratory must receive samples within 24 hours of shipment.
- To ensure tissue samples remain frozen during shipment, ice chests-used for shipping- may be lined with bubble wrap and packed with cold packs or wet ice. Ice chests must be sealed with heavy duty packaging tape to prevent tampering.
- The shipping company-receiving agent must sign the chain-of-custody form and record the date and time of taking possession of the shipment.
- When the samples arrive at the contract laboratory, the laboratory-receiving agent must sign the chain-of-custody form, record the date and time of sample receipt, and notify the designated SALG contact by email of the tissue sample arrival time. The signed chain-of-custody form must be faxed or mailed to the Seafood and Aquatic Life Group (512-834-6762) and filed in the project file.

## 10.0 Data Management and Analysis

#### <u>Data Management</u>

- A working file must be developed for each project (i.e. water body). The working file must be labeled by water body name, DSHS and/or contract laboratory account number, and grant title (grant title labeling applies only if the project is grant funded). This file must contain a project contract, a signed quality assurance project plan and associated documents (only for grant funded projects), project reports, project correspondence, field and laboratory data, and a final quantitative risk characterization.
- Field data recorded on the *DSHS SALG Fish and Shellfish Tissue Collection Data Form* (Appendix 2) and fish and shellfish tissue laboratory analysis report data (i.e. individual fish and shellfish tissue analysis reports are received from the designated laboratory by analysis type: metals, pesticides, PCBs, SVOCs, VOCs, and dioxins/furans) must be entered into a Microsoft Excel template worksheet for each chemical contaminant group (i.e. metals, pesticides, PCBs, SVOCs, VOCs, and dioxins/furans) [5]. The Microsoft Excel file must be

saved following the standard file name format *water body name and tissue collection year* (e.g. Welsh Reservoir 2003).

### Data Quality Control / Quality Assurance

 All field and laboratory report data entered must be verified for accuracy by the assigned DSHS SALG QA officer. The date and initials of the DSHS SALG staff completing each QC / QA process must be recorded on the *DSHS SALG Data Review Form* (Appendix 6).
 Follow all instructions described in Table 8 for recording the proper information in the data review form.

QC / QA Process	Documentation Instruction
Data Entered	Record two digits for month, day, and year, respectively (e.g. 08/23/03) and initials of DSHS SALG staff who completed the data entry.
Data Reviewed	Record two digits for month, day, and year, respectively (e.g. 08/23/03) and initials of DSHS SALG QA Officer.
Data Corrected	Record two digits for month, day, and year, respectively (e.g. 08/23/03) and initials of DSHS SALG staff who completed the data entry.
Corrections Verified	Record two digits for month, day, and year, respectively (e.g. 08/23/03) and initials of DSHS SALG QA Officer.

#### Table 8. Data Review Form Requirements

### Data Analysis

- Statistical procedures must be performed on IBM-compatible microcomputer using SPSS software [6]. The following descriptive statistics must be generated by water body for each detected chemical contaminant in each species at each sampling site: mean concentration, standard deviation, median, range, and minimum and maximum concentrations.
- Additional statistical analysis may be performed if needed.
- Analyzed data or raw data must be transferred to the DSHS SALG toxicologist or SALG risk assessor for health risk computations and the development of a quantitative risk characterization.

## **11.0 DSHS Project Tracking**

#### Fish or Shellfish Tissue Sample Number Reference List

• For each fish or shellfish tissue sample, DSHS SALG assigns a unique sample identification number (The DSHS SALG sample identification number is defined in Table 5. Fish and Shellfish Tissue Sample Data Requirements). The DSHS Laboratory or designated contract laboratory may also assign a unique identification number to each tissue sample. Both of these sample numbers must be recorded on the *DSHS SALG Tissue Sample Number Reference List* (Microsoft Excel electronic form) as DSHS Laboratory or DSHS SALG receives

designated contract laboratory reports. This list is used to accurately identify fish or shellfish tissue samples and facilitate communication regarding individual tissue samples between DSHS SALG and the Laboratory.

#### **Project Tracking Form**

• Project status must be tracked by water body from the date fish and shellfish tissue collection is completed to completion of the quantitative risk characterization. Project tracking information must be recorded on the *DSHS SALG Project Tracking Form* (Microsoft Excel electronic form). Follow all instructions described in Table 9 for recording the proper information on the project tracking form.

Tracking Parameter	Documentation Instruction				
Project	Record water body name (e.g. Joe Pool Reservoir).				
Funding Source	Record name of funding source (i.e. state agency, federal agency, or municipality).				
Laboratory Acct. No.	Record account number assigned by designated laboratory.				
Number of Samples	Record number of fish and/or shellfish samples.				
Analysis	Record all analyses performed for the project (e.g. metals, pesticides).				
Sampling ConductedRecord date or dates, that fish and/or shellfish collection is conducted.					
Sampling Status	Record the date that fish and/ or shellfish collection is complete.				
Laboratory Analysis Status	Record the date that Laboratory analysis data are received from the designated laboratory.				
<b>Contaminants Detected</b>	Record all chemical contaminant types detected (e.g. Hg, DDT, PCBs).				
SALG Database Entry	Record the date that data entry is complete.				
SALG Database QA/QC	Record the date that QA/QC processes are complete.				
Data to SALG Toxicologist / Risk Assessor	Record the date that field and laboratory data are transferred to the SALG Toxicologist or Risk Assessor				
TRACS Data to TCEQ	Record the date that TRACS formatted data is submitted to TCEQ. Record NA if not applicable.				
Risk Characterization Status	Record the date that the risk characterization is complete.				
Miles Surveyed	Record the water body square miles surveyed.				

#### Table 9. Project Tracking Form Requirements

#### 12.0 Laboratory Fees

The following fees are effective as of February 2007 for fish and shellfish tissue analysis conducted by the Texas A&M University Geochemical Environmental Research Group (GERG) Laboratory.

Procedure Description	Fee
Metals, Fish Tissue Panel (includes mercury)	\$160.00
Mercury (includes digestion fee)	\$53.00
Organochlorine Pesticides and PCBs, Fillet	\$785.00
Semi-Volatile Organics	\$400.00
Volatiles Organics	\$325.00
Dioxins/Furans	\$625.00

#### 2007 GERG Fish and Shellfish Tissue Analysis Fee Schedule

## 13.0 Laboratory Quality Assurance / Quality Control

The GERG Laboratory has developed *Quality Assurance Plans* (Appendix 7) that are followed by the GERG Laboratory to document the use of consistent analytical methodology to ensure accuracy and precision of data analysis. Contract laboratories must provide SALG with a similar quality assurance / quality control plan.

### References

- [USEPA] U.S. Environmental Protection Agency. Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories. Vol. 1, Fish Sampling and Analysis, 3<sup>rd</sup> ed. Washington, D.C.: 2000.
- 2. [USEPA] United States Environmental Protection Agency. Water Quality Criterion for the protection of Human Health: Methylmercury. EPA-823-R-01-001, January 2001. Office of Science and Technology, Office of Water. Washington, DC 20460. Chapter 5, pp 61-64. www.epa.gov/waterscience/criteria/methylmercury/merctitl.pdf. (Accessed February 9, 2007).
- 3. Texas Statutes: Health and Safety, Chapter 436, Subchapter D, § 436.011, §436.061 and others.
- 4. [TPWD] Texas Parks and Wildlife Department. 2006-2007 Outdoor Annual: Hunting and Fishing Regulations. Ed. J. Jefferson. Texas Monthly Custom Publishing. 2006.
- 5. SPSS<sup>®</sup> Base 10.0 ©SPSS Inc, 1999. Information available at URL: <u>http://www.spss.com</u>
- 6. Microsoft Corporation. Microsoft Excel<sup>®</sup>2000. Copyright<sup>®</sup> Microsoft Corporation 1985-1999.

# Appendix 1

Target Analyte List

## <u>Texas Department of State Health Services Seafood and Aquatic Life Group</u> <u>Fish and Shellfish Tissue Target Analyte List</u>

All chemical contaminant concentrations must be reported in **wet weight**. The laboratory methods and laboratory techniques listed are the current GERG methods and techniques used by the GERG Laboratory for metals, pesticides, polychlorinated biphenyls (PCBs), dioxins and furans, semi-volatile organic compounds (SVOCs), and volatile organic compounds (VOCs).

#### **Additional Required Parameters**

Analyte	Reporting Limit <sup>1</sup>	Extraction/Purification	Instrument Analysis	Technique
% Lipid	$NA^2$	GERG 9807	GERG 9727	NA
% Moisture	NA	NA	GERG 9415	NA

#### **Metals** (mg/kg = parts per million)

Analyte	CASRN	Reporting Limit <sup>1</sup>	Extraction/Purification	Instrument Analysis	Technique
Arsenic	7440-38-2	0.10	GERG 9408	GERG 0201	GFAAS
Cadmium	7440-43-9	0.10	GERG 9408	EPA 6020	ICP-MS
Copper	7440-50-8	0.40	GERG 9408	EPA 6020	ICP-MS
Lead	7439-92-1	0.40	GERG 9408	EPA 6020	ICP-MS
Mercury	7439-97-6	0.20	GERG 0006	GERG 0202	CVAAS
Selenium	7782-79-2	0.10	GERG 9408	GERG 0201	GFAAS
Zinc	7440-66-6	0.40	GERG 9408	EPA 6020	ICP-MS

**Pesticides** (Fg/kg = parts per billion)

Analyte	CASRN	Reporting Limit <sup>1</sup>	Extraction/Purification	Instrument Analysis	Technique
Tetrachlorobenzene 1,2,4,5	95-94-3	2.0	GERG 9807, 9720, 0009	GERG 9810	GC-ECD
Tetrachlorobenzene 1,2,3,4	634-66-2	2.0	GERG 9807, 9720, 0009	GERG 9810	GC-ECD
Pentachlorobenzene	608-93-5	2.0	GERG 9807, 9720, 0009	GERG 9810	GC-ECD
Hexachlorobenzene	118-74-1	2.0	GERG 9807, 9720, 0009	GERG 9810	GC-ECD
Alpha HCH	319-84-6	2.0	GERG 9807, 9720, 0009	GERG 9810	GC-ECD
Beta HCH	319-85-7	2.0	GERG 9807, 9720, 0009	GERG 9810	GC-ECD
Gamma HCH (Lindane)	58-89-9	2.0	GERG 9807, 9720, 0009	GERG 9810	GC-ECD
Delta HCH	319-86-8	2.0	GERG 9807, 9720, 0009	GERG 9810	GC-ECD

Analyte	CASRN	Reporting Limit <sup>1</sup>	Extraction/Purification	Instrument Analysis	Technique
Heptachlor	76-44-8	2.0	GERG 9807, 9720, 0009	GERG 9810	GC-ECD
Heptachlor Epoxide	1024-57-3	4.0	GERG 9807, 9720, 0009	GERG 9810	GC-ECD
Chlordane (Total) <sup>3</sup>	NA	10	GERG 9807, 9720, 0009	GERG 9810	GC-ECD
Aldrin	309-00-2	2.0	GERG 9807, 9720, 0009	GERG 9810	GC-ECD
Dieldrin	60-57-1	6.0	GERG 9807, 9720, 0009	GERG 9810	GC-ECD
Endrin	72-20-8	6.0	GERG 9807, 9720, 0009	GERG 9810	GC-ECD
Pentachloroanisole	1825-21-4	10	GERG 9807, 9720, 0009	GERG 9810	GC-ECD
Chlorpyrifos	2921-88-2	10	GERG 9807, 9720, 0009	GERG 9810	GC-ECD
Mirex	2385-85-5	8.0	GERG 9807, 9720, 0009	GERG 9810	GC-ECD
2,4' DDE	3424-82-6	10	GERG 9807, 9720, 0009	GERG 9810	GC-ECD
4,4' DDE	72-55-9	10	GERG 9807, 9720, 0009	GERG 9810	GC-ECD
2,4' DDD	53-19-0	10	GERG 9807, 9720, 0009	GERG 9810	GC-ECD
4,4' DDD	72-54-8	10	GERG 9807, 9720, 0009	GERG 9810	GC-ECD
2,4' DDT	789-02-6	10	GERG 9807, 9720, 0009	GERG 9810	GC-ECD
4,4' DDT	50-29-3	10	GERG 9807, 9720, 0009	GERG 9810	GC-ECD
Diazinon	333-41-5	10	GERG 9807, 9720, 0009	GERG 9810	GC-ECD
Alachlor	15972-60-8	8.0	GERG 9807, 9720, 0009	GERG 9810	GC-ECD
Methyl parathion	298-00-0	10	GERG 9807, 9720, 0009	GERG 9810	GC-ECD
Malathion	121-75-5	20	GERG 9807, 9720, 0009	GERG 9810	GC-ECD
Dacthal	1861-32-1	3.0	GERG 9807, 9720, 0009	GERG 9810	GC-ECD
Ethyl Parathion	56-38-2	10	GERG 9807, 9720, 0009	GERG 9810	GC-ECD
Endosulfan I	959-98-8	10	GERG 9807, 9720, 0009	GERG 9810	GC-ECD
Endosulfan II	33213-65-9	10	GERG 9807, 9720, 0009	GERG 9810	GC-ECD
Endosulfan Sulfate	1031-07-8	10	GERG 9807, 9720, 0009	GERG 9810	GC-ECD
Methoxychlor	72-43-5	30	GERG 9807, 9720, 0009	GERG 9810	GC-ECD
Toxaphene	8001-35-2	100	GERG 9807, 9720, 0009	GERG 9810	GC-ECD

## **Polychlorinated Biphenyls** (analyzed at $Fg/kg = parts per billion)^4$

Analyte	CASRN	Reporting Limit <sup>1</sup>	Extraction/Purification	Instrument Analysis	Technique
Aroclor 1016**	12674-11-2	40	GERG 9807, 9720, 0009	GERG 9810	GC-ECD
Aroclor 1221**	11104-28-2	40	GERG 9807, 9720, 0009	GERG 9810	GC-ECD
Aroclor 1232**	11141-16-5	40	GERG 9807, 9720, 0009	GERG 9810	GC-ECD
Aroclor 1242**	53469-21-9	40	GERG 9807, 9720, 0009	GERG 9810	GC-ECD
Aroclor 1248**	12672-29-6	40	GERG 9807, 9720, 0009	GERG 9810	GC-ECD
Aroclor 1254**	11097-69-1	40	GERG 9807, 9720, 0009	GERG 9810	GC-ECD
Aroclor 1260**	11096-82-5	40	GERG 9807, 9720, 0009	GERG 9810	GC-ECD
Aroclor 1268**	11100-14-4	40	GERG 9807, 9720, 0009	GERG 9810	GC-ECD

\*\* Aroclor is a regis tered trademark of the Monsanto Corporation

Analyte	CASRN	Reporting Limit <sup>1</sup>	Extraction/Purification	Instrument Analysis	Technique
PCB 1	2051-60-7	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 2	2051-61-8	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 3	2051-62-9	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 4	13029-08-8	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 5	16605-91-7	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 6	25569-80-6	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 7	33284-50-3	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 8	34883-43-7	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 9	34883-39-1	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 10	33146-45-1	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 11	2050-67-1	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 12	2974-92-7	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 13	2974-90-5	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 14	34883-41-5	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 15	2050-68-2	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 16	38444-78-9	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 17	37680-66-3	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 18	37680-65-2	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 19	38444-73-4	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS

Analyte	CASRN	Reporting Limit <sup>1</sup>	Extraction/Purification	Instrument Analysis	Technique
PCB 20	38444-84-7	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 21	55702-46-0	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 22	38444-85-8	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 23	55720-44-0	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 24	55702-45-9	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 25	55712-37-3	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 26	38444-81-4	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 27	38444-76-7	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 28	7012-37-5	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 29	15862-07-4	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 30	35693-92-6	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 31	16606-02-3	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 32	38444-77-8	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 33	38444-86-9	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 34	37680-68-5	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 35	37680-69-6	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 36	38444-87-0	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 37	38444-90-5	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 38	53555-66-1	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 39	38444-88-1	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 40	38444-93-8	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 41	52663-59-9	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 42	36559-22-5	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 43	70362-46-8	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 44	41464-39-5	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 45	70362-45-7	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 46	41464-47-5	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 47	2437-79-8	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 48	70362-47-9	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 49	41464-40-8	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS

Analyte	CASRN	Reporting Limit <sup>1</sup>	Extraction/Purification	Instrument Analysis	Technique
PCB 50	62796-65-0	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 51	68194-04-7	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 52	35693-99-3	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 53	41464-41-9	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 54	15968-05-5	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 55	74338-24-2	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 56	41464-43-1	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 57	70424-67-8	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 58	41464-49-7	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 59	74472-33-6	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 60	33025-41-1	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 61	33284-53-6	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 62	54230-22-7	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 63	74472-34-7	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 64	52663-58-8	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 65	33284-54-7	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 66	32598-10-0	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 67	73575-53-8	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 68	73575-52-7	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 69	60233-24-1	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 70	32598-11-1	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 71	41464-46-4	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 72	41464-42-0	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 73	74338-23-1	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 74	32690-93-0	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 75	32598-12-2	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 76	70362-48-0	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 77	32598-13-3	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 78	70362-49-1	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 79	41464-48-6	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS

Analyte	CASRN	Reporting Limit <sup>1</sup>	Extraction/Purification	Instrument Analysis	Technique
PCB 80	33284-52-5	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 81	70362-50-4	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 82	52663-62-4	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 83	60145-20-2	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 84	52663-60-2	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 85	65510-45-4	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 86	55312-69-1	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 87	38380-02-8	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 88	55215-17-3	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 89	73575-57-2	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 90	68194-07-0	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 91	68194-05-8	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 92	52663-61-3	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 93	73575-56-1	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 94	73575-55-0	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 95	38379-99-6	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 96	73575-54-9	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 97	41464-51-1	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 98	60233-25-2	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 99	38380-01-7	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 100	39485-83-1	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 101	37680-73-2	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 102	68194-06-9	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 103	60145-21-3	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 104	56558-16-8	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 105	32598-14-4	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 106	70424-69-0	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 107	70424-68-9	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 108	70362-41-3	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 109	74472-35-8	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS

Analyte	CASRN	Reporting Limit <sup>1</sup>	Extraction/Purification	Instrument Analysis	Technique	
PCB 110	38380-03-9	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 111	39635-32-0	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 112	74472-36-9	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 113	68194-10-5	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 114	74472-37-0	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 115	74472-38-1	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 116	18259-05-7	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 117	68194-11-6	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 118	31508-00-6	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 119	56558-17-9	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 120	68194-12-7	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 121	56558-18-0	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 122	76842-07-4	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 123	65510-44-3	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 124	70424-70-3	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 125	74472-39-2	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 126	57465-28-8	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 127	39635-33-1	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 128	38380-07-3	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 129	55215-18-4	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 130	52663-66-8	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 131	61798-70-7	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 132	38380-05-1	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 133	35694-04-3	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 134	52704-70-8	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 135	52744-13-5	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 136	38411-22-2	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 137	35694-06-5	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 138	35065-28-2	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 139	56030-56-9	1.0	GERG 9807, 9720, 0009	GERG 0205 HRGC/LRM		

Analyte	CASRN	Reporting Limit <sup>1</sup>	Extraction/Purification	Instrument Analysis	Technique	
PCB 140	59291-64-4	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 141	52712-04-6	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 142	41411-61-4	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 143	68194-15-0	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 144	68194-14-9	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 145	74472-40-5	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 146	51908-16-8	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 147	68194-13-8	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 148	74472-41-6	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 149	38380-04-0	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 150	68194-08-1	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 151	52663-63-5	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 152	68194-09-2	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 153	35065-27-1	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 154	60145-22-4	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 155	33979-03-2	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 156	38380-08-4	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 157	69782-90-7	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 158	74472-42-7	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 159	39635-35-3	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 160	41411-62-5	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 161	74472-43-8	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 162	39635-34-2	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 163	74472-44-9	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 164	74472-45-0	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 165	74472-46-1	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 166	41411-63-6	1.0	GERG 9807, 9720, 0009	GERG 0205 HRGC/LRM		
PCB 167	52663-72-6	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 168	59291-65-5	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 169	32774-16-6	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	

Analyte	CASRN	Reporting Limit <sup>1</sup>	Extraction/Purification	action/Purification Instrument Analysis	
PCB 170	35065-30-6	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 171	52663-71-5	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 172	52663-74-8	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 173	68194-16-1	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 174	38411-25-5	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 175	40186-70-7	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 176	52663-65-7	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 177	52663-70-4	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 178	52663-67-9	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 179	52663-64-6	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 180	35065-29-3	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 181	74472-47-2	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 182	60145-23-5	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 183	52663-69-1	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 184	74472-48-3	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 185	52712-05-7	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 186	74472-49-4	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 187	52663-68-0	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 188	74487-85-7	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 189	39635-31-9	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 190	41411-64-7	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 191	74472-50-7	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 192	74472-51-8	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 193	69782-91-8	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 194	35694-08-7	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 195	52663-78-2	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 196	42740-50-1	1.0	GERG 9807, 9720, 0009	GERG 0205 HRGC/LRM	
PCB 197	33091-17-7	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 198	68194-17-2	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS
PCB 199	52663-75-9	1.0	GERG 9807, 9720, 0009	GERG 0205 HRGC/LRM	

Analyte	CASRN	Reporting Limit <sup>1</sup>	Extraction/Purification	Instrument Analysis	Technique	
PCB 200	52663-73-7	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 201	40186-71-8	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 202	2136-99-4	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 203	52663-76-0	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 204	74472-52-9	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 205	74472-53-0	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 206	40186-72-9	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 207	52663-79-3	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 208	52663-77-1	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	
PCB 209	2051-24-3	1.0	GERG 9807, 9720, 0009	GERG 0205	HRGC/LRMS	

## **Volatile Organic Compounds** (analyzed in Fg/kg = parts per billion)

Analyte	CASRN	Reporting Limit <sup>1</sup>	Extraction/ Purification	Instrument Analysis	Technique
Chloromethane	74-87-3	50	NA	GERG 0301	HRGC/LRMS
Vinyl Chloride	75-01-4	50	NA	GERG 0301	HRGC/LRMS
Bromomethane	74-83-9	50	NA	GERG 0301	HRGC/LRMS
Chloroethane	75-00-3	50	NA	GERG 0301	HRGC/LRMS
1,1-Dichloroethene	75.35-4	20	NA	GERG 0301	HRGC/LRMS
Acetone	67-64-1	200	NA	GERG 0301	HRGC/LRMS
Iodomethane	74-88-4	50	NA	GERG 0301	HRGC/LRMS
Carbon Disulfide	75-15-0	50	NA	GERG 0301	HRGC/LRMS
Methylene Chloride	75-09-2	50	NA	GERG 0301	HRGC/LRMS
Trans-1,2-Dichloroethene	156-60-5	20	NA	GERG 0301	HRGC/LRMS
Acrylonitrile	107-13-1	20	NA	GERG 0301	HRGC/LRMS
Methyl-tert-butyl ether (MTBE)	1634-04-4	20	NA	GERG 0301	HRGC/LRMS
1,1-Dichloroethane	75-34-3	20	NA	GERG 0301	HRGC/LRMS
cis-1,2-Dichloroethene	156-59-2	20	NA	GERG 0301	HRGC/LRMS
2-Butanone (MEK)	78-93-3	100	NA	GERG 0301	HRGC/LRMS
2,2-Dichloropropane	590-20-7	20	NA	GERG 0301	HRGC/LRMS
Bromochloromethane	74-97-5	20	NA	GERG 0301	HRGC/LRMS
Analyte	CASRN	Reporting Limit <sup>1</sup>	Extraction/ Purification	Instrument Analysis	Technique
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Chloroform	67-66-3	20	NA	GERG 0301	HRGC/LRMS
1,2-Dichloroethane	107-06-2	20	NA	GERG 0301	HRGC/LRMS
Acrolein (Propenal)	107-02-8	20	NA	GERG 0301	HRGC/LRMS
Dibromomethane	74-95-3	20	NA	GERG 0301	HRGC/LRMS
Tetrahydrofuran	109-99-9	50	NA	GERG 0301	HRGC/LRMS
1,1,1-Trichloroethane	71-55-6	20	NA	GERG 0301	HRGC/LRMS
1,1-Dichloropropene	563-58-6	20	NA	GERG 0301	HRGC/LRMS
Carbon Tetrachloride	56-23-5	20	NA	GERG 0301	HRGC/LRMS
Benzene	71-43-2	20	NA	GERG 0301	HRGC/LRMS
Trichlorofluoromethane	75-69-4	50	NA	GERG 0301	HRGC/LRMS
Trichloroethene	79-01-6	20	NA	GERG 0301	HRGC/LRMS
1,2-Dichloropropane	78-87-5	20	NA	GERG 0301	HRGC/LRMS
Methyl Methacrylate	80-62-6	20	NA	GERG 0301	HRGC/LRMS
cis,-1,3-Dichloropropene	10061-01-5	100	NA	GERG 0301	HRGC/LRMS
trans-1,3-Dichloropropene	10061-02-6	100	NA	GERG 0301	HRGC/LRMS
Bromodichloromethane	75-27-4	20	NA	GERG 0301	HRGC/LRMS
1,1,2-Trichloroethane	79-00-5	20	NA	GERG 0301	HRGC/LRMS
Dichlorodifluoromethane	75-71-8	50	NA	GERG 0301	HRGC/LRMS
Ethyl Methacrylate	97-63-2	20	NA	GERG 0301	HRGC/LRMS
Dibromochloromethane	124-48-1	20	NA	GERG 0301	HRGC/LRMS
1,2-Dibromoethane	106-93-4	20	NA	GERG 0301	HRGC/LRMS
Bromoform	75-25-2	20	NA	GERG 0301	HRGC/LRMS
4-Methyl-2- Pentanone(MIBK)	108-10-1	20	NA	GERG 0301	HRGC/LRMS
Toluene	108-88-3	20	NA	GERG 0301	HRGC/LRMS
Tetrachloroethene	127-18-4	20	NA	GERG 0301	HRGC/LRMS
1,3-Dichloropropane	142-28-9	20	NA	GERG 0301	HRGC/LRMS
1,2,3-Trichloropropane	96-18-4	20	NA	GERG 0301	HRGC/LRMS
2-Hexanone	591-78-6	20	NA	GERG 0301	HRGC/LRMS
Chlorobenzene	108-90-7	20	NA	GERG 0301	HRGC/LRMS
1,1,1,2-Tetrachlorethane	930-20-6	20	NA	GERG 0301	HRGC/LRMS

Analyte	CASRN	Reporting Limit <sup>1</sup>	Extraction/ Purification	Instrument Analysis	Technique
Ethylbenzene	100-41-4	20	NA	GERG 0301	HRGC/LRMS
m-+p-Xylene	95-47-6	40	NA	GERG 0301	HRGC/LRMS
o-Xylene	100-42-5	20	NA	GERG 0301	HRGC/LRMS
Styrene	98-82-8	20	NA	GERG 0301	HRGC/LRMS
Isopropylbenzene	108-86-1	20	NA	GERG 0301	HRGC/LRMS
Bromobenzene	79-34-5	20	NA	GERG 0301	HRGC/LRMS
1,1,2,2-Tetrachloroethane	95-49-8	20	NA	GERG 0301	HRGC/LRMS
2-Chlorotoluene	106-43-4	20	NA	GERG 0301	HRGC/LRMS
4-Chlorotoluene	108-67-8	20	NA	GERG 0301	HRGC/LRMS
1,3,5-Trimethylbenzene	95-63-6	20	NA	GERG 0301	HRGC/LRMS
1,2,4-Trimethylbenzene	541-73-1	20	NA	GERG 0301	HRGC/LRMS
1,3-Dichlorobenzene	106-46-7	20	NA	GERG 0301	HRGC/LRMS
1,4-Dichlorobenzene	95-50-1	20	NA	GERG 0301	HRGC/LRMS
1,2-Dichlorobenzene	103-65-1	20	NA	GERG 0301	HRGC/LRMS
n-Proplybenzene	99-87-6	20	NA	GERG 0301	HRGC/LRMS
4-Isopropyl Toluene	98-06-6	20	NA	GERG 0301	HRGC/LRMS
tert-Butylbenzene	135-98-8	20	NA	GERG 0301	HRGC/LRMS
sec-Butylbenzene	104-51-8	20	NA	GERG 0301	HRGC/LRMS
n-Butylbenzene	96-12-8	20	NA	GERG 0301	HRGC/LRMS
1,2-Dibromo -3- Chlorpropane	87-61-6	20	NA	GERG 0301	HRGC/LRMS
1,2,3-Trichlorobenzene	120-82-1	20	NA	GERG 0301	HRGC/LRMS
1,2,4-Trichlorobenzene	87-68-3	20	NA	GERG 0301	HRGC/LRMS
Hexachlorobutadiene	91-20-3	50	NA	GERG 0301	HRGC/LRMS
Naphthalene	74-87-3	20	NA	GERG 0301	HRGC/LRMS

## **Semi-Volatile Organic Compounds** (analyzed in mg/kg = parts per million)

Analyte	CASRN	Reporting Limit <sup>1</sup> Extraction/Purification		Instrument Analysis	Technique
Benzoic acid	65-85-0	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
Phenol	108-95-2	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
2-Methylphenol	95-48-7	1.0	GERG 9807, 0009	EPA 8270C	GC/MS

Analyte	CASRN	Reporting Limit <sup>1</sup>	Extraction/Purification	Instrument Analysis	Technique
4-Methylpnenol	106-44-5	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
2,4 Dimethylphenol (or 1-4)	105-67-9	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
2-Chlorophenol	95-57-8	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
4-Chloro-3-methylphenol	59-50-7	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
2,4,-Dichlorophenol	120-83-2	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
2,6,-Dichlorophenol	87-65-0	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
2,4,6-Trichlorophenol	88-06-2	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
2,4,5-Trichlorophenol	95-95-4	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
2,3,4,6 Tetrachlorophenol	58-90-2	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
Pentachlorophenol	87-86-5	2.0	GERG 9807, 0009	EPA 8270C	GC/MS
2-Nitrophenol	88-75-5	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
4-Nitrophenol	100-02-7	4.0	GERG 9807, 0009	EPA 8270C	GC/MS
2,4 Dinitriphenol	51-28-5	2.0	GERG 9807, 0009	EPA 8270C	GC/MS
4,6,-Dinitro-2-methylphenol	534-52-1	2.0	GERG 9807, 0009	EPA 8270C	GC/MS
Bis (2-chloroethyl) ether	111-44-4	2.0	GERG 9807, 0009	EPA 8270C	GC/MS
Bis (2-chloroisopropyil) ether	108-60-1	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
Bis (2-chloroethoxy)methane	111-91-1	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
1,2 Dichlorobenzene	95-50-1	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
1,3-Dichlorobenzene	541-73-1	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
1,4 Dichlorobenzene	106-46-7	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
1,2,4,-Trichlorobenzene	120-82-1	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
1,2,4,5-Tetrachlorobenzene	95-94-3	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
Pentachlorobenzene	608-93-5	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
Pentachloronitrobenzene	82-68-8	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
Hexachloroethane	118-74-1	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
Hexachlorobutadiene	87-68-3	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
Hexachlorophene	70-30-4	1.0	GERG 9807, 0009	EPA 8270C	GC/MS

Analyte	CASRN	Reporting Limit <sup>1</sup>	Extraction/Purification	Instrument Analysis	Technique
Hexachloropropene	1888-71-7	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
Hexachlorocyclopentadiene	77-47-4	4.0	GERG 9807, 0009	EPA 8270C	GC/MS
Hexachlorobenzene	118-74-1	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
1-Chloronaphthalene	90-13-1	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
2-Chloronaphthalene	91-58-7	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
4-Chlorophenyl phenyl ether	7005-72-3	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
Naphthalene	91-20-3	0.4	GERG 9807, 0009	EPA 8270C	GC/MS
2-Methylnaphthalene	91-57-6	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
Acenaphthene	83-32-9	0.4	GERG 9807, 0009	EPA 8270C	GC/MS
Acenaphthylene	208-96-8	0.4	GERG 9807, 0009	EPA 8270C	GC/MS
Fluorene	86-73-7	0.4	GERG 9807, 0009	EPA 8270C	GC/MS
Phenanthrene	85-01-8	0.4	GERG 9807, 0009	EPA 8270C	GC/MS
Anthracene	120-12-7	0.4	GERG 9807, 0009	EPA 8270C	GC/MS
Fluoranthene	206-44-0	0.4	GERG 9807, 0009	EPA 8270C	GC/MS
Pyrene	129-00-0	0.4	GERG 9807, 0009	EPA 8270C	GC/MS
Benz(a)anthracene	56-55-3	0.4	GERG 9807, 0009	EPA 8270C	GC/MS
Chrysene	218-01-9	0.4	GERG 9807, 0009	EPA 8270C	GC/MS
Benzo(b)fluoranthene	205-99-2	0.4	GERG 9807, 0009	EPA 8270C	GC/MS
Benzo(k)fluoranthene	207-08-9	0.4	GERG 9807, 0009	EPA 8270C	GC/MS
Benzo(g,h,i)perylene	191-24-2	0.4	GERG 9807, 0009	EPA 8270C	GC/MS
Benzo(a)pyrene	50-32-8	0.4	GERG 9807, 0009	EPA 8270C	GC/MS
Dibenz(a,j)acridine	224-42-0	0.4	GERG 9807, 0009	EPA 8270C	GC/MS
Dibenz(a,h)anthracene	53-70-3	0.4	GERG 9807, 0009	EPA 8270C	GC/MS
7-12- Dimethylbenz(a)anthracene	57-97-6	0.4	GERG 9807, 0009	EPA 8270C	GC/MS
Indeno(1,2,3-cd)pyrene	193-39-5	0.4	GERG 9807, 0009	EPA 8270C	GC/MS
3-Methylcholanthrene	56-49-5	0.4	GERG 9807, 0009	EPA 8270C	GC/MS
Bis (2-ethylhexyl)adipate	103-23-1	1.0	GERG 9807, 0009	EPA 8270C	GC/MS

Analyte	CASRN	Reporting Limit <sup>1</sup>	Extraction/Purification	Instrument Analysis	Technique
Dimethyl phthalate	131-11-3	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
Diethyl phthalate	84-66-2	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
Di-n-butyl phthalate	84-74-2	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
Bis (2-ethylhexy l) phthalate	117-81-7	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
Butyl benzyl phthalate	85-68-7	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
Di-n-octyl phthalate	117-84-0	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
a-HCH	319-84-6	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
b-HCH	319-85-7	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
g-HCH (lindane)	58-89-9	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
d-HCH	319-86-8	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
Heptachlor	76-44-8	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
Heptachlor Epoxide	1024-57-3	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
Isodrin	465-73-6	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
Aldrin	309-00-2	2.0	GERG 9807, 0009	EPA 8270C	GC/MS
Dieldrin	60-57-1	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
Endrin	72-20-8	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
Endrin Aldehyde	7421-93-4	4.0	GERG 9807, 0009	EPA 8270C	GC/MS
Endrin ketone	53494-70-5	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
Endosulfan I	959-98-8	2.0	GERG 9807, 0009	EPA 8270C	GC/MS
Endosulfan II	33213-65-9	2.0	GERG 9807, 0009	EPA 8270C	GC/MS
Endosulfan sulfate	1031-07-8	2.0	GERG 9807, 0009	EPA 8270C	GC/MS
4,4'-DDE	72-55-9	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
4,4'-DDD	72-54-8	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
4,4'-DDT	50-29-3	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
4,4'-Methoxychlor	72-43-5	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
Terbufos	13071-79-9	2.0	GERG 9807, 0009	EPA 8270C	GC/MS
Fonofos (Dyfonate)	944-22-9	2.0	GERG 9807, 0009	EPA 8270C	GC/MS

Analyte	CASRN	Reporting Limit <sup>1</sup>	Extraction/Purification	Instrument Analysis	Technique
Diazinon	333-41-5	2.0	GERG 9807, 0009	EPA 8270C	GC/MS
Disulfoton	298-04-4	2.0	GERG 9807, 0009	EPA 8270C	GC/MS
Benzyl alcohol	100-51-6	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
Acetophenone	98-86-2	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
Methyl methanasulfonate	66-27-3	2.0	GERG 9807, 0009	EPA 8270C	GC/MS
Ethyl methaneasulfonate	62-50-0	2.0	GERG 9807, 0009	EPA 8270C	GC/MS
4-Bromophenyl phenyl ether	101-55-3	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
Isophorone	78-59-1	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
Phenacetin	62-44-2	2.0	GERG 9807, 0009	EPA 8270C	GC/MS
Dibenzofuran	132-64-9	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
Pronamide	23950-58-5	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
Carbazole	86-74-8	2.0	GERG 9807, 0009	EPA 8270C	GC/MS
Prometon	1610-18-0	2.0	GERG 9807, 0009	EPA 8270C	GC/MS
Pyridine	110-86-1	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
2-Picoline	109-06-8	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
Aniline	62-53-3	4.0	GERG 9807, 0009	EPA 8270C	GC/MS
2-Nitroanaline	88-74-4	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
3-Nitroaniline	99-09-2	2.0	GERG 9807, 0009	EPA 8270C	GC/MS
4-Nitroaniline	100-01-6	2.0	GERG 9807, 0009	EPA 8270C	GC/MS
4-Chloroaniline	106-47-8	4.0	GERG 9807, 0009	EPA 8270C	GC/MS
a,a-Dimethylphenylamine	122-09-8	2.0	GERG 9807, 0009	EPA 8270C	GC/MS
1,4-Phenylenediamine	624-18-0	2.0	GERG 9807, 0009	EPA 8270C	GC/MS
Diphenylamine	122-39-4	2.0	GERG 9807, 0009	EPA 8270C	GC/MS
Benzidine	92-87-5	2.0	GERG 9807, 0009	EPA 8270C	GC/MS
3,3'-Dichlorobenzidine	91-94-1	4.0	GERG 9807, 0009	EPA 8270C	GC/MS
4-Aminobiphenyl	92-67-1	2.0	GERG 9807, 0009	EPA 8270C	GC/MS
1-Naphthylamine	134-32-7	1.0	GERG 9807, 0009	EPA 8270C	GC/MS

Analyte	CASRN	Reporting Limit <sup>1</sup>	Reporting Limit <sup>1</sup> Extraction/Purification		Technique
2-Naphthylamine	91-59-8	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
p-Dimethylaminoazobenzene	60-11-7	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
Nitrobenzene	98-95-3	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
Azobenzene	103-33-3	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
2,4,-Dinitritoluene	121-14-2	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
2,6,-Dinitrotoluene	606-20-2	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
n-Nitrosodi-n-methylamine	62-75-9	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
n-Nitrosodiethylamine	55-18-5	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
N Nitrosodi-n-propylamine	621-64-7	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
n-Nitrodi-n-butylamine	924-16-3	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
n-Nitrosodiphenylamine	86-30-6	1.0	GERG 9807, 0009	EPA 8270C	GC/MS
n-Nitrosopiperidine	100-75-4	2.0	GERG 9807, 0009	EPA 8270C	GC/MS

## **Dioxins and Furans** (analyzed in pg/g = parts per trillion)

Analyte	CASRN	Reporting Limit <sup>1</sup>	Extraction/Purification	Instrument Analysis	Technique
2,3,7,8-TCDD	1746-01-6	0.5	GERG 9719	GERG 9722	HRGC/HRMS
1,2,3,7,8-PeCDD	40321-76-4	0.5	GERG 9719	GERG 9722	HRGC/HRMS
1,2,3,4,7,8-HxCDD	39227-28-6	2.5	GERG 9719	GERG 9722	HRGC/HRMS
1,2,3,6,7,8-HxCDD	57653-85-7	2.5	GERG 9719	GERG 9722	HRGC/HRMS
1,2,3,7,8,9-HxCDD	19408-74-3	2.5	GERG 9719	GERG 9722	HRGC/HRMS
1,2,3,4,6,7,8-HpCDD	35822-46-9	2.5	GERG 9719	GERG 9722	HRGC/HRMS
OCDD	3268-87-9	5.0	GERG 9719	GERG 9722	HRGC/HRMS
2,3,7,8-TCDF	51207-31-9	0.5	GERG 9719	GERG 9722	HRGC/HRMS
1,2,3,7,8-PeCDF	57117-41-6	2.5	GERG 9719	GERG 9722	HRGC/HRMS
2,3,4,7,8-PeCDF	57117-31-4	2.5	GERG 9719	GERG 9722	HRGC/HRMS
1,2,3,4,7,8-PeCDF	70648-26-9	2.5	GERG 9719	GERG 9722	HRGC/HRMS
1,2,3,6,7,8-HxCDF	57117-44-9	2.5	GERG 9719	GERG 9722	HRGC/HRMS

Analyte	CASRN	Reporting Limit <sup>1</sup>	Extraction/Purification	Instrument Analysis	Technique
2,3,4,6,7,8-HxCDF	60851-34-5	2.5	GERG 9719	GERG 9722	HRGC/HRMS
1,2,3,7,8,9-HxCDF	72918-21-9	2.5	GERG 9719	GERG 9722	HRGC/HRMS
1,2,3,4,6,7,8-HpCDF	67562-39-4	2.5	GERG 9719	GERG 9722	HRGC/HRMS
1,2,3,4,7,8,9-HpCDF	55673-89-7	2.5	GERG 9719	GERG 9722	HRGC/HRMS
OCDF	39001-02-0	5.0	GERG 9719	GERG 9722	HRGC/HRMS

<sup>1</sup> Reporting Limit = The reporting limits (RLs) listed in these tables are the specifications at or above which chemical contaminant concentrations must be quantified. Ongoing ability to recover an analyte at the reporting limit is demonstrated through analysis of a calibration check standard at the reporting limit. See the *DSHS Laboratory or designated contract laboratory Quality Control* for quality control details and acceptance criteria.

 $^{2}$ NA = not applicable

<sup>3</sup>Chlordane (total) is the sum of the primary constituents of technical –grade chlordane: alpha chlordane, gamma chlordane, cis-nonachlor, trans-nonachlor and oxychlordane, the major metabolite of chlordane.

<sup>4</sup> PCB congener information obtained from Toxicological Profile for Polychlorinated Biphenyls (Update) 1997. U.S. Department of Health and Human Services, Public Health Service, Agency for Toxic Substances and Disease Registry. pp 217-222 and Guidance for assessing chemical contaminant data for use in fish advisories. Vol. 1, Fish Sampling and Analysis, 3<sup>rd</sup> ed. Washington, D.C.: 2000. U.S. Environmental Protection Agency. p 4-53.

DSHS SALG Survey Team Fish and Shellfish Tissue Collection Data Form

	DSHS Seafood and Aquatic Life Group Fish and Shellfish Tissue Data Form									
Waterbody_			Site Name			Site Code Date Time				
TCEQ Regio	n	TCEQ Statio	n	TCE	Q Segment	t T	CEQ Seque	nce N_		
Weather			Wind Direction_			Wind Spee	d	Air temp		
Water temp	)	Sal	linity	р	H	Specific	: Conductar	nce D.O		
Hydrologic Collector(s)	conditions _ )			Fish	Observati	ons				
Sample #	Date Collected	Date Processed	Tissue Analysis	Gear Type	EPA Sp. Code	Species	Number	Composite Sample Average Size	Length (mm)	Weight (g)

Waterbody Codes for Texas Public Waters

## Waterbody Codes for Texas Public Waters

Waterbody	Water body Code
Adams Bayou	ADB
Aransas Bay	ARA
Arroyo Colorado	ARC
Arroyo Colorado (Port of Harlingen)	РОН
B.A. Steinhagen Reservoir (North of the Hwy. 190 bridge)	USR
B.A. Steinhagen Reservoir (South of the Hwy. 190 bridge to the dam)	LSR
Baffin Bay	BAF
Bastrop Bay	BAS
Big Cypress Creek	СҮС
Black Cypress Bayou	BCB
Bouton	BOU
Boykin Springs	BOY
Brakes Bayou	BRK
Brandy Branch Reservoir	BBR
Braunig Lake	BRL
Brazos River Channel (Old Channel)	OBC
Caddo Lake	CDL
Calaveras Lake	CAV
Canyon Lake	CAN
Carancahua Lake (West Galveston Bay)	CAL
Carancuhua Bay	CAR
Cement Creek Reservoir (Fort Worth area)	CCR
Chocolate Bayou	CHB
Christmas Bay	CHR
Clear Creek	CLC
Clear Lake	CLK
Clear Lake (Panola County)	CLR
Colorado River	COL
Como Lake	СОМ
Conroe Reservoir	CON
Copano Bay	СОР
Corpus Christi (Port)	PCC
Corpus Christi Bay	COR

Waterbody	Water body Code
Cow Bayou	COW
Creek Bend Resaca (Brownsville)	CBR
Daingerfield Reservoir	DAI
Delta Lake	DTL
Donna Irrigation Canal	DIC
Drum Bay	DRM
East Galveston Bay	EAS
East Matagorda Bay	EMB
Echo Lake	ECH
Ellison Creek Reservoir	ECR
Espirito Santo Bay	ESP
Forest Park Lake	FOR
Fork Reservoir	FOR
Fosdic Lake	FOS
Freeport (offshore)	FRO
Freeport Area (Caney Creek to West Gal. Bay)	FRE
French Lake (Fort Worth)	FRL
Galveston (offshore)	GAO
Galveston Bay	GAL
Greens Lake (West Galveston Bay)	GRL
Hidden Valley Resaca (Brownsville)	HID
Hills Lake	HIL
Hildalgo Settling Basin	HSB
Houston Reservoir	HOU
Houston Ship Channel	HSC
Joe Pool Reservoir	JPR
Kimball Lake	KIM
Laguna Madre	LAG
Lake O'the Pines	LOP
Lavaca Bay	LAV
Leon Creek	LEC
Livingston Reservoir	LIV
Livingston Reservoir (Trinity River confluence to approx 0.5 miles into the reservoir)	ULL
Llano Grande Lake	ШG
Lower Waterworks	LWW
Mabel Davis Park Pond	MDP
Madison Lake	LMD

Waterbody	Water body Code
Martin Creek Reservoir	MAR
Matagorda Bay	MAT
Mercedes Main Canal	MMC
Mercedes Settling Basin	MSB
Meredith Reservoir	LMR
Mesquite Bay	MES
Millwood Lake	MWL
Moses Lake	MOL
Moss Lake	MOS
Mountain Creek Lake	MCL
Nacogdoches Reservoir	NAC
Neches River	NEC
Nueces Bay	NUE
O.H. Ivie Reservoir	OHI
Palastine Reservoir	PAL
Pine Island Bayou	PIB
Port Aransas (offshore)	PAO
Port O'Conner (offshore)	POO
Powderhorn lake	PWH
Ratcliff Reservoir	RAT
Raven Reservoir	RAV
Red Bluff Reservoir	RBR
Rio Grande River	RGR
Sabine Lake	SAB
Sam Rayburn Reservoir (North of Hwy. 147 bridge)	USM
Sam Rayburn Reservoir (South of Hwy. 147 bridge to the dam)	LSM
San Antonio Bay	SAN
San Antonio River	SAR
San Jacinto River	SAJ
South Bay (Lower Laguna Madre)	SOU
Swan Lake	SWL
Tabbs Bay	TAB
Tawakoni Reservoir	TAW
Taylor Bayou	TYB
Timpson Lake	TIM

Waterbody	Water body Code
Toledo Bend Reservoir (North of the Pendelton bridge)	UTB
Toledo Bend Reservoir (South of the Pendelton bridge to the dam)	LTB
Town Lake	TNL
Trinity Bay	TRI
Twin Lakes (Town of Lytle)	TWI
Waco Lake	WAC
Welsh Reservoir	WEL
West Galveston Bay	WES

DSHS & EPA Species Code Lists

Species	DSHS Species Code	EPA Species Code
Black crappie	BLC	005
Bluegill sunfish	BLG	008
Blue catfish	BCF	067
Channel catfish	CCF	016
Common carp	CRP	012
Flathead catfish	FHC	019
Freshwater drum	FWD	020
Grey redhorse	GRH	192
Green sunfish	GRS	025
Guadalupe bass	GLB	095
Hybrid striped bass	HSB	198
Largemouth bass	LMB	031
Longear sunfish	LES	072
Longnose gar	LNG	032
Orangespotted sunfish	OSS	413
Redbreast sunfish	RBS	070
Redear sunfish	RES	040
Rio Grande cichlid	RGP	686
River carpsucker	RCS	042
Shortnose gar	SNG	107
Smallmouth bass	SMB	047
Smallmouth buffalo	BUF	048
Spotted gar	SPG	050
Spotted bass	SPB	049
Striped bass	STB	052
Blue tilapia	ТАР	054
Walleye	WAL	055
Warmouth	WAM	056
White Crappie	WHC	059
White Bass	WHB	057

DSHS and EPA Freshwater Species Code List

Species	DSHS Species Code	EPA Species Code
Atlantic croaker	CRK	115
Barracuda	BAR	111
Black drum	BDR	199
Blacktip shark	BTS	204
Blue Marlin	BLM	
Bull shark	BLS	
Bonnethead shark	BHS	713
Blue crab	BCR	226
Brown shrimp	BSH	234
Cobia / Ling	СОВ	190
Eastern oyster	EOY	217
Gafftopsail catfish	GTC	200
Greater amberjack	ABJ	110
Gulf kingfish	GKF	203
Hardhead catfish	HHC	604
King Mackerel	KMC	122
Lane snapper	LSP	
Pink Shrimp	PSH	233
Red drum	RDR	202
Red snapper	RSN	131
Sailfish	SLF	
Sand trout	STR	134
Sheepshead	SSH	078
Snook	SNK	711
Southern flounder	SOF	201
Spanish mackerel	SPM	498
Spotted seatrout	SST	142
Stone crab	SCR	
Vermilion snapper	VSP	
Wahoo	WHO	
White Marlin	WHM	317
White shrimp	WSH	232

## DSHS and EPA Estuarine and Marine Species Code List

DSHS SALG Chain-of-Custody Record Form

# **Texas Department of State Health Services** Seafood and Aquatic Life Group Chain-of-Custody Record

8407 Wall Street Austin, TX 78758 512-834-6757 fax: 512-834-6762

Account Number\_\_\_\_\_ Tissue Sample Collection Date\_\_\_\_\_

Project Contract Number\_\_\_\_\_ Water body\_\_\_\_\_

## Shipment Tracking Record

Shipment Activity	Agency/ Company	Print Name	Signature	Date & Time
Relinquished by				
Accepted by Shipper				
Accepted by lab				

### **Tissue Analysis**

Sample Number	Species	Metals (Circle Metal)	Pesticides	PCBs	SVOCs	VOCs	Dioxins
		As Cd Cu Hg Pb Se Zn					
		As Cd Cu Hg Pb Se Zn					
		As Cd Cu Hg Pb Se Zn					
		As Cd Cu Hg Pb Se Zn					
		As Cd Cu Hg Pb Se Zn					
		As Cd Cu Hg Pb Se Zn					
		As Cd Cu Hg Pb Se Zn					
		As Cd Cu Hg Pb Se Zn					
		As Cd Cu Hg Pb Se Zn					
		As Cd Cu Hg Pb Se Zn					



DSHS SALG Data Review Form

# DSHS Seafood and Aquatic Life Group Data Review Form

Parameter	Waterbody:				
Group Reviewed	Data Entered	Data Reviewed	Data Corrected	Corrections Verified	
Dioxins / Furans					
VOCs					
SVOCs					
Pesticides					
PCBs					
Metals					
Field Data					
Other:					

	Final Review of Corrections by QA Officer:	
Signature:	Date:	



Geochemical and Environmental Research Group (GERG) Texas A&M University GERG Quality Assurance Project Plan (QAPP) Appendix C

Geochemical and Environmental Research Group (GERG) Texas A&M University Quality Assurance Project Plan

## QUALITY ASSURANCE PROJECT PLAN (QAPP)

prepared by

Geochemical and Environmental Research Group Texas A&M University 833 Graham Road College Station, TX 77845

Texas Department of Health Seafood Safety Division 1100 West 49th Street Austin, TX 78756 Attn: Michael Tennant 512-719-0215

May 10, 2004

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#### FOREWORD

The attached Quality Assurance Project Plan (QAPP) is based on the standards as defined in the Geochemical and Environmental Research Group (GERG) Quality Assurance Management Plan (QAMP). Each QAPP is specifically designed for an individual project and is intended to incorporate all principles enunciated in the GERG QAMP. The detailed QAPP provides the project design and QA objectives in sufficient detail to assure accomplishment of program goals in a timely, efficient, and cost effective manner. The implementation of this QAPP will insure environmental data of the appropriate type and quality as required for its intended use. The general format of the QAPP follows the guidance contained in the U.S. EPA's document QAMS-005/80.

QAPP Authorized by

Mahlon C. Kennizutt II, Ph.D. Director, Geochemical and Environmental Research Group

5/10/04

en 2. Wate

Terry L. Wade, Ph.D Program Manager

5/11/04 Date

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#### **1.0 PROJECT DESCRIPTION**

#### 1.1 <u>General Overview</u>

This quality assurance project plan (QAPP) is provided in support of studies conducted by the Texas Department of Health - Seafood Safety Division (TDH), for the analyses of trace amounts of organic and inorganic contaminants in the tissues of fish and shellfish. The Department of Health is the Texas's agency created to protect and promote the health and safety of the people of Texas. To this end, the Department administers a variety of programs designed to identify and reduce recognized health risks to the public. To implement programs to attain this goal the TDH continually collects, analyzes, and synthesizes information that will provide for sound and timely management decisions. One element of this decision making process requires determination of organic and inorganic contaminants found in organisms.

A wide spectrum of geographic areas and contaminant problems are studied including contamination from agriculture, energy development, and industrial activities. This project proposes for the Geochemical and Environmental Research Group (GERG), Texas A&M University (TAMU), to provide high quality environmental analyses of contaminant compounds in samples received from the TDH.

#### 1.2 General Considerations

GERG will provide adequate personnel, equipment and resources to implement all trace analyses for the proposed project which are listed in Table 1.1. The analysis of all components as requested by TDH will be provided as described in this QAPP. The appropriate sample receipt, preparation equipment, and storage capacity are available at GERG. Gas chromatographs with appropriate detectors and other equipment and instruments required are available to analyze and report data from the samples generated by this project. In addition, a high resolution gas chromatography/high resolution mass spectrometer is available for dioxin/furan analyses. It is also clearly recognized that most samples submitted will arrive over a very short period of time, intermittently, each year. GERG is prepared to operate within the required time frames.

#### 1.2.1 Sample Storage and Processing Requirements

The freezer capacity needed is available to store tissue samples received, all unanalyzed portions of a sample, and all extracts/digests for analysis will be stored for at least one year after the analytical report is accepted, until disposition or return is approved by the TDH COTR. Freezing is specified as  $-20^{\circ}C \pm 10^{\circ}C$ .

Processing of samples received will be performed when requested. A sample batch is considered received (complete) when all samples have arrived at the laboratory intact and properly labeled, the sample identification number matches the delivery order received, and there is a match between the samples received with the work described in the delivery order.

All chemical contaminant concentrations will be reported on a wet weight basis. The laboratory methods and laboratory techniques listed are the current methods and techniques used by the GERG laboratory for metals, pesticides, polychlorinated biphenyls (PCBs), semi-volatile organic compounds (SVOCs), volatile organic compounds (VOCs), and dioxins/furans. For all samples, % moisture will be determined. For all organic analysis, % lipids will be determined.

### Table 1.1. Target analytes and reporting limits to be determined for the TDH program.

Analyte	*Reporting Limit	Technique
Arsenic	0.10	GFAAS
Cadmium	0.10	ICP-MS
Copper	0.40	ICP-MS
Lead	0.40	ICP-MS
Mercury	0.20	CVAAS
Selenium	0.10	GFAAS
Zinc	0.40	ICP-MS

Metals (mg/kg = parts per million) (Digestion Method: GERG 9408)

Pesticides ( $\mu$ g/kg = parts per billion)

Analyte	*Reporting Limit	Analyte	*Reporting Limit
Aldrin	2.0	Endosulfan II	10
Alachlor	8.0	Endosulfan Sulfate	10
alpha BHC	2.0	Endrin	6.0
beta BHC	2.0	Heptachlor	2.0
delta BHC	2.0	Heptachlor epoxide	4.0
Chlordane <sup>1</sup>	10	Hexachlorobenzene	2.0
Chlorpyrifos	10	Lindane	2.0
p,p'DDE	5.0	Malathion	20
p,p'DDD	10	Methoxychlor	30
p,p′DDT	10	Mirex	8.0
Dacthal	3.0	Ethyl parathion	10
Diazinon	10	Methyl parathion	10
Dieldrin	6.0	Toxaphene	100
Endosulfan I	10		

Polychlorinated Biphenyls (analyzed at  $\mu$ g/kg = parts per billion)

Analyte	*Reporting Limit
Aroclor 1016**	40
Aroclor 1221**	40
Aroclor 1232**	40
Aroclor 1242**	40
Aroclor 1248**	40
Aroclor 1254**	40
Aroclor 1260**	40

\*\* Aroclor is a registered trademark of the Monsanto Corporation

## Table 1.1. (Cont.). Additional Required PCB Parameters<sup>3</sup>

Polychlorinated biphenyls to be individually identified and quantified.

2,4' dichlorobiphenyl8 34883-43-72,2',5 trichlorobiphenyl18 37680-65-22,4,4' trichlorobiphenyl28 7012-37-53,4,4' trichlorobiphenyl37 38444-90-5
2,2',5 trichlorobiphenyl       18 37680-65-2         2,4,4' trichlorobiphenyl       28 7012-37-5         3,4,4' trichlorobiphenyl       37 38444-90-5
2,4,4' trichlorobiphenyl       28 7012-37-5         3,4,4' trichlorobiphenyl       37 38444-90-5
3,4,4' trichlorobiphenyl 37 38444-90-5
2,2',3,5' tetrachlorobiphenyl 44 41464-39-5
2,2',4,5' tetrachlorobiphenyl 49 41464-40-8
2,2',5,5' tetrachlorobiphenyl 52 35693-99-3
2,3',4,4' tetrachlorobiphenyl 66 32698-10-1
2,3',4',5 tetrachlorobiphenyl 70 32598-11-1
2,4,4',5 tetrachlorobiphenyl 74 32690-93-0
3,3',4,4' tetrachlorobiphenyl 77 32598-13-3
3,4,4',5 tetrachlorobiphenyl 81 70362-50-4
2,2',3,4,5' pentachlorobiphenyl 87 38380-02-8
2,2',3,4',5 pentachlorobiphenyl 90 68194-07-0
2,2',4,5,5' pentachlorobiphenyl 101 37680-73-2
2,3,3',4,4' pentachlorobiphenyl 105 32598-14-4
2,3,4,4',5 pentachlorobiphenyl 114 74472-37-0
2,3',4,4',5 pentachlorobiphenyl 118 31508-00-6
2,3',4,4',6 pentachlorobiphenyl 119 56558-17-9
2',3,4,4',5 pentachlorobiphenyl 123 65510-44-3
3,3',4,4',5 pentachlorobiphenyl 126 57465-28-8
2,2',3,3',4,4' hexachlorobiphenyl 128 38380-07-3
2,2',3,4,4',5' hexachlorobiphenyl 138 35065-28-2
2,2',3,5,5',6 hexachlorobiphenyl 151 52663-63-5
2,2',4,4',5,5' hexachlorobiphenyl 153 35065-27-1
2,3,3',4,4',5 hexachlorobiphenyl 156 38380-08-4
2,3,3',4,4',5' hexachlorobiphenyl 157 69782-90-7
2,3,3',4,4',6 hexachlorobiphenyl 158 74472-42-7
2,3',4,4',5,5' hexachlorobiphenyl 167 52663-72-6
2,3',4,4',5',6 hexachlorobiphenyl 168 59291-65-5
3,3',4,4',5,5' hexachlorobiphenyl 169 32774-16-6
2,2',3,3',4,4',5 heptachlorobiphenyl 170 35065-30-6
2,2',3,4,4',5,5' heptachlorobiphenyl 180 35065-29-3
2,2',3,4,4',5,6 heptachlorobiphenyl 181 74472-47-2
2,2',3,4,4',6,6' heptachlorobiphenyl 184 74472-48-3
2,2',3,4,5,5',6 heptachlorobiphenyl 185 52712-05-7
2,3,3',4,4',5,6 heptachlorobiphenyl 190 41411-64-7
2,2',3,3',4,4',5,6 octachlorobiphenyl 195 52663-78-2
2,2',3,3',4,5,6,6' octachlorobiphenyl 200 52663-73-7
2,2',3,3',4,4',5,5',6 nonachlorobiphenyl 206 40186-72-9
2,2',3,3',4,4',5,5',6,6' decachlorobiphenyl 209 2051-24-3
Analyte
-----------------------------
1,1,1,2 - Tetrachloroethane
1,1,1-Trichloroethane
1,1,2,2-Tetrachloroethane
1,1,2-Trichloroethane
1,1-Dichloroethane
1,1-Dichloroethene
1,1-Dichloropropene
1,2,3-Trichlorobenzene
1,2,3-Trichloropropane
1,2,4-Trichlorobenzene
1,2,4-Trimethylbenzene
1,2-Dibromo-3-Chloropropane
1,2-Dibromoethane
1,2-Dichlorobenzene
1,2-Dichloroethane
1,2-Dichloropropane
1,3,5-Trimethylbenzene
1,3-Dichlorobenzene
1,3-Dichloropropane
1,4-Dichlorobenzene
2,2-Dichloropropane
2-Butanone (MEK)
2-Chlorotoluene
2-Hexanone
4-Chlorotoluene
4-Isopropyl toluene
4-Methyl-2-Pentanone
Acetone
Acrylonitrile
Benzene
Bromobenzene
Bromochloromethane
Bromodichloromethane
Bromoform
Bromomethane
Carbon Disulfide

# Table 1.1. Volatile Organic Compounds (analyzed in $\mu$ g/kg = parts per billion; Cont.).

# Table 1.1. Volatile Organic Compounds (analyzed in $\mu$ g/kg = parts per billion; Cont.).

Analyte	*Reporting Limit
Carbon Tetrachloride	20
Chlorobenzene	20
Chloroethane	50
Chloroform	20
Chloromethane	50
cis-1,2-Dichloroethene	20
cis-1,3-Dichloropropene	100
Dibromochloromethane	20
Dibromomethane	20
Dichlorodifluoromethane	50
Ethyl Methacrylate	20
Ethylbenzene	20
Hexachlorobutadiene	50
Iodomethane	50
Isopropylbenzene	20
m&p-Xylene	40
Methyl Methacrylate	20
Methyl-tert-butyl ether (MTBE)	20
Methylene chloride	50
n-Butylbenzene	20
n-Propylbenzene	20
Naphthalene	20
o-Xylene	20
sec-Butylbenzene	20
Styrene	20
tert-Butylbenzene	20
Tetrachloroethene	20
Tetrahydrofuran	50
Toluene	20
trans-1,2-Dichloroethene	20
trans-1,3-Dichloropropene	100
Trichloroethene	20
Trichlorofluoromethane	50
Vinyl Chloride	50

## Table 1.1. Semi-Volatile Organic Compounds (analyzed in mg/kg = parts per million; Cont.)

Analyte	*Reporting Limit
Pyridine	1.0
N-Nitrosodimethylamine	1.0
N-Nitrosodiethylamine	1.0
Aniline	4.0
Phenol	1.0
<i>bis</i> (2-Chloroethyl)ether	2.0
2-Chlorophenol	1.0
1,3-Dichlorobenzene	1.0
1,4-Dichlorobenzene	1.0
Benzyl alcohol	1.0
1,2-Dichlorobenzene	1.0
2-Methylphenol	1.0
bis(2-Chloroisopropyl)ether	1.0
<sup>3</sup> / <sub>4</sub> -Methylphenol (coelute)	1.0
N-Nitroso-di-n-propylamine	1.0
Hexachloroethane	1.0
Nitrobenzene	1.0
Isophorone	1.0
2-Nitrophenol	1.0
2,4-Dimethylphenol	1.0
bis(2-Chloroethoxy)methane	1.0
Benzoic Acid	1.0
2,4-Dichlorophenol	1.0
1,2,4-Trichlorobenzene	1.0
Naphthalene	0.4
4-Chloroaniline	4.0
Hexachlorobutadiene	1.0
N-Nitroso-di-n-butylamine	1.0
4-Chloro-3-methylphenol	1.0
2-Methylnaphthalene	1.0
1,2,4,5-Tetrachlorobenzene	1.0
Hexachlorocyclopentadiene	4.0
2,4,6-Trichlorophenol	1.0
2,4,5-Trichlorophenol	1.0
2-Chloronaphthalene	1.0
2-Nitroaniline	1.0
Dimethylphalate	1.0
Acenaphthylene	0.4

## Table 1.1. Semi-Volatile Organic Compounds (analyzed in mg/kg = parts per million; Cont.)

Analyte	*Reporting Limit
2,6-Dinitrotoluene	1.0
3-Nitroaniline	2.0
Acenaphthene	0.4
2,4-Dinitrophenol	2.0
4-Nitrophenol	4.0
Dibenzofuran	1.0
2,4-Dinitrotoluene	1.0
Diethylphthalate	1.0
Fluorene	0.4
4-Chlorophenyl-phenylether	1.0
4-Nitroaniline	2.0
Diphenylhydrazine	1.0
4,6-Dinitro-2-methylphenol	2.0
N-Nitrosodiphenylamine	1.0
4-Bromophenyl-phenylether	1.0
Hexachlorobenzene	1.0
Pentachlorophenol	2.0
Alpha-BHC	1.0
Beta-BHC	1.0
Lindane	1.0
Delta-BHC	1.0
Phenanthrene	0.4
Anthracene	0.4
Di-n-butylphthalate	1.0
Heptachlor	1.0
Aldrin	2.0
Fluoranthene	0.4
Heptachlor epoxide	1.0
Pyrene	0.4
Alpha endosulfan	2.0
Benzidine	$ND^2$
<i>p,p</i> ′-DDE	1.0
Dieldrin	1.0
Butylbenzylphthalate	1.0
Endrin	1.0
Beta-Endosulfan	2.0
p,p ′-DDD	1.0
Endrin aldehyde	ND2

Table 1.1. Se	emi-Volatile Organic	Compounds	(analyzed in	mg/kg = pa	rts per n	nillion;
		Cont.)				

Analyte	*Reporting Limit
<i>p,p</i> ′-DDT	1.0
bis(2-Ethylhexyl)adipate	1.0
Endosulfan sulfate	2.0
Benzo[a]anthracene	0.4
3,3-Dichlorobenzidine	4.0
Chrysene	0.4
Endrin ketone	1.0
Bis(2- Ethylhexyl)phthalate	1.0
di-n-Octylphthalate	1.0
Benzo[b]fluoranthene	0.4
Benzo[k]fluoranthene	0.4
Hexachlorophene	ND <sup>2</sup>
Benzo[a]pyrene	0.4
Indeno[1,2,3-cd]pyrene	0.4
Dibenz[a,h]anthracene	0.4
Benzo(g,h,i)perylene	0.4

Dioxins (analyzed in pg/g = parts per trillion)

Analyte	*Reporting Limit
2,3,7,8-Tetrachloro-dibenzo-p-dioxin	0.5
1,2,3,7,8-Pentachloro-dibenzo-p-dioxin	0.5
1,2,3,4,7,8-Hexachloro-dibenzo-p-dioxin	2.5
1,2,3,6,7,8-Hexachloro-dibenzo-p-dioxin	2.5
1,2,3,7,8,9-Hexachloro-dibenzo-p-dioxin	2.5
1,2,3,4,6,7,8-Heptachloro-dibenzo-p-dioxin	2.5
Octachloro-dibenzo-p-dioxin (Total)	5.0

Analyte	*Reporting Limit
2,3,7,8-Tetrachloro-dibenzo-p-furan	0.5
1,2,3,7,8-Pentachloro-dibenzo-p-furan	2.5
2,3,4,7,8-Pentachloro-dibenzo-p-furan	2.5
1,2,3,4,7,8-Hexachloro-dibenzo-p-furan	2.5
1,2,3,6,7,8-Hexachloro-dibenzo-p-furan	2.5
2,3,4,6,7,8-Hexachloro-dibenzo-p-furan	2.5
1,2,3,7,8,9-Hexachloro-dibenzo-p-furan	2.5
1,2,3,4,6,7,8-Heptachloro-dibenzo-p-furan	2.5
1,2,3,4,7,8,9-Heptachloro-dibenzo-p-furan	2.5
Octachloro-dibenzo-p-furan (Total)	5.0

Table 1.1	Furans	(analvzed	in	$n\sigma/\sigma =$	parts r	per trill	ion <sup>.</sup> (	Cont)	1
1 4010 1.1.	I MIMID	( 41141 ) 204		ר יכי	parts p			<i>conc.</i> ,	/•

<sup>1</sup> Chlordane value represents total chlordane, which is the sum of the primary constituents of technical –grade chlordane: alpha chlordane, gamma chlordane, cis-nonachlor, trans-nonachlor and oxychlordane, the major metabolite of chlordane.

 $^{2}$  ND = Detection Limit not established.

<sup>3</sup> PCB congener information obtained from Toxicological Profile for Polychlorinated Biphenyls (Update) 1997. U.S. Department of Health and Human Services, Public Health Service, Agency for Toxic Substances and Disease Registry. pp 217-222 and Guidance for assessing chemical contaminant data for use in fish advisories. Vol. 1, Fish Sampling and Analysis, 3<sup>rd</sup> ed. Washington, D.C.: 2000. U.S. Environmental Protection Agency. p 4-53. \*Reporting Limit = The reporting limits (RLs) listed in these tables are the specifications at or above which chemical contaminant concentrations must be quantified. Ongoing ability to recover an analyte near the reporting limit is demonstrated through analysis of a calibration check standard at the reporting limit.

## 2.0 PROJECT ORGANIZATION AND RESPONSIBILITY

The project will be performed by personnel drawn from the Geochemical and Environmental Research Group (GERG) of the College of Geosciences at Texas A&M University. Dr. Mahlon C. Kennicutt, II is the Director of GERG which is located at 833 Graham Road in College Station, Texas, 77845. The telephone number is (979) 862-2323, and the FAX number is (979) 862-2361.

Dr. Terry L. Wade, Program Manager, will be responsible for the overall administration and execution of the project and Dr. Guy Denoux will function as the Deputy Program Manager and Data Manager. The management organization of the project is depicted in Figure 2.1.

#### 2.1 <u>Position Descriptions for Project Team</u>

The responsibility of each project team member is summarized below and the project management organization is illustrated in Figure 2.1.

## Program Manager

The Program Manager is responsible for overall administration and execution of the project and is the designated study director. Specific responsibilities include:





- Establishes and documents the roles and responsibilities of project personnel.
- Coordinates auditing of project activities
- Establishes and conducts a self-assessment program.
- Has final responsibility to insure all deliverables are provided on-time to the client.
- Establishes and develops the implementing procedures
- Approves expenditures of funds for the project.

## Deputy Program Manager

The Deputy Program Manager reports to the Program Manager and has responsibility with the Program Manager for the project in all financial, management, scientific, and quality assurance issues. The Deputy Program Manager is the responsible party in the absence of the Program Manager. The Deputy Program Manager:

- Coordinates internal and external interfaces of personnel involved with the project.
- Oversees the activities of the quality assurance unit for this project, designates personnel to perform inspections, and maintains records related to these activities.
- Ensures that the applicable QC requirements are met.
- Ensures that quality-related issues and problems are promptly identified and corrected.
- Interfaces with the QA Manager on program QA/QC considerations.
- Implements cost effective quality improvements.
- Supervises the progress of the analytical program and team.
- Assists the Organic Analytical Laboratories Manager in tracking corrective actions and analyzing data pertaining to quality.
- Provides guidance to resolve quality problems and ensure that corrective action is taken and appropriately documented in response to occurrence reports, non-conformance reports, etc.
- Identifies areas where improvement could benefit the program.

## Quality Assurance Manager

The Quality Assurance (QA) Manager is responsible for developing, enacting, and enforcing all QA/QC procedures and policies. The QA Manager ensures that all project activities are operated in a manner that provides confidence that project quality control (QC) objectives are met. The QA Manager is independent of project management, reports to the Senior Associate Director of GERG, and is responsible for ensuring all applicable QA/QC policies and directives are enforced, revised and improved to provide products of the highest quality to clients. Specific responsibilities include:

- Maintains and revises the GERG Quality Assurance Management Plan (QAMP) and the Generic Quality Assurance Manual (GQAM).
- Advises the Program Manager, the Deputy Program Manager and the project team members on QA/QC matters.
- Ensures that QA/QC requirements are effectively implemented for all project activities.

- Ensures that the QAPP is adequately developed to meet project needs and is effectively implemented.
- Coordinating, preparing, approving and reviewing QA/QC documents including all quality requirements contained in standard operating procedures.
- Identifies QA/QC requirements and assists in the development of procedures and other implementing instructions.
- Assists in the identification of problems concerning, and taking actions to eliminate or minimize potential QA problems.
- Evaluates quality performance including internal system audits, tracking of reports of QA/QC criteria, reviewing corrective actions, and overall project performance.
- Provides QA/QC training to all project personnel when required.
- Has the authority to stop the work when severe conditions adverse to quality are detected and warrant immediate action.

## Program Data Manager

The Program Data Manager reports to the Program Manager. The Program Data Manager is responsible for:

- Compiling, editing, and verifying all project data.
- Assuring data management, validation, and reporting conforms with the project requirements.
- Assuring that hard copy and electronic data formats are compatible with the intended users data requirements.
- Advising the Quality Assurance Manager on data management QA/QC issues.
- Assists in preparation of final project reports.

## Laboratory Managers

The Extraction Laboratory Manager and the Analytical Laboratory Managers are the technical supervisors responsible for the sample extract preparation and the instrumental analyses. The Laboratory Managers report to the Program Manager for this program and are responsible for:

- Supervision and coordination of all aspects of the laboratories and the analytical laboratories.
- Coordination with the Program Manager and Deputy Program Manager to submit sample extracts to the laboratory to ensure technical quality and due dates are met on all projects.
- Implementing the required standard operating procedures and the Quality Assurance Project Plan.
- Ensuring the quality of assigned work by monitoring daily performance, calibration, and QC data.
- Investigating quality problems, determining their root causes, proposing solutions, implementing corrective actions, and obtaining the concurrence of the Program Manager and the QA Manager on the appropriateness of the corrective action.

- Implementing cost effective quality improvements.
- Implementing training plans by assessing training needs, scheduling necessary training and ensuring that training is completed and documented.
- Initiating corrective actions and stop-work actions when warranted by conditions adverse to analytical quality.
- Approval of analytical data and submission of the final data to the Program Manager and Data Manager in a timely and professional manner.

## Laboratory Technician

The Laboratory Technician reports directly to his/her specific Laboratory Manager. The Laboratory Technician is responsible for:

- Being properly trained and fully knowledgeable about the SOPs required to complete the assigned work.
- Strictly adhering to SOPs.
- Identifying areas where improvement could benefit the program.
- Initiating corrective actions and stop-work actions when warranted by conditions adverse to analytical quality.
- Being familiar with the components of the project's Quality Assurance Project Plan.
- Reporting any conditions adverse to quality to the appropriate Laboratory Manager.
- Ensuring that internal chain of custody procedures are followed, and that all paperwork and forms are properly and completely maintained.
- Initiating stop-work actions when warranted by conditions adverse to analytical quality.

## Sample Control Manager and Sample Custodians

The Sample Control Manager reports to the Program Manager and is responsible for overall activities associated with sample receipt, documentation, login, preparation, storage and disposal. The Sample Custodian reports directly to the Sample Control Manager. The Sample Custodian is responsible for:

- Ensuring the integrity of project samples through all stages of the project including final archiving or other disposition.
- Logging-in, verifying chain-of-custody paperwork, and inspecting all samples for proper storage, preservation and condition.
- Maintaining all records in compliance with the Quality Assurance Project Plan.
- Advising the Sample Control Manager and the Quality Assurance Manager on issues of quality control related to sample custody procedures.
- Notifying the client of any exceptions to chain-of-custody procedures, damage to samples, and inadequate practices that jeopardize sample integrity.

## 2.2 <u>Personnel Training</u>

Personnel training and continuing education are essential elements in providing high quality analytical data. GERG provides for the selection and training of personnel so that each employee is proficient and properly trained to perform their assigned activities. Personnel selection and training procedures are explicitly stated in GERG SOP-9702.

## **3.0 QUALITY ASSURANCE OBJECTIVES**

Data quality assurance objectives specified for the TDH project are summarized in GERG standard operating procedures (SOPs) in most cases, meet or exceed these criteria. GERG procedures will be revised as necessary to meet all TDH criteria. A goal of 100% completeness is not always obtainable if, for example, no sample remains for reanalysis. Data is reported but qualified as out-of-control if no sample remains for reanalysis.

The implementation of the QA program is achieved through a team effort by the entire laboratory group. The general considerations and objectives of the overall QA/QC program are as follows:

- Sample integrity is preserved by following documented sample handling procedures relating to the preservation, custody, storage, labeling and record keeping associated with samples received by the laboratory.
- Properly approved standard analytical methods are followed. Routine analytical methods and procedures used for sample analyses are readily available and understood by all analysts using the procedures. Results generated from a method are evaluated to identify method weakness and detect needs for further analyst training.
- The analytical instrumentation is in proper working order. Instrument performance, calibration, and maintenance are documented.
- The accuracy and precision of analytical methods are recorded and maintained on a continuing basis. Accuracy and precision data are monitored using tabular formats to assess continuing performance and to detect trends. Control charts can be generated after the completion of analytical activities if required.
- Raw data is properly reduced and accurately transcribed into the proper reporting format. Various levels of data review from acquisition to the final report are incorporated to reduce the possibility of errors.

All of the above considerations are documented to validate the quality of the data.

3.1 Limits of Detection

The GERG SOPs proposed for this project have been shown in most cases to provide the required minimum limits of detection (Table 1.1). GERG procedures will be modified where necessary to add additional analytes and meet all required limits of detection. The method detection limits will be determined annually for each target compound using the EPA protocols detailed in 40 CFR Part 136, Appendix B. The method detection limit (MDL) is defined as the Student's t for 99% confidence interval times the standard deviation of at least seven replicate measurements of the same low level sample or spiked sample.

## 3.2 Precision and Accuracy Acceptance Criteria

The principal estimate of accuracy will be the recovery of spiked analytes. Program requirements for accuracy and precision criteria are summarized in Table 3.1. Some of the more volatile analytes may not meet these criteria's. Specific analytes exempted from these criteria are naphthalene, perylene, HCH's and HCB. In addition, PCB 170 is excepted due to frequent interference problems of the analyte with phthalates.

Relative percent difference (RPD) of duplicates is the principal measure of precision, as defined in QAPP Section 12.0. The required criteria for RPD are summarized in Table 3.1.

Data Quality Parameter	Method of Determination	Frequency	Required Objectives <sup>a</sup>
Accuracy • Matrix Spike	Pesticides PCB Congeners VOAs SVOAs Dioxin/Furans Trace Metals	5% of samples <sup>a</sup> ,b 5% of samples <sup>a</sup> ,b 5% of samples <sup>a</sup> ,b 5% of samples <sup>a</sup> ,b 5% of samples <sup>a</sup> ,b	40-120% Recovery 40-120% Recovery 30-150% Recovery 30-150% Recovery 40-130% Recovery 75-125% Recovery
<ul> <li><u>Precision</u></li> <li>Duplicates</li> <li>Matrix Spike Duplicates</li> </ul>		5% of samples <sup>b</sup> 5% of samples <sup>b</sup>	35% RPD <sup>c</sup> 35% RPD <sup>c</sup>

Table 3.1. QA objectives for precision and accuracy.

a - at least one per analytical batch or run sequence

b - may be waived if insufficient sample

c - relative percent difference (see QAPP Section 12.0); if concentration is less than detection limit, use half the limit of detection for calculations.

## 4.0 SAMPLING PROCEDURES

GERG is not involved in sampling. TDH will provide all samples to the laboratory with appropriate chain-of-custody or other documentation.

## **5.0 SAMPLE CUSTODY PROCEDURES**

The receiving, initial preparation, storage, tracking, archival or disposal of TDH samples are described in GERG SOP-9706 to 9712. The sample receipt date is the date that samples are received at the GERG laboratory. This date is established by the carrier or by certified mail. A diagram of the GERG sample log-in and record maintenance are shown in Figure 5.1.



Figure 5.1. Sample log-in and record maintenance.

The Sample Custodian is responsible for all aspects of sample inventory and tracking for the samples in his/her custody. The Custodian is responsible for keeping a record of all samples under his/her jurisdiction, the names of all persons having access to the samples, the movement and analyses performed (including dates and names) on the samples and the location and custodianship of samples while they were away from the primary custodian's care. After aliquoting, any remaining sample and all sample tags or labels shall be returned to the Sample Custodian to be held until indicated otherwise. The Sample Custodian is also responsible for all archiving activities.

## 6.0 CALIBRATION FREQUENCY AND PROCEDURES

All standards will be "in date" as defined in the GERG SOPs. Standard curves are used for each analyte and consist of three or more calibration points in addition to zero. The calibration correlation evaluation for linearity must meet or exceed a regression coefficient of 0.995 to be accepted as in control. Calibration is checked, at a minimum, after each ten samples as well as at the beginning and end of each analysis batch or run sequence. All analyses employ surrogate and internal standards with specific compounds detailed in the SOPs. All analyses are conducted within the established calibration range of the instrument.

## 7.0 ANALYTICAL PROCEDURES

All methods are fully described in the GERG Standard Operating Procedures. All proposed methods have been extensively intercalibrated. GERG SOPs applicable to each analysis are listed in Table 7.1. Some analyses are based on published EPA methodology.

	Item	Extraction/Purification	Instrumental Analysis
A.	Pesticides	9807, 9720, 0009	9810
B.	PCB Congener by HRGC/MS	9807, 9720, 0009	0205
C.	Quantification of Individual Aroclors	9807, 9720, 0009	9810
D.	Volatile Organic Analytes	NA	0301
E.	Semivolatile Organic Analytes	9807, 0009	EPA 8270C
F.	Dioxin and Furans	9719	9722
G.	Tissue measurements 1. % Lipids 2. % Moisture	9807 NA	9727 9415
H.	<ol> <li>Trace Metals</li> <li>Mercury</li> <li>Cadmium, Copper, Lead, Zinc</li> <li>Arsenic, Selenium</li> </ol>	0006 9408 9408	0202 In Prep* 0201

Table 7.1 Summary of GERG SOPs for the analytes of interest.

\*Based on EPA Method 6020

## 8.0 DATA REDUCTION, VALIDATION AND REPORTING

#### 8.1 Data Reduction and Validation

All sample results entering the analysis data stream are subjected to continuous validation procedures as they progress from raw data through data reduction to the final data review. The generalized validation procedure is diagrammed in Figure 8.1. The analytical group's validation process is diagrammed in Figures 8.2 and 8.3. The first level of data review validation begins with the laboratory staff. The initial data validation review identifies questionable injections or results which are outside established analytical limits (e.g., instrument calibration range) and identifies a need for re-analysis if required. After successfully passing this first level of data validation and data reduction, each analytical group initiates the second level of data validation. The data is inspected for any failure of stated QC objectives (i.e., the concentration of target analytes in the blank). If problems are identified, corrective action is initiated per the SOP. After completion of peer review, validated data are compiled and sent to the Laboratory Manager where they undergo the final data review before being entered into the database by the Data Manager. The final data review is performed by Laboratory Managers, the Deputy Program Manager, and the QA Manager after the data are entered into the database. Any errors that might occur during this process (e.g., units, conversions, formatting) are identified, returned to the Project Data Manager, corrected, and re-entered into the database.

Approved data from the final review passes to the editorial staff for report preparation. The final report is reviewed by the Program Manager who routes any corrections required to the appropriate validation level. All data which appears in the final report will have undergone three levels of data validation and two levels of data review. These validation procedures assure the completeness and integrity of project data.

#### 9.0 INTERNAL QUALITY CONTROL CHECKS

Quality control check samples and procedures include matrix spikes, laboratory spiked blanks, use of surrogate standards, procedural (method) blanks and other blanks (sampling, field, reagent and instrument), analysis of standard reference materials, use of independent standards, and calibration check standards, and detection limit determinations.

Matrix spikes (MS) are used to evaluate the effect of the sample matrix upon compounds being determined. Method blanks are used to evaluate the potential for sample contamination during preparation. Laboratory blank spikes may be used when sample availability, matrix concentration, or non-homogeneity are of concern in control monitoring.

Adequate statistical procedures are provided to monitor the precision and accuracy of the analytical data and to establish acceptable control limits. QC checks are numerous and methodology specific. The results of matrix spike sample analysis are used to demonstrate whether the laboratory method for sample preparation and analysis is working properly. The results of the MS (or MS/MSD pair) sample may be used to evaluate the accuracy (% recovery) of the analysis. The relative percent difference (RPD) determined using the concentration results of duplicate analyses (or the percent recovery for the MS/MSD pair) to evaluate precision limits

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Figure 8.1. Generalized data reduction and validation process.



Figure 8.2 Data reduction and validation process for the Organic Analytical Group.

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Figure 8.3 Data reduction and validation process for the Inorganic Analytical Group.

and is compared to requirements specified in this QAPP. Accuracy can also be evaluated based upon laboratory blank spike or SRM analyses, and these data can be compared to known concentrations.

## 9.1 Specific Requirements of this Program

GERG conforms with the following portions of 40 CFR, Part 160 that are specifically required for this program.

## Subpart B Organization and Personnel

## §160.29 Personnel

- (a) Each individual engaged in the conduct of or responsible for the supervision of a study shall have education, training, and experience, or combination thereof, to enable that individual to perform the assigned functions.
- (b) Each testing facility shall maintain a current summary of training and experience and job description for each individual engaged in or supervising the conduct of a study.
- (d) Personnel shall take necessary personal sanitation and health precautions designed to avoid contamination of test, control, and reference substances and test systems.
- (e) Personnel engaged in a study shall wear clothing appropriate for the duties they perform. Such clothing shall be changed as often as necessary to prevent microbiological, radiological, or chemical contamination of test systems and test, control, and reference substances.

## §160.31 Testing Facility Management

For each study, testing facility management shall:

- (a) Designate a study director as described in §160.33 before the study is initiated.
- (b) Replace the study director promptly if it becomes necessary to do so during the conduct of a study.
- (d) Assure that test, control, and reference substances or mixtures have been appropriately tested for identity, strength, purity, stability, and uniformity, as applicable.
- (e) Assure that personnel, resources, facilities, equipment, materials, and methodologies are available as scheduled.
- (f) Assure that personnel clearly understand the functions they are to perform.

## §160.33 Study Director

For each study, a scientist or other professional of appropriate education, training, and experience, or combination thereof, shall be identified as the study director. The study director has overall responsibility for the technical conduct of the study, as well as for the interpretation, analysis, documentation, and reporting of results, and represents the single point of study control. The study director shall assure that:

- (b) All experimental data, including observations of unanticipated responses of the test system are accurately recorded and verified.
- (c) Unforeseen circumstances that may affect the quality and integrity of the study are noted when they occur, and corrective action is taken and documented.
- (f) All raw data, documentation, protocols, specimens, and final reports are transferred to the archives during or at the close of the study.

§160.35 Quality Assurance Unit

- (a) A testing facility shall have a quality assurance unit which shall be responsible for monitoring each study to assure management that the facilities, equipment, personnel, methods, practices, records, and controls are in conformance with the regulations in this part. For any given study, the quality assurance unit shall be entirely separate from and independent of the personnel engaged in the direction and conduct of that study. The quality assurance unit shall conduct inspections and maintain records appropriate to the study.
- (b) The quality assurance unit shall:
  - (6) Review the final study report to assure that each report accurately describes the methods and standard operating procedures, and that the reported results accurately reflect the raw data of the study.

Subpart C - Facilities

§160.41 General

Each testing facility shall be of suitable size and construction to facilitate the proper conduct of studies. Testing facilities which are not located within an indoor controlled environment shall be of suitable location to facilitate the proper conduct of studies. Testing facilities shall be designed so that there is a degree of separation that will prevent any function or activity from having an adverse effect on the study.

§160.51 Specimen and Data Storage Facilities.

Space shall be provided for archives, limited to access by authorized personnel only, for the storage and retrieval of all raw data and specimens from completed studies.

Subpart D - Equipment

§160.61 Equipment Design

Equipment used in the generation, measurement, or assessment of data and equipment used for facility environmental control shall be of appropriate design and adequate capacity to function according to the protocol and shall be suitably located for operation, inspection, cleaning, and maintenance.

## §160.63 Maintenance and Calibration of Equipment

- (a) Equipment shall be adequately inspected, cleaned, and maintained. Equipment used for the generation, measurement, or assessment of data shall be adequately tested, calibrated, and/or standardized.
- (b) The written standard operating procedures required under §160.81(b)(11) shall set forth in sufficient detail the methods, materials, and schedules to be used in the routine inspection, cleaning, maintenance, testing, calibration, and/or standardization of equipment, and shall specify, when appropriate, remedial action to be taken in the event of failure or malfunction of equipment. The written standard operating procedures shall designate the person responsible for the performance of each operation.
- (c) Written records shall be maintained of all inspection, maintenance, testing, calibrating, and/or standardizing operations. These records, containing the dates of the operations, shall describe whether the maintenance operations were routine and followed the written standard operating procedures. Written records shall be kept of non-routine repairs performed on equipment as a result of failure and malfunction. Such records shall document the nature of the defect, how and when the defect was discovered, and any remedial action taken in response to the defect.

Subpart E - Testing Facilities Operation

- §160.81 Standard Operating Procedures
- (a) A testing facility shall have standard operating procedures in writing setting forth study methods that management is satisfied are adequate to insure the quality and integrity of the data generated in the course of a study. All deviations in a study from standard operating procedures shall be authorized by the study director and shall be documented in the raw data. Significant changes in established standard operating procedures shall be properly authorized in writing by management.
- (b) Standard operating procedures shall be established for, but not limited to, the following:
  - (3) Receipt, identification, storage, handling, mixing, and method of sampling of the test, control, and reference substances.
  - (5) Laboratory or other tests.
  - (11) Maintenance and calibration of equipment.
- (c) Each laboratory or other study area shall have immediately available manuals and standard operating procedures relative to the laboratory or field procedures being performed. Published literature may be used as a supplement to standard operating procedures.

§160.83 Reagents and Solutions

All reagents and solutions in the laboratory areas shall be labeled to indicate identity, titer or concentration, storage requirements, and expiration date. Deteriorated or outdated reagents and solutions shall not be used.

Subpart G - Protocol for and Conduct of a Study

§160.130 Conduct of a Study

(e) All data generated during the conduct of a study, except those that are generated by automated data collection systems, shall be recorded directly, promptly, and legibly in ink. All data entries shall be dated on the day of entry and signed or initialed by the person entering the data. Any change in entries shall be made so as not to obscure the original entry, shall indicate the reason for such change, and shall be dated and signed or identified at the time of the change. In automated data collection systems, the individual responsible for direct data input shall be identified at the time of data input. Any change in automated data entries shall be made so as not to obscure the original entry, shall indicate the reason for change, shall be dated, and the responsible individual shall be identified.

Subpart J - Records and Reports

§160.185 Reporting of Study Results

- (a) A final report shall be prepared for each study and shall include, but not necessarily be limited to, the following:
  - (6) A description of the methods used.

## 9.2 <u>Quality Control for Analytical Standards</u>

All standards are certified and/or verified against NIST or other Standard Reference Materials when available.

## 9.3 <u>Minimum Criteria for an Out-of-Control Condition</u>

A laboratory process for a particular analyte is considered out of statistical control whenever, as a minimum, any one of the following conditions is demonstrated by a control chart monitoring that analyte.

- (1) Any one point is outside of the control limits.
- (2) Any three consecutive points are outside the  $\pm$  two standard deviation warning limits.
- (3) Any six consecutive points are such that each point is larger (smaller) than its immediate predecessor.
- (4) Any obvious cyclic pattern is seen in the points.

## 9.4 <u>Reactions to Out-of-Control Statistical Conditions on Control Samples</u>

Out of control events are responded to in a number of ways as outlined in Section 13.0, Corrective Action.

#### 9.5 Administration of the Control Charts

Control charts are used to monitor all analytical streams related to this project. Control samples are run with each batch of samples. The control charts are generated by designated laboratory staff, and distributed to the QA Manager, the Deputy Program Manager, and the Program Manager. Visual examination of QC sample data on a daily basis by the instrument operator and the Laboratory Manager highlights any immediate QC problems. QC limits can be updated periodically when sufficient additional data have been generated.

## **10.0 PERFORMANCE AND SYSTEM AUDITS**

TDH may submit blank and/or control samples to provide an independent evaluation of GERG program. TDH staff may audit GERG operations at any time provided a fourteen calendar day notice is provided. GERG participates in intercomparison exercises organized by NIST and NRCC.

## **11.0 PREVENTIVE MAINTENANCE**

Maintenance logs are kept for each instrument and include documentation of column changes, detector cleaning, and parts replacement. Past calibration reports are also maintained. Spare parts and necessary maintenance items are kept in stock at all times to minimize instrument down time. All instruments are calibrated prior to or during use and must meet SOP acceptance criteria or the instrument is cleaned and/or further remedial action is taken. Each Laboratory Manager is responsible for scheduling maintenance, assigning qualified personnel to maintenance tasks and recording all maintenance activities.

## **12.0 ROUTINE PROCEDURES TO ASSESS DATA QUALITY**

Data quality is routinely assessed for precision, accuracy, and completeness. Method detection limits are also calculated annually to confirm compliance with method detection limit criteria.

## 12.1 Precision

Relative Percent Difference (RPD) is a measure of precision and can be calculated from the concentrations of field duplicates or laboratory duplicates, and from the percent recovery of matrix spike/matrix spike duplicates:

$$RPD = \frac{(C_1 - C_2) \times 100\%}{(C_1 + C_2)/2}$$

where: RPD = relative percent difference

 $C_1$  = larger of the two observed values

 $C_2$  = smaller of the two observed values.

When field or laboratory duplicates are used, concentrations less than detection limits are given the value of half the detection limit for this calculation.

## 12.2 Accuracy

Laboratory blank spikes and matrix spikes can be used to determine the accuracy of an analysis in the laboratory. For laboratory blank spikes or sample matrix spikes, the following formula is used to determine percent recovery, which is then compared to control limits based upon historical data:

$$\%R = 100\% x \left(\frac{S - U}{C_{sa}}\right)$$

where:  $\[mm] \[mm] \[mm] R = \]$  percent recovery  $\[mm] \[mm] S = \]$  measured concentration in spiked aliquot  $\[mm] U = \]$  measured concentration in unspiked aliquot  $\[mm] \[mm] \[mm] C_{sa} = \]$  actual concentration of spike added

Standard reference material (SRM) with certified analyte concentrations can also be used to determine the relative accuracy of the method. Laboratory blank spikes can also be used with the following equation to determine the accuracy of an analysis in the laboratory.

When a standard reference material (SRM) or a spiked method blank is used:

$$\%R = 100\% x \left(\frac{C_{m}}{C_{srm}}\right)$$

where:  $\[mm] R = \]$  percent recovery  $C_m = \]$  measured concentration of SRM  $C_{srm} = \]$  median concentration of the SRM

These results are then compared to control limits specified in the appropriate SRM certificate or known concentrations for laboratory blank spikes.

12.3 <u>Completeness</u>

Completeness is defined as follows for all measurements:

$$\%C = 100\% x \left(\frac{V}{n}\right)$$

where: %C = percent completeness V = number of measurements judged valid n = total number of measurements

#### 12.4 <u>Method Detection Limit (MDL)</u>

MDL is defined as follows for all measurements:

$$MDL = t_{(n-1, 1-)} = 0.99) \times S$$

where: MDL = method detection limit s = standard deviation of the replicate analyses  $t_{(n-1, 1-\alpha = 0.99)} =$  students' t-value for a one-sided 99% confidence level and a standard deviation estimate with n-1 degrees of freedom.

#### **13.0 CORRECTIVE ACTION**

Whenever a quality control sample does not meet stated project goals the procedure is reviewed to ascertain the cause of the error. If errors are discovered the analysis is repeated from the point of the error. If no error can be pinpointed the analysis is repeated. When appropriate, corrective action is applied to all samples analyzed concurrently with the sample that initiated the action. It is not sufficient to simply flag quality control errors; corrective action must be taken and documented using a Sample Action Request Form. All QC data, including SRMs, calibration checks, duplicates, laboratory blank spikes, and MS/MSD results are inspected to determine if a system-wide change is present.

Corrective action constitutes a variety of responses to noncompliance with QC requirements. Responses include replacement of GC columns, cleaning of detectors, recalibration, re-extraction of samples, and repair or replacement of parts and/or instruments as necessary. If an unacceptable "method blank" is present, analyses for the related extraction batch cease until samples are reprocessed and an acceptable method blank is produced. If the response of the calibration check standard exceeds the QC criteria, a second calibration check is analyzed. If the results are still in non-compliance a recalibration is performed. These criteria are monitored daily by the Laboratory Manager. As defined in the SOPs, the retention times for each analyte in a sample must be within the stipulated time of that observed during the most recent acceptable calibration or remedial action is initiated including leak testing and column replacement, if necessary.

Non-compliance of calibration checks or spiked blanks causes immediate cessation of analysis. Whether instrument recalibration (calibration check) or reevaluation of all sample results is necessary will be decided by a conference of the Laboratory Manager and the Program Manager. In all cases, the stated criteria must be met. These criteria may also be independently monitored by the Quality Assurance Manager to insure that QC data are being properly acquired, tabulated, and compiled.

If no errors can be found and the quality control failure appears to indicate that the quality control failure impacted a small number of the analytes within the scan, the TDH Quality Assurance Officer will be contacted for a decision. Any corrective action must be applied to all samples analyzed concurrently with the sample that initiated the action.

#### 14.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

The Quality Assurance Manager is the senior management person responsible for all QA policies at GERG. The QA Manager is not part of the analytical process and reports to the Director of GERG. The QA Manager prepares an annual report for the Director and the Senior Associate Director and also provides verbal and written reports on an as needed basis.

Changes in the SOPs must have final approval of the QA Manager. For the GERG Quality Assurance Management Plan (QAMP), the Generic Quality Assurance Manual for Laboratory Staff and Operations (GQAM), or for any QAPP, the signature of the Director of GERG is required as well.