

QUALITY ASSURANCE PROJECT PLAN

**ORGANOCHLORINE AND METAL
CONTAMINANT LEVELS IN
HUDSON RIVER, NEW YORK
REPTILES AND AMPHIBIANS**

**HUDSON RIVER NATURAL RESOURCE
DAMAGE ASSESSMENT**

HUDSON RIVER NATURAL RESOURCE TRUSTEES

STATE OF NEW YORK

U.S. DEPARTMENT OF COMMERCE

U.S. DEPARTMENT OF THE INTERIOR

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Organochlorine and Metal Contaminant Levels in Hudson River, New York Reptiles and Amphibians

Prepared for

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Environmental Conservation
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REPTILES AND AMPHIBIANS
HUDSON RIVER, NEW YORK

APPROVALS:

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9 / 8 / 98
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NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION
BUREAU OF HABITAT



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1.0 PROJECT DESCRIPTION

This project will involve the collection and contaminant analysis of snapping turtles (*Chelydra serpentina*) and green frogs (*Rana clamitans*). Sampling of both frogs and turtles will be done in three geographically distinct areas of the Hudson River in order to obtain information about the distribution of contaminant concentrations. The sampling will be performed during 1998.

1.1 INTRODUCTION

In 1987, New York State passed Section 11-0306 of the Environmental Conservation Law, known as the Hudson River Estuary Management Act. This law has resulted in the development of the Hudson River Estuary Management Plan (HREMP) which characterizes the priority problems affecting the Hudson River and identifies specific objectives to improve the estuarine ecosystem. This project has been undertaken under the auspices of this plan. The project supports the following planning objectives:

HREMP Objective

- LR-EP-8 Manage the physical and chemical properties of the estuary's water column and sediments (substrate) to ensure optimal production of the estuary's living resources.
- LR-EP-9 Reduce chemical contaminant levels to concentrations that will not impair the successful survival, reproduction and growth of sensitive species nor impair secondary consumers of fish shellfish and wildlife.

HEP Objective

- Goal H-2 Restore and maintain an ecosystem which supports an optimum diversity of living resources on a sustained basis.

1.2 INVESTIGATION OBJECTIVES

The objective of the Organochlorine and Metal Contaminant Levels in Hudson River Reptiles and Amphibians study is to provide the New York State and federal Natural Resource Trustees with body burden information to be used for analysis of contaminant caused injury to selected reptiles and amphibians within the Hudson River ecosystem (see Figure 1.1). The specific species that will be sampled are the green frog, *Rana clamitans*, and snapping turtle, *Chelydra serpentina*. The contaminants of concern are PCBs, other organochlorines, and metals, including cadmium, mercury and lead. Sampling of both frogs and turtles will be done in three geographically distinct areas of the Hudson River in order to obtain information about the distribution of contaminant related injuries.

1.3 DATA QUALITY OBJECTIVES (DQO)

The data quality objectives (DQOs) for the project include the collection, shipment, and analysis of sufficient samples to fulfill the Study Objectives identified in Section 1.2. Since the data may be used to substantiate Natural Resource Damage claims, chemical analysis of the tissue samples will use methods which meet EPA DQO Level IV or V. Level IV analyses require EPA Contract Laboratory Program (CLP) routine analytical services with rigorous quality assurance/quality control protocols and documentation. DQO Level V analyses are laboratory methods with rigorous quality assurance/quality control protocols and documentation that are not CLP routine analytical services. The DQOs for the specimen measurements, composite sample preparation, and gut contents will use methods which are equivalent to EPA DQO Level I. Level I analyses are field analyses using portable instruments, in this case portable scales and measuring rules.

2.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

2.1 PROJECT ORGANIZATION

The project management organization provides clear lines of authority and a control structure to support this study. The structure provides:

1. clearly identified lines of communication
2. management of key technical resources
3. provisions to ensure the health and safety of site workers and the public
4. project quality assurance and quality control

The organizational structure for the project team is shown in Figure 2.1.

2.2 SUBCONTRACTORS

A herpetologist has been retained to perform specimen collection and documentation of pertinent environmental conditions relative to the target species. A biomedical waste contractor for disposal of biological waste and a chemical waste contractor for disposal of spent decontamination chemicals have also been retained. Overall management and coordination and review of subcontractors' activities will be provided.

The analytical laboratory is a subcontractor to the New York State Department of Environmental Conservation (NYSDEC). The NYSDEC laboratory at Hale Creek Field Station will perform the metals analyses.

3.0 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

3.1 FIELD INVESTIGATIONS

Fifteen (15) adult snapping turtles and thirty (30) adult green frogs will be collected, five (5) turtles and ten (10) frogs at each of three locations.

Suitable habitats have been established as collection sites through consultation with the Project Manager in:

1. the upper Hudson, from Hudson Falls to the Troy Dam - Coveville
2. the mid-Hudson, from the Troy Dam to Catskill - Stockport Creek
3. the lower Hudson, from Catskill to the Tappan Zee Bridge - Vanderburgh Cove

See figures 4.1, 4.2, and 4.3.

Capture techniques will be determined by the contractor.

Each specimen will be tagged and assigned a unique identification number as soon as it is collected. The specimens will be maintained alive until samples are prepared.

New York State Amphibian & Reptile Atlas records and instructions for their use will be supplied by the Project Manager, and must be completed for each location at which collection efforts are made.

3.2 SAMPLE COLLECTION

The media being sampled and an estimate of the numbers of samples being collected are summarized in Table 7-1. In addition to the project samples, requirements and procedures for the collection of field QA/QC samples for the Site will be adhered to as discussed below. The frequency and type of QA/QC samples being collected is summarized in Table 3-1.

3.2.1 Duplicate Samples

Tissue samples will be collected from two species, green frog and snapping turtle, from three separate geographical locations. Ten frogs and five turtles will be collected from each geographical sample location. The sampling events at the three locations will take place on different days. The NYSDEC has indicated a duplicate sample requirement of 20 %. Duplicates will be prepared from the homogenized tissue samples. Two duplicates of frog muscle will be required per site, and one duplicate each for turtle muscle, turtle adipose tissue, turtle liver, and turtle kidney will be required per site.

The analysis results of the duplicate samples will be used as a check of the precision of the field sampling event. A duplicate sample will be prepared from the homogenized tissue sample. The entire tissue sample will be ground by the NYSDEC laboratory. Duplicates will be prepared from this homogenized tissue. They will be analyzed for the same parameters as the original sample.

3.2.2 Matrix Spikes

Matrix spike samples will be collected for the organic chemical analyses. One additional duplicate sample will be prepared from the homogenized tissue by the laboratory for matrix spikes and matrix spike duplicates (MS/MSD).

3.2.3 Preparation of Blanks

Tissue blanks will be prepared by the analytical laboratory performing the chemical analysis at the same time that the samples are prepared for analysis. The results from the tissue blank will be reported with the other analytical results.

Table 3-1 QA/QC Sample Summary Organochlorine and Metal Contaminant Levels in Hudson River Reptiles and Amphibians, Hudson River, New York				
Media	Samples	Duplicates		Matrix Spike
		Metals	Organics	
Muscle <i>R. clamitans</i>	30	6	6	3
Muscle <i>C. serpentina</i>	15	3	3	3
Adipose <i>C. serpentina</i>	15	3	3	3
Liver <i>C. serpentina</i>	15	3	3	3
Kidney <i>C. serpentina</i>	15	3	3	3
Total	90	18	18	15
Note: 20% QA/QC samples				

3.2.4 Trip Blanks

Trip blanks are not required for these types of analysis.

3.2.5 Field Blanks

Field blanks are not used for this type of analysis. Decontaminated, disposable scalpels will be used to obtain tissue samples.

3.2.6 Storage Blanks

Storage blanks are not required for these types of analysis.

3.3 SAMPLING TECHNIQUES

Sampling techniques and procedures are discussed in Section 4.0.

3.4 SAMPLE REPRESENTATIVENESS AND COMPLETENESS

3.4.1 Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, or an environmental condition. Representativeness is a qualitative parameter concerned with the proper design of the sampling program. The representativeness criterion is best satisfied by making certain that sampling locations are selected properly, sampling techniques are adequately described and adhered to, and a sufficient number of samples are collected. The sampling program has been designed by the NYSDEC to meet its research goals based on its knowledge of the study areas, the resources being studied, and previous research of a similar nature.

3.4.2 Completeness

With respect to data collection, completeness is a measure of the amount of valid data obtained compared to the amount that was specified or expected to be obtained under normal conditions. The measure is usually expressed as a percentage. If the data are complete, that is, if the valid data are equal in quantity to the amount specified to be collected, then it is referred to as 100 percent complete. Occasionally, completeness is something less than 100 percent due to difficulties in collection and analysis of environmental samples. In such cases, the extent of completeness must be viewed on a relative basis because the required amount of valid data anticipated or specified prior to the sampling episodes may not accurately define the amount of data necessary to render a correct decision. Given these circumstances, a completeness of 90 percent is generally acceptable and will be the standard applied for this project since the goal is to accept all sizes of snapping turtle greater than 3 lbs. In weight collected in the traps, smaller individuals may not provide sufficient volume of tissue for all of the chemical analyses. The completeness goal may need to be reassessed following the results of the field collection effort. Completeness with respect to specific measurement systems is discussed in more detail in Section 12.0.

3.4.3 Comparability

Comparability is a qualitative parameter expressing the confidence with which one data set can be compared with another. Sample data should be comparable with other measurement data for similar samples and sample conditions. This goal is achieved through using standard techniques to collect and analyze representative samples and reporting analytical results in appropriate units. Comparability is limited to the parameters of precision, accuracy, representativeness, and completeness because it is only when these parameters are known that data sets can be compared with confidence.

3.5 LABORATORY QUALITY ASSURANCE AND QUALITY CONTROL

Integrity and usefulness of the analytical results constitute the primary objectives of the analytical quality assurance and quality control processes. The primary objective of the analytical laboratory with respect to QA/QC is to achieve the acceptance criteria for a given analytical method when a sample is analyzed. The quality of the data is indicated by the parameters of representativeness, precision, accuracy, completeness, and comparability.

3.5.1 Representativeness of Analytical Measurements

Representativeness of analytical measurements will be based on a comparison of analytical results for duplicate samples.

3.5.2 Completeness of Analytical Measurements

The goal for completeness of analytical results is 100 percent. However, the objectives of this project can be addressed with a 90 percent completeness for the laboratory analyses. As noted above, there is a risk that if a number of small turtle specimens are collected, the 90 % completeness goal for the analysis of turtle tissue may not be realized.

3.5.3 Accuracy and Precision of Analytical Measurements

Accuracy and precision of analytical results will be evaluated based on criteria specified in the analytical methods used. Such criteria will include, but not be limited to, blank results, matrix spike recoveries, surrogate percent recovery values, correlation coefficients, and detection limits. The NYSDEC will determine if these parameters have been met.

3.5.4 Comparability of Analytical Measurements

The analytical results are expected to be comparable between the three specimen collections locations because the same methods will be used. The results are also expected to be compared to historical data, where available, collected at this Site.

4.0 FIELD INVESTIGATION AND SAMPLING PROCEDURES

4.1 FIELD COLLECTION

Fifteen (15) adult snapping turtles and thirty (30) adult green frogs will be collected, five (5) turtles and ten (10) frogs at each of three locations in the Hudson River (See Figure 4.1). The capture techniques will be as permitted in the New York State Biological Collectors Permit. Specimen collection will be performed by a herpetologist and the Research Supervisor. The turtles will be collected by trap and the green frogs will be collected by hand or with hand nets. The herpetologist will make written field observations during sample collection and will assign the specimen numbers. The specimens will be delivered alive and in good condition for tissue sample preparation.

Suitable habitats have been established, through consultation with the NYSDEC Project Manager, as collection sites in:

1. the upper Hudson, from Hudson Falls to the Troy Dam - Coveville
2. the mid-Hudson, from the Troy Dam to Catskill - Stockport Creek
3. the lower Hudson, from Catskill to the Tappan Zee Bridge - Vanderburgh Cove

See Figures 4.2, 4.3, and 4.4. Data that characterizes the sample location will be entered in the field as described in Section 4.3.5. In addition, photocopies of topographic maps (7½ minute quads) indicating the precise location of collections will be provided to the Project Manager.

Turtles will be trapped in 30-inch hoop traps with 1-inch mesh. Traps will be staked and baited with sardines in soy oil. Traps will be set out overnight. Baits will be changed after 24 hours, if sufficient specimens have not been collected. The number of traps set will depend on the sample area but 8 traps are estimated per collection site. Turtles larger than 3 lbs are

estimated to provide sufficient tissue for analysis. Frogs will be caught by hand or with hand nets, either from shore or from the water.

Each specimen will be tagged and assigned a unique identification number as soon as it is collected. Pre-printed, numbered labels will be used on the tags. The specimens will be weighed and measured in the field. Measurements will be reported to the nearest gram or one-quarter ounce for weight and nearest two millimeters or one quarter inch for length. Length measurements for the frogs will be taken from snout to vent. Carapace length will be recorded for turtles. The specimen number and specimen data will be recorded on field data sheets as described in Section 4.3.5. The specimens will be maintained alive until delivery to the laboratory for sample preparation. Animals will be rinsed with clean water in the field to dislodge sediment or other foreign material from their skin. The samples will be transported from the field to the laboratory under Chain-of-Custody as described in Section 5.0.

All capture equipment (nets, traps, pails, other containers, *et cetera*) will be thoroughly washed with soap and water and then rinsed with clean water before the operation is moved between collection sites.

4.2 SAMPLING PROTOCOL

The smooth functioning of the sample collection and analysis process is based on a clear understanding of the relationship between the field sampling team and the field and laboratory analysis teams and the tasks and responsibilities of each. Adherence to the protocol presented here is essential to minimize problems in maintaining data quality and integrity.

The field and sample preparation personnel will carry out the following protocols prior to initiating any field work or laboratory work involving the collection of samples:

1. Schedule the specimen collection with the tissue preparation laboratory and the tissue analyses with the analytical laboratory or laboratories designated by the NYSDEC.
2. Notify the NYSDEC Project Manager
3. Determine the type, size, and quantity of sample containers required, and the maximum holding times for each sample.
4. Identify all of the tasks, determine the equipment required for each, and make sure that it is available.
5. Read and understand all Standard Operating Procedure (SOPs) and Sampling descriptions for the necessary tasks.
6. Ensure that all measuring equipment is in proper repair, properly calibrated, supplied with fully charged batteries and replacement batteries, and that each has received the appropriate quality control checks.
7. Establish next available specimen and sample ID numbers for sampling event.
8. Obtain sample containers, trip blanks from the laboratory.
9. Obtain Chain-of-Custody Forms, chain-of-custody tapes, document envelopes, shipping forms and sealing tape.
10. If samples are to be shipped by overnight carrier, confirm the location of the carrier's service office and their hours of operation.
11. Determine that all sampling equipment and accessories have been appropriately decontaminated.

4.3 SAMPLING PROCEDURES

Specimens will be delivered alive to the tissue preparation laboratory by the herpetologist. Specimens will be maintained alive in the laboratory until they are processed for tissue preparation. The specimens will be logged into a bound laboratory sample log book by date, time, and specimen number. The collection of tissue samples will be performed as soon as possible after arrival of the specimens to the laboratory. Specimens will be processed within 24 hours of arrival to the laboratory. Specimens will be held in coolers, maintained moist, and chilled to slow metabolism.

The following data will be recorded on a *Specimen Collection Record* for each turtle or frog from which samples are prepared:

1. Tag number
2. Date and time collected
3. Species
4. Sex
5. Length and weight (carapace length for turtles; snout-vent length for frogs)
6. Method of collection
7. Location of collection (water body and distance and direction from nearest prominent and identifiable landmark)
8. Habitat (describe: substrate, dominant vegetation, water depth)
9. Gut contents (food items should be identified to lowest practical taxon and they should be counted)
10. Observed pathological lesions (if any)
11. Comments
12. Name of collector or collectors.

Data from field books and field collection sheets will be entered into the Specimen Collection Record. The data recorded on laboratory bench sheets will be added to this record after sample preparation. See section 4.3.5 for description of the sample documentation.

4.3.1 Tissue Sample Preparation

Specimens will be humanely dispatched by cooling them to slow reflexes and then pithing prior to examination. The specimen will then be rinsed and placed on a stainless steel tray. A necropsy will be performed on all specimens following guidelines agreed to by the NYSDEC. Results of the external and internal visual examination will be entered on a laboratory bench sheet. Laboratory bench sheets will be pre-numbered and retained in a looseleaf notebook. The specimen will be rinsed with distilled water prior to making any incisions. A new pair of clean, powder free, latex gloves will be worn for each specimen. Similarly, all dissection equipment (scalpels, pins, forceps, pans, *et cetera*) will be washed with soap and water, rinsed with distilled water, and then rinsed with hexane or acetone after each specimen. Tissue samples for chemical analysis will be excised using a new disposable scalpel that has been cleaned with hexane or acetone prior to use. Tissue will be placed in the hexane or acetone rinsed disposable weigh pans. A new weigh pan will be used for each type of tissue from each organism and the weigh pans will be discarded after use. Weights will be recorded to the nearest 0.5 gm.

Gut contents will be removed placed in a clean glass container, and preserved in formalin or denatured ethanol for later sorting, and identification to the lowest practical taxon. Each tissue sample will be immediately placed on ice and then frozen to -20°C as soon thereafter as is practical.

For the turtles:

- All adipose tissue will be dissected from each carcass, assembled into a composite sample and weighed. The weight will be recorded on the laboratory bench sheet. Each sample will be placed in a laboratory cleaned glass jar and labeled as described in Section 4.4.
- Kidneys and the liver will be dissected and weighed. Care will be taken to avoid the inclusion of the gall bladder in liver tissue.

- Muscle tissue will be taken from both hind limbs. If this does not provide enough material, additional muscle tissue will be taken from the forelimbs.

The sample weight will be recorded on the laboratory bench sheet. Each sample will be placed in a laboratory cleaned glass jar and labeled as described in Section 4.4 and frozen.

For the frogs, only muscle tissue samples are required. The muscle tissue will be dissected from the hind limbs and weighed. The weight will be recorded on the laboratory bench sheet. If sufficient material is not obtained from the hind limbs, additional muscle tissue will be collected from the forelimbs. Each sample will be placed in a laboratory cleaned glass jar and labeled as described in Section 4.4 and frozen.

4.3.2 Scheduling

All sampling must be scheduled in advance. The NYSDEC Project Manager will be provided with a schedule of the sampling events.

The Project Manager will contact the laboratory not less than ten days prior to the collection of samples. The Project Manager will advise the laboratory of the following information:

1. analyses to be performed
2. media to be sampled
3. sample containers and preservatives needed
4. trip, field, and storage blank requirements
5. shipping and receiving requirements

The Project Manager will advise the laboratory in advance if weekend or holiday pickup or delivery is required. The contract laboratory will send the sample containers according to the shipping requirements, and not less three days prior to the sampling event.

To the extent appropriate, sample labels and numbers, documentation, chain-of-custody and traffic reports will be filled out before sampling begins.

4.3.3 *Sample Containers*

As indicated at Section 3.2 of this QAPP, samples, where appropriate, will be collected in laboratory cleaned sample glassware where Data Quality Objectives (DQO) Level 4, or 5 analyses are required. Sample bottle requirements specific to organic and inorganic analyses are shown in Table 4-1.

Table 4-1 Sampling and Analysis Requirements Organochlorine and Metal Contaminant Levels in Hudson River Reptiles and Amphibians Hudson River, New York				
Analysis	Required Volume ¹	Container Type	Preservation	Maximum Holding Times
PCBs, Organochlorines, and pesticides	20 g ²	wide mouth amber glass jars	Freezing, -20°C	N/A if frozen
Metals - Hg, Cd, Pb	3 g	wide mouth glass jars	Freezing, -20°C	N/A if frozen
Gut contents	All per specimen	wide mouth glass jars	Formalin or 70% ethanol	None
¹ For turtle adipose tissue, and organs entire mass per specimen will be collected, to be divided equally for metals and PCB-Pesticide Analyses after grinding. ² Analysis will be performed on samples less than 20 g but detection limits will be higher.				

4.3.4 *Preservation and Storage*

Proper preservation and storage of sample containers is required to maintain sample quality. Samples shall be preserved as indicated in Table 4-1. Tissue samples shall be stored and shipped frozen to -20° Celsius from the time of preparation to the point of analysis. Gut contents will be preserved with formalin or ethanol and stored at room temperature. The appropriate Chain-of-Custody documentation will be maintained at all times.

There are two types of preservation requirements indicated in Table 4-1: temperature and chemical. To confirm temperature maintenance during field storage, a thermometer will be placed in the laboratory freezer to confirm the temperature. This temperature will be read and recorded in the appropriate laboratory log book each day that samples are being stored and that personnel are present or a minimum of three times per week.

No special monitoring is required for the chemical preservation of the gut contents.

4.3.5 Shipping

Specimens will be transferred directly from subcontractor personnel to personnel at the tissue preparation laboratory. Specimens will be transported in ventilated containers and cooled to maintain them alive and in good condition.

Samples shall be packaged to prevent damage to the sample containers, sample labels, and cooler seals. Samples will be shipped in insulated containers. "Blue ice" or dry ice coolant will be used to maintain the samples frozen during shipment. Coolers will be labeled to indicate that contents need to be maintained frozen. Samples shall be shipped by courier or overnight express carrier and delivered to the laboratory within 24 hours of dispatch from the Site. The laboratory shall be notified by phone in advance of shipment to assure that the laboratory staff will be present to receive frozen samples.

4.4 DOCUMENTATION

4.4.1 Field Documentation

Dedicated field books will be used for field collections. The field books will be used to record sampling location, date, and time, collection data needed to document the collection methods as required for the New York State Collectors permit, the environmental data required for the Specimen Collection Record (See Appendix A), tag numbers for specimens, and notes concerning the sampling event. *New York State Amphibian & Reptile Atlas* records

and instructions for their use will be supplied by the Project Manager; they must be completed for each location at which collection efforts are made. Each specimen will be tagged and assigned a unique identification number as soon as it is collected. The label information is described in Section 4.4.2 and sample labels are shown in Appendix A.

The specimens will be maintained alive until samples are prepared. Length and weight will be recorded as soon as possible after collection and before freezing. Other data should be recorded in the field upon collection. The Project Manager will provide forms for this purpose. In addition, photocopies of topographic maps (7½ minute quads) indicating the precise location of collections must be provided to the Project Manager.

4.4.2 Specimen and Sample Processing Documentation

All specimens received and samples shipped or archived in the tissue preparation laboratory during this project will be logged into the laboratory log book maintained in a bound notebook. This log will record the unique sample or specimen identification number, log-in date, log-out date, type of sample and disposition.

A pre-numbered sample label will be affixed to each specimen or sample. The label will include the following information:

1. Project: Hudson River Reptiles and Amphibians
2. Sampling location identification: Predetermined sampling location or new location, as appropriate (specimen tags only).
3. Serial No.: 000001 et. seq.
4. Sample Media: specimen or tissue type
5. Date:
6. Time:
7. Parameters to be analyzed:
8. Preservative:
9. Initials of sampler:

The following data will be recorded on a *Specimen Collection Record* for each turtle or frog from which samples are prepared:

1. Tag number

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2. Date and time collected
3. Species
4. Sex
5. Length and weight (carapace length for turtles; snout-vent length for frogs)
6. Method of collection
7. Location of collection (water body and distance and direction from nearest prominent and identifiable landmark)
8. Habitat (describe: substrate, dominant vegetation, water depth)
9. Gut contents (food items should be identified to lowest practical taxon and they should be counted)
10. Observed pathological lesions (if any)
11. Comments
12. Name of collector or collectors.

See Appendix A. The information in the specimen collection record will be linked to the electronic data file for the tissue analysis.

Laboratory bench sheets will be used to record data generated in the tissue preparation laboratory. The bench sheet pages will be pre-numbered and maintained in a looseleaf notebook. For this project bench sheets will be used to record the following information for each specimen:

- Necropsy Record
- Tissue samples taken and sample weights
- Gut Contents Identification

Data from these bench sheets will be entered into the electronic data base for this project. Data entries will be proof read by a person who did not perform the data entry.

Calibration books for scales and a daily freezer log will be maintained for this project. Copies of the freezer log pages will be provided to the NYSDEC and copies of the relevant calibration book pages will be provided, if requested.

Specimens and samples will be handled under Chain-of-Custody procedures as described in Section 5.0. Copies of the Chain-of-Custody documents will be provided to the NYSDEC Project Manager.

5.0 SAMPLE CUSTODY

Proper documentation of samples is essential to quality assurance. Each sample submitted for analysis must be accompanied by proper information and forms to ensure timely, correct and complete analysis for parameters requested. Such documentation is necessary to support subsequent legal use. Two necessary forms are 1) the Chain-of-Custody Form and 2) the Lab Sample Identification Label.

5.1 SAMPLE IDENTIFICATION

Each specimen will be identified, labeled and recorded at the time it is collected in the field as described in Section 4.1 and each tissue sample container will be labeled when it is filled in the tissue preparation laboratory. A pre-numbered sample label will be affixed to each specimen or sample. The label will include the following information:

1. Project: Hudson River Reptiles and Amphibians
2. Sampling location identification: Predetermined sampling location or new location, as appropriate (specimen tags only).
3. Serial No.: 000001 et. seq.
4. Sample Media: specimen or tissue type
5. Date:
6. Time:
7. Parameters to be analyzed:
8. Preservative:
9. Initials of sampler:

All of the information necessary to identify the sample and the sampling event will be recorded in the appropriate field notebook, field sheet, bench sheet, or laboratory log book. That information will include not only the information on the sampling label, but will also identify which samples are blanks and which are duplicates.

Once collected, preserved and labeled, the handling of the sample is governed by the chain-of-custody standard operating procedure (SOP-007) which is included in Appendix B. To comply with NYSDEC requirements, signatories to the Chain-of-Custody form must also print their signatures.

5.2 CHAIN-OF-CUSTODY PROCEDURES

The purpose of chain-of-custody procedures is to document the possession of the sample from the time of collection to the time of analysis. The objective is to maintain the integrity of the sample.

The person collecting the samples is the individual who has custody of the samples until such time as they are transferred or dispatched. The NYSDEC QA Officer determines whether the proper custody procedures have been observed and whether additional samples shall be taken if custody procedures have been broken.

The Chain-of-Custody Form shall accompany the specimens the field collection location to the tissue preparation laboratory. A second Chain-of-Custody form shall accompany sample containers from the time of sample container preparation until they are logged in at the analytical laboratory. The transfer of possession from one individual to the next is documented by signatures relinquishing and receiving possession with hour and date of the time of signing. The Chain-of-Custody Form shall include:

1. project identification
2. sampling site location
3. Serial No.
4. sample media
5. date of sample collection
6. time of sample collection
7. sample collector
8. sample description (type and quantity)

9. analyses to be performed
10. chain-of-custody seal numbers

For specimen transport, the back copy of the Chain-of-Custody Form will be detached retained by the subcontractor and filed with the sampling field notebook. The original and remaining copies will be placed in a clear plastic envelope following relinquishment endorsement and taped to the shipping container. A copy of the white original will be returned to the Subcontractor. For the tissue samples, the back copy of the Chain-of-Custody Form will be detached and retained and filed with the sample logbook. The original and remaining copies will be placed in a clear plastic envelope following relinquishment endorsement by the transporter and taped to the shipping cooler. A copy of the Chain-of-Custody form to be used on this project is provided in the SOP-007-1. Shipping containers and coolers will be sealed with a Chain of Custody tapes applied so that they must be broken to remove the contents of the shipping container.

5.3 SHIPPING CONTAINERS

Samples shall be frozen prior to shipping to maintain the temperature at -20 °C. The shipping container will be cooled with "blue ice" or similar dry cooling methods. The individual sample containers shall be wrapped in bubble pack or other shock absorbing media. It is desirable to place an absorbent and cushioning media in the bottom of the shipping cooler to absorb liquids in the event of breakage.

Coolers shall be securely sealed with suitable packing tape wrapped all the way around the shipping cooler to secure the lid during transit. It is desirable to tape over the latching handle to minimize the chance of accidental release.

The shipping containers and their contents will conform to current local, state, and federal shipping regulations.

5.4 CUSTODY SEALS

Custody seals are used to ensure that a sample shipping container has not been opened, and that custody has been maintained in transit. Following the securing of the shipping cooler lid with packing tape, custody seals will be placed across the lid opening of the shipping cooler. A minimum of two (2) custody seals will be used in case one seal is damaged during shipping even though the container lid remains closed.

The sample collector will record each custody seal number on the Chain-of-Custody Form prior to its attachment to the shipping cooler. The laboratory will compare the number of the custody seal(s) on the sample container against the Chain-of-Custody forms and report the seals' condition as intact or broken upon receipt. The laboratory will notify the Project Managers in writing, within 24 hours if a custody seal or container is received broken.

5.5 SAMPLE PACKAGING AND SHIPPING

The packaging, labeling, and shipping of hazardous wastes and substances when shipped by common carrier is regulated by the U.S. Department of Transportation (DOT) under 49 CFR. Samples from hazardous waste sites are classified as "Low", "Medium", or "High" level according to pollutant concentration. Low level samples, generally dilute in nature, are usually collected from areas surrounding a spill or disposal area. Medium level samples are generally collected on-site in areas of moderate dilution by normal environmental processes. The high level samples are generally collected from fresh spills, containers, lagoons, and waste piles, and contain greater than 15 percent of any individual chemical contaminant. Because of their potential toxicity or hazard, medium and high level samples require special handling procedures and must be shipped in compliance with DOT requirements.

The samples from this project will be considered as environmental, low level samples unless data which suggest otherwise are available.

5.5.1 Packaging

The User's Guide to the Contract Laboratory Program (U.S. EPA 1988) outlines CLP protocols for packaging and shipping or transportation of samples. They include:

1. Cool samples of low concentration to at or below 4 degrees C.
2. Pack medium and high concentration sample containers in metal cans.
3. Separate and surround cooler contents with vermiculite or equivalent packaging.
4. Fill out the Chain-of-Custody Form completely for each cooler of samples shipped.
5. Place the Chain-of-Custody form in a plastic bag with a watertight seal.
6. Include a return address label for the cooler.
7. Tape the sealed bag to the underside of the cooler lid.
8. Seal the cooler by placing custody seals across the joint between the lid and body.

5.5.2 Labeling and Marking the Cooler

The shipping labels shall be clear and correct. Conflicting labels from prior use shall be removed or covered with opaque tape.

The carrier's shipping label shall be on the top of the cooler. The name, address (delivery location), and telephone number of both the shipper and the receiving laboratory shall be included on the label. The shipper's return address shall be affixed to the top of the cooler with a durable label or with indelible felt marker. The sides of the cooler should be clearly marked with a label stating "This End Up" with arrows pointing to the top of the cooler.

5.6 RECEIVING SAMPLES AT THE ANALYTICAL LABORATORY

Upon receipt of the sample cooler at the analytical laboratory, the sample custodian shall inspect the cooler and the custody seals, noting any damage to the cooler and whether the custody seals are broken or intact. The custodian will then inspect each sample container for damage and note its condition and the condition of its custody seal. The number of each custody seal shall be verified against the number on the Chain-of-Custody Form and any discrepancies noted. Each sample container shall be inspected to verify that it has a sample tag or label and that the information is consistent with that on the Chain-of-Custody Form. Inconsistencies

between the information on the Chain-of-Custody Form and the container and seal information as received shall be reported by the laboratory to the Project Managers immediately.

If the chain-of-custody error was caused by the sampler, the sampler must correct the problem with a signed and dated correction memorandum to the Project Manager. Once validated, the correction memorandum will be sent to the NYSDEC QA/QC Manager, requesting that the modifications be made. The correction memorandum becomes a permanent part of the sample documentation and a copy of the correction memorandum, showing the date received by the laboratory, shall be submitted with the data package. Chain-of-Custody inconsistencies must be resolved before the results from the analysis are released for validation.

The laboratory will prepare Internal Chain-of-Custody (ICOC) Forms to accompany the samples through the analytical process. Copies of the ICOC will accompany the original sample, unused portions of samples, digestates, or extracts derived from the samples, raw instrumental data, and completed data packages as integral parts of the raw data. The details of the analytical laboratory QA/QC protocols will be presented in the laboratory Quality Assurance Project Plan.

6.0 CALIBRATION PROCEDURES

6.1 GENERAL

Calibration of field and laboratory equipment is necessary to ensure the accuracy and precision of the collected data. All instruments used in the sampling and analysis will have a complete maintenance and repair history through the duration of the project activities. This history will include, but will not be limited to, the following:

1. The equipment's identification by model and serial number.
2. The equipment's calibration and maintenance schedule, including the last date of routine maintenance and/or factory calibration, the name of the individual conducting the work, any deviations or abnormalities detected, and the tasks that were undertaken to correct any deficiencies.
3. A record of equipment failures and manufacturer's conducted repairs, calibrations, and maintenance and brief description of the cause of the failure that required manufacturer's service will be included, if available.

Any instrument that does not perform according to its specifications as stated by the manufacturer will be clearly labeled as such and returned for repair.

6.2 FIELD INSTRUMENTATION

The field instruments necessary for use in this investigation will be available from one or more study participants. The instruments will be maintained and calibrated in accordance with standard maintenance and calibration procedures. Manufacturers' recommendations for maintenance and calibration of instrumentation are followed where applicable. Calibration of field instruments will be performed by qualified personnel or by qualified manufacturers' representatives. Instrument calibration and maintenance procedures are kept on file. Relevant product literature including calibration and maintenance procedures is frequently an integral part of the Standard Operating Procedures, which accompany the equipment.

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Any deviation from the recommended procedures will be documented in the field book of the individual performing the calibration or maintenance, or the laboratory log maintained for the project and in the pertinent calibration book.

7.0 ANALYTICAL PROCEDURES

A summary of the analytical methods and sample requirements proposed for this project are presented in Table 7-1. The methods and holding times are summarized in Table 4-2. Method detection limits for tissue analysis proposed for this project are summarized in Table 7-2. The tissues prepared from the specimens collected for the field program (See Section 4.0) will be analyzed for PCBs, other organochlorines, metals, cadmium, mercury, and lead, percent moisture, and percent lipid. A laboratory prepared tissue blank will be used..

The analytical methods for the analysis of PCB, organochlorine pesticides, percent lipid and percent moisture content will be according to the U. S. Fish and Wildlife Service protocols based on U.S. Environmental Protection Agency methodology. The method is presented in Appendix C-1. A silicic acid column clean-up is used in the presence of PCBs to eliminate PCB interferences. Toxaphene interference will be handled by dilution unless it is encountered at concentrations greater than 50 times the concentration of other organochlorine analytes. In that case the toxaphene component will be quantified from a toxaphene standard. The clean-up procedures are described in Appendix C-1. The metals analyses for mercury, cadmium, and lead are presented in Appendix C-2. The PCB and organochlorine pesticide analysis will be performed by the contracted organics laboratory and the metals analyses will be performed by the NYSDEC laboratory at Hale Creek Field Station.

The analytical method for the metals mercury, cadmium, and lead is presented in Appendix C-2.

Media	Species	PCBs, organo-chlorines, pesticides	% Moisture	% Lipid	Mercury	Cadmium	Lead
Muscle	<i>R. clamitans</i>	36	36	36	36		
Muscle	<i>C. serpentina</i>	18	18	18	18	18	18
Adipose	<i>C. serpentina</i>	18	18	18	18	18	18
Liver	<i>C. serpentina</i>	18	18	18	18	18	18
Kidney	<i>C. serpentina</i>	18	18	18	18	18	18
TOTAL		108	108	108	108	72	72

¹ For QA/QC samples 20% duplicates, see QAPP Section 3.2

Table 7-2
Method Detection and Quantitation Limits
Organochlorine and Metal Contaminant Levels in
Hudson River Reptiles and Amphibians
Hudson River, New York

PCBS AND PESTICIDES		
Compound	Quantitation Limit (µg/Kg) ¹	MDL (µg/Kg) ²
PCB 1242	30	20
PCB 1248	30	20
PCB 1254	30	20
PCB 1260	30	20
HCB	10	2
alpha BHC	10	5
beta BHC	10	5
gamma BHC	10	5
delta BHC	10	5
alpha Chlordane	10	5
gamma Chlordane	10	5
Oxychlordane	10	5
cis-Nonachlor	10	5
trans-Nonachlor	10	5
Heptachlor epoxide	10	5
p,p'-DDT	10	2
p,p'-DDE	10	2
p,p'-DDD	10	2
o,p'-DDT	10	2
o,p'-DDE	10	2
o,p'-DDD	10	2
Endrin	10	5
Dieldrin	10	5
Toxaphene	50	50

Table 7-2
Method Detection and Quantitation Limits
Organochlorine and Metal Contaminant Levels in
Hudson River Reptiles and Amphibians
Hudson River, New York

Mirex	10	2
METALS		
Compound	Quantitation Limit (mg/Kg)	MDL (mg/Kg)
Cadmium	10	2
Mercury	10	8
Lead	15	15
OTHER PARAMETERS		
Percent Moisture	0.5	0.5
Percent Lipids	0.05	0.05
¹ The quantitation limits for PCBs, pesticides and other parameters are the “Lower limits of detection” of the proposed contractor. All values at or above the “Lower limits of detection” are to be reported by the contractor; values below the “Lower limit of detection” are to be reported as not detected. ² The values used are (except Toxaphene) those of the Department’s laboratory at the Hale Creek Field Station, Gloversville, New York.		

8.0 DATA REDUCTION, VALIDATING, EVALUATION AND REPORTING

8.1 GENERAL

All data collected for this project will be appropriately identified and validated. Where test data have been reduced, the method of reduction will be described in the text of such reports. Validation of all laboratory analytical data will be performed by the NYSDEC Data Validator.

8.2 DATA VALIDATION - LABORATORY

The Laboratory Quality Assurance Officer will perform all in-house analytical data reduction and QA/QC procedures. The Laboratory Quality Assurance Officer will be responsible for reporting any and all deficiencies to the Laboratory Project Manager, who in turn reports to the NYSDEC Data Validator, and the NYSDEC Project Manager, and NYSDEC QA/QC Manager. A deficiency report will include a discussion of why the data are suspect, the reasoning behind the possible unreliability, and possible courses of action that can be taken to remedy the situation. Any course of action to rectify suspect data shall be agreed upon by the NYSDEC Data Validator, the NYSDEC Project Manager and the Laboratory Project Manager. Any data that fail to meet the validation requirements, and cannot be adequately rectified, shall be rejected and excluded from consideration during data analysis. This information will accompany any data packages.

8.3 DATA VALIDATOR

The NYSDEC will provide the independent data validation for the analytical laboratory data.

8.4 DATA VALIDATION - FIELD MEASUREMENTS

All data from field measurements and measurements taken during tissue sample preparation will be validated by a scientist who has not been involved in the data collection. This validation will include a review of the methods and/or SOPs used for the collection of the data, and the field and laboratory notes/results that are collected. Any data

that cannot be validated will be documented and excluded from further consideration. All data entry to computer files will be checked by another person who will initial and date a hard copy of the data reviewed.

8.5 DATA EVALUATION

The contaminant data will be summarized statistically by sex and species using small sample statistical methods such as Mann-Whitney U or similar method agreed to with the NYSDEC. The sex distribution in such a small sample as 5 turtles and 10 frogs per site may not allow a statistical evaluation by sex. Qualitative discussion of the data by sex will be included.

8.6 REPORTING REQUIREMENTS

The final data report shall meet the requirements of the NYSDEC. The chemical laboratory data package will include:

1. A summary of all results showing amounts detected and detection limits.
2. A tabulation of all surrogate recoveries; matrix spikes and duplicates; trip, field, and equipment blanks; and all calibration raw data and results.
3. Chain-of-Custody Forms for each sample shipment.
4. All calculation work sheets used to reduce the raw data, including all chromatogram and instrument printouts.

Copies of the field and laboratory data generated during specimen collections and sample preparation will be provided to the NYSDEC. Copies of calculation sheets generated during data analysis will also be provided. These data reports will be available for review during data analysis.

9.0 INTERNAL QUALITY CONTROL CHECKS AND FREQUENCY

9.1 DATA HANDLING

All analytical chemistry data shall be submitted by the analytical laboratory to the NYSDEC Project Manager and NYSDEC Data Validator. Data packages shall be assembled in accordance with CLP protocols for all samples analyzed under CLP protocols. For those samples analyzed under non-CLP protocols, adequate QA procedures will be followed to allow the comparison between the non-CLP and the CLP results.

9.2 DATA VALIDATION

All chemical data included in the project database and in interim and final reports shall be validated by the NYSDEC Data Validator. This validation shall include an independent audit of all laboratory data and all QA/QC samples and procedures. This audit shall assess the validity of the data and determine its acceptability.

9.3 DATA REDUCTION

All of the chemical data and field data that are generated during the project will be entered into an electronic database. This electronic database will be provided to the NYSDEC Bureau of Habitat with the project final report. The validated chemical data will be provided by the NYSDEC for data reduction.

10.0 PERFORMANCE AND SYSTEM AUDITS

Performance and system audits for the analytical laboratory will be performed as specified by the NYSDEC. Performance and system audits for the data generated during field collection and sample preparation will consist of the review of all sampling information, raw data, data entry, and calculations by a scientist who did not participate in generation of the data.

11.0 PREVENTIVE MAINTENANCE

The purpose of preventive maintenance is to have instruments and equipment always in condition to properly and correctly carry out the function for which the unit is designed, and to do so with minimum adjustment, calibration, or down time at the time of use.

An inventory control system for instruments that may be used in the laboratory or for sampling purposes will be maintained. The inventory record will be maintained at the offices of the Project Manager. Each unit of field monitoring instruments shall have current documentation which includes:

1. type and title of unit
2. manufacturer, model number, and serial number
3. identification number
4. date of purchase
5. service Company (name, address, telephone and FAX numbers)
6. type of service policy
7. routine maintenance, servicing and calibration (timing, frequency)
8. date of and cause for last service (other than routine)
9. date of last calibration
10. adjustment required to recalibrate equipment, and any deviation to complete calibration following adjustment.

Field measurement instruments shall be maintained in accordance with SOPs and manufacturers' instructions (which may be appended to the SOP). Instruments shall be checked prior to use by the operator. The use of the instrument shall be recorded in the log book and that record shall include a report of problems encountered with the instrument, a description of the symptoms, and corrective actions taken. Problems with the instrument will be corrected before use is resumed.

Instruments shall be cleaned, repaired, and recharged (if appropriate) following use so that they are ready for the next application.

12.0 PRECISION, ACCURACY AND COMPLETENESS OF ANALYTICAL DATA

12.1 GENERAL

This section describes the QA/QC procedures that will be established to ensure that the analytical data generated for the project are accurate, precise and complete. The collection and analysis of representative samples will insure the best chance of obtaining accurate results that reflect the conditions of the specimens. The laboratory will either analyze the complete sample or a smaller portion of the sample that is representative of all that material in the container. Data that meet these criteria will be considered representative..

12.2 DEFINITIONS

The following definitions will be used to structure the QA/QC procedures.

1. Accuracy: The amount of agreement between the true value of a parameter and the measured value. Accuracy is a measurement of the bias in a parameter.
2. Precision: The measurement of the agreement between samples from the same population. Precision can be expressed as the standard deviation between independent samples or as the relative percent difference (RPD) between duplicate samples.
3. Completeness: The measure of the amount of validated data obtained compared to that which was expected to be obtained.

12.3 EQUATIONS

The following equations are required to estimate the accuracy, precision, and completeness in a QA/QC program for organic, inorganic and semi-volatile analysis of various matrices.

12.3.1 Percent Recovery

$$(\text{Spike Sample}) = \frac{\text{RSS} - \text{RUS}}{\text{SA}} \times 100$$

Where: RSS = Results of Spike Sample

RUS = Results of the Unspiked Sample

SA = Amount of Spike Added

This analysis of the chemical data will be performed as specified by the NYSDEC Data Validator

12.3.2 Relative Percent Difference

$$(\text{RPD}) = \frac{D_1 - D_2}{0.5(D_1 + D_2)} \times 100$$

Where: D_1 = First Sample Results

D_2 = Duplicate Sample Results

12.3.3 Percent Completeness

$$= \frac{\text{Number of Valid Results}}{\text{Number of Possible Results}} \times 100$$

12.4 MINIMUM REQUIREMENTS

The minimum requirements needed to demonstrate the analytical laboratory's capability to meet the QC requirements for analysis of the target compound list will be those set forth in the method protocols accepted by the NYSDEC.

12.4.1 Blank Samples

Blank samples will be used to evaluate sources of contamination inherent in laboratory or sampling procedures that are not attributable environmental contamination. Method blanks prepared from clean tissue will be used by the analytical laboratories.

12.4.2 Spiked Samples

Matrix spikes and matrix spike duplicates will be used to evaluate the accuracy and precision of the laboratory's analysis. Spiking samples and blanks prior to analysis will be used to evaluate analytical accuracy of the laboratory. Performance will be measured by the percent recovery of the spike material using Equation 12.3.1. Corrective action shall be taken if the one surrogate compound is outside the required recovery limits in either the method blank or sample. Notification of deviations will be made immediately to both the laboratory Quality Assurance Manager, the NYSDEC Project Manager, and the NYSDEC Data Validator. Method details will be addressed in the laboratory QA plans prepared by the analytical laboratory.

12.4.3 Duplicate Samples

Duplicate tissue samples will be prepared and submitted to the analytical laboratory. The number of duplicate samples will represent 20 percent of the total sample number. Enough material will be collected so that a laboratory duplicate sample may be prepared from the tissue samples submitted, after the sample is properly homogenized. Duplicate samples will be collected in the same manner, using the same equipment and by the same personnel. Duplicate samples will be analyzed using identical methods for the same parameters. These samples will be used to evaluate the analytical precision of the field sampling event.

13.0 CORRECTIVE ACTION

Any non-conformance or deficiencies detected in any activities shall be reported to the appropriate Project Manager responsible for the activity. The Project Manager will be responsible for non-conformance or deficiencies reported during field collection and sample preparation, and the NYSDEC Project Manager will be responsible for non-conformance or deficiencies reported during chemical analysis. A description of the non-conforming situation, and of the corrective actions taken, will be recorded by the project personnel in the deviation memorandum filed with the Project Manager and the NYSDEC QA/QC Manager.

14.0 QUALITY ASSURANCE REPORTS

A letter report will be submitted to the NYSDEC Project Manager that includes discussion of the data accuracy, precision, and completeness; the results of all performance audits and tests performed; and all problems discovered and the corrective actions that were taken. A summary of this report shall be included in the final report.