MEMORANDUM OF UNDERSTANDING

Among

The United States Environmental Protection Agency,
The New York State Department of Environmental Conservation and
The New York State Department of Health

I. Purpose and Scope of Work

This Memorandum of Understanding (MOU) is entered into by the United States Environmental Protection Agency (USEPA), the New York State Department of Environmental Conservation (NYSDEC) and the New York State Department of Health (NYSDOH) to delineate the responsibilities of each agency with regard to the Health Risk Assessment for the General Motors Corporation, Central Foundry Division Superfund Site, located in Massena, New York. The intent of the MOU is to ensure a coordinated approach to the timely and effective completion of the Health Risk Assessment for the Site. This memorandum of Understanding also clarifies each agency's role and responsibilities with respect to the completion of the Health Risk Assessment.

The scope of work for this Health Risk Assessment is contained and described in the following four attachments which by reference are made a part of this MOU:

1. Background and Summary - Health Risk Assessment;
2. Technical Proposal - Contaminant Monitoring in Fish;
3. Technical Proposal - Contaminant Monitoring in Wildlife; and
4. Technical Proposal - Human Breast Milk Study.

II. The Term

The MOU shall be in effect on the date it is signed by the persons authorized to sign for the USEPA, the NYSDEC and the NYSDOH. The MOU shall remain in effect until the completion of the Health Risk Assessment, unless it is mutually agreed by all of the parties to modify or to terminate the MOU.

III. Provisions

1. At the present time, the General Motors Corp. is conducting a Remedial Investigation/Feasibility Study (RI/FS) at its Central Foundry Division, Massena, New York plant site. This study is being conducted under terms of an "Administrative Order on Consent" Index No. II CERCLA-50201 dated April 16, 1985 between the United States Environmental Protection Agency Region II Office and the General Motors Corporation. This RI/FS is being conducted in accordance with the requirements of the Comprehensive Environmental Response, Compensation and Liability

Act of 1980 (CERCLA) (42 U.S.C. 9606(a)) as amended by the Superfund Amendments and Reauthorization Act (SARA) of 1986, the provisions of the Administrative Order on Consent and other applicable Federal and New York State Laws, Rules, Regulations and environmental quality guidelines. This Health Risk Assessment, as outlined in this MOU, will provide part of the information required to complete this RI/FS investigation at the General Motors Corp. - Central Foundry Division, Massena, New York plant site.

2. The USEPA will provide the overall project guidance to ensure that this project will result in information that will be suitable for inclusion in the RI/FS. Ms. Christine Visnic of the Emergency and Remedial Response Division of the USEPA, Region II, will serve as Project Manager on behalf of the USEPA and will provide the overall project guidance and the coordination of information and data concerning the Health Risk assessment among all three agencies involved in the study, among other interested parties such as the General Motors Corporation, the press and other news media, the Mohawk Nation at Akwesasne, and interested members of the public.

3. The NYSDEC will be responsible for completing the Task 1 - Contaminant Monitoring of Fish, and Task 2 - Contaminant Monitoring of Wildlife, components of the Health Risk Assessment. Mr. Jim Reagan of the Bureau of Eastern Remedial Action will serve as the Project Manager and coordinator of all the activities related to the Health Risk Assessment within the NYSDEC.

4. The NYSDOH will be responsible for completing the Task 3 - Human Breast Milk Study component of the Health Risk Assessment and for the preparation of Task 4 - the Final Overall Health Risk Assessment Report utilizing the field data obtained from Tasks 1 through 3. Mr. Gary Litwin of the Bureau of Environmental Exposure Investigation will serve as Project Manager and coordinator of all activities related to the Health Risk Assessment within the NYSDOH.

in accordance with the provisions of Section 2061(j) of the Public Health Law

5. The USEPA, NYSDEC and NYSDOH agree that complete and adequate Quality Assurance/Quality Control protocols will be developed according to the USEPA's "Interim Guidelines and Specifications for Preparing QA Project Plans" (QAM-005/80). These shall be submitted to, approved by the three Project Managers, and in place prior to performing any of the activities related to the Health Risk Assessment. Protocols will be updated and revised as necessary. These protocols will be utilized for performing the work activities for each of the individual tasks.

Superfund Amendments and Reauthorization Act (SARA) of 1986, the provisions of the Administrative Order on Consent and other applicable Federal and New York State Laws, Rules, Regulations and environmental quality guidelines. This Health Risk Assessment, as outlined in this MOU, will provide part of the information required to complete this RI/FS investigation at the General Motors Corp. - Central Foundry Division, Massena, New York plant site.

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4. The NYSDOH will be responsible for completing the Task 3 - Human Breast Milk Study component of the Health Risk Assessment in accordance with the provisions of Section 206.1(j) of the Public Health Law and for the preparation of Task 4 - the Final Overall Health Risk Assessment Report utilizing the field data obtained from Tasks 1 through 3. Mr. Gary Litwin of the Bureau of Environmental Exposure Investigation will serve as Project Manager and coordinator of all activities related to the Health Risk Assessment within the NYSDOH.

5. The USEPA, NYSDEC and NYSDOH agree that complete and adequate Quality Assurance/Quality Control protocols will be developed according to the USEPA's "Interim Guidelines and Specifications for Preparing QA Project Plans" (QAM-005/80). These shall be submitted to, approved by the three Project Managers, and in place prior to performing any of the activities related to the Health Risk Assessment. Protocols will be updated and revised as necessary. These protocols will be utilized for performing the work activities for each of the individual tasks.

6. The USEPA, the NYSDEC and the NYSDOH agree that the proposed schedule of activities related to the Health Risk Assessment found in Attachment 5 will be followed to the maximum extent possible. This schedule may be amended if modifications are agreed to by all three agencies involved in these studies.

7. The initial funding for Tasks 1, 2, and 3 is being provided from the following sources: The General Motors Corp. - $370,000 (approximately 80% of the total cost) and the New York State Department of Environmental Conservation - $91,300 (approximately 20% of the total cost). The total cost estimate for these three tasks is $461,300. The funds to perform these studies will be allocated on a One time, lump sum basis only, as follows:

Task 1 - Fish -	$271,300
Task 2 - Wildlife -	$130,000
Task 3 - Breast Milk -	$ 60,000
Subtotal (1 through 3) -	$461,300

Task 3 will be funded, in part, by a suballocation of $50,000 of appropriate funds to the NYSDOH by the NYSDEC upon execution of this MOU. The $50 000 will be used over a two-year period by NYSDOH in accordance with the expenditure plan for Task 3 included in Attachment 4.

Task 4 (Preparation of the Final Overall Health Risk Assessment Report utilizing the data and results of Tasks 1 through 3) will be performed by the New York State Department of Health (NYSDOH).

8. The USEPA, the NYSDEC and the NYSDOH agree to spend these funds only on activities related to the Health Risk Assessment.

9. The USEPA, the NYSDEC and the NYSDOH agree to exchange written progress reports and an itemization of documented expenditures incurred for activities related to the Health Risk Assessment at the General Motors Corp., Central Foundry Division Site in Massena, New York, on a ~~quarterly~~ annual basis in the format exemplified in Attachment 6, and to establish time and activity reporting codes and a cost center to document direct expenditures related to the Health Risk Assessment at this site. Reporting will follow the Federal fiscal year. Also, a complete written itemized financial account will be required from each Task Leader (Tasks 1 through 3) within one month of the completion of each of these tasks.

The first written status or progress report will be due from each of the initial three Task Leaders within 30 calendar days following the signing of this MOU by the three agencies involved.

10. All expenditures and claims for costs under this agreement are subject to review and approval by appropriate State and Federal auditors.

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6. The USEPA, the NYSDEC and the NYSDOH agree that the proposed schedule of activities related to the Health Risk Assessment found in Attachment 5 will be followed to the maximum extent possible. This schedule may be amended if modifications are agreed to by all three agencies involved in these studies.

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Task 1 - Fish -	$271,300
Task 2 - Wildlife -	$130,000
Task 3 - Breast Milk -	$ 60,000
Subtotal (1 through 3) -	$461,300

Task 3 will be funded, in part, by a suballocation of $60,000 of appropriate funds to the NYSDOH by the NYSDEC upon execution of this MOU. The $60,000 will be used over a two-year period by NYSDOH in accordance with the expenditure plan for Task 3 included in Attachment 4.

Task 4 (Preparation of the Final Overall Health Risk Assessment Report utilizing the data and results of Tasks 1 through 3) will be performed by the New York State Department of Health (NYSDOH).

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The first written status or progress report will be due from each of the initial three Task Leaders within 30 calendar days following the signing of this MOU by the three agencies involved.

11. The Project Managers for each agency shall be responsible for ensuring that the provisions of this MOU are carried out within each agency. The Project Managers for each of the three agencies shall be responsible for the proper completion of this Health Risk Assessment. All persons (including the individual Task Leaders) shall be bound by the terms of this MOU. They shall report to the Project Managers in all matters related to this Health Risk Assessment.

12. It is agreed that it shall be the stated policy of the USEPA, the NYSDEC and the NYSDOH that this Health Risk Assessment will be performed in as totally objective and scientifically neutral fashion as can be humanly and practically achieved. Steps will be taken to ensure, to the greatest extent possible, that no bias or prejudgements are introduced into these studies at any point, from the start to the end of the project. The overall fairness and objectivity of this Health Risk Assessment are of absolute and primary importance.

13. The General Motors Corp. shall be kept fully and completely informed of all aspects of each of the individual tasks and of the overall Health Risk Assessment to the greatest extent possible. The General Motors Corp. will not provide any of the direction for the performance of these studies. That direction will be the sole responsibility of the USEPA, the NYSDEC and the NYSDOH. These same three agencies will also be responsible for ensuring the technical adequacy, the scientific quality and the objectivity of the Health Risk Assessment. Comments or suggestions may be provided by the General Motors Corp. All comments provided will be given serious review and consideration by the three agencies performing the Health Risk Assessment. However, the final disposition of any comments or suggestions will be the responsibility of the three agencies involved. The General Motors Corp. will be provided with copies of all data obtained as soon as practical (in no case should this be later than the date of release of each Draft Task Report). The General Motors Corp. shall also be provided the opportunity to obtain split samples of any environmental media obtained during these studies to the greatest extent possible. The USEPA will provide the primary coordination between all of the persons involved with the Health Risk Assessment and the General Motors Corp. Direct contact by individuals will be allowed, as long as overall project guidelines are followed.

14. The Mohawk Nation at Akwesasne shall be kept fully and completely informed of all aspects of each of the individual tasks and of the overall Health Risk Assessment to the greatest extent possible. The USEPA will provide the primary coordination between all of the persons involved with the Health Risk Assessment and the Mohawk Nation.

15. No information pertaining to the Health Risk Assessment Studies will be released by anyone involved in the studies to the public, press, or other news media without the prior notification of all three agencies responsible for the completion of these studies. All of the information from these studies will be made available to the press and to the public in a uniform and orderly manner through the three agency's respective signatories or their designees. A minimum of 24 hours advance notice shall be provided to the other agencies should one agency deem it necessary to release information to the public. The advance notice shall be made to the signatory or their designee. The primary responsibility for the dissemination of this information shall rest with the USEPA.

16. All correspondence and communication of data and information will be conducted according to the chain of command procedures agreed upon at the July 14, 1987 meeting between the USEPA, the NYSDEC, and the NYSDOH. Pursuant to the above-mentioned meeting, the Task Managers shall report to the NYSDEC and NYSDOH Project Managers, who will then coordinate with EPA's Project Manager. All communication from the EPA will be directed to the NYSDEC and NYSDOH Project Managers, who will then be responsible for the prompt transmittal of this information to the Task Managers within their respective agencies.

Recommended By:

For the State of New York
Department of Environmental
Conservation

Date March 11, 1988

For the United States Environmental
Protection Agency

Date August 17, 1988

For the State of New York
Department of Health

Date May 4, 1988

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ATTACHMENT 1

BACKGROUND AND SUMMARY

ATTACHMENT 1

Background and Summary
Health Risk Assessment
GMC-CFD
Massena, New York

This three-part Health Risk Assessment is a part of the overall Remedial Investigation/Feasibility Study (RI/FS) currently being conducted by the General Motors Corp. (under terms of an April 16, 1985 Consent Order negotiated with the USEPA) at its Central Foundry Division Plant Site near Massena (St. Lawrence County), New York. This site is listed on the National Priorities List (NPL) register developed by the USEPA under Administrative Order Index No. II CERCLA-50201. It is also a Class 2 site on the list of inactive hazardous waste sites established by the Bureau of Hazardous Site Control of the NYSDEC as Site No. 6-45-007.

This Health Risk Assessment is designed to be a realistic and practical study to assess major potential pathways of exposure of human populations, and in particular the residents of the Mohawk Nation at Akwesasne, New York, to Polycholorinated-Biphenyls (PCBs) in the food chain. The study examines three primary potential routes of exposure: (1) through consumption of fish; (2) through consumption of wildlife; and (3) through consumption of human breast milk.

Agreement to perform the study was reached as the product of negotiations among the following parties during the spring and summer of 1987: the General Motors Corp.; elected representatives from the Mohawk Nation at Akwesasne, New York; the USEPA; the NYSDOH and the NYSDEC.

The costs for performing the three tasks or parts of the Health Risk Assessment have been estimated as follows: Fish - $271,300; Wildlife - $130,000; and Human Breast Milk - $60,000. The total cost for these three tasks (which does not include the costs for the final overall Health Risk Assessment) has been estimated to be $461,300. General Motors Corp. is supplying $370,000 of the funding required (or approximately 80% of the total cost estimate). The balance of the funding required - $91,300 (or approximately 20% of the total cost estimate) is being provided by the NYSDEC. It should also be recognized that substantial indirect costs will also be incurred by each of the agencies involved in the completion of the study. The entire study (including the three data collection tasks and the final overall Health Risk Assessment) is estimated to take approximately three years to complete beginning in September 1987 and ending in September 1990.

ATTACHMENT 2

CONTAMINANT MONITORING OF FISH

SAMPLING PROTOCOLS AND QUALITY ASSURANCE PLANS

For

CONTAMINANT MONITORING OF FISH FROM WATERS IN AND NEAR THE AKWESASNE RESERVATION OF THE MOHAWK NATION

One task of a three-part project to evaluate contaminant conditions in biota associated with the St. Lawrence River in the vicinity of Massena, New York involving the General Motors Central Foundry Division Superfund site and providing data for a Health Risk Assessment to be conducted by the New York State Department of Health on the consumption of fish by members of the Mohawk Nation at Akwesasne in cooperation and coordination with the United States Environmental Protection Agency, the New York State Department of Environmental Conservation, the New York State Department of Health and the Mohawk Nation at Akwesasne

Prepared by:

Ronald J. Sloan, Ph.D.
Research Scientist
Bureau of Environmental Protection
Division of Fish and Wildlife
New York State Department of Environmental Conservation
50 Wolf Road, Room 530
Albany, New York 12233-4756

February 1, 1988

CONTAMINANT MONITORING OF FISH FROM WATERS IN AND NEAR THE AKWESASNE RESERVATION OF THE MOHAWK NATION

SAMPLING PROTOCOLS AND QUALITY ASSURANCE PLANS

New York State Department of Environmental Conservation and the U.S. Environmental Protection Agency in conjunction with the Mohawk Nation at Akwesasne.

USEPA Project Manager — Christine Visnic

New York State Project Manager — James Reagan

USEPA Quality Assurance Officer — Wilbur Sellers

New York State Quality Assurance Officer — Robert Bauer

New York State Task Manager — Ronald Sloan, Ph.D.

Mohawk Nation Coordinator — Ken Jock

New York State Department of Health Health Risk Assessment Coordinator — Anthony Forti

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RECIPIENTS OF QA AND MONITORING PLANS:

Wilbur Sellers, USEPA, Region II, Edison, New Jersey
Robert Bauer, NYSDEC, Bureau of Environmental Protection, Avon, New York
Christine Visnic, USEPA, Region II, New York, New York
James Reagan, NYSDEC, Division of Remedial Action, Albany, New York
Ken Jock, St. Regis Mohawk Health Service, Hogansburg, New York
James Ransom, St. Regis Mohawk Health Service, Hogansburg, New York
Anthony Forti, NYSDOH, Bureau of Environmental Exposure Investigation, Albany, New York
Ward Stone, NYSDEC, Wildlfie Pathology, Delmar, New York
Al Schiavone, NYSDEC, Bureau of Fisheries, Region 6, Watertown, New York
Elena Kissel, USEPA, Region II, New York, New York
Leonard Charla, General Motors Corporation, Office of General Counsel, Detroit, Michigan
William McFarland, General Motors Corporation, Superfund Activities, Warren, Michigan
Douglas Premo, General Motors Corporation, Central Foundry Division, Massena, New York
Joseph Medved, Manufacturing Administration, Environmental Activities, Saginaw, Michigan
Carl Engle, General Motors Corporation, Central Foundry Division, Massena, New York
Gary Litwin, NYSDOH, Bureau of Environmental Exposure Investigation, Albany, New York
Geoff Laccetti, NYSDOH, Bureau of Environmental Exposure Investigation, Albany, New York
Dr. Edward Fitzgerald, NYSDOH, Bureau of Environmental Exposure Investigation, Albany, New York
Darrel Sweredoski, NYSDEC, Region 6, Massena, New York
Lance Turner, NYSDEC, Division of Remedial Action, Albany, New York
Larry Skinner, NYSDEC, Bureau of Environmental Protection, Albany, New York
Debbie Christian, NYSDEC, Division of Environmental Enforcement, Albany, New York
Susan McCormick, NYSDEC, Division of Construction Management, Albany, New York
Steve Patch, U.S. Fish and Wildlife Service, Cortland, New York
Samuel Jackling, NYSDEC, Bureau of Environmental Protection, Gloversville, New York
John Privatera, NYS Department of Law, Albany, New York
Doug Dillman, Hazleton Laboratories America, Madison, Wisconsin

INTRODUCTION

Previous chemical analyses of biota and other environmental strata in the Massena area on the St. Lawrence River have indicated relatively high concentrations of organochlorines, including PCB (NYSDEC 1978, 1981, 1987a). Consequently, health advisories on the consumption of fish and other wildlife are in effect (NYSDEC 1987b). Although several sources of contamination are known, the contribution of any one point source to the system is unknown.

The fish monitoring project outlined in this document, even though focusing on particular geographic points, will not, by itself, unquestionably elucidate a direct "cause-effect" relationship. In concert, however, with other studies previously performed, planned, or awaiting conception, the evaluation of fish will complement other data sets in order to better determine and resolve contaminant conditions faced by the ultimate consumer of natural resources - man. The Mohawk Nation at Akwesasne, intimately tied to the land and its produce, lies adjacent to and downstream of one specific potential generator of xenobiotics and the current project is oriented toward that site. However, to be objective other inputs are acknowledged. The study design is oriented to recognize such influences.

This revision of the sampling plan and protocols incorporates the quality assurance and quality control aspects of the project according to guidelines specified in "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans" (USEPA 1980). The major departure from the recommended format is the presentation of the project description (Section 3) by tasks:

Task 1 -	Collection of fish
Task 2 -	Preparation, shipment and chemical analyses of samples
Task 3 -	Data management and statistical analyses
Task 4 -	Reporting
Task 5 -	Administration

All sections are subject to revision including additions of passages to provide more complete descriptions, and modifications to procedures or activities. A final draft of this document is not anticipated until February 1988 primarily due to laboratory contract negotiations.

PROJECT DESCRIPTION:

Date of Initiation: Spring 1988

A. OBJECTIVES AND SCOPE

Existing data on contaminants in fish and wildlife in the vicinity of and on the Akwesasne Reservation is limited. The evaluation of wildlife is being addressed separately. Therefore, the objectives of this project concerning the fishery resource are:

To assess contamination levels of those fish species utilized by the populace adequate to derive public health risks and develop health advisories on the consumption of fish; and

To evaluate spatial relationships of contamination with respect to source(s) such as industrial discharges, leachates or contaminated sediments.

B. TASK 1 - COLLECTIONS OF FISH

Monitoring network design and rationale:

This project is envisioned as a one-time effort with sampling scheduled for the Spring of 1988. However, it may develop that additional or follow-up monitoring events are advisable. Sampling will be for fish of edible size from various locations as depicted in Figure 1 in coordination and cooperation with the Mohawk Nation at Akwesasne. A description and rationale for each location is provided in Table 1.

Data to be collected and rationale:

The following species are reported from the watershed and are available from at least some collection locations:

Smallmouth bass
Largemouth bass
Walleye
Northern pike
Muskellunge
Yellow perch
White perch
Rock bass
White bass
Pumpkinseed
American eel
Bluegill
Black crappie
White crappie
Brown bullhead
Channel catfish
White sucker
Carp
Burbot
Brown trout
Lake sturgeon
Pacific salmon

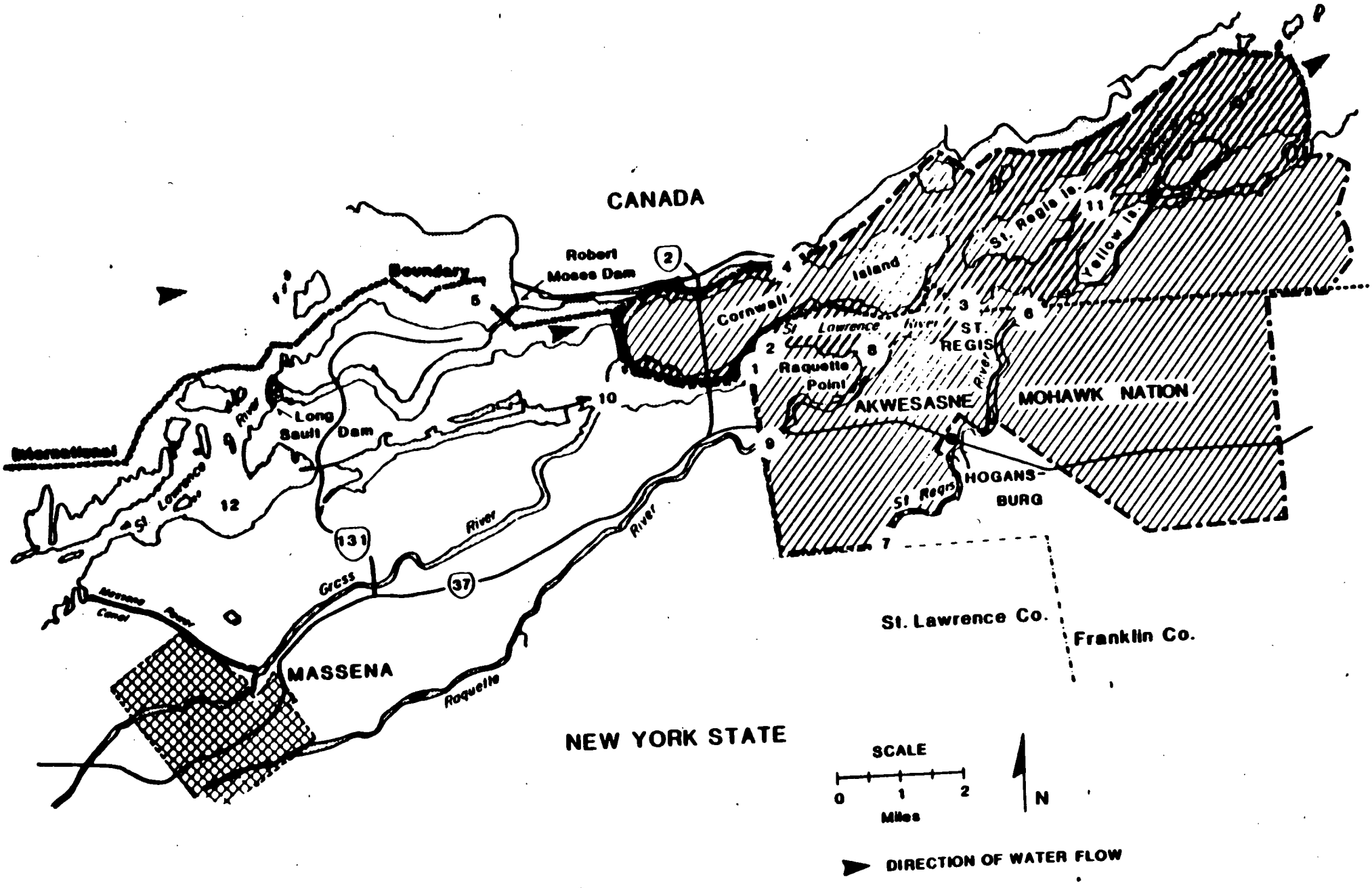

Figure 1: Sampling stations for fish from waters in or near the Mohawk Nation at Akwesasne.

Table 1. Locations, identified on Figure 1, for sampling fish in and near the Mohawk Nation at Akwesasne.

Station	Location	Reason for Inclusion
1	St. Lawrence River - mouth of unnamed tributary on Franklin/St. Lawrence County boundary.	Immediate proximity to the "Central Foundry" Superfund site.
2	St. Lawrence River - bay west of, but adjacent to, Raquette Point	Downstream, but proximate to the waste site and is heavily utilized for fishing by local populace.
3	St. Lawrence River - vicinity of St. Regis Village	Downstream of waste influences from the industrial activities along the St. Lawrence River but still on Reservation property (U.S. side) in an area intensively fished by the populace.
4	St. Lawrence River - north channel of Cornwall Island	Earlier unpublished Canadian fish data indicated lesser contamination in the north channel compared to samples in the south channel (i.e. near "Central Foundry"). The sampling point is downstream but near potential industrial sources to the north-channel.
5	St. Lawrence River - above Robert Moses Power Dam	Reflects upstream "background" conditions in the St. Lawrence River to which fish in the areas under study are exposed.
6	St. Regis River - northernmost segment near St. Regis Village	To evaluate contaminant conditions in fish from the St. Regis River near its mouth. Fish collected at this site are accessible to villagers and may not be as contaminated as fish from the St. Lawrence River.
7	St. Regis River - reach from above dam in Hogansburg to southern-most segment bordering Reservation	To evaluate contaminant conditions in fish from the St. Regis River upriver from the Reservation which are restricted from the St. Lawrence River by the dam in Hogansburg.
8	Raquette River - at mouth	An area heavily utilized for fishing. Fish have access to the St. Lawrence River and may be contaminated.

Table 1 (Continued)

9	Raquette River- upstream of Route 37 bridge in vicinity of Reservation boundary	Fish from this site are less likely (than from Station 8) to have been exposed to contamination from the St. Lawrence River
10	Grass River - at mouth	To evaluate potential additional sources of contaminants to the St. Lawrence River via the Grass River.
11	Between St. Regis and Yellow Islands or in the Snye Marsh	To determine downstream extent of contaminant conditions in fish of the Mohawk Nation at Akwesasne.
12	St. Lawrence River	A "control" site where species mix may be different from Station 5.

At each site, up to six species should be collected. With the exception of muskellunge, northern pike, and lake sturgeon a minimum of 5 fish and a maximum of 20 are required for each species at each site. Because populations of the muskellunge, pike and sturgeon are low, no more than 10 individuals should be taken from all sites; at each location where one of these species is taken a minimum of three is desirable to provide an estimate of variance.

All fish should be of edible size or legal size if applicable (i.e. bass, walleye, muskellunge, northern pike or brown trout). Large sizes are preferred.

Recognizing that diversity in habitat, water quality, and other variables influences species distributions, reasonable attempts should be made to collect fish in the following categories:

One species of restricted home range and low fat content that is common to all locations (possible species: pumpkinseed, bluegill, yellow perch, smallmouth bass, rock bass). Yellow perch is a good target species.

One species of restricted home range and high fat content that is common to all locations (possible species: American eel, carp). Carp is a good target species, although in larger waterways carp are known to travel widely (ref).

One species with wide home range and low fat content that is not common to all locations (possible species: walleye, esocids).

One species with wide home range and high fat content that is not common to all locations (possible species: channel catfish, white perch).

Two species selected on basis of availability (not common to all locations) and/or local preference (possible species: lake sturgeon, brown bullhead, black crappie, largemouth bass, smallmouth bass, white bass, Pacific salmon - i.e. coho or chinook).

These species represent those most likely to be available for harvest; however, if another species is discovered to be abundant and utilized by anglers, particularly members of the Akwesasne Nation, the species should be added or substituted as appropriate.

Fish will be collected and handled following standard procedures (Appendix A). As noted in those procedures the following data are to be recorded for each fish before being prepared for chemical analyses:

Unique tag number	Species
Date	Weight
Location	Length
Method of collection	Sex (if possible without cutting)
Collector(s)	

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Within 12 hours of capture, freeze whole at 0°F ± 10°F. In the field, place fish on ice immediately upon landing. The overall collection (Table 2) will result in a minimum of 360 individual analyses on specimens selected from the maximum number of fish available, potentially as high as 1440. The compositing procedure will result in 36 samples for the acid extractables and base/neutrals utilizing the remainders of the carcasses in order to maximize the information for a given sample location and reduce the variability inherent in analyzing individual fish. Similarly, the fillet composite for PCDD, PCDF and cogeneric PCB analyses will also result in 36 samples. Fillets (i.e. "edible portions") are appropriate since standards, guidelines or other information are available to allow an assessment of human health risk for mercury, organochlorines, dioxins and dibenzofurans. The costs for congeneric PCB analyses are being defrayed using other fiscal sources.

Field weights are the responsibility of the Field Coordinator or the Field Technician and are taken at the time the fish are placed in the freezers at Hogansburg. Scales are calibrated at each weighing session with standard weights. Accuracies sought are nearest whole gram for specimens between 50g to 250g. In keeping with the health risk assessment, fish weighing less than 50g presumably are not normally consumed and hence would not be retained. Between 250g to 500g, a 5g accuracy is adequate and above 500g, a 10g error is sufficient. For fish greater than 5kg, accuracy within 100g is sought.

Table 2. Sampling design for the collection and analysis of fish from waters in and near the lands of the Mohawk Nation at Akwesasne.

Station Number as given in Table 1 and Figure 1	Species*	Minimum Number	Maximum Number	Number of Analyses: Organochlorines	Nb, acid extractables and base neutrals**	PCDD, PCDF and Organoarsenic PCB**
1	[redacted]	[redacted]	[redacted]	[redacted]	1	1
					1	1
					1	1
	Subtotal	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
2	[redacted]	[redacted]	[redacted]	[redacted]	1	1
					1	1
					1	1
	Subtotal	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
3	[redacted]	[redacted]	[redacted]	[redacted]	1	1
					1	1
					1	1
	Subtotal	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
4	[redacted]	[redacted]	[redacted]	[redacted]	1	1
					1	1
					1	1
	Subtotal	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
5	[redacted]	[redacted]	[redacted]	[redacted]	1	1
					1	1
					1	1
	Subtotal	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
6	[redacted]	[redacted]	[redacted]	[redacted]	1	1
					1	1
					1	1
	Subtotal	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
7	[redacted]	[redacted]	[redacted]	[redacted]	1	1
					1	1
					1	1
	Subtotal	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
8	[redacted]	[redacted]	[redacted]	[redacted]	1	1
					1	1
					1	1
	Subtotal	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
9	[redacted]	[redacted]	[redacted]	[redacted]	1	1
					1	1
					1	1
	Subtotal	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
10	[redacted]	[redacted]	[redacted]	[redacted]	1	1
					1	1
					1	1
	Subtotal	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
11	[redacted]	[redacted]	[redacted]	[redacted]	1	1
					1	1
					1	1
	Subtotal	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
12	[redacted]	[redacted]	[redacted]	[redacted]	1	1
					1	1
					1	1
	Subtotal	[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
TOTAL		360	1440	360	36	36

*Letter designations are not intended to imply a specific species; species will vary between locations.
**Analysis represented by a composite of five fish

C. TASK 2 - PREPARATION, SHIPMENT, AND CHEMICAL ANALYSES OF SAMPLES

As the locked freezers at the Community Center in Hogansburg reach 1/2 capacity, samples are transported in a frozen state to the NYSDEC Hale Creek Field Station at Gloversville, New York. They are placed in the custody of a responsible person at that facility who will sign the Continuity of Evidence form.

Preparation of the samples for shipment to the contract analytical laboratory is the responsibility of the field technician with some assistance from the Hale Creek staff.

Fish will be filleted following standard procedures using the left fillet (Appendix A). At the time of filleting, reweigh and record the whole weight to determine amount of shrinkage. Record sex, sexual condition, and general condition of the organism. Weights of fillets and remaining carcass will be recorded, and these portions will be kept frozen (0°F $\pm$ 10°F) until they are either prepared for chemical extraction and analysis, or discarded. Field scales and laboratory scales at Hale Creek should be standardized with known weights each time the scales are used.

Determination of samples for individual analyses and compositing is the responsibility of the Task Manager and generally involves selection of the larger individuals of each species from each location.

Each fillet or composite sample is placed in an unused bag, tagged and refrozen in an appropriate insulated shipping container (DO NOT CLOSE THE LIDS!!). Analysis Request Forms supplied by Hazleton are completed for each container specifying fish tag numbers, location of capture, analyses to be performed, and an estimate of concentrations. The estimated levels are useful to the laboratory only for purposes of determining initial dilutions of the extracted materials. It has no bearing on the results but it does provide a mechanism to maintain lower analytical costs. The estimated concentration is derived from previous analyses on similar species in the vicinity such as those reported by NYSDEC (1987).

Shipment to Hazleton Laboratories America, Madison, WI occurs via Federal Express, on Mondays or Tuesdays. DO NOT SHIP ANY LATER IN THE WEEK. There is a remote chance that due to an error the samples could be waylaid over a weekend. Once thawed, decomposition occurs rapidly and hence, the sampling effort and the study is for naught. Likewise, holidays are also avoided for shipping. Furthermore, a "PRIORITY 1" designation is used to ensure that samples arrive in good condition. Shipping containers should be clearly labelled as property of the contract laboratory and include the full street address in the event the Federal Express label is inadvertantly lost or destroyed. Previous experience with shipments contraindicates the necessity of using dry ice to maintain the samples, provided the shipping containers are full and properly packed. If containers are less then 2/3 capacity, use dry ice to fill and submit proper ORMA hazardous materials forms to Federal Express along with the air bill. Shipping is billed to the laboratory. The laboratory is reimbursed as part of normal invoicing procedures.

Fish will be homogenized as individual fillets, extracted and analyzed by USEPA and USFDA approved techniques. Results will be reported on a wet weight and dry weight basis and percent lipid measured to allow calculation of contaminant concentrations on a lipid basis. The following analyses will be performed:

Analysis protocols	Samples
Hg and organochlorines - analyses performed by Hazleton Laboratories America, Madison, WI	left fillet of each fish collected (5 individual fish of 6 species at each site - 360 analyses).
Pb, acid extractables and base/neutrals - analyses performed by Hazleton Laboratories America	one composite of 5 carcasses of three species at each site (36 analyses).
PCDD, PCDF and congeneric PCB - laboratory to be determined	one composite of 5 left fillets of three species at each site (36 analyses).

The additional fish collected, but not selected for analysis, are to be used as back-up samples in the event of problems with analyses or finding a severely contaminated condition deserving of additional examination. Excess fish will be held at Hale Creek for up to nine months.

Laboratory procedures are outlined in more complete detail in Appendix B. Analytical results are submitted to the Task Manager for reporting requirements.

D. TASK 3 - DATA MANAGEMENT AND STATISTICAL ANALYSIS

All completed forms pertaining to collections and analytical results are sent to the Task Manager for input to Lotus 1-2-3 worksheets or a dBASE III-plus file using an IBM PS-2 Model 60. Completed files are transferred to a Statgraphics® (STSC 1986) software package for statistical purposes. By the time the analytical data comes available, PC-SAS may be operational on the PS-2. Such an installation will further facilitate the data analysis and reporting.

By convention, compounds and metals reported at the detection limit as indicated in Appendix B are utilized for statistical purposes by taking one-half the detection limit for that particular observation and using the result as an actual value. Transformations of the data to better satisfy conditions of normality will occur as needed.

Development of the data format recognizes the pathways depicted in Figure 2 that an individual fish may take during the course of the project.

Although the flow diagram is relatively straightforward, there is one area of confusion. If a specimen is selected for orgranochlorines and Hg analyses as an individual, a portion of the fillet may be composited with other samples of the same species from the same collection location. This composite is targeted for PCDD, PCDF and congeneric PCB analysis. If this selection occurs, the remainders of the carcasses of these same fish are also composited for other types of analyses (i.e. Pb, acid extractables and base neutrals).

Hence, the suggested data format which is dependent to an extent on the compounds determined, is presented in Table 3.

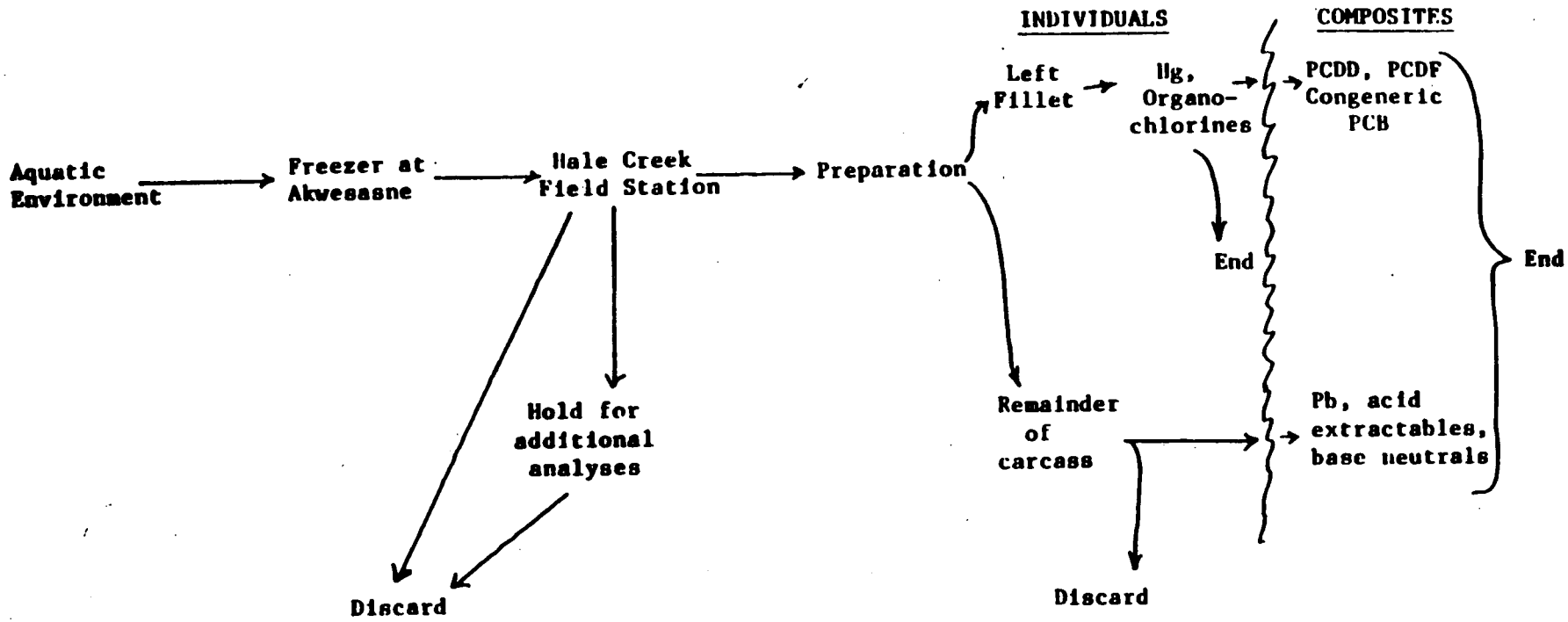

Figure 2. Pathways that an individual fish may take as a consequence of decisions on how to analyze the sample collections for the monitoring of fish from waters involving the Akwesasne Reservation.

Table 3. Suggested data format for the fish monitoring project at locations on the St. Lawrence River in and near the Mohawk Nation at Akwesasne.

Field Number	Field Description	Mnemonic	Type	Width	Decimal Places
1	Fish tag number	TAGNO	Character	6	--
2	Laboratory accession number	LABNO	Numeric	8	0
3	Collector's name	COLL	Character	20	--
4	Date collected (YYMMDD)	DATE	Numeric	6	0
5	Collection method	CMETHOD	Character	10	--
6	Location of collection	LOCATION	Character	30	--
7	Universal Transverse Mercator Coordinates (modified for New York State) - North	UTMN	Numeric	5	1
	- East	UTME	Numeric	5	1
8	Site number (as listed in the sampling protocol)	SITE	Numeric	2	0
9	Age of animal (years)	AGE	Numeric	2	0
10	Sex (male, female, unknown)	SEX	Character (M,F,U)	1	--
11	Sexual condition (Immature, Mature, MaturinG, Spent)	COND	Character (I,M,MG,S)	2	--
12	Total length (mm)	LEN	Numeric	5	0
13	Total field weight (g)	WGT	Numeric	6	0
14	Decision on whether fish is to be analyzed	TOANLY	Character (Y = yes; N = no)	1	--
15	Total weight at time of preparation (g)	PRWGT	Numeric	6	0
16	Date sample prepared for shipment (YYMMDD)	PRDATE	Numeric	6	0

Table 3 (con't)

Field Number	Field Description	Mnemonic	Type	Width	Places
17	Weight of fillet (g)	FWGT	Numeric	6	1.
18	Weight of remainder of carcass (g)	RCWGT	Numeric	6	1
19	Lipid content (%)	PCTLPD	Numeric	4	1
20	Aroclor 1221 (ppm)	AR21	Numeric	5	2
21	Aroclor 1016 (ppm)	AR16	Numeric	5	2
22	Aroclor 1248 (ppm)	AR48	Numeric	5	2
23	Aroclor 1254 (ppm)	AR54	Numeric	5	2
24	Aroclor 1260 (ppm)	AR60	Numeric	5	2
25	p, p' - DDE (ppm)	DDE	Numeric	5	3
26	trans-nonachlor (ppm)	TRANSNON	Numeric	5	3
27	Dieldrin (ppm)	DIELDRIN	Numeric	5	3
28	Mercury (ppm)	HG	Numeric	5	3
29	Decision on whether animal will appear in a composite	COMPDEC	Character (Y = yes; N = no)	1	--
30	Date fish composited (YYMMDD)	COMPDATE	Numeric	6	0
31	Number of fish in the composite; should be 5	NOINCOMP	Numeric	1	0
32	Tag number of one fish selected as an individual on which to key the others	KEYTAG	Character	6	--
33	Tag numbers of other fish in the composite sample	KEYED	Character	27	--
34	Average total length (mm) of individuals in the composite	AVLEN	Numeric	5	0

Table 3 (Con't)

Field Number	Field Description	Mnemonic	Type	Width	Places
35	Standard deviation of total length of individuals in the composite	SDLEN	Numeric	7	2
36	Minimum total length of an individual in the composite	MINLEN	Numeric	5	0
37	Maximum total length of an individual in the composite	MAXLEN	Numeric	5	0
38	Average total lab preparation weight (g) of individuals in the composite	AVWGT	Numeric	6	0
39	Standard deviation of total lab preparation weight of individuals in the composite	SDWGT	Numeric	8	2
40	Minimum total lab preparation weight of individuals in the composite	MINWGT	Numeric	6	0
41	Maximum total lab preparation weight of individuals in the composite	MAXWGT	Numeric	6	0
42	Average weight of fillets (g) in the composite	AVFWT	Numeric	6	1
43	Standard deviation of of fillet weights in the composite	SDFWF	Numeric	7	2
44	Minimum fillet weight in the composite	MINFWT	Numeric	6	1
45	Maximum fillet weight in the composite	MAXFWT	Nuemric	6	1
46	Lipid content (%)	FPCLPD	Numeric	4	1
47	2,3,7,8 - TCDD (ppt)	TCDD	Numeric	5	1

Table 3 (Con't)

Field Number	Field Description	Mnemonic	Type	Width	Places
48	Total dioxin (ppt)	DIOXTOT	Numeric	5	1
49	2,3,7,8 - TCDF (ppt)	TCDF	Numeric	5	1
50	Total dibenzofuran (ppt)	FURATOT	Numeric	5	1
51	Congeneric PCB - List dependent on analytical results				
52	Average weight of of remainders of carcasses (g) in the composite	AVRCWT	Numeric	6	1
53	Standard deviation of remainders of carcasses in the composite	SDRCWT	Numeric	7	2
54	Minimum remainder of carcass weight in the composite	MINRCWT	Numeric	6	1
55	Maximum remainder of carcass weight in the composite	MAXRCWT	Numeric	6	1
56	Lead (ppm)	PB	Numeric	5	3
57	Acid extractables - List dependent on analytical results				
58	Base neutrals - List dependent on analytical results				
59	Code for having rechecked data and verified accuracy of the record	VERIFIED	Character (Y = yes; N = no)	1	--

The variable list is dependent upon the actual array of compounds determined as a consequence of chemical analyses. Hence, the number of fields in the data file will be greater than the 58 specified.

Given the relatively small samples sizes at each location (Table 2) and problems of attaining the same species at all sites, a complete analysis of variance may not be possible. A priori comparisons may reinforce intuitive reasons for combining some locations or collections for statistical analysis. Both hydrological and biological considerations are important in such determinations. Hence, review of procedures, methodology, data and draft reports by the principal participants is critical as the project progresses.

Parametric and non-parametric statistical analysis procedures will be employed, as appropriate.

E. TASK 4 - REPORTING

Laboratory results and collection records with accompanying biological data are submitted to the Task Manager in Albany as they come available. This information as it is tabulated will be transmitted to the Bureau of Toxic Substances Assessment for health risk-assessment (HRA) purposes. A draft technical report from a resource perspective will be prepared prior to the HRA. When the HRA is completed, the two segments are to be amalgamated into a final technical report. Data developed prior to this study will also be used as background material to provide the most comprehensive package possible.

Although data will be reviewed with the interested parties during the course of the project, a public release on results and conclusions will not occur until the final report is completed and approved.

The intended audience of the final document includes members of the Mohawk Nation, scientific and technical entities, interested lay publics, and the involved or affected staffs of federal, state and industrial organizations.

F. TASK 5 - ADMINISTRATION

A schedule of activities and products resulting from this project is presented in Table 4.

Element 4 of this document provides an overview of project organization and responsibility.

Project fiscal information (includes the $180,000 available through General Motors Corporation):

	Cost	
Task 1: Collections		
Personnel (Akwesasne)		
Collection Teams (2 men per team x 4 teams x $350/wk x 9 weeks)	$25,200	
Advisor	5,000	
Coordinator	4,800	
Technician	4,500	
Administrative Overhead	12,000	
Subtotal		$51,500
Materials and Supplies		
- Gill nets and collecting equipment	5,300	
- Weighing scales and measuring boards	300	
- Thermometers (2) - mercury, -30°C to 50°C	30	
- Gas and Oil (outboard motors)	1,530	
- Life Jackets (9), fire extinguishers	260	
- Insulated Coolers (5)	320	
- Ice	500	
- Miscellaneous	1,700	
- Plastic bags	100	
Subtotal		$10,040
Travel	$ 350	
Subtotal - Task 1		$61,890

Task 2 - Preparation, Shipment and Laboratory Analyses		
Shipping	$ 850	
Laboratory Analyses		
- Hazleton (organochlorines, Pb, Hg, acid extractables, base neutrals, sample prep, lipids)	121,310	
- PCDD, PCDF, congeneric PCB (contract lab being sought)	55,000	
Travel	600	
Subtotal - Task 2		$171,760
Task 3 - Data Management and Statistical Analysis		
Personnel (30 staff days @ $200/day)	$6,000	
Travel	200	
Computer Maintenance and Software Costs	1,500	
Subtotal - Task 3		$ 7,700
Task 4 - Reporting		
Personnel (30 staff days @ $200/day)	$6,000	
Travel	250	
Subtotal - Task 4		$ 6,250
Task 5 - Admnistration		
Personnel (15 staff days at $200/day)	$3,000	
Travel	200	
Administrative overhead (all tasks exclusive of Task 1)	10,500	
Subtotal - Task 5		$ 13,700
TOTAL	$271,300	

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Table 4 Schedule of activities and products related to contaminant monitoring of fish from waters in and near the Akwesasne Reservation of the Mohawk Nation.

Activity	Month/date
Update sampling and quality assurance plan	11/1/87
Initiate contract approval process - laboratory - field crews	 9/10/87 9/29/87
Finalize contracts or letters of agreement	1/15/88
Final draft of sampling and quality assurance plan	2/1/88
Begin field sampling	4/1/88
Fnd field sampling	6/1/88
Preparation of samples for shipment to contract lab (Hazleton)	4/15-6/30/88
Shipments to Hazleton	5/1-6/30/88
All analytical results from Hazleton	8/1/88
Select representative samples for lab performance, PCDF, PCDD, Congeneric PCB	7/15/88
Analytical results from lab conducting PCDF, PCDD, and Congeneric PBC analyses	10/1/88
Submit analytical results to DOH for HRA	10/15/88
Draft technical report	11/1/88
Final technical report (DEC) and HRA (DOH)	1/1/89

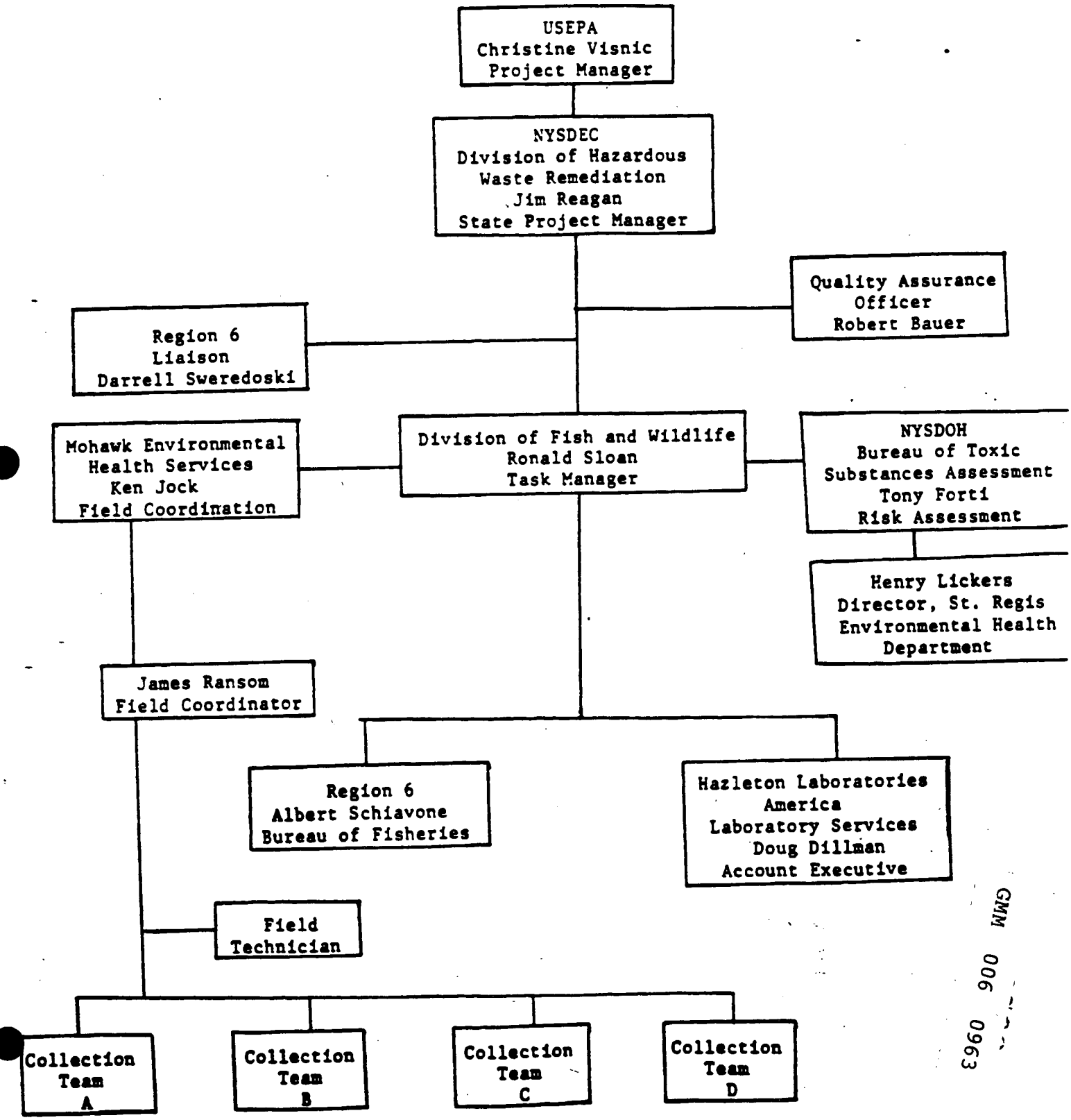
PROJECT ORGANIZATION AND RESPONSIBILITY:

Project management is depicted in Figure 3. The following is a list of key project personnel and their corresponding responsibilities:

Christine Visnic (USEPA)	Federal oversight for fiscal accountability and project QA with respect to meeting needs for Superfund
James Reagan (NYSDEC/Div. HW)	Departmental administration of funds and description of responsibilities; general oversight for project progress and integrity
Robert Bauer (NYSDEC/Div. FW)	Performance auditing Systems auditing Overall QA
Ronald Sloan (NYSDEC/Div. FW)	Overall project management Data processing Data QC Report preparation
Ken Jock (Mohawk Environmental Health Services)	Oversight of field sampling operations QC of field operations
James Ransom (Mohawk Environmental Health Services)	Coordination of field sampling Handling and preparation of samples Shipping QC of collections, preparation and shipping
Tony Forti (NYSDOH)	Evaluation of contaminants data for health risk assessment including applicable QA aspects Report preparation
Henry Lickers (St. Regis Environmental Health Dept.)	Interpretation/evaluation of contaminants data as related to Mohawk Nation affairs Input to final report
Darrell Sweredoski (NYSDEC/Reg 6)	Liaison to Region 6 to provide information to the administration; feedback mechanism to project personnel

Albert Schiavone (NYSDEC/Reg 6 FW)	Oversight for Bureau of Fisheries Technical input, review and interpretation for resource concerns
Doug Dillman (Hazleton Laboratories)	Laboratory analyses QC of laboratory operations
To be determined (Contract laboratory for PCDF, PCDD and congeneric PCB)	Laboratory analyses QC of laboratory operations

Figure 3. Organizational responsibility for persons involved with the contaminant monitoring of fish from waters in and near the Akwesasne Reservation of the Mohawk Nation.



QA OBJECTIVES FOR MEASUREMENT DATA - PRECISION, ACCURACY, COMPLETENESS, REPRESENTATIVENESS AND COMPARABILITY:

Details on the data quality requirements and assessments are provided in Table 4 for the heavy metals (lead and mercury) and the organic compound groups (organochlorines, acid extractables, base neutrals, dioxins, and dibenzofurans).

Oftentimes, due to the necessity to simplify terms, information is lost. This is the case with distinguishing between detection and quantitation limits. In practice they have been used synonymously. Realistically, the detection limits presented in Table 4 are quantitation limits. The listed values are not reflecting instrument sensitivity. For working purposes, however, the resulting analyses will provide reliable data.

For the acid extractables, base neutrals, dioxins and dibenzofurans, the analytical methodologies, particularly in biological tissues and their large scale application, are in the developmental stages. Hence, the values on accuracy and precision are rather arbitrary. As more laboratories enter into this realm of analytical measurement, and the QA database expands, the figures given will undoubtedly change. For this project, the estimates provided are felt to be reasonable targets. The higher precision limits used to judge laboratory performance were established arbitrarily, usually as a multiple by five of the quantitation limit. Until sufficient data are available to establish precision and accuracy protocols it is desirable to strive for analytical results at or below the specified limits.

Data representativeness: Fish samples shall consist of edible portions (i.e. standard fillets as indicated in Appendix A) and sizes where appropriate for human health concerns. The general sample design is provided in Table 2. Composites of the remainders of the carcasses will be anlayzed for lead since the liver, kidney and bones are likely sites for lead accumulation. If lead concentrations are high (i.e. greater than 1 ppm on a wet weight basis) comparable standard fillets will be analyzed. The 36 samples slated for acid extractable and base/neutral screens will also involve remainders of carcasses. The 36 samples for dioxin and dibenzofuran analyses will consist of composites of standard fillets.

Data comparability: Analyses will be performed for all fish with the exceptions noted above, on standard fillets. Comparisons will be made on both wet weight and lipid bases with results reported in parts per million (ppm) or ug/g. Dioxins and dibenzofurans are reported in parts per trillion (ppt) or picograms/g.

Data completeness: Data will be considered complete when the minimum required numbers of samples are collected and all results are returned from the laboratory.

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able 5: Data Quality Requirements and Assessments:

Element/Compound	Sample Matrix	Detection Limit (ppm)	Quantitation Limit (ppm)	Estimated Accuracy	Accuracy Protocol	Estimated* Precision (ppm)	Precision Protocol
Metals and Organochlorines							
Mercury	Fish tissue	0.025	0.025	±30%	A minimum of 5% of the samples analyzed shall be quality assurance for spiked recoveries.	0.063	±20%
Lead	Fish tissue	0.02	0.02	±30%		0.100	±20%
Aroclor 1016	Fish tissue	0.05	0.05	±30%		0.649	±20%
Aroclor 1221	Fish tissue	0.05	0.05	±30%		0.649	±20%
Aroclor 1232	Fish tissue	0.05	0.05	±30%		0.649	±20%
Aroclor 1242	Fish tissue	0.05	0.05	±30%		0.649	±20%
Aroclor 1248	Fish tissue	0.05	0.05	±30%		0.649	±20%
Aroclor 1254	Fish tissue	0.05	0.05	±30%		0.649	±20%
Aroclor 1260	Fish tissue	0.05	0.05	±30%		0.649	±20%
p,p' -DDT	Fish tissue	0.01	0.01	±24%		0.033	±20%
p,p' -DDE	Fish tissue	0.01	0.01	±24%		0.033	±20%
p,p' -DDD	Fish tissue	0.01	0.01	±24%		0.033	±20%
Aldrin	Fish tissue	0.01	0.01	±24%		0.050	±20%
Dieldrin	Fish tissue	0.01	0.01	±24%		0.050	±20%
α -HCH	Fish tissue	0.01	0.01	±24%		0.050	±20%
β -HCH	Fish tissue	0.01	0.01	±24%		0.050	±20%
γ -HCH	Fish tissue	0.01	0.01	±24%		0.050	±20%
δ -HCH	Fish tissue	0.01	0.01	±24%		0.050	±20%
Endrin	Fish tissue	0.01	0.01	±24%		0.050	±20%
Heptachlor	Fish tissue	0.01	0.01	±24%		0.050	±20%
Heptachlor expoxide	Fish tissue	0.01	0.01	±24%		0.050	±20%
cis-Chlordane	Fish tissue	0.01	0.01	±24%		0.050	±20%
trans-Chlordane	Fish tissue	0.01	0.01	±24%		0.050	±20%
cis-Nonachlor	Fish tissue	0.01	0.01	±24%		0.050	±20%
trans-Nonachlor	Fish tissue	0.01	0.01	±24%		0.050	±20%
Oxychlordane	Fish tissue	0.01	0.01	±24%		0.050	±20%
Compound "B"	Fish tissue	0.01	0.01	±24%		0.050	±20%
Toxaphene	Fish tissue	0.01	0.01	±24%		0.050	±20%
Mirex	Fish tissue	0.01	0.01	±24%		0.050	±20%
Hexachlorobenzene	Fish tissue	0.01	0.01	±24%		0.050	±20%

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ible 5 (Continued).

lement/Compound	Sample Matrix	Detection Limit (ppm)	Quantitation Limit (ppm)	Estimated Accuracy	Accuracy Protocol	Estimated* Precision (ppm)	Precision Protocol
cid Extractables							
-Chlorophenol	Fish tissue	0.8	0.8	±30%	A minimum of 5% of the samples analyzed shall be quality assurance for spiked recoveries.	4.0	±30%
-Nitrophenol	Fish tissue	0.8	0.8	±30%		4.0	±30%
-Nitrophenol	Fish tissue	2	2.0	±30%		10.0	±30%
,4-Dichlorophenol	Fish tissue	0.8	0.8	±30%		4.0	±30%
,4-Dimethylphenol	Fish tissue	0.8	0.8	±30%		4.0	±30%
,6-Dinitro-o-cresol	Fish tissue	2	2.0	±30%		10.0	±30%
,4-Dinitrophenol	Fish tissue	2	2.0	±30%		10.0	±30%
-Chlor-m-cresol	Fish tissue	0.8	0.8	±30%		4.0	±30%
entachlorophenol	Fish tissue	0.8	0.8	±30%		4.0	±30%
henol	Fish tissue	0.8	0.8	±30%		4.0	±30%
,4,6-Trichlorophenol	Fish tissue	0.8	0.8	±30%		4.0	±30%
-Methyl-4,5-dinitrophenol	Fish tissue	0.8	0.8	±30%		4.0	±30%
-Chloro-3-methyl phenol	Fish tissue	0.8	0.8	±30%		4.0	±30%
,4,6-Trichlorophenol	Fish tissue	0.8	0.8	±30%		4.0	±30%
Base Neutrals							
1,3-Dichlorobenzene	Fish tissue	0.8	0.8	±30%		4.0	±30%
1,4-Dichlorobenzene	Fish tissue	0.8	0.8	±30%		4.0	±30%
Acenaphthene	Fish tissue	0.8	0.8	±30%		4.0	±30%
Acenaphthylene	Fish tissue	0.8	0.8	±30%		4.0	±30%
Anthracene	Fish tissue	2	2	±30%		10.0	±30%
Benzidine	Fish tissue	0.8	0.8	±30%		4.0	±30%
Benzo(a)anthracene	Fish tissue	0.4	0.4	±30%		2.0	±30%
Benzo(a)pyrene	Fish tissue	0.4	0.4	±30%		2.0	±30%
Benzo(b)fluoranthene	Fish tissue	0.4	0.4	±30%		2.0	±30%
Benzo(g,h,i)perylene	Fish tissue	0.4	0.4	±30%		2.0	±30%
Benzo(k)fluoranthene	Fish tissue	0.4	0.4	±30%		2.0	±30%
bis(2-Chloroethoxy) methane	Fish tissue	0.8	0.8	±30%		4.0	±30%
bis(2-chloroethyl) ether	Fish tissue	0.8	0.8	±30%		4.0	±30%
bis(2-Chloroisopropyl) ether	Fish tissue	0.8	0.8	±30%		4.0	±30%
bis(2-Ethylhexyl) phthalate	Fish tissue	0.8	0.8	±30%		4.0	±30%
4-Bromophenylphenylether	Fish tissue	0.8	0.8	±30%		4.0	±30%

ble 5. (Continued).

ement/Compound	Sample Matrix	Detection Limit (ppm)	Quantitation Limit (ppm)	Estimated Accuracy	Accuracy Protocol	Estimated* Precision (ppm)	Precision Protocol
se Nuetrals (Con't)							
tylbenzylphthalate	Fish tissue	0.8	0.8	±30%	A minimum of 5% of the samples analyzed shall be quality assurance for spiked recoveries.	4.0	±30%
-Chloronaphthalene	Fish tissue	0.8	0.8	±30%		4.0	±30%
-Chlorophenylphenylether	Fish tissue	0.8	0.8	±30%		4.0	±30%
rysene	Fish tissue	0.4	0.4	±30%		2.0	±30%
lbenzo(a,h)anthracene	Fish tissue	0.4	0.4	±30%		2.0	±30%
,2-Dichlorobenzene	Fish tissue	0.8	0.8	±30%		4.0	±30%
,3-Dichlorobenzidine	Fish tissue	0.8	0.8	±30%		4.0	±30%
iethylphthalate	Fish tissue	0.8	0.8	±30%		4.0	±30%
imethylphthalate	Fish tissue	0.8	0.8	±30%		4.0	±30%
i-n-butylphthalate	Fish tissue	0.8	0.8	±30%		4.0	±30%
,6-Dinitrotoluene	Fish tissue	0.8	0.8	±30%		4.0	±30%
,4-Dinitrotoluene	Fish tissue	0.8	0.8	±30%		4.0	±30%
i-n-octylphthalate	Fish tissue	0.8	0.8	±30%		4.0	±30%
,2-Diphenylhydrazine	Fish tissue	0.4	0.4	±30%		2.0	±30%
luoranthene	Fish tissue	0.8	0.8	±30%		4.0	±30%
luorene	Fish tissue	0.8	0.8	±30%		4.0	±30%
exachlorobutadiene	Fish tissue	0.8	0.8	±30%		4.0	±30%
Hexachlorocyclopentadiene	Fish tissue	0.8	0.8	±30%		4.0	±30%
Hexachloroethane	Fish tissue	0.4	0.4	±30%		2.0	±30%
Ideno(1,2,3-ed)pyrene	Fish tissue	0.8	0.8	±30%		4.0	±30%
Isophorone	Fish tissue	0.8	0.8	±30%		4.0	±30%
Naphthalene	Fish tissue	0.8	0.8	±30%		4.0	±30%
Nitrobenzene	Fish tissue	16	16	±30%		30	±30%
N-Nitrosodimethylamine	Fish tissue	16	16	±30%		30	±30%
N-Nitrosodi-n-propylamine	Fish tissue	0.8	0.8	±30%		4.0	±30%
N-Nitrosodiphenylamine	Fish tissue	2	2	±30%		10	±30%
Phenanthrene	Fish tissue	0.4	0.4	±30%		2.0	±30%
Pyrene	Fish tissue	0.8	0.8	±30%		4.0	±30%
1,2,4-Trichlorobenzene	Fish tissue	0.8	0.8	±30%		4.0	±30%
1,2-Dichlorobenzene	Fish tissue	0.8	0.8	±30%		4.0	±30%
2,5-Dinitrotoluene	Fish tissue	0.8	0.8	±30%		4.0	±30%
Dioxins (Congeneric groups)	Fish tissue	1 ppt	5 ppt	±50%		25 ppt	±50%
2,3,7,8-TCDD	Fish tissue	1 ppt	5 ppt	±50%		25 ppt	±50%
Dibenzofurans (Congeneric groups)	Fish tissue	1 ppt	5 ppt	±50%		25 ppt	±50%
2,3,7,8-TCDF	Fish tissue	1 ppt	5 ppt	±50%		25 ppt	±50%

0690

GMW 900 0967

SAMPLING PROCEDURES

The selection of sampling sites, probable species and general experimental design were presented in Section 3. Actual sampling will utilize standard methods of netting including gill, trap and seine plus hook-and-line angling. Following the instructions in Appendix A is necessary and where applicable, fish must be of edible size. Collection data are recorded on the Fish/Wildlife Collection Record (Appendix A).

Sampling will occur during the spring spawning period. As part of the sampling process, the functions and specific activities for two key field groups are listed chronologically below:

Collectors

Capture specimen.
Identify to species
(use accepted common or scientific names as per American Fisheries Society 1980).
Jaw tag individual fish.
Bag fish individually.
Complete bag tag and attach to bag.
Fill-in collection record form as appropriate.
Initiate and sign continuity of evidence form.
Mark collection point on topographic map and attach to continuity and collection forms.
Place fish on ice or transport immediately to freezer (i.e. field coordinator or field technician).
If on ice, get to freezer within 12 hours.
Coolers used in the field must be washed with soap and water daily. Rinse with clean water. Store and use away from contact with gas and oil. All materials used for bagging and tagging of specimens cannot come in contact with other potential contaminants. Any materials becoming cross-contaminated (e.g. bags and bag tags) must be discarded. Metal items so soiled must be thoroughly washed (soap and water) or discarded.

Field technician and coordinator

Receive collections from collector(s).
Sign continuity of evidence form.
Fill-in collection record form
- measure (total length - mm)
- weigh (g)
- sex, if possible without cutting.

Retain original forms; provide copy to collector(s).
Freeze samples.
Update freezer log and keep freezers when not in attendance in locked condition.
Transfer samples in a frozen state to Hale Creek Field Station.
Prepare standard fillets (individual analyses) according to procedures put forth in Appendix A.
Prepare composite samples as discussed in Section 3.
Prior to cutting, re-weigh whole fish and record.
Sex fish and record on collection record forms.
Grind and homogenize samples.
Rinse tissue jars (See next page).
Pack homogenate, minimum of 25 grams, in glass jars with aluminum foil lined lids; DO NOT OVERPACK! Use hexane rinsed foil for lid liners.
Label the jars.
Place each jar of homogenate in an unused bag; also tag the bag.
Re-tag excess tissue in an unused bag; tag the bag.
Freeze the samples in insulated shipping containers; DO NOT CLOSE THE LIDS!

Collectors

Field technician and coordinator (cont.)

Specimens are shipped Federal Express to designated laboratory performing a particular group of analyses (e.g. Hazleton for Hg, Pb, organo-chlorines, acid extractables and base neutrals).
Samples are shipped on Mondays or Tuesdays only!
Call the laboratory on the day of shipment; provide the contact with number of coolers, total number of samples and air bill number; confirm arrival the following day; if samples are lost, initiate immediate trace.

All glass containers to be used for ground, homogenized tissues receive an acid/solvent rinse. All cutting, grinding, and homogenizing equipment are washed between each sample. Packing of shipping containers with glass sample jars is done so that breakage does not occur.

The use of plastic bags for holding frozen whole fish is a standard procedure at NYSDEC laboratories. The supplier of bags has been kept constant since 1977 and in 1986, a round-robin conducted by HLA between seven laboratories showed that storage-container type (aluminum foil, plastic bag, and glass jar) did not influence the variability of PCB content (Hill, 1986). In this project, the homogenates slated for analysis, however, will be stored in glass and the remaining excess tissue only is repackaged in unused plastic bags.

Originals of all continuity of evidence and collection record forms are sent to the Task Manager. He also receives copies of the Analysis Request forms. The Field Coordinator should recieve or retain copies of all completed forms.

The general schedule for collections and shipping is provided under Task 4 in Section 3. The maximum holding time for any sample in this project is 9 months. All samples are disposed of via incineration or through a certified waste hauler upon completion of the project.

As information on collections and data from the laboratories are received by the Task Manager, it is entered into the computer as described in Section 3, Task 4.

SAMPLE CUSTODY

The Continuity of Evidence form is one item relied upon for field sampling documentation, coupled with the Collection Record form and proper designation of sampling points on topographic sheets. Until samples are prepared for shipment and the Analysis Request forms are completed, the Continuity of Evidence form is the principal tracking tool. At the laboratory, samples are assigned a laboratory number in addition to maintaining the unique identity of the original field tag number. The dual designation then allows complete tracking from field collections through laboratory analysis and finally into the completed datafile.

Samples of Continuity of Evidence and Analysis Request forms are provided in Appendix A. A freezer log is maintained at the freezer facilities at Akwesasne and Hale Creek. A sample of that form also appears in Appendix A. Only persons associated with the project or laboratory personnel are authorized to log-in samples. These people are also responsible for maintaining the freezers in a locked condition.

Standard operating procedures at Hazleton Laboratories provides for sample security and tracking (Appendix B). This will also be a consideration for final approval of a laboratory conducting dioxin and dibenzofuran analyses.

At all points of transfer, the person taking receipt is reponsible for all samples listed. Therefore, careful checking of contents and verification is critical!

To ensure the adequate preservation of samples, freezer temperature logs are maintained on a daily basis. A sample of the freezer log is included in Appendix A. Freezer conditions are specified in Appendix A. The Field Coordinator is responsible for assuring the maintenance of this log. In case of freezer failure, all samples should be transferred immediately to the Hale Creek Field Station and the Task Manager is notified. Other back-up freezer facilities are available if necessary (e.g. other DEC laboratories or in the Bureau of Fisheries facilities in Regions 5 and 6) but these are to be used only in the event that the Hale Creek facility is also experiencing power failure or breakdown.

CALIBRATION PROCEDURES AND FREQUENCY

Refer to Appendix B.

ANALYTICAL PROCEDURES

Refer to Appendix B.

DATA REDUCTION, VALIDATION AND REPORTING

To validate data after receipt of analytical results, data are checked for obvious errors (e.g. misprints, typographical errors, and absurd values). Potential outliers are rechecked by the laboratory for mathematical errors. If no errors, the sample is rerun. Evaluation of quality control data within the specifications of Table 5 is conducted. Unacceptable results are questioned. If cause for the result is not determined or indicates a problem, the entire run involving the spurious datum is redone.

There are two situations in which data reduction occurs. At time of collection, due to sample size constraints fish are sorted to provide the species and size mixes as outlined in Section 3. The second situation involves the interpretation, graphing, tabulation, summarization and evaluation through standard statistical procedures of the biological and chemical data. This includes appropriate checks for normality, necessity for transformations or coding, and parametric vs. non-parametric tests. The influence of outliers may be lessened or eliminated via transformations (e.g. $\log_{10}$). If the outlier does present an interpretive problem and it is a valid result, the data may be analyzed both ways, with and without the particular result. In either case, the condition would appear in the technical reports resulting from the project.

INTERNAL QUALITY CONTROL CHECKS

During an analytical run of 20 samples, three are of a quality control nature: one duplicate, one spiked recovery, and a blank. They should fall within the limits specified in Table 5.

Normal operating procedures call for twice daily inspections of: Chemical assay procedures and validation, reagent preparation and labelling, controls and standards, instrument calibration and maintenance, analytical results, data recording, and analysis and archiving of data. An Internal Operating Procedure (IOP) manual detailing use, calibration and maintenance is kept with each item of analytical equipment.

PERFORMANCE AND SYSTEM AUDITS

The QA officer(s) will conduct and report on several phases of operation at the times specified:

Collections (collecting and storage)	1 visit - between April 1 and May 30, 1988
Preparation of samples	1 visit - between April 15 and June 30, 1988
PCDD and PCDF analyses	1 visit - before March 30, 1988 or utilize results from existing USEPA audits
Organochlorines and metals	None planned

PREVENTIVE MAINTENANCE PROCEDURES AND SCHEDULES

See Section 8.

ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY, AND COMPLETENESS

These items were discussed in Sections 5, 10, and 11.

CORRECTIVE ACTION

When a QC sample falls outside the control limits, the first step is to check for obvious errors in calculation or reporting. If none are found, the results on the 17 samples are voided and the samples re-run.

Any QA/QC problems and the corresponding action must be documented in writing between the parties concerned such as the Task Manager and the QA officer.

If deficiencies in laboratory or field phases are determined by the QA officer(s) during the course of the project their recommendation(s) are implemented or their concerns met as they arise.

QUALITY ASSURANCE REPORTS TO MANAGEMENT

The final report will have a section summarizing analytical QA data and interpretation of the results. Pertinent findings by the QA officer(s) from their field and laboratory system audits will also be included.

Audits by the QA officer(s) will occur in three parts: 1) during the collection periods; 2) preparation procedures at Hale Creek; and 3) analyses for dioxins and dibenzofurans. An audit was conducted on Hazleton Laboratories in 1985 during contract negotiations. Results were favorable, and hence, it will not be repeated during this project. The laboratory conducting PCDD and PCDF analyses will be audited, either by a site visit or reliance upon previous audits by the USEPA, prior to contract finalization. The laboratory conducting the PCDD and PCDF under consideration is or was under contract to USEPA for these analyses. Hence, information on previous audits should be available.

REFERENCES

Hill, S. 1986. Striped bass round-robin 1985. Hazleton Laboratories America, Inc. Madison, Wisconsin. 8 p.

New York State Department of Environmental Conservation. 1978. Toxic Substances in Fish and Wildlife, Volume 1. Tech. Rep. 78-1 (BEP), Division of Fish and Wildlife, New York State Department of Environmental Conservation, Albany, NY. 52 p.

New York State Department of Environmental Conservation. 1981. Toxic Substances in Fish and Wildlife, Volume 4, Number 1. Tech. Rep. 81-1 (BEP). Division of Fish and Wildlife, New York State Department of Environmental Conservation, Albany, NY. 138 p.

New York State Department of Environmental Conservation. 1987a. Toxic Substances in Fish and Wildlife, Volume 6. Technical Report 87-4 (BEP), Division of Fish and Wildlife, New York State Department of Environmental Conservation, Albany, NY. 182 p.

New York State Department of Environmental Conservation. 1987b. Health Advisory. New York State Fishing, Small Game Hunting, Trapping Regulations Guide. Division of Fish and Wildlife, New York State Department of Environmental Conservation, Albany, NY. 96 p.

Robins, C.R., R.M. Bailey, C.E. Bond, J.R. Brooker, E.A. Lachner, R.N. Lea and W.B. Scott. 1980. A List of Common and Scientific Names of Fishes from the United States and Canada (Fourth Ed.) Spec. Public. No. 12, Am. Fish. Soc., Bethesda, MD. 174 p.

STSC. 1986. Statgraphics: Statistical Graphics System by Statistical Graphics Corporation. STSC, A Contel Co., Rockville, MD.

U.S. Environmental Protection Agency. 1980. Interim guidelines and specifications for preparing quality assurance project plans. Office Monitoring Systems and Quality Assurance, Office of Research and Development, U.S. Environmental Protection Agency, Washington, D.C. QAMS-005/80.

APPENDIX A

STANDARD OPERATING PROCEDURES

(FISH COLLECTIONS)

COLLECTION PROCEDURES

A. Following data are to be taken on each fish collected:

1. Date collected.
2. Species identification (be explicit enough to enable assigning genus and species).
3. Total length and weight.
4. Method of collection (gill net, hook and line, etc.)
5. Sample location (Waterway and nearest prominent identifiable landmark).
6. Sex - fish may be cut enough to allow sexing but do not eviscerate.
7. Tag number (each specimen to be individually tagged with jaw tag).

The original of all collection record and continuity of evidence forms shall accompany delivery of fish to the lab. A copy shall be directed to the project leader. All necessary forms will be supplied by the Bureau of Environmental Protection.

B. Each fish to be wrapped in a plastic bag. The Bureau of Environmental Protection will supply the bags.

C. Groups of fish, by species, to be placed in one large plastic bag per sampling location. The Bureau of Environmental Protection will supply all bags.

D. Do not eviscerate.

E. All fish must be kept at a temperature below 45°F immediately following data processing. As soon as possible freeze at 0°F ± 10°F. Due to occasional freezer failures, daily freezer temperature logs are required.

F. Prior to any delivery of fish, coordinate delivery with and send copies of the collection records and continuity of evidence forms to:

Ronald Sloan, Ph.D.
Bureau of Environmental Protection
Division of Fish and Wildlife
50 Wolf Road, Room 530
Albany, New York 12233
Telephone: 518-457-1769

Samples will then be directed to the analytical facility and personnel noted on specific project descriptions.

NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION

BUREAU OF ENVIRONMENTAL PROTECTION

FISH PREPARATION PROCEDURES FOR CONTAMINANT ANALYSIS

Background

New York State DEC conducts studies requiring chemical analysis on fish tissues. Routine monitoring and surveillance studies develop data on contaminants in fish for several reasons:

1. To identify sources of environmental contamination.
2. To identify the geographic extent of environmental contamination.
3. To identify temporal trends of contaminants in fish and wildlife.
3. To provide information regarding human consumption advisories.

Chemical analyses of edible fish flesh have been determined to be the most appropriate analyses for satisfying all of these objectives. The following methodology has been developed in order to standardize the tissue under analysis and to adequately represent the contaminant levels of fish flesh. This methodology is slightly modified from the U.S. Food and Drug Administration procedures. The portion of edible flesh analyzed will be referred to as the standard fillet unless otherwise noted. For some species, the procedure is modified as indicated below.

Procedure for Standard Filleting

1. Remove scales from fish. Do not remove the skin.
2. Make a cut along the ventral midline of the fish from the vent to the base of the jaw.
3. Make diagonal cut from base of cranium following just behind gill to the ventral side just behind pectoral fin.
4. Remove the flesh and ribcage from one-half of the fish by cutting from the cranium along the spine and dorsal rays to the caudal fin. The ribs should remain on the fillet.
5. Score the skin and homogenize the entire fillet.

Modifications to Standard Fillet

Four modifications of the standard fillet procedure are designed to account for variations in fish size or known fish preparation methods for human consumption.

1. Some fish are too small to fillet by the above procedure. Fish less than approximately 6 inches long and rainbow smelt are analyzed by cutting the head off from behind the pectoral fin and eviscerating the fish. Ensure that the belly flap is retained on the carcass to be analyzed. When this modification is used, it should be noted when reporting analytical results.

2. Some species are generally eaten by skinning the fish. The skin from these species is also relatively difficult to homogenize in the sample. Hence, for the following list of species, the fish is first skinned prior to filleting:

Brown bullhead	White catfish
Yellow bullhead	Channel catfish
Atlantic sturgeon	Lake sturgeon
Black bullhead	

3. American eel are analyzed by removing the head, skin, and viscera; filleting is not attempted.

4. Forage fish and young-of-year fish are analyzed whole. This category is considered to be less than 150 mm (6 inches).

5. For large fish (i.e. greater than four feet total length) and for which grinding and homogenizing an entire fillet is inappropriate (i.e. Lake sturgeon and Atlantic sturgeon), "steaks" from three sections are removed and composited. Steaks from the anterior, middle, and posterior areas are cut one-inch thick, dorso-ventrally, with belly flap included from the pertinent sectors. The resulting mass will include the backbone. Anterior is defined as that area immediately behind the head or the base of the cranium. Middle is determined by either measuring half the distance from the base of the cranium to the distal end of the caudal peduncle or that area under the dorsal fin. Posterior is defined as that longitudinal section one inch anterior to the distal end of the caudal penduncle.

82-14-64 (7/85)

NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION

CHAIN OF CUSTODY

I, _____ (Print Name), of _____ (Print Address) have collected the on _____, 198__ from _____ in the vicinity of _____ Town of _____, _____ County.

Items: _____

_____

_____

said sample(s) were in my possession and handled according to standard procedures provided to me prior to collection. The sample(s) were placed in the custody of a representative of the New York State Department of Environmental Conservation on _____, 198__.

_____ Signature _____ Date

I, _____, have received the above mentioned samples on the date specified and have assigned identification number(s) _____ to the sample(s). I have recorded pertinent data for the sample(s) on the attached collection records. The sample(s) remained in my custody until subsequently transferred, prepared or shipped at times and dates as attested to below.

_____ Signature _____ Date

SECOND RECIPENT (Print Name)	TIME AND DATE	PURPOSE OF TRANSFER
SIGNATURE	UNIT	
THIRD RECIPENT (Print Name)	TIME AND DATE	PURPOSE OF TRANSFER
SIGNATURE	UNIT	
FOURTH RECIPENT (Print Name)	TIME AND DATE	PURPOSE OF TRANSFER
SIGNATURE	UNIT	
RECEIVED IN LABORATORY BY (Print Name)	TIME AND DATE	
SIGNATURE	UNIT	
LOGGED IN BY (Print Name)	TIME AND DATE	ACCESSION NUMBERS:
SIGNATURE	UNIT	

SEE REVERSE SIDE

GMM 006 0985

82-14-61(2/84)

FISH/WILDLIFE COLLECTION RECORD
NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION
DIVISION OF FISH AND WILDLIFE

FROM REGION_____FOR_____TOXIC SUBSTANCE MONITORING PROGRAM

BY COLLECTOR(S)_____USING_____COLLECTION METHOD.

SPECIMENS PRESERVED BY_____METHOD.

FILL IN APPROPRIATE BLANKS AS COMPLETELY AS POSSIBLE.

FOR LAB USE ONLY LAB ENTRY NO.	COLLECTION OR TAG NO.	SPECIES	DATE TAKEN	LOCATION	AGE	SEX &/OR REPROD. CONDIT.	LENGTH ( )	WEIGHT ( )	REMARKS

9860 900 GMM

FISH/WILDLIFE COLLECTION LOG
NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION
DIVISION OF FISH AND WILDLIFE

LAB NO.	TAG NO.	SPECIES	LOCATION	PROGRAM	LOG IN		PROCESSING			FINAL DISPOSITION		REMARKS
										CARCASS	GROUND	
					DATE	INIT	DATE/INIT	DATE/INIT	DATE/INIT	LOC/DATE/INIT	LOC/DATE/INIT	

App. A-6

GMM 006 0987

HAZLETON LABORATORIES AMERICA, INC.

ANALYSIS REQUEST

FOR HAZLETON USE ONLY

…RGE NUMBER: 25 43

MNEM:

…PANY NAME AND ADDRESS (1)

…ONE NUMBER

NAME OF SUBMITTER

…ND INVOICE TO

SEND REPORTS TO

…RCHASE ORDER NUMBER

DATE SENT

ENCLOSE WITH SAMPLES AND SEND TO:

HAZLETON LABORATORIES AMERICA, INC.
Chemical and BioMedical Sciences Division
3301 KINSMAN BOULEVARD
P.O. BOX 7545
MADISON, WISCONSIN 53707
(608) 241-4471

(PLEASE TYPE OR PRINT)
SHADED AREAS FOR HAZLETON USE ONLY
SEE REVERSE SIDE FOR INSTRUCTIONS

SEND ADDITIONAL FORMS | |

…HADED AREAS FOR HAZLETON USE ONLY

…RT CODE	SAMPLE DESCRIPTION (2)	SAMPLE CODE (3)	ANALYSES REQUESTED (4)	ESTIMATED LEVEL (5)

SPECIAL INSTRUCTIONS:(6)

GMM 006 0988

App. A-7

…See Back of Form

FREEZER TEMPERATURE LOG

LOCATION _____ IDENTIFICATION (for multiple units) _____

DATE	TEMPERATURE (°C/F°) (circle units used)	TIME	RECORDER

HOMOGENIZATION

A) Depending upon sample size either the large or small food chopper is used for homogenization.

B) After the sample has been put through the food chopper, the chopper should be partially disassembled so that any remaining skin material can be removed. The skin should be cut with a knife or scissors and then combined with the rest of the sample.

C) The sample is put through the food chopper three times.

D) The sample should then be homogenized with the mixer or, if the sample is too small, by hand with a spatula. 2 - 5 Minutes.

E) The homogenized sample is then subsampled into appropriate glass bottles. Generally 2 - 10 g for metals, and 20 - 40 g for organochlorine analysis.

F) The bottles are labeled with the sample Lab # and stored in the freezer.

G) All homogenization tools are to be rinsed, washed with soap solution, rinsed, rinsed with distilled water and dried between each sample.

H) The date and initials are recorded both on the collection log and the sample analysis report.

APPENDIX B

LABORATORY PROCEDURES

Hazleton Laboratories America
Madison Wisconsin
1987

ORGANOCHLORINES

The determination of organochlorine pesticides and PCBs is a frequent, high-volume analysis at HLA. Hundreds of samples per year are analyzed for organochlorine pesticides in a variety of matrices. Most of the organochlorine pesticides can be screened for in a single, multicomponent assay. HLA's method is based on the EPA procedure "Method for Organochlorine Pesticides in Industrial Effluents." This method is valid for lindane, heptachlor, aldrin, heptachlor epoxide, alpha-BHC, beta-BHC, HCB, PCNB, and trifluralin at 0.01 µg/L; endrin, dieldrin, delta-BHC, Endosulfan I, Endosulfan II, DDE, DDD, p,p'-DDT, and o,p'-DDT at 0.05 µg/L; mirex, methoxychlor, endosulfan sulfate, and endrin aldehyde at 0.1 µg/L; kelthane, kepone, and PCBs at 1.0 µg/L; and toxaphene, perthane, and strobane at 5.0 µg/L. Three injections are required to separate these organochlorine pesticides. Thiodan II requires a separate injection, as does the dieldrin, endrin, Thiodan I, and endosulfan aldehyde combination. The remaining organochlorines can be obtained in one injection. All PCBs can be quantitated in one injection.

Typical Matrix

The following method summary is typical for most matrices analyzed for organochlorine compounds. Method references for individual matrices are given following this summary.

Extraction. Each composite is ground in a Hobart or other appropriate food grinder until homogeneous. Approximately 25 g of sample are weighed into a beaker and mixed with anhydrous sodium sulfate. The sample is allowed to air dry for 48 hours. The contents of the beaker are transferred to a Whatman extraction thimble (prewashed with dichloromethane and 50:50 ethyl ether:petroleum ether) and plugged with glass wool. The thimble is placed in a desiccator for at least 12 hours, after which it is removed, placed on a Soxhlet extractor, and extracted with 50:50 ethyl ether:petroleum ether for 8 hours. The resulting solution is concentrated on a steam bath and brought to appropriate volume with petroleum ether.

Florisil Cleanup. An aliquot of the extract is placed on a previously standardized Florisil column and eluted with 1% ethyl ether in petroleum ether, followed by 20% ethyl ether in petroleum ether. The resulting elutions are concentrated on a steam bath and brought to an appropriate volume with petroleum ether. Aliquots of 10 µL or less of each elution are injected into a gas chromatograph (GC) for quantitation.

Silica Gel Separation. For samples requiring further separation, an aliquot of the first Florisil column elution is transferred to a previously standardized Silica Gel 60 column and eluted twice with petroleum ether and then with a mixture of 1% acetonitrile, 19% hexane, and 80% methylene chloride. The first two elutions are concentrated on a steam bath and brought to an appropriate volume with petroleum ether. Aliquots or 10 µL or less of each elution are injected into a GC for quantitation.

Lipid Determination. An aliquot of the original extract is transferred to a tared 2-dram vial. The solvent is removed and the vial placed in a 40°C oven for 24 hours. The vial is removed, desiccated, and reweighed and the amount of lipid calculated.

Typical Gas Chromatography

Instrument: Hewlett Packard Model 5710A equipped with a linearized Ni^{63} detector, automatic injector (or equivalent), and 3356 data system

Column No. 1: For all chlorinated insecticides and PCB except chlordane isomers

- Packing: 1.5% SP-2250/1.95% SP-2401 on 80/100 mesh Supelcoport
- Column: 6 ft x 4 mm i.d. glass
- Temperatures:
  - Column: 208°C
  - Injector: 250°C
  - Detector: 300°C
- Carrier gas: 95% argon, 5% methane
- Flow: 35 mL/minute or adjusted to give DDE retention time of approximately 10.0 minutes

Column No. 2: For chlordane isomers

- Packing: 3% OV-1 on 80/100 mesh Supelcoport
- Column: 6 ft x 4 mm i.d. glass
- Temperature:
  - Column: 200°C
  - Injector: 250°C
  - Detector: 300°C
- Carrier gas: 95% argon, 5% methane
- Flow: 33 mL/minute

References for Chlorinated Insecticide Analyses

- Fish
  - Patuxent Analytical Manual (1977).
  - Pesticide Monitoring Journal, 3, p. 148 (1969).
  - JAOAC, 53, pp. 1,300-1,303 (1970).
- Fats
  - JAOAC, 57, pp. 162-172 (1974).
  - JAOAC, 59, pp. 174-187 (1976).
  - GPC Autoprep, 1001, Operator's Manual.
  - ABC Laboratories, Inc., Application Note 1 (May 8, 1978).
- Soils
  - JAOAC, 57, pp. 608-609 (1974).

The following PCB congers will be determined on a 3% OV-1 column, 6 ft x 4 mm at 190°C.

2 Chlor
3 Chlor
4 Chlor
2,2'-Dichloro
2,5-Dichloro
3,5-Dichloro
2,4,6-Dichloro
3,3'-Dichloro
3,4'-Dichloro
2,2',5-Trichloro
4,4'-Dichloro
2,4,5-Trichloro
2,3',5-Trichloro
2,4',5-Trichloro
3,4,5-Trichloro
2',3,4-Trichloro
2,2',4,6-Tetrachloro
2,2',4,4-Tetrachloro
2,4,4',4-Tetrachloro
3,2',3',5-Tetrachloro
22',33',44',55',6-Noncahl
22',33',44',55'-Octachlo
2,3',4,5',6-Pentachlo
2,2',4,4',6,6'-Hexach
2,2',4,5,5'-Pentachlo
2,3',4,4',6-Pentachlo
2,2',3,4,5'-Pentachlo
2,2',2,2',6,6'-Hexach
2,2',4,4',5',6-Hexach
2,2',3,5,5',6-Hexachl
2,2',3,4,4',6-Hexachl
2,2',4,4',5,5'-Hexach
2,2',3,4,5,5'-Hexachl
2,2',3,4,4',5-Hexachl
2,2',3,4,4',5'-Hexach
2,2',3,3',4,5-Hexachl
2,2',3,3',4,4'-Hexacn
2,2',3,4,5,5',-Hepta
2,3,3',4,4',5-Hexachl
2,2,3,3,4,4,6-Heptach

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GENERAL ORGANICS

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2. Proposed protocols for Fish/Tissue Analysis by GC-MS, U.S. EPA Region III, September 5, 1985.

TRIANGLE LABORATORIES, INC.

4915 F Prospectus Drive
Research Triangle Park, NC 27713

Telephone: (919) 544-5729

QUALITY ASSURANCE PLAN

for

Fish Sample Analysis

SECTION 3

PROJECT DESCRIPTION

This project consists of the analysis of fish tissue samples for polychlorodibenzo-p-dioxin/furan (PCDD/F) determinations.

Triangle Laboratories will assume responsibility for these analyses. This will include provision of adequate equipment and manpower to perform the work, any consultation required for experimental design, preparation of interim and final reports, and customer support concerning the dioxin analyses performed. Strict custody procedures will be followed for samples and their corresponding data packages while in the possession of Triangle Laboratories.

Analyses will be based primarily upon gas chromatography/mass spectrometry (GC/MS) using standard operating procedures based upon EPA protocols (the EPA 600 series, including 608, 624, and 625; SW-846). The Method to be used for PCDD/F determinations is based upon EPA Method 8280 (8290) and the ASME Protocol. That is, all requirements of Method 8280 are met and concentrations of the 2,3,7,8-X isomers are reported as well.

SECTION 4

PROJECT ORGANIZATION AND RESPONSIBILITY

Triangle Labs assumes responsibility for all analyses for PCDDs and PCDFs and is therefore responsible for the implementation of an adequate Quality Assurance Program. This section discusses Triangle Labs's approach to the management of the Quality Assurance aspects of the subject analytical program, and provisions and procedures which are incorporated in the overall management structure to promote implementation of QA procedures and adherence to QA guidelines.

The management structure and organization used by Triangle Labs facilitates the development and performance of Quality Assurance/Quality Control (QA/QC) functions by accurately defining the QA/QC direct lines of communication and authority between levels of project management and the QA management structure. Triangle Labs's QA program is designed in a way that facilitates interaction between QA program personnel and the project team. QA program personnel interface independently with project team members at all levels, monitoring data representativeness, accuracy, precision, and completeness. QA program personnel are free to interact directly with project team members at any time QA considerations in one of these areas need to be addressed.

The organization of the Triangle Labs project team, including quality assurance functions, is shown in Figure 4-1. Note that the quality assurance structure is independent of the organization groups which will generate environmental measurement data during this project.

The focus of the Triangle Labs QA program is the Quality Assurance Manager. He/She is responsible for the day-to-day oversight of quality assurance activities. The Quality Assurance Manager reports directly to the President of Triangle Labs, and thus is organizationally independent of the Program Managers.

4.1 PROJECT MANAGER

J. Ronald Hass, Ph.D., President of Triangle Labs will be the Project Manager. Dr. Hass co-founded Triangle Labs in 1984. Prior to 1984, Dr. Hass was a Supervisory Research Chemist with the National Institute of Environmental Sciences, where he developed and directed an active research program addressing problems in the chemical analysis of environmental and biological samples.

Dr. Hass will supervise all technical aspects of the work, including but not limited to method development, sample analysis, interpretation of test results, and preparation of reports. Should it prove necessary, he will be available to serve as an expert witness in any legal proceedings supported by data from the analytical laboratory.

4.2 QUALITY ASSURANCE MANAGER

Hani Karam, Ph.D., serves as Quality Assurance Manager (QAM) for this project. As the QAM, he has the primary responsibility for ensuring that all reported data meet the quality assurance objectives associated with the analytical project. The QAM is independent of Program Management, reporting directly to the President. His responsibilities include:

1. Review of QA Project Plans
2. Oversight of QA aspects of projects
3. Oversight of QA aspects of all contracts
4. Recommendation and implementation of QA activities within Triangle Labs to enhance product quality.

If problems arise which have the potential to adversely affect data quality, Dr. Karam will make recommendations to the Program Managers or higher management levels as necessary to ensure that appropriate corrective actions are taken.

4.3 LABORATORY MANAGER

Donald J. Harvan, MS, acts as Laboratory Manager. This involves working with the Program Managers and the Quality Assurance Manager to ensure that project goals and objectives are recognized and that efforts necessary for their attainment are implemented. At the inception of an analytical project, Mr. Harvan will meet with the Program Managers, the Project Manager and the QAM to discuss the goals of the project, the strategy of project implementation, and necessary QA/QC.

In his capacity as Laboratory Manager, Mr. Harvan also will oversee laboratory operations associated with analytical measurements. Some of these operations include:

- supervision of the loading and conditioning of adsorbent traps and conducting subsequent test to assure the absence of contaminants.

- checking records to verify that recordkeeping practices are adequate, prescribed calibration frequencies are observed, and quality control criteria are attained and maintained.

- performing system audits of facilities and equipment to ensure that the status of these reflect the effective operation of the quality assurance program.

- conducting performance audits on the measurement systems to assess the adequacy of quality control systems

- periodic reporting to the Quality Assurance Manager, Program Managers, and Project Manager regarding the quality assurance status of on-going projects.

During the course of analytical measurements, the Program Managers will share in the responsibility of these routine quality assurance activities.

PROGRAM MANAGER

Each major analytical activity has a Program Manager. The Program Manager is responsible for the implementation of the QA Project Plan within his Program. Thus the Program Manager will oversee personnel working under his direction to ensure that all necessary quality control/quality assurance procedures are performed.

DIOXIN PROGRAM MANAGER

Yves Tondeur, Ph.D. serves as the Dioxin Program Manager. He brings 9 years of experience in applying mass spectrometry to the analysis of complex matrices of biological origin as well as PCDD/PCDF in a variety of environmental sample matrices.

TRIANGLE LABORATORIES, INC.

ORGANIZATION CHART

November 1, 1987

President & CEO, J. Ronald Hass

- Business Manager, Joel R. Duvall
  - Sue Lee
  - Amy Young
  - Dawn Westmoreland
- Organics Program Manager, Dr. Robert E. Adams
  - Catherine Lloyd
  - Kathy Averette
  - Jeanne Alston
- Dioxin Program Manager, Dr. Yves Tondeur
  - M. D. Chu
  - Dr. F. T. Varcoe
  - Otelia McDaniel
  - Diane Hutter
  - Cindi Newsome
  - Edward Wallingfor
  - Debra Murray
  - Jim Jersey
  - Takashi Fuji
  - Malinda Evans
  - George Clark
  - Jeanne Alston
  - Jim Blank
  - Mike Ackerman
  - Marc Siegberg
- Special Projects Manager, Mr. Donald J. Harv
- Quality Control Officer, Dr. Hani Karam
- Sample Custodian, Mr. M. Trent

SECTION 5

QUALITY ASSURANCE OBJECTIVES

The overall objective of these measurements is to determine the concentrations of PCDDs and PCDFs and the effectiveness of recovering the analytes. Numerical QA objectives for accuracy and precision of the calibration and analytical procedures can be established based upon previous experience in the analysis of a variety of complex sample matrices using similar procedures.

5.1 ACCURACY

Accuracy is the degree of agreement of a measurement, or average of measurements, with an accepted reference value or true value. The QA objectives for accuracy for this analysis program may be expressed in terms of the following parameters:

5.1.1 Reference Materials

Only the highest quality chemicals will be used for reference materials. Where practical, standard solutions will be traceable to EPA primary references or NBS standard reference materials.

5.1.2 Instrument Performance

The instrumental performance requirements of the EPA 600 series will be followed. In general, this will result in an initial calibration of each instrument and a regular (daily, or more frequent) demonstration of continuing calibration, and tests designed to demonstrate proper functioning of the chromatographic systems. The current version of EPA Method 8280 (8290) will be used as appropriate for specifications.

5.1.3 Analytical Performance

The overall performance of the analysis will be monitored through the regular use of blank samples and surrogate standards. The samples will be spiked with internal standards and surrogate standards (not already present in the sample). The recovery will be calculated from:

$$\% \text{ Recovery} = \frac{\text{Amount of standard found in sample}}{\text{Amount of standard added to sample}} \times 100$$

This procedure will be applied to each sample analyzed. Internal standard recoveries should be within 40 and 120%. At the option of the client, a matrix spike will be included with the spike at 2 x the expected PCDD/PCDF concentrations.

5.2 ANALYTICAL PRECISION

Precision is a measure of agreement among individual measurements of a particular sample property. Precision goals for the analyses are for the % relative standard deviation (%RSD) to be < 50% (on duplicate analyses). For the initial calibration, the %RSD for the analyte response factors will be < 25% over the full range of calibration. The internal standard recoveries will have a %RSD < 40%.

5.3 COMPLETENESS

The quality assurance objective is to obtain analytical results for 100% of the samples submitted.

5.4 REPRESENTATIVENESS

The major issues of representativeness are addressed in the sampling QA/QC plan. The major concern in the laboratory is that any sub-samples are representative of the collected samples. Thus all such samples will be throughly homogenized prior to division.

5.5 COMPARABILITY

All fish samples will be reported as total analyte as well as 2,3,7,8-substituted PCDD/PCDF congeners in ppt.

SECTION 6

SAMPLING PROCEDURES

Triangle Labs are not directly involved in sample collection. For the majority of the samples, the entire quantity available is used in the analysis. For those samples in which a subsample must be taken, effort will be exerted to ensure that the sample used for analysis is representative of the sample submitted. In cases of ambiguity (for example, multiple phases in the sample) the client will be contacted for instruction.

SECTION 7

SAMPLE CUSTODY

Triangle Labs employs systems which ensure the integrity of an environmental sample from the time of receipt at the laboratory, through analysis and reporting until final disposal. These systems are necessary to allow valid conclusions to be drawn from analytical results.

The shipping containers are inspected for external damage, evidence of leaks, status and condition of Custody Seals, etc. and any observations recorded. Each sample is given an identification number composed of the Order Number to which is appended a unique serial number for each sample. The contents of the shipping containers are compared to the bill-of-lading or Chain-of-Custody information, as appropriate. Any discrepancies are recorded and the client notified of these, if present. Otherwise, it is recorded that all sample containers were present and accounted. During the course of this inspection, the physical condition of each sample container is noted and any damaged, leaking, etc. containers are recorded. The client is informed of any compromised samples.

Samples are stored as specified by the applicable EPA Method. The laboratories and all storage areas are secured. Extractions are batched by Order Number so that there is never two different projects at the same stage of analysis simultaneously. Each sample extract is numbered with the original sample identification number. In the case of multiple fractions for a given extract, an identifier is appended to the sample number for each fraction. An example Analyst Worksheet is attached.

All chromatograms, spectra, etc. are labeled with the identification number for the originating sample-fraction. These numbers are then carried through to the interim and final reports of the results so that a paper trail can be generated for each analytical result that tracks the result back to the original sample.

At the conclusion of the analyses, all raw GC/MS data are archived by magnetic tape using an indexing system that permits rapid retrieval of the data from a particular sample. Therefore, it is not only possible, but convenient, to re-examine results should a client have later questions. An archival copy of all paper hard copies of data are organized into storage containers and placed in a secured storage area. These are organized by client and Project ID number, again to facilitate interactions with clients, should questions arise later.

Any unused extract is returned for storage until disposal/destruction is authorized.

SECTION 8

CALIBRATION PROCEDURES AND FREQUENCY

8.1 ANALYTICAL INSTRUMENTS

Calibration materials are employed which insure calibration accuracy. Materials are prepared according to prescribed procedures. Only reagents of the highest commercial quality are used for preparation of working standards. Whenever possible, the working standards are analyzed against primary standards obtained from the EPA. Only high quality volumetric glassware and balances are used for standard preparation. Our balances receive regular maintenance and are calibrated against Class S weights prior to preparation of standard solutions.

8.1.1 GC/MS Tuning Conditions and Mass Calibration

The mass calibration of a quadrupole mass spectrometer is checked daily through the use of FC-43. The initial tune of the instrument is also adjusted to give specified peak ratios for this compound, consistent with the type of analysis to be performed. The high resolution mass spectrometer is tuned to give the required static resolving power, which is checked by the peak match unit. The instrument is then mass calibrated using PFK. Mass calibration is adjusted automatically to within +/- 5 parts-per-million approximately once per second during the course of all quantitative analyses.

8.1.2 GC/MS Calibration Curves

All instruments are subjected to initial calibration procedures, the details of which are specified in each Method and/or the Project QA Plan. Curves have a minimum of three and typically five points and include a response factor for each analyte.

Continuing calibration is demonstrated on a daily basis. The maximum time between demonstrations of continuing calibrations is 12 hours. The exact specifications for continuing calibration are as specified in the relevant EPA Method and typically require response factors to agree within 30 % of the initial calibration value. In the event that continuing calibration cannot be demonstrated after corrective action (such as re-tuning instruments, maintenance of chromatographic columns, etc.) a new initial calibration curve will be generated.

All standards and samples will have internal standards added at a level approximating the mid point of the calibration curve. The response factor will be calculated from:

$$RF = A_s C_{I\,s} / A_{I\,s} C_s$$

where

$A_{I\,s}$ = area of the internal standard

A_s = area of the target compound

$C_{I\,s}$ = amount of internal standard

C_s = amount of the target compound

The response factor is used to demonstrate linearity in initial calibrations, continuing calibration and the concentrations of analytes.

SECTION 9

ANALYTICAL PROCEDURES

The following analytical procedures will be applied to samples obtained from the NYS Department of Environmental Conservation:

Matrix	Parameter	Analytical Method
Fish	PCDD/PCDF	Method 8280 (8290) or Nat. Dioxin Study

Triangle Labs have considerable experience in the application of these methods to the determination of constituents of hazardous waste and effluent samples. Therefore, analysis of spiked sample, duplicates, and surrogates will serve as indicators of method performance.

SECTION 10

DATA REDUCTION, VALIDATION, AND REPORTING

10.1 DATA REDUCTION

At each stage of the analysis, data will be reviewed for completeness and, based upon previous experience, reasonableness. Any missing data will be provided and unusual results verified. As much as possible, data reduction will be via computer programs. Where manual data manipulation procedures are required, data reduction will make use of standard forms.

At a minimum, data will be reviewed by the instrument operator, the chemist preparing the report and the QA Manager. All data will be reviewed at the earliest opportunity to facilitate corrective action.

Data reduction will follow the quidelines of the relevant EPA Reference Method. These include Methods 8280/ASME (PCDD/PCDF).

10.2 DATA VALIDATION

These tests involve the performance of complex chemical analyses by a number of chemists. For this reason data validation and coordination are very important. Internal tracking has been addressed in the SAMPLE CUSTODY section. At the conclusion of the analyses, the data are checked against the original shipping information and analytical request to be sure that the required analyses have been performed on all samples.

The validity of the data will be tested through the analysis of blank samples, duplicate samples, and matrix spikes. The blank sample results will demonstrate the absence of laboratory contamination of the samples. Duplicate analyses give a measure of analytical precision. Analysis of matrix spike samples permits a measure of accuracy.

10.3 DATA REPORTING

The data will be reported as components identified and the quantities present. The final report will include example calculations and descriptions of the equipment and procedures. Complete data packages of all raw sample and calibration data will be prepared and archived. These will be furnished upon request.

SECTION 11

INTERNAL QUALITY CONTROL CHECKS

All measurements methods employed by Triangle Labs have associated quality control checks. The extent of the activities depends upon the control status of a particular method at the time of its use. In this respect, a method is either in control or out of control.

Routine quality control activities are applied to methods in control; these routine activities generally include quality control measures recommended within published methods. For EPA methods, these are the minimum activities followed.

For methods not designated as Reference Methods, minimum quality control efforts include the following, as applicable:

1. Initial tuning, mass and response calibration of the mass spectrometer.

2. Measurement of analytical blanks, processed in the same manner as the samples.

3. Analysis of quality control samples, including matrix spikes and duplicate determinations.

For methods out of control (which includes methods under development, since these are, by definition, of undetermined quality). quality control activities in addition to those described as routine are applied. While the course for establishing (or re-establishing) control of the analysis is generally approached on a case-by-case basis, Triangle Labs has employed the following quality control activities when criteria associated with routine activities were not met, and when methods are being evaluated:

1. Replicate determinations to establish the consistency of analytical behavior.

2. Split samples to assess either the quality of the sample preparation phase or to acquire an independent check of the measurement result.

3. Spiked samples to assess the influence of matrix effects.

4. Assessment of reagent quality.

5. Evaluation of reagent quality.

Data from initial and continuing calibrations are incorporated into the final report for each project, as are results from other quality control activities including the results from the analysis of blanks and spiked samples.

SECTION 12

PERFORMANCE AND SYSTEM AUDITS

Performance and system audits are routine elements of all Triangle Labs QA/QC programs. System audits are always conducted by Program Managers before the beginning of any project. These include verification that the instruments are in proper operating conditions, that adequate supplies and spare parts are on-hand for the samples to be analyzed and that sufficient qualified personnel are available to perform the work in the specified schedule.

Laboratory performance audits are conducted prior to the implementation of any new protocol and periodically during the performance of analyses. When available, audit samples from outside sources, such as the EPA, are used to measure system performance. Otherwise, in-house audit samples are prepared and analyzed.

SECTION 13

PREVENTATIVE MAINTENANCE

Well maintained equipment is an essential ingredient in assuring the quality, completeness, and timeliness of analytical data. Triangle Labs minimizes the risk of data incompleteness through the performance of regular maintenance on all equipment, redundancy in equipment, provision of an ample stock of spare parts, and an inventory of specialized test equipment to support rapid repair, in the event unscheduled maintenance is required. Perhaps the most important feature of our equipment maintenance/repair plan is the availability on our staff of personnel capable of providing instrument service without reliance on offsite service technicians.

SECTION 14

SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY, AND COMPLETENESS

The experimental design allows the data to be grouped into sets that can be analyzed to evaluate data precision and accuracy. The central tendency and dispersion of data is assessed by reviewing the precision and accuracy. These assessments are performed on all sets of 10 or more data points.

The data sets are examined for systematic error (i.e., accuracy) by calculation of the arithmetic mean of the surrogate recoveries. Any bias in the measurements will be reflected in deviation of the mean from 100%. Where possible, accuracy is also assessed through the analysis of spiked samples. Once again, bias will be revealed by recoveries significantly different from 100%.

Precision is assessed through the analysis of duplicate samples. The % relative standard deviation is calculated for replicate analyses. A second assessment of precision is the %RSD of the surrogate recoveries.

SECTION 15

CORRECTIVE ACTION

Any sample not analyzed by the specified method will be considered for re-analysis. Equipment found to be out of calibration or operating improperly will be repaired or replaced before analyses proceed. Any data generated under these conditions will be carefully inspected to determine what repeat measurements are needed to assure data quality.

For each analytical method employed and each type of sample matrix generating 10 or more data points, mean recovery and standard deviations will be generated for the analysis of surrogates and replicate sets of sample analyses. When either the relative standard deviation or the surrogate recoveries do not meet the performance goals of the project, corrective action will be taken prior to the next lot of samples.

If weaknesses or problems become apparent during system or performance audits, corrective action must be taken immediately. Examples of corrective actions include a new initial calibration, column maintenance for GC analyses, replacement of reagents leading to unacceptable blanks, etc.

Should performance audits indicate that any methods are out of control, the Quality Assurance Manager and the Program Manager will review the audit data and determine the appropriate action to be taken. The Project Manager and the relevant chemists have daily contact with the procedures, and offer detailed familiarity with them. The QAM offers a broader background and more detached viewpoint. This combination helps in formulating quick and reliable solutions to various problems. The Project Manager and chemists will be responsible for initiating the action and the QAM will be responsible for determining if this action has resolved the problem.

SECTION 16

QUALITY ASSURANCE REPORTS TO MANAGEMENT

Quality assurance reporting and documentation are important elements of final project reports. Reports provided to agency and industrial clients contain sections which describe the QA/QC activities and criteria relevant to the measurements reported, provide results from the application of these activities, and address the impact of these results on the measurements reported.

The quality assurance procedures used in these analytical programs generate sufficient documentation to indicate data quality. Quality assurance results will be included in the final report. At a minimum, the following will be addressed in the report:

1. Precision
2. Recovery (accuracy)
3. Blanks
4. Calibration
5. Spike composition and recovery
6. GC/MS calibration and tuning
7. Internal standard response and recovery

All evidence of the execution of the Quality Assurance Plan is reviewed by management. In addition, all aspects of the project are discussed regularly by the appropriate program staff. No written record of these discussions are maintained, since all interested parties are verbally appraised at the time.

The Quality Assurance Manager makes independent checks of data quality and reports these to management. Finally, management, from time-to-time, surveys a sampling of clients to be sure that the service provided by Triangle Labs is of high quality and provided in a timely manner.

Standard Operating Procedure 201: Sample and Extract Storage.

Scope and Application: It is the purpose of this SOP to establish TLI policy regarding sample storage. The objectives of this policy are to insure that the identity and integrity of each sample is maintained and documented. Sample storage divides into three aspects. These are storage prior to any workup, storage of any extracts prior to their analysis, and storage of samples/extracts during analysis. It is necessary that all storage areas insure sample custody and integrity be maintained.

1. Initial Storage of Samples. The SOP for sample receipt and login is covered in SOP 100. This SOP describes the procedures to be followed in the storage of samples.

A. Assigned Storage Areas.

In order to minimize the possibility of sample confusion, there will be a defined storage area assigned to each customer. It will be possible to gain access to the samples of one customer without having access to any other customers samples. For customers with high sample volumes, it may be desireable to sub-divide their storage area to give independent storage for particular users or collections of users from a given customer. Within these guidelines, the size of the storage area will be determined by the volume of samples received from that customer. The samples received from occasional customers will be stored in an area designated "Miscellanous".

B. Types of Storage

Samples will be stored either in a freezer at -20 degrees C, a refrigerator at 4 degrees C or room temperature, as specified by the client. In the absence of a specification by the client, the Sample Custodian will use his best judgement for storage. Care will be taken to be sure that cross contamination does not occur during sample storage. Typical precautions will include the isolatation of samples thought to contain volatile components, addition of an extra layer to the sample container, etc. Under no circumstances will samples and standards share storage.

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C. Access to Samples.

Samples will be accessed only by TLI employees with a need to use the samples. All sample access will be under the supervision of the Sample Custodian and will be documented as described in SOP number 200-85. As soon as practical, the sample will be returned to the secured sample storage area.

2. Sample Extracts.

Great care will be used in the storage of sample extracts. Many extracts are significantly less stable than the samples from which they were extracted. At all times other than during analysis, extracts are to be stored cool or cold and protected from light. Samples known to be easily oxidized will be flushed with dry nitrogen prior to resealing.

3. Storage After Analysis.

Samples will normally be disposed of as hazardous waste after analysis. This will follow a period of storage which is either until the client has accepted all results and authorized payment of the invoice or until any post-analysis storage requirements of the contract have been fulfilled. In either event, the sample will be stored so as to maximize the probability of it maintaining its integrity. In the absence of other instructions, it will be stored in the same manner as prior to analysis.

Standard Operating Procedure No. 101-85: Internal Chain-of-Custody Documentation.

Scope and Application: It is important that TLI be able to demonstrate to any customer that the sample analyzed was the same as the sample submitted. This requires that we document that samples were either in a secured storage area or in the custody of an analyst at all times. The Sample Custodian has the responsibility of implimenting this policy.

1. Building Security.

A. Outside Entrances/Reception Area.

The outside entrance of the laboratory is to be kept locked at all times unless someone is on duty in the front offices. Visitors are announced and are normally admitted only to the reception area and/or front offices. The door connecting the reception area is equipped with an electronic lock which is to be used at all times. Only TLI employees are to be given the combination to the lock. This combination is to be changed periodically by the Sample Custodian.

B. Laboratory Access.

Access to the analytical laboratories is normally restricted to TLI employees. Exceptions will be made in the case of tours of the laboratories by prospective customers (these will be approved and coordinated by the President) and customers who have need to be present when their samples are analyzed. All visitors are to sign the Visitor Log and to be accompanied by a TLI employee at all times.

C. Sample Storage.

Samples are to be stored in an appropriate, secured area. Samples requiring refrigeration will be stored in a locked refrigerator, those requiring freezing, in a locked freezer, and all others in a locked cabinet. Regular customers will have an assigned storage area in each type of storage device, as needed, where their samples are normally stored. Access to a particular sample will require opening two key locked doors and one electronically locked door.

D. Internal Chain-of-Custody Documentation.

A sample will be defined to be in a persons custody when:

1. It is in his actual possession, or

2. It is in his view, after being in his possession, or

3. It was in his possession and then locked or sealed it up to prevent tampering, or

4. It is in a secure area.

Anytime a sample is removed from a secured storage area, the person handling the sample must document the transfer of sample custody. The Sample Custodian must authorize all transfers of sample custody. The documentation must include the time and date of sample removal, the reason the sample was removed, and the time and date the sample was returned. The person removing the samples will be responsible for following proper chain-of-custody procedures while the sample is in his possession.

DRAFT

Standard Operating Procedure No. 200. Sample Receipt and Login

Scope and Application: It is the purpose of this SOP to describe the procedures to be followed when receiving samples for analysis. The primary objective of this SOP is to provide the necessary procedures and documentation for chain of custody requirements for samples that potentially involved in litigation. These procedures will be followed for the receipt and login of all samples analyzed by TLI.

1) Receive shipment and check that number of packages corresponds with shipper's airbill. If samples are for CLP and have been shipped to North Campus, transfer to South Campus before opening.

2) Inspect coolers for damage, leakage, and chain-of custody seals.

3) Get:
   a) TLI order form
   b) TLI CLP (or dioxin) worksheet
   c) TLI chain of custody form
   d) Sample receipt log

4) Open cooler and retrieve Sample Traffic Reports, chain of custody documents, and other accompanying paperwork. For CLP samples, organize the Sample Traffic Reports in increasing order to determine the SDG number.

5) Inspect sample containers for:
   a) Breakage
   b) Leakage (quantity shipped or air bubbles in volatiles)
   c) Temperature samples were at upon receipt
   d) Sample IDs match the Sample Traffic Reports

6) In sample receipt log, record:
   a) Date
   b) TLI project number
   c) Airbill number
   d) EPA Case number and SDG number (or SAS number)
   e) Summary of the inspections from step 2
   f) TLI Sample ID (equal to Log book#-page #-sample #)
   g) Sample ID from Sample Traffic Report
   h) Sample Tag number
   i) Sample storage location
   j) Any discrepancies or problems from step 5
   k) Signature and date.

7) While recording the information of step 6f and 6g, record on the sample containers the TLI Sample ID and project #. Also record the EPA Case # (or SAS number) if it isn't already there.

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8) If problems are encountered with the above sequence, contact SMO for clarification.

9) If no problems, sign and date the Sample Traffic Reports and chain of custody documents. Write TLI project number on SMO forms, and be sure Case # and SDG # are on the forms. It may be necessary to contact SMO to determine if this is the last SDG for a given Case number.

10) Fill out the TLI internal chain of custody form.

11) Store samples as per SOP 201.

12) Make copies of all documents.

13) Send designated copy of Sample Traffic Reports and SDG summary form to SMO. The SDG summary form is sent only after ascertaining from SMO that the last sample of the SDG has been received. These forms are all sent by certified mail.

14) Give file folder containing copies of the paperwork to the appropriate program manager.

15) File copies of all documents (chain of custody, airbill, Sample Traffic Reports, log book, and other attached paperwork) in chain of custody drawer in data storage office on North Campus.

16) After certified mail has been sent, attach our copy to chain of custody information in file drawer.

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Standard Operating Procedure No. 100. Order login.

Scope and application: This SOP is to be used for recording all orders placed with TLI. For purposes of this SOP, an order is always accompanied by a purchase order number, statement of work, or other offical documentation from the customer authorizing work to be performed.

1. All information relating to the billing is recorded on a sequentially numbered "Customer Order Form" (example attached). The information concerning the work to be performed is recorded on a "Laboratory Workorder Form" (example attached). The number on the Customer Order Form becomes the TLI project number associated with that order. All records and work are to include this number for reference.

2. It is the responsibility of the Sample Custodian to inventory all in-coming sample shipments, record on the above forms the number of samples received, their storage location, and transmit these forms, along with any information that arrived with the samples to Marketing.

3. The purchase order number and price quoted for the work is added to the Customer Order Form which is then sent to the Business Manager, who records the pertinent information in the "Orders Register".

4. The Laboratory Workorder Form is completed, including the work to be performed and the target schedule. The completed form is placed in the Laboratory In-Box. This information is also entered in the Laboratory Status Log (example attached).

5. At the conclusion of the project, the Laboratory Workorder Form is returned to Billing, where the actual work performed is entered on the Customer Order Form, along with completion date and shipping information. This completed form is transmitted to the Business Manager for invoice generation.

TRIANGLE LABORATORIES, INC.

MAJOR EQUIPMENT and FACILITIES

April 1, 1986

Laboratory Facilities:

Triangle Labs are housed in 5000 square feet of space constructed in 1984. This space includes offices, wet laboratory space and instrument rooms. The laboratories are well equipped with glassware and ancillary equipment for sample preparation.

High Resolution Mass Spectrometry:

Triangle Labs owns two high resolution mass spectrometers, a 70S and 70.70H (VG Analytical). Instrument control, data acquisition and processing is by means of three VG-Analytical 11-250 data systems. This combination permits mass measurements under capillary column GC/MS conditions with a precision of 10 parts-per-million (ppm) or selected ion recording at high resolution with drift in mass limited to $\pm$ 5 ppm. Sub-picogram analytical sensitivities are routinely available by high resolution selected ion recording.

Low resolution Mass Spectrometry:

Our low resolution mass spectrometry laboratory includes two instruments. Both are fully automated for data acquisition and the preparation of reports meeting the deliverables requirements of the EPA Contract Laboratory Program. One instrument, configured for Volatile Organics Analysis, is a VG-Masslab 12-250 mass spectrometer. This instrument, with a mass range of 1200, electron impact and chemical ionization, is capable of either positive or negative ion operation. The other instrument, a VG-Masslab 20-250 mass spectrometer, is configured for Semi-Volatile Organics Analysis (i.e., BNAs), has a mass range of 2000 and is equipped with a fast atom bombardment source and a Thermospray high pressure liquid chromatography/mass spectrometry interface in addition to the standard electron impact/chemical ionization source, and positive or negative ions.

Gas Chromatography:

Currently our gas chromatography lab is equipped with two Hewlett-Packard 5890A Chromatographs with electron capture and flame ionization detectors and robotic autosamplers. Additional detectors can be fitted, if required. The chromatographs are interfaced to a Maxima Chromatography Data System.

ATTACHMENT 3

CONTAMINANT MONITORING OF WILDLIFE

Project Name: Contaminant Monitoring of wildlife from lands of the Mohawk Nation at Akwesasne

Proposed Date of Initiation: Late summer 1987 (wildlife are primarily hunted and trapped during the fall and winter months)

Project Description:

A. Objectives and Scope:

Existing data on contaminants in wildlife eaten by Mohawks is limited. This investigation will be done in coordination and with cooperation of the Mohawks. The objectives of this project are as follows:

To document the contamination levels in wildlife species, eaten by Mohawks, in a manner adequate to assess public health risks and develop health advisories on the consumption of wildlife.

To evaluate, where possible, spatial relationship of the toxics in tissue of wildlife with respect to source(s) such as industrial discharges, leachates, contaminated land and/or sediments.

B. Monitoring network design and rationale:

The project is designed as a one-time effort with sampling to begin in late summer of 1987 and end during the winter of 1988. However, it may develop that additional or follow-up monitoring events are advisable. Sampling will be for wildlife consumed by Mohawks and taken from various locations within Akwesasne.

C. Date to be collected and rationale:

The following wildlife species are taken at Akwesasne and consumed by Mohawks at Akwesasne:

Mallard Duck (F)
Black Duck (F)
Wood Duck (F)
Canada Goose (F)
Blue-winged teal
Green-winged teal
Gadwall Duck
American Wigeon Duck
Common Goldeneye Duck
Bufflehead Duck
Great Scaup Duck
Lesser Scaup Duck
Mergansers
Ruffed Grouse
Woodcock
Muskrat (F)
Beaver (F)
Cottontail Rabbit (F)
White-tailed deer (F)

*(F) Favored foods to Mohawks

D. Waterfowl

Samples of waterfowl will be taken in late August to early September, late September to mid October, late October to mid-November, and late November through December. The waterfowl will be collected from the St. Lawrence River (in the vicinity of Racquette Point), and the Snye region of the river. Twelve birds at each location during the four time periods (a total of 96 waterfowl). This sampling procedure is designed to collect toxicant data on waterfowl during the times that Mohawks normally hunt and eat waterfowl. The waterfowl species studied will, when possible, be the most favored and commonly eaten waterfowl.

The waterfowl will be placed in PCB-free plastic bags upon collection, and a standard Wildlife Pathology Unit waterproof tag will be filled out and attached to the bag. The carcasses will be accompanied by continuity of evidence forms. The waterfowl will be frozen in a freezer (with lock) until preparation. At dissection the bird will be assigned a Wildlife Pathology log number. A standard Wildlife Pathology Necropsy form will be utilized to record data on the age, sex, weight, general body condition, and any gross pathology observed. During necropsy, portions of subcutaneous fat, breast muscle, and the entire liver, and for 20 waterfowl the total plucked skin will be placed in chemically clean glass jars with an aluminum foil-lined lid. The samples will be stored at -15°C until prepared for chemical analyses.

The waterfowl tissues will be analyzed by USEPA or USFDA approved techniques. The organochlorine screen will include polychlorinated biphenyls (PCB's), octachlorostyrene, DDE, DDD, DDT, dieldrin, alph-BHC, beta-BHC, gamma-BHC, HCB, endrin, heptachlor, heptachlor epoxide, aldrin, mirex, methoychlor, toxaphene, cischlordane, trans-nonachlor, and oxychlordane. Results will be reported on a wet weight and on a lipid basis. The following analysis will be performed:

Waterfowl Analysis Protocols	Samples
organochlorine screen Hg and Pb	96 breast muscle samples
organochlorine screen	20 waterfowl skin samples and approximately 6 subcutaneous fats
Hg and Pb	96 liver samples
TCDD and TCDF	5 composites of subcutaneous fat (equal weight of fat from each bird) from five waterfowl of the same species.

E. Muskrats

A total of fifty muskrats will be collected in lots of 10 from the following areas:

1. From the unnamed tributary and small associated St. Lawrence River bay immediately east of the G.M. landfill that borders on Akwesasne.

2. Along the St. Lawrence River shoreline of eastern Raquette Point.

3. Upstream in the Raquette River within Akwesasne.

4. Upstream in the St. Regis River within Akwesasne.

5. From Akwesasne in the Snye Marsh area of the St. Lawrence River.

The muskrat carcass will be tagged, accompanied in the same manner as waterfowl carcasses. Preparation and storage methods will also be similar. Skeletal muscle sample will be taken from the hind legs.

Muskrat Analysis Protocol	Samples
organochlorine screen	50 skeletal muscle
organochlorine screen	50 liver samples
Hg, Cd, Pb	(If elevated metal levels are found the corresponding skeletal muscle sample will be run for the appropriate metal(s).
TCDD-TCDF	4 subcutaneous fat samples. 2 from muskrats taken adjacent to the GM landfill and 2 from muskrats taken in the Snye.

F. Beaver, Cottontail Rabbit, and White-tailed deer

A small sample of these species will be taken from Akwesasne. The deer population is low on Mohawk lands and the beaver are unevenly distributed.

Samples of subcutaneous fat, liver and skeletal muscle will be collected from hunter killed deer taken from Akwesasne. Basic

information on the sex, age, weight, date of death, geographic location where killed, and general body condition will be recorded.

A small sample of beaver will be taken where available from the St. Lawrence River below the G.M. plant and from the Snye.

Five cottontail rabbits will be collected from Mohawk lands adjacent to the G.M. hazardous waste site. A distant inland site in Akwesasne will be chosen for another sample of five cottontails.

Analyses Protocol

organochlorine screen	20 beaver
Hg and Pb	about 10 deer; and 10 cottontail rabbit skeletal muscle samples.
organochlorine screen	20 beaver, about 10 deer, and 10 cottontail rabbit subcutaneous fat samples.
organochlorine screen Hg and Pb	20 beaver, about 10 deer, and 10 cottontail rabbit livers
TCDD-TCDF	Pooled fat samples (2) from cottontail rabbits taken adjacent to the G.M. hazardous waste site and a distant site in Akwesasne.

G. Estimated Budget

1 Collection, Initial preparation, and sample transportation

Personnel	$12,000
Travel	1,200
Equipment (freezer)	800
Supplies (scalpels, surgical gloves, glassware plastic bags, capture supplies)	2,000
	$16,000

Laboratory Analysis	
organochlorine screens metals and TCDD and TCDF	$100,000
Project administration and reporting	$14,000
Total	$130,000

NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION
GAME RESEARCH SECTION

AUTOPSY REPORT

NUMBER	DATE	SEX	SPECIES
ANIMAL FROM			SUBMITTED BY
DATE DIED	CONDITION OF CARCASS		GROSS WEIGHT:

HISTORY

GROSS P. M. FINDINGS

HISTOPATHOLOGY: Tissues submitted – eye, brain, tongue, salivary gland, lungs, heart, esophogus, stomach (P & G), S.I., L.I., cecum, liver, spleen, kidney, U. bladder, adrenal, pancreas, ovaries, uterus, testis, lymph nodes, ( ) other.

PATH NO.

FINDINGS:

ECTOPARASITES (Species and Total Number)

ENDOPARASITES (Species and Total Number)

BACTERIOLOGY Organs	Media	Results

GROSS PHOTOGRAPHS	PHOTOMICROGRAPHS

COMMENTS:

82-15-39 (5/79) Formerly DFG-343

GMM 006 1034

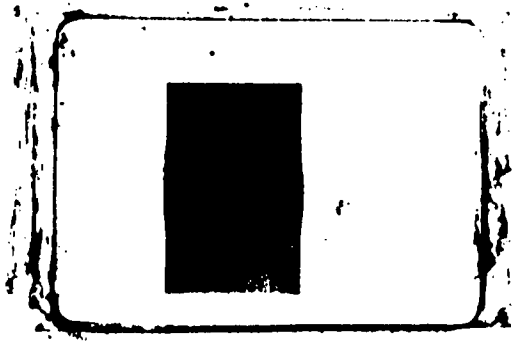
Submitted by: _____

_____

_____

Date: _____

To: New York State
Department of Environmental Conservation
Wildlife Resources Center
Wildlife Pathology Unit
Delmar, New York 12054



GMM 006 1035

[redacted]

RECORD OF SPECIMEN
for PATHOLOGICAL, TOXICOLOGICAL or PHYSIOLOGICAL EXAMINATION

Submit to Wildlife Resources Center

_____

Species _____ **Age** _____ **Sex** _____

Date of death _____ **Date submitted** _____

County _____ **Township** _____

Exact site _____

Reason(s) for submission _____

_____

Information pertinent to diagnosis (unusual behavior, suspected cause of death, etc.)

_____

_____

Animal found by:

Name _____

Address _____

Telephone No. _____

In EMERGENCY, call Area Code (518) 457-1783 (Bus.) or (518) 872-1473 (Home)

82-14-22(3/79)
Formerly FW-383

[redacted]

[redacted]

[redacted]

GMM 006 1036

ATTACHMENT 4

PARTICIPATION IN A HUMAN BREAST MILK STUDY

GENERAL MOTORS - CENTRAL FOUNDRY DIVISION
HEALTH RISK ASSESSMENT

QA/QC PROTOCOL

TASK 3 - HUMAN BREAST MILK
CONTAMINANT MONITORING STUDY

TABLE OF CONTENTS

I. INTRODUCTION

The Mohawk Nation at Akwesasne is a native American community located along the St. Lawrence River near Massena. It occupies approximately 14,000 acres in New York, Ontario, and Quebec and is inhabited by 7,000 persons. The population has become increasingly alarmed about the effects of pollution on their environment and health, given the industrialization of the area after the opening of the St. Lawrence Seaway in 1954. One issue is fluoride pollution from two aluminum plants along the River. A more recent problem is the contamination of sediment, surface soil and water from the disposal of PCBs and other chemicals at the General Motors - Central Foundry Division Superfund waste site. Superimposed upon these local concerns is the pollution of the St. Lawrence River Basin in general with organochlorines, methyl mercury, and other contaminants.

There is evidence that these chemicals have affected the local flora and fauna. High levels of fluoride have been found in vegetation, for example, and dental fluorosis has been observed in cattle from Cornwall Island (1). Elevated fat concentrations of PCBs have been detected in moles, frogs, turtles and other animals taken near the dumpsites and in fish collected from the River (2). Studies of human health effects, however, have been negative. An investigation of dental defects among Mohawk children, for instance, determined that the probable cause was the use of the antibiotic tetracycline and not fluoride exposure (3). Another study found that the concentrations of inorganic fluorides in urine and blood were no greater among Akwesasne residents than in persons who drink fluoridated water; physical examinations, radiologic and pulmonary function studies, and laboratory tests did not reveal any health conditions attributable to fluoride (4). Similar results were obtained in analyses of methyl mercury, PCBs, and mirex (5). The authors nevertheless recommended that the consumption of contaminated fish be limited, especially among children, women of childbearing age, and pregnant and nursing women. This recommendation was based in the potential of PCBs, methyl mercury, and mirex to cause adverse reproductive effects in humans or animals (6-8).

II. PROJECT DESCRIPTION

A. Objectives

The purpose of this study is to investigate the concentrations of various polychlorinated hydrocarbons in the breast milk of Mohawk women. Although breast milk contamination is a measurement of absorption and not a health effect, it is relevant to the question of adverse reproductive outcomes, since they are dependent upon dose and because breast milk is the major source of exposure to environmental contaminants among breast-fed infants (9).

The objectives are two-fold: 1) to determine if the concentrations of 75 PCB congeners, total PCB, p,p-DDE, hexachlorobenzene and mirex in milk samples are greater among Akwesasne residents compared to a semi-rural control population of women from other areas of New York, and 2) to identify dietary, residential, occupational, and other factors which correlate with contaminant levels. Pooled samples will also be analyzed for concentrations of PCDD and PCDF congeners. In addition, fat content and total milk solids will be analyzed to assess the general nutritional value of the milk.

The project is one component of a three-part Health Risk Assessment to be conducted in conjunction with the overall Remedial Investigation/Feasibility study for the General Motors - Central Foundry Division site. Together with other studies of fish and wildlife contamination, this research will assess major potential pathways of human exposure to PCBs in the food chain at Akwesasne.

B. Ascertaining and Recruiting Participants

All lactating Mohawk women who live at Akwesasne and who give birth between March 1, 1988 and February 28, 1990 will be eligible to participate. It is difficult to determine the exact number of live births each year in this population, since as citizens of a sovereign nation, Akwesasne residents are not required to register vital events with U.S. or Canadian authorities. Discussions with clergy at the local Roman Catholic church, however, indicate an increase in the number of baptisms among newborns from 74 in 1979 to 105 in 1986. The annual mean was 94. To date, the 1987 total is 100. The clergy believe that this trend is the result of an increase in the number of live births and not of an increase in the rate of baptisms, a proportion that has remained constant over time at two-thirds. This interpretation has been confirmed in discussions with local health care providers. We therefore estimate the number of live births at 100 per year. This value is conservative; the actual number is likely to be greater, perhaps as large as 150 per year. If one-half of the mothers breast-feed, the number of eligible participants will be at least 50 per year.

Potential participants will be identified through contact with the St. Regis Mohawk Health Services. WIC program, private physicians, and outreach programs. This cooperation will be obtained through the efforts of Ms. Katsi Cook, Project Director. Ms. Cook is a Mohawk midwife and Akwesasne native who maintains strong ties to the community and who will serve as a liaison between the mothers, tribal leaders and project personnel. Her employment is necessary to ensure the continuation of these relationships in the U.S. and their expansion to Canada, thereby permitting a total ascertainment of potential study participants.

II. PROJECT DESCRIPTION

B. Ascertaining and Recruiting Participants (cont.d)

The mothers will be personally contacted at home two to four weeks post-partum by project personnel regarding participation in the study. After funding has been secured, we also plan to employ a Mohawk woman full-time who has medical training (e.g., a nurse or midwife) to assist Ms. Cook in data collection. Project personnel will stress a positive theme when approaching potential participants by reassuring them that the testing is being done to help them feel confident in their decision to breast-feed a child.

No financial compensation will be offered, but we expect that at least three-quarters of all eligible women will participate. This estimate is based primarily on the fact that the Mohawk people are uniquely concerned about environmental contamination at Akwesasne and are highly motivated to address the problem. Their culture and tradition emphasize the interdependence of man and his environment. Many Mohawks also depend on local fish and game for food, adding practical and economic significance to the issue. Another incentive is the fact that participation in the study will extend the medical care routinely provided to mothers and their infants at Akwesasne to include the conduct and interpretation of additional laboratory tests and procedures of potential significance. Our anticipated response rate also reflects Ms. Cook's unique position within the community and the special relationship that she enjoys with mothers and tribal leaders. Confirming our prediction that the study will be favorably received is the fact that, although the number of women in our pilot work was small, every mother who was approached by Ms. Cook was willing to participate.

Each participant will be given literature summarizing the study (Appendix A). They will be informed about the procedures to be employed, the inherent risks, and why these risks are reasonable in relation to the anticipated benefits. Those who agree to participate will be asked to sign an informed consent form (Appendix B).

C. Procedure

Project staff will interview the participants at their homes two to four weeks post-partum, using a standard instrument (Appendix C). The interview will focus upon diet, cigarette smoking, alcohol consumption, occupational, residential, and reproductive histories, child-feeding practices, use of medications, and contact with the waste site. The medical records of selected participants may also be obtained with a signed release form (Appendix D) if circumstances indicate that additional information of a clinical nature should be acquired, e.g., the development of a health problem which interferes with nursing.

II. PROJECT DESCRIPTION

C. Procedure (cont.d)

The dietary assessment will consist of the participant's report of her consumption of various foodstuffs, emphasizing local species of fish and game. The specific method will combine a food frequency with a limited dietary history to estimate retrospectively usual intake. The food frequency will consist of a checklist of foods for which the respondent will indicate her consumption rate per week, month, or year. The dietary history will elicit information such as duration of consumption, whether diet has changed over time and what cooking or food preparation practices are used (e.g., frying vs. broiling, trimming fat off meet, eating skin of poultry). A food frequency provides a succinct picture of an individual's eating pattern on a food-specific basis, while a dietary history yields a more representative record of long-term intake (10-11). Both techniques have been useful in epidemiological research (12-13).

The data collected at the first interview will focus upon food intake during two past periods: 1) the year before the index pregnancy and 2) the nine months of pregnancy. We chose to center recall around the index pregnancy instead of simply asking each woman to report her average frequency of consumption over the last year beacause a woman may have significantly altered her diet in compliance with the health advisories to avoid local fish and game while pregnant. Questions pertaining to pre-pregnancy diet are limited to 1 year to enhance memory, but mothers will also be asked if they ate local fish and wildlife more frequently or in greater quantities at some time earlier than one year before the index pregnancy. If a woman responds affirmatively, then consumption rates during that period will also be recorded. (Earlier consumption is important because polychlorinated hydrocarbons bioaccumulate with time.)

D. Control Group

Comparative data will be collected from nursing women who live in other areas of New York State and are not exposed to toxic waste. For convenience, locations with a 100 mile radius of Albany will be selected. Under consideration are Montgomery, Schoharie, and Warren Counties. Volunteers will be recruited through contacts with the NYSDOH Albany Regional Office and local WIC clinics. Because Akwesasne is primarily rural, women from other rural areas will be selected as controls. Instead of matching the controls to Mohawk women on factors such as age and parity, however, these and other confounders will be taken into account in the analysis.

Ms. Judith Quinn of the Bureau of Environmental and Occupational Epidemiology, NYSDOH will interview the control women two to four weeks post-partum using a version of the same instrument that will be employed for the Mohawk mothers. A signed informed consent form will also be obtained.

II. PROJECT DESCRIPTION

E. Breast Milk Sampling

After the interview is completed, project personnel will instruct the Mohawk and control mothers in the use of the Marshall 900 CP Kaneson breast pump/infant nurser. They will be asked to provide at least 50 ml of breast milk, over a period of several days if necessary. The samples will be obtained after the second morning nursing (generally 9:00 a.m. to 11:00 a.m.). That period is usually when the fat content of human breast milk is the highest, and since polychlorinated hydrocarbons are lipoplilic, sampling at that time is expected to yield maximal concentrations.

The mothers will store their breast milk in a single glass container with a teflon top to prevent interference and refrigerate it until collection by study personnel. Project staff will place the samples in an insulated cooler with ice until transfer within six hours to the freezer at the Community Center in Hogansburg. There the samples will be frozen at -10° to -20°F. Shipment will occur to the Wadsworth Center for Laboratories and Research, NYSDOH via Federal Express on the last Monday or Tuesday of each month. Holiday shipping will be avoided. The samples will be packed with dry ice during shipment to prevent thawing. A "Priority 1" designation will be used to ensure that the samples arrive in good condition. Control samples will be brought to the laboratory directly after collection.

Each bottle will be labeled with the name and social security number of the participant. Responsible persons will sign the chain-of-custody form (Appendix E) at each stage. The completed interview forms will be sent under separate cover to the Bureau of Environmental and Occupational Epidemiology, NYSDOH at the end of each month.

F. Chemical Analysis

Drs. Brian Bush and Patrick O'Keefe, Wadsworth Center for Labortories and Research, NYSDOH will perform the PCB and PCDD/PCDF analyses respectively.

Preparation of Milk Sample

For the PCB analysis, 40 ml of breast milk will be used. The remaining 10 ml will be pooled with similar quantities from other women to provide the 200 ml needed for PCDD/PCDF analysis. Three composites will be formed for PCDD/PCDF determination: 1) Mohawk women who eat fish, 2) Mohawk women who do not eat fish, and 3) control mothers.

Each sample will be extracted with 50 ml of ethanol and 25 ml of hexane/benzene. After phase separation, the upper layer is decanted and dried over sodium sulfate. Two further extractions will be performed and all three extracts combined. The extracts will be evaporated to 10 ml, transferred to a graduated flask and hexane added to obtain a volume of 25 ml. Gravimetric determination will be performed by evaporating 5 ml in a tarred aluminum pan, and total milk solids will be assessed by evaporating 1 ml. The remaining sample extract will then be evaporated to 2 ml and transferred for clean-up to the top of a 1 cm diameter column of 2% deactivated florisil (10 g). For PCDD/PCDF analysis, the eluting solvent is changed to dichloromethane/acetonitrile.

II. PROJECT DESCRIPTION

Gas Chromatography for PCB Analysis

After evaporation of the eluate to 1 ml, the extract will be analyzed with a Hewlett-Packard 5840A gas chromatograph using a 50 M Apiezon L-coated glass capillary and an electron capture detector. The electron capture detector is calibrated using a 1:1:1:1 mixture of an FDA pesticide repository Arochlor composite (1221, 1016, 1254, and 1260) which has been quantitatively analyzed using 29 individual PCB congeners. The remaining congeners are estimated by extrapolation of their response factors from a line of least squares best fit. A computerized data management system reports each of 74 PCB congener concentrations, prints the congener identity which it retrieves from a peak identify file, and sums the congener concentrations to report total PCBs. Concentrations of p.p-DDE, hexachlorobenzene, and mirex are also reported. The limit of detection is 0.05 ppb for individual congeners and 1 ppb for total PCB with analytical accuracy and precision of 10% (14).

Mass Spectrometry for PCDD/PCDF Analysis

The extract is swirled with an equal volume of water twice to remove acetonitrile, dried over sodium sulfate and placed in a 150 ml centrifuge tube. Concentrated sulfuric acid is added and the tube contents are shaken. The top layer is pipetted off into a conical flask containing anhydrous granular sodium carbonate. This extraction is repeated twice more and then the combined extracts are evaporated in a Kuderna-Danish evaporator, hexane being added at intervals to exchange solvents from the dichloromethane to hexane.

After final clean-up on a semi-automated system, extracts are concentrated to volumes of 10 ul by a combination of refluxing in boiling water and vacuum evaporation. Samples are then analyzed by ion monitoring mass spectrometry using a cyanosiloxane-coated capillary column coupled to a Hewlett-Packard Mass Selection Detector. The limit of detection is 1 ppt for individual isomers and total PCDD/PCDF with analytical accuracy and precision of 50% (15).

G. Release of Results

The release of the results for the Mohawk women will be coordinated through Dr. Ben Kelly, a staff physician with the St. Regis Mohawk Health Services. Personal physicians will be the conduit for information to the control mothers. Data provided will include contaminant concentrations (expressed on both a whole milk and fat basis) and also levels of fat and total milk solids for each sample. Comparative data from other New York (16) and U.S. surveys (17, 18) will be provided for reference purposes. Information regarding any health risks associated with specific contamination levels will be transmitted with the results. Each woman will be notified when her results are mailed to Dr. Kelly or her personal physician and will be urged to telephone or meet with him or her to discuss them.

II. PROJECT DESCRIPTION

H. Statistical Analysis

T-tests for independent samples will be used to determine whether the mean concentrations of PCB congeners, total PCB, p,p-DDE, hexachlorobenzene and mirex in the breast milk (fat basis) of the Mohawk population differ significantly (two-tailed $p<0.05$) from those in the comparison group. Logarithmic transformations will be performed if necessary to normalize the distributions and stabilize the variances. Analysis of covariance will be employed to adjust for potential confounders such as maternal age (19).

Analyses will also be conducted to identify any variables that are significantly related to breast milk contaminant levels within the Mohawk and control populations. The risk factors of interest include diet, location and duration of residence, parity and other reproductive characteristics, and health habits such as cigarette smoking. The concentrations of contaminants found in samples of fish and wildlife collected and analyzed in Tasks 1 and 2 of the Health Risk Assessment will be incorporated with the dietary data whenever possible to help quantify exposure. Analysis of variance will be the primary statistical technique for categorical variables, while parametric and non-parametric correlation coefficients will be calculated for continuous factors. Methods such as covariance adjustment and partial correlation will be used to control for potential confounders.

To identify which factors may be responsible if a significant difference between the Mohawk and control populations is observed, the chi-square test will be used to compare occupational histories, reproductive experiences, diet, and health habits. If associations are found, e.g., Mohawk women may be more likely to consume local fish and game, and if the variables in question are also significant correlates of breast milk contamination levels, then multiple regression analysis of the various contaminants will be performed incorporating such variables into the models to control for their effects (20).

A mainframe IBM 4341 is available, to be supplemented with an IBM-PC for data management and analysis. SAS, SPSSX and BMDP software will be utilized. Dr. Syni-An Hwang of the Bureau of Environmental and Occupational Epidemiology, NYSDOH will direct the analysis.

I. Sample Size

Preliminary estimates indicate a likely study enrollment of three Mohawk and three control women per month. After one year, a difference between the Mohawk and control populations of approximately 7% in the mean concentration of a given pollutant will be detectable with a power of 80% and a two-tailed type I error rate of 5%, assuming a relative standard deviation of 10%. At least two years of data, however, would probably be necessary before detailed analyses of breast milk contamination levels by diet, residence, reproductive experience, and other factors would be feasible, given the likelihood of small cell frequencies.

II. PROJECT DESCRIPTION

J. Confidentiality

The data collected for this study are protected from disclosure by Section 206 (1)j of the Public Health Law. Personal identifiers will be used only to locate study subjects. The front page of the interview form contains such identifiers, but it will be separated from the remaining pages upon receipt by the New York State Department of Health. The later pages contain only an identification number. All information will be kept in locked file cabinets and will be accessible only to authorized study personnel.

K. Significance

The proposed investigation is significant for two reasons. Firstly, it provides both the Mohawk and control mothers with an important service by empirically assessing some factors pertaining to the quality of their breast milk. Such information may be helpful to a mother in her decisions concerning whether and how long to nurse her baby and may alleviate anxiety and its possible adverse effect on the mother-infant pair (21). Secondly, the study assists in the development of a database to help assess the impact of environmental pollution at Akwesasne by collecting body burden data indicative of absorption. Breast milk has not been studied in any previous examination of the health and well-being of Akwesasne residents. If high levels of contaminants are found, then follow-up studies of the infants may be warranted. It is a collabortive project involving the St. Regis Mohawk Health Services, the Environmental Health Branch of the St. Regis Band Council, and the Woman's Dance Health Project of the Seventh Generation Fund, and it reflects the public health concerns of the community.

III. TIMETABLE

During the first six months of the project (September, 1987 to February, 1988), we plan to recruit and train project personnel, pre-test interview forms, purchase equipment and supplies, select a control population, and finalize arrangements with the medical services and health care providers in the area, including Canada. Assuming a 75% participation rate, approximately three Mohawks and three controls will be identified, located, and recruited into the study, beginning in the sixth month (March, 1988) and continuing every month for two years. (February, 1990). The chemical analysis of the breast milk samples will be conducted within a month of collection. Data entry will be performed continously, with statistical analysis commencing December, 1989 and concluding six months later. A draft technical report will be available in March, 1990, with a final report completed June, 1990. As outlined in the Memorandum of Understanding between the USEPA, the NYSDEC and the NYSDOH, the public release of the findings will not occur until all involved parties have reviewed and approved the final report.

See Table 1 for a schedule of activities.

Table 1.

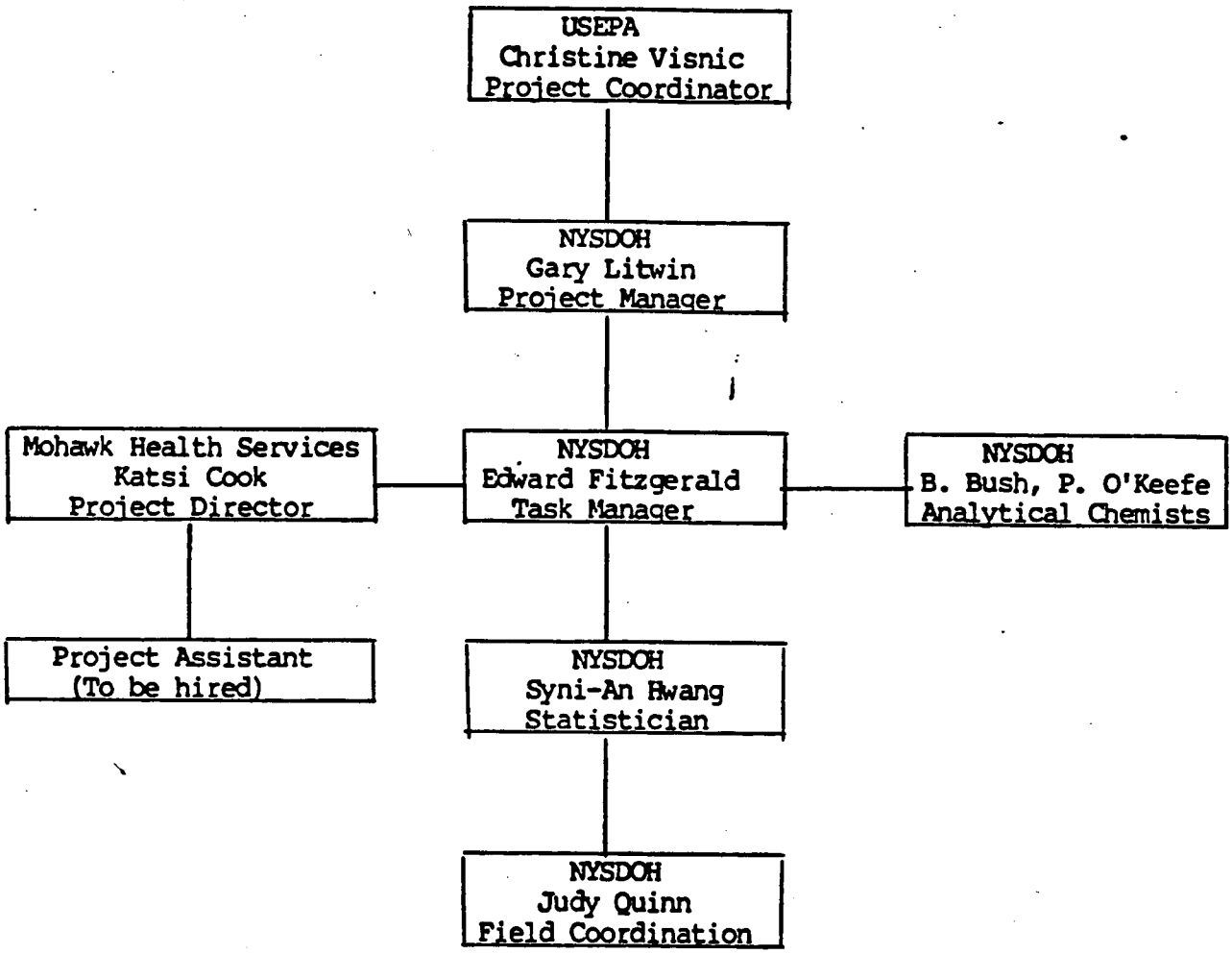
Schedule of Activities for Breast Milk Monitoring Program

Activity	Dates
Develop protocol, establish contacts with community, recruit project staff, etc.	9/87 - 2/88
Collect breast milk samples and conduct interviews	3/88 - 2/90
Analyze samples for PCBs, PCDDs, and PCDFs	4/88 - 3/90
Statistically analyze data	12/89 - 5/90
Write draft report	3/90
Write final report	6/90

IV. PROJECT ORGANIZATION AND RESPONSIBILITY

Project personnel are depicted in Figure 1. Christine Visnic of USEPA is responsible for federal oversight regarding fiscal accountability and quality assurance of the Health Risk Assessment. Gary Litwin of the NYSDOH is the departmental representative for the Health Risk Assessment and responsible for project progress and integrity. Edward Fitzgerald of the NYSDOH is responsible for overall project management, interpretation of results, and report writing. Brian Bush and Patrick O'Keefe of the NYSDOH are responsible for the chemical analysis of the breast milk samples for PCBs and PCDD/PCDFs respectively, with appropriate quality assurance/quality control procedures to ensure the validity and reliability of the data. Katsi Cook will be employed under contract with the Mohawk Health Services. She will coordinate the project at the local level, serve as a liaison between the community and central office staff, and supervise data collection. She will be assisted by a project assistant to be hired. Syni-An Hwang of the NYSDOH will be responsible for data management and statistical analysis. Judith Quinn of the NYSDOH will be responsible for interactions with the local WIC clinics that will be the source populations for the control data.

Figure 1. Organizational Responsibility for Breast Milk Monitoring Program



V. BUDGET

The expenditure plan for the project is outlined in Table 2. Approximately $50,000 will be contracted with Mohawk Health Services, with the remaining $10,000 used for supplies, equipment, and travel by the NYSDOH personnel. Note that these costs do not include the collection of control data, chemical analysis, data management, statistical analysis, reporting, or administrative costs. The total costs for the study exceed the $60,000 allocation by at least 100%.

Table 2. Expenditure Plan for Breast Milk Monitoring Program

Year 1

Contractual Services (Mohawk Health Services)			
Project Director			
40 hrs./mo. @ $13.50/hr. X 12 mos.		$6,480	
Project Assistant			
60 hrs./mo. @ $10.80/hr. X 12 mos.		7,776	
Travel			
Project Director		5,814	
12 trips from Ithaca to Akwesasne @ $450/trip	5,400		
Project Assistant			
150 mi./mo. @ 23c/mi.	414		
Telephone			
$50/mo. X 12 mos.		600	
Shipping and Handling of Specimens			
$100/mo. X 12 mos.		1,200	
Administrative Overhead			
20% of salaries		2,851	
Subtotal			$24,721
Other Than Personal Services			
Supplies		1,650	
50 Breast pumps @ $20 each	1,000		
Specimen bottles	100		
Insulated Cooler	100		
Ice	100		
Briefcase	100		
Paper, folders, etc.	250		
Equipment		5,400	
IBM-PC and related software	5,150		
File cabinet	250		
Travel			
2 Trips from Albany to Akwesasne @ $300/trip		600	
Subtotal			7,650
Total Year 1			$32,371

Year 2

Contractual Services (Mohawk Health Services)		
Project Director		$6,806
40 hrs./mo. @ $14.18/hr. X 12 mos.		
Project Assistant		8,165
60 hrs./mo. @ $11.34/hr. X 12 mos.		
Travel		5,814
Project Director	$5,400	
12 trips from Ithaca to Akwesasne @ $450/trip		
Project Assistant	414	
150 mi./mo. @ 23c/mi.		
Telephone		600
$50/mo. X 12 mos.		
Shipping and Handling of Specimens		1,200
$100/mo. X 12 mos.		
Administrative Overhead		2,994
20% of Salaries		
Subtotal		$25,5
Other Than Personal Services		
Supplies		1,450
50 Breast pumps @ $20 each	1,000	
Specimen bottles	100	
Ice	100	
Paper, folders, etc.	250	
Travel		600
2 trips from Albany to Akwesasne @ 300/trip		
Subtotal		2,0
Total Year 2		$27,6

Total - Years 1 and 2

Contractual Services (Mohawk Health Services)			
Project Director		$13,286	
Project Assistant		15,941	
Travel		11,628	
Project Director	$10,800		
Project Assistant	828		
Telephone		1,200	
Shipping and Handling of Specimens		2,400	
Administrative Overhead		5,845	
Subtotal			$50,30
Other Than Personal Services			
Supplies		3,100	
Equipment		5,400	
Travel		1,200	
Subtotal			9,70
Total Years 1 and 2			$60,00

VI. PROCEDURES FOR QUALITY ASSURANCE/QUALITY CONTROL

The interview data will be reviewed before transmittal to the NYSDOH by Katsi Cook and after receipt by Judith Quinn to ensure their completeness and accuracy. Ambiguous or missing information will be clarified through personal or telephone call-backs. Quality assurance will be maintained in the chemical analysis of breast milk samples through the use of system blanks, fortified blanks, isotopically-labeled standards, and replicate samples. The detailed QA/QC procedures for the PCB analysis are outlined in Appendix F and for the PCDD/PCDF analysis in Appendix G.

The interview and chemical data will be coded and entered onto a dBASE III file by trained personnel. All entries will be subsequently verified to eliminate errors. Edit programs will be developed to detect both out-of-range values and logical inconsistencies. Outliers and questionable results will be investigated and rerun if necessary. The statistical testing will be accompanied by graphs, residual analysis, normality checks, and other procedures to assess the validity of all assumptions.

13) Willet, W.C., Sampson, L. , Stampfer, M.J., et al. "Reproducibility and validity of a semiquantitative food frequency questionnaire." Am J Epid, 122: 51-65, 1985.

14) Bush, B., Snow, J., and Connor, S. "High resolution gas chromatographic analysis of nonpolar chlorinated hydrocarbons in human milk." J Assoc Official Anal Chem: 66: 248-255, 1983.

15) O'Keefe, P.W., Smith, R.M., Hilker, D.R. et al. "A semiautomated clean-up system for PCDD and PCDF in environmental samples. " In Chlorinated Dioxins and Dibenzofurans in the Total Environment, Keith, L.H., Rappe, C., and Choudhary, G. (eds). Boston: Butterworth Publishers, 1983.

16) Bush, B., Snow, J., Connor, S., and Koblintz, R. "Polychlorinated biphenyl congeners (PCB's), p,p-DDE, and hexachlorobenzene in human milk in three areas of upstate New York." Arch Environ Contam Toxicol, 14: 443-450, 1985.

17) Rogan, W.J., Bagniewski, A. and Danstra, T. "Pollutants in breast milk." NEJM, 302: 1450-1453, 1980.

18) Wickizer, T.M., Brilliant, L.B., Copeland, R. and Tilden, R. "Polychlorinated biphenyl contamination of nursing mother's milk in Michigan." Am J Public Health, 71: 124-126, 1981

19) Snedecor, G.W. and Cochran, W.G. Statistical Methods. Ames, IA: Iowa State University Press, 1967.

20) Kleinbaum, D.G. and Kupper, L.L. Applied Regression Analysis and Other Multivariable Methods. Belmont, CA: Wadsworth Publishing Co., 1978.

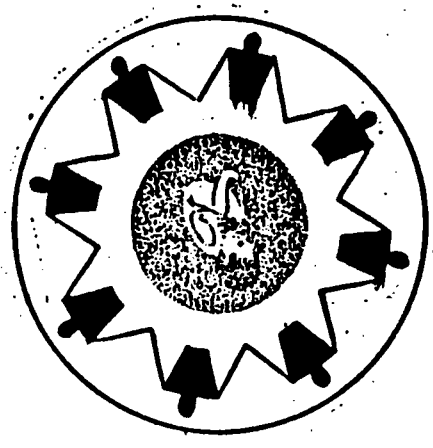
21) Barr, M. "Environmental contamination of breastmilk" (editorial). Am J Public Health, 71: 124-126, 1981.

APPENDIX A

INFORMATIONAL BROCHURE FOR MOTHER'S MILK PROJECT

The Akwesasne Mother's Milk Project is a joint effort of the St.Regis Mohawk Health Services, the Environmental Health Branch of the St.Regis Band Council, and the Akwesasne Envoronment/ MMP Seventh Generation Fund.

Akwesasne Mother's milk Project



...a mohawk women's research project for the health of our future generations

Purpose:

1. To assure Mohawk mothers of the quality of their milk.

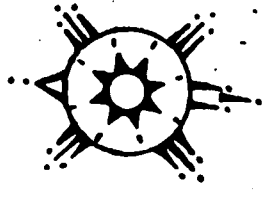
2. To develop a data base of information on mother's milk at Akwesasne to be included in ongoing environmental health studies.

Methods:

1. Once you register to participate,we will contact you by phone or mail to arrange a visit with you in your home.This is a good time to ask any questions you may have about breastfeeding or baby care.

2. A sample of your breastmilk will be collected in a small specimen bottle by hand expression or breast pump,whichever is most comfortable for you.

3. We will need to do an interview with you which asks questions like,What do you eat? Where have you lived? Have you been occupationally exposed to any known contaminants?



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WE NEED YOUR PARTICIPATION :

- If you're pregnant,or are a nursing mother, please register with the project by completing the form on the next page.

- If you have a friend or relative who is pregnant or breastfeeding,please give her a copy of this pamphlet.

- If you are interested in more information on the project,or in more information on environmental and reproductive issues, please fill in the form.

Confidential

All information obtained during the interviews and milk sample collection will be kept confidential. Each participant will be kept informed of the research process,and will receive a copy of the results of the testing of her milk sample.

Date _____

Name _____

Address _____

_____

Phone _____

[] I am pregnant and my due date is _____.

[] I am breastfeeding now and my baby is _____months old.

[] Yes,I would like more information about environmental and reproductive health issues.

If you have any questions about breastfeeding,please contact:

Beverly Cook, R.N. Clinic
Wendy Wolf, Nutritionist
Louise Cook, R.N. Medical Outreach

Phone: 358-2272

APPENDIX B

INFORMED CONSENT FORM

Participant Informed Consent Form - Akwesasne Breast Milk Monitoring Program

Participant's Name: _____

Participant's Address: _____

_____

The New State Department of Health, the St. Regis Mohawk Health Services, the Environmental Health Branch of the St. Regis Band Council, and the Women's Dance Health Project of the Seventh Generation Fund are jointly conducting a medical research study of environmental contamination in the breast milk of women who reside at Akwesasne. We would like you to participate by providing us with a sample of your breast milk. We will also ask you to complete a 20 minute interview. It includes questions about your occupational, reproductive, residential, social and dietary histories.

Dr. Ben Kelly of the St. Regis Mohawk Health Services will be given a copy of the results of the chemical analysis of your milk. It will include levels of PCB's, DDE, hexachlorobenzene, and mirex. The fat content and the total amount of solids in your milk will also be provided to Dr. Kelly. We will inform you of the date that we mail these results and recommend that you telephone or meet with him to discuss them.

Although you and your family may not benefit from this study, your participation may help to improve medical knowledge of enviromentally related illness in the future.

Any information you provide will remain strictly confidential and be used for medical research purposes only. At no time will your name be mentioned and any reports that result from this research will involve statistical information only. Your participation is completely voluntary and no penalty will be involved if you choose not to participate. You have the right to receive answers to any questions you may have concerning this study and may discontinue participation at any time.

Signature of Participant: _____

Date: _____

APPENDIX C

INTERVIEW FORM

AKWESASNE MOTHER'S MILK PROJECT

DEMOGRAPHICS

Date of interview/__/__/__/

1. Name of mother: /_____/___/_____/
   First MI Last

2. Social Security # /_____/____/_____/

3. Mailing address: /_____/
   /_____/
   /_____/

4. Home phone: /_____/_____-_____/

5. Work phone: /_____/_____-_____/

6. Date of birth: /_____/_____/_____/
   month day year

7. Height: (without shoes) /_____/_____/
   feet inches

8. Weight: (with indoor clothing before last pregnancy)
   /_____/
   lbs
   (with indoor clothing at 9 months pregnant)
   /_____/
   lbs
   (with indoor clothing currently)
   /_____/
   lbs

9. Years of school completed: /_____/

10. Current marital status: (check one)
    Married /__/ Divorced /__/
    Separated /__/ Widowed /__/
    Never married /__/

11. If currently married – name of spouse: /_____/_____/
    First Last

12. Source of health care: /_____/

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HEALTH HABITS

Now I would like to ask you some questions about your use of tobacco, alcohol, coffee, and medications for the periods before and during your last pregnancy, and since your last child was born.

Did you:	Before last pregnancy	During last pregnancy	Since last child was born
13. Smoke cigarettes?	Yes/_/No/_/	Yes/_/No/_/	Yes/_/No/_/
If yes, avg #/day:	/_____/	/_____/	/_____/
14. Drink beer?	Yes/_/No/_/	Yes/_/No/_/	Yes/_/No/_/
If yes, avg # of 12 oz cans, bottles, or glasses/wk:	/_____/	/_____/	/_____/
15. Drink wine?	Yes/_/No/_/	Yes/_/No/_/	Yes/_/No/_/
If yes, avg # of 4 oz glasses/wk:	/_____/	/_____/	/_____/
16. Drink liquor or mixed drinks?	Yes/_/No/_/	Yes/_/No/_/	Yes/_/No/_/
If yes, avg # of drinks with 1 and 1/2 oz of liquor/wk:	/_____/	/_____/	/_____/
17. Drink regular coffee?	Yes/_/No/_/	Yes/_/No/_/	Yes/_/No/_/
If yes, avg # of 8 oz cups/day:	/_____/	/_____/	/_____/
18. Use prescription medications?	Yes/_/No/_/	Yes/_/No/_/	Yes/_/No/_/
If yes, what was medication?	/_____/	/_____/	/_____/
avg # of times taken/wk:	/_____/	/_____/	/_____/
medication?	/_____/	/_____/	/_____/
avg # of times taken/wk:	/_____/	/_____/	/_____/
medication?	/_____/	/_____/	/_____/
avg # of times taken/wk:	/_____/	/_____/	/_____/

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RESIDENTIAL HISTORY

19. Starting with your present residence and working backwards, please indicate every residence you have lived at since birth, and for how many years.

Dates Years From	To	Indicate house numbers under area of of Reserve, or other address Com Isl	Snye	St Reg	Raqt Pt	US West	US East	other	What was the source of drinking water Well	River	Village	Rain	Oth
									S/_/ D/_/	SL/_/ Rq/_/ SR/_/	/_/	/_/	/_/
									S/_/ D/_/	SL/_/ Rq/_/ SR/_/	/_/	/_/	/_/
									S/_/ D/_/	SL/_/ Rq/_/ SR/_/	/_/	/_/	/_/
									S/_/ D/_/	SL/_/ Rq/_/ SR/_/	/_/	/_/	/_/
									S/_/ D/_/	SL/_/ Rq/_/ SR/_/	/_/	/_/	/_/
									S/_/ D/_/	SL/_/ Rq/_/ SR/_/	/_/	/_/	/_/

Well: S–shallow, D–deep Rivers: SL–St. Lawrence, Rq–Racquet, SR–St. Regis

20. Did you ever visit or come in contact with the General Motors Central Foundry site? Yes/__/ No/__/

If yes, what did you do there? /_____/

When? /_____/_____/
month year

How long? /_____/
number of months

OCCUPATIONAL HISTORY

23. Starting with your most recent job outside the home and working backwards, please indicate every job that you have held for one year or more.

Dates years From	To	Type of industry trade, and company	Job or position	Description of work	List any exposures to hazardous materials

REPRODUCTIVE HISTORY

22. Now I need to ask you some questions about each of your pregnancies, stillbirths, or abortions

					If live birth:		
Pg#	Outcome	Date of delivery or termination	Child's first name	Sex	Place of birth (hospital, city, state, location of home delivery	Baby's birth weight, length	
1	/_/_/_/_/ L M S A	/__/__/__/ M D Y	/_____/	/__/	/_____/ /_____/ /_____/	/____/____/ lbs inches	
2	/_/_/_/_/ L M S A	/__/__/__/ M D Y	/_____/	/__/	/_____/ /_____/ /_____/	/____/____/ lbs inches	
3	/_/_/_/_/ L M S A	/__/__/__/ M D Y	/_____/	/__/	/_____/ /_____/ /_____/	/____/____/ lbs inches	
4	/_/_/_/_/ L M S A	/__/__/__/ M D Y	/_____/	/__/	/_____/ /_____/ /_____/	/____/____/ lbs inches	
5	/_/_/_/_/ L M S A	/__/__/__/ M D Y	/_____/	/__/	/_____/ /_____/ /_____/	/____/____/ lbs inches	
6	/_/_/_/_/ L M S A	/__/__/__/ M D Y	/_____/	/__/	/_____/ /_____/ /_____/	/____/____/ lbs inches	

Key: Pg—pregnancy, L—livebirth, M—miscarriage, S—stillbirth, A—abortion,

starting with the first and including any miscarriages

			If deceased		
Delivery complications or problems in first year of life (defects prematurity, etc)	If the child was breast-fed how long? (# weeks)	the child still living?	Date of death	Place of death	Cause of death
/____/ /____/ /____/	/____/	/__/__/ Y N	/__/__/__/ M D Y	/____/	/____/
/____/ /____/ /____/	/____/	/__/__/ Y N	/__/__/__/ M D Y	/____/	/____/
/____/ /____/ /____/	/____/	/__/__/ Y N	/__/__/__/ M D Y	/____/	/____/
/____/ /____/ /____/	/____/	/__/__/ Y N	/__/__/__/ M D Y	/____/	/____/
/____/ /____/ /____/	/____/	/__/__/ Y N	/__/__/__/ M D Y	/____/	/____/
/____/ /____/ /____/	/____/	/__/__/ Y N	/__/__/__/ M D Y	/____/	/____/

DIETARY HISTORY

23. Please indicate how frequently you ate the following foods in the year before and during your last pregnancy, and what foods you have eaten in the past. Also indicate where you usually obtained these foods.

Food	Average # of times/week, month, year (fill out one), and the usual source			
	During last pregnancy	In the year before your pregnancy	Before two years ago	Number of years
Milk	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/_____/
Eggs	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/_____/
Fruits/Vegetables	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/_____/
Meats, including beef, pork, lamb, veal, luncheon meats	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/_____/
Poultry including chicken, cornish hens, turkey	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/_____/

DIETARY HISTORY (continued)

Food	Average # of times/week, month, year (fill out one), and the usual source			
	During last pregnancy	In the year before your pregnancy	Before two years ago	Number of years
Organ meats including liver heart, spleen	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/_____/
Wildlife animals: duck	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/_____/
pheasant	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/_____/
goose	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/_____/
deer	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/_____/
rabbit	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/_____/

DIETARY HISTORY (continued)

Food	Average # of times/week, month, year (fill out one), and the usual source			
	During last pregnancy	In the year before your pregnancy	Before two years ago	Number of years
muskrat	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/_____/
turtle	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/_____/
other	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/_____/
if other, specify:	/_____/	/_____/	/_____/	
Fish:				
trout	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/_____/
bass	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/_____/
perch	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/_____/

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DIETARY HISTORY (continued)

Food	Average # of times/week, month, year (fill out one), and the usual source During last pregnancy	In the year before your pregnancy	Before two years ago	Number of years
bullhead	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/_____/
pike	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/_____/
pickerel/walleye	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/_____/
sturgeon	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/_____/
other	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/__/__/__/ w m y /_____/ source	/_____/
If other, specify:	/_____/	/_____/	/_____/	

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FOOD PREPARATION

24. Please answer the following questions regarding food preparation for any items you have consumed in the past year.

Food	Do you trim fat or skin	How do you Cook*	Type of shortening used	Usual Source of shortening
Meats	Yes/__/ No/__/	B/_/F/_/R/_/S/_/	Butter Yes/_/No/_/	/_____/
		O/_____/	Lard Yes/_/No/_/	/_____/
Poultry	Yes/__/ No/__/	B/_/F/_/R/_/S/_/	Butter Yes/_/No/_/	/_____/
		O/_____/	Lard Yes/_/No/_/	/_____/
Duck	Yes/__/ No/__/	B/_/F/_/R/_/S/_/	Butter Yes/_/No/_/	/_____/
		O/_____/	Lard Yes/_/No/_/	/_____/
Pheasant	Yes/__/ No/__/	B/_/F/_/R/_/S/_/	Butter Yes/_/No/_/	/_____/
		O/_____/	Lard Yes/_/No/_/	/_____/
Goose	Yes/__/ No/__/	B/_/F/_/R/_/S/_/	Butter Yes/_/No/_/	/_____/
		O/_____/	Lard Yes/_/No/_/	/_____/
Deer	Yes/__/ No/__/	B/_/F/_/R/_/S/_/	Butter Yes/_/No/_/	/_____/
		O/_____/	Lard Yes/_/No/_/	/_____/
Rabbit	Yes/__/ No/__/	B/_/F/_/R/_/S/_/	Butter Yes/_/No/_/	/_____/
		O/_____/	Lard Yes/_/No/_/	/_____/
Muskrat	Yes/__/ No/__/	B/_/F/_/R/_/S/_/	Butter Yes/_/No/_/	/_____/
		O/_____/	Lard Yes/_/No/_/	/_____/
Turtle	Yes/__/ No/__/	B/_/F/_/R/_/S/_/	Butter Yes/_/No/_/	/_____/
		O/_____/	Lard Yes/_/No/_/	/_____/

Key: B–broil, F–fry, R–roast, S–stew, O–other

FOOD PREPARATION
(continued)

Food	Do you trim fat or skin	How do you Cook*	Type of shortening used	Usual Source of shortening
trout	Yes/__/ No/__/	B/_/F/_/R/_/S/_/ O/_____/	Butter Yes/_/No/_/ Lard Yes/_/No/_/	/_____/ /_____/
bass	Yes/__/ No/__/	B/_/F/_/R/_/S/_/ O/_____/	Butter Yes/_/No/_/ Lard Yes/_/No/_/	/_____/ /_____/
perch	Yes/__/ No/__/	B/_/F/_/R/_/S/_/ O/_____/	Butter Yes/_/No/_/ Lard Yes/_/No/_/	/_____/ /_____/
bullhead	Yes/__/ No/__/	B/_/F/_/R/_/S/_/ O/_____/	Butter Yes/_/No/_/ Lard Yes/_/No/_/	/_____/ /_____/
pike	Yes/__/ No/__/	B/_/F/_/R/_/S/_/ O/_____/	Butter Yes/_/No/_/ Lard Yes/_/No/_/	/_____/ /_____/
pickerel/ walleye	Yes/__/ No/__/	B/_/F/_/R/_/S/_/ O/_____/	Butter Yes/_/No/_/ Lard Yes/_/No/_/	/_____/ /_____/
sturgeon	Yes/__/ No/__/	B/_/F/_/R/_/S/_/ O/_____/	Butter Yes/_/No/_/ Lard Yes/_/No/_/	/_____/ /_____/
other (specify)				
/_____/	Yes/__/ No/__/	B/_/F/_/R/_/S/_/ O/_____/	Butter Yes/_/No/_/ Lard Yes/_/No/_/	/_____/ /_____/
/_____/	Yes/__/ No/__/	B/_/F/_/R/_/S/_/ O/_____/	Butter Yes/_/No/_/ Lard Yes/_/No/_/	/_____/ /_____/

Key: B–broil, F–fry, R–roast, S–stew, O–other

PAST FOOD PREPARATION

25. Prior to the past year did you prepare the following foods differently than you do now? Yes/__/ No/__/

If yes, please specify how and for how long.

Food	Did you trim fat or skin	How did you cook*	Type of shortening used	Usual Source of shortening
Meats	Yes/__/ No/__/	B/_/F/_/R/_/S/_/	Butter Yes/_/No/_/	/_____/
how long:	From/_____/	To/_____/	Lard Yes/_/No/_/	/_____/
Poultry	Yes/__/ No/__/	B/_/F/_/R/_/S/_/	Butter Yes/_/No/_/	/_____/
how long:	From/_____/	To/_____/	Lard Yes/_/No/_/	/_____/
Duck	Yes/__/ No/__/	B/_/F/_/R/_/S/_/	Butter Yes/_/No/_/	/_____/
how long:	From/_____/	To/_____/	Lard Yes/_/No/_/	/_____/
Pheasant	Yes/__/ No/__/	B/_/F/_/R/_/S/_/	Butter Yes/_/No/_/	/_____/
how long:	From/_____/	To/_____/	Lard Yes/_/No/_/	/_____/
Goose	Yes/__/ No/__/	B/_/F/_/R/_/S/_/	Butter Yes/_/No/_/	/_____/
how long:	From/_____/	To/_____/	Lard Yes/_/No/_/	/_____/
Deer	Yes/__/ No/__/	B/_/F/_/R/_/S/_/	Butter Yes/_/No/_/	/_____/
how long:	From/_____/	To/_____/	Lard Yes/_/No/_/	/_____/
Rabbit	Yes/__/ No/__/	B/_/F/_/R/_/S/_/	Butter Yes/_/No/_/	/_____/
how long:	From/_____/	To/_____/	Lard Yes/_/No/_/	/_____/
Muskrat	Yes/__/ No/__/	B/_/F/_/R/_/S/_/	Butter Yes/_/No/_/	/_____/
how long:	From/_____/	To/_____/	Lard Yes/_/No/_/	/_____/
Turtle	Yes/__/ No/__/	B/_/F/_/R/_/S/_/	Butter Yes/_/No/_/	/_____/
how long:	From/_____/	To/_____/	Lard Yes/_/No/_/	/_____/

Key: B–broil, F–fry, R–roast, S–stew

PAST FOOD PREPARATION
(continued)

Food	Did you trim fat or skin	How did you cook*	Type of shortening	Usual Source of shortening
trout	Yes/__/ No/__/	B/_/F/_/R/_/S/_/	Butter Yes/_/No/_/	/_____/
how long:	From/_____/	To/_____/	Lard Yes/_/No/_/	/_____/
bass	Yes/__/ No/__/	B/_/F/_/R/_/S/_/	Butter Yes/_/No/_/	/_____/
how long:	From/_____/	To/_____/	Lard Yes/_/No/_/	/_____/
perch	Yes/__/ No/__/	B/_/F/_/R/_/S/_/	Butter Yes/_/No/_/	/_____/
how long:	From/_____/	To/_____/	Lard Yes/_/No/_/	/_____/
bullhead	Yes/__/ No/__/	B/_/F/_/R/_/S/_/	Butter Yes/_/No/_/	/_____/
how long:	From/_____/	To/_____/	Lard Yes/_/No/_/	/_____/
pike	Yes/__/ No/__/	B/_/F/_/R/_/S/_/	Butter Yes/_/No/_/	/_____/
how long:	From/_____/	To/_____/	Lard Yes/_/No/_/	/_____/
pickerel	Yes/__/ No/__/	B/_/F/_/R/_/S/_/	Butter Yes/_/No/_/	/_____/
how long:	From/_____/	To/_____/	Lard Yes/_/No/_/	/_____/
walleye	Yes/__/ No/__/	B/_/F/_/R/_/S/_/	Butter Yes/_/No/_/	/_____/
how long:	From/_____/	To/_____/	Lard Yes/_/No/_/	/_____/
sturgeon	Yes/__/ No/__/	B/_/F/_/R/_/S/_/	Butter Yes/_/No/_/	/_____/
how long:	From/_____/	To/_____/	Lard Yes/_/No/_/	/_____/
other				
/_____/	Yes/__/ No/__/	B/_/F/_/R/_/S/_/	Butter Yes/_/No/_/	/_____/
how long:	From/_____/	To/_____/	Lard Yes/_/No/_/	/_____/
/_____/	Yes/__/ No/__/	B/_/F/_/R/_/S/_/	Butter Yes/_/No/_/	/_____/
how long:	From/_____/	To/_____/	Lard Yes/_/No/_/	/_____/

Key: B–broil, F–fry, R–roast, S–stew

APPENDIX D

MEDICAL RELEASE FORM

STATE OF NEW YORK
DEPARTMENT OF HEALTH

Bureau of Environmental and Occupational Epidemiology

Consent to Release Information

I hereby authorize the release of information from the records of (mother patient's name)_____ to Dr. Edward Fitzgerald of the New York State Department of Health for the purpose of evaluating potential health hazards related to PCBs. I understand this authorization covers only information required to made this evaluation and that the New York State Department of Health will maintain the confidentiality of this information pursuant to the Public Health Law.

Patient's Signature: _____

Address: _____

_____

Date: _____

The above records should be submitted to:

Edward Fitzgerald, Ph.D.
New York State Department of Health
2 University Plaza
Albany, New York 12237

APPENDIX E

CHAIN-OF-CUSTODY

NEW YORK STATE DEPARTMENT OF HEALTH
CENTER FOR LABORATORIES AND RESEARCH
ALBANY, N.Y. 12201

CHAIN OF CUSTODY RECORD

Must be completed for samples which might be used for enforcement proceedings or litigation.

SAMPLE ID (LAB USE ONLY)	FIELD REFERENCE NO.	DATE/TIME COLLECTED	SAMPLE COLLECTION POINT	TYPE: WATER, AIR SOIL, ETC.

SPECIFY METHOD OF PRESERVATION

- ☐ NaOH
- ☐ Cool, 4°C
- ☐ Acidification (specify)
- ☐ Other (specify

TRANSPORTING SAMPLES

DURING TRANSPORT OF THE SAMPLE FROM SAMPLING SITE TO LABORATORY, THE CHAIN OF CUSTODY MUST BE UNBROKEN. GENERALLY THIS WILL REQUIRE THAT THE SAMPLE BE DELIVERED BY THE SAMPLE COLLECTOR OF HIS DESIGNATED REPRESENTATIVE WHO WILL SIGN FOR THE RECEIPT, INTEGRITY AND TRANSFER OF THE SAMPLE DURING SHIPMENT. IF INTEGRITY OF SAMPLE IS QUESTIONED, DESCRIBE PROBLEM ON REVERSE SIDE OF THIS FORM.

CUSTODY OF SAMPLES

	NAME	AFFILIATION	DATE	TIME
1. Sample Container Prepared by				
2. Received by				
3. Received by				
4. Sample Collected by				
5. Sample Received by				
6. Sample Received by				
7. Sample Received by				
8. Sample Received by				
9. Sample Received by				
10. Sample Rec'd Lab by				
11. Sample Accessioned by				

CC-1

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APPENDIX F

QA/QC PROCEDURES FOR PCB ANALYSIS

QUALITY ASSURANCE PROCEDURES FOR HUMAN MILK ANALYSIS

IN THE HEALTH RISK ASSESSMENT AT AKWESASNE

The methods to be used have been published in the Journal of the Association of Official Analytical Chemists, Vol 65, pg 555 (1982) and Vol 66, pg 246 (1983). They are outlined below.

Control of the gas chromatograph. The electron capture detector of the chromatograph is calibrated using a 1:1:1:1 mixture of EPA pesticide repository Aroclor mixtures (Aroclors 1221, 1016, 1254 and 1260) which has been quantitatively analysed using 29 individual PCB congeners. The remaining 50 congeners were estimated by extrapolation of their response factors from a line of least squares best fit. Fluctuations in electron capture detector sensitivity are controlled by an automatic recalibration after each fourth specimen analysis. Reporting software gives the presentation shown in Fig. 1. If Total PCB deviates more than 10% from the expected value, the subsequent four specimens are reanalysed; if an individual congener deviates more than two times from the expected standard deviation for that congener, it is recalculated using peak height measurement. The standard deviation for each congener is determined when a new capillary column is installed, approximately once every three months. Quality control charts will be plotted daily for the sum of all PCB (Total PCB) and three commonly found PCB congeners (2,4,-dichlorobiphenyl, 2,4,5,2',4',5'-hexachlorobiphenyl, 2,3,4,5,2',4',5',-heptachlorobiphenyl).

Control of sample extraction. Values of PCB in this region have a mean of approximately 25 ng/g with a standard deviation of 15 ng/g. Standard human serum spiked with Aroclor 1260 is available from the National Bureau of Standards (NBS) at 106 $\pm$ 1.3 ng/g. Because of the difficulty of producing

properly characterised biologically bound standard material, the simple expedient of reconstituting NBS serum and then diluting it with cow milk will be used to produce quality control milk samples at 25 ng/g. One such sample will be processed with each batch of ten milk samples or for smaller batches one per batch. The method yields a recovery of 85 ± 8% for Total PCB. If the recovery of the quality control sample is less than 70%, then the entire batch will be rerun after trouble shooting has returned the recovery for the quality control sample to 85%. If there is an insufficient quantity of any particular sample to permit it's being rerun, its value will be adjusted arithmetically to a recovery of 85%. The interview containing information regarding diet, occupation, residential history and other risk factors will be separated from the milk samples before analysis to permit a determination of PCB concentration which is blind to exposure status.

SAMPLE TYPE: QUALITY CONTROL

DATE ANALYZED: 05/12/98
VIAL NUMBER: 2
DILUTION FACTOR: 1.000

SAMPLE NAME: MIX FR-1 200PPB

PCB SUBSTITUTION PATTERN	PPB DETECTED	% DEV. FROM AMT APPLIED	PCB SUBSTITUTION PATTERN	PPB DETECTED	% DEV. FROM AMT APPLIED
2	58.108	5.44	245/26	7.576	-4.05
2/3	19.302	8.65	234/26	9.347	-14.37
26	2.383	14.14	[illegible]	11.203	[illegible]
4	43.357	3.73	236/24	7.383	-2.38
25	2.350	-9.50	234/23	25.346	-6.31
24	8.583	1.24	236/34	4.512	-7.73
2/3	8.873	-1.24	2356/25	10.735	-3.62
25/2+23	14.754	1.13	2356/23	3.337	[illegible]
2/4	21.792	-1.77	236/245(234/235)	15.221	[illegible]
25/2	21.540	-1.20	24/34	[illegible]	-100.00
HEXACHLOROBENZENE	5.171	2.42	2345/25(CL5E)	1.532	9.46
24/2	7.267	-0.45	234/236(2356/236)	9.457	-1.51
23/2	9.600	0.00	2356/233(245/34)+2356/246	5.735	-0.26
26/4	6.132	-13.32	245/34(2346/236)	13.377	-3.13
4/4	15.132	-5.85	2346/236(236/245)	5.457	3.03
245/2	1.307	3.33	236/245(CL5D)	5.335	3.24
25/34+236/2	7.333	0.45	234/34	2.131	-72.13
24/3(CL3A)	3.143	3.03	245/245	127.025	245.37
24/25(CL3B)	1.317	-24.71	234/236(CL6A)	2.473	-0.83
25/4(24/2)	13.573	-0.74	2356/34(CL6B)	4.335	-2.31
23/3	5.833	-3.11	2345/23	[illegible]	-100.00
24/4	13.327	-1.44	234/245	25.735	-3.17
23/4	14.000	0.00	2356/233(2346/34)	5.359	-1.33
23/25	22.773	-2.25	234/234	5.341	3.77
24/25	14.947	-1.02	2356/245	11.485	-2.94
23/25	16.239	-1.75	2346/245	13.993	-1.64
24/24	5.725	1.44	2345/246	5.555	-1.32
23/24(CL4A)	5.164	1.21	2356/234	0.000	-100.00
23/23+234/2(CL4B)	3.870	-0.80	2346/234(245/345)	4.573	1.25
236/3(CL4C)	11.341	0.37	2345/2356+245/345(2345/236)	12.512	333.35
236/4(CL4D)	12.331	3.33	MIREX	3.294	-6.06
235/23(CL5A)	11.135	3.31	2345/34	4.742	1.33
OCTACHLOROSTYRENE	3.733	-4.34	2345/235	2.980	-0.34
234/25	7.334	-1.73	2345/245	27.051	-2.83
236/23	9.351	-1.39	2345/234	12.025	0.23
25/34	13.342	-2.57	2346/2356	13.959	116.55
245/4	3.331	-0.83	CL7	1.535	-53.79
24/34	4.113	-3.37	2345/2346	2.313	-19.49
34/23	4.925	-2.04	23456/245	5.224	24.14
236/236	3.273	-3.74	23456/234	3.387	1.12
245/25	17.271	-4.53	2345/2345	179.334	2566.65
245/24	5.733	-3.31			

TOTAL CL1: 192.760 CL2: 88.853 CL3: 113.625 CL4: 132.031
CL5: 129.740 CL6: 224.331 CL7: 26.713 CL8: 211.054

TOTAL PCB: 1129.577
DEVIATION FROM AMT APPLIED: 24.570 %

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APPENDIX G

QA/QC PROCEDURES FOR PCDD/PCDF ANALYSIS

QUALITY ASSURANCE

The accuracy of this analysis relies on a complex combination of liquid chromatography, high resolution gas chromatography, and low resolution mass spectrometry to remove interferences and provide a high degree of sensitivity and selectivity. Therefore, because of the complexity it is of primary importance that an isotopically labeled internal standard be added to each sample prior to sampling or analysis to provide both a qualitative check and accurate quantification when sample recovery data is variable.

Blanks

Four types of blanks are generally run: (1) a system blank prior to the use of any glassware to ensure no carry over from prior samples; (2) a method blank, run simultaneously and using the same standards, solvent, adsorbent, and glassware as the actual samples; (3) isotopically-labeled standards; and (4) benzene blanks to check for GC and syringe carry over. Because many type 3 and 4 blanks may be run, the data is usually not included in the report but is available upon request.

Fortified Blanks

A fortified blank is a method blank with added known amounts of native CDDs and CDFs. In the absence of interlaboratory studies, this provides the best available overall check on the accuracy of the method for a given group of samples.

Precision

The best measure of precision is obtained from replicate samples. The target value for this is within 20%. The recovery of the internal standard available for each sample will provide a measure of precision; however, this is based on external standardization (necessitating handling of 1 to 4 micro-

liters of total sample necessary for high sensitivity) and is known to be less precise than internal standardization.

Criteria for detection

To be detected as a CDD or CDF isomer in a sample all of the following conditions must be met:

1. co-elution on GC with appropriate standard if available,
2. proper mass within unit mass,
3. response at two ions corresponding to M, M+2; response must be in proper ratio ±20%; optional response corresponding to M-COCl as sensitivity permits,
4. Adequate recovery of the corresponding internal standard,
5. acceptable QC blanks and spikes,
6. negligible mass spectral interference,
7. signal-to-noise ratio >2.5 for both ions.

Available standards include:

	All 22 tetra CDDs
^{13}C 2378 tetra CDD	2378 tetra CDD
^{13}C 12378 penta CDD	12378 penta CDD
^{13}C 123678 hexa CDD	123678 hexa CDD
^{13}C 1234678 hepta CDD	1234678 hepta CDD
^{13}C octa CDD	octa CDD
^{13}C 2378 tetra CDF	2378 tetra CDF
^{13}C 12378 penta CDF	12378 penta CDF
	23478 penta CDF
^{13}C 123478 hexa CDF	123478 hexa CDF
	234678 hexa CDF

123678 hexa CDF

123789 hexa CDF

^{13}C 1234678 hepta CDF

1234678 hepta CDF

^{13}C octa CDF

octa CDF

Purity is checked by mass spectrometry.

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ATTACHMENT 5

HEALTH RISK ASSESSMENT

PROJECT COORDINATION AND SCHEDULING OUTLINE

ATTACHMENT 5

Table 1

General Motors Corp. - CFD-Massena, New York Facility
Health Risk Assessment
Project Coordination and Scheduling Outline

Task	USEPA Project Manager	State Project Manager	Task Manager	Start Field Work	Submit Draft Report	Submit Final Report
1 - Fish	C. Visnic USEPA	J. Reagan NYSDEC	R. Sloan NYSDEC	April 1988	November 1988	January 1989
2 - Wildlife	C. Visnic USEPA	J. Reagan NYSDEC	W. Stone NYSDEC	August 1987	August 1988	October 1988
3 - Breast Milk	C. Visnic USEPA	G. Litwin NYSDOH	E. Fitzgerald NYSDOH	September 1987	March 1990	June 1990
4 - Risk Assessment Final Report	C. Visnic USEPA	G. Litwin NYSDOH	J. Hawley NYSDOH	August 1988	June 1990	September 1990

Notes:

1. All dates are listed as of February 1988.

ATTACHMENT 5

Table 1

General Motors Corp. - CFD Massena, New York Facility
Health Risk Assessment
Project Coordination and Scheduling Outline

Task	USEPA Project Manager	State Project Manager	Task Manager	Start Field Work	Submit Draft Report	Submit Final Report
1 - Fish	C. Visnic USEPA	J. Reagan NYSDEC	R. Sloan NYSDEC	May 1988	November 1988	January 1989
2 - Wildlife	C. Visnic USEPA	J. Reagan NYSDEC	W. Stone NYSDEC	August 1987	August 1988	October 1988
3 - Breast Milk	C. Visnic USEPA	G. Litwin NYSDOH	E. Fitzgerald NYSDOH	September 1987	June 1990	September 1990
4 - Risk Assessment Final Report	C. Visnic USEPA	G. Litwin NYSDOH	J. Hawley NYSDOH	August 1988	September 1990	December 1990

Notes:

1. All dates are listed as of May 1988.

ATTACHMENT 6

HEALTH RISK ASSESSMENT

ITEMIZED EXPENDITURES

ATTACHMENT 6

MOU Among USEPA, NYSDEC and NYSDOH
Distribution of Health Risk Assessment Funds
General Motors Corporation, Central Foundry Division
Health Risk Assessment
Itemized Expenditures

Period Beginning: _____

Period Ending: _____

Activity Code								
Personnel Service								
Fringe Benefits								
Indirect Labor								
Supplies								
Travel								
Contractual								
Equipment								
Total								

CERTIFICATION: I certify to the best of my knowledge and belief that this report is correct and complete and that all expenditures are for the purpose set forth in the controlling documents:

_____

Signature of Authorized Certifying Official

_____

Typed or Printed Name, Title and Agency

_____ _____

Date Report Submitted Telephone Number

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