



DEPARTMENT OF
ECOLOGY
State of Washington

Addendum to Quality Assurance Project Plan

Analyzing Chlorinated Pesticide Residues in Fish from Washington Background Lakes: Analysis of Supplemental PBTs in Non-Background Waterbodies

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Publication Information

Addendum

This addendum is an addition to an original Quality Assurance Project Plan. The addendum is not a correction (errata) to the original plan.

This addendum is available on the Department of Ecology's website at www.ecy.wa.gov/biblio/1103108Addendum1.html

Ecology's Activity Tracker Code for this study is 02-044.

Original Publication

Quality Assurance Project Plan: Analyzing Chlorinated Pesticide Residues in Fish from Washington Background Lakes.

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DEPARTMENT OF ECOLOGY
Environmental Assessment Program

November 17, 2011

TO: Carol Kraege, Waste 2 Resources Program
Holly Davies, Waste 2 Resources Program

THROUGH: Dale Norton, Toxic Studies Unit Supervisor, Environmental Assessment Program
Will Kendra, Statewide Coordination Section Manager, Environmental Assessment Program

FROM: Art Johnson, Environmental Assessment Program

SUBJECT: Addendum to Quality Assurance Project Plan for Analyzing Chlorinated Pesticide Residues in Fish from Washington Background Lakes: Analysis of Supplemental PBTs in Non-Background Waterbodies
Activity Tracker Code: 02-044 Publication No: 11-03-108

The purpose of this addendum is to expand the chemical analyses being conducted for the chlorinated pesticide background study. The Waste 2 Resources Program has requested that fish samples be analyzed for the PBTs listed below as an extension of this project. These chemicals have either not previously been analyzed in Washington State fish or are a current focus of efforts to reduce or eliminate toxic threats.

- Hexabromocyclododecane (HBCDD)
- Tetrabromobisphenol A (TBBPA)
- Polychlorinated naphthalenes (PCNs)
- Short-chain chlorinated paraffins (SCCPs)
- Perfluorinated compounds (PFCs)
- Polybrominated diphenylethers (PBDEs)
- Cadmium (Cd) and lead (Pb)

Four fillet and four whole body samples will be prepared from bottom feeding species collected from four waterbodies with a history of toxics issues: Lake Washington, Lower Columbia River, Yakima River, and Lake Spokane. The objective is to assess the occurrence of these contaminants in fish from waterbodies where potential for detection is thought to be the greatest.

Organic compounds will be analyzed by the same laboratory contracted for the pesticide work, AXYS Analytical Services in Sidney, BC. Cadmium and lead will be analyzed by Manchester Laboratory. The QAPP addendum provides further details on how this part of the study will be conducted.

cc: Bill Kammin, Ecology Quality Assurance Officer
Stuart Magoon, Manchester Laboratory Director
Cheryl Niemi, Water Quality Program
Susan Braley, Water Quality Program
Melissa Gildersleeve, Water Quality Program
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Project Description

The Washington State Department of Ecology (Ecology) Water Quality Program (WQP) and Environmental Assessment Program (EAP) have initiated a statewide study to obtain estimates of the chlorinated pesticide background in Washington State freshwater fish. The design of this project is described in a Quality Assurance Project Plan (QAPP) (Johnson, 2011).

The Ecology Waste 2 Resources Program (W2R) and EAP saw the background study as an opportunity to obtain data on Persistent Bioaccumulative Toxins (PBTs) that have either not previously been analyzed in local fish populations or are a current focus of efforts to reduce or eliminate toxic threats. W2R and EAP selected the following PBTs for analysis, based on bioaccumulation factors and Chemical Action Plan schedules shown at www.ecy.wa.gov/programs/swfa/pbt/.

- Hexabromocyclododecane (HBCDD)
- Tetrabromobisphenol A (TBBPA)
- Polychlorinated naphthalenes (PCNs)
- Short-chain chlorinated paraffins (SCCPs)
- Perfluorinated organic compounds (PFCs)
- Polybrominated diphenylethers (PBDEs)
- Cadmium (Cd) and lead (Pb)

The objective of this effort will be to assess the occurrence of these contaminants in fish from selected urban/industrial waterbodies where potential for detection is thought to be greatest. Four fillet and four whole body samples will be obtained from four waterbodies with a history of toxics issues: Lake Washington, Lower Columbia River, Yakima River, and Lake Spokane.

The species of interest will be common carp (fillets) and largescale suckers (whole body), both of which have wide distribution in Washington lakes and rivers. As a result of their bottom-feeding habit and lipid (fat) content, carp and suckers often have higher PBT levels than other species and thus may be considered worst-case samples. As in the pesticide study, each sample will consist of a composite of five individual fish, to the extent possible.

Measurement Procedures

Organic compounds and percent lipids will be analyzed by the same laboratory contracted for the pesticide work, AXYS Analytical Services in Sidney, BC. Cadmium and lead will be analyzed by the Ecology Manchester Laboratory. Table 1 shows the methods to be used.

Table 1. Analytical Methods

Analysis	Method	Reference
HBCDD	LC-MS/MS	MLA-070 in house method
TBBPA	LC-MS/MS	MLA-079 in house method
PCNs	HRGC/HRMS	MLA-030 in house method
SCCPs	GC/MS	MLA-020 r01 in house method
PFCs	LC-MS/MS	MLA-043 in house method
PBDEs	HRGC/HRMS	MLA-033 EPA Method 1614
Percent lipids	gravimetric	Solvent extraction
Cd and Pb	ICP/MS	EPA 200.8

LC-MS/MS: liquid chromatography-mass spectrometry/mass spectrometry

HRGC/HRMS: high resolution gas chromatography/high resolution mass spectrometry

ICP/MS: inductively coupled plasma/mass spectrometry

AXYS holds accreditation with the Canadian Association for Laboratory Accreditation (CALA) for PBDEs and PFCs by HRMS in tissue. Reciprocity exists between CALA and the Ecology Laboratory Accreditation Program.

No laboratories are currently accredited to analyze HBCDD, TBBPA, PCNs, or SCCPs. (AXYS' application for accreditation from CALA for HBCDD and TBBPA in serum is currently in progress.) AXYS was the only laboratory that responded to Ecology's request for qualifications and quote. A waiver to contract with AXYS for HBCDD, TBBPA, PCNs, and SCCPs has been obtained from the Ecology Quality Assurance Officer.

The detection and reporting limits that AXYS and Manchester Laboratory anticipate achieving in the fish samples for this project are listed in Table 2.

Table 2. Anticipated Detection and Reporting Limits

Analysis	Detection Limit
HBCDD	0.25 ug/Kg reporting limit based on low calibration standard
TBBPA	2.0 ug/Kg reporting limit based on low calibration standard
PCNs	0.10 ng/Kg sample detection limit
SCCPs	0.1 ug/Kg method detection limit (tentative)
PFCs	2.5-5.0 ug/Kg reporting limit based on low calibration standard
PBDEs	1-2 ng/Kg sample detection limit
Cd and Pb	0.10 mg/Kg reporting limit
Percent lipids	0.1%

mg/Kg: parts per million; ug/Kg: parts per billion; ng/Kg: parts per trillion

Table 3 has an estimate of laboratory costs for this part of the project. The cost includes Manchester Laboratory's 25% surcharge for contracting and data review for the organic chemicals.

Table 3. Laboratory Cost Estimate

Analysis	Number of Samples*	Cost per Sample			Cost Totals
		Analysis	Electronic Deliverables	Data Package	
AXYS Laboratory					
HBCDD	9	475	15	35	4,725
TBBPA	9	352	15	35	3,591
PCNs	9	925	15	35	4,480
SCCPs	9	600	15	35	5,850
PFCs	9	425	15	35	4,275
PBDEs	9	825	15	35	7,875
Percent lipids	9	30	15	35	720
				Subtotal	31,516
				25% surcharge	7,879
				AXYS total	39,395
Manchester Laboratory					
Cd and Pb	9	68	NA	NA	612
				Project Total	\$40,007

*Four fillets, four whole fish, and one duplicate (split) sample. One additional duplicate is free of charge.

Quality Control Procedures

One fillet and one whole fish sample will be analyzed in duplicate to obtain estimates of analytical variability. Refer to the QAPP for other QC procedures to be followed.

Data Analysis

A literature search will be conducted to assess the occurrence of these chemicals in fish from waterbodies outside of Washington. The potential for risks to human health, aquatic life, and wildlife will be evaluated, to the extent that this type of information can be located. Refer to the QAPP for other aspects of data analysis.

Reports

Table 4. Revised schedule for completing field and laboratory work, data entry into EIM, and reports.

Field and laboratory work		Due date	Lead staff
Field work completed		July – Oct 2011	Michael Friese
Laboratory analyses completed		January 2012	
Environmental Information System (EIM) database			
EIM user study ID		AJOH0065	
Product		Due date	Lead staff
EIM data loaded		June 2012	Michael Friese
EIM quality assurance		June 2012	Michael Friese
EIM complete		June 2012	Michael Friese
Final report			
Author lead / support staff		Art Johnson / Michael Friese	
Schedule			
Draft due to supervisor		April 2012	
Draft due to client/peer reviewer		May 2012	
Final (all reviews done) due to publications coordinator		June 30, 2012	
Final report due on web		July 31, 2012	

References

Johnson, A., 2011. Quality Assurance Project Plan: Analyzing Chlorinated Pesticide Residues in Fish from Washington Background Lakes. Washington State Department of Ecology, Olympia, WA. Publication No. 11-03-108. www.ecy.wa.gov/biblio/1103108.html