QUALITY ASSURANCE PROJECT PLAN (QAPP)
For
CHEMICAL CONCENTRATION ASSESSMENT IN TILAPIA HARVESTED FROM THE SALTON SEA, CALIFORNIA

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Project Management

AI Project Title Tilapia Tissue Properties Project

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Project Manager / Quality Assurance for DFG: D. Crane

QA Manager: Ellis Shue – Michelson Laboratory

QA Coordinator: Dr. Barry Gump - Salton Sea Authority

Salton Sea Science Office Project Officer: Dr. Doug Barnum

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A4 PROJECT / TASK ORGANIZATION

This section of the QAPP discusses the roles and responsibilities of the Project Managers and Quality Assurance Officers. The organization chart (Fig.1) shows the relationship amongst all project participants.

Project Manager for the Center for Inland Waters: Dr. Stuart Hurlbert

Dr. S. Hurlbert will manage and have overall technical oversight of the project. He will be responsible for design and planning of the monitoring program including selection and scheduling field collection of samples, selection of chemical analyses for each sample, and selection of statistical analyses. He will be responsible for overseeing coordination of program planning, sample collection and distribution, and sample analyses between the Department of Fish and Game's (DFG) Water Pollution Control Laboratory (WPCL) and Michelson Laboratories. He will be responsible for the overall quality of the data and project activities and will consult with the Salton Sea Science Office Project Officer concerning changes in site selection, sampling and analysis. Dr. S. Hurlbert will receive the project data and write a technical report analyzing the data and describing the study results.

Quality Assurance Officer for the Salton Sea Authority: Dr. Barry Gump

Dr. Barry Gump will be responsible for reviewing the technical reports for the Salton Sea Authority. This will include reviewing project data and statistical analyses. He will be responsible for ensuring that project activities and the technical report meet the requirements of the QAPP.

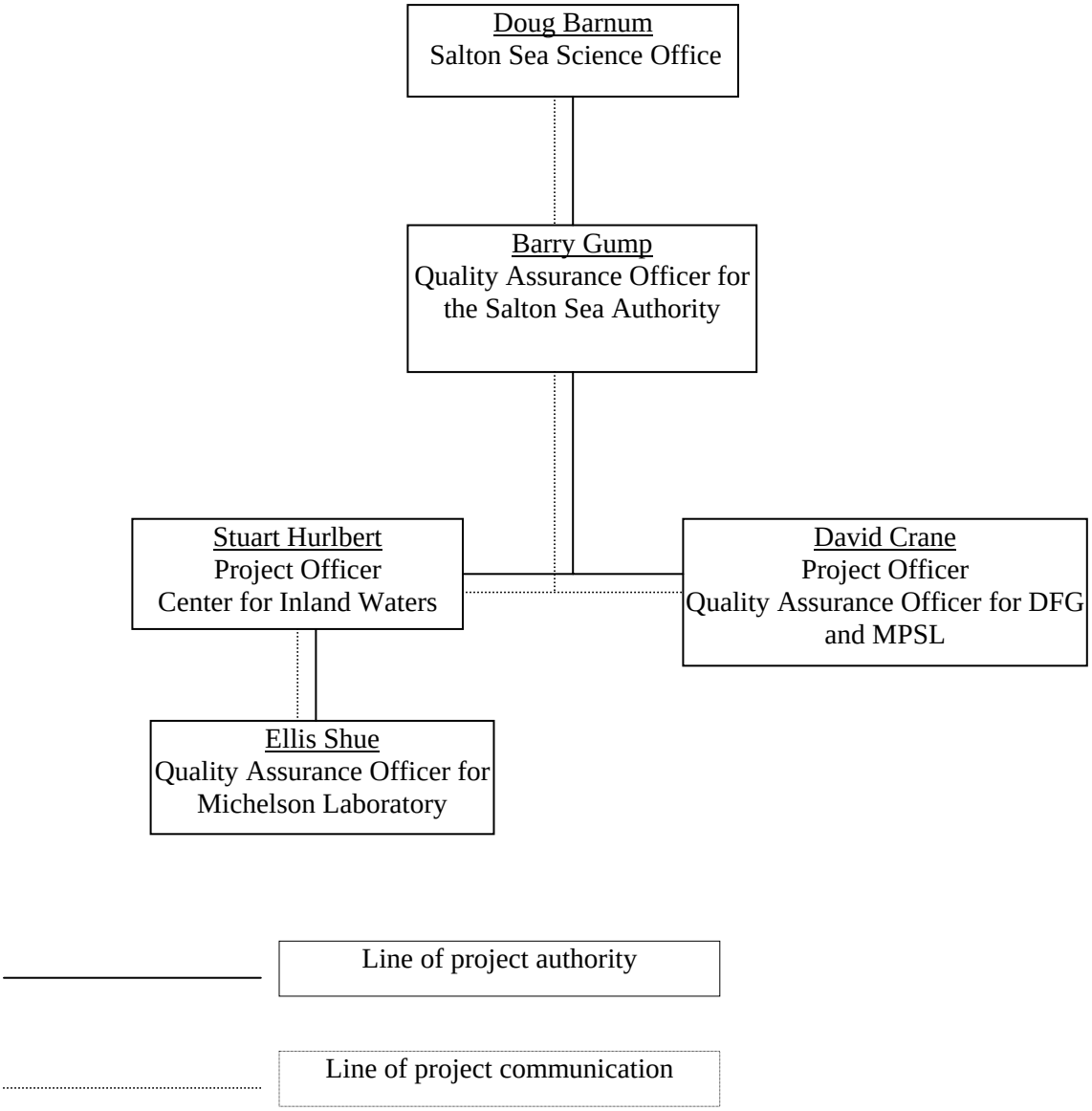
Project Manager/Quality Assurance Officer for DFG: David B. Crane

Dr. Crane manages the Water Pollution Control Laboratory (WPCL) at Rancho Cordova for DFG. Mr. Crane will be responsible for directing and assigning work tasks to DFG staff who will prepare samples for analysis, perform selected chemical analyses of metals and organic compounds, and provide overall database management. He will be responsible for coordinating preservation, logging and transportation of samples between WPCL, Marine Pollution Studies Laboratories at Moss Landing Marine Laboratories and Michelson Laboratories. Mr. Crane will transmit a database and summary report of all results to the Project managers. He will also act as the Quality Assurance Officer for DFG and MPSL in order to insure that DFG and MPSL staff implement the specifications and requirements of the QAPP.

Quality Assurance Officer for Michelson Laboratory

Ellis Shue will be responsible for reviewing all aspects of the work done by Michelson Laboratory and assuring that all specifications and requirements of the QAPP are implemented.

FIGURE 1: PROJECT ORGANIZATION CHART



A5 PROBLEM DEFINITION / BACKGROUND

Fish-based commodities such as fish meal and fish emulsion fertilizers manufactured from the tilapia (*Oreochromis mossambicus*: Cichlidae) stock present at the Salton Sea have been proposed (Hurlbert et al., 1998, Hurlbert, 2000). Besides being potentially an economically profitable enterprise, the harvesting of fish could in the long term ameliorate the eutrophication problem of the Sea (Hurlbert, 2000). Various investigators have analyzed elemental and/or organic contaminant concentrations in tilapia filets, but recent and thorough contaminant analyses in whole tilapia are lacking.

Two factors of prime importance when considering the feasibility and viability of such economic venture are the nutritional and / or qualitative characteristics as well as the contaminant concentrations of the final product. Qualitatively and nutritionally, the final product should meet or potentially exceed the quality of similar products currently on the market. With regards to contaminants, organic and inorganic toxicant concentrations in the manufactured goods should not surpass levels established to protect consumers of these resources from potential toxic effects.

The Tilapia Tissue Properties Project will provide data enabling evaluation of the feasibility of such venture, as well as providing a comprehensive assessment of elemental and organic contaminants in the Salton Sea tilapia.

A6 PROJECT / TASK DESCRIPTION AND SCHEDULE

Tilapia(*Oreochromis mossambicus*: Cichlidae) will be collected from the Salton Sea using gillnets at five fixed stations in early December and late March (corresponding to the post- and pre- spawning periods , i.e. low and high points of their lipid content). The five stations are in 7m of water or less, and are geographically dispersed to represent the north, south, east, west and central parts of the lake. As soon as the fish are removed form the nets, they will be placed on ice in labeled coolers. The freezing protocols of the fish specimens, from the field to the laboratories, is addressed in Section B2.

After each collection date, the Center for Inland Waters will send overnight the frozen specimens on dry ice to the DFG Fish and Wildlife Water Pollution Control Laboratory (WPCL) where the fish will be homogenized and the pesticide/herbicide concentrations determined. WPCL will send frozen subsamples of the fish homogenates to the Marine Pollution Studies Laboratory (MPSL) for the elemental analyses. Sample extraction, cleanup and partitioning methods for pesticide analyses were developed and validated by the Water Pollution Control Laboratory (WPCL SOP# SO-TISS Rev. 5). WPCL will analyze the homogenates and transmit the raw data and summary results in the form of a report to the Center for Inland Waters. This report will also describe sample homogenization, preparation, instrument analysis, sample results and quality control sample results. The Center for Inland Waters will summarize the DFG analytical data

and write a report which will be submitted to the Salton Sea Science Office 45 days after each fish collection.

The Center for Inland Waters will also subcontract with Michelson Laboratory for the analysis of the nutritional properties of the fish homogenates. The methods to be used for the nutritional properties are those approved by the AOAC (Association of Official Analytical Chemists), and EPA. The methods used to determine the fish tissue characteristics are presented in Table 4 (Section B4). Michelson Laboratory will analyze the homogenates and transmit the raw data and summary results in the form of a report to the Center for Inland Waters. This report will also include sample preparation, instrument analysis, sample results and quality control sample results.

The Center for Inland Waters will submit a total of 4 reports to the Salton Sea Science Office:

- an initial report summarizing the existing data from published as well as unpublished studies.
- a progress report interpreting the laboratories' analyses and findings for each of the collection dates.
- a final report summarizing and interpreting all information obtained during the project.

A7 QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT OF DATA

Measurement quality objectives (MQOs) for the various chemicals to be analyzed in the Tilapia Tissue Properties Project are presented in Table 1. The MQOs were established by obtaining estimates of the most likely data quality that is achievable based on either the instrument manufacturer's specifications, scientific experience or historical data. The detection limits are compared to the ones requested in the project RFP.

The MQOs presented in Table 1 are used as quality control criteria in laboratory measurement processes to set the bounds of acceptable measurement errors.

TABLE 1: CHEMICALS AND NUTRITIONAL CHARACTERISTICS TO BE ANALYZED AND MEASUREMENT QUALITY OBJECTIVES FOR THE TILAPIA TISSUE PROPERTIES PROJECT.

	Fish homogenate reporting limit		
ANALYTE	**Specified in RFP**	**Offered by the laboratories**	**Responsible Laboratory [a]**
INORGANIC (μg/g wet weight)			
Arsenic	0.2	0.02	MPSL
Lead	1.0	0.002	MPSL
Mercury	0.1	0.02	MPSL
Selenium	0.6	0.02	MPSL
ORGANICS (ng/g wet weight)			
DDT	5.0	5.00	WPCL
DDD	5.0	2.0	WPCL
DDE	5.0	2.0	WPCL
Toxaphene	200	50.00	WPCL
Kelthane	50	TBD[c]	WPCL
Benzene hexachloride isomers (BHC)	5.0	2.0	WPCL
Aldrin	5.0	1.0	WPCL
Dieldrin	5.0	2.0	WPCL
Endosulfan I	1.0	2.0	WPCL
Endosulfan II	1.0	10.00	WPCL
Endosulfan sulfate	1.0	10.00	WPCL
Endrin	5.0	2.0	WPCL
Heptachlor	5.0	2.0	WPCL
Heptachlor epoxide	5.0	1.0	WPCL
Hexachlorobenzene	5.0	0.3	WPCL
PCBs, total	50.0	50.00	WPCL
Dacthal (DCPA)	5.0	2.0	WPCL
Chlordane	5.0	1.0	WPCL

TABLE 1: Continuation

OTHER (Percent of wet weight, or g/100g)			
Moisture	0.5	0.5	WPCL
Lipid	0.5	0.5	WPCL
Crude Protein, minimum	0.5	0.5	ML
Crude Fat, minimum	0.5	0.5	ML
Crude Fiber, minimum	0.5	0.2	ML
Ash, maximum	0.5	0.1	ML
Calcium, maximum	0.5	0.25[b]	ML
Nitrogen, minimum	0.5	0.1	ML
Phosphorus, minimum	0.5	0.05	ML
Sodium, maximum	0.5	2.5[b]	ML
Potassium	Not specified	2.5[b]	ML
Salt (NaCl), maximum	0.5	0.05	ML

[a] WPCL = Water Pollution Control Lab, MPSL = Marine Pollution Studies Laboratory, ML = Michelson Laboratory
[b] These values are mg/100g
[c] TBD: To be determined.

Accuracy, precision and completeness requirements

Collectively, accuracy and precision can provide an estimate of the total error or uncertainty associated with an individual measured value. Measurement quality objectives for the various indicators are expressed separately as accuracy (i.e. bias) and precision requirements (Table 2). In order to evaluate the MQOs for accuracy and precision, various QA/QC samples will be collected and analyzed for most data collection activities. The different QA/QC procedures required for the complex analyses of chemical contaminants and nutritive properties in tissue samples are presented and discussed separately in Sections B5 and B7 along with a presentation of warning and control limits for the various chemistry QC sample types.

Completeness is defined as “a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under correct, normal conditions” (U.S. EPA, 1997). A completeness goal of 95% has been established for the tests that will be performed during the Tilapia Tissue Properties Project (Table 2). This goal is established in an attempt to provide a comprehensive set of data for each site evaluated for chemical contaminant concentrations in the fish homogenates. Failure to achieve this goal usually results from lost or destroyed samples. Therefore, measures to track samples during shipment and laboratory

processing will be followed to minimize data loss following successful sample collection (see Table 4, Section B3).

TABLE 2: MEASUREMENT QUALITY OBJECTIVES FOR THE TILAPIA TISSUE PROPERTIES PROJECT INDICATORS

Indicator/Data Type	Accuracy Requirement	Completeness Requirement	Precision Goal
Contaminant analyses			
Organics[WPCL]	30%	95%	30%
Trace elements[MPCL] As, Pb, Hg, Se	15%	95%	30%
Nutritional analyses[ML]	20%	95%	20%

Accuracy requirements are expressed as either maximum allowable percent deviation (%) or absolute difference (± value) from the "true" value.

Precision requirements are expressed as maximum allowable relative percent difference (RPD) or relative standard deviation (RSD) between two or more replicate measurements.

Completeness goals are the percentage of expected results to be obtained successfully.

WPCL: analyses done by Water Pollution Control Laboratory

MPSL: analyses done by Marine Pollution Studies Laboratory

ML: analyses done by Michelson Laboratories

A8 SPECIAL TRAINING REQUIREMENTS/CERTIFICATION

The WPCL and Michelson laboratories supervisors will serve as the point of contact for the program QA staff in identifying and resolving issues related to data quality including orienting staff to the QAPP requirements of the Tilapia Tissue Properties Project. To ensure that the samples are analyzed in a consistent manner throughout the duration of the project, key laboratory personnel will participate in an orientation session conducted during an initial site visit or via communications with the project staff. The purpose of the orientation session is to familiarize key laboratory personnel with the QAPP and the QA/QC program. Meetings shall be held with the laboratory at regular intervals to continually review QA/QC procedures, and to revise/update the QAPP.

WPCL and Michelson Laboratory personnel will be well versed in good laboratory practices, including standard safety procedures. It is the responsibility of the particular analytical component project officer, laboratory manager and/or supervisor to ensure that safety training is mandatory for all laboratory personnel. Each laboratory is responsible for maintaining a current safety manual in compliance with the Occupational Safety and Health Administration (OSHA), or equivalent state or local regulations. The safety manual will be readily available to laboratory personnel. Proper procedures for safe storage, handling and disposal of chemicals will be followed at all times; each chemical will be treated as a potential health hazard and good laboratory practices will be implemented accordingly.

Proper training of field personnel represents a critical aspect of quality control. To ensure comparability in data collection, the same team of field biologists will perform the fish collection during the two sampling dates. The lead field biologist is required to have formal training in boat handling and gillnetting.
The staff assigned to the various phases of this project all have demonstrated expertise in the technical requirements needed to fulfill their respective tasks.

A9 DOCUMENTATION AND RECORDS

The field operations supervisor will be responsible for recording data in a waterproof field logbook. The field logbook will include:

- date and time of start of sampling
- name of personnel
- equipment
- location of station (latitude & longitude)
- station description
- field observations (weather, water conditions, species by-catch)
- effort required for sample collection (hours)
- sex, weight, length and condition of the fish (e.g. physical abnormalities)
- problems encountered if any

Laboratory documentation will be entered into either bound laboratory notebooks or established data forms using permanent ink. Staff who record, verify or review data will sign or initial the project records. Record corrections will be made by drawing a single line through erroneous information, adding and explaining correct information, and dating and signing or initialing the correction. Project identifier numbers will be used with manual or computerized logs of data for the Tilapia Tissue Properties Project.

Raw data and records generated by or in association with the Tilapia Tissue Properties Project will be retained in a systematic form. This will include technical plans, QAPP plans, field and laboratory protocols and procedures, data (raw and final), computations and methods, communications involving changes in the project, and final reports. These records will be retained for the duration of project and filed for future retrieval. These documents will exist as hard copy and electronic documents. Hard copies of the relevant forms will be kept with the samples as well as collected in a central binder.

The data will be reported in two progress reports and a final report (see Section C2), which will exist as electronic and hard copies. These documents will be submitted to the Salton Sea Authority and the Salton Sea Science Office in a timely fashion as per contract specifications.

B MEASUREMENT/DATA ACQUISITION

This section describes the proposed sampling design for the Tilapia Tissue Properties Project as well as techniques for collection, processing and analysis of samples.

B1 SAMPLING/EXPERIMENTAL DESIGN

The five stations(FC 1-5)are in 7m of water or less, and are geographically dispersed to represent the north, south, east, west and central parts of the lake.
Their coordinates are as follow:
FC-1 116º 03.00 lat. 33º 30.128 long.
FC-2 115º 37.4 lat. 33º 12.9 long.
FC-3 115º 51.351 lat. 33º 27.002 long.
FC-4 115º 56.00 lat. 33º 19.50 long.
FC-5 115º 47.9 lat. 33º 18.01 long.
The sampling dates will be before (early spring) and after (late fall) the reproductive period.

B2 SAMPLING METHOD REQUIREMENTS

Fish collection will be done using one gillnet at each station. Each gillnet covers an area of 99 m^2 (2.4m width X 41m length). To minimize the entanglement of unwanted species, the mesh size is 50mm X 50mm (100mm stretch mesh). Gillnets will be left in the water for a minimum of one hour, and reset if the needed sample has not been obtained after the first effort.
A total of 8 tilapia will be collected at each station on two sampling dates. Six males and 2 females (the observed sex ratio during preliminary investigations), each measuring 25 to 35cm in total length, will be carefully removed from the nets at each of the stations, and immediately placed on ice in labeled coolers. Only undamaged specimens will be kept for analyses. By-catch will be either returned to the lake if they are still alive or buried in a designated area if moribund or dead. Upon return to the Salton Sea Research facility, the collected tilapia will be sexed, measured (total body length: from the anterior part of the fish to the tip of the caudal fin), weighted, doubly wrapped in heavy duty aluminum foil, packed in labeled freezer bags and immediately placed on dry ice for temporary storage. Upon return to the Biology Department at San Diego State University, California, the bagged fish will be kept in a freezer at –15^{o}C until their shipment on dry ice to the CDFG analytical laboratories. Overnight shipment will take place within 48 hours of collection. Once in the analytical laboratory, the fish will be placed in a freezer until preparation for analyses.
The fish will be allowed to thaw before being homogenized. The 8 tilapia per station will be composited into a single sample. The homogenization operation will be done according to the WPCL SOP#PREP-F, “Collection and Preparation of Fish for Trace Metal and Synthetic Organic Analysis”. Following standard procedures, 250 g

subsamples of the homogenate will be frozen and shipped on dry ice to the cooperating laboratories, the Marine Pollution Studies Laboratory at Moss Landing for elemental analyses and Michelson Laboratories in Commerce, California, for nutritive properties.

Samples to be analyzed for pesticides, herbicides, non-co-planar PCBs, and trace metals will be weighed immediately after homogenization. Samples weighed for analysis of pesticides, herbicides, and non-co-planar PCBs, will be extracted immediately. Sample aliquots will be re-frozen until the digestions for elemental analyses are scheduled. Samples will be thawed and weighed into digestion tubes or ashing beakers for trace element analysis.

Each composite sample will generate approximately 6 kg of homogenate. This is more than needed for the analyses to be carried in this project. About 500g of each homogenate will be retained in frozen storage at the WPCL facility for the eventuality that additional analyses need to be carried later on.
Sample storage temperatures and holding times are described in Table 3 of this report.

TABLE 3: SUMMARY OF CHEMISTRY SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIME CONDITIONS TO BE FOLLOWED FOR THE TILAPIA TISSUE PROPERTIES PROJECT.

Sample/ Parameter	Container	Preservation	Holding Time*	Extract/ digest Holding Time
Whole fish				
Field collection	Coolers	Ice ($0^{o}C$)	1.5 hours maximum (from nets to shore)	NA
field transit	Double wrapped in aluminum	dry ice (-15°C)	48 hours	NA
in lab	Double wrapped in aluminum	Freeze(-20°C)	6 months	NA
homogenate	glass vials	refrigerate (4°C)	1 year	as soon as possible (WPCL)
pesticide/ herbicides/ non-coplanar PCBs			60 days	as soon as possible (WPCL)
trace metals except HG			6 months	6 months (MPSL)
Hg			6 months	28 days (MPSL)
Nutritive properties	Plastic vials	Freeze ($-20^{o}C$)	6 months	As soon as possible (ML)

*No EPA criteria exists for holding times of tissue samples. This is a maximum suggested holding time.
NA: not applicable
WPCL: analyses done by Water Pollution Control Laboratory
MPSL: analyses done by Marine Pollution Studies Laboratory
ML: analyses done by Michelson Laboratories

B3 SAMPLE HANDLING AND CUSTODY REQUIREMENTS

Samples will be assigned a Field and Laboratory ID number to track them from the sampling sites through laboratory analyses and into the computer database.
Data will be recorded on field note sheets, and progress toward analyses completion will be checked off on a routine sheet that will accompany each samples (Table 4). Routine sheets will provide a standardized format to ensure consistent data recording.
A Chain-of-Custody form completed in indelible ink will accompany every sample shipping container (e.g., ice chest) used. Each person releasing a sample will sign and date the form. The receiver will also sign and date the form and retain a copy for their records. Chain-of-custody documents are maintained for each station. Each form will be a record of all samples taken for each station. Station numbers, station names, and collection dates will be included on each sheet. Additional information on the form will include: chemical analysis requested; sample processing requested; type of ice used.

TABLE 4: ROUTINE SHEET EXAMPLE

Date: Sample no: FC-
Field collection supervised by:
Station Location: Sample sent to WPCL:

Fish ID number (*)	Sex	Weight (g)	Length (mm)	Condition of fish	Wrapped/ Frozen (√)
FC# -1					
FC# -2					
FC# -3					
FC# -4					
FC# -5					
FC# -6					
FC# -7					
FC# -8					

Laboratory:	WPCL		
Date sample received:		By (name)	Date samples sent to: -MPSL: -ML(**):
Homogenization:	Date:	By:	Lab ID#:
Pesticide analyses:	Date:	By:	
QA/QC:	Date:	By:	Lab ID# of sample(s):

Laboratory:	MPSL		
Date sample received:		By (name)	
Elemental analyses:	Date:	By:	Lab ID#:
QA/QC:	Date:	By:	Lab ID# of sample(s):

Laboratory:	ML		
Date sample received:		By (name)	
Elemental/nutr. analyses:	Date:	By:	Lab ID#:
QA/QC:	Date:	By:	Lab ID# of sample(s):

(*): Refers to station designation for field ID

(**): ML = Michelson Laboratories

B4 ANALYTICAL METHODS REQUIREMENTS

The Tilapia Tissue Properties Project measures a variety of organic and inorganic contaminants in fish tissue samples, as well as nutritive qualities (Table 1). Analytical method requirements include the following laboratory procedures, manuals and logs. The general methodology for chemical analysis will be to thaw samples prior to analyses. Fish from the same site will be homogenized using a commercial meat grinder and Büchi homogenizer. The 5 sets of homogenized tilapia will then be scheduled for analyses by the laboratory technician.

Homogenates will be digested (metals) or extracted (organics) as appropriate. Subsamples for metals detection will be digested using a 4:1 nitric:perchloric acid. Analyses of Se, As, and Pb will be done using ICP-MS, while Hg analysis will be conducted using either FIMS or LLMDS, depending on the level of Hg in the samples. Subsamples for organic pesticides / herbicides will be extracted and analyzed using gas chromatography utilizing an electron capture or other appropriate detector. Extraction methods employed were developed and validated by the Water Pollution Control Laboratory (WPCL SOP# SO-TISS Rev. 5). Extract cleanup and partitioning methods are modifications of the multi-residue methods for fatty and non-fatty foods described in the U.S. Food and Drug Administration, Pesticide Analytical Manual, Vol. 1, 3rd Edition 1994, Chapter 3, Multi-residue Methods, Section 303-C1.

Methods to be used for analysis of the nutritional properties are those approved by the AOAC (Association of Official Analytical Chemists)(Table 5). These are the Official Methods of analysis of AOAC International.

TABLE 5: METHODS USED IN NUTRITIVE PROPERTIES ANALYSES

Test	Method
Crude Protein	AOAC 928.08
Crude Fat	AOAC 963.15
Crude Fiber	AOCS Ba 6-84
Ash	AOAC 923.03
Calcium	EPA 200.7
Nitrogen	AOAC 928.08
Phosphorus	AOAC 962.02
Sodium	EPA 200.7
Potassium	EPA 200.7
Salt (NaCl)	AOAC 935.47

Instrument performance information, such as baseline noise, calibration standard response, analytical precision and bias data, detection limits, etc., will be recorded in laboratory logbooks.

B5 QUALITY CONTROL REQUIREMENTS

This section presents an overview of QC protocols and requirements covering a range of activities, from sample collection and laboratory analysis to final validation of the resultant data for the Tilapia Tissue Properties Project.

In the field, the field operation supervisor will be present on the boat during the retrieval of the nets. He/she will also be responsible for recording relevant information in the log notebook, for measuring/weighting and sexing the collected fish. He/she will also be responsible for ensuring that the fish are properly wrapped/bagged and placed on dry ice in a timely fashion. Upon return to the laboratory at san Diego State University, California, the field operation supervisor will be responsible for placing the fish in a –15^{o}C freezer. He/she will also make arrangements for the shipping on dry ice to WPCL.

The QC measures include the tracking of accuracy and precision as performance indices, instrument calibration verification, the dilution of samples which exceed the instrument's calibrated range and the documentation of surrogate recoveries. Instrumental calibration is verified with continuing calibration check (CCC) solutions every 10-16 hours. The stability of all analyte calibrations are monitored through the analysis of mid—level standards on 16-20 hour intervals.
All surrogates are inspected for acceptable recoveries during sample analysis. Samples with recoveries outside the range of 50% - 150% are subjected to re-analysis or re-extraction. Marginal recoveries which are in control yet exceed the range of 60% -

120% are closely inspected and corrective action is taken as appropriate. Target analyte concentrations are corrected for surrogate recovery.

The results of the QA/QC samples will be reviewed by laboratory personnel immediately following the analysis of each sample batch. These results then will be used to determine when warning and control limit criteria have not been met and corrective actions will be taken, before processing a subsequent sample batch. When warning limit criteria have not been met, the laboratory is not obligated to halt analyses, but the analyst(s) is advised to investigate the cause of the exceedance. When control limit criteria are not met, specific corrective actions are required before the analyses may proceed.

To demonstrate and monitor statistical control of a measurement process, control charts are used. A control chart basically is a sequential plot of some sample attribute (measured value or statistic). The type of control chart used primarily by laboratory analysts is a “property” chart of individual measurements (termed an X chart). Measured values are plotted in their sequence of measurement. Three sets of limits are superimposed on the chart: 1) the “central line”, 2) the upper and lower “warning limits”, and 3) the upper and lower “control limits”. Key quality control elements for the Tilapia Tissue Properties Project are summarized in Table 5.
Control charts will be updated by laboratory personnel as soon as possible after a control sample measurement is completed. Based on the result of an individual control sample measurement, the following course of action will be taken (Taylor 1987):

If the measured value of the control sample is within the warning limits as shown in Table 5, all routine sample data since the last acceptable control sample measurement are accepted, and routine sample analyses are continued.

If the measured value of the control sample is outside of the control limits, the analysis is assumed to no longer be in a state of statistical control. All routine sample data analyzed since the last acceptable control sample measurement are suspect. Routine sample analyses are suspended until corrective action is taken. After corrective action, statistical control will be reestablished and demonstrated before sample analyses continue. The re-establishment of statistical control is demonstrated by the result of control sample measurements that are in control. Once statistical control has been demonstrated, all routine samples since the last acceptable control sample measurement are reanalyzed.

If the measured value of a control sample is outside the warning limits as shown in Table 6, but within the control limits, a second control sample is analyzed. If the second control sample measurement is within the warning limits, the analysis is assumed to be in a state of statistical control, and all routine sample data since the last acceptable control sample measurement are accepted, and routine sample analyses are continued. If the second sample measurement is outside the warning limits, it is assumed the analysis is no longer in a state of statistical control. All routine sample data analyzed since the last acceptable control sample measurement are suspect. Routine sample analyses are suspended until corrective action is taken. After corrective action, statistical control will be reestablished and demonstrated before sample analyses continue. The re-establishment of statistical

control is demonstrated by the results of three consecutive sets of control sample measurements that are in control (Taylor 1987). Once statistical control has been demonstrated, all routine samples since the last acceptable control sample measurement are reanalyzed.

Central line, warning limits, and control limits will be evaluated periodically by either the on-site Laboratory QC coordinator or the project QA staff. Central lines, warning limits, and control limits for each analyte and sample type will be redefined based on the results of quality control and quality assessment sample measurements. Current control charts will be available for review and shall be submitted routinely as a component of QA/QC reports to the project officers. Such charts will contain both the points and their associated values.

TABLE 6: KEY QUALITY CONTROL ELEMENTS FOR THE TILAPIA TISSUE PROPERTIES PROJECT CHEMICAL ANALYSES

Category	warning limit criteria	control limit criteria	QA/QC protocols[1]	Laboratory
Pesticides:				
organochlorines	d	a,f,g,h	A,B,C,D	WPCL
organophosphates	d	a, f,g,h	A,B,C,D	WPCL
herbicides	d	a, f,g,h	A,B,C,D	WPCL
non-coplanar PCBs	d	a, f,g,h	A,B,C,D	WPCL
trace metals except Hg	b,d	a,c,e,f,g,h	A,B,C,D	MPSL
Hg	b,d	a,c,e,f,g,h	A,B,C,D	MPSL

MPSL = Marine Pollution Studies Laboratory
WPCL = Water Pollution Control Laboratory

a= Calibration checks using standard solutions within ±15% of initial calibration on average for analytes; not to exceed ±25% for any single analyte.

b= Value for each analyte between 2-3sd of control chart limits for analysis of CRM or laboratory control material (LCM) for precision.

c= Value within 3sd of control chart limits for analysis of CRM or laboratory control material (LCM) for precision.

TABLE 6: Footnotes (cont.)
d= Within ±15% of true value for each analyte for relative accuracy[2]. e= Within ±20% of true value for each analyte for relative accuracy[2]. f= Analytes will be flagged if >QL; no analyte will be acceptable at >3 times MDL. g= Recovery of matrix spikes to be between 50-120% for at least 80% of the analytes. h= RPD of matrix duplicates will be ≤30% for each analyte. A= Perform all QA/QC procedures required by methods, e.g.: calculate method detection limits; analyze accuracy-based material (CRMs or laboratory control materials); calibration using standard solutions.
B= Method blank for every 10 samples or batch of samples or type of matrix.
C= Duplicate sample for every 10 samples or batch of samples or type of matrix.
D= Spiked sample for every 10 samples or batch of samples or type of matrix. Spikes made at 10 times the detection limit or at the analyte level.
1=check specific chemical methods.
2=Absolute accuracy can only be assessed using certified CRMs, hence accuracy may be relative when only "non-certified CRMs are available and used. Relative accuracy is computed by comparing the laboratory's value for each analyte against either end of the range of values (*i.e*., 95% confidence limits) reported by the certifying agency. The laboratory's value will be within ±35% of either the upper or lower 95% confidence interval value. Accuracy control limit criteria only apply for analytes having CRM concentrations 10 times the laboratory's MDL.

Matrix Spike

A laboratory fortified sample matrix (commonly called a matrix spike, or MS) and a laboratory fortified sample matrix duplicate (commonly called a matrix spike duplicate, or MSD) will be both used to evaluate the effect of the sample matrix on the recovery of the compound(s) of interest and to provide an estimate of analytical precision, if authorized and funded for the trace metals, pesticide, herbicide and non-coplanar PCB analyses. The dioxin/furan and coplanar PCB analyses use internal standards. One spiked sample will be run for every ten samples or batch of samples or type of matrix, whichever is more frequent. Each MS/MSD sample is first homogenized and then split into three subsamples. Two of these subsamples are fortified with the matrix spike solution and the third subsample is analyzed as is to provide a background concentration for each analyte of interest. The matrix spike solution will contain all the analytes of interest. The final spiked concentration of each analyte in the sample will be at least 10 times the MDL for that analyte, as previously calculated by the laboratory. Recovery data for the fortified compounds ultimately will provide a basis for determining the prevalence of matrix effects in the sediment samples analyzed during the project. If the percent recovery for any analyte in the MS or MSD is less than the recommended warning limit of 50 percent, the chromatograms and raw data quantification reports will be reviewed. If an explanation for a low percent recovery value is not discovered, the instrument response may be checked using a calibration standard. Low matrix spike recoveries may be a result of matrix interferences and further instrument response checks may not be warranted, especially if the low recovery occurs in both the MS and MSD and the other QC samples in the batch indicate that the analysis was “in control”. An explanation for low percent recovery values for MS/MSD results will be discussed in a cover letter accompanying the data package. Corrective actions taken and verification of acceptable instrument response will be included. Analysis of the MS/MSD also is useful for assessing laboratory precision. The relative percent difference (RPD) between the MS and MSD results will be less than 30 for each analyte of interest (see Table 6). The RPD is calculated as follows:

$$RPD = \frac{|C1 - C2|}{|C1 - C2| / 2} \times 100$$

where: C1 and C2 are the first and second sample results for a given analyte
If the results for any analytes do not meet the RPD < 30% control limit criteria, calculations and instruments will be checked. A repeat analysis may be required to confirm the results. Results which repeatedly fail to meet the control limit criteria indicate poor laboratory precision. In this case, the laboratory is obligated to halt the analysis of samples and eliminate the source of the imprecision before proceeding.

B6 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE REQUIREMENTS

Field operations
All sampling equipment will be made of non-contaminating materials and will be inspected prior to entering the field. Nets will be inspected for holes which will be

mended prior to being used. The boat will be visually checked for safety equipment and damage and will be tested in Mission Bay near San Diego State University prior to being taken into the field for sample collection. All ice chests and measuring devices will be thoroughly clean before and after each use.

Laboratory operations

This section addresses only general laboratory operations. Laboratories providing analytical support for the chemical analyses will have the appropriate facilities to store, prepare, and process samples, and appropriate instrumentation and staff to provide data of the required quality within the time period dictated by the project. Laboratories are expected to conduct operations using good laboratory practices, including:

A program of scheduled maintenance of analytical balances, laboratory equipment and instrumentation.

Routine checking of analytical balances using a set of standard reference weights (ASTM Class 3, NIST Class S-1, or equivalents).

Checking and recording the composition of fresh calibration standards against certified calibration check standards.

Acceptable comparisons are $\leq$ 25 percent difference between the average response factor (RF) from the initial calibration and the RF from the calibration check for pesticides, herbicides and non-coplanar PCBs, 80-120 % recovery for mercury and 90-110 % recovery for other trace elements.

Recording all analytical data in bound (where possible) logbooks, with all entries in ink.

Monitoring and documenting the temperatures of cold storage areas and freezer units once per week.

Verifying the efficiency of fume hoods.

Having a source of reagent water meeting American Society of Testing and Materials (ASTM) Type I specifications (ASTM 1984) available in sufficient quantity to support analytical operations. The conductivity of the reagent water will not exceed 18 mega-ohm at 25 ^{o}C.

Labeling all containers used in the laboratory with date prepared, contents, and initials of the individual who prepared the contents; other information as appropriate.

Dating and storing all chemicals safely upon receipt. Chemical are disposed of properly when the expiration date has expired.

QAPP, SOP's, analytical methods manuals, safety plans readily available to staff.

Laboratories will be able to provide information documenting their ability to conduct the analyses with the required level of data quality. Such information might include results from interlaboratory comparison studies, control charts and summary data of internal QA/QC checks, and results from certified reference material analyses. Laboratories will also be able to provide analytical data and associated QA/QC information in a format and time frame agreed upon with the Tilapia Tissue Properties Project Manager or designee.

B7 INSTRUMENT CALIBRATION AND FREQUENCY

Initial Demonstration of capability and calibration

Equipment will be calibrated prior to the analysis of each sample batch, after each major equipment disruption, and whenever on-going calibration checks do not meet recommended control limit criteria (Table 6). All calibration standards will be traceable to a recognized organization for the preparation and certification of QA/QC materials (*e.g*., National Institute of Standards and Technology, U.S. Environmental Protection Agency, etc.). Calibration curves will be established from a calibration blank and a minimum of three analytical standards of increasing concentration, covering the range of expected sample concentrations. The calibration curve will be well-characterized and will be established prior to the analysis of samples. Only data which results from quantification within the demonstrated working calibration range may be reported by the laboratory (*i.e*., quantification based on extrapolation is not acceptable). Samples outside the calibration range will be diluted and reanalyzed.

Initial Documentation of Method Detection Limits

Analytical chemists have coined a variety of terms to define "limits" of detection; definitions for some of the more commonly-used terms are provided in Keith et al. (1983) and in Keith (1991). The Method Detection Limit (MDL) will be used to define the analytical limit of detection. The MDL represents a quantitative estimate of low-level response detected at the maximum sensitivity of a method. The Code of Federal Regulations (40 CFR Part 136) gives the following rigorous definition: "the MDL is the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte." Confidence in the apparent analyte concentration increases as the analyte signal increases above the MDL.

The analytical laboratory will calculate and report an MDL for trace elements in fish tissue prior to the analysis of field samples (Table 1). The laboratory is required to follow the procedure specified in 40 CFR Part 136 (Federal Register, Oct. 28, 1984) to calculate MDLs for the analytical method employed. The amount of sample used in calculating the MDL will match as closely as possible the matrix of the actual field samples and the amount of sample typically used.

On-Going Demonstration of Capability

Certified Reference Materials (CRMs) generally are considered the most useful QC samples for assessing the accuracy of a given analysis (*i.e*., the closeness of a measurement to the "true" value). Certified Reference Materials can be used to assess accuracy because they have "certified" concentrations of the analytes of interest, as determined through replicate analyses by a reputable certifying agency using two independent measurement techniques for verification. CRMs available for the Tilapia Tissue Properties Project are given in Table 7.

Routine analyses of Certified Reference Materials represent a particularly vital aspect of the "performance-based" QA philosophy. At least one CRM must be analyzed along

with each batch of 25 or fewer trace element samples . For CRMs, concentrations of the target analyte will be known to the analyst(s) and will be used to provide an immediate check on performance before proceeding with a subsequent sample batch. Performance criteria for both precision and accuracy have been established for analysis of CRMs (Table 6). If the laboratory fails to meet either the precision or accuracy control limit criteria for a given analysis of the CRM , the data for the entire batch of samples is suspect. Calculations and instruments will be checked; the CRM may have to be reanalyzed to confirm the results. If the values are still outside the control limits in the repeat analysis, the laboratory is required to find and eliminate the source(s) of the problem and repeat the analysis of that batch of samples until control limits are met, before continuing with further sample processing. The results of the CRM analysis will never be used by the laboratory to "correct" the data for a given sample batch. CRM's for trace organics in fish tissue will be used if they are available.

Precision criteria: Each laboratory is expected to maintain control charts for use by analysts in monitoring the overall precision of the CRM analyses. Upper and lower control chart limits (*e.g*., warning limits and control limits) will be updated annually; control limits based on 99% percent confidence intervals around the mean are recommended. Following the analysis of all samples in a given year, an RSD (relative standard deviation, a.k.a. coefficient of variation) will be calculated for each analyte of interest in the CRM. Based on typical results obtained by experienced analysts, an overall RSD of less than 30% will be considered acceptable precision for each analyte having a CRM concentration >10 times the laboratory's MDL. Failure to meet this goal will result in a thorough review of the laboratory's control charting procedures and analytical methodology to determine if improvements in precision are possible.

Accuracy criteria: The "absolute" accuracy of an analytical method is assessed using CRMs. Based on typical results attained by experienced analysts in the past, accuracy control limit criteria have been established (Table 6). For inorganic analyses, the laboratory's value will be within <20% of either the upper or lower 95% confidence limit for the analyte of interest in the CRM. Due to the inherent variability in analyses near the method detection limit, control limit criteria for relative accuracy only apply to analytes having CRM true values which are >10 times the MDL established by the laboratory.

Continuing Calibration Checks

The initial instrument calibration performed prior to the analysis of each batch of samples is checked through the analysis of calibration check samples (*i.e*., calibration standard solutions) inserted as part of the sample stream. Calibration standard solutions used for the continuing calibration checks will contain all the analytes of interest. At a minimum, analysis of the calibration check solution will occur at the start and at the end of each sample batch. Analysts will use best professional judgment to determine if more frequent calibration checks are necessary or desirable.

If the control limit for analysis of the calibration check standard is not met (Table 6), the initial calibration will have to be repeated. If possible, the samples analyzed before the calibration check sample that failed the control limit criteria will be reanalyzed following

the re-calibration. The laboratory will begin by reanalyzing the last sample analyzed before the calibration standard which failed. If the relative percent difference (RPD) between the results of this reanalysis and the original analysis exceeds 30 percent, the instrument is assumed to have been out of control during the original analysis. If possible, reanalysis of samples will progress in reverse order until it is determined that there is less than 30 RPD between initial and reanalysis results. Only the re-analysis results will be reported by the laboratory. If it is not possible or feasible to perform reanalysis of samples, all earlier data (*i.e*., since the last successful calibration control check) is suspect. In this case, the laboratory will prepare a narrative explanation to accompany the submitted data and data results will be qualified if there is anything out of control.

Laboratory Method Blank

Laboratory method blanks are used to assess laboratory contamination during all stages of sample preparation and analysis. One laboratory method blank will be run in every sample batch. The method blank will be processed through the entire analytical procedure in a manner identical to the samples. Warning and control limits for blanks (Table 6) are based on the laboratory's method detection limits as documented prior to the analysis of samples. A method blank concentration between the MDL and 3 times the MDL for one or more of the analytes of interest will serve as a warning limit requiring further investigation based on the best professional judgment of the analyst(s). A method blank concentration equal to or greater than 3 times the MDL for one or more of the analytes of interest requires corrective action to identify and eliminate the source(s) of contamination before proceeding with sample analysis. All data should be qualified when the method blank is greater than the quantification limit or MDL.

TABLE 7: CERTIFIED REFERENCE AND LABORATORY CONTROL MATERIALS COMMONLY USED

Fish Tissue Matrices:		**Laboratories**
DORM-2	Dogfish Muscle	(MPSL)
SRM 2974	Mussel Tissue	(WPCL)
CARP-1	Fish Tissue	(MPSL)
Standard solutions:		
NIST SRM 2261	pesticides	

MPSL: Marine Pollution Studies Laboratories

WPCL: Water Pollution Control Laboratories

B8 INSPECTION/ACCEPTANCE REQUIREMENTS FOR SUPPLIES AND CONSUMABLES

The senior laboratory assistant, laboratory technician and lead chemist shall inspect supplies and consumables prior to their use in analysis. This inspection includes reviewing records and examination of deliverables. The materials description in the methods of analysis shall be used as a guideline for establishing the acceptance criteria for these materials or when specifically stated in the procurement documents, suppliers must document all requirements and specifications and the senior laboratory assistant will verify that these requirements and specifications were met. Introduction of unwanted compounds into the analytical process shall be monitored by analysis of method blanks. Purity and efficacy of reagents shall be monitored by analysis of laboratory control samples. An inventory and control system for these materials shall assure use before manufacturers' expiration dates and storage under safe and chemically compatible conditions. Certification and documentation of purity and other specification provided by the supplier will be retained.

Standards

Stock standards will be made from purchased concentrates or neat materials. The concentrates will be stored in the dark at 4°C. All standards will be prepared from material of 95% or greater purity. Newly mixed standards will be compared to an archived portion of the original solution. In general, a new standard mix is considered acceptable if the observed concentrations are within 80 - 120% of the original solution. Standards should be traceable back to the original stock solution, and certification and documentation of standards should be retained.

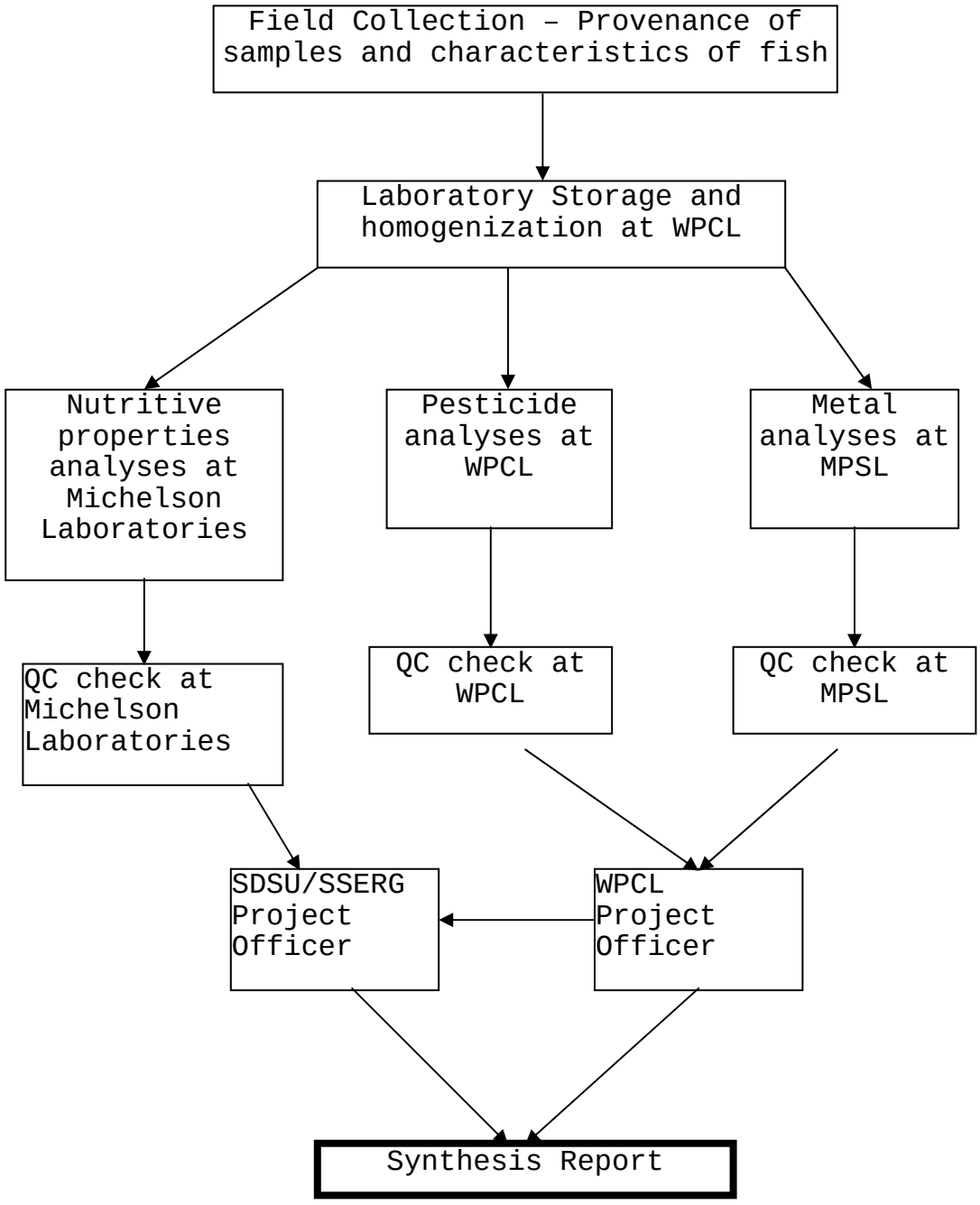
B9 DATA ACQUISITION REQUIREMENTS (NON-DIRECT MEASUREMENTS)

The Tilapia Tissue Properties Project will use historical data on chemical contaminants in tilapia and other sport fish from TSMP reports (1978-1996) as well as from other sources (Saiki, 1990; Setmire et al., 1990; Shroeder et al., 1993; Surico-Bennett, 1999; Vicario-Fisher, 1999). The data obtained from the present project will be compared to the historical data in order to potentially observe trends; this comparison will not be statistically based.

The Tilapia Tissue Properties Project will also use the best professional judgment of local experts to interpret the data.

B10 DATA MANAGEMENT

FIGURE 2: DATA MANAGEMENT PATHWAY



Data will be recorded and stored both electronically and in hard copy format. The hard copies shall be centrally stored as well as being kept with each sample set. Electronic copies will be well annotated and centrally stored. All samples will be stored, moved, and analyzed together by date as a sample set.

Laboratory personnel will verify that the measurement process was “in control” (i.e., all specified QA/QC requirements were met or acceptable deviations explained) for each batch of samples before proceeding with the analysis of a subsequent batch. In addition, each laboratory will establish a system for detecting and eliminating transcription and/or calculation errors prior to reporting data. It is recommended that an individual not involved directly in sample processing be designated as laboratory QA Officer to perform these verification checks independent of day-to-day laboratory operations.

Only data which has met QA requirements, or data which has acceptable deviations explained, will be submitted by the laboratory. When QA requirements have not been met, the samples will be reanalyzed when possible and only the results of the reanalysis will be submitted, provided they are acceptable.

Chain-of-Custody forms (COC) will be furnished to individuals collecting the samples. These forms are completed by field sampling personnel and are signed and accepted by the staff accepting the samples on behalf of the laboratory. The forms contain all pertinent information necessary for the laboratory to process the samples, such as the sample description, exact type and number of tests to run, expected date of submission of deliverable products, and other information specific to the lab/analyses being performed.

C ASSESSMENT/OVERSIGHT

C1 ASSESSMENTS AND RESPONSE ACTIONS

The quality of the project data and reports are assessed within the Tilapia Tissue Properties Project by the various project managers, QA/QC officers and peer reviewers. These assessments will be undertaken by the project officer, Dr. Barnum, the QA/QC project coordinator, Dr. Barry Gump, and the designated QA/QC project manager in the laboratories. Oversight and assessment will be accomplished through the review of project data, reports and peer review.

C2 REPORTS TO MANAGEMENT

WPCL, MPSL and Michelson Laboratories will update the Tilapia Tissue Properties Project Officer Dr. Hurlbert weekly concerning the status of their activities, performance evaluations, and data quality assessments. This will include reporting on quality assurance problems and corrective actions that may compromise the data quality or change the schedule of work for the Tilapia Tissue Properties Project. These reports will be prepared by the WPCL, MPSL and Michelson Laboratories Project Officers and may be verbal or written when requested by the Tilapia Tissue Properties Project Manager (see Section D2).

Two progress reports will be submitted approximately 45 days after each collection date to the Salton Sea Officer, Dr. Doug Barnum. These reports will document the findings from the laboratories for each set of fish. A final report, summarizing and interpreting all the information obtained during the project will be submitted approximately 90 days after the second fish collection.
This report will contain one or two manuscripts suitable for publication in a scientific journal, and appendices providing supplementary information called for in the contract with the Salton Sea Authority.

D DATA VALIDATION AND USABILITY

D1 DATA REVIEW, VALIDATION, AND VERIFICATION REQUIREMENTS

The following criteria will be followed when reviewing and validating the data upon completeness:
1-Consistency in reporting units and number of significant figures
2-all laboratories calculated percent recovery values (calibration check samples, Certified Reference Materials, matrix spikes and relative percent difference values for duplicates) are correct
3-Compare the QA/QC data against established criteria for acceptable performance, as specified earlier in this report
4-check that the reported concentrations for each analyte fall within “environmentally-realistic” ranges, based upon expert judgement and historical data.

D2 VALIDATION AND VERIFICATION METHODS

A quality assurance report will be prepared by the Tilapia Tissue Properties Project Manager/Quality Assurance Officer by each laboratory following completion of sample analysis. This report will summarize the measurement error estimates for the various data types using the QA/QC sample data. Precision, accuracy, comparability, completeness, and representativeness of the data will be addressed in this document. A separate QA report will accompany each major sampling event and will address all QA concerns relevant to the data collected during the sampling event.
In addition to the formal reports described above, the QC Officer will report regularly to the Tilapia Tissue Properties Project Manager on an informal basis, through e-mail, conference calls, and/or direct contact. One of the primary responsibilities of the QC Officer is to keep the Project Manager informed of any issue or problem which might have a negative effect on the data collected.
Validated analyses will be submitted to the Salton Sea Authority and the Salton Sea Science Office by Dr. Stuart Hurlbert through Dr. Barnum in the form of two progress reports and a final synthesis report.

D3 Reconciliation with User Requirements

The data gathered by this investigation will provide comprehensive and up-to-date information on the chemical concentrations as well as nutritive properties of the tilapia stock at the Salton Sea. The concentrations of selenium and arsenic, major elements of concern, will be documented for whole fish, and not limited to filets or edible portions alone. The data on the organic contaminants will be used to describe the present impact of agricultural run-offs on the tilapia, and could be used in models to assess contaminant impacts on the fish and avian communities. In addition to the above, the nutritive properties of whole tilapia will determine the feasibility of exploiting the stock for the manufacture of animal feed and / or fish emulsion fertilizers. The project participants reserve the right to publish these data in the scientific literature. All data gathered by this investigation will also be used to determine areas where further research is needed. The adequacy of the data gather will be determined through individual assessments by the co-principal investigators.

The results of the investigation will conform to the proposal submitted to the Salton Sea Authority and the Salton Sea Science Office in the RFP process. This proposal outlines the objectives and goals of the project as approved by the Salton Sea Authority and the Salton Sea Science Office.

The people involved in sampling, handling and analyzing the data are trained individuals. By working within the context of the aforementioned quality control methods, the data provided in the final reports will be valid, contingent upon the QC guidelines specified above.

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