

Quality Assurance Work Plan
for
NUTRIENT CYCLING IN THE SALTON SEA

Prepared for the
Salton Sea Research Management Committee

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University of California, Riverside: Michael Anderson, Chris Amrhein, Ed Betty, Paul Sternberg, Laska Whiteaker

Salton Sea Authority: Tom Kirk, Barry Gump, John Elder, Milton Friend

A4 PROJECT TASK / ORGANIZATION AND RESPONSIBILITIES

The directors and principal investigators of this project will be Dr. Michael Anderson and Dr. Chris Amrhein. Dr. Anderson will serve as co-director and co-principal investigator and will supervise field and modeling activities. Dr. Amrhein will serve as co-director and co-principal investigator and will supervise laboratory analysis. Laska Whiteaker, Staff Research Associate, will have day-to-day responsibilities for the project, and will oversee and coordinate field and laboratory efforts. Ed Betty, Staff Research Associate, will be in charge of field sampling and measurements. Paul Sternberg, Staff Research Associate, will be in charge of laboratory operations and analysis. Becky Rodrigez, Graduate Student, will be involved in field and laboratory determinations of nutrient flux. Three undergraduate students will assist in field sampling and laboratory analyses.

A5 PROBLEM DEFINITION AND BACKGROUND

The Salton Sea formed near the turn of the century through the failure of a control structure on the lower Colorado River. Diverted from its native channel, the river flowed unimpeded for ~15 months into the Salton Basin, a low-lying geological remnant of the Gulf of California. Since that time, agricultural drainage and other sources of water have been the primary inputs to this arid, closed-basin system.

The Imperial, Coachella and Mexicali Valleys which comprise the watershed of the Sea are richly productive agricultural areas (e.g., the Imperial and Coachella Valleys produce an estimated \$1.3B per year in agricultural commodities). The high rate of evapotranspiration for the basin produces agricultural drainage water significantly higher

in salinity relative to the Colorado River source water. These relatively saline inflows, combined with the effects of prolonged surface evaporation from the Sea (evaporation rate of ~1.7 m/year), have resulted in the highly saline condition presently found in the Sea (salinity ~44 ppt, or about 25% higher than that of ocean water). These high levels of salinity have threatened the viability of the sport fishery there.

In addition to the effects of salinization, other factors threaten the long-term health of the Sea. Nutrients and pesticide residues from surrounding farms and cities flow into the Sea via the three main rivers within the watershed. The high level of nutrients from agricultural drainage and municipal wastewater, combined with elevated temperatures for the area, contribute to the astonishingly high levels of productivity in the Sea. Consequences of this high productivity include nuisance algae blooms, anoxia, production of hydrogen sulfide and other malodorous compounds, and fish kills.

As a result, any effort to save the Sea will require a thorough understanding of the nutrient loading and internal recycling of nutrients. Controlling point and non-point discharges of nutrients into the Sea will be an important step in the preservation of the Sea, but questions remain about the role of internal nutrient cycling and the ultimate fate of nitrogen and phosphorus in the Sea. Specifically, the role of the sediments as a potential source and sink of nutrients to and from the water column needs to be evaluated. Toward that end, a series of studies are outlined which will provide important data on the nutrient dynamics within the Sea, and will specifically quantify the contribution of the sediments to the overall nutrient status of the Sea.

The overall objective of this study is to improve our understanding of the role of the sediments in nutrient cycling within the Salton Sea. The study specifically will:

- i. Quantify the total and available nutrient pools within the sediments of the Salton Sea;
- ii. Quantify, through field investigations, the rate of nutrient release to the overlying water column under ambient water quality conditions;
- iii. Evaluate the rate of particulate-bound nutrient deposition to the sediments;
- iv. Through lab studies, evaluate the release of nutrients under carefully controlled redox conditions, temperature, pH, and other factors;

- v. Develop a distributed-source finite-segment model which quantitatively predicts net nutrient fluxes across the sediment-water interface;
- vi. Develop a nutrient budget for the Sea, specifically quantifying the contribution of sediments to the nutrient load and overall trophic state of the Sea; and
- vii. Quantify the extent of sulfate reduction and sulfate precipitation within the Sea, and evaluate the role of “green rust” in nitrate reduction and sulfate and phosphate removal.

A6 PROJECT / TASK DESCRIPTION

A series of field and laboratory studies will quantify the concentrations, forms and flux of nutrients from sediments in the Salton Sea. The results will then be incorporated into a model that will identify the contribution of the sediments to the overall nutrient budget of the sea. Based upon available sediment (Lipton et al., 1999), water quality (Holdren, 1999) and other data, sediment samples representative of the different types present within the basin will be collected from a series of locations. Grab samples will be collected with Ekman and Ponar samplers, while intact cores will be collected using a Wildco core sampler and with divers. Sediment characterization will include particle size, organic matter content, carbonate content, total N, P and S, and empirical solid-phase speciation of N, P and S using selective extractants. *In situ* pore water samples will be collected using “peepers”. *In situ* flux chambers will be used to measure the rate of release of nutrients from sediments to the overlying water column subject to ambient water quality conditions, while sediment traps will be used to quantify the rate of particulate N and P loss from the water column. In supporting lab studies, the release of N and P from intact cores to the overlying water will be evaluated under carefully controlled redox and temperature conditions.

Laboratory and field results will then be used to develop a mathematical model of sediment nutrient flux. The model will predict N and P release as a function of local chemical and physical conditions. The data collected from the lab and field studies will then be used with available water quality data for the New, Alamo and Whitewater Rivers, the All American and East Highline Canals, and the agricultural tile drains to

construct a nutrient budget for the Sea. The results can be used to predict the nutrient status, transparency, dissolved oxygen, chlorophyll a, and other characteristics of the Sea under different scenarios and management strategies.

The time to completion of the project outlined above is 18 months and is proposed to run from February 1, 1999 – July 31, 2001. The specific timeline for each research objective is provided in Table 1. Barry Gump, the QA Coordinator for the project, will visit the laboratory and field site at least once per year.

A7 QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT DATA

Nutrient concentrations and other chemical and physical properties of water column and sediment samples will be quantified using standard methods. Detection limits and operating ranges for the analytical techniques are provided in Table 2.

Analytical precision will be evaluated by analysis of replicate samples. Duplicates will be analyzed at a frequency of no less than one per 10 samples. Duplicate analyses of field splits will be used to assess the precision of analytical methods. Duplicate analysis of a sample on the same instrument will provide instrumental precision data. The relative percent difference (RPD) of duplicates will be calculated as follows:

$$RPD = (C_1 - C_2) / ((C_1 + C_2) / 2) * 100$$

where C_1 and C_2 are concentrations of analyte in replicate samples 1 and 2. A control limit of 15% will be used for relative percent difference. Frequencies of replicate samples for various laboratory analyses are listed in Table 3.

Accuracy of analytical data will be evaluated by analyzing reference materials and spiked samples. Reference materials will be run with each batch of samples during laboratory analyses. Confidence intervals supplied with reference samples will be used as control limits at the 95% confidence level. The relative percent error (RPE) of standards will be calculated as follows:

$$RPE = (C_1 - C_0) / C_0 * 100$$

where C_1 is the concentration analyzed in the sample and C_0 is the true concentration.

Spiked samples will be used to assess the recovery of various analytes. Spikes will contain analytes at the level present in the sample, or at the concentration of the mid-

range calibration standard, whichever is higher. Spike recovery will be calculated as follows:

$$\% \text{ Recovery} = (A_s - A_o) / S * 100$$

where A_s is the amount of analyte in the spiked sample, A_o is the amount of analyte in a non-spiked sample, and S is the amount of spike added. Control limits of 90 to 110% will be used for percent recovery. Frequencies of spike samples for various laboratory analyses are listed in Table 3.

A8 SPECIAL TRAINING REQUIREMENTS

The research team is well suited for the proposed research. Dr. Anderson and Dr. Amrhein both have considerable teaching and research experience related to the chemistry of waters, sediments and soils.

Dr. Anderson teaches undergraduate courses at U.C. Riverside in limnology and environmental sampling and analysis, and advanced graduate courses in the chemistry of the solid-water interface and environmental organic chemistry. Dr. Anderson has recently completed studies on the fate of CuSO_4 in Lake Mathews (a source drinking water reservoir operated by MWD), the accumulation of Cu in fish (*Micropterus salmoides* and *Cyprinus carpio*), and the relationship between Cu content of sediments and benthic macroinvertebrate populations. The fate study included characterization of the forms of Cu within the sediments, the rate of release from the sediments focusing on chemical control, and overall lake flux and budget calculations (Anderson *et al.*, 1999). Related work has included mineral precipitation and behavior of oxyanions in agricultural evaporation basins (Levy *et al.*, 1994), and development of a hybridized Monte Carlo-finite segment model for probability-based predictions of pathogen concentrations in a reservoir (Anderson *et al.*, 1998). Dr. Anderson will have primary responsibility for field sampling, modeling and budget calculations.

Dr. Amrhein teaches graduate courses in soil chemistry and saline soils and waters and has conducted a number of studies on the chemistry of saline evaporation ponds. Recent published studies include work on mineral precipitation and solubility (e.g., Levy *et al.*, 1994; Amrhein *et al.*, 1993; Amrhein *et al.*, 1985) and the effects of redox on

metal and trace element solubility (Duff *et al.*, 1999; Amrhein *et al.*, 1993). Dr. Amrhein will oversee laboratory activities.

Mr. Ed Betty, SRA III, has extensive experience in the sampling and analysis of surface waters. He has worked on the sampling, analysis and development of a data base on water quality for the Santa Ana River (1988-1991) and lakes within the Los Angeles hydrologic unit (Lund *et al.*, 1994). He has sampled lakes throughout California including alpine lakes in the High Sierra and Lake Elsinore, near Riverside, CA. In addition to his field expertise, he has primary responsibility for GPS data acquisition and analysis and nutrient analysis using the department Technicon autoanalyzer. Mr. Betty will principally be involved in field sampling and field acquisition of data.

Mr. Paul Sternberg, SRA IV, also has considerable laboratory and field experience. He has recently evaluated the root distribution and seasonal water status in weathered granitic bedrock under chaparral (Sternberg *et al.*, 1996), has worked on in situ hydraulic and mineralogic characterization of soils and weathered rock, and is also responsible for operation of an Alpkem autoanalyzer. He is currently also an instructor of introductory geology at Fullerton College. Mr. Sternberg will have primary responsibility for the nutrient analysis of waters using an Alpkem autoanalyzer dedicated to this project. He will also be responsible for maintenance and calibration of the Alpkem and screening for QA and related analytical problems.

Laska Whiteaker, SRA I, will provide day-to-day oversight of the project. She has both academic and recent industry experience in the environmental field. Ms. Whiteaker managed the contract environmental analytical lab at the University of Arizona, and has extensive field sampling, soil and water quality analysis, wetlands delineation and QA/QC experience. She has also received awards for work on selenium and mercury bioaccumulation in the Colorado River Basin from the USEPA and for technical writing from the Air and Waste Management Association.

Becky Rodriguez, graduate student researcher in the Soil and Water Sciences program here at UCR, will assist in field sampling as well as have primary responsibility for column flux experiments.

Laboratory assistants will participate in field and laboratory sampling and analysis and will be certified divers where possible.

Vitae for the principal investigators are included in the Nutrient Cycling in the Salton Sea research proposal submitted to the Salton Sea Authority.

A9 DOCUMENTATION AND RECORDS

Samples will be labeled and recorded in a Field Log at the time of collection. A Chain-of-Custody form will be used to transfer samples collected in the field to laboratory personnel. All Field Log and Chain-of-Custody records will be kept on file for future reference. A Field Log and Chain-of-Custody form are included at the end of this report.

SECTION B: MEASUREMENT / DATA ACQUISITION ELEMENTS

B1 SAMPLING PROCESS DESIGN

Samples used to characterize sediment nutrient levels will be collected in two phases. The initial phase will be conducted using a relatively coarse regular grid of approximately 100 sites within the Salton Sea basin. Using results from the initial phase, as well as other available data, a series of local fine grids within the major sediment zones of the basin will comprise the second sediment sampling phase. Rates of nutrient release from sediments will be determined *in situ* using peepers placed at approximately 30 sites within the major sediment zones of the basin. Benthic flux chambers will be seated in sediments throughout the basin, and used *in situ* to estimate the flux of nitrogen, phosphorous and sulfur. Sediment traps will also be placed throughout the basin to quantify the rate of particulate carbon, nitrogen, phosphorous and sulfur loss from the water column. All field sampling locations will be recorded using a Trimble GeoExplorer II GPS System, and differentially corrected using the base station at the University of California, Riverside.

B2 SAMPLING METHODS REQUIREMENTS

Sediments will initially be characterized from grab samples collected at approximately 100 sites using Ekman or Ponar sediment samplers. The Ekman and Ponar sediment samplers are constructed of stainless steel, and not likely to corrode or interfere with the sediment samples. Sediment samples will be placed in 500-mL Teflon-lined glass jars, stored on ice and transported to the laboratory for analysis. Further sampling for delineation of the major sediment zones will include grab samples and intact core samples collected using a Wildco core sampler and by divers. The Wildco core sampler is fitted with a polycarbonate plastic tube, which is used for sample collection and then capped for transport to the laboratory. Duplicate field-split samples will be collected at 10% of the sites for both grab and core samples.

Peepers are equilibrium diffusion chambers, constructed from plastic vials fitted with a polycarbonate membrane. Peepers will be allowed to equilibrate for 3-6 weeks after placement in the sediments, then be removed and transported to the laboratory for analysis. Water samples will be collected from sealed benthic flux chambers placed in the sediments. Samples will be collected through sampling ports, placed in plastic bottles and transported to the laboratory for analysis. Sediment traps will be retrieved after 24-48 hours, although the sampling time may be adjusted depending on initial results. Particles within the sediment traps will be collected by washing and filtration onto glass fiber filters. All samples will be stored in a cooler on crushed ice on board and en route to the laboratory.

All field sampling locations will be recorded using a Trimble GeoExplorer II GPS System, and differentially corrected using the base station at the University of California, Riverside. This will enable us to return to sampling locations for further delineation.

B3 SAMPLE HANDLING AND CUSTODY REQUIREMENTS

Each sediment and water sample will be assigned a unique identification number. Individual sample identifications will consist of a number, the sampling date, and the latitudinal/longitudinal position recorded using a Trimble GeoExplorer II GPS System.

Identification numbers for field-splits of samples will be appended with a letter “S”.

Sample containers will be labeled at the time of collection. Labels will include the sample number, date, time and the field sampler’s initials. A Field Log will be completed during the collection of each sediment and water sample. Field Log entries will include the site number, date, time, latitude/longitude, depth to sediment, sample number, duplicate number, HydroLab water measurements, weather/site conditions, field observations/comments and the signature of the sampler. A Chain-of-Custody form will be completed for each group of samples collected on the same day, and be used to transfer samples to laboratory personnel. Chain-of-Custody entries will include the project name, field samplers, sample numbers, sample types, number of containers, comments and signatures relinquishing the samples. Samples of the Field Log and Chain-of-Custody forms to be used for this study are included at end of this report.

Field documentation will be completed using indelible ink, with any corrections made by drawing a single line through the error and entering the correct value.

Samples will be delivered to the University of California, Riverside laboratory at the end of each day to initiate processing within 24 hours.

B4 ANALYTICAL METHODS REQUIREMENTS

Standard methods will be used for all routine sediment and water quality analyses. Methodologies used are taken from Methods of Chemical Analysis for Waters and Waste (EPA-600/4-79-020) published by EPA, Standard Methods for the Examination of Water and Wastewater 17th Edition, published by American Public Health Association, American Water Works Association, and Water Pollution Control Federation, and Methods of Soil Analysis 2nd Edition published by American Society of Agronomy and Soil Science Society of America. Applicable methods for sediment analyses are listed in Table 4, while methods for water analyses are listed in Table 5.

Chemical standards will be used to calibrate instruments at the start of each run of samples. Blanks will be analyzed at a frequency of no less than one per 20 samples. Method detection limits, normal operating ranges and calibration methods for various analytes are given in Table 2.

B5 QUALITY CONTROL REQUIREMENTS

Care will be taken to ensure the collection of representative sediment and water samples. Equipment blanks will be used to aid in the identification of problems due to field contamination. Duplicate analyses of field splits will be used to assess the precision of analytical methods. Quality control in the laboratory will include analysis of reagent blanks, method blanks, sample duplicates, certified standards and spikes.

Good laboratory practices will be followed in all aspects of this project. All hoods are checked on a regular schedule by the campus Department of Health and Safety. Hazardous wastes generated during some analyses will be transferred to the Department of Health and Safety for disposal according to campus, state and federal guidelines.

As laboratory analyses are completed, the analyst will check data for quality. Corrective action will be taken by the analyst whenever the RPD of duplicates exceeds 15% and when spike recoveries fall outside of the 90-110% control limit. Samples from analyses which fail to meet quality criteria will be re-analyzed following recalibration of the instrument.

All data will be entered into computer files and materials related to analyses (raw data sheets, recorder output, computer print outs, etc.) will be kept on file for future reference. Michael Anderson will review all laboratory data approximately every week and will require additional re-analysis as warranted.

B6 INSTRUMENT / EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE REQUIREMENTS

All field instruments will be inspected and calibrated prior to visiting the field. All

laboratory equipment used to provide numerical data will be calibrated to the accuracy requirements for its use prior to, periodically during and at the end of sample analysis.

B7 INSTRUMENT CALIBRATION AND FREQUENCY

Calibration procedures in the field will include calibration of the Trimble GeoExplorer II GPS System, Quanta HydroLab and Humminbird depth finder. Field maintenance will include cleaning and monitoring of the Ekman and Ponar sediment samplers, Wildco core sampler, peepers and benthic flux chambers.

Calibration schedules will be established for all laboratory instruments, with calibration records maintained in the laboratory logbooks kept with each instrument. Information relative to instrumentation maintenance will also be recorded in the logbook kept with each instrument. The Department of Environmental Sciences has a number of service contracts with various vendors and department instruments are serviced on a regular schedule.

B8 INSPECTION / ACCEPTANCE REQUIREMENTS FOR SUPPLIES AND CONSUMABLES

Sample containers will be washed and inspected before use. All sampling containers and laboratory glassware will be washed using the appropriate detergents, followed by rinsing with tap water, acid wash, where appropriate, and final rinses with distilled water followed by deionized water. Chemical calibrations and standardizations are made using standards prepared from materials of known purity using accepted analytical techniques.

B9 DATA ACQUISITION REQUIREMENTS

Data on nutrient inputs from external sources (e.g., river inputs, atmospheric deposition) will be used for nutrient budget calculations. Results from SSA-sponsored research and other peer-reviewed studies will be used where available. Other sources

will be reviewed prior to utilization of their data.

B10 DATA MANAGEMENT

Identification numbers will be recorded on all data forms, sample tracking sheets, analytical logbooks and computer data files. A computer file will serve as the Master Log Book for the project. As laboratory analyses are completed, the resulting data will be checked for quality by Laska Whiteaker and then entered into the computer files. All raw data and data entered into the computer files will then be evaluated by either Michael Anderson or Chris Amrhein. The computer files for this project will be networked, so that Michael Anderson and Chris Amrhein will have access to all data for evaluation and modeling purposes.

Sediment characterization data (*e.g.*, organic C, inorganic C, total N, P and S, particle size, etc.) and site information (GPS) will be used in conjunction with geostatistical techniques to delineate the primary sediment types within the basin (objective *i*). Nutrient concentrations of pore water and near-sediment water column samples collected with peepers will be used to estimate the diffusive flux of nutrients out of the sediments (objective *ii*). The changes in water column concentrations of nutrients from in situ benthic flux chambers and from column flux experiments over time will be used to directly calculate nutrient flux under field and controlled-laboratory conditions (objectives *ii* and *iv*, respectively). The C, N, P and S contents of particles collected using sediments traps will be used to quantify the rate of nutrient flux to the sediments (objective *iii*).

The results from objectives *i-iv* will be used to develop a model describing N and P flux across the sediment-water interface as a function of sediment type, temperature, and water quality (objective *v*). A distributed-source approach will be adopted, in which data about the distribution and areal extent of the different types of sediments identified (objective *i*) will be combined with mathematical relationships describing nutrient flux (objectives *ii* and *iv*) and bathymetric data to estimate the Sea's total internal nutrient loading from sediments.

The model developed in objective *v* will be combined with available water quality

data for the New and Alamo Rivers, the All American and East Highline Canals, and agricultural tile drains to construct a nutrient budget for the Sea (objective vi). Atmospheric deposition will also be included. Based upon results from hydrodynamic studies and the results from the above studies, existing US Army Corps of Engineers models, *e.g.*, CE-QUAL-R1 or BATHTUB, or a new finite-segment model will be validated against existing water quality data, and then used to simulate water quality in the Sea. The results can be used to predict the nutrient status, transparency, dissolved oxygen, chlorophyll *a*, and other characteristics of the Sea under different scenarios and management strategies.

SECTION C: ASSESSMENT / OVERSIGHT ELEMENTS

C1 ASSESSMENT AND RESPONSE ACTIONS

Project assessment will include regular observation of field sampling, sample handling, sample preparation, sample analysis, data evaluation and verification of quality control. Michael Anderson will be responsible for periodic monitoring of field activities and assuring that all field personnel are adequately trained for the sampling method requirements. Chris Amrhein will be responsible for supervising laboratory activities and monitoring quality control in sample analyses. Deviations from sampling and analytical protocols will be addressed by either Michael Anderson or Chris Amrhein.

In addition to internal assessment activities, the Quality Assurance officer for the Salton Sea Authority, Barry Gump, will visit the laboratory and field site at least once per year.

C2 REPORTS TO MANAGEMENT

Sediment and water quality data and accompanying QA results generated by this project will be reported to the Salton Sea Research Management Committee on a quarterly basis, including an annual report and a final report, as outlined in Table 6.

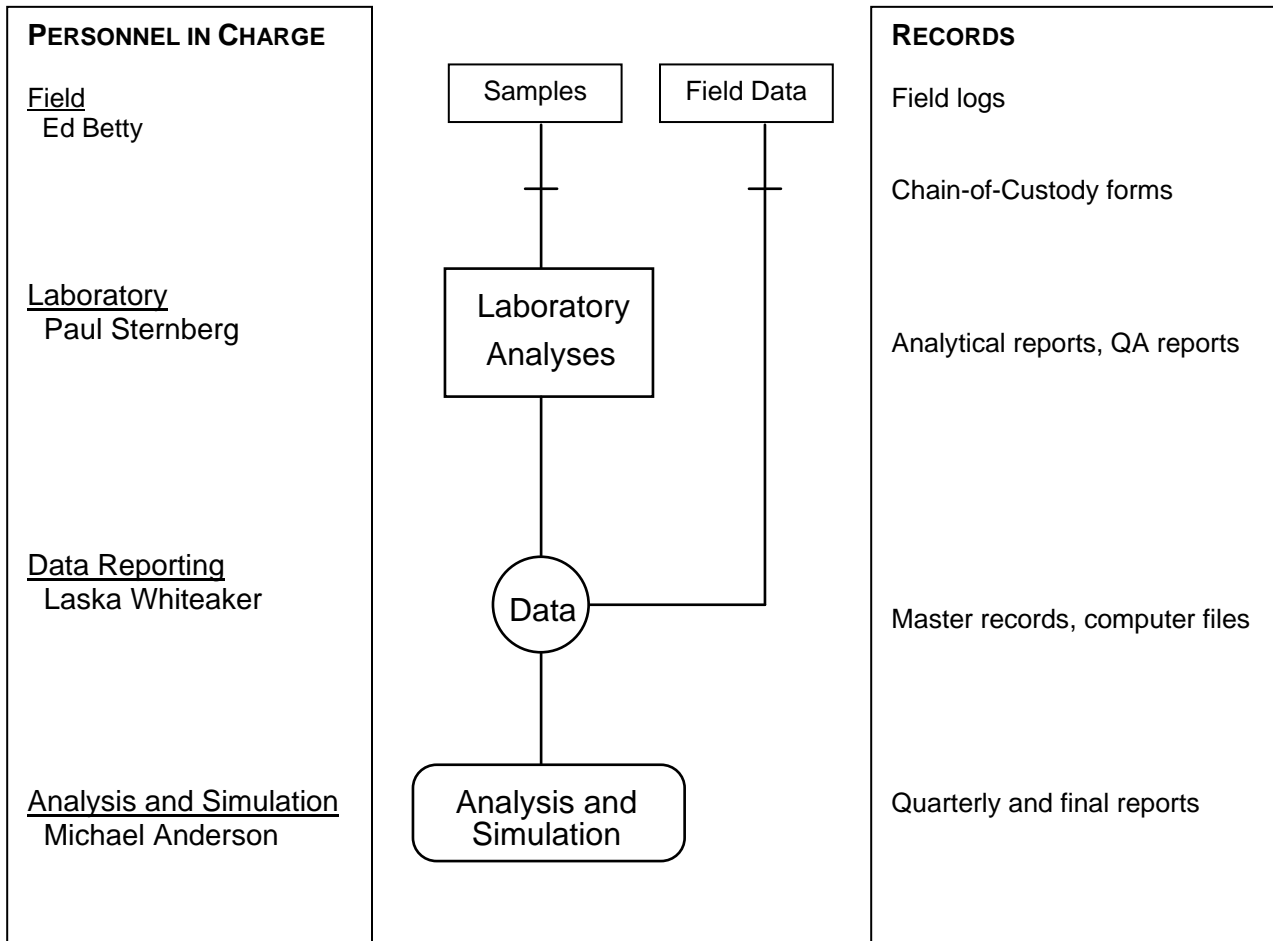
SECTION D: DATA VALIDATION AND USABILITY

D1 DATA REVIEW, VALIDATION, AND VERIFICATION REQUIREMENTS

Data review, validation and verification requirements are specified in section B5. Equipment blanks will be used to aid in the identification of problems due to field contamination. Duplicate analyses of field splits will be used to assess the precision of sampling and analytical methods. Quality control in the laboratory will include analysis of reagent blanks, method blanks, sample duplicates, certified standards and spikes.

D2 VALIDATION AND VERIFICATION METHODS

Sample and data flow, responsible personnel and data records for the project are summarized below:



D3 RECONCILIATION WITH DATA QUALITY OBJECTIVES

The data generated from the above analyses will quantify the rate of nutrient release from the sediments, which will then be used to construct a nutrient budget for the Salton Sea. Results will also be used in a lake water quality model to predict the water quality and trophic state of the Sea under different management strategies.

The project utilizes three distinct techniques for quantifying nutrient flux from the sediments. Results from the three different methods will be directly compared where possible, with the hope that all three will yield comparable estimates of flux. In the event that significant differences are observed between the flux rates determined from the three methods, the technique which yields results nearest the central tendency of the data set will be used for nutrient budget and modeling efforts. Constraints on the availability of appropriate external data may introduce significant uncertainty into nutrient budget and modeling calculations.

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Table 1. Timeline for Study (February 1, 1999 – July 31, 2001).

Objective and Task	2000						2001		
	Jan	Mar	Jun	Sep	Dec	Jan	Mar	Jun	
i Sediment Characterization		XXXXXXX							
Sediment Distribution wi/ Basin		XXXXXXX							
ii Peepers - Flux		XXX X X	XXX X X	XXX X X	XXX X X	XXX X X	XXX X		
In situ Benthic Chambers – Flux		XXX	XXX		XXX XXX X				
iii Sediment Traps – Particulate Flux		XXX	XXX	XXX	XXX X				
iv Core – Flux Experiments		XXXX	XXXX	XXXX	XXXX	X			
v Development of Flux Model			X X X	X X X X					
vi Development of Nutrient Budget				X X X	XXXXXX				
vii Evaluation of Sulfate Salt Pptn		X X X X							
Other Activities									
Chemical Analysis		X X X X X X X X X X X X	X X X X X X X X X X X X						
Report Preparation		XXX	XXX	XXX	XXX	XXXXXXX			

Table 2. Method detection limits, normal operating ranges and calibration methods for various analytes.

Measurement	Method Detection Limit	Operating Range	Calibration Method
<i>Water/Extracts</i>			
Alkalinity	10 mg/L	50-300 mg/L	Standards
Dissolved oxygen	0.01 mg/L	0-20 mg/L	External Calibration
Nitrogen			
Ammonia	0.1 mg/L	0.1-4 mg/L	Standards
Nitrate	0.1 mg/L	0.1-10 mg/L	Standards
Nitrite	0.2 mg/L	0.2-4 mg/L	Standards
pH			
Field	0.1	2-12	Buffers
Laboratory	0.01	0-14	Buffers
Phosphorous			
Total dissolved	0.1 mg/L	0.1-4 mg/L	Standards
Sulfur			
Sulfate	2.0 mg/L	2-30 mg/L	Standards
Sulfide	0.1 mg/L	0.1-2 mg/L	Standards
<i>Sediment</i>			
Inorganic Carbon	0.1 %	0-12 %	Standards
Particle size	5 %	0-100 %	N/A
Total			
Carbon	0.05 %	0-100 %	Standards
Nitrogen	0.05 %	0-100 %	Standards
Sulfur	0.3%	0-100%	Standards

Table 3. Frequency of replicates and spikes for various laboratory analyses.

Measurement	Frequency of replicates	Frequency of spikes
<i>Water/Extracts</i>		
Alkalinity	One in ten duplicated	One in ten spiked
Dissolved oxygen	Single analysis	None
Nitrogen		
Ammonia	One in ten duplicated	One in ten spiked
Nitrate	One in ten duplicated	One in ten spiked
Nitrite	One in ten duplicated	One in ten spiked
Total	One in ten duplicated	One in twenty spiked
pH		
Field	Single analysis	None
Laboratory	Single analysis	None
Phosphorous		
Total dissolved	One in ten duplicated	One in ten spiked
Sulfur		
Sulfate	One in ten duplicated	One in ten spiked
Sulfide	One in ten duplicated	One in ten spiked
<i>Sediments</i>		
Inorganic Carbon	One in ten duplicated	One in ten spiked
Particle size	One in ten duplicated	None
Total		
Carbon	One in ten duplicated	One in twenty spiked
Nitrogen	One in ten duplicated	One in twenty spiked
Sulfur	One in ten duplicated	One in twenty spiked

Table 4. Methods for the analysis of sediment samples.

Measurement	Standard Method/ Instrument	Reference
Carbon		
Total	NA 1500 CNS Analyzer	Carlo Erba, 1990
Inorganic	Pressure calcimeter	Loeppert and Suarez, 1996
Organic	Difference of total and inorganic	Nelson and Sommers, 1982
Nitrogen		
Total	NA 1500 CNS Analyzer	Carlo Erba, 1990
Particle size	Hydrometer/sieving	Gee and Bauder, 1986
Phosphorous		
Total	Ashing/extraction, Autoanalyzer	Aspila et al., 1976; APHA, 1989; Berner and Rao, 1994
Inorganic	Extraction, Autoanalyzer	Aspila et al., 1976; APHA, 1989; Berner and Rao, 1994
Organic	Difference of total and inorganic	Aspila et al., 1976; Berner and Rao, 1994
Exchangeable	Extraction, Autoanalyzer	Golterman and Booman, 1988
Iron-bound	Extraction, Autoanalyzer	Golterman and Booman, 1988
Calcium-bound	Extraction, Autoanalyzer	Golterman and Booman, 1988
Sulfur		
Total	NA 1500 CNS Analyzer	Carlo Erba, 1990

Table 5. Methods to be used for the analysis of water samples.

Measurement	EPA/Standard Method	Container	Preservative	Holding Time
Alkalinity	310.2	P	Cool, 4C	14 days
Conductance	120.1/2510B	P	Cool, 4C	28 days
Dissolved oxygen	360.1/4500-O	N/A	Determine on site	N/A
Nitrogen				
Ammonia	350.1/4500-NH ₄	P	Cool, 4C or H ₂ SO ₄ to pH,2	28 days
Nitrate	353.2/4500-NO ₃	P	Cool, 4C or H ₂ SO ₄ to pH,2	28 days
Nitrite	354.1/4500-NO ₂	P	Cool, 4C	48 hours
pH	150.1/4500-H	P	Determine on site Cool, 4C	24 hours
Phosphorous Reactive	365.3/4500-P	P	Cool, 4C or H ₂ SO ₄ to pH,2	28 days
Sulfur				
Sulfate	375.2/4500-SO ₄	P	Cool, 4C	28 days
Sulfide	4500-S ²⁻	P	Zinc acetate, 4C	48 hours
Temperature	170.1/2550B	N/A	Determine on site	N/A

Table 6. Data and document submission dates

<i>Date</i>	<i>Item</i>
May, 2000	Quarterly Report – Sediment Characterization and Distribution
August, 2000	Quarterly Report – Nutrient Flux I (Spring Data); Precipitation of Sulfate Salts
November, 2000	Quarterly Report – Nutrient Flux II (Summer-Fall Data)
February, 2000	Annual Report – Summarizing Characterization and Flux Results
April, 2001	Quarterly Report – Flux Model; Preliminary Nutrient Budget
July, 2001	Final Report
