Upper Neuse River Basin Association
Special Study Plan
Date Issued: August 17, 2015

Special Study Name, ID# and Origination:

Lake Sediment Evaluation, SS.LR.2

This Special Study was part of the Cardno FY 2015 monitoring contract and was extended into the 2016 contract. The 2015 contract provided for the collection, analysis and reporting on one set of samples. The extension into FY 2016 provided for supplemental sediment sampling in concert with sediment work the U.S. EPA may conduct on Falls Lake. Preliminary modeling efforts using the results of the sediment evaluations will help to evaluate the value of any further sediment data collection.

Responsible Contractor(s):

Cardno – responsible for study planning, general management and oversight, participation in sampling, and preliminary modeling activities.

Dr. Marc Alperin at the University of North Carolina Chapel Hill - responsible for planning, sampling, sample preparation and coordination of all laboratory analyses, data interpretation, and reporting.

Purpose of Study:

This Special Study will quantify the nutrient and organic carbon content of sediment samples from Falls Lake and use that data to help develop a more precise understanding of the spatial variability of sediment characteristics, bottom water, pore water, and benthic nutrient flux rates in Falls Lake. This evaluation will provide site-specific information which can be used to simulate spatial variability in benthic nutrient flux. The existing version of the Falls Lake Nutrient Response Model assumed uniform nutrient flux conditions throughout the lake and thus used a single set of model calibration factors. Information from this study will help develop a better understanding of the importance of internal nutrient loads to the waters of Falls Lake.

This Special Study supports these objectives of the UNRBA Monitoring Program:

- Lake response modeling,
- Support of regulatory options, and
- Source allocation and estimation of jurisdictional loading

Anticipated Schedule:

Field reconnaissance for this effort took place in May 2015 when sampling locations were determined. Sediment sampling was conducted in June 2015. Sample processing and laboratory analysis of the samples is to be conducted during July, August and September 2015. A preliminary memorandum summarizing the available results is anticipated from Dr. Alperin in September 2015 and is expected to be included in Cardno's Mid-Year data report to the UNRBA planned for release in October 2015. A final report from Dr. Alperin is expected in December 2015 (subject to completion of laboratory analyses on the anticipated schedule). Findings from the sediment evaluation will also be used as part of the Evaluation of Model Performance Special Study during FY 2016.

Summary of Study Methods:

Site Selection

Dr. Alperin and Cardno collaborated on selecting sampling sites which would maximize the ability of the analysis to identify large-scale (1000s of meters) and smaller-scale (10s of meters) patterns in potential benthic flux. Sampling locations were chosen to generally coincide with long-term DWR monitoring locations (Figure 1). The reconnaissance effort in May 2015 indicated that drowned creek and river channels contain the thickest deposits of unconsolidated sediments. Shallower areas (e.g., historic floodplains) showed little or no unconsolidated sediment in many of the areas visited, instead having hard clay or rock at or very near the substrate surface. As a result, cores were typically collected in the deepest part of the drowned channel as well as one or two locations between the old channel and lake shore. Downstream of the confluence of Beaverdam Creek with Falls Lake, the reservoir is more narrow and riverine, and only one or two coring locations were present at each location. In addition to the existing DWR stations, two additional coring sites were selected in the upper part of the lake, downstream of Ellerbe Creek and the Flat River. Selection of coring locations was facilitated with a sonar depth finder. Latitude and longitude of each sampling location was recorded from a boat-mounted GPS unit.

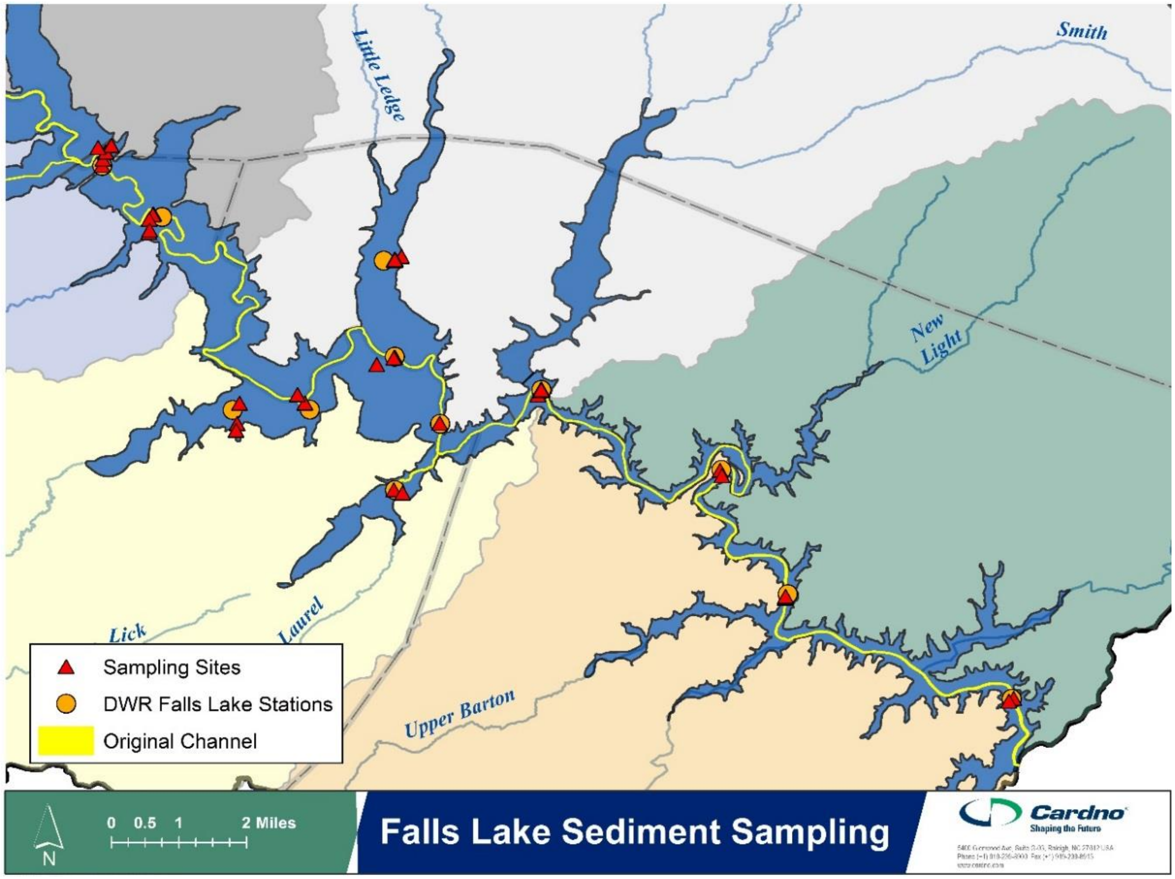

Figure 1. Sediment Coring Locations

Sediment Sample Collection

Sediment cores were collected from Falls Lake by lowering a gravity corer (Wildco K-B 20-inch model) through the water column and then allowing the weighted corer to push/settle into the lake bed sediment, thereby partially filling a plastic core liner with sediment in generally the same vertical profile as in the actual lake sediments.

As noted above, some areas of the lake do not have a layer of soft sediment and obtaining a core via the weight of the corer is not possible because the hard clay, coarse gravel, or bedrock prevent the device from penetrating the substrate. Cores were obtained from a few areas with hard clay substrates by collecting samples in water shallow enough to be waded, where it was possible to push the corer into the sediment by hand. Deeper areas with hard sediments require more specialized coring equipment not available through Dr. Alperin's laboratory and beyond the scope of this evaluation. Overall, very few locations resulted in an inability to obtain suitable samples.

Depth of cores ranged from less than 10 cm in areas with little or no soft sediment, to nearly 50 cm (the full height of the corer) in locations with deep soft sediments. Cores were immediately capped with Plexiglas o-ring seal inserts, stored in an ice-filled cooler in the field (< 6 hr), and then stored in a cold room (4°C) until processing. Core samples were transported to Dr. Alperin's laboratory in the core liners for subsampling. Special care was taken to keep the cores vertical from the time of collection to sectioning in the laboratory.

Water Quality Sample Collection

Water quality samples were collected above the sediment layer at each station. The water column depth was recorded and each water sample was collected within one meter of the bottom using a van Dorn sampler. At shallow sites (<1 m deep), water samples were collected from just below the water surface. Water samples were transferred from the van Dorn sampler to 500 mL Nalgene PETG bottles that had been pre-cleaned and rinsed with tap water followed by a triple-rinse with distilled-deionized water (DDW). Each bottle and cap was rinsed three times with bottom water before being filled with the sample. Water samples were immediately placed in an ice-filled cooler and then transferred to a cold room (4°C) upon return to the UNC laboratory the same day. All water samples were filtered within one day of collection using a 0.45 um Millipore-Millex-HN syringe filter, or equivalent, pre-rinsed with 20 mL DDW and flushed with 5 mL of sample. The filtrate (20 mL) was stored in 20 mL plastic scintillation vials or Falcon tubes (pre-cleaned) and stored in the laboratory freezer. Blanks were prepared for each site by filtering and storing 20 mL of DDW.

At each station, dissolved oxygen and temperature readings were recorded at 1-meter intervals throughout the water column using a YSI ProPlus water quality meter.

Sample Analyses

Laboratory Analyses Conducted at UNC Chapel Hill:

<u>Preparation of sediment and pore water aliquots for nutrient and carbon analysis:</u> Sediment cores were sectioned in 3-cm intervals within the top 10 cm of each core (fewer sections were obtained from the few cores with less than 10 cm of sediment). 30-mL of wet sediment was transferred to pre-cleaned 50-mL Falcon centrifuge tubes and then spun in an Eppendorf 5810R centrifuge at 5000G for 15 minutes. The supernatant was transferred to a 35 mL plastic syringe, filtered by syringe filter as described above, and stored frozen in 30-mL Falcon tubes. The sediment pellet in the centrifuge tube was frozen and then lyophilized for 72 hr in a Labonco FreezeDryer 8. The freeze-dried sediment was ground to fine powder with mortar and pestle, and stored at room temperature in 15 mL glass vials. Water and sediment subsamples were shipped to the analytical laboratory for nutrient and carbon analysis (additional details below).

Determination of Sediment Porosity: Sediment aliquots from each depth were weighed before and after drying using a Denver Instruments XS-410 balance. Mass was recorded after three consecutive measurements agreed to ±0.01 g. Each day, the balance was checked for drift using a standard brass weight. The wet sediment was dried for 24 hr at 100°C in a Fisher Scientific Isotemp oven, and then reweighed. Fifteen samples were dried for an additional 48 hr; the difference in mass between 24 and 48 hr was ~±0.01 g. Porosity values are necessary for the estimation of nutrient and carbon flux from the sediment.

Determination of Loss-on-Ignition (LOI): Dried sediment samples were combusted in a ThermoScientific Lindberg Blue M muffle furnace. The temperature was programed to increase from room temperature to 550°C in two hours, hold at 550°C for four hours, and then cool back to room temperature (typically overnight). Samples were re-weighed after sample combustion. LOI is generally highly correlated with organic carbon in sediments (which is also being quantified in this evaluation), thus it should be possible to generate a relationship between the two that will allow for rapid and inexpensive estimates of sediment organic carbon via measurement of LOI in the future.

Laboratory Analyses Conducted by Wetland Biogeochemical Analytical Services, LSU:

Chemical analysis of sediment and associated water samples will be performed by the Wetland Biogeochemical Analytical Services laboratory, which is a unit within Louisiana State University's School of the Coast & Environment (LSU). Dr. Alperin has a long-term relationship with this facility, allowing him to obtain high-quality analysis of environmental samples at a cost well below that of commercial laboratories.

The LSU lab is analyzing each bottom water and pore water sample for ammonium (EPA Method 350.1) and phosphate (EPA Method 365.5). The bottom water samples and uppermost pore water samples are analyzed for nitrate+nitrite (EPA Method 353.4). There is no value in analyzing deeper pore water samples for nitrate+nitrite, as they are not expected to be present in significant amounts under the anoxic conditions there.

Each dried sediment sample is assayed to determine % organic carbon and % nitrogen using a Costech Combustion Analyzer (EPA Method 440). Measurement of phosphorus in the solid sediment fraction is not of value for this study because the majority of the phosphorus is in mineral form or otherwise tightly bound to sediment particles; the phosphate fraction measured in the pore water is the nutrient portion available to migrate into the overlying water column.

Data Analysis

Dr. Alperin will analyze and interpret the laboratory results. Quantitative analyses and interpretation will be conventional for sediment evaluations of this type. From the nutrient and carbon concentration data, benthic flux rates will be estimated using Fick's Laws of Diffusion. Results of the analyses are expected to include tabular and graphical depictions of porosity, LOI, nutrient and organic carbon values and corresponding analysis/interpretation of nutrient lability, inventories of reactive organic nitrogen, spatial patterns, and nitrate, ammonium and phosphate flux from the sediments.

Quality Assurance/Quality Control:

Dr. Alperin conducts his laboratory work according to conventional quality control procedures for a university research institution. The LSU Wetland Biogeochemical Analytical Services laboratory has a formal Quality Control / Quality Assurance program in place under the direction of Thomas Blanchard, including Continuing Calibration Verification, Laboratory Duplicates, and Independent Calibration Verification (last update to plan: May 2015). Dr. Alperin provides the analytical laboratory with field duplicates as well as blind split samples, blanks, and standards as quality control checks.

Reporting/Deliverables:

Cardno will communicate with the UNRBA Executive Director on a regular basis on the progress of this Special Study. Status updates will be provided to the UNRBA Path Forward Committee and the Board of Directors at their regular meetings during Cardno's updates on the overall Monitoring Program status.

Discussion of the status and any available results from this Special Study will be included as part of the Mid-Year and Annual Reports.

Dr. Alperin will prepare a summary of the available results in early Fall 2015. This is expected to allow for a general assessment of the findings and spatial variability at scales ranging from centimeters within each core, to meters across the historic floodplain and channel, to kilometers along the lake's length.

The final report deliverable is anticipated in December 2015 (subject to completion of laboratory analyses in combination with Dr. Alperin's teaching schedule). It is expected to include a presentation of the raw data, determination of carbon-to-nitrogen ratios for the lake sediments and corresponding analysis/interpretation of lability, inventories of reactive organic nitrogen, estimates of nitrate, ammonium, and phosphate flux, and identification of spatial patterns observed, along with documentation of methods and a narrative interpretation of the findings. Dr. Alperin can make a presentation of the findings to the UNRBA Path Forward Committee, subject to his availability based on his teaching and research schedule.

Data from the Lake Sediment Evaluation will be included in the overall UNRBA database and will thus be available online to UNRBA members, agencies, and the general public. The final report prepared by Dr. Alperin will also be available on the UNRBA website, either as an appendix of the Annual Report produced by Cardno, or as a stand-alone document.