
ONONDAGA LAKE PRE-DESIGN INVESTIGATION:

**PHASE IV WORK PLAN ADDENDUM 8
SMU 8 HIGH-RESOLUTION CORES
Onondaga County, New York**

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TABLE OF CONTENTS

	<u>Page</u>
LIST OF ACRONYMS	iii
1.0 INTRODUCTION	1
2.0 OBJECTIVES	1
3.0 SAMPLING AND ANALYSIS WORK SCOPE	3
4.0 FIELD PROCEDURES.....	4
4.1 SEDIMENT SAMPLING AND PROCESSING	4
4.2 SAMPLE ANALYSES AND ASSESSMENT	5
5.0 HEALTH AND SAFETY.....	6
6.0 QUALITY ASSURANCE	6
7.0 DATA MANAGEMENT AND REPORTING.....	7
8.0 REFERENCES	7

TABLE OF CONTENTS (CONTINUED)

LIST OF TABLES

Table 1 2008 MNR Sampling and Analysis Summary

Table 2 Descriptions of 2008 MNR Sediment Sampling Locations

LIST OF FIGURES

Figure 1 Cesium-137 Subsurface Core Results in SMU 8 Sediment From 1988

Figure 2 2008 SMU 8 MNR Sediment Core Locations

LIST OF APPENDICES

Appendix A Parsons' Standard Operations Procedure for Sediment Core Sampling and for Processing Sediment Samples by Depth

Appendix B Laboratory Standard Operating Procedures For Measurement of Lead-210 and Cesium-137

LIST OF ACRONYMS

BSQV	bioaccumulation-based sediment quality value
cm/yr	Centimeters per year
CPOI	Chemical Parameters of Interest
dpm	disintegrations per minute (2.22 dpm = 1 picocurie or 1 pCi)
FS	Feasibility Study
GPS	Global Positioning System
mg/kg	milligrams per kilogram
MNR	Monitored Natural Recovery
NYSDEC	New York State Department of Environmental Conservation
pCi	picocurie (1 pCi = 2.22 DPM)
PEC	probable effect concentration
PDI	Pre-Design Investigation
ppm	parts per million (1 ppm is the same as 1 mg/kg in sediment)
QA/QC	Quality Assurance / Quality Control
RI	Remedial Investigation
ROD	Record of Decision
SMU	Sediment Management Unit
SOP	Standard Operating Procedure
USEPA	United States Environmental Protection Agency

Note: One centimeter (cm) of length is approximately equivalent to 0.4 inch. One inch is approximately equivalent to 2.5 cm. Thirty cm is approximately equivalent to 12 inches or 1 ft.

WORK PLAN FOR SMU 8 HIGH-RESOLUTION CORES

1.0 INTRODUCTION

This Work Plan describes sampling and analysis to be conducted in Sediment Management Unit (SMU) 8 of Onondaga Lake during 2008 consistent with the monitoring and contingency approach approved by the State of New York Department of Environmental Conservation (NYSDEC) for monitoring natural recovery in SMU 8 (Parsons, 2008a). SMU 8 accounts for approximately 65% of the lake's surface area and is the deepest water portion of Onondaga Lake where water depths exceed 9 meters (30 ft). SMU 8 is also referred to as the profundal zone of the lake where, based on variations in lake water temperature, waters become vertically stratified each year from late spring until fall.

The purpose of the sediment sampling and analysis described in this Work Plan is to better quantify accumulation rates to date in SMU 8 as part of the ongoing evaluation of monitored natural recovery (MNR). MNR in SMU 8 is one of the elements of the remedy for Onondaga Lake specified in the Record of Decision (ROD) for the Onondaga Lake Bottom Subsite (NYSDEC and USEPA, 2005). Sampling and analysis described herein will help determine if any additional remedial measures will be needed in SMU 8 in the future, such as additional thin-layer capping. Other Honeywell efforts to be used to help assess MNR include previous and future monitoring and modeling efforts and microbead marker placement and monitoring work described in a separate work plan (Parsons, 2008b).

The remedy for Onondaga Lake, as specified in the ROD, includes MNR in SMU 8 to achieve the mercury probable effect concentration (PEC) of 2.2 milligrams per kilogram (mg/kg or parts per million) in the lake's profundal zone (where water depths exceed 9 meters or 30 ft) and to achieve the bioaccumulation-based sediment quality value (BSQV) for mercury of 0.8 mg/kg on an area-wide basis, within ten years following the remediation of upland sources, dredging and/or isolation capping of littoral sediment (in SMUs 1 through 7), and initial thin layer capping in the profundal zone. Dredging of littoral sediment is scheduled to begin by 2012, and capping is scheduled to be completed following the completion of dredging. The starting point for baseline monitoring of natural recovery following active remediation will begin after the completion of dredging and capping.

2.0 OBJECTIVES

SMU 8 sediment will be sampled at various locations during November 2008 and analyzed for two radioisotopes (lead-210 and cesium-137), mercury, bulk density, and water content. Results from the radioisotope and mercury analyses will be used to update previous quantifications of sedimentation (i.e., sediment accumulation) rates in SMU 8. Results from

mercury and bulk density analyses will also be used to further describe ongoing natural recovery. Results from this sampling and analysis will, in combination with other relevant data, provide the basis for refining the MNR assessment and predictions described in the Feasibility Study (FS) Report for Onondaga Lake (Parsons, 2004).

Sediment sampling and analysis described in this work plan will provide additional basis for quantifying accumulation rates and, in particular, accumulation rates since the most recent radioisotope work was conducted during the 1990's. The average sediment accumulation rate in SMU 8 since 1986, when Honeywell's predecessor company discontinued soda ash manufacturing near the lakeshore, is approximately 0.3 to 0.6 centimeter per year (cm/yr) at the South Deep station based on available data summarized in the FS Report (see Appendix N in Parsons, 2004). This sampling and analysis effort will provide basis for evaluating whether recent sedimentation history differs from that observed in cores collected during the late 1980's and early 1990's.

The first use of mercury at the former Willis Avenue Plant was in 1946 or 1947 based on memos from a Honeywell predecessor company indicating the mercury cell plant was under construction in 1946. Other information available about mercury loadings to the lake is summarized in Section 1 of the Onondaga Lake Remedial Investigation Report (TAMS Consultants and YEC, 2002). Mercury cell operation took place at the former Willis Avenue Plant from 1946-1947 until 1977. Mercury cell operation took place at the former Bridge Street Plant from 1953 until 1988. Effluent mercury loadings were reduced from 21 pounds per day to 1 pound per day based on a 1970 consent order and further reduced at the former Bridge Street plant to 0.028 pound per day in 1977.

Sediment accumulation will be evaluated by measuring concentrations of lead-210, cesium-137, and mercury. Lead-210 is a naturally-occurring radioactive element that has a half life of 22.3 years. Cesium-137 is a thermonuclear byproduct with a half life of 30.3 years. Cesium deposition began in approximately the year 1952, increased significantly in 1954-1955 (see Figure 1), and peaked during the years 1963-1964 (USGS, 2008). Mercury loadings to the lake from Honeywell predecessor company operations increased in the late 1940's and early 1950's and later declined significantly in the year 1970 based on substantial reductions in discharges of mercury to the lake that Honeywell's predecessor company instituted at that time. Measuring levels of these two radioisotopes and variability of sediment mercury concentrations with depth will provide multiple lines of evidence for quantifying parameters needed as input to the MNR Model (use of which is discussed in Parsons, 2004 and in Parsons, 2008a).

In addition to demonstrating long-term sediment stability, radioisotope core data provide a method to quantify sediment accumulation rates for quiescent waters. Accumulation rates measured by cores shown in Appendix N of the Onondaga Lake FS Report (Parsons, 2004) vary in a range of approximately 0.3 cm/yr to 0.6 cm/yr (average 0.4 cm/yr) after 1986 when

Honeywell's predecessor ceased manufacturing of soda ash. Rates after 1986 are all based on data from Hairston *et al*, (1999) which is the only radioisotope or deep mercury coring work performed since 1992 (with the exception of a single core for cesium-137 analysis collected by Honeywell in 1996 that included a possible error in sample labeling).

Data from high-resolution cores rely on known chemical markers to establish the time span (and thereby accumulation rate) represented by the core or portions of the core. A limitation in the Onondaga Lake system is that for cesium-137 and mercury, the last known marker is from at least 20 years ago. Most of the discrete cesium-137 and mercury markers for Onondaga Lake include events that occurred until the mid-to-late 1980's; thus, they have little use in establishing the time span in more recent (upper) sections of such cores. Similarly, lead-210 data cannot easily resolve time spans of only a few years given its half life of 22.3 years, typical SMU 8 accumulation rates of 0.3 to 0.6 cm per year, and given the minimum length of vertical sample intervals extractable from sediment cores.

While developing the work scope presented herein, prior input from NYSDEC has been considered. In particular, NYSDEC input dated August 17, 2006 was assessed as this work scope was developed.

3.0 SAMPLING AND ANALYSIS WORK SCOPE

Sediment cores will be collected at a total of six locations as shown on Figure 2. These sample locations will provide widespread distribution throughout SMU 8 with more locations in the southern half of the lake than in the northern half. One of the sample locations (at ST-51 in the South Basin) is co-located with the one of the two previous radioisotope analyses conducted in SMU 8 during 1992. Table 1 provides a summary of the sampling and analysis work scope. Table 2 provides a summary basis for coring at the six locations identified in Figure 2.

Sediment at five of the six locations will be collected to a depth of 100 cm (1 meter or 3.3 ft). Sediment at the sixth location (i.e., OL-STA-80073 in the Ninemile Creek Outlet area), will be collected to a depth of 200 cm (2 meters or 6.6 ft).

With the exception of samples from location OL-STA-80073, the vertical intervals for conducting laboratory analyses will be every two cm (0.8 inch) within the top 10 cm (4 inches) and every other 2 cm from 10 to 70 cm (28 inches). The current average sediment accumulation rate in SMU 8 is approximately 0.3 to 0.6 cm per year, while prior sediment accumulation rates may have been as high as one cm per year. Each centimeter of accumulated sediment represents a timeframe of 1.6 to 3.3 years for the uppermost 10 to 20 cm of sediment and a timeframe of approximately one to two years for deeper sediment. Based on averages of these ranges, the top 70 cm likely represent approximately 120 years of sediment accumulation. Reaching deep enough to obtain 120 years of sediment would improve the radioisotope modeling analysis explained in Section 5.2.

For location OL-STA-80073, the vertical intervals for conducting laboratory analyses will be every two cm (0.8 inch) within the top 10 cm (4 inches) and every other 2 cm from 10 to 100 cm (39 inches).

With the exception of samples from location OL-STA-80073, sediment from the top 50 cm will be analyzed for lead-210, cesium-137, mercury, and bulk density, and water content. From depths of 50 cm to 70 cm, sediment will be analyzed for lead-210, mercury, bulk density, and water content given this sediment likely represents accumulation from years prior to the 1950's. Sediment from intervals not analyzed within the top 70 cm and sediment below 70 cm to a depth of 100 cm will be retained in case future analyses are warranted.

For location OL-STA-80073, sediment samples from the top 70 cm will be analyzed for lead-210, cesium-137, mercury, and bulk density, and water content. From depths of 70 cm to 100 cm, sediment will be analyzed for lead-210, mercury, bulk density, and water content. Sediment from intervals not analyzed within the top 100 cm and sediment below 100 cm to a depth of 200 cm will be retained in case future analyses are warranted.

Analyses of cesium-137 and lead-210 will be conducted based on the SOPs presented in Appendix B. Analyses for mercury, bulk density, and water content will be conducted using standard USEPA and ASTM procedures respectively that have been used previously and continue to be used successfully as part of the Onondaga Lake pre-design investigation work efforts and as part of the Onondaga Lake baseline monitoring effort. Test America will conduct the analyses for mercury (USEPA SW-846, Method 7471A) and water content (ASTM D2216). Flett Research will conduct the analyses for radioisotopes, water content, and bulk density. Analyses for bulk density will be conducted by weighing, drying, and then reweighing a typical sediment volume of five milliliters.

4.0 FIELD PROCEDURES

4.1 Sediment Sampling and Processing

Push coring devices have been used successfully from a sampling boat as part of previous pre-design investigation work with Onondaga Lake sediment. If the push corer can not sufficiently penetrate into the SMU 8 sediment, a VibracoreTM unit will be added to provide additional weight over the push corer to promote additional penetration. Sample management, equipment decontamination, and other field procedures not specified in this Work Plan will follow procedures provided in the Onondaga Lake PDI Standard Operating Procedures (SOP) (Parsons, 2005b). If smearing is significant during processing, sediment from the inner section of each core will be used for mercury and radioisotope analysis.

Sample boat positioning and sediment sample locations will be determined using a Trimble geographic positioning system (GPS) receiver (or equivalent) interfaced with a navigation and

data logging system. Differential GPS coordinate positions and water depths will be reported for each sediment sampling location.

SMU 8 shallow sediments are generally very soft and fluffy. If sediment samples cannot be collected to the desired depth with at least 80 to 90% sample recovery in a relatively undisturbed condition, the sediment sampler will be moved approximately 10 ft to a new location where a second attempt will be made to collect a suitable sample. If the second attempt is also not successful, a third attempt will be made at a location approximately 10 ft away from the original sample location in another direction. In addition, sediment compaction due to sampling will be measured by measuring the penetration depth on the outside of the sampling device and comparing penetration depth of the sampler with sediment depth inside the sampler.

Two sampling tubes with an inside diameter of 3.5 inches will be used to collect sediment from each location. Sufficient sample dry weights needed by the laboratories from a 2-cm interval can be obtained from a single core. Once collected, sampling tubes containing sediment will be capped on both ends and stored vertically on the boat and in a cooler with ice. Sediment depth within each tube will be measured soon after collection. Samples will be processed from one of the two tubes. Sediment from intervals not being analyzed and sediment cores that are not processed (*i.e.*, the duplicate core from each location) will be archived for possible future analysis as warranted. Sample processing onshore will start with measuring the total depth of sediment within each tube. Sediment for analysis will then be cut or extruded from one sampling tube per sample location into 2-cm slices of sediment using the sample processing SOP from the 2007 MNR sampling effort (Parsons, 2008a) slightly modified as presented in Appendix A. Sample management, equipment decontamination, and other field procedures not specified in this work plan will follow procedures provided in the Onondaga Lake PDI Standard Operating Procedures (Parsons, 2005b). Archived samples and cores will be kept vertical and refrigerated at the Parsons' field office located at the lakeshore. Caps on the archived sample tubes will be sealed completely and inspected at regular time intervals.

4.2 Sample Analyses and Assessment

Analyses of cesium-137, lead-210, bulk density, and water content will be conducted by Flett Research located in Winnipeg, Manitoba, Canada. Analyses of mercury will be conducted consistent with previous Onondaga Lake pre-design work efforts at Test America's Pittsburgh, PA or at another Test America laboratory approved for mercury analysis.

Flett Research will also assess the lead-210 data using a slope regression model. The model is an iterative best-fit digital model designed to process date for cases where background activity is not known. The purpose of the modeling is to estimate the age at the bottom of each core section. The sediment accumulation rate will also be estimated in units such as dry grams per square cm per year. Lead-210 is most often the primary method used to calculate sediment age

and accumulation rates. Cesium-137 measurements serve to check lead-210 estimates, and in some cases, are used to help choose one of several possible lead-210 interpretations. The dry bulk density profiles can also be important in interpreting the core's history, and like cesium-137, may help in deciding which of several possible lead-210 interpretations is most likely. SMU 8 sediment is expected to be relatively homogeneous, so Flett Research will measure bulk density by homogenizing each wet sample well, weighing a precise volume of the wet sediment, drying, and then reweighing the sample.

The lead-210 model to be used by Flett Research does not depend upon the assumption of a constant sediment accumulation rate. However, this model does require that background be achieved. In shorter cores, it may be possible to use the linear regression model, together with Ra-226 measurements, to determine average sediment accumulation rates and approximate ages of sediment. Not all cores provide data that are useful in calculating ages or sediment accumulation rates. The model is based on the assumption that a perfect core will exhibit a linear decrease in the logarithm of the sample's excess activity as a function of cumulative dry weight of the sediment sample. If background activity is known based on the results, a series of sediment age calculations will be developed as a function of sediment depth.

Analytical results will be shared with the SMU 8 Technical Work Group to determine which, if any, of the archived samples warrant analysis for mercury, lead-210, or cesium-137.

5.0 HEALTH AND SAFETY

Health and Safety Plans for personnel working for Honeywell must be consistent with the Honeywell Syracuse Portfolio Health and Safety Plan (Honeywell, Parsons, and O'Brien & Gere, 2007). If not already available, the subcontractor will prepare a Safety Plan to be implemented by all subcontracted personnel. Any task outside of the work scope incorporated into the Project Safety Plan will have a new Job Safety Analysis completed and reviewed before the task begins. The Parsons Project Safety Plan will be strictly followed by Parsons personnel. Copies of the Subcontractor Safety Plan and the Project Safety Plan have been provided to NYSDEC and will be maintained at the support zone onshore and on the work boat.

Field crew members will need to have successfully completed a minimum of the 24-hour HAZWOPER training consistent with Occupational Safety and Health Administration requirements for workers potentially exposed to SMU 8 sediment.

6.0 QUALITY ASSURANCE

The support zone and facilities established by Honeywell for the pre-design investigation efforts will be used as needed for the sediment sampling work described in this Work Plan. Work efforts not specifically described herein, such as decontamination and waste management activities, will be conducted in accordance with Phase I PDI Work Plan (Parsons, 2005a,

Appendix A). Laboratory analyses of lead-210 and cesium-137 will be conducted in accordance with the SOPs presented in Appendix B and with the SOPs for sediment mercury analysis presented in the quality assurance project plan for Book 2 of the 2008 Baseline Monitoring Work Plan (Parsons et al, 2008). Field quality assurance and quality control will consist of the collection and analysis of field duplicates and other quality assurance samples as appropriate.

7.0 DATA MANAGEMENT AND REPORTING

Sample names, QA/QC procedures, sample collection, data entry, and data validation for this portion of the work will be conducted in accordance with the Phase I PDI Work Plan (Parsons, 2005a, Appendix A and Appendix B). Any deviations from these procedures will be discussed with NYSDEC prior to execution of the work.

The data will be organized into a compilation of laboratory and field generated data in electronic file format. Electronic data files will be generated by the analytical laboratories, while pertinent field data will be entered into electronic format during collection. Data will be added to Locus Focus through an input module of the system by the Data Manager. Access to the input module will be restricted to the Syracuse Portfolio Data Managers or delegates. Chemical analytical data will be loaded/entered into a database.

Analytical data generated during this investigation will be reviewed for usability. All analytes will be subject to Level III validation as described in the PDI QAPP (Parsons 2005). In addition, ten percent of the total mercury data will be validated based on Level IV protocols. The validated results will be incorporated into the Locus Focus database by Parsons following validation. Once the data have been validated, results will be reviewed with the Onondaga Lake SMU 8 Technical Work Group. The work group will receive interim updates as warranted as the work continues. In addition, results from the work effort described in this Work Plan will be documented in a submittal most likely in conjunction with other Phase IV Pre-Design Investigation work efforts.

Unvalidated and validated data will be submitted to NYSDEC consistent in content and timing with submissions being provided for other pre-design investigation and baseline monitoring efforts for Onondaga Lake. Analytical data generated during this investigation will be reviewed and validated as described in detail in the QAPP associated with the pre-design investigation work.

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Appendix A Phase I Sampling And Analysis Plan
Appendix B Quality Assurance Project Plan
Appendix C Project Safety Plan Updated March 2007.
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**TABLE 1
2008 MNR SAMPLING AND ANALYSIS SUMMARY**

Objectives	Update accumulation rates obtained during the 1990's and quantify corresponding indicator sediment chemical concentrations.
Sample collection method	Push corer with 3.5-inch inside diameter polycarbonate tubes.
Sample depth intervals for analyses	Every 2 cm from the sediment surface (or mudline) to 70 cm below the mudline except at location OL-STA-80073 in the Ninemile Creek Outlet area where samples will be analyzed from the top 100 cm.
Field observations to be recorded	Lake water surface elevation, water depth to top of sediment, and sample position coordinates. Sediment depth collected within each core tube measured on the sampling boat during collection measured again at the sample processing location onshore
Laboratory analyses	Lead-210, cesium-137, mercury, bulk density, and water content for every 2 cm of sediment depth to 10 cm and for every other 2-cm depth interval below 10 cm to 50 cm (to 70 cm for the Ninemile Creek Outlet location). Lead-210, mercury, bulk density and water content only for every other 2 cm depth below 50 cm to 70 cm (and from 70 to 100 cm for the Ninemile Creek Outlet location).
Quality assurance	Analyze one field duplicate core from one of the six locations. Archive sections not analyzed and archive a second core from each sampling location. Conduct matrix spikes and matrix spike duplicates for mercury analyses as appropriate consistent with previous Onondaga Lake sediment mercury analytical efforts.

Note: Based on 0.3 to 0.6 cm per year of sediment accumulation since 1986, the average accumulation indicates the last 22 years can be accounted for in the top 10 cm. Based on 0.5 to one cm per year of sediment accumulation prior to 1986, the average accumulation from 1954 to 1986 (32 years) can be accounted for in the next 24 cm.

TABLE 2
DESCRIPTION OF 2008 MNR SEDIMENT SAMPLING LOCATIONS

<u>SMU 8 Location</u>	<u>Vicinity</u>	<u>Basis for Selected Location</u>
North Basin		
OL-STA-80068	Center	Near OL-STA-80068 where 9.2(J) mg/kg and 46.9(J) mg/kg mercury were measured in sediment at 4 to 10 cm and 10 to 15 cm respectively during November 2007.
Ninemile Creek (NMC) Outlet		
OL-STA-80073	Center	A core collected in 1992 in this area did not show distinct trends. However, the rate of sediment deposition may be higher in this area than elsewhere in SMU 8. Recurring large storms and/or rapid snowmelt peak flows from Ninemile Creek tend to mix lake sediment adjoining the creek mouth. Given a potentially higher deposition rate here than elsewhere in SMU 8, the depth of this core will be 200 cm instead of 100 cm. Samples will be analyzed to a depth of 100 cm, and sediment below 100 cm will be archived. Microbeads are planned to be placed near OL-STA-80073.
Saddle		
OL-STA-80103	Center	Area could have different accumulation characteristics than North Basin or South Basin. November 2007 sediment mercury concentrations in the Saddle at OL-STA-80075 were 1.6(J) to 2.1(J) mg/kg in the top 15 cm.
South Basin		
OL-STA-80076	North center	Near OL-STA-80076 where 6.2(J) mg/kg mercury was measured in sediment at 10 to 15 cm (November 2007)). Also near OL-MB-80097 where microbeads are planned to be placed.
ST51	Center	Near ST-51 where lead-210 and cesium-137 were analyzed during September 1992.
South Corner		
OL-STA-80089	South Center	Near OL-STA-80089 where mercury in the top 15 cm during November 2007 was 1.6 to 2.5(J) mg/kg.

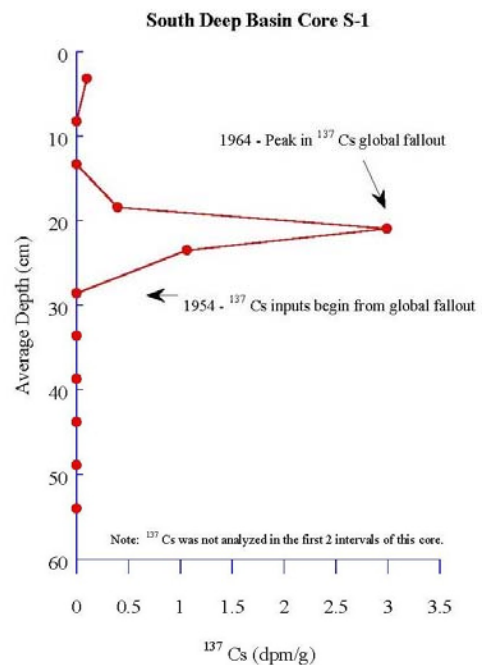
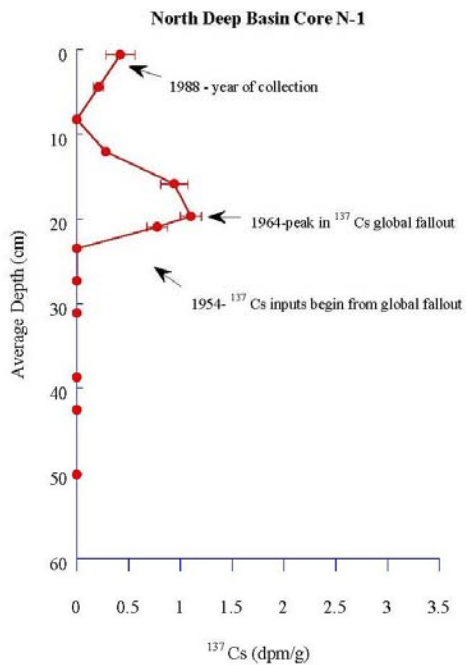
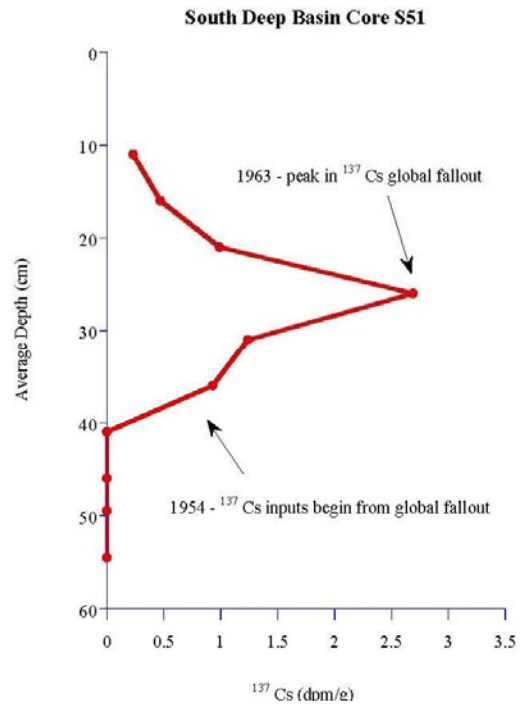
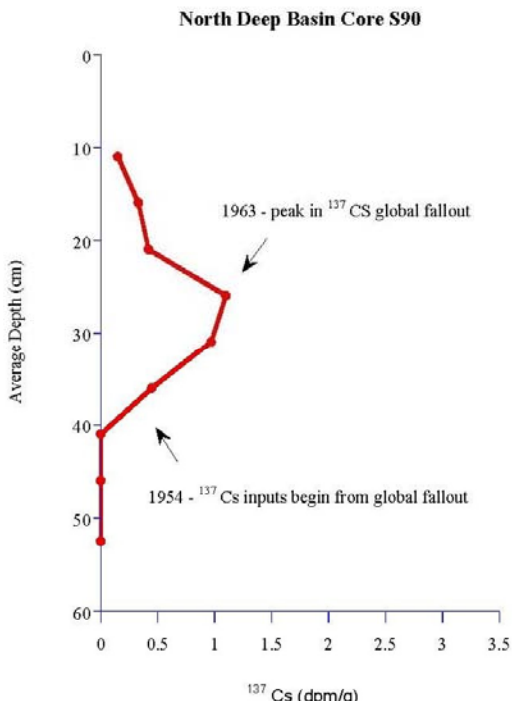
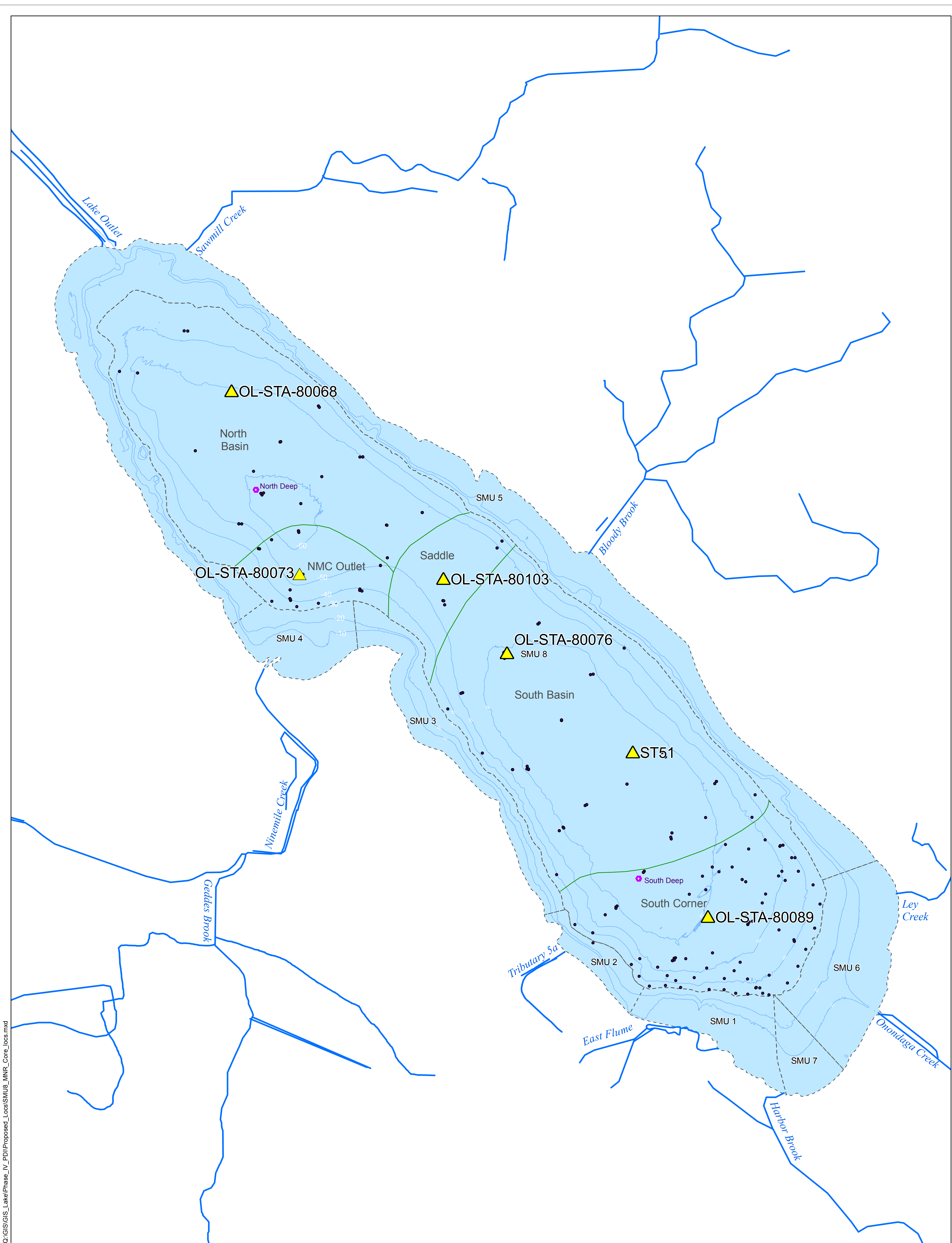


FIGURE 1

**ONONDAGA LAKE
SYRACUSE, NEW YORK**

**CESIUM-137 SUBSURFACE CORE
RESULTS IN SMU 8 SEDIMENT
FROM 1988**

Source: TAMS, 2002



C:\GIS\GIS_Lake\Phase_IV_PDI\Proposed_Locs\SMU8_MNR_Core_Locs.mxd

▲ 2008 MNR Sediment Core Location

HISTORICAL SAMPLE LOCATIONS (RI TO PHASE III PDI)

- Phase III PDI Box Core Sample Location
- Historical Sample Location (RI to Phase II)

● North and South Deeps

- NYSDEC Demarcation for SMU 8
- 10 ft. Bathymetric contour



FIGURE 2

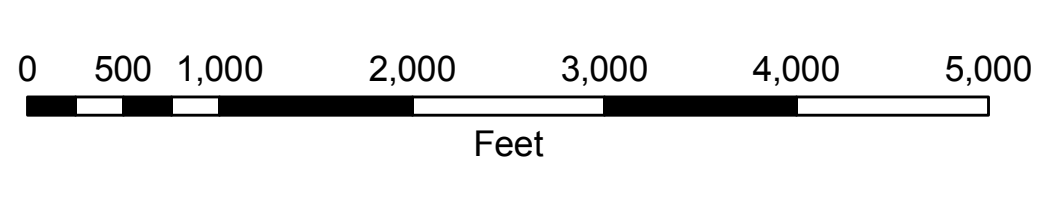
Honeywell

Onondaga Lake
Syracuse, New York

2008 SMU 8 MNR
Sediment Core Locations

PARSONS

290 ELWOOD DAVIS RD, SUITE 312, LIVERPOOL, NY 13088 Phone:(315)451-9560



NOTES

1. Bathymetry contour interval = 10 feet.
2. Water depth based on average lake elevation of 362.82 feet.

APPENDIX A

STANDARD OPERATIONS PROCEDURE FOR SEDIMENT CORE SAMPLING AND FOR PROCESSING SEDIMENT SAMPLES BY DEPTH

Sediment cores will be collected using the push core technique. If the weight of the core is not sufficient to penetrate as needed into the sediment, a Vibracore™ unit will be used without using the vibration option. The Vibracore™ unit has more weight than the push core.

(Note: The SOP text below for sediment processing is very similar to the SOP presented initially during late 2007 in Parsons, 2008c.)

Sediment sample processing will be done at the lake shoreline. The inner sediment within each tube will be collected for analysis of lead-210, cesium-137, bulk density, mercury, and water content.. As needed, the outer sediment from each tube can be collected for physical analyses. This will eliminate the potential of introducing contaminants from a shallower zone through smearing near the sediment/sample tube interface. Clean stainless steel, glass, or Teflon coated implements will be used to slice and transfer each sample. A semi-circular stainless steel spatula or similar device will be used to slice the core segments, one segment at a time. Contact between sampler gloves and sample will be avoided. For each sample location, the procedure for processing the tubes of sediment will be as follows:

1. Carefully remove excess water (if any) that has accumulated at the top of each tube without disturbing underlying sediment. A pipette-like device should be adequate for slowly and carefully removing excess water.
2. Record total sediment depth (or top of sediment position) within each tube and note any settling which may have occurred between the top of sediment in the core tube recorded on the boat and the top of sediment in the core tube recorded as part of this step prior to beginning sample extrusion.
3. As appropriate, measure and mark one or more short measuring tubes at 2 cm intervals on the barrel of each measuring tube.
4. Maintaining its vertical position, place each individual tube containing sediment in an extrusion device over clean plastic or aluminum foil.
5. With an extrusion device (see Mudroch and MacKnight, 1994 for an example), push from the bottom of the tube so the top two cm of sediment can be sliced with a wide

knife or spatula into a short, separate measuring tube (or portion of a measuring tube) and so a cap can be positioned over the sediment.

6. Place the measuring tube containing sampled sediment into a holding container. Replace the cap onto the original tube to hold in place the rest of the sediment core and associated water.
7. Weigh the empty sample containers.
8. With minimal sample disturbance, use an appropriate knife or similar device to carefully and quickly remove sufficient sediment from the inner 70 to 80% of the sample volume over the entire depth interval of the sample and place the inner sample volume carefully into a previously-weighed sample container.
9. Composite remaining sample as needed for physical analyses as needed.
10. Weigh the containers containing sample to confirm sufficient sample quantity is being provided to the laboratory for analyses.
11. Repeat Steps 4 through 9 above for the next 2 cm interval as needed.

Once each container is full and ready for analysis, apply a label (if not pre-applied), and refrigerate as soon as practical.

The above procedure will be continued until all sample intervals have been collected. Any unused sediment will be properly managed in accordance with the appropriate laboratory SOP.

The following alternate sediment processing procedure may be used although it was less preferred during the 2008 microbead marker pre-mobilization field tests (see Parsons, 2008b for the pre-mobilization field test work scope):

1. Tip the side of the tube away from the portion of the tube to be cut so water will not spill out during Steps 2 through 4 below.
2. Make two longitudinal cuts through the wall of the tube from the top of the tube to a level below the top of the sediment that is close to the bottom of the sediment interval being sampled. The horizontal distance between the two longitudinal cuts will be approximately 1/3 to 1/2 of the tube's lateral circumference.
3. Make one lateral cut along the base of the two longitudinal cuts.
4. Keeping the tube tipped, remove the section of the tube between the two longitudinal cuts.
5. If the solids content of the sediment is low and sediment can not be scooped without slumping, place one or more thin discs horizontally into the tube at the base of the vertical interval of sediment being sampled.

6. Remove the sediment sample with a small spoon or equivalent into an appropriate sample holding container.
7. Remove excess water and cut and remove excess remaining tube length no longer needed above the sediment interval most recently sampled.
8. As needed, repeat Steps 1 through 7 above to obtain a sample of sediment the next sampling interval within the tube.

Additional Reference:

Mudroch, A. and S.D. MacKnight (editors), 1994. Handbook of Techniques for Aquatic Sediments Sampling. Lewis Publishers (CRC Press). Second edition.

APPENDIX B

STANDARD OPERATING PROCEDURES FOR LABORATORY MEASUREMENT OF LEAD-210 AND CESIUM-137

(Prepared by Flett Research, Ltd.)

Overview of the Pb-210 Method as Performed at Flett Research Ltd, Winnipeg, Canada (2008)

Last Modified by R. Flett Nov. 21, 2008

This method is based upon the method of Eakins and Morrison (1978) wherein polonium-210 (the granddaughter of Pb-210) is distilled out of sediments at high temperature, acid digested and finally plated onto silver disks for analysis by alpha spectrometry. It is assumed that Po-210 is in secular equilibrium (i.e. at the same activity) with the Pb-210, a situation expected when sediments are > 2 years old.

Field Collection

Samples should be collected in clean jars. The jars should be waterproof so that no water is lost from the samples – try to keep the rims of the jars clean when closing so that no particles are on the sealing surfaces. Avoid loss of water from the core slices as this will affect the bulk density which in turn will affect the Pb-210 dating calculations. The samples do not require special storage conditions. Holding times are unlimited.

Distillation

A 0.1 - 0.5 g sample of dry sediment (or wet sample providing the equivalent amount of dry sediment) is put into a test tube, treated with hydrochloric acid and spiked with a precisely known aliquot (10 - 20 DPM) of Po-209 tracer (NIST SRM 4326 or NIST traceable). The sample is dried overnight in a heating block at about 100 - 120 C. A small Pyrex wool plug is pushed down the test tube on top of the sample and a second Pyrex plug, dampened with water, is inserted in the top most portion of the test tube. The tube is then placed horizontally in a tube furnace at about 500 C for 1 hour (only the bottom half of the tube is actually in the furnace). The volatile chloride form of polonium distills out of the sediment and condenses on the cooler Pyrex plug in the portion of the tube out of the furnace.

Acid Digestion

After the tubes have cooled, the tube is etched 2 in. from the bottom and the lower section, which contains the sediment, is broken off and discarded. The upper section is placed into a 400 ml tall form beaker, the Pyrex wool forced out of the tube onto the floor of the beaker with a clean glass rod, and 25 ml of conc. HNO₃ is added to the beaker. The beaker, covered with a watch glass, is heated on a hotplate so that it gently refluxes for about 3 hours, or until all brown colouration has disappeared. When cool, the HNO₃ and Pyrex wool is poured into a funnel into a 200 ml beaker. The 400 ml beaker is rinsed 3 times with about 5 ml of 1.5 N HNO₃ and the rinses are poured over the Pyrex wool in the funnel so that any polonium absorbed to the wool is washed into the beaker. The wool in the funnel, following several more rinses with 1.5 N HNO₃, is then squeezed mostly dry with a glass stirring rod, the funnel removed and the wool discarded.

The acid polonium solution is evaporated to dryness, and then made up to 20 ml with 1.5 N HCl and heated gently until any precipitate is dissolved. The warm solution is poured into a 25 ml beaker which already contains a silver disk and the 200 ml beaker is rinsed with 3 X 2 ml rinses of ascorbic acid solution which are poured into the small beaker. The 25 ml beaker is heated just to boiling for a period of 3 hours. After cooling, the fluid is poured out of the beaker and the disks are rinsed with distilled water and then methanol. They are placed on a paper towel and dry overnight at room temperature.

Counting by Octet PC Alpha Spectrometer

The silver disks are placed into an Ortec 'Octet' alpha spectrometer (one silver disk in an evacuated chamber at a time) for a 30,000 – 60,000 second counting period. The samples are monitored for Po-210 and Po-209 isotopes with a 300 mm² PIPS or surface barrier detectors.

Calibration

The Alpha spectrometer is calibrated with the Po-209 and Po-210 peaks. Resolution is variable and depends upon plating chemistry but the peaks are typically well separated. A small number of counts from the Po-210 peak do stretch down into the Po-209 ROI but they are not significant enough to account for. Peak shift is determined on every sample. If necessary, the 44 channel ROI for each peak is repositioned such that less than 4 empty channels are present on the high energy end of each ROI. Pulser is available but not employed. If the high energy end of peak is not well defined, the planchette is recounted to detect/correct for instrument drift. If necessary, the sample will be reprocessed completely.

Data Handling

The data are then entered into an Excel97 spreadsheet which plots the activity vs. depth (actually cumulative mass) and which also allows modelling the data to select the most probable sedimentation accumulation rate. The Po-209 counts allow precise measurement of the overall chemical yield and thereby permits calculation of the specific activity of the Po-210 in the original sediment sample. The Appleby and Blais publications (see below) are useful references describing the modelling procedures.

Tracer recovery is normally variable. Recoveries of +/- 50 % of core mean are considered to be acceptable. Recoveries outside of these limits will be examined and samples may be repeated to determine whether the difficulty stems from a processing problem or a poor sample.

Detection Limit

The minimum detection limit (MDL) for a 0.5 g sample is between 0.1 – 0.2 DPM Po-210/g dry sample at a 95 % confidence level for a 30,000 sec counting time. This can vary slightly and depends upon the detector and the recovery efficiency of each sample.

Quality Assurance

A duplicate is run every 10th sample provided sufficient material is available. A blank is run every 20th sample. Detector blanks are run every 90 days. All data are reviewed by chief scientist before release. Sample duplicates will be repeated if different by > 3 X mean SD of the duplicates. Laboratory Control Samples (LCS) are not normally run for accuracy determination because only the relatively loosely bound Po-210 (of atmospheric origin) is being targeted for measurement. It is expected that Po-210 of geological origin would be less completely recovered using the specified methods.

Laboratory duplicate samples will be prepared and analyzed (if there is sample volume available) at a frequency of one per twenty samples to check the precision of the methods. If the LD is outside criteria (for accuracy and precision as specified in the QAPP), perform additional duplicate analyses. If still unable to meet QA criteria, contact client.

References

1. Eakins, J.D. and R.T. Morrison. 1978. A new procedure for the determination of Lead-210 in lake and marine sediments. *International Journal of Applied Radiation and Isotopes*. 29: 531 - 536.
2. Appleby, P.G. and F. Oldfield, 1978. The calculation of Lead-210 dates assuming a constant rate of supply of unsupported Pb-210 to the sediment *Catena* 5: 1-8
3. Flynn, W.W. 1968. The determination of low levels of Polonium-210 in environmental materials. *Anal. Chim. Acta* . 43: 221 – 227.
4. Blais, Jules M., Jacob Kalff, R. Jack Cornett & R. Douglas Evans. 1995. Evaluation of Pb-210 dating in lake sediments using stable Pb, Ambrosia pollen, and Cs-137. *J. Paleolimnol.* 13: 169 – 178.

Overview of Cs-137 Measurement in Sediment Core Sections by Gamma Spectrometry Using a HPGe (High Purity Germanium) Coaxial Detector as Performed at Flett Research Ltd., Winnipeg, Canada (2008)

Field Sample Collection:

Samples should be collected in clean jars. The jars should be waterproof so that no water is lost from the samples – try to keep the rims of the jars clean when closing so that no particles are on the sealing surfaces. If Pb-210 analyses are also being performed, avoid loss of water from the core slices as this will affect the bulk density which in turn will affect the Pb-210 dating calculations. The samples do not require special storage conditions. Holding times are unlimited.

Scope:

Cs-137 is a product of nuclear fission not found in the environment prior to the first above ground nuclear bomb test in 1945. Subsequent above ground testing of fusion bombs in the early 1950's served to introduce larger quantities of Cs-137 high into the atmosphere and cause widespread distribution of the radioisotope around the earth. Cs-137 is most easily measured by counting the gamma emissions at 662 KeV that are emitted in 85.2 % of the decays. The gamma radiation is relatively strong and therefore penetrates through several centimeters of sediment material with little attenuation. For our purposes, self absorption of the gamma radiation by the sediment material is considered to be similar to that occurring in the spiked clay reference material employed for calibrating the counting efficiency. The detection limit is on the order of 0.5 DPM/g for an 80000 second counting period when measuring 10 g of sample.

Cs-137 has a half life 30 years and therefore even the earliest sediment Cs-137 is expected to be present at a minimum of 25 % of the original activity when it entered the environment. Nuclear atmospheric fallout records indicate that Cs-137 deposition significantly increased about 1955, initially peaked in 1959, decreased and then peaked at much higher levels in 1963. A rapid drop in Cs-137 inputs occurred after 1963 due to the above ground nuclear test ban, and by 1969, inputs of the isotope were at similar levels to those in 1954.

Overview:

Our HPGe gamma detector assemblies are club shaped and designed to operate vertically, with the narrow part sitting in a dewar of liquid nitrogen that serves to cool the upper part containing the HPGe crystal. The heat is pulled from the crystal by a thick copper rod that runs down the length of the cryostat into the dewar. The detector crystal sits in a 3 inch diameter cylindrical casing that projects about 12 inches vertically above the dewar. We have constructed a 2 inch thick lead castle around the detector so that detection of external radiation from the environment is minimized. The samples sit on the top of the detector, similar to a cookie on a small table. Typically, samples placed directly on the detector will weigh less than 100 g and have smooth upper and lower surfaces. Access to the detector (for changing samples) is gained by removing the lead castletop (approx. 40 lb).

The shape and placement of the sample in relation to the detector are very important and significantly affect the counting efficiency. Ideally the shape and placement of the sample and calibration standard should be identical. However, in actual practice, the amount of sample available is often variable and therefore a set of calibration standards of various sizes needs to be employed. All samples and standards that are analyzed by HPGe detectors are cylindrical shapes of 2.187 in. diameter. Only the thickness of the cylinder varies. Four different thicknesses of NIST Cs-137 spiked clay reference material are used to establish the counting efficiency as a function of sample thickness. A polynomial regression of the four point efficiency curve yields an equation which is used to determine the Cs-137 counting efficiency for each sample.

Wet sediment samples are homogenized in sample container, sub-sampled for bulk density if Pb-210 analyses are also being done, with the remainder dried to constant weight at 48 - 60 C and then ground with mortar and pestle. The density of samples can be highly variable and therefore all samples are compressed at about 3000 psi in a special hydraulic press that produces pancakes of 2.187 in diameter of maximum and relatively similar density. The pancakes are of the best possible counting geometry and permit the highest counting efficiencies. The pancakes are often durable enough to be placed directly into our standard 2.178 in diameter aluminum planchet (sample cup). In the case of very sandy samples, the press procedure does not yield a monolithic pancake and it is necessary to pour the sample into a planchet and tamp it flat and level with a cylinder of the same diameter. The thickness of all samples is recorded for later use in the calculation of counting efficiency. Standards of known thickness and weight are created in exactly the same manner but are hermetically sealed to prevent any possible leakage of the material onto the detector. Since the standards are created only once but used many times, an additional thin polyethylene membrane encloses the standards to increase the durability.

At the beginning of a sample batch, a gamma spectrum for a CANMET Th-U ore reference material standard is acquired with the HPGe detector and associated software. This spectrum allows energy calibration of the detector. The energy calibration is done 295.2, 352, 609.4 and 1120.4 KeV or other prominent peaks in the U-Th spectrum. The ROI for the Cs-137 is set around the Cs-137 standard peak. The spectrum span is 2507 KeV for GEM and 4658 KeV for GMX detectors. Next a series of 4 different thicknesses of NIST Cs-137 spiked clay standard are used to determine the counting efficiency of the detector for Cs-137 in samples of varying thickness. The spectra are also used to confirm the precise spectral position of the 661.6 KeV Cs-137 peak. Detector resolution is determined using the highest Cs-137 standard. The FWHM is displayed automatically by the Gamma Vision 5 software. The GMX detector (at 661 KeV) has a FWHM resolution of about 1.8 KeV; the GEM detector has a resolution of about 3 KeV. Next, an 80,000 sec. background spectrum is run on an empty planchet to determine background peak intensities, which for Cs-137 is normally undetectable. Subsequent background spectra of 80,000 sec. duration are normally acquired as a matter of course during analysis of cores i.e. in most cores it is usual to obtain some samples that contain no detectable Cs-137. In the case that Cs-137 has been detected in all the previous 30 samples, then a detector blank of 80,000 sec will be acquired to check the detector for contamination. Provided no contamination is found, counting will continue. If contamination is found, then the detector assembly will be cleaned and re-evaluated for contamination. Data from the previous 30 samples will be examined to determine a possible contamination source and samples recounted where necessary to assure accurate measurements are reported. The counting efficiency as a function of sample thickness is redetermined about every 90 days and applied to subsequent samples. Data taken from the HPGe system is entered onto EXCEL97 spreadsheets which calculate the Cs-137 detector efficiency, and finally, the original activity of Cs-137 in

the sample at the time of sampling.

ROIs of about 3 FWHM are employed to allow for some drift of peak position across the energy spectrum without losing counts. If the always present background peaks at 186 KeV (U-235/Ra-226), 510.7 KeV (Tl-208) or 1460.8 KeV (K-40) are seen to drift further than 1.5 KeV from their known values, then a new energy calibration will be performed.

When calibration is conducted as outlined above, it is extremely unlikely that a false positive will occur from another nuclide. The most likely false positive would be from Bi-214 at 665.6 KeV but this peak is seldom seen in the sediments we have examined. If visual inspection of the Cs-137 ROI indicates possible overlap with the 656.6 photopeak, then we would determine the Bi-214 activity at 609.4 KeV and subtract the appropriate number of counts from the Cs-137 peak.

Detection Limits

The detection limit is on the order of 0.5 DPM/g (95 % confidence) for an 80,000 sec counting period when measuring 10 g of sample. If sufficient sample volume is available, and upon direction from the client the detection limit may be lowered in certain samples by counting longer and/or by counting larger samples.

Quality Assurance

Characterized material from the EML proficiency sample program for Cs-137 in soil and plant materials will be used as a Laboratory Control Samples (LCS). The LCS will be prepared in the same geometry as the field samples to check the accuracy of the method. The frequency will be one per twenty samples. If the LCS is outside of the control limits (for accuracy and precision as specified in the QAPP) the laboratory will check instrument parameters, sensitivity and linearity and correct any problems. The laboratory will also contact the client and maintain written documentation.

Laboratory duplicate samples will be prepared and analyzed (if there is sample volume available) at a frequency of one per twenty samples to check the precision of the methods. If the LD is outside criteria (as specified in the QAPP) and the LCS is within criteria the laboratory will contact the client.