ONONDAGA LAKE

MICROBEAD MARKER WORK PLAN FOR MONITORING NATURAL RECOVERY IN SMU 8
Syracuse, New York

Prepared For:

Honeywell

5000 Brittonfield Parkway
East Syracuse, NY 13057

Prepared By:

PARSONS

290 Elwood Davis Road, Suite 312
Liverpool, New York 13088
(315) 451-9560 Tel
(315) 451-9570 Fax

ANCHOR ENVIRONMENTAL, L.L.C.

23 South Wenatchee Avenue, Suite 120
Wenatchee, WA 98801
(509) 888-2070 Tel
(509) 888-2211 Fax

ENVIRONMENTAL TRACING SYSTEMS

The Coach House Bannachra
Helensburgh G84 9EF UK
+44 (0)1389 711001 Tel
+44 (0)1389 850551 Fax

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LIST OF ACRONYMS

CPOI	Chemical Parameters of Interest
ETS	Environmental Tracing Systems, Ltd. (Helensburgh, Scotland, UK)
FS`	Feasibility Study
GPS	Global Positioning System
mg/Kg	milligrams per kilogram
MNR	Monitored Natural Recovery
MSDS	Material Safety Data Sheet
NYSDEC	New York State Department of Environmental Conservation
PDI	Pre-Design Investigation
PEC	Probable Effect Concentration
ppm	parts per million (1 ppm is the same as 1 mg/Kg)
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 is approximately equivalent to 0.4 inch, and one inch is approximately equivalent to 2.5 centimeters.

MICROBEAD MARKER WORK PLAN FOR MONITORING NATURAL RECOVERY IN SMU 8

1.0 INTRODUCTION

This work plan describes field tests and placement of microbead markers in Sediment Management Unit (SMU) 8 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 is the deeper water portion of Onondaga Lake where water depths exceed 9 meters (30 feet), which is also referred to as the profundal zone of the lake where waters become vertically stratified each year from late spring until fall. Within the monitoring and contingency approach, the term "tracer" was used to denote what is now being called microbead marker (so as to not be confused with dye tracer work to be done to address a different objective).

The primary purpose of this work plan is to describe the utility and implementation of microbead markers in SMU 8. In particular, results from the pre-mobilization field tests described in Section 4.1 will have a significant effect on marker implementation. Post 2008 activities described in this work plan will be re-evaluated, based on the results of the 2008 pre-mobilization field tests, to determine if the post 2008 activities remain appropriate.

The purpose of placing and monitoring a microbead marker is to measure ongoing sedimentation rates as part of the evaluation of monitored natural recovery (MNR) in SMU 8. MNR is one of the elements of the remedy for Onondaga Lake specified in the Record of Decision for the Onondaga Lake Bottom Subsite (NYSDEC and USEPA, 2005). This microbead marker work will help determine, along with other monitoring and modeling efforts, if any additional remedial measures will be needed in SMU 8 in the future, such as additional thin-layer capping.

The remedy for Onondaga Lake, as specified in the Record of Decision, includes MNR in SMU 8 to achieve the mercury probable effect concentration (PEC) of 2.2 milligrams per kilogram (mg/kg or part per million) in the lake's profundal zone (where water depths exceed 9 meters or 30 feet) and to achieve the bioaccumulation-based sediment quality value (BSQV) for mercury of 0.8 mg/kg on an area-wide basis, within 10 years following the remediation of upland sources, dredging and/or isolation capping of littoral sediment, and initial thin layer capping in the profundal zone. Dredging is to begin during May 2012 and be completed within four years. Capping will begin during this four-year dredging period and is anticipated to be completed within two years of the completion of dredging. Baseline monitoring of natural recovery will begin once dredging and capping are complete. In the interim, Honeywell will track the course of natural recovery during the remedy design and construction phase through

continued monitoring and MNR evaluations and, as needed, contingency actions will be implemented if it appears that MNR is not on the expected course. Sediment from the top 15 centimeters (i.e., 6 inches) was sampled for Honeywell in November 2007 at 26 locations and analyzed, as reported in the Phase III Addendum 6 Data Summary Report (Parsons, 2008b). Additional sediment sampling will be conducted in the future. Radioisotope and mercury cores are being planned for 2008 to record recent trends in sedimentation and sediment chemical concentrations as an update of information obtained from prior coring efforts in SMU 8. A separate work plan is being prepared for this additional coring effort. Microbead marker deployment is intended to track the course of MNR during the design and construction phases of the remedy and also during the MNR period following construction through 2027.

2.0 MICROBEAD MARKER OBJECTIVES

Placement of a microbead marker is consistent with a mechanistic/predictive approach to MNR evaluation, and data associated with marker placement would be primarily used to better understand the mechanism of sedimentation and its dynamics. These data would be used to adjust/refine MNR model input parameters and update model predictions.

Microbead marker data can be used to quantify sedimentation rates and mixed layer depths to provide refinements to the MNR assessment and predictions described in the Feasibility Study Report for Onondaga Lake (Parsons, 2004). Use of microbeads to further assess bioturbation in the littoral (shallower) zone of Onondaga Lake is being evaluated separately from this microbead work effort for SMU 8.

Deploying microbead markers at specific locations throughout SMU 8 would help establish a new marker appropriate to assess surface sediment changes over the next 15 to 20 years. The average sediment settling rate in SMU 8 since 1986 is approximately 0.3 to 0.6 centimeter per year at the South Deep station based on available data summarized in the Feasibility Study Report (Parsons, 2004, Appendix N). Given this average annual sedimentation rate generally observed in SMU 8 and resolution of 0.5 to 1 cm available for sampled core intervals, it would likely take at least two years to quantify a preliminary sedimentation rate based on sampling of such markers which is consistent with the schedule for monitoring natural recovery presented in the Phase III PDI Addendum 6 work plan (Parsons, 2008a). This sediment sampling resolution of 0.5 to 1 cm will be further assessed during the pre-mobilization field tests.

A microbead marker can also provide a way to measure mixing due to the combined effects of bottom lake water circulation, bioturbation, and/or ebullition. Bottom water circulation measurements made in SMU 8 during late September and early October 2007 show mean currents at the Saddle between the South Basin and the North Basin of 3.0 centimeters per second and mean currents at South Deep of 1.4 centimeters per second over the sampling period with clear evidence of a seiche. Effects of water currents on vertical sediment mixing have not yet been assessed. Bioturbation can only be taking place if sediment organisms are present that cause vertical movement of SMU 8 sediment. Bioturbation is likely not taking place in SMU 8

at present, because anoxic conditions exist in the hypolimnion during summer stratification that prevent biological activity. Ebullition (release of gas upward from underlying sediment), like bioturbation, may encourage vertical mixing of SMU 8 sediment. However, measurements of ebullition in SMU 8 during 2006 and 2007 by Upstate Freshwater Institute show that ebullition was 80% lower during 2007 than during 2006 (Upstate Freshwater Institute, 2008a). Significant water circulation, bioturbation and/or ebullition could limit the ability to quantify sedimentation rates over time as the vertical position of a placed marker becomes progressively more mixed.

3.0 MICROBEAD MARKER DESCRIPTION AND APPLICATION WITHIN SMU 8

Two different fluorescent microbead markers with different densities, sizes, and/or shapes may be placed onto the surface of SMU 8 sediment during the spring or early summer of 2009 . Pre-mobilization field tests described in Section 4.1 will provide additional information for determining feasibility of effective marker sampling and placement and also whether placing two markers is warranted. One potential marker (hereafter called Marker A), would mimic the SMU 8 sediment by being a fine (clay-silt)-sized microbead. The more Marker A would be disturbed by processes including bioturbation, bottom currents or ebullition, the more Marker A would become mixed within the surface sediment making it more difficult to quantify sedimentation rate. In addition, being silt-clay in size, Marker A particles would not be visible and detection of the marker would need to be by collection of a bed sediment sample followed by analysis at the ETS laboratory in Scotland. A second potential marker (hereafter called Marker B) would have a much larger (sand) grain size and perhaps be more plate-like in shape having a much higher erosion shear stress and, therefore, being less prone to vertical mixing than Marker A. Marker B would have an added advantage of being able to be detected visibly at the project site, either *in situ* using an underwater camera or by collecting a sediment sample for visual observation.

Pending results from the pre-mobilization field tests described in Section 4.1, the characteristics of the two potential microbead markers could be as follows.

- Marker A consisting of microbeads each with a particle diameter distributed relatively evenly between 2 to 60 microns (clay – silt) and a dry density of 2.6 grams per cubic centimeter (i.e., 160 pounds per cubic foot) based on the results from particle size and specific gravity analyses completed on 11 surface sediment samples collected throughout SMU 8 during November 2007 (Parsons, 2008b). The purpose of this marker is to mimic movement of SMU 8 sediment and, therefore, be subject to any activity that SMU 8 sediment may undergo (e.g. mixing and resuspension due to bottom currents, bioturbation, and/or ebullition).
- Marker B consisting of microbeads with a particle diameter of 200 to 300 microns (or 0.2 to 0.3 millimeters). Other properties, such as specific gravity and particle shape may differ from Marker A properties as needed so that Marker B would not settle differently on average from SMU 8 sediment or Marker A.

To quantify mixed layer depth, Marker A must not move faster or slower than SMU 8 surface sediment. Due to the small size of the SMU 8 sediment particles (silt and clay), Marker A would not be visible. Marker A would provide basis for quantifying mixed layer depth. If significant vertical mixing is taking place, the boundary for sedimentation would likely 'blur' as Marker A becomes mixed with SMU 8 sediment. Marker B, on the other hand, would be much less likely to be affected by natural vertical mixing due to its larger particle size. Marker B could also be visible and, therefore, observable from collecting and examining sediment samples without conducting laboratory analyses of the marker following placement.

Both microbead markers would be manufactured in Scotland by Environmental Tracing Systems, Ltd, using non-toxic polyester-based polymers that are known to be highly resistant to photodegradation and biochemical breakdown even when exposed to the natural elements. The markers would include a naturally-occurring mineral such as barium sulfate to adjust physical properties of the marker (such as dry density) as needed to meet project objectives. The two markers would be manufactured with the same fluorescent pigment label. The fluorescent pigment will comprise approximately 3 percent by weight of the final microbead marker composition. The fluorescent chemical signature will be thermoset into the pigment polymer and will represent less than 5 percent by weight of the overall pigment. With this design, if the microbead does break down gradually over time, the pigment would still retain its fluorescent property. Additional details of the polymer and the method of application will be provided following the pre-mobilization field tests described in Section 4.1. A material safety data sheet, which includes aquatic toxicity information, is provided in Appendix A. After the specific marker(s) is identified from the pre-mobilization field tests, one or more additional material safety data sheets will be provided as warranted. Marker properties will be further reviewed with NYSDEC once the specific markers are identified and prior to marker placement.

Baseline physical process data are being collected as part of other Honeywell SMU 8 work efforts to help explain, as needed, any unusual behavior of the microbeads. Conductivity, temperature, and turbidity-surrogate parameters are being measured in the water column by Upstate Freshwater Institute from mid-April through November at various SMU 8 locations and water depths. Likewise, ebullition is being quantified based on the volume of total gas collected (Upstate Freshwater Institute and Syracuse University, 2008).

Near-bottom water velocities will also be measured as was done during the fall of 2007 as part of the sediment incubation-related field work. Near bottom water velocities will be measured adjacent to a microbead labeled location in the same manner described in the work plan for sediment incubations (Parsons, 2007b).

4.0 MICROBEAD MARKER FIELD COMPONENTS

4.1 Pre-Mobilization Field Tests

ETS and Parsons will perform field tests prior to marker placement to help confirm that available microbead markers will serve as suitable markers of sedimentation and vertical sediment mixing for approximately 20 years following marker placement. To confirm the type, size, and density of the markers to be manufactured and released, a microbead pre-mobilization field test will be conducted during the fall of 2008. The four tasks to be carried out by ETS during the microbead pre-application field survey are as follows:

a. Collect approximately 5 to 10 sediment cores (in 4 to 6-inch diameter clear core tubes) from each of the designated SMU 8 microbead marker locations. Keep the cores in a vertical position and undisturbed as much as reasonably possible while transporting and staging the sediment cores from the boat to a work area onshore. Minimize and measure sediment compaction during sample transport and processing. Handle samples carefully by slowly siphoning off most but not all of the overlying water, maintaining the sampling tubes in a vertical position until the samples are segmented into sections, and minimizing movement of the sampling tubes on the boat and at the shoreline where the samples will be processed. Once the core tubes are onshore, use microbead marker material available from past ETS projects to simulate (within the core tubes) placing a layer of microbeads on top of the sediment cores. Subsequently, release SMU 8 surface sediment into the sediment cores at a rate in excess of natural sedimentation levels to allow the SMU 8 sediment to settle on top of the microbead marker. Observe and/or measure for approximately 24 hours whether the integrity of the beads is maintained or whether mixing takes place. ETS will use particles with a range of densities and sizes to assess whether having a density or size that is too high will affect the settling and/or consolidation of the material. Of particular interest will be how the microbeads position themselves with the top of the sediment. This is the reason behind trying a range of particles both similar to and different from Marker A and Marker B. Either a TracerCam will be used on site to determine marker presence or some or all of the core sub-sample will be sent to the ETS laboratory in Scotland for analysis and cross-referencing. The ETS laboratory based analysis is more accurate than the field TracerCam method.

b. Collect sediment grab and core samples from representative lake tributaries (including Ninemile Creek and Onondaga Creek) and also lake sediment samples from nearshore and from SMU 8. Review locations for tributary samples with NYSDEC prior to collecting the samples as part of the pre-mobilization field tests. Determine background fluorescence using a fluorescence microscope (see Appendix B) to assess whether any fluorescent particles or species are present in those sediments that may interfere with the measurement and/or detection limits of the fluorescent microbead markers. Given historical land use in the lake watershed, it is important that this assessment be made in advance of manufacturing the microbead markers to ensure the fluorescent spectra of the microbead markers are measurable without significant interferences.

c. Test placing mimic marker material in a 1 to 2 millimeter thick layer on top of sediment at one or more representative SMU 8 locations by mixing with SMU 8 sediment an inert golden or white natural sand and a light/white fine inert mineral (such as a talc or fluorite mineral) to mimic Marker A and Marker B. The mimic markers will have properties similar to Marker A and Marker B, although the characteristics of Marker A and Marker B may change as a result of the pre-mobilization field tests. If marker characteristics change as a result of the pre-mobilization field tests, marker characteristics will be modified accordingly and reviewed with NYSDEC prior to implementation. Both mimic markers will be placed in a test area of SMU 8 to be identified using the method described below in Section 4.3. A sufficient quantity of mimic marker B will be placed to allow visual assessment without the marker being masked by the sediment. The marker placement method may need to be modified based on pre-mobilization field test results. Following placement, the marked sediment will be core sampled to assess the homogeneity of the marker layer over the test bed and also to validate the marker sampling procedure. In the short term, the golden or white sand and the light/white mineral will likely be visibly distinguishable from SMU 8 sediment.

d. Test use of vibracore and/or box core sediment sampling equipment to assess their utility to quantify sedimentation rate (see Section 4.4).

Cores collected and then segmented as part of the pre-mobilization field tests will be shipped to Scotland following onsite testing to fine tune a method of sub-sampling so a more detailed sample processing SOP can be developed and implemented as needed.

Once results from these pre-mobilization field tests are available, four important items for this work scope will be more specifically identified and reported to NYSDEC: (1) whether to place one or two microbead markers in SMU 8; (2) more specific marker characteristics; (3) marker quantity and placement method; and (4) more specific information about analyses to be used to determine marker presence. Additional information will be submitted to NYSDEC following the pre-mobilization field tests. Following submittal of results and recommendations from the pre-mobilization field tests, feedback from the agencies will be needed before manufacturing of the microbead marker(s) can begin.

Following the manufacturing of the marker(s), characterization tests would be conducted prior to placement. These tests would include laboratory testing of marker particle size and specific gravity to compare with SMU 8 sediment characterization data.

4.2 Mobilization and Marker Positioning

Marker preparation would consist of mixing marker and SMU 8 sediment onshore within an area approximately 30 m by 30 m in size provided with potable water electrical power, and a mixer to be used to wet both markers. The mixing area ground surface would be covered with a tarpaulin. A sufficient quantity of sediment would be collected from SMU 8 prior to marker placement to mix with the sediment markers at a ratio of approximately one sediment particle to

every two microbead particles. SMU 8 sediment and the markers need to be mixed in order to ensure the microbeads adsorb any available organic charge in a well mixed manner. Once wetted and mixed, the marked sediment would be stored temporarily in sealable buckets or drums, washed down, and then loaded onto the boat to be used for marker placement.

As has been the case for previous Honeywell pre-design investigation efforts in Onondaga Lake, the boat to be used to place the microbeads would be able to work safely throughout SMU 8. The boat would also include a mechanized A-frame capable of handling a vibracore or box core sampler and the underwater marker placement equipment described in Section 4.3 below.

Microbead marker would be placed at a total of eight locations as described in Table 2 and shown in Figure 1. These locations would not be co-located with previously-sampled locations and provide widespread distribution throughout SMU 8 with more locations near the sources of chemical parameters of interest in the southern half of the lake. Each of the sample locations would be in the vicinity of SMU 8 locations sampled during 2007 as indicated in Table 2.

Vessel positioning and the determination of microbead marker placement locations would be accomplished utilizing a Trimble geographic positioning system (GPS) receiver (or equivalent) interfaced with a navigation and data logging system. Differential GPS coordinates and water depth would be reported for each marker placement location.

4.3 Marker Placement

One or two microbead markers would be placed on SMU 8 sediment in a controlled manner and distributed uniformly over a pre-determined bottom area at each location. To achieve effective, uniform distribution, microbeads would be pumped down a hose to a horizontal bar approximately 3 to 5 meters in length which would be towed 2 to 3 meters above the SMU 8 sediment mudline. The hose would have multiple discharge ports mounted along the length of the bar, ensuring that the microbeads are spread over the bar width in a uniform manner. As described in Section 4.1 of this work plan, ETS would test this equipment and the placement method prior to the release of any microbeads to ensure the placement method works successfully and achieves the intended marker distribution. Other methods are available for placing microbead markers, but the placement method described herein should be able to be effectively implemented in Onondaga Lake. As with marker particle characteristics, any revision to the maker placement method identified as a result of the pre-mobilization field tests would be reviewed with NYSDEC prior to marker placement

In order to pump the marker and sediment down the hose to the multiple discharge bar described above, the material would need to be fluidized. The marker and sediment would be fluidized by adding marker into a larger barrel on board the work boat to which water would be added and mixed with the marker. A circulation pump would be used to keep everything in suspension and well mixed in the barrel prior to release. Lake water to be mixed with the marker would be pumped from below the thermocline in order to match the temperature into which the

microbeads are being discharged. Another pump with variable speed would be used to pump the marked sediment slurry down the hose which would split into 5 to 10 nozzles mounted along the bar. Each nozzle would discharge the same volume of marker and sediment. The bar with the nozzles would need to be streamlined so that the bar remains at a suitable height above the sediment and does not yaw or pitch. The boat would need to either proceed along a pre-determined path very slowly or pass multiple times over each marker placement location. One challenge is to have the boat move very slowly and compensate for the limited steerage caused by the bar. As described in Section 4.1, a pre-mobilization field test would be conducted with just the bar and the surrogate golden sand and white mineral markers prior to releasing a fluorescent marker. Any changes to this method of release would be made based on results from the pre-mobilization field tests. The method for marker placement may be revised if an alternate method to uniformly place marker particles is identified as a result of the pre-mobilization field tests.

The size of the marker placement area would be approximately 125 square meters (140 square yards) at each of the eight locations. An area of 125 square meters would allow a sub-area of 3 meters by 3 meters (i.e., 10 feet by 10 feet) to be available for each of 14 future marker sampling efforts without sampling the same sub-area twice. Depending on the extent the microbead markers become compacted, this density of microbead placement would result in an approximate microbead layer thickness of 0.75 to 2.5 millimeters based on a marker particle size of 0.25 millimeter (i.e., Marker B). This density of placement is equivalent to approximately 200 microbead particles per square millimeter (or 130,000 particles per square inch).

4.4 Marker Sampling and Analyses

Within a few weeks after placing both markers, one additional core would be collected from each of the marker placement locations to confirm the homogeneity of the microbeads and compaction or mixing during placement. In addition, core samples would be collected during the same timeframe from locations immediately surrounding two to four of the marker placement locations to confirm that there has been little spread of the microbeads beyond the microbead locations as a result of placement.

To further confirm placement effectiveness and assess effects of lake turnover and winter mixing, a single core sample would be collected from each of the eight marker placement locations approximately 4 to 6 months following marker placement. In order to assess variability, duplicate samples would be collected at two of the eight locations in order to provide information on variability and sampling error. Marker sampling would be repeated approximately 12 months following marker placement at four of the marker placement locations to establish the effects of one year of lake stratification. Subsequently, marker sampling would be conducted during 2011 and every three years thereafter as presented in the MNR Work Plan (Parsons, 2008a).

Unless otherwise stated, the sampling and analysis activities described in this work plan will be conducted in accordance with the procedures outlined in the Phase I PDI Work Plan (Parsons, 2005a). Based on results from the pre-mobilization field tests, a vibracore or box core sampling device will be used to collect sediment samples from the top few centimeters in SMU 8. Both sampling devices have been used successfully as part of previous pre-design investigation work with Onondaga Lake sediment.

Vibracore or box core sampler penetration at each sample location will be to a depth at least 18 inches below the mudline to ensure successful retrieval of an intact core of the top few inches of sediment. SMU 8 shallow sediments are generally very soft and fluffy. If shallow 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.

A sufficient number of samples will be collected to meet laboratory analysis requirements. Sampling tubes will then be capped on both ends and stored vertically on the boat and on ice prior to processing onshore and then shipment of processed samples to the laboratory in Scotland. Sample processing onshore will include segmenting each core into vertical intervals using the sample processing SOP from the 2007 MNR sampling effort (Parsons, 2008a). 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).

The sediment depth interval to be analyzed will be refined as needed following the pre-mobilization field tests. At this time, a sediment thickness of 0.5 centimeter appears to be sufficient for the marker detection analyses that need to be conducted.

Marker A would be detected by ETS in Scotland using fluorescence microscopy and/or analytical flow cytometry and image analysis providing a count and microbead particle distribution if required. Marker B would be detected by ETS in Scotland using fluorescent magnification and microscopy. Laboratory procedures to be used are presented in Appendix B. Analyses for the presence of the marker(s) will be conducted in ETS' ISO 9001 accredited laboratory, based in Scotland. ETS' analytical techniques have been proven to be accurate and reliable on more than 60 sediment transport studies. The analytical techniques and monitoring equipment to be used will be selected and optimized to achieve needed sensitivity and detection limits.

Upon receipt of each sample onshore, the sample will be extruded using the procedure implemented with the Phase III MNR samples collected in SMU 8 during November 2007 (Parsons, 2008a). Alternatively, two-part core tubes may be used that interlock and can be easily split into two parts leaving an exposed surface of sediment avoiding the need for extruding the sediment. The boundary for the coarse fraction will then be measured and recorded and then a sub-sample will be obtained from the core sample based on the boundary of the coarse fraction by cutting layers with a stainless steel knife and discarding any fragments. Following shipment of the samples to the ETS lab in Scotland, each sub-sample of sediment will be weighed, labeled, and dried to a constant weight noting weight loss due to drying. Each sub-sample will be analyzed using either fluorescence magnification or microscopy to count the respective number of coarse and fine microbeads. Counts will be expressed as numbers of marker particles per dry gram.

4.5 Post Marker Placement Control Check

In addition to collecting sediment samples from SMU 8 following marker placement, a control check would be conducted for one or both of the microbead markers. The purpose of this control check is to confirm marker longevity over time. Sufficient quantity of SMU 8 surface sediment would be collected and placed in 20 tubs or buckets. The control check would be set up as follows: (1) a layer of SMU 8 surface sediment would be placed in the 20 tubs or bucket; (2) a 1 to 2-millimeter thick layer of microbeads would be placed on the sediment surface; (3). a small additional quantity of SMU 8 surface sediment would be added to each tub to represent sediment that is settling in SMU 8 over time; and (4) the tubs or buckets would be stored in a cool, dark area at ETS away from light that could breakdown the marker over time. During each year following marker placement until the MNR time period is complete, a sediment-marker sample would be collected from one of the tubs or buckets, and a determination would be made as to the extent the two microbead markers remain intact in SMU 8 and detectable with unchanged properties.

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). 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 (such as microbead handling) 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 final Subcontractor Safety Plan and the final Project Safety Plan will be available for viewing at the support zone onshore and on the work boat prior to the commencement of fieldwork associated with the microbead marker evaluation/investigation.

Microbead marker work crew members will need to have successfully completed 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 microbead marker 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 procedures will be conducted in accordance with the SOP included in Appendix B. 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.

ETS will conduct the laboratory analyses in Scotland. Analytical data generated during this investigation will be reviewed for usability. Following laboratory analyses, the data will be assessed to determine whether the extent of microbead markers in each of the marker locations is relatively uniform.

Once the data have been checked, 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, the status of the microbead marker placement and monitoring work will be documented in a report at the same time the other SMU 8 monitored natural recovery field efforts planned for 2008 are documented.

8.0 REFERENCES

Honeywell, Parsons, and O'Brien & Gere, 2007. Honeywell Syracuse Portfolio Health and Safety Program. February 15, 2007 (or a more recent version if available in the future).

NYSDEC and USEPA Region 2. 2005. Record of Decision. Onondaga Lake Bottom Subsite of the Onondaga Lake Superfund Site. July 2005.

Parsons, 2004. Feasibility Study Report for Onondaga Lake. Prepared for Honeywell. November 2004. (MNR is described in detail in Appendix N.)

Parsons. 2005a. Onondaga Lake Pre-Design Investigation: Phase I Work Plan. Prepared for Honeywell. September 2005.

Appendix A Phase I Sampling And Analysis Plan

Appendix B Quality Assurance Project Plan

Appendix C Project Safety Plan Updated March 2007.

Parsons. 2005b. Onondaga Lake Pre-Design Investigation: Standard Operating Procedures. Prepared for Honeywell. November 2005

Parsons. 2006. Onondaga Lake Pre-Design Investigation: Phase II Work Plan. Prepared for Honeywell. September 2006.

Parsons, 2007a, *Onondaga* Lake Pre-Design Investigation: Phase III Work Plan. Prepared for Honeywell, Morristown, New Jersey. Syracuse, New York. May 2007.

Parsons, 2007b. Work Plan for Onondaga Lake SMU 8 Sediment Incubations and Supporting Studies. Prepared for Honeywell, Morristown, New Jersey. Syracuse, New York. Revised December 2007.

Parsons, 2008a. Onondaga Lake Pre-Design Investigation: Phase III Work Plan – Addendum 6. SMU 8 Sampling to Monitor Natural Recovery. Prepared for Honeywell. Prepared in association with Exponent and Anchor Environmental. Revised January 2008.

Parsons, 2008b. Onondaga Lake Pre-Design Investigation: Phase III Addendum 6 Data Summary Report. Prepared for Honeywell. Prepared in association with Exponent and Anchor Environmental. March 2008. Draft.

TAMS Consultants and YEC, Inc., 2002. Onondaga Lake Remedial Investigation Report. Prepared for the State of New York Department of Environmental Conservation. Based on an original document prepared by Exponent for Honeywell. December 2002.

Upstate Freshwater Institute, 2008a. Presentation by Dave Matthews to the Onondaga Lake Baseline and Long-Term Monitoring Technical Work Group. Syracuse, NY. January 17, 2008.

Upstate Freshwater Institute, 2008b. Onondaga Lake SMU 8 Dye Tracer Analysis Prepared for Honeywell Prepared by Upstate Freshwater Institute. Syracuse, NY April 2008. Draft.

Upstate Freshwater Institute and Syracuse University, 2008. Onondaga Lake Baseline Monitoring Book 1. Deep Basin Water and Zooplankton Monitoring Work Plan for 2008. Prepared for Honeywell. Prepared by Steven Effler and C. Driscoll. March 2008. Draft.

USEPA. 2005. Contaminated Sediment Remediation Guidance for Hazardous Waste Sites. EPA-540-R-05-012. Office of Solid Waste and Emergency Response. OSWER 9355.0-85. December 2005.

TABLE 1

SMU 8 MICROBEAD MARKER PLACEMENT SUMMARY

Marker types	Two markers may be placed: (1) clay-silt particle size and resembling SMU 8 sediment characteristics; and (2) sand particle size but also resembling settling characteristics of SMU 8 sediment without possible vertical mixing due to water movement, bioturbation, and/or gas ebullition.
Marker objectives	Marker (1) would assess combined effects of vertical mixing and sedimentation Marker (2) would assess sedimentation without vertical mixing
Marker placement locations and size	Eight each approximately 100 square meters in area (see Figure 1).
Sample collection method	Vibracore™ or box core sampler with a 3.5-inch inside diameter polycarbonate tubes.
Sample depth intervals for analyses (in centimeters below the mudline)	To be determined from pre-mobilization test results. Samples will be segmented by sediment depth onshore prior to shipment.
Field observations to be recorded	Water surface elevation and depth to top of sediment.
Laboratory analyses	For Marker A: fluorescence microscopy and/or analytical flow cytometry. Image analysis can also be sued to provide a count and microbead particle distribution if required. For Marker B: fluorescent magnification and microscopy
Quality assurance	Post marker placement control check (see Section 4.5) Field duplicates as appropriate

TABLE 2
DESCRIPTION OF MICROBEAD MARKER PLACEMENT LOCATIONS

SMU 8 Location	Vicinity	Basis for Selected Location
North Basin		
OL-MB-80093	North end	Near OL-STA-80067 where 46.6(J) mg/Kg of mercury was measured in sediment at 10 to 15 cm below top of sediment (November 2007).
OL-MB-80094	East Side	Near OL-STA-80069 where 17.3(J) mg/Kg of mercury was measured in sediment at 10 to 15 cm below top of sediment (November 2007).
OL-MB-80095	Center	Near OL-STA-80070 where 32.3(J) mg/Kg mercury was measured in sediment at 4 to 10 cm (November 2007)). Also, adjacent to North Deep and approximately 600 ft from OL-STA-80071 and S90 where a high resolution core was collected during 1992.
Ninemile Creek (NMC) Outlet		
OL-MB-80096	Near SMU 4	Near OL-STA-80073 and in the center of the Ninemile Creek outlet area.
Saddle		
None	-	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-MB-80097	North central	Near OL-STA-80076 where 6.2(J) mg/Kg of mercury was measured at 10 to 15 cm.
OL-MB-80098	West Side (near SMU 3)	Near OL-STA-80079 where 6(J) and 57.1(J) mg/Kg of mercury were measured at 4 to 10 and 10 to 15 cm respectively (November 2007).
OL-MB-80099	Center	Near OL-STA-80081 where 21(J) mg/Kg of mercury was measured at 10 to 15 cm below top of sediment (November 2007).
South Corner		
OL-MB-80100	South Center	Near South Deep and OL-STA-80086.

Lake Outlet
Sawmill Creek
OL-MB-80093
North Basin
OL-MB-80094
North Deep
OL-MB-80095
SMU 5
Bloody Brook
60
OL-MB-80096
NMC Outlet
50
40
30
20
10
Saddle
SMU 5
SMU 4
OL-MB-80097
SMU 8
South Basin
SMU 3
OL-MB-80098
OL-MB-80099
Ninemile Creek
OL-MB-80100
South Deep
South Corner
Geddes Brook
Tributary 5a
SMU 2
SMU 6
SMU 1
East Flume
SMU 7
Harbor Brook

Q:\GIS\GIS_Lake\Phase_IV_PDI\SMU8.mxd

PROPOSED MICROBEAD MARKER PLACEMENT LOCATION

- Proposed Marker Placement Location (OL-MB-800XX)

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

0 485 970 1,940 2,910 3,880 4,850
Feet

NOTES

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

FIGURE 1

Honeywell

Onondaga Lake
Syracuse, New York

SMU 8
Proposed MNR Microbead
Marker Placement Locations

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

APPENDIX A

REPRESENTATIVE MICROBEAD MARKER MATERIAL SAFETY DATA SHEET (MSDS)

This MSDS refers to regulatory mechanisms operating in the United Kingdom because that is where the microbeads would be manufactured.

EEC – European Economic Community - European Directives are numbered and always end in EEC or EC.

CHIP – Chemicals (Hazard Information and Packaging for Supply) Regulations – regulations regarding supply of chemicals.

The UK Health and Safety Executive sets Occupational Exposure Limits.

Needed gloves and goggles are disposable.

Note: The “elevated temperature” referred to in Section 9 is the decomposition point which is above 190 degrees Celsius as noted in Section 8.

MATERIAL SAFETY DATA SHEET

1. Product/Manufacturer's Details:

SERIES NAME:	EcoTrace Yellow A2 – 7 Fluorescent Tracer
APPLICATION:	Particle tracing
MANUFACTURER'S NAME:	c/o ETS Ltd., The Coach House, Bannachra, Helensburgh, Argyll, Scotland, G84 9EF
TELEPHONE:	+44 (0)1389 711001
FAX:	+44 (0)1389 850551
e-mail:	enquiries@environmentaltracing.com
CONTACT:	FRASER TAYLOR

2. Composition/Information on Ingredients

COMPOSITION:	Solid solution of fluorescent dyes in thermoplastic polyester
HAZARDOUS INGREDIENTS:	Does not contain any substances presenting a health hazard within the meaning of the Dangerous Substance Directive 67/548/EEC as amended by the Seventh Amendment 92/32/EEC

3. First Aid Measures

GENERAL:	In all cases of doubt or when symptoms persist, seek medical attention. Never give anything by mouth to an unconscious person.
INHALATION:	Remove to fresh air, keep patient warm and at rest; if breathing is irregular or stopped, administer artificial respiration. Give nothing by mouth. If unconscious, place in recovery position and seek medical advice.
EYE CONTACT:	Irrigate copiously with clean fresh water for at least 10 minutes holding the eyelids apart and seek medical advice
SKIN CONTACT:	Wash skin thoroughly with soap and water or use recognised skin cleaner. Do NOT use solvents or thinners
INGESTION:	If accidentally swallowed give two glasses of water to drink. Do NOT induce vomiting. If symptoms persist seek medical advice

4. Fire Fighting Measures

EXTINGUISHING MEDIA:	Foam, CO_2 powders, water fog
PRECAUTIONS:	Exposure to decomposition products may cause a health hazard (Section 9)

5. Accidental Release Measures

PERSONAL PRECAUTIONS:	Refer to protective measures listed in Section 7. Avoid dust formation. Take precautionary measures against static discharges
METHODS FOR CLEANING UP:	Contain spillage with suitable dust binding materials such as sand/sawdust and dispose in accordance with Section 12. Clean affected areas with water/biodegradable surfactant solution – avoid use of solvents Refer to protective measures listed in Section 7. Avoid dust formation. Take precautionary measures against static discharges
METHODS FOR CLEANING UP:	Contain spillage with suitable dust binding materials such as sand/sawdust and dispose in accordance with Section 12. Clean affected areas with water

6. Handling and Storage

HANDLING:	Avoid dust formation. Take precautionary measures against static discharges
STORAGE:	Store in a dry well ventilated place away from sources of heat and direct sunlight. Keep away from sources of ignition. Keep away from strong oxidising agents and alkaline and acidic materials. Containers, which are open, should be closed and kept upright to prevent leakage and control contamination. Keep in original packaging.

7. Exposure Controls/Personal Protection

ENGINEERING MEASURES:	Provide local exhaust ventilation if required. See exposure limits	
EXPOSURE LIMITS:	SHORT TERM EXPOSURE LIMITS	LONG TERM EXPOSURE LIMITS
Total inhalable dust:	10mg/m^3	10mg/m^3
Respirable dust:	5mg/m^3	5mg/m^3
RESPIRATORY PROTECTION:	Provide local extraction if required. See exposure limits. If exposure limits are likely to be exceeded then ensure that masks are used – EN 143 type P2 is recommended	
HAND PROTECTION:	Wear gloves	
EYE PROTECTION:	Wear goggles	
GENERAL SAFETY MEASURES:	The usual precautions for the handling of chemicals and powders must be observed	

8. Physical and Chemical Properties

FORM:	Coloured fine powder
SOFTENING POINT:	Not applicable – Thermoset product
DECOMPOSITION POINT:	Above 190^{o}C
SOLUBILITY IN WATER:	None
pH VALUE:	6-7.5 (5% in water @ 25^{o}C)
SPECIFIC GRAVITY	c.1.0 up to 2.65 (g/cm^3) @ 20^{o}C
FLASH POINT:	Not applicable
ODOUR:	Slight smell
VISCOSITY:	Not applicable
BOILING POINT:	Not applicable
VAPOUR DENSITY:	Not applicable
VAPOUR PRESSURE:	Not applicable
EXPLOSION HAZARD:	Dust explosion hazard if stored in large quantities
MIN. EXPLOSIBLE CONC.:	67-75 g/M^3
MINIMUM IGNITION ENERGY:	7-10m Joules

9. Stability and Reactivity

CONDITIONS CONTRIBUTING TO INSTABILITY:	Product is stable under recommended storage and handling conditions. If exposed to elevated temperatures gas can be liberated – in these cases suitable control procedures should be implemented.
MATERIALS TO AVOID:	Keep product away from strong oxidising agents, strongly alkaline and strongly acidic materials
HAZARDOUS DECOMPOSITION PRODUCTS:	Fumes may contain oxides of sulphur, carbon and nitrogen.

10. Toxicological Information

ACUTE ORAL TOXICITY LD50:	More than 16g/Kg*
ACUTE DERMAL TOXICITY LD50:	More than 23g/Kg*
ACUTE DUST INHALATION LC50:	More than 4.4mg/l (4 hours)*
EYE IRRITATION:	No significant irritation
HEAVY METAL CONTENT:	Typical Analysis Expressed in mg/kg Antimony <1, Arsenic <1, Barium 1, Cadmium <1 Chromium <1, Lead <1, Mercury <1, Selenium <2
FREE PRIMARY AROMATIC AMINE:	Less than 0.1% w/w typical analysis
*NOTES:	The values for acute oral toxicity, acute dermal toxicity and acute dust inhalation refer to tests conducted on representative samples. These tests resulted in NO DEATHS OF THE TEST ANIMALS.

11. Ecological Information

Tests have been carried out by CEFAS Weymouth and ETS Ltd. exposing Pacific Oysters to high concentrations of EcoTrace tracer particles. The results indicated <5% uptake of available tracer particles peaking at 2 hours after exposure followed by dsicharge in the faecal strands. EcoTrace particles were depurated when the oysters were placed in clean water over a 5 day period with trace levels remaining in the Oysters after depuration. Further details are available from ETS Ltd.

12. Disposal considerations

Waste and emptied containers should be disposed of in accordance with current regulations

13. Transport information

Considered as Non-Hazardous under Transport Regulations

14. Regulatory Information

LABELLING ACCORDIING TO EU DIRECTIVES: Not subject to labelling
NATIONAL LEGISLATION/REGULATIONS: This product is classified as NON-HAZARDOUS under the UK 'Chemicals (Hazard Information and Packaging/Regulations' CHIP Regulations

15. Other Information

The information in this MSDS is based on the present state of our knowledge and on current EEC and National Laws. It is the responsibility of the user to ensure that their employees are aware of the content of this MSDS and also to ensure that any additional local rules and regulations are satisfied. The information contained herein is provided in accordance with the current legal requirement and should not be considered as a guarantee of the product's properties or performance. The information in this Safety Data Sheet is pursuant to:

a) The Chemicals (Hazard Information and Packaging) Regulation 1994
b) Article 27 of the Dangerous Substances Directive – 67/548/EEC as amended by the Seventh Amendment – 92/32/EEC (Official Journal No. L154. 5 June 1992 P1)
c) Article 10 of the Dangerous Preparation Directive – 88/379/EEC (Official Journal No. L187. 16 July 1988 P14)
d) The Safety Data Sheets Directive – 91/155/EEC as amended by Directive 93/112/EEC (Official Journal No. L314. 16 December 1993, P38)

APPENDIX B

STANDARD OPERATING PROCEDURE FOR LABORATORY MEASUREMENT OF FLUORESCENCE

REVISED AUGUST 2008

APPENDIX B

STANDARD OPERATING PROCEDURE

QP014

Fluorescence Microscopy and Fluorescence Magnification Methods for the Enumeration of Artificial Fluorescent Tracer Particles in Sediment Samples

Environmental Tracing Systems Ltd

The Coach House Bannachra Helensburgh G84 9EF UK

Tel: 00 44 1389 711001 e-mail: enquiries@environmentaltracing.com

Environmental
Tracing Systems Ltd

TABLE OF CONTENTS

1. SCOPE AND APPLICABILITY

This Standard Operating Procedure (SOP) details the operational methods for enumerating artificial fluorescent particles within an environmental sample and is valid for sediment core samples, sediment grab samples and sediment trap samples

This method has been adapted specifically for the two tracers "Marker A" and "Marker B" proposed for the study at Onondaga Lake, NY.

2. SUMMARY OF METHOD

Fluorescent microscopy method

The ETS microscope with camera and an eight place automated stage is a completely automated system capable of differentiating between several colours within the same sample at the same time. A software program specifically written for ETS is used to control the scanning of the slides; maintaining the sample in focus throughout the scan. Progress can be seen in real time on the computer screen, the software determines a grid of 'fields of view' in which all the surface of the sample will be scanned. The stage moves from one field of view to the next, where the whole field of view is checked against the pre-determined parameters for each colour before moving onto the next field of view. If a positive particle is indicated within the field of view the software captures the image of the particle and determines the size, allows post-analysis checks to be undertaken. At the end of each scan summary size distribution histograms for each colour are exported to spreadsheet.

Fluorescent magnification method

Samples are dried and spread to a sediment monolayer and examined for tracer particles by using a magnifying ultraviolet lamp. Particles identified by this method are then collected and examined by fluorescence microscopy to confirm identification as tracer particles.

Proper techniques for reception, preserving and storage sediment samples are important if the integrity of the sample is to be maintained. Procedures for preparation and analysis of sediment samples are given along with quality control information.

3. RECEIPT OF SAMPLES

It is anticipated that all sediment core samples collected will have underwent some initial processing, prior to being received by the ETS Laboratory. This initial

processing will involve sectioning the core into 0.5 cm or 1 cm subsections – each of these subsections is to be handled as an individual sample for analysis purposes.

Receipt of all samples must be logged as per ETS SOP QA003 *Receipt of samples document*, the key points are given below.

3.1. Sample arrival

On arrival all samples are inspected and compared to any information provided by the client. Any discrepancies between the documentation and the samples will be reported to the client, analysis will be put 'on hold' until the matter is resolved.

3.2. Samples Login

All samples are allocated a unique number, linked to any information provided by the client. Within the login sheet, notes are made concerning the samples such as: sample constituency, sample variation, missing samples etc. All samples are kept in a cool, dark storage room, refrigerated or frozen (as required) while awaiting preparation and analysis.

4. SAMPLE PREPARATION

Each sample is checked for integrity and the outside of the sample container is wiped down to reduce the possibility of contamination of the sample contents.

1. Each of the following two methods is undertaken for the enumeration of Marker A and Marker B concentrations within each sample: The number of slides for enumeration from each sample for Marker A will be provided based on results from site-specific information to be collected during the pre-mobilization field tests.

4.1. Marker A

This tracer is designed to mimic fine cohesive lake sediment found in Onondaga Lake.

4.1.1. Mass of sample used

The total wet weight of sample received is determined and recorded. The contents of each sample container is thoroughly mixed and a homogenous sub-sample removed. The mass of this sub-sample is determined and recorded. The remainder of the sample is retained for gravimetric analysis and determination of Marker B concentration.

4.1.2. Sample dilution

The sub-sample removed is added to a measured volume of deionised water and thoroughly mixed by placing in an ultrasonic bath for 10 minutes. The volume of this sediment suspension used for analysis depends on the sediment characteristics and concentration and within the original sample. The aim is to produce a sediment monolayer on each filter membrane during sample filtration to ensure maximum detection by the microscope image analysis software.

4.1.3. Sample filtration

The filtration unit consists of a two-piece polypropylene unit which is held together with a clamp during filtration. The base unit has a platform on which a single use membrane filter is placed. Once the filter membrane is in place then the upper section is connected to the base section and held in place with a clamp, thus holding the filter membrane in place and providing an area for sample introduction. The bottom section is permanently connected to a vacuum manifold, the vacuum facilitates the removal of excess fluid from the sample. The manifold is situated over a draining rack so that the base section of the filtration unit can be easily cleaned in situ.

A 0.45µm pore size GFC membrane filter membrane is prepared for each sample as per the procedure detailed in ETS SOP QA002 "*Gravimetric Analysis"*. Each filter membrane is dried, weighed and identified with the sample reference prior to use. The filtration unit is prepared, cleaned

thoroughly and the vacuum pumping apparatus connected. The filter membrane is placed in the polypropylene filter housing and clamped into place. The sediment sub-sample dilution is then carefully poured into the filter, washing the sides of the filter housing with de-ionised water. The vacuum pump is then switched on and operated until all the water has been removed from the filter. The pump is then stopped, the vacuum removed by allowing air pressure to equilibrate and the filter housing unclamped.

Between sample filtrations, the upper section of the filtration unit is removed from the base unit and rinsed with deionized water, followed by an acetone rinse. The upper section is then submerged in an ultrasonic bath for 5 minutes. The unit is then once again rinsed with acetone followed by a final deionized water rinse to remove any solvent residue prior to being reused. This washing cycle is performed at the start and finish of an analysis run and between every sample.

4.1.4. Preparation of slides for enumeration by fluorescence microscopy

The membrane is carefully removed from the filter housing and transferred to a labelled microscope slide. A coverslip is placed onto the slide, covering the filter membrane, and secured in place with adhesive.

4.2. Marker B

Particle size for Marker B will be significantly larger than for Marker A. Marker B will be a sand-sized fluorescent particle tracer.

4.2.1. Preparation for enumeration by fluorescence magnification

5. FOLLOWING PREPARATION OF SLIDE(S) FOR ENUMERATION OF MARKER A, THE REMAINING WET SAMPLE IS PREPARED FOR GRAVIMETRIC ANALYSIS AND FOR DETERMINATION OF MARKER B CONCENTRATION. SEE ETS SOP QA002 *"GRAVIMETRIC*

***ANALYSIS"* FOR DETAILED PROCEDURE. THE GRAVIMETRIC ANALYSIS USES A FURTHER SMALL SUB-SAMPLE OF THE ORIGINAL SAMPLE – THE REMAINING WET SAMPLE IS OVEN DRIED IN ITS ENTIRETY AND REWEIGHED. IF THE SAMPLE IS COMPOSED OF MAINLY SAND, THERE IS NO FURTHER SAMPLE PREPARATION REQUIRED. IF THE SAMPLE IS COMPOSED OF MAINLY COHESIVE SILT, THE DRIED SAMPLE IS PULVERISED IN A CLEAN MORTAR AND PESTLE TO FACILITATE COUNTING.SAMPLE ANALYSIS BY FLUORESCENCE MICROSCOPY**

5.1. Details of Equipment used

The fluorescent microscope to be used for enumeration of Marker A counts is a Leica Microscope DM600B with a DFC280 camera and an eight place automated stage. This is a completely automated system capable of differentiating between several colours simultaneously within the same sample.

5.2. Details of Microscope Control and Image Analysis Software

The proprietary image analysis software used to interpret the images generated from the Microscope and Digital Camera is Leica QWin Pro version 3.2.1, developed by Leica Microsystems (Switzerland) Ltd.

This software has been modified by Leica specifically for ETS to control the scanning of the slides.

5.3. Microscope Set-up

The Fluorescence Microscope is switched on for 30 minutes prior to analysis to allow the lamp to stabilize. The correct filter set for the tracer(s) is selected and placed in the relevant position.

Up to eight prepared microscope slides are installed within the automated stage and sample reference details for each slide are manually entered into the control software. The slides are cross-checked to confirm correct labelling and documentation and the slide integrity is also checked.

The control software then moves the microscope lens to one edge of the first filter membrane on the first slide; the operator then fine tunes the positioning of the microscope lens to the exact edge of the filter membrane. This is repeated for each edge of each membrane for each slide, with the whole process taking about one minute to complete. This provides the control software with exact details of which regions of the automated stage to examine.

The parameters to identify a positive tracer particle are then selected, these parameters characterise (in very basic terms) the ratio of primary colours contained within the microscope image and the minimum and maximum dimensions of particles to be accepted. These parameters are set-up as part of the calibration routine by analysing prepared samples of the tracer (Marker) to be studied.

5.4. Microscope Sample Analysis

The control software then commences scanning the slides for images that conform to the parameters selected during the set-up routine. The software automatically maintains the sample in focus throughout the scan.

Progress can be seen in real time on the computer screen, the software determines a grid of 'field of views' in which all the surface of the sample will be scanned. The stage moves from one field of view to the next, where the whole field of view is checked against the pre-determined parameters for each colour before moving onto the next field of view. If a positive particle is indicated within the field of view then the software records the number of positive particles, the software also sizes the particles at this point. An image of any field of view containing positive particles is saved to the hard disk of the computer, allowing post-analysis checks to be undertaken, as part of quality assurance procedures or to examine an individual tracer particle image. At the end of the scan a summary containing, for each colour, the number of positive particles determined and the size band they fall into is automatically written into an excel spreadsheet.

fluorescent microscope and image analysis software described above, to confirm that the particles being identified and counted are tracer particles.

7. QUALITY CONTROL MEASURES

Throughout the analysis procedures outlined above, control samples and blank samples are prepared to ensure that all equipment and laboratory working area remained clean and uncontaminated with fluorescent material from other samples, laboratory equipment or personnel. If contamination is observed then all samples analyzed since the previous clear blank are reanalyzed following identification and elimination of source of contamination.

All samples must have a unique number and correspond to the relevant documentation.

All results, descriptions and comments concerning each sample are cross-referenced to its unique sample reference number.

A 'Clean Laboratory' policy is in effect minimizing cross-contamination.

All measuring equipment is calibrated and checked before use.

Cross-checks are performed to ensure documentation, sample labelling coincide throughout analysis.

A random selection (10%) of the original samples will be selected for replicate analysis, with a proportion of the prepared samples re-examined by a second analyst.

Quality control limits for replicated analyses will be provided based on results from site-specific information to be collected during the pre-mobilization field tests.

8. SAMPLE STORAGE

Following analysis, all samples will be placed in storage in a cool dark room, refrigerator or freezer, as required, until the final report has been agreed by all parties concerned.

9. REPORTING

Results from all the experiments are collated into an excel worksheet and reported to the Project Manager.

Tracer particle counts from both the fluorescence microscopy and by magnifying ultraviolet lamp are recorded into a notebook before being transcribed into a prepared Microsoft Excel spreadsheet, along with additional information provided or measured. Such additional information will include:

- Unique sample reference number
- Field sample label
- Description of sample and comments on appearance
- Date and time sample collected (if provided)
- Geographical position of sampling location (if provided)
- Mass of wet sample received
- Gravimetric determination – percentage mass lost on drying
- Mass of sample analysed
- Tracer counts determined by analysis

The tracer counts are then reported as "number of tracer particles per gram **dry** weight of core/grab" or as "number of tracer particles per gram **wet** weight of core/grab" as required.

For analyses performed by fluorescence microscopy, the counts are first given as counts per wet weight of sample received. The gravimetric determination is then used to convert this to counts per gram dry weight.

6. SAMPLE ANALYSIS BY FLUORESCENCE MAGNIFICATION

Where larger sand-sized particles (over 63 μm), such as Marker B particles, are to be enumerated the following procedure is adopted.

6.1. Preparation of workspace for Fluorescent Magnification analysis

The work area is prepared for the inspection of the dried sediment by fluorescence magnification as described below.

The work area, table or bench, is thoroughly cleaned prior to commencing sediment analysis. A large sheet of heavy paper is placed on the work area and a sheet of aluminium foil is placed on the heavy paper. The edges of the paper and aluminium are folded inwards to secure the foil in place – this creates a temporary clean work area that is used for the examination of a single sample and is discarded after single use.

The operator examines the temporary workspace with the magnifying ultraviolet lamp to ensure there are no contaminating fluorescent particles visible prior to sample analysis.

6.2. Fluorescent Magnification analysis

The prepared dried sediment sample is then placed onto the prepared work area and is carefully spread, a small mass at a time, across the workspace in a monolayer using a prepared piece of thick card. The viewing room is darkened and the operator inspects the sediment sample through the magnifying ultraviolet lamp. Any sand-sized fluorescent particles glow intensely and are very clearly visible through the lamp.

Typically, the first ten fluorescent particles are collected by tweezers and placed on a microscope slide prepared with double-sided tape. This allows characterization of the collected particles in terms of size and colour using the

For analyses performed with magnifying ultraviolet lamp the total number of particles counted is divided by the dry mass of sediment analysed to give counts per gram dry weight. The gravimetric determination is then used to convert this to counts per gram wet weight of sample received.

10. RELATED DOCUMENTS

ETS Standard Operating Procedures:

QA003 : Receipt of Samples

QA004 : Safety Code of Practice

QP002 : Gravimetric Analysis