# Addendum No. 6 Sampling and Analysis Plan and Quality Assurance Project Plan

# South State Street Manufactured Gas Plant Remedial Investigation/Feasibility Study Bellingham, Washington

June 7, 2016

Prepared for

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and

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# Addendum No. 6 Sampling and Analysis Plan and **Quality Assurance Project Plan** South State Street Manufactured Gas Plant **Bellingham, Washington**

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- A MW-46 Conductivity and Tidal Survey

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### Attachment <u>Title</u>

1 Standard Operating Procedure – Separation of Solids by Centrifuge Modified EPA-823-B-010002 Method

### LIST OF ABBREVIATIONS AND ACRONYMS

ARI	Analytical Resources, Inc.
City	City of Bellingham
Ecology	Washington State Department of Ecology
ЕРА	Environmental Protection Agency
ft	foot or feet
HASP	Health and Safety Plan
ICPMS	inductively-coupled plasma mass spectrometry
LAI	Landau Associates, Inc.
μg/L	micrograms per liter
MTCA	Model Toxics Control Act
РАН	polycyclic aromatic hydrocarbon
PSE	Puget Sound Energy
QA/QC	quality assurance/quality control
QAPP	quality assurance project plan
RI/FS	remedial investigation and feasibility study
SAP	sampling and analysis plan
SSSMGP	South State Street Manufactured Gas Plant
SOP	standard operating procedure
WAC	Washington Administrative Code
WISHA	Washington Industrial Safety and Health Act
WSDOT	Washington State Department of Transportation

# **1.0 INTRODUCTION**

This document is an addendum to the sampling and analysis plan (SAP) and quality assurance project plan (QAPP) for the South State Street Manufactured Gas Plant (SSSMGP) Site remedial investigation and feasibility study (RI/FS) in Bellingham, Washington (Figure 1). It outlines additional sampling and testing activities proposed for the SSSMGP Site, activities supplementary to work conducted under the August 6, 2010 Work Plan (Herrenkohl Consulting and LAI 2010c) and subsequent addenda, based on recommendations in the RI Interim Data Report (Herrenkohl Consulting and LAI 2011c), and further discussions with the Washington State Department of Ecology (Ecology).

This addendum provides specific guidance for field methodology and quality assurance procedures that will be followed by Landau Associates, Inc. (LAI) and subcontractors. LAI is conducting this work under contract with the City of Bellingham, Parks and Recreation Department (City), with direction from the Ecology Toxics Cleanup Program. The addendum to the SAP and QAPP was prepared in accordance with an Agreed Order and Scope of Work negotiated between the City, Puget Sound Energy (PSE), and Ecology (signed April 30, 2010; Document No. 7655), and was developed to meet the requirements of an RI/FS as defined by the Washington State Model Toxics Control Act (MTCA) Cleanup Regulation (Washington Administrative Code [WAC] 173-340) (Ecology 2007).

Several documents are cited repeatedly in this addendum. Altogether, these documents are referred to as the Work Plans for the SSSMGP Site RI/FS:

- Work Plan for the RI/FS of the SSSMGP Site Bellingham, Washington (August 6, 2010). The Work Plan provides information on existing data for the SSSMGP Site and the sampling strategy and design to meet the data needs for completing the RI/FS. The Work Plan also describes the project management strategy for implementing and reporting RI/FS activities for the Site, including project team responsibilities and schedule.
- SAP (Appendix B of the Work Plan) for the RI/FS of the SSSMGP Site, Bellingham, Washington (August 6, 2010). The SAP describes the procedures for conducting field activities and presents the proposed laboratory analyses for samples collected in the field.
- QAPP (Appendix C of the Work Plan) for the RI/FS of the SSSMGP Site, Bellingham, Washington (August 6, 2010). The QAPP describes analytical method reporting limit goals, field and laboratory quality assurance/quality control (QA/QC) requirements and reporting requirements for the RI/FS for the Site.
- Project Health and Safety Plan (HASP; Appendix D of the Work Plan) for the RI/FS of the SSSMGP Site, Bellingham, Washington (August 6, 2010). The HASP was prepared in accordance with WAC 173-340-810, applicable Washington Industrial Safety and Health Act (WISHA) regulations, and project requirements. It addresses those activities associated with work to be performed at the Site.

The Work Plan described an environmental investigation designed to meet the data needs for completing the RI/FS. Data collected in that investigation were summarized in the Interim Data Report (Herrenkohl Consulting and LAI 2011c). An evaluation of that data resulted in the identification of remaining data gaps and recommendations for additional investigation activities to fulfill the data

needs for completing the RI/FS. Those additional investigation activities were conducted in July 2011, February 2012, October 2012, February 2013, and September 2015 in accordance with SAP and QAPP Addendums No. 1, No. 2, No. 3, No. 4, and No. 5 (Herrenkohl Consulting and LAI 2011a, b, 2012, 2013, 2015); the September 2015 investigation addressed sediment quality data gaps identified by Ecology after review of the draft RI report.

Sampling and testing completed in accordance with Addendum No. 1 included laboratory analysis of some archived soil and sediment samples, additional soil vapor sampling, and a bivalve reconnaissance within the pocket beach area of the Site. For Addendum No. 2, soil and sediment borings were drilled and monitoring wells installed at four additional locations (HS/MW-46, HS/MW-53, HS/MW-54 and HS/MW-55) to further delineate contamination associated with the Site and define the Site boundary.<sup>1</sup> For Addendum No. 3, soil borings were drilled (GP-56 and GP-57) to further delineate soil and groundwater contamination associated with the Site and further define the Site boundary. An evaluation of vapor intrusion as a potential exposure pathway at the Spinnaker Reach Condominiums was completed in accordance with Addendum No. 4.

The City and PSE submitted a draft RI report to Ecology in April 2014 for review. The draft RI report included an evaluation of data collected and analyzed as described in the original Work Plan and subsequent four addenda. After receiving Ecology's preliminary comments on the draft RI report in August 2014, the City and PSE provided a response to those preliminary comments and met with Ecology to discuss specific comments on December 3, 2014 and February 24, 2015. Based on those meetings and written communications including a revised set of comment responses by Ecology on May 14, 2015, the City and PSE agreed to conduct supplemental sampling to address groundwater quality data gaps and sediment sampling to further define the Site boundary and nature and extent of contamination in sediments.

To address these data gaps, Addendum No. 5 was prepared and reviewed by Ecology; Ecology's review comments were addressed in the final Addendum No. 5, submitted to Ecology on September 10, 2015. Sampling and testing proposed in Addendum No. 5 included:

- Collection and analysis of additional surface sediment and sediment core samples to further define the Site boundary and the nature and extent of sediment contamination, and to support evaluation of the direct contact and seafood consumption pathways for human health exposure.
- Characterization of water quality conditions at the point of discharge from groundwater to surface water downgradient of MW-28 and in other near-shore wells at the Site.

The sediment sampling and analysis program was completed in September 2015. Initial reporting on the results of the sediment sampling activities was submitted to Ecology in support of

<sup>&</sup>lt;sup>1</sup> For the purpose of this document, the term "boundary" refers to the Preliminary SSSMGP Site Boundary as set out in Exhibit A to the 2010 Agreed Order. The Preliminary SSSMGP Site Boundary may not encompass the full lateral extent of contamination associated with the former MGP. The final Site boundary will be based on the full extent of contamination and could be larger or smaller than that originally identified in Exhibit A of the Agreed Order.

recommendations for additional analysis of select archived samples (LAI 2015). Archived sample analysis is underway; data will be reviewed and discussed with Ecology, as appropriate.

During review of Addendum No. 5, Ecology requested that an additional monitoring well (i.e., MW-58) be installed north of existing MW-28 to provide additional information regarding the potential presence and quality of shallow groundwater in the northern portion of the lower park. To support effective installation and sampling of that newly-requested well, this addendum has been prepared to support the additional groundwater well installation and water quality evaluation approximately 100 feet (ft) of MW-28; the information in this addendum supersedes the groundwater investigation strategy outlined in the previously-approved Addendum No. 5.

# 2.0 GROUNDWATER INVESTIGATION

One new monitoring well (MW-58) and two temporary well points (MGP-WP-01 and MGP-WP-02) will be installed and groundwater samples will be collected from these three new locations for laboratory analysis. The new monitoring well will be located approximately 100 ft north of MW-28 to provide additional information regarding the potential presence of shallow groundwater and its associated water quality in the northern portion of the lower park. The two temporary well points will be located along the shoreline, approximately 50 ft and 100 ft north of MW-46, to provide additional groundwater data downgradient of MW-28.

Additional groundwater samples will be collected from the existing near-shore monitoring wells simultaneous with sampling of the new monitoring well and temporary well points. The locations of proposed new monitoring well MW-58, the two temporary well points, and the existing Site wells proposed for additional groundwater sampling are shown on Figure 1. Proposed well locations may be adjusted based on field conditions. The remainder of this section describes the planned temporary well point installation and construction and the sampling and analysis procedures that differ from those described in the original SAP and QAPP documentation (Herrenkohl Consulting and LAI 2010a, b).

# 2.1 Monitoring Well Installation and Development

To further evaluate groundwater elevations and characterize potential water quality north of MW-28, one monitoring well (MW-58) will be installed approximately 100 ft north of MW-28 along the existing walking path. Monitoring well installation and development procedures will follow standard procedures in the SAP (Herrenkohl Consulting and LAI 2010c). If groundwater is present in proposed well MW-58, the well will be surveyed by a professional surveyor so the groundwater elevations in the lower park can be evaluated as part of planned groundwater sampling activities.

# 2.2 Well Point Installation and Construction

To further characterize water quality conditions at the point of discharge from groundwater to surface water potentially downgradient of MW-28 (i.e., north of MW-46), groundwater from two beach locations (MGP-WP-01 and MGP-WP-02) will be sampled from temporary hand-driven well points. Sampling of the temporary wells will be timed so that groundwater samples are collected at the point in the tidal cycle when groundwater discharge is greatest, relative to mixing with bay water at the point of discharge (as discussed Section 2.3).

A stainless steel well point consisting of a 0.010-inch slot size, wire-wrapped screen above a drive point will be hand driven at each of the two locations. The well points will be installed 1.5 ft to 2 ft below the water interface by hammer-driving the well point to depth. Alternatively, hand-auger borings will be advanced using a 2-inch outside-diameter auger attached to a rotohammer and stand. Once drilled to depth, the auger will be removed and the well point will be advanced into the open hole. Pre-drilling the boring with the rotohammer/auger setup would likely increase the turbidity of groundwater initially; however, this approach will be used as backup in the event that driving the well point screen is initially unsuccessful due to site conditions (e.g., coarse-grained materials). A sediment sample will be collected and archived from each well point location from the depth interval at which the groundwater samples will be collected (i.e., soil from the top foot in which groundwater infiltration is observed in the well hole).

## 2.3 Groundwater Sampling

Two rounds of groundwater samples will be collected for analysis from the new monitoring well MW-58 (if sufficient groundwater is present to allow for sampling) and each of the two temporary well points following installation. Groundwater samples will also be collected during the two rounds from existing Site wells MW-28, MW-34, MW-36, MW-40, MW-42, MW-46, and MW-55 (Figure 1). Water level data will also be measured from these monitoring wells and the two temporary well points following procedures in the original SAP (Herrenkohl Consulting and LAI 2010c).

Samples will be collected using dedicated polyethylene tubing and a peristaltic pump. Field geochemical parameters (i.e., pH, ferrous iron, dissolved oxygen, oxygen-reduction potential, conductivity, and turbidity) will be measured in groundwater collected from the new monitoring well, two temporary well points, and seven selected existing Site wells.

The first round of groundwater sampling will be scheduled for the second quarter of 2016 (i.e., May/June 2016). The second round of groundwater sampling will be scheduled for third quarter 2016. The groundwater samples will be collected using the procedures described in the original SAP with the following exceptions for sample timing and tubing placement, as described below.

### 2.3.1 Groundwater Sampling Timing and Tubing Placement

Groundwater samples will be collected at the point in the tidal cycle when bay water dilution of the groundwater is the least. The point during the tidal cycle of least bay water dilution was determined during the 2010 evaluation on tidal influence on groundwater levels (LAI 2010), and during the 2015 MW-46 conductivity and tidal study (LAI 2016, see Appendix A), as noted below. Sample tubing will be placed at the center of the screened portion of the well, unless as discussed in the 2015 MW-46 conductivity and tidal study, and as indicated below.

### 2.3.1.1 Wells MW-34, MW-36, MW-40, MW-42, MW-55

Near-shore wells (excluding MW-28, MW-46, MW-58, and the two temporary well points) will be sampled at a time centered on approximately 15 minutes after the predicted low tide, as described in the original SAP (Herrenkohl Consulting and LAI 2010b).

### 2.3.1.2 MW-28 and MW-58

The 2010 evaluation of tidal influence on groundwater elevations (LAI 2010) determined that there was no tidal influence of groundwater at MW-28; therefore, MW-28 and planned new well MW-58 will not be sampled at a specific time relative to the predicted tide.

### 2.3.1.3 MW-46, MGP-WP-01, and MGP-WP-02

As indicated in the MW-46 conductivity and tidal survey (LAI 2016), MW-46 will be sampled approximately 3 to 3.5 hours following the predicted low tide and the inlet of the sample tubing will be placed in the top foot of the water column. Well points MGP-WP-01 and MGP-WP-02 are in the same vicinity as MW-46, and will be sampled in the same manner in accordance with the conclusions presented in the technical memorandum summarizing the 2015 conductivity and tidal survey (LAI 2016, see Appendix A).

### 2.4 Laboratory Analyses

The groundwater samples collected from the new temporary well points and new/existing monitoring wells will be analyzed for:

- Hardness by Standard Method 6010C.
- Total and dissolved arsenic, lead, selenium, and silver by U.S. Environmental Protection Agency (EPA) Method 6020 with a collision cell.
- Benzene, ethylbenzene, toluene, and total xylenes (BTEX) by EPA Method 8260.
- Polycyclic aromatic hydrocarbons (PAHs) by EPA Method 8270D with low-level analysis (selective ion monitoring [SIM]) and silica-gel cleanup.
- Total and Weak Acid Dissociable (WAD) cyanide by Standard Method 4500CNI.

Samples collected for dissolved metals and WAD cyanide analysis will be filtered in the field. Since water samples at stations MW-46, MGP-WP-01, and MGP-WP-02 are expected to be turbid upon collection, samples will be centrifuged at the laboratory before analysis, following Analytical Resources, Inc. (ARI) standard operating procedure (SOP) 1151 for separation of solids by centrifuge (Modified Method EPA-823-B-01-002–refer to Attachment 1). In addition, samples collected for WAD cyanide analysis from the other seven sampling locations will also be centrifuged in the laboratory before analysis.

Target reporting limits for each analysis will be those specified in the original QAPP (Herrenkohl Consulting and LAI 2010a) with the following exceptions:

- For the total and dissolved metals the laboratory will use EPA Method 6020 with a collision cell inductively-coupled plasma mass spectrometry (ICPMS) in an attempt to achieve reporting limits of between 0.1 and 2.0 micrograms per liter (μg/L) depending on the metal being analyzed.
- For PAHs, the laboratory will use EPA Method 8270D–SIM analysis with a silica gel cleanup in an attempt to achieve a reporting limit of 0.01 μg/L.

# 2.5 Well Point Abandonment

Temporary well points will be removed from the sediment after sampling is complete and the holes will be filled with hydrated bentonite chips. The locations of the temporary well points will be measured by surveyors to evaluate groundwater elevations and allow re-installation of temporary well points and follow-up second round sampling at the same locations.

# 2.6 Investigation-Derived Waste

Investigation-derived waste, including remaining sediment, purge water, and decontamination water, will be containerized in Washington State Department of Transportation (WSDOT)-approved 55-gallon drums, labeled, and stored onsite pending analysis for proper disposal in accordance with applicable regulations.

### **3.0 REFERENCES**

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APPENDIX A

# **MW-46 Conductivity and Tidal Survey**

## **Technical Memorandum**

то:	John Guenther, Washington State Department of Ecology
FROM:	Chip Halbert, PE and Dylan Frazer, LG
DATE:	May 5, 2016
RE:	MW-46 Conductivity and Tidal Survey South State Street Manufactured Gas Plant Bellingham, Washington Project No. 0015015.070

As stated in the *South State Street Manufactured Gas Plant Remedial Investigation/Feasibility Study Work Plan* (RI/FS Work Plan) (Herrenkohl Consulting and LAI 2010), groundwater sampling at near-shore wells at the South State Street Manufactured Gas Plant (SSSMGP) site in Bellingham, Washington (Site; Figure 1) will be conducted at times coinciding with low tide events. A 2010 tidal study was conducted to observe the nature of tidal influences on groundwater elevations in monitoring wells (Herrenkohl Consulting and LAI 2010). A second tidal survey was conducted in 2015 at the request of the Washington State Department of Ecology (Ecology), who anticipated that a correlation between conductivity and predicted tidal cycles would further reinforce the presumed time period during which groundwater discharges to Bellingham Bay are at their peak.

This technical memorandum presents the results of the 2015 conductivity and tidal survey for use in scheduling groundwater monitoring events at MW-46, and other nearby sampling points (as appropriate), such that they coincide with the time of maximum groundwater discharge at each sampling location.

## Methodology

A tidal and conductivity survey was conducted from December 7 through 14, 2015 at MW-46. A preliminary review of the initial data collected on December 8, 2015 indicated that conductivity levels in the well were more stable than anticipated. After discussions with Ecology, representatives of Landau Associates, Inc. (LAI) collected supplemental data at MW-46, which included conductivity relative to depth in the well's water column, conductivity relative to time within the predicted tidal cycle, and groundwater elevation relative to time within the predicted tidal cycle.

On December 9, 2015 a datalogger set to record conductivity at 5-second intervals was lowered slowly through the water column in MW-46. The datalogger's rate of descent was slow enough to evaluate stratification of conductivity throughout the water column. After the water column survey was completed, MW-46 was redeveloped. A Waterra<sup>©</sup> pump was used for redevelopment, and MW-46 was pumped dry approximately 10 times.

Following redevelopment, the conductivity datalogger was re-installed in the well. Based on discussions with Ecology, this datalogger was set at a fixed depth of approximately 8 ft below the top



of well casing, within the depth horizon of the more permeable sand layer. A second, pressure-only datalogger was installed at the bottom of the water column to measure groundwater elevation changes as a result of the tidal cycle. The dataloggers were set to collect data every minute, and recorded data until they were removed from MW-46 on the morning of December 14, 2015. Data discussed below include those collected after an approximately 48-hour post-development period of recharge and equilibration; the survey period therefore extended from the evening of December 11, 2015 to the morning of December 14, 2015.

One final check of potential conductivity stratification of the water column was conducted on December 14, 2015 after removal of the two dataloggers. This monitoring of conductivity stratification was conducted approximately 1 hour after the predicted morning high tide on December 14, 2015.

## **Data Results**

### **Conductivity Data and Predicted Tide**

As presented on Figure 2, the conductivity measured in the water column within MW-46 at approximately 8 ft below the top of the well casing ranged from a maximum value of 21,000 microSiemens per centimeter ( $\mu$ S/cm) to a minimum value of 19,855  $\mu$ S/cm. Figure 2 illustrates the minor variability of the conductivity as measured during the survey. Conductivity levels in MW-46 do not oscillate on a regular (tidal-like) cycle. For comparison purposes, conductivity values measured at Site wells, based on the most recent groundwater sampling event at each location (i.e., either the March 2011 or the February 2012 sampling event, depending on the location), are presented on Figure 1. The ranges of conductivity levels are helpful in representing areas in which mixing with seawater is negligible, moderate, and high.

### **Groundwater Elevations and Predicted Tide**

As presented on Figure 3, the water levels measured by a pressure transducer placed within MW-46 ranged from approximately 6 to 10 ft mean lower low water (mllw, approximately a 4-ft swing), in a pattern that generally follows the tidal cycle. Three extreme tidal lows were recorded during the December 11 through December 14 survey period; minimum groundwater elevations in MW-46 follow the extreme tidal lows with an approximately 3- to 3.5-hour lag time.

### Water Column Conductivity Survey

As presented on Figure 4, the conductivity in the water column in MW-46, as measured approximately one hour after high tide on December 14, 2015, is stratified into three distinct layers. From the top of the water column (approximately 9.5 ft mllw) to approximately 8 ft mllw, the conductivity was measured at approximately 14,500  $\mu$ S/cm; relative to sea water conductivity of approximately 55,000  $\mu$ S/cm, this conductivity range indicates a predominant presence of groundwater with moderate sea water mixing. From 8 ft mllw to 5.5 ft mllw, the conductivity was measured at

approximately 16,000  $\mu$ S/cm, again reflecting moderate seawater mixing. A transition zone appears to exist between 5.5 ft mllw and 3.5 ft mllw, where conductivity steadily increases from approximately 16,000  $\mu$ S/cm to approximately 20,500  $\mu$ S/cm; this transition zone reflects the point of the water column in which, at the time of data collection, seawater mixing increases from a moderate level to a higher level. Conductivity was measured at approximately 20,500  $\mu$ S/cm from 3.5 ft mllw to the bottom of the well, which is at approximately -1 ft mllw.

## **Analysis and Recommendations**

Contrary to the presumed result of the investigation, conductivity is not a good indicator in MW-46 of the relative amount of groundwater discharge as compared to the predicted tides. No apparent correlation was identified between conductivity and tidal cycles over a several-day period (Figure 2). However, as compared to the most recently-measured Site groundwater conductivity data presented on Figure 1 (Herrenkohl Consulting and LAI 2014), the groundwater in MW-46 does appear to be a mixture of groundwater and sea water.

As illustrated on Figure 1, the measured conductivities in MW-46 are less than typical sea water conductivity (approximately 55,000  $\mu$ S/cm), and also less than those values measured at monitoring wells in the lower park portion of the Site (i.e., MW-34, MW-36, MW-38, MW-40, MW-42, MW-54, and MW-55), which range between approximately 23,000 and 36,200  $\mu$ S/cm (indicating a high degree of sea water mixing). The MW-46 conductivity values are greater than conductivities in wells MW-19, MW-24, MW-28, MW-29, MW-31, and MW-53, which range between approximately 300 and 6,700  $\mu$ S/cm (indicating limited to no sea water mixing). The MW-46 conductivity values are similar to the values measured in MW-45, indicating a moderate degree of sea water mixing at both locations.

Groundwater elevation data collected indicate that the groundwater elevation in MW-46 follows a tidal cycle; the lowest groundwater elevation appears to follow the predicted low tide by approximately 3 to 3.5 hours (Figure 3). The water column survey indicates the presence of three distinct layers of conductivity stratification in MW-46 (Figure 4). As illustrated by the historically-measured conductivities noted on Figure 1, the conductivity readings in the well do indicate moderate mixing with sea water in the upper horizons of the well and higher mixing with sea water in the deeper horizons. Based on ranges of conductivities in other near-shore wells shown on Figure 1, mixing of groundwater and sea water appears to occur in MW-46 to a lesser degree than might be expected. The upper portions of the water column have the lowest conductivity in MW-46 (i.e., a higher proportion of groundwater).

The data and analyses provided above indicate that a groundwater sample collected from MW-46 will have the least sea water dilution and be most representative of groundwater quality if:

• MW-46 is sampled at the time when the groundwater gradient into MW-46 is the steepest, which is approximately 3 to 3.5 hours following the predicted lower low tide, and

• Groundwater is sampled from within the top foot of the MW-46 water column, which is the portion of the water column with the lowest conductivity, and thus the greatest proportion of groundwater and least sea water influence.

The results of the MW-46 tidal and conductivity survey will be used to support planning and strategy for future sampling events as documented in subsequent Sampling and Analysis Plan Addenda, as appropriate.

## Limitations

This technical memorandum has been prepared for the exclusive use of the City of Bellingham for specific application to the South State Street Manufactured Gas Plant Site. No other party is entitled to rely on the information, conclusions, and recommendations included in this document without the express written consent of Landau Associates. Further, the reuse of information, conclusions, and recommendations provided herein for extensions of the project or for any other project, without review and authorization by Landau Associates, shall be at the user's sole risk. Landau Associates warrants that within the limitations of scope, schedule, and budget, our services have been provided in a manner consistent with that level of care and skill ordinarily exercised by members of the profession currently practicing in the same locality under similar conditions as this project. We make no other warranty, either express or implied.

This document has been prepared under the supervision and direction of the following key staff.

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Attachments:Figure 1 – Site Plan and Most Recently Sampled Conductivity ValuesFigure 2 – MW-46 Conductivity and Predicted TidesFigure 3 – MW-46 Groundwater Elevation and Predicted TidesFigure 4 – MW-46 Conductivity vs. Elevation in Water Column

## References

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Landau Associates Δ

ATTACHMENT 1

Standard Operating Procedure – Separation of Solids by Centrifuge Modified EPA-823-B-010002 Method

# **Standard Operating Procedure**

# Separation of Solids by Centrifuge Modified EPA-823-B-01-002 Methods

**SOP 1151 Revision 001** 

Revision Date: 4/17/09

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Approvals:

Laboratory / Section Manager

## **Annual Review**

SOP Number:		
Title:	Separation of Solids by Centrifuge	
Revision:	001	
Revision Date:	4/17/09	
Effective date:		

The ARI employee named below certifies that this SOP is accurate, complete and requires no revisions.

Reviewer's Name	Reviewer's Signature	Date
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### 1.0 PURPOSE AND SCOPE

This procedure describes the method, materials, equipment, and special conditions required to isolate and separate the solids in a given sample. This procedure is for extraction of solid material from a matrix in preparation for various chemical and physical analyses.

### 2.0 EQUIPMENT

- 2.1 Refrigerated centrifuge
- 2.2 500 ml decontaminated glass centrifuge bottles
- 2.3 Unused 1 L amber glass bottles
- 2.4 Balance for balancing the centrifuge bottles, accurate to 0.1 g
- 2.5 Decontaminated spoons
- 2.6 Aluminum foil
- 2.7 Unused glass sample jars of appropriate sizes
- 2.8 Deionized (DI) water

### **3.0 DEFINITIONS**

- 3.1 Test Environment
- 3.1.1 The test environment should be a fairly constant temperature of approximately 4° C during centrifuging. Fluctuations in temperature may introduce changes in chemical makeup that are of practical significance.

### 4.0 DOCUMENTATION

4.1 1151F - Separation of Solids by Centrifuging Data Sheet

### 5.0 PROCEDURE

- 5.1 Decontamination
- 5.1.1 Decontamination of glass centrifuge bottles must include the following steps:
  - wash the bottles with a Citranox detergent and rinse with tap water to remove any residue
  - soak with a 5% nitric acid solution for at least four hours
  - rinse with DI water
  - air dry
  - rinse with dichloromethane
  - air dry
- 5.1.2 Decontamination of miscellaneous spoons must include the following steps:
  - wash the spoons with a detergent and rinse with DI water to remove any residue
  - air dry
  - rinse with dichloromethane
- 5.2 Sediment samples will be maintained at 4°C prior to centrifuging. Care must be taken at each step to ensure that the temperature of the sample is maintained at or below 10°C during processing.
- 5.3 Remove the samples from the cooler/refrigerator. Verify sample ID numbers, and notify supervisor for ID discrepancies. Review the job folder and check for any possible special client instructions that might require additional quality control samples, special reporting limits, or special sample handling. Obtain blank data sheets and record all handwritten raw data including project information, date, technician initials, sample IDs, etc.
- 5.4 Sample preparation



- 5.4.1 For a given sample, label a 1 L amber glass bottle with the both the ARI and client sample IDs, the date, and a note indicating "water from centrifuging for solids."
- 5.4.2 Open the sample bottle. Without pouring out any sediment, pour the top water layer, if present, into the labeled 1 L amber glass bottle.
- 5.4.3 Pour any extra water volume into the evaporation pans for disposal.
- 5.4.4 Pour all sediment and other solid material from the sample bottle into a decontaminated glass centrifuge bottle that has been labeled with the ARI sample ID. You may use a decontaminated spoon to scrape out solid material; save the spoon for later use on a piece of aluminum foil labeled with the sample ID. You may also use some of the saved sample water in the amber glass bottle to rinse the sample bottle out.
- 5.4.5 There are usually two sample bottles provided for any given sample. Repeat steps 5.4.15.4.4 for the second sample bottle, using the same centrifuge and amber glass bottles. Only
  1 L of saved sample water is required, but another centrifuge bottle may be used if there is too much solid material present.
- 5.4.6 Repeat steps 5.4.1 through 5.4.5 for all samples.
- 5.4.7 Equalize the weights of all glass centrifuge bottles with a balance; the bottle weights must be within 0.1 g of each other. To balance each sample, you may use the corresponding saved sample water from the amber glass bottles. Remember to include the centrifuge bottle caps when balancing.
- 5.5 Centrifuging
- 5.5.1 The process of centrifugation separates the solids and liquid phases based on their specific gravity. During the spinning process, the denser materials move to the bottom of the bottle while the lighter materials float to the top. Extraction requires a significant difference between the specific gravity of the matrix and the specific gravity of the separated material. Some organic materials may not be significantly denser than the pore water and thus may not separate. For instance, seed pods generally float on top of the water layer. The rate of separation in a centrifuge depends on temperature, the speed and radius of the rotor, and variations in the specific gravity and particle size of the materials. Also, the centrifuge rotor's design affects calculations, as the angle of the rotor, and thus the effective radius, will change the centrifugal force.
- 5.5.2 The temperature of the centrifuge should be set to 4°C. It should be allowed come to this temperature prior to use.
- 5.5.3 Pad the centrifuge wells with rubber pads on the bottom and with foam on the sides to prevent breakage. Ensure the glass centrifuge bottles do not touch any metal of the centrifuge well. Place the glass centrifuge bottles within the padded wells, and place the wells within the centrifuge.
- 5.5.4 Turn on the centrifuge and set the dials to spin at 1000 times the force of gravity for 30 minutes. Record the sample centrifugation start time, as well as all other pertinent information, such as centrifugation speed, temperature, duration, etc.
- 5.5.5 If necessary, repeat steps 5.5.2-5.5.4 for additional samples. If centrifuging a sample twice in order to appropriately balance the centrifuge, record the second starting time rather than the first.
- 5.6 Extraction
- 5.6.1 After the samples have been centrifuged for 30 minutes, remove the samples from the centrifuge.
- 5.6.2 For a given sample, pour the separated water from the centrifuge bottle into the corresponding amber glass bottle. Pour any excess into the evaporation pans for disposal.
- 5.6.3 Estimate the amount of isolated solids to determine the appropriately sized sample jar. Label the sample jar with the corresponding stickers. If stickers have not been provided, label the jar using a sample label with the ARI and client sample IDs, the ARI job number, and sampling date and time (same as the decant time on the data sheet, step 5.6.7). Label the sample jar lid with the ARI job number and sample ID using permanent marker.



- 5.6.4 Place the labeled sample jar without the lid on the balance. Zero out the balance.
- 5.6.5 Using either the corresponding spoon saved from step 5.4.4 or an unused decontaminated spoon, transfer all solid material from the centrifuge bottle into the labeled sample jar. Record the sample weight under "Estimated Recovery" on the data sheet. Remove the sample from the balance and screw on the appropriate lid.
- 5.6.6 If necessary, a sample's solid material may be spooned into more than one sample jar. Repeat steps 5.6.3-5.6.5 and add the individual "Estimated Recovery" weights to obtain a total "Estimated Recovery" weight. Record the total weight on the data sheet instead of the individual weights.
- 5.6.7 Record the time at step 5.6.5 under "Decant Time" on the data sheet.
- 5.6.8 Repeat steps 5.6.2-5.6.7 for all samples.
- 5.7 Keep the workstation clean. Initial and date the data sheet and record any spills or corrective actions.

### 6.0 SAFETY

6.1 Keep all laboratory areas clean and follow all safety requirements.

### 7.0 REFERENCE

7.1 EPA-823-B-01-002, Methods for Collection, Storage and Manipulation of Sediments for Chemical and Toxicological Analyses: Technical Manual, 2001