

**The Bioavailability of PAHs
In The
Powell River Watershed**

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Executive Summary

The Powell River TMDL identified high polycyclic / polynuclear aromatic hydrocarbon (PAH) levels in sediment samples as a “possible stressor” to the benthic macroinvertebrate communities in the Powell River. A Monitoring Plan was developed due to different interpretations of the data.

Biological Monitoring, Inc. of Blacksburg, Virginia was contracted by the Virginia Division of Mined Land Reclamation to implement a study addressing the PAH portion of this Monitoring Plan in the Powell River Watershed. This study addressed PAH bioavailability in the watershed. It should be recognized that this study served as a screening tool. No attempt was made to evaluate PAH effect concentrations, to determine if PAH was metabolized or if equilibrium was attained, or determine the sources of PAH.

Macroinvertebrates were collected at each of the stations identified in the Monitoring Plan. Wavy-Rayed Lamprussels, *Lampsilis fasciola*, were placed *in situ* at each of these same stations for two months. After this exposure period, the mussels were brought back to the laboratory and prepared for PAH analysis.

Five PAH compounds, collectively, were detected in mussels and benthic macroinvertebrates. These data may suggest that PAHs were bioavailable in the Powell River system. On the other hand, the Monitoring Plan states that “significantly higher values in the fauna collected from sites with high PAH values would indicate bioavailability”. These criteria were not met in this screening study. Furthermore, the distribution of the PAH data does not suggest PAH bioavailability. Therefore, one may also conclude that PAHs are not bioavailable. As such, these results do not warrant elevating the status of PAH from a “possible stressor” to a “probable stressor”.

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1.0 Introduction

The Bull Creek, Levisa Fork, Pound River and Powell River TMDLs were submitted to the United States Environmental Protection Agency in 2011 as phased TMDLs. The Powell TMDL identified high polycyclic/polynuclear aromatic hydrocarbon (PAH) levels in sediment samples as a “possible stressor” to the benthic macroinvertebrate communities in the Powell River. Due to uncertainties and differences of interpretation regarding the data, a Monitoring Plan was developed. Biological Monitoring, Inc. (BMI) of Blacksburg, Virginia was contracted by the Virginia Division of Mined Land Reclamation (DMLR) to implement a study addressing the PAH bioavailability portion of this Monitoring Plan in the Powell River. It should be recognized that this study served as a screening tool for PAH bioavailability rather than definitively addressing PAHs’ role as a stressor or to identify any sources of PAH.

PAHs include several chemical compounds that are all planar molecules composed of two or more aromatic (benzene) rings, sharing at least one pair of carbon atoms. The environmentally significant PAHs are those molecules which contain two (e.g., naphthalene) to seven benzene rings (e.g., coronene). Structures and chemistries vary significantly within this range. For instance, PAH resistance to oxidation, reduction, and vaporization increases with increasing molecular weight, whereas the aqueous solubility of these compounds decreases. As a result, PAHs differ in their behavior, distribution in the environment, and their effects on biological systems. PAHs can be divided into two groups based on their physical, chemical, and biological characteristics. The lower molecular weight PAHs (e.g., two- to three-ring groups of PAHs such as naphthalenes, fluorenes, phenanthrenes, and anthracenes) are acutely toxic to aquatic organisms, whereas the high molecular weight, four- to seven-ring PAHs (from chrysenes to coronenes) are not.¹

The benthic portion of the phased Powell TMDL stated, “dissolved acute toxicity studies indicate that PAH toxicity to invertebrates is inconsistent.” According to the Powell TMDL, there are apparently no studies available on the chronic toxicity of dissolved PAHs, and very few on the acute toxicity to the benthic community. Due to the inconclusive research on their toxicity to benthics, and due to the unidentified sources in the Powell River, PAHs were considered as a “possible stressor” in the phased TMDL.

The most common PAH, and that most commonly found in the Powell River, was naphthalene. Naphthalene has the lowest molecular weight of any PAH and is highly volatile. It is typically a white solid substance that evaporates easily, used in mothballs, moth flakes and tar camphor. It usually enters the environment by the burning of wood or fossil fuels, and is present in both coal and petroleum. Due to its high volatility, naphthalene typically evaporates from rivers or lakes within two weeks. It also binds very weakly to soil and sediments.

Naphthalene and 2-methyl naphthalene were detected in sediment samples taken at several sites and at different times in the Powell River Watershed during TMDL development. Therefore it was speculated that either there is an active, widely distributed source of naphthalene in the watershed, or that the naphthalene being detected is bound up in coal sediments and is not bioavailable.^{2,3} The objective of the monitoring effort was to determine whether PAHs are bioavailable in the Powell River system. It was not within the scope of this study to determine the fate of or the sources of PAH in the Powell River Watershed.

2.0 Methods and Materials

2.1 General

This study addressed PAH bioavailability at five stations. Station locations are shown on the map in Figure 1. A summary of station attributes is presented as Table 1. Photographs of these stations are presented as Figures 2-6.

The sampling locations were specified in the Monitoring Plan. These sites were located at or near stations from the Powell River TMDL. These stations were recommended in the Monitoring Plan because of their sediment PAH levels. Callahan Creek, Roaring Fork, and Powell River 1 (downstream) were designated as having high sediment PAH levels in the TMDL. South Fork of the Powell River and Powell River 2 (upstream) were designated as having low sediment PAH levels in the TMDL.

The Monitoring Plan suggested the use of field collected macroinvertebrates and, if available, mussels to serve as the indicator target organisms. Both macroinvertebrates and mussels were employed to address PAH bioavailability. Macroinvertebrates were sampled and mussels were placed at each of the five station locations cited in the Monitoring Plan. The overall study design incorporated US EPA bioaccumulation study guidelines.^{4,5}

2.2 Macroinvertebrate Study

2.2.1 Collection

Macroinvertebrates were collected on November 28th, 2012. Based upon the quantitation limits achieved by the contract laboratory, a second collection was necessary. This occurred on January 29th, 2013.

The macroinvertebrate study directly surveyed PAH levels in population samples of the benthic organisms at the five stations identified in the Monitoring Plan. Composite samples of benthic macroinvertebrates were collected at each of the five stations using a 0.5 m wide rectangular kick-net with 500- μ m mesh openings. Samples were collected by placing the net on the bottom of the stream, downstream of the area to be sampled, and vigorously kicking the cobble substrate

to agitate and dislodge organisms. Where appropriate, large rocks and debris were removed and discarded after any attached macroinvertebrates were brushed off into the net. All available habitats were sampled at each station. The collected samples were subsequently placed in five-gallon polypropylene plastic containers containing site water, and aerated for the transport to BMI's laboratory for processing.

2.2.2 Preparation for Analysis

The macroinvertebrates were sorted from the debris and placed in appropriate vessels containing BMI culture water (Table 2). BMI culture water is prepared from Blacksburg, Virginia municipal tap water. The tap water is dechlorinated using activated carbon, deionized using mixed bed deionization tanks to a resistivity of > 1 megohm-cm, and polished with a Millipore, Milli Q UV Plus and 0.2 micron filter to a final resistivity of ≥ 18 megohm-cm. This water is referred to as BMI Deionized (DI) water. The DI water is then reconstituted to Moderately Hard Reconstituted Water (MHRW) as per EPA Acute and Chronic Toxicity Manuals.^{6,7} MHRW is used at BMI for culturing and as dilution water in testing.

The macroinvertebrates were maintained in BMI culture water at 15 ± 1 °C for two hours to enable gut purging. This gut purging allowed for sediment removal from the digestive tract, and reflects US EPA guidance.^{4,5} After purging, the macroinvertebrates were frozen and shipped to a laboratory contracted by DMLR (REIC Laboratory in Beaver, WV) for PAH analysis via EPA Method SW 8270D. Table 3 lists the PAH analytes with their respective target detection limits.

2.3 Mussel Study

Freshwater mussels were exposed *in situ* using mussel silos (Figure 7) placed at the monitoring stations. The Monitoring Plan suggested that mussels be used for the study. Other research suggests that mussels would be a good indicator organism due to the fact that they are filter-feeders which consume microorganisms in the water column as well as suspended particles and pollutants.⁸

The mussel portion of the study was designed following discussions with key malacologists and toxicologists at the USGS Columbia Environmental Research Center, the Virginia Department of Game & Inland Fisheries Freshwater Mussel Program, the Biology Department at Missouri State

University, and Virginia Tech's Freshwater Mollusk Conservation Center. The silo design was modified from prior designs developed by groups such as Barnhart et al.⁹ The silos consisted of a hemispherical concrete dome with an inner chamber housing the mussels. The dome shape causes water flowing over the hemisphere to speed up, creating a Bernoulli Effect that draws water upward through the chamber. The silos were constructed with channels underneath to allow flow into the chambers. Figure 7 depicts the expected flow of water through the silos.

2.3.1 Silo Construction

For each silo, a 10-cm-diameter PVC pipe was cut into an 11.5 cm section and capped at one end with a 10 cm PVC grid (floor drain). The section was placed grid-end first into a 28-cm-diameter hemispherical mold (stainless steel mixing bowl). After securing the section into position with a weight, concrete was poured into the mold around it. Nylon rope was inserted into the wet concrete to create handles for transporting the finished silos. Blocks of wood were then pressed into the concrete to create channels that would allow water to flow into the center. Once the concrete was dry, the domes were removed from the molds. Excess concrete was removed by chiseling or sanding. The domes were then sprayed with flat black paint and allowed to dry.

Inner chambers to house the mussels were constructed of 7.5-cm-diameter PVC pipe, cut into 8.5 cm sections and capped at both ends with 7.5 cm PVC grids. These inner chambers were placed inside the larger 10 cm PVC sections within the silos. Finally the 10 cm PVC pipe was capped with a second 10 cm PVC grid.

2.3.2 Mussel Preparation and Handling

On November 16, Wavy-Rayed Lampmussels (*Lampsilis fasciola*) were obtained from the VA Tech Freshwater Mollusk Conservation Center in Blacksburg. The mussels had shell lengths ranging from approximately 2 to 2.5 cm and bodies weighing approximately 0.5 g each. They were placed in BMI culture water and maintained at $15 \pm 1^\circ\text{C}$.

2.3.2.1 Control Groups

A group of these mussels was isolated and designated as a control. The whole bodies were removed from each mussel shell and frozen on November 27, 2012. Approximately 20 g (wet weight) of mussel tissue represented this control group. This sample was delivered to the

contract laboratory, along with the first group of macroinvertebrates collected, for PAH analysis (EPA Method SW 8270D) on December 7, 2012.

The contract laboratory did not achieve the required target detection limit when analyzing the control group. BMI therefore prepared a second group of controls for the contract laboratory to analyze. This second group had been maintained in BMI culture water for approximately two months, and had been fed algae and yeast-cerophyl-trout chow during the holding period. The whole bodies were removed from each mussel shell and frozen on January 11, 2013. This sample was shipped overnight to the contract laboratory for PAH analysis on January 22, 2013.

2.3.2.2 Experimental Groups

Twenty mussels were placed in each of ten PVC chambers. The mussels were delivered in BMI culture water to the five stations identified in the Monitoring Plan on November 28, 2012. Upon arrival, each chamber was inserted into a silo. Two silos were placed in the stream bed at each station and staked down.

After one month, on December 31, 2012, BMI personnel returned to observe the two mussel silos located at each of the five stations. Debris was removed from the stakes and any sediment was removed from the mussel chambers. Since no mussels were found to be agape, the decision was made to leave them in place for one additional month.

The mussels were collected after two months of continuous exposure on January 29, 2013. The inner PVC chambers housing the mussels were removed from the silos and placed directly into five-gallon polypropylene plastic containers containing site water. These containers were aerated for transport to BMI's laboratory in Blacksburg.

The mussels were removed from the PVC chambers and the two samples from each station were composited. The composite samples were placed in appropriate vessels containing BMI culture water (15 ± 1 °C) for six hours to enable gut purging. This gut purging allows for sediment removal from the digestive tract, and reflects US EPA guidance.^{4,5} The whole bodies were removed from the shells and frozen on January 30, 2013. These samples were shipped overnight to the contract laboratory for PAH analysis on February 11, 2013.

3.0 Results

3.1 Macroinvertebrate Study Data

Appendix A presents the three data reports generated by the contract laboratory. The first report was dated January 7 & 8, 2013. This data set represented the first set of macroinvertebrate collections and a control group of mussels. Target Practical Quantitation Limits (PQLs) were not achieved by these analyses. The second report was dated February 7, 2013. This data represents the second set of control group mussels. This set was sent to the contract laboratory independent of other samples to assure desired PQLs were achievable. The third data report was dated February 27, 2013. This data represents the analyses from the second set of macroinvertebrate collections and the mussels retrieved from the silos.

Fluoranthene, phenanthrene, and pyrene were detected in the benthic macroinvertebrate samples collected. Fluoranthene was measured only in the Callahan Creek sample at a concentration of 0.317 mg/Kg. Phenanthrene was also measured only in the Callahan Creek sample at a concentration of 0.317 mg/Kg. Pyrene was found in the sample from Callahan Creek at 0.286 mg/Kg. Pyrene was detected in the sample from Powell River 1 at 0.07716 mg/Kg. However, this reported concentration was flagged by the contract laboratory as an estimate. All PAH compounds were reported as not detected in the samples from Powell River 2, Roaring Fork, and South Fork Powell. Data are presented as Table 4. The analytical data are presented as Appendix A.

3.2 Mussel Study Data

Appendix A presents the three data reports generated by the contract laboratory. The first report was dated January 7 & 8, 2013. This data set represented the first set of macroinvertebrate collections and a control group of mussels. Target Practical Quantitation Limits (PQLs) were not achieved by these analyses. The second report was dated February 7, 2013. This data represents the second set of control group mussels. This set was sent to the contract laboratory independent of other samples to assure desired PQLs were achievable. The third data report was dated February 27, 2013. This data represents the analyses from the second set of macroinvertebrate collections and the mussels retrieved from the silos.

Upon retrieval at the end of the study, all but three mussels survived. Furthermore, upon dissecting and preparing the mussels for analysis, it was observed that they appeared plump and robust.

PAHs were not detected in the control group of mussels. Benzo(a)pyrene and indeno(1,2,3-cd)pyrene were detected in the mussel samples collected. Benzo(a)pyrene was detected in the mussel tissue from three different stations. In the Powell River 2 sample, the concentration of benzo(a)pyrene was 0.128 mg/Kg. The Callahan Creek sample concentration was 0.164 mg/Kg, and the South Fork Powell sample concentration was 0.161 mg/Kg. Indeno(1,2,3-cd)pyrene was found in mussel tissue samples from the Callahan Creek, South Fork Powell, and Powell River 1 stations. In the Callahan Creek sample, the concentration was 0.238 mg/Kg. The South Fork Powell sample concentration was 0.249 mg/Kg, while The Powell River 1 sample concentration was 0.328 mg/Kg. All PAH compounds were reported as not detected in the sample from Roaring Fork. Data are presented as Table 5. The analytical data are presented as Appendix A.

4.0 Discussion

Bioavailability refers to the bioaccessibility of substances that are absorbed or adsorbed by an organism with the potential for distribution, metabolism, elimination, and bioaccumulation. Bioaccumulation occurs when an organism absorbs or adsorbs a substance at a rate greater than that at which the substance is lost. Bioaccumulation refers to the build-up of substances in an organism from all sources. Bioconcentration is a related but more specific term, referring to uptake and accumulation of a substance from water alone. The fact that a substance is bioavailable, or even that it bioaccumulates, doesn't necessarily mean there will be a negative effect.

The issue raised by the TMDL was whether PAHs constitute a "probable stressor" to the benthic communities in the Powell River Watershed. However, this study was designed to address whether PAHs in the Powell River Watershed are bioavailable. This study was designed to serve as a screening tool rather than to definitively quantify PAHs' role in benthic impairment. No attempt was made to determine whether the concentrations found in the tissue were at levels that may affect benthic populations. No attempt was made to determine if PAH was metabolized, eliminated, or if equilibrium was attained. No attempt was made to determine the sources or fate of PAH in the Powell River Watershed. Finally, no attempt was made to confirm the sediment PAH findings of the TMDL.

Nonetheless, these screening data suggest some PAHs may be bioavailable in the Powell River Watershed, simply because some were found in the tissues analyzed. On the other hand, the Monitoring Plan states that "significantly higher values in the fauna collected from sites with high PAH values would indicate bioavailability". These criteria were not met in this screening study. Therefore, one may also conclude that PAHs are not bioavailable. This is further evidenced by one of the sites designated as having high sediment PAH levels (Roaring Fork) producing results of no detects in both groups of study organisms.

The Powell River TMDL and subsequent Monitoring Plan specifically identified naphthalene as a PAH of particular concern. This was due to the high levels detected in the Powell River

sediments at several sites and at different times. However, naphthalene was not detected in either mussel or macroinvertebrate tissues in this study.

Although initially the contract laboratory did not achieve adequate PQLs, they ultimately achieved Practical Quantitation Limits (PQLs) lower than the target detection limits requested as can be seen presented in Table 4 and 5. Of the seventeen PAHs assessed for this study, only five were detected. Additionally, all detected values were below the target detection limits of 0.33 mg/Kg specified in the Monitoring Plan.

Mussels and macroinvertebrates accumulated different PAH compounds. This may be attributed to different mechanisms of bioaccumulation. Three PAH compounds were detected only in macroinvertebrate samples. Those compounds were fluoranthene, phenanthrene, and pyrene. Two PAH compounds were detected only in mussel samples. Those compounds were benzo(a)pyrene and indeno(1,2,3-cd)pyrene.

Three of the five stations in this study were categorized in the Powell River TMDL as having “high” levels of sediment PAHs. Those stations were Callahan Creek, Roaring Fork, and Powell River 1. All five PAH compounds found in this study were detected in the Callahan Creek samples. None of the PAHs screened were detected in the Roaring Fork samples. The Powell River 1 samples had only indeno(1,2,3-cd)pyrene in the mussels.

Two stations in the study were categorized as “low” levels of sediment PAHs in the Powell River TMDL and served as a reference for this study. These stations were South Fork Powell River and Powell River 2. None of the PAH compounds were detected in the benthic macroinvertebrate samples for these two stations. Benzo(a)pyrene and indeno(1,2,3-cd)pyrene were detected in mussels in the South Fork Powell River samples. Only benzo(a)pyrene was found in the Powell River 2 mussel sample. Since these two stations served as a reference for this study, the detection of these two compounds requires consideration when evaluating these results.

One might be tempted to conclude that some of the PAHs are bioavailable to some of the biota in the Powell River Watershed simply based on their presence in tissues analyzed. On the other hand, one might also conclude that PAHs are not bioavailable based on the suggested criteria for determining bioavailability as presented in the Monitoring Plan, as well as, the distribution of the PAH data as compared to the expectations presented in the Monitoring Plan. It should also be noted that Naphthalene, the predominant form of PAH identified in Powel River sediments in the TMDL, were not detected in the tissues assessed in this study. This study does not provide sufficient evidence that PAH should be elevated to a “probable stressor”. However, additional studies would be necessary to address:

- Are the results repeatable?
- Are current conditions reflective of the levels of PAHs in sediment presented in the TMDL?
- What are the aquatic life-based concentrations of concern for PAH bioaccumulation?
- Are there seasonal considerations?
- Were steady state kinetics of the PAHs reached?
- What are the sources of PAHs? and,
- What are the routes of exposure to aquatic organisms of specific PAHs?

5.0 Literature Cited

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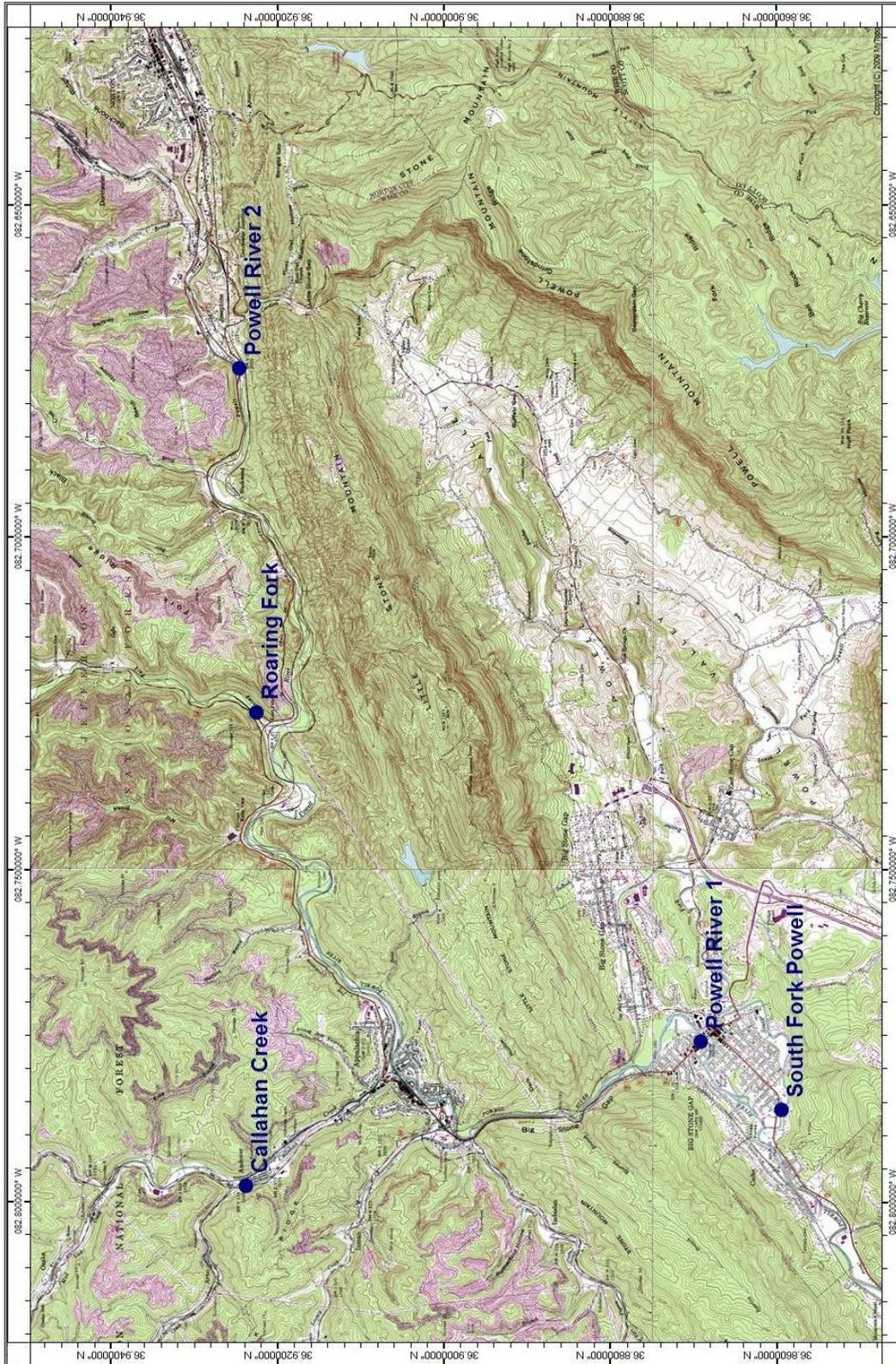


Figure 1. Study Area Map



(a) Facing upstream

(b) Facing downstream

Figure 2. Powell River 2



(a) Facing upstream

(b) Facing downstream

Figure 3. Roaring Fork



(a) Facing upstream

(b) Facing downstream

Figure 4. Callahan Creek



(a) Facing upstream

(b) Right ascending view from an island

Figure 5. Powell River 1



(a) Facing upstream

(b) Facing downstream

Figure 6. South Fork Powell

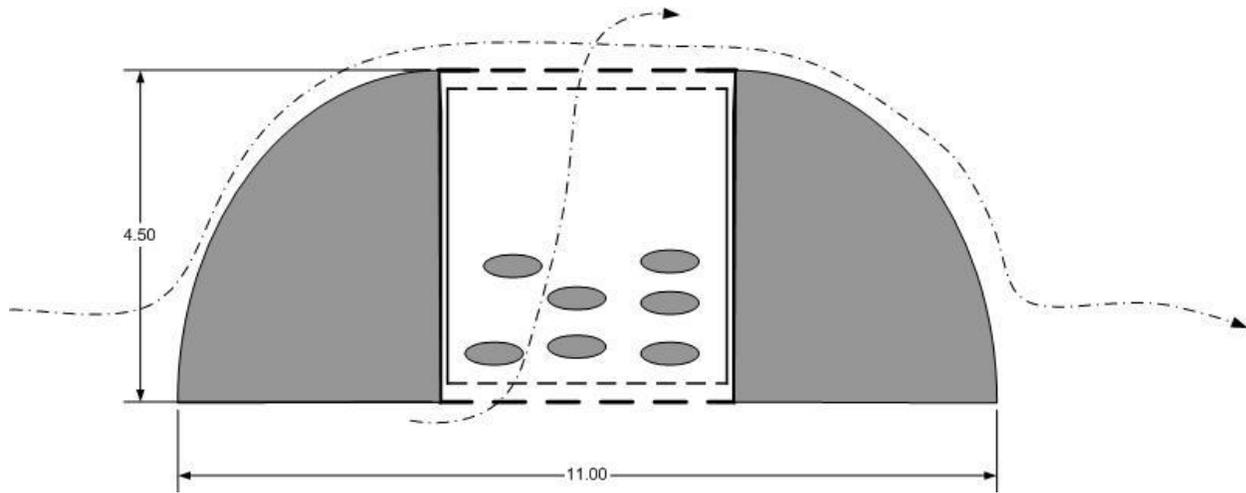


Figure 7. Mussel Silo Design

Dashed lines indicate direction of flow when silos are placed on the bottom of a moving stream.

Table 1. Station Attributes

| Station ID | Water Body | PAH Level | Latitude | Longitude |
|-------------|-------------------------|-----------|----------|-----------|
| 6BCAL001.57 | Callahan Creek | High* | 36.9237 | -82.7975 |
| 6BRIN000.31 | Roaring Fork | High* | 36.9225 | -82.7264 |
| 6BPOW179.20 | Powell River 1 | High* | 36.8691 | -82.7758 |
| 6BPLL000.27 | South Fork Powell River | Low | 36.8594 | -82.7861 |
| 6BPOW193.38 | Powell River 2 | Low | 36.9246 | -82.6746 |

* According to the Powell River TMDL, "High" indicates that the PAH levels at these stations exceed VADEQ screening values

Table 2. Preparation of BMI Culture Water

| | Reagent Added (mg/L) | | | | Approximate Final Water Quality | | |
|-------------|----------------------|--|-------------------|-----|---------------------------------|----------|------------|
| | NaHCO ₃ | CaSO ₄ + 2H ₂ O | MgSO ₄ | KCl | pH | Hardness | Alkalinity |
| MHRW | 96.0 | 60.0 | 60.0 | 4.0 | 7.4-7.8 | 80-100 | 57-64 |

Table 3. PAH analytes listed with target detection limits.

| PAH Analyte | Target Detection Limit (ppb) |
|------------------------|-------------------------------------|
| Acenaphthene | 330 |
| Acenaphthylene | 330 |
| Anthracene | 330 |
| Benzo(a)anthracene | 330 |
| Benzo(a)pyrene | 330 |
| Benzo(b)fluoranthene | 330 |
| Benzo(g,h,i)perylene | 330 |
| Benzo(k)fluoranthene | 330 |
| Chrysene | 330 |
| Dibenzo(a,h)anthracene | 330 |
| Fluoranthene | 330 |
| Fluorene | 330 |
| Indeno(1,2,3-cd)pyrene | 330 |
| Naphthalene | 330 |
| Phenanthrene | 330 |
| Pyrene | 330 |
| 2-Methyl naphthalene | 83 |

Table 4. Benthic Macroinvertebrate Results

| STATION | | | | | | | | | | | | |
|------------------------|--------|--------|--------------|--------|----------------|--------|--------|---------------------|-----------|--------|-------|--|
| | PR 2 | | Roaring Fork | | Callahan Creek | | PR 1 | | SF Powell | | Units | |
| | PQL | Result | PQL | Result | PQL | Result | PQL | Result | PQL | Result | | |
| Percent Moisture | 0.5 | 84 | 0.5 | 80 | 0.5 | 90 | 0.5 | 86 | 0.5 | 88 | wt% | |
| Anthracene | 0.167 | ND | 0.108 | ND | 0.157 | ND | 0.154 | ND | 0.137 | ND | mg/Kg | |
| Acenaphthene | 0.167 | ND | 0.108 | ND | 0.157 | ND | 0.154 | ND | 0.137 | ND | mg/Kg | |
| Acenaphthylene | 0.167 | ND | 0.108 | ND | 0.157 | ND | 0.154 | ND | 0.137 | ND | mg/Kg | |
| Benzo(a)anthracene | 0.167 | ND | 0.108 | ND | 0.157 | ND | 0.154 | ND | 0.137 | ND | mg/Kg | |
| Benzo(a)pyrene | 0.167 | ND | 0.108 | ND | 0.157 | ND | 0.154 | ND | 0.137 | ND | mg/Kg | |
| Benzo(b)fluoranthene | 0.167 | ND | 0.108 | ND | 0.157 | ND | 0.154 | ND | 0.137 | ND | mg/Kg | |
| Benzo(g,h,i)perylene | 0.167 | ND | 0.108 | ND | 0.157 | ND | 0.154 | ND | 0.137 | ND | mg/Kg | |
| Benzo(k)fluoranthene | 0.167 | ND | 0.108 | ND | 0.157 | ND | 0.154 | ND | 0.137 | ND | mg/Kg | |
| Chrysene | 0.167 | ND | 0.108 | ND | 0.157 | ND | 0.154 | ND | 0.137 | ND | mg/Kg | |
| Dibenzo(a,h)anthracene | 0.167 | ND | 0.108 | ND | 0.157 | ND | 0.154 | ND | 0.137 | ND | mg/Kg | |
| Fluoranthene | 0.167 | ND | 0.108 | ND | 0.157 | 0.317 | 0.154 | ND | 0.137 | ND | mg/Kg | |
| Fluorene | 0.167 | ND | 0.108 | ND | 0.157 | ND | 0.154 | ND | 0.137 | ND | mg/Kg | |
| Indeno(1,2,3-cd)pyrene | 0.167 | ND | 0.108 | ND | 0.157 | ND | 0.154 | ND | 0.137 | ND | mg/Kg | |
| 2-Methyl naphthalene | 0.0846 | ND | 0.0545 | ND | 0.0795 | ND | 0.0778 | ND | 0.0696 | ND | mg/Kg | |
| Naphthalene | 0.167 | ND | 0.108 | ND | 0.157 | ND | 0.154 | ND | 0.137 | ND | mg/Kg | |
| Phenanthrene | 0.167 | ND | 0.108 | ND | 0.157 | 0.317 | 0.154 | ND | 0.137 | ND | mg/Kg | |
| Pyrene | 0.167 | ND | 0.108 | ND | 0.157 | 0.286 | 0.154 | 0.0776 ^J | 0.137 | ND | mg/Kg | |

ND: Not Detected at the PQL or MDL

J: Analyte concentration is reported, and is less than the PQL and greater than or equal to the MDL. The result reported is an estimate

Table 5. Mussel Results

| | STATION | | | | | | | | | | | |
|------------------------|---------|--------|--------------|--------|----------------|--------|--------|--------|-----------|--------|-------|--|
| | PR 2 | | Roaring Fork | | Callahan Creek | | PR 1 | | SF Powell | | | |
| | PQL | Result | PQL | Result | PQL | Result | PQL | Result | PQL | Result | Units | |
| Percent Moisture | 0.5 | 87 | 0.5 | 90 | 0.5 | 89 | 0.5 | 90 | 0.5 | 89 | wt% | |
| Anthracene | 0.127 | ND | 0.165 | ND | 0.148 | ND | 0.162 | ND | 0.145 | ND | mg/Kg | |
| Acenaphthene | 0.127 | ND | 0.165 | ND | 0.148 | ND | 0.162 | ND | 0.145 | ND | mg/Kg | |
| Acenaphthylene | 0.127 | ND | 0.165 | ND | 0.148 | ND | 0.162 | ND | 0.145 | ND | mg/Kg | |
| Benzo(a)anthracene | 0.127 | ND | 0.165 | ND | 0.148 | ND | 0.162 | ND | 0.145 | ND | mg/Kg | |
| Benzo(a)pyrene | 0.127 | 0.128 | 0.165 | ND | 0.148 | 0.164 | 0.162 | ND | 0.145 | 0.161 | mg/Kg | |
| Benzo(b)fluoranthene | 0.127 | ND | 0.165 | ND | 0.148 | ND | 0.162 | ND | 0.145 | ND | mg/Kg | |
| Benzo(g,h,i)perylene | 0.127 | ND | 0.165 | ND | 0.148 | ND | 0.162 | ND | 0.145 | ND | mg/Kg | |
| Benzo(k)fluoranthene | 0.127 | ND | 0.165 | ND | 0.148 | ND | 0.162 | ND | 0.145 | ND | mg/Kg | |
| Chrysene | 0.127 | ND | 0.165 | ND | 0.148 | ND | 0.162 | ND | 0.145 | ND | mg/Kg | |
| Dibenzo(a,h)anthracene | 0.127 | ND | 0.165 | ND | 0.148 | ND | 0.162 | ND | 0.145 | ND | mg/Kg | |
| Fluoranthene | 0.127 | ND | 0.165 | ND | 0.148 | ND | 0.162 | ND | 0.145 | ND | mg/Kg | |
| Fluorene | 0.127 | ND | 0.165 | ND | 0.148 | ND | 0.162 | ND | 0.145 | ND | mg/Kg | |
| Indeno(1,2,3-cd)pyrene | 0.127 | ND | 0.165 | ND | 0.148 | 0.238 | 0.162 | 0.328 | 0.145 | 0.249 | mg/Kg | |
| 2-Methyl naphthalene | 0.0642 | ND | 0.0835 | ND | 0.0747 | ND | 0.0821 | ND | 0.145 | ND | mg/Kg | |
| Naphthalene | 0.127 | ND | 0.165 | ND | 0.148 | ND | 0.162 | ND | 0.145 | ND | mg/Kg | |
| Phenanthrene | 0.127 | ND | 0.165 | ND | 0.148 | ND | 0.162 | ND | 0.145 | ND | mg/Kg | |
| Pyrene | 0.127 | ND | 0.165 | ND | 0.148 | ND | 0.162 | ND | 0.145 | ND | mg/Kg | |

ANALYTE

ND: Not Detected at the PQL or MDL

J: Analyte concentration is reported, and is less than the PQL and greater than or equal to the MDL. The result reported is an estimate

Appendix A: Lab Reports



Improving the environment, one client at a time...

225 Industrial Park Drive
Beaver, WV 25813
TEL: 304.255.2500
FAX: 304.255.2572

3029-C Peters Creek Road
Roanoke, VA 24019
TEL: 540.777.1276
FAX: 540.400.8508

101 17th Street
Ashland, KY 41101
TEL: 606.393.5027

1557 Commerce Road, Suite 201
Verona, VA 24482
TEL: 540.248.0183

December 18, 2012

Mr. Joey O'Quinn
VIRGINIA DEPARTMENT OF MINES, MINERALS, A
P.O. DRAWER 900
3405 MTN EMPIRE ROAD
BIG STONE GAP VA 24219

TEL: (276) 523-8179

FAX

RE: DMME-HARMON

Order No.: 1212762

Dear Mr. Joey O'Quinn:

REI Consultants, Inc. received 6 sample(s) on 12/7/2012 for the analyses presented in the following report.

Please note two changes you may see on your report.

- Results for "Dissolved" parameters will be shown under a separate sample ID, rather than as a separate analysis under the same sample ID. The sample ID for "Dissolved" parameters will include "Field Filtered" or "Lab Filtered", as appropriate.
- Metals results will no longer be identified as "Total" or "Total Recoverable". The methods have not been changed, only their appearance on the report.

If you have any questions regarding these results, please do not hesitate to call.

Sincerely,

Scott Gross

Project Manager





Improving the environment, one client at a time...

225 Industrial Park Drive
Beaver, WV 25813
TEL: 304.255.2500
FAX: 304.255.2572

3029-C Peters Creek Road
Roanoke, VA 24019
TEL: 540.777.1276
FAX: 540.400.8508

101 17th Street
Ashland, KY 41101
TEL: 606.393.5027

1557 Commerce Road, Suite 201
Verona, VA 24482
TEL: 540.248.0183

Report Narrative

Project Manager:: **Scott Gross**

WO#: **1212762**

Date: **12/18/2012**

CLIENT: VIRGINIA DEPARTMENT OF MINES,
Project: DMME-HARMON

The analytical results presented in this report relate only to the samples documented herein. All analyses were performed using documented laboratory SOPs that incorporate appropriate quality control procedures as described in the applicable methods. Any deviation from compliance or method modification is explained below and/or identified within the body of this report by a qualifier footnote which is defined at the bottom of each page.

All sample results are reported on an "as-received" wet weight basis unless otherwise noted.

Results reported for sums of individual parameters, such as Total Trihalomethanes (TTHM) and Total Haloacetic Acids (HAA5), may vary slightly from the sum of the individual parameter results. This apparent anomaly is caused by rounding individual results and summations at reporting, as required by EPA.

Following standard laboratory protocol, sample preservation, such as pH, is verified at time of extraction or analysis based on client requested parameters. Improper preservation is noted on the analytical bench sheet, extraction log, or preservation log and client is notified by close of following business day. All results are reported using preservation compliant samples unless otherwise noted in the analytical report.

The test results in this report meet all NELAP requirements for parameters for which accreditations are required or available. Any exceptions are noted in this report. This report may not be reproduced, except in full, without the written approval of REIC.

In compliance with federal guidelines and standard operating procedures, all reports, including raw data and supporting quality control, will be disposed of after five years unless otherwise arranged by the client via written notification or contract requirement.

If you have any questions please contact the project manager whose name is listed above.

Beaver, WV: WVDHHR 00412CM, WVDEP 060, VADCLS 00281, KYDEP 90039, TNDEQ TN02926, NCDWQ 466, PADEP 68-00839, FLDOH (NELAC) E87958,
VADCLS (VELAP) 460148
Bioassay (Beaver, WV) WVDEP 060, FLDOH (NELAC) E871055, VADCLS (VELAP) 460149
Roanoke, VA: VADCLS (VELAP) 460150
Verona, VA: VADCLS (VELAP) 460157
Ashland, KY: KYDEP 00094

CLIENT: VIRGINIA DEPARTMENT OF MINES, MINERALS, A **WorkOrder** 1212762 **Lab ID** 1212762-01A
Client Sample ID: L. FASCIOLA CONTROL GROUP **DateReceived** 12/7/2012
Project: DMME-HARMON **Collection Date:** 11/27/2012 3:00:00 PM
Site ID: **Matrix:** TISSUE

| Analyses | Result | Units | Qual | PQL | MCL | Prep Date | Date Analyzed |
|---------------------------------------|--------|-----------|-----------------|----------|-----|--------------------|-------------------|
| PERCENT MOISTURE | | | SM2540 B | | | Analyst: SL | |
| Percent Moisture | 89 | wt% | | 0.5 | NA | | 12/14/12 12:00 AM |
| SEMIVOLATILE ORGANIC COMPOUNDS | | | SW8270D | | | Analyst: JD | |
| Acenaphthene | ND | mg/Kg-dry | | 25.2 | NA | 12/14/12 8:27 AM | 12/17/12 5:11 PM |
| Acenaphthylene | ND | mg/Kg-dry | | 25.2 | NA | 12/14/12 8:27 AM | 12/17/12 5:11 PM |
| Anthracene | ND | mg/Kg-dry | | 25.2 | NA | 12/14/12 8:27 AM | 12/17/12 5:11 PM |
| Benzo(a)anthracene | ND | mg/Kg-dry | | 25.2 | NA | 12/14/12 8:27 AM | 12/17/12 5:11 PM |
| Benzo(a)pyrene | ND | mg/Kg-dry | | 25.2 | NA | 12/14/12 8:27 AM | 12/17/12 5:11 PM |
| Benzo(b)fluoranthene | ND | mg/Kg-dry | | 25.2 | NA | 12/14/12 8:27 AM | 12/17/12 5:11 PM |
| Benzo(g,h,i)perylene | ND | mg/Kg-dry | | 25.2 | NA | 12/14/12 8:27 AM | 12/17/12 5:11 PM |
| Benzo(k)fluoranthene | ND | mg/Kg-dry | | 25.2 | NA | 12/14/12 8:27 AM | 12/17/12 5:11 PM |
| Chrysene | ND | mg/Kg-dry | | 25.2 | NA | 12/14/12 8:27 AM | 12/17/12 5:11 PM |
| Dibenzo(a,h)anthracene | ND | mg/Kg-dry | | 25.2 | NA | 12/14/12 8:27 AM | 12/17/12 5:11 PM |
| Fluoranthene | ND | mg/Kg-dry | | 25.2 | NA | 12/14/12 8:27 AM | 12/17/12 5:11 PM |
| Fluorene | ND | mg/Kg-dry | | 25.2 | NA | 12/14/12 8:27 AM | 12/17/12 5:11 PM |
| Indeno(1,2,3-cd)pyrene | ND | mg/Kg-dry | | 25.2 | NA | 12/14/12 8:27 AM | 12/17/12 5:11 PM |
| Naphthalene | ND | mg/Kg-dry | | 25.2 | NA | 12/14/12 8:27 AM | 12/17/12 5:11 PM |
| Phenanthrene | ND | mg/Kg-dry | | 25.2 | NA | 12/14/12 8:27 AM | 12/17/12 5:11 PM |
| Pyrene | ND | mg/Kg-dry | | 25.2 | NA | 12/14/12 8:27 AM | 12/17/12 5:11 PM |
| Surr: 2-Fluorophenol | 76.6 | %REC | | 54.1-110 | NA | 12/14/12 8:27 AM | 12/17/12 5:11 PM |
| Surr: Phenol-d5 | 90.3 | %REC | | 60.1-110 | NA | 12/14/12 8:27 AM | 12/17/12 5:11 PM |
| Surr: 2,4,6-Tribromophenol | 90.6 | %REC | | 58.8-128 | NA | 12/14/12 8:27 AM | 12/17/12 5:11 PM |
| Surr: Nitrobenzene-d5 | 78.9 | %REC | | 68.2-123 | NA | 12/14/12 8:27 AM | 12/17/12 5:11 PM |
| Surr: 2-Fluorobiphenyl | 87.8 | %REC | | 68.4-116 | NA | 12/14/12 8:27 AM | 12/17/12 5:11 PM |
| Surr: 4-Terphenyl-d14 | 83.0 | %REC | | 52.2-121 | NA | 12/14/12 8:27 AM | 12/17/12 5:11 PM |

Key: MCL Maximum Contaminant Level **Qualifiers:** B Analyte detected in the associated Method Blank
 MDL Minimum Detection Limit E Estimated Value above quantitation range
 NA Not Applicable H Holding times for preparation or analysis exceeded
 ND Not Detected at the PQL or MDL S Spike/Surrogate Recovery exceeds REIC control limits
 PQL Practical Quantitation Limit * Value exceeds MCL or Regulatory Limits Page 2 of 7
 TIC Tentatively Identified Compound, Estimated Concentration

| | | | | | |
|--------------------------|---|-------------------------|-----------------------|---------------|-------------|
| CLIENT: | VIRGINIA DEPARTMENT OF MINES, MINERALS, A | WorkOrder | 1212762 | Lab ID | 1212762-02A |
| Client Sample ID: | POWELL RIVER 2 | DateReceived | 12/7/2012 | | |
| Project: | DMME-HARMON | Collection Date: | 11/28/2012 9:00:00 AM | | |
| Site ID: | | Matrix: | TISSUE | | |

| Analyses | Result | Units | Qual | PQL | MCL | Prep Date | Date Analyzed |
|---------------------------------------|--------|-----------|-----------------|----------|-----|--------------------|-------------------|
| PERCENT MOISTURE | | | SM2540 B | | | Analyst: SL | |
| Percent Moisture | 90 | wt% | | 0.5 | NA | | 12/14/12 12:00 AM |
| SEMIVOLATILE ORGANIC COMPOUNDS | | | SW8270D | | | Analyst: JD | |
| Acenaphthene | ND | mg/Kg-dry | | 31.2 | NA | 12/14/12 8:27 AM | 12/17/12 5:39 PM |
| Acenaphthylene | ND | mg/Kg-dry | | 31.2 | NA | 12/14/12 8:27 AM | 12/17/12 5:39 PM |
| Anthracene | ND | mg/Kg-dry | | 31.2 | NA | 12/14/12 8:27 AM | 12/17/12 5:39 PM |
| Benzo(a)anthracene | ND | mg/Kg-dry | | 31.2 | NA | 12/14/12 8:27 AM | 12/17/12 5:39 PM |
| Benzo(a)pyrene | ND | mg/Kg-dry | | 31.2 | NA | 12/14/12 8:27 AM | 12/17/12 5:39 PM |
| Benzo(b)fluoranthene | ND | mg/Kg-dry | | 31.2 | NA | 12/14/12 8:27 AM | 12/17/12 5:39 PM |
| Benzo(g,h,i)perylene | ND | mg/Kg-dry | | 31.2 | NA | 12/14/12 8:27 AM | 12/17/12 5:39 PM |
| Benzo(k)fluoranthene | ND | mg/Kg-dry | | 31.2 | NA | 12/14/12 8:27 AM | 12/17/12 5:39 PM |
| Chrysene | ND | mg/Kg-dry | | 31.2 | NA | 12/14/12 8:27 AM | 12/17/12 5:39 PM |
| Dibenzo(a,h)anthracene | ND | mg/Kg-dry | | 31.2 | NA | 12/14/12 8:27 AM | 12/17/12 5:39 PM |
| Fluoranthene | ND | mg/Kg-dry | | 31.2 | NA | 12/14/12 8:27 AM | 12/17/12 5:39 PM |
| Fluorene | ND | mg/Kg-dry | | 31.2 | NA | 12/14/12 8:27 AM | 12/17/12 5:39 PM |
| Indeno(1,2,3-cd)pyrene | ND | mg/Kg-dry | | 31.2 | NA | 12/14/12 8:27 AM | 12/17/12 5:39 PM |
| Naphthalene | ND | mg/Kg-dry | | 31.2 | NA | 12/14/12 8:27 AM | 12/17/12 5:39 PM |
| Phenanthrene | ND | mg/Kg-dry | | 31.2 | NA | 12/14/12 8:27 AM | 12/17/12 5:39 PM |
| Pyrene | ND | mg/Kg-dry | | 31.2 | NA | 12/14/12 8:27 AM | 12/17/12 5:39 PM |
| Surr: 2-Fluorophenol | 62.3 | %REC | | 54.1-110 | NA | 12/14/12 8:27 AM | 12/17/12 5:39 PM |
| Surr: Phenol-d5 | 74.4 | %REC | | 60.1-110 | NA | 12/14/12 8:27 AM | 12/17/12 5:39 PM |
| Surr: 2,4,6-Tribromophenol | 104 | %REC | | 58.8-128 | NA | 12/14/12 8:27 AM | 12/17/12 5:39 PM |
| Surr: Nitrobenzene-d5 | 94.3 | %REC | | 68.2-123 | NA | 12/14/12 8:27 AM | 12/17/12 5:39 PM |
| Surr: 2-Fluorobiphenyl | 84.2 | %REC | | 68.4-116 | NA | 12/14/12 8:27 AM | 12/17/12 5:39 PM |
| Surr: 4-Terphenyl-d14 | 95.1 | %REC | | 52.2-121 | NA | 12/14/12 8:27 AM | 12/17/12 5:39 PM |

| | | | | | |
|-------------|-----|--|--------------------|---|--|
| Key: | MCL | Maximum Contaminant Level | Qualifiers: | B | Analyte detected in the associated Method Blank |
| | MDL | Minimum Detection Limit | | E | Estimated Value above quantitation range |
| | NA | Not Applicable | | H | Holding times for preparation or analysis exceeded |
| | ND | Not Detected at the PQL or MDL | | S | Spike/Surrogate Recovery exceeds REIC control limits |
| | PQL | Practical Quantitation Limit | | * | Value exceeds MCL or Regulatory Limits |
| | TIC | Tentatively Identified Compound, Estimated Concentration | | | |

CLIENT: VIRGINIA DEPARTMENT OF MINES, MINERALS, A **WorkOrder** 1212762 **Lab ID** 1212762-03A
Client Sample ID: ROARING FORK **DateReceived** 12/7/2012
Project: DMME-HARMON **Collection Date:** 11/28/2012 10:30:00 AM
Site ID: **Matrix:** TISSUE

| Analyses | Result | Units | Qual | PQL | MCL | Prep Date | Date Analyzed |
|---------------------------------------|--------|-----------|-----------------|----------|-----|--------------------|-------------------|
| PERCENT MOISTURE | | | SM2540 B | | | Analyst: SL | |
| Percent Moisture | 91 | wt% | | 0.5 | NA | | 12/14/12 12:00 AM |
| SEMIVOLATILE ORGANIC COMPOUNDS | | | SW8270D | | | Analyst: JD | |
| Acenaphthene | ND | mg/Kg-dry | | 34.7 | NA | 12/14/12 8:27 AM | 12/17/12 6:06 PM |
| Acenaphthylene | ND | mg/Kg-dry | | 34.7 | NA | 12/14/12 8:27 AM | 12/17/12 6:06 PM |
| Anthracene | ND | mg/Kg-dry | | 34.7 | NA | 12/14/12 8:27 AM | 12/17/12 6:06 PM |
| Benzo(a)anthracene | ND | mg/Kg-dry | | 34.7 | NA | 12/14/12 8:27 AM | 12/17/12 6:06 PM |
| Benzo(a)pyrene | ND | mg/Kg-dry | | 34.7 | NA | 12/14/12 8:27 AM | 12/17/12 6:06 PM |
| Benzo(b)fluoranthene | ND | mg/Kg-dry | | 34.7 | NA | 12/14/12 8:27 AM | 12/17/12 6:06 PM |
| Benzo(g,h,i)perylene | ND | mg/Kg-dry | | 34.7 | NA | 12/14/12 8:27 AM | 12/17/12 6:06 PM |
| Benzo(k)fluoranthene | ND | mg/Kg-dry | | 34.7 | NA | 12/14/12 8:27 AM | 12/17/12 6:06 PM |
| Chrysene | ND | mg/Kg-dry | | 34.7 | NA | 12/14/12 8:27 AM | 12/17/12 6:06 PM |
| Dibenzo(a,h)anthracene | ND | mg/Kg-dry | | 34.7 | NA | 12/14/12 8:27 AM | 12/17/12 6:06 PM |
| Fluoranthene | ND | mg/Kg-dry | | 34.7 | NA | 12/14/12 8:27 AM | 12/17/12 6:06 PM |
| Fluorene | ND | mg/Kg-dry | | 34.7 | NA | 12/14/12 8:27 AM | 12/17/12 6:06 PM |
| Indeno(1,2,3-cd)pyrene | ND | mg/Kg-dry | | 34.7 | NA | 12/14/12 8:27 AM | 12/17/12 6:06 PM |
| Naphthalene | ND | mg/Kg-dry | | 34.7 | NA | 12/14/12 8:27 AM | 12/17/12 6:06 PM |
| Phenanthrene | ND | mg/Kg-dry | | 34.7 | NA | 12/14/12 8:27 AM | 12/17/12 6:06 PM |
| Pyrene | ND | mg/Kg-dry | | 34.7 | NA | 12/14/12 8:27 AM | 12/17/12 6:06 PM |
| Surr: 2-Fluorophenol | 82.8 | %REC | | 54.1-110 | NA | 12/14/12 8:27 AM | 12/17/12 6:06 PM |
| Surr: Phenol-d5 | 78.9 | %REC | | 60.1-110 | NA | 12/14/12 8:27 AM | 12/17/12 6:06 PM |
| Surr: 2,4,6-Tribromophenol | 103 | %REC | | 58.8-128 | NA | 12/14/12 8:27 AM | 12/17/12 6:06 PM |
| Surr: Nitrobenzene-d5 | 85.8 | %REC | | 68.2-123 | NA | 12/14/12 8:27 AM | 12/17/12 6:06 PM |
| Surr: 2-Fluorobiphenyl | 80.4 | %REC | | 68.4-116 | NA | 12/14/12 8:27 AM | 12/17/12 6:06 PM |
| Surr: 4-Terphenyl-d14 | 95.1 | %REC | | 52.2-121 | NA | 12/14/12 8:27 AM | 12/17/12 6:06 PM |

Key: MCL Maximum Contaminant Level **Qualifiers:** B Analyte detected in the associated Method Blank
 MDL Minimum Detection Limit E Estimated Value above quantitation range
 NA Not Applicable H Holding times for preparation or analysis exceeded
 ND Not Detected at the PQL or MDL S Spike/Surrogate Recovery exceeds REIC control limits
 PQL Practical Quantitation Limit * Value exceeds MCL or Regulatory Limits Page 4 of 7
 TIC Tentatively Identified Compound, Estimated Concentration

CLIENT: VIRGINIA DEPARTMENT OF MINES, MINERALS, A **WorkOrder** 1212762 **Lab ID** 1212762-04A
Client Sample ID: CALLAHAN CREEK **DateReceived** 12/7/2012
Project: DMME-HARMON **Collection Date:** 11/28/2012 1:30:00 PM
Site ID: **Matrix:** TISSUE

| Analyses | Result | Units | Qual | PQL | MCL | Prep Date | Date Analyzed |
|---------------------------------------|--------|-----------|-----------------|----------|-----|--------------------|-------------------|
| PERCENT MOISTURE | | | SM2540 B | | | Analyst: SL | |
| Percent Moisture | 92 | wt% | | 0.5 | NA | | 12/14/12 12:00 AM |
| SEMIVOLATILE ORGANIC COMPOUNDS | | | SW8270D | | | Analyst: JD | |
| Acenaphthene | ND | mg/Kg-dry | | 4.16 | NA | 12/14/12 8:27 AM | 12/17/12 6:33 PM |
| Acenaphthylene | ND | mg/Kg-dry | | 4.16 | NA | 12/14/12 8:27 AM | 12/17/12 6:33 PM |
| Anthracene | ND | mg/Kg-dry | | 4.16 | NA | 12/14/12 8:27 AM | 12/17/12 6:33 PM |
| Benzo(a)anthracene | ND | mg/Kg-dry | | 4.16 | NA | 12/14/12 8:27 AM | 12/17/12 6:33 PM |
| Benzo(a)pyrene | ND | mg/Kg-dry | | 4.16 | NA | 12/14/12 8:27 AM | 12/17/12 6:33 PM |
| Benzo(b)fluoranthene | ND | mg/Kg-dry | | 4.16 | NA | 12/14/12 8:27 AM | 12/17/12 6:33 PM |
| Benzo(g,h,i)perylene | ND | mg/Kg-dry | | 4.16 | NA | 12/14/12 8:27 AM | 12/17/12 6:33 PM |
| Benzo(k)fluoranthene | ND | mg/Kg-dry | | 4.16 | NA | 12/14/12 8:27 AM | 12/17/12 6:33 PM |
| Chrysene | ND | mg/Kg-dry | | 4.16 | NA | 12/14/12 8:27 AM | 12/17/12 6:33 PM |
| Dibenzo(a,h)anthracene | ND | mg/Kg-dry | | 4.16 | NA | 12/14/12 8:27 AM | 12/17/12 6:33 PM |
| Fluoranthene | ND | mg/Kg-dry | | 4.16 | NA | 12/14/12 8:27 AM | 12/17/12 6:33 PM |
| Fluorene | ND | mg/Kg-dry | | 4.16 | NA | 12/14/12 8:27 AM | 12/17/12 6:33 PM |
| Indeno(1,2,3-cd)pyrene | ND | mg/Kg-dry | | 4.16 | NA | 12/14/12 8:27 AM | 12/17/12 6:33 PM |
| Naphthalene | ND | mg/Kg-dry | | 4.16 | NA | 12/14/12 8:27 AM | 12/17/12 6:33 PM |
| Phenanthrene | ND | mg/Kg-dry | | 4.16 | NA | 12/14/12 8:27 AM | 12/17/12 6:33 PM |
| Pyrene | ND | mg/Kg-dry | | 4.16 | NA | 12/14/12 8:27 AM | 12/17/12 6:33 PM |
| Surr: 2-Fluorophenol | 58.5 | %REC | | 54.1-110 | NA | 12/14/12 8:27 AM | 12/17/12 6:33 PM |
| Surr: Phenol-d5 | 75.8 | %REC | | 60.1-110 | NA | 12/14/12 8:27 AM | 12/17/12 6:33 PM |
| Surr: 2,4,6-Tribromophenol | 93.5 | %REC | | 58.8-128 | NA | 12/14/12 8:27 AM | 12/17/12 6:33 PM |
| Surr: Nitrobenzene-d5 | 95.6 | %REC | | 68.2-123 | NA | 12/14/12 8:27 AM | 12/17/12 6:33 PM |
| Surr: 2-Fluorobiphenyl | 96.2 | %REC | | 68.4-116 | NA | 12/14/12 8:27 AM | 12/17/12 6:33 PM |
| Surr: 4-Terphenyl-d14 | 97.4 | %REC | | 52.2-121 | NA | 12/14/12 8:27 AM | 12/17/12 6:33 PM |

Key: MCL Maximum Contaminant Level **Qualifiers:** B Analyte detected in the associated Method Blank
 MDL Minimum Detection Limit E Estimated Value above quantitation range
 NA Not Applicable H Holding times for preparation or analysis exceeded
 ND Not Detected at the PQL or MDL S Spike/Surrogate Recovery exceeds REIC control limits
 PQL Practical Quantitation Limit * Value exceeds MCL or Regulatory Limits Page 5 of 7
 TIC Tentatively Identified Compound, Estimated Concentration

CLIENT: VIRGINIA DEPARTMENT OF MINES, MINERALS, A **WorkOrder** 1212762 **Lab ID** 1212762-05A
Client Sample ID: POWELL RIVER 1 **DateReceived** 12/7/2012
Project: DMME-HARMON **Collection Date:** 11/28/2012 2:30:00 PM
Site ID: **Matrix:** TISSUE

| Analyses | Result | Units | Qual | PQL | MCL | Prep Date | Date Analyzed |
|---------------------------------------|--------|-----------|-----------------|----------|-----|--------------------|-------------------|
| PERCENT MOISTURE | | | SM2540 B | | | Analyst: SL | |
| Percent Moisture | 87 | wt% | | 0.5 | NA | | 12/14/12 12:00 AM |
| SEMIVOLATILE ORGANIC COMPOUNDS | | | SW8270D | | | Analyst: JD | |
| Acenaphthene | ND | mg/Kg-dry | | 2.56 | NA | 12/14/12 8:27 AM | 12/17/12 7:00 PM |
| Acenaphthylene | ND | mg/Kg-dry | | 2.56 | NA | 12/14/12 8:27 AM | 12/17/12 7:00 PM |
| Anthracene | ND | mg/Kg-dry | | 2.56 | NA | 12/14/12 8:27 AM | 12/17/12 7:00 PM |
| Benzo(a)anthracene | ND | mg/Kg-dry | | 2.56 | NA | 12/14/12 8:27 AM | 12/17/12 7:00 PM |
| Benzo(a)pyrene | ND | mg/Kg-dry | | 2.56 | NA | 12/14/12 8:27 AM | 12/17/12 7:00 PM |
| Benzo(b)fluoranthene | ND | mg/Kg-dry | | 2.56 | NA | 12/14/12 8:27 AM | 12/17/12 7:00 PM |
| Benzo(g,h,i)perylene | ND | mg/Kg-dry | | 2.56 | NA | 12/14/12 8:27 AM | 12/17/12 7:00 PM |
| Benzo(k)fluoranthene | ND | mg/Kg-dry | | 2.56 | NA | 12/14/12 8:27 AM | 12/17/12 7:00 PM |
| Chrysene | ND | mg/Kg-dry | | 2.56 | NA | 12/14/12 8:27 AM | 12/17/12 7:00 PM |
| Dibenzo(a,h)anthracene | ND | mg/Kg-dry | | 2.56 | NA | 12/14/12 8:27 AM | 12/17/12 7:00 PM |
| Fluoranthene | ND | mg/Kg-dry | | 2.56 | NA | 12/14/12 8:27 AM | 12/17/12 7:00 PM |
| Fluorene | ND | mg/Kg-dry | | 2.56 | NA | 12/14/12 8:27 AM | 12/17/12 7:00 PM |
| Indeno(1,2,3-cd)pyrene | ND | mg/Kg-dry | | 2.56 | NA | 12/14/12 8:27 AM | 12/17/12 7:00 PM |
| Naphthalene | ND | mg/Kg-dry | | 2.56 | NA | 12/14/12 8:27 AM | 12/17/12 7:00 PM |
| Phenanthrene | ND | mg/Kg-dry | | 2.56 | NA | 12/14/12 8:27 AM | 12/17/12 7:00 PM |
| Pyrene | ND | mg/Kg-dry | | 2.56 | NA | 12/14/12 8:27 AM | 12/17/12 7:00 PM |
| Surr: 2-Fluorophenol | 61.6 | %REC | | 54.1-110 | NA | 12/14/12 8:27 AM | 12/17/12 7:00 PM |
| Surr: Phenol-d5 | 70.4 | %REC | | 60.1-110 | NA | 12/14/12 8:27 AM | 12/17/12 7:00 PM |
| Surr: 2,4,6-Tribromophenol | 111 | %REC | | 58.8-128 | NA | 12/14/12 8:27 AM | 12/17/12 7:00 PM |
| Surr: Nitrobenzene-d5 | 106 | %REC | | 68.2-123 | NA | 12/14/12 8:27 AM | 12/17/12 7:00 PM |
| Surr: 2-Fluorobiphenyl | 94.0 | %REC | | 68.4-116 | NA | 12/14/12 8:27 AM | 12/17/12 7:00 PM |
| Surr: 4-Terphenyl-d14 | 94.8 | %REC | | 52.2-121 | NA | 12/14/12 8:27 AM | 12/17/12 7:00 PM |

Key: MCL Maximum Contaminant Level **Qualifiers:** B Analyte detected in the associated Method Blank
 MDL Minimum Detection Limit E Estimated Value above quantitation range
 NA Not Applicable H Holding times for preparation or analysis exceeded
 ND Not Detected at the PQL or MDL S Spike/Surrogate Recovery exceeds REIC control limits
 PQL Practical Quantitation Limit * Value exceeds MCL or Regulatory Limits Page 6 of 7
 TIC Tentatively Identified Compound, Estimated Concentration

| | | | | | |
|--------------------------|---|-------------------------|-----------------------|---------------|-------------|
| CLIENT: | VIRGINIA DEPARTMENT OF MINES, MINERALS, A | WorkOrder | 1212762 | Lab ID | 1212762-06A |
| Client Sample ID: | SOUTH FORK POWELL | DateReceived | 12/7/2012 | | |
| Project: | DMME-HARMON | Collection Date: | 11/28/2012 3:00:00 PM | | |
| Site ID: | | Matrix: | TISSUE | | |

| Analyses | Result | Units | Qual | PQL | MCL | Prep Date | Date Analyzed |
|---------------------------------------|--------|-----------|-----------------|----------|-----|--------------------|-------------------|
| PERCENT MOISTURE | | | SM2540 B | | | Analyst: SL | |
| Percent Moisture | 90 | wt% | | 0.5 | NA | | 12/14/12 12:00 AM |
| SEMIVOLATILE ORGANIC COMPOUNDS | | | SW8270D | | | Analyst: JD | |
| Acenaphthene | ND | mg/Kg-dry | | 28.5 | NA | 12/14/12 8:27 AM | 12/17/12 7:27 PM |
| Acenaphthylene | ND | mg/Kg-dry | | 28.5 | NA | 12/14/12 8:27 AM | 12/17/12 7:27 PM |
| Anthracene | ND | mg/Kg-dry | | 28.5 | NA | 12/14/12 8:27 AM | 12/17/12 7:27 PM |
| Benzo(a)anthracene | ND | mg/Kg-dry | | 28.5 | NA | 12/14/12 8:27 AM | 12/17/12 7:27 PM |
| Benzo(a)pyrene | ND | mg/Kg-dry | | 28.5 | NA | 12/14/12 8:27 AM | 12/17/12 7:27 PM |
| Benzo(b)fluoranthene | ND | mg/Kg-dry | | 28.5 | NA | 12/14/12 8:27 AM | 12/17/12 7:27 PM |
| Benzo(g,h,i)perylene | ND | mg/Kg-dry | | 28.5 | NA | 12/14/12 8:27 AM | 12/17/12 7:27 PM |
| Benzo(k)fluoranthene | ND | mg/Kg-dry | | 28.5 | NA | 12/14/12 8:27 AM | 12/17/12 7:27 PM |
| Chrysene | ND | mg/Kg-dry | | 28.5 | NA | 12/14/12 8:27 AM | 12/17/12 7:27 PM |
| Dibenzo(a,h)anthracene | ND | mg/Kg-dry | | 28.5 | NA | 12/14/12 8:27 AM | 12/17/12 7:27 PM |
| Fluoranthene | ND | mg/Kg-dry | | 28.5 | NA | 12/14/12 8:27 AM | 12/17/12 7:27 PM |
| Fluorene | ND | mg/Kg-dry | | 28.5 | NA | 12/14/12 8:27 AM | 12/17/12 7:27 PM |
| Indeno(1,2,3-cd)pyrene | ND | mg/Kg-dry | | 28.5 | NA | 12/14/12 8:27 AM | 12/17/12 7:27 PM |
| Naphthalene | ND | mg/Kg-dry | | 28.5 | NA | 12/14/12 8:27 AM | 12/17/12 7:27 PM |
| Phenanthrene | ND | mg/Kg-dry | | 28.5 | NA | 12/14/12 8:27 AM | 12/17/12 7:27 PM |
| Pyrene | ND | mg/Kg-dry | | 28.5 | NA | 12/14/12 8:27 AM | 12/17/12 7:27 PM |
| Surr: 2-Fluorophenol | 60.4 | %REC | | 54.1-110 | NA | 12/14/12 8:27 AM | 12/17/12 7:27 PM |
| Surr: Phenol-d5 | 85.0 | %REC | | 60.1-110 | NA | 12/14/12 8:27 AM | 12/17/12 7:27 PM |
| Surr: 2,4,6-Tribromophenol | 102 | %REC | | 58.8-128 | NA | 12/14/12 8:27 AM | 12/17/12 7:27 PM |
| Surr: Nitrobenzene-d5 | 112 | %REC | | 68.2-123 | NA | 12/14/12 8:27 AM | 12/17/12 7:27 PM |
| Surr: 2-Fluorobiphenyl | 90.7 | %REC | | 68.4-116 | NA | 12/14/12 8:27 AM | 12/17/12 7:27 PM |
| Surr: 4-Terphenyl-d14 | 90.3 | %REC | | 52.2-121 | NA | 12/14/12 8:27 AM | 12/17/12 7:27 PM |

| | | | | | |
|-------------|-----|--|--------------------|---|--|
| Key: | MCL | Maximum Contaminant Level | Qualifiers: | B | Analyte detected in the associated Method Blank |
| | MDL | Minimum Detection Limit | | E | Estimated Value above quantitation range |
| | NA | Not Applicable | | H | Holding times for preparation or analysis exceeded |
| | ND | Not Detected at the PQL or MDL | | S | Spike/Surrogate Recovery exceeds REIC control limits |
| | PQL | Practical Quantitation Limit | | * | Value exceeds MCL or Regulatory Limits |
| | TIC | Tentatively Identified Compound, Estimated Concentration | | | |



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1557 Commerce Road, Suite 201
Verona, VA 24482
TEL: 540.777.1276

16 Commerce Drive
Westover, WV 26501
TEL: 304.241.5861

Thursday, February 07, 2013

Mr. Joey O'Quinn
VIRGINIA DEPARTMENT OF MINES, MINERALS, AND ENERGY
P.O. DRAWER 900
3405 MTN EMPIRE ROAD
BIG STONE GAP, VA 24219

TEL: (276) 523-8179
FAX:

RE: DMME-HARMON
Work Order #: 1301J88

Dear Mr. Joey O'Quinn:

REI Consultants, Inc. received 1 sample(s) on 1/23/2013 for the analyses presented in the following report.

Sincerely,

Scott Gross
Project Manager



Client: VIRGINIA DEPARTMENT OF MINES, MINERALS, AND ENERGY

Project: DMME-HARMON

The analytical results presented in this report relate only to the samples documented herein. All analyses were performed using documented laboratory SOPs that incorporate appropriate quality control procedures as described in the applicable methods. Any deviation from compliance or method modification is explained below and/or identified within the body of this report by a qualifier footnote which is defined at the bottom of each page.

All sample results are reported on an "as-received" wet weight basis unless otherwise noted.

Results reported for sums of individual parameters, such as Total Trihalomethanes (TTHM) and Total Haloacetic Acids (HAA5), may vary slightly from the sum of the individual parameter results. This apparent anomaly is caused by rounding individual results and summations at reporting, as required by EPA.

Following standard laboratory protocol, sample preservation, such as pH, is verified at time of extraction or analysis based on client requested parameters. Improper preservation is noted on the analytical bench sheet, extraction log, or preservation log and client is notified by close of following business day. All results are reported using preservation compliant samples unless otherwise noted in the analytical report.

The test results in this report meet all NELAP requirements for parameters for which accreditations are required or available. Any exceptions are noted in this report. This report may not be reproduced, except in full, without the written approval of REIC.

In compliance with federal guidelines and standard operating procedures, all reports, including raw data and supporting quality control, will be disposed of after five years unless otherwise arranged by the client via written notification or contract requirement.

If you have any questions please contact the project manager whose name is listed above.

REI Consultants, Inc. - Analytical Report

WO#: 1301J88

Date Reported: 2/7/2013

| | | | |
|--------------------------|---|-------------------------|-----------------------|
| Client: | VIRGINIA DEPARTMENT OF MINES, MINERALS, AND ENERGY | Collection Date: | 1/11/2013 4:00:00 PM |
| Project: | DMME-HARMON | Date Received: | 1/23/2013 10:12:56 AM |
| Lab ID: | 1301J88-01A | Matrix: | TISSUE |
| Client Sample ID: | L. FASCIOLA CONTROL GROUP | Site ID: | |

| Analysis | Result | MDL | PQL | MCL | Qual | Units | Date Analyzed |
|--|--------|-------------------------|--------|----------------|------|--------------------|--------------------|
| PERCENT MOISTURE | | Method: SM2540 B | | | | Analyst: SL | |
| Percent Moisture | 89 | NA | 0.5 | NA | | wt% | 1/23/2013 12:00 AM |
| POLYNUCLEAR AROMATIC HYDROCARBONS (SIM) | | Method: SW8270D | | SW3550B | | Analyst: JD | |
| Anthracene | ND | NA | 0.134 | NA | | mg/Kg-dry | 1/24/2013 1:46 AM |
| Acenaphthene | ND | NA | 0.134 | NA | | mg/Kg-dry | 1/24/2013 1:46 AM |
| Acenaphthylene | ND | NA | 0.134 | NA | | mg/Kg-dry | 1/24/2013 1:46 AM |
| Benzo(a)anthracene | ND | NA | 0.134 | NA | | mg/Kg-dry | 1/24/2013 1:46 AM |
| Benzo(a)pyrene | ND | NA | 0.134 | NA | | mg/Kg-dry | 1/24/2013 1:46 AM |
| Benzo(b)fluoranthene | ND | NA | 0.134 | NA | | mg/Kg-dry | 1/24/2013 1:46 AM |
| Benzo(g,h,i)perylene | ND | NA | 0.134 | NA | | mg/Kg-dry | 1/24/2013 1:46 AM |
| Benzo(k)fluoranthene | ND | NA | 0.134 | NA | | mg/Kg-dry | 1/24/2013 1:46 AM |
| Chrysene | ND | NA | 0.134 | NA | | mg/Kg-dry | 1/24/2013 1:46 AM |
| Dibenzo(a,h)anthracene | ND | NA | 0.134 | NA | | mg/Kg-dry | 1/24/2013 1:46 AM |
| Fluoranthene | ND | NA | 0.134 | NA | | mg/Kg-dry | 1/24/2013 1:46 AM |
| Fluorene | ND | NA | 0.134 | NA | | mg/Kg-dry | 1/24/2013 1:46 AM |
| Indeno(1,2,3-cd)pyrene | ND | NA | 0.134 | NA | | mg/Kg-dry | 1/24/2013 1:46 AM |
| 2-Methylnaphthalene | ND | NA | 0.0678 | NA | | mg/Kg-dry | 2/6/2013 4:19 PM |
| Naphthalene | ND | NA | 0.134 | NA | | mg/Kg-dry | 1/24/2013 1:46 AM |
| Phenanthrene | ND | NA | 0.134 | NA | | mg/Kg-dry | 1/24/2013 1:46 AM |
| Pyrene | ND | NA | 0.134 | NA | | mg/Kg-dry | 1/24/2013 1:46 AM |
| Surr: Nitrobenzene-d5 | 104 | NA | | NA | | %REC | 1/24/2013 1:46 AM |
| Surr: 2-Fluorobiphenyl | 92.2 | NA | | NA | | %REC | 1/24/2013 1:46 AM |
| Surr: 4-Terphenyl-d14 | 93.3 | NA | | NA | | %REC | 1/24/2013 1:46 AM |

Notes:

Insufficient sample was available to prepare and analyze a matrix spiked quality control sample. Accuracy assessment was based on a lab control sample.

380278

CHAIN OF CUSTODY RECORD



Research Environmental & Industrial Consultants, Inc.
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 Roanoke, VA 24019
 540-777-1276

MORGANTOWN Service Center
 16 Commerce Drive
 Westover, WV 26501
 304-241-5861

Client: DMLR (BMI) PO # _____
 Contact Person Joey O'Quinn / Dave Gruber Phone 540-953-2821
 QUOTE # _____ Fax: _____ Email: bmi@biomon.com
 Address 1800 Kraft Dr., Suite 104 City Blacksburg State VA Zip 24060
 Billing Address (if different) DMLR
 City _____ State _____ Zip _____
 Site ID & State _____ Project ID DMME-Harmon Sampler JR (BMI)

SAMPLE LOG & ANALYSIS REQUEST

TURNAROUND TIME RUSH TURNAROUND

NORMAL 5 DAY 3 DAY 2 DAY 1 DAY

*Rush work needs prior laboratory approval and will incur additional charges

ANALYSIS & METHOD REQUESTED

PAH (see Attached)

| SAMPLE ID | No. & Type of Containers | Sampling Date/Time | Matrix | Sample Comp/Grab | X | | | | | | |
|---------------------------|--------------------------|--------------------|--------|------------------|---|--|--|--|--|--|--|
| L. fasciola Control Group | 1/Glass | 1/11/13 1600 | Tissue | Grab | X | | | | | | |
| | | | | | | | | | | | |
| | | | | | | | | | | | |
| | | | | | | | | | | | |
| | | | | | | | | | | | |
| | | | | | | | | | | | |
| | | | | | | | | | | | |
| | | | | | | | | | | | |

- ENTER PRESERVATIVE CODE:**
- 0 None
 - 1 Hydrochloric Acid
 - 2 Nitric Acid
 - 3 Sulfuric Acid
 - 4 Sodium Thiosulfate
 - 5 Sodium Hydroxide
 - 6 Zinc Acetate
 - 7 EDTA
 - 8 Ascorbic Acid

COMMENTS:
 Results to Ed Kirk & Crystal Bennett

All analytical requests are subject to REIC's Standard Terms and Conditions. Temperature at arrival: -1.0 C ICED? Y N

| | | | | | |
|---|--|---|-----------------------------------|---|--|
| 1 <u>Wendy R Boylan</u> <small>Relinquished by (signature)</small> | 22 JAN 13 <small>Date/Time</small> 1400 | 2 _____ <small>Relinquished by (signature)</small> | _____ <small>Date/Time</small> | FAX RESULTS <input type="checkbox"/> | EMAIL RESULTS <input type="checkbox"/> |
| <u>Kathleen Berry</u> <small>Received by (signature)</small> | 1-23-13 <small>Date/Time</small> 9:58a | _____ <small>Received by (signature)</small> | _____ <small>Date/Time</small> | SHIPMENT <input type="checkbox"/> Hand Delivered <input type="checkbox"/> Courier <input checked="" type="checkbox"/> UPS <input type="checkbox"/> FEDEX <input type="checkbox"/> USPS <input type="checkbox"/> OTHER | |

COC-NCR-061312



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TEL: 540.777.1276

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TEL: 304.241.5861

Wednesday, February 27, 2013

Mr. Joey O'Quinn
VIRGINIA DEPARTMENT OF MINES, MINERALS, AND ENERGY
P.O. DRAWER 900
3405 MTN EMPIRE ROAD
BIG STONE GAP, VA 24219

TEL: (276) 523-8179
FAX:

RE: DMME-HARMON
Work Order #: 1302960

Dear Mr. Joey O'Quinn:

REI Consultants, Inc. received 10 sample(s) on 2/12/2013 for the analyses presented in the following report.

Sincerely,

Scott Gross
Project Manager



Client: VIRGINIA DEPARTMENT OF MINES, MINERALS, AND ENERGY

Project: DMME-HARMON

The analytical results presented in this report were produced using documented laboratory SOPs that incorporate appropriate quality control procedures as described in the applicable methods. Verification of required sample preservation (as required) is recorded on associated laboratory logs. Any deviation from compliance or method modification is identified within the body of this report by a qualifier footnote which is defined at the bottom of this page.

All sample results for solid samples are reported on an "as-received" wet weight basis unless otherwise noted.

Results reported for sums of individual parameters, such as TTHM and HAA5, may vary slightly from the sum of the individual parameter results, due to rounding of individual results, as required by EPA.

The test results in this report meet all NELAP (and/or VELAP) requirements for parameters except as noted in this report.

This report may not be reproduced, except in full, without the written approval of REIC.

DEFINITIONS:

MCL: Maximum Contaminant Level

MDL: Method Detection Limit; The lowest concentration of analyte that can be detected by the method in the applicable matrix.

Mg/Kg or mg/L: Units of part per million (PPM) - milligram per Kilogram (weight/weight) or milligram per Liter (weight/volume).

NA: Not Applicable

ND: Not Detected at the PQL or MDL

PQL: Practical Quantitation Limit; The lowest verified limit to which data is quantified without qualifications. Analyte concentrations below PQL are reported either as ND or as a number with a "J" qualifier.

Qual: Qualifier that applies to the analyte reported.

TIC: Tentatively Identified Compound, Estimated Concentration

Ug/Kg or ug/L: Units of part per billion (PPB) - microgram per kilogram (weight/weight) or microgram per liter (weight/volume).

QUALIFIERS:

*: Reported value exceeds required MCL

B: Analyte detected in the associated Method Blank at a concentration > 1/2 the PQL

E: Analyte concentration reported that exceeds the upper calibration standard. Greater uncertainty is associated with this result and data should be considered estimated.

H: Holding time for preparation or analysis has been exceeded.

J: Analyte concentration is reported, and is less than the PQL and greater than or equal to the MDL. The result reported is an estimate.

S: % REC (% recovery) exceeds control limits

CERTIFICATIONS:

Beaver, WV: WVDHHR 00412CM, WVDEP 060, VADCLS 00281, KYDEP 90039, TNDEQ TN02926, NCDWQ 466, PADEP 68-00839, FLDOH (NELAC) E87958, VADCLS(VELAP) 460148

Bioassay (Beaver, WV): WVDEP 060, FLDOH (NELAC) E871055, VADCLS(VELAP) 460149

Roanoke, VA: VADCLS(VELAP) 460150

Verona, VA: VADCLS(VELAP) 460151

Ashland, KY: KYDEP 00094

REI Consultants, Inc. - Analytical Report

WO#: 1302960

Date Reported: 2/27/2013

| | | | |
|--------------------------|---|-------------------------|-----------------------|
| Client: | VIRGINIA DEPARTMENT OF MINES, MINERALS, AND ENERGY | Collection Date: | 1/29/2013 12:00:00 AM |
| Project: | DMME-HARMON | Date Received: | 2/12/2013 10:58:30 AM |
| Lab ID: | 1302960-01A | Matrix: | Biological Tissue |
| Client Sample ID: | ROARING FORK | Site ID: | |

| Analysis | Result | MDL | PQL | MCL | Qual | Units | Date Analyzed |
|--|--------|-------------------------|----------|----------------|------|--------------------|--------------------|
| PERCENT MOISTURE | | Method: SM2540 B | | | | Analyst: SL | |
| Percent Moisture | 90 | NA | 0.5 | NA | | wt% | 2/21/2013 12:00 AM |
| POLYNUCLEAR AROMATIC HYDROCARBONS (SIM) | | Method: SW8270D | | SW3550B | | Analyst: JD | |
| Anthracene | ND | NA | 0.165 | NA | | mg/Kg-dry | 2/26/2013 1:52 AM |
| Acenaphthene | ND | NA | 0.165 | NA | | mg/Kg-dry | 2/26/2013 1:52 AM |
| Acenaphthylene | ND | NA | 0.165 | NA | | mg/Kg-dry | 2/26/2013 1:52 AM |
| Benzo(a)anthracene | ND | NA | 0.165 | NA | | mg/Kg-dry | 2/26/2013 1:52 AM |
| Benzo(a)pyrene | ND | NA | 0.165 | NA | | mg/Kg-dry | 2/26/2013 1:52 AM |
| Benzo(b)fluoranthene | ND | NA | 0.165 | NA | | mg/Kg-dry | 2/26/2013 1:52 AM |
| Benzo(g,h,i)perylene | ND | NA | 0.165 | NA | | mg/Kg-dry | 2/26/2013 1:52 AM |
| Benzo(k)fluoranthene | ND | NA | 0.165 | NA | | mg/Kg-dry | 2/26/2013 1:52 AM |
| Chrysene | ND | NA | 0.165 | NA | | mg/Kg-dry | 2/26/2013 1:52 AM |
| Dibenzo(a,h)anthracene | ND | NA | 0.165 | NA | | mg/Kg-dry | 2/26/2013 1:52 AM |
| Fluoranthene | ND | NA | 0.165 | NA | | mg/Kg-dry | 2/26/2013 1:52 AM |
| Fluorene | ND | NA | 0.165 | NA | | mg/Kg-dry | 2/26/2013 1:52 AM |
| Indeno(1,2,3-cd)pyrene | ND | NA | 0.165 | NA | | mg/Kg-dry | 2/26/2013 1:52 AM |
| 2-Methylnaphthalene | ND | NA | 0.0835 | NA | | mg/Kg-dry | 2/26/2013 3:28 PM |
| Naphthalene | ND | NA | 0.165 | NA | | mg/Kg-dry | 2/26/2013 1:52 AM |
| Phenanthrene | ND | NA | 0.165 | NA | | mg/Kg-dry | 2/26/2013 1:52 AM |
| Pyrene | ND | NA | 0.165 | NA | | mg/Kg-dry | 2/26/2013 1:52 AM |
| Surr: Nitrobenzene-d5 | 96.7 | NA | 60.3-147 | NA | | %REC | 2/26/2013 1:52 AM |
| Surr: 2-Fluorobiphenyl | 88.9 | NA | 75.1-110 | NA | | %REC | 2/26/2013 1:52 AM |
| Surr: 4-Terphenyl-d14 | 85.6 | NA | 52.6-111 | NA | | %REC | 2/26/2013 1:52 AM |

Notes:

Insufficient sample was available to prepare and analyze a matrix spiked quality control sample. Accuracy assessment was based on a lab control sample.

REI Consultants, Inc. - Analytical Report

WO#: 1302960

Date Reported: 2/27/2013

| | | | |
|--------------------------|---|-------------------------|-----------------------|
| Client: | VIRGINIA DEPARTMENT OF MINES, MINERALS, AND ENERGY | Collection Date: | 1/29/2013 12:00:00 AM |
| Project: | DMME-HARMON | Date Received: | 2/12/2013 10:58:30 AM |
| Lab ID: | 1302960-02A | Matrix: | Biological Tissue |
| Client Sample ID: | ROARING FORK 2 | Site ID: | |

| Analysis | Result | MDL | PQL | MCL | Qual | Units | Date Analyzed |
|--|--------|-------------------------|----------|----------------|------|--------------------|--------------------|
| PERCENT MOISTURE | | Method: SM2540 B | | | | Analyst: SL | |
| Percent Moisture | 80 | NA | 0.5 | NA | | wt% | 2/21/2013 12:00 AM |
| POLYNUCLEAR AROMATIC HYDROCARBONS (SIM) | | Method: SW8270D | | SW3550B | | Analyst: JD | |
| Anthracene | ND | NA | 0.108 | NA | | mg/Kg-dry | 2/26/2013 2:17 AM |
| Acenaphthene | ND | NA | 0.108 | NA | | mg/Kg-dry | 2/26/2013 2:17 AM |
| Acenaphthylene | ND | NA | 0.108 | NA | | mg/Kg-dry | 2/26/2013 2:17 AM |
| Benzo(a)anthracene | ND | NA | 0.108 | NA | | mg/Kg-dry | 2/26/2013 2:17 AM |
| Benzo(a)pyrene | ND | NA | 0.108 | NA | | mg/Kg-dry | 2/26/2013 2:17 AM |
| Benzo(b)fluoranthene | ND | NA | 0.108 | NA | | mg/Kg-dry | 2/26/2013 2:17 AM |
| Benzo(g,h,i)perylene | ND | NA | 0.108 | NA | | mg/Kg-dry | 2/26/2013 2:17 AM |
| Benzo(k)fluoranthene | ND | NA | 0.108 | NA | | mg/Kg-dry | 2/26/2013 2:17 AM |
| Chrysene | ND | NA | 0.108 | NA | | mg/Kg-dry | 2/26/2013 2:17 AM |
| Dibenzo(a,h)anthracene | ND | NA | 0.108 | NA | | mg/Kg-dry | 2/26/2013 2:17 AM |
| Fluoranthene | ND | NA | 0.108 | NA | | mg/Kg-dry | 2/26/2013 2:17 AM |
| Fluorene | ND | NA | 0.108 | NA | | mg/Kg-dry | 2/26/2013 2:17 AM |
| Indeno(1,2,3-cd)pyrene | ND | NA | 0.108 | NA | | mg/Kg-dry | 2/26/2013 2:17 AM |
| 2-Methylnaphthalene | ND | NA | 0.0545 | NA | | mg/Kg-dry | 2/26/2013 3:56 PM |
| Naphthalene | ND | NA | 0.108 | NA | | mg/Kg-dry | 2/26/2013 2:17 AM |
| Phenanthrene | ND | NA | 0.108 | NA | | mg/Kg-dry | 2/26/2013 2:17 AM |
| Pyrene | ND | NA | 0.108 | NA | | mg/Kg-dry | 2/26/2013 2:17 AM |
| Surr: Nitrobenzene-d5 | 103 | NA | 60.3-147 | NA | | %REC | 2/26/2013 2:17 AM |
| Surr: 2-Fluorobiphenyl | 88.9 | NA | 75.1-110 | NA | | %REC | 2/26/2013 2:17 AM |
| Surr: 4-Terphenyl-d14 | 87.8 | NA | 52.6-111 | NA | | %REC | 2/26/2013 2:17 AM |

Notes:

Insufficient sample was available to prepare and analyze a matrix spiked quality control sample. Accuracy assessment was based on a lab control sample.

REI Consultants, Inc. - Analytical Report

WO#: 1302960

Date Reported: 2/27/2013

| | | | |
|--------------------------|---|-------------------------|-----------------------|
| Client: | VIRGINIA DEPARTMENT OF MINES, MINERALS, AND ENERGY | Collection Date: | 1/29/2013 12:00:00 AM |
| Project: | DMME-HARMON | Date Received: | 2/12/2013 10:58:30 AM |
| Lab ID: | 1302960-03A | Matrix: | Biological Tissue |
| Client Sample ID: | PR-DS | Site ID: | |

| Analysis | Result | MDL | PQL | MCL | Qual | Units | Date Analyzed |
|--|--------|-------------------------|----------|----------------|------|--------------------|--------------------|
| PERCENT MOISTURE | | Method: SM2540 B | | | | Analyst: SL | |
| Percent Moisture | 90 | NA | 0.5 | NA | | wt% | 2/21/2013 12:00 AM |
| POLYNUCLEAR AROMATIC HYDROCARBONS (SIM) | | Method: SW8270D | | SW3550B | | Analyst: JD | |
| Anthracene | ND | NA | 0.162 | NA | | mg/Kg-dry | 2/26/2013 2:43 AM |
| Acenaphthene | ND | NA | 0.162 | NA | | mg/Kg-dry | 2/26/2013 2:43 AM |
| Acenaphthylene | ND | NA | 0.162 | NA | | mg/Kg-dry | 2/26/2013 2:43 AM |
| Benzo(a)anthracene | ND | NA | 0.162 | NA | | mg/Kg-dry | 2/26/2013 2:43 AM |
| Benzo(a)pyrene | ND | NA | 0.162 | NA | | mg/Kg-dry | 2/26/2013 2:43 AM |
| Benzo(b)fluoranthene | ND | NA | 0.162 | NA | | mg/Kg-dry | 2/26/2013 2:43 AM |
| Benzo(g,h,i)perylene | ND | NA | 0.162 | NA | | mg/Kg-dry | 2/26/2013 2:43 AM |
| Benzo(k)fluoranthene | ND | NA | 0.162 | NA | | mg/Kg-dry | 2/26/2013 2:43 AM |
| Chrysene | ND | NA | 0.162 | NA | | mg/Kg-dry | 2/26/2013 2:43 AM |
| Dibenzo(a,h)anthracene | ND | NA | 0.162 | NA | | mg/Kg-dry | 2/26/2013 2:43 AM |
| Fluoranthene | ND | NA | 0.162 | NA | | mg/Kg-dry | 2/26/2013 2:43 AM |
| Fluorene | ND | NA | 0.162 | NA | | mg/Kg-dry | 2/26/2013 2:43 AM |
| Indeno(1,2,3-cd)pyrene | 0.328 | NA | 0.162 | NA | | mg/Kg-dry | 2/26/2013 2:43 AM |
| 2-Methylnaphthalene | ND | NA | 0.0821 | NA | | mg/Kg-dry | 2/26/2013 4:24 PM |
| Naphthalene | ND | NA | 0.162 | NA | | mg/Kg-dry | 2/26/2013 2:43 AM |
| Phenanthrene | ND | NA | 0.162 | NA | | mg/Kg-dry | 2/26/2013 2:43 AM |
| Pyrene | ND | NA | 0.162 | NA | | mg/Kg-dry | 2/26/2013 2:43 AM |
| Surr: Nitrobenzene-d5 | 98.9 | NA | 60.3-147 | NA | | %REC | 2/26/2013 2:43 AM |
| Surr: 2-Fluorobiphenyl | 86.7 | NA | 75.1-110 | NA | | %REC | 2/26/2013 2:43 AM |
| Surr: 4-Terphenyl-d14 | 88.9 | NA | 52.6-111 | NA | | %REC | 2/26/2013 2:43 AM |

Notes:

Insufficient sample was available to prepare and analyze a matrix spiked quality control sample. Accuracy assessment was based on a lab control sample.

REI Consultants, Inc. - Analytical Report

WO#: 1302960

Date Reported: 2/27/2013

| | | | |
|--------------------------|---|-------------------------|-----------------------|
| Client: | VIRGINIA DEPARTMENT OF MINES, MINERALS, AND ENERGY | Collection Date: | 1/29/2013 12:00:00 AM |
| Project: | DMME-HARMON | Date Received: | 2/12/2013 10:58:30 AM |
| Lab ID: | 1302960-04A | Matrix: | Biological Tissue |
| Client Sample ID: | PR-DS 2 | Site ID: | |

| Analysis | Result | MDL | PQL | MCL | Qual | Units | Date Analyzed |
|--|--------|-------------------------|----------|----------------|------|--------------------|--------------------|
| PERCENT MOISTURE | | Method: SM2540 B | | | | Analyst: SL | |
| Percent Moisture | 86 | NA | 0.5 | NA | | wt% | 2/21/2013 12:00 AM |
| POLYNUCLEAR AROMATIC HYDROCARBONS (SIM) | | Method: SW8270D | | SW3550B | | Analyst: JD | |
| Anthracene | ND | NA | 0.154 | NA | | mg/Kg-dry | 2/26/2013 3:08 AM |
| Acenaphthene | ND | NA | 0.154 | NA | | mg/Kg-dry | 2/26/2013 3:08 AM |
| Acenaphthylene | ND | NA | 0.154 | NA | | mg/Kg-dry | 2/26/2013 3:08 AM |
| Benzo(a)anthracene | ND | NA | 0.154 | NA | | mg/Kg-dry | 2/26/2013 3:08 AM |
| Benzo(a)pyrene | ND | NA | 0.154 | NA | | mg/Kg-dry | 2/26/2013 3:08 AM |
| Benzo(b)fluoranthene | ND | NA | 0.154 | NA | | mg/Kg-dry | 2/26/2013 3:08 AM |
| Benzo(g,h,i)perylene | ND | NA | 0.154 | NA | | mg/Kg-dry | 2/26/2013 3:08 AM |
| Benzo(k)fluoranthene | ND | NA | 0.154 | NA | | mg/Kg-dry | 2/26/2013 3:08 AM |
| Chrysene | ND | NA | 0.154 | NA | | mg/Kg-dry | 2/26/2013 3:08 AM |
| Dibenzo(a,h)anthracene | ND | NA | 0.154 | NA | | mg/Kg-dry | 2/26/2013 3:08 AM |
| Fluoranthene | ND | NA | 0.154 | NA | | mg/Kg-dry | 2/26/2013 3:08 AM |
| Fluorene | ND | NA | 0.154 | NA | | mg/Kg-dry | 2/26/2013 3:08 AM |
| Indeno(1,2,3-cd)pyrene | ND | NA | 0.154 | NA | | mg/Kg-dry | 2/26/2013 3:08 AM |
| 2-Methylnaphthalene | ND | NA | 0.0778 | NA | | mg/Kg-dry | 2/26/2013 4:52 PM |
| Naphthalene | ND | NA | 0.154 | NA | | mg/Kg-dry | 2/26/2013 3:08 AM |
| Phenanthrene | ND | NA | 0.154 | NA | | mg/Kg-dry | 2/26/2013 3:08 AM |
| Pyrene | 0.0776 | NA | 0.154 | NA | J | mg/Kg-dry | 2/26/2013 3:08 AM |
| Surr: Nitrobenzene-d5 | 101 | NA | 60.3-147 | NA | | %REC | 2/26/2013 3:08 AM |
| Surr: 2-Fluorobiphenyl | 88.9 | NA | 75.1-110 | NA | | %REC | 2/26/2013 3:08 AM |
| Surr: 4-Terphenyl-d14 | 87.8 | NA | 52.6-111 | NA | | %REC | 2/26/2013 3:08 AM |

Notes:

Insufficient sample was available to prepare and analyze a matrix spiked quality control sample. Accuracy assessment was based on a lab control sample.

REI Consultants, Inc. - Analytical Report

WO#: 1302960

Date Reported: 2/27/2013

| | | | |
|--------------------------|---|-------------------------|-----------------------|
| Client: | VIRGINIA DEPARTMENT OF MINES, MINERALS, AND ENERGY | Collection Date: | 1/29/2013 12:00:00 AM |
| Project: | DMME-HARMON | Date Received: | 2/12/2013 10:58:30 AM |
| Lab ID: | 1302960-05A | Matrix: | Biological Tissue |
| Client Sample ID: | PR-US | Site ID: | |

| Analysis | Result | MDL | PQL | MCL | Qual | Units | Date Analyzed |
|--|--------|-------------------------|----------|----------------|------|--------------------|--------------------|
| PERCENT MOISTURE | | Method: SM2540 B | | | | Analyst: SL | |
| Percent Moisture | 87 | NA | 0.5 | NA | | wt% | 2/21/2013 12:00 AM |
| POLYNUCLEAR AROMATIC HYDROCARBONS (SIM) | | Method: SW8270D | | SW3550B | | Analyst: JD | |
| Anthracene | ND | NA | 0.127 | NA | | mg/Kg-dry | 2/26/2013 3:34 AM |
| Acenaphthene | ND | NA | 0.127 | NA | | mg/Kg-dry | 2/26/2013 3:34 AM |
| Acenaphthylene | ND | NA | 0.127 | NA | | mg/Kg-dry | 2/26/2013 3:34 AM |
| Benzo(a)anthracene | ND | NA | 0.127 | NA | | mg/Kg-dry | 2/26/2013 3:34 AM |
| Benzo(a)pyrene | 0.128 | NA | 0.127 | NA | | mg/Kg-dry | 2/26/2013 3:34 AM |
| Benzo(b)fluoranthene | ND | NA | 0.127 | NA | | mg/Kg-dry | 2/26/2013 3:34 AM |
| Benzo(g,h,i)perylene | ND | NA | 0.127 | NA | | mg/Kg-dry | 2/26/2013 3:34 AM |
| Benzo(k)fluoranthene | ND | NA | 0.127 | NA | | mg/Kg-dry | 2/26/2013 3:34 AM |
| Chrysene | ND | NA | 0.127 | NA | | mg/Kg-dry | 2/26/2013 3:34 AM |
| Dibenzo(a,h)anthracene | ND | NA | 0.127 | NA | | mg/Kg-dry | 2/26/2013 3:34 AM |
| Fluoranthene | ND | NA | 0.127 | NA | | mg/Kg-dry | 2/26/2013 3:34 AM |
| Fluorene | ND | NA | 0.127 | NA | | mg/Kg-dry | 2/26/2013 3:34 AM |
| Indeno(1,2,3-cd)pyrene | ND | NA | 0.127 | NA | | mg/Kg-dry | 2/26/2013 3:34 AM |
| 2-Methylnaphthalene | ND | NA | 0.0642 | NA | | mg/Kg-dry | 2/26/2013 5:19 PM |
| Naphthalene | ND | NA | 0.127 | NA | | mg/Kg-dry | 2/26/2013 3:34 AM |
| Phenanthrene | ND | NA | 0.127 | NA | | mg/Kg-dry | 2/26/2013 3:34 AM |
| Pyrene | ND | NA | 0.127 | NA | | mg/Kg-dry | 2/26/2013 3:34 AM |
| Surr: Nitrobenzene-d5 | 102 | NA | 60.3-147 | NA | | %REC | 2/26/2013 3:34 AM |
| Surr: 2-Fluorobiphenyl | 90.0 | NA | 75.1-110 | NA | | %REC | 2/26/2013 3:34 AM |
| Surr: 4-Terphenyl-d14 | 87.8 | NA | 52.6-111 | NA | | %REC | 2/26/2013 3:34 AM |

Notes:

Insufficient sample was available to prepare and analyze a matrix spiked quality control sample. Accuracy assessment was based on a lab control sample.

REI Consultants, Inc. - Analytical Report

WO#: 1302960

Date Reported: 2/27/2013

| | | | |
|--------------------------|---|-------------------------|-----------------------|
| Client: | VIRGINIA DEPARTMENT OF MINES, MINERALS, AND ENERGY | Collection Date: | 1/29/2013 12:00:00 AM |
| Project: | DMME-HARMON | Date Received: | 2/12/2013 10:58:30 AM |
| Lab ID: | 1302960-06A | Matrix: | Biological Tissue |
| Client Sample ID: | PR-US 2 | Site ID: | |

| Analysis | Result | MDL | PQL | MCL | Qual | Units | Date Analyzed |
|--|--------|-------------------------|----------|----------------|------|--------------------|--------------------|
| PERCENT MOISTURE | | Method: SM2540 B | | | | Analyst: SL | |
| Percent Moisture | 84 | NA | 0.5 | NA | | wt% | 2/21/2013 12:00 AM |
| POLYNUCLEAR AROMATIC HYDROCARBONS (SIM) | | Method: SW8270D | | SW3550B | | Analyst: JD | |
| Anthracene | ND | NA | 0.167 | NA | | mg/Kg-dry | 2/26/2013 3:59 AM |
| Acenaphthene | ND | NA | 0.167 | NA | | mg/Kg-dry | 2/26/2013 3:59 AM |
| Acenaphthylene | ND | NA | 0.167 | NA | | mg/Kg-dry | 2/26/2013 3:59 AM |
| Benzo(a)anthracene | ND | NA | 0.167 | NA | | mg/Kg-dry | 2/26/2013 3:59 AM |
| Benzo(a)pyrene | ND | NA | 0.167 | NA | | mg/Kg-dry | 2/26/2013 3:59 AM |
| Benzo(b)fluoranthene | ND | NA | 0.167 | NA | | mg/Kg-dry | 2/26/2013 3:59 AM |
| Benzo(g,h,i)perylene | ND | NA | 0.167 | NA | | mg/Kg-dry | 2/26/2013 3:59 AM |
| Benzo(k)fluoranthene | ND | NA | 0.167 | NA | | mg/Kg-dry | 2/26/2013 3:59 AM |
| Chrysene | ND | NA | 0.167 | NA | | mg/Kg-dry | 2/26/2013 3:59 AM |
| Dibenzo(a,h)anthracene | ND | NA | 0.167 | NA | | mg/Kg-dry | 2/26/2013 3:59 AM |
| Fluoranthene | ND | NA | 0.167 | NA | | mg/Kg-dry | 2/26/2013 3:59 AM |
| Fluorene | ND | NA | 0.167 | NA | | mg/Kg-dry | 2/26/2013 3:59 AM |
| Indeno(1,2,3-cd)pyrene | ND | NA | 0.167 | NA | | mg/Kg-dry | 2/26/2013 3:59 AM |
| 2-Methylnaphthalene | ND | NA | 0.0846 | NA | | mg/Kg-dry | 2/26/2013 5:47 PM |
| Naphthalene | ND | NA | 0.167 | NA | | mg/Kg-dry | 2/26/2013 3:59 AM |
| Phenanthrene | ND | NA | 0.167 | NA | | mg/Kg-dry | 2/26/2013 3:59 AM |
| Pyrene | ND | NA | 0.167 | NA | | mg/Kg-dry | 2/26/2013 3:59 AM |
| Surr: Nitrobenzene-d5 | 100 | NA | 60.3-147 | NA | | %REC | 2/26/2013 3:59 AM |
| Surr: 2-Fluorobiphenyl | 86.7 | NA | 75.1-110 | NA | | %REC | 2/26/2013 3:59 AM |
| Surr: 4-Terphenyl-d14 | 85.6 | NA | 52.6-111 | NA | | %REC | 2/26/2013 3:59 AM |

Notes:

Insufficient sample was available to prepare and analyze a matrix spiked quality control sample. Accuracy assessment was based on a lab control sample.

REI Consultants, Inc. - Analytical Report

WO#: 1302960

Date Reported: 2/27/2013

| | | | |
|--------------------------|---|-------------------------|-----------------------|
| Client: | VIRGINIA DEPARTMENT OF MINES, MINERALS, AND ENERGY | Collection Date: | 1/29/2013 12:00:00 AM |
| Project: | DMME-HARMON | Date Received: | 2/12/2013 10:58:30 AM |
| Lab ID: | 1302960-07A | Matrix: | Biological Tissue |
| Client Sample ID: | CALLAHAN CREEK | Site ID: | |

| Analysis | Result | MDL | PQL | MCL | Qual | Units | Date Analyzed |
|--|--------|-------------------------|----------|----------------|------|--------------------|--------------------|
| PERCENT MOISTURE | | Method: SM2540 B | | | | Analyst: SL | |
| Percent Moisture | 89 | NA | 0.5 | NA | | wt% | 2/21/2013 12:00 AM |
| POLYNUCLEAR AROMATIC HYDROCARBONS (SIM) | | Method: SW8270D | | SW3550B | | Analyst: JD | |
| Anthracene | ND | NA | 0.148 | NA | | mg/Kg-dry | 2/26/2013 4:24 AM |
| Acenaphthene | ND | NA | 0.148 | NA | | mg/Kg-dry | 2/26/2013 4:24 AM |
| Acenaphthylene | ND | NA | 0.148 | NA | | mg/Kg-dry | 2/26/2013 4:24 AM |
| Benzo(a)anthracene | ND | NA | 0.148 | NA | | mg/Kg-dry | 2/26/2013 4:24 AM |
| Benzo(a)pyrene | 0.164 | NA | 0.148 | NA | | mg/Kg-dry | 2/26/2013 4:24 AM |
| Benzo(b)fluoranthene | ND | NA | 0.148 | NA | | mg/Kg-dry | 2/26/2013 4:24 AM |
| Benzo(g,h,i)perylene | ND | NA | 0.148 | NA | | mg/Kg-dry | 2/26/2013 4:24 AM |
| Benzo(k)fluoranthene | ND | NA | 0.148 | NA | | mg/Kg-dry | 2/26/2013 4:24 AM |
| Chrysene | ND | NA | 0.148 | NA | | mg/Kg-dry | 2/26/2013 4:24 AM |
| Dibenzo(a,h)anthracene | ND | NA | 0.148 | NA | | mg/Kg-dry | 2/26/2013 4:24 AM |
| Fluoranthene | ND | NA | 0.148 | NA | | mg/Kg-dry | 2/26/2013 4:24 AM |
| Fluorene | ND | NA | 0.148 | NA | | mg/Kg-dry | 2/26/2013 4:24 AM |
| Indeno(1,2,3-cd)pyrene | 0.238 | NA | 0.148 | NA | | mg/Kg-dry | 2/26/2013 4:24 AM |
| 2-Methylnaphthalene | ND | NA | 0.0747 | NA | | mg/Kg-dry | 2/26/2013 6:15 PM |
| Naphthalene | ND | NA | 0.148 | NA | | mg/Kg-dry | 2/26/2013 4:24 AM |
| Phenanthrene | ND | NA | 0.148 | NA | | mg/Kg-dry | 2/26/2013 4:24 AM |
| Pyrene | ND | NA | 0.148 | NA | | mg/Kg-dry | 2/26/2013 4:24 AM |
| Surr: Nitrobenzene-d5 | 97.8 | NA | 60.3-147 | NA | | %REC | 2/26/2013 4:24 AM |
| Surr: 2-Fluorobiphenyl | 87.8 | NA | 75.1-110 | NA | | %REC | 2/26/2013 4:24 AM |
| Surr: 4-Terphenyl-d14 | 85.6 | NA | 52.6-111 | NA | | %REC | 2/26/2013 4:24 AM |

Notes:

Insufficient sample was available to prepare and analyze a matrix spiked quality control sample. Accuracy assessment was based on a lab control sample.

REI Consultants, Inc. - Analytical Report

WO#: 1302960

Date Reported: 2/27/2013

| | | | |
|--------------------------|---|-------------------------|-----------------------|
| Client: | VIRGINIA DEPARTMENT OF MINES, MINERALS, AND ENERGY | Collection Date: | 1/29/2013 12:00:00 AM |
| Project: | DMME-HARMON | Date Received: | 2/12/2013 10:58:30 AM |
| Lab ID: | 1302960-08A | Matrix: | Biological Tissue |
| Client Sample ID: | CALLAHAN CREEK 2 | Site ID: | |

| Analysis | Result | MDL | PQL | MCL | Qual | Units | Date Analyzed |
|--|--------|-------------------------|----------|----------------|------|--------------------|--------------------|
| PERCENT MOISTURE | | Method: SM2540 B | | | | Analyst: SL | |
| Percent Moisture | 90 | NA | 0.5 | NA | | wt% | 2/21/2013 12:00 AM |
| POLYNUCLEAR AROMATIC HYDROCARBONS (SIM) | | Method: SW8270D | | SW3550B | | Analyst: JD | |
| Anthracene | ND | NA | 0.157 | NA | | mg/Kg-dry | 2/26/2013 4:50 AM |
| Acenaphthene | ND | NA | 0.157 | NA | | mg/Kg-dry | 2/26/2013 4:50 AM |
| Acenaphthylene | ND | NA | 0.157 | NA | | mg/Kg-dry | 2/26/2013 4:50 AM |
| Benzo(a)anthracene | ND | NA | 0.157 | NA | | mg/Kg-dry | 2/26/2013 4:50 AM |
| Benzo(a)pyrene | ND | NA | 0.157 | NA | | mg/Kg-dry | 2/26/2013 4:50 AM |
| Benzo(b)fluoranthene | ND | NA | 0.157 | NA | | mg/Kg-dry | 2/26/2013 4:50 AM |
| Benzo(g,h,i)perylene | ND | NA | 0.157 | NA | | mg/Kg-dry | 2/26/2013 4:50 AM |
| Benzo(k)fluoranthene | ND | NA | 0.157 | NA | | mg/Kg-dry | 2/26/2013 4:50 AM |
| Chrysene | ND | NA | 0.157 | NA | | mg/Kg-dry | 2/26/2013 4:50 AM |
| Dibenzo(a,h)anthracene | ND | NA | 0.157 | NA | | mg/Kg-dry | 2/26/2013 4:50 AM |
| Fluoranthene | 0.317 | NA | 0.157 | NA | | mg/Kg-dry | 2/26/2013 4:50 AM |
| Fluorene | ND | NA | 0.157 | NA | | mg/Kg-dry | 2/26/2013 4:50 AM |
| Indeno(1,2,3-cd)pyrene | ND | NA | 0.157 | NA | | mg/Kg-dry | 2/26/2013 4:50 AM |
| 2-Methylnaphthalene | ND | NA | 0.0795 | NA | | mg/Kg-dry | 2/26/2013 6:43 PM |
| Naphthalene | ND | NA | 0.157 | NA | | mg/Kg-dry | 2/26/2013 4:50 AM |
| Phenanthrene | 0.317 | NA | 0.157 | NA | | mg/Kg-dry | 2/26/2013 4:50 AM |
| Pyrene | 0.286 | NA | 0.157 | NA | | mg/Kg-dry | 2/26/2013 4:50 AM |
| Surr: Nitrobenzene-d5 | 108 | NA | 60.3-147 | NA | | %REC | 2/26/2013 4:50 AM |
| Surr: 2-Fluorobiphenyl | 90.0 | NA | 75.1-110 | NA | | %REC | 2/26/2013 4:50 AM |
| Surr: 4-Terphenyl-d14 | 88.9 | NA | 52.6-111 | NA | | %REC | 2/26/2013 4:50 AM |

Notes:

Insufficient sample was available to prepare and analyze a matrix spiked quality control sample. Accuracy assessment was based on a lab control sample.

REI Consultants, Inc. - Analytical Report

WO#: 1302960

Date Reported: 2/27/2013

| | | | |
|--------------------------|---|-------------------------|-----------------------|
| Client: | VIRGINIA DEPARTMENT OF MINES, MINERALS, AND ENERGY | Collection Date: | 1/29/2013 12:00:00 AM |
| Project: | DMME-HARMON | Date Received: | 2/12/2013 10:58:30 AM |
| Lab ID: | 1302960-09A | Matrix: | Biological Tissue |
| Client Sample ID: | SFP | Site ID: | |

| Analysis | Result | MDL | PQL | MCL | Qual | Units | Date Analyzed |
|--|--------|-------------------------|----------|----------------|------|--------------------|--------------------|
| PERCENT MOISTURE | | Method: SM2540 B | | | | Analyst: SL | |
| Percent Moisture | 89 | NA | 0.5 | NA | | wt% | 2/21/2013 12:00 AM |
| POLYNUCLEAR AROMATIC HYDROCARBONS (SIM) | | Method: SW8270D | | SW3550B | | Analyst: JD | |
| Anthracene | ND | NA | 0.145 | NA | | mg/Kg-dry | 2/26/2013 5:15 AM |
| Acenaphthene | ND | NA | 0.145 | NA | | mg/Kg-dry | 2/26/2013 5:15 AM |
| Acenaphthylene | ND | NA | 0.145 | NA | | mg/Kg-dry | 2/26/2013 5:15 AM |
| Benzo(a)anthracene | ND | NA | 0.145 | NA | | mg/Kg-dry | 2/26/2013 5:15 AM |
| Benzo(a)pyrene | 0.161 | NA | 0.145 | NA | | mg/Kg-dry | 2/26/2013 5:15 AM |
| Benzo(b)fluoranthene | ND | NA | 0.145 | NA | | mg/Kg-dry | 2/26/2013 5:15 AM |
| Benzo(g,h,i)perylene | ND | NA | 0.145 | NA | | mg/Kg-dry | 2/26/2013 5:15 AM |
| Benzo(k)fluoranthene | ND | NA | 0.145 | NA | | mg/Kg-dry | 2/26/2013 5:15 AM |
| Chrysene | ND | NA | 0.145 | NA | | mg/Kg-dry | 2/26/2013 5:15 AM |
| Dibenzo(a,h)anthracene | ND | NA | 0.145 | NA | | mg/Kg-dry | 2/26/2013 5:15 AM |
| Fluoranthene | ND | NA | 0.145 | NA | | mg/Kg-dry | 2/26/2013 5:15 AM |
| Fluorene | ND | NA | 0.145 | NA | | mg/Kg-dry | 2/26/2013 5:15 AM |
| Indeno(1,2,3-cd)pyrene | 0.249 | NA | 0.145 | NA | | mg/Kg-dry | 2/26/2013 5:15 AM |
| 2-Methylnaphthalene | ND | NA | 0.0735 | NA | | mg/Kg-dry | 2/26/2013 7:11 PM |
| Naphthalene | ND | NA | 0.145 | NA | | mg/Kg-dry | 2/26/2013 5:15 AM |
| Phenanthrene | ND | NA | 0.145 | NA | | mg/Kg-dry | 2/26/2013 5:15 AM |
| Pyrene | ND | NA | 0.145 | NA | | mg/Kg-dry | 2/26/2013 5:15 AM |
| Surr: Nitrobenzene-d5 | 102 | NA | 60.3-147 | NA | | %REC | 2/26/2013 5:15 AM |
| Surr: 2-Fluorobiphenyl | 92.2 | NA | 75.1-110 | NA | | %REC | 2/26/2013 5:15 AM |
| Surr: 4-Terphenyl-d14 | 86.7 | NA | 52.6-111 | NA | | %REC | 2/26/2013 5:15 AM |

Notes:

Insufficient sample was available to prepare and analyze a matrix spiked quality control sample. Accuracy assessment was based on a lab control sample.

REI Consultants, Inc. - Analytical Report

WO#: 1302960

Date Reported: 2/27/2013

| | | | |
|--------------------------|---|-------------------------|-----------------------|
| Client: | VIRGINIA DEPARTMENT OF MINES, MINERALS, AND ENERGY | Collection Date: | 1/29/2013 12:00:00 AM |
| Project: | DMME-HARMON | Date Received: | 2/12/2013 10:58:30 AM |
| Lab ID: | 1302960-10A | Matrix: | Biological Tissue |
| Client Sample ID: | SFP 2 | Site ID: | |

| Analysis | Result | MDL | PQL | MCL | Qual | Units | Date Analyzed |
|--|--------|-------------------------|----------|----------------|------|--------------------|--------------------|
| PERCENT MOISTURE | | Method: SM2540 B | | | | Analyst: SL | |
| Percent Moisture | 88 | NA | 0.5 | NA | | wt% | 2/21/2013 12:00 AM |
| POLYNUCLEAR AROMATIC HYDROCARBONS (SIM) | | Method: SW8270D | | SW3550B | | Analyst: JD | |
| Anthracene | ND | NA | 0.137 | NA | | mg/Kg-dry | 2/26/2013 5:41 AM |
| Acenaphthene | ND | NA | 0.137 | NA | | mg/Kg-dry | 2/26/2013 5:41 AM |
| Acenaphthylene | ND | NA | 0.137 | NA | | mg/Kg-dry | 2/26/2013 5:41 AM |
| Benzo(a)anthracene | ND | NA | 0.137 | NA | | mg/Kg-dry | 2/26/2013 5:41 AM |
| Benzo(a)pyrene | ND | NA | 0.137 | NA | | mg/Kg-dry | 2/26/2013 5:41 AM |
| Benzo(b)fluoranthene | ND | NA | 0.137 | NA | | mg/Kg-dry | 2/26/2013 5:41 AM |
| Benzo(g,h,i)perylene | ND | NA | 0.137 | NA | | mg/Kg-dry | 2/26/2013 5:41 AM |
| Benzo(k)fluoranthene | ND | NA | 0.137 | NA | | mg/Kg-dry | 2/26/2013 5:41 AM |
| Chrysene | ND | NA | 0.137 | NA | | mg/Kg-dry | 2/26/2013 5:41 AM |
| Dibenzo(a,h)anthracene | ND | NA | 0.137 | NA | | mg/Kg-dry | 2/26/2013 5:41 AM |
| Fluoranthene | ND | NA | 0.137 | NA | | mg/Kg-dry | 2/26/2013 5:41 AM |
| Fluorene | ND | NA | 0.137 | NA | | mg/Kg-dry | 2/26/2013 5:41 AM |
| Indeno(1,2,3-cd)pyrene | ND | NA | 0.137 | NA | | mg/Kg-dry | 2/26/2013 5:41 AM |
| 2-Methylnaphthalene | ND | NA | 0.0696 | NA | | mg/Kg-dry | 2/26/2013 7:38 PM |
| Naphthalene | ND | NA | 0.137 | NA | | mg/Kg-dry | 2/26/2013 5:41 AM |
| Phenanthrene | ND | NA | 0.137 | NA | | mg/Kg-dry | 2/26/2013 5:41 AM |
| Pyrene | ND | NA | 0.137 | NA | | mg/Kg-dry | 2/26/2013 5:41 AM |
| Surr: Nitrobenzene-d5 | 110 | NA | 60.3-147 | NA | | %REC | 2/26/2013 5:41 AM |
| Surr: 2-Fluorobiphenyl | 87.8 | NA | 75.1-110 | NA | | %REC | 2/26/2013 5:41 AM |
| Surr: 4-Terphenyl-d14 | 88.9 | NA | 52.6-111 | NA | | %REC | 2/26/2013 5:41 AM |

Notes:

Insufficient sample was available to prepare and analyze a matrix spiked quality control sample. Accuracy assessment was based on a lab control sample.

380279 1/2

CHAIN OF CUSTODY RECORD



Research Environmental & Industrial Consultants, Inc.

MAIN LABORATORY & CORPORATE HEADQUARTERS:

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800-999-0105 • 304-255-2500 • www.reiclabs.com

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1557 Commerce Rd., Ste 201
Verona, VA 24482
540-248-0183

ROANOKE Service Center
3029-C Peters Creek Rd
Roanoke, VA 24019
540-777-1276

MORGANTOWN Service Center
16 Commerce Drive
Westover, WV 26501
304-241-5861

Client: DMLR (BMI) PO# _____
Contact Person Joey O'Quinn / Dave Gruber Phone 540-953-2821
QUOTE # _____ Fax: _____ Email: bmi@biomon.com
Address 1800 Kraft Dr, Suite 104 City Blacksburg State VA Zip 24060
Billing Address (if different) DMLR
City _____ State _____ Zip _____
Site ID & State _____ Project ID DMME - Harmon Sampler JR/TPT(BMI)

SAMPLE LOG & ANALYSIS REQUEST

TURNAROUND TIME

NORMAL

RUSH TURNAROUND

5 DAY 3 DAY 2 DAY 1 DAY

*Rush work needs prior laboratory approval and will incur additional charges

ANALYSIS & METHOD REQUESTED
PAH (see attached)

| SAMPLE ID | No. & Type of Containers | Sampling Date/Time | Matrix | Sample Comp/Grab | X | | | | | | | | | | |
|------------------|--------------------------|--------------------|--------|------------------|---|--|--|--|--|--|--|--|--|--|--|
| Roaring Fork | 1/Glass | 29 JAN 13 | Tissue | Grab | X | | | | | | | | | | |
| Roaring Fork 2 | 1/Glass | 29 JAN 13 | Tissue | Grab | X | | | | | | | | | | |
| PR-DS | 1/Glass | 29 JAN 13 | Tissue | Grab | X | | | | | | | | | | |
| PR-DS 2 | 1/Glass | 29 JAN 13 | Tissue | Grab | X | | | | | | | | | | |
| PR-US | 1/Glass | 29 JAN 13 | Tissue | Grab | X | | | | | | | | | | |
| PR-US 2 | 1/Glass | 29 JAN 13 | Tissue | Grab | X | | | | | | | | | | |
| Callahan Creek | 1/Glass | 29 JAN 13 | Tissue | Grab | X | | | | | | | | | | |
| Callahan Creek 2 | 1/Glass | 29 JAN 13 | Tissue | Grab | X | | | | | | | | | | |
| SFP | 1/Glass | 29 JAN 13 | Tissue | Grab | X | | | | | | | | | | |

- ENTER PRESERVATIVE CODE:**
- 0 None
 - 1 Hydrochloric Acid
 - 2 Nitric Acid
 - 3 Sulfuric Acid
 - 4 Sodium Thiosulfate
 - 5 Sodium Hydroxide
 - 6 Zinc Acetate
 - 7 EDTA
 - 8 Ascorbic Acid

COMMENTS:
results to Ed Kirk & Crystal Bennett
#2 Denotes Benthics (Others are mussels)

All analytical requests are subject to REIC's Standard Terms and Conditions.

Temperature at arrival: _____ °C ICED? Y___ N___

| | | | | | |
|---|-------------------------------|--|--------------------|--|--|
| 1 <u>Wendie R Boylan</u> Relinquished by (signature) | <u>11 FEB 13</u> Date/Time | 2 _____ Relinquished by (signature) | _____ Date/Time | FAX RESULTS <input type="checkbox"/> | EMAIL RESULTS <input type="checkbox"/> |
| _____ Received by (signature) | _____ Date/Time | _____ Received by (signature) | _____ Date/Time | SHIPMENT <input type="checkbox"/> Hand Delivered <input type="checkbox"/> Courier <input type="checkbox"/> UPS <input type="checkbox"/> FEDEX <input type="checkbox"/> USPS <input type="checkbox"/> OTHER | |

COC-NCR-061312



380280 2/2

CHAIN OF CUSTODY RECORD



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3029-C Peters Creek Rd
Roanoke, VA 24019
540-777-1276

MORGANTOWN Service Center
16 Commerce Drive
Westover, WV 26501
304-241-5861

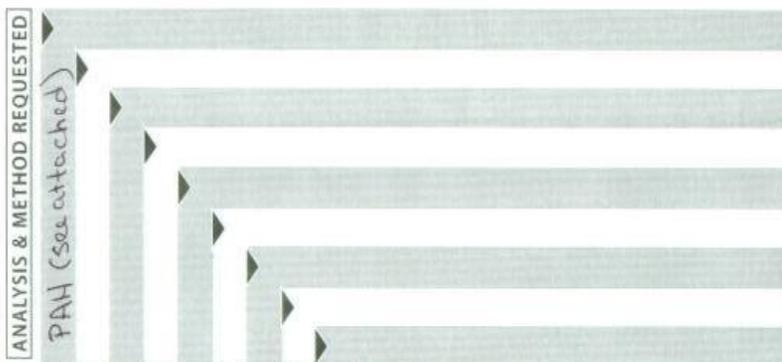
Client: DMLR PO # _____
Contact Person: Joey O'Quinn / Dave Gruber Phone: 540-953-2821
QUOTE # _____ Fax: _____ Email: bmi@biomon.com
Address: 1800 Kraft Dr, Suite 104 City: Blacksburg State: VA Zip: 24060
Billing Address (if different): DMLR
City: _____ State: _____ Zip: _____
Site ID & State: _____ Project ID: DMME-Harmon Sampler: JR/TPT (BME)

SAMPLE LOG & ANALYSIS REQUEST

TURNAROUND TIME NORMAL RUSH TURNAROUND

5 DAY 3 DAY 2 DAY 1 DAY

*Rush work needs prior laboratory approval and will incur additional charges



| SAMPLE ID | No. & Type of Containers | Sampling Date/Time | Matrix | Sample Comp/Grab | | | | | | |
|-----------|--------------------------|--------------------|--------|------------------|---|--|--|--|--|--|
| SFP 2 | 1 Glass | 29 JAN 13 | Tissue | Grab | X | | | | | |
| | | | | | | | | | | |
| | | | | | | | | | | |
| | | | | | | | | | | |
| | | | | | | | | | | |
| | | | | | | | | | | |
| | | | | | | | | | | |

- ENTER PRESERVATIVE CODE:**
- 0 None
 - 1 Hydrochloric Acid
 - 2 Nitric Acid
 - 3 Sulfuric Acid
 - 4 Sodium Thiosulfate
 - 5 Sodium Hydroxide
 - 6 Zinc Acetate
 - 7 EDTA
 - 8 Ascorbic Acid

COMMENTS:
results to Ed Kirk &
Crystal Bennett
#2 Denotes Benthics
(Others are mussels)

All analytical requests are subject to REIC's Standard Terms and Conditions. Temperature at arrival: °C ICED? Y ___ N ___

| | | | | | |
|--|---|--|-----------------|--|--|
| 1 <u>Wendy R. Boyle</u> Relinquished by (signature) | <u>11 FEB 13</u> Date/Time <u>11:5</u> | 2 _____ Relinquished by (signature) | _____ Date/Time | FAX RESULTS <input type="checkbox"/> | EMAIL RESULTS <input type="checkbox"/> |
| _____ Received by (signature) | _____ Date/Time | _____ Received by (signature) | _____ Date/Time | SHIPMENT <input type="checkbox"/> Hand Delivered <input type="checkbox"/> Courier <input type="checkbox"/> UPS <input type="checkbox"/> FEDEX <input type="checkbox"/> USPS <input type="checkbox"/> OTHER | |

COC-NCR-061312



DBPix Evaluation

Laboratory Analyses

All laboratory analyses should be performed by a VELAP certified laboratory. In all cases, samples should be kept below 4°C during delivery to the lab. Table 4 indicates the methods to be used for analysis and the detection limits needed. PAH analysis will include quantification of the following components:

- ✓ Acenaphthene
- ✓ Acenaphthylene
- ✓ Anthracene
- ✓ Benzo(a)anthracene
- ✓ Benzo(a)pyrene
- ✓ Benzo(b)fluoranthene
- ✓ Benzo(g,h,i)perylene
- ✓ Benzo(k)fluoranthene
- ✓ Chrysene
- ✓ Dibenzo(a,h)anthracene
- ✓ Fluoranthene
- ✓ Fluorene
- ✓ Indeno(1,2,3-cd)pyrene
- ✓ Naphthalene
- ✓ Phenanthrene
- ✓ Pyrene

2-methyl naphthalene

Table 4. Methods and approximate detection limits for parameters measured in the laboratory.

| Parameter | Approximate Detection Limit(s) | Method |
|--------------|--|-------------------------|
| Conductivity | Lower = 10 umhos/cm Upper = 10,000 umhos/cm | Standard Methods 2510 |
| TDS | Lower = 10mg/L | Standard Methods 2540 C |
| TSS | Lower = 1mg/L | Standard Methods 2540 D |
| PAHs | Lower = 0.33 ppm | Standard Methods 8270 |

2-methyl naphthalene Lower = 83 ppb